

Crystal structure of synthetic hafnon, HfSiO_4 , comparison with zircon and the actinide orthosilicates

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Abstract

A crystal structure refinement of a synthetic hafnon, HfSiO_4 , space group $I4_1/AMD$ with $a = 6.5725(7)\text{\AA}$, $c = 5.9632(4)\text{\AA}$, $Z = 4$, $D = 6.97 \text{ gm cm}^{-3}$, produced positional parameters for oxygen of $y = 0.0655(13)$ and $z = 0.1948(14)$ at $R = 0.054$. Hafnon has a zircon structure and the similar ionic radii of Hf (0.83\AA) and Zr (0.84\AA) accounts for the interatomic distances and angles of hafnon and zircon being identical within the stated errors, although hafnon has systematically smaller distances and corresponds to a zircon at 29.5 kbar pressure. Combining the hafnon, zircon and thorite structure refinements allows prediction of the oxygen positional parameters and bond lengths for the actinide (Pa, U, Np, Pu and Am) orthosilicates as well as possible extent of the solid solution among these zircon-structure silicates.

Introduction

The most common compositional substitution for Zr in zircon is Hf (see Speer, 1980a for discussion and references). Most natural zircons have a Zr:Hf ratio of 40:1, which corresponds to their crustal abundance ratio. Synthetic HfSiO_4 is isostructural with zircon and was called hafnon by Curtis *et al.* (1954), a name eventually approved for the rare, natural end member by the International Mineralogical Association. Hafnon is found in pegmatites with Ta and Nb minerals, in particular where there is enrichment in the pegmatite of the even-odd pair $^{72}\text{Hf}-^{73}\text{Ta}$ over the $^{40}\text{Zr}-^{41}\text{Nb}$ pair.

Hf and Zr are extremely similar in their chemical properties; Hf was distinguished from Zr only in 1922. This similarity is a result of the similarity in their electronic structures and ionic radii which permits complete miscibility in most Zr-Hf compounds. While there is little change in the geometry and dimensions of these structures with chemistry, there are greater changes in some physical properties (*e.g.*, density and thermal neutron capture cross section). Zirconium has a cross section of 0.18 barn whereas Hf has a larger value of 115 barn. The presence of Hf in zircon increases its susceptibility to metamictization, and Hf compositional zoning causes a nonuniform susceptibility. Hf-rich zones in natural zircons are usually more metamict, contain

greater compositional substitutions for Zr and Si and yield discordant ages (see Speer, 1980a, chemical zoning). A crystal structure refinement of hafnon was performed to determine if any geometrical or dimensional differences accompany the physical property differences between hafnon and zircon. The comparison of the hafnon structure with that of zircon and thorite permits an understanding of the crystal chemistry of the solid solutions among hafnon, zircon and the actinide orthosilicates.

Experimental

Hafnon crystals were grown in a platinum crucible from a Li_2MoO_4 melt which was about 5 percent by weight $\text{HfO}_2\cdot\text{SiO}_2$. The starting material was spectroscopic grade, 99.99% HfO_2 from Apache Chemicals, Inc. and Puratronic SiO_2 from Johnson Matthey Chemicals, Ltd. The crucible was held at 1000°C for four days in an air-atmosphere muffle furnace. The crystals were separated from the quenched flux in hot water.

The crystals were tetragonal dipyramids, and wavelength dispersive electron microprobe scans of the crystals detected only Hf and Si. The space group of hafnon, determined by precession method, is $I4_1/AMD$. Unit-cell parameters were obtained by least-squares refinement of 20 automatically-centered reflections in the 2θ range of 74 to 93° (Table 1). Data were collected on a crystal $0.12 \times 0.14 \times$

