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(BIS(TRIMETHYLSILYL)AMIDO)URANIUM(IV)

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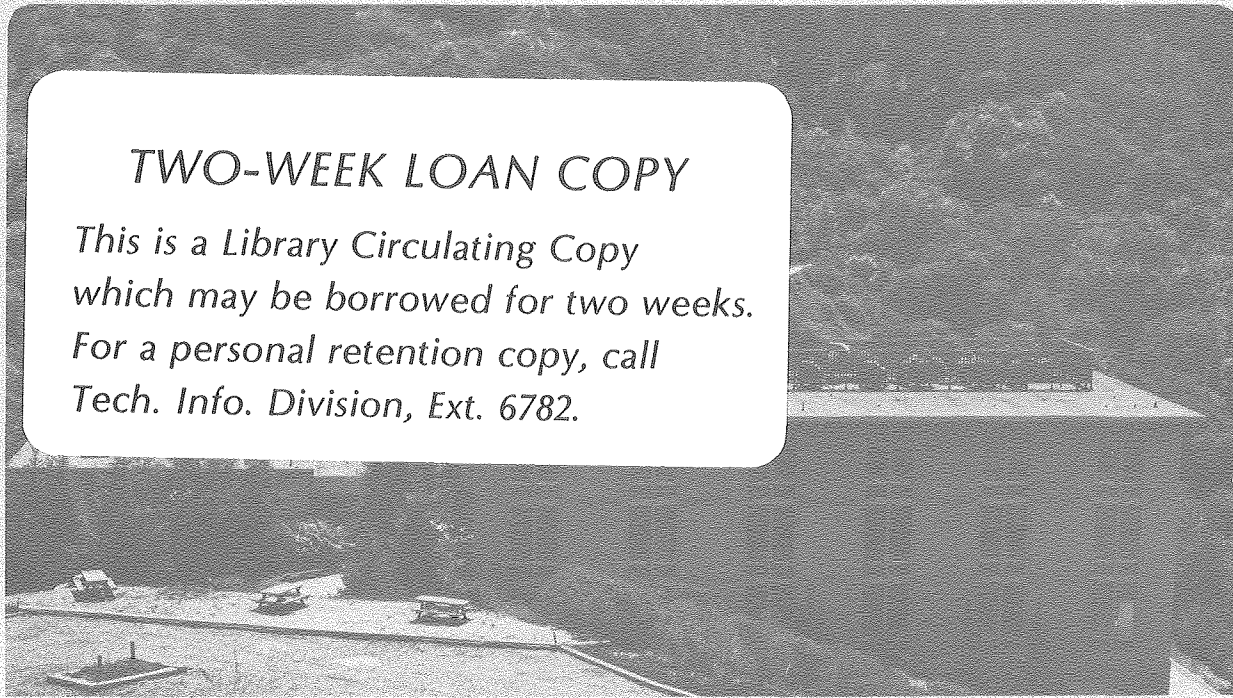
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CRYSTAL AND MOLECULAR STRUCTURE OF
HYDRIDOTRIS[BIS(TRIMETHYLSILYL)AMIDO]URANIUM(IV)

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New hydride derivatives of thorium (IV) and uranium (IV), $\text{HM}[\text{N}(\text{SiMe}_3)_2]_3$, have recently been prepared.¹ This paper describes the crystal structure of the uranium species, though the hydride-ion was not located, and shows that the thorium analogue is isostructural. The reaction chemistry and spectroscopy leave no doubt that these derivatives are authentic examples of four-coordinate, monomeric hydride, compounds.^{1,2}

EXPERIMENTAL SECTION

Hydridotris[bis(trimethylsilyl)amido]thorium(IV), hydridotris[bis(trimethylsilyl)amido]uranium(IV), and tris[bis(trimethylsilyl)amido]uranium(III) were prepared as previously described.^{1,3} The crystals used in the X-ray analysis were grown from pentane (-10°C). The needles were loaded into quartz capillaries under argon and the capillaries were sealed.

A brown, hexagonal-shaped crystal of the uranium hydride, ~ 0.15 mm across and ~ 0.4 mm long, was examined with a Picker FACS-I automatic diffractometer equipped with a graphite monochromator and a Mo X-ray tube ($\lambda(\text{K}\alpha_1)$ 0.70930 \AA). ω scans of several low-angle reflections showed peaks with half-widths of 0.13° and 0.11° for an $h00$ and 001 type reflections, respectively. The space group is $\text{P}\bar{3}1\text{c}$. The setting angles of 12 manually centered reflections ($20.0^\circ < 2\theta < 30.3^\circ$) were used to determine by least squares the cell parameters $\underline{a} = 16.402(8) \text{ \AA}$ and $\underline{c} = 8.501(4) \text{ \AA}$, and $V = 1980.6 \text{ \AA}^3$. There are two molecules in the unit cell.

Intensity data were collected using the θ - 2θ scan technique with a scan speed on $2^\circ/\text{min}$ on 2θ . Each peak was scanned from 0.75° before the $K\alpha_1$ peak to 0.75° after the $K\alpha_2$ peak, and backgrounds were counted for 10 s at each end of the scan range, offset by 0.5° . The needle direction of the crystal was approximately parallel to the ϕ axis of the diffractometer. The temperature during data collection was $21 \pm 1^\circ\text{C}$. Three standard reflections, (300, 060 and 002), were measured after every 200th scan; a decay of about 1 percent in all three standards was observed, and the data were corrected accordingly. An absorption correction⁴ ($\mu = 41 \text{ cm}^{-1}$) was applied and ranged from 1.65 to 1.75. A total of 3569 scans, not including standards, resulted in 879 unique reflections, 521 of which had intensities greater than 3σ .

