Crystal structure of $C_{11}H_{19}NO_4$ — ban0832

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Abstract

The crystal structure of $C_{11}H_{19}NO_4$ is reported.

Comment

The crystallographic asymmetric unit consists of one $C_{11}H_{19}NO_4$ molecule.

Experimental

The compound was prepared by AL. The sample ID is AlinfragB.

Refinement

The compound is enantiometrically pure but the anomolous dispersion terms are very low for all elements in the structure and so the absolute configuration can not be determined in this experiment. Consequently Friedel-pair reflections have been averaged and the Flack parameter has not been refined. The absolute configuration of the molecule has been assigned on the basis of the synthetic precursors.

Refinement of a totally ordered model gave large displacement parameters for C5 and C6 and a very short C5═C6 distance. It therefore appears that there is disorder of the —CH_2—CH═CH_2 unit. Consequently atoms C4, C5 and C6 were each split over two sites and connectivities established to describe two alternative alignments of the chain. Restraints were imposed so equivalent bond lengths should tend to their respective mean values and so displacement parameters for adjacent atom sites should tend to be similar. The relative occupancies of the two images were refined. Metric parameters in this section of the structure have low precision.

H atoms were included at geometrically determined positions and ride on the atoms to which they are respectively attached. Difference-fourier maps suggested that the H atoms on C14 were disordered so the major sites were assigned occupancies of 0.7 and sites between them were assigned occupancies of 0.3.

The largest peaks in the final difference electron density map are located along C—C bonds or within the disorder.

Computing details

Data collection: *COLLECT* (Nonius, 1997-2001).; cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP*II (Johnson 1976) in *TEXSAN* (MSC, 1992-1997); software used to prepare material for publication: *CRYSTALS* (Betteridge *et al.*, 2003).

(ban0832)

Crystal data

$C_{11}H_{19}NO_4$	V = 1265.32 (5) Å^3
M_r = 229.28	Z = 4
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$
a = 8.0952 (2) Å	μ = 0.09 mm^{-1}
b = 9.7080 (2) Å	T = 200 K
c = 16.1006 (4) Å	0.45 × 0.32 × 0.21 mm

Data collection

Area diffractometer	1682 independent reflections
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	1460 reflections with $I > 2.0\sigma(I)$
T_{min} = 0.969, T_{max} = 0.983	R_{int} = 0.034
18889 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030	H-atom parameters constrained
$wR(F^2)$ = 0.084	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
S = 0.95	$\Delta\rho_{min}$ = −0.13 e Å^{-3}
1682 reflections	Absolute structure: The enantiomer has been assigned by reference to an unchanging chiral centre in the synthetic procedure.
173 parameters	
60 restraints	

Table 1
Selected geometric parameters (Å, °)

O7—C1	1.2229 (19)	C2—C3	1.537 (2)
O8—C2	1.4189 (19)	C3—C41	1.509 (5)
O8—C9	1.4459 (18)	C3—C42	1.532 (8)
O10—C3	1.4290 (17)	C9—C11	1.509 (2)
O10—C9	1.4252 (18)	C9—C12	1.506 (2)

O15—N13	1.3998 (19)	C41—C51	1.498 (6)
O15—C16	1.442 (2)	C42—C52	1.494 (8)
N13—C1	1.343 (2)	C51—C61	1.296 (5)
N13—C14	1.445 (2)	C52—C62	1.300 (7)
C1—C2	1.529 (2)		
C2—O8—C9	109.46 (11)	O10—C3—C41	113.9 (3)
C3—O10—C9	106.60 (10)	C2—C3—C42	122.1 (4)
N13—O15—C16	109.60 (12)	O10—C3—C42	103.1 (6)
O15—N13—C1	119.60 (13)	O8—C9—O10	105.36 (11)
O15—N13—C14	115.29 (15)	O8—C9—C11	108.33 (12)
C1—N13—C14	124.63 (15)	O10—C9—C11	111.53 (13)
N13—C1—O7	120.75 (15)	O8—C9—C12	109.54 (13)
N13—C1—C2	117.11 (13)	O10—C9—C12	108.39 (12)
O7—C1—C2	122.14 (15)	C11—C9—C12	113.36 (14)
C1—C2—O8	109.46 (13)	C3—C41—C51	108.9 (4)
C1—C2—C3	112.61 (12)	C3—C42—C52	120.6 (7)
O8—C2—C3	103.15 (12)	C41—C51—C61	124.5 (5)
C2—C3—O10	101.71 (11)	C42—C52—C62	128.3 (8)
C2—C3—C41	113.7 (2)		

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supplementary materials

Crystal structure of $C_{11}H_{19}NO_4$ — ban0832

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Crystal data

$C_{11}H_{19}NO_4$	$D_x = 1.203$ Mg m^{-3}
$M_r = 229.28$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 11432 reflections
$a = 8.0952$ (2) Å	$\theta = 3$–27º
$b = 9.7080$ (2) Å	$\mu = 0.09$ mm^{-1}
$c = 16.1006$ (4) Å	$T = 200$ K
$V = 1265.32$ (5) Å^3	Block, colourless
$Z = 4$	0.45 × 0.32 × 0.21 mm
$F_{000} = 496$	

Data collection

Area diffractometer	1460 reflections with $I > 2.0\sigma(I)$
Monochromator: graphite	$R_{int} = 0.034$
$T = 200$ K	$\theta_{max} = 27.5$º
φ and ω scans with CCD	$\theta_{min} = 3.3$º
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	$h = -10 \rightarrow 10$
$T_{min} = 0.969$, $T_{max} = 0.983$	$k = -12 \rightarrow 12$
18889 measured reflections	$l = -20 \rightarrow 20$
1682 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.030$	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.05P)^2 + 0.0P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$wR(F^2) = 0.084$	$(\Delta/\sigma)_{max} = 0.006$
$S = 0.95$	$\Delta\rho_{max} = 0.14$ e Å^{-3}
1682 reflections	$\Delta\rho_{min} = -0.13$ e Å^{-3}
173 parameters	Extinction correction: None

60 restraints

Absolute structure: The enantiomer has been assigned by reference to an unchanging chiral centre in the synthetic procedure.

Primary atom site location: structure-invariant direct methods

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O7	0.52514 (16)	0.11862 (11)	0.11567 (7)	0.0506	
O8	0.33816 (14)	0.33013 (11)	0.18203 (7)	0.0460	
O10	0.40623 (12)	0.20606 (10)	0.29755 (6)	0.0373	
O15	0.81155 (15)	0.38789 (12)	0.11707 (7)	0.0532	
N13	0.7330 (2)	0.26523 (15)	0.09404 (8)	0.0518	
C1	0.5901 (2)	0.22964 (15)	0.13056 (8)	0.0395	
C2	0.51267 (18)	0.33433 (15)	0.18990 (10)	0.0392	
C3	0.54097 (17)	0.29742 (15)	0.28166 (9)	0.0378	
C9	0.26833 (19)	0.25832 (16)	0.25244 (9)	0.0397	
C11	0.1706 (2)	0.36031 (17)	0.30337 (12)	0.0517	
C12	0.1668 (2)	0.13825 (18)	0.22238 (12)	0.0551	
C14	0.8264 (3)	0.17742 (19)	0.03882 (11)	0.0612	
C16	0.7868 (3)	0.49033 (19)	0.05344 (11)	0.0653	
C41	0.7084 (8)	0.2353 (9)	0.2988 (4)	0.0364	0.610 (7)
C42	0.6915 (13)	0.2145 (15)	0.3105 (6)	0.0440	0.390 (7)
C51	0.7381 (5)	0.2358 (4)	0.3906 (2)	0.0482	0.610 (7)
C52	0.7083 (7)	0.1712 (7)	0.3992 (4)	0.0503	0.390 (7)
C61	0.7868 (5)	0.1298 (5)	0.4330 (2)	0.0650	0.610 (7)
C62	0.8128 (7)	0.2154 (8)	0.4543 (3)	0.0625	0.390 (7)
H21	0.5549	0.4290	0.1779	0.0471*	
H31	0.5273	0.3822	0.3162	0.0454*	0.61
H32	0.5292	0.3820	0.3167	0.0454*	0.39
H111	0.1213	0.3124	0.3524	0.0620*	
H112	0.2454	0.4356	0.3229	0.0620*	
H113	0.0805	0.4005	0.2685	0.0620*	
H121	0.1186	0.0887	0.2711	0.0661*	
H122	0.2388	0.0738	0.1902	0.0661*	
H123	0.0757	0.1726	0.1859	0.0661*	
H141	0.7607	0.0929	0.0260	0.0735*	0.7
H142	0.9326	0.1505	0.0661	0.0735*	0.7
H143	0.8506	0.2282	−0.0138	0.0735*	0.7
H144	0.9278	0.2271	0.0198	0.0735*	0.3
H145	0.8591	0.0914	0.0688	0.0735*	0.3
H146	0.7570	0.1530	−0.0104	0.0735*	0.3
H161	0.8429	0.5779	0.0700	0.0783*	
H162	0.8344	0.4566	−0.0001	0.0783*	
H163	0.6658	0.5076	0.0463	0.0783*	
H411	0.7957	0.2909	0.2704	0.0438*	0.61
H412	0.7115	0.1385	0.2776	0.0438*	0.61
H421	0.7910	0.2714	0.2973	0.0528*	0.39

H422	0.6934	0.1283	0.2765	0.0528*	0.39
H511	0.7190	0.3239	0.4213	0.0579*	0.61
H521	0.6289	0.0991	0.4185	0.0603*	0.39
H611	0.8041	0.1378	0.4943	0.0781*	0.61
H612	0.8072	0.0400	0.4044	0.0781*	0.61
H621	0.8106	0.1772	0.5120	0.0748*	0.39
H622	0.8956	0.2875	0.4390	0.0748*	0.39

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O7	0.0687 (8)	0.0349 (5)	0.0481 (6)	−0.0039 (6)	−0.0046 (6)	−0.0016 (5)
O8	0.0418 (6)	0.0453 (6)	0.0508 (6)	0.0041 (5)	−0.0007 (5)	0.0161 (5)
O10	0.0320 (5)	0.0372 (5)	0.0427 (5)	−0.0012 (4)	−0.0034 (4)	0.0056 (4)
O15	0.0562 (7)	0.0538 (7)	0.0496 (6)	−0.0124 (6)	0.0087 (6)	−0.0009 (5)
N13	0.0643 (10)	0.0409 (7)	0.0502 (7)	−0.0037 (7)	0.0188 (7)	−0.0040 (6)
C1	0.0504 (9)	0.0333 (7)	0.0348 (7)	0.0009 (7)	−0.0024 (6)	0.0051 (6)
C2	0.0385 (7)	0.0292 (6)	0.0499 (8)	−0.0022 (6)	0.0054 (7)	0.0037 (6)
C3	0.0346 (7)	0.0347 (7)	0.0441 (7)	−0.0053 (6)	0.0022 (6)	−0.0060 (6)
C9	0.0342 (7)	0.0352 (7)	0.0497 (8)	−0.0021 (6)	−0.0048 (6)	0.0098 (6)
C11	0.0389 (8)	0.0457 (8)	0.0704 (11)	0.0025 (7)	0.0066 (8)	0.0066 (8)
C12	0.0474 (9)	0.0490 (9)	0.0691 (11)	−0.0109 (8)	−0.0156 (9)	0.0043 (9)
C14	0.0817 (13)	0.0560 (10)	0.0460 (9)	0.0116 (11)	0.0179 (9)	0.0002 (8)
C16	0.0860 (15)	0.0492 (10)	0.0605 (10)	−0.0130 (10)	0.0210 (11)	0.0068 (8)
C41	0.031 (2)	0.045 (2)	0.0327 (17)	−0.0063 (17)	0.0001 (14)	−0.0037 (14)
C42	0.031 (3)	0.051 (4)	0.050 (4)	−0.004 (3)	0.007 (2)	−0.003 (3)
C51	0.0484 (18)	0.054 (2)	0.0419 (15)	−0.0032 (17)	−0.0088 (13)	−0.0037 (15)
C52	0.045 (3)	0.051 (3)	0.055 (3)	−0.004 (2)	−0.012 (2)	0.010 (3)
C61	0.073 (2)	0.071 (2)	0.0503 (19)	−0.010 (2)	−0.0108 (18)	0.0124 (18)
C62	0.059 (3)	0.077 (4)	0.052 (3)	−0.008 (3)	−0.004 (2)	0.001 (3)

Geometric parameters (Å, °)

O7—C1	1.2229 (19)	C14—H141	1.000
O8—C2	1.4189 (19)	C14—H142	1.000
O8—C9	1.4459 (18)	C14—H143	1.000
O10—C3	1.4290 (17)	C14—H144	1.000
O10—C9	1.4252 (18)	C14—H145	1.000
O15—N13	1.3998 (19)	C14—H146	1.000
O15—C16	1.442 (2)	C16—H161	1.000
N13—C1	1.343 (2)	C16—H162	1.000
N13—C14	1.445 (2)	C16—H163	1.000
C1—C2	1.529 (2)	C41—C51	1.498 (6)
C2—C3	1.537 (2)	C41—H411	1.000
C2—H21	1.000	C41—H412	1.000
C3—C41	1.509 (5)	C42—C52	1.494 (8)
C3—C42	1.532 (8)	C42—H421	1.000
C3—H31	1.000	C42—H422	1.000
C3—H32	1.000	C51—C61	1.296 (5)

C9—C11	1.509 (2)	C51—H511	1.000
C9—C12	1.506 (2)	C52—C62	1.300 (7)
C11—H111	1.000	C52—H521	1.000
C11—H112	1.000	C61—H611	1.000
C11—H113	1.000	C61—H612	1.000
C12—H121	1.000	C62—H621	1.000
C12—H122	1.000	C62—H622	1.000
C12—H123	1.000		
O7···C16^{i}	3.501 (2)	O10···C16^{ii}	3.547 (2)
O7···C14^{i}	3.563 (2)	O15···C12^{iii}	3.553 (2)
O7···C3^{ii}	3.570 (2)	C1···C14^{i}	3.579 (2)
O8···C14^{i}	3.558 (2)	C14···C61^{iv}	3.555 (5)
O8···C52^{iii}	3.580 (7)	C16···C62^{v}	3.370 (7)
O8···C61^{iii}	3.594 (4)		
C2—O8—C9	109.46 (11)	H122—C12—H123	109.5
C3—O10—C9	106.60 (10)	N13—C14—H141	109.5
N13—O15—C16	109.60 (12)	N13—C14—H142	109.5
O15—N13—C1	119.60 (13)	H141—C14—H142	109.5
O15—N13—C14	115.29 (15)	N13—C14—H143	109.5
C1—N13—C14	124.63 (15)	H141—C14—H143	109.5
N13—C1—O7	120.75 (15)	H142—C14—H143	109.5
N13—C1—C2	117.11 (13)	N13—C14—H144	109.5
O7—C1—C2	122.14 (15)	N13—C14—H145	109.5
C1—C2—O8	109.46 (13)	H144—C14—H145	109.5
C1—C2—C3	112.61 (12)	N13—C14—H146	109.5
O8—C2—C3	103.15 (12)	H144—C14—H146	109.5
C1—C2—H21	110.5	H145—C14—H146	109.5
O8—C2—H21	110.5	O15—C16—H161	109.5
C3—C2—H21	110.5	O15—C16—H162	109.5
C2—C3—O10	101.71 (11)	H161—C16—H162	109.5
C2—C3—C41	113.7 (2)	O15—C16—H163	109.5
O10—C3—C41	113.9 (3)	H161—C16—H163	109.5
C2—C3—C42	122.1 (4)	H162—C16—H163	109.5
O10—C3—C42	103.1 (6)	C3—C41—C51	108.9 (4)
C2—C3—H31	109.1	C3—C41—H411	109.6
O10—C3—H31	109.1	C51—C41—H411	109.6
C41—C3—H31	109.1	C3—C41—H412	109.6
C2—C3—H32	109.6	C51—C41—H412	109.6
O10—C3—H32	109.6	H411—C41—H412	109.5
C42—C3—H32	109.6	C3—C42—C52	120.6 (7)
O8—C9—O10	105.36 (11)	C3—C42—H421	106.6
O8—C9—C11	108.33 (12)	C52—C42—H421	106.6
O10—C9—C11	111.53 (13)	C3—C42—H422	106.6
O8—C9—C12	109.54 (13)	C52—C42—H422	106.7
O10—C9—C12	108.39 (12)	H421—C42—H422	109.5
C11—C9—C12	113.36 (14)	C41—C51—C61	124.5 (5)
C9—C11—H111	109.5	C41—C51—H511	117.8

C9—C11—H112	109.5	C61—C51—H511	117.8
H111—C11—H112	109.5	C42—C52—C62	128.3 (8)
C9—C11—H113	109.5	C42—C52—H521	115.8
H111—C11—H113	109.5	C62—C52—H521	115.9
H112—C11—H113	109.5	C51—C61—H611	120.0
C9—C12—H121	109.5	C51—C61—H612	120.0
C9—C12—H122	109.5	H611—C61—H612	120.0
H121—C12—H122	109.5	C52—C62—H621	120.0
C9—C12—H123	109.5	C52—C62—H622	120.0
H121—C12—H123	109.5	H621—C62—H622	120.0
O7—C1—N13—O15	−174.9 (1)	C1—C2—C3—C41	37.1 (4)
O7—C1—N13—C14	−3.2 (2)	C1—C2—C3—C42	28.0 (6)
O7—C1—C2—O8	−35.7 (2)	C2—O8—C9—C11	113.0 (1)
O7—C1—C2—C3	78.4 (2)	C2—O8—C9—C12	−122.9 (1)
O8—C2—C1—N13	143.4 (1)	C2—C1—N13—C14	177.6 (1)
O8—C2—C3—O10	32.1 (1)	C2—C3—O10—C9	−37.2 (1)
O8—C2—C3—C41	155.0 (4)	C2—C3—C41—C51	166.3 (4)
O8—C2—C3—C42	145.9 (6)	C2—C3—C42—C52	−175.2 (7)
O8—C9—O10—C3	28.3 (1)	C3—O10—C9—C11	−89.1 (1)
O10—C3—C2—C1	−85.8 (1)	C3—O10—C9—C12	145.5 (1)
O10—C3—C41—C51	−77.8 (5)	C3—C2—O8—C9	−15.8 (1)
O10—C3—C42—C52	−62 (1)	C3—C41—C51—C61	130.2 (5)
O10—C9—O8—C2	−6.5 (1)	C3—C42—C52—C62	−110 (1)
O15—N13—C1—C2	5.9 (2)	C9—O10—C3—C41	−160.0 (3)
N13—C1—C2—C3	−102.5 (1)	C9—O10—C3—C42	−164.5 (4)
C1—N13—O15—C16	−104.4 (2)	C14—N13—O15—C16	83.2 (2)
C1—C2—O8—C9	104.2 (1)		

Symmetry codes: (i) *x*−1/2, −*y*+1/2, −*z*; (ii) −*x*+1, *y*−1/2, −*z*+1/2; (iii) −*x*+1, *y*+1/2, −*z*+1/2; (iv) −*x*+3/2, −*y*, *z*−1/2; (v) −*x*+3/2, −*y*+1, *z*−1/2.

Crystal structure of $C_{19}H_{24}O_7.1.111CHCl_3$ — ban0910

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Abstract

The crystal structure of $C_{19}H_{24}O_7.1.111CHCl_3$ is reported.

Comment

The crystallographic asymmetric unit consists of one $C_{19}H_{24}O_7$ molecule, one localized but disordered chloroform molecule of crystallization and a chain of disordered chloroform molecules of crystallization running through the cell.

Experimental

The compound was prepared by AL. The sample ID is AL-589.

Refinement

The compound is enantiometrically pure. The absolute structure of the crystal has been determined by refinement of the Flack parameter and this establishes the absolute configuration of the molecule. It is in agreement with the configuration expected on the basis of the synthetic precursors.

There are two regions of disordered solvate molecules in the structure. One is localized, and is centred at about (0.10,0.07,0.17). There are three images of chloroform consisting of sites Cl51, Cl52, Cl53 and C53; Cl51, Cl52, Cl54 and C54; and Cl61, Cl62, Cl63 and C61. Some sites are common to two of the images. The relative occupancies of the sites were refined. Restraints were imposed on bonded distances and angles and on displacement parameters as appropriate.

There is also appears to be a chain of disordered atom sites running through the structure down the 3-fold screw axis 1/3,1/3,*z*. The sites are about 1.8Å apart suggesting they might arise from chloroform molecules which are not maintaining a regular stacking. The model presented here has C71 and Cl71 on a common site so a typical molecule will be C71, Cl71(2/3 − *y*,1/3 + *x*-*y*,1/3 + *z*), Cl71(2/3 − *x* + *y*,1/3 − *x*,1/3 + *z*) and Cl72, giving a rough tetrahedron about C71. The next chloroform image along the chain would be C71(2/3 − *x* + *y*,1/3 − *x*,1/3 + *z*), Cl71, Cl71(?) and Cl72(2/3 − *x* + *y*,1/3 − *x*,1/3 + *z*) and so on. The relative occupancies have been chosen to support this theory though their absolute values are arbitrary. Restraints were imposed on displacement parameters.

The H atoms of $C_{19}H_{24}O_7$ were all located in difference maps, but those attached to carbon atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their

geometry (C—H in the range 0.93–0.98, O—H = 0.82 Å) and with U_{iso}(H) in the range 1.2–1.5 times U_{eq} of the parent atom, after which the positions were refined with riding restraints and the displacement parameters were held fixed. H atoms of the solvate molecules were included at calculated positions and ride on the atom to which they bonded.

The largest peaks in the final difference electron density map are located within the disorder.

Computing details

Data collection: *COLLECT* (Nonius, 1997-2001).; cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP*II (Johnson 1976) in *TEXSAN* (MSC, 1992-1997); software used to prepare material for publication: *CRYSTALS* (Betteridge *et al.*, 2003).

