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4-*N*-Acetylamino-5-[*N*-acetyl-*N*-(tetra-*O*-acetyl-β-D-glucopyranosyl)amino]-1,3-dimethyluracil

Adolfo Sánchez Rodrigo,[a] Manuel Nogueras Montiel,[a] Justo Cobo,[a] John N. Low[b] and Christopher Glidewell[c]*

[a]Departamento de Química Inorgánica y Orgánica, Universidad de Jaén, 23071 Jaén, Spain, [b]Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, and [c]School of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland

Correspondence e-mail: cg@st-andrews.ac.uk

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.035
wR factor = 0.087
Data-to-parameter ratio = 8.8

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

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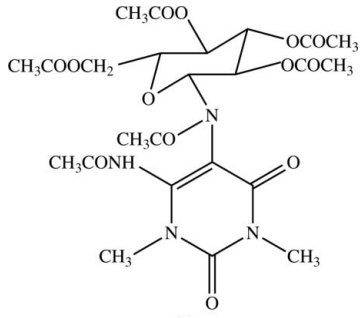
The title compound, $C_{24}H_{32}N_4O_{13}$, crystallizes with two molecules in the asymmetric unit. These have very similar conformations, and each contains an intramolecular N—H···O hydrogen bond. There are no significant intermolecular interactions.

Comment

The title compound, (I), has been prepared for use as an intermediate in the synthesis of nucleoside analogues with potential antitumour or antiviral applications.



(I)

Compound (I) crystallizes with two independent molecules (Fig. 1) in the space group $P2_1$, and the two molecules have very similar conformations. In the glucopyranose rings, the ring-puckering parameters (Cremer & Pople, 1975) in molecules 1 and 2 (containing N11 and N31 respectively, Fig. 1) are, for the atom sequences (O25, C21–C25) and (O45, C41–C45), $\theta = 3.5\ (2)$ and $4.5\ (2)^\circ$, respectively, and $\varphi = 104\ (4)$ and $342\ (3)^\circ$, respectively: thus each ring has an almost perfect chair conformation, for which the ideal value of θ is zero.

The conformation adopted by all the substituents exocyclic to these chair rings are again similar for the two molecules, as shown by the leading torsion angles (Table 1), but the differences between them are sufficient to preclude any additional symmetry. The bond distances within the uracil rings are also very similar for the two molecules.

In each molecule there is a single intramolecular N—H···O hydrogen bond (Table 2), forming an *S*(7) motif (Bernstein *et al.*, 1995), and these interactions may have an influence on the overall molecular conformations.

There are no significant direction-specific intermolecular interactions.

Experimental

Crystals of compound (I) were prepared according to a published procedure (Melgarejo Sampedro *et al.*, 1982).

Crystal data

$C_{24}H_{32}N_4O_{13}$
M_r = 584.54
Monoclinic, $P2_1$
a = 8.8490 (1) Å
b = 17.8429 (2) Å
c = 18.3058 (2) Å
β = 103.7830 (8)°
V = 2807.11 (6) Å^3
Z = 4
D_x = 1.383 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6642 reflections
θ = 2.9–27.5°
μ = 0.11 mm^{-1}
T = 120 (2) K
Block, colourless
0.22 × 0.20 × 0.18 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.954, T_{max} = 0.980
46614 measured reflections
6642 independent reflections
5791 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 27.5°
$h = -11 \rightarrow 10$
$k = -22 \rightarrow 23$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.087
S = 1.08
6642 reflections
755 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0458P)^2 + 0.4658P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N11—C12	1.382 (3)	N31—C32	1.368 (4)
C12—N13	1.386 (3)	C32—N33	1.396 (3)
N13—C14	1.376 (3)	N33—C34	1.374 (3)
C14—C15	1.361 (3)	C34—C35	1.362 (3)
C15—C16	1.449 (3)	C35—C36	1.448 (3)
C16—N11	1.400 (3)	C36—N31	1.389 (3)
C15—C14—N14—C141	−116.1 (2)	C35—C34—N34—C341	−120.4 (2)
C15—N15—C21—C22	−35.7 (3)	C35—N35—C41—C42	−27.7 (3)
C22—C21—N15—C151	151.98 (19)	C42—C41—N35—C351	145.74 (19)
C21—C22—O22—C221	129.62 (18)	C41—C42—O42—C421	132.84 (19)
C22—C23—O23—C231	−150.68 (18)	C42—C43—O43—C431	−146.41 (19)
C23—C24—O24—C241	104.9 (2)	C43—C44—O44—C441	100.1 (2)
C24—C25—C26—O26	61.3 (3)	C44—C45—C46—O46	49.3 (3)
C25—C26—O26—C261	−136.4 (2)	C45—C46—O46—C461	−124.2 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N14—H14···O25	0.88	2.00	2.787 (3)	148
N34—H34···O45	0.88	2.06	2.867 (2)	152

All H atoms were located in difference maps, and then treated as riding atoms with distances C—H = 0.98 (CH_3), 0.99 (CH_2) or 1.00 Å (CH), and N—H = 0.88 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N) or 1.5U_{eq}(methyl C). In the absence of significant anomalous scattering, the Flack (1983) parameter was indeterminate (Flack & Bernardinelli, 2000); hence, the Friedel equivalents were merged prior to the final refinements. The absolute configuration was assigned by reference to the known configuration of the chiral starting material. There is evidence for considerable libration of the acetyl groups bonded to O26 and O46.

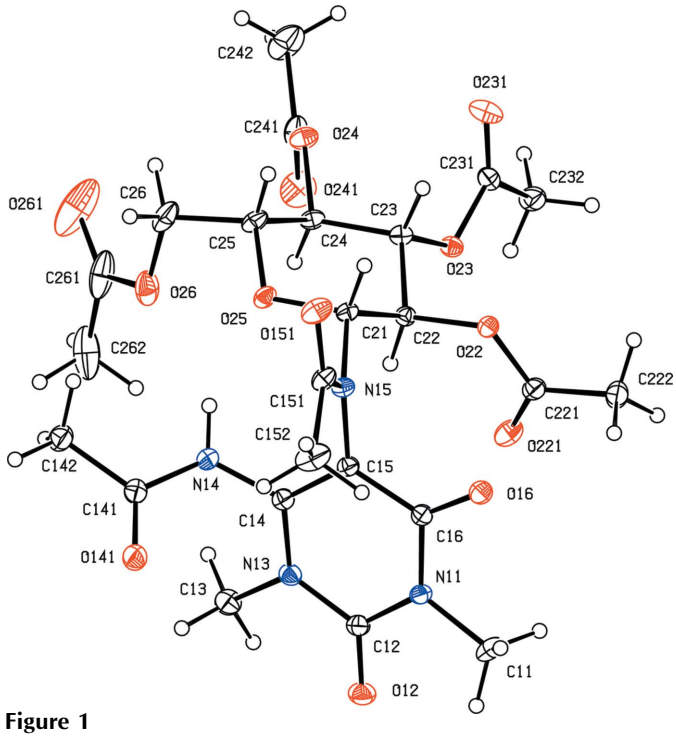


Figure 1
One of the two independent molecules of compound (I), *viz*. molecule 1, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

