Crystal structure of $C_{20}H_{19}NO_9$ — ban0718

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Abstract

The crystal structure of $C_{20}H_{19}NO_9$ is reported.

Computing details

Data collection: *COLLECT* (Nonius BV, 1997); 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* (Watkin *et al.* 2003); molecular graphics: *ORTEP*II (Johnson 1976) in *TEXSAN* (MSC, 1992-1997); software used to prepare material for publication: *CRYSTALS* (Watkin *et al.* 2003).

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Crystal data

$C_{20}H_{19}NO_9$	V = 1910.48 (7) Å^3
M_r = 417.37	Z = 4
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$
a = 8.0014 (1) Å	μ = 0.12 mm^{-1}
b = 8.2641 (2) Å	T = 200 K
c = 28.8922 (6) Å	0.46 × 0.11 × 0.07 mm

Data collection

Nonius KappaCCD diffractometer	2526 independent reflections
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	1904 reflections with $I > 2.0\sigma(I)$
T_{min} = 0.965, T_{max} = 0.993	R_{int} = 0.043
20908 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029	H atoms treated by a mixture of independent and constrained refinement

$wR(F^2) = 0.034$	$\Delta\rho_{max} = 0.17$ e Å^{-3}
$S = 1.16$	$\Delta\rho_{min} = -0.14$ e Å^{-3}
1904 reflections	Absolute structure: ?
287 parameters	Flack parameter: ?
12 restraints	Rogers parameter: ?

Selected geometric parameters (Å, °)

O11—C10	1.381 (2)	C1—C2	1.502 (2)
O11—C12	1.432 (3)	C1—C17	1.338 (3)
O13—C12	1.435 (3)	C2—C3	1.521 (3)
O13—C14	1.363 (2)	C3—C4	1.512 (2)
O18—C2	1.468 (2)	C4—C5	1.524 (2)
O18—C19	1.349 (2)	C5—C17	1.513 (3)
O20—C19	1.201 (3)	C7—C8	1.490 (3)
O22—C3	1.447 (2)	C8—C9	1.403 (3)
O22—C23	1.351 (5)	C8—C16	1.404 (3)
O22—C231	1.324 (12)	C9—C10	1.361 (3)
O24—C23	1.182 (6)	C10—C14	1.391 (3)
O24—C231	1.296 (16)	C14—C15	1.375 (3)
O26—C4	1.443 (2)	C15—C16	1.410 (3)
O26—C27	1.365 (2)	C16—C17	1.486 (2)
O28—C27	1.202 (3)	C19—C21	1.488 (3)
O30—C7	1.230 (2)	C23—C25	1.480 (5)
O241—C231	1.190 (13)	C25—C231	1.516 (12)
N6—C5	1.456 (2)	C27—C29	1.491 (3)
N6—C7	1.347 (3)		
C10—O11—C12	105.53 (16)	C8—C9—C10	116.47 (18)
C12—O13—C14	105.88 (16)	O11—C10—C9	128.56 (19)
C2—O18—C19	116.90 (14)	O11—C10—C14	109.24 (17)
C3—O22—C23	117.9 (2)	C9—C10—C14	122.21 (17)
C3—O22—C231	118.3 (6)	O13—C12—O11	107.64 (16)
C4—O26—C27	114.86 (15)	C10—C14—O13	110.09 (17)
C5—N6—C7	124.70 (17)	C10—C14—C15	122.45 (18)
C2—C1—C17	123.77 (16)	O13—C14—C15	127.41 (17)
C1—C2—O18	109.22 (14)	C14—C15—C16	116.71 (16)
C1—C2—C3	114.08 (15)	C15—C16—C8	119.93 (16)
O18—C2—C3	103.24 (14)	C15—C16—C17	120.78 (16)
C2—C3—O22	107.21 (14)	C8—C16—C17	119.25 (16)
C2—C3—C4	110.50 (14)	C5—C17—C16	116.14 (15)
O22—C3—C4	109.01 (14)	C5—C17—C1	121.16 (16)
C3—C4—O26	111.05 (13)	C16—C17—C1	122.67 (16)
C3—C4—C5	110.41 (15)	O18—C19—O20	122.92 (18)
O26—C4—C5	107.68 (15)	O18—C19—C21	111.74 (18)
C4—C5—N6	108.69 (15)	O20—C19—C21	125.3 (2)
C4—C5—C17	109.90 (15)	O22—C23—O24	122.8 (4)
N6—C5—C17	111.96 (14)	O22—C23—C25	111.4 (4)
N6—C7—O30	121.78 (18)	O24—C23—C25	125.8 (4)

N6—C7—C8	116.04 (16)	O26—C27—O28	123.0 (2)
O30—C7—C8	121.99 (17)	O26—C27—C29	112.24 (19)
C7—C8—C9	116.12 (16)	O28—C27—C29	124.7 (2)
C7—C8—C16	121.52 (16)	C25—C231—O241	126.5 (10)
C9—C8—C16	122.12 (17)	O22—C231—O241	122.7 (10)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H61···O20^{i}	0.79 (3)	2.25 (3)	2.924 (2)	143 (2)
N6—H61···O26	0.79 (3)	2.50 (3)	2.795 (2)	103 (2)

Symmetry codes: (i) *x*+1, *y*, *z*.

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supplementary materials

Crystal structure of $C_{20}H_{19}NO_9$ — ban0718

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Comment

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 crystallographic asymmetric unit consists of one molecule of $C_{20}H_{19}NO_9$. Disorder is observed for atoms O24 and C23. Each of these atoms has been assigned two sites. Relative populations were refined. Distances and angles were restrained for the minor sites. Anisotropic displacement parameters of C24 and C241 were constrained to be equal and those of O23 and O231 were restrained to be near-equal.

A difference electron-density map revealed most H atoms, including the preferred orientations of each methyl group. H atoms were included at calculated positions. The H bonded to N6 which was refined positionally, but the remainder ride on the atom to which they are attached during refinement. A difference map at this stage revealed electron density between the H atoms of the methyl groups. Consequently the present methyl H atoms were assigned occupancies of 0.7 and additional H sites with occupancies 0.3 were placed between them.

The largest peaks in the final difference electron density map are located between C atoms or adjacent to the methyl of the disordered group.

Experimental

The compound was prepared by *MM* and recrystallized from methanol. The sample ID is *MM6*–486.

Refinement

H atoms attached to C were included at calculated positions and ride on the atoms to which they are attached. The H attached to N6 is refined positionally.

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Crystal data

$C_{20}H_{19}NO_9$	$D_x = 1.451$ Mg m^{-3}
$M_r = 417.37$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 23127 reflections
$a = 8.0014$ (1) Å	$\theta = 2.8$–27.5º
$b = 8.2641$ (2) Å	$\mu = 0.12$ mm^{-1}

c = 28.8922 (6) Å	T = 200 K
V = 1910.48 (7) Å^3	Needle, colourless
Z = 4	0.46 × 0.11 × 0.07 mm
F_{000} = 872	

Data collection

Nonius KappaCCD diffractometer	1904 reflections with $I > 2.0\sigma(I)$
Monochromator: graphite	R_{int} = 0.043
T = 200 K	θ_{max} = 27.5º
φ and ω scans with CCD	θ_{min} = 2.6º
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	h = −10→10
T_{min} = 0.965, T_{max} = 0.993	k = −10→10
20908 measured reflections	l = −22→37
2526 independent reflections	

Refinement

Refinement on F	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	Method, part 1, Chebychev polynomial, (Carruthers & Watkin, 1979, *P*rince, 1982) [*w*eight] = 1.0/ [A_0*T_0(x) + A_1*T_1(x) ··· + A_{n-1}]*T_{n-1}(x)] where A_i are the Chebychev coefficients listed below and x = F /Fmax Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta$F$/6*sigma$F$)^2]^2 A_i are: 0.963 0.518 0.752
$R[F^2 > 2\sigma(F^2)]$ = 0.029	$(\Delta/\sigma)_{max}$ = 0.004
$wR(F^2)$ = 0.034	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
S = 1.16	$\Delta\rho_{min}$ = −0.14 e Å^{-3}
1904 reflections	Extinction correction: None
287 parameters	
12 restraints	
Primary atom site location: structure-invariant direct methods	
Hydrogen site location: inferred from neighbouring sites	

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

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
O11	0.7956 (2)	1.1766 (2)	0.60000 (5)	0.0496	
O13	0.52661 (18)	1.1458 (2)	0.57191 (5)	0.0434	
O18	0.51863 (16)	0.87063 (15)	0.32946 (4)	0.0305	
O20	0.25121 (19)	0.90947 (19)	0.34963 (6)	0.0459	
O22	0.67558 (18)	0.49488 (14)	0.37419 (4)	0.0321	

O24	0.7362 (13)	0.3601 (5)	0.30963 (16)	0.0649	0.73 (2)
O26	0.98835 (17)	0.63688 (15)	0.34664 (4)	0.0318	
O28	0.9511 (2)	0.7436 (2)	0.27548 (5)	0.0509	
O30	1.19193 (18)	0.9611 (2)	0.47422 (5)	0.0440	
O241	0.632 (4)	0.3629 (13)	0.3089 (4)	0.0741	0.27 (2)
N6	1.0184 (2)	0.8346 (2)	0.42466 (6)	0.0331	
C1	0.5645 (2)	0.8339 (2)	0.41164 (6)	0.0264	
C2	0.5366 (2)	0.7494 (2)	0.36627 (6)	0.0268	
C3	0.6844 (2)	0.6482 (2)	0.35012 (6)	0.0272	
C4	0.8471 (2)	0.7308 (2)	0.36225 (6)	0.0272	
C5	0.8627 (2)	0.7506 (2)	0.41453 (6)	0.0271	
C7	1.0501 (2)	0.9159 (2)	0.46410 (6)	0.0313	
C8	0.9018 (2)	0.9593 (2)	0.49271 (6)	0.0293	
C9	0.9348 (2)	1.0424 (3)	0.53404 (6)	0.0351	
C10	0.7996 (3)	1.0970 (3)	0.55799 (7)	0.0354	
C12	0.6259 (3)	1.2280 (3)	0.60589 (7)	0.0413	
C14	0.6372 (2)	1.0773 (3)	0.54182 (6)	0.0318	
C15	0.6012 (2)	0.9935 (2)	0.50194 (6)	0.0307	
C16	0.7383 (2)	0.9312 (2)	0.47698 (6)	0.0259	
C17	0.7125 (2)	0.8407 (2)	0.43314 (6)	0.0257	
C19	0.3683 (3)	0.9431 (2)	0.32549 (7)	0.0329	
C21	0.3674 (4)	1.0664 (3)	0.28791 (7)	0.0499	
C23	0.7049 (10)	0.3587 (5)	0.34960 (17)	0.0349	0.73 (2)
C25	0.6904 (3)	0.2125 (2)	0.37895 (9)	0.0431	
C27	1.0289 (3)	0.6574 (3)	0.30112 (7)	0.0376	
C29	1.1769 (3)	0.5589 (3)	0.28732 (8)	0.0510	
C231	0.661 (2)	0.3611 (14)	0.3493 (5)	0.0349	0.27 (2)
H11	0.4668 (2)	0.8882 (2)	0.42664 (6)	0.0317*	
H21	0.4339 (2)	0.6807 (2)	0.36799 (6)	0.0322*	
H31	0.6776 (2)	0.6303 (2)	0.31594 (6)	0.0327*	
H41	0.8504 (2)	0.8399 (2)	0.34729 (6)	0.0326*	
H51	0.8666 (2)	0.6409 (2)	0.42907 (6)	0.0326*	
H61	1.099 (4)	0.816 (3)	0.4095 (9)	0.0397*	
H91	1.0514 (2)	1.0606 (3)	0.54531 (6)	0.0421*	
H121	0.5862 (3)	1.1996 (3)	0.63768 (7)	0.0495*	
H122	0.6176 (3)	1.3477 (3)	0.60128 (7)	0.0495*	
H151	0.4835 (2)	0.9776 (2)	0.49115 (6)	0.0368*	
H211	0.2543 (4)	1.1174 (3)	0.28600 (7)	0.0598*	0.7000
H212	0.4529 (4)	1.1514 (3)	0.29474 (7)	0.0598*	0.7000
H213	0.3945 (4)	1.0131 (3)	0.25775 (7)	0.0598*	0.7000
H214	0.4804 (4)	1.0710 (3)	0.27314 (7)	0.0598*	0.3000
H215	0.3392 (4)	1.1747 (3)	0.30121 (7)	0.0598*	0.3000
H216	0.2821 (4)	1.0361 (3)	0.26414 (7)	0.0598*	0.3000
H251	0.7128 (3)	0.1141 (2)	0.35981 (9)	0.0517*	0.5100
H252	0.7737 (3)	0.2186 (2)	0.40471 (9)	0.0517*	0.5100
H253	0.5751 (3)	0.2062 (2)	0.39216 (9)	0.0517*	0.5100
H254	0.6630 (3)	0.2453 (2)	0.41140 (9)	0.0517*	0.2200
H255	0.7988 (3)	0.1523 (2)	0.37864 (9)	0.0517*	0.2200
H256	0.5997 (3)	0.1413 (2)	0.36665 (9)	0.0517*	0.2200

H257	0.6786 (3)	0.1131 (2)	0.35951 (9)	0.0517*	0.2700
H258	0.8057 (3)	0.2169 (2)	0.39228 (9)	0.0517*	0.2700
H259	0.6067 (3)	0.2097 (2)	0.40463 (9)	0.0517*	0.2700
H291	1.2592 (3)	0.6293 (3)	0.27080 (8)	0.0612*	0.7000
H292	1.2304 (3)	0.5120 (3)	0.31558 (8)	0.0612*	0.7000
H293	1.1403 (3)	0.4695 (3)	0.26633 (8)	0.0612*	0.7000
H294	1.2782 (3)	0.5993 (3)	0.30397 (8)	0.0612*	0.3000
H295	1.1571 (3)	0.4430 (3)	0.29558 (8)	0.0612*	0.3000
H296	1.1946 (3)	0.5685 (3)	0.25317 (8)	0.0612*	0.3000

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O11	0.0336 (8)	0.0761 (11)	0.0391 (7)	0.0001 (8)	−0.0038 (6)	−0.0253 (8)
O13	0.0316 (7)	0.0579 (9)	0.0406 (7)	−0.0022 (8)	0.0023 (6)	−0.0181 (7)
O18	0.0284 (6)	0.0343 (7)	0.0288 (6)	0.0007 (6)	−0.0024 (5)	0.0065 (5)
O20	0.0330 (8)	0.0557 (9)	0.0492 (8)	0.0051 (7)	0.0038 (7)	0.0045 (8)
O22	0.0414 (8)	0.0227 (6)	0.0321 (6)	−0.0041 (6)	−0.0005 (6)	0.0008 (5)
O24	0.110 (5)	0.0383 (15)	0.0466 (17)	0.008 (2)	0.024 (2)	−0.0067 (12)
O26	0.0306 (6)	0.0311 (6)	0.0337 (6)	0.0025 (6)	0.0022 (6)	−0.0028 (6)
O28	0.0535 (10)	0.0596 (10)	0.0396 (8)	0.0034 (9)	0.0043 (8)	0.0080 (8)
O30	0.0238 (6)	0.0602 (10)	0.0481 (8)	−0.0016 (7)	−0.0040 (6)	−0.0160 (8)
O241	0.141 (14)	0.033 (4)	0.048 (4)	−0.013 (7)	−0.010 (7)	−0.012 (3)
N6	0.0228 (7)	0.0408 (8)	0.0357 (8)	0.0002 (7)	0.0014 (7)	−0.0087 (7)
C1	0.0263 (8)	0.0262 (8)	0.0266 (8)	−0.0013 (7)	0.0000 (7)	0.0012 (7)
C2	0.0266 (8)	0.0273 (8)	0.0266 (8)	−0.0031 (8)	−0.0023 (7)	0.0032 (7)
C3	0.0311 (9)	0.0248 (8)	0.0258 (8)	−0.0013 (8)	−0.0001 (7)	0.0027 (7)
C4	0.0292 (9)	0.0228 (8)	0.0295 (8)	0.0009 (7)	0.0022 (7)	−0.0010 (7)
C5	0.0265 (8)	0.0250 (8)	0.0300 (9)	−0.0013 (8)	−0.0011 (7)	0.0001 (7)
C7	0.0243 (8)	0.0349 (9)	0.0346 (9)	−0.0003 (8)	−0.0026 (8)	−0.0014 (8)
C8	0.0268 (8)	0.0306 (9)	0.0305 (9)	0.0000 (7)	−0.0027 (7)	0.0003 (8)
C9	0.0281 (9)	0.0459 (11)	0.0313 (9)	−0.0004 (8)	−0.0068 (8)	−0.0052 (9)
C10	0.0324 (9)	0.0427 (10)	0.0310 (9)	−0.0022 (9)	−0.0023 (8)	−0.0065 (8)
C12	0.0390 (11)	0.0471 (11)	0.0377 (11)	−0.0010 (10)	0.0003 (9)	−0.0107 (9)
C14	0.0277 (8)	0.0370 (9)	0.0308 (9)	−0.0005 (8)	0.0011 (8)	−0.0019 (8)
C15	0.0255 (8)	0.0350 (9)	0.0314 (8)	−0.0034 (8)	−0.0003 (7)	−0.0011 (8)
C16	0.0252 (8)	0.0248 (8)	0.0279 (8)	−0.0029 (7)	−0.0027 (7)	0.0030 (7)
C17	0.0266 (8)	0.0223 (7)	0.0281 (8)	−0.0012 (7)	0.0015 (7)	0.0033 (7)
C19	0.0341 (9)	0.0354 (9)	0.0290 (8)	0.0021 (8)	−0.0036 (8)	−0.0036 (8)
C21	0.0554 (13)	0.0558 (13)	0.0384 (11)	0.0157 (12)	−0.0053 (10)	0.0124 (10)
C23	0.032 (3)	0.0266 (9)	0.0465 (12)	−0.0003 (15)	0.0051 (16)	−0.0067 (9)
C25	0.0414 (12)	0.0265 (9)	0.0613 (13)	−0.0034 (9)	0.0026 (11)	0.0016 (9)
C27	0.0382 (10)	0.0379 (9)	0.0366 (9)	−0.0057 (10)	0.0042 (9)	−0.0053 (9)
C29	0.0456 (13)	0.0561 (13)	0.0513 (12)	0.0047 (12)	0.0095 (11)	−0.0150 (11)
C231	0.032 (3)	0.0266 (9)	0.0465 (12)	−0.0003 (15)	0.0051 (16)	−0.0067 (9)

