



Crystal structure of dioxidobis(pentane-2,4-dionato- κ^2O,O')[1-phenyl-3-(pyridin-4-yl)propane- κN]uranium(VI)

Takeshi Kawasaki^a and Takafumi Kitazawa^{a,b*}

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^aDepartment of Chemistry, Faculty of Science, Toho University, 2-2-1 Miyama, Funabashi, Chiba 274-8510, Japan, and
^bResearch Center for Materials with Integrated Properties, Toho University, Miyama, Funabashi, Chiba 274-8510, Japan.
*Correspondence e-mail: kitazawa@chem.sci.toho-u.ac.jp

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In the title compound, $[\text{UO}_2(\text{C}_5\text{H}_7\text{O}_2)_2(\text{C}_{14}\text{H}_{15}\text{N})]$, the uranyl(VI) unit ($[\text{O}=\text{U}=\text{O}]^{2+}$) is coordinated to two acetylacetonate (acac) anions and one 1-phenyl-3-(pyridin-4-yl)propane (ppp) molecule. The geometry around the U atom is UNO_6 pentagonal-bipyramidal; two uranyl(VI) O atoms are located at the axial positions, whereas four O atoms from two chelating bidentate acac ligands and one N atom of a ppp ligand form the equatorial plane.

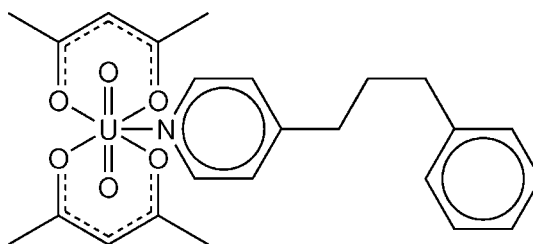
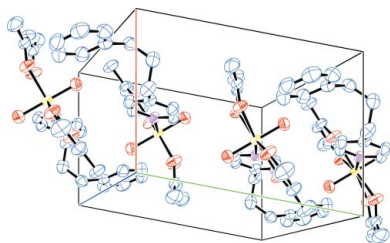
Keywords: crystal structure; pentane-2,4-dionate; 1-phenyl-3-(pyridin-4-yl)propane; uranium(VI) complex

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1. Chemical context

The structural properties of uranyl(VI) complexes are interesting from the viewpoint of nuclear fuels reprocessing and actinide waste treatment. In most commercial reprocessing plants, spent nuclear fuels are treated by the Purex method, in which uranium and plutonium are extracted from a nitric acid solution of spent nuclear fuels using tributyl-phosphate/*n*-dodecane. Uranium in the nitric acid solution exists as uranyl(VI) ($[\text{O}=\text{U}=\text{O}]^{2+}$) complexes. However, the Purex method has a few problems; for example, as the processing takes place on a relatively large scale, a large amount of extractant is necessary (Ikeda *et al.*, 2004; Suzuki *et al.*, 2012). Attempts to find other suitable coordinating ligands are therefore being undertaken. A number of structural studies of uranyl(VI) β -diketonate complexes have been reported by ourselves and others (Alcock *et al.*, 1984, 1987; Huuskonen *et al.*, 2007; Kannan *et al.*, 2001; Kawasaki & Kitazawa, 2008; Kawasaki *et al.*, 2010; Sidorenko *et al.*, 2009; Tahir *et al.*, 2006; Takao & Ikeda, 2008). In particular, acetylacetonate (acac), is the simplest β -diketonate ligand and an important coordinating ligand for uranium.



We report herein the synthesis and crystal structure of a novel uranyl(VI) acetylacetonate (acac) complex with the pyridine-based ligand ppp [ppp = 1-phenyl-3-(pyridin-4-yl)propane] (Seth, 2014), namely, $[\text{UO}_2(\text{acac})_2(\text{ppp})]$.