Table 2. Hafnon Refinement

D%

HAFFNON REFINEMENT

PARAMETERS AFTER LEAST SQUARES CYCLE 3

SCALE FACTOR 1 G FOR ISOTROPIC EXTINCTION

VALUE, V -0.600374 0.961591

CHANGE, C -0.000000 0.0

STD DEV, E 0.006095 0.0

DAMPING, D 1.000000 0.0

HF	(ATOM NO. 1)	F REAL	F IMAG	MULT	X	Y	Z	B(1,1)	B(2,2)	B(3,3)	B(1,2)	B(1,3)	B(2,3)
V	1.00000	6.18500	0.0	0.0	0.0	0.75000	0.12500	0.00351	0.00351	0.00342	0.0	0.0	0.0
C	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.00014	0.00014	0.00019	0.0	0.0	0.0
E	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.00000	1.00000	1.00000	0.0	0.0	0.0
S	0.0	b(I,J)/(2 PI**2)	ASTAR(I) ASTAR(J)), ANGSTROMS SQUARED					0.00769	0.00769	0.00616	0.0	0.0	0.0
SI	(ATOM NO. 2)	F REAL	F IMAG	MULT	X	Y	Z	B	B	B	B	B	B
V	2.00000	0.0	0.0	0.12500	0.0	0.75000	0.62500	0.05450	0.05450	0.00003	0.0	0.0	0.0
C	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.08637	0.08637	0.00000	0.0	0.0	0.0
E	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.00000	1.00000	1.00000	0.0	0.0	0.0
S	0.0	b/(8 PI**2), ANGSTROM UNITS SQUARED						0.00069	0.00069	0.00069	0.0	0.0	0.0
U	(ATOM NO. 3)	F REAL	F IMAG	MULT	X	Y	Z	B	B	B	B	B	B
V	3.00000	0.0	0.0	0.50000	0.0	0.65554	0.19477	0.72782	0.72782	-0.00000	0.0	0.0	0.0
C	0.0	0.0	0.0	0.0	0.0	0.00000	0.00000	0.00126	0.00126	0.00144	0.00144	0.00097	0.00097
E	0.0	0.0	0.0	0.0	0.0	0.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
S	0.0	b/(8 PI**2), ANGSTROM UNITS SQUARED						0.00922	0.00922	0.00922	0.00922	0.00922	0.00922

ESTIMATED AGREEMENT FACTORS BASED ON PARAMETERS AFTER CYCLE 3

SUM(W*(10-C)**2) IS 0.3864E-04

SQRTH(SUM(W*(10-C)**2)/(NU-NVT)) IS 4.4402

TIME FOR MATRIX INVERSION WAS 0 SEC.

