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STRUCTURE OF TRIS(DIS(TRIMETHYLSILYL)AMIDO)NEODYMIUM (III), Nd[N(Si(CE3)3)2]3

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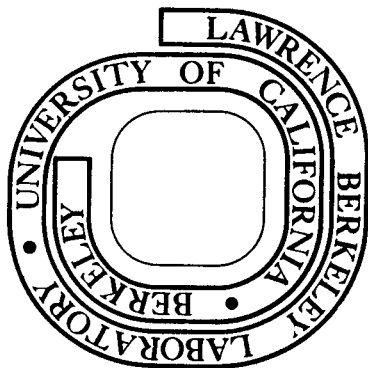
Richard A. Andersen, David H. Templeton, and
Allan Zalkin

March 1978

Prepared for the U. S. Department of Energy
under Contract W-7405-ENG-48

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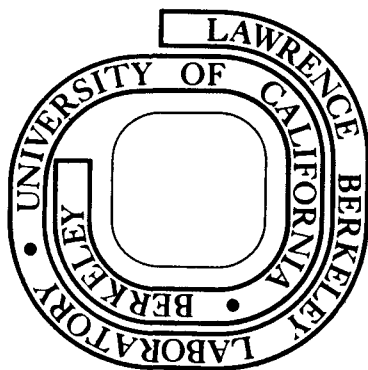
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Contribution from Materials and Molecular
Research Division, Lawrence Berkeley Laboratory,
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University of California, Berkeley, California 94720

Structure of Tris(bis(trimethylsilyl)amido)neodymium(III),
 $\text{Nd}[\text{N}(\text{Si}(\text{CH}_3)_2)_3]_3^1$

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 $\text{M}[\text{N}(\text{SiMe}_3)_2]_3$ are planar.⁴ We describe the crystal structure of $\text{Nd}[\text{N}(\text{SiMe}_3)_2]_3$ and show that it is also non-planar.

EXPERIMENTAL

The $\text{Nd}[\text{N}(\text{SiMe}_3)_2]_3$ 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 $\text{HgCo}(\text{CNS})_4$.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 ($\lambda(\text{K}\alpha_1)$ 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 001 type reflections respectively. The space group was identified as $P\bar{3}1c$. The setting angles of 12 manually centered reflections ($19^\circ < 2\theta < 25^\circ$) were used to determine by least squares the cell parameters $a = 16.476(13)$ Å, $c = 8.485(7)$ Å, and $V = 1995$ Å³. 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 $21 \pm 1^\circ\text{C}$. 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 percent 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 $\sum w(|F_o| - |F_c|)^2 / \sum w F_o^2$ 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 Turner⁶ were used, and dispersion corrections⁷ 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 Eu³ compounds. The Nd atom is on a crystallographic 3-fold axis and is bonded to three nitrogen atoms. In the Sc and Eu isomorphs⁴ the metal atoms are disordered in the z direction, and were treated as two half atoms $\sim 0.6 \text{ \AA}$ above and below the plane at $z = 1/4$. This structure is similar with Nd 0.34 \AA 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 \AA^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 Rodesiler⁹ 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 $3 e/\text{\AA}^3$, and three peaks between 0.4 and $1.0 e/\text{\AA}^3$ 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 $\theta = 12^\circ\text{K}$. 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

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Table I. Positional and Thermal Parameters^a with Estimated Deviations^b for Nd[N(Si(CH₃)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)

^aThe anisotropic temperature factor has the form $\exp(-0.25(B_{11}h^2a^{*2} + 2B_{12}hka^*b^* + \dots))$.

^bHere and in the following tables the numbers in parenthesis are the estimated standard deviations in the least significant digit.

