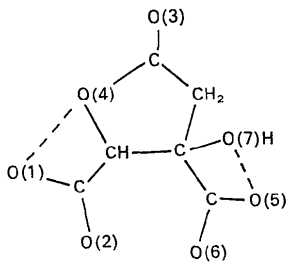
Table 6. *Comparison of the surroundings of the lactone ion*



	Hibiscus	Garcinia
O(1)	H bond from O(*W*2)	H bond from O(*W*3)
	Ca coordination	H bond from O(7)
O(2)	H bond from O(*W*1)	H bond from O(*W*1)
	H bond from O(*W*1′)	H bond from O(*W*1′)
	Ca coordination	H bond to O(*W*4)
O(3)	H bond from O(*W*3) or O(*W*4)	Ca coordination
O(4)	Nothing	Nothing
O(5)	Ca coordination	Ca coordination
	Ca′ coordination	Ca′ coordination
O(6)	Ca coordination	H bond from O(*W*2)
O(7)	H bond to O(*W*2)	H bond to O(1)
	Ca coordination	Ca coordination

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The Crystal Structure of Manganese Chlorophosphate, $Mn_2(PO_4)Cl$

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Manganese chlorophosphate, $Mn_2(PO_4)Cl$, crystallizes in the space group $Pna2_1$, with unit-cell dimensions $a = 11{\cdot}364$ (2), $b = 8{\cdot}0965$ (7), and $c = 4{\cdot}9053$ (4) Å ($Z = 4$). A three-dimensional structural analysis, using automatic diffractometer data, has been completed and refined by full-matrix least-squares procedures to a residual $R = 0{\cdot}030$ ($R_w = 0{\cdot}049$). The structural unit can be described as two distorted edge-sharing octahedra of anions about the two non-equivalent manganese atoms, which are further linked through edge sharing by phosphate tetrahedra.

Introduction

Many minerals having the stoichiometry $M_2(XO_4)Z$ (where M is a divalent metal or metals, X is P, As, or V, and Z is a halogen or hydroxyl group) are known (Richmond, 1940), but few crystal structures have been reported. Recently, several crystallographic structures for relevant phosphate materials have been published: triplite, $(Mn, Fe)_2(PO_4)F$, (Waldrop, 1969); triploidite, $(Mn, Fe)_2(PO_4)(OH)$, (Waldrop, 1970); wagnerite, $Mg_2(PO_4)F$, (Coda, Giuseppetti & Tadini, 1967); tar-

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† See footnote p. 2525.

buttite, $Zn_2(PO_4)(OH)$, (Cocco, Fanfani & Zanazzi, 1966); and the structure of a synthetic analogue of the mineral specdiosite, $Ca_2(PO_4)Cl$, (Greenblatt, Banks & Post, 1967). Although $Mn_2(PO_4)Cl$ seems to have no mineral analog, it represents a new structure-type for materials of this stoichiometry and could serve as a model for unknown mineral structures.

Klement & Haselbeck (1965) first reported the preparation of $Mn_2(PO_4)Cl$ without description of its properties. Recently, its crystal growth and optical properties have been reported (Kreidler, Meehan, Wilson & Fries, 1971). We have grown single crystals of $Mn_2(PO_4)Cl$ by a similar technique; this paper describes its crystal structure.

Experimental

Preparation and crystal growth

Single crystals of manganese chlorophosphate were grown by standard flux melt techniques using excess manganese chloride as the flux. Manganese(II) phosphate trihydrate was prepared in aqueous solution at 100°C by reaction of manganese(II) sulfate monohydrate and disodium hydrogen phosphate (all starting materials were Analytical Reagent Grade, Mallinckrodt Chemical Works) and was then dehydrated at 750°C. Manganese(II) chloride tetrahydrate was carefully heated at 240°C to form the anhydrous halide.

A mixture of 25 mol.% $Mn_3(PO_4)_2$ – 75 mol.% $MnCl_2$ was placed in a 25 cm^3 platinum crucible, tightly covered to inhibit the volatilization of the halide, and inserted into a silicon carbide resistance-heated furnace which was brought to a temperature of 900°C under a flowing dry nitrogen atmosphere. After soaking at this temperature for four hours, the furnace was cooled at a rate of 8 °C.hr^{-1} to 650°C and then shut off. The crystals were separated from the melt by repeated washing with hot distilled water. Typically, crystals were of needle shape with dimensions $0{\cdot}03 \times 0{\cdot}03 \times 0{\cdot}15$ cm; no attempt was made to maximize the crystal size.

X-ray diffraction data

A crystal was ground to a 0·011 (2) cm sphere. Weissenberg and precession photographs revealed orthorhombic symmetry with systematic absences corresponding to the space groups *Pnam* or $Pna2_1$. Since a piezoelectric effect was observed (using a Giebe–Scheibe detector), the noncentrosymmetric space group was selected for refinement.

The lattice parameters were determined in a *PICK* II least-squares refinement program, using 48 reflections within the angular range $36° < 2\theta < 54°$; the reflections were automatically centered on a Picker FACS-I four-circle diffractometer using Mo $K\alpha_1$ radiation. At 22°C the lattice parameters are $a = 11{\cdot}364$ (2), $b = 8{\cdot}0965$ (7), and $c = 4{\cdot}9053$ (4) Å, where the figures in parentheses represent the standard deviations in the last reported figure. The calculated density, with $Z = 4$, is 3·536 g.cm^{-3} and the measured density (buoyant force) is 3·51 (1) g.cm^{-3}. This agreement is satisfactory since all the crystals contained visible voids.