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Crystal data

$C_{19}H_{24}O_7 \cdot 1.111(CHCl_3)$	$\gamma = 120^o$
$M_r = 496.99$	$V = 5327.6\ (2)$ Å^3
Trigonal, $R\bar{3}$	$Z = 9$
$a = 34.6694\ (10)$ Å	Mo $K\alpha$
$b = 34.6694\ (10)$ Å	$\mu = 0.46$ mm^{-1}
$c = 5.11810\ (10)$ Å	$T = 200$ K
$\alpha = 90^o$	$0.35 \times 0.07 \times 0.06$ mm
$\beta = 90^o$	

Data collection

Area diffractometer	4121 independent reflections
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	2923 reflections with $I > 2.0\sigma(I)$
$T_{min} = 0.915$, $T_{max} = 0.980$	$R_{int} = 0.041$
18939 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$	H-atom parameters constrained
$wR(F^2) = 0.120$	$\Delta\rho_{max} = 0.41$ e Å^{-3}
$S = 0.95$	$\Delta\rho_{min} = -0.33$ e Å^{-3}
4121 reflections	Absolute structure: Flack (1983), 2049 Friedel-pairs
347 parameters	Flack parameter: 0.02 (10)
130 restraints	

Table 1

Selected geometric parameters (Å, °)

Cl51—C53	1.760 (13)	O26—C17	1.362 (5)
Cl51—C54	1.784 (16)	C1—C18	1.465 (5)
Cl52—C53	1.984 (14)	C3—C4	1.525 (5)
Cl52—C54	1.788 (15)	C3—C20	1.515 (5)
Cl53—C53	1.799 (14)	C4—C5	1.475 (5)
Cl54—C54	1.876 (16)	C5—C6	1.328 (5)
Cl61—C61	1.841 (19)	C6—C7	1.482 (5)
Cl62—C61	1.768 (19)	C7—C8	1.505 (5)
Cl63—C61	1.768 (19)	C8—C9	1.526 (5)
Cl71—C71^{i}	1.865 (10)	C9—C10	1.516 (5)
Cl71—C71^{ii}	1.865 (10)	C10—C11	1.518 (5)
Cl72—C71	1.79 (3)	C11—C12	1.531 (5)
O2—C1	1.331 (4)	C12—C13	1.517 (5)
O2—C3	1.480 (4)	C13—C14	1.378 (5)
O19—C1	1.229 (4)	C13—C18	1.434 (5)
O21—C7	1.226 (4)	C14—C15	1.404 (6)
O22—C8	1.412 (4)	C15—C16	1.368 (6)
O23—C9	1.439 (4)	C16—C17	1.387 (6)
O24—C15	1.361 (5)	C17—C18	1.417 (5)
O24—C25	1.434 (5)		
C1—O2—C3	116.9 (3)	C14—C13—C18	119.1 (3)
C15—O24—C25	116.6 (4)	C13—C14—C15	121.4 (4)
O2—C1—O19	121.5 (3)	C14—C15—O24	114.5 (4)
O2—C1—C18	115.7 (3)	C14—C15—C16	120.9 (4)
O19—C1—C18	122.7 (3)	O24—C15—C16	124.6 (4)
O2—C3—C4	105.3 (3)	C15—C16—C17	118.7 (4)
O2—C3—C20	109.6 (3)	C16—C17—O26	115.5 (3)
C4—C3—C20	113.9 (3)	C16—C17—C18	122.5 (4)
C3—C4—C5	115.8 (3)	O26—C17—C18	122.0 (4)
C4—C5—C6	125.3 (3)	C1—C18—C13	125.9 (3)
C5—C6—C7	122.6 (3)	C1—C18—C17	116.7 (3)
C6—C7—O21	121.2 (3)	C13—C18—C17	117.3 (3)
C6—C7—C8	117.5 (3)	Cl53—C53—Cl51	105.5 (8)
O21—C7—C8	121.3 (3)	Cl53—C53—Cl52	104.6 (8)
C7—C8—O22	110.9 (3)	Cl51—C53—Cl52	99.0 (7)
C7—C8—C9	110.8 (3)	Cl54—C54—Cl52	99.7 (9)
O22—C8—C9	110.9 (3)	Cl54—C54—Cl51	103.2 (8)
C8—C9—O23	110.3 (3)	Cl52—C54—Cl51	105.9 (10)
C8—C9—C10	113.7 (3)	Cl61—C61—Cl63	103.9 (12)
O23—C9—C10	107.6 (3)	Cl61—C61—Cl62	97.6 (11)
C9—C10—C11	111.5 (3)	Cl63—C61—Cl62	104.2 (13)
C10—C11—C12	110.9 (3)	Cl71^{i}—C71—Cl71^{ii}	139.1 (12)
C11—C12—C13	117.2 (3)	Cl71^{i}—C71—Cl72	96.5 (13)
C12—C13—C14	119.6 (3)	Cl71^{ii}—C71—Cl72	115.0 (13)
C12—C13—C18	121.3 (3)		

Symmetry codes: (i) −*y*+2/3, *x*−*y*+1/3, *z*+1/3; (ii) −*x*+*y*+1/3, −*x*+2/3, *z*−1/3.

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supplementary materials

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Crystal data

$C_{19}H_{24}O_7 \cdot 1.111(CHCl_3)$	$Z = 9$
$M_r = 496.99$	$F_{000} = 2325.813$
Trigonal, $R3$	$D_x = 1.394$ Mg m^{-3}
$a = 34.6694$ (10) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 34.6694$ (10) Å	Cell parameters from 10011 reflections
$c = 5.11810$ (10) Å	$\theta = 3$–25º
$\alpha = 90$º	$\mu = 0.46$ mm^{-1}
$\beta = 90$º	$T = 200$ K
$\gamma = 120$º	Needle, colourless
$V = 5327.6$ (2) Å^3	0.35 × 0.07 × 0.06 mm

Data collection

Area diffractometer	2923 reflections with $I > 2.0\sigma(I)$
Monochromator: graphite	$R_{int} = 0.041$
$T = 200$ K	$\theta_{max} = 25.0$º
φ and ω scans with CCD	$\theta_{min} = 3.1$º
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	$h = -41 \rightarrow 41$
$T_{min} = 0.915$, $T_{max} = 0.980$	$k = -37 \rightarrow 41$
18939 measured reflections	$l = -5 \rightarrow 6$
4121 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.049$	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.07P)^2 + 0.0P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$wR(F^2) = 0.120$	$(\Delta/\sigma)_{max} = 0.030$
$S = 0.95$	$\Delta\rho_{max} = 0.41$ e Å^{-3}
4121 reflections	$\Delta\rho_{min} = -0.33$ e Å^{-3}
347 parameters	Extinction correction: None
130 restraints	Absolute structure: Flack (1983), 2049 Friedel-pairs

Primary atom site location: structure-invariant direct methods

Flack parameter: 0.02 (10)

Special details

Refinement. The absolute structure of the crystal has been determined by refinement of the Flack parameter and this establishes the absolute configuration of the molecule. It is in agreement with the configuration expected on the basis of the synthetic precursors.

There are two regions of disordered solvate molecules on the structure. One is localized, and is centred at about (0.10,0.07,0.17). There are three images of chloroform consisting of sites Cl51, Cl52, Cl53 and C53; Cl51, Cl52, Cl54 and C54; and Cl61, Cl62, Cl63 and C61. Some sites are common to two images. The relative occupancies of the sites were refined. Restraints were imposed on bonded distances and angles and on displacement parameters as appropriate.

There is also appears to be a chain of disordered atom sites running through the structure down the 3-fold screw axis 1/3,1/3,*z*. The sites are about 1.8Å apart suggesting they might arise from chloroform molecules which are not maintaining a regular stacking. The model presented here has C71 and Cl71 on a common site so a typical molecule will be C71, Cl71(2/3 − *y*,1/3 + *x*-*y*,1/3 + *z*), Cl71(2/3 − *x* + *y*,1/3 − *x*,1/3 + *z*) and Cl72, giving a rough tetrahedron about C71. The next chloroform image along the chain would be C71(2/3 − *x* + *y*,1/3 − *x*,1/3 + *z*), Cl71, Cl71(?) and Cl72(2/3 − *x* + *y*,1/3 − *x*,1/3 + *z*) and so on. The relative occupancies have been chosen to support this theory though their absolute values are arbitrary. Restraints were imposed on displacement parameters.

The H atoms of $C_{19}H_{24}O_7$ were all located in difference maps, but those attached to carbon atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98, O—H = 0.82 Å) and with U_{iso}(H) in the range 1.2–1.5 times U_{eq} of the parent atom, after which the positions were refined with riding restraints and the displacement parameters were held fixed. H atoms of the solvate molecules were included at calculated positions and ride on the atom to which they bonded.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl51	0.52894 (7)	0.49859 (7)	−0.0588 (7)	0.0994	0.734 (5)
Cl52	0.51689 (10)	0.41103 (8)	−0.0677 (8)	0.1194	0.734 (5)
Cl53	0.4629 (2)	0.43544 (18)	0.2797 (13)	0.1157	0.458 (7)
Cl54	0.4457 (2)	0.4224 (3)	0.086 (2)	0.1253	0.276 (6)
Cl61	0.4807 (6)	0.3980 (3)	−0.1646 (18)	0.1749	0.265 (5)
Cl62	0.5133 (7)	0.4869 (5)	−0.218 (3)	0.2420	0.265 (5)
Cl63	0.5016 (7)	0.4526 (4)	0.287 (2)	0.1884	0.265 (5)
Cl71	0.3458 (5)	0.3332 (5)	−0.260 (3)	0.2125	0.2222
Cl72	0.4045 (10)	0.3713 (10)	−0.251 (5)	0.1808	0.1111
O2	0.42262 (8)	0.63208 (8)	0.4424 (7)	0.0391	
O19	0.34949 (9)	0.59801 (10)	0.3640 (7)	0.0533	
O21	0.57132 (10)	0.70647 (10)	0.9889 (7)	0.0558	
O22	0.62358 (8)	0.67757 (9)	0.7956 (7)	0.0462	
O23	0.59857 (8)	0.63642 (9)	0.2811 (6)	0.0446	
O24	0.39971 (11)	0.47173 (12)	−0.2640 (8)	0.0769	
O26	0.31609 (9)	0.53789 (10)	0.0229 (7)	0.0627	
C1	0.38506 (13)	0.59845 (13)	0.3475 (8)	0.0409	
C3	0.42014 (13)	0.67036 (13)	0.5505 (8)	0.0424	
C4	0.46747 (12)	0.70980 (12)	0.5336 (8)	0.0395	
C5	0.50131 (13)	0.70573 (12)	0.6867 (8)	0.0404	

C6	0.53799 (13)	0.70814 (12)	0.5898 (8)	0.0396	
C7	0.56933 (12)	0.70100 (12)	0.7516 (9)	0.0409	
C8	0.59828 (12)	0.68641 (12)	0.6141 (8)	0.0382	
C9	0.57026 (12)	0.64581 (12)	0.4426 (8)	0.0363	
C10	0.54010 (12)	0.60392 (12)	0.5957 (8)	0.0395	
C11	0.50114 (12)	0.57068 (13)	0.4313 (8)	0.0415	
C12	0.46641 (12)	0.58531 (13)	0.3990 (9)	0.0427	
C13	0.42749 (12)	0.55779 (12)	0.2180 (8)	0.0408	
C14	0.42838 (14)	0.52617 (14)	0.0591 (9)	0.0495	
C15	0.39334 (15)	0.50047 (14)	−0.1138 (9)	0.0525	
C16	0.35629 (15)	0.50487 (15)	−0.1231 (9)	0.0560	
C17	0.35425 (12)	0.53585 (14)	0.0393 (9)	0.0474	
C18	0.38968 (12)	0.56409 (13)	0.2079 (8)	0.0402	
C20	0.40172 (15)	0.65973 (14)	0.8258 (8)	0.0516	
C25	0.36518 (19)	0.4453 (2)	−0.4471 (11)	0.0910	
C53	0.4836 (5)	0.4438 (4)	−0.050 (3)	0.1291	0.458 (7)
C54	0.5075 (5)	0.4484 (5)	0.126 (4)	0.0913	0.276 (6)
C61	0.5236 (9)	0.4511 (7)	−0.022 (4)	0.1895	0.265 (5)
C71	0.3458 (5)	0.3332 (5)	−0.260 (3)	0.2125	0.1111
H31	0.4000	0.6762	0.4388	0.0522*	
H41	0.4664	0.7359	0.6036	0.0487*	
H42	0.4758	0.7146	0.3496	0.0487*	
H51	0.4971	0.7015	0.8682	0.0496*	
H61	0.5431	0.7133	0.4121	0.0505*	
H81	0.6188	0.7115	0.4997	0.0443*	
H91	0.5523	0.6536	0.3261	0.0465*	
H101	0.5290	0.6115	0.7523	0.0475*	
H102	0.5580	0.5913	0.6561	0.0480*	
H111	0.5120	0.5686	0.2597	0.0530*	
H112	0.4870	0.5413	0.5163	0.0529*	
H121	0.4815	0.6159	0.3310	0.0534*	
H122	0.4541	0.5849	0.5753	0.0535*	
H141	0.4526	0.5214	0.0641	0.0600*	
H161	0.3325	0.4882	−0.2391	0.0590*	
H201	0.4006	0.6846	0.8998	0.0819*	
H202	0.4209	0.6531	0.9329	0.0813*	
H203	0.3719	0.6338	0.8204	0.0818*	
H221	0.6510	0.6950	0.7821	0.0658*	
H231	0.6102	0.6536	0.1584	0.0737*	
H251	0.3738	0.4272	−0.5491	0.1241*	
H252	0.3597	0.4640	−0.5664	0.1242*	
H253	0.3381	0.4258	−0.3527	0.1237*	
H261	0.3180	0.5524	0.1538	0.0923*	
H531	0.4617	0.4363	−0.1816	0.1515*	0.458
H541	0.5179	0.4521	0.3016	0.1323*	0.276
H611	0.5538	0.4592	−0.0497	0.2038*	0.265
H711	0.3445	0.3057	−0.2249	0.2457*	0.1111

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl51	0.0681 (13)	0.0643 (13)	0.168 (2)	0.0350 (11)	0.0054 (13)	0.0098 (13)
Cl52	0.1051 (19)	0.0676 (15)	0.200 (3)	0.0539 (14)	−0.0298 (18)	−0.0365 (16)
Cl53	0.122 (4)	0.096 (3)	0.137 (4)	0.060 (3)	0.039 (3)	0.014 (3)
Cl54	0.082 (4)	0.102 (5)	0.197 (7)	0.050 (4)	0.027 (4)	0.035 (5)
Cl61	0.268 (12)	0.101 (6)	0.147 (6)	0.086 (7)	0.032 (7)	−0.007 (5)
Cl62	0.299 (12)	0.169 (8)	0.207 (9)	0.079 (8)	0.020 (9)	−0.001 (7)
Cl63	0.248 (12)	0.153 (7)	0.165 (7)	0.101 (8)	−0.003 (8)	−0.019 (6)
Cl71	0.204 (12)	0.189 (12)	0.258 (13)	0.108 (10)	−0.052 (11)	−0.035 (9)
Cl72	0.188 (16)	0.185 (17)	0.227 (18)	0.137 (14)	0.001 (15)	0.018 (15)
O2	0.0392 (15)	0.0419 (15)	0.0411 (13)	0.0240 (13)	0.0028 (11)	0.0021 (11)
O19	0.0392 (17)	0.0581 (18)	0.0649 (17)	0.0259 (15)	0.0056 (13)	0.0048 (14)
O21	0.0651 (19)	0.072 (2)	0.0348 (15)	0.0380 (17)	−0.0008 (12)	0.0011 (12)
O22	0.0284 (13)	0.0599 (17)	0.0440 (13)	0.0173 (13)	0.0001 (11)	0.0147 (12)
O23	0.0492 (16)	0.0648 (18)	0.0353 (12)	0.0401 (15)	0.0078 (11)	0.0092 (12)
O24	0.066 (2)	0.072 (2)	0.081 (2)	0.0260 (18)	−0.0106 (17)	−0.0392 (18)
O26	0.0395 (17)	0.073 (2)	0.0708 (19)	0.0251 (16)	−0.0126 (14)	−0.0028 (16)
C1	0.032 (2)	0.041 (2)	0.048 (2)	0.0175 (19)	0.0023 (17)	0.0083 (17)
C3	0.048 (2)	0.048 (2)	0.042 (2)	0.033 (2)	0.0075 (17)	0.0048 (17)
C4	0.047 (2)	0.040 (2)	0.0366 (18)	0.0257 (19)	0.0063 (16)	0.0016 (16)
C5	0.050 (2)	0.050 (2)	0.0267 (17)	0.029 (2)	−0.0004 (16)	−0.0023 (15)
C6	0.046 (2)	0.044 (2)	0.0326 (18)	0.0259 (19)	0.0016 (16)	0.0009 (16)
C7	0.043 (2)	0.036 (2)	0.034 (2)	0.0119 (18)	0.0022 (16)	0.0043 (15)
C8	0.029 (2)	0.045 (2)	0.0360 (18)	0.0151 (17)	0.0008 (15)	0.0106 (16)
C9	0.037 (2)	0.052 (2)	0.0307 (16)	0.0299 (19)	0.0023 (15)	0.0071 (15)
C10	0.035 (2)	0.044 (2)	0.0429 (19)	0.0223 (18)	0.0001 (16)	0.0060 (16)
C11	0.042 (2)	0.043 (2)	0.047 (2)	0.0267 (19)	−0.0002 (17)	0.0002 (16)
C12	0.038 (2)	0.044 (2)	0.047 (2)	0.0213 (19)	−0.0048 (17)	−0.0051 (17)
C13	0.036 (2)	0.044 (2)	0.0378 (19)	0.0158 (18)	−0.0035 (15)	−0.0003 (16)
C14	0.044 (2)	0.044 (2)	0.058 (2)	0.021 (2)	−0.0032 (19)	−0.0079 (19)
C15	0.051 (3)	0.048 (3)	0.051 (2)	0.019 (2)	0.0011 (19)	−0.0086 (19)
C16	0.047 (3)	0.054 (3)	0.049 (2)	0.011 (2)	−0.0090 (19)	−0.004 (2)
C17	0.037 (2)	0.055 (3)	0.045 (2)	0.019 (2)	−0.0008 (18)	0.0084 (19)
C18	0.033 (2)	0.041 (2)	0.0392 (19)	0.0123 (18)	−0.0025 (16)	0.0031 (16)
C20	0.056 (3)	0.059 (3)	0.046 (2)	0.033 (2)	0.0130 (19)	0.0014 (19)
C25	0.083 (4)	0.086 (4)	0.083 (4)	0.026 (3)	−0.019 (3)	−0.046 (3)
C53	0.115 (6)	0.086 (5)	0.171 (6)	0.039 (5)	0.029 (5)	−0.012 (5)
C54	0.075 (5)	0.064 (5)	0.175 (6)	0.065 (4)	0.004 (5)	−0.010 (5)
C61	0.245 (10)	0.131 (7)	0.178 (8)	0.083 (8)	0.010 (8)	−0.009 (7)
C71	0.204 (12)	0.189 (12)	0.258 (13)	0.108 (10)	−0.052 (11)	−0.035 (9)

Geometric parameters (Å, °)

Cl51—C53	1.760 (13)	C6—H61	0.926
Cl51—C54	1.784 (16)	C7—C8	1.505 (5)
Cl52—C53	1.984 (14)	C8—C9	1.526 (5)

Cl52—C54	1.788 (15)	C8—H81	0.993
Cl53—C53	1.799 (14)	C9—C10	1.516 (5)
Cl54—C54	1.876 (16)	C9—H91	0.991
Cl61—C61	1.841 (19)	C10—C11	1.518 (5)
Cl62—C61	1.768 (19)	C10—H101	0.980
Cl63—C61	1.768 (19)	C10—H102	0.973
Cl71—C71^{i}	1.865 (10)	C11—C12	1.531 (5)
Cl71—C71^{ii}	1.865 (10)	C11—H111	0.971
Cl72—C71	1.79 (3)	C11—H112	0.985
O2—C1	1.331 (4)	C12—C13	1.517 (5)
O2—C3	1.480 (4)	C12—H121	0.981
O19—C1	1.229 (4)	C12—H122	0.995
O21—C7	1.226 (4)	C13—C14	1.378 (5)
O22—C8	1.412 (4)	C13—C18	1.434 (5)
O22—H221	0.835	C14—C15	1.404 (6)
O23—C9	1.439 (4)	C14—H141	0.934
O23—H231	0.819	C15—C16	1.368 (6)
O24—C15	1.361 (5)	C16—C17	1.387 (6)
O24—C25	1.434 (5)	C16—H161	0.942
O26—C17	1.362 (5)	C17—C18	1.417 (5)
O26—H261	0.819	C20—H201	0.961
C1—C18	1.465 (5)	C20—H202	0.975
C3—C4	1.525 (5)	C20—H203	0.974
C3—C20	1.515 (5)	C25—H251	0.971
C3—H31	0.998	C25—H252	0.975
C4—C5	1.475 (5)	C25—H253	0.969
C4—H41	0.992	C53—H531	0.950
C4—H42	0.975	C54—H541	0.950
C5—C6	1.328 (5)	C61—H611	0.950
C5—H51	0.940	C71—H711	0.950
C6—C7	1.482 (5)		
Cl51···O21^{iii}	3.330 (4)	O2···C20^{v}	3.478 (6)
Cl52···O26^{ii}	3.236 (5)	O19···C3^{viii}	3.315 (6)
Cl52···C17^{ii}	3.578 (5)	O19···C4^{viii}	3.358 (6)
Cl53···Cl62^{iv}	3.12 (2)	O19···C20^{v}	3.402 (5)
Cl53···Cl72^{iv}	3.21 (3)	O21···C9^{iv}	3.121 (6)
Cl53···Cl61^{iv}	3.31 (1)	O21···C6^{iv}	3.296 (6)
Cl53···C53^{iv}	3.49 (2)	O21···O23^{iv}	3.365 (5)
Cl54···O24	3.38 (1)	O21···C8^{iv}	3.500 (6)
Cl61···Cl63^{v}	3.26 (1)	O22···O23^{ix}	2.665 (4)
Cl61···C16^{ii}	3.40 (1)	O22···O23^{iv}	2.779 (4)
Cl61···C25^{vi}	3.56 (1)	O22···O22^{ix}	3.424 (5)
Cl62···Cl63^{v}	2.74 (2)	O22···O22^{x}	3.424 (5)
Cl62···O21^{iii}	3.50 (2)	O23···C8^{x}	3.194 (5)
Cl62···C54^{v}	3.58 (3)	O26···C4^{xi}	3.517 (6)

