Acta Cryst. (1963). **16**, 264

The Crystal Structure of Myo-Inositol Dihydrate

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(*Received* 14 *May* 1962)

A complete structure determination has been made of myo-inositol dihydrate, which grows in monoclinic crystals of space group $P2_1/a$ with

$$a = 9{\cdot}144,\ b = 16{\cdot}814,\ c = 6{\cdot}531\ \text{Å};\ \beta = 111^\circ\ 49'.$$

Cell contents: four molecules of $C_6H_6(OH)_6.2H_2O$, density 1·54 g.cm.$^{-3}$. The molecule is of the expected chair form, with one axial hydroxyl group and five equatorial ones. Twelve out of the sixteen hydrogen atoms were located by a three-dimensional Fourier synthesis. The structure forms a complete system of hydrogen bonds. Each water molecule possesses four bonds in an approximately tetrahedral arrangement, the two water molecules joined together by a hydrogen bond, forming a dimer. The water molecules lie in channels parallel to the *c* axis.

Introduction

Inositol compounds are widely distributed in nature both as the free base and as phosphoric esters. They have been isolated from such diverse sources as ox-brain, egg-yolk, yeast and the berries of mistletoe. Angyal (1957) says of myo-inositol that no living tissue, plant or animal, which has been examined by an adequate method, has been found to lack it. In conjunction with biotin and vitamin B_1 myo-inositol seems to be a growth-regulating factor in certain plants and some of the phosphates appear to play important parts in human physiological and pathological processes. Many of the chemical and physical properties of the several isomers of inositol have been described by Fleury & Balatre (1947), and Angyal (1957) has discussed the reactions of inositols and related compounds from a stereochemical point of view. Law (1960) has reviewed the stereochemistry of some inositol phosphates. Myo-inositol was first isolated by Scherer (1850) and its chemical structure was worked out by Pasternak (1942). The results of an X-ray determination of the crystal structure as reported below confirm Pasternak's conclusions. The molecular conformation is also established as the chair form with one –OH group axial and five equatorial. When recrystallized from water at temperatures above 50 °C., crystals of the anhydrous compound are obtained, but at lower temperatures a hydrated compound $C_6H_{12}O_6.2H_2O$ results. This dihydrate is stable at room temperatures in atmospheres of high humidity but loses both molecules of water spontaneously in normal room conditions. The work described below was carried out on the hydrated compound.

Space-group and unit-cell dimensions

Rotation and Weissenberg photographs were taken about the three axes. A thin coating of vaseline on the crystal surface was found to delay the dehydration for about one week. The unit-cell dimensions were obtained from high-order reflexions. The crystal is monoclinic with:

$$a = 9{\cdot}144 \pm 0{\cdot}005,\ b = 16{\cdot}814 \pm 0{\cdot}005\ ,$$
$$c = 6{\cdot}531 \pm 0{\cdot}004\ \text{Å};\ \beta = 111^\circ\ 49' \pm 5'.$$

The space group is $P2_1/a$ in agreement with that reported by White (1931) and the substance has a density of 1·54 ± 0·01 g.cm.$^{-3}$, so that there are four molecules of $C_6H_6(OH)_6.2H_2O$ in the unit cell. The calculated density is also 1·54 g.cm.$^{-3}$.

The intensities were visually estimated using the triplefilm-overlay Weissenberg method. In addition to the three zero layer-lines, three upper layer-lines were measured on oscillation photographs taken about the *c*-axis. The three Patterson projections were calculated as well as a three-dimensional Patterson function with about 60% of the total number of reflexions possible.

Interpretation of the Patterson maps

The first clue to the interpretation of the Patterson projections came from the *b*-axis projection. This map has a marked symmetry about lines perpendicular to the *a*-axis which pass through points on that axis at $x = 0$, 1/8, 1/4 etc. These lines are shown as dotted lines in Fig. 1(*a*). This symmetry is also very obvious in the zero layer-line Weissenberg photograph about