Diffraction intensities were measured using Zr-filtered Mo $K\alpha$ radiation at a take-off angle of 2·5° with the diffractometer operating in the θ–2θ scan mode. Scans were made at 1° per min over 1·5° with allowance for dispersion, and with 40-sec background counts taken at both ends of the scan. Of the 1136 independent available data investigated in the angular range $2\theta <$ 71°, 1097 were considered observable according to the criterion $|F_o| > 0{\cdot}675\sigma_F$, where σ_F is defined as $0{\cdot}02|F_o| + (C + k^2B)^{1/2}/2|F_o|\mathrm{Lp}$; the total scan count is C, k is the ratio of scanning time to the total background time, and B is the total background count. Three reflections were systematically monitored and no random variations in intensity greater than 3·5% were observed over the entire data collection period; the mean variation was very much smaller.

The intensity data were corrected for Lorentz and polarization effects, and absorption corrections (*International Tables for X-ray Crystallography*, 1968) were applied for a spherical crystal with $\mu R = 0{\cdot}74$; the maximum absorption correction applied was 2·0% of $|F_o|$.

Determination and refinement of the structure

A straightforward analysis of the Patterson map revealed the positions of the four heavy atoms in the asymmetric unit. A least-squares refinement and difference Fourier synthesis were then calculated using manganese atoms in the two positions which contributed most strongly to the Patterson map, and a chlorine and a phosphorus atom in the other two positions. Since the four most intense image peaks in the difference Fourier map were in reasonable anion positions, oxygen atoms were placed at these sites.

Three cycles of least-squares refinement (Busing, Martin & Levy, 1962*a*) of these positions using a $1/\sigma^2$ weighting scheme, zerovalent scattering factors for Mn, P, Cl, and O (Cromer & Mann, 1968), isotropic temperature factors, and corrections for secondary extinction and anomalous dispersion, yielded a residual $R = 0{\cdot}051$ and a weighted residual $R_w = 0{\cdot}071$. Since the space group was non-centric, the refinement was also carried out using all negative coordinates; the results were $R = 0{\cdot}050$ and $R_w = 0{\cdot}069$. According to Hamilton's significance test (Hamilton, 1965), the first cell may be rejected at less than the 0·005 significance level. Hence, the second set of coordinates was selected for anisotropic refinement.

The anisotropic refinement, based on a data to parameter ratio of 15 to 1 with 73 independently varied parameters, yielded a final $R = 0{\cdot}030$ and $R_w = 0{\cdot}049$ for the observed data. Calculated and observed structure factors are listed in Table 1. In the final refinement, the maximum extinction correction (Zachariasen, 1968) was 64% of $|F_c|$ for the 040 reflection.

Table 2 presents the final atomic coordinates and anisotropic thermal parameters.

Table 1. *Observed and calculated structure factors*

H	K	L	FOBS	FCAL
0	0	-8	371	376
0	0	-6	535	528
0	0	-4	1212	1231
0	0	-2	1303	1269
0	1	-7	237	242
0	1	-3	523	507
0	1	-1	309	300
0	2	-6	26	59
0	2	-4	27	31
0	2	-2	84	84
0	3	-7	243	236
0	3	-5	262	266
0	3	-3	796	792
0	3	-1	771	778
0	4	-6	408	414
0	4	-4	1138	1144
0	4	-2	697	692
0	4	0	1961	1970
0	5	-7	255	253
0	5	-5	275	271
0	5	-3	425	430
0	5	-1	300	303
0	6	-6	79	79
0	6	-4	48	43
0	6	-2	136	146
0	6	0	63	52
0	7	-5	349	343
0	7	-3	676	682
0	7	-1	875	899
0	8	-6	243	244
0	8	-4	441	443
0	8	-2	223	225
0	8	0	805	830
0	9	-5	226	224
0	9	-3	227	221
0	9	-1	278	284
0	10	-4	90	86
0	10	-2	49	32
0	10	0	72	65
0	11	-3	529	541
0	11	-1	534	542
0	12	-2	253	261
0	12	0	265	272
0	13	-1	77	68
1	1	-7	119	124
1	1	-6	148	144
1	1	-5	84	80
1	1	-4	91	91
1	1	-3	340	337
1	1	-2	369	366
1	1	-1	286	286
1	1	0	307	321
1	2	-7	199	192
1	2	-6	569	571
1	2	-5	650	658
1	2	-4	301	295
1	2	-3	739	740
1	2	-2	972	967
1	2	-1	1519	1527
1	2	0	404	408
1	3	-7	100	101
1	3	-6	121	121
1	3	-5	186	184
1	3	-4	215	217
1	3	-3	518	527
1	3	-2	132	133
1	3	-1	479	483
1	3	0	261	267
1	4	-7	34	38
1	4	-6	23	26
1	4	-5	34	52
1	4	-4	82	88
1	4	-3	46	47
1	4	-2	76	72
1	4	-1	67	69
1	4	0	108	110
1	5	-7	201	192
1	5	-6	262	258
1	5	-5	251	246
1	5	-4	254	256
1	5	-3	337	340
1	5	-2	610	612
1	5	-1	242	234
1	5	0	523	527
1	6	-7	85	90
1	6	-6	245	252
1	6	-5	365	368
1	6	-4	175	172
1	6	-3	294	297
1	6	-2	517	526
1	6	-1	717	712
1	6	0	290	304
1	7	-6	138	133
1	7	-5	163	169
1	7	-4	251	252
1	7	-3	329	335
1	7	-2	181	182
1	7	-1	465	477
1	7	0	272	274
1	8	-5	63	53
1	8	-4	67	64
1	8	-3	33	24
1	8	-2	16	40
1	8	-1	42	52
1	8	0	212	116
1	9	-5	261	259
1	9	-4	266	270
1	9	-3	197	198
1	9	-2	513	519
1	9	-1	322	323
1	9	0	381	378
1	10	-5	139	143
1	10	-4	233	229
1	10	-3	58	49
1	10	-2	54	51
1	10	-1	208	202
1	10	0	192	203
1	11	-4	150	145
1	11	-3	220	220
1	11	-2	97	95
1	11	-1	223	230
1	11	0	153	148

