azolin-5-one (Sabelli & Zanazzi, 1969*a*) and by 2·45° for 3-phenylisoxazolin-5-one (Cannas, Biagini & Marongiu, 1969). The intermolecular distances less than 3·8 Å are listed in Table 6 and shown in Figs. 6 and 7. The shortest intermolecular distances are as follows:

$C(14^{i})\cdots O(2^{iii})$, 3·350; $C(4^{i})\cdots O(2^{vi})$, 3·355;
$C(6^{i})\cdots N(2^{ii})$, 3·405; $O(1^{i})\cdots O(2^{iii})$, 3·423;
$C(12^{i})\cdots O(2^{ix})$, 3·452 Å.

Computation

The main part of the calculations was performed on an IBM 7040 computer with the use of the program *ERBR* 1 (Van den Hende, 1961) for structure factor and least-squares refinement and the program *ERFR* 2 (Sly, Shoemaker & Van den Hende, 1962) for Fourier summation. The final block-diagonal least-squares refinements were carried out with the program *HBLS* (Ashida, 1964). Thermal ellipsoids were drawn by the CDC 3600 computer at C. Itoh Electronic Computing Service Co. Ltd. using the program *ORTEP* (Johnson, 1965).

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The Crystal Structure of Cs_2MnBr_4

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The crystal structure of Cs_2MnBr_4, determined from Weissenberg data, is orthorhombic with $a=10·150$ (23), $b=7·806$ (11) and $c=13·70$ (8) Å. The space group is *Pnma* and $Z=4$. The structure is isomorphous with that of Cs_2ZnBr_4.

Introduction

The material examined in this work was prepared by heating a mixture, consisting of stoichiometric amounts of CsBr and $MnBr_2$, in a sealed evacuated silica tube until molten at about 650°C. The specimen was then cooled slowly (10°C per hour) to room temperature and single crystals suitable for X-ray study were selected from the yellow crystalline mass. Because the material was very unstable in the presence of water vapour, each crystal was sealed in a Lindemann glass tube containing P_2O_5.

The space lattice and unit-cell dimensions were determined from rotation and Weissenberg photographs taken with Cu $K\alpha$ radiation ($\lambda = 1{\cdot}5418$ Å). The unit cell was found to be very similar in size to those determined for Cs_2ZnBr_4 and Cs_2CuBr_4 by Morosin & Lingafelter (1959, 1960) as seen in Table 1.

Table 1. *Unit-cell data*

	a	*b*	*c*
Cs_2MnBr_4	10·150 (23) Å	7·806 (11) Å	13·70 (8) Å
Cs_2ZnBr_4	10·196 (18)	7·770 (6)	13·517 (23)
Cs_2CuBr_4	10·195 (12)	7·965 (5)	13·936 (15)

Since the conditions for the observed reflexions were the same as those noted by Morosin & Lingafelter in their structure analyses of the zincate and the cuprate, it was assumed at the outset that the manganate was isomorphous with the latter and this indeed proved to be the case.

Structure analysis

The macroscopic density was determined by weighing a sample of the material in air and in toluene. Good agreement between the observed and calculated densities was obtained by assigning four molecules of Cs_2MnBr_4 per unit cell.

Intensity data were collected from *c*-axis equi-inclination Weissenberg multiple-film photographs taken with Mo $K\alpha$ radiation. The systematically absent reflexions were consistent with space groups *Pnma* and $Pn2_1a$; the centrosymmetrical space group *Pnma* (No. 62) was chosen since the structures of Cs_2ZnBr_4 and Cs_2CuBr_4 were based on this. The intensities of 380 reflexions on layer lines 0 to 7 were measured using a Joyce–Loebl flying spot microdensitometer, visual estimates being made for very weak spots; about 126 reflexions were too weak to be recorded. These data were corrected for the Lorentz–polarization factor and for absorption by applying the correction factors given by Bond (1959) for a cylindrical crystal. The appropriate crystal data are presented in Table 2.

Table 2. *Crystal data*

Formula: Cs_2MnBr_4, F.W. 640·42
$D_0 = 3{\cdot}81$ g.cm^{-3}, $D_x = 3{\cdot}92$ g.cm^{-3}, $Z = 4$
Mean $r = 0{\cdot}005$ cm
$\mu = 235$ cm^{-1} ($\lambda = 0{\cdot}7107$ Å)

The structure determination began with a three-dimensional least-squares refinement using individual isotropic temperature factors and assuming initial atomic parameters similar to those of the zincate and the cuprate. Atomic scattering factors for Cs^+, Mn^{2+} and Br^- were taken from *International Tables for X-ray Crystallography* (1962) and inter-layer scaling was achieved by scaling the observed to the calculated structure factors. After 10 cycles of refinement the reliability index $\sum||F_o| - |F_c||/\sum|F_o|$ reduced to 10·8%, at which stage the shifts in the positional parameters were all less than one tenth of the standard deviations and those in the thermal parameters less than one thirtieth. An electron density projection along **b** was finally calculated and, as this did not reveal any spurious peaks, the structure was considered to be correct. Final atomic parameters are given in Table 3; observed and calculated structure factors are compared in Table 4.

