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4-Nitrobenzyl 5-oxo-2-(2-oxopiperidin-1-yl)-tetrahydrofuran-2-carboxylate

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Key indicators

Single-crystal X-ray study
T = 200 K
Mean σ(C–C) = 0.006 Å
Disorder in main residue
R factor = 0.135
wR factor = 0.310
Data-to-parameter ratio = 17.7

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

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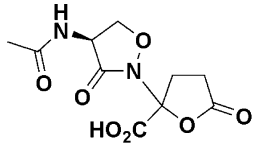
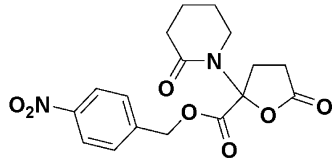
The title compound, $C_{17}H_{18}N_2O_7$, is a synthetic racemic analogue of lactivicin, a natural product antibiotic that targets penicillin-binding proteins. There are two almost identical molecules in the asymmetric unit.

Comment

Lactivicin [LTV, (1)] is a natural product antibiotic that targets penicillin-binding proteins (PBPs), a class of enzymes involved in the final steps of bacterial cell wall biosynthesis (Nozaki *et al.*, 1987, 1989). As part of a research programme aimed at identifying new antibiotics, we are interested in antibiotics that do not possess a β-lactam ring. We have prepared an LTV analogue from the title compound, (2), where a six-membered cyclic hydroxamate unit acts as a surrogate of the naturally occurring isoxazolidine-3-one core (Wolfe, Akuche *et al.*, 2003; Wolfe, Wilson *et al.*, 2003).



(1)



(2)

The structure of (2) contains two molecules in the asymmetric unit (Fig. 1). Bond lengths and angles are unremarkable, the largest differences from the *Mogul* norms (Bruno *et al.*, 2004) being for C15—C16 (0.06 Å; *Mogul* s.u. 0.04 Å) and C18—C171—N12 (5.7°; *Mogul* s.u. 1.5°).

As is common in Z' = 2 structures (Collins, 2006), one molecule of (2) is well ordered and the other has resolvable disorder. If the minor component of the disorder is selected, the two molecules have very similar geometries (Fig. 2), with the major discrepancy being in the orientation of the nitro group. If this group is also omitted, the two molecules are essentially identical (r.m.s. positional deviation = 0.10 Å, r.m.s. bond length deviation = 0.016 Å and r.m.s. torsion angle deviation = 3.07°) (Collins *et al.*, 2006) and related by a pseudo glide plane at (0.42 − x, 0.50 + y, 0.00 + z) (Fig. 3).

There are no hydrogen bonds in the crystal structure of (2), which consists of bilayers with the nitro groups dominating the exposed faces (Fig. 4).

Experimental

Compound (2) was prepared by coupling δ-valerolactam (300 mg, 3.02 mmol) and 1-(4-nitrobenzyl)-2-oxoglutarate (1.10 g, 3.93 mmol) in the presence of *N*,*N*′-dicyclohexylcarbodiimide (811 mg, 3.93 mmol) in CH_2Cl_2 (15 ml). The reaction mixture was stirred at

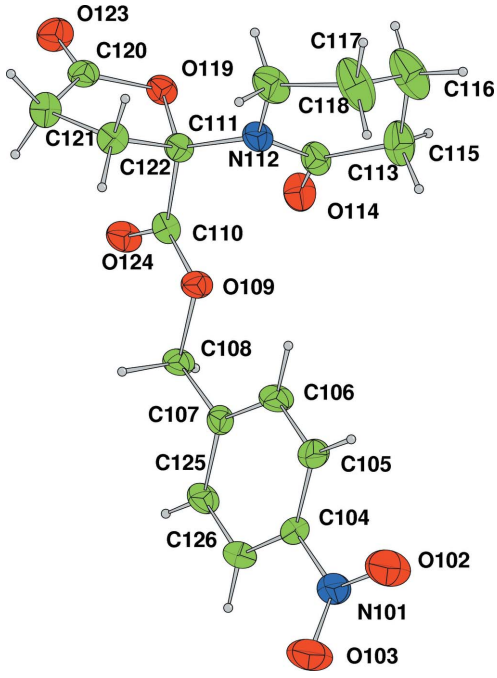


Figure 1
The structure of one molecule of the asymmetric unit of the title compound, (2), with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

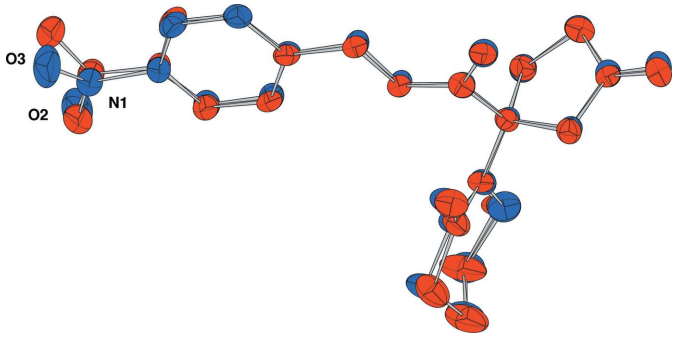


Figure 2
A least-squares fit of the minor component of the disordered molecule (blue) to the undisordered molecule. The major differences in conformation are in the nitro group. H atoms have been omitted.

room temperature under N_2 for 18 h, after which H_2O (40 ml) and CH_2Cl_2 (40 ml) were added. The aqueous layer was extracted with CH_2Cl_2 (100 ml) and the combined organic layers were washed with brine (2 × 40 ml), dried ($MgSO_4$), filtered and concentrated *in vacuo*. Purification by flash column chromatography (MeOH–CH_2Cl_2, 1:99 *v*/*v*) afforded (2) as a white solid (280 mg, 26%). Suitable crystals were obtained upon recrystallization from MeOH (m.p. 390–391 K).