The atomic positions of U, Si, N and C were taken from the isomorphous Nd(III) compound⁵ and used to start the least squares refinement of the structure. The full-matrix least-squares program minimizes the function $\sum w(|F_o| - |F_c|)^2 / \sum w F_o^2$ and the structure was refined with anisotropic temperature factors to an R value, $R = \sum ||F_o| - |F_c|| / \sum |F_o|$, of 0.091 for 608 ($F^2 > 2\sigma$) data. A difference Fourier calculation showed a large peak, $3 \text{ e}\text{\AA}^3$, with coordinates near 0,0,0.3. This peak is in the channel at the origin, parallel to the z axis, which in this structure type⁵⁻⁸ no ordered matter has been observed, and which is large enough to fit molecules the size of benzene.⁸ To compensate for the electron density of the largest peak in the difference Fourier, $3 \text{ e}\text{\AA}^3$, a pseudo-atom, with

the scattering power of carbon and an isotropic thermal parameter, was included in the least-squares calculations. The R value after a series of least-squares refinement reduced R to 0.071. The methyl hydrogen atoms were not observed in the difference Fourier, but their estimated positions were calculated with C-H bond distances of 0.95Å and assuming a staggered arrangement of tetrahedral C-H bonds to the Si-C tetrahedral bonds. The estimated hydrogen atom positions were included in the least-squares refinements but not refined. One isotropic thermal parameter for all 9 hydrogen atoms was included and refined. Because of large observed discrepancies among some of the lower 2θ angle data, probably due to inaccurate absorption corrections, all 17 data with $\sin\theta/\lambda < 0.14$ were deleted. The final R value for 498 data, $F^2 > 3\sigma$, was 0.044, and for all 897 data it was 0.095. The weighted R_w , $[\sum w(|F_o| - |F_c|)^2 / \sum w |F_o|^2]^{1/2}$, was 0.053 for 48 parameters. The goodness-of-fit was 1.24 and in the last cycle no parameter changed more than 0.01σ .

The expressions used in processing the data and estimating the weights are given in the supplementary material; the "ignorance factor," p , was set to 0.06. Scattering factors from Doyle and Turner⁹ were used, and dispersion corrections¹⁰ were applied. The largest peak in the final difference Fourier was $\sim 0.8 \text{ e}\text{\AA}^{-3}$ about 1Å from the U atom. The second and third peaks were each $\sim 0.3 \text{ e}\text{\AA}^{-3}$ and in the channel along the z axis; none of the residual density in the hole could be related to any chemically reasonable structure.

Weissenberg photography, using $\text{CuK}\alpha$ X-rays, was used to determine the cell dimensions of the isomorphous complexes of the U(III) and Th(IV). Table 1 summarizes these results. Unfortunately the crystal quality of these compounds was very poor and crystal structure determinations were not attempted.

RESULTS AND DISCUSSION

Atomic parameters, distances and angles are listed in Table II-IV. An ORTEP drawing of the structure and a packing diagram are shown in Figures I and II. The uranium atom is on a crystallographic 3-fold axis and bonded to three nitrogen atoms. It is disordered in the z direction at locations 0.51 Å above and below the plane at $z = 1/4$. The electron density in the channel, that runs parallel to \underline{c} at the origin, is attributed to pentane solvent molecules in highly disordered arrangements.

Although $[(\text{Me}_3\text{Si})_2\text{N}]_3\text{UH}$ is isostructural with the binary silylamides of the lanthanide derivatives which have been structurally characterized (Nd^5 , Eu^6 , and Yb^7), the uranium species is tetravalent. The uranium-nitrogen bond length of 2.24 Å is very close to those previously found for terminal, tetravalent uranium-nitrogen bond lengths in $[\text{U}(\text{NEt}_2)_4]_2$, $[\text{U}(\text{MeNCH}_2\text{CH}_2\text{NMe})_2]_3$, $[\text{U}(\text{MeNCH}_2\text{CH}_2\text{NMe})_2]_4$, and $[\text{U}(\text{NPh}_2)_4]$ which are 2.22 Å,¹¹ 2.21 Å,¹² 2.27 Å,¹³ and 2.24 Å,¹⁴ respectively. The thorium (IV)-nitrogen bond length in the six coordinate tetrahydroborate derivative, $\text{Th}[\text{N}(\text{SiMe}_3)_2]_3\text{BH}_4$, is 2.32 Å.¹⁵ The ionic radius of thorium(IV) is estimated to be ca. 0.05 Å larger than that of uranium(IV).¹⁶ Hence, a uranium(IV)-nitrogen bond length of ca. 2.27 Å can be estimated, in good agreement with the observed value. Since the ionic radius of uranium(III) is ca. 0.08 Å longer than that of thorium(IV), a uranium(III)-nitrogen bond length can be estimated to be ca. 2.4 Å, much longer than that observed.

Thus, the U-N bond length in $[(\text{Me}_3\text{Si})_2\text{N}]_3\text{UH}$ supports the identification as a compound of tetravalent uranium.

Unfortunately, a suitable crystal for a structure determination of the authentic uranium(III) silylamide, $\text{U}[\text{N}(\text{SiMe}_3)_2]_3$, could not be obtained, but it was found to be isostructural with that of the hydride, $\text{HU}[\text{N}(\text{SiMe}_3)_2]_3$. However, the larger cell dimensions and volume (Table 1) of the trivalent species are consistent with the fact that uranium(III) is larger than uranium(IV). Further, the similarity of the cell dimensions and volume of the isostructural thorium hydride, $[(\text{Me}_3\text{Si})_2\text{N}]_3\text{ThH}$, with those of $[(\text{Me}_3\text{Si})_2\text{N}]_3\text{U}$ indicates that uranium(III) is similar in size to thorium(IV).

The hydride ion position could not be located, which is not surprising considering the usual difficulty in finding hydrogen atoms in structures containing very heavy atoms, especially in this case where the considerable disorder degrades the resolving power of the diffraction data.

Supplementary Material Available: Data processing formulas and the listing of structure factor amplitudes (5 pages). Ordering information is given on a current masthead.

ACKNOWLEDGMENT

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Table I. Cell Dimensions (\AA) of the Tris(bis(trimethylsilyl)amido Complexes of U(III), U(IV) and Th(IV).