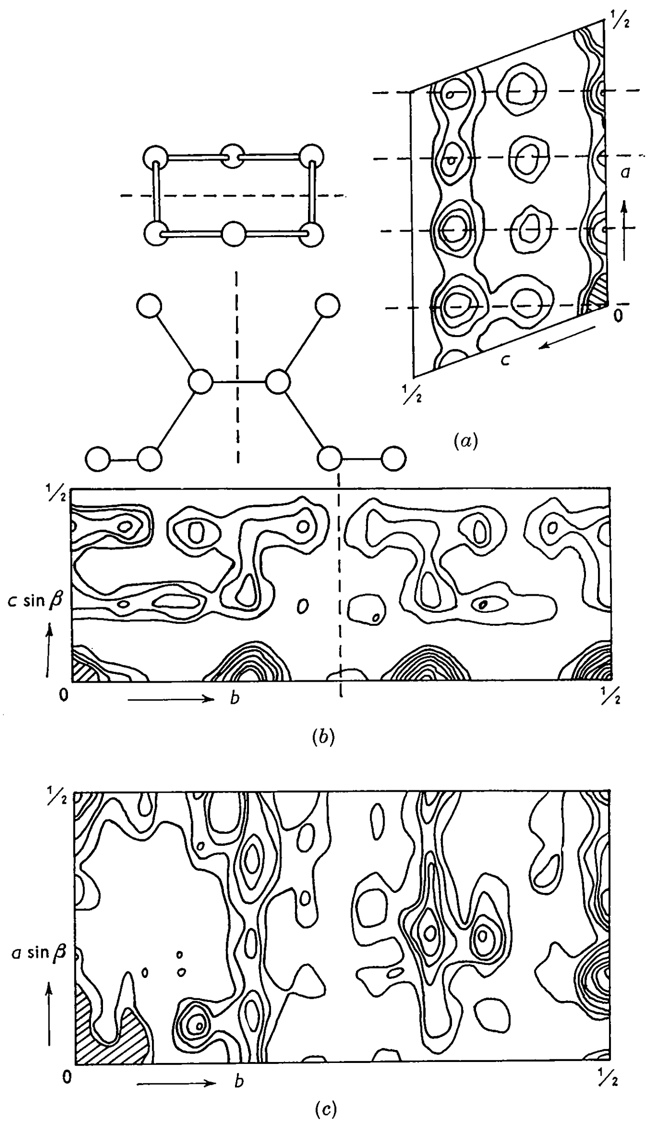

Fig. 1. (*a*) Patterson projection down the *b*-axis, and projection of carbon ring in a suitable orientation to fit the Patterson peaks (mirror symmetry lines dotted). (*b*) *a*-axis Patterson projection, and orientation of the ring. (*c*) *c*-axis Patterson projection.

the *b*-axis, in which the line of spots with $h=4l$ forms an almost perfect symmetry line. Indeed it is only too easy to take this line as an axial line in the photograph, but a careful inspection of the intensities reveals the lack of perfect symmetry, and this also shows in the positions of the very high order reflexions, a fact which underlines the importance of the use of crystals large enough to give these orders. Furthermore the arrangement of the peaks in the *b*-axis Patterson projection strongly suggests that the ring has a rectangular appearance (Fig. 1(*a*), left) in this projection.

Both the *a*- and the *c*-axis projections show peaks which agree with this orientation of the ring. Fig. 1(*b*) shows the *a*-axis Patterson projection, together with a diagram of the molecule (half of the ring only) in the appropriate orientation. It is clear that there is a good general fit of the ring vectors, and also that the extra pseudo-symmetry plane in this projection (again shown dotted) is accounted for. In addition to this the *a*-axis Patterson projection shows large peaks at $y=1/6$, $1/3$ and $1/2$ and $z=0$. These are the centre-to-centre vectors between one molecule and its equivalent, firstly across the glide plane (vector at $y=1/6$), secondly across the centre of symmetry (vector at $y=1/3$) and finally between molecules

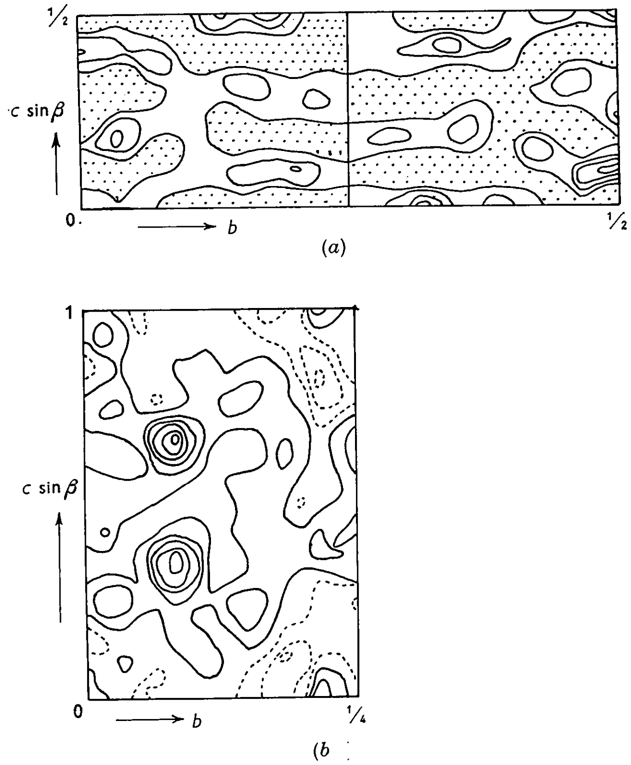

Fig. 2. (*a*) *a*-axis Patterson projection using only intensities with *k* odd. (*b*) *a*-axis difference map, showing water positions.