Geometric parameters (Å, °)

O11—C10	1.381 (2)	C9—C10	1.361 (3)

O11—C12	1.432 (3)	C9—H91	1.000
O13—C12	1.435 (3)	C10—C14	1.391 (3)
O13—C14	1.363 (2)	C12—H121	1.000
O18—C2	1.468 (2)	C12—H122	1.000
O18—C19	1.349 (2)	C14—C15	1.375 (3)
O20—C19	1.201 (3)	C15—C16	1.410 (3)
O22—C3	1.447 (2)	C15—H151	1.000
O22—C23	1.351 (5)	C16—C17	1.486 (2)
O22—C231	1.324 (12)	C19—C21	1.488 (3)
O24—C23	1.182 (6)	C21—H211	1.000
O24—C231	1.296 (16)	C21—H212	1.000
O26—C4	1.443 (2)	C21—H213	1.000
O26—C27	1.365 (2)	C21—H214	1.000
O28—C27	1.202 (3)	C21—H215	1.000
O30—C7	1.230 (2)	C21—H216	1.000
O241—C231	1.190 (13)	C23—C25	1.480 (5)
N6—C5	1.456 (2)	C25—C231	1.516 (12)
N6—C7	1.347 (3)	C25—H251	1.000
N6—H61	0.79 (3)	C25—H252	1.000
C1—C2	1.502 (2)	C25—H253	1.000
C1—C17	1.338 (3)	C25—H254	1.000
C1—H11	1.000	C25—H255	1.000
C2—C3	1.521 (3)	C25—H256	1.000
C2—H21	1.000	C25—H257	1.000
C3—C4	1.512 (2)	C25—H258	1.000
C3—H31	1.000	C25—H259	1.000
C4—C5	1.524 (2)	C27—C29	1.491 (3)
C4—H41	1.000	C29—H291	1.000
C5—C17	1.513 (3)	C29—H292	1.000
C5—H51	1.000	C29—H293	1.000
C7—C8	1.490 (3)	C29—H294	1.000
C8—C9	1.403 (3)	C29—H295	1.000
C8—C16	1.404 (3)	C29—H296	1.000
O11···C231^{i}	3.29 (2)	O20···C4^{iv}	3.573 (2)
O11···C25^{i}	3.345 (3)	O22···C9^{ii}	3.292 (2)
O11···O22^{i}	3.436 (2)	O24···C21^{vi}	3.396 (5)
O11···C23^{i}	3.596 (8)	O26···C12^{i}	3.491 (3)
O13···C25^{ii}	3.259 (3)	O28···C29^{vii}	3.336 (3)
O13···O26^{ii}	3.330 (2)	O28···C21^{vi}	3.463 (3)
O13···O22^{ii}	3.415 (2)	O30···C15^{viii}	3.382 (2)
O13···C23^{ii}	3.431 (7)	O30···C12^{ix}	3.498 (3)
O13···C5^{ii}	3.550 (2)	O30···C16^{i}	3.555 (2)
O18···C25^{iii}	3.452 (2)	O241···C21^{vi}	3.26 (1)
O20···N6^{iv}	2.924 (2)	O241···C21^{x}	3.29 (2)
O20···C27^{iv}	3.077 (3)	N6···C15^{i}	3.506 (2)
O20···O26^{iv}	3.083 (2)	C1···C25^{iii}	3.420 (2)

O20···C12^{v}	3.411 (3)	C7···C15^{i}	3.546 (2)
O20···C29^{iv}	3.462 (3)	C12···C25^{ii}	3.546 (3)
O20···O28^{iv}	3.498 (2)	C17···C25^{iii}	3.453 (2)
C10—O11—C12	105.53 (16)	C15—C16—C8	119.93 (16)
C12—O13—C14	105.88 (16)	C15—C16—C17	120.78 (16)
C2—O18—C19	116.90 (14)	C8—C16—C17	119.25 (16)
C3—O22—C23	117.9 (2)	C5—C17—C16	116.14 (15)
C3—O22—C231	118.3 (6)	C5—C17—C1	121.16 (16)
C4—O26—C27	114.86 (15)	C16—C17—C1	122.67 (16)
C5—N6—C7	124.70 (17)	O18—C19—O20	122.92 (18)
C5—N6—H61	119.4 (19)	O18—C19—C21	111.74 (18)
C7—N6—H61	114.2 (19)	O20—C19—C21	125.3 (2)
C2—C1—C17	123.77 (16)	C19—C21—H211	109.5
C2—C1—H11	118.1	C19—C21—H212	109.5
C17—C1—H11	118.1	H211—C21—H212	109.5
C1—C2—O18	109.22 (14)	C19—C21—H213	109.5
C1—C2—C3	114.08 (15)	H211—C21—H213	109.5
O18—C2—C3	103.24 (14)	H212—C21—H213	109.5
C1—C2—H21	110.0	C19—C21—H214	109.5
O18—C2—H21	110.0	C19—C21—H215	109.5
C3—C2—H21	110.0	H214—C21—H215	109.5
C2—C3—O22	107.21 (14)	C19—C21—H216	109.5
C2—C3—C4	110.50 (14)	H214—C21—H216	109.5
O22—C3—C4	109.01 (14)	H215—C21—H216	109.5
C2—C3—H31	110.0	O22—C23—O24	122.8 (4)
O22—C3—H31	110.0	O22—C23—C25	111.4 (4)
C4—C3—H31	110.0	O24—C23—C25	125.8 (4)
C3—C4—O26	111.05 (13)	C23—C25—H251	109.5
C3—C4—C5	110.41 (15)	C23—C25—H252	109.5
O26—C4—C5	107.68 (15)	H251—C25—H252	109.5
C3—C4—H41	109.2	C23—C25—H253	109.5
O26—C4—H41	109.2	H251—C25—H253	109.5
C5—C4—H41	109.2	H252—C25—H253	109.5
C4—C5—N6	108.69 (15)	C23—C25—H254	109.5
C4—C5—C17	109.90 (15)	C23—C25—H255	109.5
N6—C5—C17	111.96 (14)	C23—C25—H256	109.5
C4—C5—H51	108.7	C231—C25—H257	109.5
N6—C5—H51	108.7	C231—C25—H258	109.5
C17—C5—H51	108.7	C231—C25—H259	109.5
N6—C7—O30	121.78 (18)	H254—C25—H255	109.5
N6—C7—C8	116.04 (16)	H254—C25—H256	109.5
O30—C7—C8	121.99 (17)	H255—C25—H256	109.5
C7—C8—C9	116.12 (16)	H257—C25—H258	109.5
C7—C8—C16	121.52 (16)	H257—C25—H259	109.5
C9—C8—C16	122.12 (17)	H258—C25—H259	109.5
C8—C9—C10	116.47 (18)	O26—C27—O28	123.0 (2)
C8—C9—H91	121.8	O26—C27—C29	112.24 (19)
C10—C9—H91	121.8	O28—C27—C29	124.7 (2)

O11—C10—C9	128.56 (19)	C27—C29—H291	109.5
O11—C10—C14	109.24 (17)	C27—C29—H292	109.5
C9—C10—C14	122.21 (17)	H291—C29—H292	109.5
O13—C12—O11	107.64 (16)	C27—C29—H293	109.5
O13—C12—H121	109.9	H291—C29—H293	109.5
O11—C12—H121	109.9	H292—C29—H293	109.5
O13—C12—H122	109.9	C27—C29—H294	109.5
O11—C12—H122	109.9	C27—C29—H295	109.5
H121—C12—H122	109.5	H294—C29—H295	109.5
C10—C14—O13	110.09 (17)	C27—C29—H296	109.5
C10—C14—C15	122.45 (18)	H294—C29—H296	109.5
O13—C14—C15	127.41 (17)	H295—C29—H296	109.5
C14—C15—C16	116.71 (16)	C25—C231—O241	126.5 (10)
C14—C15—H151	121.6	O22—C231—O241	122.7 (10)
C16—C15—H151	121.6		
O11—C10—C9—C8	177.6 (2)	C2—C1—C17—C16	177.3 (2)
O11—C10—C14—O13	1.4 (3)	C2—C3—O22—C23	136.8 (4)
O11—C10—C14—C15	−176.1 (2)	C2—C3—O22—C231	119.7 (8)
O11—C12—O13—C14	−11.9 (2)	C2—C3—C4—C5	61.3 (2)
O13—C12—O11—C10	12.7 (2)	C3—O22—C23—C25	−179.7 (3)
O13—C14—C10—C9	−178.7 (2)	C3—O22—C231—C25	168.0 (6)
O13—C14—C15—C16	−178.8 (2)	C3—C2—O18—C19	158.5 (1)
O18—C2—C1—C17	−105.6 (2)	C3—C2—C1—C17	9.3 (2)
O18—C2—C3—O22	−159.9 (1)	C3—C4—O26—C27	82.3 (2)
O18—C2—C3—C4	81.4 (2)	C3—C4—C5—C17	−55.6 (2)
O20—C19—O18—C2	−1.9 (3)	C4—O26—C27—C29	179.3 (2)
O22—C3—C2—C1	81.7 (2)	C4—C3—O22—C23	−103.6 (4)
O22—C3—C4—O26	63.1 (2)	C4—C3—O22—C231	−120.7 (8)
O22—C3—C4—C5	−56.3 (2)	C4—C5—N6—C7	158.5 (2)
O24—C23—O22—C3	−0(1)	C4—C5—C17—C16	−154.3 (1)
O24—C231—O22—C3	34 (2)	C5—N6—C7—C8	−17.3 (2)
O26—C4—C3—C2	−179.3 (1)	C5—C4—O26—C27	−156.7 (2)
O26—C4—C5—N6	60.2 (2)	C5—C17—C16—C8	14.3 (2)
O26—C4—C5—C17	−176.9 (1)	C5—C17—C16—C15	−167.9 (2)
O28—C27—O26—C4	−1.8 (3)	C7—N6—C5—C17	37.0 (2)
O30—C7—N6—C5	167.5 (2)	C7—C8—C9—C10	173.5 (2)
O30—C7—C8—C9	−5.5 (3)	C7—C8—C16—C15	−171.3 (2)
O30—C7—C8—C16	169.0 (2)	C7—C8—C16—C17	6.5 (2)
O241—C231—O22—C3	−11 (3)	C8—C9—C10—C14	−2.3 (3)
N6—C5—C4—C3	−178.4 (1)	C8—C16—C15—C14	−1.4 (3)
N6—C5—C17—C1	148.5 (2)	C9—C8—C16—C15	2.8 (3)
N6—C5—C17—C16	−33.4 (2)	C9—C8—C16—C17	−179.3 (2)
N6—C7—C8—C9	179.3 (2)	C9—C10—O11—C12	171.4 (2)
N6—C7—C8—C16	−6.2 (2)	C9—C10—C14—C15	3.8 (4)
C1—C2—O18—C19	−79.8 (2)	C10—C9—C8—C16	−1.0 (3)
C1—C2—C3—C4	−37.0 (2)	C10—C14—O13—C12	6.6 (2)
C1—C17—C5—C4	27.7 (2)	C10—C14—C15—C16	−1.8 (3)
C1—C17—C16—C8	−167.7 (2)	C12—O11—C10—C14	−8.8 (2)
C1—C17—C16—C15	10.1 (3)	C12—O13—C14—C15	−176.1 (2)

C2—O18—C19—C21	178.4 (2)	C14—C15—C16—C17	−179.2 (2)
C2—C1—C17—C5	−4.8 (3)		

Symmetry codes: (i) *x*+1/2, −*y*+3/2, −*z*+1; (ii) *x*−1/2, −*y*+3/2, −*z*+1; (iii) *x*, *y*+1, *z*; (iv) *x*−1, *y*, *z*; (v) *x*−1/2, −*y*+5/2, −*z*+1; (vi) −*x*+1, *y*−1/2, −*z*+1/2; (vii) −*x*+2, *y*+1/2, −*z*+1/2; (viii) *x*+1, *y*, *z*; (ix) *x*+1/2, −*y*+5/2, −*z*+1; (x) *x*, *y*−1, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H61···O20^{viii}	0.79 (3)	2.25 (3)	2.924 (2)	143 (2)
N6—H61···O26	0.79 (3)	2.50 (3)	2.795 (2)	103 (2)

Symmetry codes: (viii) *x*+1, *y*, *z*.

Crystal structure of $C_{20}H_{14}BrClN_2O_8$ — ban0810

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Abstract

The crystal structure of $C_{20}H_{14}BrClN_2O_8$ is reported.

Comment

The crystallographic asymmetric unit consists of one molecule of $C_{20}H_{14}BrClN_2O_8$.

Experimental

The compound was prepared by *MM* and recrystallized from ethylacetate/hexane. The sample ID is *MM6*–506.

Refinement

The compound is enantiometrically pure. The absolute structure of the crystal has been determined by refinement of the Flack parameter (Flack, 1983) and this establishes the absolute configuration of the molecule.

All hydrogen atoms were observed in difference electron density maps prior to their inclusion. H atoms were included at calculated positions and then were refined positionally with $U_{iso}(H) = 1.2 \times U_{eq}$(adjacent atom).

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

(ban0810)

Crystal data

$C_{20}H_{14}BrClN_2O_8$ $V = 2080.18\ (6)\ Å^3$

M_r = 525.70	Z = 4
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$
a = 6.8298 (1) Å	μ = 2.16 mm^{-1}
b = 14.9660 (3) Å	T = 200 K
c = 20.3511 (3) Å	0.46 × 0.10 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer	6060 independent reflections
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	5043 reflections with $I > 2.0\sigma(I)$
T_{min} = 0.554, T_{max} = 0.836	R_{int} = 0.043
28527 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026	Only H-atom coordinates refined
wR = 0.030	$\Delta\rho_{max}$ = 0.54 e Å^{-3}
S = 1.11	$\Delta\rho_{min}$ = −0.79 e Å^{-3}
5043 reflections	Absolute structure: Flack (1983), 2628 Friedel-pairs
332 parameters	Flack parameter: −0.014 (5)

Table 1
Selected geometric parameters (Å, °)

Br8—C4	1.9037 (14)	C3—C4	1.501 (2)
Cl7—C3	1.8150 (15)	C4—C5	1.324 (2)
O9—C1	1.4519 (19)	C5—C6	1.503 (2)
O9—C10	1.3417 (19)	C10—C12	1.488 (2)
O11—C10	1.197 (2)	C12—C13	1.395 (2)
O19—N18	1.204 (3)	C12—C17	1.401 (2)
O20—N18	1.212 (2)	C13—C14	1.388 (2)
O21—C2	1.4464 (18)	C14—C15	1.383 (2)
O21—C22	1.3577 (19)	C15—C16	1.373 (3)
O23—C22	1.206 (2)	C16—C17	1.392 (2)
O31—N30	1.214 (3)	C22—C24	1.491 (2)
O32—N30	1.216 (3)	C24—C25	1.393 (2)
N18—C15	1.475 (2)	C24—C29	1.400 (2)
N30—C27	1.472 (2)	C25—C26	1.386 (2)
C1—C2	1.517 (2)	C26—C27	1.380 (2)
C1—C6	1.521 (2)	C27—C28	1.385 (3)
C2—C3	1.540 (2)	C28—C29	1.387 (3)
C1—O9—C10	117.32 (13)	O11—C10—C12	124.08 (16)
C2—O21—C22	118.03 (12)	C10—C12—C13	121.13 (14)
O20—N18—O19	123.17 (19)	C10—C12—C17	118.08 (15)
O20—N18—C15	118.50 (19)	C13—C12—C17	120.71 (15)
O19—N18—C15	118.33 (17)	C12—C13—C14	119.75 (14)

