Supporting Information for:

Identification of the U(V) Complex $(C_5Me_5)_2U^{V}I(=NSiMe_3)$ in the Reaction of $(C_5Me_5)_2U^{III}I(THF)$ with N_3SiMe_3

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Experimental Details

Caution! ^{238}U is an alpha emitter with a half-life of approximately $4.5x10^9$ years. Samples should only be handled in laboratories equipped with appropriate radiological safety controls and radiation-counting equipment.

All syntheses and manipulations described below were conducted under argon with rigorous exclusion of air and water using standard glovebox and vacuum line techniques. Solvents were sparged with UHP argon and dried by passage through columns containing Q-5 and molecular sieves prior to use. Deuterated NMR solvents were dried over NaK alloy, degassed by three freeze-pump-thaw cycles, and vacuum transferred before use. ^1H spectra were recorded on a CRYO500 MHz at 298 K and referenced to residual protio-solvent resonances. $(C_5Me_5)_2U^{III}I(THF)$[1,2] was prepared as previously described. N_3SiMe_3 was degassed by three freeze-pump-thaw cycles before use. PhN=NPh was sublimed prior to use. N_3Ad was used as received.

Crystallization of $(C_5Me_5)_2U^{V}I(=NSiMe_3)$, 1. $(C_5Me_5)_2U^{III}I(THF)$ (50 mg, 0.071 mmol) was dissolved in toluene (2 mL) to form a dark green solution. N_3SiMe_3 (9 mg, 0.078 mmol) was dissolved in toluene (0.5 mL) and added to the stirred solution. The solution became brown. The solution was stirred for two hours and then dried under vacuum. Hexane (5 mL) was added to form a brown suspension. The mixture was centrifuged to remove the solids and dried under vacuum. ^1H NMR and IR spectroscopy showed the presence of a new paramagnetic compound. Brown X-ray quality crystals of **1** were grown from a concentrated hexane solution at −35 °C.

Reaction of $(C_5Me_5)_2U^{III}I(THF)$ and N_3SiMe_3. $(C_5Me_5)_2U^{III}I(THF)$ (100 mg, 0.141 mmol) was dissolved in toluene (5 mL) to form a dark green solution. N_3SiMe_3 (18 mg, 0.16 mmol) was dissolved in toluene (0.5 mL) and added to the stirred solution by pipet. The solution

became dark brown immediately. The solution was stirred for 30 minutes and then dried under vacuum. Hexane (8 mL) was added and the solution was stirred for one hour. Red solids were removed by filtration and the solution was dried to yield brown solids (68 mg, 67%). The ^{1}H NMR spectrum in C_6D_6 showed a ratio of roughly 3:4 of $(C_5Me_5)_2U^{VI}(=NSiMe_3)_2$ (δ 4.91 ppm, C_5Me_5)[3] to $(C_5Me_5)_2U^{IV}I_2$ (δ 17.99 ppm, C_5Me_5).[4]

Reaction of $(C_5Me_5)_2U^{III}I(THF)$ and N_3Ad. As above, $(C_5Me_5)_2U^{III}I(THF)$ (50 mg, 0.071 mmol) and N_3Ad (13 mg, 0.073 mmol) were combined in toluene (5 mL) to form a brown solution. Slight gas evolution was observed. The solution was dried and the products were extracted into hexane (47 mg, 85%). The ^{1}H NMR spectrum in C_6D_6 showed a ratio of roughly 3:4 $(C_5Me_5)_2U^{VI}(=NAd)_2$ (δ 4.07 ppm, C_5Me_5)[5] to $(C_5Me_5)_2U^{IV}I_2$ (δ 18.00 ppm, C_5Me_5).[4] Brown crystals of $(C_5Me_5)_2U^{VI}(=NAd)_2$ were grown from a concentrated toluene solution at −35 °C and identified by X-ray diffraction.[5]

Reaction of $(C_5Me_5)_2UI^{III}(THF)$ and PhN=NPh. As above, $(C_5Me_5)_2U^{III}I(THF)$ (50 mg, 0.071 mmol) and PhN=NPh (6.5 mg, 0.036 mmol) were combined in toluene (5 mL) to form a red/brown solution. The solution was stirred for 30 minutes and then dried. The products were extracted into hexane, filtered, and dried to yield dark brown solids (39 mg, 76%). The ^{1}H NMR spectrum in C_6D_6 showed a ratio of roughly 2:3 $(C_5Me_5)_2U^{VI}(=NPh)_2$ (δ 4.11 ppm, C_5Me_5)[6] to $(C_5Me_5)_2U^{IV}I_2$ (δ 17.95 ppm, C_5Me_5).[4]

