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Crystal and Molecular Structure of the Cd Salt of D(—)-Phosphoglyceric Acid

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The crystal and molecular structure of the cadmium salt of D(−)-phosphoglyceric acid has been determined by three-dimensional X-ray methods. The crystals are monoclinic with unit cell dimensions $a = 7.888$ Å, $b = 8.530$ Å, $c = 7.733$ Å and $\beta = 92.75°$. The space group is $P2_1$ with two molecules per unit cell. The structure determination was based on 916 reflections, and a final R-factor of 0.068 was reached by least squares refinements. The cadmium ion is surrounded by seven oxygen atoms, arranged as a pentagonal bipyramid. The crystals were found to contain six molecules of water per unit cell.

Our interest in 3-phosphoglyceric acid, $H_2PO_4CH_2CHOHCOOH$, was stimulated by the important role this molecule plays in the energy metabolism. The compound is an intermediate in carbohydrate metabolism and an end product in the oxidation of 3-phosphoglyceraldehyde *via* 1,3-di-phosphoglyceric acid, the energy of oxidation being used in the formation of adenosine-triphosphate (ATP) from adenosine-diphosphate. It is also a starting point in the formation of phospho-enol-pyruvic acid by the isomerisation to 2-phosphoglyceric acid.

3-Phosphoglyceric acid has one asymmetric carbon atom and thus exists in two optically active forms. The D-form, which is characterized by $[\alpha]_D^{25} = -14.5°$, was studied. Attempts to crystallize salts formed by this compound and one of the lighter metal ions, especially Mg and Ca, were unsuccessful. Good crystals were obtained only of the barium and cadmium salts, so we chose to investigate the cadmium compound. The cadmium ion has 46 electrons constituting 27 % of the total, and thus the interesting part of the structure was expected to suffer from relatively large standard deviations in bond and angle values.

EXPERIMENTAL

Small prismatic crystals were obtained by slow diffusion of ethanol into a solution of the compound in water. They were easy to cut, and pieces of approximately $0.10 \times 0.10 \times 0.20$ mm were used in collecting the X-ray data. The crystals proved to be

unstable, and three different specimens were used to obtain the intensity data. Unit cell dimensions were determined by least squares analysis of 26 lines on a Guinier photograph taken at room temperature with Cu$K\alpha$ radiation ($a = 1.54050$ Å) using KCl as a standard. The intensity data were obtained by using multi-film, integrating equi-inclination Weissenberg technique with Ni-filtered Cu$K\alpha$ radiation at room temperature. The layers $h0l$ to $h3l$ were recorded with the first crystal, $h3l$ to $h5l$ with the second, and $0kl$ and $1kl$ with the third. The intensities were measured photometrically except for the weakest reflections which were estimated visually with the use of a calibrated scale. The total number of reflections obtainable with copper radiation is 1324. Of these, 916 might occur on the recorded films. 842 reflections were actually observed to be above the background level, hence 74 reflections were recorded as zero observed. The intensities were corrected for absorption effects.

The full matrix least squares program used in the parameter refinement procedure [1] minimizes the functions $\sum W^2(F_o - F_c)^2$. The weight W applied to the structure factors was constant for $|F_o| < 30.0$, and proportional to $|F_o|^{-2/3}$ for larger values of F_o. Non-observed reflections were included with a structure factor of the most probable value [2] and assigned a weight of one third of the weight given to the observed reflections. The atomic form factors used were those of Hanson *et al.*,[3] except for the cadmium ion where the values were those given by Cramer *et al.*[4]

The density of the crystals was measured by flotation methods.

CRYSTAL DATA

Asymmetric unit: $C_3H_5O_7PCd,3H_2O$. Crystal system: monoclinic. Unit cell dimensions: $a = 7.888(0.002)$Å; $b = 8.530(0.002)$Å; $c = 7.733(0.002)$Å; $\beta = 92.75(0.01°)$. The figures in parentheses are estimated standard deviations. Molecular weight: 350.4. Cell volume: 519.8 Å^3. Measured density: 2.203 g/cm^3. $F_{000} = 340$. Absent reflections: $0k0$ when $k = 2n + 1$. Space group: $P2_1$.

STRUCTURE DETERMINATION

The space groups compatible with the systematically absent reflections are $P2_1/m$ and $P2_1$. The optical activity excludes mirror symmetry, hence the space group is $P2_1$. The position of the cadmium ion and the phosphorus atom were located from a Patterson map. A subsequent Fourier in the $h0l$ projection indicated the position of the entire molecule and also the location of one molecule of water. The suggested structure yielded an R-factor of 0.15 based on the three-dimensional data. A three-dimensional Fourier synthesis strongly indicated the presence of more water molecules, and the introduction of one additional water molecule led to an R-factor of 0.09.

Also a third molecule of water was included from considerations of a model of the coordination around the cadmium ion and from indications from a three-dimensional difference map. This gave an R-factor of 0.08.

The hydrogen atoms could not be located by Fourier methods and were placed in expected positions and given a temperature factor of 5.0 or 4.0.

The final refinements with anisotropic thermal parameters for all the atoms except hydrogen gave an R-factor of 0.068. The final parameters are listed in Table 2 together with their standard deviations. A comparison of observed and calculated structure factors is given in Table 1, the figures for the non-observed reflections being those actually used in the least squares calculations.

Table 1. Observed and calculated structure factors. The columns are h, k, l, F_o and F_c.

0	0	1	718	741
0	0	2	551	524
0	0	3	77	60
0	0	4	495	479
0	0	5	490	481
0	0	6	272	291
0	0	7	104	98
0	0	8	186	185
0	0	9	177	187
0	1	1	331	349
0	1	2	729	669
0	1	3	608	607
0	1	4	337	357
0	1	5	159	147
0	1	6	454	451
0	1	7	261	266
0	1	8	175	166
0	1	9	126	121
0	2	0	1161	1076
0	2	1	839	735
0	2	2	129	159
0	2	3	465	448
0	2	4	388	382
0	2	5	403	430
0	2	6	331	336
0	2	7	67	64
0	2	8	176	179
0	2	9	149	176
0	3	1	517	515
0	3	2	454	442
0	3	3	558	560
0	3	4	251	255
0	3	5	154	183
0	3	6	167	177
0	3	7	319	325
0	3	8	217	207
0	3	9	72	64
0	4	0	564	501
0	4	1	651	620
0	4	2	267	261
0	4	3	239	256
0	4	4	403	418
0	4	5	403	409
0	4	6	235	241
0	4	7	74	58
0	4	8	91	100
0	4	9	113	122
0	5	1	515	505
0	5	2	502	493
0	5	3	229	223
0	5	4	197	205
0	5	5	172	160
0	5	6	204	209
0	5	7	255	281
0	5	8	124	147
0	6	0	442	445
0	6	1	295	275
0	6	2	84	83
0	6	3	210	224
0	6	4	379	391
0	6	5	296	299
0	7	1	295	310
0	7	2	288	268
0	7	3	260	237
0	7	4	242	195
0	7	6	91	110
0	7	7	162	184
0	8	0	264	275
0	8	1	261	243
0	8	2	127	139
0	8	3	124	131
0	8	4	278	240
0	8	5	185	181
0	9	1	91	120
0	9	2	114	143
0	9	3	128	143
0	10	0	240	224
0	10	1	113	123
1	0	-6	338	345
1	0	-5	665	700
1	0	-4	430	464
1	0	-3	214	210
1	0	-2	586	580
1	0	-1	851	834
1	0	0	601	705
1	0	1	621	579
1	0	3	575	586
1	0	4	610	595
1	0	5	307	297
1	0	7	99	93
1	0	8	123	109
1	0	9	191	201
1	1	-9	155	154
1	1	-8	305	298
1	1	-7	249	257
1	1	-6	111	132
1	1	-5	314	326
1	1	-4	471	476
1	1	-3	311	322
1	1	-2	400	404
1	1	-1	390	387
1	1	0	597	599
1	1	1	984	923
1	1	2	562	512
1	1	3	529	491
1	1	4	214	210
1	1	5	183	185
1	1	6	390	383
1	1	7	411	434
1	1	8	153	151
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1	2	-9	107	115
1	2	-7	119	117
1	2	-6	247	274
1	2	-5	614	612
1	2	-4	579	572
1	2	-3	86	107
1	2	-2	460	461
1	2	-1	530	480
1	2	0	703	699
1	2	1	712	668
1	2	2	362	369
1	2	3	872	817
1	2	4	464	439
1	2	5	201	199
1	2	6	128	134
1	2	7	159	150
1	2	8	193	195
1	2	9	157	166
1	3	-9	135	131
1	3	-8	205	215
1	3	-7	195	199
1	3	-6	217	235
1	3	-5	114	91
1	3	-4	397	443
1	3	-3	704	739
1	3	-2	375	393
1	3	-1	124	116
1	3	0	131	109
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1	3	2	793	804
1	3	3	280	284
1	3	4	160	150
1	3	5	216	220
1	3	6	319	348
1	3	7	265	288
1	3	8	108	113
1	3	9	81	44
1	4	-9	158	127
1	4	-7	151	155
1	4	-6	300	296
1	4	-5	349	355
1	4	-4	215	239
1	4	-3	62	66
1	4	-2	360	359
1	4	-1	654	693
1	4	0	972	859
1	4	1	351	338
1	4	2	337	340
1	4	3	407	427
1	4	4	407	411
1	4	5	344	337
1	4	6	155	140
1	4	7	87	92
1	4	8	181	195
1	4	9	112	136
1	5	-8	135	146
1	5	-7	173	194
1	5	-6	168	156
1	5	-5	121	85
1	5	-4	378	397
1	5	-3	397	412
1	5	-2	284	280
1	5	-1	82	61
1	5	0	210	212
1	5	1	260	254
1	5	2	502	474
1	5	3	277	279
1	5	5	183	169
1	5	6	282	275
1	5	7	176	164
1	5	8	72	79
1	6	-7	118	106
1	6	-6	224	222
1	6	-5	238	202
1	6	-4	155	160
1	6	-2	174	163
1	6	-1	501	506
1	6	0	453	458
1	6	2	189	196
1	6	3	209	206
1	6	4	305	309
1	6	5	238	243
1	6	6	89	87
1	6	7	81	70
1	6	8	131	170
1	7	-7	105	133
1	7	-6	111	121
1	7	-4	313	309
1	7	-3	286	289
1	7	-2	135	127
1	7	0	78	75
1	7	1	208	191
1	7	2	374	361
1	7	3	275	263
1	7	5	137	147
1	7	6	132	135
1	7	7	126	146
1	8	-6	99	108
1	8	-5	162	189
1	8	-4	147	147
1	8	-2	198	183
1	8	-1	347	374
1	8	0	293	306
1	8	1	89	102
1	8	2	113	103
1	8	3	222	226
1	8	4	189	210
1	8	5	156	149
1	8	6	77	85
1	9	-4	150	147
1	9	-3	186	180
1	9	-2	133	119
1	9	0	132	104
1	9	1	176	169
1	9	2	198	185
1	9	3	131	123
1	9	5	99	100
1	10	-1	163	171
1	10	0	153	163
1	10	1	115	72
1	10	3	117	122
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2	0	-8	105	92
2	0	-7	163	164
2	0	-6	354	346
2	0	-5	228	231
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2	0	-3	146	141
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2	0	-1	1252	1560
2	0	0	70	27
2	0	1	205	207
2	0	2	520	537
2	0	3	965	961
2	0	4	533	516
2	0	5	140	140
2	0	6	93	68
2	0	7	237	230
2	0	8	246	235
2	0	9	99	104
2	1	-9	132	140
2	1	-8	232	215
2	1	-7	174	175
2	1	-6	72	39
2	1	-5	290	312
2	1	-4	696	717
2	1	-3	595	596
2	1	-2	78	50
2	1	-1	286	301
2	1	0	222	215
2	1	1	578	642
2	1	2	822	825
2	1	3	315	297
2	1	4	278	276
2	1	5	398	347
2	1	6	277	274
2	1	7	208	211
2	1	8	58	57
2	1	9	115	111
2	2	-9	86	95
2	2	-8	104	93
2	2	-7	216	227
2	2	-6	235	253
2	2	-5	337	341
2	2	-4	276	286
2	2	-3	296	346
2	2	-2	710	647
2	2	-1	576	479
2	2	0	444	427
2	2	1	219	213
2	2	2	700	618
2	2	3	539	500
2	2	4	461	452
2	2	5	225	213
2	2	6	145	125
2	2	7	239	236
2	2	8	224	237
2	2	9	101	112
2	3	-9	205	179
2	3	-8	225	189
2	3	-7	128	120
2	3	-6	71	59
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2	3	-4	456	517
2	3	-3	357	374
2	3	-2	95	87
2	3	-1	186	180
2	3	0	235	256
2	3	1	591	582
2	3	2	592	616
2	3	3	100	111
2	3	4	137	136
2	3	5	244	250
2	3	6	313	334
2	3	7	186	196
2	3	8	87	50
2	3	9	103	87
2	4	-8	64	63
2	4	-7	196	203
2	4	-6	256	254
2	4	-5	328	320
2	4	-4	151	153
2	4	-3	382	393
2	4	-2	565	550
2	4	-1	548	531
2	4	0	458	431
2	4	1	150	162
2	4	2	326	346
2	4	3	556	519
2	4	4	410	405
2	4	5	217	204
2	4	7	161	175
2	4	8	156	165
2	5	-8	176	190
2	5	-7	105	123
2	5	-5	167	173
2	5	-4	272	243
2	5	-3	393	392
2	5	-2	198	220
2	5	-1	158	147
2	5	0	467	429
2	5	1	491	433
2	5	2	249	229
2	5	3	183	172
2	5	4	159	145
2	5	5	367	366
2	5	6	281	269
2	5	7	132	136
3	0	-8	111	91
3	0	-7	352	330
3	0	-6	270	282
3	0	-5	72	80
3	0	-4	162	172
3	0	-3	592	648
3	0	-2	801	866
3	0	-1	603	696
3	0	0	47	42
3	0	1	399	391
3	0	2	551	560
3	0	3	646	662
3	0	4	255	241
3	0	7	200	201
3	0	8	244	231
3	0	9	83	89
3	1	-9	226	224
3	1	-8	175	139
3	1	-6	196	177
3	1	-5	266	269
3	1	-4	278	284
3	1	-3	403	411
3	1	-1	621	676
3	1	0	297	358
3	1	1	244	279
3	1	2	451	459
3	1	3	82	65
3	1	4	409	400
3	1	5	545	536
3	1	6	313	307
3	1	7	90	68
3	1	8	92	104
3	1	9	117	128
3	2	-8	139	127
3	2	-7	260	274
3	2	-6	318	325
3	2	-5	186	188
3	2	-4	150	164
3	2	-3	481	450
3	2	-2	642	588
3	2	-1	645	610
3	2	0	356	354
3	2	1	454	434
3	2	2	715	681
3	2	3	448	419
3	2	4	218	213
3	2	6	238	252
3	2	7	182	185
3	2	8	192	191
3	3	-9	168	164
3	3	-8	167	130

Table 1. Continued.

3	3	-7	71	56
3	3	-6	138	138
3	3	-5	281	282
3	3	-4	431	440
3	3	-3	241	274
3	3	-2	75	63
3	3	-1	379	358
3	3	0	721	716
3	3	1	382	380
3	3	2	200	207
3	3	3	84	72
3	3	4	315	319
3	3	5	378	409
3	3	6	252	258
3	3	7	139	110
3	3	8	115	95
3	4	-8	168	149
3	4	-7	186	203
3	4	-6	229	234
3	4	-5	121	121
3	4	-4	142	131
3	4	-3	322	320
3	4	-2	574	573
3	4	-1	452	482
3	4	0	260	269
3	4	1	221	227
3	4	2	465	476
3	4	3	516	498
3	4	4	321	304
3	4	6	221	215
3	4	7	164	172
3	4	8	116	134
3	5	-8	125	140
3	5	-6	171	180
3	5	-5	256	266
3	5	-4	388	389
3	5	-3	189	214
3	5	-2	55	71
3	5	-1	193	201
3	5	0	432	386
3	5	1	446	418
3	5	2	181	140
3	5	4	226	207
3	5	5	304	267
3	5	6	181	159
3	5	7	78	103
4	0	-9	113	86
4	0	-8	157	150
4	0	-7	259	228
4	0	-6	196	192
4	0	-5	124	111
4	0	-4	179	175
4	0	-3	743	751
4	0	-2	627	691
4	0	-1	41	32
4	0	0	193	208
4	0	1	315	312
4	0	2	618	622
4	0	3	431	432
4	0	5	224	229
4	0	6	335	335
4	0	7	157	155
4	1	-9	122	134
4	1	-8	97	91
4	1	-7	117	114
4	1	-6	334	328
4	1	-5	293	296
4	1	-4	109	123
4	1	-3	110	99
4	1	-2	165	199
4	1	-1	474	478
4	1	0	815	813
4	1	1	439	434
4	1	2	129	135
4	1	3	291	271
4	1	4	349	327
4	1	5	360	359
4	1	6	214	223
4	1	8	186	184
4	2	-8	160	170
4	2	-7	182	202
4	2	-6	220	225
4	2	-5	167	173
4	2	-4	265	262
4	2	-3	444	418
4	2	-2	475	481
4	2	-1	279	273
4	2	0	269	279
4	2	1	363	349
4	2	2	308	300
4	2	3	359	333
4	2	5	81	61
4	2	6	236	237
4	2	7	300	293
4	2	8	198	207
4	3	-8	68	69
4	3	-7	112	80
4	3	-6	303	295
4	3	-5	343	320
4	3	-4	274	245
4	3	-2	181	187
4	3	-1	378	388
4	3	0	469	521
4	3	1	277	312
4	3	2	131	117
4	3	3	139	129
4	3	4	326	326
4	3	5	333	327
4	3	6	168	176
4	3	7	123	83
4	3	8	120	122
4	4	-8	129	139
4	4	-7	197	207
4	4	-6	135	125
4	4	-5	179	154
4	4	-4	217	219
4	4	-3	318	309
4	4	-2	411	407
4	4	-1	264	269
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4	4	2	448	425
4	4	3	259	267
4	4	4	160	123
4	4	5	140	117
4	4	6	203	195
4	4	7	198	190
4	5	-6	127	130
4	5	-5	273	272
4	5	-4	236	250
4	5	-2	159	146
4	5	-1	389	383
4	5	0	235	230
4	5	1	161	152
4	5	2	209	163
4	5	3	236	206
4	5	4	389	361
4	5	5	217	221
5	0	-8	222	200
5	0	-7	179	171
5	0	-5	241	247
5	0	-4	277	258
5	0	-3	311	291
5	0	-2	267	270
5	0	-1	87	59
5	0	0	424	449
5	0	1	725	772
5	0	2	356	365
5	0	3	113	109
5	0	4	65	52
5	0	5	110	75
5	0	6	321	296
5	0	7	256	224
5	1	-8	63	51
5	1	-7	122	120
5	1	-6	214	216
5	1	-5	367	345
5	1	-4	209	218
5	1	-3	250	245
5	1	-2	331	347
5	1	-1	243	254
5	1	0	325	326
5	1	1	252	243
5	1	2	185	219
5	1	3	339	345
5	1	4	429	413
5	1	5	201	205
5	1	6	62	42
5	1	7	155	146
5	1	8	133	126
5	2	-8	135	170
5	2	-7	143	170
5	2	-5	183	188
5	2	-4	285	319
5	2	-3	248	267
5	2	-2	320	325
5	2	-1	147	158
5	2	0	422	437
5	2	1	570	568
5	2	2	303	261
5	2	3	163	173
5	2	4	146	130
5	2	5	225	243
5	2	6	227	218
5	2	7	198	200
5	3	-7	137	120
5	3	-6	303	251
5	3	-5	260	251
5	3	-4	121	108
5	3	-3	115	112
5	3	-2	298	301
5	3	-1	427	418
5	3	0	306	323
5	3	1	140	157
5	3	2	262	260
5	3	3	309	300
5	3	4	291	295
5	3	5	213	224
5	3	6	95	73
5	3	7	139	131
5	4	-4	352	323
5	4	-3	351	340
5	4	-2	182	181
5	4	-1	135	109
5	4	0	242	258
5	4	1	334	371
5	4	2	390	398
5	4	3	191	205
5	4	4	140	124
5	4	5	237	203
5	4	6	191	177
5	4	7	119	128
5	5	-7	110	122
5	5	-6	195	193
5	5	-5	185	165
5	5	-2	112	138
5	5	-1	314	317
5	5	0	278	284
5	5	1	89	67
5	5	2	185	176
5	5	3	211	205
5	5	4	209	201
5	5	5	149	137
6	0	-8	99	76
6	0	-7	91	75
6	0	-5	239	223
6	0	-4	549	530
6	0	-3	307	285
6	0	-2	177	173
6	0	-1	174	161
6	0	0	302	293
6	0	1	465	469
6	0	2	319	331
6	0	4	245	252
6	0	5	260	249
6	0	6	127	101
6	0	7	77	54
6	1	-8	95	108
6	1	-7	194	179
6	1	-6	172	148
6	1	-5	93	81
6	1	-4	83	61
6	1	-3	140	161
6	1	-2	277	264
6	1	-1	406	386
6	1	0	170	161
6	1	1	151	155
6	1	2	340	337
6	1	3	293	291
6	1	4	210	217
6	1	5	116	116
6	1	6	108	82
6	1	7	204	211
6	2	-7	84	88
6	2	-5	220	227
6	2	-4	344	359
6	2	-3	268	280
6	2	-2	101	83
6	2	-1	170	174
6	2	0	196	216
6	2	1	301	314
6	2	2	245	259
6	2	3	119	89
6	2	4	212	211
6	2	5	187	198
6	2	6	123	137
6	3	-7	175	156
6	3	-6	205	167
6	3	-5	175	147
6	3	-3	273	263
6	3	-2	288	279
6	3	-1	237	237
6	3	0	96	92
6	3	1	120	110
6	3	2	175	177
6	3	3	298	288
6	3	4	228	221
6	3	5	122	113
6	3	6	66	51
6	3	7	135	159
6	4	-6	59	67
6	4	-5	176	160
6	4	-4	210	202
6	4	-3	199	190
6	4	-2	75	86
6	4	-1	157	175
6	4	0	324	338
6	4	1	317	319
6	4	2	168	182
6	4	4	152	149
6	4	5	175	156
6	4	6	166	173
6	4	-6	150	160
6	5	-5	133	122
6	5	-3	207	221
6	5	-2	252	268
6	5	-1	108	119
6	5	0	139	126
6	5	2	188	208
6	5	3	265	284
6	5	4	160	159
7	0	-7	82	60
7	0	-6	140	118
7	0	-5	173	150
7	0	-4	220	209
7	0	-3	112	97
7	0	-2	102	61
7	0	-1	311	300
7	0	0	369	360
7	0	1	94	68
7	0	4	209	192
7	0	5	235	200
7	0	6	127	118
7	1	-7	109	117
7	1	-6	158	144
7	1	-4	168	157
7	1	-3	259	268
7	1	-2	256	233
7	1	-1	126	125
7	1	0	94	69
7	1	1	124	142
7	1	2	324	326
7	1	3	280	279
7	1	4	128	119
7	1	5	75	62
7	1	6	111	113
7	2	-6	111	106
7	2	-5	134	161
7	2	-4	179	192
7	2	-3	177	189
7	2	-1	282	304
7	2	0	231	241
7	2	1	144	137
7	2	2	82	76
7	2	3	149	147
7	2	4	181	197
7	2	5	163	173
7	2	6	96	113
7	3	-6	99	93
7	3	-4	136	121
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7	3	-1	151	169
7	3	0	95	69
7	3	1	171	161
7	3	2	266	251
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7	3	6	106	118
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7	4	-4	175	173
7	4	-3	137	129
7	4	-2	156	145
7	4	-1	219	222
7	4	0	224	233
7	4	1	240	243
7	4	3	113	121
7	4	4	175	177
7	4	5	164	172
7	5	-3	76	123
7	5	-2	216	226
7	5	-1	113	119
7	5	1	79	86
7	5	2	133	149
7	5	3	151	157
7	5	4	61	60
8	0	-6	167	155
8	0	-5	212	181
8	0	-3	76	64
8	0	-2	154	137
8	0	-1	306	267
8	0	0	269	253
8	0	1	111	114
8	0	2	125	110
8	0	3	197	208
8	0	4	181	172
8	0	5	134	125
8	1	-5	57	45
8	1	-4	107	95
8	1	-3	234	221
8	1	-2	229	207
8	1	-1	76	59
8	1	0	141	129
8	1	1	155	149
8	1	2	162	156
8	1	3	167	174
8	1	5	122	134
8	2	-6	95	149
8	2	-5	120	150
8	2	-4	80	95
8	2	-2	131	129
8	2	-1	177	194
8	2	0	177	204
8	2	1	120	101
8	2	2	89	97
8	2	3	153	166
8	2	4	116	144
8	2	5	74	90
8	3	-5	83	68
8	3	-4	107	125
8	3	-3	199	169
8	3	-2	155	150
8	3	0	120	101
8	3	1	132	147
8	3	2	176	166
8	3	3	128	121
8	3	4	82	61
8	4	-5	100	113
8	4	-4	95	100
8	4	-2	139	135
8	4	-1	207	214
8	4	0	175	182
8	4	1	71	68
8	4	3	120	136
8	4	4	117	157
8	5	-4	104	111
8	5	-3	79	116
8	5	-2	87	95
8	5	1	165	191
8	5	2	145	179
9	0	-3	123	111
9	0	-2	235	212
9	0	-1	176	157
9	0	3	158	148
9	0	4	107	155
9	1	-4	94	102
9	1	-3	59	65

Table 1. Continued.

9	1	-2	99	76	0	6	6	52	59 *	3	2	5	64	50 *	6	3	-4	49	36 *
9	1	0	188	168	0	6	7	41	39 *	3	4	5	61	40 *	6	4	-7	30	41 *
9	1	1	232	223	0	6	8	26	58 *	3	5	-7	64	26 *	6	5	-4	66	30 *
9	1	2	105	116	0	7	5	67	31 *	3	5	3	66	37 *	6	5	1	68	67 *
9	2	-3	75	90	0	8	6	34	67 *	3	5	8	39	39 *	7	0	2	25	17 *
9	2	-2	155	183	0	9	4	52	113 *	4	0	4	24	59 *	7	0	3	25	27 *
9	2	-1	128	139	0	9	5	33	16 *	4	1	7	41	38 *	7	1	-5	40	18 *
9	2	0	122	94	0	10	2	48	48 *	4	3	-7	45	60 *	7	2	-7	31	48 *
9	2	2	148	113	0	10	3	43	65 *	4	3	-3	43	39 *	7	2	-2	64	49 *
9	2	3	89	109	1	0	-8	24	26 *	4	5	-7	58	54 *	7	4	2	53	44 *
9	3	-3	106	99	1	0	-7	25	9 *	4	5	-3	74	73 *	7	5	0	63	85 *
9	3	-2	61	66	1	0	2	54	20 *	4	5	6	66	85 *	8	0	-4	20	17 *
9	3	0	136	132	1	0	6	24	53 *	4	5	7	67	80 *	8	1	4	33	20 *
9	3	1	171	174	1	2	-8	59	53 *	5	0	-6	25	17 *	8	2	-3	55	37 *
9	3	2	122	144	1	4	-8	51	42 *	5	2	-6	62	80 *	8	3	-1	45	12 *
9	3	3	59	59	2	4	-9	31	63 *	5	2	8	31	50 *	8	4	-3	42	31 *
9	4	-2	88	115	2	4	6	60	62 *	5	3	-8	29	37 *	8	4	2	43	53 *
9	4	-1	157	173	2	5	-6	66	83 *	5	4	-6	58	69 *	8	5	-1	50	45 *
9	4	0	89	105	2	5	8	50	42 *	5	5	-4	75	100 *	8	5	0	50	60 *
9	4	1	39	49	3	0	-9	35	22 *	5	5	-3	73	56 *	9	0	0	19	34 *
9	4	2	101	122	3	0	5	25	4 *	5	5	6	49	86 *	9	0	1	19	10 *
10	0	-1	158	142	3	0	6	25	1 *	6	0	-6	23	9 *	9	0	2	18	30 *
10	1	-1	85	91	3	1	-7	44	36 *	6	0	3	25	40 *	9	1	-1	35	29 *
10	1	0	77	85	3	1	-2	29	20 *	6	2	-6	57	56 *	9	2	-4	43	65 *
										6	2	7	37	59 *	10	0	0	11	20 *

DISCUSSION

The water content found does not agree with that reported by Neuberg and Kobel [5] and also leads to a calculated density (2.238 g/cm³) slightly greater than the measured density (2.203 g/cm³). However, three water molecules per glycerophosphate molecule give the best agreement between the calculated and the measured density and this is also supported by crystallographic evidence. The fact that the crystals are not stable may indicate that part of the water is easily lost, which may explain the low density measurement and the low water content in properly dried material.

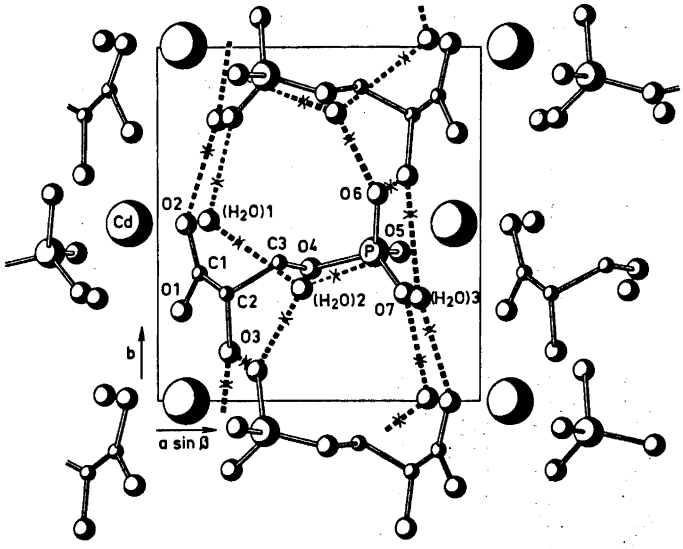

Fig. 1. The structure of the Cd-salt of 3-phosphoglyceric acid viewed along the *c*-axis.

Table 2. Final parameters and their estimated standard deviations. The temperature factor has the form of $\exp-(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{12}hk+B_{13}hl+B_{23}kl)$. The values have been multiplied by 10^{-4}.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cd	0724	0000	1055	0116	0095	0103	0015	0004	0002
	1	0	1	1	2	2	7	2	6
P	6695	4227	2403	0105	0120	0085	0008	0005	0021
	6	8	6	7	10	7	14	11	15
C1	1247	3752	1694	0192	0211	0167	−0293	0126	0227
	33	44	31	44	68	40	108	68	91
C2	2094	3068	3393	0093	0077	0212	0002	0008	−0009
	24	30	30	27	42	42	68	53	70
C3	3788	3915	3814	0110	0222	0120	0039	−0003	0033
	25	40	24	28	57	30	75	47	71
O1	0801	2622	0558	0140	0119	0284	−0091	−0021	−0013
	19	24	25	25	32	38	52	52	59
O2	0950	5122	1577	0179	0044	0148	0067	−0050	0037
	18	33	17	23	26	20	76	34	62
O3	2257	1429	3258	0253	0020	0183	0026	−0073	−0033
	26	19	23	35	26	31	64	54	44
O4	4739	3822	2271	0120	0265	0134	−0139	0011	−0031
	18	29	18	23	43	23	56	37	57
O5	7121	4287	0579	0115	0224	0126	−0076	0025	0017
	17	22	17	21	42	22	42	35	45
O6	6833	5892	3294	0244	0174	0132	−0001	−0011	0065
	24	26	21	36	39	27	68	53	62
O7	7658	3151	3646	0167	0151	0160	0135	−0078	−0009
	20	24	20	26	36	27	38	44	51
$(H_2O)1$	8441	0181	2701	0181	0251	0192	0223	0027	−0143
	18	38	19	25	53	27	88	42	86
$(H_2O)2$	5614	8258	1510	0158	0173	0287	−0074	−0129	0029
	21	28	27	28	43	41	58	55	66
$(H_2O)3$	1852	7976	2879	0369	0025	0205	0050	−0068	−0050
	31	21	26	56	24	34	79	72	50

	x	y	z	B
H1(C3)	3558	5154	4116	4.0
H2(C3)	4434	3449	4920	4.0
H(C2)	1258	3316	4437	4.0
H(O3)	7878	5278	6616	5.0
H(O6)	7162	6086	4539	5.0
H1$(H_2O)1$	8143	1291	3059	5.0
H2$(H_2O)1$	7459	9513	2287	5.0
H1$(H_2O)2$	6083	7348	2196	5.0
H2$(H_2O)2$	4615	8635	0743	5.0
H1$(H_2O)3$	2030	8039	4552	5.0
H2$(H_2O)3$	1523	6928	2389	5.0

Table 3. Interatomic distances (Å) and bond angles (°). Standard deviations in parenthesis.

C1 – O1	1.34 (3)	O1 – C1 – O2	128 (3)
C1 – O2	1.19 (4)	O1 – C1 – C2	112 (3)
C1 – C2	1.56 (4)	O2 – C1 – C2	120 (2)
C2 – C3	1.54 (3)	C1 – C1 – C3	110 (2)
C2 – O3	1.41 (3)	C1 – C2 – O3	110 (2)
C3 – O4	1.44 (2)	O3 – C2 – C3	114 (2)
P – O4	1.58 (2)	C2 – C3 – O4	106 (2)
P – O5	1.47 (1)	C3 – O4 – P	119 (1)
P – O6	1.58 (2)	O4 – P – O5	102 (1)
P – O7	1.51 (2)	O4 – P – O6	106 (1)
		O4 – P – O7	112 (1)
		O5 – P – O6	112 (1)
		O5 – P – O7	120 (1)
		O6 – P – O7	104 (1)

The structure as viewed down the *c*-axis is illustrated in Fig. 1. Table 3 gives the interatomic distances and bond angles. The bond lengths and angles are also shown in Fig. 2. The components of the thermal vibrational r.m.s. amplitudes along the axes of the unit cell are listed in Table 4. Fig. 3 illustrates the vibrational ellipsoids of the atoms. Despite the fact that the data were collected mainly along one zone axis and from different crystals, the vibrational amplitudes seem reasonable.

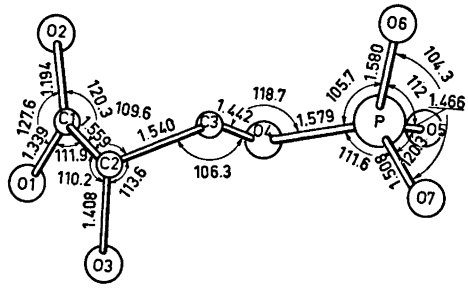

Fig. 2. Schematical drawing of the molecule showing interatomic distances and bond angles.

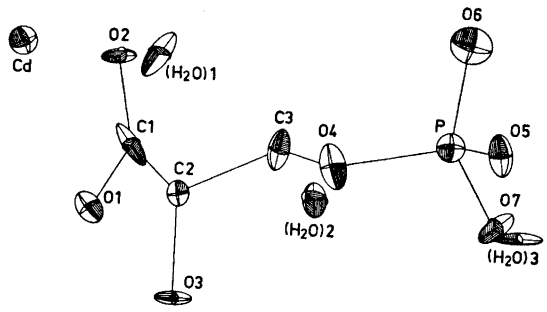

Fig. 3. Vibrational ellipsoids of the atoms in the $C_3H_6O_7PCd,3H_2O$ crystal structure.

Table 4. Temperature factors in the direction of the vibrational axes and the components of the vibrational amplitudes (in Å) along the crystallographic axes.

	3.08	0.1590	0.1041	−0.0467
Cd	2.70	−0.0823	0.1553	0.0533
	2.41	0.0689	−0.0221	0.1623
	3.55	0.0204	−0.2080	0.0376
P	2.60	0.1792	0.0127	−0.0187
	1.96	0.0214	0.0291	0.1545
	6.66	−0.0372	0.0031	0.2910
O1	4.67	0.1692	−0.1734	0.0314
	2.22	0.1189	0.1176	0.0195
	5.02	0.2213	0.0393	−0.1043
O2	3.41	0.0801	0.0713	0.1820
	0.88	0.0306	−0.0978	0.0269
	6.91	0.2640	0.0237	−0.1195
O3	3.90	0.1006	−0.0160	0.2024
	0.51	−0.0031	0.0801	0.0079
	8.38	0.1061	−0.3075	0.0236
O4	3.19	−0.0269	0.0027	0.1979
	2.32	0.1605	0.0565	0.0286
	6.80	−0.0726	0.2839	0.0134
O5	3.08	0.0847	0.0110	0.1823
	2.49	0.1544	0.0439	−0.0658
	6.14	−0.2702	0.0458	0.0396
O6	5.35	0.0570	0.2397	0.0865
	2.81	0.0228	−0.0666	0.1762
	6.45	0.1948	0.1818	−0.0947
O7	3.88	−0.0134	0.1206	0.1849
	2.14	0.1203	−0.0901	0.0731
	9.77	0.1633	0.2914	−0.1027
$(H_2O)1$	4.65	0.1365	−0.0001	0.2073
	1.96	−0.1080	0.0882	0.0681
	8.05	−0.1376	0.0840	0.2692
$(H_2O)2$	5.06	0.0513	−0.2290	0.0977
	2.86	0.1677	0.0641	0.0720
	9.61	0.3338	0.0327	−0.0811
$(H_2O)3$	4.69	0.0704	−0.0287	0.2351
	0.60	−0.0049	0.0860	0.0119
	10.76	0.2078	−0.2671	0.1577
C1	2.92	0.1117	0.0001	−0.1512
	1.07	0.0685	0.0802	0.0521
	5.07	0.0018	0.0107	−0.2531
C2	2.31	0.1642	0.0484	0.0111
	2.23	−0.0477	0.1612	0.0042
	6.57	0.0373	0.2844	0.0323
C3	2.99	0.1084	0.0032	−0.1564
	2.52	0.1467	−0.0296	0.1046

The crystal structure appears to be governed by the coordination around the cadmium ion. The hydrogen bonds between glycerophosphate molecules are mainly formed through bridging water molecules, whereas three of the glycerophosphate molecules are coordinated to the same cadmium ion. Each of the glycerophosphate molecules is in turn coordinated to three different cadmium ions through four of its oxygen atoms (O1, O2, O3, and O5). One of these (O1) also participates in coordination to two different cadmium ions.

The cadmium ion is coordinated to seven oxygen atoms which form a slightly distorted pentagonal bipyramid as illustrated in Fig. 4. The distances

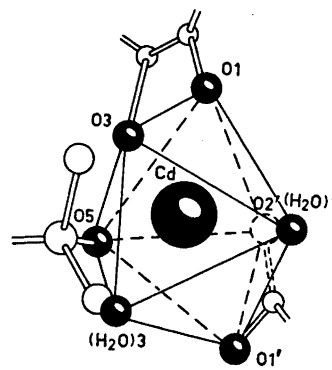

Fig. 4. The coordination of the cadmium ion.

between the cadmium ion and the oxygen atoms as well as those between the oxygen atoms are given in Table 5. Five of the oxygen atoms coordinated to cadmium are lying in a plane through cadmium. The deviations from a least squares plane through these atoms are less than 0.1 Å.

The mean Cd – O distance is 2.346 Å, which is in agreement with the sum of the van der Waals radii for the atoms (O: 1.40 Å, Cd: 0.97 Å). Since estimated standard deviations in the Cd-O distances are about 0.02 Å, there are three

Table 5. Distances in Å between oxygen atoms coordinated to cadmium, and between these oxygen atoms and cadmium.

$(H_2O)1-(H_2O)3$	3.28	Cd – O1	2.27
$(H_2O)1-O1$	3.29	Cd – O1′	2.64
$(H_2O)1-O1'$	3.41	Cd – O2′	2.38
$(H_2O)1-O2$	3.37	Cd – O3	2.38
$(H_2O)1-O3$	3.20	Cd – O3	2.38
$(H_2O)-O3$	2.98	Cd – O5	2.25
$(H_2O)3-O1$	3.32	Cd – $(H_2O)1$	2.26
$(H_2O)3-O5$	3.04	Cd – $(H_2O)3$	2.38
O1 – O5	3.23		
O1′ – O5	3.42		
O3 – O5	3.54		
O2 – O5	3.16		
O1 – O3	2.55		
O1 – O2′	2.99		
O1 – O2′	2.27		

Cd-O distances significantly shorter than the others, namely Cd – $(H_2O)1$, Cd – O1, and Cd – O5. This may be explained by the fact that $(H_2O)1$ and O5 represent the apices in the bi-pyramid, where the approach to cadmium is less hindered by neighbouring oxygens. Also O1 and O5 are probably the ionized acid groups in the glycerophosphate molecule as judged from the pK_s values of the three acid groups in this compound: these values are 3.42 for the carboxyl group, and 1.42 and 5.593 for the two phosphate acid groups.[6] A coordination very similar to this has been reported for the calcium ion in several compounds [8,10-12] with Ca – O distances of approximately 2.4 Å. A close packed five-membered ring of oxygen atoms with radius of 1.40 Å leaves room for a central sphere of radius 0.98 Å, corresponding closely to the ionic radii of cadmium (0.97 Å) and calcium (0.99 Å). We have, however, not been able to crystallize the calcium salt of phosphoglyceric acid. Both for calcium and cadmium also six-coordination has been reported.[7,10]

In the glyceric acid part of the molecule the characteristic feature is the planar configuration of the –C(OH)COO- group. The distances from a least squares plane through these atoms are given in Table 6. An approximately planar arrangement of the carboxyl group and one of the bonds in an adjacent sp^3 hybridized carbon atom have often been reported.[13,8,14,15]

Table 6. Distances from least squares plane through the $-C(OH)COO^-$ group.

Atom	O1	O2	C1	C2	O3	Cd
Distances in Å	0.01	0.001	0.05	0.01	0.003	0.5

As the carboxyl group is believed to be ionized, one should expect the two C – O distances to be equal. In the present study these distances are not significantly different,[16] but a difference in the two distances would not be surprising when it is considered that O1 is involved in coordination to two cadmium ions.

Table 7. Hydrogen bond distances in Å and some of the angles. Standard deviations in parenthesis.

$(H_2O)1 - O7$	2.72 (4)
$(H_2O)1 - (H_2O)2$	2.88 (3)
$(H_2O)2 - O5$	2.77 (2)
$(H_2O)2 - O6$	2.60 (3)
$(H_2O)3 - O7$	2.70 (3)
$(H_2O)3 - O2$	2.72 (3)
$(H_2O)3 - O3$	2.98 (3)
O6 – O3	2.77 (3)
$O7 - (H_2O)1 - (H_2O)2$	115°
$O5 - (H_2O)2 - O6$	144°
$O7 - (H_2O)3 - O2$	116°
$C2 - O3 - (H_2O)3$	133°
P – O6 – O3	125°

The structure of the phosphate group is in accordance with earlier structure determinations. The conformation of the group is such as to give maximum overlap of $3p-3d$-orbitals as described by Collin,[9] and the bond lengths reported here are in agreement with those found in other phosphate groups.[17]

The hydrogen bonds are illustrated by broken lines in Fig. 1, where crosses indicate the assumed hydrogen positions. The oxygen – oxygen distances are listed in Table 7. Except for the distances between oxygens coordinated to cadmium the only oxygen – oxygen distance shorter than 3 Å, and not included in the hydrogen bond system, is the $(H_2O)_2-O4$ of 2.963 Å.

Molecules related by the screw axis are linked together by the O6 – H···O3 bonds, which seem to be the only "direct" hydrogen bonds between adjacent glycerophosphate molecules. All other hydrogen bonds between these molecules are bridged by water molecules which generate a hydrogen bond system in the general direction of the *b*- and *c*-axes. In the direction of the *a*-axis the crystal seems to be held together mainly by the coordination forces to cadmium.

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