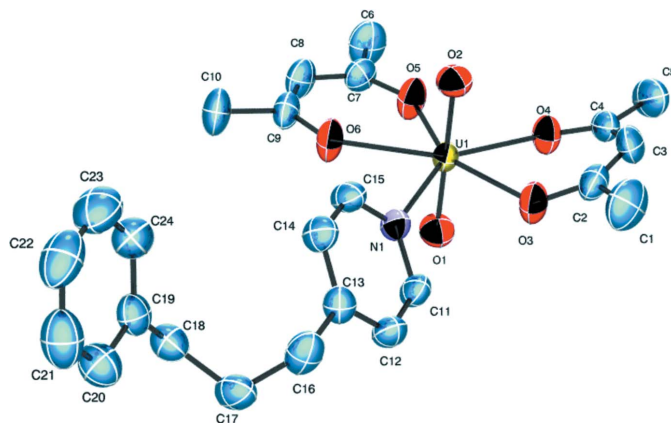


Figure 1
The molecular structure of $[\text{UO}_2(\text{acac})_2(\text{ppp})]$. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

2. Structural commentary

The title compound of formula $[\text{UO}_2(\text{C}_5\text{H}_7\text{O}_2)_2(\text{C}_{14}\text{H}_{15}\text{N})]$, is constructed from one uranyl(VI) ($[\text{O}=\text{U}=\text{O}]^{2+}$) unit, two acetylacetonate anions and one molecule of ppp (Fig. 1). The uranium(VI) atom exhibits a pentagonal-bipyramidal coordination geometry: two uranyl(VI) oxygen atoms (O1 and O2) are located in the axial positions and four oxygen atoms (O3, O4, O5 and O6) from two chelating bidentate acac ions, together with one nitrogen atom (N1) of the ppp molecule, form the equatorial plane. The bond lengths around U1 (Table 1) decrease in the order $\text{U}-\text{N} > \text{U}-\text{O}_{\text{acac}} > \text{U}=\text{O}$. The dihedral angle between the pyridine ring of the ppp molecule and the equatorial plane around U1 is $49.43(12)^\circ$. The above structural properties are similar to those in the majority of previously characterised $[\text{UO}_2(\text{acac})_2L]$ (L = pyridine derivative ligand) complexes (Alcock *et al.*, 1984; Kawasaki & Kitazawa, 2008; Kawasaki *et al.*, 2010). The conformation of the ppp molecule is GG' (Fig. 2). The dihedral angle between the pyridine ring and the phenyl ring in the ppp molecule is $26.96(13)^\circ$.

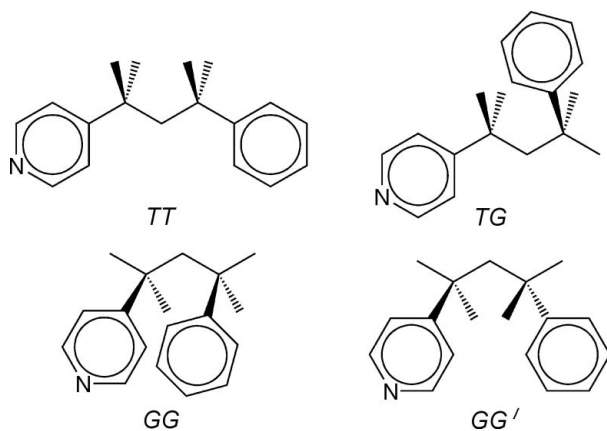


Figure 2
The four possible conformations that the ppp ligand can form (based on Carlucci *et al.*, 2002). In the title compound, the conformation is GG' .

Table 1
Selected geometric parameters (\AA , $^\circ$).

U1—O1	1.773 (3)	U1—O5	2.348 (2)
U1—O2	1.777 (3)	U1—O6	2.354 (2)
U1—O3	2.330 (2)	U1—N1	2.610 (3)
U1—O4	2.360 (2)		
O1—U1—O2	179.19 (11)	O1—U1—N1	86.45 (11)
O3—U1—O4	70.88 (9)	O2—U1—N1	92.74 (11)
O3—U1—O6	138.83 (9)	O3—U1—N1	69.37 (9)
O4—U1—O5	79.13 (9)	O6—U1—N1	70.15 (9)
O5—U1—O6	70.91 (9)		

3. Supramolecular features

A packing diagram of title complex is shown in Fig. 3. The molecules are stacked along the b axis, held together by van der Waals' interactions only. Significant intermolecular $\pi-\pi$ and $\text{C}-\text{H}\cdots\pi$ interactions are not found.