Cl63···C53^{iv}	3.44 (2)	O26···C4^{viii}	3.596 (6)
Cl71···Cl71^{vii}	3.494 (7)	C1···C20^{v}	3.279 (6)
Cl71···Cl71^{vi}	3.494 (7)	C6···C61^{ix}	3.37 (3)
Cl72···C25^{vi}	3.54 (3)	C14···C25^{iv}	3.592 (7)
Cl72···O24	3.57 (4)	C71···C71^{vii}	3.494 (7)
Cl72···C25	3.60 (4)	C71···C71^{vi}	3.494 (7)
C1—O2—C3	116.9 (3)	C13—C12—H122	107.7
C8—O22—H221	113.2	H121—C12—H122	109.4
C9—O23—H231	114.9	C12—C13—C14	119.6 (3)
C15—O24—C25	116.6 (4)	C12—C13—C18	121.3 (3)
C17—O26—H261	101.4	C14—C13—C18	119.1 (3)
O2—C1—O19	121.5 (3)	C13—C14—C15	121.4 (4)
O2—C1—C18	115.7 (3)	C13—C14—H141	120.2
O19—C1—C18	122.7 (3)	C15—C14—H141	118.4
O2—C3—C4	105.3 (3)	C14—C15—O24	114.5 (4)
O2—C3—C20	109.6 (3)	C14—C15—C16	120.9 (4)
C4—C3—C20	113.9 (3)	O24—C15—C16	124.6 (4)
O2—C3—H31	109.1	C15—C16—C17	118.7 (4)
C4—C3—H31	109.6	C15—C16—H161	122.4
C20—C3—H31	109.2	C17—C16—H161	118.9
C3—C4—C5	115.8 (3)	C16—C17—O26	115.5 (3)
C3—C4—H41	106.4	C16—C17—C18	122.5 (4)
C5—C4—H41	106.9	O26—C17—C18	122.0 (4)
C3—C4—H42	107.6	C1—C18—C13	125.9 (3)
C5—C4—H42	110.4	C1—C18—C17	116.7 (3)
H41—C4—H42	109.6	C13—C18—C17	117.3 (3)
C4—C5—C6	125.3 (3)	C3—C20—H201	110.5
C4—C5—H51	118.1	C3—C20—H202	109.0
C6—C5—H51	116.6	H201—C20—H202	109.2
C5—C6—C7	122.6 (3)	C3—C20—H203	108.8
C5—C6—H61	117.6	H201—C20—H203	110.0
C7—C6—H61	119.7	H202—C20—H203	109.4
C6—C7—O21	121.2 (3)	O24—C25—H251	109.7
C6—C7—C8	117.5 (3)	O24—C25—H252	111.2
O21—C7—C8	121.3 (3)	H251—C25—H252	108.5
C7—C8—O22	110.9 (3)	O24—C25—H253	109.2
C7—C8—C9	110.8 (3)	H251—C25—H253	108.7
O22—C8—C9	110.9 (3)	H252—C25—H253	109.5
C7—C8—H81	106.8	Cl53—C53—Cl51	105.5 (8)
O22—C8—H81	109.0	Cl53—C53—Cl52	104.6 (8)
C9—C8—H81	108.4	Cl51—C53—Cl52	99.0 (7)
C8—C9—O23	110.3 (3)	Cl53—C53—H531	114.9
C8—C9—C10	113.7 (3)	Cl51—C53—H531	115.9
O23—C9—C10	107.6 (3)	Cl52—C53—H531	115.2
C8—C9—H91	107.0	Cl54—C54—Cl52	99.7 (9)
O23—C9—H91	107.9	Cl54—C54—Cl51	103.2 (8)
C10—C9—H91	110.2	Cl52—C54—Cl51	105.9 (10)

C9—C10—C11	111.5 (3)	Cl54—C54—H541	115.7
C9—C10—H101	110.0	Cl52—C54—H541	115.3
C11—C10—H101	109.7	Cl51—C54—H541	115.2
C9—C10—H102	107.8	Cl61—C61—Cl63	103.9 (12)
C11—C10—H102	111.2	Cl61—C61—Cl62	97.6 (11)
H101—C10—H102	106.6	Cl63—C61—Cl62	104.2 (13)
C10—C11—C12	110.9 (3)	Cl61—C61—H611	117.2
C10—C11—H111	109.1	Cl63—C61—H611	123.6
C12—C11—H111	108.8	Cl62—C61—H611	106.6
C10—C11—H112	109.5	Cl71^{i}—C71—Cl71^{ii}	139.1 (12)
C12—C11—H112	108.9	Cl71^{i}—C71—Cl72	96.5 (13)
H111—C11—H112	109.6	Cl71^{ii}—C71—Cl72	115.0 (13)
C11—C12—C13	117.2 (3)	Cl71^{i}—C71—H711	99.3
C11—C12—H121	108.1	Cl71^{ii}—C71—H711	99.3
C13—C12—H121	106.9	Cl72—C71—H711	101.5
C11—C12—H122	107.4		
O2—C1—C18—C13	15.8 (6)	C1—C18—C17—C16	173.6 (4)
O2—C1—C18—C17	−161.3 (4)	C3—O2—C1—C18	171.4 (3)
O2—C3—C4—C5	−62.0 (5)	C3—C4—C5—C6	122.4 (4)
O19—C1—O2—C3	−4.5 (6)	C4—C5—C6—C7	−176.1 (3)
O19—C1—C18—C13	−168.3 (4)	C5—C4—C3—C20	58.1 (6)
O19—C1—C18—C17	14.6 (6)	C5—C6—C7—C8	156.0 (3)
O21—C7—C6—C5	−23.4 (5)	C6—C7—C8—C9	−51.7 (4)
O21—C7—C8—O22	4.2 (4)	C7—C8—C9—C10	−68.1 (5)
O21—C7—C8—C9	127.7 (4)	C8—C9—C10—C11	157.4 (4)
O22—C8—C7—C6	−175.3 (3)	C9—C10—C11—C12	−75.1 (5)
O22—C8—C9—O23	−65.5 (4)	C10—C11—C12—C13	174.3 (3)
O22—C8—C9—C10	55.4 (5)	C11—C12—C13—C14	−10.7 (5)
O23—C9—C8—C7	170.9 (3)	C11—C12—C13—C18	169.5 (3)
O23—C9—C10—C11	−80.1 (5)	C12—C13—C14—C15	−179.1 (4)
O24—C15—C14—C13	177.9 (4)	C12—C13—C18—C17	−178.0 (4)
O24—C15—C16—C17	−179.4 (4)	C13—C14—C15—C16	−2.5 (7)
O26—C17—C16—C15	−178.9 (4)	C13—C18—C17—C16	−3.8 (6)
O26—C17—C18—C1	−5.3 (6)	C14—C13—C18—C17	2.3 (6)
O26—C17—C18—C13	177.4 (4)	C14—C15—O24—C25	−178.9 (4)
C1—O2—C3—C4	−156.3 (4)	C14—C15—C16—C17	1.0 (6)
C1—O2—C3—C20	80.8 (5)	C15—C14—C13—C18	0.7 (6)
C1—C18—C13—C12	4.9 (6)	C15—C16—C17—C18	2.1 (7)
C1—C18—C13—C14	−174.8 (4)	C16—C15—O24—C25	1.4 (6)

Symmetry codes: (i) −*y*+2/3, *x*−*y*+1/3, *z*+1/3; (ii) −*x*+*y*+1/3, −*x*+2/3, *z*−1/3; (iii) −*y*+4/3, *x*−*y*+2/3, *z*−4/3; (iv) *x*, *y*, *z*+1; (v) *x*, *y*, *z*−1; (vi) −*x*+*y*+1/3, −*x*+2/3, *z*+2/3; (vii) −*y*+2/3, *x*−*y*+1/3, *z*−2/3; (viii) −*x*+*y*, −*x*+1, *z*; (ix) −*x*+*y*+2/3, −*x*+4/3, *z*+1/3; (x) −*y*+4/3, *x*−*y*+2/3, *z*−1/3; (xi) −*x*+*y*, −*x*+1, *z*−1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O22—H221···O23^{ix}	0.84	1.88	2.665 (4)	157
O23—H231···O22^{v}	0.82	1.99	2.779 (4)	161

O26—H261···O19	0.82	1.77	2.514 (5)	150

Symmetry codes: (ix) −*x*+*y*+2/3, −*x*+4/3, *z*+1/3; (v) *x*, *y*, *z*−1.

Crystal structure of $C_{11}H_{21}NO_5$ — ban0834

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Abstract

The crystal structure of $C_{11}H_{21}NO_5$ is reported.

Comment

The crystallographic asymmetric unit consists of one $C_{11}H_{21}NO_5$ molecule.

Experimental

The compound was prepared by AL. The sample ID is AL486.weinrebOH.

Refinement

The compound is enantiometrically pure but the anomolous dispersion terms are very low for all elements in the structure and so the absolute configuration can not be determined in this experiment. Consequently Friedel-pair reflections have been averaged and the Flack parameter has not been refined. The absolute configuration of the molecule has been assigned on the basis of the synthetic precursors.

After refinement of a totally ordered model, a difference electron density map showed a significant peak near H62, which possibly indicated an alternative site for the alcohol O atom. This site was incorporated into the refinement model with an isotropic displacement parameter. Restraints were imposed on the C—O length and C—C—O angle of the minor site in subsequent refinement in which the relative occupancies of the two O sites were refined.

H atoms attached to C atoms were included at geometrically determined positions and that on O17 at the position indicated in a difference map. They were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98 and O—H 0.82 Å) and U_{iso}(H) (in the range 1.2–1.5 times U_{eq} of the parent atom). H sites on C6 were reset to calculated positions. In the final refinement all H atoms ride on the atoms to which they are respectively attached.

The largest peaks in the final difference electron density map are located along C—C bonds.

Computing details

Data collection: *COLLECT* (Nonius, 1997-2001).; cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP*II (Johnson 1976) in *TEXSAN* (MSC, 1992-1997); software used to prepare material for publication: *CRYSTALS* (Betteridge *et al.*, 2003).

(ban0834)

Crystal data

$C_{11}H_{21}NO_5$	$V = 656.93\ (9)$ Å^3
$M_r = 247.29$	$Z = 2$
Monoclinic, $P2_1$	Mo $K\alpha$
$a = 8.2018\ (6)$ Å	$\mu = 0.10$ mm^{-1}
$b = 7.7781\ (7)$ Å	$T = 200$ K
$c = 10.6923\ (8)$ Å	0.33 × 0.17 × 0.04 mm
$\beta = 105.615\ (5)^o$	

Data collection

Area diffractometer	1245 independent reflections
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	1010 reflections with $I > 2.0\sigma(I)$
$T_{min} = 0.978$, $T_{max} = 0.996$	$R_{int} = 0.069$
8789 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$	H-atom parameters constrained
$wR(F^2) = 0.071$	$\Delta\rho_{max} = 0.16$ e Å^{-3}
$S = 0.83$	$\Delta\rho_{min} = -0.14$ e Å^{-3}
1242 reflections	Absolute structure: The enantiomer has been assigned by reference to an unchanging chiral centre in the synthetic procedure.
160 parameters	
3 restraints	

Table 1
Selected geometric parameters (Å, °)

O7—C1	1.227 (3)	N13—C1	1.343 (3)
O8—C2	1.417 (3)	N13—C14	1.453 (3)
O8—C9	1.443 (3)	C1—C2	1.526 (3)
O10—C3	1.440 (3)	C2—C3	1.548 (3)

O10—C9	1.412 (3)	C3—C4	1.518 (3)
O15—N13	1.400 (2)	C4—C5	1.520 (3)
O15—C16	1.443 (3)	C5—C6	1.502 (3)
O17—C6	1.428 (3)	C9—C11	1.512 (4)
O18—C6	1.407 (19)	C9—C12	1.500 (3)
C2—O8—C9	109.31 (17)	C2—C3—C4	117.98 (19)
C3—O10—C9	107.06 (15)	O10—C3—C4	108.35 (17)
N13—O15—C16	108.5 (2)	C3—C4—C5	113.10 (19)
O15—N13—C1	117.53 (17)	C4—C5—C6	112.13 (19)
O15—N13—C14	114.29 (19)	C5—C6—O17	112.9 (2)
C1—N13—C14	123.59 (19)	C5—C6—O18	111.6 (10)
N13—C1—O7	120.1 (2)	O8—C9—O10	104.52 (17)
N13—C1—C2	117.1 (2)	O8—C9—C11	109.9 (2)
O7—C1—C2	122.7 (2)	O10—C9—C11	111.7 (2)
C1—C2—O8	109.0 (2)	O8—C9—C12	107.8 (2)
C1—C2—C3	111.25 (18)	O10—C9—C12	109.09 (18)
O8—C2—C3	104.78 (16)	C11—C9—C12	113.4 (2)
C2—C3—O10	102.13 (18)		

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supplementary materials

Crystal structure of $C_{11}H_{21}NO_5$ — ban0834

Andrew Lin, Martin G. Banwell and Anthony C. Willis

(ban0834)

Crystal data

$C_{11}H_{21}NO_5$	F_{000} = 268
M_r = 247.29	D_x = 1.250 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.2018 (6) Å	Cell parameters from 20531 reflections
b = 7.7781 (7) Å	θ = 3–25º
c = 10.6923 (8) Å	μ = 0.10 mm^{-1}
β = 105.615 (5)º	T = 200 K
V = 656.93 (9) Å^3	Plate, colourless
Z = 2	0.33 × 0.17 × 0.04 mm

Data collection

Area diffractometer	1010 reflections with $I > 2.0\sigma(I)$
Monochromator: graphite	R_{int} = 0.069
T = 200 K	θ_{max} = 25.0º
φ and ω scans with CCD	θ_{min} = 2.6º
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	h = −9→9
T_{min} = 0.978, T_{max} = 0.996	k = −8→9
8789 measured reflections	l = −12→12
1245 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.031	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.03P)^2 + 0.0P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$wR(F^2)$ = 0.071	$(\Delta/\sigma)_{max}$ = 0.001
S = 0.83	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
1242 reflections	$\Delta\rho_{min}$ = −0.14 e Å^{-3}
160 parameters	Extinction correction: Larson (1970), Equation 22
3 restraints	Extinction coefficient: 300 (30)

Primary atom site location: structure-invariant direct methods

Absolute structure: The enantiomer has been assigned by reference to an unchanging chiral centre in the synthetic procedure.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O7	0.7323 (2)	0.9231 (4)	0.84194 (18)	0.0653	
O8	0.6279 (2)	0.8798 (3)	0.57741 (17)	0.0599	
O10	0.82441 (18)	0.6696 (3)	0.60629 (14)	0.0455	
O15	0.39132 (19)	0.6467 (3)	0.81322 (14)	0.0494	
O17	1.0355 (2)	0.1004 (3)	0.86720 (18)	0.0560	0.928 (4)
O18	0.843 (3)	0.195 (3)	0.953 (2)	0.067 (9)*	0.072 (4)
N13	0.5117 (2)	0.7728 (4)	0.86657 (18)	0.0475	
C1	0.6139 (3)	0.8257 (4)	0.7950 (2)	0.0461	
C2	0.5783 (3)	0.7556 (4)	0.6570 (2)	0.0466	
C3	0.6892 (3)	0.5964 (4)	0.6513 (2)	0.0426	
C4	0.7664 (3)	0.5002 (4)	0.7767 (2)	0.0435	
C5	0.8604 (3)	0.3383 (4)	0.7571 (2)	0.0478	
C6	0.9545 (3)	0.2579 (4)	0.8837 (2)	0.0515	
C9	0.7543 (3)	0.8064 (4)	0.5221 (2)	0.0473	
C11	0.6723 (3)	0.7437 (5)	0.3858 (2)	0.0633	
C12	0.8881 (3)	0.9401 (4)	0.5283 (2)	0.0589	
C14	0.5564 (3)	0.7822 (5)	1.0074 (2)	0.0679	
C16	0.2256 (3)	0.7244 (5)	0.7825 (3)	0.0774	
H17	0.9523	0.0285	0.8397	0.0838*	0.928
H18	0.8026	0.0960	0.9123	0.0796*	0.072
H21	0.4603	0.7266	0.6206	0.0536*	
H31	0.6243	0.5131	0.5847	0.0508*	
H41	0.8427	0.5744	0.8409	0.0525*	
H42	0.6721	0.4646	0.8113	0.0527*	
H51	0.9361	0.3673	0.7038	0.0570*	
H52	0.7818	0.2554	0.7116	0.0565*	
H61	1.0382	0.3365	0.9293	0.0614*	0.928
H62	0.8765	0.2357	0.9333	0.0614*	0.928
H63	1.0261	0.3419	0.9354	0.0614*	0.072
H64	1.0213	0.1656	0.8667	0.0614*	0.072
H111	0.7577	0.6890	0.3506	0.0915*	
H112	0.6249	0.8392	0.3314	0.0922*	
H113	0.5845	0.6632	0.3869	0.0918*	
H121	0.9726	0.8914	0.4939	0.0891*	
H122	0.9346	0.9746	0.6174	0.0880*	
H123	0.8388	1.0380	0.4769	0.0883*	
H141	0.4604	0.7657	1.0425	0.1006*	
H142	0.5988	0.8968	1.0309	0.1014*	
H143	0.6417	0.6981	1.0458	0.1015*	
H161	0.1475	0.6335	0.7517	0.1140*	
H162	0.2047	0.7774	0.8568	0.1136*	
H163	0.2128	0.8044	0.7138	0.1136*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O7	0.0531 (11)	0.0612 (12)	0.0785 (13)	−0.0142 (10)	0.0124 (10)	−0.0067 (11)
O8	0.0575 (10)	0.0633 (12)	0.0697 (11)	0.0205 (10)	0.0355 (9)	0.0249 (10)
O10	0.0375 (8)	0.0533 (10)	0.0485 (8)	0.0049 (8)	0.0162 (7)	0.0105 (9)
O15	0.0424 (9)	0.0553 (11)	0.0525 (9)	−0.0056 (9)	0.0159 (7)	−0.0036 (9)
O17	0.0489 (11)	0.0497 (12)	0.0690 (12)	0.0067 (10)	0.0153 (9)	0.0065 (10)
N13	0.0410 (10)	0.0549 (13)	0.0467 (11)	−0.0028 (11)	0.0122 (9)	−0.0080 (11)
C1	0.0389 (12)	0.0451 (14)	0.0538 (14)	0.0040 (12)	0.0118 (10)	0.0014 (12)
C2	0.0385 (12)	0.0547 (15)	0.0480 (14)	0.0036 (13)	0.0137 (10)	0.0091 (13)
C3	0.0370 (12)	0.0491 (14)	0.0432 (11)	−0.0039 (12)	0.0132 (10)	0.0023 (11)
C4	0.0427 (12)	0.0465 (13)	0.0433 (12)	0.0016 (12)	0.0151 (10)	0.0048 (11)
C5	0.0496 (13)	0.0483 (14)	0.0483 (13)	0.0026 (12)	0.0178 (10)	0.0036 (12)
C6	0.0499 (13)	0.0499 (14)	0.0536 (14)	0.0055 (14)	0.0121 (11)	0.0015 (13)
C9	0.0427 (12)	0.0556 (16)	0.0459 (13)	0.0066 (13)	0.0158 (10)	0.0082 (12)
C11	0.0619 (16)	0.0750 (19)	0.0490 (15)	−0.0002 (16)	0.0080 (12)	0.0097 (14)
C12	0.0585 (16)	0.0641 (18)	0.0581 (15)	−0.0040 (14)	0.0226 (13)	0.0068 (14)
C14	0.0644 (17)	0.092 (2)	0.0482 (14)	−0.0002 (18)	0.0177 (13)	−0.0154 (17)
C16	0.0386 (13)	0.121 (3)	0.0739 (18)	0.0072 (17)	0.0182 (12)	−0.0164 (19)

Geometric parameters (Å, °)

O7—C1	1.227 (3)	C5—C6	1.502 (3)
O8—C2	1.417 (3)	C5—H51	0.975
O8—C9	1.443 (3)	C5—H52	0.948
O10—C3	1.440 (3)	C6—H61	0.950
O10—C9	1.412 (3)	C6—H62	0.950
O15—N13	1.400 (2)	C6—H63	0.950
O15—C16	1.443 (3)	C6—H64	0.950
O17—C6	1.428 (3)	C9—C11	1.512 (4)
O17—H17	0.870	C9—C12	1.500 (3)
O18—C6	1.407 (19)	C11—H111	0.979
O18—H18	0.900	C11—H112	0.959
N13—C1	1.343 (3)	C11—H113	0.957
N13—C14	1.453 (3)	C12—H121	0.947
C1—C2	1.526 (3)	C12—H122	0.964
C2—C3	1.548 (3)	C12—H123	0.962
C2—H21	0.967	C14—H141	0.967
C3—C4	1.518 (3)	C14—H142	0.965
C3—H31	1.003	C14—H143	0.965
C4—C5	1.520 (3)	C16—H161	0.951
C4—H41	0.982	C16—H162	0.950
C4—H42	0.983	C16—H163	0.947
O7···O18^{i}	2.48 (2)	O15···C14^{vi}	3.385 (4)
O7···O17^{i}	2.792 (3)	O15···O18^{vii}	3.56 (3)
O7···C6^{i}	3.141 (4)	O17···C16^{viii}	3.545 (4)