^cSymmetry 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

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

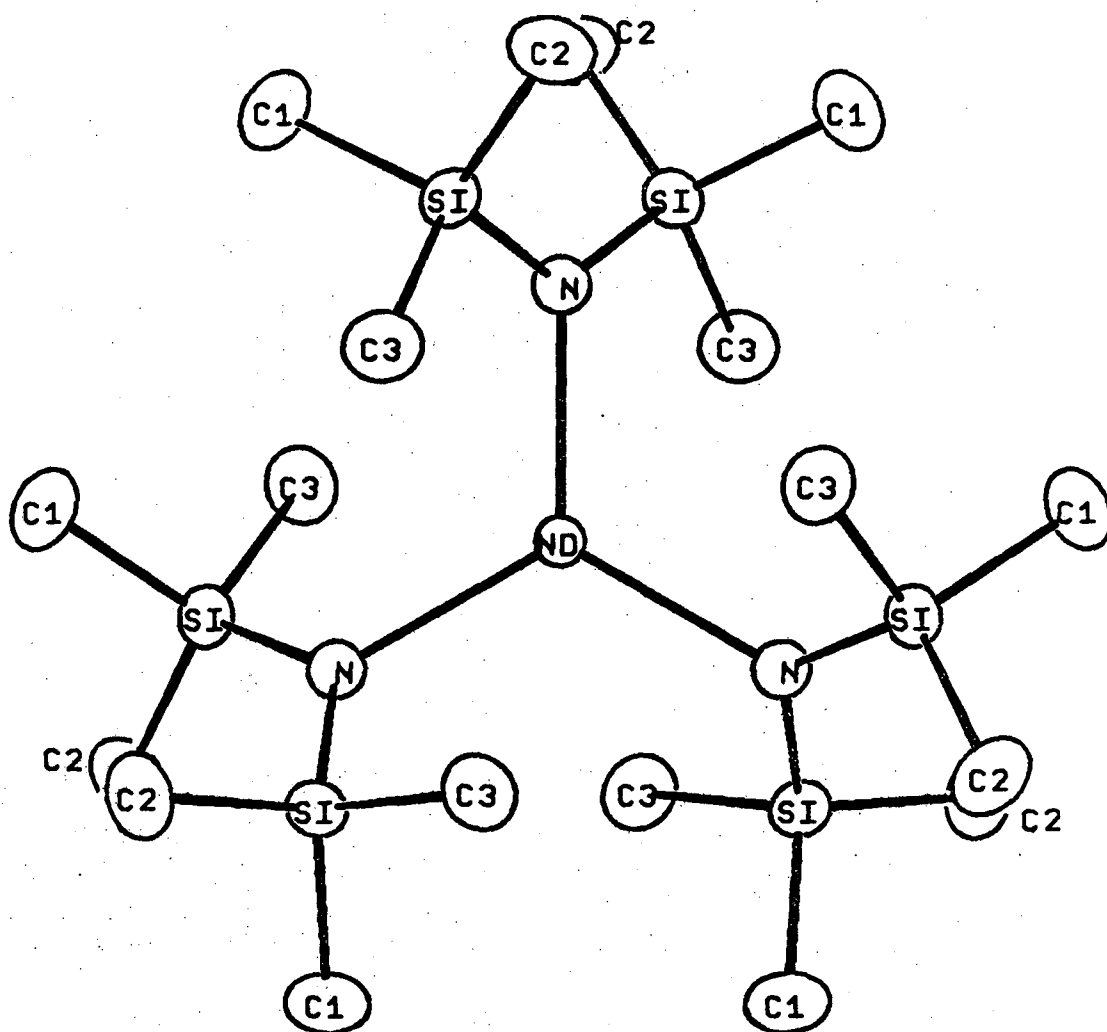
Table III. Selected Angles (deg.)

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)

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

FIGURE CAPTION

Fig. 1. ORTEP view of $\text{Nd}(\text{N}(\text{SiMe}_3)_2)_3$ down the c axis.

