Dalton Transactions

Synthesis and structural study of new metallasilsesquioxanes of potassium and uranium

Stephan Gießmann, Volker Lorenz, Phil Liebing, Liane Hilfert, Axel Fischer, and Frank T. Edelmann*

Chemisches Institut der Otto-von-Guericke-Universität, 39106 Magdeburg, Germany.

Supporting Information

STRUCTURE REPORT for Compound 2

STRUCTURE REPORT for Compound 3

STRUCTURE REPORT for Compound 2

Crystallographer: A. Fischer, P. Liebing
ID code: LOKA **Date:** 20 December 2016
Compound: $(Cy_7Si_7O_{12})_2K_6(DME)_4$ (**2**)
Formula sum: $C_{100}H_{194}K_6O_{32}Si_{14}$
Formula moieties: $C_{100}H_{194}K_6O_{32}Si_{14}$

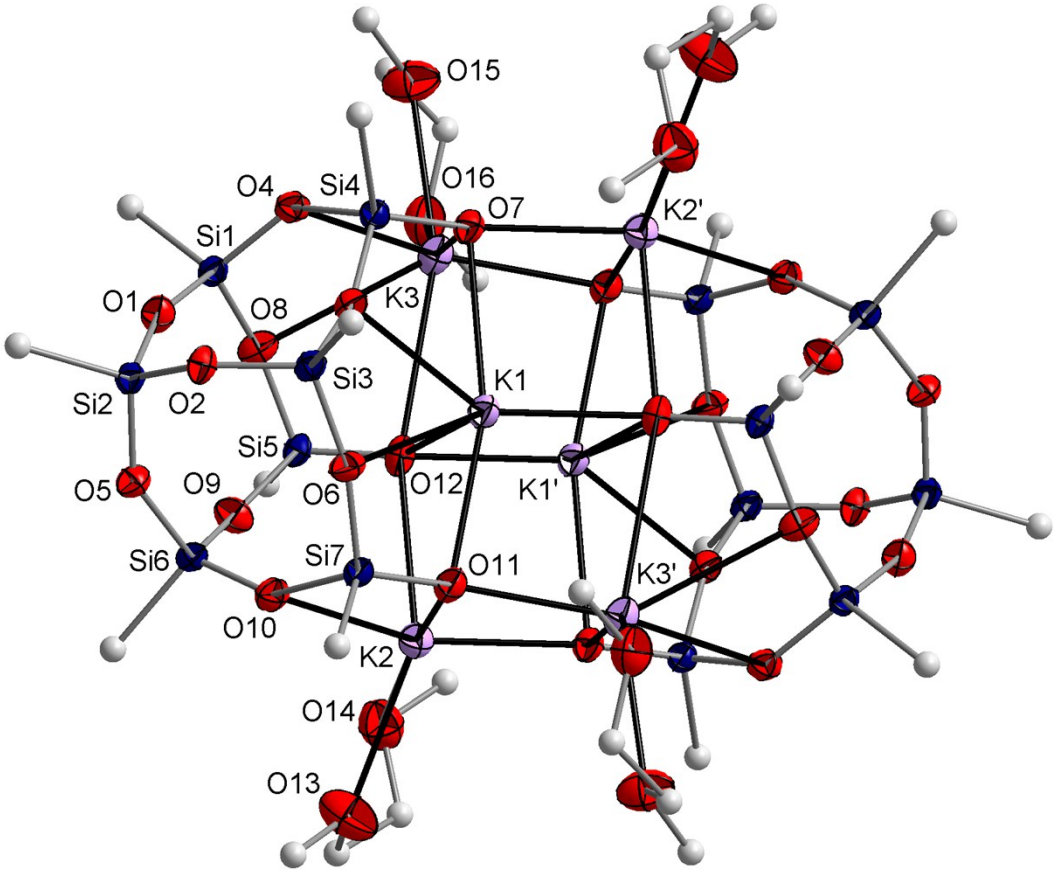

Figure S1. Molecular structure of **2** in the crystal showing the atom-labeling scheme (displacement ellipsoids of K, Si and O drawn at the 50% probability level, H atoms and peripheral C atoms of the cyclohexyl groups omitted for clarity). Symmetry code: ‘ 1–x, 2–y, 1–z.

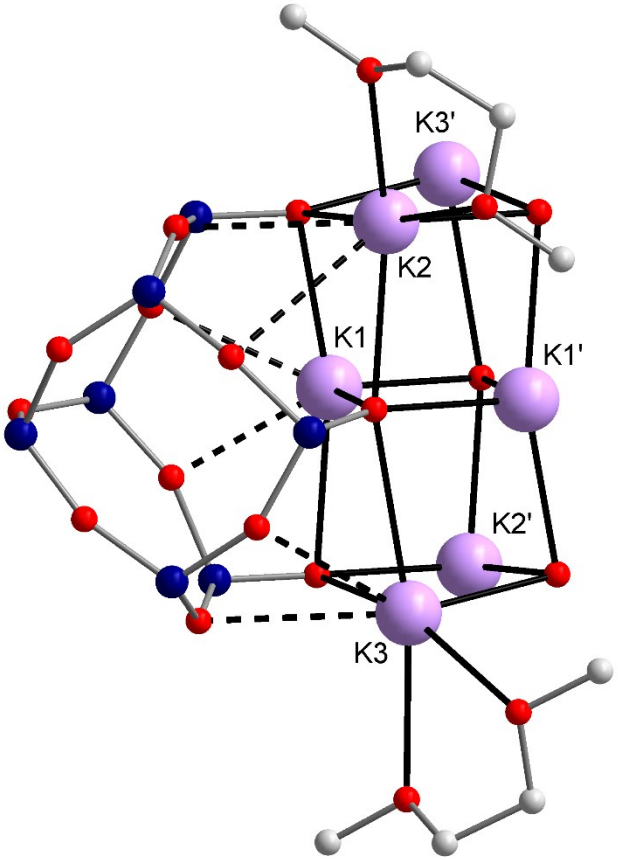

Figure S2. Representation of the coordination sphere of the potassium atoms in compound **2**.

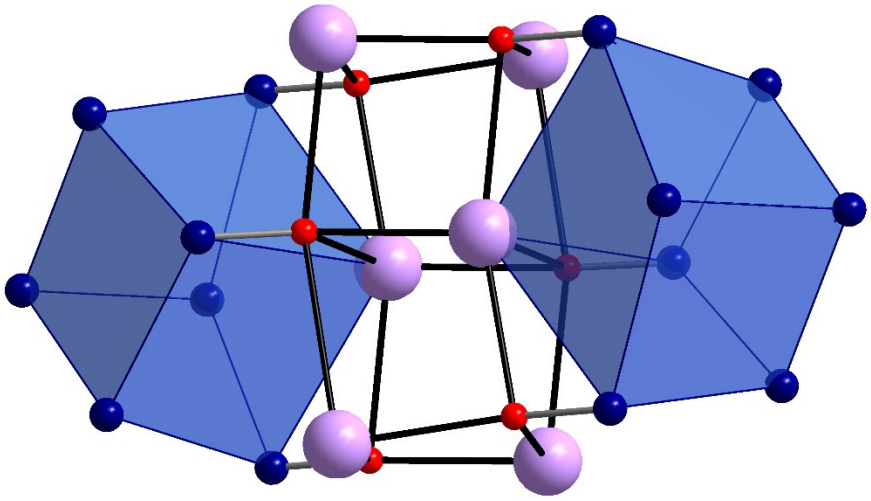

Figure S3. Representation of the interconnection of the two cube-shaped Si_7K frameworks in compound **2**.