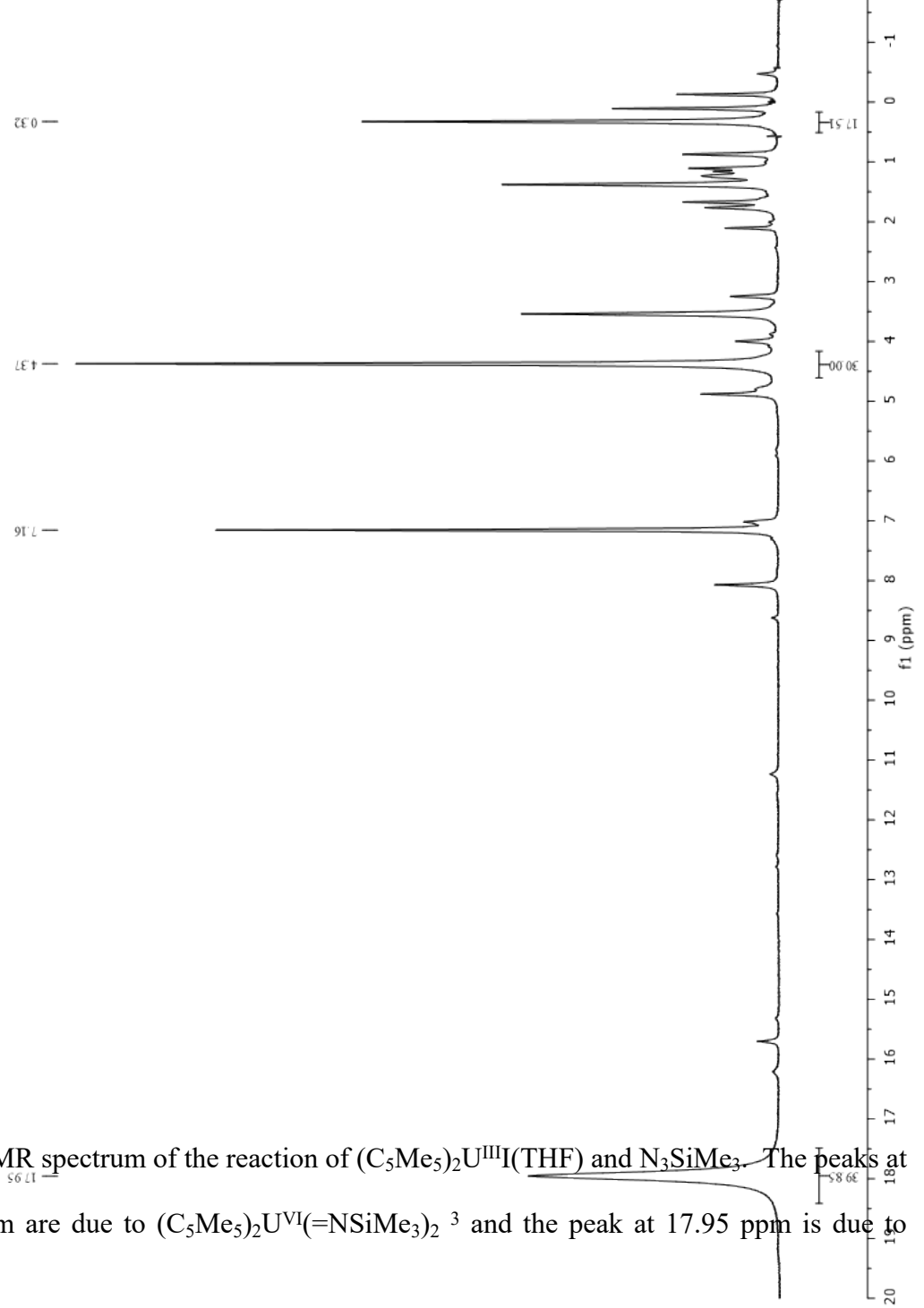

Figure S1: ^{1}H NMR spectrum of the reaction of $(C_5Me_5)_2U^{III}I(THF)$ and N_3SiMe_3. The peaks at 4.37 and 0.32 ppm are due to $(C_5Me_5)_2U^{VI}(=NSiMe_3)_2$ [3] and the peak at 17.95 ppm is due to $(C_5Me_5)_2U^{IV}I_2$.[4]

