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Preprint

STRUCTURE OF TRIS(BIS(TRIMETHYLSILYL)AMIDO)NEODYMIUM(III), $Nd[N(Si(CH_3)_3)_2]_3$

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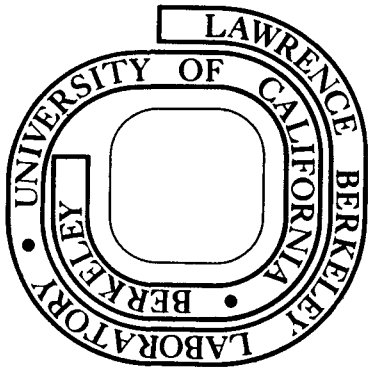

LBL-7603

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and the Department of Chemistry,
University of California, Berkeley, California 94720

Structure of Tris(bis(trimethylsilyl)amido)neodymium(III), $Nd[N(Si(CH_3)_3)_2]_3$[1]

By Richard A. Andersen*, David H. Templeton* and Allan Zalkin*

Neodymium tris[di(trimethylsilyl)amide] is the only known monomeric, three coordinate derivative of this lanthanide element.[2] The structures of the europium (III)[3] and ytterbium (III)[4] derivatives have been examined by X-ray crystallographic techniques and they, along with the scandium (III) analogue,[3] have been shown to have MN_3 skeletons which are not planar. In contrast all other crystallographically known tris-silylamides of the type $M[N(SiMe_3)_2]_3$ are planar.[4] We describe the crystal structure of $Nd[N(SiMe_3)_2]_3$ and show that it is also non-planar.

EXPERIMENTAL

The $Nd[N(SiMe_3)_2]_3$ was prepared as previously described,[2] m.p. 157-161° (lit.[2] 161-164°). The crystal used in the X-ray analysis was taken from a batch crystallized from pentane (0°C).

Magnetic susceptibility measurements were obtained with a PAR model 155 vibrating sample magnetometer employing a homogeneous magnetic field produced by a Varian Associates 12-inch electromagnet capable of a maximum field strength of 12.5 kG. The magnetometer was calibrated with $HgCo(CNS)_4$.[5] A variable temperature liquid helium system produced sample temperatures in the range 4-100°K. The temperature was measured with a calibrated GaAs diode.

A hexagonal needle shaped crystal, 0.09 mm across and 0.3 mm long, was sealed inside a quartz capillary in an argon filled dry box. It was examined with a Picker FACS-I automatic diffractometer equipped with a graphite monochromator and a Mo x-ray tube ($\lambda(K\alpha_1)$ 0.7093 Å). ω scans of several low-angle reflections showed peaks with half-widths of 0.16° and 0.21° for an h00 and 00l type reflections respectively. The space group was identified as $P\bar{3}1c$. The setting angles of 12 manually centered reflections ($19° < 2\theta < 25°$) were used to determine by least squares the cell parameters $\underline{a}$ = 16.476(13) Å, $\underline{c}$ = 8.485(7) Å, and V = 1995 $Å^3$. For Z = 2 and a molecular weight of 625.4 the calculated density is 1.04 g cm^{-3}.

Intensity data were collected using the θ-2θ scan technique with a scan speed of 2°/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 phi axis of the diffractometer. The temperature during data collection was 21 ± 1°C. Three standard reflections, (300, 060 and 002), were measured after every 200th scan; no significant variation was observed in the intensities of the first two reflections, and a 5 pecent decay in intensity was observed for the 002 reflection. A linear decay correction of about 4% was applied uniformly to the data. The absorption coefficient is estimated to be 15 cm^{-1}. Because of the diffraction geometry and the small crystal dimensions the absorption is small and no correction was deemed necessary. A total of 3404 scans, not including standards, resulted in 917 unique reflections, 535 of which were greater than 2σ.

The positions of the Nd, N, and Si atoms were deduced from a three dimensional Patterson function. The carbon atoms were obtained from a subsequent least squares and Fourier calculation. A series of least-squares refinements in which the function $\Sigma w(|F_o|-|F_c|)^2/\Sigma wF_o^2$ was minimized converged to the final structure. The expressions that were 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 Turner[6] were used, and dispersion corrections[7] were applied. Hydrogen atoms could not be located, and were not included. Because of the large residuals exhibited by several of

the low angle intensities, all 35 data whose $(\sin\theta)/\lambda$ is less than 0.127, were deleted. The discrepancy index for 522 data are

$$R = \Sigma||F_o|-|F_c||/\Sigma|F_o| = 0.076$$

$$R_w = [\Sigma w(|F_o|-|F_c|)^2/\Sigma w|F_o|^2]^{1/2} = 0.091$$

R for all 882 data is 0.13. The error in an observation of unit weight is 2.0. In the last cycle no parameter changed more than 0.001σ.