4. Synthesis and crystallization

The title complex was synthesized according to literature procedures (Alcock *et al.*, 1984, 1987; Kawasaki & Kitazawa, 2008; Kawasaki *et al.*, 2010). To 10 ml of a methanolic solution containing 1 mmol $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was added 3 mmol of acetylacetonate and 3 mmol of 1-phenyl-3-(pyridin-4-yl)propane in 5 ml MeOH. The solvent evaporated slowly at room temperature for a few days and orange crystal were obtained.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were placed at calculated positions [$\text{C}(\text{CH})-\text{H} = 0.93$, $\text{C}(\text{CH}_2)-\text{H} = 0.97$ and $\text{C}(\text{CH}_3)-\text{H} = 0.96\text{\AA}$] and allowed to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{CH}, \text{CH}_2)$ and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{CH}_3)$.

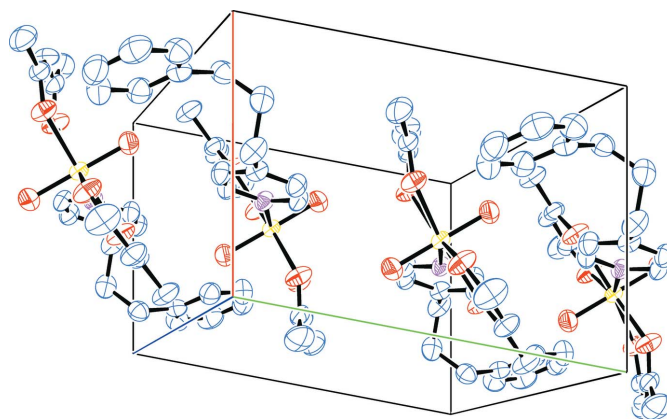


Figure 3
A packing diagram of the title complex (red line: a axis; green line: b axis; blue line: c axis). Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

Table 2
Experimental details.

Crystal data	
Chemical formula	[UO ₂ (C ₅ H ₇ O ₂) ₂ (C ₁₄ H ₁₅ N)]
<i>M_r</i>	665.51
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	297
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.2100 (16), 11.530 (2), 14.516 (3)
α , β , γ (°)	108.67 (3), 98.50 (3), 100.81 (3)
<i>V</i> (Å ³)	1246.4 (4)
<i>Z</i>	2
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	6.55
Crystal size (mm)	0.47 × 0.29 × 0.26
Data collection	
Diffractometer	Bruker SMART APEXII
Absorption correction	Analytical (<i>XPREP</i> ; Bruker, 2007)
<i>T</i> _{min} , <i>T</i> _{max}	0.149, 0.281
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	9353, 6948, 6026
<i>R</i> _{int}	0.015
(sin θ / λ) _{max} (Å ⁻¹)	0.722
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.027, 0.056, 0.99
No. of reflections	6948
No. of parameters	293
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.88, -0.64

Computer programs: *APEX2*, *SAINT* and *XSCANS* (Bruker, 2007), *SHELXS97*, *SHELXL97* and *SHELXTL* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009).

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Crystal structure of dioxidobis(pentane-2,4-dionato- κ^2O,O')[1-phenyl-3-(pyridin-4-yl)propane- κN]uranium(VI)

Takeshi Kawasaki and Takafumi Kitazawa

Computing details

Data collection: *APEX2* (Bruker, 2007) and *XSCANS* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007) and *XSCANS* (Bruker, 2007); data reduction: *APEX2* (Bruker, 2007) and *SAINTE* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

Dioxidobis(pentane-2,4-dionato- κ^2O,O')[1-phenyl-3-(pyridin-4-yl)propane- κN]uranium(VI)

Crystal data

[U(C₅H₇O₂)₂O₂(C₁₄H₁₅N)]

$M_r = 665.51$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 8.2100$ (16) Å

$b = 11.530$ (2) Å

$c = 14.516$ (3) Å

$\alpha = 108.67$ (3)°

$\beta = 98.50$ (3)°

$\gamma = 100.81$ (3)°

$V = 1246.4$ (4) Å³

$Z = 2$

$F(000) = 640$

$D_x = 1.773$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4311 reflections

$\theta = 2.6$ – 28.5 °

$\mu = 6.55$ mm⁻¹

$T = 297$ K

Block, orange

$0.47 \times 0.29 \times 0.26$ mm

Data collection

Bruker SMART APEXII
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.333 pixels mm⁻¹

