pounds and those for the germanium compounds. In all it is felt that considerable reliance can be placed on both the tin and the germanium assignments.

Earlier, in the discussion of the germanium assignments, it was pointed out that the metal–halogen and metal–oxygen stretching bands occur at higher frequencies and the oxygen–hydrogen stretching bands at lower frequencies in the hemiporphyrazine. This led to the suggestion that the substituent–germanium bond is stronger in the hemiporphyrazines than in the phthalocyanines and that the pyridine rings of the hemiporphyrazine macrocycle retain much of their ordinary character in the macrocycle. The newly completed set of assignments for the tin complexes is seen to parallel the set for the germanium complexes and to lead to analogous conclusions. Both sets, then, suggest that the resonance form of Figure 1c is more important than that of Figure 1b for the tin and germanium hemiporphyrazines.

The infrared data also provide evidence supporting the formulation of the dehydration product of hpSn$(OH)_2$, $(hpSnO)_x$, as a polymer. The spectrum of this compound, while similar to that of hpSn$(OH)_2$, shows a broadening of some of the ligand bands and the absence of others. It also shows a relatively strong and broad nonligand absorption at 895 cm^{-1}. These observations all are in keeping with a polymeric formulation for the compound, the broadenings and absences being attributable to coupling and the 895 cm^{-1} absorption (by analogy with $(hpGeO)_x$[2]) to the Sn–O asymmetric vibration. (Using a line of reasoning similar to that applied to $(hpGeO)_x$, further evidence for the polymeric nature of $(hpSnO)_x$ is provided by the prominent 3.67-A line in its powder pattern. Such reasoning also leads to an Sn–O bond length of 1.84 A.)

Finally, it is worth noting that the spectra of the two forms of hpLiH show bands identifiable as N–H bands. For γ-hpLiH bands at 598 and 3419 cm^{-1} can be assigned to the bending and stretching modes, while for β-hpLiH bands at 582 and 3418 cm^{-1} can be similarly assigned. The bands given comparable asignments for hpH_2 are at 574 and 3452 cm^{-1}, respectively.[2]

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Structure Determination and Crystal Preparation of Monoclinic Rare Earth Sesquisulfides

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A method is described by which single crystals of the monoclinic lanthanide sesquisulfides were easily prepared. The crystal structure of one member, Ho_2S_3, was determined using three-dimensional X-ray data. The monoclinic crystals have cell dimensions $a = 17.50 \pm 0.03$, $b = 4.002 \pm 0.005$, $c = 10.15 \pm 0.02$ A, and $\beta = 99.4 \pm 0.2°$. The space group is $P2_1/m$ with 12 Ho atoms and 18 S atoms per cell. All atoms are in the e positions: $x, {}^1/_4, z;\ \bar{x}, {}^3/_4, \bar{z}$. Half of the holmium atoms are six-coordinated and the others are seven-coordinated, while two-thirds of the sulfur atoms are four-coordinated and the remainder are five-coordinated. Thus the average coordination number of holmium is $6^1/_2$; that of sulfur is $4^1/_3$. These coordination numbers are intermediate between those shown by the sesquisulfides of larger atomic radii (defect Th_3P_4 structure) and those of the smaller atomic radii lanthanides (α-Al_2O_3, Sc_2S_3 structures).

Introduction

The sesquisulfides of the four lanthanide elements Y, Ho, Er, Tm have been prepared previously in single-crystal form and the dimensions of their monoclinic unit cells reported.[1,2] However, the crystal structure of this group has been unknown and forms the largest single gap in our knowledge of the crystal chemistry of the lanthanide sesquichalcogenides. The situation with regard to the sulfides is shown in Table I where the notation is the same as that used in discussing the sesquichalcogenides as a whole.[3] The non rare earth elements Y and Sc are placed in positions corresponding to their ionic radii. Since it was established[4] that Ce_2S_3 has a defect Th_3P_4 structure it has been found that the sesquisulfides of the ten elements from La to Dy crystallize in this structure type. Yb_2S_3 and Lu_2S_3 (E) have the α-Al_2O_3 structure type[5] while Sc_2S_3 (F)[6] is isostructural with a large number of rare earth sesquiselenides and sesquitellurides.