H	K	L	FOBS	FCAL
1	12	-2	53	35
1	12	-1	71	64
1	12	0	41	57
1	13	-1	89	76
1	13	0	216	228
2	0	-7	537	545
2	0	-6	116	114
2	0	-5	666	659
2	0	-4	346	345
2	0	-3	1174	1147
2	0	-2	510	509
2	0	-1	1260	1258
2	1	-7	40	15
2	1	-6	121	109
2	1	-5	85	84
2	1	-4	152	150
2	1	-3	57	55
2	1	-2	390	393
2	1	-1	315	314
2	1	0	546	550
2	2	-7	18	23
2	2	-6	21	10
2	2	-5	44	38
2	2	-3	53	59
2	2	-2	30	20
2	2	-1	98	95
2	2	0	39	34
2	3	-7	100	95
2	3	-6	224	220
2	3	-5	243	245
2	3	-4	274	267
2	3	-3	234	236
2	3	-2	201	203
2	3	-1	182	166
2	3	0	366	357
2	4	-7	444	455
2	4	-6	70	72
2	4	-5	481	484
2	4	-4	270	268
2	4	-3	638	636
2	4	-2	441	435
2	4	-1	735	752
2	4	0	472	456
2	5	-6	225	225
2	5	-5	103	95
2	5	-4	332	336
2	5	-3	63	61
2	5	-2	598	609
2	5	-1	124	130
2	5	0	671	680
2	6	-7	52	41
2	6	-6	27	17
2	6	-5	51	61
2	6	-4	50	46
2	6	-3	81	83
2	6	-2	19	37
2	6	-1	127	121
2	6	0	20	2
2	7	-6	301	303
2	7	-5	71	64
2	7	-4	448	443
2	7	-3	64	57
2	7	-2	378	386
2	7	-1	240	240
2	7	0	460	455
2	8	-6	32	28
2	8	-5	179	183
2	8	-4	147	150
2	8	-3	107	113
2	8	-2	226	227
2	8	-1	170	167
2	8	0	101	81
2	9	-5	93	96
2	9	-4	285	287
2	9	-3	93	90
2	9	-2	420	426
2	9	-1	34	27
2	9	0	394	400
2	10	-5	22	22
2	10	-4	17	26
2	10	-2	50	45
2	10	-1	43	37
2	10	0	66	60
2	11	-4	331	323
2	11	-3	44	44
2	11	-2	257	256
2	11	-1	68	74
2	11	0	345	336
2	12	-3	157	163
2	12	-2	140	140
2	12	-1	290	297
2	12	0	73	36
2	13	-1	70	64
2	13	0	206	196
3	1	-7	70	79
3	1	-6	165	157
3	1	-5	135	129
3	1	-4	285	281
3	1	-3	135	135
3	1	-2	246	248
3	1	-1	493	487
3	1	0	731	706
3	2	-7	308	321
3	2	-6	480	482
3	2	-5	394	386
3	2	-4	442	431
3	2	-3	1130	1128
3	2	-2	1447	1450
3	2	-1	1266	1273
3	2	0	195	222
3	3	-7	167	161
3	3	-6	270	268
3	3	-5	216	221
3	3	-4	203	202
3	3	-3	207	214
3	3	-2	167	170
3	3	-1	426	421
3	3	0	53	42
3	4	-7	63	50
3	4	-6	36	23
3	4	-5	45	51
3	4	-4	55	58