List of Tables

Table S1. Crystallographic Data and Details on Structure Refinement for Compound **2**.

formula sum	$C_{100}H_{194}K_6O_{32}Si_{14}$
formula weight	2536.40
crystal color / shape / size (mm)	colorless plates / 0.25 × 0.14 × 0.08
crystal system	triclinic
space group	$P\bar{1}$
unit cell parameters	
a (Å)	15.1248(6)
b (Å)	15.1905(6)
c (Å)	16.8898(6)
α (deg)	101.994(3)
β (deg)	96.467(3)
γ (deg)	115.554(3)
unit cell volume V (Å^3)	3333.3(2)
molecules per cell z	1
crystallographic density ρ_{calcd} (g cm^{-3})	1.264
absorption coefficient μ (mm^{-1})	0.389
diffractometer	Bruker Smart CCD System
radiation (λ [Å])	graphite-monochromated Mo-K_α (0.71073)
temperature (°C)	–73(2)
scan type[a]	ω scans
completeness of dataset	98.0%
θ range of data collection (deg)	1.551 … 25.000
reflections collected[a]	17619 ($-17 \le h \le 15$, $-15 \le k \le 18$, $-20 \le l \le 18$)
independent reflections	11495 (R_{int} = 0.0470)
independent reflections with $I>2\sigma(I)$	6880
structure solution method	direct methods (SHELXS-97[b])
refinement method	full-matrix least-squares on F^2 (SHELXL-2016/4[b])
absorption correction method[a]	multi-scan
range of transmission factors	0.9 … 1.0
data / parameters / restraints	11495 / 685 / 0
goodness of fit (GooF) [all data]	1.037
final R values	
R_1 [all data, $I \ge 2\sigma(I)$]	0.1268, 0.0617
wR_2 [all data, $I \ge 2\sigma(I)$]]	0.1275, 0.1015
largest difference peak and hole	0.371 and –0.400 e Å^{-3}

Refinement special details: Reflections (0 0 1), (-1 1 0) and (1 0 0) are strongly disagreeing with the structural model and were therefore omitted for refinement.

[a] Bruker (2001). *AXS SMART and SADABS.* Bruker AXS Inc., Madison, Wisconsin, USA.

[b] G. M. Sheldrick, *Acta Cryst.* 2015, **C71**, 3–8.

Table S2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters for Compound **2**.

Atom	x	y	z	U_{eq} / Å^2
K1	0.53345(7)	1.00149(7)	0.41131(6)	0.0283(2)
K2	0.31787(8)	1.07651(7)	0.45142(6)	0.0325(3)
K3	0.40675(8)	0.73496(7)	0.47589(6)	0.0324(3)
O1	0.3430(2)	0.8997(2)	0.44119(18)	0.0308(8)
O2	0.4752(2)	1.1411(2)	0.38004(18)	0.0294(7)
O3	0.5602(2)	0.8258(2)	0.40064(17)	0.0271(7)
O4	0.5019(2)	0.86529(19)	0.26379(17)	0.0259(7)
O5	0.4830(2)	1.0228(2)	0.24442(18)	0.0302(8)
O6	0.3994(2)	0.6808(2)	0.28220(18)	0.0267(7)
O7	0.4019(2)	0.8532(2)	0.11930(17)	0.0266(7)
O8	0.3091(2)	1.0237(2)	0.25761(18)	0.0292(7)
O9	0.2767(2)	0.7009(2)	0.16979(18)	0.0313(8)
O10	0.2555(2)	0.7180(2)	0.32722(18)	0.0318(8)
O11	0.2273(2)	0.8407(2)	0.14449(18)	0.0324(8)
O12	0.2075(2)	0.8593(2)	0.30147(19)	0.0327(8)
O13	0.1896(3)	1.1629(3)	0.4292(3)	0.0641(12)
O14	0.1440(3)	1.0296(3)	0.5314(2)	0.0550(10)
O15	0.4055(3)	0.5495(3)	0.4733(2)	0.0563(11)
O16	0.2849(3)	0.6022(3)	0.5693(2)	0.0477(9)
SI1	0.28469(9)	0.66028(8)	0.25068(8)	0.0230(3)
SI2	0.28338(9)	0.77507(9)	0.11196(8)	0.0233(3)
SI3	0.49740(9)	0.92757(9)	0.19727(8)	0.0232(3)
SI4	0.51493(9)	0.77862(9)	0.30388(8)	0.0237(3)
SI5	0.24371(9)	0.81734(9)	0.37549(8)	0.0225(3)
SI6	0.21497(9)	0.91045(9)	0.22633(8)	0.0230(3)
SI7	0.43366(9)	1.09654(9)	0.28305(8)	0.0236(3)
C11	0.1938(3)	0.5220(3)	0.2183(3)	0.0298(11)
C12	0.1691(4)	0.4749(3)	0.2902(3)	0.0431(14)
C13	0.0899(4)	0.3637(4)	0.2603(4)	0.070(2)
C14	0.1220(5)	0.3018(4)	0.1971(5)	0.077(2)
C15	0.1430(5)	0.3459(4)	0.1251(4)	0.071(2)
C16	0.2236(4)	0.4580(3)	0.1547(3)	0.0435(14)
C21	0.2264(3)	0.6975(3)	0.0018(3)	0.0259(10)
C22	0.1254(3)	0.6023(4)	–0.0114(3)	0.0417(13)
C23	0.0820(4)	0.5380(4)	–0.1027(3)	0.0533(15)
C24	0.1576(4)	0.5099(4)	–0.1371(3)	0.0586(16)
C25	0.2558(4)	0.6034(4)	–0.1280(3)	0.0535(16)
C26	0.3004(4)	0.6675(4)	–0.0367(3)	0.0435(14)
C31	0.6181(3)	0.9759(3)	0.1630(3)	0.0282(11)
C32	0.7065(3)	1.0554(4)	0.2358(3)	0.0432(13)
C33	0.8086(4)	1.0902(4)	0.2113(4)	0.0591(17)
C34	0.8077(4)	1.1310(4)	0.1366(4)	0.0564(16)
C35	0.7211(4)	1.0529(4)	0.0643(3)	0.0471(14)
C36	0.6195(3)	1.0190(4)	0.0888(3)	0.0367(12)
C41	0.5865(3)	0.7292(3)	0.2398(3)	0.0259(11)
C42	0.6122(4)	0.6533(4)	0.2710(3)	0.0400(13)
C43	0.6760(4)	0.6210(4)	0.2197(3)	0.0567(16)
C44	0.6228(5)	0.5749(4)	0.1282(3)	0.0546(16)
C45	0.5937(4)	0.6464(4)	0.0940(3)	0.0416(13)
C46	0.5315(3)	0.6804(3)	0.1467(3)	0.0336(12)
C51	0.1301(3)	0.7638(3)	0.4201(3)	0.0304(11)

Table S2 (continued). Atomic Coordinates and Equivalent Isotropic Displacement Parameters for Compound **2**.

Atom	x	y	z	U_{eq} / Å^2
C52	0.0369(3)	0.6775(4)	0.3547(3)	0.0404(13)
C53	–0.0569(4)	0.6363(4)	0.3916(4)	0.0617(17)
C54	–0.0344(4)	0.6036(5)	0.4669(4)	0.0638(18)
C55	0.0550(4)	0.6883(4)	0.5329(3)	0.0516(15)
C56	0.1492(4)	0.7298(4)	0.4961(3)	0.0403(13)
C61	0.0953(3)	0.9159(3)	0.1951(3)	0.0269(11)
C62	0.0053(3)	0.8102(3)	0.1546(3)	0.0456(14)
C63	–0.0934(4)	0.8151(4)	0.1329(4)	0.0622(17)
C64	–0.0859(4)	0.8846(4)	0.0779(4)	0.0540(15)
C65	0.0029(4)	0.9905(4)	0.1182(3)	0.0501(15)
C66	0.1007(4)	0.9853(3)	0.1388(3)	0.0383(12)
C71	0.4533(3)	1.1913(3)	0.2221(3)	0.0269(11)
C72	0.4094(4)	1.2618(3)	0.2554(3)	0.0393(13)
C73	0.4217(5)	1.3396(4)	0.2078(3)	0.0543(16)
C74	0.3766(4)	1.2885(4)	0.1156(3)	0.0510(15)
C75	0.4231(4)	1.2219(4)	0.0810(3)	0.0451(14)
C76	0.4103(4)	1.1421(3)	0.1281(3)	0.0369(12)
C100	0.1704(5)	1.1912(5)	0.3575(4)	0.084(2)
C101	0.1125(6)	1.1429(6)	0.4697(5)	0.092(3)
C102	0.1370(7)	1.1199(6)	0.5477(4)	0.103(3)
C103	0.1773(5)	1.0090(5)	0.6041(4)	0.0722(19)
C200	0.4264(5)	0.5006(5)	0.4019(4)	0.089(2)
C201	0.3309(4)	0.4789(4)	0.5038(4)	0.0551(16)
C202	0.3222(4)	0.5344(4)	0.5838(4)	0.0557(16)

Table S3. Selected Interatomic Distances (pm) for Compound **2**.