O32—N30—O31	123.32 (18)	C13—C14—C15	118.21 (16)
O32—N30—C27	118.10 (19)	N18—C15—C14	117.43 (17)
O31—N30—C27	118.57 (17)	N18—C15—C16	119.05 (15)
O9—C1—C2	105.21 (12)	C14—C15—C16	123.52 (16)
O9—C1—C6	111.05 (13)	C15—C16—C17	118.33 (15)
C2—C1—C6	111.79 (13)	C12—C17—C16	119.46 (16)
C1—C2—O21	109.88 (13)	O21—C22—O23	124.40 (15)
C1—C2—C3	110.39 (12)	O21—C22—C24	110.77 (13)
O21—C2—C3	104.72 (12)	O23—C22—C24	124.83 (14)
C2—C3—Cl7	108.68 (11)	C22—C24—C25	121.26 (13)
C2—C3—C4	112.05 (12)	C22—C24—C29	118.15 (14)
Cl7—C3—C4	109.87 (10)	C25—C24—C29	120.60 (15)
Br8—C4—C3	115.35 (11)	C24—C25—C26	120.02 (13)
Br8—C4—C5	119.96 (12)	C25—C26—C27	118.41 (15)
C3—C4—C5	124.68 (14)	N30—C27—C26	117.77 (16)
C4—C5—C6	122.72 (15)	N30—C27—C28	119.40 (15)
C1—C6—C5	111.02 (14)	C26—C27—C28	122.83 (15)
O9—C10—O11	124.63 (17)	C27—C28—C29	118.66 (15)
O9—C10—C12	111.29 (14)	C24—C29—C28	119.47 (16)

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supplementary materials

Crystal structure of $C_{20}H_{14}BrClN_2O_8$ — ban0810

Maria Matveenko, Martin G. Banwell and Anthony C. Willis

(ban0810)

Crystal data

$C_{20}H_{14}BrClN_2O_8$	$D_x = 1.678$ Mg m^{-3}
$M_r = 525.70$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 29036 reflections
$a = 6.8298$ (1) Å	$\theta = 2.6$–30º
$b = 14.9660$ (3) Å	$\mu = 2.16$ mm^{-1}
$c = 20.3511$ (3) Å	$T = 200$ K
$V = 2080.18$ (6) Å^3	Needle, colourless
$Z = 4$	0.46 × 0.10 × 0.10 mm
$F_{000} = 1056$	

Data collection

Nonius KappaCCD diffractometer	5043 reflections with $I > 2.0\sigma(I)$
Monochromator: graphite	$R_{int} = 0.043$
$T = 200$ K	$\theta_{max} = 30.0$º
φ and ω scans with CCD	$\theta_{min} = 2.7$º
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	$h = -9 \rightarrow 9$
$T_{min} = 0.554$, $T_{max} = 0.836$	$k = -20 \rightarrow 21$
28527 measured reflections	$l = -28 \rightarrow 28$
6060 independent reflections	

Refinement

Refinement on F	Only H-atom coordinates refined
Least-squares matrix: full	Method, part 1, Chebychev polynomial, (Watkin, 1994, *P*rince, 1982) [*w*eight] = 1.0/[A_0*T_0(x) + A_1*T_1(x) ··· + A_{n-1}]*T_{n-1}(x)] where A_i are the Chebychev coefficients listed below and x = F /Fmax Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta$F$/6*sigma$F$)^2]^2 A_i are: 2.04 -0.263 1.78
$R[F^2 > 2\sigma(F^2)] = 0.026$	$(\Delta/\sigma)_{max} = 0.003$
wR = 0.030	$\Delta\rho_{max} = 0.54$ e Å^{-3}
$S = 1.11$	$\Delta\rho_{min} = -0.79$ e Å^{-3}
5043 reflections	Extinction correction: None

332 parameters	Absolute structure: Flack (1983), 2628 Friedel-pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.014 (5)
Hydrogen site location: inferred from neighbouring sites	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br8	0.70273 (3)	0.537712 (13)	0.944739 (9)	0.0449
Cl7	1.08490 (6)	0.57245 (3)	0.83102 (2)	0.0416
O9	0.7144 (2)	0.68614 (7)	0.66598 (6)	0.0356
O11	0.7767 (4)	0.83227 (10)	0.68115 (8)	0.0711
O19	0.7174 (5)	0.73629 (15)	0.33476 (9)	0.0927
O20	0.7052 (3)	0.87771 (13)	0.34307 (9)	0.0655
O21	0.62714 (17)	0.51623 (7)	0.71925 (5)	0.0313
O23	0.74596 (17)	0.49804 (8)	0.61599 (6)	0.0356
O31	−0.2100 (2)	0.29173 (10)	0.64945 (9)	0.0525
O32	−0.1094 (3)	0.26118 (16)	0.55250 (10)	0.0743
N18	0.7125 (3)	0.80366 (13)	0.36705 (8)	0.0482
N30	−0.0888 (3)	0.29626 (11)	0.60593 (9)	0.0437
C1	0.7447 (2)	0.66763 (10)	0.73527 (8)	0.0314
C2	0.7948 (2)	0.56901 (10)	0.73869 (7)	0.0288
C3	0.8358 (2)	0.54109 (10)	0.81023 (7)	0.0304
C4	0.6954 (2)	0.58362 (10)	0.85754 (7)	0.0312
C5	0.5736 (2)	0.64933 (11)	0.84256 (8)	0.0336
C6	0.5625 (3)	0.68966 (12)	0.77505 (9)	0.0357
C10	0.7418 (3)	0.77049 (11)	0.64565 (9)	0.0376
C12	0.7253 (2)	0.77757 (10)	0.57291 (8)	0.0332
C13	0.7209 (3)	0.70145 (10)	0.53338 (8)	0.0334
C14	0.7175 (3)	0.71031 (11)	0.46553 (9)	0.0364
C15	0.7164 (3)	0.79556 (12)	0.43928 (9)	0.0370
C16	0.7170 (3)	0.87185 (11)	0.47677 (9)	0.0396
C17	0.7241 (3)	0.86308 (11)	0.54485 (9)	0.0384
C22	0.6192 (2)	0.48627 (10)	0.65634 (8)	0.0295
C24	0.4337 (2)	0.43638 (10)	0.64429 (7)	0.0283
C25	0.2907 (2)	0.42893 (10)	0.69282 (7)	0.0305
C26	0.1189 (2)	0.38280 (11)	0.68007 (8)	0.0328
C27	0.0942 (2)	0.34523 (11)	0.61868 (9)	0.0330
C28	0.2343 (3)	0.35101 (13)	0.56965 (9)	0.0394
C29	0.4066 (3)	0.39677 (12)	0.58264 (8)	0.0356
H11	0.868 (4)	0.6993 (16)	0.7497 (11)	0.0376*
H21	0.903 (3)	0.5508 (16)	0.7105 (11)	0.0346*
H31	0.837 (3)	0.4753 (16)	0.8135 (12)	0.0364*
H51	0.486 (4)	0.6693 (17)	0.8718 (13)	0.0404*
H61	0.536 (4)	0.7555 (19)	0.7771 (13)	0.0428*
H62	0.441 (4)	0.6648 (17)	0.7545 (13)	0.0428*
H131	0.722 (4)	0.6443 (17)	0.5530 (12)	0.0400*
H141	0.711 (4)	0.6604 (17)	0.4366 (13)	0.0437*

H161	0.699 (4)	0.9310 (19)	0.4565 (12)	0.0476*
H171	0.728 (4)	0.9144 (18)	0.5727 (13)	0.0461*
H251	0.304 (3)	0.4541 (14)	0.7348 (11)	0.0366*
H261	0.025 (4)	0.3744 (16)	0.7104 (12)	0.0394*
H281	0.212 (4)	0.3242 (17)	0.5283 (13)	0.0472*
H291	0.510 (4)	0.4029 (17)	0.5507 (13)	0.0428*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br8	0.05866 (11)	0.04393 (9)	0.03201 (7)	−0.00228 (8)	−0.00189 (7)	0.00580 (7)
Cl7	0.03200 (17)	0.0481 (2)	0.0446 (2)	0.00087 (16)	−0.00751 (15)	−0.00354 (17)
O9	0.0457 (6)	0.0291 (5)	0.0321 (5)	−0.0033 (5)	−0.0026 (5)	0.0039 (4)
O11	0.140 (2)	0.0319 (6)	0.0417 (7)	−0.0098 (9)	−0.0056 (10)	0.0007 (5)
O19	0.171 (3)	0.0694 (11)	0.0380 (8)	−0.0352 (15)	0.0022 (12)	0.0060 (8)
O20	0.0725 (10)	0.0703 (10)	0.0536 (9)	0.0093 (10)	0.0038 (9)	0.0319 (8)
O21	0.0335 (5)	0.0322 (5)	0.0283 (5)	−0.0064 (4)	−0.0012 (4)	−0.0035 (4)
O23	0.0374 (6)	0.0352 (5)	0.0342 (5)	−0.0045 (4)	0.0037 (4)	−0.0022 (4)
O31	0.0379 (6)	0.0444 (7)	0.0752 (10)	−0.0095 (6)	0.0007 (8)	0.0019 (6)
O32	0.0723 (12)	0.0906 (13)	0.0602 (10)	−0.0337 (11)	−0.0167 (9)	−0.0212 (10)
N18	0.0482 (8)	0.0574 (10)	0.0391 (7)	−0.0098 (8)	−0.0012 (7)	0.0145 (7)
N30	0.0404 (8)	0.0369 (7)	0.0539 (9)	−0.0046 (6)	−0.0140 (7)	0.0011 (6)
C1	0.0368 (9)	0.0269 (6)	0.0304 (6)	−0.0016 (5)	0.0000 (5)	0.0013 (5)
C2	0.0291 (6)	0.0273 (5)	0.0301 (6)	−0.0043 (6)	−0.0029 (6)	−0.0027 (5)
C3	0.0331 (7)	0.0258 (6)	0.0324 (6)	−0.0011 (5)	−0.0048 (5)	−0.0004 (6)
C4	0.0336 (7)	0.0307 (7)	0.0294 (6)	−0.0043 (6)	−0.0023 (6)	−0.0004 (5)
C5	0.0333 (7)	0.0336 (7)	0.0339 (7)	−0.0014 (6)	0.0007 (6)	−0.0024 (6)
C6	0.0369 (8)	0.0340 (8)	0.0361 (7)	0.0056 (6)	0.0009 (6)	0.0032 (6)
C10	0.0487 (10)	0.0268 (7)	0.0374 (8)	0.0007 (6)	0.0002 (6)	0.0036 (6)
C12	0.0329 (7)	0.0297 (7)	0.0370 (7)	0.0030 (6)	0.0017 (6)	0.0057 (5)
C13	0.0362 (7)	0.0282 (6)	0.0357 (7)	0.0011 (6)	0.0020 (6)	0.0040 (5)
C14	0.0371 (8)	0.0351 (7)	0.0370 (7)	−0.0023 (7)	0.0018 (7)	0.0026 (6)
C15	0.0301 (7)	0.0441 (8)	0.0368 (7)	−0.0014 (6)	0.0015 (7)	0.0112 (6)
C16	0.0386 (8)	0.0365 (7)	0.0438 (8)	−0.0007 (7)	0.0004 (7)	0.0142 (6)
C17	0.0438 (8)	0.0285 (6)	0.0430 (9)	0.0022 (6)	0.0029 (7)	0.0065 (6)
C22	0.0327 (7)	0.0266 (6)	0.0292 (6)	0.0018 (5)	−0.0022 (5)	−0.0017 (5)
C24	0.0303 (6)	0.0258 (6)	0.0288 (6)	0.0027 (5)	−0.0013 (5)	−0.0022 (5)
C25	0.0326 (6)	0.0306 (6)	0.0283 (6)	−0.0016 (6)	−0.0011 (6)	−0.0036 (5)
C26	0.0315 (7)	0.0329 (7)	0.0341 (7)	0.0014 (6)	−0.0001 (6)	−0.0007 (6)
C27	0.0294 (7)	0.0295 (7)	0.0401 (8)	0.0003 (6)	−0.0066 (6)	−0.0023 (6)
C28	0.0407 (9)	0.0432 (8)	0.0342 (8)	0.0016 (7)	−0.0043 (6)	−0.0123 (6)
C29	0.0337 (7)	0.0438 (8)	0.0294 (7)	0.0001 (7)	0.0002 (6)	−0.0078 (6)

Geometric parameters (Å, °)

Br8—C4	1.9037 (14)	C6—H61	1.00 (3)
Cl7—C3	1.8150 (15)	C6—H62	1.00 (3)
O9—C1	1.4519 (19)	C10—C12	1.488 (2)
O9—C10	1.3417 (19)	C12—C13	1.395 (2)

O11—C10	1.197 (2)	C12—C17	1.401 (2)
O19—N18	1.204 (3)	C13—C14	1.388 (2)
O20—N18	1.212 (2)	C13—H131	0.94 (2)
O21—C2	1.4464 (18)	C14—C15	1.383 (2)
O21—C22	1.3577 (19)	C14—H141	0.95 (3)
O23—C22	1.206 (2)	C15—C16	1.373 (3)
O31—N30	1.214 (3)	C16—C17	1.392 (2)
O32—N30	1.216 (3)	C16—H161	0.98 (3)
N18—C15	1.475 (2)	C17—H171	0.95 (3)
N30—C27	1.472 (2)	C22—C24	1.491 (2)
C1—C2	1.517 (2)	C24—C25	1.393 (2)
C1—C6	1.521 (2)	C24—C29	1.400 (2)
C1—H11	1.01 (2)	C25—C26	1.386 (2)
C2—C3	1.540 (2)	C25—H251	0.94 (2)
C2—H21	0.97 (2)	C26—C27	1.380 (2)
C3—C4	1.501 (2)	C26—H261	0.90 (3)
C3—H31	0.99 (2)	C27—C28	1.385 (3)
C4—C5	1.324 (2)	C28—C29	1.387 (3)
C5—C6	1.503 (2)	C28—H281	0.94 (3)
C5—H51	0.90 (3)	C29—H291	0.97 (3)
Br8···O23^{i}	3.543 (1)	O20···C1^{v}	3.591 (2)
Cl7···O31^{ii}	3.414 (2)	O23···O31^{iii}	3.176 (2)
Cl7···C5^{iii}	3.538 (2)	O23···N30^{iii}	3.230 (2)
Cl7···N30^{ii}	3.587 (2)	O23···C27^{iii}	3.300 (2)
O9···N18^{iv}	3.471 (2)	O23···C26^{iii}	3.341 (2)
O9···O20^{iv}	3.490 (2)	O31···C22^{vi}	3.140 (2)
O9···N18^{v}	3.497 (2)	O31···C6^{vii}	3.239 (2)
O9···O19^{v}	3.588 (4)	O31···C24^{vi}	3.259 (2)
O11···C25^{ii}	2.980 (2)	O31···C5^{vii}	3.276 (2)
O11···C26^{ii}	3.010 (2)	O31···C29^{vi}	3.343 (2)
O11···O19^{iv}	3.196 (4)	O32···C28^{viii}	3.184 (3)
O19···C10^{v}	3.274 (4)	N18···C10^{v}	3.411 (3)
O19···C6^{iv}	3.432 (3)	C12···C14^{iv}	3.456 (2)
O20···C25^{iv}	3.041 (2)	C12···C15^{iv}	3.537 (2)
O20···C24^{iv}	3.201 (2)	C12···C14^{v}	3.560 (2)
O20···C2^{v}	3.356 (2)	C13···C15^{iv}	3.430 (3)
O20···C22^{iv}	3.484 (2)	C13···C15^{v}	3.491 (3)
O20···O21^{iv}	3.526 (2)	C13···C16^{iv}	3.567 (3)
O20···C6^{iv}	3.571 (3)	C14···C17^{v}	3.551 (3)
C1—O9—C10	117.32 (13)	O11—C10—C12	124.08 (16)
C2—O21—C22	118.03 (12)	C10—C12—C13	121.13 (14)
O20—N18—O19	123.17 (19)	C10—C12—C17	118.08 (15)
O20—N18—C15	118.50 (19)	C13—C12—C17	120.71 (15)
O19—N18—C15	118.33 (17)	C12—C13—C14	119.75 (14)
O32—N30—O31	123.32 (18)	C12—C13—H131	119.7 (15)