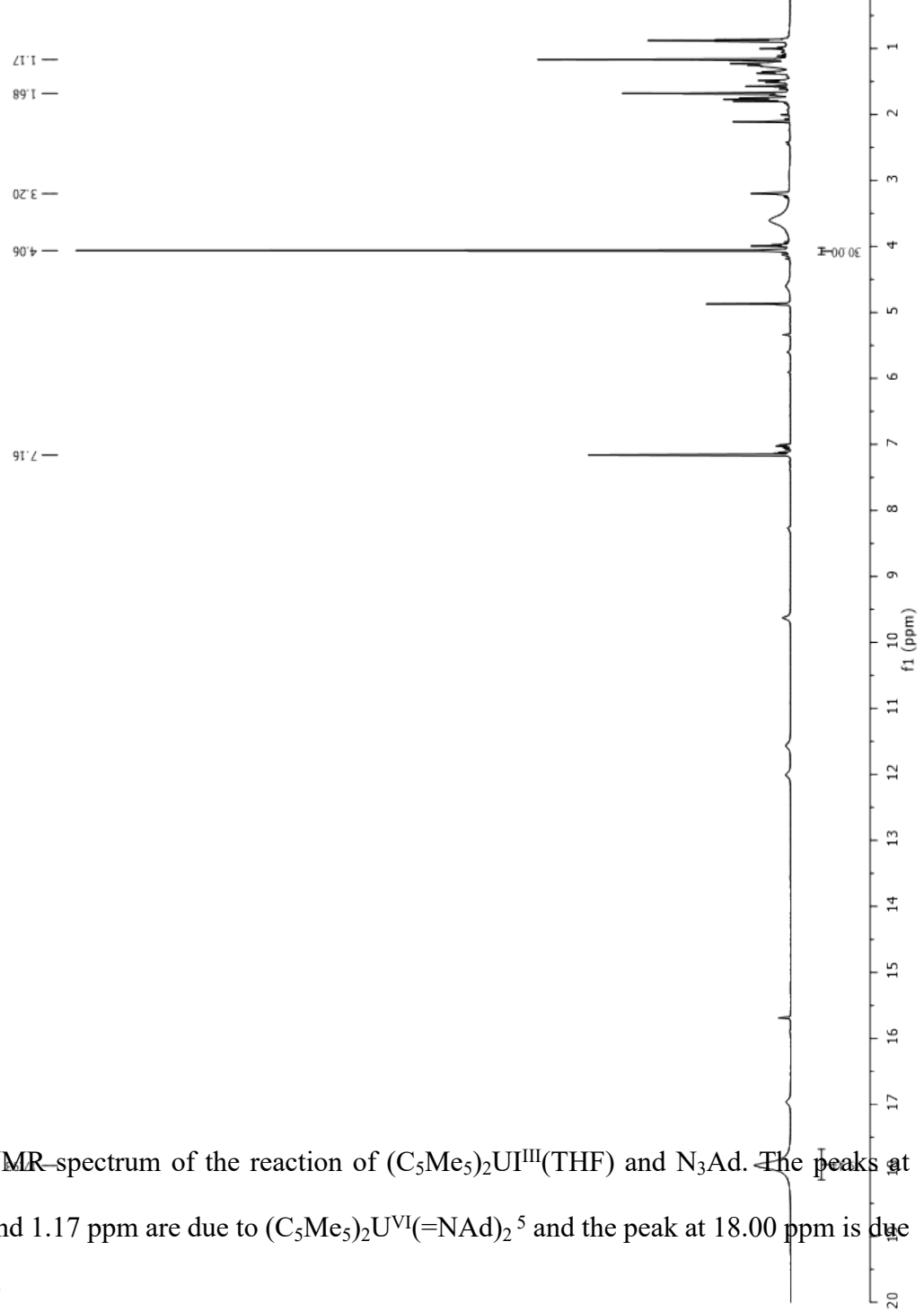

Figure S2: ^{1}H NMR spectrum of the reaction of $(C_5Me_5)_2UI^{III}(THF)$ and N_3Ad. The peaks at 4.07, 3.20, 1.68, and 1.17 ppm are due to $(C_5Me_5)_2U^{VI}(=NAd)_2$ [5] and the peak at 18.00 ppm is due to $(C_5Me_5)_2U^{IV}I_2$.[4]

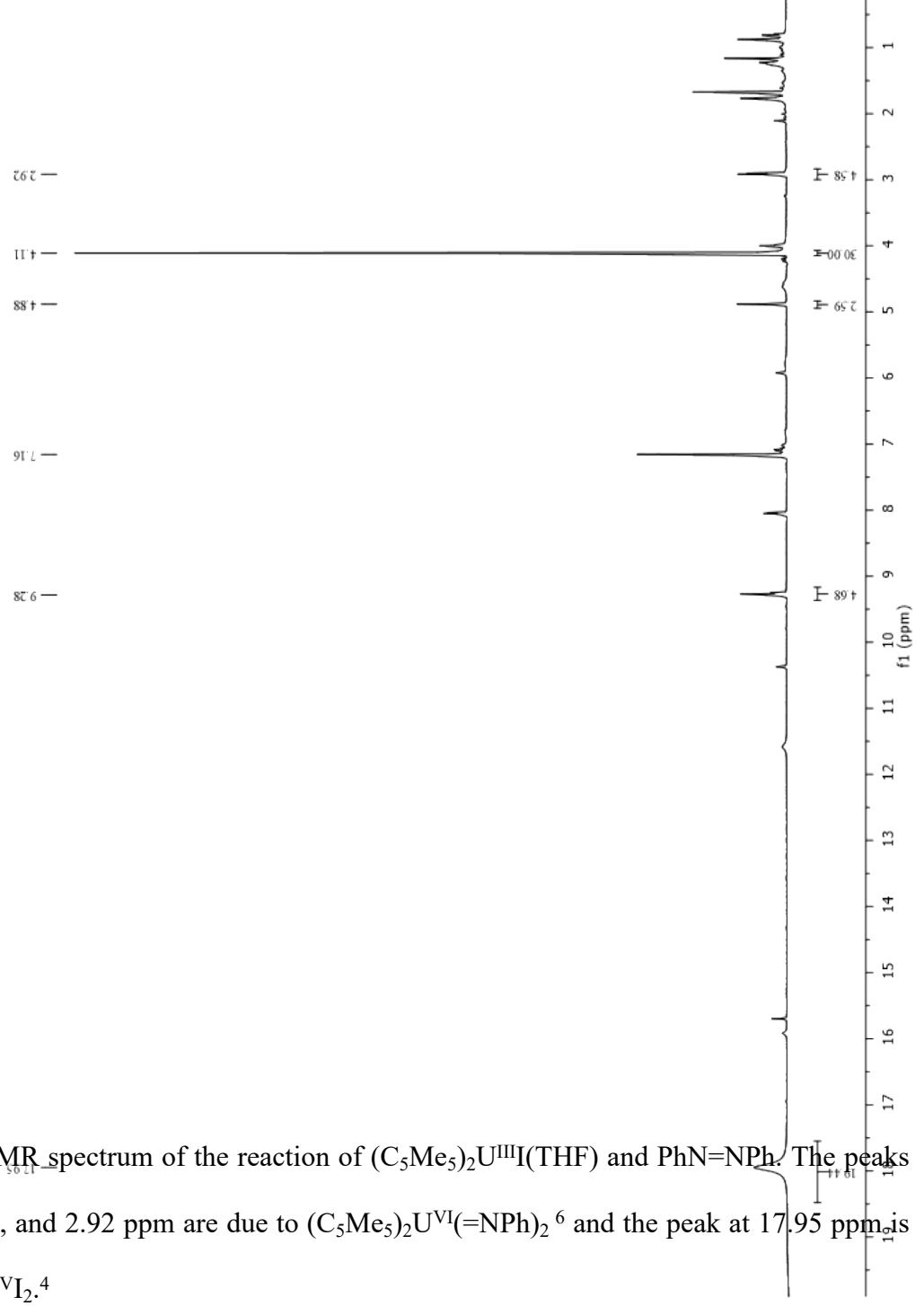

Figure S3: ^{1}H NMR spectrum of the reaction of $(C_5Me_5)_2U^{III}I(THF)$ and PhN=NPh. The peaks at 9.28, 4.88, 4.11, and 2.92 ppm are due to $(C_5Me_5)_2U^{VI}(=NPh)_2$ [6] and the peak at 17.95 ppm is due to $(C_5Me_5)_2U^{IV}I_2$.[4]

X-ray Data Collection, Structure Solution and Refinement for **1**.

A brown crystal of approximate dimensions 0.059 x 0.091 x 0.213 mm was mounted in a cryoloop and transferred to a Bruker SMART APEX II diffractometer system. The APEX2[7] program package was used to determine the unit-cell parameters and for data collection (120 sec/frame scan time). The raw frame data was processed using SAINT[8] and SADABS[9] to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL[10] program

package. The diffraction symmetry was 2/*m* and the systematic absences were consistent with the monoclinic space groups $P2_1$ and $P2_1/m$. It was later determined that space group $P2_1/m$ was correct.

The structure was solved by direct methods and refined on F^2 by full-matrix least-squares techniques. The analytical scattering factors[11] for neutral atoms were used throughout the analysis. Hydrogen atoms were included using a riding model. There were two molecules of the formula-unit present, each located on a mirror plane. The pentamethylcyclopentadienyl ligands were disordered and included using multiple components, partial site-occupancy-factors, geometric and displacements constraints.