Atom 1	Atom 2	Distance
K1	O1	278.4(3)
K1	O1’	263.2(3)
K1	O2	274.4(3)
K1	O3	284.0(3)
K1	O4	273.0(3)
K1	O5	295.6(3)
K2	O1	284.4(3)
K2	O2	268.3(3)
K2	O3’	262.8(3)
K2	O8	317.7(3)
K2	O12	330.3(3)
K2	O13	280.4(4)
K2	O14	297.2(4)
K3	O1	317.8(3)
K3	O2’	262.1(3)
K3	O3	274.5(3)
K3	O6	318.0(3)
K3	O10	308.8(3)
K3	O15	280.1(3)
K3	O16	295.4(3)
SI1	O6	162.7(3)
SI1	O9	162.4(3)
SI1	O10	162.1(3)
SI2	O7	164.0(3)
SI2	O9	161.7(3)
SI2	O11	162.0(3)
SI3	O4	162.6(3)
SI3	O5	161.4(3)
SI3	O7	162.5(3)
SI4	O3	157.8(3)
SI4	O4	167.2(3)
SI4	O6	166.9(3)
SI5	O1	157.4(3)
SI5	O10	165.2(3)
SI5	O12	165.7(3)
SI6	O8	161.4(3)
SI6	O11	163.9(3)
SI6	O12	161.1(3)
SI7	O2	157.4(3)
SI7	O5	166.3(3)
SI7	O8	166.8(3)

‘1–x, 2–y, 1–z

Table S4. Selected Interatomic Angles (deg.) for Compound **2**.

Atom 1	Atom 2	Atom 3	Angle
O1	K1	O1’	105.76(8)
O1	K1	O2	86.04(9)
O1’	K1	O2	101.93(9)
O1	K1	O3	90.26(8)
O1’	K1	O3	93.43(9)
O1	K1	O4	97.09(9)
O1’	K1	O4	142.76(9)
O1	K1	O5	101.50(9)
O1’	K1	O5	142.95(9)
O2	K1	O3	164.63(9)
O2	K1	O4	108.74(9)
O2	K1	O5	55.27(8)
O3	K1	O4	56.91(8)
O3	K1	O5	111.20(8)
O4	K1	O5	54.44(8)
O1	K2	O2	86.00(8)
O1	K2	O3’	93.42(9)
O1	K2	O8	84.09(8)
O1	K2	O12	51.74(8)
O1	K2	O13	149.10(11)
O1	K2	O14	101.12(9)
O2	K2	O3’	90.23(9)
O2	K2	O8	53.70(8)
O2	K2	O12	89.49(8)
O2	K2	O13	114.80(10)
O2	K2	O14	172.87(10)
O3’	K2	O8	143.92(8)
O3’	K2	O12	145.09(8)
O3’	K2	O13	108.30(11)
O3’	K2	O14	89.05(10)
O8	K2	O12	47.62(7)
O8	K2	O13	90.34(10)
O8	K2	O14	126.82(10)
O12	K2	O13	103.41(11)
O12	K2	O14	95.16(9)
O13	K2	O14	58.84(11)
O1	K3	O2’	91.63(8)
O1	K3	O3	84.26(8)
O1	K3	O6	84.42(7)
O1	K3	O10	50.47(7)
O1	K3	O15	162.43(11)
O1	K3	O16	115.63(9)
O2’	K3	O3	89.03(9)
O2’	K3	O6	142.34(8)
O2’	K3	O10	142.03(9)
O2’	K3	O15	104.22(11)
O2’	K3	O16	87.10(9)
O3	K3	O6	53.32(8)
O3	K3	O10	89.15(8)
O3	K3	O15	103.27(9)
O3	K3	O16	159.83(10)
O6	K3	O10	49.50(7)
O6	K3	O15	87.47(9)
O6	K3	O16	128.21(9)
O10	K3	O15	113.09(10)
O10	K3	O16	105.92(9)
O15	K3	O16	58.77(10)
K1	O1	K1’	74.24(8)
K1	O1	K2	89.74(8)
K1’	O1	K2	86.49(8)
K1	O1	K3	86.81(8)
K1’	O1	K3	78.85(8)
K2	O1	K3	165.33(11)
K1	O1	SI5	127.10(15)
K1’	O1	SI5	157.16(16)
K2	O1	SI5	100.30(14)
K3	O1	SI5	93.13(12)
K1	O2	K2	94.06(9)
K1	O2	K3’	87.59(9)
K2	O2	K3’	90.33(9)
K1	O2	SI7	99.95(13)
K2	O2	SI7	107.86(15)
K3’	O2	SI7	159.58(16)
K1	O3	K2’	86.66(8)
K1	O3	K3	94.65(8)
K2’	O3	K3	88.85(9)
K1	O3	SI4	95.05(13)
K2’	O3	SI4	164.30(16)
K3	O3	SI4	106.53(14)
K1	O4	SI3	102.17(12)
K1	O4	SI4	96.90(13)
SI3	O4	SI4	159.74(18)
SI3	O5	SI7	163.6(2)
SI1	O6	SI4	136.78(17)
SI2	O7	SI3	133.64(18)
SI6	O8	SI7	147.90(18)
SI1	O9	SI2	161.8(2)
SI1	O10	SI5	150.20(19)
SI2	O11	SI6	145.1(2)
SI5	O12	SI6	158.3(2)
O6	SI1	O9	109.80(15)
O6	SI1	O10	107.86(17)
O9	SI1	O10	111.78(16)
O7	SI2	O9	109.41(16)
O7	SI2	O11	109.05(15)
O9	SI2	O11	108.81(16)
O4	SI3	O5	107.27(15)
O4	SI3	O7	109.17(15)
O5	SI3	O7	109.89(16)
O3	SI4	O4	109.56(15)

‘ 1–x, 2–y, 1–z.

Table S4 (continued). Selected Interatomic Angles (deg.) for Compound **2**.

Atom 1	Atom 2	Atom 3	Angle
O3	SI4	O6	111.65(15)
O4	SI4	O6	105.56(15)
O1	SI5	O10	111.87(16)
O1	SI5	O12	114.24(16)
O10	SI5	O12	106.01(16)
O8	SI6	O11	111.15(15)
O8	SI6	O12	108.63(17)
O11	SI6	O12	110.59(16)
O2	SI7	O5	109.94(16)
O2	SI7	O8	111.72(16)
O5	SI7	O8	106.58(15)

Table S5. Derived H Atom Coordinates and Isotropic Displacement Parameters for Compound **2**.

Atom	x	y	z	U_{iso} / Å^2
H10A	0.226989	1.204.872	0.330282	0.100
H10B	0.108657	1.135.800	0.319087	0.100
H10C	0.162349	1.252.690	0.373142	0.100
H10D	0.050211	1.084.137	0.433024	0.111
H10E	0.099985	1.202.575	0.482547	0.111
H10F	0.201823	1.176.568	0.582566	0.123
H10G	0.084190	1.113.891	0.579034	0.123
H10H	0.180515	0.944792	0.588473	0.087
H10I	0.244243	1.064.414	0.633740	0.087
H10J	0.129995	1.003.525	0.640330	0.087
H11	0.129199	0.515665	0.189016	0.036
H12A	0.231355	0.480649	0.322171	0.052
H12B	0.144667	0.513788	0.328127	0.052
H13A	0.025079	0.358191	0.234199	0.084
H13B	0.080062	0.335524	0.308296	0.084
H14A	0.183200	0.301653	0.224699	0.092
H14B	0.067843	0.230436	0.176169	0.092
H15A	0.080136	0.340282	0.094521	0.086
H15B	0.166071	0.306262	0.086440	0.086
H16A	0.233107	0.485530	0.106325	0.052
H16B	0.288406	0.462938	0.180214	0.052
H20A	0.478190	0.552007	0.382844	0.107
H20B	0.450437	0.454135	0.416012	0.107
H20C	0.364825	0.462030	0.357554	0.107
H20D	0.349688	0.426162	0.512634	0.066
H20E	0.265460	0.444780	0.462926	0.066
H20F	0.276059	0.485062	0.609058	0.067
H20G	0.389081	0.573074	0.622912	0.067
H20H	0.244972	0.698914	0.631676	0.078
H20I	0.222285	0.602383	0.665775	0.078
H20J	0.335172	0.691945	0.684615	0.078
H21	0.213290	0.741514	–0.029374	0.031
H22A	0.134887	0.559867	0.022611	0.050
H22B	0.076413	0.623454	0.008316	0.050
H23A	0.019884	0.475152	–0.107034	0.064
H23B	0.064027	0.576863	–0.135937	0.064
H24A	0.170411	0.465446	–0.107290	0.070
H24B	0.129270	0.471317	–0.196640	0.070

Table S5 (continued). Derived H Atom Coordinates and Isotropic Displacement Parameters for Compound **2**.