Table 3. *Final atomic parameters*

Origin at $\bar{1}$. Standard deviations are given in parentheses.

Equipoint		*x*	*y*	*z*	*B* (Å^2)
Cs(1)	4(*c*)	0·1315 (11)	$\frac{1}{4}$	0·0975 (10)	3·89 (22)
Cs(2)	4(*c*)	−0·0200 (8)	$\frac{1}{4}$	0·6749 (9)	2·55 (17)
Mn	4(*c*)	0·2291 (18)	$\frac{1}{4}$	0·4239 (19)	1·51 (38)
Br(1)	4(*c*)	−0·0121 (18)	$\frac{1}{4}$	0·4061 (19)	4·15 (39)
Br(2)	4(*c*)	0·3156 (18)	$\frac{1}{4}$	0·5907 (19)	4·37 (42)
Br(3)	8(*d*)	0·3222 (11)	0·5024 (16)	0·3440 (13)	4·25 (29)

Table 4. *Observed and calculated structure factors*

H	K	F_o	F_o
L=0			
0	2	388	-455
0	4	492	635
0	6	172	-217
0	8	199	227
2	0	30	-25
4	0	94	90
6	0	314	310
8	0	91	58
10	0	43	36
2	1	58	-50
2	2	233	-245
2	3	44	40
2	5	52	-38
2	6	114	-108
4	1	85	75
4	2	173	-164
4	3	66	-57
4	4	58	72
4	5	62	57
4	6	81	-92
6	1	239	216
6	2	85	-63
6	3	188	-181
6	4	231	210
6	5	136	123
6	7	77	-77
6	8	55	73
8	1	137	120
8	2	211	-195
8	3	123	-107
8	4	62	48
8	5	79	81
8	6	114	-105
8	7	61	-60
12	1	125	104
12	2	95	26
L=1			
2	0	39	-40
3	0	424	-454
4	0	122	-123
5	0	97	-81
8	0	24	-22
9	0	115	-93
10	0	119	-115
2	1	113	99
3	1	234	-235
4	1	127	126
5	1	37	41
6	1	79	77
8	1	55	-49
9	1	131	-102
11	1	53	-46
1	2	111	103
2	2	289	313
3	2	190	191
4	2	169	-154
5	2	200	195
6	2	75	76
8	2	91	82
9	2	31	31
1	3	30	-32
2	3	90	-94
3	3	174	181
4	3	119	-106
6	3	69	-63
8	3	43	36
9	3	106	87
10	3	28	-25
3	4	246	-280
4	4	91	-91
5	4	61	-62
8	4	26	-19
9	4	57	-69
10	4	95	-87
2	5	71	67
3	5	112	-124
4	5	60	75
5	5	24	27
6	5	52	57
9	5	60	-61
1	6	43	40
2	6	132	135
3	6	58	87
4	6	65	-63
5	6	105	100
2	7	47	-56
3	7	66	80
4	7	57	-50
3	8	79	-93
L=2			
0	2	177	-171
0	6	60	-64
2	0	93	-93
3	0	129	-134
4	0	49	-49
5	0	115	121
6	0	63	-57
7	0	104	108
1	1	119	-109
2	1	64	-58
3	1	37	-28
4	1	42	-43
5	1	138	130
7	1	128	-126
10	1	64	-58
11	1	31	30
1	2	275	314
2	2	181	180
3	2	49	49
4	2	72	74
5	2	261	-274
7	2	91	-76
1	3	101	99
2	3	41	50
4	3	39	31
5	3	99	-97
7	3	123	108
10	3	57	59
1	4	48	33
2	4	61	-70
3	4	97	-86
4	4	38	-35
5	4	86	90
6	4	48	-51
7	4	71	77
1	5	63	-69
2	5	39	-32
3	5	28	-31
5	5	61	70
7	5	71	-92
1	6	117	118
2	6	96	91
[illegible]	6	137	-139
1	7	61	52
7	7	52	57
L=3			
0	3	246	231
0	5	135	-157
0	7	114	101
2	0	192	-212
3	0	334	411
4	0	156	143
6	0	73	71
7	0	69	77
8	0	54	49
9	0	82	75
1	1	51	59
2	1	130	-113
3	1	87	83
4	1	35	-31
5	1	51	65
7	1	60	62
8	1	103	-153
9	1	122	[illegible]
11	1	50	[illegible]
1	2	236	-238
2	2	228	[illegible]
3	2	31	-35
4	2	198	-187
5	2	207	-211
6	2	58	-46
7	2	50	-50
10	2	33	14
1	3	37	-30
2	3	64	81
3	3	64	-72
5	3	62	-61
7	3	54	-50
8	3	138	134
9	3	80	-75
11	3	51	-48
1	4	46	55
2	4	120	-133
3	4	239	259
4	4	95	94
6	4	40	46
7	4	37	44
8	4	[illegible]	30
9	4	58	54
1	5	40	39
2	5	46	-53
3	5	47	47
5	5	41	40