Table 1. *List of parameters*

Atom	x/a	y/b	z/c	Atom	x/a	y/b	z/c
C_1	0·319	0·417	$0{\cdot}\overline{009}$	H_1*	0·35	0·47	$0{\cdot}\overline{02}$
C_2	0·262	0·362	$0{\cdot}\overline{210}$	H_2	0·18	0·32	$0{\cdot}\overline{21}$
C_3	0·395	0·307	$0{\cdot}\overline{192}$	H_3	0·49	0·35	$0{\cdot}\overline{20}$
C_4	0·450	0·256	0·011	H_4*	0·48	0·18	0·02
C_5	0·499	0·311	0·197	H_5	0·59	0·35	0·21
C_6	0·373	0·369	0·200	H_6	0·29	0·32	0·21
O_1	0·192	0·466	$0{\cdot}\overline{020}$	H_7	0·27	0·00	$0{\cdot}\overline{10}$
O_2	0·226	0·412	$0{\cdot}\overline{406}$	H_8	0·08	0·42	0·50
O_3	0·346	0·253	$0{\cdot}\overline{385}$	H_9	0·20	0·25	0·50
O_4	0·323	0·207	0·015	H_{10}	0·35	0·15	0·15
O_5	0·545	0·266	0·402	H_{11}	0·48	0·25	$0{\cdot}\overline{42}$
O_6	0·436	0·424	0·381	H_{12}	0·35	0·42	0·50
O_7	0·410	0·086	0·357	H_{13}	0·34	0·02	0·34
O_8	0·255	0·080	$0{\cdot}\overline{321}$	H_{14}	0·35	0·08	0·50
				H_{15}*	0·25	0·11	$0{\cdot}\overline{12}$
				H_{16}*	0·13	0·08	0·50

Table 2. *Calculated and observed structure factors*

Each (*hkl*) block is headed by its common *h* and *l* indices, and the three columns give the values of *k*, $10F_c$ and $10|F_o|$. For unobserved intensities the minimum observable $10F_o$ is given. Intensities affected by extinction are indicated by an asterisk