Atom	x	y	z	U_{iso} / Å^2
H25A	0.244149	0.645011	–0.161785	0.064
H25B	0.304255	0.582321	–0.149076	0.064
H26A	0.362495	0.729997	–0.033089	0.052
H26B	0.319172	0.628304	–0.004339	0.052
H31	0.631628	0.916577	0.145875	0.034
H32A	0.695558	1.115.366	0.254992	0.052
H32B	0.707848	1.025.918	0.282662	0.052
H33A	0.862435	1.143.927	0.258798	0.071
H33B	0.823318	1.031.962	0.197909	0.071
H34A	0.872182	1.148.279	0.119415	0.068
H34B	0.800949	1.193.970	0.152113	0.068
H35A	0.731728	0.992800	0.045154	0.057
H35B	0.720122	1.082.649	0.017546	0.057
H36A	0.605853	1.077.818	0.102963	0.044
H36B	0.565453	0.966369	0.040844	0.044
H41	0.651752	0.789179	0.242932	0.031
H42A	0.649377	0.684792	0.329912	0.048
H42B	0.549049	0.592250	0.268009	0.048
H43A	0.741639	0.680981	0.226342	0.068
H43B	0.689135	0.570437	0.240525	0.068
H44A	0.561453	0.510254	0.121026	0.066
H44B	0.667665	0.559198	0.096093	0.066
H45A	0.554015	0.611239	0.035962	0.050
H45B	0.655420	0.706942	0.093678	0.050
H46A	0.466089	0.620701	0.141245	0.040
H46B	0.517779	0.730234	0.125341	0.040
H51	0.113149	0.820373	0.439267	0.037
H52A	0.051868	0.621189	0.331985	0.048
H52B	0.022308	0.703249	0.308255	0.048
H53A	–0.077404	0.689929	0.407810	0.074
H53B	–0.113297	0.577556	0.349010	0.074
H54A	–0.094342	0.580460	0.490998	0.077
H54B	–0.020761	0.545153	0.449132	0.077
H55A	0.068963	0.662396	0.579385	0.062
H55B	0.039125	0.744165	0.555059	0.062
H56A	0.205450	0.788090	0.539179	0.048
H56B	0.169646	0.675943	0.480338	0.048
H61	0.080559	0.945437	0.247510	0.032
H62A	–0.001461	0.767740	0.192999	0.055
H62B	0.017991	0.777187	0.103203	0.055
H63A	–0.148313	0.745705	0.103862	0.075
H63B	–0.110385	0.840374	0.184890	0.075
H64A	–0.149035	0.889895	0.068748	0.065
H64B	–0.077248	0.855003	0.023139	0.065
H65A	0.009067	1.033.017	0.079842	0.060
H65B	–0.009717	1.023.136	0.169777	0.060
H66A	0.155916	1.054.828	0.167042	0.046
H66B	0.116689	0.959444	0.086460	0.046
H71	0.527564	1.234.556	0.230884	0.032
H72A	0.336860	1.219.967	0.252103	0.047
H72B	0.443100	1.298.121	0.314701	0.047

Table S5 (continued). Derived H Atom Coordinates and Isotropic Displacement Parameters for Compound **2**.

Atom	**x**	**y**	**z**	U_{iso} / Å^2
H73A	0.388438	1.379.969	0.229606	0.065
H73B	0.494229	1.386.895	0.216433	0.065
H74A	0.388620	1.340.943	0.085947	0.061
H74B	0.302894	1.246.094	0.106276	0.061
H75A	0.495726	1.265.295	0.085709	0.054
H75B	0.390693	1.186.789	0.021313	0.054
H76A	0.337808	1.093.874	0.117834	0.044
H76B	0.444811	1.103.022	0.106444	0.044

STRUCTURE REPORT for Compound 3

Crystallographer: A. Fischer, P. Liebing
ID code: ULO **Date:** 20 December 2016
Compound: $(Cy_7Si_7O_{12})_2U$ (**3**)
Formula sum: $C_{90}H_{170}N_2O_{24}Si_{14}U$ [+ solvent]
Formula moieties: $C_{84}H_{154}O_{24}Si_{14}U$, $C_6H_{16}N_2$, [solvent]

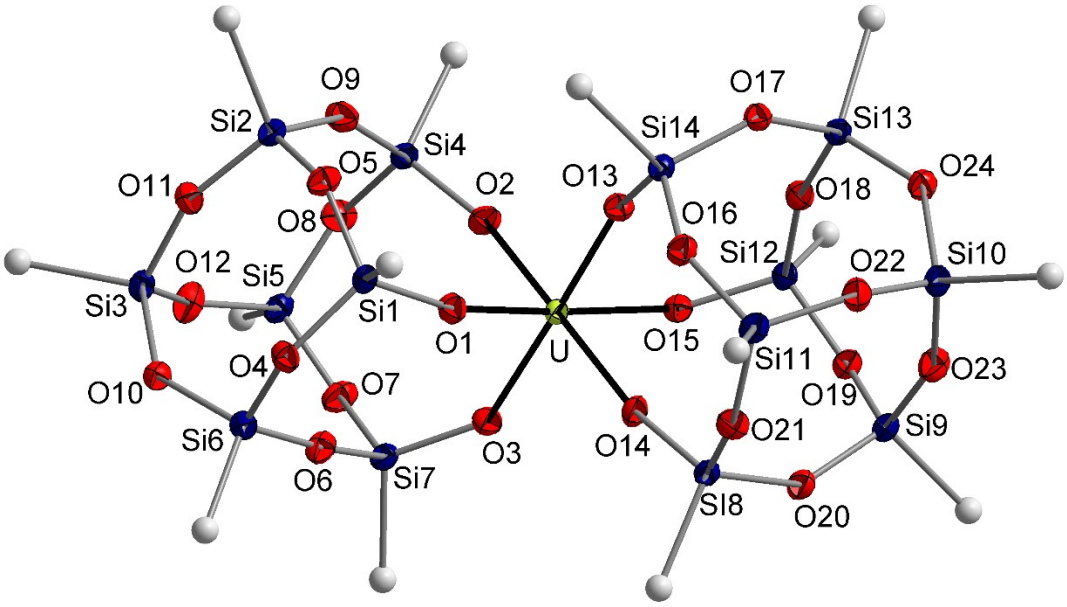

Figure S4. Molecular structure of **3** in the crystal showing the atom labeling scheme (displacement ellipsoids of U, Si and O drawn at the 50% probability level, H atoms and peripheral C atoms of the cyclohexyl groups omitted for clarity).

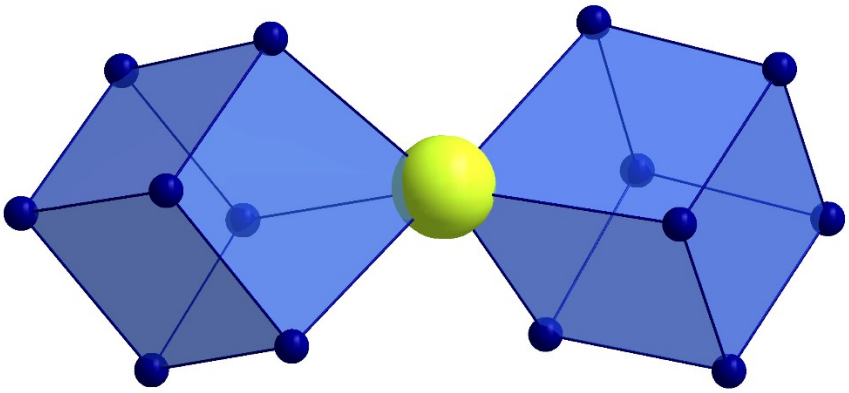

Figure S5. Representation of the interconnection of the two cube-shaped Si_7U frameworks in compound **3**.