ω scans

Absorption correction: analytical
(XPREP; Bruker, 2007)

$T_{\min} = 0.149$, $T_{\max} = 0.281$

9353 measured reflections

6948 independent reflections

6026 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.015$

$\theta_{\max} = 30.9$ °, $\theta_{\min} = 1.9$ °

$h = -11 \rightarrow 9$

$k = -16 \rightarrow 15$

$l = -14 \rightarrow 20$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.027$

$wR(F^2) = 0.056$

$S = 0.99$

6948 reflections

293 parameters

0 restraints

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

$$w = 1/[\sigma^2(F_o^2) + (0.0257P)^2]$$

where $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\max} = 0.003$$

$$\Delta\rho_{\max} = 0.88 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.64 \text{ e } \text{\AA}^{-3}$$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
U1	0.488854 (15)	0.690915 (11)	0.372080 (8)	0.03519 (4)
O1	0.3391 (3)	0.5504 (2)	0.3581 (2)	0.0535 (6)
O2	0.6386 (3)	0.8327 (2)	0.38786 (19)	0.0525 (6)
O3	0.6977 (3)	0.6473 (3)	0.47398 (18)	0.0566 (7)
O4	0.6471 (3)	0.5741 (2)	0.27211 (18)	0.0521 (6)
O5	0.3569 (4)	0.6784 (3)	0.21310 (17)	0.0579 (7)
O6	0.2846 (3)	0.8105 (3)	0.38825 (18)	0.0567 (7)
N1	0.4613 (4)	0.7730 (3)	0.55740 (19)	0.0397 (6)
C1	0.9705 (6)	0.6794 (5)	0.5706 (3)	0.0810 (14)
H1A	0.9878	0.7680	0.6071	0.121*
H1B	1.0774	0.6618	0.5601	0.121*
H1C	0.9231	0.6316	0.6080	0.121*
C2	0.8488 (5)	0.6426 (3)	0.4705 (3)	0.0493 (9)
C3	0.9047 (5)	0.6043 (4)	0.3827 (3)	0.0573 (10)
H3	1.0188	0.6043	0.3872	0.069*
C4	0.8004 (5)	0.5661 (3)	0.2886 (3)	0.0497 (9)
C5	0.8646 (7)	0.5049 (5)	0.1981 (4)	0.0757 (14)
H5A	0.7867	0.4249	0.1584	0.114*
H5B	0.9744	0.4920	0.2186	0.114*
H5C	0.8738	0.5586	0.1595	0.114*
C6	0.2066 (7)	0.6847 (5)	0.0645 (3)	0.0796 (15)
H6A	0.3049	0.7172	0.0433	0.119*
H6B	0.1140	0.7170	0.0434	0.119*
H6C	0.1752	0.5940	0.0354	0.119*
C7	0.2466 (5)	0.7251 (4)	0.1764 (3)	0.0496 (9)
C8	0.1637 (5)	0.8067 (4)	0.2321 (3)	0.0560 (10)
H8	0.0878	0.8375	0.1980	0.067*
C9	0.1856 (4)	0.8459 (3)	0.3350 (3)	0.0473 (8)
C10	0.0891 (6)	0.9354 (4)	0.3879 (3)	0.0670 (12)
H10A	-0.0194	0.8881	0.3894	0.101*