During exploratory synthetic work on the lanthanide sulfides a method was discovered by which single crystals of the structurally unknown group D could be prepared relatively easily, and a crystal structure determination of one member, Ho_2S_3, was undertaken.

Experimental Section

Crystal Preparation.—The anhydrous lanthanide chloride was melted in a carbon boat at 1100° and a slow flow of H_2S was passed

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[1] J. Flahaut, M. Guittard, J. Loriers, and M. Patrie, *Compt. Rend.*, **245**, 2291 (1957).

[2] M. Picon, L. Domange, J. Flahaut, M. Patrie, and M. Guittard, *Bull. Soc. Chim. France*, **27**, 221 (1960).

[3] J. P. Dismukes and J. G. White, *Inorg. Chem.*, **4**, 970 (1965).

[4] W. H. Zachariasen, *Acta Cryst.*, **2**, 57 (1949).

[5] J. Flahaut, L. Domange, and M. Pardo, *Compt. Rend.*, **258**, 594 (1964).

[6] J. P. Dismukes and J. G. White, *Inorg. Chem.*, **3**, 1220 (1964).

TABLE I

CRYSTAL STRUCTURE TYPES OF THE LANTHANIDE SESQUISULFIDES[a]

	La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	(Y)	Ho	Er	Tm	Yb	Lu	(Sc)
M_2S_3	A	A	A	A	A	A	A	A	A	A	D	D	D	D	E	E	F

[a] A, defect Th_3P_4 structure; D, Ho_2S_3 structure; E, α-Al_2O_3 structure; F, Sc_2S_3 structure.

over the molten chloride. A passage of 30 cc/min for 3 hr has yielded good results although these conditions can be changed within wide limits. On removal of the boat from the reaction tube, a continuous layer of sulfide was found covering the boat. Underneath this layer were found well-defined crystals of the sulfide usually as needles but sometimes broadened into platelets. Crystals of Y_2S_3, Ho_2S_3, Er_2S_3, and Tm_2S_3 were prepared by this method.

Crystal Data for Ho_2S_3.—The crystal data for Ho_2S_3 are: mol wt, 426.05; absent spectra, $0k0$ with k odd; monoclinic, space group $P2_1/m$, $Z = 6$; $a = 17.50 \pm 0.03$, $b = 4.002 \pm 0.005$, $c = 10.15 \pm 0.02$ A, $\beta = 99.4 \pm 0.2°$; $V = 701.1$ A^3; $d_{\text{calcd}} = 6.06$ g cm^{-3}.

The crystals of holmium sesquisulfide used for X-ray measurements were in the form of long needles elongated in the direction of the monoclinic b axis. No pyroelectric effect could be detected, suggesting a centrosymmetric space group.

Three-dimensional X-ray intensity data were recorded with Zr-filtered Mo Kα radiation using the equiinclination Weissenberg technique. The multiple-film method was used and the intensities were estimated visually by comparison with a standard scale. The bulk of the data was obtained from a crystal 0.05 × 0.07 mm in cross section and 0.5 mm long set for rotation about the b (needle) axis. The zones $h0l$–$h5l$ were measured from this crystal and approximate absorption corrections were made by treating the crystal as a cylinder of equal cross-sectional area. For rotation about the c axis a suitably shaped crystal could not be obtained and the one actually used to record the $hk0$, $hk1$, and $hk2$ zones measured 0.03 × 0.08 mm in cross section. As a consequence, there was considerable error in the correlation of the intensities in the different layers of the b-axis rotation, and at a later stage in the analysis these were separately scaled against the calculated values. For the structure analysis the reflections with $d \geqslant 0.665$ A were used although some photographs showed reflections of considerably smaller spacings.