List of Tables

Table S6. Crystallographic Data and Details on Structure Refinement for Compound **3**.

formula sum	$C_{90}H_{170}N_2O_{24}Si_{14}U$ [+ solvent]
formula weight	2295.56 [+ solvent]
crystal color / shape / size (mm)	colorless plates / 0.24 × 0.26 × 0.04
crystal system	monoclinic
space group	$P2_1/c$
unit cell parameters	
a (Å)	25.601(1)
b (Å)	15.067(1)
c (Å)	30.835(1)
α (deg)	90
β (deg)	102.059(3)
γ (deg)	90
unit cell volume V (Å^3)	11632(1)
molecules per cell z	4
crystallographic density ρ_{calcd} (g cm^{-3})	1.311
absorption coefficient μ (mm^{-1})	1.603
diffractometer	Bruker Smart CCD System
radiation (λ [Å])	graphite-monochromated Mo-K_α (0.71073)
temperature (°C)	–130(2)
scan type[c]	ω scans
completeness of dataset	99.9%
θ range of data collection (deg)	1.424 … 25.000
reflections collected[c]	71138 ($-23 \le h \le 32$, $-18 \le k \le 19$, $-39 \le l \le 25$)
independent reflections	25375 (R_{int} = 0.0513)
independent reflections with $I>2\sigma(I)$	18932
structure solution method	heavy atom methods (SHELXS-2013[d])
refinement method	full-matrix least-squares on F^2 (SHELXL-2016/4[d])
absorption correction method[c]	multi-scan
range of transmission factors	0.76 … 1.00
data / parameters / restraints	25375 / 1323 / 307
goodness of fit (GooF) [all data]	1.070
final R values	
R_1 [all data, $I \ge 2\sigma(I)$]	0.0707, 0.0415
wR_2 [all data, $I \ge 2\sigma(I)$]]	0.0851, 0.0747
largest difference peak and hole	0.831 and –1.289 e Å^{-3}

Refinement special details: Reflections (0 0 2) and (1 0 0) are strongly disagreeing with the structure model and were therefore omitted for refinement. The cyclohexyl groups C61–C66, C91–C96 and C111–C116 are disordered over each two orientations. The site occupancy factors of the disordered atoms were refined freely, and restraints on C-C distances and displacement parameters were applied on the same (SADI, SIMU and EADP restraints in ShelXL[d]). C61 was refined as not disordered (EXYZ restraint in ShelXL[d]). Diffuse solvent electron density was considered using the SQUEEZE routine of the software Platon[e]. The

solvent content of the crystals was calculated to approx. 0.5 molecules of *n*-hexane per formula unit.

[c] Bruker (2001). *AXS SMART and SADABS.* Bruker AXS Inc., Madison, Wisconsin, USA.

[d] G. M. Sheldrick, G. M. *Acta Cryst.* 2015, **C71**, 3–8.

[e] A. L. Spek, *Acta Cryst.* 2015, **C71**, 9-18.

Table S7. Atomic Coordinates and Equivalent Isotropic Displacement Parameters for Compound **3**.

Atom	x	y	z	U_{eq} / Å^2
U	0.26227(2)	0.55705(2)	0.26761(2)	0.01462(4)
O1	0.28835(10)	0.44085(16)	0.23963(7)	0.0205(5)
O2	0.19201(10)	0.56336(17)	0.21788(8)	0.0225(6)
O3	0.29914(11)	0.63986(16)	0.22490(8)	0.0214(6)
O4	0.32713(10)	0.36802(15)	0.17506(8)	0.0206(6)
O5	0.22994(10)	0.32364(16)	0.18491(8)	0.0228(6)
O6	0.33222(11)	0.54116(15)	0.16408(8)	0.0238(6)
O7	0.25848(11)	0.66575(17)	0.13918(8)	0.0267(6)
O8	0.16028(11)	0.60196(17)	0.13230(8)	0.0263(6)
O9	0.15073(10)	0.43818(17)	0.16022(8)	0.0248(6)
O10	0.30245(10)	0.43773(17)	0.09449(7)	0.0231(6)
O11	0.20662(11)	0.38388(16)	0.10272(8)	0.0231(6)
O12	0.22015(11)	0.54719(16)	0.07679(8)	0.0270(6)
O13	0.22865(10)	0.47179(15)	0.30753(8)	0.0205(6)
O14	0.33252(10)	0.56841(16)	0.31726(7)	0.0214(6)
O15	0.23382(10)	0.67276(15)	0.30367(7)	0.0184(5)
O16	0.29622(10)	0.37583(16)	0.36773(8)	0.0223(6)
O17	0.20070(10)	0.42203(15)	0.38101(7)	0.0183(5)
O18	0.19732(10)	0.59790(15)	0.37132(8)	0.0206(6)
O19	0.27190(10)	0.72251(16)	0.38693(8)	0.0208(6)
O20	0.36887(10)	0.65694(16)	0.39212(8)	0.0232(6)
O21	0.38066(10)	0.48273(16)	0.38980(8)	0.0230(6)
O22	0.32056(10)	0.44630(17)	0.44781(8)	0.0245(6)
O23	0.31580(11)	0.62120(16)	0.45576(8)	0.0258(6)
O24	0.22801(10)	0.52601(16)	0.45075(8)	0.0226(6)
N1	0.28219(13)	0.8268(2)	0.28420(10)	0.0240(7)
N2	0.17315(15)	0.9152(2)	0.23013(10)	0.0303(8)
SI1	0.29004(4)	0.35103(6)	0.21145(3)	0.0168(2)
SI2	0.18185(4)	0.35468(6)	0.14497(3)	0.0179(2)
SI3	0.23869(4)	0.44515(7)	0.07400(3)	0.0202(2)
SI4	0.14801(4)	0.54214(6)	0.17331(3)	0.0175(2)
SI5	0.20504(4)	0.63141(7)	0.10502(3)	0.0198(2)
SI6	0.34170(4)	0.44569(7)	0.14323(3)	0.0202(2)
SI7	0.31213(4)	0.63895(7)	0.17569(3)	0.0193(2)
SI8	0.37947(4)	0.57329(7)	0.36110(3)	0.0182(2)
SI9	0.32717(4)	0.69525(7)	0.42075(3)	0.0191(2)
SI10	0.29266(4)	0.52720(7)	0.46955(3)	0.0211(2)
SI11	0.34523(4)	0.40635(7)	0.40739(3)	0.0202(2)
SI12	0.21831(4)	0.68815(6)	0.35193(3)	0.0171(2)

Table S7 (continued). Atomic Coordinates and Equivalent Isotropic Displacement Parameters for Compound **3**.