	U(III)	U(IV)	Th(IV)
Color	Dark red	Brown	White
a	16.56(5)	16.402(8) \AA	16.47(3) \AA
c	8.43(4)	8.501(4) \AA	8.53(3)
V	2002 \AA^3	1981 \AA^3	2004 \AA^3

Table II. Positional and Thermal Parameters^a with Estimated Deviations.^b

Atom	x	y	z
U	2/3	1/3	.3100(1)
N	.5133(6)	.2567	1/4
Si	.4575(2)	.2881(2)	.1105(3)
C(1)	.4016(9)	.3520(9)	.197(1)
C(2)	.3660(8)	.1863(9)	-.002(1)
C(3)	.5488(9)	.3682(8)	-.029(1)
H(1) ^c	.4489	.4084	.2463
H(2)	.3565	.3137	.2728
H(3)	.3723	.3683	.1162
H(4)	.3197	.1433	.0688
H(5)	.3948	.1558	-.0547
H(6)	.3383	.2077	-.0763
H(7)	.5773	.3363	-.0763
H(8)	.5946	.4218	.026
H(9)	.5203	.3869	-.1076
χ^2	0	0	.313(2)

Table II. Continued

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
U ^d	5.65(5)	5.65	4.54(5)	2.825	0	0
N ^e	5.2(5)	4.5(3)	4.5(4)	2.6	0	-.3(3)
Si	6.2(1)	6.3(1)	5.4(1)	3.4(1)	-1.1(1)	-.3(1)
C(1)	10.8(8)	11.1(8)	8.5(6)	7.9(7)	-.5(6)	-.5(6)
C(2)	10.2(8)	9.7(8)	9.1(6)	4.1(7)	-3.8(6)	-1.9(6)
C(3)	10.2(8)	9.3(7)	6.3(5)	4.0(6)	.9(5)	1.9(5)

^aThe anisotropic temperature factor has the form $\exp(-.25(B_{11}h^2\underline{a}^{*2} + 2 B_{12}hka^*b^* + \dots))$.

^bHere and in the following tables the number in parentheses is the estimated standard deviation of the least significant digit.

^cIsotropic temperature parameters for H and X are 15(2) Å² and 3.0(4) Å² respectively. X is the pseudoatom introduced to compensate for the largest electron density peak in the channel; it does not represent any chemically recognizable structure.

^dSymmetry conditions of the special position; $B_{11} = B_{22} = 2B_{12}$, and $B_{13} = B_{23} = 0$.

^eSymmetry conditions of the special position; $x = 2y$, $B_{11} = 2B_{12}$, and $B_{13} = 0$.

Table III. Interatomic Distances (Å).

		Corrected ^a
U-3N	2.237(9)	2.240
N-2Si	1.727(5)	1.740
Si-C(1)	1.86(1)	1.89
Si-C(2)	1.86(1)	1.90
Si-C(3)	1.84(1)	1.88

^aAdjusted for thermal motion assuming the "riding model".

Table IV. Selected Angles (deg).

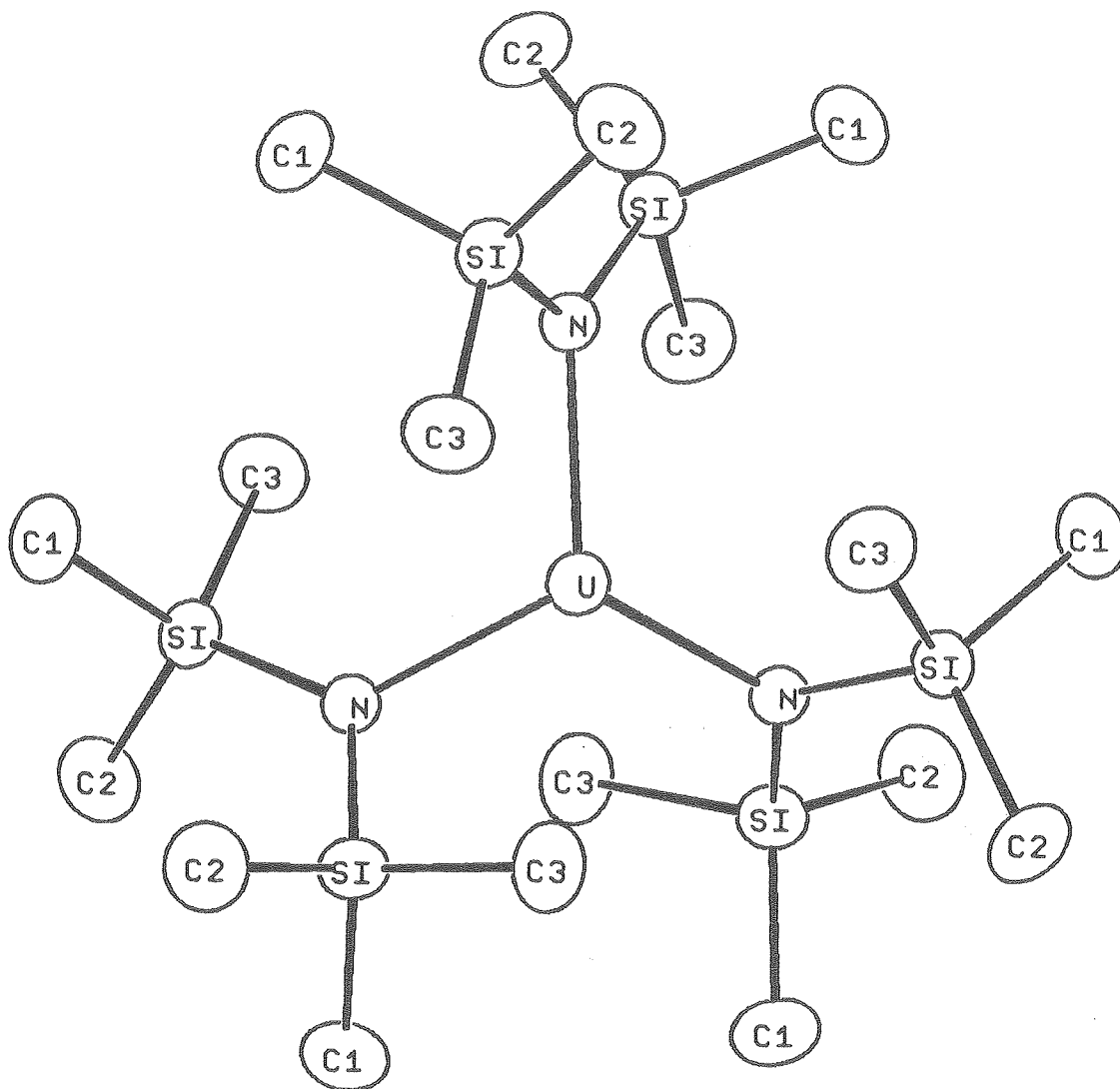
N-U-N	115.0(1)
U-N-Si	127.1(3)
U-N-Si ^a	106.9(2)
Si-N-Si ^a	125.3(6)
N-Si-C(1)	112.5(4)
N-Si-C(2)	113.3(5)
N-Si-C(3)	106.8(5)
C(1)-Si-C(2)	107.9(6)
C(1)-Si-C(3)	107.8(6)
C(2)-Si-C(3)	108.3(5)

^aAtom at position x, x-y, 1/2-z.