Least-squares analysis yielded wR2 = 0.1737 and Goof = 1.075 for 153 variables refined against 6170 data (0.82 Å), R1 = 0.0670 for those 4634 data with $I > 2.0\sigma(I)$.

There were several high residuals present in the final difference-Fourier map. It was not possible to determine the nature of the residuals although it was probable that *n*-hexane solvent was present. The SQUEEZE[12a] routine in the PLATON[12b] program package was used to account for the electrons in the solvent accessible voids.

Table S1: Crystal data and structure refinement for **1**.

Identification code	jcw104	
Empirical formula	C_{23} H_{39} I N Si U	
Formula weight	722.57	
Temperature	133(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/m$	
Unit cell dimensions	a = 10.0187(12) Å	α = 90°.
	b = 15.8538(19) Å	β = 101.6286(18)°.

	c = 19.991(2) Å γ = 90°.
Volume	3110.2(6) Å^3
Z	4
Density (calculated)	1.543 Mg/m^3
Absorption coefficient	6.257 mm^{-1}
F(000)	1372
Crystal color	brown
Crystal size	0.213 x 0.091 x 0.059 mm^3
Theta range for data collection	1.040 to 25.740°
Index ranges	$-12 \le h \le 12$, $-19 \le k \le 19$, $-24 \le l \le 24$
Reflections collected	35670
Independent reflections	6170 [R(int) = 0.0722]
Completeness to theta = 25.242°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.2594 and 0.1951
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6170 / 0 / 153
Goodness-of-fit on F^2	1.075
Final R indices [I>2sigma(I) = 4634 data]	R1 = 0.0670, wR2 = 0.1616
R indices (all data, 0.82 Å)	R1 = 0.0911, wR2 = 0.1737
Largest diff. peak and hole	2.934 and -2.413 e.Å^{-3}

Table S2: Bond lengths [Å] and angles [°] for **1**.

U(1)-Cnt1	2.525	U(1)-C(3B)#1	2.85(3)
U(1)-N(1)	2.061(10)	U(1)-C(5)	2.877(18)
U(1)-C(2)#1	2.69(4)	U(1)-C(5)#1	2.88(4)
U(1)-C(2)	2.692(19)	U(1)-C(4)	2.898(19)
U(1)-C(1B)#1	2.731(13)	U(1)-C(4)#1	2.90(4)
U(1)-C(1B)	2.731(13)	U(1)-I(1)	3.1137(14)
U(1)-C(5B)	2.745(11)	Si(1)-N(1)	1.662(12)
U(1)-C(5B)#1	2.75(3)	Si(1)-C(11)	1.84(3)
U(1)-C(1)	2.750(17)	Si(1)-C(12)#1	1.84(3)
U(1)-C(1)#1	2.750(17)	Si(1)-C(12)	1.84(3)
U(1)-C(3)#1	2.79(4)	C(1)-C(2)	1.403(10)
U(1)-C(3)	2.79(2)	C(1)-C(5)	1.403(10)
U(1)-C(2B)#1	2.80(3)	C(1)-C(6)	1.492(11)
U(1)-C(2B)	2.797(13)	C(2)-C(3)	1.403(10)
U(1)-C(4B)	2.820(11)	C(2)-C(7)	1.492(10)
U(1)-C(4B)#1	2.82(3)	C(3)-C(4)	1.403(10)
U(1)-C(3B)	2.852(13)	C(3)-C(8)	1.492(11)

C(4)-C(5)	1.403(10)	C(13)-C(14)	1.402(6)
C(4)-C(9)	1.492(11)	C(13)-C(17)	1.402(6)
C(5)-C(10)	1.492(10)	C(13)-C(19)	1.490(6)
C(1B)-C(5B)	1.410(6)	C(14)-C(15)	1.402(6)
C(1B)-C(2B)	1.410(6)	C(14)-C(20)	1.490(6)
C(1B)-C(6B)	1.490(7)	C(15)-C(16)	1.402(6)
C(2B)-C(3B)	1.410(6)	C(15)-C(21)	1.490(6)
C(2B)-C(7B)	1.490(7)	C(16)-C(17)	1.402(6)
C(3B)-C(4B)	1.410(6)	C(16)-C(22)	1.490(6)
C(3B)-C(8B)	1.490(7)	C(17)-C(18)	1.490(6)
C(4B)-C(5B)	1.410(6)	C(13B)-C(17B)	1.409(10)
C(4B)-C(9B)	1.490(7)	C(13B)-C(14B)	1.409(10)
C(5B)-C(10B)	1.490(7)	C(13B)-C(19B)	1.488(11)
U(2)-Cnt2	2.513	C(14B)-C(15B)	1.409(10)
U(2)-N(2)	2.100(13)	C(14B)-C(20B)	1.488(11)
U(2)-C(13B)	2.665(18)	C(15B)-C(16B)	1.409(10)
U(2)-C(13B)#2	2.665(18)	C(15B)-C(21B)	1.488(11)
U(2)-C(14)#2	2.71(2)	C(16B)-C(17B)	1.409(10)
U(2)-C(14)	2.709(11)	C(16B)-C(22B)	1.488(11)
U(2)-C(17B)#2	2.73(4)	C(17B)-C(18B)	1.488(11)
U(2)-C(17B)	2.735(19)		
U(2)-C(15)	2.748(10)	Cnt1-U(1)-I(1)	111.3
U(2)-C(15)#2	2.75(2)	Cnt1-U(1)-N(1)	103.1
U(2)-C(14B)#2	2.76(4)	Cnt1-U(1)-Cnt1	132.1
U(2)-C(14B)	2.759(17)	N(1)-U(1)-C(2)#1	122.3(10)
U(2)-C(13)	2.782(11)	N(1)-U(1)-C(2)	122.3(4)
U(2)-C(13)#2	2.782(11)	C(2)#1-U(1)-C(2)	115.4(12)
U(2)-C(16)	2.844(10)	N(1)-U(1)-C(1B)#1	89.2(2)
U(2)-C(16)#2	2.84(2)	N(1)-U(1)-C(1B)	89.2(2)
U(2)-C(17)#2	2.86(3)	C(1B)#1-U(1)-C(1B)	178.3(5)
U(2)-C(17)	2.864(11)	N(1)-U(1)-C(5B)	79.7(3)
U(2)-C(16B)#2	2.87(4)	C(1B)#1-U(1)-C(5B)	149.1(4)
U(2)-C(16B)	2.868(19)	C(1B)-U(1)-C(5B)	29.84(16)
U(2)-C(15B)#2	2.88(4)	N(1)-U(1)-C(5B)#1	79.7(7)
U(2)-C(15B)	2.882(18)	C(1B)#1-U(1)-C(5B)#1	29.8(6)
U(2)-I(2)	3.1074(14)	C(1B)-U(1)-C(5B)#1	149.1(6)
Si(2)-N(2)	1.619(14)	C(5B)-U(1)-C(5B)#1	119.2(7)
Si(2)-C(23)	1.85(2)	N(1)-U(1)-C(1)	93.1(4)
Si(2)-C(24)	1.85(2)	C(2)#1-U(1)-C(1)	144.4(11)
Si(2)-C(24)#2	1.85(2)	C(2)-U(1)-C(1)	29.9(3)