H	K	L	FOBS	FCAL
3	4	-3	65	62
3	4	-2	31	15
3	4	-1	83	84
3	4	0	126	128
3	5	-7	167	173
3	5	-6	167	163
3	5	-5	293	287
3	5	-4	440	444
3	5	-3	345	342
3	5	-2	444	441
3	5	-1	530	539
3	5	0	785	788
3	6	-7	229	231
3	6	-6	363	368
3	6	-5	161	156
3	6	-4	48	55
3	6	-3	593	600
3	6	-2	635	636
3	6	-1	649	646
3	6	0	211	209
3	7	-6	213	210
3	7	-5	171	172
3	7	-4	159	160
3	7	-3	323	325
3	7	-2	223	225
3	7	-1	305	309
3	7	0	57	56
3	8	-6	17	15
3	8	-5	17	43
3	8	-4	61	59
3	8	-3	68	72
3	8	-2	55	29
3	8	-1	20	25
3	8	0	74	65
3	9	-5	326	321
3	9	-4	440	433
3	9	-3	396	396
3	9	-2	381	389
3	9	-1	347	347
3	9	0	559	576
3	10	-5	98	96
3	10	-4	116	117
3	10	-3	262	269
3	10	-2	240	230
3	10	-1	162	163
3	10	0	305	305
3	11	-4	85	90
3	11	-3	151	166
3	11	-2	107	103
3	11	-1	181	176
3	11	0	27	31
3	12	-3	76	69
3	12	-2	73	71
3	12	-1	37	47
3	13	0	384	396
4	0	-7	224	223
4	0	-6	445	441
4	0	-5	164	146
4	0	-4	1140	1135
4	0	-3	736	731
4	0	-2	1451	1437
4	0	-1	681	675
4	0	0	1582	1554
4	1	-7	111	113
4	1	-6	154	153
4	1	-5	165	169
4	1	-4	247	249
4	1	-3	230	220
4	1	-2	168	171
4	1	-1	312	295
4	1	0	661	648
4	2	-7	37	14
4	2	-6	32	26
4	2	-5	24	4
4	2	-4	45	45
4	2	-3	42	40
4	2	-2	65	69
4	2	-1	42	39
4	2	0	34	43
4	3	-7	232	230
4	3	-6	210	211
4	3	-5	164	160
4	3	-4	360	367
4	3	-3	434	431
4	3	-2	46	27
4	3	-1	685	682
4	3	0	410	434
4	4	-7	159	166
4	4	-6	370	375
4	4	-5	76	81
4	4	-4	632	623
4	4	-3	619	631
4	4	-2	878	887
4	4	-1	569	562
4	4	0	1436	1415
4	5	-7	183	177
4	5	-6	140	139
4	5	-5	277	280
4	5	-4	183	180
4	5	-3	276	277
4	5	-2	110	113
4	5	-1	384	398
4	5	0	344	344
4	6	-6	80	80
4	6	-5	29	12
4	6	-4	42	55
4	6	-3	78	87
4	6	-2	64	66
4	6	-1	78	74
4	6	0	90	90
4	7	-6	111	112
4	7	-5	339	341
4	7	-4	204	206
4	7	-3	647	654
4	7	-2	146	154
4	7	-1	615	609
4	7	0	467	488
4	8	-6	108	104
4	8	-5	67	65
4	8	-4	249	245
4	8	-3	401	404
4	8	-2	259	251

H	K	L	FOBS	FCAL
4	8	-1	293	291
4	8	0	391	386
4	9	-5	236	237
4	9	-4	140	128
4	9	-3	261	257
4	9	-2	45	34
4	9	-1	235	235
4	9	0	139	136
4	10	-4	40	27
4	10	-3	78	92
4	10	-2	46	50
4	10	-1	65	71
4	10	0	48	47
4	11	-4	161	170
4	11	-3	412	417
4	11	-2	64	64
4	11	-1	460	466
4	11	0	267	269
4	12	-2	90	85
4	12	-1	144	147
4	12	0	75	62
5	1	-7	74	69
5	1	-6	102	100
5	1	-5	211	214
5	1	-4	45	44
5	1	-3	198	197
5	1	-2	253	259
5	1	-1	77	78
5	1	0	238	229
5	2	-7	338	336
5	2	-6	290	288
5	2	-5	480	490
5	2	-4	614	623
5	2	-3	565	567
5	2	-2	241	248
5	2	-1	887	891
5	2	0	748	740
5	3	-7	107	112
5	3	-6	101	99
5	3	-5	154	154
5	3	-4	288	291
5	3	-3	282	287
5	3	-2	206	205
5	3	-1	549	548
5	3	0	343	350
5	4	-6	57	48
5	4	-5	30	26
5	4	-4	44	47
5	4	-3	60	56
5	4	-2	111	120
5	4	-1	51	47
5	4	0	103	106
5	5	-7	167	168
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5	8	-1	33	28
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5	9	-4	184	189
5	9	-3	229	231
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5	9	-1	190	186
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5	10	0	106	117
5	11	-3	171	176
5	11	-2	157	159
5	11	-1	218	209
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5	12	-1	39	22
5	12	0	29	12
6	0	-7	413	411
6	0	-6	214	218
6	0	-5	713	714
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6	0	-3	689	702
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6	0	-1	1646	1600
6	0	0	223	212
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6	1	-6	42	51
6	1	-5	210	221
6	1	-4	231	229
6	1	-3	124	118
6	1	-2	112	113
6	1	-1	156	156
6	1	0	169	193
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6	2	-5	29	38
6	2	-4	16	23
6	2	-3	49	51
6	2	-2	20	18
6	2	-1	61	60
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6	4	-7	215	216
6	4	-6	106	103
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6	4	-1	816	813
6	4	0	163	159
6	5	-6	124	122
6	5	-5	143	146
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6	5	-3	88	92
6	5	-2	306	312
6	5	-1	218	209
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6	6	-3	91	83
6	6	-1	88	88
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6	7	-2	563	572
6	7	-1	123	116
6	7	0	483	484
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6	8	-4	200	195
6	8	-3	198	188
6	8	-2	250	256
6	8	-1	279	276
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6	9	-4	247	245
6	9	-3	70	64
6	9	-2	233	242
6	9	-1	165	186
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6	10	-2	21	26
6	10	-1	48	38
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6	11	-2	423	430
6	11	-1	144	144
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6	12	-2	163	170
6	12	-1	165	160
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7	1	-6	212	210
7	1	-5	175	179
7	1	-4	291	284
7	1	-3	248	256
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7	1	-1	331	326
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7	2	-7	361	355
7	2	-6	353	352
7	2	-5	232	238
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7	2	-3	816	820
7	2	-2	779	780
7	2	-1	859	867
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7	3	-2	398	403
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7	3	0	259	244
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7	5	-5	294	295
7	5	-4	402	398
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7	5	0	632	625
7	6	-6	199	204
7	6	-5	94	96
7	6	-4	446	448
7	6	-3	569	567
7	6	-2	395	389
7	6	-1	470	459
7	6	0	640	628
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7	7	-5	155	159
7	7	-4	49	47
7	7	-3	164	175
7	7	-2	269	269
7	7	-1	171	166
7	8	-5	60	57
7	8	-4	54	48
7	8	-3	63	63
7	8	-2	33	34
7	8	-1	73	80
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7	9	-4	312	316
7	9	-3	350	358
7	9	-2	213	215
7	9	-1	506	514
7	9	0	605	605