Atom	x	y	z	U_{eq} / Å^2
SI13	0.18730(4)	0.51587(6)	0.40276(3)	0.0170(2)
SI14	0.23401(4)	0.39211(6)	0.34355(3)	0.0174(2)
C11	0.31649(15)	0.2530(2)	0.24530(11)	0.0189(8)
C12	0.32516(18)	0.1737(2)	0.21624(13)	0.0312(10)
C13	0.34065(19)	0.0902(3)	0.24412(15)	0.0380(11)
C14	0.38965(19)	0.1060(3)	0.28057(15)	0.0423(12)
C15	0.3821(2)	0.1847(3)	0.30918(14)	0.0443(12)
C16	0.36665(18)	0.2686(3)	0.28138(13)	0.0327(10)
C21	0.13248(16)	0.2641(2)	0.12905(12)	0.0234(8)
C22	0.0871(2)	0.2935(3)	0.09083(16)	0.0472(14)
C23	0.0464(2)	0.2188(3)	0.07632(18)	0.0582(16)
C24	0.0731(2)	0.1364(4)	0.06443(17)	0.0623(17)
C25	0.1157(2)	0.1058(3)	0.10298(19)	0.0570(15)
C26	0.15774(19)	0.1784(3)	0.11759(16)	0.0409(11)
C31	0.22325(17)	0.4045(3)	0.01580(11)	0.0251(9)
C32	0.16711(19)	0.4300(3)	-0.00832(13)	0.0383(11)
C33	0.1528(2)	0.3907(3)	-0.05501(14)	0.0437(13)
C34	0.1585(2)	0.2908(3)	-0.05348(14)	0.0397(12)
C35	0.2151(2)	0.2649(3)	-0.03264(14)	0.0460(13)
C36	0.2313(2)	0.3036(3)	0.01468(13)	0.0410(12)
C41	0.07975(15)	0.5620(2)	0.18280(12)	0.0233(8)
C42	0.06798(18)	0.4940(3)	0.21751(14)	0.0348(10)
C43	0.01344(19)	0.5093(3)	0.22912(16)	0.0430(12)
C44	0.00889(19)	0.6022(3)	0.24593(15)	0.0414(11)
C45	0.0173(2)	0.6705(3)	0.21070(17)	0.0477(13)
C46	0.07220(18)	0.6569(3)	0.19779(15)	0.0355(10)
C51	0.17679(16)	0.7214(2)	0.06592(11)	0.0215(8)
C52	0.15159(19)	0.7959(2)	0.08849(13)	0.0335(11)
C53	0.1250(2)	0.8679(3)	0.05647(14)	0.0385(11)
C54	0.0843(2)	0.8291(3)	0.01865(15)	0.0443(12)
C55	0.1107(2)	0.7599(3)	-0.00535(14)	0.0572(17)
C56	0.1366(2)	0.6856(3)	0.02617(14)	0.0419(12)
C61A	0.41163(16)	0.4319(3)	0.13868(12)	0.0379(10)
C61B	0.41163(16)	0.4319(3)	0.13868(12)	0.0379(10)
C62A	0.4551(5)	0.4153(14)	0.1789(4)	0.0359(18)
C62B	0.4480(3)	0.4344(9)	0.1847(3)	0.0359(18)
C63A	0.5101(6)	0.3895(11)	0.1733(5)	0.046(3)
C63B	0.5065(4)	0.4264(6)	0.1812(3)	0.040(2)
C64A	0.5270(4)	0.4447(10)	0.1382(3)	0.056(3)
C64B	0.5154(3)	0.3471(6)	0.1533(3)	0.055(2)
C65A	0.4868(3)	0.4423(10)	0.0957(3)	0.048(3)
C65B	0.4789(3)	0.3486(7)	0.1080(2)	0.056(2)
C66A	0.4315(4)	0.4668(8)	0.1003(3)	0.040(3)
C66B	0.4203(3)	0.3561(5)	0.1101(2)	0.0416(19)
C71	0.36562(16)	0.7196(2)	0.17043(12)	0.0215(8)
C72	0.41821(18)	0.7019(3)	0.20413(15)	0.0360(11)
C73	0.4629(2)	0.7657(3)	0.19746(19)	0.0551(15)
C74	0.4718(2)	0.7614(3)	0.1503(2)	0.0600(16)
C75	0.4207(2)	0.7803(3)	0.11657(18)	0.0502(14)
C76	0.37581(18)	0.7180(3)	0.12277(13)	0.0310(10)
C81	0.44491(16)	0.5896(3)	0.34573(12)	0.0252(9)

Table S7 (continued). Atomic Coordinates and Equivalent Isotropic Displacement Parameters for Compound **3**.

Atom	x	y	z	U_{eq} / Å^2
C82	0.49196(19)	0.5888(4)	0.38564(16)	0.0607(17)
C83	0.5456(2)	0.6047(5)	0.37181(19)	0.0719(19)
C84	0.55449(18)	0.5391(3)	0.33727(15)	0.0426(12)
C85	0.50863(19)	0.5404(4)	0.29737(15)	0.0462(13)
C86	0.45522(18)	0.5239(3)	0.31057(14)	0.0347(10)
C91A	0.3552(5)	0.8003(16)	0.4493(7)	0.027(3)
C91B	0.3571(7)	0.7905(17)	0.4536(8)	0.026(3)
C92A	0.3196(9)	0.8364(16)	0.4791(7)	0.030(3)
C92B	0.3249(11)	0.8261(17)	0.4862(8)	0.031(4)
C93A	0.3429(6)	0.9196(15)	0.5040(7)	0.0414(16)
C93B	0.3486(7)	0.9107(16)	0.5096(8)	0.0414(16)
C94A	0.4030(3)	0.9183(6)	0.5235(3)	0.041(2)
C94B	0.3672(4)	0.9786(6)	0.4791(3)	0.043(2)
C95A	0.4342(4)	0.8897(5)	0.4888(3)	0.040(2)
C95B	0.4059(4)	0.9367(5)	0.4535(3)	0.044(2)
C96A	0.4148(3)	0.7995(5)	0.4693(3)	0.035(2)
C96B	0.3793(4)	0.8587(5)	0.4258(3)	0.033(2)
C101	0.30593(16)	0.5137(2)	0.53081(11)	0.0214(8)
C102	0.28291(18)	0.4248(2)	0.54335(12)	0.0290(10)
C103	0.29216(18)	0.4117(3)	0.59344(12)	0.0305(10)
C104	0.26905(19)	0.4894(3)	0.61520(13)	0.0352(10)
C105	0.29260(19)	0.5770(3)	0.60417(13)	0.0348(11)
C106	0.28367(18)	0.5903(3)	0.55386(12)	0.0293(9)
C111	0.3898(7)	0.3127(15)	0.4225(7)	0.028(3)
C112	0.4465(6)	0.3342(10)	0.4491(4)	0.053(3)
C113	0.4449(5)	0.3431(7)	0.4988(3)	0.063(3)
C114	0.4219(4)	0.2585(6)	0.5158(3)	0.061(3)
C115	0.3671(4)	0.2376(7)	0.4862(3)	0.054(3)
C116	0.3661(9)	0.2263(11)	0.4361(4)	0.045(3)
C121	0.16580(16)	0.7751(2)	0.34842(12)	0.0212(8)
C122	0.10978(18)	0.7384(3)	0.32866(15)	0.0369(11)
C123	0.06566(19)	0.8086(3)	0.32523(14)	0.0429(12)
C124	0.06588(19)	0.8502(3)	0.36979(14)	0.0419(12)
C125	0.1197(2)	0.8879(3)	0.38985(16)	0.0497(14)
C126	0.16481(19)	0.8193(3)	0.39318(14)	0.0399(12)
C131	0.11660(15)	0.5130(2)	0.40774(12)	0.0206(8)
C132	0.08068(16)	0.4938(3)	0.36203(12)	0.0291(9)
C133	0.02143(18)	0.4909(3)	0.36364(14)	0.0425(12)
C134	0.01129(19)	0.4227(3)	0.39678(15)	0.0483(13)
C135	0.04564(19)	0.4394(4)	0.44273(14)	0.0497(13)
C136	0.10506(17)	0.4455(3)	0.44176(13)	0.0326(9)
C141	0.20678(15)	0.2886(2)	0.31533(12)	0.0204(8)
C142	0.14644(18)	0.2934(3)	0.29675(16)	0.0394(11)
C143	0.1263(2)	0.2098(3)	0.27072(17)	0.0542(15)
C144	0.1389(2)	0.1277(3)	0.29889(17)	0.0465(13)
C145	0.1990(2)	0.1201(3)	0.31775(15)	0.0389(11)
C146	0.22083(19)	0.2037(2)	0.34292(14)	0.0328(10)
C211	0.3902(8)	0.3071(15)	0.4252(7)	0.043(4)
C212	0.4363(5)	0.3297(9)	0.4638(4)	0.057(3)
C213	0.4733(4)	0.2498(6)	0.4755(4)	0.067(3)
C214	0.4429(4)	0.1689(7)	0.4864(4)	0.067(3)

Table S7 (continued). Atomic Coordinates and Equivalent Isotropic Displacement Parameters for Compound **3**.

Atom	x	y	z	U_{eq} / Å^2
C215	0.3970(4)	0.1474(6)	0.4487(4)	0.071(3)
C216	0.3615(8)	0.2303(10)	0.4419(6)	0.065(4)
C301	0.26373(18)	0.8457(3)	0.23526(13)	0.0313(10)
C302	0.20350(19)	0.8395(3)	0.21980(13)	0.0330(10)
C303	0.34046(18)	0.8058(3)	0.29514(15)	0.0355(10)
C304	0.27007(19)	0.9029(2)	0.31192(13)	0.0297(10)
C305	0.1833(2)	0.9942(3)	0.20517(15)	0.0406(11)
C306	0.11625(19)	0.8945(3)	0.21950(15)	0.0444(12)

Table S8. Selected Interatomic Distances (pm) for Compound **2**.