RESULTS AND DISCUSSION

Atomic parameters, distances and angles are listed in Tables I-III. The molecular structure of this Nd complex (Fig. 1) is similar to that found in the Al,[8] Fe,[9] Sc,[3] and Eu[3] compounds. The Nd atom is on a crystallographic 3-fold axis and is bonded to three nitrogen atoms. In the Sc and Eu isomorphs[4] the metal atoms are disordered in the z direction, and were treated as two half atoms ~0.6 Å above and below the plane at z = 1/4. This structure is similar with Nd 0.34 Å above and below z = 1/4. Originally the Nd atom was treated as an anisotropic atom at z = 1/4 which resulted in thermal parameters B_{11} and B_{33} being 3.5 and 16.0 $Å^2$ respectively. When the Nd atom was treated isotropically as two half atoms disordered across the plane at z = 1/4 the subsequent least-squares refinement resulted in the R factor going from 0.084 for the ordered to 0.076 for the disordered description.

A large channel that runs up the z axis, at the origin of the unit cell, is characteristic of the structures of these hexagonal $M[N(SiMe_3)_2]_3$ complexes. Hursthouse and Rodesiler[9] have shown that in the case of the iron complex the channel is large enough to accommodate a benzene ring with the plane of the ring perpendicular to the z axis; they could not find any ordered solvent in the channel. A search of the final electron density and difference maps for the Nd structure showed one peak at 0,0,1/4 of about 3 e/Å^3, and three peaks between 0.4 and 1.0 e/Å^3 just off the axis. The pattern of these peaks did not resemble any reasonable molecule that might have been used in the synthesis. It must be presumed that the channel, if occupied, contains solvent molecules that are so irregularly located as to be virtually invisible to the x-ray diffraction technique. The large R factor, and the large error of a reflection of unit weight may be a result of this unresolved structure.

The variable temperature (4.2 - 89.6°K) magnetic susceptibility follows Curie-Weiss behavior, $\chi = \frac{C_M}{T+\theta}$, $C_M = 1.33$ and $\theta = 12°K$. The magnetic moment, μ_{eff}, is 3.27 B.M.

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

ACKNOWLEDGEMENT

We thank Dr. N. M. Edelstein for useful discussions.

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Table I. Positional and Thermal Parameters[a] with Estimated Deviations[b] for $Nd[N(Si(CH_3)_3)_2]_3$

ATOM	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Nd[c]	2/3	1/3	.2903(3)	3.49(7)					
N[c]	.508(1)	.254	1/4	4.3(7)	4.7(6)	2.4(7)	2.15	0	.3(5)
Si	.4543(3)	.2866(3)	.1131(5)	4.5(2)	5.4(2)	4.0(2)	2.8(2)	-.6(2)	-.1(2)
C(1)	.401(1)	.353(1)	.202(2)	8(1)	9(1)	8(1)	6(1)	1(1)	-1(1)
C(2)	.364(1)	.187(1)	-.005(2)	6(1)	8(1)	7(1)	2(1)	-4(1)	-1(1)
C(3)	.546(1)	.367(1)	-.033(2)	7(1)	8(1)	4(1)	4(1)	1(1)	3(1)

[a] The anisotropic temperature factor has the form $\exp(-0.25(B_{11}\underline{h}^2\underline{a}^{*2} + 2B_{12}\underline{hka}^*\underline{b}^* + \ldots))$.

[b] Here and in the following tables the numbers in parenthesis are the estimated standard deviations in the least significant digit.

[c] Symmetry conditions of the special positions for N; $x = 2y$, $B_{11} = 2B_{12}$, and $B_{13} = 0$.

Table II. Interatomic Distances (Å)

		Corrected[a]
Nd - 3N	2.29(2)	2.29
N - 2Si	1.70(1)	1.71
Si - C(1)	1.88(2)	1.91
Si - C(2)	1.86(2)	1.90
Si - C(3)	1.89(2)	1.91

[a]Adjusted for thermal motion assuming the "riding" model.

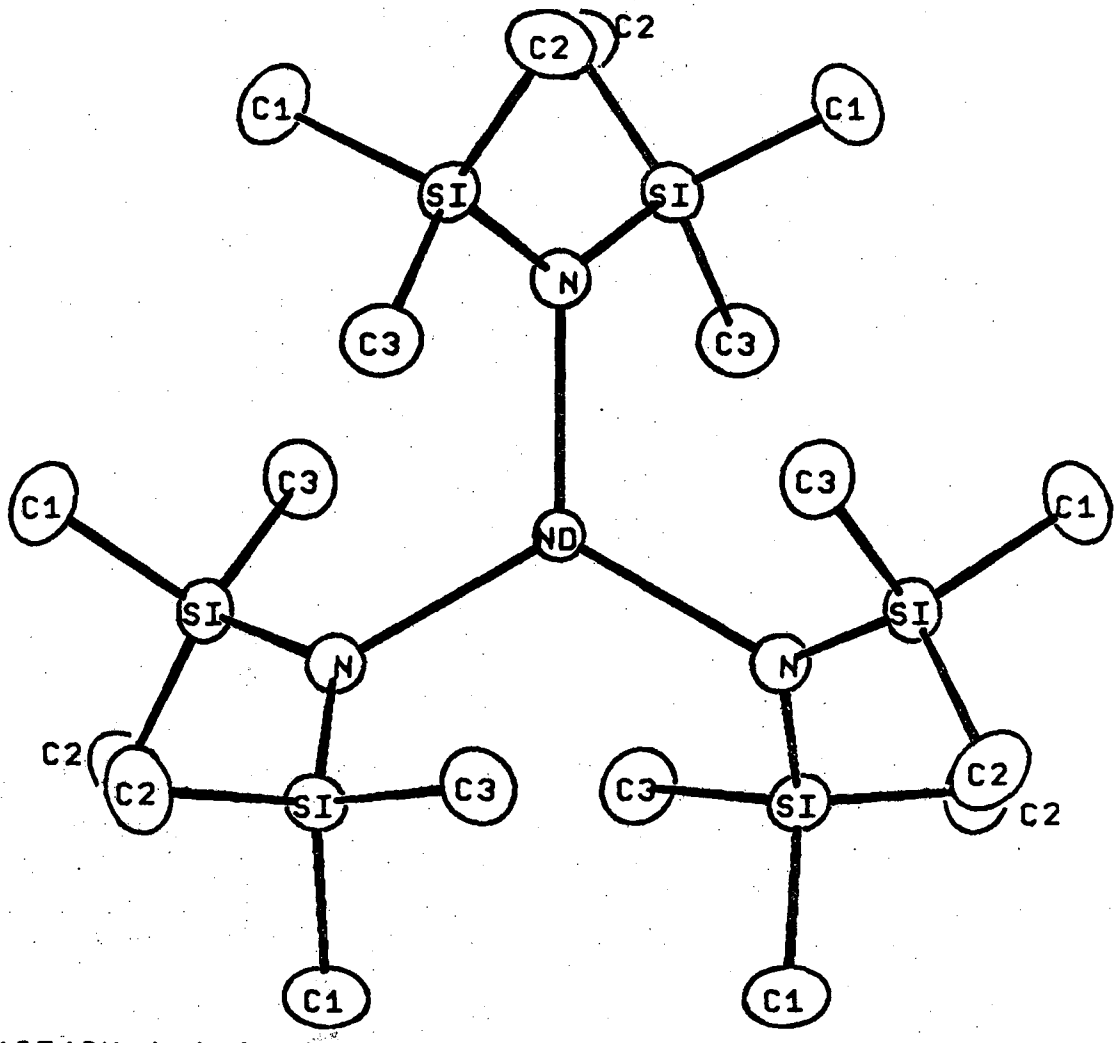
Table III. Selected Angles (deg.)

N - Nd - N	117.8(1)
Nd - N - Si	123.2(5)
Nd - N - Si[a]	110.1(4)
Si - N - Si[a]	126.4(9)
N - Si - C(1)	112.4(6)
N - Si - C(2)	114.0(7)
N - Si - C(3)	108.0(7)
C(1) - Si - C(2)	108.9(9)
C(1) - Si - C(3)	107.2(8)
C(2) - Si - C(3)	105.9(8)

[a]Atom at position x, x-y, 1/2 - z.

FIGURE CAPTION

Fig. 1. ORTEP view of $Nd(N(SiMe_3)_2)_3$ down the c axis.



$ND(N(SI(CH_3)_3)_2)_3$

XBL 782-7162

Fig. 1

SUPPLEMENTARY MATERIALS FOR THE PAPER

Structure of Tris(bis(trimethylsily)amido)neodymium(III), $Nd[N(SI(CH_3)_3)_2]_3$

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DATA PROCESSING FORMULAE

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

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

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

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

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

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

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

When $S(F_a^2) > 4\sigma(F_a^2)$, $\sigma(F_a^2)$ is replaced by $S(F_a^2)$.

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

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

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

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

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

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

C = counts recorded during a scan

I = individual raw intensity, background removed.

t_c = scan count time

t_b = background count time

B_1 = individual background count

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

F = structure factor

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

A = absorption correction

Lp = Lorentz and polarization corrections

θ_m = monochromater angle

θ = crystal diffraction angle

S = scatter

a = average

q = additional uncertainty that affects the weak intensities

p = estimate of non-statistical errors

wtg = weighting factors in least squares

OBSERVED STRUCTURE FACTORS, STANDARD DEVIATIONS, AND DIFFERENCES (ALL X 6.0)
F(0,0,0) = 3276

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

L	FOB	SG	DEL
H,K=	0,	5	
2	406	0	131*
4	208	7	22
6	94	12	-37
8	114	12	-9
H,K=	1,	0	
1	1048	0	12*
2	102	0	-147*
3	334	11	45
4	62	8	-34
5	43	13	36*
6	105	8	47
7	172	10	2
8	38	31	32*
9	69	20	-5
H,K=	1,	1	
0	914	0	306*
2	231	15	93
4	226	9	27
6	85	28	-42*
8	103	22	-19
H,K=	2,	-1	
2	986	30	146
4	352	11	20
6	67	20	63*
8	63	30	41*
H,K=	2,	0	
0	373	0	-266*
1	297	0	-8*
2	75	47	-37*
3	508	16	10
4	92	5	-16
5	138	6	-1
6	101	8	60
7	93	19	-8
8	48	20	32*
9	62	26	-24*
H,K=	2,	1	
0	449	0	219*
1	155	0	58*
2	51	71	-75*
3	206	8	-22
4	215	7	2
5	284	10	3
6	47	35	40*
7	77	44	1*
8	115	9	-43
9	110	15	-24
H,K=	2,	2	
0	531	0	159*
2	0	69	-26*
4	445	14	7
6	127	10	-32
8	193	11	3
H,K=	3,	-1	
1	860	0	38*
2	461	15	-40
3	557	17	11
4	256	8	-18
5	40	20	24*
6	80	11	-37
7	116	21	-12
8	65	15	-19
9	26	56	19*
H,K=	3,	0	
0	482	0	186*
1	404	0	35*
2	899	27	140
3	180	7	-7
4	299	9	-4
5	53	15	37
6	58	12	23
7	144	13	2
8	139	12	-18
H,K=	3,	1	
0	331	0	-100*
1	142	23	12
2	298	11	67
3	377	12	18
4	69	8	-18
5	160	7	6
6	232	8	22
7	113	22	-29
8	54	64	6*
H,K=	3,	2	
0	118	20	-91
1	166	12	-46
2	259	9	7
3	281	9	4
4	41	16	19*
5	205	9	2
6	26	45	-14*
7	26	73	12*
8	0	62	-29*
H,K=	3,	3	
0	612	19	53
2	800	24	81
4	105	11	1
6	53	39	45*
8	59	48	19*
H,K=	4,	-2	
2	502	16	-68
4	296	10	-4
6	305	11	-19
8	25	55	6*
H,K=	4,	-1	
1	591	19	64
2	668	21	22
3	373	12	10
4	78	9	12
5	17	36	-14*
6	51	26	-18*
7	133	21	-25
8	42	48	8*
H,K=	4,	0	
0	313	13	26
1	851	26	46
2	32	42	0*
3	179	6	-11
4	169	6	-8
5	65	10	-33
6	94	8	-31
7	27	67	7*
8	76	14	-11
H,K=	4,	1	
0	560	17	-21
1	21	59	-42*
2	508	16	24
3	85	7	0
4	95	7	19
5	281	10	-5
6	48	23	-9*
7	25	69	-9*
8	39	51	-21*
H,K=	4,	2	
0	277	9	0
1	638	20	-2
2	13	33	-15*
3	284	9	3
4	246	8	10
5	195	9	2
6	99	11	38
7	6	61	-6*
8	43	51	-29*
H,K=	4,	3	
0	283	9	-33
1	558	17	36
2	521	16	-8
3	452	14	-3
4	33	48	-8*
5	86	11	-11
6	53	55	48*
7	76	19	-5
8	46	51	33*
H,K=	4,	4	
0	600	18	72
2	388	12	27
4	431	14	-19
6	33	80	21*
8	0	63	-52*
H,K=	5,	-2	
1	678	21	9
2	168	8	54
3	139	6	-9
4	117	8	12
5	54	24	30*
6	41	42	31*
7	48	72	-16*
8	41	47	10*
H,K=	5,	-1	
1	394	13	46
2	459	14	35
3	209	7	15
4	34	34	-20*
5	78	19	0
6	33	43	-8*
7	0	63	-36*
8	34	57	8*
H,K=	5,	0	
0	28	47	-99*
1	529	16	11
2	403	12	20
3	45	10	29
4	38	16	2*
5	38	39	-11*
6	123	8	22
7	39	49	-1*
8	35	45	27*
H,K=	5,	1	
0	366	12	-38
1	362	11	-35
2	406	13	1
3	49	15	-21*
4	237	8	-2
5	59	15	1
6	96	19	-8
7	18	59	-7*
8	79	20	49
H,K=	5,	2	
0	529	16	60
1	243	8	2
2	605	19	36
3	46	23	13*
4	116	8	-18
5	194	8	-2
6	87	22	37
7	81	11	12
8	55	67	19*
H,K=	5,	3	
0	39	11	19
1	493	15	19
2	136	6	-16
3	252	8	-12
4	347	11	2
5	157	8	-3
6	72	53	2*
7	26	43	14*
8	56	35	-1*
H,K=	5,	4	
0	289	9	-58
1	176	7	-10
2	51	12	34
3	233	9	-13
4	101	9	-22
5	200	8	1
6	103	22	1
7	51	39	26*
H,K=	5,	5	
0	382	12	44
2	213	8	45
4	194	11	-27
6	51	63	7*
H,K=	6,	-3	
2	264	10	-6
4	223	9	4
6	65	28	-12*
8	58	51	-11*
H,K=	6,	-2	
1	341	11	26
2	481	15	14
3	95	7	4
4	192	7	-14
5	237	9	-4
6	76	14	-2
7	70	29	-3*
8	43	48	33*
H,K=	6,	-1	
1	389	12	-35
2	262	8	60
3	221	7	-17
4	26	36	18*
5	85	12	1
6	203	8	23
7	24	47	0*
8	40	62	-2*
H,K=	6,	0	
0	817	25	86
1	25	35	-12*
2	184	6	2
3	37	15	28*

STRUCTURE FACTORS CONTINUED FOR

L	FOB	SG	DEL
4	143	6	-2
5	37	38	16*
6	31	52	27*
7	15	38	13*
8	34	50	-12*
H,K=		6,	1
0	255	8	-45
1	458	14	20
2	0	33	-63*
3	352	11	-1
4	163	8	-7
H,K=		7,	-2
1	23	28	-7*
2	281	9	29
3	393	12	3
4	260	9	1
5	92	10	-12
6	107	21	-9
7	69	19	-4
8	68	23	-22*
H,K=		7,	-1
1	297	9	34
2	120	6	-31
3	219	7	10
4	82	14	13
5	184	8	13
6	50	64	12*
7	34	42	19*
8	0	57	-38*
H,K=		7,	0
0	30	30	-41*
1	378	12	7
2	181	6	-24
3	251	8	-22
4	345	11	-1
5	28	35	18*
6	51	66	4*
7	51	19	26*
8	96	13	-8
H,K=		7,	1
0	366	11	37
1	230	8	16
2	401	13	41
3	184	7	12
4	266	9	-11
5	81	14	10
6	68	34	24*
7	43	65	-16*
H,K=		7,	2
0	132	5	-42
1	237	8	5
2	221	8	-40
3	149	8	7
4	46	27	33*
5	125	11	10
6	70	13	14
7	5	51	0*
H,K=		7,	3
0	261	9	-32
1	473	15	6
2	33	44	23*
3	86	10	14
4	54	24	34*
5	34	74	15*
6	141	8	11
7	36	67	33*
H,K=		7,	4
0	472	15	34
1	121	8	8
2	335	11	20
3	102	9	-2
4	39	56	6*
5	64	37	2*
6	41	50	9*
7	26	52	1*
H,K=		7,	5
0	198	8	-24
1	225	8	2
2	224	8	-8
3	217	8	7
4	43	73	-10*
5	0	45	-21*
6	0	57	-12*
H,K=		7,	6
0	21	37	2*
1	229	9	-7
2	120	11	-15
3	261	12	3
4	83	28	0*
5	37	50	-3*
6	59	37	0*
H,K=		7,	7
0	269	9	-1
2	249	13	13
4	0	58	-61*
H,K=		8,	-4
2	538	17	19
4	390	12	-12
6	60	88	24*
8	37	64	-21*
H,K=		8,	-3
1	464	14	13
2	195	7	-37
3	266	9	0
4	280	9	5
5	61	17	-16
6	45	68	36*
7	27	46	-6*
8	93	17	4
H,K=		8,	-2
1	122	6	-9
2	117	6	-25
3	327	11	0
4	97	9	5
5	43	31	10*
6	51	68	45*
7	32	54	-17*
8	41	57	35*
H,K=		8,	-1
1	36	23	13*
2	309	10	19
3	200	8	5
4	248	8	6
5	112	9	-5
6	44	56	41*
7	61	25	-4*
H,K=		8,	0
0	349	11	-33
1	83	6	-26
2	28	30	3*
3	501	15	-8
4	102	8	6
5	124	9	2
6	35	48	24*
7	133	11	-9
H,K=		8,	1
0	66	9	29
1	273	9	-8
2	184	8	-30
3	95	9	-14
4	178	7	-6
5	38	71	30*
6	28	54	20*
7	19	54	-23*
H,K=		8,	2
0	532	16	47
1	22	43	-9*
2	226	8	12
3	42	42	8*
4	60	20	50*
5	29	57	0*
6	42	31	37*
7	36	59	35*
H,K=		8,	3
0	304	10	-27
1	376	12	-9
2	73	11	7
3	57	17	34*
4	63	42	51*
5	13	54	-3*
6	59	34	31*
7	0	57	-11*
H,K=		8,	4
0	37	44	-4*
1	237	8	6
2	107	13	-13
3	105	12	3
4	25	64	-21*
5	40	45	8*
6	45	57	0*
H,K=		8,	5
0	144	7	9
1	52	40	2*
2	191	9	16
3	52	58	51*
4	100	11	-14
5	59	26	44*
6	0	58	-4*
H,K=		8,	6
0	142	9	-12
1	136	18	0
2	75	37	-41*
3	124	10	-8
4	67	20	45*
5	0	52	-34*
H,K=		8,	7
0	150	8	-5
1	245	8	4
2	60	16	-19
3	78	16	19
4	8	51	-5*
H,K=		8,	8
0	337	12	21
2	164	12	17
H,K=		9,	-4
1	329	18	8
2	333	10	-29
3	443	14	-9
4	58	17	-1
5	55	40	8*
6	37	56	-33*
7	65	21	12*
H,K=		9,	-3
1	368	12	12
2	502	15	35
3	60	13	12
4	79	16	-12
5	117	14	-5
6	16	69	-14*
7	97	14	69
H,K=		9,	-2
1	393	12	9
2	25	35	-65*
3	223	8	-9
4	29	40	14*
5	59	37	51*
6	94	13	9
7	48	58	23*
H,K=		9,	-1
1	215	8	-1
2	235	8	-19
3	113	8	-19
4	208	8	-11
5	51	61	1*
6	56	25	41*