O32—N30—C27	118.10 (19)	C14—C13—H131	120.5 (15)
O31—N30—C27	118.57 (17)	C13—C14—C15	118.21 (16)
O9—C1—C2	105.21 (12)	C13—C14—H141	122.8 (15)
O9—C1—C6	111.05 (13)	C15—C14—H141	119.0 (15)
C2—C1—C6	111.79 (13)	N18—C15—C14	117.43 (17)
O9—C1—H11	108.2 (13)	N18—C15—C16	119.05 (15)
C2—C1—H11	104.8 (13)	C14—C15—C16	123.52 (16)
C6—C1—H11	115.2 (13)	C15—C16—C17	118.33 (15)
C1—C2—O21	109.88 (13)	C15—C16—H161	120.9 (15)
C1—C2—C3	110.39 (12)	C17—C16—H161	120.4 (15)
O21—C2—C3	104.72 (12)	C12—C17—C16	119.46 (16)
C1—C2—H21	114.6 (14)	C12—C17—H171	119.5 (16)
O21—C2—H21	106.7 (14)	C16—C17—H171	121.0 (16)
C3—C2—H21	110.1 (13)	O21—C22—O23	124.40 (15)
C2—C3—Cl7	108.68 (11)	O21—C22—C24	110.77 (13)
C2—C3—C4	112.05 (12)	O23—C22—C24	124.83 (14)
Cl7—C3—C4	109.87 (10)	C22—C24—C25	121.26 (13)
C2—C3—H31	109.7 (14)	C22—C24—C29	118.15 (14)
Cl7—C3—H31	103.5 (14)	C25—C24—C29	120.60 (15)
C4—C3—H31	112.7 (14)	C24—C25—C26	120.02 (13)
Br8—C4—C3	115.35 (11)	C24—C25—H251	123.0 (14)
Br8—C4—C5	119.96 (12)	C26—C25—H251	117.0 (14)
C3—C4—C5	124.68 (14)	C25—C26—C27	118.41 (15)
C4—C5—C6	122.72 (15)	C25—C26—H261	123.0 (16)
C4—C5—H51	121.0 (16)	C27—C26—H261	118.5 (16)
C6—C5—H51	116.1 (16)	N30—C27—C26	117.77 (16)
C1—C6—C5	111.02 (14)	N30—C27—C28	119.40 (15)
C1—C6—H61	112.5 (15)	C26—C27—C28	122.83 (15)
C5—C6—H61	111.4 (15)	C27—C28—C29	118.66 (15)
C1—C6—H62	112.0 (15)	C27—C28—H281	120.2 (17)
C5—C6—H62	106.0 (15)	C29—C28—H281	121.2 (17)
H61—C6—H62	104 (2)	C24—C29—C28	119.47 (16)
O9—C10—O11	124.63 (17)	C24—C29—H291	117.7 (15)
O9—C10—C12	111.29 (14)	C28—C29—H291	122.8 (15)
Br8—C4—C3—Cl7	−69.8 (1)	N30—C27—C26—C25	179.9 (1)
Br8—C4—C3—C2	169.3 (1)	N30—C27—C28—C29	−179.5 (2)
Br8—C4—C5—C6	179.0 (1)	C1—O9—C10—C12	174.3 (1)
Cl7—C3—C2—O21	160.91 (9)	C1—C2—O21—C22	97.6 (1)
Cl7—C3—C2—C1	−80.9 (1)	C1—C2—C3—C4	40.7 (2)
Cl7—C3—C4—C5	109.4 (1)	C1—C6—C5—C4	−18.1 (2)
O9—C1—C2—O21	−66.4 (1)	C2—O21—C22—C24	−177.8 (1)
O9—C1—C2—C3	178.6 (1)	C2—C1—O9—C10	−155.2 (1)
O9—C1—C6—C5	165.4 (1)	C2—C1—C6—C5	48.2 (2)
O9—C10—C12—C13	−11.5 (2)	C2—C3—C4—C5	−11.5 (2)
O9—C10—C12—C17	171.6 (2)	C3—C2—O21—C22	−143.9 (1)
O11—C10—O9—C1	−5.1 (3)	C3—C2—C1—C6	−60.8 (2)
O11—C10—C12—C13	167.9 (2)	C3—C4—C5—C6	−0.1 (2)
O11—C10—C12—C17	−9.0 (3)	C6—C1—O9—C10	83.7 (2)
O19—N18—C15—C14	−2.6 (3)	C10—C12—C13—C14	−176.1 (2)

O19—N18—C15—C16	178.0 (3)	C10—C12—C17—C16	177.4 (2)
O20—N18—C15—C14	177.7 (2)	C12—C13—C14—C15	−0.7 (3)
O20—N18—C15—C16	−1.7 (3)	C12—C17—C16—C15	−1.6 (3)
O21—C2—C1—C6	54.2 (2)	C13—C12—C17—C16	0.5 (3)
O21—C2—C3—C4	−77.5 (1)	C13—C14—C15—C16	−0.5 (3)
O21—C22—C24—C25	3.2 (2)	C14—C13—C12—C17	0.7 (3)
O21—C22—C24—C29	−176.7 (1)	C14—C15—C16—C17	1.7 (3)
O23—C22—O21—C2	2.7 (2)	C22—C24—C25—C26	179.4 (1)
O23—C22—C24—C25	−177.3 (1)	C22—C24—C29—C28	−179.1 (2)
O23—C22—C24—C29	2.8 (2)	C24—C25—C26—C27	−0.1 (2)
O31—N30—C27—C26	0.6 (2)	C24—C29—C28—C27	−0.6 (3)
O31—N30—C27—C28	179.9 (2)	C25—C24—C29—C28	1.0 (3)
O32—N30—C27—C26	−178.3 (2)	C25—C26—C27—C28	0.6 (3)
O32—N30—C27—C28	1.0 (3)	C26—C25—C24—C29	−0.7 (2)
N18—C15—C14—C13	−179.9 (2)	C26—C27—C28—C29	−0.2 (3)
N18—C15—C16—C17	−178.9 (2)		

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

Crystal structure of $C_9H_7IO_5$ — ban0739A

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Abstract

The crystal structure of $C_9H_7IO_5$ is reported.

Comment

The crystallographic asymmetric unit consists of one molecule of $C_9H_7IO_5$.

Experimental

The compound was prepared by *MM* and recrystallized from hexanes/dichloromethane. The sample ID is *MM6*–535.

Refinement

The space group is noncentrosymmetric, so the Flack parameter has been refined. (Flack, 1983) The molecule itself has no chiral centres.

A difference electron-density map revealed most H atoms, including the preferred orientations of the methyl group. H atoms were included at calculated positions and then were refined positionally with $U_{iso}(H) = 1.2 \times U_{eq}$(adjacent atom).

The largest peaks in the final difference electron density map are located close to the I atom.

Computing details

Data collection: *COLLECT* (Nonius BV, 1997); 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* (Watkin *et al.* 2003); molecular graphics: *ORTEP*II (Johnson 1976) in *TEXSAN* (MSC, 1992-1997); software used to prepare material for publication: *CRYSTALS* (Watkin *et al.* 2003).

(ban0739A)

Crystal data

$C_9H_7IO_5$ $V = 958.11\ (7)\ Å^3$

M_r = 322.06	Z = 4
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$
a = 4.1771 (2) Å	μ = 3.34 mm^{-1}
b = 6.9480 (3) Å	T = 200 K
c = 33.0128 (15) Å	0.44 × 0.14 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer	2147 independent reflections
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	1877 reflections with $I > 3.0\sigma(I)$
T_{min} = 0.482, T_{max} = 0.861	R_{int} = 0.122
10078 measured reflections	

Refinement

R = 0.030	Only H-atom coordinates refined
wR = 0.034	$\Delta\rho_{max}$ = 0.92 e Å^{-3}
S = 1.12	$\Delta\rho_{min}$ = −1.14 e Å^{-3}
1877 reflections	Absolute structure: Flack (1983), 807 Friedel-pairs
158 parameters	Flack parameter: −0.02 (4)

Selected geometric parameters (Å, °)

I15—C10	2.119 (4)	O13—C14	1.446 (6)
O5—C4	1.385 (6)	C1—C2	1.478 (6)
O5—C6	1.446 (7)	C2—C3	1.423 (7)
O7—C6	1.445 (7)	C2—C10	1.427 (7)
O7—C8	1.374 (6)	C3—C4	1.375 (7)
O11—C3	1.354 (6)	C4—C8	1.365 (8)
O12—C1	1.216 (7)	C8—C9	1.381 (7)
O13—C1	1.331 (6)	C9—C10	1.394 (7)
C4—O5—C6	104.9 (4)	O5—C4—C3	126.6 (5)
C6—O7—C8	105.6 (4)	O5—C4—C8	110.8 (5)
C1—O13—C14	115.3 (4)	C3—C4—C8	122.5 (5)
O13—C1—O12	121.5 (4)	O5—C6—O7	107.9 (4)
O13—C1—C2	115.1 (4)	O7—C8—C4	110.4 (4)
O12—C1—C2	123.4 (4)	O7—C8—C9	126.9 (5)
C1—C2—C3	115.5 (4)	C4—C8—C9	122.6 (5)
C1—C2—C10	126.3 (4)	C8—C9—C10	116.4 (5)
C3—C2—C10	118.2 (4)	C2—C10—I15	125.9 (4)
C2—C3—O11	124.8 (4)	C2—C10—C9	122.5 (4)
C2—C3—C4	117.8 (4)	I15—C10—C9	111.6 (4)
O11—C3—C4	117.4 (4)		

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supplementary materials

Crystal structure of $C_9H_7IO_5$ — ban0739A

Maria Matveenko, Martin G. Banwell and Anthony C. Willis

(ban0739A)

Crystal data

$C_9H_7IO_5$	$D_x = 2.233$ Mg m^{-3}
$M_r = 322.06$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 35510 reflections
$a = 4.1771$ (2) Å	$\theta = 2.6$–$27.5°$
$b = 6.9480$ (3) Å	$\mu = 3.34$ mm^{-1}
$c = 33.0128$ (15) Å	$T = 200$ K
$V = 958.11$ (7) Å^3	Needle, colourless
$Z = 4$	0.44 × 0.14 × 0.05 mm
$F_{000} = 616$	

Data collection

Nonius KappaCCD diffractometer	1877 reflections with $I > 3.0\sigma(I)$
Monochromator: graphite	$R_{int} = 0.122$
$T = 200$ K	$\theta_{max} = 27.4°$
φ and ω scans with CCD	$\theta_{min} = 3.2°$
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	$h = -5 \rightarrow 5$
$T_{min} = 0.482$, $T_{max} = 0.861$	$k = -9 \rightarrow 9$
10078 measured reflections	$l = -41 \rightarrow 42$
2147 independent reflections	

Refinement

Refinement on F	Only H-atom coordinates refined
Least-squares matrix: full	Method, part 1, Chebychev polynomial, (Carruthers & Watkin, 1979, *P*rince, 1982) [*w*eight] = 1.0/ $[A_0*T_0(x) + A_1*T_1(x) \cdots + A_{n-1}]*T_{n-1}(x)]$ where A_i are the Chebychev coefficients listed below and x = F /Fmax Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta*F*/6*sigma*F*)^2]^2 A_i are: 0.910 -0.188 0.611
R = 0.030	$(\Delta/\sigma)_{max} = 0.002$
wR = 0.034	$\Delta\rho_{max} = 0.92$ e Å^{-3}
$S = 1.12$	$\Delta\rho_{min} = -1.14$ e Å^{-3}
1877 reflections	Extinction correction: None

158 parameters
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
Absolute structure: Flack (1983), 807 Friedel-pairs
Flack parameter: −0.02 (4)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
I15	0.34757 (7)	0.30827 (4)	0.452591 (9)	0.0294
O5	0.8066 (10)	0.1784 (6)	0.27591 (10)	0.0374
O7	0.4881 (11)	−0.0498 (6)	0.30876 (11)	0.0412
O11	1.0002 (11)	0.5311 (6)	0.31079 (11)	0.0368
O12	1.0163 (11)	0.7312 (6)	0.37533 (13)	0.0406
O13	0.7102 (9)	0.6607 (5)	0.42837 (11)	0.0332
C1	0.8276 (13)	0.6232 (7)	0.39173 (15)	0.0284
C2	0.7136 (11)	0.4422 (7)	0.37319 (14)	0.0260
C3	0.8176 (14)	0.4085 (7)	0.33276 (14)	0.0289
C4	0.7259 (13)	0.2394 (7)	0.31451 (16)	0.0311
C6	0.6402 (18)	−0.0023 (8)	0.27075 (16)	0.0374
C8	0.5395 (13)	0.1059 (7)	0.33358 (15)	0.0291
C9	0.4324 (14)	0.1289 (8)	0.37288 (16)	0.0319
C10	0.5209 (11)	0.2990 (7)	0.39231 (13)	0.0263
C14	0.8519 (16)	0.8239 (8)	0.44881 (17)	0.0368
H1	1.081 (17)	0.613 (11)	0.328 (2)	0.0440*
H61	0.795 (17)	−0.096 (10)	0.259 (2)	0.0450*
H62	0.457 (18)	0.006 (11)	0.251 (2)	0.0450*
H91	0.310 (16)	0.033 (10)	0.387 (2)	0.0380*
H141	0.764 (18)	0.821 (11)	0.470 (2)	0.0480*
H142	1.05 (2)	0.805 (11)	0.451 (2)	0.0480*
H143	0.816 (19)	0.933 (12)	0.436 (2)	0.0480*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I15	0.03242 (15)	0.02954 (14)	0.02612 (14)	−0.00104 (13)	0.00266 (13)	−0.00166 (13)
O5	0.054 (2)	0.0347 (18)	0.0234 (15)	−0.006 (2)	0.0076 (16)	−0.0067 (15)
O7	0.063 (2)	0.0326 (19)	0.0282 (19)	−0.015 (2)	0.0068 (18)	−0.0086 (16)
O11	0.052 (2)	0.032 (2)	0.0265 (18)	−0.0105 (18)	0.0063 (18)	0.0006 (15)
O12	0.053 (2)	0.0310 (19)	0.038 (2)	−0.0135 (17)	0.0050 (18)	−0.0044 (15)
O13	0.042 (2)	0.0269 (17)	0.0304 (17)	−0.0048 (15)	0.0063 (15)	−0.0058 (14)
C1	0.030 (2)	0.0243 (19)	0.031 (2)	−0.002 (2)	−0.003 (2)	−0.0012 (17)
C2	0.029 (2)	0.026 (2)	0.023 (2)	−0.0029 (19)	−0.0025 (17)	0.0009 (18)
C3	0.035 (3)	0.024 (2)	0.028 (2)	−0.007 (2)	0.004 (2)	0.0017 (17)
C4	0.038 (3)	0.029 (2)	0.027 (2)	0.0001 (18)	−0.0003 (19)	0.0002 (18)
C6	0.055 (3)	0.032 (2)	0.025 (2)	−0.006 (3)	0.007 (3)	−0.0083 (19)
C8	0.038 (2)	0.025 (2)	0.025 (2)	0.000 (2)	−0.002 (2)	−0.0035 (18)
C9	0.042 (3)	0.026 (2)	0.027 (2)	−0.0028 (19)	−0.001 (2)	−0.0013 (18)
C10	0.031 (2)	0.025 (2)	0.022 (2)	0.001 (2)	0.0029 (17)	0.001 (2)

C14	0.047 (3)	0.028 (2)	0.035 (2)	−0.005 (3)	−0.005 (3)	−0.008 (2)

Geometric parameters (Å, °)

I15—C10	2.119 (4)	C2—C10	1.427 (7)
O5—C4	1.385 (6)	C3—C4	1.375 (7)
O5—C6	1.446 (7)	C4—C8	1.365 (8)
O7—C6	1.445 (7)	C6—H61	1.00 (7)
O7—C8	1.374 (6)	C6—H62	1.02 (8)
O11—C3	1.354 (6)	C8—C9	1.381 (7)
O11—H1	0.86 (8)	C9—C10	1.394 (7)
O12—C1	1.216 (7)	C9—H91	0.97 (7)
O13—C1	1.331 (6)	C14—H141	0.80 (8)
O13—C14	1.446 (6)	C14—H142	0.85 (9)
C1—C2	1.478 (6)	C14—H143	0.87 (8)
C2—C3	1.423 (7)		
O5···O11^{i}	3.145 (5)	O11···C3^{iii}	3.593 (7)
O5···C6^{ii}	3.283 (7)	O12···C9^{vii}	3.265 (7)
O5···O7^{iii}	3.434 (6)	O12···O13^{iii}	3.422 (6)
O5···C6^{iv}	3.555 (8)	O12···C1^{iii}	3.513 (7)
O5···O7^{ii}	3.591 (5)	O12···C2^{iii}	3.539 (7)
O7···O12^{v}	3.321 (6)	O12···C8^{viii}	3.556 (7)
O7···O12^{vi}	3.466 (6)	C2···C10^{iii}	3.572 (7)
O7···O11^{v}	3.555 (6)	C3···C9^{iii}	3.482 (8)
O11···C6^{iv}	3.091 (7)	C4···C8^{iii}	3.579 (8)
C4—O5—C6	104.9 (4)	O5—C6—H62	113 (4)
C6—O7—C8	105.6 (4)	O7—C6—H62	105 (4)
C3—O11—H1	107 (5)	H61—C6—H62	105 (5)
C1—O13—C14	115.3 (4)	O7—C8—C4	110.4 (4)
O13—C1—O12	121.5 (4)	O7—C8—C9	126.9 (5)
O13—C1—C2	115.1 (4)	C4—C8—C9	122.6 (5)
O12—C1—C2	123.4 (4)	C8—C9—C10	116.4 (5)
C1—C2—C3	115.5 (4)	C8—C9—H91	124 (4)
C1—C2—C10	126.3 (4)	C10—C9—H91	120 (4)
C3—C2—C10	118.2 (4)	C2—C10—I15	125.9 (4)
C2—C3—O11	124.8 (4)	C2—C10—C9	122.5 (4)
C2—C3—C4	117.8 (4)	I15—C10—C9	111.6 (4)
O11—C3—C4	117.4 (4)	O13—C14—H141	102 (6)
O5—C4—C3	126.6 (5)	O13—C14—H142	109 (5)
O5—C4—C8	110.8 (5)	H141—C14—H142	112 (7)
C3—C4—C8	122.5 (5)	O13—C14—H143	113 (5)
O5—C6—O7	107.9 (4)	H141—C14—H143	111 (7)
O5—C6—H61	107 (4)	H142—C14—H143	110 (7)
O7—C6—H61	119 (4)		
I15—C10—C2—C1	−0.1 (7)	O12—C1—C2—C10	172.7 (5)
I15—C10—C2—C3	177.8 (4)	O13—C1—C2—C3	175.2 (4)
I15—C10—C9—C8	−178.6 (4)	O13—C1—C2—C10	−6.8 (7)

O5—C4—C3—O11	1.5 (8)	C1—C2—C3—C4	178.1 (5)
O5—C4—C3—C2	−178.7 (5)	C1—C2—C10—C9	−177.9 (5)
O5—C4—C8—O7	0.2 (6)	C2—C1—O13—C14	172.3 (4)
O5—C4—C8—C9	178.3 (5)	C2—C3—C4—C8	0.5 (8)
O5—C6—O7—C8	5.3 (6)	C2—C10—C9—C8	−0.5 (8)
O7—C6—O5—C4	−5.2 (6)	C3—C2—C10—C9	0.1 (7)
O7—C8—C4—C3	−179.1 (5)	C3—C4—O5—C6	−177.6 (6)
O7—C8—C9—C10	178.8 (5)	C3—C4—C8—C9	−1.0 (9)
O11—C3—C2—C1	−2.0 (8)	C4—C3—C2—C10	−0.0 (7)
O11—C3—C2—C10	179.8 (5)	C4—C8—O7—C6	−3.4 (6)
O11—C3—C4—C8	−179.4 (5)	C4—C8—C9—C10	1.0 (8)
O12—C1—O13—C14	−7.2 (7)	C6—O5—C4—C8	3.2 (6)
O12—C1—C2—C3	−5.3 (8)	C6—O7—C8—C9	178.5 (6)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O11—H1···O12	0.87 (7)	1.79 (7)	2.545 (6)	144 (7)

Crystal structure of $C_{17}H_{20}NO_4^+C_2HO_4^-\cdot 0.09H_2O$ — ban0821

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Abstract

The crystal structure of $C_{17}H_{20}NO_4^+C_2HO_4^-\cdot 0.09H_2O$ is reported.