(0 k 0)		
2	231-	214
4	42	49
6	629-	*462
8	86-	99
10	49	39
12	176	202
14	43	49
16	25	49
18	255-	252
20	25-	57
(1 k 0)		
1	109	96
2	277	257
3	242-	222
4	152	144
5	106	113
6	97-	109
7	92-	89
8	144	166
10	70	69
11	47-	41
12	17	18
13	58	53
15	24	36
16	0	18
17	93	81
18	19	18
19	99-	83
20	89-	91
21	44	39
(2 k 0)		
0	187-	234
1	482	*381
2	299-	275
3	34	53
4	188	190
5	79	120
6	35	39
7	167-	180
8	23	18
9	152-	154
10	41-	63
11	68-	69
12	32	33
13	3-	18
14	63	85
15	27	18
16	91	85
17	133	148
18	63	47
19	53-	57
20	7	18
21	13	18
(3 k 0)		
1	142	164
2	26-	18
3	13-	18
4	59-	41
5	189	200
6	61	124
7	171-	186
8	7-	33
9	21	18
10	80-	69
11	7-	20
13	33-	47
14	86-	90
15	38-	59
16	37	33
17	28-	18
18	50	39
19	3	19
20	41	39
(4 k 0)		
0	75	49
1	70-	91
2	75	41
3	314	275
4	34-	49
5	51	63
6	30-	18
7	66	77
8	48-	33
9	52-	47
10	12	18
11	16-	18
12	59	49
13	66	71
14	29	47
15	3	18
16	26-	18
17	10	27
18	16-	27
19	21-	33
20	28-	27
(5 k 0)		
1	73-	71
2	8	18
3	16-	18
4	31-	39
5	75-	79
6	17-	18
7	10	33
8	9	18
9	8-	18
10	30-	41
11	56	57
12	12	18
13	64	71
14	38-	18
15	19-	18
16	49	38
17	32	18
18	35-	33
19	12-	27
(6 k 0)		
0	53-	45
1	75-	67
2	133-	127
3	10	18
4	170	166
5	34-	18
6	103	99
7	6	19
8	32	27
9	61-	49
10	24-	33
11	54-	63
12	91-	79
13	27-	41
14	83-	77
15	27	41
16	17	18
17	11	18
18	27	27
(7 k 0)		
1	54	49
2	10	18
3	15-	45
4	201	186
5	20-	18
6	0	18
7	5-	18
8	1-	18
9	9	18
10	95-	81
11	37	41
13	44-	39
14	86	71
15	6-	18
16	1-	19
(8 k 0)		
1	9	19
2	11-	18
3	37-	33
4	102-	105
5	42	18
6	87	79
7	34-	33
8	6	18
9	36	18
10	21	18
11	11-	19
12	32-	33
13	34	39
14	14	39
(9 k 0)		
1	33-	18
2	2-	18
3	65-	81
4	50-	53
5	98	75
6	60-	81
7	67	57
8	4	18
9	16-	27
10	34	39
11	13-	27
(10 k 0)		
1	43-	59
2	46	27
3	14-	45
4	36-	33
5	63-	77
6	97	83
7	39	45
8	87-	97
(0 k 1)		
0	24	27
1	8	18
2	67-	59
3	5-	18
4	118	115
5	14	27
6	360	299
7	23-	18
8	52-	49
9	31	37
10	22	18
11	23-	18
12	244-	224
13	28	18
14	35-	18
15	1	18
16	3-	18
17	17	27
18	1-	18
19	50-	47
20	32	18
21	19	19
(1 k 1)		
1	41-	47
2	11-	18
3	45	53
4	197	208
5	174-	178
6	9	18
7	48	53
8	116	105
9	59-	57
10	1-	18
11	97	79
12	44	37
13	3-	18
14	38	49
15	44	41
16	6-	18
17	9	18
18	25-	18
19	5-	18
20	39-	37
21	31-	27
(2 k 1)		
0	72-	53
1	217-	198
2	175	166
3	343-	303
4	276-	271
5	214-	198
6	85-	105
7	84	75
8	8-	18
9	18	19
10	45	37
11	154	128
12	83	67
13	93-	93
14	25	18
15	44-	37
16	10	18
17	76-	69
18	17-	18
19	37	19
20	12-	19
(3 k 1)		
1	6-	19
2	6-	19
3	3-	18
4	5-	18
5	154	158
6	107-	67
7	158	140
8	56-	47
9	11-	38
10	67	79
11	148-	144
12	2-	18
13	120-	99
14	31	18
15	41	37
16	28-	47
17	20-	18
18	19-	27
19	43	27
(4 k 1)		
0	5	18
1	17	18
2	26	37
3	63	103
4	8	18
5	51	53
6	63-	69
7	85-	79
8	39	47
9	257	240
10	79-	57
11	55-	57
12	71	79
13	20	18
14	11	18
15	15-	18
16	35	31
17	2-	18
18	28-	19
19	0	31
(5 k 1)		
1	11-	18
2	90-	87
3	63	65
4	105-	103
5	118-	123
6	22-	41
7	92-	79
8	24	18
9	73-	83
10	146	150
11	137	130
12	113-	115
13	74	79
14	0	18
15	18	18
16	45-	31
17	7	18
(6 k 1)		
0	169	132
1	63	105
2	113	77
3	115-	91
4	34-	65
5	142	150
6	84-	57
7	182-	174
8	184-	212
9	49	37
10	128	122
11	97-	83
12	40	27
13	79	67
14	40	37
15	64-	49
16	3	18
17	26-	31
(7 k 1)		
1	13	18
2	32-	18
3	8-	18
4	109	128
5	38	41
6	32	37
7	21	7
8	3-	18
9	37-	18
10	11-	18
11	4	18
12	47-	37
13	45-	59
14	95	95
15	40	41
(8 k 1)		
0	92-	57
1	55	93
2	13-	18
3	34	18
4	62-	71
5	76	63
6	102	117
7	39-	31
8	15-	18
9	43-	27
10	9	18
11	43-	37
12	51-	75
13	4-	18
(9 k 1)		
1	79	73
2	4-	31
3	31-	41
4	31-	27
5	11-	31
6	3	18
7	75-	57