N(1)-U(1)-C(1)#1	93.1(4)	C(4B)-U(1)-C(4B)#1	83.0(6)
C(2)#1-U(1)-C(1)#1	29.9(10)	N(1)-U(1)-C(3B)	127.7(3)
C(2)-U(1)-C(1)#1	144.4(7)	C(1B)#1-U(1)-C(3B)	132.9(4)
C(1)-U(1)-C(1)#1	167.4(8)	C(1B)-U(1)-C(3B)	48.2(3)
N(1)-U(1)-C(3)#1	121.3(8)	C(5B)-U(1)-C(3B)	48.1(2)
C(2)#1-U(1)-C(3)#1	29.6(6)	C(5B)#1-U(1)-C(3B)	119.6(7)
C(2)-U(1)-C(3)#1	107.0(10)	C(2B)#1-U(1)-C(3B)	107.8(7)
C(1)-U(1)-C(3)#1	134.7(8)	C(2B)-U(1)-C(3B)	28.89(16)
C(1)#1-U(1)-C(3)#1	48.4(10)	C(4B)-U(1)-C(3B)	28.79(15)
N(1)-U(1)-C(3)	121.3(4)	C(4B)#1-U(1)-C(3B)	90.9(6)
C(2)#1-U(1)-C(3)	107.0(11)	N(1)-U(1)-C(3B)#1	127.7(5)
C(2)-U(1)-C(3)	29.6(3)	C(1B)#1-U(1)-C(3B)#1	48.2(6)
C(1)-U(1)-C(3)	48.4(4)	C(1B)-U(1)-C(3B)#1	132.9(5)
C(1)#1-U(1)-C(3)	134.7(6)	C(5B)-U(1)-C(3B)#1	119.6(5)
C(3)#1-U(1)-C(3)	86.9(10)	C(5B)#1-U(1)-C(3B)#1	48.1(6)
N(1)-U(1)-C(2B)#1	118.7(6)	C(2B)#1-U(1)-C(3B)#1	28.9(4)
C(1B)#1-U(1)-C(2B)#1	29.5(7)	C(2B)-U(1)-C(3B)#1	107.8(5)
C(1B)-U(1)-C(2B)#1	152.1(7)	C(4B)-U(1)-C(3B)#1	90.9(6)
C(5B)-U(1)-C(2B)#1	148.5(7)	C(4B)#1-U(1)-C(3B)#1	28.8(3)
C(5B)#1-U(1)-C(2B)#1	48.6(6)	C(3B)-U(1)-C(3B)#1	84.7(6)
N(1)-U(1)-C(2B)	118.7(2)	N(1)-U(1)-C(5)	77.2(4)
C(1B)#1-U(1)-C(2B)	152.1(4)	C(2)#1-U(1)-C(5)	153.2(11)
C(1B)-U(1)-C(2B)	29.53(17)	C(2)-U(1)-C(5)	48.0(4)
C(5B)-U(1)-C(2B)	48.6(3)	C(1)-U(1)-C(5)	28.8(2)
C(5B)#1-U(1)-C(2B)	148.5(7)	C(1)#1-U(1)-C(5)	163.8(7)
C(2B)#1-U(1)-C(2B)	122.6(7)	C(3)#1-U(1)-C(5)	126.3(9)
N(1)-U(1)-C(4B)	102.5(3)	C(3)-U(1)-C(5)	47.2(4)
C(1B)#1-U(1)-C(4B)	131.5(4)	N(1)-U(1)-C(5)#1	77.2(9)
C(1B)-U(1)-C(4B)	48.5(2)	C(2)#1-U(1)-C(5)#1	48.0(9)
C(5B)-U(1)-C(4B)	29.31(16)	C(2)-U(1)-C(5)#1	153.2(11)
C(5B)#1-U(1)-C(4B)	105.7(7)	C(1)-U(1)-C(5)#1	163.8(11)
C(2B)#1-U(1)-C(4B)	119.5(7)	C(1)#1-U(1)-C(5)#1	28.8(10)
C(2B)-U(1)-C(4B)	47.9(2)	C(3)#1-U(1)-C(5)#1	47.2(8)
N(1)-U(1)-C(4B)#1	102.5(6)	C(3)-U(1)-C(5)#1	126.3(11)
C(1B)#1-U(1)-C(4B)#1	48.5(5)	C(5)-U(1)-C(5)#1	135.0(11)
C(1B)-U(1)-C(4B)#1	131.5(5)	N(1)-U(1)-C(4)	93.3(4)
C(5B)-U(1)-C(4B)#1	105.7(6)	C(2)#1-U(1)-C(4)	125.7(11)
C(5B)#1-U(1)-C(4B)#1	29.3(4)	C(2)-U(1)-C(4)	47.7(4)
C(2B)#1-U(1)-C(4B)#1	47.9(6)	C(1)-U(1)-C(4)	47.3(4)
C(2B)-U(1)-C(4B)#1	119.5(5)	C(1)#1-U(1)-C(4)	143.0(6)