^{1}H NMR (400 MHz, $CDCl_3$, δ, p.p.m.): 1.83 (4H, *m*, CH_2-4′, CH_2-5′), 2.40 (3H, *m*, CH_2-3′, CH-3 or CH-4), 2.64 (1H, *m*, CH-3 or CH-4), 2.80 (1H, *m*, CH-3 or CH-4), 3.33 (2H, *m*, CH-3 or CH-4, CH-6′), 3.49 (1H, *m*, CH-6′), 5.30 (2H, *dd*, *J* = 13.3 and 6.1 Hz, OCH_2Ar), 7.50 (2H, *d*, *J* = 8.7 Hz, 2 × *o*-ArH), 8.23 (2H, *d*, *J* = 8.7 Hz, 2 × *m*-ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ, p.p.m.): 20.3 (CH_2), 23.0 (CH_2), 27.7 (CH_2), 31.0 (CH_2), 32.7 (CH_2), 44.2 (CH_2N), 66.4 (CH_2Ar), 93.5 (C2), 123.9 (2 × ArCH), 128.3 (2 × ArCH), 142.3 (ArC), 147.8 (ArC), 166.7 (C═O), 171.6 (C═O), 173.8 (C═O); IR (NaCl, ν, cm^{-1}): 1794 (C═O), 1753 (C═O), 1659 (C═O), 1521, 1348; MS *m*/*z* (ES+) 385, $[M+Na]^+$; HRMS *m*/*z* (ES^+): found 363.1180 $[M+H]^+$; $C_{17}H_{19}N_2O_7$ requires 363.1187.

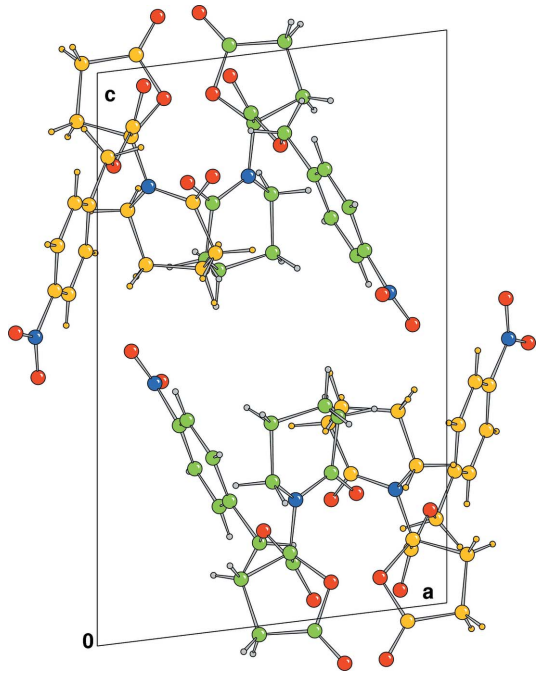


Figure 3
A view along the pseudo-glide plane (0.42 − *x*, 0.50 + *y*, 0.00 + *z*) that relates the two independent molecules. C atoms in the independent molecules are coloured green (disordered) and orange.

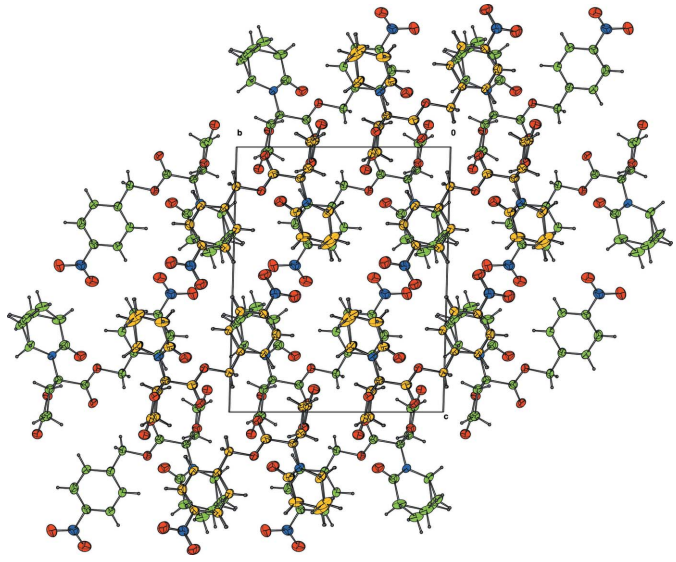


Figure 4
A cross section through the bilayers in the structure of (2). The interface rich in nitro groups lies parallel to *ab* at *c* = 0.5.