HAFNUN REFINEMENT

Table 2. Hafnun Refinement (cont.) D%

Y(CALC) BASED ON PARAMETERS BEFORE CYCLE FOR ACCEPTANCE OF DATUM, Q IS CALC F**2/SIN 2 THETA		Y(OBS)		Y(CALC)		A	B	OBS-CALC	SIG(0)	(0-C)/SIG(0)	I0	IL	Q	EXTINCTION
1	1	5	5	7	7	-76.05181	-16.0031	8.3969	1.1804	7.1138	1	1	8823.39	0.4288
1	1	5	5	7	7	78.01730	71.0108	69.9087	66.3369	14.7413	1	1	4538.63	0.5449
1	1	5	5	7	7	57.9828	57.9828	58.7577	57.2530	13.2123	0	0	9292	2234.63
1	1	5	5	7	7	53.9038	53.9038	54.9631	-33.0100	-11.5222	-1	0	0593	0.6537
1	1	5	5	7	7	51.6180	51.6180	51.6616	-51.6616	-14.9035	-1	0	5944	0.8371
1	1	5	5	7	7	49.862	49.862	49.887	49.887	11.3741	-1	0	2122	7978.19
1	1	5	5	7	7	47.2384	47.2384	47.2604	47.2604	11.2072	-1	0	0725	0.4408
1	1	5	5	7	7	44.1260	44.1260	44.1475	44.1475	10.86199	-1	0	0749	0.0000
1	1	5	5	7	7	40.8055	40.8055	40.8240	40.8240	10.0072	-1	0	0725	0.6343
1	1	5	5	7	7	38.5246	38.5246	38.5443	38.5443	9.20608	-1	0	0749	0.2000
1	1	5	5	7	7	34.0740	34.0740	34.0947	34.0947	8.2065	-1	0	0749	0.5061
1	1	5	5	7	7	31.9472	31.9472	31.9672	31.9672	7.20672	-1	0	0749	1.78670
1	1	5	5	7	7	29.1950	29.1950	29.1950	29.1950	6.20672	-1	0	0749	0.4927
1	1	5	5	7	7	26.4959	26.4959	26.4959	26.4959	5.20672	-1	0	0749	0.3250
1	1	5	5	7	7	23.9151	23.9151	23.9151	23.9151	4.20672	-1	0	0749	0.0000
1	1	5	5	7	7	21.3035	21.3035	21.3035	21.3035	3.20672	-1	0	0749	0.5037
1	1	5	5	7	7	18.7947	18.7947	18.7947	18.7947	2.20672	-1	0	0749	2.0244.43
1	1	5	5	7	7	16.2037	16.2037	16.2037	16.2037	1.20672	-1	0	0749	0.8031
1	1	5	5	7	7	13.7125	13.7125	13.7125	13.7125	0.20672	-1	0	0749	1.9139.74
1	1	5	5	7	7	11.2027	11.2027	11.2027	11.2027	-0.20672	-1	0	0749	2.5508.48
1	1	5	5	7	7	8.7027	8.7027	8.7027	8.7027	-1.20672	-1	0	0749	1.4640.04
1	1	5	5	7	7	6.2026	6.2026	6.2026	6.2026	-2.20672	-1	0	0749	0.5677.32
1	1	5	5	7	7	3.7019	3.7019	3.7019	3.7019	-3.20672	-1	0	0749	2.310.42
1	1	5	5	7	7	1.2010	1.2010	1.2010	1.2010	-4.20672	-1	0	0749	4.605.58
1	1	5	5	7	7	-0.7009	-0.7009	-0.7009	-0.7009	-5.20672	-1	0	0749	2.5508.48
1	1	5	5	7	7	-3.2010	-3.2010	-3.2010	-3.2010	-6.20672	-1	0	0749	0.2000
1	1	5	5	7	7	-6.7010	-6.7010	-6.7010	-6.7010	-7.20672	-1	0	0749	0.5037