H	K	L	FOBS	FCAL
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7	10	-2	137	135
7	10	-1	164	154
7	10	0	271	260
7	11	-3	145	138
7	11	-2	142	146
7	11	-1	101	96
7	11	0	47	9
7	12	-1	52	45
7	12	0	75	87
8	0	-7	148	139
8	0	-6	601	606
8	0	-5	219	218
8	0	-4	423	418
8	0	-3	448	441
8	0	-2	1098	1106
8	0	-1	338	328
8	0	0	722	713
8	1	-7	55	53
8	1	-6	200	201
8	1	-5	76	65
8	1	-4	225	222
8	1	-3	215	217
8	1	-2	129	141
8	1	-1	221	221
8	1	0	378	373
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8	2	-6	27	29
8	2	-4	33	42
8	2	-3	37	34
8	2	-2	43	33
8	2	-1	22	32
8	2	0	88	89
8	3	-7	130	121
8	3	-6	131	133
8	3	-5	216	214
8	3	-4	124	127
8	3	-3	291	297
8	3	-2	281	294
8	3	-1	221	215
8	3	0	615	612
8	4	-6	409	410
8	4	-5	60	63
8	4	-4	336	329
8	4	-3	294	287
8	4	-2	825	823
8	4	-1	431	420
8	4	0	275	257
8	5	-6	166	161
8	5	-5	217	209
8	5	-4	129	127
8	5	-3	334	341
8	5	-2	118	109
8	5	-1	416	408
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8	6	-5	50	54
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8	6	-1	62	65
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8	7	-5	316	314
8	7	-4	224	231
8	7	-3	334	332
8	7	-2	141	145
8	7	-1	465	455
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8	8	-4	40	8
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8	8	-2	269	266
8	8	-1	215	207
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8	9	-1	307	309
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8	10	-3	52	62
8	10	-2	30	21
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8	11	-2	106	116
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9	5	-5	92	84
9	5	-4	284	288
9	5	-3	219	220
9	5	-2	125	130
9	5	-1	163	176
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H	K	L	FOBS	FCAL
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9	6	-3	115	115
9	6	-2	150	163
9	6	-1	218	214
9	6	0	183	184
9	7	-5	168	169
9	7	-4	226	227
9	7	-3	127	133
9	7	-2	367	368
9	7	-1	268	262
9	7	0	285	280
9	8	-4	63	64
9	8	-3	46	32
9	8	-2	71	68
9	8	-1	20	30
9	8	0	124	65
9	9	-4	330	332
9	9	-3	218	213
9	9	-2	212	199
9	9	-1	214	218
9	9	0	212	208
9	10	-2	69	56
9	10	-1	268	278
9	11	-2	229	227
9	11	-1	119	125
9	11	0	212	212
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10	0	-2	342	348
10	0	-1	959	957
10	0	0	369	357
10	1	-6	68	84
10	1	-5	36	37
10	1	-4	222	219
10	1	-3	166	169
10	1	-2	190	188
10	1	-1	87	83
10	1	0	189	190
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10	2	-1	38	35
10	3	-6	204	207
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10	4	-5	444	443
10	4	-4	139	140
10	4	-3	473	479
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10	5	-3	122	119
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10	5	-1	101	104
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10	7	-5	62	56
10	7	-4	371	371
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10	7	-2	617	614
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10	8	-2	159	144
10	8	-1	339	338
10	8	0	266	253
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11	3	-1	135	136
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11	5	-1	460	465
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11	6	-5	148	148
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11	6	-3	34	30
11	6	-2	257	256
11	6	-1	216	212
11	6	0	708	695
11	7	-4	187	184
11	7	-3	201	196
11	7	-2	238	238
11	7	-1	148	147
11	7	0	125	122
11	8	-4	67	63
11	8	-3	58	77
11	8	-2	22	27
11	8	-1	78	80
11	8	0	67	74
11	9	-3	425	432
11	9	-2	155	154
11	9	-1	418	413
11	9	0	134	118
11	10	-2	52	92
11	10	-1	115	102
11	10	0	266	268
12	0	-6	444	443
12	0	-5	123	125
12	0	-4	425	433
12	0	-3	75	76
12	0	-2	472	477
12	0	-1	397	398
12	0	0	490	481
12	1	-6	81	89
12	1	-5	67	60
12	1	-4	51	43
12	1	-3	265	258
12	1	-2	163	170
12	1	-1	206	205
12	1	0	62	50
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12	2	-4	35	37
12	2	-3	28	23
12	2	-2	41	47
12	2	-1	53	33
12	2	0	39	26
12	3	-5	217	211
12	3	-4	115	110
12	3	-3	154	147
12	3	-2	239	251
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12	4	-1	154	149
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12	6	-3	28	38
12	6	-2	57	72
12	6	-1	74	85
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13	1	-4	155	152
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13	1	-1	150	149
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13	3	-5	150	152
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13	3	-3	81	83
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13	5	-2	137	140
13	5	-1	258	270
13	5	0	253	259
13	6	-4	160	159
13	6	-3	241	239
13	6	-2	372	380
13	6	-1	145	138
13	6	0	78	91
13	7	-3	89	85
13	7	-2	269	271
13	7	-1	105	102
13	7	0	230	224
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H	K	L	FOBS	FCAL
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14	0	-3	621	619
14	0	-2	341	349
14	0	-1	779	779
14	1	-5	167	163
14	1	-4	119	114
14	1	-3	97	94
14	1	-2	109	96
14	1	-1	225	219
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14	2	-1	33	26
14	2	0	41	10
14	3	-4	320	324
14	3	-3	99	97
14	3	-2	233	225
14	3	-1	274	281
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14	4	-4	49	49
14	4	-3	564	566
14	4	-2	293	286
14	4	-1	523	524
14	5	-4	143	147
14	5	-3	85	77
14	5	-2	169	160
14	5	-1	134	131
14	5	0	78	75
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14	6	-2	76	83
14	6	-1	36	27
14	6	0	37	56
14	7	-3	131	126
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14	7	-1	195	199
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14	8	-2	185	175
14	8	-1	231	229
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15	1	-3	158	155
15	1	-2	118	121
15	1	-1	114	125
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15	2	-1	158	162
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15	5	-2	144	138
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15	6	0	306	293
15	7	-2	143	140
15	7	-1	149	154
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16	0	-1	111	101
16	0	0	436	408
16	1	-4	43	37
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16	1	-2	109	102
16	1	-1	182	180
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16	2	-1	46	24
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16	5	-1	276	276
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16	6	0	52	33
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17	1	-2	198	199
17	1	-1	174	172
17	1	0	79	44
17	2	-3	371	369
17	2	-2	334	336
17	2	-1	423	426
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17	3	0	166	154
17	4	-2	43	50
17	4	-1	43	28
17	4	0	60	64
17	5	-1	225	209
17	5	0	176	167
18	0	-2	210	210
18	0	-1	413	403
18	0	0	279	370
18	1	-1	170	160
18	1	0	43	83
18	2	0	51	49
18	3	0	318	304

Discussion

Manganese atoms lie at the centers of highly distorted octahedra; Mn(1) is coordinated by four oxygen atoms at an average distance of 2·146 Å, one oxygen atom at 2·507 Å and one chlorine atom at 2·653 Å; Mn(2) is coordinated by three oxygens at an average distance of 2·145 Å, one oxygen at 2·475 Å and two chlorines

Table 2. *Fractional atomic coordinates* ($\times 10^4$) *and anisotropic thermal parameters* ($\times 10^2$)

Numbers in parentheses are estimated standard deviations in the last significant figure.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Mn(1)	8801 (1)	8962 (1)	½	51 (2)	82 (2)	77 (2)	2 (1)	13 (2)	− 8 (3)
Mn(2)	6243 (1)	8458 (1)	42 (2)	71 (2)	72 (2)	77 (2)	1 (1)	−16 (2)	− 5 (3)
P	6642 (1)	6218 (1)	4539 (2)	44 (3)	43 (3)	44 (4)	− 5 (2)	2 (2)	− 7 (3)
Cl	9341 (1)	6206 (1)	7647 (4)	91 (3)	100 (3)	167 (4)	−21 (3)	−54 (3)	− 1 (4)
O(1)	7737 (3)	9636 (4)	8552 (8)	86 (10)	59 (9)	93 (11)	− 8 (8)	12 (9)	−24 (9)
O(2)	6494 (3)	6208 (4)	7632 (8)	112 (11)	60 (9)	45 (10)	− 4 (8)	18 (9)	− 3 (9)
O(3)	5443 (3)	6242 (4)	2982 (8)	72 (10)	161 (12)	55 (11)	− 1 (9)	− 4 (9)	3 (9)
O(4)	7297 (3)	7766 (4)	3534 (8)	118 (11)	96 (11)	54 (10)	−20 (9)	−12 (9)	− 1 (9)

at 2·475 and 2·650 Å (the chlorine atoms are *cis*). Table 3 lists all the pertinent angles and distances for the manganese polyhedra.