Crystal data

$C_{17}H_{18}N_2O_7$
M_r = 362.34
Triclinic, $P\overline{1}$
a = 9.2366 (9) Å
b = 12.0658 (10) Å
c = 15.0535 (15) Å
α = 88.416 (3)°
β = 82.933 (3)°
γ = 89.663 (7)°
V = 1664.3 (3) Å^3
Z = 4
Mo *K*α radiation
μ = 0.11 mm^{-1}
T = 200 (2) K
0.65 × 0.08 × 0.03 mm

Data collection

Nonius KappaCCD area-detector diffractometer	30982 measured reflections 8658 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003) T_{min} = 0.82, T_{max} = 1.00	8640 reflections with $I > -3\sigma(I)$ R_{int} = 0.067

Refinement

$R[F^2 > -3\sigma(F^2)]$ = 0.136	489 parameters
$wR(F^2)$ = 0.310	H-atom parameters constrained
S = 1.01	$\Delta\rho_{max}$ = 0.88 e Å^{-3}
8640 reflections	$\Delta\rho_{min}$ = −0.55 e Å^{-3}

Table 1
Comparison of in-house and National Crystallography Service results.

Entries in the rows containing 'shifts' are the differences between the results of data set 1 and the corresponding column.

Quantity	In-house data set 1	In-house data set 2	NCS data set
`diffrn_measured_fraction_theta_full`	0.77	0.95	0.98
Reflections in refinement	5596	7047	8640
Data collection (h)	20	21	3
Temperature (K)	150	200	200
$R(2\sigma)$	0.125	0.095	0.110
wR(all)	0.273	0.261	0.311
θ_{max} (°)	27.5	27.6	29.3
Occupancy of C160 and C170	0.682 (10)	0.679 (9)	0.665 (9)
Twin fraction	0.838 (8)	0.842 (7)	0.709 (5)
Mean shift in atomic coordinate (Å)		0.01	0.02
r.m.s. shift in atomic coordinate (Å)		0.01	0.02
Maximum shift (Å)		C171, 0.025 (1)	O102, 0.042 (1)
Maximum *Mogul* discrepancies (*Mogul* s.u. in parentheses)			
C18—C171—N12 angle (°)	6.17 (1.46)	6.32 (1.46)	5.75 (1.46)
C15—C161 bond length (Å)	0.07 (4)	0.05 (4)	0.06 (4)

Compound (2) crystallizes as thin plates which are always twinned. Initial structure determination and refinement were from data collected on an in-house Nonius KappaCCD diffractometer using a sealed-tube source (data set 1 in Table 1). The low completeness even at $\theta = 25°$ generated two level A *checkCIF* alerts. We were advised to obtain new data from the UK EPSRC National Crystallography Service (NCS) rotating anode diffractometer. A new sample was prepared (also twinned) and a suitable crystal was selected (data set 2 in Table 1). It was observed that the mosaicity deteriorated reversibly on lowering the temperature: the optimal mosaic spread occurred at 200 K. The structure from the NCS data, which generated no level A alerts, is reported in this paper. All three analyses yield the same structural information, although the rotating anode data are undoubtably crystallographically superior (Table 1). The quality of an analysis is undoubtedly limited by the quality of the samples nature provides.

H atoms were located in a difference map, but those attached to C atoms were repositioned geometrically and initially refined with soft restraints on bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98 Å and O—H = 0.82 Å) and U_{iso}(H) values (in the range 1.2–1.5 times U_{eq} of the parent atom), after which the positions were refined with riding constraints. The disordered atoms were refined with bond length similarity and anisotropic displacement parameter similarity restraints. C160, C170 and attached H atoms are disordered over two sites, with occupancy factors 0.665(9) and 0.335(9).

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

The authors gratefully acknowledge the effort of the EPSRC National Crystallography Service for collecting data from this difficult sample. We acknowledge the MRC for a Capacity Building Studentship to SJS and the European Union for funding to DSF through the FP6 Integrated Project EUR-INTAFAR (Project No. LSHM-CT-2004-512138) under the thematic priority Life Sciences, Genomics and Biotechnology for Health.

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supporting information

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4-Nitrobenzyl 5-oxo-2-(2-oxopiperidin-1-yl)tetrahydrofuran-2-carboxylate

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4-Nitrobenzyl 5-oxo-2-(2-oxopiperidin-1-yl)tetrahydrofuran-2-carboxylate

Crystal data

$C_{17}H_{18}N_2O_7$
$M_r = 362.34$
Triclinic, $P\overline{1}$
$a = 9.2366$ (9) Å
$b = 12.0658$ (10) Å
$c = 15.0535$ (15) Å
$\alpha = 88.416$ (3)°
$\beta = 82.933$ (3)°
$\gamma = 89.663$ (7)°
$V = 1664.3$ (3) Å^3
$Z = 4$
$F(000) = 760$
$D_x = 1.446$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5599 reflections
$\theta = 5$–27°
$\mu = 0.11$ mm^{-1}
$T = 200$ K
Plate, colourless
0.65 × 0.08 × 0.03 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.82$, $T_{max} = 1.00$
30982 measured reflections
8658 independent reflections
8640 reflections with $I > -3\sigma(I)$
$R_{int} = 0.067$
$\theta_{max} = 29.3°$, $\theta_{min} = 3.0°$
$h = -12 \rightarrow 9$
$k = -16 \rightarrow 16$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.136$
$wR(F^2) = 0.310$
$S = 1.02$
8640 reflections
489 parameters
60 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + (0.14P)^2 + 4.3P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} = 0.000171$
$\Delta\rho_{max} = 0.88$ e Å^{-3}
$\Delta\rho_{min} = -0.55$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C104	1.1211 (5)	1.1478 (3)	0.3709 (3)	0.0302	
C126	1.1137 (5)	1.2094 (3)	0.2928 (3)	0.0342	