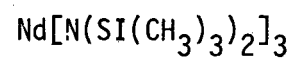


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Fig. 1

SUPPLEMENTARY MATERIALS FOR THE PAPER

Structure of Tris(bis(trimethylsily)amido)neodymium(III),



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DATA PROCESSING FORMULAE

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

L_p = Lorentz and polarization corrections

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	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL			
	H,K= 0, 0			6	127	10	-32	8	25	55	6*	2	388	12	27	H,K= 5, 3						
2	406	0	131*	8	193	11	3	H,K= 4, -1	4	431	14	-19	0	39	11	19						
4	208	7	22	H,K= 3, -1	1	591	19	64	6	33	80	21*	1	493	15	19						
6	94	12	-37	1	860	0	38*	2	668	21	22	8	0	63	-52*	2	136	6	-16			
8	114	12	-9	2	461	15	-40	3	373	12	10	H,K= 5, -2	3	252	8	-12						
H,K= 1, 0				3	557	17	11	4	78	9	12	1	678	21	9	4	347	11	2			
110	48	0	12*	4	256	8	-18	5	17	36	-14*	2	168	8	54	5	157	8	-3			
2	102	0	-147*	5	40	20	24*	6	51	26	-18*	3	139	6	-9	6	72	53	2*			
3	334	11	45	6	80	11	-37	7	133	21	-25	4	117	8	12	7	26	43	14*			
4	62	8	-34	7	116	21	-12	8	42	48	8*	5	54	24	30*	8	56	35	-1*			
5	43	13	36*	8	65	15	-19	H,K= 4, 0	6	41	42	31*	H,K= 5, 4									
6	105	8	47	9	26	56	19*	0	313	13	26	7	48	72	-16*	0	289	9	-50			
7	172	10	2	H,K= 3, 0	0	482	0	186*	1	851	26	46	8	41	47	10*	1	176	7	-10		
8	38	31	32*	0	482	0	186*	2	32	42	0*	H,K= 5, -1	2	51	12	34						
9	69	20	-5	1	404	0	35*	3	179	6	-11	1	394	13	46	3	233	9	-13			
H,K= 1, 1				2	899	27	140	4	169	6	-8	2	459	14	35	4	181	9	-22			
0	914	0	306*	3	180	7	-7	5	65	10	-33	3	209	7	15	5	200	8	1			
2	231	15	93	4	299	9	-4	6	94	8	-31	4	34	34	-20*	6	103	22	1			
4	226	9	27	5	53	15	37	7	27	67	7*	5	78	19	0	7	51	39	26*			
6	85	28	-42*	6	58	12	23	8	76	14	-11	6	33	43	-8*	H,K= 5, 5						
8	103	22	-19	7	144	13	2	H,K= 4, 1	7	0	63	-36*	0	382	12	44						
H,K= 2, -1				8	139	12	-18	0	560	17	-21	8	34	57	8*	2	213	8	45			