Table 3. *Bond distances, polyhedral edge lengths, and bond angles for manganese atomic positions*

Numbers in parentheses are estimated standard deviations in the last significant figure.

(i) Interatomic distances

Mn(1)–O(1)	2·190 (4) Å	Mn(2)–O(1)	2·050 (4) Å
Mn(1)–O(2)	2·184 (3)	Mn(2)–O(2)	2·191 (3)
Mn(1)–O(3)	2·119 (4)	Mn(2)–O(3)	2·475 (4)
Mn(1)–O(3′)	2·507 (4)	Mn(2)–O(4)	2·164 (4)
Mn(1)–O(4)	2·092 (3)	Mn(2)–Cl	2·475 (1)
Mn(1)–Cl	2·653 (2)	Mn(2)–Cl′	2·650 (2)

Mn(1) octahedron		Mn(2) octahedron	
O(4)–O(3)	3·675 (5) Å	O(4)–O(3)	2·456 (5) Å
O(4)–O(2)	3·138 (5)	O(4)–O(2)	3·287 (5)
O(4)–O(1)	2·933 (5)	O(4)–O(1)	2·918 (5)
O(4)–Cl	3·326 (4)	O(4)–Cl′	3·378 (4)
O(3)–O(2)	2·969 (5)	O(3)–O(2)	2·884 (5)
O(3)–O(3′)	3·328 (5)	O(3)–Cl	3·563 (4)
O(3)–Cl	3·328 (4)	O(3)–Cl′	4·030 (4)
O(2)–O(1)	3·289 (5)	O(2)–O(1)	3·146 (5)
O(2)–O(3′)	2·884 (5)	O(2)–Cl	3·221 (4)
O(1)–O(3′)	2·458 (5)	O(1)–Cl	3·944 (4)
O(1)–Cl	3·351 (4)	O(1)–Cl′	3·351 (4)
O(3′)–Cl	4·088 (4)	Cl—Cl′	3·475 (2)

(ii) Angles

Mn(1) octahedron		Mn(2) octahedron	
O(4)–Mn(1)–O(3)	121·5 (1)°	O(4)–Mn(2)–O(3)	63·5 (1)°
O(4)–Mn(1)–O(2)	94·4 (1)	O(4)–Mn(2)–O(2)	98·0 (1)
O(4)–Mn(1)–O(1)	86·4 (1)	O(4)–Mn(2)–O(1)	86·8 (1)
O(4)–Mn(1)–Cl	88·2 (1)	O(4)–Mn(2)–Cl′	88·5 (1)
O(3)–Mn(1)–O(2)	87·2 (1)	O(3)–Mn(2)–O(2)	76·0 (1)
O(3)–Mn(1)–O(3′)	91·6 (1)	O(3)–Mn(2)–Cl	92·1 (1)
O(3)–Mn(1)–Cl	87·7 (1)	O(3)–Mn(2)–Cl′	103·6 (1)
O(2)–Mn(1)–O(1)	97·5 (1)	O(2)–Mn(2)–O(1)	94·9 (1)
O(2)–Mn(1)–O(3′)	75·5 (1)	O(2)–Mn(2)–Cl	87·0 (1)
O(1)–Mn(1)–Cl	87·0 (1)	O(1)–Mn(2)–Cl	119·7 (1)
O(1)–Mn(1)–O(3′)	87·0 (1)	O(1)–Mn(2)–Cl′	89·4 (1)
O(3′)–Mn(1)–Cl	62·7 (1)	Cl—Mn(2)–Cl′	85·30 (3)
O(4)–Mn(1)–O(3′)	145·2 (1)	O(4)–Mn(2)–Cl	152·6 (1)
O(3)–Mn(1)–O(1)	151·3 (2)	O(3)–Mn(2)–O(1)	146·8 (1)
O(2)–Mn(1)–Cl	174·9 (2)	O(2)–Mn(2)–Cl′	172·4 (1)

The discrete phosphate tetrahedra are quite regular with an average bond length of 1·542 Å and an average angle of 109·42° (+3·58, −4·62°). This average

Table 4. *Bond distances, polyhedral edge lengths, and bond angles for the phosphate tetrahedron*

Numbers in parentheses are estimated standard deviations in the last significant figure.

(i) Interatomic distances

P–O(1)	1·541 (3) Å	O(1)–O(2)	2·528 (5) Å
P–O(2)	1·526 (4)	O(1)–O(3)	2·458 (5)
P–O(3)	1·561 (4)	O(1)–O(4)	2·535 (5)
P–O(4)	1·539 (4)	O(2)–O(3)	2·575 (6)
		O(2)–O(4)	2·542 (5)
		O(3)–O(4)	2·456 (5)

(ii) Angles

O(1)–P–O(2)	111·0 (2)°
O(1)–P–O(3)	104·8 (2)
O(1)–P–O(4)	110·8 (2)
O(2)–P–O(3)	113·0 (2)
O(2)–P–O(4)	112·1 (2)
O(3)–P–O(4)	104·8 (2)