H10B	0.0723	0.9919	0.3531	0.101*
H10C	0.1526	0.9833	0.4549	0.101*
C11	0.4545 (5)	0.6962 (3)	0.6101 (3)	0.0449 (8)
H11	0.4583	0.6129	0.5791	0.054*
C12	0.4422 (5)	0.7361 (4)	0.7083 (3)	0.0479 (8)
H12	0.4371	0.6798	0.7421	0.058*
C13	0.4373 (4)	0.8594 (3)	0.7568 (2)	0.0434 (8)
C14	0.4484 (5)	0.9381 (3)	0.7028 (3)	0.0476 (8)
H14	0.4501	1.0227	0.7333	0.057*
C15	0.4570 (5)	0.8929 (3)	0.6042 (3)	0.0470 (8)
H15	0.4599	0.9474	0.5688	0.056*
C16	0.4135 (6)	0.9028 (4)	0.8628 (3)	0.0576 (10)
H16A	0.4701	0.9919	0.8956	0.069*
H16B	0.4675	0.8568	0.8984	0.069*
C17	0.2268 (6)	0.8831 (4)	0.8692 (3)	0.0586 (10)
H17A	0.1655	0.7980	0.8253	0.070*
H17B	0.2195	0.8901	0.9367	0.070*
C18	0.1404 (5)	0.9765 (4)	0.8412 (3)	0.0536 (9)
H18A	0.1565	0.9747	0.7759	0.064*
H18B	0.0192	0.9492	0.8361	0.064*
C19	0.2056 (5)	1.1116 (4)	0.9144 (3)	0.0488 (8)
C20	0.1846 (6)	1.1420 (4)	1.0114 (3)	0.0621 (11)
H20	0.1317	1.0788	1.0318	0.075*
C21	0.2424 (6)	1.2672 (5)	1.0796 (3)	0.0750 (14)
H21	0.2255	1.2868	1.1442	0.090*
C22	0.3239 (6)	1.3604 (5)	1.0503 (4)	0.0772 (14)
H22	0.3654	1.4431	1.0952	0.093*
C23	0.3428 (7)	1.3292 (5)	0.9534 (4)	0.0796 (14)
H23	0.3952	1.3923	0.9328	0.096*
C24	0.2867 (6)	1.2082 (4)	0.8869 (3)	0.0627 (11)
H24	0.3030	1.1900	0.8222	0.075*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
U1	0.03593 (7)	0.03608 (7)	0.03601 (7)	0.01545 (5)	0.00830 (5)	0.01242 (5)
O1	0.0494 (15)	0.0444 (15)	0.0616 (16)	0.0058 (12)	0.0129 (12)	0.0154 (12)
O2	0.0563 (16)	0.0411 (14)	0.0611 (16)	0.0096 (12)	0.0197 (13)	0.0182 (12)
O3	0.0536 (16)	0.078 (2)	0.0503 (14)	0.0391 (15)	0.0132 (12)	0.0262 (14)
O4	0.0537 (16)	0.0575 (16)	0.0478 (14)	0.0285 (13)	0.0151 (12)	0.0124 (12)
O5	0.0669 (18)	0.0722 (19)	0.0417 (13)	0.0421 (15)	0.0081 (12)	0.0175 (13)
O6	0.0607 (17)	0.0762 (19)	0.0453 (13)	0.0456 (15)	0.0134 (12)	0.0208 (13)
N1	0.0469 (16)	0.0383 (15)	0.0385 (14)	0.0166 (13)	0.0148 (12)	0.0141 (12)
C1	0.067 (3)	0.093 (4)	0.075 (3)	0.026 (3)	-0.012 (2)	0.029 (3)
C2	0.046 (2)	0.043 (2)	0.062 (2)	0.0183 (16)	0.0049 (17)	0.0212 (17)
C3	0.0358 (19)	0.065 (3)	0.079 (3)	0.0201 (18)	0.0187 (19)	0.030 (2)
C4	0.052 (2)	0.044 (2)	0.068 (2)	0.0230 (17)	0.0303 (19)	0.0257 (18)
C5	0.093 (4)	0.072 (3)	0.086 (3)	0.043 (3)	0.054 (3)	0.032 (3)