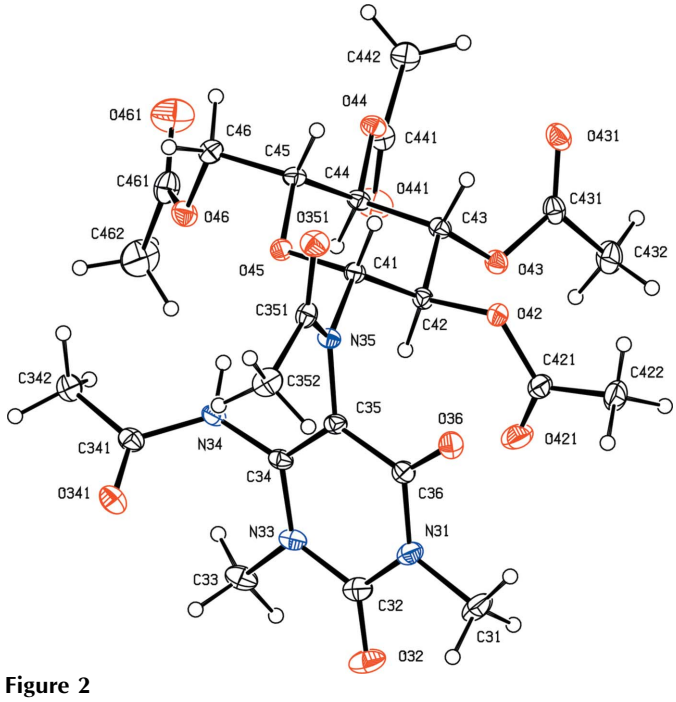


Figure 2
One of the two independent molecules of compound (I), *viz*. molecule 2, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); soft-

ware used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton, England. The authors thank the staff for all their help and advice. ASR, MNM and JC thank the Consejería de Innovación, Ciencia y Empresa (Junta de Andalucía, Spain) and the Universidad de Jaén for financial support.

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Monoclinic, $P2_1$
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$a = 8.8490$ (1) Å
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$c = 18.3058$ (2) Å
$\beta = 103.7830$ (8)°
$V = 2807.11$ (6) Å^3
$Z = 4$
$F(000) = 1232$
$D_x = 1.383$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6642 reflections
$\theta = 2.9$–27.5°
$\mu = 0.11$ mm^{-1}
$T = 120$ K
Block, colourless
0.22 × 0.20 × 0.18 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Radiation source: Bruker-Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.954$, $T_{max} = 0.980$
46614 measured reflections
6642 independent reflections
5791 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.9°$
$h = -11 \rightarrow 10$
$k = -22 \rightarrow 23$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.087$
$S = 1.08$
6642 reflections
755 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0458P)^2 + 0.4658P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N11	0.9337 (2)	0.42742 (12)	0.83728 (10)	0.0222 (4)
C11	0.8141 (3)	0.38198 (16)	0.85942 (15)	0.0339 (6)
C12	1.0086 (2)	0.48112 (14)	0.88748 (12)	0.0225 (5)
O12	0.96987 (19)	0.49537 (11)	0.94561 (9)	0.0310 (4)
N13	1.1308 (2)	0.51828 (11)	0.86795 (10)	0.0221 (4)
C13	1.1979 (3)	0.58236 (15)	0.91553 (14)	0.0294 (5)
C14	1.1807 (2)	0.50003 (13)	0.80455 (12)	0.0201 (4)
N14	1.3161 (2)	0.53533 (11)	0.79646 (11)	0.0230 (4)
C141	1.4590 (3)	0.52158 (14)	0.84579 (13)	0.0238 (5)
O141	1.47059 (19)	0.48102 (11)	0.89996 (9)	0.0308 (4)
C142	1.5929 (3)	0.56229 (16)	0.82671 (15)	0.0307 (5)
C15	1.0978 (2)	0.45189 (12)	0.75244 (12)	0.0190 (4)
N15	1.1450 (2)	0.43373 (10)	0.68527 (10)	0.0185 (4)
C151	1.2325 (3)	0.37007 (13)	0.67975 (13)	0.0238 (5)
O151	1.2766 (2)	0.35754 (10)	0.62307 (10)	0.0310 (4)
C152	1.2678 (3)	0.31951 (16)	0.74688 (17)	0.0383 (7)
C21	1.1210 (2)	0.48574 (12)	0.62313 (12)	0.0190 (4)
C22	0.9712 (2)	0.53131 (12)	0.61122 (11)	0.0183 (4)
O22	0.84262 (17)	0.47985 (9)	0.59061 (8)	0.0210 (3)
C221	0.7297 (3)	0.48458 (14)	0.62960 (13)	0.0246 (5)
O221	0.7331 (2)	0.53006 (11)	0.67844 (10)	0.0335 (4)
C222	0.6069 (3)	0.42667 (17)	0.60451 (15)	0.0356 (6)
C23	0.9632 (3)	0.58685 (13)	0.54700 (12)	0.0202 (4)
O23	0.82640 (17)	0.63257 (9)	0.54400 (8)	0.0209 (3)
C231	0.7520 (3)	0.66120 (13)	0.47597 (12)	0.0227 (5)
O231	0.7882 (2)	0.64737 (11)	0.41852 (9)	0.0351 (4)
C232	0.6224 (3)	0.71199 (16)	0.48277 (14)	0.0309 (5)
C24	1.1071 (3)	0.63619 (13)	0.56516 (12)	0.0211 (4)
O24	1.1150 (2)	0.68135 (9)	0.50130 (9)	0.0276 (4)
C241	1.0788 (3)	0.75487 (14)	0.50305 (16)	0.0316 (6)
O241	1.0356 (3)	0.78322 (11)	0.55370 (13)	0.0471 (5)
C242	1.0978 (4)	0.79121 (19)	0.4323 (2)	0.0530 (9)
C25	1.2550 (3)	0.58918 (14)	0.58160 (14)	0.0254 (5)
C26	1.3980 (3)	0.63618 (16)	0.60911 (17)	0.0363 (6)
O26	1.3865 (2)	0.67209 (11)	0.67852 (12)	0.0399 (5)
C261	1.4265 (3)	0.74464 (17)	0.6863 (2)	0.0535 (9)
O261	1.4703 (3)	0.77954 (14)	0.6391 (2)	0.0902 (11)
C262	1.4111 (4)	0.7743 (2)	0.7600 (3)	0.0689 (12)
O25	1.25068 (17)	0.53662 (9)	0.63982 (9)	0.0223 (3)
C16	0.9674 (2)	0.41112 (13)	0.76804 (12)	0.0200 (4)
O16	0.89333 (18)	0.36325 (9)	0.72684 (9)	0.0256 (3)
N31	0.7874 (2)	0.58238 (13)	0.27061 (12)	0.0281 (4)
C31	0.9234 (3)	0.61973 (18)	0.25399 (18)	0.0394 (7)
C32	0.8188 (3)	0.52956 (15)	0.32636 (14)	0.0301 (5)
O32	0.9519 (2)	0.51303 (12)	0.35935 (12)	0.0414 (5)