Comment

The crystallographic asymmetric unit consists of two $C_{17}H_{20}NO_4^+$ cations, two $C_2HO_4^-$ anions, and one site of water of crystallization which is partially occupied.

Experimental

The compound was prepared by *MM* and recrystallized from tetrahydrofuran/water. The sample ID is *MM9*–846-salt.

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.

One inner reflection appeared to be unreliably measured and was omitted from the dataset.

All H atoms were located in a difference electron density map, but those attached to carbon atoms were included at geometrically determined positions. 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, N—H in the range 0.86–0.89 and O—H = 0.82 Å) and U_{iso}(H) (in the range 1.2–1.5 times U_{eq} of the parent atom). Later, the positions of H atoms attached to C were refined with riding constraints while the coordinates of the other H atoms were refined without constraints.

A difference map calculated after inclusion of the anions and cations, revealed a peak of significant electron density remote from other atoms. This has been interpreted to be a water molecule with an occupancy considerably less than one. The occupancy of this atom site refined to a final value of 0.173 (8). The hydrogen atoms of this molecule were not included in the model.

The largest peaks in the final difference electron density map are located along C—C bonds or adjacent to one of the oxalates.

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).

(ban0821)

Crystal data

$C_{17}H_{20}NO_4^+ \cdot C_2HO_4^- \cdot 0.09(H_2O)$	$V = 1757.71\ (5)$ Å^3
$M_r = 392.95$	$Z = 4$
Monoclinic, $P2_1$	Mo $K\alpha$
$a = 8.54860\ (10)$ Å	$\mu = 0.12$ mm^{-1}
$b = 16.0954\ (3)$ Å	$T = 200$ K
$c = 12.7901\ (2)$ Å	0.46 × 0.45 × 0.11 mm
$\beta = 92.8111\ (12)^o$	

Data collection

Area diffractometer	4160 independent reflections
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	3826 reflections with $I > 2.0\sigma(I)$
$T_{min} = 0.954$, $T_{max} = 0.987$	$R_{int} = 0.039$
37573 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.027$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.068$	$\Delta\rho_{max} = 0.19$ e Å^{-3}
$S = 0.94$	$\Delta\rho_{min} = -0.16$ e Å^{-3}
4159 reflections	Absolute structure: The enantiomer has been assigned by reference to an unchanging chiral centre in the synthetic procedure.
533 parameters	
1 restraint	

Table 1
Selected geometric parameters (Å, °)

O11—C10	1.378 (2)	C3—C4	1.537 (2)

O11—C12	1.443 (2)	C4—C5	1.517 (2)
O13—C12	1.430 (3)	C5—C19	1.508 (2)
O13—C14	1.378 (2)	C7—C8	1.514 (2)
O20—C2	1.4240 (19)	C8—C9	1.412 (3)
O21—C3	1.423 (2)	C8—C16	1.397 (2)
O21—C22	1.417 (3)	C9—C10	1.369 (3)
O111—C110	1.382 (2)	C10—C14	1.385 (3)
O111—C112	1.437 (2)	C14—C15	1.369 (3)
O113—C112	1.432 (2)	C15—C16	1.405 (2)
O113—C114	1.379 (2)	C16—C17	1.519 (3)
O120—C102	1.428 (2)	C17—C18	1.535 (2)
O121—C103	1.422 (2)	C17—C19	1.515 (2)
O121—C122	1.411 (2)	C101—C102	1.505 (2)
O201—C202	1.327 (3)	C101—C119	1.324 (3)
O203—C202	1.211 (3)	C102—C103	1.531 (2)
O205—C204	1.229 (2)	C103—C104	1.536 (2)
O206—C204	1.259 (2)	C104—C105	1.517 (2)
O301—C302	1.303 (2)	C105—C119	1.509 (2)
O303—C302	1.207 (2)	C107—C108	1.518 (2)
O305—C304	1.250 (2)	C108—C109	1.409 (3)
O306—C304	1.251 (2)	C108—C116	1.394 (2)
N6—C5	1.531 (2)	C109—C110	1.369 (3)
N6—C7	1.498 (2)	C110—C114	1.384 (3)
N6—C18	1.507 (2)	C114—C115	1.375 (3)
N106—C105	1.536 (2)	C115—C116	1.411 (2)
N106—C107	1.501 (2)	C116—C117	1.520 (2)
N106—C118	1.514 (2)	C117—C118	1.532 (2)
C1—C2	1.505 (2)	C117—C119	1.512 (2)
C1—C19	1.326 (2)	C202—C204	1.547 (3)
C2—C3	1.528 (3)	C302—C304	1.555 (3)
C10—O11—C12	105.71 (15)	C102—C101—C119	122.61 (16)
C12—O13—C14	106.05 (15)	C101—C102—O120	108.81 (15)
C3—O21—C22	113.22 (15)	C101—C102—C103	112.99 (15)
C110—O111—C112	105.14 (14)	O120—C102—C103	112.27 (14)
C112—O113—C114	105.09 (15)	C102—C103—O121	106.78 (13)
C103—O121—C122	114.01 (14)	C102—C103—C104	111.14 (13)
C5—N6—C7	112.03 (14)	O121—C103—C104	111.06 (15)
C5—N6—C18	105.61 (13)	C103—C104—C105	105.87 (13)
C7—N6—C18	108.70 (13)	N106—C105—C104	115.39 (13)
C105—N106—C107	110.39 (13)	N106—C105—C119	103.11 (12)
C105—N106—C118	105.38 (13)	C104—C105—C119	110.35 (15)
C107—N106—C118	108.32 (14)	N106—C107—C108	110.57 (14)
C2—C1—C19	122.54 (16)	C107—C108—C109	117.51 (16)
C1—C2—O20	108.66 (14)	C107—C108—C116	121.50 (16)
C1—C2—C3	112.97 (14)	C109—C108—C116	120.99 (16)
O20—C2—C3	112.22 (14)	C108—C109—C110	116.95 (17)
C2—C3—O21	106.80 (13)	O111—C110—C109	128.37 (17)
C2—C3—C4	110.67 (15)	O111—C110—C114	109.41 (15)
O21—C3—C4	112.17 (15)	C109—C110—C114	122.22 (17)

C3—C4—C5	106.98 (13)	O111—C112—O113	107.59 (14)
N6—C5—C4	114.02 (13)	C110—C114—O113	110.00 (16)
N6—C5—C19	102.77 (13)	C110—C114—C115	122.12 (16)
C4—C5—C19	110.80 (15)	O113—C114—C115	127.87 (17)
N6—C7—C8	110.46 (14)	C114—C115—C116	116.72 (17)
C7—C8—C9	117.77 (16)	C115—C116—C108	120.98 (16)
C7—C8—C16	120.96 (16)	C115—C116—C117	119.65 (16)
C9—C8—C16	121.26 (16)	C108—C116—C117	119.21 (15)
C8—C9—C10	116.59 (17)	C116—C117—C118	109.08 (15)
O11—C10—C9	127.96 (18)	C116—C117—C119	110.01 (13)
O11—C10—C14	109.94 (16)	C118—C117—C119	98.52 (14)
C9—C10—C14	122.09 (18)	C117—C118—N106	100.60 (12)
O11—C12—O13	108.16 (15)	C117—C119—C105	106.86 (14)
C10—C14—O13	110.00 (17)	C117—C119—C101	129.97 (16)
C10—C14—C15	122.35 (16)	C105—C119—C101	122.37 (16)
O13—C14—C15	127.56 (17)	O201—C202—O203	120.7 (2)
C14—C15—C16	117.03 (16)	O201—C202—C204	116.39 (17)
C15—C16—C8	120.62 (16)	O203—C202—C204	122.94 (19)
C15—C16—C17	119.48 (15)	C202—C204—O206	116.19 (17)
C8—C16—C17	119.90 (14)	C202—C204—O205	116.45 (17)
C16—C17—C18	108.77 (14)	O206—C204—O205	127.34 (19)
C16—C17—C19	111.40 (14)	O301—C302—O303	126.89 (18)
C18—C17—C19	97.59 (13)	O301—C302—C304	111.20 (15)
C17—C18—N6	99.99 (13)	O303—C302—C304	121.91 (16)
C17—C19—C5	106.41 (13)	C302—C304—O306	116.50 (16)
C17—C19—C1	129.32 (17)	C302—C304—O305	117.73 (14)
C5—C19—C1	122.80 (16)	O306—C304—O305	125.77 (18)

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supplementary materials

Crystal structure of $C_{17}H_{20}NO_4^+C_2HO_4^-\cdot0.09H_2O$ — ban0821

Maria Matveenko, Martin G. Banwell and Anthony C. Willis

(ban0821)

Crystal data

$C_{17}H_{20}NO_4^+\cdot C_2HO_4^-\cdot0.09(H_2O)$	F_{000} = 826.773
M_r = 392.95	D_x = 1.484 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.54860 (10) Å	Cell parameters from 38429 reflections
b = 16.0954 (3) Å	θ = 2.6–27.5º
c = 12.7901 (2) Å	μ = 0.12 mm^{-1}
β = 92.8111 (12)º	T = 200 K
V = 1757.71 (5) Å^3	Plate, colourless
Z = 4	0.46 × 0.45 × 0.11 mm

Data collection

Area diffractometer	3826 reflections with $I > 2.0\sigma(I)$
Monochromator: graphite	R_{int} = 0.039
T = 200 K	θ_{max} = 27.5º
φ and ω scans with CCD	θ_{min} = 2.7º
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	h = −11→9
T_{min} = 0.954, T_{max} = 0.987	k = −20→20
37573 measured reflections	l = −16→16
4160 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.027	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.0P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$wR(F^2)$ = 0.068	$(\Delta/\sigma)_{max}$ = 0.022
S = 0.94	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
4159 reflections	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
533 parameters	Extinction correction: None

1 restraint	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)
O11	−0.0506 (2)	0.15137 (10)	0.30697 (12)	0.0460	
O13	0.08360 (18)	0.26031 (10)	0.23219 (11)	0.0421	
O20	0.56881 (14)	0.48020 (10)	0.70140 (11)	0.0345	
O21	0.44105 (15)	0.34204 (9)	0.77222 (10)	0.0331	
O111	0.29546 (16)	0.09480 (9)	0.49565 (11)	0.0379	
O113	0.33672 (17)	0.19665 (10)	0.62183 (11)	0.0408	
O120	0.15108 (16)	0.59504 (10)	0.31740 (11)	0.0404	
O121	0.11717 (13)	0.47081 (9)	0.18172 (9)	0.0289	
O201	0.33634 (18)	0.08273 (11)	1.04488 (12)	0.0456	
O203	0.2203 (2)	0.20474 (11)	1.02793 (13)	0.0489	
O205	0.25121 (16)	0.05318 (9)	0.84012 (11)	0.0380	
O206	0.05680 (15)	0.14493 (9)	0.84856 (12)	0.0412	
O301	−0.08311 (16)	0.27796 (10)	0.88586 (11)	0.0376	
O303	−0.22011 (18)	0.25372 (11)	1.02806 (12)	0.0452	
O305	−0.21289 (14)	0.42368 (9)	0.85799 (10)	0.0317	
O306	−0.33923 (14)	0.41202 (9)	1.00537 (10)	0.0321	
O999	0.6846 (18)	0.1016 (9)	0.0655 (9)	0.0822	0.173 (8)
N6	−0.04988 (15)	0.38015 (10)	0.69254 (12)	0.0266	
N106	0.56248 (16)	0.35253 (10)	0.19636 (12)	0.0269	
C1	0.30508 (19)	0.48472 (12)	0.63269 (14)	0.0282	
C2	0.41040 (18)	0.48479 (12)	0.73054 (14)	0.0278	
C3	0.37013 (19)	0.41628 (13)	0.80746 (14)	0.0290	
C4	0.1918 (2)	0.40751 (14)	0.81331 (14)	0.0325	
C5	0.12898 (18)	0.37881 (12)	0.70635 (13)	0.0254	
C7	−0.1130 (2)	0.30266 (12)	0.64104 (14)	0.0284	
C8	−0.06109 (19)	0.29677 (12)	0.52981 (14)	0.0264	
C9	−0.0893 (2)	0.22151 (12)	0.47543 (14)	0.0302	
C10	−0.0352 (2)	0.21654 (12)	0.37661 (14)	0.0311	
C12	0.0303 (2)	0.17687 (14)	0.21592 (16)	0.0384	
C14	0.04464 (19)	0.28141 (13)	0.33205 (13)	0.0289	
C15	0.06974 (18)	0.35572 (12)	0.38214 (14)	0.0280	
C16	0.01486 (18)	0.36316 (12)	0.48347 (13)	0.0259	
C17	0.04047 (18)	0.44387 (12)	0.54341 (14)	0.0273	
C18	−0.08863 (19)	0.45302 (12)	0.62195 (14)	0.0293	
C19	0.17823 (18)	0.43743 (11)	0.62210 (13)	0.0255	
C22	0.4271 (3)	0.27445 (15)	0.84220 (18)	0.0461	
C101	0.38892 (19)	0.51834 (12)	0.34145 (14)	0.0273	
C102	0.2873 (2)	0.56899 (12)	0.26607 (14)	0.0291	
C103	0.24652 (18)	0.52341 (12)	0.16322 (14)	0.0271	
C104	0.38671 (18)	0.47296 (12)	0.12751 (13)	0.0274	

C105	0.41649 (18)	0.40636 (11)	0.21003 (13)	0.0242
C107	0.5259 (2)	0.26271 (12)	0.21512 (14)	0.0298
C108	0.48122 (19)	0.24937 (12)	0.32726 (14)	0.0262
C109	0.4101 (2)	0.17328 (12)	0.35142 (15)	0.0291
C110	0.3662 (2)	0.16294 (12)	0.45211 (15)	0.0290
C112	0.2517 (2)	0.12196 (13)	0.59719 (16)	0.0362
C114	0.3905 (2)	0.22368 (12)	0.52780 (14)	0.0295
C115	0.46165 (19)	0.29808 (12)	0.50689 (14)	0.0289
C116	0.50704 (18)	0.31043 (12)	0.40346 (14)	0.0264
C117	0.57389 (19)	0.39384 (12)	0.37230 (14)	0.0271
C118	0.68067 (19)	0.38103 (13)	0.28092 (14)	0.0308
C119	0.44814 (17)	0.44546 (12)	0.31617 (13)	0.0247
C122	0.0494 (2)	0.43351 (16)	0.09049 (16)	0.0409
C202	0.2452 (2)	0.13660 (13)	0.99221 (16)	0.0350
C204	0.17832 (19)	0.10810 (12)	0.88346 (15)	0.0310
C302	−0.18271 (19)	0.29666 (12)	0.95608 (14)	0.0280
C304	−0.25101 (18)	0.38530 (12)	0.93780 (13)	0.0261
H11	0.3287	0.5216	0.5789	0.0357*
H21	0.3959	0.5372	0.7671	0.0328*
H31	0.4151	0.4290	0.8786	0.0340*
H41	0.1465	0.4617	0.8312	0.0384*
H42	0.1670	0.3671	0.8665	0.0385*
H61	−0.093 (3)	0.3862 (15)	0.7530 (19)	0.0403*
H71	−0.2248	0.3047	0.6409	0.0341*
H72	−0.0753	0.2557	0.6802	0.0340*
H91	−0.1405	0.1761	0.5048	0.0346*
H121	0.1211	0.1396	0.2081	0.0467*
H122	−0.0433	0.1750	0.1512	0.0462*
H151	0.1185	0.4016	0.3504	0.0336*
H171	0.0462	0.4915	0.4959	0.0317*
H181	−0.0772	0.5047	0.6615	0.0343*
H182	−0.1956	0.4480	0.5905	0.0353*
H201	0.616 (3)	0.4677 (18)	0.760 (2)	0.0524*
H221	0.4970	0.2308	0.8186	0.0700*
H222	0.4589	0.2921	0.9137	0.0680*
H223	0.3185	0.2529	0.8401	0.0696*
H1011	0.4115	0.5413	0.4087	0.0332*
H1021	0.3475	0.6178	0.2475	0.0339*
H1031	0.2100	0.5644	0.1078	0.0325*
H1041	0.4788	0.5090	0.1218	0.0327*
H1042	0.3630	0.4484	0.0578	0.0314*
H1051	0.3297	0.3687	0.2117	0.0295*
H1061	0.605 (3)	0.3566 (16)	0.1283 (19)	0.0401*
H1071	0.6213	0.2309	0.2017	0.0352*
H1072	0.4392	0.2471	0.1656	0.0359*
H1091	0.3954	0.1312	0.3007	0.0354*
H1121	0.2873	0.0796	0.6480	0.0448*
H1122	0.1374	0.1335	0.5960	0.0441*
H1151	0.4772	0.3396	0.5598	0.0351*

H1171	0.6264	0.4219	0.4335	0.0326*
H1181	0.7273	0.4328	0.2593	0.0368*
H1182	0.7600	0.3381	0.2907	0.0362*
H1201	0.075 (3)	0.6005 (18)	0.268 (2)	0.0600*
H1221	−0.0492	0.4074	0.1092	0.0607*
H1222	0.0279	0.4758	0.0362	0.0609*
H1223	0.1206	0.3914	0.0636	0.0590*
H1224	0.1654	0.3222	0.6933	0.0297*
H2011	0.338 (3)	0.030 (2)	1.004 (2)	0.0687*
H3011	−0.029 (3)	0.223 (2)	0.895 (2)	0.0586*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O11	0.0691 (10)	0.0367 (8)	0.0333 (8)	−0.0092 (7)	0.0145 (7)	−0.0085 (7)
O13	0.0560 (8)	0.0431 (8)	0.0283 (7)	−0.0064 (7)	0.0139 (6)	−0.0062 (6)
O20	0.0229 (6)	0.0413 (8)	0.0398 (8)	−0.0012 (5)	0.0056 (5)	0.0101 (6)
O21	0.0342 (6)	0.0299 (7)	0.0351 (7)	−0.0014 (5)	0.0025 (5)	0.0047 (6)
O111	0.0511 (8)	0.0292 (7)	0.0340 (7)	−0.0052 (6)	0.0093 (6)	0.0043 (6)
O113	0.0572 (9)	0.0368 (8)	0.0292 (7)	−0.0072 (7)	0.0113 (6)	0.0023 (6)
O120	0.0387 (7)	0.0470 (9)	0.0354 (8)	0.0166 (6)	0.0010 (6)	−0.0115 (7)
O121	0.0227 (5)	0.0373 (7)	0.0268 (6)	0.0000 (5)	0.0031 (4)	−0.0032 (5)
O201	0.0539 (9)	0.0422 (9)	0.0400 (8)	−0.0025 (7)	−0.0052 (7)	0.0034 (7)
O203	0.0673 (10)	0.0383 (9)	0.0418 (9)	−0.0039 (8)	0.0091 (7)	−0.0091 (7)
O205	0.0418 (7)	0.0365 (8)	0.0359 (7)	0.0097 (6)	0.0042 (6)	−0.0054 (6)
O206	0.0336 (7)	0.0339 (8)	0.0555 (9)	0.0039 (6)	−0.0027 (6)	−0.0082 (7)
O301	0.0432 (7)	0.0329 (7)	0.0380 (7)	0.0107 (6)	0.0149 (6)	0.0029 (6)
O303	0.0461 (8)	0.0465 (9)	0.0441 (8)	0.0027 (7)	0.0130 (6)	0.0149 (7)
O305	0.0320 (6)	0.0350 (7)	0.0289 (6)	0.0068 (5)	0.0091 (5)	0.0035 (6)
O306	0.0274 (6)	0.0398 (7)	0.0299 (7)	0.0026 (5)	0.0095 (5)	−0.0044 (6)
O999	0.124 (12)	0.074 (9)	0.049 (8)	0.017 (8)	0.011 (7)	−0.004 (6)
N6	0.0218 (6)	0.0329 (8)	0.0255 (7)	−0.0002 (6)	0.0055 (5)	0.0004 (6)
N106	0.0225 (6)	0.0317 (8)	0.0270 (7)	0.0044 (6)	0.0056 (5)	0.0020 (6)
C1	0.0270 (8)	0.0270 (9)	0.0311 (9)	0.0000 (7)	0.0079 (6)	0.0013 (7)
C2	0.0239 (7)	0.0272 (9)	0.0328 (9)	−0.0012 (7)	0.0047 (6)	−0.0012 (8)
C3	0.0265 (8)	0.0349 (10)	0.0257 (8)	−0.0040 (7)	0.0020 (6)	−0.0025 (8)
C4	0.0280 (8)	0.0448 (11)	0.0253 (8)	−0.0052 (8)	0.0064 (6)	−0.0027 (8)
C5	0.0204 (7)	0.0316 (9)	0.0246 (8)	−0.0002 (6)	0.0050 (6)	0.0010 (7)
C7	0.0251 (8)	0.0326 (9)	0.0279 (9)	−0.0040 (7)	0.0053 (6)	0.0017 (7)
C8	0.0237 (7)	0.0313 (9)	0.0244 (8)	0.0007 (6)	0.0033 (6)	0.0017 (7)
C9	0.0327 (9)	0.0311 (9)	0.0271 (9)	−0.0022 (7)	0.0036 (7)	0.0039 (8)
C10	0.0327 (9)	0.0312 (10)	0.0292 (9)	0.0016 (7)	0.0018 (7)	−0.0026 (8)
C12	0.0453 (11)	0.0403 (11)	0.0300 (10)	0.0026 (9)	0.0055 (8)	−0.0026 (9)
C14	0.0253 (8)	0.0387 (10)	0.0230 (8)	0.0036 (7)	0.0032 (6)	0.0017 (8)
C15	0.0229 (7)	0.0349 (10)	0.0263 (8)	−0.0014 (7)	0.0022 (6)	0.0042 (8)
C16	0.0222 (7)	0.0299 (9)	0.0256 (8)	0.0007 (6)	0.0007 (6)	0.0021 (7)
C17	0.0274 (8)	0.0288 (9)	0.0259 (8)	−0.0005 (7)	0.0036 (6)	0.0031 (7)
C18	0.0266 (8)	0.0304 (9)	0.0312 (9)	0.0036 (7)	0.0040 (6)	0.0011 (7)

C19	0.0250 (7)	0.0267 (9)	0.0252 (8)	0.0007 (6)	0.0058 (6)	−0.0010 (7)
C22	0.0525 (12)	0.0380 (12)	0.0466 (12)	−0.0100 (10)	−0.0096 (10)	0.0123 (10)
C101	0.0263 (8)	0.0291 (9)	0.0264 (8)	−0.0032 (7)	0.0005 (6)	−0.0020 (7)
C102	0.0289 (8)	0.0263 (9)	0.0323 (9)	0.0019 (7)	0.0027 (7)	−0.0011 (7)
C103	0.0241 (7)	0.0312 (9)	0.0265 (8)	0.0031 (7)	0.0051 (6)	0.0036 (7)
C104	0.0235 (7)	0.0347 (10)	0.0246 (8)	0.0037 (7)	0.0069 (6)	0.0032 (7)
C105	0.0190 (7)	0.0291 (9)	0.0247 (8)	0.0027 (6)	0.0045 (6)	−0.0007 (7)
C107	0.0317 (8)	0.0287 (9)	0.0296 (9)	0.0035 (7)	0.0078 (7)	0.0002 (8)
C108	0.0243 (7)	0.0279 (9)	0.0266 (9)	0.0046 (6)	0.0029 (6)	0.0008 (7)
C109	0.0316 (8)	0.0270 (9)	0.0286 (9)	0.0030 (7)	0.0017 (7)	−0.0013 (7)
C110	0.0304 (8)	0.0244 (9)	0.0324 (9)	0.0005 (7)	0.0026 (7)	0.0051 (7)
C112	0.0407 (10)	0.0348 (10)	0.0338 (10)	−0.0003 (8)	0.0088 (8)	0.0048 (8)
C114	0.0328 (8)	0.0328 (10)	0.0233 (8)	0.0039 (7)	0.0040 (7)	0.0038 (7)
C115	0.0303 (8)	0.0298 (9)	0.0263 (9)	0.0016 (7)	−0.0004 (7)	0.0003 (7)
C116	0.0236 (7)	0.0284 (9)	0.0271 (9)	0.0027 (7)	−0.0001 (6)	0.0019 (7)
C117	0.0234 (7)	0.0291 (9)	0.0287 (9)	−0.0006 (6)	−0.0019 (6)	0.0018 (7)
C118	0.0206 (7)	0.0354 (10)	0.0362 (10)	0.0008 (7)	0.0001 (6)	0.0052 (8)
C119	0.0207 (7)	0.0289 (9)	0.0244 (8)	−0.0032 (6)	0.0011 (6)	0.0021 (7)
C122	0.0322 (9)	0.0538 (13)	0.0360 (10)	−0.0015 (9)	−0.0035 (8)	−0.0086 (10)
C202	0.0372 (9)	0.0353 (11)	0.0331 (10)	−0.0070 (8)	0.0087 (8)	0.0002 (8)
C204	0.0282 (8)	0.0283 (9)	0.0370 (10)	−0.0036 (7)	0.0070 (7)	0.0007 (8)
C302	0.0252 (8)	0.0324 (10)	0.0264 (9)	−0.0017 (7)	0.0026 (6)	−0.0003 (7)
C304	0.0193 (7)	0.0331 (9)	0.0261 (8)	−0.0017 (6)	0.0029 (6)	−0.0033 (7)

Geometric parameters (Å, °)

O11—C10	1.378 (2)	C9—H91	0.940
O11—C12	1.443 (2)	C10—C14	1.385 (3)
O13—C12	1.430 (3)	C12—H121	0.990
O13—C14	1.378 (2)	C12—H122	1.016
O20—C2	1.4240 (19)	C14—C15	1.369 (3)
O20—H201	0.86 (3)	C15—C16	1.405 (2)
O21—C3	1.423 (2)	C15—H151	0.949
O21—C22	1.417 (3)	C16—C17	1.519 (3)
O111—C110	1.382 (2)	C17—C18	1.535 (2)
O111—C112	1.437 (2)	C17—C19	1.515 (2)
O113—C112	1.432 (2)	C17—H171	0.982
O113—C114	1.379 (2)	C18—H181	0.975
O120—C102	1.428 (2)	C18—H182	0.984
O120—H1201	0.89 (3)	C22—H221	0.979
O121—C103	1.422 (2)	C22—H222	0.983
O121—C122	1.411 (2)	C22—H223	0.991
O201—C202	1.327 (3)	C101—C102	1.505 (2)
O201—H2011	1.00 (4)	C101—C119	1.324 (3)
O203—C202	1.211 (3)	C101—H1011	0.947
O205—C204	1.229 (2)	C102—C103	1.531 (2)
O206—C204	1.259 (2)	C102—H1021	0.975
O301—C302	1.303 (2)	C103—C104	1.536 (2)
O301—H3011	1.01 (3)	C103—H1031	1.006

O303—C302	1.207 (2)	C104—C105	1.517 (2)
O305—C304	1.250 (2)	C104—H1041	0.983
O306—C304	1.251 (2)	C104—H1042	0.987
N6—C5	1.531 (2)	C105—C119	1.509 (2)
N6—C7	1.498 (2)	C105—H1051	0.959
N6—C18	1.507 (2)	C107—C108	1.518 (2)
N6—H61	0.88 (2)	C107—H1071	0.985
N106—C105	1.536 (2)	C107—H1072	0.984
N106—C107	1.501 (2)	C108—C109	1.409 (3)
N106—C118	1.514 (2)	C108—C116	1.394 (2)
N106—H1061	0.96 (2)	C109—C110	1.369 (3)
C1—C2	1.505 (2)	C109—H1091	0.942
C1—C19	1.326 (2)	C110—C114	1.384 (3)
C1—H11	0.938	C112—H1121	0.980
C2—C3	1.528 (3)	C112—H1122	0.993
C2—H21	0.975	C114—C115	1.375 (3)
C3—C4	1.537 (2)	C115—C116	1.411 (2)
C3—H31	0.991	C115—H1151	0.956
C4—C5	1.517 (2)	C116—C117	1.520 (2)
C4—H41	0.985	C117—C118	1.532 (2)
C4—H42	0.972	C117—C119	1.512 (2)
C5—C19	1.508 (2)	C117—H1171	0.992
C5—H1224	0.979	C118—H1181	0.970
C7—C8	1.514 (2)	C118—H1182	0.971
C7—H71	0.957	C122—H1221	0.982
C7—H72	0.954	C122—H1222	0.984
C8—C9	1.412 (3)	C122—H1223	0.984
C8—C16	1.397 (2)	C202—C204	1.547 (3)
C9—C10	1.369 (3)	C302—C304	1.555 (3)
O11···C18^{i}	3.511 (2)	O206···C102^{i}	3.446 (2)
O11···C1^{i}	3.562 (2)	O206···C302	3.510 (2)
O13···O203^{ii}	3.048 (2)	O301···N6	2.995 (2)
O13···C122	3.330 (3)	O301···C7	3.154 (2)
O13···C109	3.415 (2)	O301···C4	3.308 (2)
O13···O121	3.464 (2)	O301···C5	3.406 (2)
O13···C108	3.558 (2)	O301···C204	3.532 (2)
O13···O303^{ii}	3.592 (2)	O303···O999^{ix}	2.63 (1)
O20···O305^{iii}	2.820 (2)	O303···C107^{ix}	3.312 (2)
O20···C18^{iii}	3.177 (2)	O303···N106^{ix}	3.318 (2)
O20···C109^{iv}	3.187 (2)	O303···C12^{viii}	3.374 (2)
O20···O111^{iv}	3.375 (2)	O305···N6	2.683 (2)
O20···C110^{iv}	3.595 (2)	O305···C18	3.283 (2)
O21···O113	3.131 (2)	O305···C7	3.529 (2)
O21···C304^{iii}	3.369 (2)	O305···C4	3.543 (2)
O21···O305^{iii}	3.371 (2)	O305···C3^{x}	3.593 (2)
O21···C115	3.480 (2)	O306···N106^{ix}	2.791 (2)

O111···C18^{i}	3.215 (2)	O306···C104^{ix}	3.041 (2)
O111···C101^{v}	3.547 (2)	O306···C105^{ix}	3.429 (2)
O113···C22	3.146 (3)	O306···C3^{x}	3.460 (2)
O120···O206^{vi}	2.818 (2)	O306···C122^{viii}	3.462 (2)
O120···C7^{vi}	3.402 (2)	O306···C118^{ix}	3.555 (2)
O120···C9^{vi}	3.403 (2)	O306···C22^{x}	3.583 (3)
O121···O206^{vi}	3.187 (2)	O999···C104^{xi}	3.26 (1)
O121···C15	3.204 (2)	O999···C103^{xi}	3.26 (1)
O121···O205^{vi}	3.416 (2)	O999···C2^{v}	3.35 (1)
O121···C204^{vi}	3.428 (2)	O999···C3^{v}	3.44 (1)
O201···O306^{vii}	2.822 (2)	O999···C107	3.53 (1)
O201···O999^{viii}	2.99 (2)	C2···C109^{iv}	3.580 (3)
O201···O305^{vii}	3.056 (2)	C12···C202^{ii}	3.535 (3)
O201···C304^{vii}	3.270 (3)	C14···C118^{x}	3.533 (2)
O203···C12^{viii}	3.001 (3)	C14···C109	3.574 (2)
O203···C22	3.231 (3)	C15···C118^{x}	3.533 (2)
O203···O301	3.310 (2)	C22···C202	3.364 (3)
O203···C107^{viii}	3.580 (2)	C22···C304^{iii}	3.453 (3)
O205···O306^{vii}	3.080 (2)	C22···C204	3.476 (3)
O205···C118^{v}	3.241 (2)	C22···C302^{iii}	3.592 (3)
O205···C112	3.299 (2)	C101···C112^{iv}	3.550 (2)
O205···C104^{v}	3.361 (2)	C122···C304^{ii}	3.243 (2)
O205···C122^{i}	3.365 (2)	C122···C302^{ii}	3.378 (3)
O206···O301	2.510 (2)	C122···C204^{vi}	3.444 (3)
O206···C103^{i}	3.246 (2)		
C10—O11—C12	105.71 (15)	O21—C22—H222	109.7
C12—O13—C14	106.05 (15)	H221—C22—H222	110.4
C2—O20—H201	101.6 (17)	O21—C22—H223	111.1
C3—O21—C22	113.22 (15)	H221—C22—H223	109.0
C110—O111—C112	105.14 (14)	H222—C22—H223	110.0
C112—O113—C114	105.09 (15)	C102—C101—C119	122.61 (16)
C102—O120—H1201	106.5 (17)	C102—C101—H1011	117.1
C103—O121—C122	114.01 (14)	C119—C101—H1011	120.2
C202—O201—H2011	108.2 (18)	C101—C102—O120	108.81 (15)
C302—O301—H3011	115.9 (15)	C101—C102—C103	112.99 (15)
C5—N6—C7	112.03 (14)	O120—C102—C103	112.27 (14)
C5—N6—C18	105.61 (13)	C101—C102—H1021	107.3
C7—N6—C18	108.70 (13)	O120—C102—H1021	109.1
C5—N6—H61	111.3 (15)	C103—C102—H1021	106.1
C7—N6—H61	108.6 (16)	C102—C103—O121	106.78 (13)
C18—N6—H61	110.5 (16)	C102—C103—C104	111.14 (13)
C105—N106—C107	110.39 (13)	O121—C103—C104	111.06 (15)
C105—N106—C118	105.38 (13)	C102—C103—H1031	109.9
C107—N106—C118	108.32 (14)	O121—C103—H1031	106.9

C105—N106—H1061	114.3 (14)	C104—C103—H1031	110.9
C107—N106—H1061	107.5 (15)	C103—C104—C105	105.87 (13)
C118—N106—H1061	110.8 (13)	C103—C104—H1041	110.5
C2—C1—C19	122.54 (16)	C105—C104—H1041	111.2
C2—C1—H11	117.9	C103—C104—H1042	110.6
C19—C1—H11	119.5	C105—C104—H1042	111.4
C1—C2—O20	108.66 (14)	H1041—C104—H1042	107.4
C1—C2—C3	112.97 (14)	N106—C105—C104	115.39 (13)
O20—C2—C3	112.22 (14)	N106—C105—C119	103.11 (12)
C1—C2—H21	108.3	C104—C105—C119	110.35 (15)
O20—C2—H21	108.4	N106—C105—H1051	106.3
C3—C2—H21	106.1	C104—C105—H1051	111.0
C2—C3—O21	106.80 (13)	C119—C105—H1051	110.4
C2—C3—C4	110.67 (15)	N106—C107—C108	110.57 (14)
O21—C3—C4	112.17 (15)	N106—C107—H1071	107.0
C2—C3—H31	110.6	C108—C107—H1071	109.9
O21—C3—H31	108.2	N106—C107—H1072	107.4
C4—C3—H31	108.4	C108—C107—H1072	110.8
C3—C4—C5	106.98 (13)	H1071—C107—H1072	111.1
C3—C4—H41	109.4	C107—C108—C109	117.51 (16)
C5—C4—H41	110.6	C107—C108—C116	121.50 (16)
C3—C4—H42	110.2	C109—C108—C116	120.99 (16)
C5—C4—H42	110.4	C108—C109—C110	116.95 (17)
H41—C4—H42	109.2	C108—C109—H1091	121.2
N6—C5—C4	114.02 (13)	C110—C109—H1091	121.8
N6—C5—C19	102.77 (13)	O111—C110—C109	128.37 (17)
C4—C5—C19	110.80 (15)	O111—C110—C114	109.41 (15)
N6—C5—H1224	108.5	C109—C110—C114	122.22 (17)
C4—C5—H1224	109.6	O111—C112—O113	107.59 (14)
C19—C5—H1224	111.0	O111—C112—H1121	107.6
N6—C7—C8	110.46 (14)	O113—C112—H1121	107.5
N6—C7—H71	108.1	O111—C112—H1122	110.0
C8—C7—H71	109.8	O113—C112—H1122	109.6
N6—C7—H72	108.9	H1121—C112—H1122	114.4
C8—C7—H72	109.6	C110—C114—O113	110.00 (16)
H71—C7—H72	109.9	C110—C114—C115	122.12 (16)
C7—C8—C9	117.77 (16)	O113—C114—C115	127.87 (17)
C7—C8—C16	120.96 (16)	C114—C115—C116	116.72 (17)
C9—C8—C16	121.26 (16)	C114—C115—H1151	121.3
C8—C9—C10	116.59 (17)	C116—C115—H1151	122.0
C8—C9—H91	122.9	C115—C116—C108	120.98 (16)
C10—C9—H91	120.5	C115—C116—C117	119.65 (16)
O11—C10—C9	127.96 (18)	C108—C116—C117	119.21 (15)
O11—C10—C14	109.94 (16)	C116—C117—C118	109.08 (15)
C9—C10—C14	122.09 (18)	C116—C117—C119	110.01 (13)
O11—C12—O13	108.16 (15)	C118—C117—C119	98.52 (14)
O11—C12—H121	108.6	C116—C117—H1171	110.9
O13—C12—H121	109.8	C118—C117—H1171	113.6
O11—C12—H122	110.3	C119—C117—H1171	114.1

O13—C12—H122	109.1	C117—C118—N106	100.60 (12)
H121—C12—H122	110.9	C117—C118—H1181	111.8
C10—C14—O13	110.00 (17)	N106—C118—H1181	109.0
C10—C14—C15	122.35 (16)	C117—C118—H1182	115.8
O13—C14—C15	127.56 (17)	N106—C118—H1182	108.3
C14—C15—C16	117.03 (16)	H1181—C118—H1182	110.7
C14—C15—H151	122.8	C117—C119—C105	106.86 (14)
C16—C15—H151	120.1	C117—C119—C101	129.97 (16)
C15—C16—C8	120.62 (16)	C105—C119—C101	122.37 (16)
C15—C16—C17	119.48 (15)	O121—C122—H1221	107.6
C8—C16—C17	119.90 (14)	O121—C122—H1222	110.2
C16—C17—C18	108.77 (14)	H1221—C122—H1222	109.6
C16—C17—C19	111.40 (14)	O121—C122—H1223	110.2
C18—C17—C19	97.59 (13)	H1221—C122—H1223	110.1
C16—C17—H171	111.5	H1222—C122—H1223	109.1
C18—C17—H171	112.9	O201—C202—O203	120.7 (2)
C19—C17—H171	113.8	O201—C202—C204	116.39 (17)
C17—C18—N6	99.99 (13)	O203—C202—C204	122.94 (19)
C17—C18—H181	111.3	C202—C204—O206	116.19 (17)
N6—C18—H181	109.9	C202—C204—O205	116.45 (17)
C17—C18—H182	114.1	O206—C204—O205	127.34 (19)
N6—C18—H182	110.7	O301—C302—O303	126.89 (18)
H181—C18—H182	110.5	O301—C302—C304	111.20 (15)
C17—C19—C5	106.41 (13)	O303—C302—C304	121.91 (16)
C17—C19—C1	129.32 (17)	C302—C304—O306	116.50 (16)
C5—C19—C1	122.80 (16)	C302—C304—O305	117.73 (14)
O21—C22—H221	106.5	O306—C304—O305	125.77 (18)
O11—C10—C9—C8	−178.4 (2)	C7—N6—C5—C19	104.8 (2)
O11—C10—C14—O13	−0.0 (2)	C7—N6—C18—C17	−79.6 (1)
O11—C10—C14—C15	176.7 (2)	C7—C8—C9—C10	−177.3 (2)
O11—C12—O13—C14	3.7 (2)	C7—C8—C16—C15	176.8 (2)
O13—C12—O11—C10	−3.7 (2)	C7—C8—C16—C17	−2.5 (2)
O13—C14—C10—C9	−179.2 (2)	C8—C7—N6—C18	50.9 (2)
O13—C14—C15—C16	178.1 (2)	C8—C9—C10—C14	0.6 (3)
O20—C2—C1—C19	134.0 (2)	C8—C16—C15—C14	0.3 (2)
O20—C2—C3—O21	−41.8 (2)	C8—C16—C17—C18	−27.4 (2)
O20—C2—C3—C4	−164.1 (2)	C8—C16—C17—C19	79.0 (2)
O21—C3—C2—C1	81.5 (2)	C9—C8—C16—C15	−2.0 (2)
O21—C3—C4—C5	−54.5 (2)	C9—C8—C16—C17	178.8 (2)
O111—C110—C109—C108	−179.7 (2)	C9—C10—O11—C12	−178.5 (2)
O111—C110—C114—O113	0.1 (2)	C9—C10—C14—C15	−2.4 (3)
O111—C110—C114—C115	178.9 (2)	C10—C9—C8—C16	1.5 (2)
O111—C112—O113—C114	16.2 (2)	C10—C14—O13—C12	−2.3 (2)
O113—C112—O111—C110	−16.2 (2)	C10—C14—C15—C16	1.9 (2)
O113—C114—C110—C109	−179.4 (2)	C12—O11—C10—C14	2.4 (2)
O113—C114—C115—C116	179.6 (2)	C12—O13—C14—C15	−178.9 (2)
O120—C102—C101—C119	131.6 (2)	C14—C15—C16—C17	179.5 (1)
O120—C102—C103—O121	−40.9 (2)	C15—C16—C17—C18	153.3 (1)
O120—C102—C103—C104	−162.2 (2)	C15—C16—C17—C19	−100.2 (2)

O121—C103—C102—C101	82.7 (2)	C18—N6—C5—C19	−13.4 (2)
O121—C103—C104—C105	−53.8 (2)	C101—C102—C103—C104	−38.6 (2)
O201—C202—C204—O205	−23.2 (3)	C101—C119—C105—C104	28.5 (2)
O201—C202—C204—O206	158.0 (2)	C101—C119—C117—C116	118.7 (2)
O203—C202—C204—O205	155.1 (2)	C101—C119—C117—C118	−127.3 (2)
O203—C202—C204—O206	−23.6 (3)	C102—C101—C119—C105	−1.0 (3)
O301—C302—C304—O305	4.7 (2)	C102—C101—C119—C117	167.4 (2)
O301—C302—C304—O306	−174.9 (1)	C102—C103—O121—C122	170.9 (2)
O303—C302—C304—O305	−176.0 (2)	C102—C103—C104—C105	64.9 (2)
O303—C302—C304—O306	4.4 (2)	C103—C102—C101—C119	6.1 (2)
N6—C5—C4—C3	−170.7 (2)	C103—C104—C105—C119	−58.2 (2)
N6—C5—C19—C1	147.4 (2)	C104—C103—O121—C122	−67.8 (2)
N6—C5—C19—C17	−19.9 (2)	C104—C105—N106—C107	−136.4 (1)
N6—C7—C8—C9	170.2 (1)	C104—C105—N106—C118	106.8 (2)
N6—C7—C8—C16	−8.6 (2)	C104—C105—C119—C117	−142.2 (1)
N6—C18—C17—C16	64.8 (2)	C105—N106—C107—C108	−63.3 (2)
N6—C18—C17—C19	−50.9 (2)	C105—N106—C118—C117	39.7 (2)
N106—C105—C104—C103	−174.6 (1)	C105—C119—C117—C116	−71.5 (2)
N106—C105—C119—C101	152.3 (2)	C105—C119—C117—C118	42.4 (2)
N106—C105—C119—C117	−18.4 (2)	C107—N106—C105—C119	103.2 (2)
N106—C107—C108—C109	166.1 (1)	C107—N106—C118—C117	−78.4 (2)
N106—C107—C108—C116	−12.9 (2)	C107—C108—C109—C110	−178.2 (2)
N106—C118—C117—C116	65.4 (2)	C107—C108—C116—C115	178.5 (2)
N106—C118—C117—C119	−49.3 (2)	C107—C108—C116—C117	3.1 (2)
C1—C2—C3—C4	−40.8 (2)	C108—C107—N106—C118	51.6 (2)
C1—C19—C5—C4	25.3 (2)	C108—C109—C110—C114	−0.3 (3)
C1—C19—C17—C16	124.5 (2)	C108—C116—C115—C114	−0.4 (2)
C1—C19—C17—C18	−121.9 (2)	C108—C116—C117—C118	−30.9 (2)
C2—C1—C19—C5	−0.7 (3)	C108—C116—C117—C119	76.2 (2)
C2—C1—C19—C17	163.5 (2)	C109—C108—C116—C115	−0.5 (3)
C2—C3—O21—C22	173.9 (2)	C109—C108—C116—C117	−175.8 (2)
C2—C3—C4—C5	64.7 (2)	C109—C110—O111—C112	−170.5 (2)
C3—C2—C1—C19	8.8 (2)	C109—C110—C114—C115	−0.6 (3)
C3—C4—C5—C19	−55.3 (2)	C110—C109—C108—C116	0.9 (2)
C4—C3—O21—C22	−64.7 (2)	C110—C114—O113—C112	−10.2 (2)
C4—C5—N6—C7	−135.2 (2)	C110—C114—C115—C116	1.0 (3)
C4—C5—N6—C18	106.6 (2)	C112—O111—C110—C114	10.0 (2)
C4—C5—C19—C17	−142.1 (1)	C112—O113—C114—C115	171.1 (2)
C5—N6—C7—C8	−65.5 (2)	C114—C115—C116—C117	174.9 (1)
C5—N6—C18—C17	40.8 (2)	C115—C116—C117—C118	153.7 (1)
C5—C19—C17—C16	−69.3 (2)	C115—C116—C117—C119	−99.3 (2)
C5—C19—C17—C18	44.4 (2)	C118—N106—C105—C119	−13.5 (2)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H61···O305	0.88 (2)	1.83 (2)	2.683 (3)	163 (2)

O20—H201···O305^{iii}	0.86 (3)	2.01 (3)	2.820 (3)	158 (2)
N106—H1061···O306^{xii}	0.96 (2)	1.89 (2)	2.791 (3)	155 (2)
O120—H1201···O206^{vi}	0.89 (3)	1.95 (3)	2.818 (3)	162 (3)
O201—H2011···O306^{vii}	1.00 (3)	1.90 (3)	2.822 (3)	152 (2)
O301—H3011···O206	1.01 (3)	1.58 (3)	2.510 (3)	151 (2)

Symmetry codes: (iii) $x+1, y, z$; (xii) $x+1, y, z-1$; (vi) $-x, y+1/2, -z+1$; (vii) $-x, y-1/2, -z+2$.

Crystal structure of $C_{17}H_{19}NO_4$ — ban0813

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Abstract

The crystal structure of $C_{17}H_{19}NO_4$ is reported.

Comment

The crystallographic asymmetric unit consists of one molecule of $C_{17}H_{19}NO_4$.

Experimental

The compound was prepared by *MM* and recrystallized from tetrahydrofuran. The sample ID is *MM9*–828.

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.

The H atoms were all located in a difference map, 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 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, with the exception of the alcohol H which was refined without constraints.

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).

(ban0813)

Crystal data

$C_{17}H_{19}NO_4$	$V = 1464.22\ (16)$ Å^3
$M_r = 301.34$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$
$a = 10.5581\ (6)$ Å	$\mu = 0.10$ mm^{-1}
$b = 10.9515\ (8)$ Å	$T = 200$ K
$c = 12.6633\ (8)$ Å	$0.30 \times 0.10 \times 0.04$ mm

Data collection

Nonius KappaCCD diffractometer	1493 independent reflections
Absorption correction: multi-scan multi-scan from symmetry-related measurements Sortav (Blessing 1995, 1997)	1139 reflections with $I > 2.0\sigma(I)$
$T_{min} = 0.821$, $T_{max} = 0.996$	$R_{int} = 0.073$
10548 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$	$\Delta\rho_{max} = 0.24$ e Å^{-3}
$wR(F^2) = 0.067$	$\Delta\rho_{min} = -0.21$ e Å^{-3}
$S = 0.82$	Absolute structure: The enantiomer has been assigned by reference to an unchanging chiral centre in the synthetic procedure.
1485 reflections	
202 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °)

C1—C2	1.504 (3)	C8—C16	1.397 (3)
C1—C19	1.324 (3)	C9—C10	1.354 (4)
C2—C3	1.541 (3)	C10—O11	1.389 (3)
C2—O20	1.441 (3)	C10—C14	1.383 (4)
C3—C4	1.516 (3)	O11—C12	1.421 (4)
C3—O21	1.438 (3)	C12—O13	1.438 (3)
C4—C5	1.520 (3)	O13—C14	1.385 (3)
C5—N6	1.493 (3)	C14—C15	1.371 (3)
C5—C19	1.511 (3)	C15—C16	1.398 (3)
N6—C7	1.481 (3)	C16—C17	1.516 (3)
N6—C18	1.494 (3)	C17—C18	1.534 (3)
C7—C8	1.518 (4)	C17—C19	1.510 (3)
C8—C9	1.411 (4)	O21—C22	1.432 (3)
C2—C1—C19	122.2 (2)	C9—C10—C14	122.2 (3)

C1—C2—C3	114.3 (2)	O11—C10—C14	109.3 (2)
C1—C2—O20	105.1 (2)	C10—O11—C12	105.7 (2)
C3—C2—O20	110.1 (2)	O11—C12—O13	108.7 (2)
C2—C3—C4	113.3 (2)	C12—O13—C14	104.8 (2)
C2—C3—O21	111.3 (2)	O13—C14—C10	110.4 (2)
C4—C3—O21	106.1 (2)	O13—C14—C15	127.9 (2)
C3—C4—C5	108.9 (2)	C10—C14—C15	121.7 (3)
C4—C5—N6	114.6 (2)	C14—C15—C16	117.3 (2)
C4—C5—C19	108.1 (2)	C15—C16—C8	120.9 (2)
N6—C5—C19	105.95 (19)	C15—C16—C17	120.7 (2)
C5—N6—C7	111.51 (19)	C8—C16—C17	118.4 (2)
C5—N6—C18	103.36 (18)	C16—C17—C18	109.35 (19)
C7—N6—C18	107.27 (19)	C16—C17—C19	110.51 (19)
N6—C7—C8	114.6 (2)	C18—C17—C19	98.4 (2)
C7—C8—C9	119.8 (2)	C17—C18—N6	102.17 (18)
C7—C8—C16	119.9 (2)	C5—C19—C17	106.1 (2)
C9—C8—C16	120.3 (2)	C5—C19—C1	123.0 (2)
C8—C9—C10	117.5 (2)	C17—C19—C1	129.6 (2)
C9—C10—O11	128.5 (2)	C3—O21—C22	113.1 (2)

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supplementary materials

Crystal structure of $C_{17}H_{19}NO_4$ — ban0813

Maria Matveenko, Martin G. Banwell and Anthony C. Willis

(ban0813)

Crystal data

$C_{17}H_{19}NO_4$	$D_x = 1.367$ Mg m^{-3}
$M_r = 301.34$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 17341 reflections
$a = 10.5581$ (6) Å	$\theta = 2.6$–25º
$b = 10.9515$ (8) Å	$\mu = 0.10$ mm^{-1}
$c = 12.6633$ (8) Å	$T = 200$ K
$V = 1464.22$ (16) Å^3	Plate, colourless
$Z = 4$	0.30 × 0.10 × 0.04 mm
$F_{000} = 640$	

Data collection

Nonius KappaCCD diffractometer	1139 reflections with $I > 2.0\sigma(I)$
Monochromator: graphite	$R_{int} = 0.073$
$T = 200$ K	$\theta_{max} = 25.0$º
φ and ω scans with CCD	$\theta_{min} = 2.7$º
Absorption correction: multi-scan multi-scan from symmetry-related measurements Sortav (Blessing 1995, 1997)	$h = -12\rightarrow11$
$T_{min} = 0.821$, $T_{max} = 0.996$	$k = -13\rightarrow13$
10548 measured reflections	$l = -15\rightarrow15$
1493 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.033$	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.02P)^2 + 0.0P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$wR(F^2) = 0.067$	$(\Delta/\sigma)_{max} = 0.0003$
$S = 0.82$	$\Delta\rho_{max} = 0.24$ e Å^{-3}
1485 reflections	$\Delta\rho_{min} = -0.21$ e Å^{-3}
202 parameters	Extinction correction: None

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}
C1	0.7597 (2)	0.7954 (2)	0.6960 (2)	0.0302
C2	0.7074 (2)	0.9122 (2)	0.74066 (19)	0.0332
C3	0.6598 (2)	1.0035 (3)	0.6569 (2)	0.0302
C4	0.7195 (2)	0.9847 (2)	0.54920 (19)	0.0290
C5	0.6953 (2)	0.8543 (2)	0.51359 (18)	0.0265
N6	0.75402 (18)	0.82162 (19)	0.41015 (15)	0.0288
C7	0.6735 (2)	0.7357 (2)	0.34980 (19)	0.0316
C8	0.6588 (2)	0.6106 (2)	0.39992 (19)	0.0288
C9	0.5712 (2)	0.5265 (3)	0.3574 (2)	0.0342
C10	0.5598 (2)	0.4170 (2)	0.4062 (2)	0.0345
O11	0.48097 (18)	0.32007 (18)	0.38025 (15)	0.0485
C12	0.4944 (3)	0.2342 (3)	0.4636 (3)	0.0570
O13	0.59876 (18)	0.27077 (18)	0.52923 (16)	0.0499
C14	0.6296 (2)	0.3871 (2)	0.4949 (2)	0.0347
C15	0.7167 (2)	0.4659 (2)	0.5373 (2)	0.0314
C16	0.7315 (2)	0.5792 (2)	0.48803 (19)	0.0269
C17	0.8234 (2)	0.6729 (2)	0.53162 (19)	0.0299
C18	0.8712 (2)	0.7541 (3)	0.44128 (19)	0.0322
C19	0.7535 (2)	0.7692 (2)	0.59400 (19)	0.0271
O20	0.81019 (18)	0.96491 (19)	0.79996 (14)	0.0404
O21	0.52532 (16)	0.99294 (18)	0.64155 (15)	0.0401
C22	0.4532 (3)	1.0598 (3)	0.7179 (2)	0.0551
H1	0.776 (3)	1.035 (3)	0.836 (2)	0.0599*
H11	0.7992	0.7396	0.7443	0.0349*
H21	0.6387	0.8921	0.7912	0.0395*
H31	0.6814	1.0888	0.6806	0.0350*
H41	0.8105	0.9974	0.5546	0.0336*
H42	0.6852	1.0430	0.4986	0.0339*
H51	0.6025	0.8418	0.5089	0.0311*
H71	0.7102	0.7264	0.2786	0.0377*
H72	0.5891	0.7748	0.3417	0.0382*
H91	0.5222	0.5462	0.2983	0.0390*
H121	0.4182	0.2329	0.5067	0.0677*
H122	0.5110	0.1514	0.4348	0.0673*
H151	0.7642	0.4447	0.5971	0.0358*
H171	0.8928	0.6337	0.5736	0.0349*
H181	0.9356	0.8137	0.4680	0.0392*
H182	0.9066	0.7065	0.3815	0.0388*
H221	0.3629	1.0492	0.7025	0.0812*
H222	0.4769	1.1474	0.7136	0.0809*
H223	0.4724	1.0286	0.7884	0.0807*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0320 (14)	0.0260 (15)	0.0325 (15)	−0.0030 (12)	−0.0022 (13)	0.0018 (12)
C2	0.0340 (15)	0.0380 (17)	0.0278 (13)	−0.0050 (14)	0.0008 (12)	−0.0027 (13)
C3	0.0284 (13)	0.0283 (15)	0.0340 (14)	−0.0031 (12)	−0.0034 (12)	−0.0047 (13)
C4	0.0267 (14)	0.0290 (15)	0.0313 (14)	0.0018 (13)	0.0000 (12)	0.0031 (12)
C5	0.0230 (13)	0.0319 (15)	0.0246 (13)	0.0003 (11)	0.0033 (12)	0.0007 (12)
N6	0.0260 (11)	0.0335 (12)	0.0269 (11)	0.0032 (10)	0.0015 (10)	−0.0024 (10)
C7	0.0325 (14)	0.0350 (16)	0.0272 (13)	0.0021 (13)	−0.0008 (12)	−0.0028 (13)
C8	0.0271 (13)	0.0322 (15)	0.0270 (14)	0.0031 (12)	0.0026 (12)	−0.0046 (13)
C9	0.0283 (14)	0.0403 (18)	0.0339 (15)	0.0047 (14)	−0.0061 (13)	−0.0072 (15)
C10	0.0286 (15)	0.0317 (16)	0.0434 (16)	−0.0008 (13)	−0.0030 (13)	−0.0091 (15)
O11	0.0444 (12)	0.0346 (11)	0.0665 (14)	−0.0068 (10)	−0.0167 (11)	−0.0056 (11)
C12	0.0487 (19)	0.0397 (19)	0.083 (2)	−0.0126 (16)	−0.0149 (19)	0.000 (2)
O13	0.0500 (12)	0.0322 (12)	0.0674 (15)	−0.0092 (10)	−0.0178 (11)	0.0040 (11)
C14	0.0349 (16)	0.0275 (15)	0.0415 (16)	0.0016 (13)	0.0009 (14)	−0.0023 (14)
C15	0.0293 (14)	0.0319 (15)	0.0331 (15)	0.0031 (13)	−0.0031 (13)	−0.0008 (13)
C16	0.0223 (12)	0.0303 (14)	0.0282 (14)	0.0022 (12)	0.0019 (12)	−0.0041 (13)
C17	0.0263 (13)	0.0312 (15)	0.0323 (14)	0.0013 (12)	−0.0036 (12)	0.0005 (13)
C18	0.0247 (13)	0.0361 (17)	0.0359 (15)	0.0025 (13)	0.0021 (12)	0.0001 (14)
C19	0.0243 (12)	0.0281 (14)	0.0290 (14)	−0.0030 (12)	−0.0012 (11)	0.0005 (13)
O20	0.0426 (12)	0.0434 (12)	0.0352 (10)	0.0047 (10)	−0.0092 (10)	−0.0123 (9)
O21	0.0263 (9)	0.0489 (13)	0.0452 (11)	0.0048 (9)	0.0000 (9)	−0.0170 (11)
C22	0.0394 (17)	0.073 (2)	0.0533 (19)	0.0105 (17)	0.0092 (15)	−0.0221 (19)

Geometric parameters (Å, °)

C1—C2	1.504 (3)	C9—H91	0.935
C1—C19	1.324 (3)	C10—O11	1.389 (3)
C1—H11	0.960	C10—C14	1.383 (4)
C2—C3	1.541 (3)	O11—C12	1.421 (4)
C2—O20	1.441 (3)	C12—O13	1.438 (3)
C2—H21	0.992	C12—H121	0.973
C3—C4	1.516 (3)	C12—H122	0.992
C3—O21	1.438 (3)	O13—C14	1.385 (3)
C3—H31	1.007	C14—C15	1.371 (3)
C4—C5	1.520 (3)	C15—C16	1.398 (3)
C4—H41	0.973	C15—H151	0.938
C4—H42	0.974	C16—C17	1.516 (3)
C5—N6	1.493 (3)	C17—C18	1.534 (3)
C5—C19	1.511 (3)	C17—C19	1.510 (3)
C5—H51	0.991	C17—H171	1.002
N6—C7	1.481 (3)	C18—H181	1.001
N6—C18	1.494 (3)	C18—H182	0.992
C7—C8	1.518 (4)	O20—H1	0.96 (3)
C7—H71	0.987	O21—C22	1.432 (3)
C7—H72	0.994	C22—H221	0.980

C8—C9	1.411 (4)	C22—H222	0.993
C8—C16	1.397 (3)	C22—H223	0.978
C9—C10	1.354 (4)		
O11···C7^{i}	3.464 (3)	O20···C5^{iii}	3.353 (3)
O13···C4^{ii}	3.392 (3)	O20···C9^{iv}	3.402 (3)
O13···C3^{ii}	3.405 (4)	O21···C18^{v}	3.327 (4)
O13···O21^{ii}	3.447 (3)	O21···C12^{vi}	3.488 (4)
O20···N6^{iii}	2.806 (3)	O21···C17^{v}	3.557 (3)
O20···C4^{iii}	3.219 (3)	O21···C16^{v}	3.597 (3)
O20···C7^{iii}	3.343 (3)	C1···C22^{vii}	3.592 (4)
C2—C1—C19	122.2 (2)	C9—C10—C14	122.2 (3)
C2—C1—H11	117.4	O11—C10—C14	109.3 (2)
C19—C1—H11	120.4	C10—O11—C12	105.7 (2)
C1—C2—C3	114.3 (2)	O11—C12—O13	108.7 (2)
C1—C2—O20	105.1 (2)	O11—C12—H121	110.1
C3—C2—O20	110.1 (2)	O13—C12—H121	108.2
C1—C2—H21	108.8	O11—C12—H122	110.5
C3—C2—H21	110.5	O13—C12—H122	109.4
O20—C2—H21	107.7	H121—C12—H122	109.9
C2—C3—C4	113.3 (2)	C12—O13—C14	104.8 (2)
C2—C3—O21	111.3 (2)	O13—C14—C10	110.4 (2)
C4—C3—O21	106.1 (2)	O13—C14—C15	127.9 (2)
C2—C3—H31	108.9	C10—C14—C15	121.7 (3)
C4—C3—H31	107.5	C14—C15—C16	117.3 (2)
O21—C3—H31	109.7	C14—C15—H151	121.3
C3—C4—C5	108.9 (2)	C16—C15—H151	121.4
C3—C4—H41	109.1	C15—C16—C8	120.9 (2)
C5—C4—H41	108.7	C15—C16—C17	120.7 (2)
C3—C4—H42	110.4	C8—C16—C17	118.4 (2)
C5—C4—H42	111.0	C16—C17—C18	109.35 (19)
H41—C4—H42	108.6	C16—C17—C19	110.51 (19)
C4—C5—N6	114.6 (2)	C18—C17—C19	98.4 (2)
C4—C5—C19	108.1 (2)	C16—C17—H171	111.8
N6—C5—C19	105.95 (19)	C18—C17—H171	113.8
C4—C5—H51	108.3	C19—C17—H171	112.3
N6—C5—H51	109.0	C17—C18—N6	102.17 (18)
C19—C5—H51	110.9	C17—C18—H181	110.4
C5—N6—C7	111.51 (19)	N6—C18—H181	109.2
C5—N6—C18	103.36 (18)	C17—C18—H182	112.8
C7—N6—C18	107.27 (19)	N6—C18—H182	111.7
N6—C7—C8	114.6 (2)	H181—C18—H182	110.2
N6—C7—H71	108.2	C5—C19—C17	106.1 (2)
C8—C7—H71	109.2	C5—C19—C1	123.0 (2)
N6—C7—H72	107.1	C17—C19—C1	129.6 (2)
C8—C7—H72	109.9	C2—O20—H1	106.2 (18)
H71—C7—H72	107.6	C3—O21—C22	113.1 (2)
C7—C8—C9	119.8 (2)	O21—C22—H221	108.8

C7—C8—C16	119.9 (2)	O21—C22—H222	108.9
C9—C8—C16	120.3 (2)	H221—C22—H222	110.4
C8—C9—C10	117.5 (2)	O21—C22—H223	109.2
C8—C9—H91	121.2	H221—C22—H223	109.9
C10—C9—H91	121.3	H222—C22—H223	109.6
C9—C10—O11	128.5 (2)		
O11—C10—C9—C8	179.8 (2)	C4—C5—N6—C7	−145.2 (2)
O11—C10—C14—O13	0.0 (3)	C4—C5—N6—C18	99.9 (2)
O11—C10—C14—C15	179.2 (2)	C4—C5—C19—C17	−134.8 (2)
O11—C12—O13—C14	10.3 (3)	C5—N6—C7—C8	−65.9 (2)
O13—C12—O11—C10	−10.4 (3)	C5—N6—C18—C17	42.5 (2)
O13—C14—C10—C9	179.4 (2)	C5—C19—C17—C16	−78.1 (2)
O13—C14—C15—C16	179.7 (2)	C5—C19—C17—C18	36.2 (2)
O20—C2—C1—C19	−127.2 (2)	C7—N6—C5—C19	95.7 (2)
O20—C2—C3—O21	−145.6 (2)	C7—N6—C18—C17	−75.4 (2)
O20—C2—C3—C4	94.9 (2)	C7—C8—C9—C10	−178.7 (2)
O21—C3—C2—C1	96.3 (2)	C7—C8—C16—C15	178.1 (2)
O21—C3—C4—C5	−65.8 (3)	C7—C8—C16—C17	0.4 (3)
N6—C5—C4—C3	−177.5 (2)	C8—C7—N6—C18	46.6 (2)
N6—C5—C19—C1	156.1 (2)	C8—C9—C10—C14	0.5 (4)
N6—C5—C19—C17	−11.5 (2)	C8—C16—C15—C14	0.8 (3)
N6—C7—C8—C9	171.6 (2)	C8—C16—C17—C18	−30.5 (3)
N6—C7—C8—C16	−8.2 (3)	C8—C16—C17—C19	76.7 (3)
N6—C18—C17—C16	67.0 (2)	C9—C8—C16—C15	−1.7 (3)
N6—C18—C17—C19	−48.3 (2)	C9—C8—C16—C17	−179.4 (2)
C1—C2—C3—C4	−23.1 (3)	C9—C10—O11—C12	−172.9 (3)
C1—C19—C5—C4	32.8 (3)	C9—C10—C14—C15	−1.5 (4)
C1—C19—C17—C16	115.4 (3)	C10—C9—C8—C16	1.0 (3)
C1—C19—C17—C18	−130.2 (2)	C10—C14—O13—C12	−6.4 (3)
C2—C1—C19—C5	0.8 (3)	C10—C14—C15—C16	0.8 (3)
C2—C1—C19—C17	165.2 (2)	C12—O11—C10—C14	6.4 (3)
C2—C3—O21—C22	83.2 (3)	C12—O13—C14—C15	174.6 (3)
C2—C3—C4—C5	56.6 (2)	C14—C15—C16—C17	178.4 (2)
C3—C2—C1—C19	−6.3 (3)	C15—C16—C17—C18	151.8 (2)
C3—C4—C5—C19	−59.6 (2)	C15—C16—C17—C19	−101.0 (2)
C4—C3—O21—C22	−153.2 (2)	C18—N6—C5—C19	−19.3 (2)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O20—H1···N6^{iii}	0.96 (3)	1.86 (3)	2.806 (4)	167 (3)

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