O7···C6^{ii}	3.578 (3)	O17···C14^{ix}	3.547 (3)
O7···C5^{i}	3.587 (4)	O18···C16^{vi}	3.04 (2)
O8···C3^{iii}	3.487 (3)	O18···C14^{vi}	3.48 (3)
O10···C16^{iv}	3.350 (3)	O18···C1^{x}	3.60 (2)
O10···C12^{v}	3.556 (3)		
C2—O8—C9	109.31 (17)	C5—C6—H62	108.6
C3—O10—C9	107.06 (15)	O17—C6—H62	108.6
N13—O15—C16	108.5 (2)	H61—C6—H62	109.5
C6—O17—H17	104.1	C5—C6—H63	108.9
C6—O18—H18	104.8	O18—C6—H63	108.8
O15—N13—C1	117.53 (17)	C5—C6—H64	108.9
O15—N13—C14	114.29 (19)	O18—C6—H64	109.1
C1—N13—C14	123.59 (19)	H63—C6—H64	109.5
N13—C1—O7	120.1 (2)	O8—C9—O10	104.52 (17)
N13—C1—C2	117.1 (2)	O8—C9—C11	109.9 (2)
O7—C1—C2	122.7 (2)	O10—C9—C11	111.7 (2)
C1—C2—O8	109.0 (2)	O8—C9—C12	107.8 (2)
C1—C2—C3	111.25 (18)	O10—C9—C12	109.09 (18)
O8—C2—C3	104.78 (16)	C11—C9—C12	113.4 (2)
C1—C2—H21	112.5	C9—C11—H111	109.4
O8—C2—H21	109.6	C9—C11—H112	109.7
C3—C2—H21	109.5	H111—C11—H112	108.6
C2—C3—O10	102.13 (18)	C9—C11—H113	110.0
C2—C3—C4	117.98 (19)	H111—C11—H113	109.8
O10—C3—C4	108.35 (17)	H112—C11—H113	109.3
C2—C3—H31	109.8	C9—C12—H121	107.9
O10—C3—H31	109.1	C9—C12—H122	109.0
C4—C3—H31	109.0	H121—C12—H122	111.0
C3—C4—C5	113.10 (19)	C9—C12—H123	109.5
C3—C4—H41	111.3	H121—C12—H123	109.6
C5—C4—H41	109.4	H122—C12—H123	109.7
C3—C4—H42	106.8	N13—C14—H141	113.0
C5—C4—H42	107.4	N13—C14—H142	106.8
H41—C4—H42	108.7	H141—C14—H142	107.2
C4—C5—C6	112.13 (19)	N13—C14—H143	111.0
C4—C5—H51	108.4	H141—C14—H143	108.5
C6—C5—H51	111.7	H142—C14—H143	110.3
C4—C5—H52	109.5	O15—C16—H161	105.8
C6—C5—H52	107.0	O15—C16—H162	111.2
H51—C5—H52	108.0	H161—C16—H162	110.8
C5—C6—O17	112.9 (2)	O15—C16—H163	110.6
C5—C6—O18	111.6 (10)	H161—C16—H163	107.2
C5—C6—H61	108.6	H162—C16—H163	110.9
O17—C6—H61	108.6		
O7—C1—N13—O15	−172.8 (3)	C1—N13—O15—C16	−110.2 (3)
O7—C1—N13—C14	−18.2 (5)	C1—C2—O8—C9	120.9 (2)
O7—C1—C2—O8	−32.0 (4)	C1—C2—C3—C4	19.7 (3)

O7—C1—C2—C3	83.1 (4)	C2—O8—C9—C11	97.9 (2)
O8—C2—C1—N13	149.7 (2)	C2—O8—C9—C12	−138.2 (2)
O8—C2—C3—O10	18.7 (2)	C2—C1—N13—C14	160.2 (3)
O8—C2—C3—C4	137.4 (2)	C2—C3—O10—C9	−33.2 (2)
O8—C9—O10—C3	35.1 (2)	C2—C3—C4—C5	175.3 (2)
O10—C3—C2—C1	−98.9 (2)	C3—O10—C9—C11	−83.8 (2)
O10—C3—C4—C5	−69.4 (3)	C3—O10—C9—C12	150.1 (2)
O10—C9—O8—C2	−22.2 (2)	C3—C2—O8—C9	1.8 (2)
O15—N13—C1—C2	5.6 (4)	C3—C4—C5—C6	172.6 (2)
O17—C6—C5—C4	176.6 (2)	C4—C3—O10—C9	−158.5 (2)
O18—C6—C5—C4	66 (1)	C14—N13—O15—C16	92.9 (3)
N13—C1—C2—C3	−95.2 (3)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O17—H17···O7^{x}	0.87	1.99	2.793 (4)	153
O18—H18···O7^{x}	0.90	1.57	2.472 (4)	180

Symmetry codes: (x) *x*, *y*−1, *z*.

Crystal structure of $C_{17}H_{24}N_2O_6Se$ — ban0915

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Abstract

The crystal structure of $C_{17}H_{24}N_2O_6Se$ is reported.

Comment

The crystallographic asymmetric unit consists of four $C_{17}H_{24}N_2O_6Se$ molecules, one of which shows some disorder.

Parts of the structure show pseudo-symmetry relationships. Each molecule, however, has features that make it significantly different from all the others. The molecule containing Se7 is very similar to that containing Se107 except that the five-membered ring has a different conformation (an envelope on O12 for the former, twisted on O116—C113 for the latter). These molecules differ substantially from the molecules containing Se207 and Se307 with regard to the location of the $SeC_6H_4NO_2$ unit relative to the five-membered ring (−72° for torsion angle Se7—C8—C9—C10 and −71° for the Se107 counterpart, compared with −165°, −179°, and −160° for Se207, Se307 and Se407, respectively). The molecule containing Se207 differs from that containing Se307 in that torsion angle C218—N219—O220—C221 is −114° *cf.* 119°.

Reflections with h+k=2n tend to be significantly stronger than those with h+k=2n+1, but the agreement factors for each group are in line with expectation for their respective average intensities. An approximate C-centring in observed in the final structure.

Experimental

The compound was prepared by AL. The sample ID is AL-633.

Refinement

The compound is enantiometrically pure. The absolute structure of the crystal has been determined by refinement of the Flack parameter and this establishes the absolute configuration of the molecule.

There is disorder in the molecule with atoms labelled X3xx. Besides the major locations for atoms Se307, C308 and C309 there are also alternative sites (labelled Se407, C408, C409) which correspond to an alternative conformation of this section of the molecule. Restraints were imposed on distances, angles and displacement parameters for disordered sites and the relative occupancies of the two conformations were refined.

H atoms were included at calculated positions and ride on the atom to which they bonded.

The largest peaks in the final difference electron density map are located near Se atoms.

Computing details

Data collection: *COLLECT* (Nonius, 1997-2001).; cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP*II (Johnson 1976) in *TEXSAN* (MSC, 1992-1997); software used to prepare material for publication: *CRYSTALS* (Betteridge *et al.*, 2003).

(ban0915)

Crystal data

$C_{17}H_{24}N_2O_6Se$	$\gamma = 96.309\ (2)^o$
$M_r = 431.35$	$V = 1908.18\ (11)$ Å^3
Triclinic, $P\overline{1}$	$Z = 4$
$a = 8.0087\ (2)$ Å	Mo $K\alpha$
$b = 14.3159\ (5)$ Å	$\mu = 2.00$ mm^{-1}
$c = 17.0739\ (6)$ Å	$T = 200$ K
$\alpha = 100.0775\ (14)^o$	0.30 × 0.10 × 0.08 mm
$\beta = 93.859\ (2)^o$	

Data collection

Area diffractometer	16425 independent reflections
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	12385 reflections with $I > 2.0\sigma(I)$
$T_{min} = 0.713$, $T_{max} = 0.881$	$R_{int} = 0.045$
36797 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$	H-atom parameters constrained
$wR(F^2) = 0.092$	$\Delta\rho_{max} = 0.94$ e Å^{-3}
$S = 1.00$	$\Delta\rho_{min} = -0.82$ e Å^{-3}
16425 reflections	Absolute structure: Flack (1983), 7715 Friedel-pairs
967 parameters	Flack parameter: 0.000 (5)
42 restraints	

Table 1
Selected geometric parameters (Å, °)

Se7—C6	1.905 (4)	N319—C323	1.433 (6)
Se7—C8	1.954 (4)	N324—C301	1.459 (5)

Se107—C106	1.915 (4)	C1—C2	1.388 (6)
Se107—C108	1.964 (4)	C1—C6	1.402 (6)
Se207—C206	1.899 (4)	C2—C3	1.365 (7)
Se207—C208	1.952 (4)	C3—C4	1.380 (6)
Se307—C306	1.806 (4)	C4—C5	1.381 (6)
Se307—C308	1.942 (5)	C5—C6	1.409 (5)
Se407—C306	2.054 (5)	C8—C9	1.523 (5)
Se407—C408	1.930 (8)	C9—C10	1.525 (5)
O12—C11	1.442 (4)	C10—C11	1.520 (5)
O12—C13	1.426 (4)	C11—C17	1.558 (5)
O16—C13	1.429 (4)	C13—C14	1.508 (5)
O16—C17	1.414 (4)	C13—C15	1.503 (6)
O20—N19	1.414 (4)	C17—C18	1.517 (5)
O20—C21	1.436 (5)	C101—C102	1.395 (6)
O22—C18	1.220 (5)	C101—C106	1.393 (5)
O25—N24	1.225 (5)	C102—C103	1.375 (6)
O26—N24	1.223 (5)	C103—C104	1.386 (6)
O112—C111	1.444 (4)	C104—C105	1.374 (5)
O112—C113	1.429 (4)	C105—C106	1.393 (5)
O116—C113	1.428 (4)	C108—C109	1.504 (5)
O116—C117	1.441 (4)	C109—C110	1.522 (5)
O120—N119	1.409 (4)	C110—C111	1.513 (5)
O120—C121	1.423 (5)	C111—C117	1.556 (5)
O122—C118	1.215 (5)	C113—C114	1.520 (5)
O125—N124	1.240 (5)	C113—C115	1.502 (5)
O126—N124	1.232 (5)	C117—C118	1.513 (5)
O212—C211	1.426 (5)	C201—C202	1.389 (6)
O212—C213	1.422 (5)	C201—C206	1.399 (5)
O216—C213	1.434 (5)	C202—C203	1.372 (6)
O216—C217	1.409 (5)	C203—C204	1.394 (6)
O220—N219	1.418 (4)	C204—C205	1.361 (6)
O220—C221	1.401 (5)	C205—C206	1.417 (5)
O222—C218	1.215 (5)	C208—C209	1.533 (6)
O225—N224	1.224 (4)	C209—C210	1.518 (5)
O226—N224	1.233 (5)	C210—C211	1.514 (5)
O312—C311	1.436 (5)	C211—C217	1.541 (5)
O312—C313	1.418 (4)	C213—C214	1.521 (6)
O316—C313	1.446 (5)	C213—C215	1.517 (7)
O316—C317	1.417 (5)	C217—C218	1.527 (5)
O320—N319	1.413 (4)	C301—C302	1.378 (6)
O320—C321	1.421 (6)	C301—C306	1.397 (5)
O322—C318	1.217 (4)	C302—C303	1.369 (6)
O325—N324	1.227 (5)	C303—C304	1.368 (7)
O326—N324	1.221 (5)	C304—C305	1.373 (7)
N19—C18	1.364 (5)	C305—C306	1.417 (6)
N19—C23	1.435 (5)	C308—C309	1.543 (9)
N24—C1	1.463 (5)	C309—C310	1.521 (8)
N119—C118	1.374 (5)	C310—C311	1.505 (6)
N119—C123	1.442 (5)	C310—C409	1.627 (11)

N124—C101	1.455 (5)	C311—C317	1.539 (5)
N219—C218	1.353 (5)	C313—C314	1.519 (5)
N219—C223	1.446 (5)	C313—C315	1.507 (6)
N224—C201	1.456 (5)	C317—C318	1.534 (5)
N319—C318	1.351 (5)	C408—C409	1.508 (13)
C6—Se7—C8	100.01 (17)	Se107—C108—C109	116.0 (3)
C106—Se107—C108	100.24 (17)	C108—C109—C110	113.6 (3)
C206—Se207—C208	101.90 (17)	C109—C110—C111	112.4 (3)
C306—Se307—C308	103.6 (3)	C110—C111—O112	106.9 (3)
C306—Se407—C408	102.6 (4)	C110—C111—C117	116.5 (3)
C11—O12—C13	108.9 (3)	O112—C111—C117	103.5 (3)
C13—O16—C17	111.4 (3)	O112—C113—O116	104.3 (3)
N19—O20—C21	109.5 (3)	O112—C113—C114	109.9 (3)
C111—O112—C113	109.1 (3)	O116—C113—C114	111.4 (3)
C113—O116—C117	107.3 (3)	O112—C113—C115	108.8 (3)
N119—O120—C121	109.7 (3)	O116—C113—C115	108.2 (3)
C211—O212—C213	107.5 (3)	C114—C113—C115	113.7 (3)
C213—O216—C217	110.0 (3)	C111—C117—O116	104.4 (3)
N219—O220—C221	109.9 (3)	C111—C117—C118	115.4 (3)
C311—O312—C313	108.0 (3)	O116—C117—C118	106.5 (3)
C313—O316—C317	109.5 (3)	C117—C118—N119	114.8 (3)
N319—O320—C321	109.1 (4)	C117—C118—O122	125.1 (4)
O20—N19—C18	115.3 (3)	N119—C118—O122	120.0 (4)
O20—N19—C23	112.8 (3)	N224—C201—C202	116.9 (4)
C18—N19—C23	123.1 (3)	N224—C201—C206	120.6 (4)
O25—N24—O26	122.6 (4)	C202—C201—C206	122.5 (4)
O25—N24—C1	118.0 (4)	C201—C202—C203	119.7 (4)
O26—N24—C1	119.4 (4)	C202—C203—C204	119.4 (4)
O120—N119—C118	115.1 (3)	C203—C204—C205	120.8 (4)
O120—N119—C123	114.2 (3)	C204—C205—C206	121.7 (4)
C118—N119—C123	123.5 (3)	C205—C206—C201	115.9 (4)
O125—N124—O126	122.5 (4)	C205—C206—Se207	120.9 (3)
O125—N124—C101	118.3 (4)	C201—C206—Se207	123.3 (3)
O126—N124—C101	119.1 (4)	Se207—C208—C209	104.9 (3)
O220—N219—C218	117.5 (3)	C208—C209—C210	113.9 (3)
O220—N219—C223	113.4 (4)	C209—C210—C211	114.9 (3)
C218—N219—C223	125.6 (4)	C210—C211—O212	109.4 (3)
O226—N224—O225	122.1 (4)	C210—C211—C217	116.2 (3)
O226—N224—C201	118.4 (3)	O212—C211—C217	101.5 (3)
O225—N224—C201	119.5 (4)	O216—C213—O212	105.6 (3)
O320—N319—C318	118.7 (3)	O216—C213—C214	108.8 (4)
O320—N319—C323	115.2 (4)	O212—C213—C214	110.5 (3)
C318—N319—C323	122.8 (4)	O216—C213—C215	109.9 (4)
O325—N324—O326	122.7 (4)	O212—C213—C215	108.1 (4)
O325—N324—C301	118.6 (4)	C214—C213—C215	113.7 (4)
O326—N324—C301	118.7 (4)	C211—C217—O216	103.3 (3)
N24—C1—C2	117.8 (4)	C211—C217—C218	114.2 (3)
N24—C1—C6	119.7 (4)	O216—C217—C218	109.6 (3)
C2—C1—C6	122.5 (4)	C217—C218—N219	117.2 (4)

C1—C2—C3	120.4 (4)	C217—C218—O222	122.7 (4)
C2—C3—C4	119.1 (4)	N219—C218—O222	120.0 (4)
C3—C4—C5	120.8 (4)	N324—C301—C302	116.4 (4)
C4—C5—C6	121.9 (4)	N324—C301—C306	120.9 (4)
C5—C6—C1	115.2 (4)	C302—C301—C306	122.7 (4)
C5—C6—Se7	120.6 (3)	C301—C302—C303	120.1 (4)
C1—C6—Se7	124.2 (3)	C302—C303—C304	119.1 (5)
Se7—C8—C9	114.9 (3)	C303—C304—C305	121.6 (5)
C8—C9—C10	112.7 (3)	C304—C305—C306	120.9 (4)
C9—C10—C11	113.7 (3)	Se407—C306—C305	128.1 (3)
C10—C11—O12	107.6 (3)	Se407—C306—C301	116.0 (3)
C10—C11—C17	117.0 (3)	C305—C306—C301	115.5 (3)
O12—C11—C17	103.6 (3)	C305—C306—Se307	116.9 (3)
O16—C13—O12	105.9 (3)	C301—C306—Se307	127.2 (3)
O16—C13—C14	108.7 (3)	Se307—C308—C309	103.8 (4)
O12—C13—C14	110.9 (3)	C308—C309—C310	106.8 (5)
O16—C13—C15	110.1 (3)	C309—C310—C311	122.7 (5)
O12—C13—C15	107.4 (3)	C310—C311—O312	109.5 (3)
C14—C13—C15	113.5 (3)	C310—C311—C317	116.5 (4)
C11—C17—O16	104.5 (3)	O312—C311—C317	101.2 (3)
C11—C17—C18	113.0 (3)	O316—C313—O312	105.4 (3)
O16—C17—C18	109.3 (3)	O316—C313—C314	108.0 (3)
C17—C18—N19	115.5 (4)	O312—C313—C314	110.5 (3)
C17—C18—O22	123.5 (4)	O316—C313—C315	109.6 (3)
N19—C18—O22	120.9 (4)	O312—C313—C315	109.6 (3)
N124—C101—C102	115.9 (4)	C314—C313—C315	113.4 (4)
N124—C101—C106	120.5 (4)	C311—C317—O316	103.8 (3)
C102—C101—C106	123.6 (4)	C311—C317—C318	112.9 (3)
C101—C102—C103	118.4 (4)	O316—C317—C318	108.9 (3)
C102—C103—C104	119.9 (4)	C317—C318—N319	117.9 (3)
C103—C104—C105	120.4 (4)	C317—C318—O322	122.1 (4)
C104—C105—C106	122.3 (4)	N319—C318—O322	120.0 (4)
C101—C106—C105	115.5 (4)	Se407—C408—C309	121.2 (8)
C101—C106—Se107	124.0 (3)	Se407—C408—C409	115.8 (7)
C105—C106—Se107	120.5 (3)	C408—C409—C310	108.8 (7)

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supplementary materials

Crystal structure of $C_{17}H_{24}N_2O_6Se$ — ban0915

Andrew Lin, Martin G. Banwell and Anthony C. Willis

(ban0915)

Crystal data

$C_{17}H_{24}N_2O_6Se$	$Z = 4$
$M_r = 431.35$	$F_{000} = 888$
Triclinic, $P\overline{1}$	$D_x = 1.501$ Mg m^{-3}
$a = 8.0087$ (2) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 14.3159$ (5) Å	Cell parameters from 58776 reflections
$c = 17.0739$ (6) Å	$\theta = 2.6$–27.5°
$\alpha = 100.0775$ (14)°	$\mu = 2.00$ mm^{-1}
$\beta = 93.859$ (2)°	$T = 200$ K
$\gamma = 96.309$ (2)°	Rod, orange
$V = 1908.18$ (11) Å^3	0.30 × 0.10 × 0.08 mm

Data collection

Area diffractometer	12385 reflections with $I > 2.0\sigma(I)$
Monochromator: graphite	$R_{int} = 0.045$
$T = 200$ K	$\theta_{max} = 27.5^\circ$
φ and ω scans with CCD	$\theta_{min} = 2.6^\circ$
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	$h = -9 \rightarrow 10$
$T_{min} = 0.713$, $T_{max} = 0.881$	$k = -18 \rightarrow 18$
36797 measured reflections	$l = -22 \rightarrow 22$
16425 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.03P)^2 + 0.14P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.043$	$(\Delta/\sigma)_{max} = 0.038$
$wR(F^2) = 0.092$	$\Delta\rho_{max} = 0.94$ e Å^{-3}
$S = 1.00$	$\Delta\rho_{min} = -0.82$ e Å^{-3}
16425 reflections	Extinction correction: Larson (1970), Equation 22
967 parameters	Extinction coefficient: 92 (7)
42 restraints	Absolute structure: Flack (1983), 7715 Friedel-pairs

Primary atom site location: structure-invariant direct methods	Flack parameter: 0.000 (5)
Hydrogen site location: inferred from neighbouring sites	

Special details

Refinement. There is disorder in the molecule with atoms labelled X3xx. Besides the major locations for atoms Se307, C308 and C309 there are also alternative sites (labelled Se407, C408, C409) which correspond to an alternative conformation of this section of the molecule. Restraints were imposed on distances, angles and displacement parameters for disordered sites and the relative occupancies of the two conformations were refined.