C6	0.092 (4)	0.107 (4)	0.046 (2)	0.048 (3)	0.004 (2)	0.029 (2)
C7	0.048 (2)	0.056 (2)	0.0462 (19)	0.0167 (18)	0.0018 (16)	0.0232 (17)
C8	0.055 (2)	0.064 (3)	0.052 (2)	0.030 (2)	0.0006 (18)	0.0204 (19)
C9	0.0377 (18)	0.043 (2)	0.058 (2)	0.0173 (15)	0.0035 (16)	0.0130 (17)
C10	0.062 (3)	0.068 (3)	0.071 (3)	0.040 (2)	0.011 (2)	0.014 (2)
C11	0.057 (2)	0.0391 (19)	0.0473 (19)	0.0229 (16)	0.0172 (17)	0.0184 (15)
C12	0.060 (2)	0.049 (2)	0.0470 (19)	0.0214 (18)	0.0182 (17)	0.0262 (17)
C13	0.0427 (19)	0.048 (2)	0.0362 (16)	0.0154 (16)	0.0057 (14)	0.0101 (15)
C14	0.060 (2)	0.0379 (19)	0.0438 (18)	0.0158 (17)	0.0158 (17)	0.0096 (15)
C15	0.062 (2)	0.0379 (19)	0.0488 (19)	0.0157 (17)	0.0215 (17)	0.0192 (16)
C16	0.076 (3)	0.066 (3)	0.0361 (18)	0.034 (2)	0.0081 (18)	0.0178 (18)
C17	0.079 (3)	0.055 (2)	0.051 (2)	0.021 (2)	0.029 (2)	0.0227 (19)
C18	0.052 (2)	0.057 (2)	0.053 (2)	0.0113 (18)	0.0174 (18)	0.0191 (19)
C19	0.0391 (19)	0.058 (2)	0.048 (2)	0.0161 (17)	0.0076 (16)	0.0160 (18)
C20	0.063 (3)	0.065 (3)	0.056 (2)	0.015 (2)	0.018 (2)	0.017 (2)
C21	0.073 (3)	0.084 (4)	0.058 (3)	0.033 (3)	0.011 (2)	0.007 (2)
C22	0.069 (3)	0.051 (3)	0.094 (4)	0.018 (2)	-0.001 (3)	0.009 (3)
C23	0.081 (3)	0.053 (3)	0.103 (4)	0.013 (2)	0.019 (3)	0.029 (3)
C24	0.064 (3)	0.063 (3)	0.069 (3)	0.021 (2)	0.019 (2)	0.030 (2)

Geometric parameters (Å, °)

U1—O1	1.773 (3)	C10—H10A	0.9600
U1—O2	1.777 (3)	C10—H10B	0.9600
U1—O3	2.330 (2)	C10—H10C	0.9600
U1—O4	2.360 (2)	C11—C12	1.376 (5)
U1—O5	2.348 (2)	C11—H11	0.9300
U1—O6	2.354 (2)	C12—C13	1.382 (5)
U1—N1	2.610 (3)	C12—H12	0.9300
O3—C2	1.260 (4)	C13—C14	1.375 (5)
O4—C4	1.272 (4)	C13—C16	1.512 (5)
O5—C7	1.271 (4)	C14—C15	1.375 (5)
O6—C9	1.251 (4)	C14—H14	0.9300
N1—C11	1.342 (4)	C15—H15	0.9300
N1—C15	1.342 (4)	C16—C17	1.528 (6)
C1—C2	1.519 (5)	C16—H16A	0.9700
C1—H1A	0.9600	C16—H16B	0.9700
C1—H1B	0.9600	C17—C18	1.519 (5)
C1—H1C	0.9600	C17—H17A	0.9700
C2—C3	1.384 (6)	C17—H17B	0.9700
C3—C4	1.386 (6)	C18—C19	1.517 (6)
C3—H3	0.9300	C18—H18A	0.9700
C4—C5	1.501 (5)	C18—H18B	0.9700
C5—H5A	0.9600	C19—C20	1.382 (5)
C5—H5B	0.9600	C19—C24	1.389 (6)
C5—H5C	0.9600	C20—C21	1.407 (6)
C6—C7	1.505 (5)	C20—H20	0.9300
C6—H6A	0.9600	C21—C22	1.375 (7)