Structure Determination and Refinement

One very striking feature of the experimental data was the parallelism of the intensities of the $hk0$, $hk2$, and $hk4$ zones and also of the $hk1$, $hk3$, and $hk5$ zones. A careful examination of the intensities along reciprocal lattice lines perpendicular to b showed no discontinuity in the series $k = 0, 2, 4$ and $k = 1, 3, 5$. This was very strong evidence that, of the two space groups $P2_1$ and $P2_1/m$ formally possible from the observed extinctions, the second was correct and all atoms were in the mirror planes in the positions e: $x, {}^1/_4, z$; $\bar{x}, {}^3/_4, \bar{z}$.

The sections of the three-dimensional Patterson function at $y = 0$ and $y = {}^1/_2$ were computed, using data sharpened to correspond to atoms at rest. For 12 Ho atoms in the twofold positions given above, the Patterson section at $y = 0$ should contain 15 independent vectors between Ho atoms in the same plane, while that at $y = {}^1/_2$ should contain 21 vectors between Ho atoms, 15 between independent atoms in the two planes $y = {}^1/_4$, $y = {}^3/_4$, and 6 of the type $2x, {}^1/_2, 2z$ between the pairs of atoms related by a screw axis. After a few trials, all such vectors could be identified and the positions of all of the Ho atoms determined. The S atom positions were then found from an electron density map prepared using the phases calculated from the positions of the Ho atoms.

Refinement was carried out through six cycles of computation of ρ_{obsd} and $\rho_{\text{obsd}} - \rho_{\text{calcd}}$ three-dimensional electron density maps using separate isotropic temperature factors for each atom. The procedure by which the data of separate reciprocal lattice layers had been scaled separately against the calculated values would, of course, vitiate any attempt to determine an anisotropic component of the temperature factor in the y direction. No pronounced anisotropic effects were observed in the xz plane.

The final atomic parameters are given in Table II with their standard deviations.[7] The observed and calculated values of the structure factors are listed in Table III. The atomic scattering factors used are those given in the "International Tables"[8] modified by anomalous dispersion corrections.[9] For 1920 observed reflections $R = 0.097$.

TABLE II

ATOMIC PARAMETERS FOR Ho_2S

Atom	x	y	z	σ, A	B, A^2
Ho(1)	0.0208	0.2500	0.1894	0.002	1.2
Ho(2)	0.4300	0.2500	0.1242	0.002	1.2
Ho(3)	0.7806	0.2500	0.8292	0.002	1.3
Ho(4)	0.7203	0.2500	0.1872	0.002	1.3
Ho(5)	0.3981	0.2500	0.4867	0.002	1.4
Ho(6)	0.1156	0.2500	0.5494	0.002	1.2
S(1)	0.5756	0.2500	0.0531	0.009	1.4
S(2)	0.8731	0.2500	0.2683	0.011	1.4
S(3)	0.9672	0.2500	0.6167	0.009	1.1
S(4)	0.2808	0.2500	0.6372	0.009	1.3
S(5)	0.2689	0.2500	0.0081	0.008	1.2
S(6)	0.6306	0.2500	0.7108	0.009	1.2
S(7)	0.9239	0.2500	0.9506	0.011	1.3
S(8)	0.1825	0.2500	0.3192	0.012	1.4
S(9)	0.5319	0.2500	0.3678	0.009	1.3

Discussion

A view of the crystal structure of Ho_2S_3 in the direction of the crystal b axis is shown in Figure 1. The atoms indicated by full circles represent the atoms in the plane $y = {}^1/_4$, while the dashed circles represent the screw axis related atoms in the plane $y = {}^3/_4$. The circles are approximately half the ionic size for the two types of atoms.

It is clear that the structure is not a simple close-packed one, and, in fact, the coordination system is quite complex. The atoms Ho(3), Ho(4), Ho(5) are bonded to six S atoms while the other three Ho atoms are in sevenfold coordination. Of the S atoms, S(3), S(5), and S(6) are each in fivefold coordination while the

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(7) D. W. J. Cruickshank, *Acta Cryst.*, **2**, 65 (1949).

(8) "International Tables for X-ray Crystallography," Vol. III, The Kynoch Press, Birmingham, England, 1962.

(9) C. H. Dauben and D. H. Templeton, *Acta Cryst.*, **8**, 841 (1955).

Table III

Calculated and Observed Structure Factors for Ho_2S_3 ($F(000) = 545$)

h	k	l	F_c	F_o
0	0	14	-31	35
1			-23	28
3			61	56
4			-32	28
0	0	13	-41	37
2			-32	34
3			-30	35
0	0	12	-23	25
1			36	33
2			-53	49
4			28	26
6			-29	26
7			-71	65
9			-35	31
0	0	11	61	60
1			50	50
3			-56	49
4			54	49
5			-43	38
12			-69	67
13			-39	36
0	0	10	20	23
1			-21	23
2			21	23
3			56	54
4			40	41
5			33	34
6			99	98
9			-28	25
10			34	36
11			57	50
12			-34	26
13			33	27
14			24	27
17			-42	37
0	0	9	-40	32
2			82	79
3			-31	25
5			-67	58
6			31	26
7	0	9	72	62
8			-43	36
9			75	76
11			57	61
12			25	25
15			-20	27
16			52	52
17			35	39
0	0	8	-30	44
1			-135	126
4			-60	54
5			21	21
6			-30	25
7			17	22
9			-37	30
10			-39	41
11			-40	37
12			28	24
13			37	35
14			-19	26
16			37	38
2	0	7	-37	27
3			-32	25
5			-24	20
6			-130	128
7			-39	32
8			19	21
9			43	36
10			-40	34
11			-38	32
12			48	43
13			-67	65
14			-15	25
15			-58	62
19			48	50
20			-32	36
0	0	6	130	107
1			18	17
2			-191	187
3			27	23
5	0	6	109	104
6			-54	50
8			61	59
9			-95	98
11			-63	63
12			22	22
13			-21	23
15			43	42
16			-77	91
17			-22	26
20			-52	52
21			-27	28
22			35	37
0	0	5	42	34
1			158	139
2			19	16
3			100	86
4			97	87
5			-37	30
6			43	41
10			126	133
11			52	55
13			-26	22
14			34	30
16			-106	121
19			36	37
23			-52	40
2	0	4	-34	31
3			30	28
4			-146	152
5			42	40
6			135	144
7			46	46
8			75	78
9			-32	26
10			142	148
12			-50	52
13			57	56
15			110	118
16			36	33
17	0	4	27	28
19			-36	29
20			46	63
21			-25	27
26			36	44
0	0	3	-212	166
2			61	58
3			-32	31
5			-129	140
6			78	79
7			-68	77
8			-81	84
10			-58	67
11			108	110
12			46	47
13			41	49
16			42	62
18			-36	37
19			25	25
20			66	81
21			36	34
22			33	35
24			24	28
0	0	2	-10	13
1			-116	116
2			16	21
3			-110	129
4			-80	87
5			-86	103
6			-84	96
8			75	79
9			-17	17
10			-94	106
11			-54	63
12			14	19
14			-100	107
16			41	41
18			23	24
19			-44	40
21			-28	30
23	0	2	34	35
24			-30	32
25			48	40
1	0	1	-29	39
2			-28	44
3			-36	54
4			229	222
5			-26	27
6			-189	202
7			-66	52
8			-64	68
9			26	28
10			-175	195
11			33	34
12			33	36
13			-34	34
16			34	35
15			-80	94
17			-95	114
20			-66	79
22			26	30
24			-40	39
25			-24	28
2	0	0	14	29
3			26	40
4			22	27
5			78	101
6			-131	155
7			128	154
8			47	65
9			33	42
10			86	93
12			-66	78
13			-74	89
14			33	41
16			-56	57
18			52	58
20			-90	110
21			-31	29
22			-29	30
24	0	0	-38	39
2	0	$\bar{1}$	-29	41
3			82	105
4			54	52
5			89	112
6			162	173
7			82	90
8			-116	128
9			36	37
10			61	70
11			25	17
12			65	46
13			24	19
14			205	228
18			-43	40
19			39	39
20			-35	35
21			60	36
22			-22	26
24			37	38
25			-43	39
26			-32	33
2	0	$\bar{2}$	103	91
3			36	43
4			-227	207
5			52	54
6			-88	86
8			-49	61
10			170	179
11			-29	25
12			56	70