Reflections with h+k=2n tend to be significantly stronger than those with h+k=2n+1, but the agreement factors for each group are in line with expectation for their respective average intensities.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Se7	0.50031 (8)	0.00013 (4)	0.59997 (4)	0.0452	
Se107	−0.00165 (7)	0.50869 (4)	0.57994 (4)	0.0434	
Se207	0.99976 (8)	0.24098 (5)	0.35189 (4)	0.0477	
Se307	0.4809 (3)	0.7305 (2)	0.35237 (11)	0.0472	0.573 (8)
Se407	0.5348 (6)	0.7602 (3)	0.34595 (16)	0.0540	0.427 (8)
O12	0.3683 (3)	0.27728 (18)	0.78221 (15)	0.0366	
O16	0.5531 (4)	0.3923 (2)	0.8629 (2)	0.0640	
O20	0.7591 (3)	0.17742 (19)	0.94061 (15)	0.0396	
O22	0.8272 (4)	0.3216 (2)	0.79962 (18)	0.0540	
O25	0.4346 (5)	0.0445 (3)	0.3499 (2)	0.0960	
O26	0.5804 (4)	0.0254 (3)	0.4548 (2)	0.0633	
O112	−0.1213 (3)	0.76845 (18)	0.77986 (15)	0.0350	
O116	0.0955 (3)	0.88330 (19)	0.82739 (16)	0.0428	
O120	0.2673 (3)	0.68955 (19)	0.94859 (16)	0.0390	
O122	0.3840 (4)	0.8108 (2)	0.79831 (18)	0.0530	
O125	−0.1414 (5)	0.5108 (3)	0.3234 (2)	0.0787	
O126	0.0340 (4)	0.5363 (3)	0.4295 (2)	0.0641	
O212	0.4615 (3)	0.13161 (18)	0.13481 (16)	0.0400	
O216	0.1842 (3)	0.1515 (2)	0.1192 (2)	0.0542	
O220	0.3267 (4)	0.4316 (2)	0.21762 (18)	0.0536	
O222	0.3005 (4)	0.2834 (2)	0.03151 (18)	0.0541	
O225	1.4377 (4)	0.2413 (3)	0.5239 (2)	0.0612	
O226	1.3164 (4)	0.2429 (3)	0.4080 (2)	0.0681	
O312	−0.0121 (3)	0.62587 (18)	0.12612 (15)	0.0375	
O316	−0.2880 (3)	0.62833 (19)	0.08516 (17)	0.0421	
O320	−0.3571 (4)	0.8865 (2)	0.1867 (2)	0.0627	
O322	−0.1282 (4)	0.7863 (2)	0.03370 (17)	0.0520	
O325	0.9351 (4)	0.7494 (3)	0.5244 (2)	0.0701	
O326	0.8161 (4)	0.7305 (3)	0.4052 (2)	0.0720	
N19	0.8559 (4)	0.2332 (2)	0.89526 (19)	0.0372	
N24	0.4453 (6)	0.0276 (3)	0.4178 (2)	0.0569	

N119	0.3793 (4)	0.7350 (2)	0.9028 (2)	0.0401
N124	−0.1104 (5)	0.5178 (3)	0.3965 (2)	0.0474
N219	0.2979 (5)	0.4034 (2)	0.13350 (19)	0.0446
N224	1.3107 (4)	0.2412 (3)	0.4797 (2)	0.0462
N319	−0.2482 (5)	0.8815 (2)	0.1247 (2)	0.0478
N324	0.8090 (4)	0.7386 (2)	0.4772 (3)	0.0457
C1	0.2889 (6)	0.0107 (3)	0.4556 (3)	0.0414
C2	0.1377 (6)	0.0099 (3)	0.4106 (3)	0.0542
C3	−0.0122 (6)	−0.0044 (4)	0.4430 (3)	0.0545
C4	−0.0113 (5)	−0.0185 (3)	0.5209 (3)	0.0486
C5	0.1387 (5)	−0.0179 (3)	0.5660 (3)	0.0420
C6	0.2962 (5)	−0.0028 (3)	0.5351 (3)	0.0401
C8	0.4089 (5)	−0.0222 (3)	0.6993 (2)	0.0424
C9	0.3542 (5)	0.0655 (3)	0.7503 (2)	0.0399
C10	0.5030 (5)	0.1378 (3)	0.7899 (2)	0.0408
C11	0.4514 (4)	0.2284 (3)	0.8377 (2)	0.0332
C13	0.4008 (5)	0.3778 (3)	0.8113 (2)	0.0382
C14	0.2611 (5)	0.4126 (3)	0.8592 (2)	0.0454
C15	0.4268 (6)	0.4253 (3)	0.7405 (3)	0.0563
C17	0.5958 (4)	0.3051 (3)	0.8819 (2)	0.0355
C18	0.7677 (5)	0.2881 (3)	0.8541 (3)	0.0363
C21	0.8235 (6)	0.2023 (3)	1.0231 (2)	0.0527
C23	0.9969 (5)	0.1888 (3)	0.8656 (3)	0.0513
C101	−0.2507 (5)	0.5029 (3)	0.4447 (2)	0.0383
C102	−0.4114 (6)	0.4951 (3)	0.4053 (3)	0.0499
C103	−0.5488 (5)	0.4803 (3)	0.4478 (3)	0.0527
C104	−0.5253 (5)	0.4733 (3)	0.5276 (3)	0.0471
C105	−0.3653 (5)	0.4811 (3)	0.5647 (2)	0.0402
C106	−0.2215 (5)	0.4958 (3)	0.5247 (2)	0.0345
C108	−0.0617 (5)	0.4801 (3)	0.6836 (2)	0.0419
C109	−0.1090 (5)	0.5624 (3)	0.7420 (2)	0.0383
C110	0.0392 (5)	0.6378 (3)	0.7759 (2)	0.0378
C111	−0.0142 (5)	0.7232 (3)	0.8294 (2)	0.0341
C113	−0.0801 (4)	0.8698 (3)	0.8012 (2)	0.0329
C114	−0.1844 (5)	0.9092 (3)	0.8677 (3)	0.0482
C115	−0.1026 (5)	0.9128 (3)	0.7276 (2)	0.0421
C117	0.1281 (5)	0.8049 (3)	0.8665 (2)	0.0350
C118	0.3054 (5)	0.7846 (3)	0.8506 (2)	0.0371
C121	0.3121 (6)	0.7261 (4)	1.0312 (2)	0.0538
C123	0.5280 (5)	0.6887 (3)	0.8877 (3)	0.0469
C201	1.1469 (5)	0.2383 (3)	0.5120 (3)	0.0388
C202	1.1450 (5)	0.2366 (3)	0.5931 (3)	0.0423
C203	0.9942 (6)	0.2326 (3)	0.6268 (3)	0.0468
C204	0.8454 (5)	0.2326 (3)	0.5795 (2)	0.0443
C205	0.8476 (5)	0.2350 (3)	0.5003 (2)	0.0404
C206	1.0002 (5)	0.2388 (3)	0.4628 (2)	0.0356
C208	0.7619 (5)	0.2463 (3)	0.3221 (3)	0.0438
C209	0.7532 (5)	0.2551 (3)	0.2338 (2)	0.0426
C210	0.5896 (5)	0.2863 (3)	0.2035 (3)	0.0418

C211	0.4373 (5)	0.2113 (3)	0.1941 (2)	0.0355	
C213	0.2997 (5)	0.0818 (3)	0.1058 (3)	0.0467	
C214	0.2532 (6)	0.0034 (3)	0.1530 (3)	0.0720	
C215	0.3011 (7)	0.0454 (4)	0.0169 (3)	0.0768	
C217	0.2704 (5)	0.2393 (3)	0.1603 (2)	0.0392	
C218	0.2923 (5)	0.3093 (3)	0.1026 (2)	0.0389	
C221	0.1838 (6)	0.4672 (4)	0.2486 (3)	0.0600	
C223	0.3345 (6)	0.4810 (3)	0.0909 (3)	0.0576	
C301	0.6438 (5)	0.7348 (3)	0.5085 (2)	0.0356	
C302	0.6408 (6)	0.7297 (3)	0.5882 (3)	0.0488	
C303	0.4909 (7)	0.7258 (3)	0.6223 (3)	0.0595	
C304	0.3453 (6)	0.7284 (3)	0.5764 (3)	0.0650	
C305	0.3455 (6)	0.7322 (3)	0.4966 (3)	0.0607	
C306	0.4982 (5)	0.7367 (3)	0.4592 (2)	0.0436	
C308	0.2453 (7)	0.7432 (5)	0.3272 (4)	0.0336	0.573 (8)
C309	0.2389 (9)	0.7626 (6)	0.2411 (4)	0.0379	0.573 (8)
C310	0.0571 (6)	0.7751 (4)	0.2177 (3)	0.0652	
C311	−0.0763 (5)	0.6920 (3)	0.1866 (2)	0.0427	
C313	−0.1514 (5)	0.5700 (3)	0.0780 (2)	0.0407	
C314	−0.2036 (6)	0.4800 (3)	0.1106 (3)	0.0586	
C315	−0.1102 (6)	0.5502 (3)	−0.0077 (3)	0.0531	
C317	−0.2350 (5)	0.7140 (3)	0.1407 (2)	0.0387	
C318	−0.1989 (5)	0.7961 (3)	0.0950 (2)	0.0375	
C321	−0.2752 (7)	0.9494 (4)	0.2555 (3)	0.0790	
C323	−0.2447 (8)	0.9588 (4)	0.0815 (3)	0.0718	
C408	0.3243 (12)	0.8017 (8)	0.3129 (7)	0.0630	0.427 (8)
C409	0.1943 (14)	0.7249 (8)	0.2654 (8)	0.0531	0.427 (8)
H21	0.1386	0.0200	0.3541	0.0670*	
H31	−0.1212	−0.0047	0.4107	0.0646*	
H41	−0.1206	−0.0292	0.5450	0.0570*	
H51	0.1355	−0.0285	0.6223	0.0493*	
H81	0.4977	−0.0464	0.7317	0.0508*	
H82	0.3086	−0.0721	0.6854	0.0508*	
H91	0.2875	0.0444	0.7930	0.0473*	
H92	0.2817	0.0972	0.7156	0.0473*	
H101	0.5719	0.1069	0.8267	0.0479*	
H102	0.5730	0.1560	0.7473	0.0479*	
H111	0.3711	0.2110	0.8767	0.0395*	
H141	0.2866	0.4831	0.8789	0.0556*	
H142	0.2518	0.3791	0.9057	0.0556*	
H143	0.1521	0.3986	0.8247	0.0556*	
H151	0.4500	0.4962	0.7588	0.0690*	
H152	0.5248	0.4017	0.7131	0.0690*	
H153	0.3230	0.4098	0.7024	0.0690*	
H171	0.5976	0.3076	0.9408	0.0415*	
H211	0.7551	0.1628	1.0551	0.0633*	
H212	0.9438	0.1897	1.0284	0.0633*	
H213	0.8166	0.2716	1.0430	0.0633*	
H231	1.0608	0.2311	0.8341	0.0626*	

H232	1.0727	0.1785	0.9115	0.0626*
H233	0.9555	0.1258	0.8305	0.0626*
H1021	−0.4264	0.5001	0.3477	0.0597*
H1031	−0.6655	0.4745	0.4211	0.0621*
H1041	−0.6253	0.4626	0.5584	0.0562*
H1051	−0.3518	0.4760	0.6224	0.0477*
H1081	0.0373	0.4562	0.7090	0.0504*
H1082	−0.1598	0.4287	0.6734	0.0504*
H1091	−0.1584	0.5366	0.7874	0.0461*
H1092	−0.1955	0.5935	0.7143	0.0461*
H1101	0.1216	0.6083	0.8077	0.0452*
H1102	0.0947	0.6601	0.7305	0.0452*
H1111	−0.0786	0.7016	0.8726	0.0401*
H1141	−0.1550	0.9801	0.8824	0.0595*
H1142	−0.1599	0.8796	0.9155	0.0595*
H1143	−0.3070	0.8937	0.8491	0.0595*
H1151	−0.0742	0.9838	0.7419	0.0510*
H1152	−0.2224	0.8968	0.7043	0.0510*
H1153	−0.0263	0.8863	0.6876	0.0510*
H1171	0.1198	0.8232	0.9253	0.0420*
H1211	0.2325	0.6936	1.0636	0.0644*
H1212	0.3056	0.7965	1.0424	0.0644*
H1213	0.4297	0.7138	1.0458	0.0644*
H1231	0.6007	0.7250	0.8550	0.0563*
H1232	0.5922	0.6871	0.9397	0.0563*
H1233	0.4941	0.6219	0.8580	0.0563*
H2021	1.2529	0.2382	0.6266	0.0500*
H2031	0.9912	0.2298	0.6848	0.0569*
H2041	0.7355	0.2307	0.6042	0.0531*
H2051	0.7388	0.2341	0.4676	0.0484*
H2081	0.7218	0.3031	0.3550	0.0526*
H2082	0.6918	0.1867	0.3294	0.0526*
H2091	0.8486	0.3031	0.2263	0.0506*
H2092	0.7661	0.1914	0.2014	0.0506*
H2101	0.5663	0.3435	0.2420	0.0502*
H2102	0.6052	0.3043	0.1502	0.0502*
H2111	0.4214	0.1901	0.2461	0.0440*
H2141	0.1392	−0.0309	0.1319	0.0857*
H2142	0.2513	0.0325	0.2106	0.0857*
H2143	0.3385	−0.0428	0.1476	0.0857*
H2151	0.1882	0.0100	−0.0049	0.0870*
H2152	0.3896	0.0014	0.0078	0.0870*
H2153	0.3265	0.1007	−0.0107	0.0870*
H2171	0.2053	0.2663	0.2051	0.0476*
H2211	0.2050	0.4869	0.3079	0.0756*
H2212	0.1596	0.5237	0.2246	0.0756*
H2213	0.0850	0.4163	0.2354	0.0756*
H2231	0.3116	0.4566	0.0322	0.0724*
H2232	0.4557	0.5082	0.1034	0.0724*

H2233	0.2614	0.5320	0.1076	0.0724*	
H3021	0.7484	0.7289	0.6213	0.0570*	
H3031	0.4878	0.7212	0.6799	0.0727*	
H3041	0.2361	0.7275	0.6015	0.0769*	
H3051	0.2364	0.7320	0.4643	0.0684*	
H3081	0.2089	0.7977	0.3644	0.0426*	0.573
H3082	0.1720	0.6829	0.3302	0.0426*	0.573
H3091	0.3155	0.8219	0.2385	0.0456*	0.573
H3092	0.2738	0.7072	0.2043	0.0456*	0.573
H3101	0.0152	0.8116	0.2663	0.0720*	0.573
H3102	0.0622	0.8152	0.1752	0.0720*	0.573
H3103	0.0113	0.8268	0.2542	0.0720*	0.427
H3104	0.1074	0.8020	0.1731	0.0720*	0.427
H3111	−0.1095	0.6592	0.2313	0.0517*	
H3141	−0.3019	0.4415	0.0760	0.0703*	
H3142	−0.2361	0.4980	0.1663	0.0703*	
H3143	−0.1070	0.4413	0.1110	0.0703*	
H3151	−0.2087	0.5108	−0.0415	0.0620*	
H3152	−0.0851	0.6120	−0.0267	0.0620*	
H3153	−0.0096	0.5146	−0.0117	0.0620*	
H3171	−0.3240	0.7281	0.1780	0.0474*	
H3211	−0.3518	0.9532	0.2996	0.0910*	
H3212	−0.2469	1.0145	0.2427	0.0910*	
H3213	−0.1693	0.9248	0.2728	0.0910*	
H3231	−0.1646	0.9491	0.0391	0.0905*	
H3232	−0.2066	1.0205	0.1190	0.0905*	
H3233	−0.3602	0.9607	0.0561	0.0905*	
H4081	0.2772	0.8314	0.3622	0.0704*	0.427
H4082	0.3513	0.8511	0.2790	0.0704*	0.427
H4091	0.1349	0.6882	0.3032	0.0578*	0.427
H4092	0.2485	0.6795	0.2269	0.0578*	0.427

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se7	0.0344 (2)	0.0453 (3)	0.0536 (4)	0.0048 (2)	0.0022 (2)	0.0037 (3)
Se107	0.0334 (2)	0.0463 (3)	0.0473 (3)	0.0052 (2)	0.0024 (2)	−0.0001 (3)
Se207	0.0359 (2)	0.0696 (4)	0.0402 (2)	0.0074 (2)	0.00191 (19)	0.0170 (2)
Se307	0.0416 (8)	0.0621 (11)	0.0385 (6)	0.0037 (6)	0.0011 (5)	0.0135 (6)
Se407	0.0658 (17)	0.0562 (15)	0.0393 (9)	0.0133 (12)	−0.0038 (10)	0.0060 (8)
O12	0.0370 (14)	0.0295 (15)	0.0411 (15)	0.0024 (11)	−0.0082 (12)	0.0057 (12)
O16	0.0491 (18)	0.0304 (16)	0.105 (3)	0.0029 (13)	−0.0326 (18)	0.0068 (17)
O20	0.0383 (15)	0.0435 (17)	0.0380 (15)	0.0029 (12)	0.0025 (12)	0.0119 (13)
O22	0.0572 (19)	0.056 (2)	0.0509 (19)	0.0002 (15)	0.0027 (15)	0.0222 (16)
O25	0.092 (3)	0.150 (4)	0.072 (3)	0.053 (3)	0.042 (2)	0.054 (3)
O26	0.047 (2)	0.076 (3)	0.066 (2)	0.0055 (18)	0.0162 (18)	0.0062 (19)
O112	0.0300 (13)	0.0315 (15)	0.0423 (15)	0.0067 (11)	−0.0064 (11)	0.0054 (12)
O116	0.0401 (15)	0.0363 (16)	0.0522 (17)	−0.0014 (12)	−0.0050 (12)	0.0163 (14)

O120	0.0344 (14)	0.0399 (17)	0.0429 (16)	−0.0013 (12)	−0.0006 (12)	0.0128 (13)
O122	0.0492 (18)	0.066 (2)	0.0486 (18)	0.0062 (15)	0.0153 (14)	0.0206 (16)
O125	0.087 (3)	0.114 (3)	0.047 (2)	0.040 (2)	0.0208 (19)	0.024 (2)
O126	0.059 (2)	0.072 (2)	0.056 (2)	−0.0040 (17)	0.0233 (17)	−0.0016 (18)
O212	0.0390 (15)	0.0329 (16)	0.0460 (16)	0.0052 (12)	−0.0053 (12)	0.0045 (13)
O216	0.0370 (16)	0.0443 (19)	0.081 (2)	−0.0029 (13)	−0.0113 (15)	0.0212 (17)
O220	0.061 (2)	0.054 (2)	0.0467 (18)	0.0203 (15)	−0.0007 (14)	0.0044 (15)
O222	0.083 (2)	0.0455 (19)	0.0359 (18)	0.0145 (16)	0.0019 (16)	0.0114 (15)
O225	0.0334 (18)	0.068 (2)	0.081 (3)	0.0091 (16)	−0.0138 (17)	0.016 (2)
O226	0.042 (2)	0.102 (3)	0.062 (2)	0.0100 (18)	0.0115 (16)	0.019 (2)
O312	0.0349 (14)	0.0347 (15)	0.0413 (15)	0.0070 (11)	−0.0043 (11)	0.0040 (12)
O316	0.0357 (15)	0.0343 (16)	0.0545 (18)	0.0034 (12)	−0.0088 (13)	0.0092 (14)
O320	0.072 (2)	0.056 (2)	0.064 (2)	0.0233 (18)	0.0249 (18)	0.0068 (17)
O322	0.077 (2)	0.0400 (18)	0.0454 (18)	0.0184 (15)	0.0221 (16)	0.0129 (14)
O325	0.0347 (19)	0.077 (3)	0.097 (3)	0.0093 (17)	−0.0076 (19)	0.013 (2)
O326	0.062 (2)	0.093 (3)	0.064 (3)	0.012 (2)	0.0211 (19)	0.018 (2)
N19	0.0275 (16)	0.043 (2)	0.0424 (19)	0.0029 (14)	0.0017 (13)	0.0123 (16)
N24	0.063 (3)	0.062 (3)	0.054 (3)	0.022 (2)	0.024 (2)	0.017 (2)
N119	0.0304 (17)	0.050 (2)	0.044 (2)	0.0070 (15)	0.0014 (14)	0.0168 (17)
N124	0.051 (2)	0.050 (2)	0.042 (2)	0.0150 (18)	0.0131 (18)	0.0012 (18)
N219	0.066 (2)	0.036 (2)	0.036 (2)	0.0186 (17)	0.0086 (17)	0.0088 (16)
N224	0.0287 (19)	0.043 (2)	0.066 (3)	0.0062 (15)	0.0016 (17)	0.009 (2)
N319	0.071 (3)	0.033 (2)	0.045 (2)	0.0160 (17)	0.0206 (18)	0.0081 (17)
N324	0.036 (2)	0.032 (2)	0.069 (3)	0.0029 (15)	0.0028 (19)	0.0100 (19)
C1	0.046 (2)	0.037 (2)	0.044 (2)	0.0135 (19)	0.0103 (19)	0.0046 (19)
C2	0.059 (3)	0.059 (3)	0.049 (3)	0.026 (2)	0.006 (2)	0.010 (2)
C3	0.054 (3)	0.060 (3)	0.047 (3)	0.017 (2)	−0.002 (2)	0.001 (2)
C4	0.034 (2)	0.048 (3)	0.061 (3)	0.0013 (18)	0.0028 (19)	0.004 (2)
C5	0.035 (2)	0.042 (2)	0.047 (2)	−0.0029 (17)	−0.0009 (18)	0.007 (2)
C6	0.035 (2)	0.033 (2)	0.051 (3)	0.0032 (17)	0.0028 (18)	0.0040 (19)
C8	0.052 (2)	0.036 (2)	0.040 (2)	0.0056 (18)	−0.0062 (19)	0.0095 (19)
C9	0.039 (2)	0.033 (2)	0.046 (2)	0.0012 (17)	−0.0033 (18)	0.0085 (19)
C10	0.035 (2)	0.032 (2)	0.053 (2)	0.0087 (16)	−0.0053 (17)	0.0014 (19)
C11	0.0254 (19)	0.037 (2)	0.037 (2)	0.0060 (16)	−0.0067 (15)	0.0089 (18)
C13	0.032 (2)	0.033 (2)	0.047 (2)	−0.0013 (16)	−0.0035 (17)	0.0090 (19)
C14	0.050 (2)	0.040 (2)	0.050 (3)	0.0084 (19)	0.0081 (19)	0.013 (2)
C15	0.069 (3)	0.048 (3)	0.056 (3)	0.002 (2)	0.019 (2)	0.019 (2)
C17	0.031 (2)	0.033 (2)	0.039 (2)	0.0066 (16)	−0.0053 (16)	0.0001 (18)
C18	0.035 (2)	0.035 (2)	0.036 (2)	0.0001 (17)	−0.0025 (18)	0.004 (2)
C21	0.068 (3)	0.054 (3)	0.036 (2)	0.009 (2)	−0.006 (2)	0.012 (2)
C23	0.038 (2)	0.043 (3)	0.075 (3)	0.0084 (19)	0.015 (2)	0.011 (2)
C101	0.049 (3)	0.031 (2)	0.035 (2)	0.0099 (18)	0.0123 (18)	0.0013 (18)
C102	0.053 (3)	0.053 (3)	0.044 (3)	0.017 (2)	−0.005 (2)	0.006 (2)
C103	0.039 (2)	0.055 (3)	0.061 (3)	0.011 (2)	−0.006 (2)	0.005 (2)
C104	0.033 (2)	0.049 (3)	0.059 (3)	0.0032 (18)	0.0036 (19)	0.010 (2)
C105	0.039 (2)	0.040 (2)	0.040 (2)	0.0002 (18)	0.0024 (18)	0.0069 (19)
C106	0.038 (2)	0.027 (2)	0.037 (2)	0.0035 (16)	0.0029 (17)	0.0011 (17)
C108	0.049 (2)	0.040 (2)	0.038 (2)	0.0166 (19)	−0.0043 (18)	0.0043 (19)
C109	0.043 (2)	0.032 (2)	0.040 (2)	0.0018 (17)	−0.0002 (17)	0.0101 (18)