7	5	46	46
8	5	105	-97
9	5	60	57
1	6	109	-116
2	6	119	108
4	6	74	-89
5	6	111	-108
2	8	41	-47
3	8	100	101
L=4			
0	2	249	189
0	4	375	-327
0	6	69	92
0	8	94	-114
2	0	48	45
3	0	153	-141
4	0	22	20
5	0	81	100
6	0	197	-172
7	0	201	-191
8	0	25	-20
9	0	52	-50
11	0	79	-61
1	1	224	205
2	1	67	60
3	1	174	184
5	1	276	255
6	1	140	-132
8	1	37	-36
9	1	[illegible]	[illegible]
11	1	[illegible]	[illegible]
12	1	[illegible]	[illegible]
1	2	33	[illegible]
2	2	96	115
3	2	194	176
4	2	42	32
5	2	63	55
8	2	101	126
9	2	105	92
11	2	70	72
1	3	174	-169
2	3	27	-43
3	3	138	-146
5	3	224	-217
6	3	[illegible]	103
8	3	30	33
9	3	41	-43
12	3	48	39
1	4	145	-145
3	4	123	-131
5	4	57	66
6	4	120	-117
7	4	130	-139
11	4	45	-48
1	5	105	115
2	5	44	36
3	5	84	112
5	5	139	152
6	5	58	-69
1	6	26	-16
2	6	41	52
3	6	92	93
8	6	50	56
9	6	61	51
1	7	40	-79
3	7	57	-65
5	7	68	-103
L=5			
0	3	138	134
0	5	117	-111
2	0	126	-154
3	0	111	115
4	0	212	232
5	0	41	37
7	0	97	-84
8	0	39	38
10	0	69	67
2	1	104	-91
3	1	132	131
4	1	133	-121
5	1	104	-116
7	1	87	-83
8	1	54	-61
9	1	43	32
1	2	160	155
2	2	116	-113
3	2	158	-155
4	2	33	39
6	2	70	-67
7	2	61	80
8	2	101	-97
1	3	52	23
2	3	93	80
3	3	108	-106
4	3	108	105
5	3	84	100
7	3	99	70
8	3	53	56
1	4	109	-98
2	4	101	-96
3	4	68	68
4	4	159	159
7	4	83	-65
10	4	48	47
2	5	39	-49
3	5	58	73
4	5	52	-77
5	5	74	-75
1	6	78	79
2	6	56	-51
3	6	45	-39
L=6			
0	2	315	325
0	6	137	141
2	0	96	92
3	0	25	51
4	0	61	69
5	0	25	20
6	0	74	88
8	0	70	-74
9	0	46	55
11	0	34	26
2	1	70	94
3	1	94	-100
4	1	70	59
5	1	94	-80
6	1	29	-21
10	1	104	106
11	1	34	-31
1	2	136	-143
2	2	252	-250
3	2	22	-24
4	2	99	-116
5	2	47	33
6	2	69	67
7	2	59	-43
8	2	29	-33
9	2	36	-30
1	3	23	29
2	3	69	-85
3	3	105	86
4	3	57	-43
5	3	50	65
10	3	95	-91
1	4	26	29
2	4	80	75
4	4	55	50
6	4	65	69
8	4	57	-52
1	5	38	-24
2	5	67	54
3	5	46	-58
5	5	57	-48
10	5	58	74
1	6	69	-68
2	6	139	-134
4	6	65	-59
2	7	47	-43
3	7	57	41
L=7			
0	3	259	-221
0	5	136	166
0	7	107	-103
2	0	142	171
4	0	196	-197
5	0	80	98
6	0	76	-100
8	0	54	-45
10	0	51	-56
1	1	82	-67
2	1	216	196
3	1	98	92
4	1	86	69
5	1	50	-49
6	1	84	84
8	1	147	125
1	2	67	75
2	2	34	-42
3	2	150	-161
4	2	83	65
6	2	126	128
7	2	30	38
8	2	56	71
9	2	60	-62
1	3	51	52
2	3	159	-166
3	3	82	-77
4	3	63	-61
5	3	39	46
6	3	79	-71
8	3	123	-108
2	4	101	112
4	4	137	-137
5	4	63	69
6	4	54	-74
1	5	82	-47
2	5	128	116
3	5	51	58
4	5	43	48
6	5	58	56
8	5	73	78
1	6	44	35
3	6	69	-70
6	6	66	69

Discussion

The structure consists of Cs^+ and $MnBr_4^{2-}$ ions, each Cs(1) having eleven nearest Br neighbours and each Cs(2) nine. The atomic parameters are so similar to those given by Morosin & Lingafelter for Cs_2ZnBr_4, that any discussion can be confined to a comparison between the two sets of structural data.