C125	1.0633 (5)	1.1573 (3)	0.2220 (3)	0.0314
C107	1.0230 (4)	1.0467 (3)	0.2283 (2)	0.0256
C106	1.0328 (5)	0.9877 (3)	0.3086 (3)	0.0335
C105	1.0818 (5)	1.0383 (3)	0.3799 (3)	0.0346
C108	0.9688 (5)	0.9961 (3)	0.1490 (3)	0.0288
O109	0.9525 (3)	0.8785 (2)	0.16551 (18)	0.0279
C110	0.8977 (4)	0.8250 (3)	0.1015 (3)	0.0269
O124	0.8672 (3)	0.8660 (2)	0.0324 (2)	0.0328
C111	0.9033 (4)	0.6984 (3)	0.1178 (2)	0.0257
N112	0.8532 (4)	0.6651 (3)	0.2090 (2)	0.0291
C113	0.7255 (5)	0.7145 (4)	0.2461 (3)	0.0319
C115	0.6619 (6)	0.6772 (5)	0.3391 (3)	0.0448
C116	0.6997 (7)	0.5576 (6)	0.3637 (4)	0.0614
C117	0.8603 (7)	0.5442 (6)	0.3454 (4)	0.0628
C118	0.9129 (6)	0.5628 (4)	0.2466 (3)	0.0419
O114	0.6694 (4)	0.7894 (3)	0.2053 (2)	0.0386
C122	1.0584 (4)	0.6620 (3)	0.0798 (3)	0.0288
C121	1.0447 (5)	0.6530 (4)	−0.0199 (3)	0.0330
C120	0.8875 (5)	0.6341 (3)	−0.0234 (3)	0.0283
O119	0.8094 (3)	0.6504 (2)	0.05860 (18)	0.0279
O123	0.8271 (4)	0.6076 (3)	−0.0857 (2)	0.0375
N101	1.1773 (5)	1.1992 (3)	0.4472 (3)	0.0409
O102	1.1686 (6)	1.1484 (3)	0.5184 (2)	0.0643
O103	1.2341 (5)	1.2893 (3)	0.4362 (3)	0.0591
H1261	1.1419	1.2839	0.2888	0.0420*
H1251	1.0573	1.1978	0.1686	0.0397*
H1061	1.0059	0.9130	0.3137	0.0389*
H1051	1.0887	0.9987	0.4339	0.0407*
H1081	1.0372	1.0117	0.0949	0.0362*
H1082	0.8747	1.0286	0.1392	0.0363*
H1151	0.6985	0.7249	0.3810	0.0544*
H1152	0.5563	0.6844	0.3419	0.0539*
H1161	0.6666	0.5423	0.4259	0.0742*
H1162	0.6542	0.5072	0.3255	0.0742*
H1171	0.9070	0.5985	0.3793	0.0746*
H1172	0.8886	0.4690	0.3638	0.0742*
H1181	1.0187	0.5673	0.2399	0.0527*
H1182	0.8820	0.5021	0.2108	0.0529*
H1221	1.1311	0.7158	0.0923	0.0354*
H1222	1.0844	0.5895	0.1045	0.0354*
H1211	1.0713	0.7230	−0.0504	0.0403*
H1212	1.1019	0.5952	−0.0493	0.0402*
N1	0.1649 (5)	0.7142 (3)	0.4458 (3)	0.0460
O2	0.0978 (6)	0.6609 (3)	0.5068 (3)	0.0647
O3	0.1849 (7)	0.8156 (3)	0.4474 (3)	0.0775
C4	0.2346 (5)	0.6562 (3)	0.3672 (3)	0.0330
C5	0.2574 (5)	0.5439 (3)	0.3747 (3)	0.0336
C6	0.3308 (5)	0.4893 (3)	0.3028 (3)	0.0305