C110	0.036 (2)	0.036 (2)	0.041 (2)	0.0073 (16)	−0.0009 (16)	0.0055 (18)
C111	0.032 (2)	0.033 (2)	0.036 (2)	−0.0001 (16)	−0.0077 (17)	0.0074 (18)
C113	0.035 (2)	0.032 (2)	0.030 (2)	0.0014 (16)	0.0011 (15)	0.0048 (17)
C114	0.059 (3)	0.038 (2)	0.051 (3)	0.014 (2)	0.015 (2)	0.009 (2)
C115	0.050 (3)	0.042 (3)	0.035 (2)	0.013 (2)	−0.0006 (18)	0.0079 (19)
C117	0.034 (2)	0.035 (2)	0.035 (2)	0.0001 (16)	−0.0036 (16)	0.0100 (18)
C118	0.033 (2)	0.041 (3)	0.035 (2)	−0.0013 (17)	−0.0029 (17)	0.0055 (19)
C121	0.058 (3)	0.068 (3)	0.035 (2)	0.002 (2)	−0.003 (2)	0.014 (2)
C123	0.038 (2)	0.043 (3)	0.060 (3)	0.0094 (18)	0.0084 (19)	0.006 (2)
C201	0.033 (2)	0.030 (2)	0.051 (3)	0.0017 (16)	−0.0038 (18)	0.006 (2)
C202	0.047 (2)	0.037 (2)	0.041 (2)	0.0076 (19)	−0.0088 (19)	0.006 (2)
C203	0.058 (3)	0.046 (3)	0.038 (2)	0.014 (2)	0.000 (2)	0.009 (2)
C204	0.048 (2)	0.041 (3)	0.044 (2)	0.0125 (19)	0.0113 (19)	0.001 (2)
C205	0.033 (2)	0.042 (2)	0.046 (2)	0.0089 (17)	−0.0004 (17)	0.007 (2)
C206	0.0276 (19)	0.035 (2)	0.045 (2)	0.0035 (15)	−0.0015 (16)	0.0096 (18)
C208	0.039 (2)	0.046 (3)	0.047 (3)	0.0065 (19)	−0.0067 (19)	0.012 (2)
C209	0.041 (2)	0.049 (3)	0.037 (2)	0.0021 (19)	−0.0037 (18)	0.011 (2)
C210	0.044 (2)	0.036 (2)	0.045 (2)	0.0067 (18)	−0.0042 (19)	0.010 (2)
C211	0.038 (2)	0.037 (2)	0.035 (2)	0.0108 (17)	0.0034 (16)	0.0121 (18)
C213	0.049 (2)	0.030 (2)	0.058 (3)	0.0014 (18)	−0.016 (2)	0.009 (2)
C214	0.070 (3)	0.045 (3)	0.099 (4)	−0.011 (3)	−0.021 (3)	0.032 (3)
C215	0.086 (4)	0.058 (3)	0.075 (4)	0.005 (3)	−0.032 (3)	−0.004 (3)
C217	0.038 (2)	0.039 (2)	0.042 (2)	0.0059 (18)	0.0029 (17)	0.0116 (19)
C218	0.034 (2)	0.045 (3)	0.042 (2)	0.0093 (17)	0.0038 (17)	0.014 (2)
C221	0.059 (3)	0.067 (3)	0.062 (3)	0.034 (2)	0.017 (2)	0.016 (3)
C223	0.064 (3)	0.048 (3)	0.069 (3)	0.013 (2)	0.007 (3)	0.029 (3)
C301	0.029 (2)	0.031 (2)	0.047 (2)	0.0069 (16)	0.0033 (17)	0.0028 (18)
C302	0.054 (3)	0.037 (3)	0.051 (3)	0.004 (2)	−0.009 (2)	0.002 (2)
C303	0.072 (3)	0.045 (3)	0.064 (3)	0.008 (2)	0.028 (3)	0.009 (2)
C304	0.056 (3)	0.050 (3)	0.087 (4)	0.007 (2)	0.025 (3)	−0.003 (3)
C305	0.033 (2)	0.048 (3)	0.090 (4)	0.012 (2)	−0.006 (2)	−0.017 (3)
C306	0.037 (2)	0.038 (2)	0.049 (3)	0.0120 (18)	−0.0097 (18)	−0.007 (2)
C308	0.026 (3)	0.031 (4)	0.042 (4)	0.000 (3)	−0.007 (3)	0.007 (3)
C309	0.036 (4)	0.032 (5)	0.040 (5)	−0.012 (3)	−0.003 (3)	0.005 (3)
C310	0.054 (3)	0.059 (3)	0.066 (3)	0.014 (2)	−0.024 (2)	−0.024 (3)
C311	0.050 (2)	0.045 (3)	0.035 (2)	0.0124 (19)	0.0031 (18)	0.0075 (19)
C313	0.043 (2)	0.030 (2)	0.049 (2)	0.0027 (17)	−0.0084 (18)	0.0133 (19)
C314	0.062 (3)	0.028 (2)	0.086 (4)	0.001 (2)	−0.007 (3)	0.018 (2)
C315	0.057 (3)	0.052 (3)	0.045 (3)	0.009 (2)	−0.005 (2)	−0.002 (2)
C317	0.042 (2)	0.036 (2)	0.041 (2)	0.0081 (18)	0.0031 (18)	0.0116 (19)
C318	0.045 (2)	0.032 (2)	0.036 (2)	0.0073 (17)	0.0010 (18)	0.0074 (18)
C321	0.082 (4)	0.091 (4)	0.055 (3)	0.015 (3)	0.009 (3)	−0.018 (3)
C323	0.109 (5)	0.044 (3)	0.073 (4)	0.026 (3)	0.026 (3)	0.024 (3)
C408	0.063 (6)	0.057 (7)	0.066 (6)	0.007 (5)	−0.003 (5)	0.008 (5)
C409	0.048 (6)	0.048 (7)	0.057 (7)	0.004 (5)	−0.001 (5)	−0.005 (6)

Geometric parameters (Å, °)

Se7—C6	1.905 (4)	C108—H1082	1.000

Se7—C8	1.954 (4)	C109—C110	1.522 (5)
Se107—C106	1.915 (4)	C109—H1091	1.000
Se107—C108	1.964 (4)	C109—H1092	1.000
Se207—C206	1.899 (4)	C110—C111	1.513 (5)
Se207—C208	1.952 (4)	C110—H1101	1.000
Se307—C306	1.806 (4)	C110—H1102	1.000
Se307—C308	1.942 (5)	C111—C117	1.556 (5)
Se407—C306	2.054 (5)	C111—H1111	1.000
Se407—C408	1.930 (8)	C113—C114	1.520 (5)
O12—C11	1.442 (4)	C113—C115	1.502 (5)
O12—C13	1.426 (4)	C114—H1141	1.000
O16—C13	1.429 (4)	C114—H1142	1.000
O16—C17	1.414 (4)	C114—H1143	1.000
O20—N19	1.414 (4)	C115—H1151	1.000
O20—C21	1.436 (5)	C115—H1152	1.000
O22—C18	1.220 (5)	C115—H1153	1.000
O25—N24	1.225 (5)	C117—C118	1.513 (5)
O26—N24	1.223 (5)	C117—H1171	1.000
O112—C111	1.444 (4)	C121—H1211	1.000
O112—C113	1.429 (4)	C121—H1212	1.000
O116—C113	1.428 (4)	C121—H1213	1.000
O116—C117	1.441 (4)	C123—H1231	1.000
O120—N119	1.409 (4)	C123—H1232	1.000
O120—C121	1.423 (5)	C123—H1233	1.000
O122—C118	1.215 (5)	C201—C202	1.389 (6)
O125—N124	1.240 (5)	C201—C206	1.399 (5)
O126—N124	1.232 (5)	C202—C203	1.372 (6)
O212—C211	1.426 (5)	C202—H2021	1.000
O212—C213	1.422 (5)	C203—C204	1.394 (6)
O216—C213	1.434 (5)	C203—H2031	1.000
O216—C217	1.409 (5)	C204—C205	1.361 (6)
O220—N219	1.418 (4)	C204—H2041	1.000
O220—C221	1.401 (5)	C205—C206	1.417 (5)
O222—C218	1.215 (5)	C205—H2051	1.000
O225—N224	1.224 (4)	C208—C209	1.533 (6)
O226—N224	1.233 (5)	C208—H2081	1.000
O312—C311	1.436 (5)	C208—H2082	1.000
O312—C313	1.418 (4)	C209—C210	1.518 (5)
O316—C313	1.446 (5)	C209—H2091	1.000
O316—C317	1.417 (5)	C209—H2092	1.000
O320—N319	1.413 (4)	C210—C211	1.514 (5)
O320—C321	1.421 (6)	C210—H2101	1.000
O322—C318	1.217 (4)	C210—H2102	1.000
O325—N324	1.227 (5)	C211—C217	1.541 (5)
O326—N324	1.221 (5)	C211—H2111	1.000
N19—C18	1.364 (5)	C213—C214	1.521 (6)
N19—C23	1.435 (5)	C213—C215	1.517 (7)
N24—C1	1.463 (5)	C214—H2141	1.000
N119—C118	1.374 (5)	C214—H2142	1.000

N119—C123	1.442 (5)	C214—H2143	1.000
N124—C101	1.455 (5)	C215—H2151	1.000
N219—C218	1.353 (5)	C215—H2152	1.000
N219—C223	1.446 (5)	C215—H2153	1.000
N224—C201	1.456 (5)	C217—C218	1.527 (5)
N319—C318	1.351 (5)	C217—H2171	1.000
N319—C323	1.433 (6)	C221—H2211	1.000
N324—C301	1.459 (5)	C221—H2212	1.000
C1—C2	1.388 (6)	C221—H2213	1.000
C1—C6	1.402 (6)	C223—H2231	1.000
C2—C3	1.365 (7)	C223—H2232	1.000
C2—H21	1.000	C223—H2233	1.000
C3—C4	1.380 (6)	C301—C302	1.378 (6)
C3—H31	1.000	C301—C306	1.397 (5)
C4—C5	1.381 (6)	C302—C303	1.369 (6)
C4—H41	1.000	C302—H3021	1.000
C5—C6	1.409 (5)	C303—C304	1.368 (7)
C5—H51	1.000	C303—H3031	1.000
C8—C9	1.523 (5)	C304—C305	1.373 (7)
C8—H81	1.000	C304—H3041	1.000
C8—H82	1.000	C305—C306	1.417 (6)
C9—C10	1.525 (5)	C305—H3051	1.000
C9—H91	1.000	C308—C309	1.543 (9)
C9—H92	1.000	C308—H3081	1.000
C10—C11	1.520 (5)	C308—H3082	1.000
C10—H101	1.000	C309—C310	1.521 (8)
C10—H102	1.000	C309—H3091	1.000
C11—C17	1.558 (5)	C309—H3092	1.000
C11—H111	1.000	C310—C311	1.505 (6)
C13—C14	1.508 (5)	C310—C409	1.627 (11)
C13—C15	1.503 (6)	C310—H3101	1.000
C14—H141	1.000	C310—H3102	1.000
C14—H142	1.000	C310—H3103	1.000
C14—H143	1.000	C310—H3104	1.000
C15—H151	1.000	C311—C317	1.539 (5)
C15—H152	1.000	C311—H3111	1.000
C15—H153	1.000	C313—C314	1.519 (5)
C17—C18	1.517 (5)	C313—C315	1.507 (6)
C17—H171	1.000	C314—H3141	1.000
C21—H211	1.000	C314—H3142	1.000
C21—H212	1.000	C314—H3143	1.000
C21—H213	1.000	C315—H3151	1.000
C23—H231	1.000	C315—H3152	1.000
C23—H232	1.000	C315—H3153	1.000
C23—H233	1.000	C317—C318	1.534 (5)
C101—C102	1.395 (6)	C317—H3171	1.000
C101—C106	1.393 (5)	C321—H3211	1.000
C102—C103	1.375 (6)	C321—H3212	1.000
C102—H1021	1.000	C321—H3213	1.000

C103—C104	1.386 (6)	C323—H3231	1.000
C103—H1031	1.000	C323—H3232	1.000
C104—C105	1.374 (5)	C323—H3233	1.000
C104—H1041	1.000	C408—C409	1.508 (13)
C105—C106	1.393 (5)	C408—H4081	0.992
C105—H1051	1.000	C408—H4082	1.004
C108—C109	1.504 (5)	C409—H4091	1.011
C108—H1081	1.000	C409—H4092	0.997
O12···C202^{i}	3.507 (5)	O225···C6^{ii}	3.595 (6)
O12···C23^{i}	3.585 (5)	O226···N24^{ii}	3.384 (6)
O22···C108^{ii}	3.351 (5)	O226···C103^{viii}	3.386 (6)
O22···C203	3.422 (6)	O226···C2^{ii}	3.491 (6)
O22···C14^{ii}	3.588 (5)	O226···C1^{ii}	3.545 (6)
O25···O226^{i}	3.111 (6)	O312···C121^{vii}	3.462 (5)
O25···C321^{iii}	3.207 (7)	O316···C223^{i}	3.508 (5)
O25···C408^{iv}	3.43 (1)	O322···C123^{vi}	3.572 (5)
O25···C214	3.496 (6)	O325···C4^{ix}	3.315 (6)
O25···N224^{i}	3.543 (5)	O325···C305^{ii}	3.382 (6)
O26···C3^{ii}	3.352 (6)	O325···C304^{ii}	3.404 (6)
O26···C205	3.426 (5)	O325···C5^{ix}	3.476 (6)
O26···O225^{i}	3.454 (6)	O326···N124^{ii}	3.145 (6)
O26···C4^{ii}	3.545 (5)	O326···C101^{ii}	3.434 (6)
O112···C302^{i}	3.597 (5)	N24···N224^{i}	3.373 (6)
O122···C8^{v}	3.157 (5)	N24···C408^{iv}	3.41 (1)
O122···C303	3.247 (6)	N124···N324^{i}	3.369 (5)
O125···C221	3.055 (7)	N224···C1^{ii}	3.237 (6)
O125···O326^{i}	3.274 (6)	N224···C104^{viii}	3.373 (6)
O125···C314	3.576 (6)	N224···C2^{ii}	3.418 (6)
O126···O325^{i}	3.396 (6)	N324···C101^{ii}	3.298 (5)
O126···C221	3.413 (6)	N324···C4^{ix}	3.545 (5)
O126···O326^{i}	3.510 (6)	C1···C408^{iv}	3.56 (1)
O126···C305	3.515 (5)	C1···C201^{i}	3.564 (6)
O126···C103^{ii}	3.527 (5)	C2···C201^{i}	3.412 (6)
O126···N324^{i}	3.580 (5)	C2···C206^{i}	3.556 (6)
O216···C21^{vi}	3.436 (5)	C3···C206^{i}	3.428 (7)
O222···C23^{vi}	3.575 (5)	C3···C201^{i}	3.532 (7)
O222···C11^{vii}	3.587 (5)	C5···C202^{i}	3.586 (6)
O225···N24^{ii}	3.280 (5)	C101···C301^{i}	3.521 (6)
O225···C104^{viii}	3.291 (6)	C102···C301^{i}	3.539 (6)
O225···C205^{ii}	3.343 (5)	C102···C306^{i}	3.586 (6)
O225···C1^{ii}	3.359 (6)	C105···C302^{i}	3.506 (6)
O225···C204^{ii}	3.360 (5)	C108···C203^{i}	3.592 (6)

O225···C105^{viii}	3.547 (6)	C115···C302^{i}	3.568 (5)
C6—Se7—C8	100.01 (17)	O120—C121—H1211	109.5
C106—Se107—C108	100.24 (17)	O120—C121—H1212	109.5
C206—Se207—C208	101.90 (17)	H1211—C121—H1212	109.5
C306—Se307—C308	103.6 (3)	O120—C121—H1213	109.5
C306—Se407—C408	102.6 (4)	H1211—C121—H1213	109.5
C11—O12—C13	108.9 (3)	H1212—C121—H1213	109.5
C13—O16—C17	111.4 (3)	N119—C123—H1231	109.5
N19—O20—C21	109.5 (3)	N119—C123—H1232	109.5
C111—O112—C113	109.1 (3)	H1231—C123—H1232	109.5
C113—O116—C117	107.3 (3)	N119—C123—H1233	109.5
N119—O120—C121	109.7 (3)	H1231—C123—H1233	109.5
C211—O212—C213	107.5 (3)	H1232—C123—H1233	109.5
C213—O216—C217	110.0 (3)	N224—C201—C202	116.9 (4)
N219—O220—C221	109.9 (3)	N224—C201—C206	120.6 (4)
C311—O312—C313	108.0 (3)	C202—C201—C206	122.5 (4)
C313—O316—C317	109.5 (3)	C201—C202—C203	119.7 (4)
N319—O320—C321	109.1 (4)	C201—C202—H2021	120.2
O20—N19—C18	115.3 (3)	C203—C202—H2021	120.2
O20—N19—C23	112.8 (3)	C202—C203—C204	119.4 (4)
C18—N19—C23	123.1 (3)	C202—C203—H2031	120.3
O25—N24—O26	122.6 (4)	C204—C203—H2031	120.3
O25—N24—C1	118.0 (4)	C203—C204—C205	120.8 (4)
O26—N24—C1	119.4 (4)	C203—C204—H2041	119.6
O120—N119—C118	115.1 (3)	C205—C204—H2041	119.6
O120—N119—C123	114.2 (3)	C204—C205—C206	121.7 (4)
C118—N119—C123	123.5 (3)	C204—C205—H2051	119.2
O125—N124—O126	122.5 (4)	C206—C205—H2051	119.1
O125—N124—C101	118.3 (4)	C205—C206—C201	115.9 (4)
O126—N124—C101	119.1 (4)	C205—C206—Se207	120.9 (3)
O220—N219—C218	117.5 (3)	C201—C206—Se207	123.3 (3)
O220—N219—C223	113.4 (4)	Se207—C208—C209	104.9 (3)
C218—N219—C223	125.6 (4)	Se207—C208—H2081	110.6
O226—N224—O225	122.1 (4)	C209—C208—H2081	110.6
O226—N224—C201	118.4 (3)	Se207—C208—H2082	110.6
O225—N224—C201	119.5 (4)	C209—C208—H2082	110.6
O320—N319—C318	118.7 (3)	H2081—C208—H2082	109.5
O320—N319—C323	115.2 (4)	C208—C209—C210	113.9 (3)
C318—N319—C323	122.8 (4)	C208—C209—H2091	108.3
O325—N324—O326	122.7 (4)	C210—C209—H2091	108.4
O325—N324—C301	118.6 (4)	C208—C209—H2092	108.4
O326—N324—C301	118.7 (4)	C210—C209—H2092	108.3
N24—C1—C2	117.8 (4)	H2091—C209—H2092	109.5
N24—C1—C6	119.7 (4)	C209—C210—C211	114.9 (3)
C2—C1—C6	122.5 (4)	C209—C210—H2101	108.1
C1—C2—C3	120.4 (4)	C211—C210—H2101	108.1
C1—C2—H21	119.8	C209—C210—H2102	108.1
C3—C2—H21	119.8	C211—C210—H2102	108.1