FIGURE CAPTION

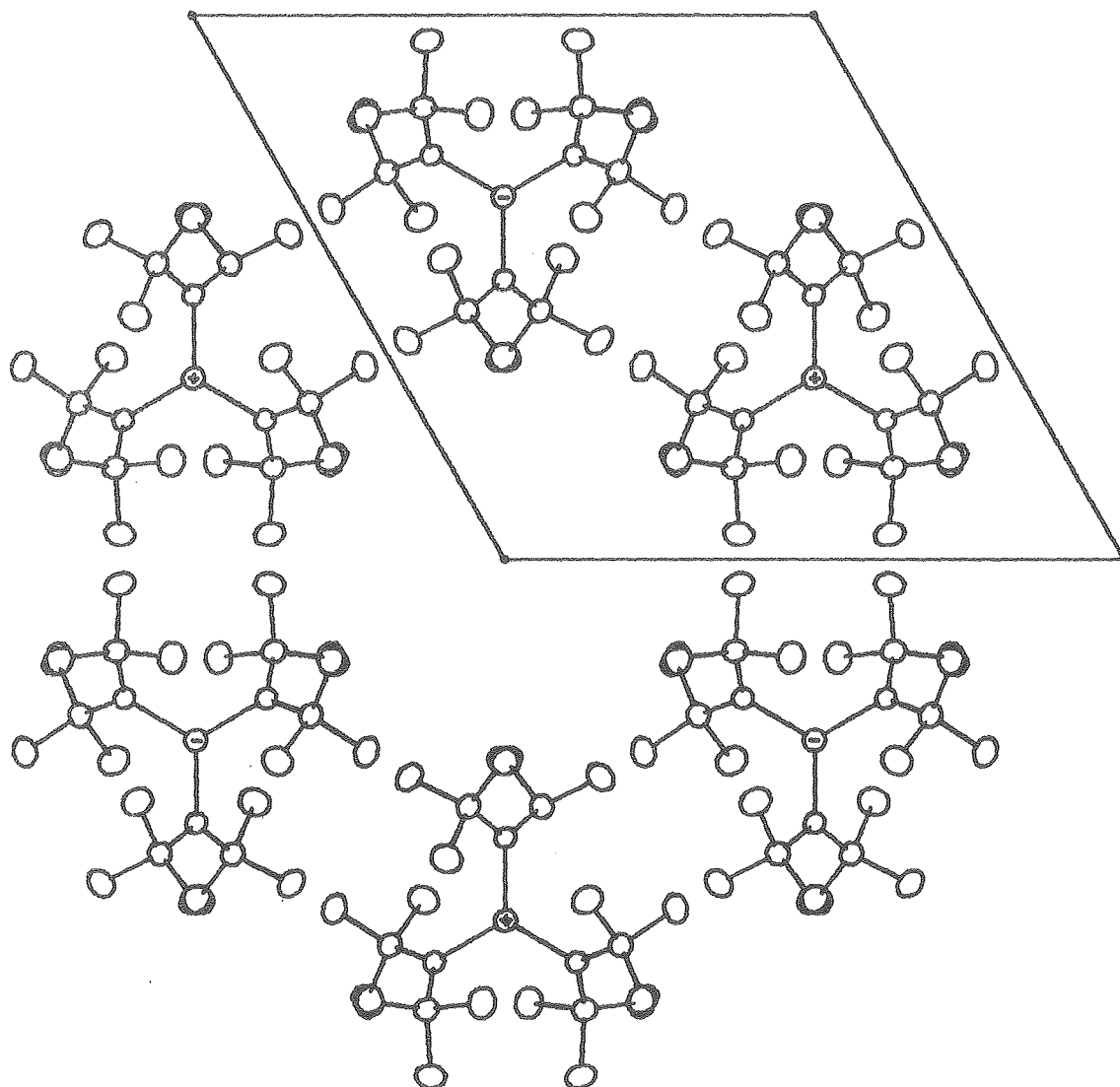
Fig. 1. ORTEP view down, but slightly off, the c axis.

Fig. 2. Packing diagram as seen down the c axis.



XBL 802-8029

Fig. 1



XBL 802-8030

Fig. 2

SUPPLEMENTARY MATERIALS FOR THE PAPER

Crystal and Molecular Structure of
Hydridotris[bis(trimethylsilyl)amido]uranium(IV)

by Richard A. Andersen, Allan Zalkin,^{*} and David H. Templeton

DATA PROCESSING FORMULAE

$$I = C - (t_c/2t_b)(B_1+B_2)$$

$$\sigma(B) = \text{Max}[(t_c/2t_b)(B_1+B_2)^{\frac{1}{2}}, (t_c/2t_b)|B_1-B_2|]$$

$$\sigma(I) = [0 + \sigma^2(B)]^{\frac{1}{2}}$$

$$F^2 = (D \cdot A/Lp)I$$

$$\sigma(F^2) = (D \cdot A/Lp)\sigma(I)$$

$$F_a^2 = \Sigma F^2/n$$

$$\sigma(F_a^2) = [\Sigma \sigma^2(F^2)/n]^{\frac{1}{2}} \quad \text{When } S(F_a^2) > 4\sigma(F_a^2), \sigma(F_a^2) \text{ is replaced by } S(F_a^2).$$

$$S(F_a^2) = [\Sigma |F^2 - F_a^2|^2/n(n-1)]^{\frac{1}{2}}$$

$$\sigma(F_o^2) = [\sigma^2(F_a^2) + (pF_a^2)^2 + q^2]^{\frac{1}{2}}$$

$$F_o = (F_a^2)^{\frac{1}{2}}$$

$$\sigma(F) = F_o - [F_a^2 - \sigma(F_o^2)]^{\frac{1}{2}} \text{ when } \sigma(F_o^2) \leq F_a^2 \text{ or } [\sigma(F_a^2)]^{\frac{1}{2}} \text{ when } \sigma(F_a^2) > F_a^2$$

$$Lp = [\cos^2 2\theta_m + \cos^2 2\theta]/[\sin 2\theta (1 + \cos^2 2\theta_m)]$$

$$\text{wtg} = 1/\sigma^2(F)$$

C = counts recorded during a scan

θ_m = monochromater angle

I = individual raw intensity,
background removed.