N33	0.6916 (2)	0.49383 (12)	0.34391 (11)	0.0258 (4)
C33	0.7291 (3)	0.43388 (16)	0.40016 (14)	0.0348 (6)
C34	0.5417 (2)	0.51248 (13)	0.30817 (12)	0.0215 (4)
N34	0.4231 (2)	0.47380 (11)	0.33021 (10)	0.0232 (4)
C341	0.3989 (3)	0.47838 (14)	0.40223 (13)	0.0260 (5)
O341	0.4779 (2)	0.51943 (11)	0.44949 (9)	0.0341 (4)
C342	0.2728 (3)	0.42821 (16)	0.41584 (14)	0.0330 (6)
C35	0.5133 (2)	0.56315 (12)	0.25089 (12)	0.0202 (4)
N35	0.3594 (2)	0.57824 (11)	0.20807 (10)	0.0197 (4)
C351	0.2876 (3)	0.64703 (13)	0.21261 (13)	0.0234 (5)
O351	0.1687 (2)	0.66401 (10)	0.16741 (10)	0.0317 (4)
C352	0.3607 (3)	0.69583 (15)	0.27864 (16)	0.0345 (6)
C41	0.2791 (2)	0.52555 (13)	0.15225 (12)	0.0197 (4)
C42	0.3861 (2)	0.47726 (13)	0.11738 (12)	0.0198 (4)
O42	0.46192 (17)	0.52602 (9)	0.07462 (8)	0.0230 (3)
C421	0.6186 (3)	0.51840 (16)	0.08439 (14)	0.0308 (6)
O421	0.6951 (2)	0.47453 (13)	0.12746 (11)	0.0405 (5)
C422	0.6813 (3)	0.5733 (2)	0.03738 (16)	0.0430 (7)
C43	0.2922 (2)	0.41926 (13)	0.06475 (12)	0.0205 (4)
O43	0.40424 (18)	0.37114 (10)	0.04277 (9)	0.0241 (3)
C431	0.3674 (3)	0.34180 (14)	−0.02775 (13)	0.0267 (5)
O431	0.2442 (2)	0.35106 (10)	−0.07145 (9)	0.0313 (4)
C432	0.5003 (3)	0.29657 (17)	−0.04135 (16)	0.0381 (6)
C44	0.1912 (2)	0.37420 (13)	0.10573 (12)	0.0208 (4)
O44	0.08784 (18)	0.32624 (9)	0.05350 (9)	0.0238 (3)
C441	0.1278 (3)	0.25301 (14)	0.05120 (14)	0.0268 (5)
O441	0.2397 (2)	0.22611 (11)	0.09376 (12)	0.0431 (5)
C442	0.0179 (3)	0.21355 (16)	−0.01072 (15)	0.0343 (6)
C45	0.0903 (2)	0.42695 (13)	0.13932 (12)	0.0210 (4)
C46	−0.0091 (3)	0.38646 (14)	0.18349 (14)	0.0269 (5)
O46	0.0868 (2)	0.33473 (10)	0.23564 (9)	0.0303 (4)
C461	0.0440 (3)	0.26187 (16)	0.23186 (16)	0.0362 (6)
O461	−0.0687 (3)	0.23867 (12)	0.18726 (14)	0.0560 (6)
C462	0.1527 (4)	0.2170 (2)	0.2892 (2)	0.0630 (10)
O45	0.19311 (17)	0.47638 (9)	0.19027 (8)	0.0208 (3)
C36	0.6397 (3)	0.60310 (13)	0.23045 (12)	0.0231 (5)
O36	0.6200 (2)	0.65264 (10)	0.18299 (9)	0.0288 (4)
H11A	0.8177	0.3906	0.9127	0.051*
H11B	0.8332	0.3288	0.8515	0.051*
H11C	0.7114	0.3961	0.8289	0.051*
H13A	1.2222	0.6227	0.8838	0.044*
H13B	1.2934	0.5665	0.9514	0.044*
H13C	1.1229	0.6005	0.9431	0.044*
H14	1.3170	0.5518	0.7513	0.028*
H14A	1.6909	0.5433	0.8582	0.046*
H14B	1.5840	0.6160	0.8359	0.046*
H14C	1.5913	0.5541	0.7736	0.046*
H15A	1.3369	0.3454	0.7891	0.057*

H15B	1.3188	0.2739	0.7349	0.057*
H15C	1.1708	0.3061	0.7606	0.057*
H21	1.1220	0.4577	0.5759	0.023*
H22	0.9669	0.5585	0.6584	0.022*
H22A	0.5070	0.4459	0.6106	0.053*
H22B	0.6344	0.3813	0.6350	0.053*
H22C	0.5987	0.4147	0.5515	0.053*
H23	0.9550	0.5599	0.4983	0.024*
H23A	0.5233	0.6851	0.4677	0.046*
H23B	0.6206	0.7556	0.4501	0.046*
H23C	0.6381	0.7287	0.5351	0.046*
H24	1.1052	0.6690	0.6092	0.025*
H24A	1.0866	0.8456	0.4362	0.080*
H24B	1.0181	0.7723	0.3896	0.080*
H24C	1.2013	0.7796	0.4248	0.080*
H25	1.2631	0.5617	0.5351	0.030*
H26A	1.4922	0.6043	0.6180	0.044*
H26B	1.4057	0.6745	0.5710	0.044*
H26C	1.3990	0.8289	0.7569	0.103*
H26D	1.5045	0.7616	0.7989	0.103*
H26E	1.3197	0.7519	0.7729	0.103*
H31A	0.8885	0.6570	0.2143	0.059*
H31B	0.9891	0.5824	0.2372	0.059*
H31C	0.9832	0.6446	0.2995	0.059*
H33A	0.6442	0.3973	0.3913	0.052*
H33B	0.7426	0.4553	0.4506	0.052*
H33C	0.8256	0.4090	0.3962	0.052*
H34	0.3436	0.4602	0.2938	0.028*
H34A	0.2798	0.4250	0.4700	0.050*
H34B	0.2849	0.3781	0.3961	0.050*
H34C	0.1712	0.4487	0.3903	0.050*
H35A	0.2964	0.7406	0.2787	0.052*
H35B	0.4650	0.7109	0.2749	0.052*
H35C	0.3681	0.6678	0.3254	0.052*
H41	0.2047	0.5533	0.1115	0.024*
H42	0.4655	0.4518	0.1579	0.024*
H42A	0.7429	0.5467	0.0077	0.065*
H42B	0.7474	0.6099	0.0701	0.065*
H42C	0.5948	0.5994	0.0035	0.065*
H43	0.2256	0.4444	0.0195	0.025*
H43A	0.5783	0.3301	−0.0537	0.057*
H43B	0.4623	0.2620	−0.0833	0.057*
H43C	0.5475	0.2679	0.0040	0.057*
H44	0.2586	0.3434	0.1464	0.025*
H44A	0.0343	0.1593	−0.0050	0.051*
H44B	0.0359	0.2297	−0.0591	0.051*
H44C	−0.0892	0.2256	−0.0089	0.051*
H45	0.0221	0.4570	0.0983	0.025*