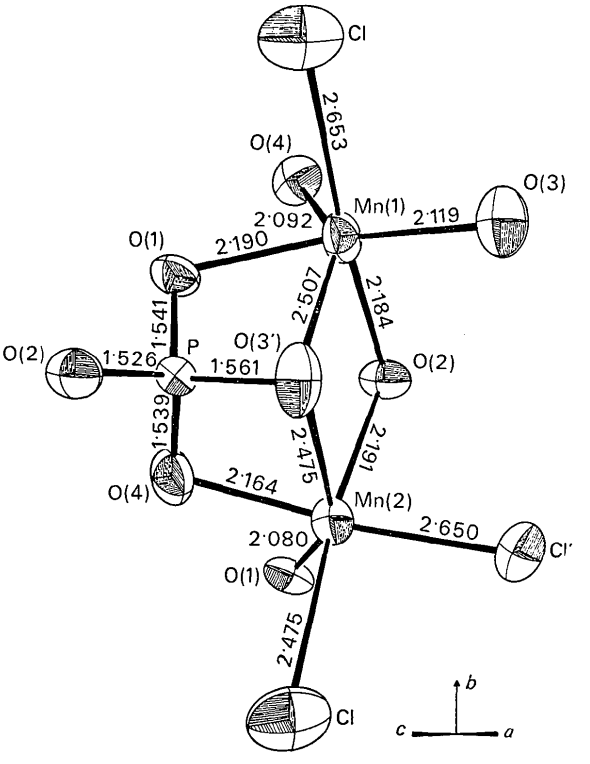

Fig. 1. The structural unit of $Mn_2(PO_4)Cl$.

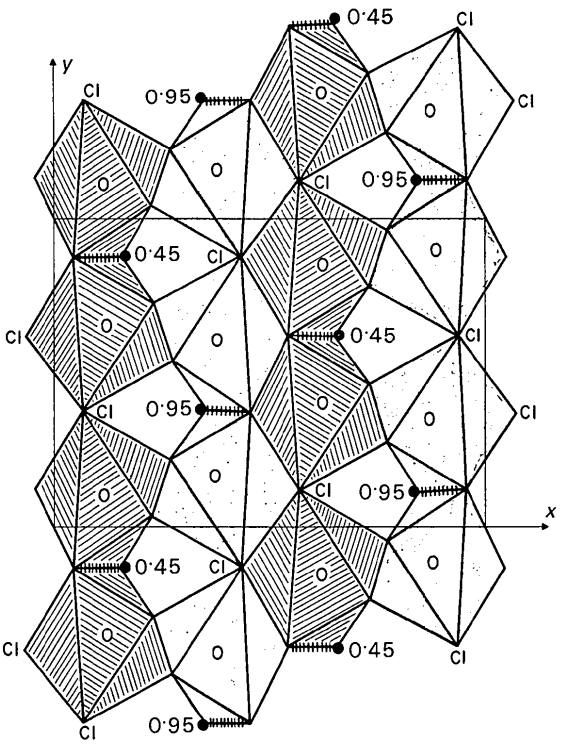

Fig. 2. A polyhedral representation of the crystal structure of $Mn_2(PO_4)Cl$ viewed down the *c* axis. Manganese atoms are represented by open circles, phosphorus atoms by solid circles. All corners not labeled Cl are oxygen atoms. The numbers indicate the *z* coordinates of the phosphorus atoms.

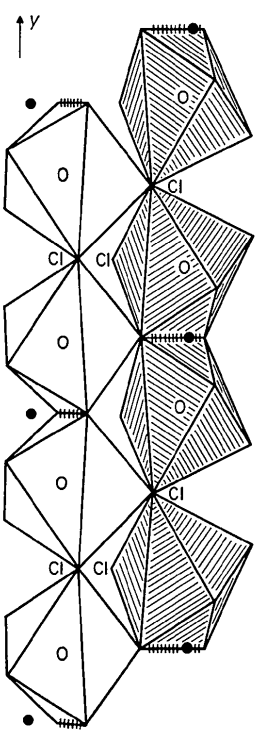

Fig. 3. The central portion of Fig. 2 rotated 10° clockwise about *b* to demonstrate the non-sharing of edges (see text).

bond distance is typical for discrete phosphate tetrahedra. Table 4 lists the tetrahedral bond angles and distances. The standard deviations of bond lengths and bond angles were computed by the function and error program, *ORFFE*, of Busing, Martin & Levy (1962*b*).

The building block of the structure is shown in Fig. 1. It is composed of the phosphorus tetrahedron and the two distorted octahedra of anions about the manganese atoms with which it shares edges; the two octahedra also share edges. The apparent mirror symmetry of the block is illusory, since for example, O(3) and Cl′ do not identify.

The entire structure can be formed by linking together these units through corner-sharing. A projection of the octahedra down the *c* axis is shown in Fig. 2. In this figure the edges shared by octahedra are shown by ticked lines. The manganese atoms at the centers of the shaded and hatched octahedra lie at $z=0{\cdot}50$ and $z=0{\cdot}00$ respectively, and are related by the twofold screw axis. Hence the shaded and hatched octahedra do not share edges, as demonstrated in Fig. 3, which is the central portion of Fig. 2 with a 10° clockwise rotation about *b*. The shaded and hatched octahedra which appeared to share edges in Fig. 2 are seen to share only corners.

The structure of $Mn_2(PO_4)Cl$ does not seem to be related to either the structure of chlorospodiosite (Greenblatt, Banks & Post, 1967), wagnerite (Coda, Giuseppetti & Tadini, 1967), or triplite/triploidite (Waldrop, 1969, 1970).

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