C(3)#1-U(1)-C(4)	98.2(9)	C(12)#1-Si(1)-C(12)	109(2)
C(3)-U(1)-C(4)	28.5(2)	Si(1)-N(1)-U(1)	164.0(7)
C(5)-U(1)-C(4)	28.1(2)	C(2)-C(1)-C(5)	108.0
C(5)#1-U(1)-C(4)	119.4(11)	C(2)-C(1)-C(6)	126.0
N(1)-U(1)-C(4)#1	93.3(9)	C(5)-C(1)-C(6)	126.0
C(2)#1-U(1)-C(4)#1	47.7(8)	C(2)-C(1)-U(1)	72.8(6)
C(2)-U(1)-C(4)#1	125.7(10)	C(5)-C(1)-U(1)	80.6(6)
C(1)-U(1)-C(4)#1	143.0(8)	C(6)-C(1)-U(1)	113.0(6)
C(1)#1-U(1)-C(4)#1	47.3(10)	C(1)-C(2)-C(3)	108.0
C(3)#1-U(1)-C(4)#1	28.5(5)	C(1)-C(2)-C(7)	126.0
C(3)-U(1)-C(4)#1	98.2(10)	C(3)-C(2)-C(7)	126.0
C(5)-U(1)-C(4)#1	119.4(9)	C(1)-C(2)-U(1)	77.4(6)
C(5)#1-U(1)-C(4)#1	28.1(5)	C(3)-C(2)-U(1)	78.9(6)
C(4)-U(1)-C(4)#1	95.9(10)	C(7)-C(2)-U(1)	110.5(6)
N(1)-U(1)-I(1)	82.4(3)	C(4)-C(3)-C(2)	108.0
C(2)#1-U(1)-I(1)	95.3(10)	C(4)-C(3)-C(8)	126.0
C(2)-U(1)-I(1)	95.3(4)	C(2)-C(3)-C(8)	126.0
C(1B)#1-U(1)-I(1)	90.2(2)	C(4)-C(3)-U(1)	80.2(6)
C(1B)-U(1)-I(1)	90.2(2)	C(2)-C(3)-U(1)	71.5(6)
C(5B)-U(1)-I(1)	116.5(2)	C(8)-C(3)-U(1)	114.6(6)
C(5B)#1-U(1)-I(1)	116.5(6)	C(3)-C(4)-C(5)	108.0
C(1)-U(1)-I(1)	85.0(4)	C(3)-C(4)-C(9)	126.0
C(1)#1-U(1)-I(1)	85.0(4)	C(5)-C(4)-C(9)	126.0
C(3)#1-U(1)-I(1)	124.9(8)	C(3)-C(4)-U(1)	71.3(6)
C(3)-U(1)-I(1)	124.9(3)	C(5)-C(4)-U(1)	75.1(6)
C(2B)#1-U(1)-I(1)	92.4(6)	C(9)-C(4)-U(1)	119.4(6)
C(2B)-U(1)-I(1)	92.4(2)	C(1)-C(5)-C(4)	108.0
C(4B)-U(1)-I(1)	137.7(2)	C(1)-C(5)-C(10)	126.0
C(4B)#1-U(1)-I(1)	137.7(5)	C(4)-C(5)-C(10)	126.0
C(3B)-U(1)-I(1)	119.5(2)	C(1)-C(5)-U(1)	70.6(6)
C(3B)#1-U(1)-I(1)	119.5(5)	C(4)-C(5)-U(1)	76.8(6)
C(5)-U(1)-I(1)	106.3(3)	C(10)-C(5)-U(1)	118.5(6)
C(5)#1-U(1)-I(1)	106.3(10)	C(5B)-C(1B)-C(2B)	108.0
C(4)-U(1)-I(1)	132.0(3)	C(5B)-C(1B)-C(6B)	126.0
C(4)#1-U(1)-I(1)	132.0(8)	C(2B)-C(1B)-C(6B)	126.0
N(1)-Si(1)-C(11)	110.3(10)	C(5B)-C(1B)-U(1)	75.6(4)
N(1)-Si(1)-C(12)#1	111.8(9)	C(2B)-C(1B)-U(1)	77.8(4)
C(11)-Si(1)-C(12)#1	106.8(9)	C(6B)-C(1B)-U(1)	112.9(4)
N(1)-Si(1)-C(12)	111.8(8)	C(3B)-C(2B)-C(1B)	108.0
C(11)-Si(1)-C(12)	106.8(9)	C(3B)-C(2B)-C(7B)	126.0