C6—H6B	0.9600	C21—H21	0.9300
C6—H6C	0.9600	C22—C23	1.375 (7)
C7—C8	1.385 (5)	C22—H22	0.9300
C8—C9	1.388 (5)	C23—C24	1.362 (6)
C8—H8	0.9300	C23—H23	0.9300
C9—C10	1.504 (5)	C24—H24	0.9300
O1—U1—O2	179.19 (11)	O6—C9—C8	122.9 (3)
O1—U1—O3	91.86 (12)	O6—C9—C10	116.8 (3)
O1—U1—O4	91.38 (11)	C8—C9—C10	120.3 (3)
O1—U1—O5	89.82 (12)	C9—C10—H10A	109.5
O1—U1—O6	92.85 (12)	C9—C10—H10B	109.5
O2—U1—O3	87.91 (12)	H10A—C10—H10B	109.5
O2—U1—O4	89.27 (11)	C9—C10—H10C	109.5
O2—U1—O5	90.77 (12)	H10A—C10—H10C	109.5
O2—U1—O6	86.82 (11)	H10B—C10—H10C	109.5
O3—U1—O4	70.88 (9)	N1—C11—C12	122.5 (3)
O3—U1—O5	149.99 (9)	N1—C11—H11	118.8
O3—U1—O6	138.83 (9)	C12—C11—H11	118.8
O4—U1—O5	79.13 (9)	C11—C12—C13	120.3 (3)
O4—U1—O6	149.71 (9)	C11—C12—H12	119.8
O5—U1—O6	70.91 (9)	C13—C12—H12	119.8
O1—U1—N1	86.45 (11)	C14—C13—C12	116.7 (3)
O2—U1—N1	92.74 (11)	C14—C13—C16	122.3 (3)
O3—U1—N1	69.37 (9)	C12—C13—C16	121.0 (3)
O4—U1—N1	140.08 (8)	C15—C14—C13	120.7 (3)
O5—U1—N1	140.62 (9)	C15—C14—H14	119.6
O6—U1—N1	70.15 (9)	C13—C14—H14	119.6
C2—O3—U1	132.2 (2)	N1—C15—C14	122.3 (3)
C4—O4—U1	132.7 (2)	N1—C15—H15	118.9
C7—O5—U1	137.4 (2)	C14—C15—H15	118.9
C9—O6—U1	139.2 (2)	C13—C16—C17	113.2 (3)
C11—N1—C15	117.5 (3)	C13—C16—H16A	108.9
C11—N1—U1	120.6 (2)	C17—C16—H16A	108.9
C15—N1—U1	121.9 (2)	C13—C16—H16B	108.9
C2—C1—H1A	109.5	C17—C16—H16B	108.9
C2—C1—H1B	109.5	H16A—C16—H16B	107.7
H1A—C1—H1B	109.5	C18—C17—C16	113.9 (3)
C2—C1—H1C	109.5	C18—C17—H17A	108.8
H1A—C1—H1C	109.5	C16—C17—H17A	108.8
H1B—C1—H1C	109.5	C18—C17—H17B	108.8
O3—C2—C3	123.9 (4)	C16—C17—H17B	108.8
O3—C2—C1	115.6 (4)	H17A—C17—H17B	107.7
C3—C2—C1	120.5 (4)	C19—C18—C17	114.2 (3)
C2—C3—C4	123.8 (3)	C19—C18—H18A	108.7
C2—C3—H3	118.1	C17—C18—H18A	108.7
C4—C3—H3	118.1	C19—C18—H18B	108.7
O4—C4—C3	124.5 (3)	C17—C18—H18B	108.7

O4—C4—C5	115.8 (4)	H18A—C18—H18B	107.6
C3—C4—C5	119.7 (4)	C20—C19—C24	117.9 (4)
C4—C5—H5A	109.5	C20—C19—C18	120.4 (4)
C4—C5—H5B	109.5	C24—C19—C18	121.7 (4)
H5A—C5—H5B	109.5	C19—C20—C21	120.9 (4)
C4—C5—H5C	109.5	C19—C20—H20	119.5
H5A—C5—H5C	109.5	C21—C20—H20	119.5
H5B—C5—H5C	109.5	C22—C21—C20	119.7 (5)
C7—C6—H6A	109.5	C22—C21—H21	120.1
C7—C6—H6B	109.5	C20—C21—H21	120.1
H6A—C6—H6B	109.5	C21—C22—C23	118.8 (5)
C7—C6—H6C	109.5	C21—C22—H22	120.6
H6A—C6—H6C	109.5	C23—C22—H22	120.6
H6B—C6—H6C	109.5	C24—C23—C22	121.7 (5)
O5—C7—C8	124.5 (3)	C24—C23—H23	119.1
O5—C7—C6	115.5 (4)	C22—C23—H23	119.1
C8—C7—C6	120.0 (3)	C23—C24—C19	120.8 (4)
C7—C8—C9	124.8 (3)	C23—C24—H24	119.6
C7—C8—H8	117.6	C19—C24—H24	119.6
C9—C8—H8	117.6		