C7	0.3788 (4)	0.5476 (3)	0.2239 (3)	0.0257	
C8	0.4643 (5)	0.4938 (3)	0.1450 (3)	0.0277	
O9	0.4762 (3)	0.3770 (2)	0.16513 (18)	0.0277	
C10	0.5603 (4)	0.3223 (3)	0.1028 (3)	0.0258	
C11	0.5518 (4)	0.1965 (3)	0.1211 (2)	0.0248	
N12	0.5677 (4)	0.1641 (3)	0.2127 (2)	0.0302	
C13	0.6698 (5)	0.2216 (4)	0.2520 (3)	0.0334	
O14	0.7412 (4)	0.2956 (3)	0.2108 (2)	0.0400	
C15	0.6893 (6)	0.1921 (5)	0.3482 (3)	0.0478	
C18	0.5015 (6)	0.0592 (4)	0.2490 (3)	0.0427	
O19	0.6692 (3)	0.1476 (2)	0.06104 (18)	0.0276	
C20	0.6230 (5)	0.1312 (3)	−0.0202 (3)	0.0294	
C21	0.4627 (5)	0.1489 (4)	−0.0147 (3)	0.0313	
C22	0.4120 (4)	0.1582 (3)	0.0856 (3)	0.0286	
O23	0.7076 (4)	0.1051 (2)	−0.0832 (2)	0.0359	
O24	0.6195 (3)	0.3617 (2)	0.03365 (19)	0.0327	
C25	0.3497 (5)	0.6606 (3)	0.2169 (3)	0.0312	
C26	0.2788 (5)	0.7162 (3)	0.2889 (3)	0.0339	
H51	0.2240	0.5056	0.4272	0.0402*	
H61	0.3497	0.4129	0.3072	0.0378*	
H81	0.5602	0.5257	0.1337	0.0337*	
H82	0.4147	0.5053	0.0926	0.0338*	
H151	0.6517	0.2522	0.3854	0.0593*	
H152	0.7927	0.1851	0.3541	0.0590*	
H181	0.3961	0.0634	0.2505	0.0510*	
H182	0.5372	0.0004	0.2092	0.0509*	
H211	0.4447	0.2178	−0.0471	0.0391*	
H212	0.4182	0.0880	−0.0411	0.0391*	
H221	0.3799	0.0861	0.1112	0.0356*	
H222	0.3329	0.2112	0.0982	0.0361*	
H251	0.3793	0.6992	0.1622	0.0386*	
H261	0.2623	0.7928	0.2854	0.0420*	
C160	0.5793 (10)	0.1121 (7)	0.3972 (5)	0.0516	0.665 (9)
C170	0.5749 (11)	0.0176 (6)	0.3322 (5)	0.0507	0.665 (9)
H1601	0.6095	0.0865	0.4538	0.0612*	0.665 (9)
H1602	0.4848	0.1484	0.4069	0.0610*	0.665 (9)
H1701	0.5180	−0.0429	0.3617	0.0603*	0.665 (9)
H1702	0.6728	−0.0078	0.3134	0.0598*	0.665 (9)
C161	0.6519 (17)	0.0660 (9)	0.3702 (11)	0.0518	0.335 (9)
C171	0.4986 (17)	0.0390 (14)	0.3510 (7)	0.0480	0.335 (9)
H1611	0.6604	0.0496	0.4328	0.0620*	0.335 (9)
H1612	0.7201	0.0200	0.3329	0.0621*	0.335 (9)
H1711	0.4286	0.0882	0.3835	0.0570*	0.335 (9)
H1712	0.4735	−0.0372	0.3679	0.0572*	0.335 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C104	0.035 (2)	0.0268 (18)	0.0280 (19)	−0.0041 (15)	0.0014 (15)	−0.0066 (15)
C126	0.041 (2)	0.0238 (18)	0.037 (2)	−0.0074 (16)	−0.0013 (18)	0.0004 (16)
C125	0.040 (2)	0.0257 (18)	0.0279 (19)	−0.0029 (16)	−0.0048 (16)	0.0083 (15)
C107	0.0291 (19)	0.0231 (17)	0.0235 (17)	0.0031 (13)	0.0007 (14)	0.0024 (13)
C106	0.051 (3)	0.0188 (17)	0.031 (2)	−0.0022 (16)	−0.0066 (18)	0.0055 (15)
C105	0.050 (3)	0.0257 (19)	0.0271 (19)	−0.0020 (17)	−0.0012 (17)	−0.0011 (15)
C108	0.038 (2)	0.0202 (16)	0.0281 (19)	−0.0021 (14)	−0.0057 (16)	0.0036 (14)
O109	0.0368 (16)	0.0221 (12)	0.0255 (13)	−0.0021 (10)	−0.0076 (11)	0.0023 (10)
C110	0.0243 (19)	0.0300 (18)	0.0262 (18)	−0.0011 (14)	−0.0024 (14)	0.0032 (14)