8	29-	31
9	11	18
10	10	31
(10 k 1)		
0	43	27
1	11-	37
($\bar{1}$ k 1)		
1	33	109
2	222-	164
3	160-	115
4	162-	123
5	31-	49
6	26-	27
7	26-	27
8	125-	127
9	13	18
10	58-	47
11	80	79
12	50	47
13	42-	47
14	22-	18
15	7-	41
16	26	18
17	37-	47
18	80-	73
19	1-	37
20	63	59
21	67	47
($\bar{2}$ k 1)		
0	282-	204
1	455-	*333
2	265-	273
3	43-	47
4	177	140
5	45-	47
6	13	18
7	144	132
8	78	65
9	165	164
10	42-	49
11	27	18
12	81	77
13	23	31
14	104-	91
15	76-	69
16	85	75
17	81-	69
18	1	18
19	33	31
20	75	53
21	40	19
($\bar{3}$ k 1)		
1	2-	18
2	87-	69
3	4	18
4	222	164
5	316	333
6	112-	140
7	143-	144
8	171	200
9	9	27
10	14-	31
11	3	18
12	12-	18
13	92-	75
14	40-	18
15	41-	37
16	46	49
17	14-	18
18	19-	18
19	32	49
20	47	37
($\bar{4}$ k 1)		
0	132-	89
1	14	18
2	120-	144
4	128	117
5	146-	166
6	126	115
7	117-	138
8	72	65
9	318-	321
10	36-	18
11	19	18
12	51	37
13	101-	109
14	73-	69
15	234	202
16	19	18
17	28	18
18	28	18
20	26	19
($\bar{5}$ k 1)		
1	42-	57
2	42	37
3	82-	67
4	163	182
5	113-	162
6	150-	154
7	94	99
8	106	122
9	26	18
10	8-	18
11	3-	18
12	27-	18
13	47	18
14	23-	18
15	10	18
16	31	65
17	24	37
18	17-	37
19	21-	27
($\bar{5}$ k 1)		
0	195	220
1	164-	234
2	89	87
3	96	103
4	95-	131
5	20-	18
6	57-	65
7	69	87
8	67-	77
9	43	59
10	61	75
11	11	18
12	23-	49
13	17-	49
14	82	85
15	1	18
16	56-	57
17	35-	37
18	32-	27
($\bar{7}$ k 1)		
1	3-	18
2	3-	18
3	40	41
4	1	18
5	39	49
6	19	18
7	14	18
8	63-	59
9	7-	18
10	41-	49
11	61-	53
12	15-	37
13	2-	18
14	6-	18
15	9-	18
16	47	31
17	30	19
($\bar{8}$ k 1)		
1	12	18
2	10	18
3	57	57
4	15-	18
5	29	37
6	56-	47
7	36-	37
8	36	41
9	34-	31
10	3-	27
11	51-	47
12	49	49
13	17	18
15	35	27
($\bar{9}$ k 1)		
1	19	41
2	11	37
3	39-	47
4	52	49
5	40	18
6	16	18
7	36-	18
8	18	18
($\overline{10}$ k 1)		
0	88	67
1	39-	31
2	9	47
3	54-	53
4	27	31
5	13-	27
6	28-	31
7	33	47
8	57-	27
(0 k 2)		
1	33-	18
2	17	18
3	68	75
4	299	244
5	42	47
6	163-	210
7	56	53
8	25	33
9	40-	43
10	17-	18
11	12	18
12	15	27
13	29-	18
14	59-	79
15	54	43
16	84-	97
17	25-	18
18	3	18
19	29	18
20	28	39
(1 k 2)		
1	71-	85
2	28	39
3	76	92
4	310	297
5	184-	156
6	134	109
7	23-	19
8	76-	67
9	94	91
10	89-	85
11	1	18
12	33-	18
13	128	118
14	147	134
15	116-	107
16	33-	53
17	42	39
18	21	18
(2 k 2)		
0	83-	79
1	197	162
2	242-	208
3	31	18
4	140	138
5	246	234
6	41-	27
7	221-	180
8	313	330
9	175-	174
11	136-	178
12	15	18
13	65	69
14	33-	33
15	97	93
16	83	72
17	23	18
18	23	27
(3 k 2)		
1	92	63
2	59-	39
3	34-	33
4	16-	39
5	68	59
6	50-	39
7	14-	27
8	19	33
9	93	83
10	94	85
11	151-	154
12	104-	97
13	20	18
14	32	33
15	31-	39
16	44-	69
(4 k 2)		
0	127-	113
1	56-	47
2	62-	59
3	112-	83
4	42	120
5	16	27
6	43	47
7	30-	39
8	104	91
9	315	281
10	19-	20
11	29-	43
12	43-	43
13	3	18
14	44-	27
16	8-	18
17	18-	18
(5 k 2)		
1	87-	69
2	69	75
3	21-	18
4	63	52
5	6-	18
6	40-	57
7	15-	18
8	107-	83
9	48	30
10	27	18
11	11	18
12	15-	18
13	25	18
14	44	47
15	34-	33
16	33	27
(6 k 2)		
0	109	18
1	54-	93
2	28-	18
3	5	18
4	53	50
5	50-	57
6	4-	18
7	8-	18
8	25-	27
9	21-	18
10	12-	18
11	13	18
12	42-	47
13	23-	18
14	22	27
(7 k 2)		
1	22-	18
2	27-	47
3	11	18
4	8	18
5	50-	47
6	35-	49
7	9	18
8	92	75
9	2	18
10	42	39
11	40-	39
12	27	19
(8 k 2)		
0	46-	18
1	3	18
2	26-	45
3	49-	45
4	26	57
5	43-	27
6	63-	45
7	28	49
8	31	33
9	17	27
(9 k 2)		
1	37-	18
2	47-	49
3	35	43
4	33-	39
5	31	19
($\bar{1}$ k 2)		
1	58-	60
2	63-	59
4	323	355
5	51-	75
6	34	33
7	37-	43
8	34-	39
9	45-	47
10	96	75
11	12-	39
12	102-	79
13	89	91
14	94	69
15	1-	27
16	20	18
17	8	27
18	32	18
19	6-	18
20	42	39