Atom 1	Atom 2	Distance
U	O1	212.0(2)
U	O2	210.8(2)
U	O3	216.8(2)
U	O13	208.5(2)
U	O14	211.1(2)
U	O15	226.8(2)
SI1	O1	161.4(2)
SI1	O4	163.4(3)
SI1	O5	163.8(3)
SI2	O5	161.8(3)
SI2	O9	161.1(3)
SI2	O11	162.4(3)
SI3	O10	162.6(3)
SI3	O11	161.7(3)
SI3	O12	161.7(3)
SI4	O2	161.6(2)
SI4	O8	163.6(3)
SI4	O9	162.3(3)
SI5	O7	162.7(3)
SI5	O8	161.9(3)
SI5	O12	163.0(3)
SI6	O4	162.0(3)
SI6	O6	161.5(3)
SI6	O10	162.8(2)

Atom 1	Atom 2	Distance
SI7	O3	162.0(3)
SI7	O6	162.5(3)
SI7	O7	163.4(3)
SI8	O14	161.2(2)
SI8	O20	163.9(3)
SI8	O21	162.3(3)
SI9	O19	162.5(3)
SI9	O20	162.7(3)
SI9	O23	162.0(3)
SI10	O22	162.6(3)
SI10	O23	162.6(3)
SI10	O24	163.5(3)
SI11	O16	162.5(3)
SI11	O21	162.7(3)
SI11	O22	162.6(3)
SI12	O15	163.5(3)
SI12	O18	162.2(3)
SI12	O19	164.2(3)
SI13	O17	163.2(2)
SI13	O18	162.4(3)
SI13	O24	162.9(3)
SI14	O13	162.2(2)
SI14	O16	163.0(3)
SI14	O17	163.6(3)

Table S9. Selected Interatomic Angles (deg.) for Compound **3**.

Atom 1	Atom 2	Atom 3	Angle
O1	U	O2	91.99(9)
O1	U	O3	90.80(9)
O1	U	O13	86.30(9)
O1	U	O14	93.96(9)
O1	U	O15	174.14(9)
O2	U	O3	86.89(10)
O2	U	O13	93.59(10)
O2	U	O14	172.76(9)
O2	U	O15	90.54(9)
O3	U	O13	177.07(9)
O3	U	O14	88.90(9)
O3	U	O15	94.62(9)
O13	U	O14	90.91(10)
O13	U	O15	88.27(9)
O14	U	O15	83.94(9)
U	O1	SI1	163.27(16)
U	O2	SI4	161.21(16)
U	O3	SI7	139.88(14)
SI1	O4	SI6	140.30(16)
SI1	O5	SI2	142.54(17)
SI6	O6	SI7	167.52(18)
SI5	O7	SI7	147.08(18)
SI4	O8	SI5	145.47(19)
SI2	O9	SI4	151.17(18)
SI3	O10	SI6	136.81(16)
SI2	O11	SI3	157.21(18)
SI3	O12	SI5	150.89(17)
U	O13	SI14	150.93(16)
U	O14	SI8	169.99(16)
U	O15	SI12	135.98(13)
SI11	O16	SI14	145.98(17)
SI13	O17	SI14	135.59(16)
SI12	O18	SI13	164.61(17)
SI9	O19	SI12	146.91(16)
SI8	O20	SI9	142.86(18)
SI8	O21	SI11	145.90(18)
SI10	O22	SI11	150.19(18)
SI9	O23	SI10	154.17(17)
SI10	O24	SI13	136.96(17)
O1	SI1	O4	108.85(13)
O1	SI1	O5	110.63(14)
O4	SI1	O5	108.44(13)
O5	SI2	O9	110.35(14)
O5	SI2	O11	109.04(15)
O9	SI2	O11	108.80(14)
O10	SI3	O11	109.35(14)
O10	SI3	O12	109.07(14)
O11	SI3	O12	108.99(15)
O2	SI4	O8	109.63(14)
O2	SI4	O9	110.12(14)
O8	SI4	O9	108.32(14)
O7	SI5	O8	109.97(14)
O7	SI5	O12	109.39(15)
O8	SI5	O12	109.67(15)
O4	SI6	O6	109.23(13)
O4	SI6	O10	109.28(14)
O6	SI6	O10	108.55(14)
O3	SI7	O6	110.51(13)
O3	SI7	O7	109.62(15)
O6	SI7	O7	109.36(14)
O14	SI8	O20	109.83(14)
O14	SI8	O21	110.16(14)
O20	SI8	O21	108.35(14)
O19	SI9	O20	108.87(13)
O19	SI9	O23	110.36(15)
O20	SI9	O23	110.30(14)
O22	SI10	O23	109.26(15)
O22	SI10	O24	110.16(14)
O23	SI10	O24	108.30(14)
O16	SI11	O21	110.24(14)
O16	SI11	O22	108.61(14)
O21	SI11	O22	109.07(14)
O15	SI12	O18	112.27(13)
O15	SI12	O19	108.33(14)
O18	SI12	O19	108.53(13)
O17	SI13	O18	109.94(13)
O17	SI13	O24	107.76(13)
O18	SI13	O24	108.66(14)
O13	SI14	O16	111.06(14)
O13	SI14	O17	107.31(13)
O16	SI14	O17	108.79(13)

Table S10. Derived H Atom Coordinates and Isotropic Displacement Parameters for Compound **3**.

Atom	x	y	z	U_{iso} / Å^2
H01R	0.280848	0.802926	0.218217	0.038
H01S	0.275520	0.906035	0.228912	0.038
H02A	0.244077	0.423005	0.530629	0.035
H02B	0.299840	0.375308	0.530232	0.035
H02R	0.195243	0.830919	0.187246	0.040
H02S	0.191025	0.785947	0.233245	0.040
H03A	0.330955	0.406986	0.605923	0.037
H03B	0.275031	0.355808	0.599969	0.037
H03R	0.347265	0.752596	0.278920	0.043
H03S	0.351799	0.795303	0.327084	0.043
H03T	0.360579	0.855901	0.286583	0.043
H04A	0.229771	0.490718	0.604733	0.042
H04B	0.276863	0.480959	0.647759	0.042
H04R	0.232799	0.921141	0.301633	0.036
H04S	0.293695	0.952833	0.309186	0.036
H04T	0.275818	0.884390	0.343023	0.036
H05A	0.331369	0.577964	0.617185	0.042
H05B	0.275697	0.626380	0.617390	0.042
H05R	0.161590	1.043.775	0.212092	0.049
H05S	0.173779	0.981573	0.173318	0.049
H05T	0.221182	1.010.012	0.213464	0.049
H06A	0.244906	0.596016	0.541465	0.035
H06B	0.301070	0.646220	0.547696	0.035
H06R	0.095978	0.945260	0.227146	0.053
H06S	0.109383	0.842406	0.236490	0.053
H06T	0.105140	0.881992	0.187717	0.053
H101	0.345515	0.512202	0.541918	0.026
H11	0.287876	0.234560	0.261094	0.023
H111	0.397427	0.295834	0.393046	0.034
H121	0.173667	0.822479	0.327978	0.025
H12A	0.353738	0.188229	0.200170	0.037
H12B	0.291946	0.162275	0.193974	0.037
H12K	0.459244	0.390412	0.438278	0.064
H12L	0.471468	0.286303	0.445051	0.064
H12M	0.456683	0.380477	0.455504	0.068
H12N	0.421856	0.347167	0.489934	0.068
H131	0.107112	0.573249	0.417334	0.025
H13A	0.348010	0.041445	0.224740	0.046
H13B	0.310431	0.071895	0.257538	0.046
H13K	0.481486	0.353546	0.516249	0.076
H13L	0.422593	0.394747	0.503067	0.076
H13M	0.490172	0.235938	0.450214	0.081
H13N	0.501969	0.264703	0.501344	0.081
H141	0.223768	0.282183	0.289089	0.024
H14A	0.420948	0.116723	0.267089	0.051
H14B	0.396941	0.052299	0.299346	0.051
H14K	0.446522	0.208119	0.515204	0.073
H14L	0.418069	0.267393	0.546788	0.073
H14M	0.467427	0.117379	0.492113	0.080
H14N	0.429206	0.180401	0.513583	0.080
H15A	0.353759	0.170803	0.325635	0.053

Table S10 (continued). Derived H Atom Coordinates and Isotropic Displacement Parameters for Compound **3**.

Atom	x	y	z	U_{iso} / Å^2
H15B	0.415617	0.195612	0.331125	0.053
H15K	0.342077	0.285914	0.489775	0.064
H15L	0.353398	0.182276	0.497178	0.064
H15M	0.376744	0.095622	0.456155	0.085
H15N	0.410090	0.133742	0.421409	0.085
H16A	0.360067	0.317635	0.300912	0.039
H16B	0.396562	0.286211	0.267419	0.039
H16K	0.387853	0.174633	0.430955	0.054
H16L	0.329062	0.217892	0.419188	0.054
H16M	0.328085	0.217183	0.420217	0.078
H16N	0.352001	0.246841	0.470345	0.078
H21	0.116275	0.251742	0.155256	0.028
H211	0.405167	0.286508	0.399489	0.051
H22A	0.102325	0.311954	0.065259	0.057
H22B	0.068622	0.345419	0.100407	0.057
H22K	0.109485	0.714438	0.298714	0.044
H22L	0.101974	0.688805	0.347381	0.044
H23A	0.019301	0.238671	0.050375	0.070
H23B	0.027901	0.205354	0.100702	0.070
H23K	0.071086	0.855290	0.304011	0.051
H23L	0.030498	0.780623	0.313766	0.051
H24A	0.046180	0.088914	0.056029	0.075
H24B	0.089419	0.148520	0.038613	0.075
H24K	0.038797	0.898047	0.366234	0.050
H24L	0.056148	0.804913	0.389972	0.050
H25A	0.098956	0.090001	0.128142	0.068
H25B	0.133212	0.051962	0.094331	0.068
H25K	0.119209	0.910638	0.419884	0.060
H25L	0.127117	0.938598	0.371612	0.060
H26A	0.176941	0.189746	0.093406	0.049
H26B	0.184118	0.157540	0.143801	0.049
H26K	0.160259	0.772940	0.414846	0.048
H26L	0.199521	0.848927	0.404394	0.048
H31	0.249104	0.432912	–0.000180	0.030
H32A	0.141085	0.408843	0.009026	0.046
H32B	0.164379	0.495464	–0.010283	0.046
H32K	0.086444	0.540298	0.340847	0.035
H32L	0.091122	0.436107	0.350986	0.035
H33A	0.176583	0.415997	–0.073357	0.052
H33B	0.115554	0.406668	–0.068938	0.052
H33K	0.009941	0.550053	0.372022	0.051
H33L	0.000155	0.475981	0.333882	0.051
H34A	0.148920	0.266496	–0.083943	0.048
H34B	0.133782	0.265239	–0.036060	0.048
H34K	0.019130	0.362781	0.386566	0.058
H34L	–0.026885	0.424337	0.398493	0.058
H35A	0.218137	0.199445	–0.031355	0.055
H35B	0.239619	0.287449	–0.051056	0.055
H35K	0.040132	0.390637	0.462821	0.060
H35L	0.034179	0.495425	0.454694	0.060
H36A	0.269389	0.289755	0.026823	0.049

Table S10 (continued). Derived H Atom Coordinates and Isotropic Displacement Parameters for Compound **3**.

Atom	x	y	z	U_{iso} / Å^2
H36B	0.209869	0.274750	0.033928	0.049
H36K	0.117844	0.386347	0.434655	0.039
H36L	0.125259	0.462515	0.471585	0.039
H41	0.053709	0.551456	0.154240	0.028
H42A	0.096126	0.498659	0.244830	0.042
H42B	0.069231	0.433231	0.205594	0.042
H42K	0.127607	0.300720	0.321487	0.047
H42L	0.138344	0.345785	0.277106	0.047
H43A	0.008339	0.466194	0.252111	0.052
H43B	–0.015037	0.499194	0.202459	0.052
H43K	0.087208	0.214164	0.259753	0.065
H43L	0.143159	0.204975	0.244684	0.065
H44A	–0.026861	0.610758	0.252801	0.050
H44B	0.036019	0.611454	0.273552	0.050
H44K	0.126439	0.074628	0.280766	0.056
H44L	0.119650	0.130115	0.323572	0.056
H45A	–0.011547	0.664214	0.183969	0.057
H45B	0.015459	0.731230	0.222512	0.057
H45K	0.205905	0.068374	0.337959	0.047
H45L	0.217795	0.109945	0.293195	0.047
H46A	0.101038	0.671242	0.223610	0.043
H46B	0.075210	0.698370	0.173539	0.043
H46K	0.260161	0.198815	0.352042	0.039
H46L	0.206242	0.208262	0.370149	0.039
H51	0.206999	0.747967	0.054390	0.026
H52A	0.179622	0.823539	0.111532	0.040
H52B	0.124565	0.769858	0.103487	0.040
H53A	0.152672	0.899855	0.044432	0.046
H53B	0.107280	0.911293	0.072727	0.046
H54A	0.054920	0.801481	0.030263	0.053
H54B	0.068972	0.876788	–0.002252	0.053
H55A	0.083713	0.733825	–0.029708	0.069
H55B	0.138429	0.788776	–0.018622	0.069
H56A	0.154822	0.643532	0.009641	0.050
H56B	0.108267	0.652657	0.036855	0.050
H61A	0.408090	0.368876	0.128369	0.045
H61B	0.420603	0.486391	0.123181	0.045
H62A	0.458796	0.469816	0.197257	0.043
H62B	0.442351	0.367902	0.196425	0.043
H62C	0.442520	0.490762	0.199651	0.043
H62D	0.438681	0.384851	0.202838	0.043
H63A	0.510267	0.326049	0.164996	0.055
H63B	0.535936	0.397538	0.201779	0.055
H63C	0.529074	0.420332	0.211315	0.047
H63D	0.517378	0.481256	0.167804	0.047
H64A	0.561655	0.422547	0.133138	0.068
H64B	0.532186	0.506889	0.148653	0.068
H64C	0.509238	0.291859	0.168847	0.066
H64D	0.553021	0.346938	0.149847	0.066
H65A	0.498241	0.483604	0.074518	0.058
H65B	0.486072	0.381807	0.083134	0.058

Table S10 (continued). Derived H Atom Coordinates and Isotropic Displacement Parameters for Compound **3**.

Atom	x	y	z	U_{iso} / Å^2
H65C	0.488678	0.399424	0.091001	0.067
H65D	0.484304	0.293564	0.091994	0.067
H66A	0.406532	0.447135	0.072978	0.048
H66B	0.429536	0.532379	0.101123	0.048
H66C	0.408501	0.300410	0.122062	0.050
H66D	0.398693	0.364833	0.079778	0.050
H71	0.352912	0.780398	0.176207	0.026
H72A	0.412023	0.708900	0.234582	0.043
H72B	0.429680	0.639919	0.200832	0.043
H73A	0.496525	0.750018	0.218395	0.066
H73B	0.453302	0.827150	0.204091	0.066
H74A	0.485076	0.701646	0.144705	0.072
H74B	0.499334	0.805254	0.146670	0.072
H75A	0.409636	0.842510	0.119802	0.060
H75B	0.427289	0.772977	0.086272	0.060
H76A	0.342556	0.735100	0.101760	0.037
H76B	0.384952	0.656753	0.115480	0.037
H81	0.444097	0.650105	0.332240	0.030
H82A	0.486513	0.635456	0.406860	0.073
H82B	0.493108	0.530771	0.400891	0.073
H83A	0.575024	0.599713	0.398212	0.086
H83B	0.546139	0.665609	0.359884	0.086
H84A	0.588127	0.553635	0.327805	0.051
H84B	0.558107	0.478707	0.350254	0.051
H85A	0.507613	0.598733	0.282437	0.055
H85B	0.514482	0.494196	0.276066	0.055
H86A	0.426098	0.528826	0.283932	0.042
H86B	0.454749	0.462762	0.322230	0.042
H91A	0.351437	0.844740	0.424741	0.032
H91B	0.389740	0.765069	0.473197	0.031
H92A	0.283951	0.850377	0.460844	0.037
H92B	0.314932	0.790184	0.500818	0.037
H92C	0.323153	0.780074	0.508772	0.038
H92D	0.287917	0.838383	0.470085	0.038
H93A	0.335162	0.970671	0.483430	0.050
H93B	0.324008	0.930140	0.528426	0.050
H93C	0.321410	0.938950	0.523677	0.050
H93D	0.379325	0.894406	0.533450	0.050
H94A	0.410498	0.876694	0.548939	0.049
H94B	0.414789	0.978212	0.534603	0.049
H94C	0.385066	1.028.656	0.497130	0.052
H94D	0.335881	1.002.327	0.457955	0.052
H95A	0.429541	0.934465	0.464829	0.049
H95B	0.472656	0.886138	0.502608	0.049
H95C	0.417107	0.981614	0.433899	0.053
H95D	0.438167	0.915644	0.474587	0.053
H96A	0.422374	0.753843	0.492875	0.042
H96B	0.434572	0.783309	0.446085	0.042
H96C	0.405723	0.829646	0.411153	0.040
H96D	0.349879	0.881338	0.402248	0.040