2	986	30	146	H,K= 3, 1	1	21	59	-42*	H,K= 5, 0	0	28	47	-99*	4	194	11	-27					
4	352	11	20	0	331	0	-100*	2	508	16	24	0	28	47	-99*	6	51	63	7*			
6	67	20	63*	1	142	23	12	3	85	7	0	1	529	16	11	H,K= 6, -3						
8	63	30	41*	2	298	11	67	4	95	7	19	2	403	12	20	2	264	10	-6			
H,K= 2, 0				3	377	12	18	5	281	10	-5	3	45	16	29	4	223	9	4			
0	373	0	-266*	4	69	8	-18	6	48	23	-9*	4	38	16	2*	6	65	28	-12*			
1	297	0	-8*	5	160	7	6	7	25	69	-9*	5	38	39	-11*	8	58	51	-11*			
2	75	47	-37*	6	232	8	22	8	39	51	-21*	6	123	8	22	H,K= 6, -2						
3	508	16	16	7	113	22	-29	H,K= 4, 2	0	277	9	0	7	39	49	-1*	1	341	11	26		
4	92	5	-16	8	54	64	6*	0	277	9	0	8	35	45	27*	2	481	15	14			
5	138	6	-1	H,K= 3, 2	0	118	20	-91	1	638	20	-2	H,K= 5, 1	0	366	12	-38	3	95	7	4	
6	101	8	60	0	118	20	-91	2	13	33	-15*	0	366	12	-38	4	192	7	-14			
7	93	19	-8	1	166	12	-46	3	284	9	3	1	362	11	-35	5	237	9	-4			
8	48	20	32*	2	259	9	7	4	246	8	10	2	406	13	1	6	76	14	-2			
9	62	26	-24*	3	281	9	4	5	195	9	2	3	49	15	-21*	7	70	29	-3*			
H,K= 2, 1				4	41	16	19*	6	99	11	38	4	237	8	-2	8	43	48	33*			
0	449	0	219*	5	205	9	2	7	6	61	-6*	5	59	15	1	H,K= 6, -1						
1	155	0	58*	6	26	45	-14*	8	43	51	-29*	6	96	19	-8	1	389	12	-35			
2	51	71	-75*	7	26	73	12*	H,K= 4, 3	0	283	9	-33	7	18	59	-7*	2	262	8	60		
3	206	8	-22	8	0	62	-29*	0	283	9	-33	8	79	20	49	3	221	7	-17			
4	215	7	2	H,K= 3, 3	0	612	19	53	1	558	17	36	H,K= 5, 2	4	26	36	10*					
5	284	10		0	612	19	53	2	521	16	-8	0	529	16	60	5	85	12	1			
6	47	35	40*	2	800	24	81	3	452	14	-3	1	243	8	2	6	203	8	23			
7	77	44	1*	4	105	11	1	4	33	48	-8*	2	605	19	36	7	24	47	0*			
8	115	9	-43	6	53	39	45*	5	86	11	-11	3	46	23	13*	8	40	62	-2*			
9	110	15	-24	8	59	48	19*	6	53	55	48*	4	116	8	-18	H,K= 6, 0						
H,K= 2, 2				7	76	19	-5	7	76	19	-5	5	194	8	-2	0	817	25	86			
0	531	0	159*	2	502	16	-68	8	46	51	33*	6	87	22	37	1	25	35	-12*			
2	0	69	-26*	4	296	10	-4	H,K= 4, 4	0	600	18	72	7	81	11	12	2	184	6	2		
4	445	14	7	6	305	11	-19	0	600	18	72	8	55	67	19*	3	37	15	28*			

STRUCTURE FACTORS CONTINUED FOR

L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL		
4	143	6	-2	6	129	15	31	H,K=	7,	3	4	97	9	5	6	45	57	0*			
5	37	38	16*	H,K=	7,	-3	0	261	9	-32	5	43	31	10*	H,K=	8,	5				
6	31	52	27*	1	462	14	32	1	473	15	6	6	51	68	45*	8	144	7	9		
7	15	38	13*	2	64	8	-34	2	33	44	23*	7	32	54	-17*	1	52	40	2*		
8	34	50	-12*	3	401	12	-10	3	86	10	14	8	41	57	35*	2	191	9	16		
H,K=	6,	1		4	50	16	-8*	4	54	24	34*	H,K=	8,	-1	3	52	58	51*			
0	255	8	-45	5	211	8	-11	5	34	74	15*	1	36	23	13*	4	100	11	-14		
1	458	14	20	6	143	14	27	6	141	8	11	2	309	10	19	5	59	26	44*		
2	0	33	-63*	7	59	21	3*	7	36	67	33*	3	200	8	5	6	0	58	-4*		
3	352	11	-1	8	40	49	0*	H,K=	7,	4	4	248	8	6	H,K=	8,	6				
4	163	8	-7	H,K=	7,	-2	0	472	15	34	5	112	9	-5	8	142	9	-12			
5	42	45	-8*	1	23	28	-7*	1	121	8	8	6	44	56	41*	1	136	18	0		
6	66	67	50*	2	281	9	29	2	335	11	20	7	61	25	-4*	2	75	37	-41*		
7	38	45	-9*	3	393	12	3	3	102	9	-2	H,K=	8,	0	3	124	10	-8			
8	33	62	0*	4	260	9	1	4	39	56	6*	0	349	11	-33	4	67	20	45*		
H,K=	6,	2		5	92	10	-12	5	64	37	2*	1	83	6	-26	5	0	52	-34*		
0	256	8	-41	6	107	21	-9	6	41	50	9*	2	28	30	3*	H,K=	8,	7			
1	377	12	-8	7	69	19	-4	7	26	52	1*	3	501	15	-8	0	150	8	-5		
2	141	6	-28	8	68	23	-22*	H,K=	7,	5	4	102	8	6	1	245	8	4			
3	345	11	-4	H,K=	7,	-1	0	198	8	-24	5	124	9	2	2	60	16	-19			
4	56	19	9*	1	297	9	34	1	225	8	2	6	35	48	24*	3	78	16	19		
5	103	10	2	2	120	6	-31	2	224	8	-8	7	133	11	-9	4	8	51	-5*		
6	85	32	20*	3	219	7	10	3	217	8	7	H,K=	8,	1	H,K=	8,	8				
7	74	19	26	4	82	14	13	4	43	73	-10*	0	66	9	29	0	337	12	21		
8	41	70	30*	5	184	8	13	5	0	45	-21*	1	273	9	-8	2	164	12	17		
H,K=	6,	3		6	50	64	12*	6	8	57	-12*	2	184	8	-30	H,K=	9,	-4			
0	461	14	55	7	34	42	19*	H,K=	7,	6	3	95	9	-14	1	329	10	8			
1	0	38	-44*	8	0	57	-38*	0	21	37	2*	4	178	7	-6	2	333	10	-29		
2	250	9	33	H,K=	7,	0	1	229	9	-7	5	38	71	30*	3	443	14	-9			
3	185	8	5	0	30	30	-41*	2	120	11	-15	6	28	54	20*	4	58	17	-1		
4	219	8	0	1	378	12	7	3	261	12	3	7	19	54	-23*	5	55	40	8*		
5	94	17	17	2	181	6	-24	4	83	28	0*	H,K=	8,	2	6	37	56	-33*			
6	63	66	-25*	3	251	8	-22	5	37	50	-3*	0	532	16	47	7	65	21	12*		
7	98	14	20	4	345	11	-1	6	59	37	0*	1	22	43	-9*	H,K=	9,	-3			
H,K=	6,	4		5	28	35	18*	H,K=	7,	7	2	226	8	12	1	368	12	12			
0	117	6	-36	6	51	66	4*	0	269	9	-1	3	42	42	8*	2	502	15	35		
1	241	8	-7	7	51	19	26*	2	249	13	13	4	60	20	50*	3	60	13	12		
2	197	8	-23	8	96	13	-8	4	0	58	-61*	5	29	57	0*	4	79	16	-12		
3	46	22	11*	H,K=	7,	1	H,K=	8,	-4	6	42	31	37*	7	36	59	35*	5	117	14	-5
4	134	8	-7	0	366	11	37	2	538	17	19	7	36	59	35*	6	16	69	-14*		
5	156	16	10	1	230	8	16	4	390	12	-12	H,K=	8,	3	7	97	14	69			
6	67	27	-1*	2	401	13	41	6	60	88	24*	0	304	10	-27	H,K=	9,	-2			
7	0	58	-25*	3	184	7	12	8	37	64	-21*	1	376	12	-9	1	393	12	9		
H,K=	6,	5		4	266	9	-11	H,K=	8,	-3	2	73	11	7	2	25	35	-65*			
0	45	13	-33*	5	81	14	10	1	464	14	13	3	57	17	34*	3	223	8	-9		
1	346	11	2	6	68	34	24*	2	195	7	-37	4	63	42	51*	4	29	40	14*		
2	134	8	-28	7	43	65	-16*	3	266	9	0	5	13	54	-3*	5	59	37	51*		
3	280	9	-9	H,K=	7,	2	4	280	9	5	7	0	57	-11*	6	94	13	9			
4	47	57	18*	0	132	5	-42	5	61	17	-16	7	0	57	-11*	7	48	58	23*		
5	15	69	4*	1	237	8	5	6	45	68	36*	H,K=	8,	4	H,K=	9,	-1				
6	31	49	8*	2	221	8	-40	7	27	46	-6*	0	37	44	-4*	1	215	8	-1		
7	77	22	40	3	149	8	7	8	93	17	4	1	237	8	6	2	235	8	-19		
H,K=	6,	6		4	46	27	33*	H,K=	8,	-2	2	107	13	-13	3	113	8	-19			
0	152	9	20	5	125	11	10	1	122	6	-9	3	105	12	3	4	208	8	-11		
1	490	16	17	6	70	13	14	2	117	6	-25	4	25	64	-21*	5	51	61	1*		
4	160	28	13	7	5	51	0*	3	327	11	0	5	40	45	8*	6	56	25	41*		