13			-19	19
14			-38	66
15			67	75
17			59	59
19			58	63
20			85	105
22			-32	30
24			30	31
2	0	$\bar{3}$	-62	57
3	0	$\bar{3}$	-34	30
4			-48	43
5			-45	45
6			77	70
7			-65	64
9			-100	102
10			-107	107
12			21	18
14			-34	31
15			32	29
16			75	88
18			-84	99
21			23	25
22			19	26
24			49	52
26			37	37
1	0	$\bar{4}$	54	66
2			14	14
3			30	22
4			-18	14
5			-72	65
6			-150	137
7			-74	70
8			89	84
9			-89	87
10			-57	60
11			-25	24
12			50	49
13			-23	20
14			-136	134
16			-45	45
19			-16	24
20			24	25
21			-51	49
22			37	40
23			-25	31
26			-54	52
1	0	$\bar{5}$	57	48
2			-127	118
4			228	179
5	0	$\bar{5}$	-61	54
6			53	51
7			21	17
8			83	77
10			-154	164
11			22	19
12			-99	101
14			24	21
16			-24	22
17			-48	48
18			30	31
19			-57	61
20			-78	91
21			-24	30
22			23	26
26			-26	32
1	0	$\bar{6}$	-46	37
2			96	81
3			47	38
4			153	142
5			34	31
7			16	18
8			-36	30
9			40	32
10			122	111
11			64	62
13			63	66
14			33	28
16			-63	60
18			75	82
19			-23	25
20			26	29
24			-55	55
3	0	7	-83	74
4			-56	50
5			-19	18
6			121	103
7			17	19
8			-31	[illegible]
9			103	[illegible]
11	0	$\bar{7}$	34	29
12			-81	91
14			79	79
16			119	129
18			45	45
19			27	25
22			-40	38
23			47	45
24			44	39
1	0	$\bar{8}$	-74	72
2			88	80
3			-43	38
4			-42	38
5			46	41
6			-42	42
8			-101	96
12			57	57
13			-23	23
17			-24	25
18			-37	36
19			35	33
20			68	59
23			32	32
1	0	$\bar{9}$	24	21
2			-41	36
3			-33	30
4			-38	50
5			-37	32
6			-27	25
8			29	25
9			-24	22
10			-60	56
13			-83	86
14			-50	50
15			-17	24
16			63	63
18			-20	26
19			23	26
20			-80	31
21			19	27
22	0	$\bar{9}$	-30	36
3	0	$\bar{10}$	66	61
4			40	34
5			59	58
6			-85	77
7			36	32
8			25	23
9			-86	83
10			-31	27
11			-34	34
12			64	66
13			-28	25
14			-36	32
16			-64	59
18			-30	34
20			-25	27
22			46	49
1	0	$\bar{11}$	67	63
2			-64	60
3			84	87
4			36	34
5			-39	34
8			108	117
9			26	24
10			42	38
11			22	25
12			41	36
16			-35	31
17			34	27
18			32	27
20			-45	43
1	0	$\bar{12}$	-35	35
2			-38	35
4			20	25
5			41	35
6			50	41
10			57	49
11			-37	37
13			55	50
14			42	44
15	0	$\bar{12}$	48	52
16			-29	32
17			32	32
1	0	$\bar{13}$	45	40
3			-29	26
4			-36	34
5			-21	26
6			30	26
7			-67	64
9			20	26
11			38	38
12			-33	31
13			23	27
14			-38	36
1	0	$\bar{14}$	-29	32
3			-63	67
10			-37	32
5	0	$\bar{15}$	-28	28
6			-26	28
10			-35	33
0	1	14	81	87
1			-22	27
2			20	31
3			32	35
6			-24	28
0	1	13	-19	26
1			-28	26
2			-23	26
5			40	46
6			39	46
9			36	44
2	1	12	13	25
3			-33	33
4			-69	68
5			30	30
6			-37	39
7			-33	40
8			-21	26
10			62	62
11			22	27
12	1	12	21	28
0	1	11	-71	61
1			45	45
2			-47	48
3			-24	28
4			-52	47
6			38	32
7			-25	29
9			-38	45
10			-41	45
12			-23	31
13			-23	31
14			-17	28
1	1	10	20	22
2			42	41
3			20	26
4			21	27
5			-48	47
6			-37	36
8			20	28
9			-40	47
11			21	25
12			18	26
13			-23	27
14			-52	53
16			-46	55
2	1	9	-52	46
3			36	39
4			68	64
5			-68	66
6			68	66
7			43	46
8			53	54
10			28	24
11			33	29
12			-50	52
13			25	30
18			22	36
0	1	8	31	23
1			-91	80
2	1	8	93	89
3			40	37
4			65	58
8			-39	40
9			63	62
10			45	46
11			29	27
12			23	24
13			89	100
14			24	25
15			-39	39
16			-24	26
17			20	26
18			29	31
19			-22	28
2	1	7	-29	24
3			-107	102
4			-52	48
5			-41	42
6			38	36
7			-61	55
9			114	117
12			-55	53
13			29	27
14			45	39
16			69	48
18			41	43
19			26	31
0	1	6	-25	21
1			-41	36
2			44	38
3			-108	95
4			-24	23
5			79	77
6			-74	78
7			-24	25
8			-142	156
9			-21	23
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5			29	28
7			-20	23
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11			-95	94
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13			39	40
14			27	24
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21			-21	28
23			38	38
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7			42	38
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9			-45	44
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12			25	24
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5			-30	26
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11			36	37
13			36	35
14			19	21
16			-36	38
18			44	45
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3	4	$\bar{10}$	38	36
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4			21	28
7			42	46
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1			34	30
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5			-35	35
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8			46	51
10			45	48
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13			-44	49
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2			38	33
3			28	32
4			17	19
5			-16	17
8			-14	13
9			-33	38
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13			-23	23
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3			26	25
4			9	9
7			116	122
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14			38	39
1	5	$\bar{3}$	-61	47
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10			25	25
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13			53	57
14			22	19
2	5	$\bar{4}$	-22	20
3			-50	37
4			-32	30
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9			35	35
11			-65	66
12			16	15
13			-28	27
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2			-48	41
5			44	42
7			-46	45
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11			-26	26
12			-16	15
1	5	$\bar{6}$	40	36
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11			16	15
12			-26	23
3	5	$\bar{7}$	38	37
4			24	19
6			56	56
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1	5	$\bar{8}$	-19	15
2			47	47
5			-54	52
7			43	45