STRUCTURE FACTORS CONTINUED FOR

L	FOB	SG	DEL
7	69	21	41*
H,K=	9,	0	
0	144	7	21
1	31	32	7*
2	302	10	24
3	171	7	7
4	155	8	-9
5	57	59	10*
6	43	19	29*
7	60	23	29*
H,K=	9,	1	
0	26	37	-42*
1	346	11	3
2	60	14	17
3	163	7	-2
4	59	36	-3*
5	10	69	6*
6	26	50	16*
7	57	41	1*
H,K=	9,	2	
0	261	8	-18
1	273	9	-4
2	210	8	-2
3	100	12	12
4	60	70	25*
5	49	23	0*
6	32	51	-31*
H,K=	9,	3	
0	360	11	17
1	97	9	0
2	180	8	23
3	89	24	39
4	52	64	-7*
5	61	17	-14
6	73	21	37
H,K=	9,	4	
0	135	7	2
1	71	20	7
2	53	71	43*
3	107	15	-12
4	30	47	6*
5	81	20	11
6	101	16	-7
H,K=	9,	5	
0	42	54	3*
1	52	71	-4*
2	0	61	-7*
3	82	14	-31
4	85	36	-4*
5	115	13	2
H,K=	9,	6	
0	226	8	6
1	27	41	14*
2	105	11	-9
3	36	62	3*
4	114	12	18

L	FOB	SG	DEL
H,K=	9,	7	
0	111	9	-10
1	220	9	9
2	109	18	27
3	42	49	9*
H,K=	9,	8	
0	99	10	-3
1	197	13	5
2	62	34	2*
H,K=	10,	-5	
2	509	17	18
4	151	12	6
6	23	55	20*
H,K=	10,	-4	
1	572	18	11
2	0	43	-41*
3	251	9	4
4	120	10	-14
5	63	66	14*
6	37	53	-12*
7	66	22	64*
H,K=	10,	-3	
1	345	11	-3
2	298	10	-30
3	139	8	0
4	116	9	10
5	29	72	-7*
6	56	21	13*
7	53	44	30*
H,K=	10,	-2	
1	24	42	16*
2	260	9	23
3	48	23	21*
4	53	20	16*
5	46	63	29*
6	51	31	4*
7	33	61	24*
H,K=	10,	-1	
1	211	8	6
2	111	8	-20
3	137	8	4
4	66	32	15*
5	95	33	-2*
6	0	49	-20*
7	34	50	29*
H,K=	10,	0	
0	299	10	-15
1	153	6	6
2	119	6	-10
3	238	8	7
4	33	58	-2*
5	95	11	5
6	52	55	37*
H,K=	10,	1	
0	285	9	17
1	184	7	6