($\bar{2}$ k 2)		
0	117-	60
1	264	186
2	203	174
3	230	234
4	253-	234
5	180	172
6	114-	129
7	120-	136
8	55-	59
9	50-	53
10	37	18
11	86-	67
12	108	91
13	53	39
14	94	91
15	55	18
16	1	18
17	41	18
18	13	18
($\bar{3}$ k 2)		
1	42	75
2	80	75
3	3	18
4	119	130
5	137	128
6	44	47
7	7-	18
8	20-	33
9	13	18
10	33	39
11	44-	53
12	22-	18
13	90-	79
14	51	47
15	31	18
16	22-	18
17	37-	18
19	42	27
($\bar{4}$ k 2)		
0	102	97
1	100	79
3	287-	270
4	7-	18
5	65-	67
6	82-	71
7	65-	59
8	26-	18
9	74	83
10	0	18
11	18	18
12	33	39
13	78-	71
14	12-	18
15	20-	18
16	21	47
17	5-	18
18	36-	19
19	39	27
($\bar{5}$ k 2)		
1	24-	18
2	25	33
3	6	18
4	112	117
5	156-	194
6	109-	93
7	41	43
8	11	18
10	23	18
13	53	47
14	57	47
15	87	69
($\bar{6}$ k 2)		
0	196	204
1	147-	136
3	71	91
4	117-	101
5	6-	18
6	67-	57
7	72	73
8	43-	27
9	43	49
10	23-	18
11	30	18
12	1	18
13	5	18
14	72	71
15	35	45
16	82-	63
($\bar{7}$ k 2)		
1	31	59
2	123-	111
3	73	63
4	33-	67
5	70-	77
6	12	18
7	36	49
8	29-	18
9	23-	18
10	8-	18
11	3	18
12	17	18
13	19-	18
14	3	18
15	13-	18
16	12-	39
($\bar{8}$ k 2)		
0	356-	373
1	141-	95
2	85	95
3	79-	83
4	31-	47
5	76-	57
6	128	140
7	43	33
8	14-	27
9	68	75
10	1-	18
11	37	39
12	48-	18
($\bar{9}$ k 2)		
1	30-	25
2	21	18
3	49	73
4	37	39
5	24-	49
6	41-	39
7	20	18
8	46	43
9	19-	33
10	28	43
11	7	33
($\overline{10}$ k 2)		
0	41-	83
1	63	47
2	69	47
3	31	39
4	25-	33
5	12	27
6	33	57
7	52-	18
8	1	27
9	30-	33
10	8	17
(0 k 3)		
0	375	359
1	62-	47
2	98	75
3	8	29
4	126	120
5	10-	18
6	348-	264
7	7-	18
8	50-	55
9	36	43
10	7-	18
11	27	18
12	157	107
13	42	55
14	15	18
15	37-	47
16	74-	65
17	9-	18
18	65-	37
(1 k 3)		
1	151-	131
2	115-	131
3	68	59
4	69	55
5	19	18
6	37-	18
7	80	63
8	119	107
9	47-	53
10	13	18
11	66-	53
12	50	47
13	64	47
14	1	18
15	13	18
16	48-	47
17	67	43
(2 k 3)		
0	75-	55
1	5	18
2	74	55
3	10-	29
4	87-	75
5	35-	47
6	5-	18
7	17-	18
8	12-	18
9	66-	53
10	7	18
11	36-	29
12	27	43
13	24-	18
14	33	18
15	28	18
17	21	23
(3 k 3)		
1	113	87
2	23-	83
3	38	18
4	32	29
5	9	18
6	9-	18
7	91-	75
8	40	47
9	10-	18
10	55-	47
11	26	18
12	4	18
13	29-	18
14	10-	18
15	14	23
(4 k 3)		
0	18	18
1	57-	37
2	47	53
3	149	129
4	44-	37
5	39	18
6	45-	47
7	58	55
8	37-	18
9	74-	65
10	3	18
11	4	18
12	39	37
13	59	53
14	20	29
(5 k 3)		
1	19	18
2	45-	50
3	37-	63
4	95-	79
5	84-	75
6	51	65
7	36	45
8	3-	18
(6 k 3)		
1	125	95
6	33	18
7	38	23
8	39	37
9	26-	29
(7 k 3)		
1	5	23
2	151	101
3	55	43
6	10	37
7	1	18
($\bar{1}$ k 3)		
1	5	37
2	62	124
3	137	154
4	132-	117
5	119-	113
6	83-	63
7	84-	63
8	41-	47
9	43	43
10	35-	18
11	11-	18
12	35	18
13	121	85
14	93-	65
15	93-	71
16	76	59
18	22-	37
($\bar{2}$ k 3)		
0	209-	178
1	159-	222
2	133-	212
3	76-	47
4	56	71
5	302-	262
6	22-	23
7	232	174
8	248	210
9	123	105
10	172-	158
11	155	117
13	125-	107
15	74-	75
16	58	71
18	53	23
($\bar{3}$ k 3)		
1	11	18
2	11	18
3	60-	37
4	11	18
5	89	117
6	1-	18
7	96	59
8	57	55
9	76	71
10	13-	18
11	194-	192
12	135	111
13	29-	18
14	30-	37
15	1	18
16	1	18
18	46-	23
($\bar{4}$ k 3)		
0	23	18
1	31	23
2	14	23
3	104-	53
4	9	23
5	30-	53
6	1-	18
7	48	63
8	44	43
9	204-	208
10	1-	18
11	83	75
12	58-	55
13	8-	18
14	16	18
15	25	37
($\bar{5}$ k 3)		
1	19-	43
5	104-	85
6	35	43
7	33-	53
9	53	47
10	73-	75
11	74	75
13	64	55
15	50-	37
($\bar{6}$ k 3)		
1	52	55
2	132-	131
4	135	162
5	65	43
6	69	75
7	27	23
8	39	37
9	45	55
12	86-	75
13	40	55
($\bar{7}$ k 3)		
4	94-	87
5	47	75
10	32	37
11	44-	59
($\bar{8}$ k 3)		
0	57-	37
2	15-	75
3	42	65
6	49-	65
7	28-	55
8	30	47
9	52-	55
11	26-	65
12	63	75
($\bar{9}$ k 3)		
1	66-	47

related by the screw axis (vector at $y=1/2$). Thus the centre of the molecule lies in the plane $z=0$, and with a y parameter of very nearly 1/6. The x parameter of the ring centre can be obtained by inspection of the c-axis Patterson projection, since the largest peaks at $y=1/3$ on this map (Fig. 1(*c*)) lie at $x=\pm 0{\cdot}235$. The value $-0{\cdot}235$ is twice the x parameter of the ring centre.

By these considerations a good approximation to the structure can be reached, but this has a higher symmetry than that of the true molecule, whose axial hydroxyl group destroys the symmetry plane perpendicular to the a-axis. Also the centre of the molecule may have a small shift from the position $y=1/6$, $z=0$, and its orientation may not be exactly perpendicular to a. There may also be a small rotation about the c-axis direction. These points were settled by the use of a Patterson projection down the a-axis using only the $0kl$ reflexions with k odd. This map is shown in Fig. 2(*a*), and instead of a mirror-plane at $y=1/4$ it has a reversal mirror-plane in this position, showing a symmetry discussed by Cochran (1952). The map shows that the centre-to-centre peak at $y=1/3$, $z=0$ is still on the b-axis, whilst the Harker peak at $y=1/2$ is moved off the b-axis. This indicates strongly that the deviation from perfect symmetry takes the form of a small rotation of the molecule about its centre, the centre remaining on the b-axis.