C2—C3—C4	119.1 (4)	H2101—C210—H2102	109.5
C2—C3—H31	120.5	C210—C211—O212	109.4 (3)
C4—C3—H31	120.4	C210—C211—C217	116.2 (3)
C3—C4—C5	120.8 (4)	O212—C211—C217	101.5 (3)
C3—C4—H41	119.6	C210—C211—H2111	109.8
C5—C4—H41	119.6	O212—C211—H2111	109.8
C4—C5—C6	121.9 (4)	C217—C211—H2111	109.8
C4—C5—H51	119.0	O216—C213—O212	105.6 (3)
C6—C5—H51	119.0	O216—C213—C214	108.8 (4)
C5—C6—C1	115.2 (4)	O212—C213—C214	110.5 (3)
C5—C6—Se7	120.6 (3)	O216—C213—C215	109.9 (4)
C1—C6—Se7	124.2 (3)	O212—C213—C215	108.1 (4)
Se7—C8—C9	114.9 (3)	C214—C213—C215	113.7 (4)
Se7—C8—H81	108.1	C213—C214—H2141	109.5
C9—C8—H81	108.1	C213—C214—H2142	109.5
Se7—C8—H82	108.1	H2141—C214—H2142	109.5
C9—C8—H82	108.1	C213—C214—H2143	109.4
H81—C8—H82	109.5	H2141—C214—H2143	109.5
C8—C9—C10	112.7 (3)	H2142—C214—H2143	109.5
C8—C9—H91	108.6	C213—C215—H2151	109.5
C10—C9—H91	108.7	C213—C215—H2152	109.4
C8—C9—H92	108.7	H2151—C215—H2152	109.5
C10—C9—H92	108.7	C213—C215—H2153	109.5
H91—C9—H92	109.5	H2151—C215—H2153	109.5
C9—C10—C11	113.7 (3)	H2152—C215—H2153	109.5
C9—C10—H101	108.4	C211—C217—O216	103.3 (3)
C11—C10—H101	108.4	C211—C217—C218	114.2 (3)
C9—C10—H102	108.4	O216—C217—C218	109.6 (3)
C11—C10—H102	108.4	C211—C217—H2171	109.9
H101—C10—H102	109.5	O216—C217—H2171	109.8
C10—C11—O12	107.6 (3)	C218—C217—H2171	109.9
C10—C11—C17	117.0 (3)	C217—C218—N219	117.2 (4)
O12—C11—C17	103.6 (3)	C217—C218—O222	122.7 (4)
C10—C11—H111	109.4	N219—C218—O222	120.0 (4)
O12—C11—H111	109.5	O220—C221—H2211	109.5
C17—C11—H111	109.4	O220—C221—H2212	109.5
O16—C13—O12	105.9 (3)	H2211—C221—H2212	109.5
O16—C13—C14	108.7 (3)	O220—C221—H2213	109.5
O12—C13—C14	110.9 (3)	H2211—C221—H2213	109.5
O16—C13—C15	110.1 (3)	H2212—C221—H2213	109.5
O12—C13—C15	107.4 (3)	N219—C223—H2231	109.5
C14—C13—C15	113.5 (3)	N219—C223—H2232	109.4
C13—C14—H141	109.5	H2231—C223—H2232	109.5
C13—C14—H142	109.5	N219—C223—H2233	109.5
H141—C14—H142	109.5	H2231—C223—H2233	109.5
C13—C14—H143	109.5	H2232—C223—H2233	109.5
H141—C14—H143	109.5	N324—C301—C302	116.4 (4)
H142—C14—H143	109.5	N324—C301—C306	120.9 (4)
C13—C15—H151	109.5	C302—C301—C306	122.7 (4)

C13—C15—H152	109.5	C301—C302—C303	120.1 (4)
H151—C15—H152	109.5	C301—C302—H3021	119.9
C13—C15—H153	109.5	C303—C302—H3021	119.9
H151—C15—H153	109.5	C302—C303—C304	119.1 (5)
H152—C15—H153	109.5	C302—C303—H3031	120.4
C11—C17—O16	104.5 (3)	C304—C303—H3031	120.5
C11—C17—C18	113.0 (3)	C303—C304—C305	121.6 (5)
O16—C17—C18	109.3 (3)	C303—C304—H3041	119.2
C11—C17—H171	110.0	C305—C304—H3041	119.2
O16—C17—H171	109.9	C304—C305—C306	120.9 (4)
C18—C17—H171	109.9	C304—C305—H3051	119.6
C17—C18—N19	115.5 (4)	C306—C305—H3051	119.5
C17—C18—O22	123.5 (4)	Se407—C306—C305	128.1 (3)
N19—C18—O22	120.9 (4)	Se407—C306—C301	116.0 (3)
O20—C21—H211	109.5	C305—C306—C301	115.5 (3)
O20—C21—H212	109.5	C305—C306—Se307	116.9 (3)
H211—C21—H212	109.5	C301—C306—Se307	127.2 (3)
O20—C21—H213	109.5	Se307—C308—C309	103.8 (4)
H211—C21—H213	109.5	Se307—C308—H3081	111.0
H212—C21—H213	109.5	C309—C308—H3081	110.7
N19—C23—H231	109.5	Se307—C308—H3082	110.6
N19—C23—H232	109.5	C309—C308—H3082	111.1
H231—C23—H232	109.5	C308—C309—C310	106.8 (5)
N19—C23—H233	109.5	C308—C309—H3091	110.4
H231—C23—H233	109.5	C310—C309—H3091	110.3
H232—C23—H233	109.5	C308—C309—H3092	109.9
N124—C101—C102	115.9 (4)	C310—C309—H3092	110.0
N124—C101—C106	120.5 (4)	H3091—C309—H3092	109.5
C102—C101—C106	123.6 (4)	C309—C310—C311	122.7 (5)
C101—C102—C103	118.4 (4)	C309—C310—H3101	106.2
C101—C102—H1021	120.8	C311—C310—H3101	106.1
C103—C102—H1021	120.8	C309—C310—H3102	105.9
C102—C103—C104	119.9 (4)	C311—C310—H3102	106.1
C102—C103—H1031	120.1	H3101—C310—H3102	109.5
C104—C103—H1031	120.1	C409—C310—H3103	111.6
C103—C104—C105	120.4 (4)	C409—C310—H3104	111.1
C103—C104—H1041	119.8	H3103—C310—H3104	109.5
C105—C104—H1041	119.8	C310—C311—O312	109.5 (3)
C104—C105—C106	122.3 (4)	C310—C311—C317	116.5 (4)
C104—C105—H1051	118.8	O312—C311—C317	101.2 (3)
C106—C105—H1051	118.9	C310—C311—H3111	109.7
C101—C106—C105	115.5 (4)	O312—C311—H3111	109.7
C101—C106—Se107	124.0 (3)	C317—C311—H3111	109.7
C105—C106—Se107	120.5 (3)	O316—C313—O312	105.4 (3)
Se107—C108—C109	116.0 (3)	O316—C313—C314	108.0 (3)
Se107—C108—H1081	107.8	O312—C313—C314	110.5 (3)
C109—C108—H1081	107.8	O316—C313—C315	109.6 (3)
Se107—C108—H1082	107.8	O312—C313—C315	109.6 (3)
C109—C108—H1082	107.8	C314—C313—C315	113.4 (4)

H1081—C108—H1082	109.5	C313—C314—H3141	109.5
C108—C109—C110	113.6 (3)	C313—C314—H3142	109.5
C108—C109—H1091	108.4	H3141—C314—H3142	109.5
C110—C109—H1091	108.4	C313—C314—H3143	109.4
C108—C109—H1092	108.4	H3141—C314—H3143	109.5
C110—C109—H1092	108.4	H3142—C314—H3143	109.5
H1091—C109—H1092	109.5	C313—C315—H3151	109.5
C109—C110—C111	112.4 (3)	C313—C315—H3152	109.5
C109—C110—H1101	108.7	H3151—C315—H3152	109.5
C111—C110—H1101	108.7	C313—C315—H3153	109.5
C109—C110—H1102	108.7	H3151—C315—H3153	109.5
C111—C110—H1102	108.8	H3152—C315—H3153	109.5
H1101—C110—H1102	109.5	C311—C317—O316	103.8 (3)
C110—C111—O112	106.9 (3)	C311—C317—C318	112.9 (3)
C110—C111—C117	116.5 (3)	O316—C317—C318	108.9 (3)
O112—C111—C117	103.5 (3)	C311—C317—H3171	110.3
C110—C111—H1111	109.9	O316—C317—H3171	110.3
O112—C111—H1111	109.9	C318—C317—H3171	110.3
C117—C111—H1111	109.9	C317—C318—N319	117.9 (3)
O112—C113—O116	104.3 (3)	C317—C318—O322	122.1 (4)
O112—C113—C114	109.9 (3)	N319—C318—O322	120.0 (4)
O116—C113—C114	111.4 (3)	O320—C321—H3211	109.4
O112—C113—C115	108.8 (3)	O320—C321—H3212	109.5
O116—C113—C115	108.2 (3)	H3211—C321—H3212	109.5
C114—C113—C115	113.7 (3)	O320—C321—H3213	109.4
C113—C114—H1141	109.5	H3211—C321—H3213	109.5
C113—C114—H1142	109.5	H3212—C321—H3213	109.5
H1141—C114—H1142	109.5	N319—C323—H3231	109.4
C113—C114—H1143	109.5	N319—C323—H3232	109.5
H1141—C114—H1143	109.5	H3231—C323—H3232	109.5
H1142—C114—H1143	109.5	N319—C323—H3233	109.5
C113—C115—H1151	109.5	H3231—C323—H3233	109.5
C113—C115—H1152	109.5	H3232—C323—H3233	109.5
H1151—C115—H1152	109.5	Se407—C408—C309	121.2 (8)
C113—C115—H1153	109.5	Se407—C408—C409	115.8 (7)
H1151—C115—H1153	109.5	Se407—C408—H4081	106.8
H1152—C115—H1153	109.5	C409—C408—H4081	109.3
C111—C117—O116	104.4 (3)	Se407—C408—H4082	107.2
C111—C117—C118	115.4 (3)	C409—C408—H4082	107.9
O116—C117—C118	106.5 (3)	H4081—C408—H4082	109.8
C111—C117—H1171	110.0	C408—C409—C310	108.8 (7)
O116—C117—H1171	110.1	C408—C409—H4091	109.2
C118—C117—H1171	110.1	C310—C409—H4091	109.2
C117—C118—N119	114.8 (3)	C408—C409—H4092	110.8
C117—C118—O122	125.1 (4)	C310—C409—H4092	110.0
N119—C118—O122	120.0 (4)	H4091—C409—H4092	108.8
Se7—C6—C1—N24	0.5 (6)	C1—C6—Se7—C8	179.3 (4)
Se7—C6—C1—C2	−178.7 (3)	C1—C6—C5—C4	−0.5 (6)
Se7—C6—C5—C4	178.7 (3)	C2—C1—C6—C5	0.4 (6)

Se7—C8—C9—C10	−71.6 (4)	C2—C3—C4—C5	0.2 (7)
Se107—C106—C101—N124	2.2 (6)	C3—C2—C1—C6	−0.0 (7)
Se107—C106—C101—C102	−178.8 (3)	C3—C4—C5—C6	0.2 (7)
Se107—C106—C105—C104	178.7 (3)	C5—C6—Se7—C8	0.3 (4)
Se107—C108—C109—C110	−70.6 (4)	C6—Se7—C8—C9	−80.1 (3)
Se207—C206—C201—N224	1.1 (6)	C8—C9—C10—C11	177.0 (3)
Se207—C206—C201—C202	−179.9 (3)	C9—C10—C11—C17	178.5 (3)
Se207—C206—C205—C204	179.5 (3)	C10—C11—O12—C13	−148.1 (3)
Se207—C208—C209—C210	−164.8 (3)	C10—C11—C17—C18	14.1 (5)
Se307—C306—C301—N324	7.4 (6)	C11—O12—C13—C14	−94.1 (3)
Se307—C306—C301—C302	−172.9 (4)	C11—O12—C13—C15	141.3 (3)
Se307—C306—C305—C304	174.6 (3)	C11—C17—O16—C13	−0.9 (4)
Se307—C308—C309—C310	−179.1 (4)	C13—O12—C11—C17	−23.6 (3)
Se407—C306—C301—N324	−6.6 (5)	C13—O16—C17—C18	120.3 (3)
Se407—C306—C301—C302	173.1 (4)	C14—C13—O16—C17	105.6 (4)
Se407—C306—C305—C304	−171.3 (4)	C15—C13—O16—C17	−129.4 (4)
Se407—C408—C409—C310	−160.8 (7)	C17—C18—N19—C23	163.0 (3)
O12—C11—C10—C9	−65.5 (4)	C18—N19—O20—C21	−121.4 (4)
O12—C11—C17—O16	14.7 (3)	C21—O20—N19—C23	90.0 (4)
O12—C11—C17—C18	−104.1 (4)	C101—C102—C103—C104	−0.2 (6)
O12—C13—O16—C17	−13.6 (4)	C101—C106—Se107—C108	−175.6 (4)
O16—C13—O12—C11	23.6 (4)	C101—C106—C105—C104	0.5 (6)
O16—C17—C11—C10	132.8 (3)	C102—C101—C106—C105	−0.7 (6)
O16—C17—C18—O22	−25.5 (5)	C102—C103—C104—C105	0.0 (6)
O16—C17—C18—N19	153.4 (3)	C103—C102—C101—C106	0.5 (6)
O20—N19—C18—O22	−163.1 (3)	C103—C104—C105—C106	−0.2 (7)
O20—N19—C18—C17	18.0 (4)	C105—C106—Se107—C108	6.3 (4)
O22—C18—N19—C23	−18.1 (5)	C106—Se107—C108—C109	−83.8 (3)
O22—C18—C17—C11	90.4 (5)	C108—C109—C110—C111	175.6 (3)
O25—N24—C1—C2	3.3 (6)	C109—C110—C111—C117	−179.5 (3)
O25—N24—C1—C6	−175.9 (4)	C110—C111—O112—C113	−138.5 (3)
O26—N24—C1—C2	−177.4 (4)	C110—C111—C117—C118	−5.5 (5)
O26—N24—C1—C6	3.3 (6)	C111—O112—C113—C114	−88.9 (3)
O112—C111—C110—C109	−64.4 (4)	C111—O112—C113—C115	146.0 (3)
O112—C111—C117—O116	−6.0 (3)	C111—C117—O116—C113	24.8 (3)
O112—C111—C117—C118	−122.6 (3)	C113—O112—C111—C117	−15.0 (3)
O112—C113—O116—C117	−34.4 (3)	C113—O116—C117—C118	147.4 (3)
O116—C113—O112—C111	30.7 (3)	C114—C113—O116—C117	84.1 (4)
O116—C117—C111—C110	111.1 (4)	C115—C113—O116—C117	−150.3 (3)
O116—C117—C118—O122	−18.0 (5)	C117—C118—N119—C123	164.5 (3)
O116—C117—C118—N119	159.8 (3)	C118—N119—O120—C121	−119.8 (4)
O120—N119—C118—O122	−166.1 (3)	C121—O120—N119—C123	88.8 (4)
O120—N119—C118—C117	16.0 (4)	C201—C202—C203—C204	−1.5 (6)
O122—C118—N119—C123	−17.5 (5)	C201—C206—Se207—C208	−177.7 (4)
O122—C118—C117—C111	97.4 (5)	C201—C206—C205—C204	1.0 (6)
O125—N124—C101—C102	−8.7 (6)	C202—C201—C206—C205	−1.5 (6)
O125—N124—C101—C106	170.4 (4)	C202—C203—C204—C205	1.0 (6)
O126—N124—C101—C102	171.7 (4)	C203—C202—C201—C206	1.8 (6)
O126—N124—C101—C106	−9.2 (6)	C203—C204—C205—C206	−0.8 (7)

O212—C211—C210—C209	−63.6 (5)	C205—C206—Se207—C208	4.0 (4)
O212—C211—C217—O216	31.0 (4)	C206—Se207—C208—C209	176.3 (3)
O212—C211—C217—C218	−87.9 (4)	C208—C209—C210—C211	−71.8 (5)
O212—C213—O216—C217	−4.2 (5)	C209—C210—C211—C217	−177.7 (3)
O216—C213—O212—C211	25.5 (4)	C210—C211—O212—C213	−158.0 (4)
O216—C217—C211—C210	149.5 (3)	C210—C211—C217—C218	30.6 (4)
O216—C217—C218—O222	−28.0 (5)	C211—O212—C213—C214	−92.0 (4)
O216—C217—C218—N219	150.2 (4)	C211—O212—C213—C215	143.1 (4)
O220—N219—C218—O222	−165.9 (4)	C211—C217—O216—C213	−16.7 (4)
O220—N219—C218—C217	15.8 (5)	C213—O212—C211—C217	−34.7 (4)
O222—C218—N219—C223	−8.3 (7)	C213—O216—C217—C218	105.3 (4)
O222—C218—C217—C211	87.4 (4)	C214—C213—O216—C217	114.5 (4)
O225—N224—C201—C202	0.5 (6)	C215—C213—O216—C217	−120.5 (4)
O225—N224—C201—C206	179.6 (4)	C217—C218—N219—C223	173.5 (4)
O226—N224—C201—C202	−179.9 (4)	C218—N219—O220—C221	−114.3 (4)
O226—N224—C201—C206	−0.9 (6)	C221—O220—N219—C223	85.4 (5)
O312—C311—C310—C309	−46.9 (6)	C301—C302—C303—C304	−0.9 (6)
O312—C311—C310—C409	−69.1 (6)	C301—C306—Se307—C308	−177.4 (4)
O312—C311—C317—O316	30.8 (4)	C301—C306—Se407—C408	−160.7 (4)
O312—C311—C317—C318	−87.1 (3)	C301—C306—C305—C304	1.4 (6)
O312—C313—O316—C317	−4.1 (4)	C302—C301—C306—C305	−0.5 (6)
O316—C313—O312—C311	25.4 (4)	C302—C303—C304—C305	1.8 (6)
O316—C317—C311—C310	149.5 (3)	C303—C302—C301—C306	0.3 (7)
O316—C317—C318—O322	−40.4 (5)	C303—C304—C305—C306	−2.1 (6)
O316—C317—C318—N319	139.5 (3)	C305—C306—Se307—C308	10.3 (4)
O320—N319—C318—O322	168.2 (3)	C305—C306—Se407—C408	12.0 (5)
O320—N319—C318—C317	−11.8 (5)	C306—Se307—C308—C309	166.7 (4)
O322—C318—N319—C323	9.7 (6)	C306—Se407—C408—C409	−94.8 (9)
O322—C318—C317—C311	74.4 (4)	C308—Se407—C408—C409	−43.3 (8)
O325—N324—C301—C302	−9.2 (5)	C308—C309—C310—C311	−83.3 (6)
O325—N324—C301—C306	170.6 (4)	C309—C310—C311—C317	−160.9 (5)
O326—N324—C301—C302	170.5 (4)	C310—C311—O312—C313	−158.3 (4)
O326—N324—C301—C306	−9.7 (6)	C310—C311—C317—C318	31.6 (5)
N19—C18—C17—C11	−90.7 (4)	C311—O312—C313—C314	−91.0 (4)
N24—C1—C2—C3	−179.2 (4)	C311—O312—C313—C315	143.3 (4)
N24—C1—C6—C5	179.6 (4)	C311—C310—C409—C408	−170.3 (8)
N119—C118—C117—C111	−84.8 (4)	C311—C317—O316—C313	−16.8 (4)
N124—C101—C102—C103	179.6 (4)	C313—O312—C311—C317	−34.7 (4)
N124—C101—C106—C105	−179.7 (4)	C313—O316—C317—C318	103.8 (4)
N219—C218—C217—C211	−94.4 (4)	C314—C313—O316—C317	114.0 (3)
N224—C201—C202—C203	−179.2 (4)	C315—C313—O316—C317	−122.0 (3)
N224—C201—C206—C205	179.5 (4)	C317—C311—C310—C409	176.9 (5)
N319—C318—C317—C311	−105.6 (4)	C317—C318—N319—C323	−170.3 (4)
N324—C301—C302—C303	−179.9 (4)	C318—N319—O320—C321	118.8 (4)
N324—C301—C306—C305	179.7 (4)	C321—O320—N319—C323	−81.1 (5)
C1—C2—C3—C4	−0.3 (7)		

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

Crystal structure of $C_{23}H_{28}O_7$ — ban1009

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The crystal structure of $C_{23}H_{28}O_7$ is reported.

Comment

The crystallographic asymmetric unit consists of one $C_{23}H_{28}O_7$ molecule.

Experimental

The compound was prepared by AL and recrystallised from ethyl acetate and petroleum spirits. The sample ID is AL-156.

Crystal data

$C_{23}H_{28}O_7$	$V = 2181.25\ (7)$ Å^3
$M_r = 416.47$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.6558\ (1)$ Å	$\mu = 0.09$ mm^{-1}
$b = 8.9051\ (2)$ Å	$T = 200$ K
$c = 28.2982\ (5)$ Å	$0.44 \times 0.07 \times 0.06$ mm

Data collection

Area diffractometer	2937 independent reflections
Absorption correction: Integration via Gaussian method (Coppens, 1970) implemented in *maXus* (2000)	2429 reflections with $I > 2.0\sigma(I)$
$T_{min} = 0.975$, $T_{max} = 0.995$	$R_{int} = 0.053$
27170 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$	0 restraints
$wR(F^2) = 0.081$	H-atom parameters constrained
$S = 1.01$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
2937 reflections	$\Delta\rho_{min} = -0.25$ e Å^{-3}
271 parameters	Absolute structure: The enantiomer has been assigned by reference to an unchanging chiral centre in the synthetic procedure.