STRUCTURE FACTORS CONTINUED FOR

L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL
7	69	21	41*	H,K= 9,	7	2	374	12	25	6	57	28	9*	1	96	9	-7		
	H,K= 9,	0		0	111	9	-10	3	91	12	20	7	45	50	15*	2	23	48	-1*
0	144	7	21	1	220	9	9	4	114	22	-9		H,K= 11,	-3	3	148	10	15	
1	31	32	7*	2	109	18	27	5	124	8	6	1	354	11	-2	4	52	52	9*
2	302	10	24	3	42	49	9*	6	60	31	33*	2	130	9	-11		H,K= 11,	5	
3	171	7	7		H,K= 9,	8			H,K= 10,	2		3	49	21	35*	0	151	8	11
4	155	8	-9	6	99	10	-3	0	101	10	-6	4	50	60	-2*	1	76	20	18
5	57	59	10*	1	197	13	5	1	330	11	8	5	0	56	-5*	2	152	11	25
6	43	19	29*	2	62	34	2*	2	85	16	1	6	52	37	44*	3	62	35	27*
7	60	23	29*		H,K= 10,	-5		3	162	19	1	7	40	51	9*		H,K= 11,	6	
	H,K= 9,	1		2	509	17	18	4	94	22	-12		H,K= 11,	-2	0	44	28	22*	
0	26	37	-42*	4	151	12	6	5	66	26	35*	1	318	10	11	1	147	10	2
1	346	11	3	6	23	55	20*	6	29	69	-23*	2	122	9	23	2	81	19	-6
2	60	14	17		H,K= 10,	-4			H,K= 10,	3		3	113	10	1		H,K= 12,	-6	
3	163	7	-2	1	572	18	11	0	66	23	14*	4	39	68	26*	2	0	47	-19*
4	59	36	-3*	2	0	43	-41*	1	119	12	4	5	6	47	-4*	4	215	12	-1
5	10	69	6*	3	251	9	4	2	100	25	-20	6	99	14	24	6	54	69	-8*
6	26	50	16*	4	120	10	-14	3	80	18	-24		H,K= 11,	-1		H,K= 12,	-5		
7	57	41	1*	5	63	66	14*	4	60	20	8*	1	32	42	30*	1	86	9	6
	H,K= 9,	2		6	37	53	-12*	5	30	55	-47*	2	201	9	25	2	41	35	24*
0	261	8	-18	7	66	22	64*		H,K= 10,	4		3	134	12	16	3	107	11	10
1	273	9	-4		H,K= 10,	-3		0	164	11	12	4	134	18	-10	4	79	41	-7*
2	210	8	-2	1	345	11	-3	1	36	46	30*	5	21	52	6*	5	132	9	-12
3	100	12	12	2	298	10	-30	2	0	59	-56*	6	26	51	-9*	6	29	50	-7*
4	60	70	25*	3	139	8	0	3	32	43	18*		H,K= 11,	0		H,K= 12,	-4		
5	49	23	0*	4	116	9	10	4	166	12	2	0	126	8	11	1	314	10	1
6	32	51	-31*	5	29	72	-7*	5	53	49	38*	1	265	9	2	2	59	17	-5
	H,K= 9,	3		6	56	21	13*		H,K= 10,	5		2	227	8	-8	3	98	14	-4
0	360	11	17	7	53	44	30*	0	43	32	-3*	3	153	10	-13	4	48	65	0*
1	97	9	0		H,K= 10,	-2		1	51	33	-25*	4	137	8	-7	5	33	44	17*
2	180	8	23	1	24	42	16*	2	69	13	3	5	64	18	7	6	10	50	3*
3	89	24	39	2	260	9	23	3	75	19	-12	6	25	50	10*		H,K= 12,	-3	
4	52	64	-7*	3	48	23	21*	4	119	13	-5		H,K= 11,	1		1	145	8	-7
5	61	17	-14	4	53	20	16*		H,K= 10,	6		0	169	7	1	2	194	8	18
6	73	21	37	5	46	63	29*	0	117	8	-2	1	256	9	7	3	71	32	6*
	H,K= 9,	4		6	51	31	4*	1	131	11	2	2	141	15	-3	4	0	68	-43*
0	135	7	2	7	33	61	24*	2	77	17	25	3	223	12	0	5	48	27	-17*
1	71	20	7		H,K= 10,	-1		3	139	11	13	4	27	49	17*	6	45	55	42*
2	53	71	43*	1	211	8	6		H,K= 10,	7		5	65	24	-8*		H,K= 12,	-2	
3	107	15	-12	2	111	8	-20	0	144	8	-4	6	55	37	45*	1	118	9	-5
4	30	47	6*	3	137	8	4	1	0	56	-10*		H,K= 11,	2		2	185	8	-12
5	81	20	11	4	66	32	15*	2	169	12	4	0	233	9	19	3	71	40	9*
6	101	16	-7	5	95	33	-2*		H,K= 11,	-5		1	33	62	-24*	4	26	47	-17*
	H,K= 9,	5		6	0	49	-20*	1	213	8	8	2	263	11	-3	5	93	15	15
0	42	54	3*	7	34	50	29*	2	185	8	-30	3	0	48	-5*	6	60	33	20*
1	52	71	-4*		H,K= 10,	0		3	104	10	1	4	62	44	-16*		H,K= 12,	-1	
2	0	61	-7*	0	299	10	-15	4	65	19	44*	5	0	67	-19*	1	129	8	-11
3	82	14	-31	1	153	6	6	5	120	15	-17		H,K= 11,	3		2	56	61	29*
4	85	36	-4*	2	119	6	-10	6	50	39	25*	0	134	12	-20	3	152	13	-10
5	115	13	2	3	238	8	7	7	18	51	-17*	1	123	15	-6	4	67	14	8
	H,K= 9,	6		4	33	58	-2*		H,K= 11,	-4		2	105	11	2	5	129	17	13
0	226	8	6	5	95	11	5	1	60	19	-8*	3	105	12	-3	6	41	52	-21*
1	27	41	14*	6	52	55	37*	2	294	9	13	4	53	37	20*		H,K= 12,	0	
2	105	11	-9		H,K= 10,	1		3	0	44	-5*	5	85	19	20	0	205	10	25
3	36	62	3*	0	285	9	17	4	84	15	56		H,K= 11,	4		1	37	52	32*
4	114	12	18	1	184	7	6	5	31	62	-6*	0	130	7	-3	2	236	10	-2