θ = crystal diffraction angle

t_c = scan count time

S = scatter

t_b = background count time

a = average

B_1 = individual background count

q = additional uncertainty that
affects the weak intensities

$\sigma(B)$ = estimated standard deviation of the total background count

p = estimate of non-statistical errors

F = structure factor

wtg = weighting factors in least squares

D = decay correction; an empirically applied correction obtained from the fluctuations of the standard reflections.

A = absorption correction

Lp = Lorentz and polarization corrections

OBSERVED STRUCTURE FACTORS, STANDARD DEVIATIONS, AND DIFFERENCES (ALL X 6.0)
 U(IV) (N(SI(CH3)3)2)3 H F(0,0,0) = 4259

FOB AND FCA ARE THE OBSERVED AND CALCULATED STRUCTURE FACTORS.
 SG = ESTIMATED STANDARD DEVIATION OF FCB. DEL = /FOB/ - /FCA/.
 * INDICATES ZERO WEIGHTED DATA.

L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL
H,K=	0,	0		6	144	9	20	6	489	15	1	0	656	20	8	8	129	10	-3
2	447	0	182*	8	286	10	-2	8	138	14	-6	2	425	13	13	H,K=	5,	3	
4	43	16	22*	H,K=	3,	-1		H,K=	4,	-1		4	228	9	4	0	51	11	-14*
6	149	8	9	1	972	0	0*	1	732	0	21*	6	189	12	13	1	546	17	10
8	53	35	-32*	2	509	16	-20	2	646	20	25	8	142	11	-1	2	144	6	-13
H,K=	1,	0		3	492	15	19	3	318	10	2	H,K=	5,	-2		3	194	7	2
11	214	0	20*	4	144	6	-10	4	148	6	2	1	765	23	-7	4	232	8	4
2	121	0	-100*	5	207	7	7	5	172	7	-5	2	111	5	-3	5	31	39	-4*
3	266	8	37	6	195	7	-8	6	133	9	-18	3	91	6	-2	6	133	8	2
4	40	9	38*	7	251	9	-7	7	281	9	-2	4	44	12	20*	7	113	9	6
5	186	6	-7	8	120	9	8	8	49	36	17*	5	209	7	-2	8	91	13	9
6	46	13	-16*	9	68	26	2*	H,K=	4,	0		6	99	9	-4	H,K=	5,	4	
7	74	10	12	H,K=	3,	0		0	377	12	-17	7	196	10	-1	0	318	10	-11
8	51	24	-10*	0	685	0	74*	1	937	29	-6	8	84	16	10*	1	245	8	-4
9	123	8	-7	1	358	0	21*	2	41	11	5*	H,K=	5,	-1		2	40	14	15*
H,K=	1,	1		2	937	29	44	3	142	5	-1	1	367	11	38	3	190	7	1
01	146	0	253*	3	180	6	-3	4	79	7	3	2	501	15	-16	4	58	18	9*
2	307	0	103*	4	125	7	20	5	112	5	-5	3	173	6	11	5	44	49	5*
4	68	15	55*	5	29	31	17*	6	194	7	-11	4	130	7	0	6	20	45	1*
6	153	8	-1	6	287	9	8	7	164	8	-7	5	72	9	-1	7	133	9	-3
8	227	9	-12	7	111	9	-1	8	104	9	-9	6	191	7	8	H,K=	5,	5	
H,K=	2,	-1		8	245	8	-10	H,K=	4,	1		7	31	46	20*	0	439	14	10
21	027	0	127*	9	60	40	45*	0	703	21	-30	8	150	8	-1	2	189	9	4
4	162	6	38	H,K=	3,	1		1	36	18	33*	H,K=	5,	0		4	90	12	-8
6	266	9	5	0	426	0	-11*	2	518	16	5	0	129	5	7	6	89	16	-7
8	141	10	-22	1	279	0	24*	3	78	9	2	1	602	18	11	H,K=	6,	-3	
H,K=	2,	0		2	298	9	29	4	205	7	6	2	419	13	24	2	299	9	-8
0	496	0	-213*	3	323	10	11	5	243	8	4	3	86	6	1	4	48	18	-9*
1	484	0	10*	4	17	30	12*	6	181	7	6	4	119	5	2	6	159	8	8
2	45	0	-15*	5	53	14	-7*	7	21	40	-2*	5	205	7	2	8	185	11	12
3	452	14	37	6	66	16	-4*	8	162	8	-17	6	36	27	18*	H,K=	6,	-2	
4	22	26	7*	7	270	9	-7	H,K=	4,	2		7	101	10	-16	1	433	13	14
5	85	7	-5	8	102	10	0	0	349	11	20	8	75	11	16	2	466	14	4
6	51	13	-19*	H,K=	3,	2		1	677	21	6	H,K=	5,	1		3	114	6	-13
7	245	8	-2	0	232	8	6	2	37	15	5*	0	419	13	9	4	84	7	5
8	47	19	-5*	1	292	9	-15	3	207	7	9	1	442	14	-21	5	35	44	-11*
9	127	9	3	2	246	8	12	4	144	7	11	2	374	12	14	6	143	7	-1
H,K=	2,	1		3	218	7	-1	5	325	10	-4	3	38	15	9*	7	74	19	-24*
0	292	0	113*	4	30	33	-9*	6	26	40	-11*	4	287	9	-5	8	31	43	-20*
1	110	0	7*	5	20	40	2*	7	118	8	-19	5	107	8	-1	H,K=	6,	-1	
2	120	8	-14	6	138	8	-1	8	106	11	-1	6	155	7	-4	1	512	16	-19
3	162	6	8	7	171	7	-1	H,K=	4,	3		7	139	7	-9	2	230	7	6
4	123	6	5	8	72	16	-1*	0	326	10	-5	8	33	43	12*	3	136	5	-7
5	69	9	14	H,K=	3,	3		1	622	19	9	H,K=	5,	2		4	37	28	-1*
6	93	8	-14	0	722	22	-20	2	495	15	8	0	619	19	5	5	89	9	11
7	225	9	-11	2	750	23	-0	3	341	11	1	1	237	8	-3	6	79	12	19
8	159	8	-10	4	64	13	2*	4	123	6	1	2	598	18	12	7	121	8	5
9	154	9	-2	6	207	8	-5	5	88	10	-4	3	42	13	-8*	8	91	16	6
H,K=	2,	2		8	157	10	-1	6	93	9	7	4	41	18	-5*	H,K=	6,	0	
0	709	0	38*	H,K=	4,	-2		7	182	8	-10	5	174	7	5	0	878	27	-14
2	105	8	-6	2	596	18	-16	8	35	47	2*	6	226	8	6	1	18	26	-19*
4	274	9	19	4	81	11	-10	H,K=	4,	4		7	68	14	1*	2	229	7	-2

STRUCTURE FACTORS CONTINUED FOR
U(IV) (N(SI(CH3)3)2)3 H

L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL
3	27	22	15*	4	0	51	-37*	7	113	9	7	3	243	8	-3	5	78	13	16
4	40	15	6*	6	183	9	13	H,K=	7,	3	4	52	13	36*	6	60	23	1*	
5	23	33	-2*	H,K=	7,	-3	0	298	10	-2	5	116	7	11	H,K=	8,	5		
6	175	7	2	1	524	16	10	1	442	14	-2	6	51	40	-12*	0	171	7	3
7	39	35	38*	2	57	8	-9	2	34	24	4*	7	156	8	2	1	36	36	-23*
8	149	7	4	3	311	10	-1	3	68	10	18	8	60	19	13*	2	156	8	-3
H,K=	6,		1	4	23	39	14*	4	55	17	17*	H,K=	8,	-1	3	36	43	30*	
0	293	9	-15	5	36	28	-0*	5	94	18	7*	1	27	31	-14*	4	23	43	10*
1	536	16	12	6	40	28	18*	6	56	22	4*	2	300	9	7	5	41	48	33*
2	46	10	-15*	7	188	7	-3	7	105	10	14	3	165	6	-0	6	118	11	22
3	238	8	8	8	112	13	28	H,K=	7,	4	4	79	9	-19	H,K=	8,			6
4	75	8	-12	H,K=	7,	-2	0	485	15	2	5	92	11	4	0	138	7	10	
5	193	8	6	1	17	26	16*	1	89	8	-19	6	182	7	-1	1	133	8	8
6	86	12	4	2	300	9	1	2	283	9	11	7	70	17	18*	2	78	11	-4
7	150	8	2	3	346	11	-7	3	88	12	1	H,K=	8,	0	3	88	11	9	
8	75	16	9*	4	93	7	-2	4	69	13	18*	0	361	11	-12	4	53	23	39*
H,K=	6,		2	5	95	8	2	5	47	26	-12*	1	162	6	1	5	73	16	24*
0	308	10	-1	6	117	9	24	6	107	10	18	2	38	16	8*	H,K=	8,	7	
1	410	13	-10	7	58	19	7*	7	0	44	-12*	3	390	12	-5	0	113	8	8
2	163	6	-15	8	176	8	-5	H,K=	7,	5	4	130	6	3	1	191	7	-4	
3	269	9	-10	H,K=	7,	-1	0	198	7	-2	5	55	12	14*	2	54	32	2*	
4	42	21	7*	1	369	11	12	1	233	8	5	6	55	19	2*	3	19	41	-4*
5	64	12	9*	2	158	7	-11	2	183	7	-8	7	220	8	8	4	70	17	49*
6	35	42	13*	3	119	6	5	3	128	7	-1	H,K=	8,	1	H,K=	8,	8		
7	149	8	-7	4	15	36	9*	4	48	31	45*	0	52	10	12*	0	260	10	-3
8	52	25	13*	5	43	37	26*	5	75	14	9*	1	302	9	-6	2	111	12	9
H,K=	6,		3	6	107	9	9	6	48	51	-7*	2	181	7	-0	H,K=	9,	-4	
0	499	15	1	7	144	8	3	H,K=	7,	6	3	59	16	4*	1	371	12	-9	
1	21	29	-5*	8	57	22	-1*	0	42	43	17*	4	111	7	-9	2	286	9	-11
2	264	8	5	H,K=	7,	0	1	238	8	-2	5	128	8	14	3	338	11	-9	
3	151	6	-7	0	107	5	-3	2	106	9	-3	6	90	13	10	4	49	15	38*
4	89	8	-6	1	437	13	-15	3	163	8	-3	7	121	10	-5	5	105	10	22
5	71	18	10*	2	175	6	-3	4	34	39	8*	H,K=	8,	2	6	116	10	6	
6	79	12	-10	3	168	6	-3	5	115	10	10	0	514	16	4	7	143	8	-2
7	56	19	-7*	4	213	7	5	6	74	15	-6*	1	25	33	-5*	H,K=	9,	-3	
H,K=	6,		4	5	122	6	-5	H,K=	7,	7	2	210	7	6	1	318	10	6	
0	133	6	-21	6	119	9	4	0	238	9	2	3	65	11	22	2	456	14	7
1	266	8	1	7	146	7	5	2	207	9	-1	4	93	10	14	3	77	8	4
2	178	7	-19	8	111	9	5	4	58	60	40*	5	15	43	-15*	4	48	18	28*
3	33	34	12*	H,K=	7,	1	H,K=	8,	-4	6	118	9	-8	5	94	14	6		
4	59	14	-11*	0	449	14	14	2	501	15	17	7	36	49	35*	6	168	8	-9
5	31	56	4*	1	169	6	-4	4	203	9	-18	H,K=	8,	3	7	37	53	15*	
6	107	10	-1	2	393	12	11	6	213	9	8	0	279	9	-6	H,K=	9,	-2	
7	98	12	21	3	152	6	9	8	153	11	-2	1	357	11	1	1	412	13	2
H,K=	6,		5	4	96	7	-17	H,K=	8,	-3	2	43	18	-20*	2	70	8	-5	
0	108	8	-4	5	21	45	-23*	1	494	15	7	3	47	17	17*	3	157	6	-5
1	341	11	-4	6	201	9	0	2	215	7	-9	4	35	38	-4*	4	53	14	-2*
2	104	8	-5	7	63	17	24*	3	171	7	10	5	62	17	-9*	5	132	9	9
3	180	7	-11	H,K=	7,	2	4	184	8	-7	6	72	13	36*	6	34	48	18*	
4	56	20	49*	0	149	7	-21	5	66	14	4*	7	52	33	-12*	7	121	9	2
5	77	18	3*	1	286	9	0	6	104	9	6	H,K=	8,	4	H,K=	9,	-1		
6	47	24	22*	2	213	7	-6	7	151	8	2	0	62	15	6*	1	239	8	-8
7	126	13	19	3	102	7	-2	8	89	14	-3	1	248	8	-3	2	225	7	-3
H,K=	6,		6	4	55	13	22*	H,K=	8,	-2	2	76	9	3	3	80	10	6	
0	200	8	-8	5	34	48	11*	1	228	7	-11	3	23	39	-19*	4	134	7	-7
2	396	13	-3	6	185	13	4	2	105	5	4	4	31	49	23*	5	90	14	7