Table 1
Selected geometric parameters (Å, º)

C1—O2	1.353 (2)	C11—C12	1.534 (3)
C1—C18	1.488 (3)	C12—C13	1.518 (3)
C1—O19	1.206 (2)	C13—C14	1.386 (3)
O2—C3	1.461 (2)	C13—C18	1.410 (3)
C3—C4	1.518 (3)	C14—C15	1.390 (3)
C3—C20	1.513 (3)	C15—C16	1.383 (3)
C4—C5	1.462 (3)	C15—O27	1.365 (2)
C5—C6	1.196 (3)	C16—C17	1.389 (3)

C6—C7	1.453 (3)	C17—C18	1.411 (3)
C7—C8	1.514 (3)	C17—O29	1.358 (2)
C7—O21	1.222 (2)	O22—C23	1.448 (3)
C8—C9	1.545 (3)	C23—O24	1.431 (2)
C8—O22	1.417 (2)	C23—C25	1.508 (3)
C9—C10	1.521 (3)	C23—C26	1.507 (3)
C9—O24	1.431 (2)	O27—C28	1.431 (3)
C10—C11	1.526 (3)	O29—C30	1.433 (3)

O2—C1—C18	112.83 (17)	C12—C13—C18	123.98 (18)
O2—C1—O19	122.49 (19)	C14—C13—C18	119.25 (17)
C18—C1—O19	124.68 (18)	C13—C14—C15	121.25 (19)
C1—O2—C3	116.28 (15)	C14—C15—C16	120.31 (19)
O2—C3—C4	105.42 (16)	C14—C15—O27	115.66 (19)
O2—C3—C20	111.06 (18)	C16—C15—O27	124.04 (18)
C4—C3—C20	113.75 (18)	C15—C16—C17	119.26 (18)
C3—C4—C5	112.72 (18)	C16—C17—C18	121.28 (19)
C4—C5—C6	177.8 (2)	C16—C17—O29	123.08 (18)
C5—C6—C7	172.1 (2)	C18—C17—O29	115.62 (18)
C6—C7—C8	114.15 (18)	C1—C18—C17	117.80 (18)
C6—C7—O21	122.7 (2)	C1—C18—C13	123.56 (16)
C8—C7—O21	123.16 (19)	C17—C18—C13	118.56 (18)
C7—C8—C9	111.63 (16)	C8—O22—C23	109.38 (14)
C7—C8—O22	111.81 (16)	O22—C23—O24	104.88 (15)
C9—C8—O22	103.52 (16)	O22—C23—C25	109.31 (18)
C8—C9—C10	118.79 (17)	O24—C23—C25	108.17 (16)
C8—C9—O24	100.44 (14)	O22—C23—C26	108.66 (16)
C10—C9—O24	108.95 (15)	O24—C23—C26	111.61 (16)
C9—C10—C11	113.12 (15)	C25—C23—C26	113.81 (18)
C10—C11—C12	112.07 (15)	C23—O24—C9	107.54 (14)
C11—C12—C13	111.85 (16)	C15—O27—C28	117.77 (18)
C12—C13—C14	116.77 (18)	C17—O29—C30	117.74 (18)

The compound is enantiometrically pure but the anomolous dispersion terms are very low for all elements in the structure and so the absolute configuration can not be determined in this experiment. Consequently Friedel-pair reflections have been averaged and the Flack parameter has not been refined. The absolute configuration of the molecule has been assigned on the basis of the synthetic precursors.

The H atoms were all located in a difference map, but were repositioned geometrically. They were initially refined with soft restraints on the bond lengths and angles to regularise their geometry (C—H in the range 0.93–0.98Å) and with U_{iso}(H) in the range 1.2-1.5 times U_{eq} of the parent atom, after which the positions were refined with riding constraints.

The final difference electron density map is reasonably flat with the largest peaks distributed randomly through the structure.

Data collection: *COLLECT* (Nonius, 2001).; cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP*-II (Johnson 1976) in *TEXSAN* (MSC, 1992-1997); software used to prepare material for publication: *CRYSTALS* (Betteridge *et al.*, 2003).

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supplementary materials

Crystal structure of $C_{23}H_{28}O_7$ — ban1009

Andrew Lin, Martin G. Banwell and Anthony C. Willis*

(ban1009)

Crystal data

$C_{23}H_{28}O_7$	D_x = 1.268 Mg m^{-3}
M_r = 416.47	Mo $K\alpha$ radiation, λ = 0.71073 Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 33266 reflections
a = 8.6558 (1) Å	θ = 2.6–27.5°
b = 8.9051 (2) Å	μ = 0.09 mm^{-1}
c = 28.2982 (5) Å	T = 200 K
V = 2181.25 (7) Å^3	Needle, Colourless
Z = 4	0.44 × 0.07 × 0.06 mm
F(000) = 888	

Data collection

Area diffractometer	2429 reflections with $I > 2.0\sigma(I)$
graphite	R_{int} = 0.053
φ and ω scans with CCD	θ_{max} = 27.9°, θ_{min} = 2.7°
Absorption correction: Integration via Gaussian method (Coppens, 1970) implemented in *maXus* (2000)	h = −11→11
T_{min} = 0.975, T_{max} = 0.995	k = −11→11
27170 measured reflections	l = −36→37
2937 independent reflections	

Refinement

Refinement on F^2	Primary atom site location: Structure-invariant direct methods
Least-squares matrix: Full	Hydrogen site location: Inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.038	H-atom parameters constrained
$wR(F^2)$ = 0.081	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.35P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max}$ = 0.001
2937 reflections	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
271 parameters	$\Delta\rho_{min}$ = −0.25 e Å^{-3}
0 restraints	Absolute structure: The enantiomer has been assigned by reference to an unchanging chiral centre in the synthetic procedure.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.7010 (2)	−0.0169 (2)	0.46441 (7)	0.0361
O2	0.64477 (16)	−0.01570 (15)	0.50903 (4)	0.0358
C3	0.6812 (2)	−0.1464 (2)	0.53817 (7)	0.0379
C4	0.5654 (3)	−0.1431 (2)	0.57842 (7)	0.0424
C5	0.5856 (3)	−0.0132 (2)	0.60940 (7)	0.0404
C6	0.6067 (3)	0.0910 (3)	0.63530 (7)	0.0423
C7	0.6121 (3)	0.2255 (2)	0.66431 (7)	0.0378
C8	0.4938 (2)	0.3433 (2)	0.65102 (7)	0.0356

C9	0.5630 (2)	0.4635 (2)	0.61778 (6)	0.0320
C10	0.6896 (2)	0.4174 (2)	0.58344 (6)	0.0319
C11	0.6271 (2)	0.3486 (2)	0.53792 (6)	0.0318
C12	0.7571 (2)	0.3009 (2)	0.50425 (7)	0.0343
C13	0.6969 (2)	0.2682 (2)	0.45493 (7)	0.0332
C14	0.6733 (2)	0.3908 (2)	0.42560 (7)	0.0372
C15	0.6124 (2)	0.3735 (2)	0.38047 (7)	0.0371
C16	0.5738 (2)	0.2324 (3)	0.36387 (7)	0.0385
C17	0.6009 (2)	0.1078 (2)	0.39220 (7)	0.0357
C18	0.6644 (2)	0.1228 (2)	0.43787 (6)	0.0330
O19	0.77237 (18)	−0.12181 (18)	0.44857 (5)	0.0489
C20	0.8478 (3)	−0.1428 (3)	0.55429 (8)	0.0494
O21	0.70607 (19)	0.24255 (18)	0.69609 (5)	0.0478
O22	0.44478 (17)	0.42745 (16)	0.69081 (5)	0.0420
C23	0.5217 (2)	0.5719 (2)	0.69080 (7)	0.0370
O24	0.62616 (15)	0.56578 (16)	0.65165 (4)	0.0344
C25	0.6154 (3)	0.5878 (3)	0.73545 (7)	0.0537
C26	0.4019 (3)	0.6931 (2)	0.68438 (8)	0.0459
O27	0.5947 (2)	0.50268 (18)	0.35509 (5)	0.0497
C28	0.5299 (3)	0.4911 (3)	0.30870 (7)	0.0585
O29	0.56516 (19)	−0.03445 (17)	0.37882 (5)	0.0465
C30	0.5093 (3)	−0.0567 (3)	0.33168 (8)	0.0561
H31	0.6642	−0.2382	0.5186	0.0431*
H42	0.5808	−0.2323	0.5978	0.0511*
H41	0.4598	−0.1428	0.5650	0.0509*
H81	0.4051	0.2962	0.6368	0.0407*
H91	0.4764	0.5107	0.5999	0.0371*
H101	0.7596	0.3489	0.5986	0.0384*
H102	0.7480	0.5073	0.5757	0.0368*
H111	0.5615	0.2628	0.5453	0.0381*
H112	0.5646	0.4227	0.5219	0.0384*
H121	0.8083	0.2120	0.5169	0.0405*
H122	0.8334	0.3816	0.5018	0.0392*
H141	0.6987	0.4890	0.4368	0.0440*
H161	0.5304	0.2209	0.3337	0.0456*
H201	0.8684	−0.2246	0.5755	0.0735*
H202	0.8669	−0.0499	0.5709	0.0729*
H203	0.9161	−0.1474	0.5271	0.0744*
H251	0.6644	0.6855	0.7360	0.0790*
H252	0.5469	0.5827	0.7622	0.0795*
H253	0.6926	0.5072	0.7376	0.0793*
H261	0.4497	0.7930	0.6847	0.0684*
H262	0.3315	0.6861	0.7107	0.0678*
H263	0.3478	0.6780	0.6544	0.0671*
H282	0.5360	0.5912	0.2949	0.0868*
H281	0.5919	0.4190	0.2900	0.0867*
H283	0.4237	0.4556	0.3099	0.0879*
H302	0.4926	−0.1624	0.3278	0.0826*
H301	0.5872	−0.0214	0.3090	0.0831*
H303	0.4138	−0.0007	0.3267	0.0835*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0320 (10)	0.0374 (11)	0.0388 (10)	−0.0012 (9)	0.0031 (9)	−0.0034 (9)
O2	0.0393 (8)	0.0321 (8)	0.0360 (7)	0.0024 (6)	0.0047 (6)	0.0020 (6)
C3	0.0438 (11)	0.0279 (10)	0.0421 (11)	−0.0007 (9)	−0.0013 (10)	0.0018 (9)

C4	0.0512 (12)	0.0307 (11)	0.0452 (11)	−0.0065 (10)	0.0022 (10)	0.0043 (10)
C5	0.0483 (12)	0.0328 (11)	0.0401 (11)	−0.0002 (10)	0.0052 (10)	0.0090 (10)
C6	0.0509 (13)	0.0356 (12)	0.0403 (11)	0.0026 (10)	0.0044 (10)	0.0063 (10)
C7	0.0436 (11)	0.0350 (11)	0.0350 (10)	−0.0035 (10)	0.0090 (10)	0.0052 (9)
C8	0.0340 (10)	0.0344 (11)	0.0385 (10)	−0.0039 (9)	0.0027 (9)	−0.0028 (9)
C9	0.0333 (10)	0.0308 (10)	0.0318 (10)	−0.0017 (9)	−0.0015 (8)	−0.0037 (8)
C10	0.0297 (9)	0.0332 (11)	0.0328 (10)	−0.0031 (8)	0.0006 (8)	0.0000 (9)
C11	0.0298 (9)	0.0339 (10)	0.0318 (9)	−0.0016 (8)	−0.0003 (8)	−0.0012 (9)
C12	0.0307 (10)	0.0360 (11)	0.0362 (10)	−0.0010 (9)	0.0021 (8)	−0.0027 (9)
C13	0.0267 (9)	0.0394 (11)	0.0335 (10)	0.0001 (8)	0.0072 (8)	−0.0037 (9)
C14	0.0364 (10)	0.0355 (12)	0.0398 (10)	0.0005 (10)	0.0066 (9)	−0.0058 (9)
C15	0.0360 (10)	0.0395 (12)	0.0356 (10)	0.0050 (10)	0.0070 (9)	0.0033 (9)
C16	0.0336 (11)	0.0481 (13)	0.0337 (10)	0.0010 (10)	0.0033 (9)	−0.0036 (10)
C17	0.0322 (10)	0.0357 (11)	0.0392 (10)	−0.0008 (9)	0.0059 (9)	−0.0069 (9)
C18	0.0290 (9)	0.0363 (11)	0.0337 (10)	0.0004 (9)	0.0054 (8)	−0.0016 (9)
O19	0.0546 (9)	0.0417 (9)	0.0505 (9)	0.0132 (8)	0.0121 (8)	−0.0009 (8)
C20	0.0465 (12)	0.0464 (13)	0.0552 (13)	0.0012 (11)	−0.0057 (11)	0.0042 (12)
O21	0.0551 (9)	0.0489 (9)	0.0394 (8)	0.0060 (8)	−0.0019 (8)	0.0033 (7)
O22	0.0471 (8)	0.0392 (8)	0.0398 (7)	−0.0023 (7)	0.0143 (7)	−0.0024 (7)
C23	0.0396 (11)	0.0386 (11)	0.0327 (10)	−0.0005 (9)	0.0061 (9)	−0.0028 (9)
O24	0.0358 (7)	0.0353 (7)	0.0320 (6)	−0.0053 (6)	0.0029 (6)	−0.0062 (6)
C25	0.0589 (14)	0.0639 (16)	0.0382 (11)	0.0054 (14)	−0.0011 (11)	−0.0116 (11)
C26	0.0479 (13)	0.0429 (13)	0.0469 (12)	0.0039 (11)	0.0085 (11)	−0.0057 (10)
O27	0.0640 (10)	0.0441 (9)	0.0409 (8)	0.0022 (8)	−0.0005 (8)	0.0055 (7)
C28	0.0757 (17)	0.0602 (16)	0.0396 (12)	0.0012 (14)	−0.0016 (12)	0.0110 (12)
O29	0.0510 (9)	0.0434 (9)	0.0452 (8)	−0.0037 (8)	−0.0048 (7)	−0.0097 (7)
C30	0.0556 (14)	0.0600 (16)	0.0526 (13)	0.0024 (13)	−0.0105 (12)	−0.0229 (12)

Geometric parameters (Å, º)

C1—O2	1.353 (2)	C13—C18	1.410 (3)
C1—C18	1.488 (3)	C14—C15	1.390 (3)
C1—O19	1.206 (2)	C14—H141	0.956
O2—C3	1.461 (2)	C15—C16	1.383 (3)
C3—C4	1.518 (3)	C15—O27	1.365 (2)
C3—C20	1.513 (3)	C16—C17	1.389 (3)
C3—H31	0.998	C16—H161	0.938
C4—C5	1.462 (3)	C17—C18	1.411 (3)
C4—H42	0.974	C17—O29	1.358 (2)
C4—H41	0.989	C20—H201	0.960
C5—C6	1.196 (3)	C20—H202	0.966
C6—C7	1.453 (3)	C20—H203	0.972
C7—C8	1.514 (3)	O22—C23	1.448 (3)
C7—O21	1.222 (2)	C23—O24	1.431 (2)
C8—C9	1.545 (3)	C23—C25	1.508 (3)
C8—O22	1.417 (2)	C23—C26	1.507 (3)
C8—H81	0.962	C25—H251	0.968
C9—C10	1.521 (3)	C25—H252	0.964
C9—O24	1.431 (2)	C25—H253	0.983
C9—H91	0.998	C26—H261	0.981
C10—C11	1.526 (3)	C26—H262	0.965
C10—H101	0.961	C26—H263	0.979
C10—H102	0.972	O27—C28	1.431 (3)
C11—C12	1.534 (3)	C28—H282	0.974
C11—H111	0.974	C28—H281	0.990
C11—H112	0.967	C28—H283	0.973
C12—C13	1.518 (3)	O29—C30	1.433 (3)

C12—H121	0.976	C30—H302	0.959
C12—H122	0.979	C30—H301	0.984
C13—C14	1.386 (3)	C30—H303	0.976
O19···C4^{i}	3.376 (3)	O24···C30^{ii}	3.351 (3)
O19···C9^{ii}	3.441 (2)	O24···C4^{iv}	3.360 (2)
O21···C28^{ii}	3.493 (3)	O27···C20^{v}	3.564 (3)
O21···C26^{iii}	3.537 (3)	O29···C10^{v}	3.577 (2)
O2—C1—C18	112.83 (17)	C13—C14—H141	119.3
O2—C1—O19	122.49 (19)	C15—C14—H141	119.5
C18—C1—O19	124.68 (18)	C14—C15—C16	120.31 (19)
C1—O2—C3	116.28 (15)	C14—C15—O27	115.66 (19)
O2—C3—C4	105.42 (16)	C16—C15—O27	124.04 (18)
O2—C3—C20	111.06 (18)	C15—C16—C17	119.26 (18)
C4—C3—C20	113.75 (18)	C15—C16—H161	120.4
O2—C3—H31	107.9	C17—C16—H161	120.4
C4—C3—H31	109.5	C16—C17—C18	121.28 (19)
C20—C3—H31	109.0	C16—C17—O29	123.08 (18)
C3—C4—C5	112.72 (18)	C18—C17—O29	115.62 (18)
C3—C4—H42	108.4	C1—C18—C17	117.80 (18)
C5—C4—H42	107.0	C1—C18—C13	123.56 (16)
C3—C4—H41	108.8	C17—C18—C13	118.56 (18)
C5—C4—H41	109.7	C3—C20—H201	110.4
H42—C4—H41	110.1	C3—C20—H202	109.2
C4—C5—C6	177.8 (2)	H201—C20—H202	108.3
C5—C6—C7	172.1 (2)	C3—C20—H203	109.9
C6—C7—C8	114.15 (18)	H201—C20—H203	110.4
C6—C7—O21	122.7 (2)	H202—C20—H203	108.6
C8—C7—O21	123.16 (19)	C8—O22—C23	109.38 (14)
C7—C8—C9	111.63 (16)	O22—C23—O24	104.88 (15)
C7—C8—O22	111.81 (16)	O22—C23—C25	109.31 (18)
C9—C8—O22	103.52 (16)	O24—C23—C25	108.17 (16)
C7—C8—H81	109.9	O22—C23—C26	108.66 (16)
C9—C8—H81	110.9	O24—C23—C26	111.61 (16)
O22—C8—H81	108.8	C25—C23—C26	113.81 (18)
C8—C9—C10	118.79 (17)	C23—O24—C9	107.54 (14)
C8—C9—O24	100.44 (14)	C23—C25—H251	109.4
C10—C9—O24	108.95 (15)	C23—C25—H252	108.9
C8—C9—H91	108.0	H251—C25—H252	107.5
C10—C9—H91	109.3	C23—C25—H253	110.5
O24—C9—H91	111.0	H251—C25—H253	111.0
C9—C10—C11	113.12 (15)	H252—C25—H253	109.5
C9—C10—H101	109.8	C23—C26—H261	111.0
C11—C10—H101	110.3	C23—C26—H262	107.2
C9—C10—H102	107.2	H261—C26—H262	108.5
C11—C10—H102	109.0	C23—C26—H263	109.6
H101—C10—H102	107.2	H261—C26—H263	109.5
C10—C11—C12	112.07 (15)	H262—C26—H263	111.0
C10—C11—H111	109.9	C15—O27—C28	117.77 (18)
C12—C11—H111	110.1	O27—C28—H282	106.2
C10—C11—H112	108.7	O27—C28—H281	109.0
C12—C11—H112	107.9	H282—C28—H281	110.5
H111—C11—H112	108.0	O27—C28—H283	111.2
C11—C12—C13	111.85 (16)	H282—C28—H283	111.2
C11—C12—H121	109.3	H281—C28—H283	108.7
C13—C12—H121	109.8	C17—O29—C30	117.74 (18)

C11—C12—H122	109.6	O29—C30—H302	107.0
C13—C12—H122	107.9	O29—C30—H301	109.5
H121—C12—H122	108.4	H302—C30—H301	110.1
C12—C13—C14	116.77 (18)	O29—C30—H303	110.4
C12—C13—C18	123.98 (18)	H302—C30—H303	111.0
C14—C13—C18	119.25 (17)	H301—C30—H303	108.9
C13—C14—C15	121.25 (19)		

O2—C1—C18—C13	52.3 (2)	C6—C7—C8—C9	−96.0 (2)
O2—C1—C18—C17	−131.0 (2)	C7—C8—O22—C23	102.0 (2)
O2—C3—C4—C5	−65.5 (2)	C7—C8—C9—C10	31.7 (2)
O19—C1—O2—C3	2.7 (3)	C8—O22—C23—C25	−120.0 (2)
O19—C1—C18—C13	−128.0 (2)	C8—O22—C23—C26	115.3 (2)
O19—C1—C18—C17	48.7 (3)	C8—C9—O24—C23	−37.5 (2)
O21—C7—C8—O22	−32.7 (3)	C8—C9—C10—C11	83.6 (2)
O21—C7—C8—C9	82.8 (2)	C9—O24—C23—C25	144.0 (2)
O22—C8—C7—C6	148.6 (2)	C9—O24—C23—C26	−90.1 (2)
O22—C8—C9—O24	33.6 (2)	C9—C8—O22—C23	−18.4 (2)
O22—C8—C9—C10	152.1 (1)	C9—C10—C11—C12	−179.1 (1)
O22—C23—O24—C9	27.4 (2)	C10—C9—O24—C23	−162.9 (1)
O24—C9—C8—C7	−86.8 (2)	C10—C11—C12—C13	−166.2 (1)
O24—C9—C10—C11	−162.4 (1)	C11—C12—C13—C14	81.5 (2)
O24—C23—O22—C8	−4.1 (2)	C11—C12—C13—C18	−98.4 (2)
O27—C15—C14—C13	180.0 (2)	C12—C13—C14—C15	−177.3 (2)
O27—C15—C16—C17	178.1 (2)	C12—C13—C18—C17	176.6 (2)
O29—C17—C16—C15	179.4 (2)	C13—C14—C15—C16	0.0 (3)
O29—C17—C18—C1	6.3 (2)	C13—C18—C17—C16	1.5 (3)
O29—C17—C18—C13	−176.8 (2)	C14—C13—C18—C17	−3.4 (3)
C1—O2—C3—C4	−161.6 (2)	C14—C15—O27—C28	−178.9 (2)
C1—O2—C3—C20	74.7 (2)	C14—C15—C16—C17	−2.0 (3)
C1—C18—C13—C12	−6.7 (3)	C15—C14—C13—C18	2.6 (3)
C1—C18—C13—C14	173.3 (2)	C15—C16—C17—C18	1.2 (3)
C1—C18—C17—C16	−175.4 (2)	C16—C15—O27—C28	1.0 (3)
C3—O2—C1—C18	−177.6 (1)	C16—C17—O29—C30	5.5 (3)
C5—C4—C3—C20	56.4 (2)	C18—C17—O29—C30	−176.2 (2)

Symmetry codes: (i) *x*+1/2, −*y*−1/2, −*z*+1; (ii) *x*+1/2, −*y*+1/2, −*z*+1; (iii) −*x*+1, *y*−1/2, −*z*+3/2; (iv) *x*, *y*+1, *z*; (v) *x*−1/2, −*y*+1/2, −*z*+1.