1	1	5	5	7	7	-10.2010	-10.2010	-10.2010	-10.2010	-8.20672	-1	0	0749	0.0000
1	1	5	5	7	7	-13.7010	-13.7010	-13.7010	-13.7010	-7.20672	-1	0	0749	0.8031
1	1	5	5	7	7	-17.2010	-17.2010	-17.2010	-17.2010	-6.20672	-1	0	0749	1.9139.74
1	1	5	5	7	7	-20.7010	-20.7010	-20.7010	-20.7010	-5.20672	-1	0	0749	2.5508.48
1	1	5	5	7	7	-24.2010	-24.2010	-24.2010	-24.2010	-4.20672	-1	0	0749	1.4640.04
1	1	5	5	7	7	-27.7010	-27.7010	-27.7010	-27.7010	-3.20672	-1	0	0749	0.5677.32
1	1	5	5	7	7	-31.2010	-31.2010	-31.2010	-31.2010	-2.20672	-1	0	0749	2.310.42
1	1	5	5	7	7	-34.7010	-34.7010	-34.7010	-34.7010	-1.20672	-1	0	0749	4.605.58
1	1	5	5	7	7	-38.2010	-38.2010	-38.2010	-38.2010	0.20672	-1	0	0749	2.5508.48
1	1	5	5	7	7	-41.7010	-41.7010	-41.7010	-41.7010	-0.20672	-1	0	0749	0.2000
1	1	5	5	7	7	-45.2010	-45.2010	-45.2010	-45.2010	1.20672	-1	0	0749	0.5037
1	1	5	5	7	7	-48.7010	-48.7010	-48.7010	-48.7010	2.20672	-1	0	0749	1.9139.74
1	1	5	5	7	7	-52.2010	-52.2010	-52.2010	-52.2010	3.20672	-1	0	0749	2.5508.48
1	1	5	5	7	7	-55.7010	-55.7010	-55.7010	-55.7010	4.20672	-1	0	0749	1.4640.04
1	1	5	5	7	7	-59.2010	-59.2010	-59.2010	-59.2010	5.20672	-1	0	0749	0.5677.32
1	1	5	5	7	7	-62.7010	-62.7010	-62.7010	-62.7010	6.20672	-1	0	0749	2.310.42
1	1	5	5	7	7	-66.2010	-66.2010	-66.2010	-66.2010	7.20672	-1	0	0749	4.605.58
1	1	5	5	7	7	-70.7010	-70.7010	-70.7010	-70.7010	8.20672	-1	0	0749	2.5508.48
1	1	5	5	7	7	-74.2010	-74.2010	-74.2010	-74.2010	9.20672	-1	0	0749	0.2000
1	1	5	5	7	7	-77.7010	-77.7010	-77.7010	-77.7010	10.20672	-1	0	0749	0.5037
1	1	5	5	7	7	-81.2010	-81.2010	-81.2010	-81.2010	11.20672	-1	0	0749	1.9139.74
1	1	5	5	7	7	-84.7010	-84.7010	-84.7010	-84.7010	12.20672	-1	0	0749	2.5508.48
1	1	5	5	7	7	-88.2010	-88.2010	-88.2010	-88.2010	13.20672	-1	0	0749	1.4640.04
1	1	5	5	7	7	-91.7010	-91.7010	-91.7010	-91.7010	14.20672	-1	0	0749	0.5677.32
1	1	5	5	7	7	-95.2010	-95.2010	-95.2010	-95.2010	15.20672	-1	0	0749	2.310.42
1	1	5	5	7	7	-98.7010	-98.7010	-98.7010	-98.7010	16.20672	-1	0	0749	4.605.58
1	1	5	5	7	7	-102.2010	-102.2010	-102.2010	-102.2010	17.20672	-1	0	0749	2.5508.48
1	1	5	5	7	7	-105.7010	-105.7010	-105.7010	-105.7010	18.20672	-1	0	0749	0.2000
1	1	5	5	7	7	-109.2010	-109.2010	-109.2010	-109.2010	19.20672	-1	0	0749	0.5037
1	1	5	5	7	7	-112.7010	-112.7010	-112.7010	-112.7010	20.20672	-1	0	0749	1.9139.74
1	1	5	5	7	7	-116.2010	-116.2010	-116.2010	-116.2010	21.20672	-1	0	0749	2.5508.48
1	1	5	5	7	7	-120.7010	-120.7010	-120.7010	-120.7010	22.20672	-1	0	0749	1.4640.04
1	1	5	5	7	7	-124.2010	-124.2010	-124.2010	-124.2010	23.20672	-1	0	0749	0.5677.32
1	1	5	5	7	7	-127.7010	-127.7010	-127.7010	-127.7010	24.20672	-1	0	0749	2.310.42
1	1	5	5	7	7	-131.2010	-131.2010	-131.2010	-131.2010	25.20672	-1	0	0749	4.605.58
1	1	5	5	7	7	-134.7010	-134.7010	-134.7010	-134.7010	26.20672	-1	0	0749	2.5508.48
1	1	5	5	7	7	-138.2010	-138.2010	-138.2010	-138.2010	27.20672	-1	0	0749	0.2000
1	1	5	5	7	7	-141.7010	-141.7010	-141.7010	-141.7010	28.20672	-1	0	0749	0.5037
1	1	5	5	7	7	-145.2010	-145.2010	-145.2010	-145.2010	29.20672	-1	0	0749	1.9139.74
1	1	5	5	7	7	-148.7010	-148.7010	-148.7010	-148.7010	30.20672	-1	0	0749	2.5508.48
1	1	5	5	7	7	-152.2010	-152.2010	-152.2010	-152.2010	31.20672	-1	0	0749	1.4640.04
1	1	5	5	7	7	-155.7010	-155.7010	-155.7010	-155.7010	32.20672	-1	0	0749	0.5677.32
1	1	5	5	7	7	-159.2010	-159.2010	-159.2010	-159.2010	33.20672	-1	0	0749	2.310.42
1	1	5	5	7	7	-162.7010	-162.7010	-162.7010	-162.7010	34.20672	-1	0	0749	4.605.58
1	1	5	5	7	7	-166.2010	-166.2010	-166.2010	-166.2010	35.20672	-1	0	0749	2.5508.48
1	1	5	5	7	7	-170.7010	-170.7010	-170.7010	-170.7010	36.20672	-1	0	0749	0.2000
1	1	5	5	7	7	-174.2010	-174.2010	-174.2010	-174.2010	37.20672	-1	0	0749	0.5037
1	1	5	5	7	7	-177.7010	-177.7010	-177.7010	-177.7010	38.20672	-1	0	0749	1.9139.74
1	1	5	5	7	7	-181.2010	-181.2010	-181.2010	-181.2010	39.20672	-1	0	0749	2.5508.48
1	1	5	5	7	7	-184.7010	-184.7010	-184.7010	-184.7010	40.20672	-1	0	0749	1.4640.04
1	1	5	5	7	7	-188.2010	-188.2010	-188.2010	-188.2010	41.20672	-1	0	0749	0.5677.32
1	1	5	5	7	7	-191.7010	-191.7010	-191.7010	-191.7010	42.20672	-1	0	0749	2.310.42
1	1	5	5	7	7	-195.2010	-195.2010	-195.2010	-195.2010	43.20672</				

Table 2. Hafnon Refinement (cont.)

Table 2. Haffon Refinement (cont.)

	Y(UBS)	Y(CALC)	A	B	OBSS-CALC	SIG(0)	(0-C)/SIG(0)	IQ	IL	Q	EXTINCTION
100	100	100	108.7926	103.0011	-101.5392	-17.2923	-5.7915	1.2907	4.4870	1	4819
100	100	100	131.0611	137.1427	-135.7123	-19.7561	-6.0815	-3.209	-2.209	1	9874
100	100	100	130.0022	137.0590	-17.0590	0.0000	0.0000	0.0000	0.0000	0	3874
100	100	100	130.2722	137.2779	-17.2779	5.0249	-4.0249	-6.2196	-5.2196	1	9582
100	100	100	130.2779	137.3714	-17.3714	13.8731	-4.8731	-6.4642	-5.4642	1	6993
100	100	100	130.2779	137.794	-17.794	-4.7476	-2.7476	-6.4222	-5.4222	1	7152
100	100	100	130.2779	137.794	-17.794	-12.7793	-3.7793	-3.1462	-2.1462	1	6779
100	100	100	130.2779	137.794	-17.794	-13.7140	-3.7140	-3.1462	-2.1462	1	9874
100	100	100	130.2779	137.794	-17.794	-14.7132	-4.7132	-3.1462	-2.1462	1	3874
100	100	100	130.2779	137.794	-17.794	-15.7128	-5.7128	-3.1462	-2.1462	1	9582
100	100	100	130.2779	137.794	-17.794	-16.7124	-6.7124	-3.1462	-2.1462	1	6993
100	100	100	130.2779	137.794	-17.794	-17.7120	-7.7120	-3.1462	-2.1462	1	7152
100	100	100	130.2779	137.794	-17.794	-18.7116	-8.7116	-3.1462	-2.1462	1	6779
100	100	100	130.2779	137.794	-17.794	-19.7112	-9.7112	-3.1462	-2.1462	1	9874
100	100	100	130.2779	137.794	-17.794	-20.7108	-10.7108	-3.1462	-2.1462	1	3874
100	100	100	130.2779	137.794	-17.794	-21.7104	-11.7104	-3.1462	-2.1462	1	9582
100	100	100	130.2779	137.794	-17.794	-22.7100	-12.7100	-3.1462	-2.1462	1	6993
100	100	100	130.2779	137.794	-17.794	-23.7096	-13.7096	-3.1462	-2.1462	1	7152
100	100	100	130.2779	137.794	-17.794	-24.7092	-14.7092	-3.1462	-2.1462	1	6779
100	100	100	130.2779	137.794	-17.794	-25.7088	-15.7088	-3.1462	-2.1462	1	9874
100	100	100	130.2779	137.794	-17.794	-26.7084	-16.7084	-3.1462	-2.1462	1	3874
100	100	100	130.2779	137.794	-17.794	-27.7080	-17.7080	-3.1462	-2.1462	1	9582
100	100	100	130.2779	137.794	-17.794	-28.7076	-18.7076	-3.1462	-2.1462	1	6993
100	100	100	130.2779	137.794	-17.794	-29.7072	-19.7072	-3.1462	-2.1462	1	7152
100	100	100	130.2779	137.794	-17.794	-30.7068	-20.7068	-3.1462	-2.1462	1	6779
100	100	100	130.2779	137.794	-17.794	-31.7064	-21.7064	-3.1462	-2.1462	1	9874
100	100	100	130.2779	137.794	-17.794	-32.7060	-22.7060	-3.1462	-2.1462	1	3874
100	100	100	130.2779	137.794	-17.794	-33.7056	-23.7056	-3.1462	-2.1462	1	9582
100	100	100	130.2779	137.794	-17.794	-34.7052	-24.7052	-3.1462	-2.1462	1	6993
100	100	100	130.2779	137.794	-17.794	-35.7048	-25.7048	-3.1462	-2.1462	1	7152
100	100	100	130.2779	137.794	-17.794	-36.7044	-26.7044	-3.1462	-2.1462	1	6779
100	100	100	130.2779	137.794	-17.794	-37.7040	-27.7040	-3.1462	-2.1462	1	9874
100	100	100	130.2779	137.794	-17.794	-38.7036	-28.7036	-3.1462	-2.1462	1	3874
100	100	100	130.2779	137.794	-17.794	-39.7032	-29.7032	-3.1462	-2.1462	1	9582
100	100	100	130.2779	137.794	-17.794	-40.7028	-30.7028	-3.1462	-2.1462	1	6993
100	100	100	130.2779	137.794	-17.794	-41.7024	-31.7024	-3.1462	-2.1462	1	7152
100	100	100	130.2779	137.794	-17.794	-42.7020	-32.7020	-3.1462	-2.1462	1	6779
100	100	100	130.2779	137.794	-17.794	-43.7016	-33.7016	-3.1462	-2.1462	1	9874
100	100	100	130.2779	137.794	-17.794	-44.7012	-34.7012	-3.1462	-2.1462	1	3874
100	100	100	130.2779	137.794	-17.794	-45.7008	-35.7008	-3.1462	-2.1462	1	9582
100	100	100	130.2779	137.794	-17.794	-46.7004	-36.7004	-3.1462	-2.1462	1	6993
100	100	100	130.2779	137.794	-17.794	-47.7000	-37.7000	-3.1462	-2.1462	1	7152
100	100	100	130.2779	137.794	-17.794	-48.7000	-38.7000	-3.1462	-2.1462	1	6779
100	100	100	130.2779	137.794	-17.794	-49.7000	-39.7000	-3.1462	-2.1462	1	9874
100	100	100	130.2779	137.794	-17.794	-50.7000	-40.7000	-3.1462	-2.1462	1	3874
100	100	100	130.2779	137.794	-17.794	-51.7000	-41.7000	-3.1462	-2.1462	1	9582
100	100	100	130.2779	137.794	-17.794	-52.7000	-42.7000	-3.1462	-2.1462	1	6993
100	100	100	130.2779	137.794	-17.794	-53.7000	-43.7000	-3.1462	-2.1462	1	7152
100	100	100	130.2779	137.794	-17.794	-54.7000	-44.7000	-3.1462	-2.1462	1	6779
100	100	100	130.2779	137.794	-17.794	-55.7000	-45.7000	-3.1462	-2.1462	1	9874
100	100	100	130.2779	137.794	-17.794	-56.7000	-46.7000	-3.1462	-2.1462	1	3874
100	100	100	130.2779	137.794	-17.794	-57.7000	-47.7000	-3.1462	-2.1462	1	9582
100	100	100	130.2779	137.794	-17.794	-58.7000	-48.7000	-3.1462	-2.1462	1	6993
100	100	100	130.2779	137.794	-17.794	-59.7000	-49.7000	-3.1462	-2.1462	1	7152
100	100	100	130.2779	137.794	-17.794	-60.7000	-50.7000	-3.1462	-2.1462	1	6779
100	100	100	130.2779	137.794	-17.794	-61.7000	-51.7000	-3.1462	-2.1462	1	9874
100	100	100	130.2779	137.794	-17.794	-62.7000	-52.7000	-3.1462	-2.1462	1	3874
100	100	100	130.2779	137.794	-17.794	-63.7000	-53.7000	-3.1462	-2.1462	1	9582
100	100	100	130.2779	137.794	-17.794	-64.7000	-54.7000	-3.1462	-2.1462	1	6993
100	100	100	130.2779	137.794	-17.794	-65.7000	-55.7000	-3.1462	-2.1462	1	7152
100	100	100	130.2779	137.794	-17.794	-66.7000	-56.7000	-3.1462	-2.1462	1	6779
100	100	100	130.2779	137.794	-17.794	-67.7000	-57.7000	-3.1462	-2.1462	1	9874
100	100	100	130.2779	137.794	-17.794	-68.7000	-58.7000	-3.1462	-2.1462	1	3874
100	100	100	130.2779	137.794	-17.794	-69.7000	-59.7000	-3.1462	-2.1462	1	9582
100	100	100	130.2779	137.794	-17.794	-70.7000	-60.7000	-3.1462	-2.1462	1	6993
100	100	100	130.2779	137.794	-17.794	-71.7000	-61.7000	-3.1462	-2.1462	1	7152
100	100	100	130.2779	137.794	-17.794	-72.7000	-62.7000	-3.1462	-2.1462	1	6779
100	100	100	130.2779	137.794	-17.794	-73.7000	-63.7000	-3.1462	-2.1462	1	9874
100	100	100	130.2779	137.794	-17.794	-74.7000	-64.7000	-3.1462	-2.1462	1	3874
100	100	100	130.2779	137.794	-17.794	-75.7000	-65.7000	-3.1462	-2.1462	1	9582
100	100	100	130.2779	137.794	-17.794	-76.7000	-66.7000	-3.1462	-2.1462	1	6993
100	100	100	130.2779	137.794	-17.794	-77.7000	-67.7000	-3.1462	-2.1462	1	7152
100	100	100	130.2779	137.794	-17.794	-78.7000	-68.7000	-3.1462	-2.1462	1	6779
100	100	100	130.2779	137.794	-17.794	-79.7000	-69.7000	-3.1462	-2.1462	1	9874
100	100	100	130.2779	137.794	-17.794	-80.7000	-70.7000	-3.1462	-2.1462	1	3874
100	100	100	130.2779	137.794	-17.794	-81.7000	-71.7000	-3.1462	-2.1462	1	9582
100	100	100	130.2779	137.794	-17.794	-82.7000	-72.7000	-3.1462	-2.1462	1	6993
100	100	100	130.2779	137.794	-17.794	-83.7000	-73.7000	-3.1462	-2.1462	1	7152
100	100	100	130.2779	137.794	-17.794	-84.7000	-74.7000	-3.1462	-2.1462	1	6779
100	100	100	130.2779	137.794	-17.794	-85.7000	-75.7000	-3.1462	-2.1462	1	9874
100	100	100	130.2779	137.794	-17.794	-86.7000	-76.7000	-3.1462	-2.1462	1	3874
100	100	100	130.2779	137.794	-17.794	-87.7000	-77.7000	-3.1462	-2.1462	1	9582
100	100	100	130.2779	137.794	-17.794	-88.7000	-78.7000	-3.1462	-2.1462	1	6993
100	100	100	130.2779	137.794	-17.794	-89.7000	-79.7000	-3.1462	-2.1462	1	7152
100	100	100	130.2779	137.794	-17.794	-90.7000	-80.7000	-3.1462	-2.1462	1	6779
100	100	100	130.2779	137.794	-17.794	-91.7000	-81.7000	-3.1462	-2.1462	1	9874
100	100	100	130.2779	137.794	-17.794	-92.7000	-82.7000	-3.1462	-2.1462	1	3874
100	100	100	130.2779	137.794	-17.794	-9					