STRUCTURE FACTORS CONTINUED FOR
U (IV) (N(SI(CH3)3)2)3 H

L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL
6	90	12	3	4	48	28	26*	1	166	6	13	5	38	46	-11*	0	97	9	14
7	119	10	6	H,K=	9,	7		2	284	9	-6	6	82	15	1*	1	44	45	-37*
	H,K=	9,	0	0	91	12	-2	3	53	15	9*	7	30	45	23*	2	45	29	34*
0	200	7	-0	1	166	9	13	4	20	44	6*	H,K=	11,	-3	3	82	12	12	
1	17	32	16*	2	67	15	18*	5	81	16	0*	1	363	11	5	4	22	44	14*
2	278	9	7	3	60	32	50*	6	133	10	10	2	103	8	-5	H,K=	11,	5	
3	179	6	8	H,K=	9,	8		H,K=	10,	2		3	23	36	-19*	0	90	11	-25
4	69	13	23	0	90	11	14	0	99	9	-1	4	48	21	46*	1	69	16	35*
5	35	43	6*	1	146	8	4	1	314	10	6	5	92	13	-3	2	69	16	-14*
6	160	7	10	2	63	25	31*	2	32	40	-12*	6	59	23	22*	3	60	19	30*
7	41	43	9*	H,K=	10,	-5		3	92	13	7	7	67	30	14*	H,K=	11,	6	
	H,K=	9,	1	2	465	14	12	4	28	40	-31*	H,K=	11,	-2	0	31	45	17*	
0	84	8	-4	4	0	48	-48*	5	123	9	21	1	290	9	3	1	108	10	6
1	323	10	-2	6	167	9	22	6	29	50	19*	2	62	12	-12*	2	70	23	21*
2	36	37	12*	H,K=	10,	-4		H,K=	10,	3		3	69	11	20	H,K=	12,	-6	
3	125	9	2	1	570	18	-10	0	86	11	24	4	15	48	-18*	2	48	45	-9*
4	25	40	12*	2	28	37	-15*	1	129	8	2	5	88	11	7	4	75	24	-11*
5	98	10	3	3	173	7	-5	2	94	9	5	6	38	42	19*	6	88	16	31*
6	37	46	-12*	4	63	12	-22*	3	38	41	-12*	H,K=	11,	-1	H,K=	12,	-5		
7	132	10	14	5	178	8	14	4	13	44	1*	1	36	27	24*	1	117	7	-10
	H,K=	9,	2	6	43	44	17*	5	25	47	16*	2	153	7	-3	2	30	35	12*
0	262	9	18	7	80	15	-2*	H,K=	10,	4		3	70	11	-21	3	60	13	5*
1	273	9	-3	H,K=	10,	-3		0	152	7	-7	4	22	41	-4*	4	36	40	3*
2	170	7	-1	1	348	11	-12	1	22	42	16*	5	38	49	9*	5	20	47	6*
3	51	15	25*	2	271	9	-10	2	24	39	-26*	6	98	10	20	6	45	28	31*
4	69	14	5*	3	84	11	-15	3	57	18	47*	H,K=	11,	0	H,K=	12,	-4		
5	35	45	-17*	4	134	7	1	4	61	33	-1*	0	76	13	-4	1	284	9	-3
6	98	11	22	5	86	12	6	5	25	46	4*	1	257	8	8	2	58	19	4*
	H,K=	9,	3	6	89	11	5	H,K=	10,	5		2	162	6	-3	3	39	41	-6*
0	367	11	-0	7	74	20	-2*	0	7	41	-32*	3	87	10	-4	4	37	45	20*
1	70	14	0*	H,K=	10,	-2		1	73	13	19	4	59	12	-14*	5	79	19	15*
2	147	10	7	1	33	35	8*	2	27	40	-12*	5	60	16	25*	6	79	15	38*
3	67	17	34*	2	244	8	4	3	60	16	17*	6	75	13	16	H,K=	12,	-3	
4	41	28	5*	3	20	35	13*	4	88	15	17*	H,K=	11,	1	1	87	9	-30	
5	67	14	12*	4	85	9	25	H,K=	10,	6		0	180	8	23	2	133	8	-7
6	76	14	10*	5	31	47	19*	0	97	11	21	1	210	7	-8	3	58	20	0*
	H,K=	9,	4	6	96	13	-0	1	96	15	4	2	122	9	13	4	63	27	13*
0	128	7	-2	7	43	46	37*	2	53	21	29*	3	132	8	-18	5	67	14	18*
1	90	9	-1	H,K=	10,	-1		3	66	16	-6*	4	32	42	-5*	6	109	12	18
2	29	47	13*	1	226	8	7	H,K=	10,	7		5	29	42	1*	H,K=	12,	-2	
3	64	20	11*	2	65	12	-17	0	123	9	-10	6	46	47	7*	1	142	8	13
4	27	40	23*	3	102	7	3	1	50	25	44*	H,K=	11,	2	2	135	7	-7	
5	62	19	37*	4	53	15	42*	2	113	10	3	0	220	8	0	3	31	43	16*
6	43	44	3*	5	28	47	0*	H,K=	11,	-5		1	0	38	-23*	4	20	46	9*
	H,K=	9,	5	6	70	15	10*	1	252	8	5	2	207	8	-6	5	34	44	24*
0	32	39	10*	7	103	12	12	2	188	7	-9	3	38	39	29*	6	93	11	22
1	68	19	-0*	H,K=	10,	0		3	79	10	4	4	36	41	31*	H,K=	12,	-1	
2	41	52	28*	0	257	8	5	4	6	43	-25*	5	32	46	30*	1	123	7	-10
3	60	15	3*	1	158	6	-7	5	42	43	32*	H,K=	11,	3	2	42	25	26*	
4	54	19	21*	2	135	6	9	6	66	17	30*	0	109	8	-7	3	101	9	1
5	40	43	21*	3	129	6	-16	7	97	13	4	1	130	9	14	4	50	30	27*
	H,K=	9,	6	4	29	41	24*	H,K=	11,	-4		2	77	15	9*	5	30	44	21*
0	187	8	2	5	59	12	26*	1	86	7	1	3	62	17	10*	6	50	51	31*
1	30	43	13*	6	49	19	12*	2	274	9	-4	4	38	43	34*	H,K=	12,	0	
2	59	17	-31*	H,K=	10,	1		3	27	35	21*	5	41	44	24*	0	181	7	-10
3	31	41	12*	0	295	9	-6	4	72	14	5*	H,K=	11,	4	1	32	42	19*	