O124	0.0407 (17)	0.0284 (14)	0.0307 (15)	−0.0028 (12)	−0.0123 (12)	0.0069 (11)
C111	0.0273 (19)	0.0265 (17)	0.0236 (17)	−0.0026 (14)	−0.0057 (14)	0.0012 (14)
N112	0.0368 (19)	0.0272 (16)	0.0236 (15)	−0.0036 (13)	−0.0064 (13)	0.0048 (12)
C113	0.031 (2)	0.039 (2)	0.0256 (19)	−0.0077 (16)	−0.0051 (15)	0.0020 (16)
C115	0.039 (3)	0.066 (3)	0.029 (2)	−0.005 (2)	−0.0027 (18)	0.005 (2)
C116	0.056 (3)	0.081 (4)	0.047 (3)	−0.018 (3)	−0.010 (3)	0.029 (3)
C117	0.060 (4)	0.079 (4)	0.047 (3)	−0.004 (3)	−0.006 (3)	0.036 (3)
C118	0.052 (3)	0.038 (2)	0.037 (2)	−0.002 (2)	−0.010 (2)	0.0157 (19)
O114	0.0356 (17)	0.0427 (17)	0.0361 (16)	0.0034 (13)	−0.0004 (13)	0.0035 (13)
C122	0.028 (2)	0.0312 (19)	0.0280 (19)	0.0028 (15)	−0.0081 (15)	0.0009 (15)
C121	0.031 (2)	0.037 (2)	0.030 (2)	0.0030 (16)	−0.0049 (16)	0.0012 (16)
C120	0.038 (2)	0.0206 (16)	0.0264 (18)	−0.0007 (14)	−0.0058 (15)	0.0016 (14)
O119	0.0269 (14)	0.0293 (13)	0.0281 (14)	−0.0042 (10)	−0.0061 (11)	0.0014 (11)
O123	0.0428 (18)	0.0380 (16)	0.0338 (16)	−0.0021 (13)	−0.0123 (13)	−0.0028 (13)
N101	0.057 (3)	0.0347 (19)	0.0304 (19)	−0.0113 (17)	−0.0030 (17)	−0.0016 (15)
O102	0.111 (4)	0.051 (2)	0.0343 (19)	−0.029 (2)	−0.021 (2)	0.0071 (16)
O103	0.090 (3)	0.041 (2)	0.048 (2)	−0.027 (2)	−0.017 (2)	0.0016 (16)
N1	0.065 (3)	0.042 (2)	0.033 (2)	0.023 (2)	−0.0112 (19)	−0.0071 (17)
O2	0.096 (3)	0.057 (2)	0.0348 (19)	0.013 (2)	0.016 (2)	−0.0009 (17)
O3	0.142 (5)	0.038 (2)	0.052 (2)	0.026 (2)	−0.004 (3)	−0.0106 (18)
C4	0.044 (2)	0.0278 (19)	0.0278 (19)	0.0083 (16)	−0.0061 (17)	−0.0037 (15)
C5	0.046 (3)	0.0283 (19)	0.0253 (19)	0.0010 (17)	0.0015 (17)	0.0018 (15)
C6	0.041 (2)	0.0206 (17)	0.0288 (19)	0.0016 (15)	−0.0021 (16)	0.0010 (14)
C7	0.0268 (19)	0.0237 (17)	0.0272 (18)	−0.0021 (13)	−0.0066 (14)	0.0017 (14)
C8	0.035 (2)	0.0193 (16)	0.0277 (18)	0.0001 (14)	0.0015 (15)	0.0014 (14)
O9	0.0320 (15)	0.0224 (12)	0.0274 (13)	0.0021 (10)	0.0010 (11)	0.0011 (10)
C10	0.0269 (19)	0.0241 (17)	0.0265 (18)	−0.0001 (13)	−0.0036 (14)	0.0013 (14)
C11	0.0281 (19)	0.0241 (17)	0.0215 (17)	0.0019 (13)	0.0002 (13)	0.0011 (13)
N12	0.042 (2)	0.0237 (15)	0.0242 (16)	0.0008 (13)	−0.0041 (14)	0.0044 (12)
C13	0.035 (2)	0.034 (2)	0.030 (2)	0.0067 (16)	−0.0052 (16)	0.0029 (16)
O14	0.0377 (18)	0.0494 (19)	0.0343 (16)	−0.0076 (14)	−0.0120 (13)	0.0063 (14)
C15	0.051 (3)	0.063 (3)	0.030 (2)	−0.004 (2)	−0.0130 (19)	0.010 (2)
C18	0.059 (3)	0.032 (2)	0.035 (2)	−0.0019 (19)	0.000 (2)	0.0128 (17)
O19	0.0279 (14)	0.0290 (13)	0.0259 (13)	0.0048 (10)	−0.0030 (10)	−0.0012 (10)
C20	0.043 (2)	0.0185 (16)	0.0262 (18)	−0.0022 (14)	−0.0023 (16)	0.0042 (14)
C21	0.031 (2)	0.035 (2)	0.0289 (19)	−0.0006 (16)	−0.0072 (15)	0.0010 (16)