L	FOB	SG	DEL
2	374	12	25
3	91	12	20
4	114	22	-9
5	124	8	6
6	60	31	33*
H,K=	10,	2	
0	101	10	-6
1	330	11	8
2	85	16	1
3	162	19	1
4	94	22	-12
5	66	26	35*
6	29	69	-23*
H,K=	10,	3	
0	66	23	14*
1	119	12	4
2	100	25	-20
3	80	18	-24
4	60	20	8*
5	30	55	-47*
H,K=	10,	4	
0	164	11	12
1	36	46	30*
2	0	59	-56*
3	32	43	18*
4	166	12	2
5	53	49	38*
H,K=	10,	5	
0	43	32	-3*
1	51	33	-25*
2	69	13	3
3	75	19	-12
4	119	13	-5
H,K=	10,	6	
0	117	8	-2
1	131	11	2
2	77	17	25
3	139	11	13
H,K=	10,	7	
0	144	8	-4
1	0	56	-10*
2	169	12	4
H,K=	11,	-5	
1	213	8	8
2	185	8	-30
3	104	10	1
4	65	19	44*
5	120	15	-17
6	50	39	25*
7	18	51	-17*
H,K=	11,	-4	
1	60	19	-8*
2	294	9	13
3	0	44	-5*
4	84	15	56
5	31	62	-6*

L	FOB	SG	DEL
6	57	28	9*
7	45	50	15*
H,K=	11,	-3	
1	354	11	-2
2	130	9	-11
3	49	21	35*
4	50	60	-2*
5	0	56	-5*
6	52	37	44*
7	40	51	9*
H,K=	11,	-2	
1	318	10	11
2	122	9	23
3	113	10	1
4	39	68	26*
5	6	47	-4*
6	99	14	24
H,K=	11,	-1	
1	32	42	30*
2	201	9	25
3	134	12	16
4	134	18	-10
5	21	52	6*
6	26	51	-9*
H,K=	11,	0	
0	126	8	11
1	265	9	2
2	227	8	-8
3	153	10	-13
4	137	8	-7
5	64	18	7
6	25	50	10*
H,K=	11,	1	
0	169	7	1
1	256	9	7
2	141	15	-3
3	223	12	0
4	27	49	17*
5	65	24	-8*
6	55	37	45*
H,K=	11,	2	
0	233	9	19
1	33	62	-24*
2	263	11	-3
3	0	48	-5*
4	62	44	-16*
5	0	67	-19*
H,K=	11,	3	
0	134	12	-20
1	123	15	-6
2	105	11	2
3	105	12	-3
4	53	37	20*
5	85	19	20
H,K=	11,	4	
0	130	7	-3

L	FOB	SG	DEL
1	96	9	-7
2	23	48	-1*
3	148	10	15
4	52	52	9*
H,K=	11,	5	
0	151	8	11
1	76	20	18
2	152	11	25
3	62	35	27*
H,K=	11,	6	
0	44	28	22*
1	147	10	2
2	81	19	-6
H,K=	12,	-6	
2	0	47	-19*
4	215	12	-1
6	54	69	-8*
H,K=	12,	-5	
1	86	9	6
2	41	35	24*
3	107	11	10
4	79	41	-7*
5	132	9	-12
6	29	50	-7*
H,K=	12,	-4	
1	314	10	1
2	59	17	-5
3	98	14	-4
4	48	65	0*
5	33	44	17*
6	10	50	3*
H,K=	12,	-3	
1	145	8	-7
2	194	8	18
3	71	32	6*
4	0	68	-43*
5	48	27	-17*
6	45	55	42*
H,K=	12,	-2	
1	118	9	-5
2	185	8	-12
3	71	40	9*
4	26	47	-17*
5	93	15	15
6	60	33	20*
H,K=	12,	-1	
1	129	8	-11
2	56	61	29*
3	152	13	-10
4	67	14	8
5	129	17	13
6	41	52	-21*
H,K=	12,	0	
0	205	10	25
1	37	52	32*
2	236	10	-2