C(1B)-C(2B)-C(7B)	126.0	C(14)-U(2)-C(15)	29.75(14)
C(3B)-C(2B)-U(1)	77.7(4)	N(2)-U(2)-C(15)#2	79.7(6)
C(1B)-C(2B)-U(1)	72.6(4)	C(14)#2-U(2)-C(15)#2	29.8(4)
C(7B)-C(2B)-U(1)	115.8(4)	C(14)-U(2)-C(15)#2	147.5(6)
C(2B)-C(3B)-C(4B)	108.0	C(15)-U(2)-C(15)#2	117.8(6)
C(2B)-C(3B)-C(8B)	126.0	N(2)-U(2)-C(14B)#2	93.8(11)
C(4B)-C(3B)-C(8B)	126.0	C(13B)-U(2)-C(14B)#2	142.7(12)
C(2B)-C(3B)-U(1)	73.4(4)	C(13B)#2-U(2)-C(14B)#2	30.1(11)
C(4B)-C(3B)-U(1)	74.4(4)	C(17B)#2-U(2)-C(14B)#2	49.0(10)
C(8B)-C(3B)-U(1)	118.1(4)	C(17B)-U(2)-C(14B)#2	137.1(11)
C(5B)-C(4B)-C(3B)	108.0	N(2)-U(2)-C(14B)	93.8(4)
C(5B)-C(4B)-C(9B)	126.0	C(13B)-U(2)-C(14B)	30.1(3)
C(3B)-C(4B)-C(9B)	126.0	C(13B)#2-U(2)-C(14B)	142.7(7)
C(5B)-C(4B)-U(1)	72.4(4)	C(17B)#2-U(2)-C(14B)	137.1(9)
C(3B)-C(4B)-U(1)	76.8(4)	C(17B)-U(2)-C(14B)	49.0(4)
C(9B)-C(4B)-U(1)	116.8(4)	C(14B)#2-U(2)-C(14B)	162.1(11)
C(4B)-C(5B)-C(1B)	108.0	N(2)-U(2)-C(13)	118.8(2)
C(4B)-C(5B)-C(10B)	126.0	C(14)#2-U(2)-C(13)	152.0(7)
C(1B)-C(5B)-C(10B)	126.0	C(14)-U(2)-C(13)	29.54(15)
C(4B)-C(5B)-U(1)	78.3(4)	C(15)-U(2)-C(13)	48.4(2)
C(1B)-C(5B)-U(1)	74.5(4)	C(15)#2-U(2)-C(13)	146.9(6)
C(10B)-C(5B)-U(1)	113.6(4)	N(2)-U(2)-C(13)#2	118.8(2)
Cnt2-U(2)-I(2)	110.8	C(14)#2-U(2)-C(13)#2	29.5(7)
Cnt2-U(2)-N(2)	102.8	C(14)-U(2)-C(13)#2	152.0(4)
Cnt2-U(2)-Cnt2	133.4	C(15)-U(2)-C(13)#2	146.9(3)
N(2)-U(2)-C(13B)	122.8(4)	C(15)#2-U(2)-C(13)#2	48.4(6)
N(2)-U(2)-C(13B)#2	122.8(4)	C(13)-U(2)-C(13)#2	122.5(4)
C(13B)-U(2)-C(13B)#2	114.4(8)	N(2)-U(2)-C(16)	102.3(3)
N(2)-U(2)-C(14)#2	89.2(6)	C(14)#2-U(2)-C(16)	130.0(6)
N(2)-U(2)-C(14)	89.2(2)	C(14)-U(2)-C(16)	48.1(2)
C(14)#2-U(2)-C(14)	177.2(7)	C(15)-U(2)-C(16)	28.97(14)
N(2)-U(2)-C(17B)#2	120.2(8)	C(15)#2-U(2)-C(16)	104.3(6)
C(13B)-U(2)-C(17B)#2	108.5(10)	C(13)-U(2)-C(16)	47.5(2)
C(13B)#2-U(2)-C(17B)#2	30.2(9)	C(13)#2-U(2)-C(16)	118.3(3)
N(2)-U(2)-C(17B)	120.2(4)	N(2)-U(2)-C(16)#2	102.3(5)
C(13B)-U(2)-C(17B)	30.2(3)	C(14)#2-U(2)-C(16)#2	48.1(6)
C(13B)#2-U(2)-C(17B)	108.5(7)	C(14)-U(2)-C(16)#2	130.0(4)
C(17B)#2-U(2)-C(17B)	89.2(11)	C(15)-U(2)-C(16)#2	104.3(5)
N(2)-U(2)-C(15)	79.7(3)	C(15)#2-U(2)-C(16)#2	29.0(4)
C(14)#2-U(2)-C(15)	147.5(7)	C(13)-U(2)-C(16)#2	118.3(5)

C(13)#2-U(2)-C(16)#2 47.5(5)
C(16)-U(2)-C(16)#2 81.9(6)
N(2)-U(2)-C(17)#2 127.3(5)
C(14)#2-U(2)-C(17)#2 47.9(6)
C(14)-U(2)-C(17)#2 131.9(4)
C(15)-U(2)-C(17)#2 118.2(5)
C(15)#2-U(2)-C(17)#2 47.6(5)
C(13)-U(2)-C(17)#2 107.2(5)
C(13)#2-U(2)-C(17)#2 28.7(4)
C(16)-U(2)-C(17)#2 89.9(5)
C(16)#2-U(2)-C(17)#2 28.4(3)
N(2)-U(2)-C(17) 127.3(3)
C(14)#2-U(2)-C(17) 131.9(6)
C(14)-U(2)-C(17) 47.9(2)
C(15)-U(2)-C(17) 47.6(2)
C(15)#2-U(2)-C(17) 118.2(6)
C(13)-U(2)-C(17) 28.71(14)
C(13)#2-U(2)-C(17) 107.2(4)
C(16)-U(2)-C(17) 28.44(14)
C(16)#2-U(2)-C(17) 89.9(5)
C(17)#2-U(2)-C(17) 84.0(5)
N(2)-U(2)-C(16B)#2 91.5(9)
C(13B)-U(2)-C(16B)#2 129.2(11)
C(13B)#2-U(2)-C(16B)#2 48.5(10)
C(17B)#2-U(2)-C(16B)#2 29.0(5)
C(17B)-U(2)-C(16B)#2 102.0(11)
C(14B)#2-U(2)-C(16B)#2 47.7(9)
C(14B)-U(2)-C(16B)#2 148.1(9)
N(2)-U(2)-C(16B) 91.5(4)
C(13B)-U(2)-C(16B) 48.5(4)
C(13B)#2-U(2)-C(16B) 129.2(7)
C(17B)#2-U(2)-C(16B) 102.0(10)
C(17B)-U(2)-C(16B) 29.0(2)
C(14B)#2-U(2)-C(16B) 148.1(10)
C(14B)-U(2)-C(16B) 47.7(4)
C(16B)#2-U(2)-C(16B) 100.8(10)
N(2)-U(2)-C(15B)#2 76.6(10)
C(13B)-U(2)-C(15B)#2 156.0(12)
C(13B)#2-U(2)-C(15B)#2 48.3(11)
C(17B)#2-U(2)-C(15B)#2 47.8(9)
C(17B)-U(2)-C(15B)#2 130.3(11)
C(14B)#2-U(2)-C(15B)#2 28.8(6)
C(14B)-U(2)-C(15B)#2 168.1(11)
C(16B)#2-U(2)-C(15B)#2 28.4(6)
C(16B)-U(2)-C(15B)#2 124.5(11)
N(2)-U(2)-C(15B) 76.6(4)
C(13B)-U(2)-C(15B) 48.3(4)
C(13B)#2-U(2)-C(15B) 156.0(7)
C(17B)#2-U(2)-C(15B) 130.3(9)
C(17B)-U(2)-C(15B) 47.8(4)
C(14B)#2-U(2)-C(15B) 168.1(11)
C(14B)-U(2)-C(15B) 28.8(2)
C(16B)#2-U(2)-C(15B) 124.5(10)
C(16B)-U(2)-C(15B) 28.4(2)
C(15B)#2-U(2)-C(15B) 139.7(11)
N(2)-U(2)-I(2) 81.7(3)
C(13B)-U(2)-I(2) 94.6(4)
C(13B)#2-U(2)-I(2) 94.6(4)
C(14)#2-U(2)-I(2) 91.1(6)
C(14)-U(2)-I(2) 91.1(2)
C(17B)#2-U(2)-I(2) 124.8(9)
C(17B)-U(2)-I(2) 124.8(4)
C(15)-U(2)-I(2) 117.0(2)
C(15)#2-U(2)-I(2) 117.0(5)
C(14B)#2-U(2)-I(2) 82.5(10)
C(14B)-U(2)-I(2) 82.5(4)
C(13)-U(2)-I(2) 93.8(2)
C(13)#2-U(2)-I(2) 93.8(2)
C(16)-U(2)-I(2) 138.4(2)
C(16)#2-U(2)-I(2) 138.4(4)
C(17)#2-U(2)-I(2) 120.9(5)
C(17)-U(2)-I(2) 120.9(2)
C(16B)#2-U(2)-I(2) 129.4(9)
C(16B)-U(2)-I(2) 129.4(4)
C(15B)#2-U(2)-I(2) 102.7(10)
C(15B)-U(2)-I(2) 102.7(4)
N(2)-Si(2)-C(23) 110.9(10)
N(2)-Si(2)-C(24) 111.9(7)
C(23)-Si(2)-C(24) 107.3(8)
N(2)-Si(2)-C(24)#2 111.9(7)