STRUCTURE FACTORS CONTINUED FOR

L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL	L	F08	SG	DEL
3	19	45	6*	5	32	70	24*	5	0	75	-8*	1	181	10	5				
4	143	7	-3		H,K=	13,	-2		H,K=	14,	-3	2	60	61	49*				
5	46	58	31*	1	42	56	9*	1	193	11	5	3	86	16	8				
	H,K=	12,	1	2	139	17	-9	2	45	32	-1*		H,K=	15,	-1				
0	149	11	-14	3	32	41	-1*	3	156	8	1	1	91	16	-20				
1	191	9	-1	4	99	19	-28	4	71	21	9*	2	109	13	0				
2	158	10	-10	5	32	51	20*	5	0	52	-19*	3	86	25	5				
3	157	8	-6		H,K=	13,	-1		H,K=	14,	-2		H,K=	15,	0				
4	49	51	45*	1	134	17	-12	1	103	9	-21	0	106	14	10				
5	31	51	-16*	2	21	53	13*	2	161	10	11	1	0	47	-32*				
	H,K=	12,	2	3	147	8	0	3	50	54	10*	2	125	10	-8				
0	33	37	-20*	4	68	37	22*	4	64	24	8*		H,K=	16,	-8				
1	314	10	14	5	71	23	-20*		H,K=	14,	-1	2	151	13	6				
2	0	49	-43*		H,K=	13,	0	1	72	18	29		H,K=	16,	-7				
3	128	11	10	0	51	25	-9*	2	121	11	5	1	202	10	-3				
4	95	15	9	1	190	8	-9	3	56	61	-28*	2	0	52	-36*				
	H,K=	12,	3	2	119	8	7	4	84	27	4*	3	60	26	5*				
0	235	8	5	3	83	14	-4		H,K=	14,	0		H,K=	16,	-6				
1	48	42	13*	4	108	13	1	0	119	15	-9	1	102	13	-3				
2	145	13	-1		H,K=	13,	1	1	161	8	10	2	58	35	-27*				
3	0	50	-12*	0	241	8	4	2	63	21	-4*	3	106	17	-7				
4	31	51	-22*	1	105	10	-8	3	128	10	10		H,K=	16,	-5				
	H,K=	12,	4	2	209	10	7		H,K=	14,	1	1	75	18	30				
0	11	43	-9*	3	43	50	32*	0	95	13	17	2	85	17	-14				
1	197	11	22	4	35	51	-32*	1	164	10	5	3	39	55	-21*				
2	10	56	-18*		H,K=	13,	2	2	41	71	5*		H,K=	16,	-4				
3	82	20	-1	0	127	8	1	3	116	14	5	1	113	13	-17				
	H,K=	12,	5	1	168	10	16		H,K=	14,	2	2	21	49	-39*				
0	106	14	-5	2	144	10	5	0	150	10	0	3	0	74	-37*				
1	81	18	-15	3	84	17	8	1	0	63	-5*		H,K=	16,	-3				
	H,K=	13,	-6		H,K=	13,	3		H,K=	15,	-7	1	109	13	-15				
1	85	11	1	0	122	11	-3	1	258	10	12	2	54	38	5*				
2	41	52	25*	1	152	10	11	2	111	12	-9		H,K=	16,	-2				
3	113	21	-9	2	104	22	23	3	0	49	-59*	1	70	24	54*				
4	114	13	-4		H,K=	13,	4	4	34	50	3*		H,K=	17,	-8				
5	150	10	9	0	168	9	10		H,K=	15,	-6	1	164	9	20				
6	75	35	66*		H,K=	14,	-7	1	25	49	-10*	2	46	62	-26*				
	H,K=	13,	-5	2	152	18	8	2	232	8	5		H,K=	17,	-7				
1	127	8	3	4	126	16	10	3	0	49	-6*	1	34	68	31*				
2	205	9	9		H,K=	14,	-6	4	0	51	-44*	2	86	17	-16				
3	24	68	-5*	1	181	13	9		H,K=	15,	-5		H,K=	17,	-6				
4	154	9	-12	2	44	64	-55*	1	204	8	-2	1	57	28	-4*				
5	42	57	40*	3	155	8	-8	2	79	23	-8	2	84	27	24*				
6	78	24	46*	4	106	21	26	3	154	11	-5		H,K=	17,	-5				
	H,K=	13,	-4	5	43	55	36*	4	92	15	-6	1	78	18	4				
1	185	8	11		H,K=	14,	-5		H,K=	15,	-4								
2	191	12	-8	1	248	12	9	1	134	8	2								
3	186	11	-11	2	140	12	-16	2	88	15	-10								
4	20	55	11*	3	208	8	2	3	133	11	-13								
5	37	50	28*	4	47	58	-2*	4	45	63	23*								
6	12	57	-10*	5	34	50	26*		H,K=	15,	-3								
	H,K=	13,	-3		H,K=	14,	-4	1	73	17	13								
1	274	10	9	1	0	63	-18*	2	116	15	11								
2	79	34	7*	2	296	10	6	3	22	49	1*								
3	134	11	-11	3	50	20	29*	4	70	22	18*								
4	66	15	-9	4	134	11	11		H,K=	15,	-2								

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