Table 2. Hafnon Refinement (cont.)

HAFFNAN REFINEMENT

TIME FOR MATRIX SETUP WAS 0 SEC. NUMBER OF DEGREES OF FREEDOM IS 196 NUMBER OF PARAMETERS VARIED IN THIS CYCLE WAS 7, TOTAL NUMBER OF PARAMETERS FITTED TO DATA SET TAKEN AS 7

Table 2. Haffnian Refinement (cont.) D%

***** MEASURES OF AGREEMENT BEFORE CYCLE 4 *****

FOR REFLECTIONS ACCEPTED BY LOGIC	R(F)	R(F**2)	WR(YO)	SIG 1
ALL INTENSITIES (203 REFLECTIONS)				
RATIO	0.0536	0.1224	0.0611	4.4402
NUMERATOR	0.950988535E 03	0.116261000E 06	0.386413354E 04	
DENOMINATOR	0.121445195E 05	0.949494000E 06	0.103377856E 07	
GREATER THAN SIGMA (203 REFLECTIONS)				
RATIO	0.0536	0.1224	0.0611	4.4402
NUMERATOR	0.650998535E 03	0.116261000E 06	0.386413354E 04	
DENOMINATOR	0.121445195E 05	0.949494000E 06	0.103377856E 07	
NO TEST ON LOGIC				
ALL INTENSITIES (203 REFLECTIONS)				
RATIO	0.0536	0.1224	0.0611	4.4402
NUMERATOR	0.650998535E 03	0.116261000E 06	0.386413354E 04	
DENOMINATOR	0.121445195E 05	0.949494000E 06	0.103377856E 07	
GREATER THAN SIGMA (203 REFLECTIONS)				
RATIO	0.0536	0.1224	0.0611	4.4402
NUMERATOR	0.650998535E 03	0.116261000E 06	0.386413354E 04	
DENOMINATOR	0.121445195E 05	0.949494000E 06	0.103377856E 07	

HAFFNAN REFINEMENT

ELEMENTS OF CORRELATION MATRIX OF MAGNITUDE GREATER THAN 0.30

SCALE	1	=	^{HF} _{B11}	0.7309
		-	^{HF} _{B33}	0.6952
HF	511	=	^{HF} _{B33}	0.3440
HF	833	=	^{HF} _B	0.6291

CORRELATION MATRIX

1	1.0000	0.7309	0.6952	0.3440	0.0199	-0.0075	0.2471
2	0.0	1.0000	0.6291	0.7757	0.0134	-0.0110	0.2216
3	0.0	0.0	1.0000	0.5990	0.0142	0.0413	0.2277
4	0.0	0.0	0.0	1.0000	-0.0156	0.0162	0.0806
5	0.0	0.0	0.0	0.0	1.0000	-0.0383	0.0160
6	0.0	0.0	0.0	0.0	0.0	1.0000	0.0501
7	0.0	0.0	0.0	0.0	0.0	0.0	1.0000

Table 2. Hafnon Refinement (cont.) D%