C22	0.028 (2)	0.0272 (18)	0.0294 (19)	−0.0018 (14)	0.0000 (15)	0.0025 (15)
O23	0.0420 (18)	0.0325 (15)	0.0309 (15)	0.0018 (12)	0.0054 (13)	−0.0014 (12)
O24	0.0388 (17)	0.0288 (14)	0.0284 (14)	0.0001 (12)	0.0025 (12)	0.0069 (11)
C25	0.039 (2)	0.0276 (19)	0.0270 (19)	0.0011 (16)	−0.0061 (16)	0.0048 (15)
C26	0.039 (2)	0.0237 (18)	0.040 (2)	0.0069 (16)	−0.0107 (18)	0.0008 (16)
C160	0.062 (4)	0.063 (4)	0.028 (3)	−0.003 (3)	0.000 (3)	0.009 (3)
C170	0.070 (4)	0.044 (3)	0.035 (3)	−0.001 (3)	−0.001 (3)	0.015 (2)
C161	0.061 (5)	0.064 (4)	0.030 (4)	−0.001 (4)	−0.006 (4)	0.015 (4)
C171	0.062 (5)	0.043 (4)	0.036 (3)	−0.001 (4)	−0.002 (4)	0.016 (4)

Geometric parameters (Å, º)

C104—C126	1.382 (6)	C5—H51	0.925
C104—C105	1.371 (5)	C6—C7	1.389 (5)
C104—N101	1.471 (5)	C6—H61	0.939
C126—C125	1.383 (6)	C7—C8	1.505 (5)
C126—H1261	0.935	C7—C25	1.391 (5)
C125—C107	1.385 (5)	C8—O9	1.440 (4)
C125—H1251	0.937	C8—H81	0.962
C107—C106	1.398 (5)	C8—H82	0.966
C107—C108	1.496 (5)	O9—C10	1.329 (5)
C106—C105	1.375 (6)	C10—C11	1.536 (5)
C106—H1061	0.934	C10—O24	1.202 (5)
C105—H1051	0.940	C11—N12	1.447 (5)
C108—O109	1.439 (4)	C11—O19	1.457 (4)
C108—H1081	0.982	C11—C22	1.536 (6)
C108—H1082	0.977	N12—C13	1.374 (6)
O109—C110	1.326 (5)	N12—C18	1.471 (5)
C110—O124	1.204 (5)	C13—O14	1.223 (5)
C110—C111	1.542 (5)	C13—C15	1.513 (6)
C111—N112	1.441 (5)	C15—H151	0.965
C111—C122	1.542 (6)	C15—H152	0.973
C111—O119	1.451 (4)	C15—C160	1.515 (7)
N112—C113	1.380 (6)	C15—H151	0.965
N112—C118	1.476 (5)	C15—H152	0.973
C113—C115	1.509 (6)	C15—C161	1.579 (9)
C113—O114	1.227 (5)	C18—H181	0.972
C115—C116	1.528 (8)	C18—H182	0.971
C115—H1151	0.957	C18—C170	1.565 (7)
C115—H1152	0.974	C18—H181	0.972
C116—C117	1.483 (9)	C18—H182	0.971
C116—H1161	0.961	C18—C171	1.545 (9)
C116—H1162	0.981	O19—C20	1.364 (5)
C117—C118	1.518 (7)	C20—C21	1.487 (6)
C117—H1171	0.976	C20—O23	1.200 (5)
C117—H1172	0.984	C21—C22	1.531 (6)
C118—H1181	0.972	C21—H211	0.975
C118—H1182	0.985	C21—H212	0.964

C122—C121	1.527 (6)	C22—H221	0.975
C122—H1221	0.975	C22—H222	0.972
C122—H1222	0.981	C25—C26	1.384 (6)
C121—C120	1.479 (6)	C25—H251	0.945
C121—H1211	0.969	C26—H261	0.937
C121—H1212	0.958	C160—C170	1.526 (8)
C120—O119	1.369 (5)	C160—H1601	0.971
C120—O123	1.201 (5)	C160—H1602	0.971
N101—O102	1.213 (5)	C170—H1701	0.967
N101—O103	1.208 (5)	C170—H1702	0.965
N1—O2	1.213 (6)	C161—C171	1.519 (9)
N1—O3	1.240 (6)	C161—H1611	0.971
N1—C4	1.469 (6)	C161—H1612	0.972
C4—C5	1.373 (6)	C171—H1711	0.971
C4—C26	1.385 (6)	C171—H1712	0.969
C5—C6	1.385 (6)		

C126—C104—C105	122.6 (4)	C6—C7—C8	122.5 (3)
C126—C104—N101	119.6 (4)	C6—C7—C25	119.6 (4)
C105—C104—N101	117.8 (4)	C8—C7—C25	117.8 (3)
C104—C126—C125	117.8 (4)	C7—C8—O9	108.5 (3)
C104—C126—H1261	120.5	C7—C8—H81	110.0
C125—C126—H1261	121.7	O9—C8—H81	109.5
C126—C125—C107	121.4 (4)	C7—C8—H82	109.6
C126—C125—H1251	119.0	O9—C8—H82	110.0
C107—C125—H1251	119.6	H81—C8—H82	109.1
C125—C107—C106	118.8 (4)	C8—O9—C10	113.8 (3)
C125—C107—C108	118.4 (3)	O9—C10—C11	111.4 (3)
C106—C107—C108	122.8 (3)	O9—C10—O24	125.7 (3)
C107—C106—C105	120.7 (4)	C11—C10—O24	122.1 (3)
C107—C106—H1061	119.6	C10—C11—N12	113.6 (3)
C105—C106—H1061	119.7	C10—C11—O19	106.4 (3)
C106—C105—C104	118.8 (4)	N12—C11—O19	109.6 (3)
C106—C105—H1051	120.8	C10—C11—C22	105.9 (3)
C104—C105—H1051	120.4	N12—C11—C22	116.3 (3)
C107—C108—O109	109.0 (3)	O19—C11—C22	104.2 (3)
C107—C108—H1081	110.0	C11—N12—C13	116.0 (3)
O109—C108—H1081	110.8	C11—N12—C18	118.4 (3)
C107—C108—H1082	110.0	C13—N12—C18	124.0 (3)
O109—C108—H1082	109.6	N12—C13—O14	120.8 (4)
H1081—C108—H1082	107.4	N12—C13—C15	118.2 (4)
C108—O109—C110	114.6 (3)	O14—C13—C15	121.0 (4)
O109—C110—O124	125.6 (4)	C13—C15—H151	108.6
O109—C110—C111	111.3 (3)	C13—C15—H152	109.7
O124—C110—C111	122.2 (4)	H151—C15—H152	107.1
C110—C111—N112	112.9 (3)	C13—C15—C160	116.3 (5)
C110—C111—C122	105.9 (3)	H151—C15—C160	91.5
N112—C111—C122	117.1 (3)	H152—C15—C160	120.9