STRUCTURE FACTORS CONTINUED FOR

L	FOB	SG	DEL
3	19	45	6*
4	143	7	-3
5	46	58	31*
H,K=	12,	1	
0	149	11	-14
1	191	9	-1
2	158	10	-10
3	157	8	-6
4	49	51	45*
5	31	51	-16*
H,K=	12,	2	
0	33	37	-20*
1	314	10	14
2	0	49	-43*
3	128	11	10
4	95	15	9
H,K=	12,	3	
0	235	8	5
1	48	42	13*
2	145	13	-1
3	0	50	-12*
4	31	51	-22*
H,K=	12,	4	
0	11	43	-9*
1	197	11	22
2	10	56	-18*
3	82	20	-1
H,K=	12,	5	
0	106	14	-5
1	81	18	-15
H,K=	13,	-6	
1	85	11	1
2	41	52	25*
3	113	21	-9
4	114	13	-4
5	150	10	9
6	75	35	66*
H,K=	13,	-5	
1	127	8	3
2	205	9	9
3	24	68	-5*
4	154	9	-12
5	42	57	40*
6	78	24	46*
H,K=	13,	-4	
1	185	8	11
2	191	12	-8
3	186	11	-11
4	20	55	11*
5	37	50	28*
6	12	57	-10*
H,K=	13,	-3	
1	274	10	9
2	79	34	7*
3	134	11	-11
4	66	15	-9
5	32	70	24*
H,K=	13,	-2	
1	42	56	9*
2	139	17	-9
3	32	41	-1*
4	99	19	-28
5	32	51	20*
H,K=	13,	-1	
1	134	17	-12
2	21	53	13*
3	147	8	0
4	68	37	22*
5	71	23	-20*
H,K=	13,	0	
0	51	25	-9*
1	190	8	-9
2	119	8	7
3	83	14	-4
4	108	13	1
H,K=	13,	1	
0	241	8	4
1	105	10	-8
2	209	10	7
3	43	50	32*
4	35	51	-32*
H,K=	13,	2	
0	127	8	1
1	168	10	16
2	144	10	5
3	84	17	8
H,K=	13,	3	
0	122	11	-3
1	152	10	11
2	104	22	23
H,K=	13,	4	
0	168	9	10
H,K=	14,	-7	
2	152	18	8
4	126	16	10
H,K=	14,	-6	
1	181	13	9
2	44	64	-55*
3	155	8	-8
4	106	21	26
5	43	55	36*
H,K=	14,	-5	
1	248	12	9
2	140	12	-16
3	208	8	2
4	47	58	-2*
5	34	50	26*
H,K=	14,	-4	
1	0	63	-18*
2	296	10	6
3	50	20	29*
4	134	11	11
5	0	75	-8*
H,K=	14,	-3	
1	193	11	5
2	45	32	-1*
3	156	8	1
4	71	21	9*
5	0	52	-19*
H,K=	14,	-2	
1	103	9	-21
2	161	10	11
3	50	54	10*
4	64	24	8*
H,K=	14,	-1	
1	72	18	29
2	121	11	5
3	56	61	-28*
4	84	27	4*
H,K=	14,	0	
0	119	15	-9
1	161	8	10
2	63	21	-4*
3	128	10	10
H,K=	14,	1	
0	95	13	17
1	164	10	5
2	41	71	5*
3	116	14	5
H,K=	14,	2	
0	150	10	0
1	0	63	-5*
H,K=	15,	-7	
1	258	10	12
2	111	12	-9
3	0	49	-59*
4	34	50	3*
H,K=	15,	-6	
1	25	49	-10*
2	232	8	5
3	0	49	-6*
4	0	51	-44*
H,K=	15,	-5	
1	204	8	-2
2	79	23	-8
3	154	11	-5
4	92	15	-6
H,K=	15,	-4	
1	134	8	2
2	88	15	-10
3	133	11	-13
4	45	63	23*
H,K=	15,	-3	
1	73	17	13
2	116	15	11
3	22	49	1*
4	70	22	18*
H,K=	15,	-2	
1	181	10	5
2	60	61	49*
3	86	16	8
H,K=	15,	-1	
1	91	16	-20
2	109	13	0
3	86	25	5
H,K=	15,	0	
0	106	14	10
1	0	47	-32*
2	125	10	-8
H,K=	16,	-8	
2	151	13	6
H,K=	16,	-7	
1	202	10	-3
2	0	52	-36*
3	60	26	5*
H,K=	16,	-6	
1	102	13	-3
2	58	35	-27*
3	106	17	-7
H,K=	16,	-5	
1	75	18	30
2	85	17	-14
3	39	55	-21*
H,K=	16,	-4	
1	113	13	-17
2	21	49	-39*
3	0	74	-37*
H,K=	16,	-3	
1	109	13	-15
2	54	38	5*
H,K=	16,	-2	
1	70	24	54*
H,K=	17,	-8	
1	164	9	20
2	46	62	-26*
H,K=	17,	-7	
1	34	68	31*
2	86	17	-16
H,K=	17,	-6	
1	57	28	-4*
2	84	27	24*
H,K=	17,	-5	
1	78	18	4

L	FOB	SG	DEL

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