STRUCTURE FACTORS CONTINUED FOR
U(IV) (N(SI(CH3)3)2)3 H

L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL
2	176	7	-18	4	42	39	16*	H,K=	14,	-3	3	56	31	19*					
3	48	17	39*	5	84	14	17	1	152	8	-3	H,K=	15,	-1					
4	41	22	-10*	H,K=	13,	-2	2	32	49	-11*	1	62	22	-27*					
5	49	21	48*	1	21	38	-8*	3	83	12	-4	2	85	14	13				
H,K=	12,	1		2	99	12	-17	4	36	51	23*	H,K=	15,	0					
0	100	8	-10	3	28	43	14*	5	76	15	31*	0	88	19	-9*				
1	185	7	10	4	25	41	-7*	H,K=	14,	-2	1	46	27	33*					
2	107	10	-15	5	42	47	35*	1	82	17	-28*	H,K=	16,	-8					
3	92	16	-6	H,K=	13,	-1	2	108	10	11	2	102	15	-0					
4	29	44	12*	1	126	7	1	3	41	44	25*	H,K=	16,	-7					
5	54	35	21*	2	31	49	16*	4	20	44	4*	1	169	8	10				
H,K=	12,	2		3	74	14	-14*	H,K=	14,	-1	2	15	48	-9*					
0	73	11	30	4	0	51	-5*	1	56	24	31*	3	68	15	54*				
1	232	8	-3	5	22	47	7*	2	69	14	-27*	H,K=	16,	-6					
2	58	21	28*	H,K=	13,	0	3	74	14	10	1	66	20	-31*					
3	68	22	5*	0	84	11	33	4	39	43	15*	2	76	14	24*				
4	2	48	-30*	1	150	7	-15	H,K=	14,	0	3	56	20	-0*					
H,K=	12,	3		2	85	10	8	0	74	16	-27*	H,K=	16,	-5					
0	192	8	3	3	65	11	12	1	136	8	14	1	48	23	16*				
1	30	41	-4*	4	62	17	9*	2	39	27	-1*	2	70	18	-13*				
2	110	11	2	H,K=	13,	1	3	67	14	-6*	3	51	33	12*					
3	28	42	21*	0	199	8	-3	H,K=	14,	1	H,K=	16,	-4						
4	31	50	22*	1	80	13	0	0	86	11	35	1	110	10	5				
H,K=	12,	4		2	160	8	-3	1	148	9	4	2	62	17	35*				
0	54	20	36*	3	26	44	11*	2	44	46	25*	3	40	46	30*				
1	129	9	13	4	8	43	4*	H,K=	14,	2	H,K=	16,	-3						
2	44	46	27*	H,K=	13,	2	0	138	9	6	1	65	18	-25*					
3	65	19	33*	0	41	10	-5	1	28	47	17*	2	78	15	43*				
H,K=	12,	5		1	129	8	7	H,K=	15,	-7	H,K=	16,	-2						
0	104	10	28	2	106	11	10	1	191	7	-2	1	45	51	30*				
1	64	17	3*	3	44	47	9*	2	83	13	12	H,K=	17,	-8					
H,K=	13,	-6		H,K=	13,	3	3	44	36	21*	1	122	11	2					
1	87	9	-1	0	111	12	27	4	35	42	-1*	2	44	46	3*				
2	50	17	30*	1	110	11	-1	H,K=	15,	-6	H,K=	17,	-7						
3	66	20	1*	2	66	18	29*	1	32	42	26*	1	34	46	33*				
4	52	19	-4*	H,K=	14,	-7	2	169	7	-5	2	40	47	-33*					
5	34	45	-8*	2	103	14	-9	3	61	16	57*	H,K=	17,	-6					
6	57	41	18*	4	29	70	-16*	4	44	33	26*	1	69	15	14*				
H,K=	13,	-5		H,K=	14,	-6	H,K=	15,	-5	2	40	51	10*						
1	89	9	-6	1	129	7	-3	1	165	9	3	H,K=	17,	-5					
2	159	7	-4	2	65	13	-3*	2	79	15	6*	1	47	34	-3*				
3	44	32	20*	3	82	13	-16	3	95	10	5								
4	56	23	2*	4	0	42	-29*	4	53	22	18*								
5	41	43	39*	5	52	29	6*	H,K=	15,	-4									
6	105	12	5	H,K=	14,	-5	1	123	8	-1									
H,K=	13,	-4		1	200	8	-4	2	52	23	-15*								
1	163	7	8	2	109	11	13	3	81	12	0								
2	125	8	-22	3	105	10	-20	4	46	49	31*								
3	100	13	-6	4	45	46	33*	H,K=	15,	-3									
4	0	41	-27*	5	80	14	18	1	58	21	20*								
5	80	13	20	H,K=	14,	-4	2	80	12	-7									
6	58	22	18*	1	31	46	1*	3	26	46	9*								
H,K=	13,	-3		2	227	9	1	4	39	43	29*								
1	228	8	-1	3	11	40	3*	H,K=	15,	-2									
2	60	21	34*	4	18	41	-6*	1	148	9	15								
3	46	25	-33*	5	43	48	36*	2	46	49	19*								