C110—C111—O119	106.4 (3)	C13—C15—H151	108.6
N112—C111—O119	109.4 (3)	C13—C15—H152	109.7
C122—C111—O119	104.4 (3)	H151—C15—H152	107.1
C111—N112—C113	115.6 (3)	C13—C15—C161	110.5 (7)
C111—N112—C118	118.9 (4)	H151—C15—C161	124.0
C113—N112—C118	123.1 (4)	H152—C15—C161	95.6
N112—C113—C115	118.1 (4)	N12—C18—H181	109.7
N112—C113—O114	120.4 (4)	N12—C18—H182	108.2
C115—C113—O114	121.5 (4)	H181—C18—H182	108.2
C113—C115—C116	114.1 (5)	N12—C18—C170	110.4 (5)
C113—C115—H1151	108.1	H181—C18—C170	121.7
C116—C115—H1151	108.2	H182—C18—C170	97.2
C113—C115—H1152	106.8	N12—C18—H181	109.7
C116—C115—H1152	109.1	N12—C18—H182	108.2
H1151—C115—H1152	110.6	H181—C18—H182	108.2
C115—C116—C117	108.0 (5)	N12—C18—C171	115.9 (7)
C115—C116—H1161	109.7	H181—C18—C171	95.0
C117—C116—H1161	110.7	H182—C18—C171	118.7
C115—C116—H1162	109.2	C11—O19—C20	109.7 (3)
C117—C116—H1162	108.2	O19—C20—C21	110.2 (3)
H1161—C116—H1162	110.8	O19—C20—O23	120.7 (4)
C116—C117—C118	111.2 (5)	C21—C20—O23	129.1 (4)
C116—C117—H1171	108.9	C20—C21—C22	104.5 (3)
C118—C117—H1171	108.6	C20—C21—H211	108.5
C116—C117—H1172	110.0	C22—C21—H211	111.1
C118—C117—H1172	108.9	C20—C21—H212	109.9
H1171—C117—H1172	109.3	C22—C21—H212	112.9
C117—C118—N112	112.8 (5)	H211—C21—H212	109.6
C117—C118—H1181	107.8	C11—C22—C21	101.9 (3)
N112—C118—H1181	109.2	C11—C22—H221	111.6
C117—C118—H1182	111.3	C21—C22—H221	110.1
N112—C118—H1182	105.7	C11—C22—H222	111.6
H1181—C118—H1182	110.0	C21—C22—H222	112.9
C111—C122—C121	102.0 (3)	H221—C22—H222	108.7
C111—C122—H1221	111.3	C7—C25—C26	120.6 (4)
C121—C122—H1221	113.5	C7—C25—H251	119.2
C111—C122—H1222	112.0	C26—C25—H251	120.2
C121—C122—H1222	109.8	C4—C26—C25	118.4 (4)
H1221—C122—H1222	108.3	C4—C26—H261	120.5
C122—C121—C120	104.5 (3)	C25—C26—H261	121.1
C122—C121—H1211	109.5	C15—C160—C170	103.8 (6)
C120—C121—H1211	108.3	C15—C160—H1601	111.2
C122—C121—H1212	114.8	C170—C160—H1601	111.8
C120—C121—H1212	110.7	C15—C160—H1602	109.1
H1211—C121—H1212	108.8	C170—C160—H1602	110.2
C121—C120—O119	110.6 (3)	H1601—C160—H1602	110.5
C121—C120—O123	128.8 (4)	C160—C170—C18	109.5 (6)
O119—C120—O123	120.5 (4)	C160—C170—H1701	109.4

C111—O119—C120	109.5 (3)	C18—C170—H1701	108.9
C104—N101—O102	118.9 (4)	C160—C170—H1702	109.6
C104—N101—O103	118.8 (4)	C18—C170—H1702	110.0
O102—N101—O103	122.2 (4)	H1701—C170—H1702	109.5
O2—N1—O3	123.7 (4)	C15—C161—C171	111.2 (10)
O2—N1—C4	119.1 (4)	C15—C161—H1611	109.3
O3—N1—C4	117.0 (4)	C171—C161—H1611	109.4
N1—C4—C5	118.6 (4)	C15—C161—H1612	109.2
N1—C4—C26	119.4 (4)	C171—C161—H1612	108.2
C5—C4—C26	122.0 (4)	H1611—C161—H1612	109.5
C4—C5—C6	119.2 (4)	C18—C171—C161	104.8 (10)
C4—C5—H51	120.1	C18—C171—H1711	110.4
C6—C5—H51	120.7	C161—C171—H1711	110.0
C5—C6—C7	120.1 (4)	C18—C171—H1712	111.0
C5—C6—H61	120.4	C161—C171—H1712	111.2
C7—C6—H61	119.5	H1711—C171—H1712	109.3