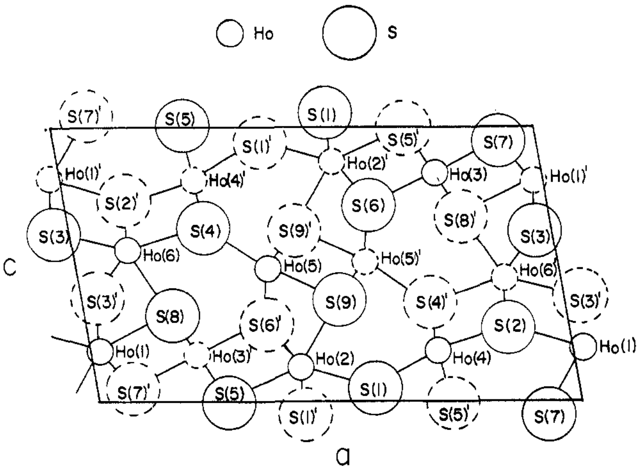

Figure 1.—The crystal structure of Ho_2S_3 projected down the crystal *b* axis. Full circles represent atoms in the plane $y = {}^1/_4$; dashed circles, those in the plane $y = {}^3/_4$.

TABLE IV

AVERAGE COORDINATION NUMBERS IN LANTHANIDE SESQUISULFIDES

Structure type	Av coordn no. of metal	Av coordn no. of S
Ce_2S_3 (defect Th_3P_4)	8	$5^1/_3$
Ho_2S_3	$6^1/_2$	$4^1/_3$
Yb_2S_3 (α-Al_2O_3)	6	4
Sc_2S_3	6	4

others are fourfold coordinated by Ho atoms. A consequence of the peculiar coordination is the occurrence of large empty channels running through the crystal in the *y* direction. The bond angles show considerable distortion. Of the three octahedrally coordinated Ho atoms Ho(3) has a maximum deviation of 8.0° and an average deviation of 4.2° from 90° bond angles. The least distorted S-centered tetrahedron has one angle almost 20° greater than the regular tetrahedron angle. The average Ho–S bond distance is 2.76 A.

The most remarkable feature of the Ho_2S_3 structure is the odd-numbered coordination of half the Ho atoms and one-third of the S atoms. In Table IV the average coordination numbers are given for the four principal structure types of the rare earth sesquisulfides. For the Ce_2S_3 group the S atoms are ideally in sixfold coordination positions but one-ninth of the metal atoms are randomly missing. The coordination numbers of the Ho_2S_3 group are intermediate between those of the Ce_2S_3 group with larger metal radii and those of the Yb_2S_3 group with smaller metal radii. In a very recent abstract[10] it has been found that in the α form of Gd_2S_3 half of the Gd atoms are seven-coordinated and the other half, eight-coordinated. The coordination in this crystal is thus intermediate between that of the Ho_2S_3 structure and that in the defect Th_3P_4 structure.

It is intended to carry out more sophisticated analyses, using counter-measured intensity data, on crystals of the end members of the series, Y_2S_3 and Tm_2S_3, in the hope that a detailed comparison of the bonding and the thermal vibrations may shed light on why the Ho_2S_3 structure becomes unstable with respect to the Ce_2S_3 and Yb_2S_3 structures.

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[10] C. A. Prewitt and A. W. Sleight, American Crystallographic Association Winter Meeting, Atlanta, Ga., Jan 1967.

CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
UNIVERSITY OF ALBERTA, EDMONTON, ALBERTA, CANADA

Organometallic Compounds with Metal–Metal Bonds. IX. Pentafluorophenyltin Derivatives of Manganese Carbonyl[1]

BY J. A. J. THOMPSON AND W. A. G. GRAHAM

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Compounds of the series $(C_6H_5)_n(C_6F_5)_{3-n}SnMn(CO)_5$ ($n = 0, 1, 2$) have been prepared by reactions of C_6F_5Li or C_6F_5MgBr with $(C_6H_5)_nCl_{3-n}SnMn(CO)_5$. Carbonyl stretching frequencies are assigned and approximate force constants given. A linear relation between carbonyl stretching frequency and electronegativity of substituents on tin suggests a Pauling electronegativity of about 2.4 for the C_6F_5 group. The compounds $(C_6F_5)_2Sn[Mn(CO)_5]_2$ and $(OC)_5Mn(C_6H_5)_2Sn$-$Sn(C_6H_5)_2$-$Mn(CO)_5$ have also been prepared, the latter containing a four-metal chain. In both of the bis-pentacarbonylmanganese compounds, the number of strong carbonyl bands is greater than five, suggesting that a substantial coupling of $Mn(CO)_5$ groups takes place across the intervening tin atoms.

Introduction

Numerous pentafluorophenyl organometallic compounds of both main and transition group metals have been reported in recent years. Convenient synthetic methods are available, and the compounds are normally more stable than their hydrocarbon analogs.[2] A recent communication from this laboratory suggested that a combination of coupling constant and chemical shift

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[1] Part VIII: J. A. J. Thompson and W. A. G. Graham, *Inorg. Chem.*, **6**, 1365 (1967).

[2] For reviews, *cf.* P. M. Treichel and F. G. A. Stone, *Advan. Organometal. Chem.*, **1**, 143 (1964); F. G. A. Stone, *Endeavour*, **25**, 33 (1966). For recent leading references, *cf.* F. J. Hopton, A. H. Rest, D. T. Rosevear, and F. G. A. Stone, *J. Chem. Soc., Sect. A*, 1326 (1966); C. Tamborski, E. J. Soloski, and S. M. Dec, *J. Organometal. Chem.* (Amsterdam), **4**, 446 (1965).