The water-molecule positions were obtained from difference maps, of which the a-axis projection is shown in Fig. 2(*b*). The water molecules come out extremely clearly.

Fourier and least-squares refinements were carried out on the carbon and oxygen atom positions using the three-dimensional data on the DEUCE computer at the University of Glasgow (programmes devised by Rollett (1961). A three-dimensional difference synthesis was computed, and from it twelve of the hydrogen atoms appeared, in positions predicted on the assumption of normal bond-lengths. The remaining four hydrogen atoms (shown by an asterisk in Table 1) did not show up in the difference maps. Their parameters are merely suggested positions. Projections down the a and c axes were also refined by the use of Curtis's (1959) programme, and gave R indexes of 15 and 16% respectively, neglecting the H atoms. The final list of parameters is shown in Table 1. The R index from the three-dimensional data is 19·7%, the complete results being shown in Table 2.

Description of the structure

The six-membered carbon ring of the inositol molecule in this structure is of the 'chair' form, and all the –OH groups are equatorial except that on C_4, which is axial. Pasternak's structure is thus fully confirmed.

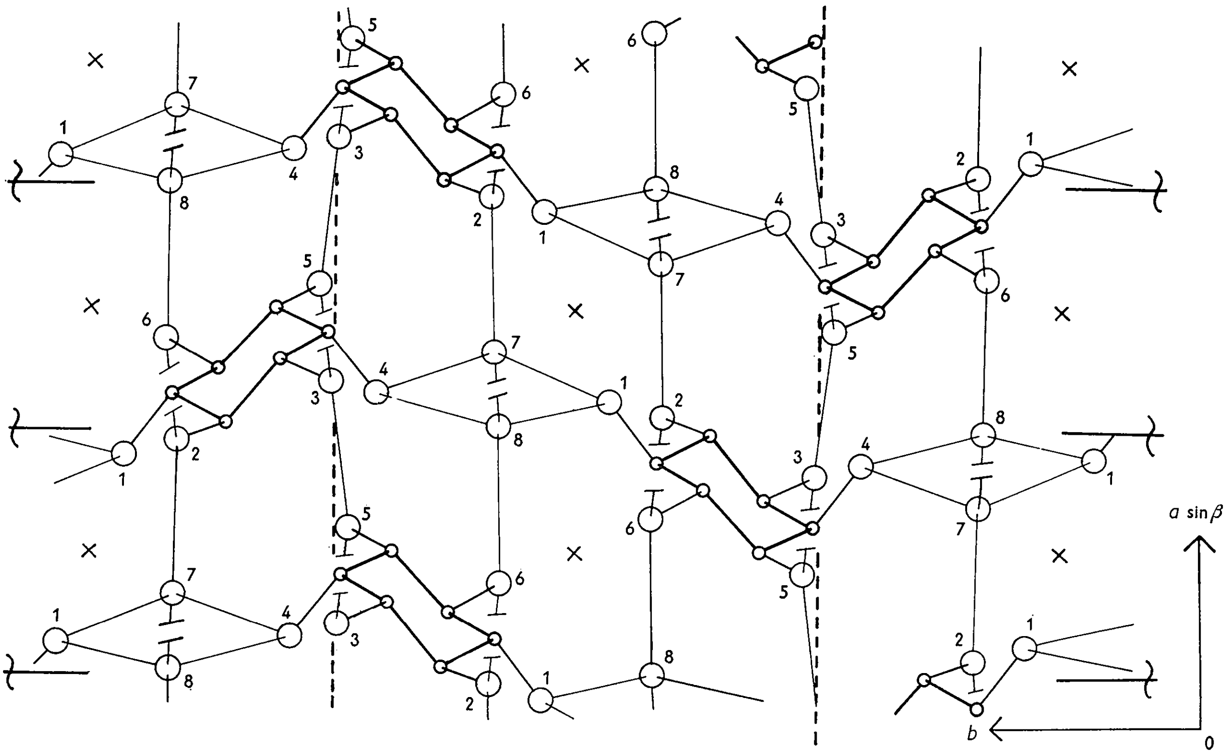

Fig. 3. A projection of the complete structure down the c-axis. The carbon atoms are shown as small circles, and the OH groups as larger circles. A bond shown terminated by a cross line is going to an atom in front of or behind the atom shown in the figure.

The following are the interatomic distances within the molecule:

C_1–C_2 = 1·53 Å C_1–O_1 = 1·40 Å
C_2–C_3 = 1·50 C_2–O_2 = 1·46
C_3–C_4 = 1·49 C_3–O_3 = 1·47
C_4–C_5 = 1·45 C_4–O_4 = 1·44
C_5–C_6 = 1·52 C_5–O_5 = 1·45
C_6–C_1 = 1·49 C_6–O_6 = 1·43

The averages of these distances are: C–C = 1·50 Å and C–O = 1·44 Å to the nearest 0·01 Å, and the standard deviations of these bond lengths worked out by the least-squares formula:

$$\sigma(x/a) = \Sigma w(F_o-F_c)^2/(n-s)\Sigma w[\partial\Delta F/\partial(x/a)]^2$$

came to an average of 0·02 Å. Thus there seems to be a slight but definite shortening of the C–C bonds in the molecule from the single bond value of 1·54 Å. The angles between the bonds to the individual carbon atoms are however quite close to the tetrahedral angle, the minimum of these angles being 105° and the maximum 120°.

The structure is best displayed by a view down the shortest axis—the *c*-axis of length 6·53 Å. In this projection the cell appears with the dimensions $a \sin\beta = 8{\cdot}489$ and $b = 16{\cdot}81$ Å. In the *c* direction perpendicular to the paper (Fig. 3) the molecules are bonded to each other through contacts O_3–O_5 = 2·68 and O_2–O_6 = 2·77 Å, thus forming continuous chains along the *c*-axis. In the figure these bonds are shown as lines which are broken where they go to atoms in molecules above or below the actual molecule shown. It is these contacts which cause the mean plane of the molecule to tilt some 22° ($=\beta-90°$) from the *c*-axis and to lie almost perpendicular to the *a*-axis.

The oxygen atoms 3 and 5 of the molecules in these chains are very close to the *a* glide planes (shown by dashed lines in Fig. 3) and bonds O_3–O_5 (of length 2·605 Å) are formed across this glide plane. These bonds not only link together the chains related to each other by the glide planes, but also, of course, hold together successive chains with the *a*-axis separation. This double series of molecular chains is operated on by either the centres of symmetry or by the screw axes parallel to *b* to give a parallel double series which is bonded to the first by the water-molecules 7 and 8. These molecules are in pairs in the structure with an O_7–O_8 distance of 2·94 Å, and each water molecule makes three additional bonds, thus giving a tetrahedral disposition of the bonds from each molecule. The angles between the bonds vary, however, from 89° to 114° for O_8 and from 89° to 125° for O_7. The bond-distances are quite close to the accepted O–O distances, being as follows:

O_7–O_1 = 2·88 Å O_8–O_1 = 2·84 Å
O_7–O_4 = 2·90 O_8–O_4 = 2·96
O_7–O_2 = 2·73 O_8–O_6 = 2·84
O_7–O_8 = 2·94

It is interesting that the bonds from O_7 and O_8 to these other oxygens are staggered with respect to each other just as are the bonds from C–H in a molecule such as ethane C_2H_6. The bonds to these water molecules link the structure together in all directions. Altogether there are ten unique O–O distances of lengths between 2·60 and 2·96 Å (the mean of the ten distances being 2·80 Å) and these distances correspond to the bonds formed by the ten hydrogen atoms of the hydroxyl groups and water molecules. As can be seen from Fig. 3 the water molecules 7 and 8 have a rather easy 'escape path' parallel to the *c*-axis and this probably accounts for the high water-vapour pressure of the crystal.

One of us (T. R. L.) is indebted to I.C.I. Ltd. for financial assistance in the purchase of computing time, and to Dr A. R. Curtis for the computer programme. A. M. was in receipt of a D.S.I.R. Studentship during the years 1958–60.

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