H46A	−0.0574	0.4232	0.2115	0.032*
H46B	−0.0932	0.3587	0.1487	0.032*
H46C	0.1498	0.1646	0.2727	0.095*
H46D	0.1219	0.2199	0.3371	0.095*
H46E	0.2585	0.2367	0.2957	0.095*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0198 (9)	0.0288 (10)	0.0191 (9)	0.0010 (8)	0.0065 (7)	0.0038 (8)
C11	0.0324 (13)	0.0385 (15)	0.0363 (14)	−0.0042 (12)	0.0191 (11)	0.0055 (12)
C12	0.0204 (10)	0.0279 (12)	0.0197 (10)	0.0061 (9)	0.0056 (8)	0.0028 (9)
O12	0.0294 (9)	0.0424 (11)	0.0227 (8)	0.0060 (8)	0.0094 (7)	0.0000 (7)
N13	0.0214 (9)	0.0252 (10)	0.0196 (8)	0.0030 (8)	0.0049 (7)	−0.0017 (8)
C13	0.0330 (13)	0.0272 (13)	0.0285 (12)	−0.0001 (11)	0.0087 (10)	−0.0056 (10)
C14	0.0204 (10)	0.0189 (11)	0.0213 (10)	0.0029 (8)	0.0053 (8)	0.0028 (8)
N14	0.0225 (9)	0.0246 (10)	0.0225 (9)	−0.0028 (8)	0.0065 (7)	0.0024 (8)
C141	0.0238 (10)	0.0222 (11)	0.0262 (11)	−0.0005 (9)	0.0072 (9)	−0.0056 (10)
O141	0.0261 (8)	0.0342 (10)	0.0306 (9)	−0.0010 (8)	0.0037 (7)	0.0067 (8)
C142	0.0260 (12)	0.0341 (14)	0.0338 (13)	−0.0020 (11)	0.0105 (10)	−0.0003 (11)
C15	0.0186 (10)	0.0201 (11)	0.0188 (10)	0.0033 (8)	0.0054 (8)	0.0033 (8)
N15	0.0222 (9)	0.0158 (9)	0.0189 (8)	0.0023 (7)	0.0075 (7)	0.0020 (7)
C151	0.0245 (11)	0.0189 (11)	0.0311 (12)	0.0010 (9)	0.0124 (9)	0.0022 (9)
O151	0.0367 (9)	0.0242 (9)	0.0385 (10)	0.0059 (8)	0.0214 (8)	0.0011 (8)
C152	0.0442 (15)	0.0298 (14)	0.0467 (16)	0.0162 (12)	0.0221 (13)	0.0153 (12)
C21	0.0223 (10)	0.0171 (10)	0.0190 (10)	−0.0006 (9)	0.0079 (8)	0.0013 (8)
C22	0.0202 (10)	0.0169 (11)	0.0178 (9)	−0.0009 (8)	0.0048 (8)	0.0004 (8)
O22	0.0218 (7)	0.0201 (8)	0.0210 (7)	−0.0037 (6)	0.0051 (6)	0.0012 (6)
C221	0.0204 (10)	0.0288 (13)	0.0250 (11)	0.0041 (9)	0.0059 (9)	0.0088 (10)
O221	0.0314 (9)	0.0349 (10)	0.0395 (10)	0.0028 (8)	0.0190 (8)	0.0008 (9)
C222	0.0237 (12)	0.0434 (16)	0.0364 (14)	−0.0070 (11)	0.0011 (10)	0.0132 (12)
C23	0.0242 (11)	0.0181 (10)	0.0192 (10)	0.0018 (9)	0.0071 (8)	0.0008 (9)
O23	0.0228 (7)	0.0210 (8)	0.0187 (7)	0.0027 (6)	0.0044 (6)	0.0022 (6)
C231	0.0270 (11)	0.0170 (11)	0.0214 (11)	−0.0030 (9)	0.0002 (9)	0.0027 (9)
O231	0.0504 (11)	0.0337 (10)	0.0195 (8)	0.0097 (9)	0.0051 (8)	0.0009 (7)
C232	0.0283 (12)	0.0347 (14)	0.0296 (12)	0.0053 (11)	0.0071 (10)	0.0105 (11)
C24	0.0272 (11)	0.0168 (11)	0.0212 (10)	0.0014 (9)	0.0095 (9)	0.0046 (9)
O24	0.0358 (9)	0.0216 (8)	0.0277 (8)	0.0004 (7)	0.0123 (7)	0.0083 (7)
C241	0.0246 (12)	0.0198 (12)	0.0477 (16)	−0.0014 (10)	0.0036 (11)	0.0078 (11)
O241	0.0585 (13)	0.0217 (10)	0.0628 (13)	0.0037 (9)	0.0181 (11)	0.0002 (10)
C242	0.0512 (18)	0.0389 (17)	0.073 (2)	0.0054 (15)	0.0228 (16)	0.0313 (17)
C25	0.0278 (12)	0.0217 (12)	0.0303 (12)	0.0021 (9)	0.0143 (10)	0.0090 (10)
C26	0.0249 (12)	0.0330 (14)	0.0540 (17)	0.0003 (11)	0.0153 (12)	0.0174 (13)
O26	0.0329 (10)	0.0285 (10)	0.0522 (12)	−0.0107 (8)	−0.0019 (9)	0.0062 (9)
C261	0.0239 (13)	0.0250 (15)	0.109 (3)	0.0006 (12)	0.0101 (16)	0.0023 (18)
O261	0.0767 (18)	0.0241 (12)	0.191 (3)	−0.0010 (12)	0.073 (2)	0.0165 (17)
C262	0.0379 (17)	0.045 (2)	0.112 (3)	−0.0048 (15)	−0.0069 (19)	−0.020 (2)
O25	0.0213 (7)	0.0208 (8)	0.0259 (8)	−0.0002 (6)	0.0080 (6)	0.0073 (7)

C16	0.0186 (10)	0.0198 (11)	0.0210 (10)	0.0028 (9)	0.0034 (8)	0.0054 (9)
O16	0.0275 (8)	0.0236 (9)	0.0254 (8)	−0.0045 (7)	0.0058 (7)	0.0024 (7)
N31	0.0203 (9)	0.0318 (11)	0.0322 (10)	−0.0026 (9)	0.0063 (8)	−0.0091 (9)
C31	0.0235 (12)	0.0430 (16)	0.0549 (17)	−0.0101 (12)	0.0154 (12)	−0.0140 (14)
C32	0.0247 (12)	0.0291 (13)	0.0343 (13)	0.0048 (10)	0.0028 (10)	−0.0119 (11)
O32	0.0220 (8)	0.0410 (11)	0.0568 (12)	0.0068 (8)	0.0005 (8)	−0.0081 (10)
N33	0.0237 (10)	0.0252 (10)	0.0246 (9)	0.0021 (8)	−0.0020 (8)	−0.0033 (8)
C33	0.0359 (14)	0.0311 (14)	0.0311 (13)	0.0067 (11)	−0.0042 (11)	0.0029 (11)
C34	0.0221 (10)	0.0201 (11)	0.0200 (10)	0.0014 (9)	0.0003 (8)	−0.0051 (9)
N34	0.0239 (9)	0.0248 (10)	0.0182 (9)	−0.0022 (8)	−0.0002 (7)	0.0012 (8)
C341	0.0274 (11)	0.0242 (12)	0.0244 (11)	0.0062 (10)	0.0023 (9)	0.0039 (10)
O341	0.0427 (10)	0.0342 (10)	0.0238 (8)	−0.0014 (9)	0.0049 (7)	−0.0037 (8)
C342	0.0338 (13)	0.0368 (15)	0.0287 (12)	−0.0002 (11)	0.0080 (10)	0.0050 (11)
C35	0.0188 (10)	0.0199 (11)	0.0210 (10)	−0.0015 (9)	0.0026 (8)	−0.0052 (9)
N35	0.0195 (8)	0.0187 (9)	0.0194 (9)	0.0000 (7)	0.0014 (7)	−0.0017 (7)
C351	0.0246 (11)	0.0203 (11)	0.0278 (11)	0.0009 (9)	0.0111 (9)	0.0027 (9)
O351	0.0300 (9)	0.0290 (9)	0.0345 (9)	0.0103 (8)	0.0045 (7)	0.0023 (8)
C352	0.0339 (13)	0.0267 (13)	0.0436 (15)	−0.0003 (11)	0.0104 (12)	−0.0106 (11)
C41	0.0199 (10)	0.0195 (11)	0.0191 (10)	−0.0016 (9)	0.0034 (8)	0.0013 (9)
C42	0.0217 (10)	0.0196 (11)	0.0181 (10)	−0.0007 (9)	0.0047 (8)	0.0017 (9)
O42	0.0218 (7)	0.0267 (9)	0.0213 (7)	−0.0036 (7)	0.0066 (6)	0.0003 (7)
C421	0.0238 (11)	0.0411 (15)	0.0292 (12)	−0.0053 (11)	0.0094 (10)	−0.0156 (12)
O421	0.0231 (8)	0.0505 (12)	0.0469 (11)	0.0029 (9)	0.0061 (8)	−0.0090 (10)
C422	0.0385 (15)	0.060 (2)	0.0354 (14)	−0.0221 (14)	0.0191 (12)	−0.0139 (14)
C43	0.0212 (10)	0.0201 (11)	0.0191 (10)	0.0021 (9)	0.0028 (8)	0.0001 (9)
O43	0.0256 (8)	0.0252 (9)	0.0217 (7)	0.0024 (7)	0.0060 (6)	−0.0028 (7)
C431	0.0357 (13)	0.0224 (12)	0.0242 (11)	−0.0041 (10)	0.0114 (10)	−0.0015 (9)
O431	0.0372 (10)	0.0328 (10)	0.0234 (8)	−0.0057 (8)	0.0064 (7)	−0.0040 (7)
C432	0.0443 (15)	0.0375 (15)	0.0370 (14)	0.0038 (13)	0.0185 (12)	−0.0096 (12)
C44	0.0218 (10)	0.0193 (11)	0.0191 (10)	−0.0022 (9)	0.0007 (8)	−0.0010 (9)
O44	0.0254 (8)	0.0209 (8)	0.0230 (8)	−0.0033 (7)	0.0016 (6)	−0.0020 (6)
C441	0.0327 (13)	0.0197 (12)	0.0287 (12)	−0.0023 (10)	0.0089 (10)	−0.0006 (10)
O441	0.0492 (12)	0.0245 (10)	0.0480 (11)	0.0074 (9)	−0.0038 (9)	−0.0015 (9)
C442	0.0391 (14)	0.0262 (13)	0.0376 (14)	−0.0090 (11)	0.0091 (11)	−0.0087 (11)
C45	0.0188 (10)	0.0213 (11)	0.0211 (10)	−0.0021 (9)	0.0014 (8)	−0.0010 (9)
C46	0.0237 (11)	0.0282 (13)	0.0290 (12)	−0.0030 (10)	0.0067 (9)	0.0000 (10)
O46	0.0324 (9)	0.0314 (10)	0.0257 (8)	−0.0070 (8)	0.0041 (7)	0.0058 (7)
C461	0.0409 (15)	0.0307 (14)	0.0413 (15)	−0.0063 (12)	0.0183 (12)	0.0046 (12)
O461	0.0584 (14)	0.0305 (11)	0.0720 (15)	−0.0144 (11)	0.0011 (12)	−0.0006 (11)
C462	0.057 (2)	0.056 (2)	0.073 (2)	−0.0048 (18)	0.0116 (18)	0.040 (2)
O45	0.0211 (7)	0.0202 (8)	0.0209 (7)	−0.0042 (6)	0.0049 (6)	−0.0013 (6)
C36	0.0235 (11)	0.0236 (12)	0.0223 (11)	−0.0033 (9)	0.0055 (9)	−0.0094 (9)
O36	0.0332 (9)	0.0261 (9)	0.0283 (8)	−0.0067 (7)	0.0097 (7)	−0.0033 (7)

Geometric parameters (Å, º)

N11—C12	1.382 (3)	N31—C32	1.368 (4)
C12—N13	1.386 (3)	C32—N33	1.396 (3)

N13—C14	1.376 (3)	N33—C34	1.374 (3)
C14—C15	1.361 (3)	C34—C35	1.362 (3)
C15—C16	1.449 (3)	C35—C36	1.448 (3)
C16—N11	1.400 (3)	C36—N31	1.389 (3)
N11—C11	1.465 (3)	N31—C31	1.469 (3)
C11—H11A	0.98	C31—H31A	0.98
C11—H11B	0.98	C31—H31B	0.98
C11—H11C	0.98	C31—H31C	0.98
C12—O12	1.220 (3)	C32—O32	1.225 (3)
N13—C13	1.474 (3)	N33—C33	1.467 (3)
C13—H13A	0.98	C33—H33A	0.98
C13—H13B	0.98	C33—H33B	0.98
C13—H13C	0.98	C33—H33C	0.98
C14—N14	1.392 (3)	C34—N34	1.394 (3)
N14—C141	1.389 (3)	N34—C341	1.387 (3)
N14—H14	0.88	N34—H34	0.8799
C141—O141	1.212 (3)	C341—O341	1.219 (3)
C141—C142	1.500 (3)	C341—C342	1.497 (4)
C142—H14A	0.98	C342—H34A	0.98
C142—H14B	0.98	C342—H34B	0.98
C142—H14C	0.98	C342—H34C	0.98
C15—N15	1.427 (3)	C35—N35	1.426 (3)
N15—C151	1.392 (3)	N35—C351	1.394 (3)
N15—C21	1.444 (3)	N35—C41	1.445 (3)
C151—O151	1.213 (3)	C351—O351	1.212 (3)
C151—C152	1.496 (3)	C351—C352	1.505 (3)
C152—H15A	0.98	C352—H35A	0.98
C152—H15B	0.98	C352—H35B	0.98
C152—H15C	0.98	C352—H35C	0.98
C21—O25	1.438 (3)	C41—O45	1.444 (3)
C21—C22	1.526 (3)	C41—C42	1.528 (3)
C21—H21	1.00	C41—H41	1.00
C22—O22	1.441 (3)	C42—O42	1.439 (3)
C22—C23	1.526 (3)	C42—C43	1.520 (3)
C22—H22	1.00	C42—H42	1.00
O22—C221	1.362 (3)	O42—C421	1.362 (3)
C221—O221	1.202 (3)	C421—O421	1.199 (3)
C221—C222	1.491 (4)	C421—C422	1.496 (4)
C222—H22A	0.98	C422—H42A	0.98
C222—H22B	0.98	C422—H42B	0.98
C222—H22C	0.98	C422—H42C	0.98
C23—O23	1.450 (3)	C43—O43	1.440 (3)
C23—C24	1.518 (3)	C43—C44	1.525 (3)
C23—H23	1.00	C43—H43	1.00
O23—C231	1.362 (3)	O43—C431	1.359 (3)
C231—O231	1.196 (3)	C431—O431	1.201 (3)
C231—C232	1.490 (3)	C431—C432	1.495 (4)
C232—H23A	0.98	C432—H43A	0.98

C232—H23B	0.98	C432—H43B	0.98
C232—H23C	0.98	C432—H43C	0.98
C24—O24	1.435 (3)	C44—O44	1.438 (3)
C24—C25	1.523 (3)	C44—C45	1.524 (3)
C24—H24	1.00	C44—H44	1.00
O24—C241	1.353 (3)	O44—C441	1.357 (3)
C241—O241	1.196 (3)	C441—O441	1.205 (3)
C241—C242	1.493 (4)	C441—C442	1.484 (4)
C242—H24A	0.98	C442—H44A	0.98
C242—H24B	0.98	C442—H44B	0.98
C242—H24C	0.98	C442—H44C	0.98
C25—O25	1.427 (3)	C45—O45	1.439 (3)
C25—C26	1.501 (4)	C45—C46	1.512 (3)
C25—H25	1.00	C45—H45	1.00
C26—O26	1.448 (4)	C46—O46	1.448 (3)
C26—H26A	0.99	C46—H46A	0.99
C26—H26B	0.99	C46—H46B	0.99
O26—C261	1.340 (4)	O46—C461	1.351 (3)
C261—O261	1.203 (5)	C461—O461	1.202 (4)
C261—C262	1.485 (6)	C461—C462	1.478 (4)
C262—H26C	0.98	C462—H46C	0.98
C262—H26D	0.98	C462—H46D	0.98
C262—H26E	0.98	C462—H46E	0.98
C16—O16	1.222 (3)	C36—O36	1.222 (3)

C12—N11—C16	125.05 (19)	C32—N31—C36	125.2 (2)
C12—N11—C11	117.59 (18)	C32—N31—C31	115.9 (2)
C16—N11—C11	117.3 (2)	C36—N31—C31	118.9 (2)
N11—C11—H11A	109.5	N31—C31—H31A	109.5
N11—C11—H11B	109.5	N31—C31—H31B	109.5
H11A—C11—H11B	109.5	H31A—C31—H31B	109.5
N11—C11—H11C	109.5	N31—C31—H31C	109.5
H11A—C11—H11C	109.5	H31A—C31—H31C	109.5
H11B—C11—H11C	109.5	H31B—C31—H31C	109.5
O12—C12—N11	122.3 (2)	O32—C32—N31	122.3 (2)
O12—C12—N13	121.6 (2)	O32—C32—N33	120.7 (2)
N11—C12—N13	116.10 (18)	N31—C32—N33	117.1 (2)
C14—N13—C12	122.11 (19)	C34—N33—C32	121.4 (2)
C14—N13—C13	121.10 (19)	C34—N33—C33	122.8 (2)
C12—N13—C13	116.64 (18)	C32—N33—C33	115.8 (2)
N13—C13—H13A	109.5	N33—C33—H33A	109.5
N13—C13—H13B	109.5	N33—C33—H33B	109.5
H13A—C13—H13B	109.5	H33A—C33—H33B	109.5
N13—C13—H13C	109.5	N33—C33—H33C	109.5
H13A—C13—H13C	109.5	H33A—C33—H33C	109.5
H13B—C13—H13C	109.5	H33B—C33—H33C	109.5
C15—C14—N13	120.84 (19)	C35—C34—N33	120.5 (2)
C15—C14—N14	123.00 (19)	C35—C34—N34	122.57 (19)

N13—C14—N14	116.12 (19)	N33—C34—N34	116.8 (2)
C141—N14—C14	121.60 (19)	C341—N34—C34	122.88 (19)
C141—N14—H14	117.4	C341—N34—H34	117.4
C14—N14—H14	116.9	C34—N34—H34	116.0
O141—C141—N14	121.6 (2)	O341—C341—N34	121.2 (2)
O141—C141—C142	124.2 (2)	O341—C341—C342	124.4 (2)
N14—C141—C142	114.2 (2)	N34—C341—C342	114.4 (2)
C141—C142—H14A	109.5	C341—C342—H34A	109.5
C141—C142—H14B	109.5	C341—C342—H34B	109.5
H14A—C142—H14B	109.5	H34A—C342—H34B	109.5
C141—C142—H14C	109.5	C341—C342—H34C	109.5
H14A—C142—H14C	109.5	H34A—C342—H34C	109.5
H14B—C142—H14C	109.5	H34B—C342—H34C	109.5
C14—C15—N15	121.86 (19)	C34—C35—N35	121.69 (19)
C14—C15—C16	120.02 (19)	C34—C35—C36	120.9 (2)
N15—C15—C16	117.79 (19)	N35—C35—C36	117.39 (19)
C151—N15—C15	122.01 (18)	C351—N35—C35	121.52 (18)
C151—N15—C21	116.55 (17)	C351—N35—C41	117.44 (18)
C15—N15—C21	120.98 (17)	C35—N35—C41	120.70 (18)
O151—C151—N15	120.7 (2)	O351—C351—N35	120.9 (2)
O151—C151—C152	123.0 (2)	O351—C351—C352	122.7 (2)
N15—C151—C152	116.3 (2)	N35—C351—C352	116.3 (2)
C151—C152—H15A	109.5	C351—C352—H35A	109.5
C151—C152—H15B	109.5	C351—C352—H35B	109.5
H15A—C152—H15B	109.5	H35A—C352—H35B	109.5
C151—C152—H15C	109.5	C351—C352—H35C	109.5
H15A—C152—H15C	109.5	H35A—C352—H35C	109.5
H15B—C152—H15C	109.5	H35B—C352—H35C	109.5
O25—C21—N15	106.16 (17)	O45—C41—N35	106.34 (16)
O25—C21—C22	108.43 (17)	O45—C41—C42	107.99 (18)
N15—C21—C22	114.49 (17)	N35—C41—C42	114.37 (17)
O25—C21—H21	109.2	O45—C41—H41	109.3
N15—C21—H21	109.2	N35—C41—H41	109.3
C22—C21—H21	109.2	C42—C41—H41	109.3
O22—C22—C21	107.64 (17)	O42—C42—C43	108.81 (16)
O22—C22—C23	108.88 (16)	O42—C42—C41	107.58 (18)
C21—C22—C23	109.58 (17)	C43—C42—C41	110.40 (17)
O22—C22—H22	110.2	O42—C42—H42	110.0
C21—C22—H22	110.2	C43—C42—H42	110.0
C23—C22—H22	110.2	C41—C42—H42	110.0
C221—O22—C22	117.10 (17)	C421—O42—C42	117.39 (19)
O221—C221—O22	122.7 (2)	O421—C421—O42	123.3 (2)
O221—C221—C222	125.7 (2)	O421—C421—C422	125.3 (2)
O22—C221—C222	111.6 (2)	O42—C421—C422	111.4 (2)
C221—C222—H22A	109.5	C421—C422—H42A	109.5
C221—C222—H22B	109.5	C421—C422—H42B	109.5
H22A—C222—H22B	109.5	H42A—C422—H42B	109.5
C221—C222—H22C	109.5	C421—C422—H42C	109.5

H22A—C222—H22C	109.5	H42A—C422—H42C	109.5
H22B—C222—H22C	109.5	H42B—C422—H42C	109.5
O23—C23—C24	109.34 (18)	O43—C43—C42	105.90 (17)
O23—C23—C22	106.22 (16)	O43—C43—C44	110.25 (18)
C24—C23—C22	108.99 (17)	C42—C43—C44	110.08 (17)
O23—C23—H23	110.7	O43—C43—H43	110.2
C24—C23—H23	110.7	C42—C43—H43	110.2
C22—C23—H23	110.7	C44—C43—H43	110.2
C231—O23—C23	117.58 (16)	C431—O43—C43	118.04 (17)
O231—C231—O23	123.7 (2)	O431—C431—O43	123.7 (2)
O231—C231—C232	124.9 (2)	O431—C431—C432	126.1 (2)
O23—C231—C232	111.46 (19)	O43—C431—C432	110.2 (2)
C231—C232—H23A	109.5	C431—C432—H43A	109.5
C231—C232—H23B	109.5	C431—C432—H43B	109.5
H23A—C232—H23B	109.5	H43A—C432—H43B	109.5
C231—C232—H23C	109.5	C431—C432—H43C	109.5
H23A—C232—H23C	109.5	H43A—C432—H43C	109.5
H23B—C232—H23C	109.5	H43B—C432—H43C	109.5
O24—C24—C23	110.38 (18)	O44—C44—C45	107.13 (17)
O24—C24—C25	105.31 (17)	O44—C44—C43	109.95 (17)
C23—C24—C25	111.12 (19)	C45—C44—C43	109.94 (19)
O24—C24—H24	110.0	O44—C44—H44	109.9
C23—C24—H24	110.0	C45—C44—H44	109.9
C25—C24—H24	110.0	C43—C44—H44	109.9
C241—O24—C24	117.9 (2)	C441—O44—C44	117.69 (18)
O241—C241—O24	123.4 (2)	O441—C441—O44	122.9 (2)
O241—C241—C242	127.7 (3)	O441—C441—C442	126.4 (2)
O24—C241—C242	108.9 (2)	O44—C441—C442	110.6 (2)
C241—C242—H24A	109.5	C441—C442—H44A	109.5
C241—C242—H24B	109.5	C441—C442—H44B	109.5
H24A—C242—H24B	109.5	H44A—C442—H44B	109.5
C241—C242—H24C	109.5	C441—C442—H44C	109.5
H24A—C242—H24C	109.5	H44A—C442—H44C	109.5
H24B—C242—H24C	109.5	H44B—C442—H44C	109.5
O25—C25—C26	106.7 (2)	O45—C45—C46	107.73 (18)
O25—C25—C24	109.56 (17)	O45—C45—C44	107.40 (17)
C26—C25—C24	112.0 (2)	C46—C45—C44	113.1 (2)
O25—C25—H25	109.5	O45—C45—H45	109.5
C26—C25—H25	109.5	C46—C45—H45	109.5
C24—C25—H25	109.5	C44—C45—H45	109.5
O26—C26—C25	108.2 (2)	O46—C46—C45	109.44 (18)
O26—C26—H26A	110.1	O46—C46—H46A	109.8
C25—C26—H26A	110.1	C45—C46—H46A	109.8
O26—C26—H26B	110.1	O46—C46—H46B	109.8
C25—C26—H26B	110.1	C45—C46—H46B	109.8
H26A—C26—H26B	108.4	H46A—C46—H46B	108.2
C261—O26—C26	116.7 (3)	C461—O46—C46	117.8 (2)
O261—C261—O26	123.0 (4)	O461—C461—O46	122.9 (3)

O261—C261—C262	125.7 (3)	O461—C461—C462	126.1 (3)
O26—C261—C262	111.2 (3)	O46—C461—C462	111.0 (3)
C261—C262—H26C	109.5	C461—C462—H46C	109.5
C261—C262—H26D	109.5	C461—C462—H46D	109.5
H26C—C262—H26D	109.5	H46C—C462—H46D	109.5
C261—C262—H26E	109.5	C461—C462—H46E	109.5
H26C—C262—H26E	109.5	H46C—C462—H46E	109.5
H26D—C262—H26E	109.5	H46D—C462—H46E	109.5
C25—O25—C21	114.74 (17)	C45—O45—C41	112.60 (15)
O16—C16—N11	120.4 (2)	O36—C36—N31	121.7 (2)
O16—C16—C15	124.4 (2)	O36—C36—C35	123.5 (2)
N11—C16—C15	115.17 (19)	N31—C36—C35	114.8 (2)
C16—N11—C12—O12	176.6 (2)	C36—N31—C32—O32	179.2 (2)
C11—N11—C12—O12	−5.9 (3)	C31—N31—C32—O32	−1.6 (3)
C16—N11—C12—N13	−3.2 (3)	C36—N31—C32—N33	0.1 (3)
C11—N11—C12—N13	174.2 (2)	C31—N31—C32—N33	179.3 (2)
O12—C12—N13—C14	176.5 (2)	O32—C32—N33—C34	179.1 (2)
N11—C12—N13—C14	−3.6 (3)	N31—C32—N33—C34	−1.8 (3)
O12—C12—N13—C13	−7.8 (3)	O32—C32—N33—C33	−3.2 (3)
N11—C12—N13—C13	172.08 (19)	N31—C32—N33—C33	175.9 (2)
C12—N13—C14—C15	9.6 (3)	C32—N33—C34—C35	4.4 (3)
C13—N13—C14—C15	−165.9 (2)	C33—N33—C34—C35	−173.1 (2)
C12—N13—C14—N14	−172.39 (19)	C32—N33—C34—N34	−179.6 (2)
C13—N13—C14—N14	12.1 (3)	C33—N33—C34—N34	2.9 (3)
C15—C14—N14—C141	−116.1 (2)	C35—C34—N34—C341	−120.4 (2)
N13—C14—N14—C141	65.9 (3)	N33—C34—N34—C341	63.6 (3)
C14—N14—C141—O141	−3.5 (3)	C34—N34—C341—O341	3.0 (3)
C14—N14—C141—C142	177.8 (2)	C34—N34—C341—C342	−175.8 (2)
N13—C14—C15—N15	178.24 (19)	N33—C34—C35—N35	173.9 (2)
N14—C14—C15—N15	0.3 (3)	N34—C34—C35—N35	−1.9 (3)
N13—C14—C15—C16	−8.6 (3)	N33—C34—C35—C36	−5.2 (3)
N14—C14—C15—C16	173.5 (2)	N34—C34—C35—C36	179.0 (2)
C14—C15—N15—C151	95.6 (3)	C34—C35—N35—C351	111.6 (2)
C16—C15—N15—C151	−77.7 (3)	C36—C35—N35—C351	−69.3 (3)
C14—C15—N15—C21	−76.4 (3)	C34—C35—N35—C41	−75.3 (3)
C16—C15—N15—C21	110.3 (2)	C36—C35—N35—C41	103.8 (2)
C15—N15—C151—O151	−176.9 (2)	C35—N35—C351—O351	167.5 (2)
C21—N15—C151—O151	−4.7 (3)	C41—N35—C351—O351	−5.8 (3)
C15—N15—C151—C152	3.0 (3)	C35—N35—C351—C352	−15.9 (3)
C21—N15—C151—C152	175.3 (2)	C41—N35—C351—C352	170.74 (19)
C151—N15—C21—O25	−88.4 (2)	C351—N35—C41—O45	−95.2 (2)
C15—N15—C21—O25	83.9 (2)	C35—N35—C41—O45	91.4 (2)
C15—N15—C21—C22	−35.7 (3)	C35—N35—C41—C42	−27.7 (3)
C22—C21—N15—C151	151.98 (19)	C42—C41—N35—C351	145.74 (19)
O25—C21—C22—O22	176.96 (15)	O45—C41—C42—O42	175.45 (15)
N15—C21—C22—O22	−64.7 (2)	N35—C41—C42—O42	−66.4 (2)
O25—C21—C22—C23	58.7 (2)	O45—C41—C42—C43	56.9 (2)

N15—C21—C22—C23	177.02 (18)	N35—C41—C42—C43	175.01 (17)
C21—C22—O22—C221	129.62 (18)	C43—C42—O42—C421	−107.5 (2)
C23—C22—O22—C221	−111.7 (2)	C41—C42—O42—C421	132.84 (19)
C22—O22—C221—O221	1.3 (3)	C42—O42—C421—O421	−1.5 (3)
C22—O22—C221—C222	−178.03 (19)	C42—O42—C421—C422	−179.14 (19)
O22—C22—C23—O23	67.9 (2)	O42—C42—C43—O43	69.3 (2)
C21—C22—C23—O23	−174.63 (16)	C41—C42—C43—O43	−172.81 (16)
O22—C22—C23—C24	−174.41 (17)	O42—C42—C43—C44	−171.48 (17)
C21—C22—C23—C24	−56.9 (2)	C41—C42—C43—C44	−53.6 (2)
C24—C23—O23—C231	91.9 (2)	C42—C43—O43—C431	−146.41 (19)
C22—C23—O23—C231	−150.68 (18)	C44—C43—O43—C431	94.5 (2)
C23—O23—C231—O231	4.2 (3)	C43—O43—C431—O431	−3.4 (3)
C23—O23—C231—C232	−175.15 (19)	C43—O43—C431—C432	177.3 (2)
O23—C23—C24—O24	−72.9 (2)	O43—C43—C44—O44	−71.0 (2)
C22—C23—C24—O24	171.37 (17)	C42—C43—C44—O44	172.54 (17)
O23—C23—C24—C25	170.63 (17)	O43—C43—C44—C45	171.30 (16)
C22—C23—C24—C25	54.9 (2)	C42—C43—C44—C45	54.8 (2)
C23—C24—O24—C241	104.9 (2)	C45—C44—O44—C441	−140.4 (2)
C25—C24—O24—C241	−135.0 (2)	C43—C44—O44—C441	100.1 (2)
C24—O24—C241—O241	−2.7 (4)	C44—O44—C441—O441	4.7 (3)
C24—O24—C241—C242	178.8 (2)	C44—O44—C441—C442	−174.13 (19)
O24—C24—C25—O25	−174.16 (18)	O44—C44—C45—O45	−178.58 (16)
C23—C24—C25—O25	−54.6 (2)	C43—C44—C45—O45	−59.1 (2)
O24—C24—C25—C26	67.7 (2)	O44—C44—C45—C46	62.7 (2)
C23—C24—C25—C26	−172.82 (19)	C43—C44—C45—C46	−177.84 (18)
O25—C25—C26—O26	−58.6 (2)	O45—C45—C46—O46	−69.2 (2)
C24—C25—C26—O26	61.3 (3)	C44—C45—C46—O46	49.3 (3)
C25—C26—O26—C261	−136.4 (2)	C45—C46—O46—C461	−124.2 (2)
C26—O26—C261—O261	−0.2 (4)	C46—O46—C461—O461	0.3 (4)
C26—O26—C261—C262	−179.4 (2)	C46—O46—C461—C462	−179.4 (2)
C26—C25—O25—C21	−179.44 (19)	C46—C45—O45—C41	−172.08 (18)
C24—C25—O25—C21	59.1 (2)	C44—C45—O45—C41	65.8 (2)
N15—C21—O25—C25	175.11 (17)	N35—C41—O45—C45	172.14 (16)
C22—C21—O25—C25	−61.4 (2)	C42—C41—O45—C45	−64.7 (2)
C12—N11—C16—O16	−179.0 (2)	C32—N31—C36—O36	177.5 (2)
C11—N11—C16—O16	3.5 (3)	C31—N31—C36—O36	−1.7 (3)
C12—N11—C16—C15	3.9 (3)	C32—N31—C36—C35	−0.8 (3)
C11—N11—C16—C15	−173.5 (2)	C31—N31—C36—C35	−180.0 (2)
C14—C15—C16—O16	−174.9 (2)	C34—C35—C36—O36	−174.9 (2)
N15—C15—C16—O16	−1.4 (3)	N35—C35—C36—O36	6.0 (3)
C14—C15—C16—N11	2.1 (3)	C34—C35—C36—N31	3.4 (3)
N15—C15—C16—N11	175.51 (18)	N35—C35—C36—N31	−175.75 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N14—H14···O25	0.88	2.00	2.787 (3)	148
N34—H34···O45	0.88	2.06	2.867 (2)	152