C(23)-Si(2)-C(24)#2	107.3(8)	C(18)-C(17)-U(2)	118.5(3)
C(24)-Si(2)-C(24)#2	107.3(18)	C(17B)-C(13B)-C(14B)	108.0
Si(2)-N(2)-U(2)	163.7(7)	C(17B)-C(13B)-C(19B)	126.0
C(14)-C(13)-C(17)	108.0	C(14B)-C(13B)-C(19B)	126.0
C(14)-C(13)-C(19)	126.0	C(17B)-C(13B)-U(2)	77.6(6)
C(17)-C(13)-C(19)	126.0	C(14B)-C(13B)-U(2)	78.7(6)
C(14)-C(13)-U(2)	72.3(3)	C(19B)-C(13B)-U(2)	110.4(6)
C(17)-C(13)-U(2)	78.9(3)	C(15B)-C(14B)-C(13B)	108.0
C(19)-C(13)-U(2)	115.0(3)	C(15B)-C(14B)-C(20B)	126.0
C(15)-C(14)-C(13)	108.0	C(13B)-C(14B)-C(20B)	126.0
C(15)-C(14)-C(20)	126.0	C(15B)-C(14B)-U(2)	80.4(6)
C(13)-C(14)-C(20)	126.0	C(13B)-C(14B)-U(2)	71.3(6)
C(15)-C(14)-U(2)	76.7(3)	C(20B)-C(14B)-U(2)	114.5(6)
C(13)-C(14)-U(2)	78.1(4)	C(14B)-C(15B)-C(16B)	108.0
C(20)-C(14)-U(2)	111.8(3)	C(14B)-C(15B)-C(21B)	126.0
C(14)-C(15)-C(16)	108.0	C(16B)-C(15B)-C(21B)	126.0
C(14)-C(15)-C(21)	126.0	C(14B)-C(15B)-U(2)	70.7(5)
C(16)-C(15)-C(21)	126.0	C(16B)-C(15B)-U(2)	75.3(6)
C(14)-C(15)-U(2)	73.6(3)	C(21B)-C(15B)-U(2)	119.8(6)
C(16)-C(15)-U(2)	79.3(3)	C(17B)-C(16B)-C(15B)	108.0
C(21)-C(15)-U(2)	113.5(3)	C(17B)-C(16B)-C(22B)	126.0
C(15)-C(16)-C(17)	108.0	C(15B)-C(16B)-C(22B)	126.0
C(15)-C(16)-C(22)	126.0	C(17B)-C(16B)-U(2)	70.2(6)
C(17)-C(16)-C(22)	126.0	C(15B)-C(16B)-U(2)	76.4(6)
C(15)-C(16)-U(2)	71.7(3)	C(22B)-C(16B)-U(2)	119.2(5)
C(17)-C(16)-U(2)	76.6(3)	C(16B)-C(17B)-C(13B)	108.0
C(22)-C(16)-U(2)	117.6(3)	C(16B)-C(17B)-C(18B)	126.0
C(13)-C(17)-C(16)	108.0	C(13B)-C(17B)-C(18B)	126.0
C(13)-C(17)-C(18)	126.0	C(16B)-C(17B)-U(2)	80.8(6)
C(16)-C(17)-C(18)	126.0	C(13B)-C(17B)-U(2)	72.1(6)
C(13)-C(17)-U(2)	72.4(3)	C(18B)-C(17B)-U(2)	113.4(6)
C(16)-C(17)-U(2)	75.0(3)		

Symmetry transformations used to generate equivalent atoms:
#1 x,-y+1/2,z #2 x,-y+3/2,z

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Definitions:

$wR2 = [\Sigma[w(F_o^2-F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$

$R1 = \Sigma||F_o|-|F_c|| / \Sigma|F_o|$

$Goof = S = [\Sigma[w(F_o^2-F_c^2)^2] / (n-p)]^{1/2}$ where n is the number of reflections and p is the total number of parameters refined.