Bond lengths and angles are given in Table 5. The average Cs(1)–Br and Cs(2)–Br distances (4·04 and 3·73 Å, respectively) agree, within one standard devia-

Table 5. *Bond lengths and angles*

Standard deviations are given in parentheses.

Mn–Br tetrahedron

Mn^{i}	Br(1^{i})	2·460 (27) Å	Br(1^{i})–Mn^{i}–Br(2^{i})	116·7 (1·0)°
	Br(2^{i})	2·448 (36)	Br(1^{i})–Mn^{i}–Br(3^{i,iii})	109·9 (9)
	Br(3^{i,iii})	2·444 (19)	Br(2^{i})–Mn^{i}–Br(3^{i,iii})	106·2 (9)
Br(1^{i})	Br(2^{i})	4·178 (30)	Br(3^{i})–Mn^{i}–Br(3^{iii})	107·5 (8)
	Br(3^{i,iii})	4·015 (20)		
Br(2^{i})	Br(3^{i,iii})	3·913 (28)		
Br(3^{i})	Br(3^{iii})	3·940 (18)		

Cs(1)–Br distances

Cs(1^{ii})	Br(1^{ii})	4·472 (29) Å
	Br(1^{ix})	3·618 (21)
	Br(2^{i})	4·113 (25)
	Br(2^{vi,viii})	3·941 (3)
	Br(3^{i,iii})	3·792 (15)
	Br(3^{ii,iv})	4·363 (20)
	Br(3^{vi,viii})	4·002 (20)

Cs(2)–Br distances

Cs(2^{x})	Br(1^{v,vii})	4·070 (8) Å
	Br(1^{x})	3·685 (29)
	Br(2^{i})	3·619 (27)
	Br(2^{x})	3·596 (21)
	Bı(3^{v,vii})	3·635 (13)
	Br(3^{vi,viii})	3·624 (17)

i	x	y	z
ii	$\frac{1}{2}+x$	y	$\frac{1}{2}-z$
iii	x	$\frac{1}{2}-y$	z
iv	$\frac{1}{2}+x$	$\frac{1}{2}-y$	$\frac{1}{2}-z$
v	$\frac{1}{2}-x$	$1-y$	$\frac{1}{2}+z$
vi	$1-x$	$1-y$	$1-z$
vii	$\frac{1}{2}-x$	$-\frac{1}{2}+y$	$\frac{1}{2}+z$
viii	$1-x$	$-\frac{1}{2}+y$	$1-z$
ix	$1+x$	y	z
x	$\frac{1}{2}+x$	y	$\frac{3}{2}-z$

tion, with those observed for the zincate. The latter distance almost equals the sum of the ionic radii (3·63 Å) given by Clark (1955). The angular distortion of the tetrahedral unit, which occurs in the two structures, has been adequately explained by Morosin & Lingafelter.

Finally, it is interesting to note that Pauling's second rule is not quite obeyed in this structure; the sum of the bond strengths to Br(1), Br(2) and Br(3) are 201/198, 197/198 and 197/198, respectively.

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The Crystal and Molecular Structure of Dodecachloropentasilane Silicon Tetrachloride

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The structure of dodecachloropentasilane silicon tetrachloride, $Si_5Cl_{12}.SiCl_4$, has been determined and refined by three-dimensional least-squares methods, using 67 reflexions, to an R index of 0·044. The substance crystallizes in the cubic space group $F\bar{4}3c$, with $a=17·344$ Å. The two types of molecule, Si_5Cl_{12} and $SiCl_4$, are simply stacked together with intermolecular distances corresponding to van der Waals type interactions.

Introduction

The perchloropolysilanes, Si_nCl_{2n+2}, could be described as the chlorosilicon analogues of the alkanes, C_nH_{2n+2}. But whereas C–C linkages are very common, Si–Si linkages are somewhat rare, Si–O bonds being much preferred when possible.

Some of the higher perchloropolysilanes form 1:1 adducts with silicon tetrachloride (Urry, 1964) which are crystalline and suitable for study by X-ray methods. For example, dodecachloropentasilane silicon tetrachloride was prepared. It was of interest to see which isomer of the dodecachloropentasilane was formed: n-pentyl, isopentyl or neopentyl: