bonded S···H contacts of 2·91 Å, in agreement with the smaller van der Waals radius.

There is no simple explanation for the differences in the observed lengths of P–P and P–C bonds in molecules I and II.

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The Crystal Structure of Glucitol–Pyridine

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The crystal structure of a 1:1 glucitol–pyridine complex, $C_6H_{14}O_6.C_5H_5N$, has been determined by the application of the symbolic addition and tangent formulas to diffractometer Cu $K\alpha$ data. The anisotropic refinement terminated at $R=0·04$. The space group is $P2_1$ with two molecules in a unit cell of dimensions $a=4·652$ (3), $b=10·207$ (4), $c=13·439$ (5) Å, $\beta=90·3$ (3)°. The structure consists of layers of hydrogen-bonded glucitol molecules separated by layers of pyridine molecules stacked in a herringbone arrangement. The two layers are linked by O–H···N hydrogen bonds of 2·814 Å. The glucitol molecule has a non-planar carbon chain such that the parallel alignment of C(1)–OH to C(3)–OH and C(2)–OH to C(4)–OH is avoided.

Introduction

Glucitol forms a 1:1 solvent complex on crystallization from pyridine, which has been used for purification (Strain, 1934, 1937). A similar derivative has been reported for 2-deoxyglucitol (Wolfrom, Konigsberg, Moody & Goepp, 1946). In addition to the intrinsic interest in the structure of the complex, this compound provides an opportunity to examine the conformation of the glucitol molecule in a crystal-field environment different from that in the crystal structure of D-glucitol itself, which has also been determined (Jeffrey & Park, 1970).

Crystal data

Large, transparent monoclinic crystals of

$$C_6H_{14}O_6.C_5H_5N,$$

m.p. 76°C, which decomposed on exposure to air, were obtained by slowly cooling a saturated solution of D-glucitol in pyridine. They gave the following data:

Space group $P2_1$, from systematic absences $0k0$ absent for k odd

$a=4·652$ (3) Å $\quad V=638·15$ Å^3

$b=10·207$ (4) $\quad Z=2$

$c=13·439$ (5) $\quad D_m=1·365$ g.cm^{-3} at 23°C

Table 1. *Fractional atomic coordinates and anisotropic thermal parameters in glucitol–pyridine complex*

Key to atomic numbering is given in Fig. 1. The estimated standard deviations given in parentheses refer to the last decimal positions. The temperature factor expression used was

$$\exp\left[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)\right].$$

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	0·7081 (5)	0·3858 (4)	0·1738 (2)	0·0360 (10)	0·0081 (3)	0·0032 (1)	0·0022 (4)	−0·0010 (3)	0·0008 (1)
C(2)	0·6172 (5)	0·5249 (3)	0·1488 (2)	0·0301 (9)	0·0066 (2)	0·0031 (1)	−0·0005 (4)	−0·0008 (3)	−0·0001 (1)
C(3)	0·4552 (4)	0·5333 (3)	0·0491 (2)	0·0226 (8)	0·0046 (2)	0·0032 (1)	−0·0003 (3)	−0·0003 (3)	−0·0000 (1)
C(4)	0·6480 (4)	0·5077 (3)	−0·0411 (2)	0·0239 (9)	0·0047 (2)	0·0029 (1)	0·0000 (3)	−0·0003 (2)	0·0000 (1)
C(5)	0·4666 (5)	0·4724 (3)	−0·1330 (2)	0·0260 (9)	0·0050 (2)	0·0033 (1)	0·0004 (3)	−0·0014 (3)	0·0000 (1)
C(6)	0·6427 (6)	0·4486 (3)	−0·2269 (2)	0·0422 (11)	0·0074 (3)	0·0027 (1)	−0·0008 (5)	−0·0006 (3)	0·0002 (1)
C(7)	0·1481 (9)	0·3656 (5)	0·4298 (3)	0·0816 (23)	0·0140 (5)	0·0067 (2)	0·0010 (9)	−0·0054 (6)	−0·0008 (3)
C(8)	0·0782 (9)	0·3502 (7)	0·5276 (3)	0·0671 (19)	0·0199 (7)	0·0070 (2)	0·0039 (10)	0·0006 (5)	−0·0030 (4)
C(9)	0·1977 (9)	0·2507 (5)	0·5809 (2)	0·0725 (19)	0·0179 (6)	0·0041 (2)	−0·0076 (9)	0·0020 (4)	−0·0008 (3)
C(10)	0·3849 (9)	0·1682 (5)	0·5346 (3)	0·0857 (23)	0·0136 (4)	0·0055 (2)	−0·0018 (9)	−0·0020 (5)	0·0002 (3)
C(11)	0·4420 (9)	0·1893 (5)	0·4351 (3)	0·0775 (21)	0·0146 (5)	0·0059 (2)	−0·0032 (9)	0·0027 (5)	−0·0024 (3)
N	0·3257 (7)	0·2856 (5)	0·3819 (2)	0·0829 (17)	0·0159 (4)	0·0040 (1)	−0·0102 (8)	0·0014 (4)	0·0002 (2)
O(1)	0·4718 (4)	0·2983 (3)	0·1792 (1)	0·0548 (10)	0·0059 (2)	0·0028 (1)	−0·0018 (3)	0·0010 (2)	0·0001 (1)
O(2)	0·4398 (4)	0·5685 (3)	0·2279 (1)	0·0566 (10)	0·0067 (2)	0·0027 (1)	0·0019 (3)	0·0012 (2)	−0·0004 (1)
O(3)	0·3337 (3)	0·6615 (3)	0·0406 (1)	0·0254 (6)	0·0053 (1)	0·0034 (1)	0·0011 (2)	−0·0016 (2)	−0·0005 (1)
O(4)	0·8273 (3)	0·6179 (3)	−0·0614 (1)	0·0212 (6)	0·0055 (2)	0·0039 (1)	−0·0011 (2)	−0·0002 (2)	0·0003 (1)
O(5)	0·2948 (3)	0·3601 (3)	−0·1106 (1)	0·0241 (6)	0·0056 (2)	0·0041 (1)	−0·0005 (3)	−0·0013 (2)	−0·0002 (1)
O(6)	0·8043 (3)	0·3305 (3)	−0·2228 (1)	0·0332 (7)	0·0073 (2)	0·0037 (1)	−0·0001 (3)	0·0002 (2)	−0·0012 (1)
H(C1)	0·828	0·363	0·120						
H(C1′)	0·830	0·393	0·234						
H(C2)	0·809	0·574	0·143						
H(C3)	0·299	0·468	0·052						
H(C4)	0·783	0·435	−0·022						
H(C5)	0·345	0·536	−0·143						
H(C6)	0·770	0·524	−0·239						
H(C6′)	0·534	0·442	−0·287						
H(C7)	0·044	0·439	0·398						
H(C8)	−0·051	0·435	0·562						
H(C9)	0·143	0·242	0·649						
H(C10)	0·471	0·097	0·575						
H(C11)	0·604	0·141	0·390						
H(O1)	0·424	0·292	0·226						
H(O2)	0·436	0·649	0·227						
H(O3)	0·163	0·655	0·017						
H(O4)	0·740	0·671	−0·061						
H(O5)	0·388	0·307	−0·095						
H(O6)	0·947	0·346	−0·179						

$\beta = 90{\cdot}3\ (3)^\circ$ $D_x = 1{\cdot}359\ \mathrm{g.cm^{-3}}$
μ Cu $K\alpha = 9{\cdot}49\ \mathrm{cm^{-1}}$

(the e.s.d.'s given in parentheses refer to the last decimal).

Experimental

The unit-cell dimensions and intensities were measured on a Picker FACS I diffractometer using a crystal of dimensions $0{\cdot}43 \times 0{\cdot}52 \times 0{\cdot}37$ mm which was coated

Table 2. *Observed and calculated structure factors*

Columns are: index l, $10|F_{\rm obs}|$, $10|F_{\rm calc}|$, A, B.

H= 0 K= 0				
1	350	400	400	0
2	227	256	256	0
3	415	498	498-	0
4	42	42	42	0
5	335	383	383	0
6	211	221	221-	0
7	105	107	107-	0
8	108	109	109-	0
9	5*	6	6-	0
10	160	158	158-	0
11	178	183	183-	0
12	100	101	101-	0
13	5*	7	7	0
14	30	29	29-	0
15	77	78	78-	0
H= 0 K= 1				
1	75	81	74	33-
2	168	164	137	91
3	105	99	76-	63
4	197	208	78	192
5	264	267	258	69
6	127	129	85-	97-
7	130	132	58-	119-
8	199	198	163-	113-
9	138	136	136	4
10	190	198	190	55
11	108	108	93	54
12	63	60	60-	3-
13	39	37	9-	36-
14	33	33	15-	30-
15	59	62	62	3
H= 0 K= 2				
0	189	182	24-	180-
1	425	525	501	157
2	347	385	121-	366
3	339	392	17	392
4	218	229	99	206
5	148	150	141-	51-
6	76	76	22	72-
7	65	66	4-	66-
8	138	136	83	107-
9	95	99	1-	99-
10	181	192	119-	151-
11	163	168	144-	87-
12	58	61	43-	42-
13	32	33	31	11
14	47	48	38	29
15	29	30	3	30
H= 0 K= 3				
1	254	267	266	21
2	153	154	134	77-
3	141	146	146-	7
4	115	122	103-	65-
5	69	59	30-	51-
6	107	103	37	96
7	171	169	79-	150
8	175	176	74-	160
9	114	116	103	52
10	70	67	60	30-
11	79	78	69	35
12	70	70	46-	53
13	44	44	17-	40
14	45	45	45	7-
15	43	43	7-	42-
H= 0 K= 4				
0	286	271	164-	215
1	113	109	42-	101
2	254	260	89-	244-
3	177	175	89	151-
4	109	110	100-	47-
5	143	139	98	99
6	74	73	72	11-
7	144	148	90	118-
8	50	51	48	17
9	63	61	59-	14-
10	38	38	25-	29
11	50	49	49-	5-
12	16	14	11-	10
13	36	36	6-	36
14	20	22	22	4-
H= 0 K= 5				
1	91	92	92	10
2	48	50	1	50-
3	131	119	23-	117-
4	139	137	84-	108-
5	58	60	0	60
6	41	44	12	43-
7	56	56	21-	52-
8	76	75	61	44-
9	40	40	40-	2
10	124	128	45	120
11	118	114	114-	6-
12	54	54	53-	10
13	50	52	49-	17-
14	22	21	10	19
H= 0 K= 6				
0	248	239	239	7
1	136	134	131	30-
2	157	157	149	50
3	79	70	39-	58-
4	32	33	32	5-
5	71	68	38-	56-
6	43	41	39	9-
7	83	81	13	80
8	26	24	20-	13
9	48	49	6-	48
10	64	67	64-	21-
11	33	31	29-	12-
12	81	81	79-	18
13	46	44	34-	27-
H= 0 K= 7				
1	188	195	147	129
2	175	183	156	94
3	27	27	27	0
4	35	34	34	5-
5	80	82	80	17-
6	83	84	68	48
7	67	65	47	46-
8	51	50	5-	50-
9	73	74	73	9
10	22	25	23	9-
11	42	42	6-	41
12	84	83	82-	11-
H= 0 K= 8				
0	39	41	2-	41
1	74	74	60	44
2	143	142	122	72
3	72	71	43	57
4	94	98	97-	12
5	97	101	84-	57
6	79	77	0	77
7	46	46	45	7
8	68	69	20-	66-
9	135	135	63-	119-
10	51	51	15-	49-
11	18	17	1	17
H= 0 K= 9				
1	85	82	69	43-
2	83	82	78	24
3	111	112	72	86
4	125	126	20-	125
5	102	105	55-	90
6	53	53	41-	33
7	48	48	6	48
8	51	51	35	37
9	42	39	18	35
10	11	13	1	13
H= 0 K= 10				
0	46	45	37-	25-
1	34	33	3-	33
2	12	12	6-	10
3	42	42	3-	41
4	36	37	26-	26
5	25	25	25	1
6	49	49	48	9-
7	38	37	10	35-
8	57	59	47-	35-
H= 0 K= 11				
1	69	71	71-	10-
2	31	33	22-	24-
3	11	11	8	7
4	18	18	17	7
5	74	77	72-	28
6	84	87	79-	35
H= 0 K= 12				
0	35	34	26-	21
1	22	22	19-	11
H= 1 K= 0				
0	552	711	711-	0
1	456	522	522-	0
2	187	191	191	0
3	243	252	252	0
4	187	187	187-	0
5	509	602	602-	0
6	498	559	559-	0
7	72	71	71-	0
8	22	20	20-	0
9	10	4	4-	0
10	4*	7	7	0
11	73	78	78	0
12	144	148	148	0
13	86	87	87	0
14	17	16	16	0
15	62	61	61	0
H= 1 K= 1				
0	276	292	246-	156
1	381	433	362	238
2	386	421	420-	36-
3	124	125	15-	124-
4	103	95	46	84
5	200	209	17-	208
6	212	226	135	182
7	72	67	30-	60
8	90	92	37	84-
9	55	56	47-	30-
10	151	154	95-	120-
11	74	76	56-	51-
12	49	48	34	35-
13	80	78	51	59
14	31	31	7	30
15	43	41	41-	5-
H= 1 K= 2				
0	291	299	183-	236-
1	239	254	168-	191
2	451	502	87	495-
3	199	199	16-	198-
4	461	477	125-	460-
5	180	178	134-	117-
6	88	79	49	62
7	69	72	0	72-
8	48	49	27-	41-
9	79	78	20-	76-
10	94	93	32	88
11	154	158	146	60
12	39	38	38	1
13	42	45	34-	29-
14	38	39	22-	33
15	70	69	3-	69
H= 1 K= 3				
0	186	188	118	147
1	405	461	458-	57-
2	241	235	231-	45
3	137	138	46-	130
4	142	146	133	44-
5	115	110	105	33-
6	111	115	95-	64-
7	37	36	20	30-
8	66	70	18	67
9	71	68	1	68-
10	56	56	35-	43
11	77	75	23	71-
12	66	66	64	17-
13	88	87	51	71-
14	36	36	25-	26-
15	35	36	32-	17-
H= 1 K= 4				
0	245	232	231-	24-
1	232	230	125-	193-
2	265	231	132-	208
3	133	131	76	107
4	150	150	42	144
5	136	127	99	79-
6	117	108	33	103-
7	80	79	26	75
8	80	88	63	62
9	85	86	26	82
10	99	99	97	16
11	33	35	30-	18-
12	22	22	22-	5-
13	53	53	52-	5
14	34	32	28-	15
H= 1 K= 5				
0	25	26	1-	26-
1	141	135	134-	14-
2	40	35	35-	9-
3	78	95	61	73-
4	64	65	64	10-
5	153	145	123-	76-
6	67	74	73-	6-
7	77	75	6-	75
8	78	71	43	56
9	85	82	22	79
10	54	54	15-	51-
11	53	50	43	27-
12	60	58	58	1
13	41	42	36	22
14	30	30	20-	22
H= 1 K= 6				
0	91	82	82-	4
1	154	143	119-	78
2	153	144	133-	56
3	31	23	20-	11-
4	56	51	50	12-
5	58	56	55-	5-
6	177	178	154-	89
7	123	124	123-	16
8	31	32	25-	20
9	68	62	59	17
10	4*	6	2	5
11	47	48	16	45
12	18	19	11	16-
13	48	48	48	3
H= 1 K= 7				
0	79	77	77	5
1	79	74	19	72-
2	86	79	25-	75-
3	47	46	37	28
4	72	73	12	65
5	87	86	42	75
6	75	79	78-	10-
7	71	70	66-	20-
8	46	46	44-	13-
9	56	53	37-	38-
10	49	51	28-	42-
11	71	72	70-	16-
12	25	22	15	16-
H= 1 K= 8				
0	131	130	88	95-
1	69	68	68	10-
2	60	59	9	58-
3	122	118	82-	84-
4	113	115	38-	109-
5	36	37	1	37-
6	49	50	33-	37-
7	73	74	70-	24
8	63	65	59-	27
9	46	46	11	44
10	42	43	27	33
11	46	46	18	43-
H= 1 K= 9				
0	73	76	59-	45
1	121	121	84-	87
2	109	106	80-	67
3	49	48	30-	38
4	49	48	42	22-
5	80	80	50	62-
6	34	34	34	1-
7	45	43	42-	12-
8	73	70	53-	45-
9	98	99	31-	94-
10	63	67	7-	66-
H= 1 K= 10				
0	47	48	1-	48
1	38	38	28	27-
2	66	65	0	65-
3	48	46	30-	36-
4	4*	7	6-	6-
5	16	14	7-	12-
6	24	22	0	22-
7	17	18	9-	15-
8	44	45	20	40
H= 1 K= 11				
0	73	75	64-	40
1	38	40	31-	25
2	29	30	20-	22
3	61	61	55-	27-
4	59	63	51-	38-
5	26	26	4	26-
H= -1 K= 0				
1	157	169	169-	0
2	77	82	82	0
3	118	113	113	0
4	244	243	243	0
5	292	312	312	0
6	434	480	480	0
7	90	62	62	0
8	4*	6	6	0
9	52	58	58	0
10	149	152	152	0
11	62	62	62	0
12	25	20	20-	0
13	86	89	89-	0
14	65	65	65	0
15	25	26	26	0
H= -1 K= 1				
1	193	191	191-	5-
2	277	294	195	219
3	63	58	48-	32
4	477	547	530-	137-
5	245	251	246-	48
6	149	149	94-	112
7	210	212	134	165
8	41	44	23	37
9	106	105	86-	61
10	41	43	43-	4
11	48	46	46	4-
12	97	94	61	72-
13	44	41	21	35-
14	16	15	11	11-
15	16	18	17	3
H= -1 K= 2				
1	353	380	250-	286
2	176	186	12	184
3	272	280	39	277
4	68	72	5	72
5	219	219	219	8-
6	97	96	41	87
7	101	83	21	81
8	118	117	105-	51
9	96	93	62-	69-
10	123	124	121	27
11	135	137	128	51
12	83	80	50	63
13	13	8	8-	2-
14	87	85	67-	52-
15	28	28	4	28-
H= -1 K= 3				
1	296	302	193-	232-
2	265	272	46	268-
3	67	63	19-	59-
4	158	160	56	150
5	269	280	280-	3-
6	138	138	104-	91-
7	185	181	36	177-
8	61	51	10-	50-
9	20	22	6-	22-
10	83	85	72-	44
11	48	47	28	38-
12	57	59	24	53
13	37	34	16	30
14	61	63	1	63
15	64	63	39	49
H= -1 K= 4				
1	102	101	88-	50-
2	54	53	2	53
3	102	91	14	90-
4	68	64	7	64-
5	143	145	120-	81-
6	82	76	67-	37
7	34	32	21-	24
8	52	53	24-	48-
9	99	97	88	39
10	51	52	12	51-
11	122	122	122	2
12	88	88	69	55-
13	42	42	42	8-
14	30	31	1-	31
H= -1 K= 5				
1	143	136	91-	99
2	123	125	117-	43
3	182	171	170	22
4	144	143	73	123-
5	123	126	124	54-
6	62	64	43	47
7	56	54	50	22
8	30	30	28	9-
9	144	147	105-	102-
10	86	86	61-	60-
11	55	54	23-	49
12	73	73	65	33
13	41	40	15	36
14	20	21	16-	13-
H= -1 K= 6				
1	206	198	37-	194-
2	245	245	132-	207-
3	145	138	19-	137-
4	81	81	70	41-
5	141	139	124	62
6	89	89	87	20
7	22	21	7	19
8	110	110	104	36
9	46	49	49	0
10	118	119	87	81
11	24	24	2-	24
12	46	45	34	29
13	39	38	38	5-
H= -1 K= 7				
1	100	95	77-	56-
2	67	65	64-	12
3	91	92	92-	8
4	88	92	90-	19
5	28	30	15-	25-
6	106	107	97	46-
7	94	94	75	56
8	20	22	16-	15
9	105	105	85-	61
10	31	31	28-	13
11	52	52	52	4-
12	59	59	56	19-
H= -1 K= 8				
1	117	114	101-	53-
2	73	68	53-	43-
3	75	75	15	73
4	133	135	125	49
5	81	79	74	27-
6	33	32	26-	17-
7	33	33	13	30
8	89	91	1-	91
9	84	87	18	85
10	56	55	39-	40
11	37	37	23-	29
H= -1 K= 9				
1	96	94	32-	89-
2	90	91	58-	70-
3	95	93	85-	38-
4	18	19	13-	13-
5	37	37	2	37-
6	33	35	7-	34-
7	24	22	15-	15-
8	57	59	27-	52
9	35	36	19	31
10	4*	6	5	4
H= -1 K= 10				
1	36	36	14	33
2	23	22	7	21
3	76	77	75	20-
4	35	36	32	16
5	45	47	40-	25
6	57	58	58-	4-
7	33	33	31-	10-
8	26	28	27	6-
H= -1 K= 11				
1	57	60	60	0
2	12	13	4-	12-
3	22	22	17-	15-
4	14	15	5-	15-
5	33	33	32	9-
H= 2 K= 0				
0	131	113	113-	0
1	107	106	106-	0
2	194	205	205-	0
3	166	181	181-	0
4	109	104	104	0
5	158	158	158	0
6	313	316	316	0
7	99	95	95	0
8	23	22	22	0
9	40	43	43	0
10	19	19	19	0
11	67	59	59	0
12	4*	2	2-	0
13	19	20	20	0
14	17	18	18	0
H= 2 K= 1				
0	56	51	17-	48
1	189	169	148-	82
2	272	205	171-	113-
3	150	148	94-	114
4	84	88	8-	87-
5	163	166	19-	165-
6	194	202	29	199-
7	65	63	19	60-
8	118	116	85	79
9	86	89	88	11
10	22	20	11-	17-
11	45	43	36	23-
12	63	62	62-	5
13	16	13	13	4-
14	12	11	7-	9-
H= 2 K= 2				
0	177	172	126	119
1	47	42	12	40
2	95	99	71	68-
3	126	123	80-	94-
4	50	51	37	35-
5	174	170	154	72
6	41	41	3-	41-
7	36	35	2-	35-
8	96	96	33-	90
9	97	98	32	93
10	138	139	29	136
11	57	58	58-	0
12	75	78	59-	52
13	12	14	1-	14
14	29	32	32	4
H= 2 K= 3				
0	274	266	189-	178-
1	247	239	139	194-
2	233	235	107	209
3	130	129	13	128-
4	159	163	162-	15-
5	149	139	16-	138-
6	117	119	112	40-
7	174	175	175	8
8	113	114	73	88-
9	16	17	7-	15
10	35	37	16	33
11	95	96	29-	91
12	41	42	10-	41
13	38	38	38-	4
14	16	16	15-	5
H= 2 K= 4				
0	206	190	186	40
1	300	298	293	54
2	70	67	8-	67-
3	49	54	54	8
4	62	62	39-	46
5	72	69	66	20
6	91	92	87	30
7	29	27	4-	27-
8	76	77	21-	74-
9	77	82	65-	51-
10	84	85	23-	82-
11	35	32	21-	24
12	21	22	3-	22
13	24	25	24-	7
H= 2 K= 5				
0	39	35	9-	34-
1	155	141	123	69-
2	268	257	229	117-
3	106	98	67	71
4	48	46	5-	46
5	84	79	18	77
6	83	81	80	14-
7	85	84	14	83-
8	63	64	64-	4-
9	58	59	52-	27
10	88	87	23	84
11	52	54	40	36
12	25	23	17-	16-
13	69	68	54-	41-
H= 2 K= 6				
0	48	44	34-	28-
1	137	145	124-	76-
2	94	88	51	72
3	151	148	41	142
4	182	185	61	175
5	103	101	39	93
6	105	105	100	32-
7	156	155	148	45-
8	52	55	45	31-
9	39	34	29-	18-
10	50	50	31-	39-
11	56	58	33	48-
12	67	66	53	39-
H= 2 K= 7				
0	81	80	77	22-
1	72	73	31-	66
2	16	15	9-	12
3	42	40	20-	34-
4	71	66	1-	66-
5	45	45	21-	40-
6	37	38	37-	10-
7	37	35	8-	35
8	19	19	6-	18
9	34	33	24	24
10	12	11	10	4
11	33	32	20	24-
H= 2 K= 8				
0	94	91	89-	20
1	105	103	94-	43
2	50	53	50-	17
3	51	51	26	44
4	81	81	36	72
5	49	49	15-	47
6	38	36	4	36
7	20	22	7	21-
8	44	43	43	1
9	25	24	23	9
10	27	27	1	27
H= 2 K= 9				
0	32	31	8-	30-
1	49	51	49	16-
2	12	8	6	6-
3	55	57	8-	56-
4	75	73	41-	60-
5	48	48	30-	37-
6	29	28	28	4-
7	64	83	74	37-
8	84	83	81	18-
H= 2 K= 10				
0	26	26	20	17-
1	18	20	2	19-
2	19	22	13-	18
3	28	31	5-	31
4	24	25	20-	16-
5	29	30	20-	22-
6	34	33	28-	18-
H= 2 K= 11				
0	31	32	29	14-
1	38	37	31	21-
2	30	31	22	22-
H= -2 K= 0				
1	155	151	151-	0
2	79	71	71	0
3	71	65	65-	0
4	4*	5	5	0
5	176	172	172-	0
6	114	113	113-	0
7	11	12	12	0
8	55	52	52-	0
9	44	42	42-	0
10	77	80	80-	0
11	20	19	19-	0
12	4*	3	3	0
13	4*	4	4	0
14	4*	1	1	0
H= -2 K= 1				
1	259	263	26-	262-
2	88	80	43	67-
3	101	99	93	35-
4	186	172	163	55-
5	111	110	6-	109-
6	42	44	21-	39-
7	121	121	14-	120
8	102	94	74-	58
9	4*	7	5-	5
10	127	124	115-	48-
11	63	65	60-	25
12	66	65	37-	53
13	46	45	39-	22
14	18	17	15	7-
H= -2 K= 2				
1	122	111	40	103-
2	182	188	34-	185-
3	196	196	183	71-
4	106	110	109-	8
5	164	161	159-	25
6	207	202	167-	114-
7	21	20	15-	13
8	165	162	157	39
9	115	116	15	115
10	91	90	58-	69-
11	108	110	108-	23-
12	51	51	20-	47-
13	27	26	16	20
14	25	25	23	10-
H= -2 K= 3				
1	383	393	169	355
2	106	97	41	87
3	18	12	2-	12
4	54	47	14-	45-
5	150	145	141	34-
6	79	76	1-	76-
7	151	156	38-	151-
8	55	54	1-	54-
9	89	91	48-	78-
10	16	15	14-	4-
11	86	85	69-	50-
12	40	43	29-	32-
13	31	32	31	8-
14	21	22	11-	19-
H= -2 K= 4				
1	165	159	109	117
2	67	60	7	59
3	177	175	175-	6-
4	161	142	86-	113
5	165	163	137-	89
6	105	108	81-	71
7	58	57	13-	56-
8	59	61	22-	57-
9	71	69	28	63-
10	44	40	20-	35
11	16	15	0	15-
12	29	29	24-	16
13	29	32	11-	30-
H= -2 K= 5				
1	67	59	58	11
2	27	24	19-	14
3	117	110	52-	96
4	125	129	100	82
5	50	48	45	15-
6	87	88	45-	76-
7	112	112	84-	74-
8	48	45	13	43-
9	156	156	152	34
10	102	103	103	5-
11	25	27	20	18-
12	32	32	7-	32-
13	30	29	9-	28-
H= -2 K= 6				
1	38	36	22	28
2	78	72	23	68
3	138	131	100-	86
4	164	164	163-	6-
5	90	91	63-	66-
6	90	89	11-	88-
7	131	103	36-	95-
8	104	101	101-	10-
9	48	47	44-	19-
10	40	40	19	35-
11	49	50	33	37-
12	13	11	8-	7-
H= -2 K= 7				
1	42	40	33-	24-
2	20	21	18-	10
3	137	135	111	77-
4	91	93	80	49-
5	34	34	16-	30
6	84	85	59-	61
7	80	80	56-	57
8	46	45	44	10
9	48	49	45	19-
10	34	35	31	17-
11	4*	4	3	3-
H= -2 K= 8				
1	28	28	28	4-
2	53	53	39	36-
3	71	71	38	60-
4	54	52	1-	52-
5	46	47	2	47-
6	83	81	22	78-
7	37	38	14-	35-
8	52	52	27	45-
9	4*	2	1	2
10	16	15	11	9-
H= -2 K= 9				
1	78	75	9-	75
2	54	58	58	1-
3	43	46	33	32-
4	24	24	16	18-
5	30	29	22	19
6	20	21	3-	21
7	39	40	32	23-
8	33	33	11-	31-
H= -2 K= 10				
1	35	34	13	31
2	43	43	6-	43
3	36	35	34-	10
4	14	16	1-	15-
5	27	28	28	3
6	46	48	20	44
H= -2 K= 11				
1	23	25	8	24
2	60	59	37	46
H= 3 K= 0				
0	30	29	29-	0
1	37	38	38-	0
2	23	23	23	0
3	45	45	45	0
4	16	15	15-	0
5	76	78	78-	0
6	4*	4	4-	0
7	13	13	13	0
8	50	49	49	0
9	31	30	30	0
10	30	29	29-	0
11	22	24	24	0
12	81	81	81-	0
13	28	28	28-	0
H= 3 K= 1				
0	81	79	49-	63-
1	74	71	67	23-
2	105	104	104	3
3	176	174	173	16-
4	66	66	31	58-
5	30	28	28	3
6	73	74	70-	25
7	61	60	58	14
8	51	51	48	18-
9	11	12	11	4-
10	53	49	9	48
11	55	51	33-	39
12	36	33	19	27
13	23	19	12-	16
H= 3 K= 2				
0	40	42	32	28-
1	34	32	8-	32-
2	66	61	42-	45
3	48	49	14-	47
4	60	57	11-	55
5	30	30	28-	10-
6	66	67	67-	1-
7	50	53	10-	52
8	21	20	6	19
9	15	13	3	13-
10	48	49	2	49-
11	35	33	21-	25-
12	53	55	52	16
H= 3 K= 3				
0	52	51	47	20
1	60	55	45	32
2	88	87	66-	56-
3	77	77	75	17-
4	143	144	76	122
5	232	235	55	229
6	215	215	36-	212
7	121	122	103-	65
8	23	21	19-	10-
9	20	22	21	6
10	18	16	17	4
11	20	22	22-	1-
12	41	42	38-	18-
H= 3 K= 4				
0	110	104	11	104-
1	74	70	24	65-
2	77	80	78	17-
3	53	51	51	4
4	140	140	24	138-
5	104	104	63-	82-
6	105	101	101-	8
7	105	103	79-	66
8	58	59	11-	58
9	16	15	11-	10-
10	28	30	21-	21
11	36	35	28-	22
12	15	16	14	7
H= 3 K= 5				
0	49	48	46	15
1	88	67	76-	38-
2	83	75	47-	59
3	165	168	168-	12
4	64	64	14	62
5	45	43	7-	42-
6	69	68	25-	63
7	115	114	72-	89
8	84	83	79-	23
9	45	42	9-	41-
10	78	80	33-	73-
11	37	37	26-	24-
H= 3 K= 6				
0	82	82	53	63
1	120	119	105	55
2	88	90	86	28-
3	115	116	16	115-
4	97	93	46-	81-
5	66	62	15-	60-
6	33	35	1-	35
7	32	31	6-	31
8	42	45	40-	20-
9	31	30	18-	24-
10	31	31	22-	22-
H= 3 K= 7				
0	46	43	26-	34
1	4*	7	7	1
2	58	59	26-	53-
3	35	37	19	31
4	36	35	32-	13
5	22	20	16-	12-
6	40	37	17-	32-
7	34	34	9-	33-
8	11	10	0	10
9	25	26	26-	2
H= 3 K= 8				
0	57	56	17	53
1	33	34	29	18
2	34	33	33-	3
3	60	58	57-	7
4	20	20	18-	8-
5	27	24	24	1-
6	46	47	42	22-
7	22	21	10	19-
8	4*	3	1	3-
H= 3 K= 9				
0	11	13	9	10
1	17	17	16	6
2	13	16	16	3-
3	19	18	13	12
4	23	24	10	22
5	25	25	5	25
H= -3 K= 0				
1	117	109	109	0
2	93	93	93	0
3	54	52	52	0
4	46	48	48-	0
5	17	16	16	0
6	131	127	127	0
7	12	11	11	0
8	85	80	80	0
9	82	81	81-	0
10	33	34	34	0
11	4*	3	3-	0
12	4*	3	3	0
13	11	7	7	0
H= -3 K= 1				
1	44	46	45-	13
2	124	123	110	56-
3	56	52	26	46-
4	118	112	101	48
5	109	111	78	79
6	32	32	30	11
7	108	107	82	68-
8	48	46	20-	42-
9	17	13	7-	10-
10	42	42	32-	28
11	19	17	17	2
12	33	35	30	18
13	23	23	13	20
H= -3 K= 2				
1	54	56	11-	55-
2	100	101	75-	68-
3	118	112	101-	47-
4	98	98	83	52
5	196	200	160	121
6	121	120	87	82
7	31	31	17	25
8	102	99	96-	26-
9	46	45	30	33
10	27	27	6	26
11	18	20	17	11-
12	46	47	8	46-
13	42	42	13-	39-
H= -3 K= 3				
1	34	32	17-	28
2	37	38	2	38
3	84	77	60	48
4	44	43	6-	42
5	139	132	34	127
6	146	143	64	127
7	88	88	3	88
8	35	38	20-	32
9	70	73	45-	57
10	28	27	12	24
11	15	14	14	0
12	63	61	6-	61-
H= -3 K= 4				
1	66	59	40-	43-
2	99	99	54-	82
3	56	55	51	20
4	101	100	69	73-
5	125	126	108	64-
6	37	38	15	35
7	86	87	18-	85
8	55	55	17-	52
9	23	19	18-	7
10	41	40	35-	18
11	45	43	21-	37
12	22	24	23-	5
H= -3 K= 5				
1	20	23	21	10
2	52	49	26-	42
3	147	150	148-	23
4	182	182	176-	45
5	102	100	100-	9
6	53	52	27	44
7	71	73	23	69
8	41	39	16-	36
9	39	38	31-	21-
10	40	38	4	37-
11	25	25	9	23-
H= -3 K= 6				
1	113	114	7	114
2	85	86	82	25
3	77	77	77	8-
4	67	62	55	28-
5	27	28	27-	6-
6	47	49	8-	49
7	21	20	17	10
8	18	20	5	19-
9	54	54	47-	26-
10	41	40	35-	21
H= -3 K= 7				
1	75	73	14-	72-
2	40	40	31-	26-
3	86	87	61-	61
4	81	83	76-	35
5	38	38	36-	11-
6	32	31	9-	29-
7	57	56	50	25-
8	26	26	25	3-
9	24	23	19	13-
H= -3 K= 8				
1	28	28	12	26
2	37	37	30-	22
3	4*	6	4-	4
4	15	12	8	9
5	28	28	3	27
6	34	33	21	25
7	26	27	5	27
8	23	21	11	18
H= -3 K= 9				
1	19	21	9-	18-
2	14	16	16-	1
3	18	17	12-	13-
4	13	12	6	11-
5	18	15	6	14
H= 4 K= 0				
0	37	37	37-	0
1	80	81	81	0
2	19	19	19	0
3	49	51	51-	0
4	74	70	70-	0
5	31	32	32	0
6	28	27	27	0
7	18	16	16	0
8	52	54	54-	0
9	31	31	31-	0
10	21	23	23	0
H= 4 K= 1				
0	26	21	21	1-
1	18	17	15	9
2	49	46	37	27-
3	31	32	31	3
4	23	21	15	15-
5	32	31	26	18-
6	53	52	6	52-
7	25	25	19-	17-
8	81	83	83-	3
9	70	70	69-	11
10	17	17	17-	2-
H= 4 K= 2				
0	67	66	29-	60-
1	100	99	43-	89-
2	48	46	7-	46-
3	44	44	41	16
4	65	65	31	57
5	35	35	7	34
6	29	28	28	3-
7	18	20	10	18-
8	25	26	26	6
9	18	16	2-	16
10	4*	8	8-	2-
H= 4 K= 3				
0	149	49	4-	149
1	82	83	14-	82
2	73	76	23	73
3	33	33	33	2-
4	21	22	8	21-
5	101	102	9	102-
6	102	100	12	99-
7	59	59	3	59-
8	23	22	8-	21
9	33	35	35-	3
10	64	64	13-	62-
H= 4 K= 4				
0	26	24	8-	22
1	112	112	82-	77
2	65	64	22-	60
3	35	35	34-	3-
4	52	53	26	46
5	73	73	4-	73
6	32	34	3	34
7	33	31	19	25-
8	31	31	4-	31-
9	30	31	4	31
H= 4 K= 5				
0	16	16	8-	14
1	82	82	49-	66
2	79	78	57-	53
3	35	38	35-	15
4	35	33	16-	29-
5	60	60	11-	59-
6	28	25	6	24-
7	59	58	57	12-
8	83	84	83	15-
H= 4 K= 6				
0	22	20	20-	0
1	42	43	4-	43
2	48	52	48-	20
3	50	53	24-	48
4	50	52	52-	4
5	23	21	19-	8-
6	62	64	42-	49-
7	69	71	43-	56-
H= 4 K= 7				
0	59	57	30-	48-
1	30	29	29-	1-
2	30	30	8-	29
3	23	27	20-	18
4	43	43	14-	41-
5	24	26	26-	3-
H= 4 K= 8				
0	25	25	12-	23-
1	27	28	12-	26-
2	4*	7	3	7-
H= -4 K= 0				
1	85	80	80-	0
2	119	120	120-	0
3	20	18	18-	0
4	15	14	14-	0
5	56	56	56-	0
6	5*	7	7-	0
7	4*	7	7	0
8	70	67	67	0
9	47	47	47	0
10	54	52	52	0
H= -4 K= 1				
1	32	31	31-	0
2	77	78	64-	45
3	30	29	1	29
4	50	48	27	39-
5	94	96	20-	94-
6	91	93	26-	89-
7	81	83	75-	36-
8	65	64	64	8
9	50	50	45	21
10	51	54	48	26
H= -4 K= 2				
1	25	28	2-	28-
2	64	64	61	20
3	18	13	6	11
4	45	45	43-	14-
5	57	57	53-	21-
6	58	62	45-	43-
7	14	12	2	12
8	4*	9	4-	8
9	30	30	11	28
10	24	22	18	12
H= -4 K= 3				
1	75	72	16-	70-
2	48	48	14	46-
3	48	49	26	41-
4	84	85	48	70-
5	69	67	15	65-
6	25	24	21-	13
7	37	37	14	34
8	15	15	13	6
9	34	37	26	26-
10	10	9	9	3
H= -4 K= 4				
1	78	80	48	64-
2	104	103	69	77-
3	56	56	54	14
4	32	33	1	33
5	31	31	6-	30-
6	57	57	22-	53-
7	47	49	25-	42-
8	32	32	29-	14
9	15	14	6-	13-
H= -4 K= 5				
1	44	44	15	42-
2	44	45	36	30-
3	90	88	87	17-
4	61	59	59	3
5	43	44	38	22
6	23	22	13	18-
7	4*	6	1	6-
8	30	31	28-	12
H= -4 K= 6				
1	34	33	21-	26-
2	4*	11	9-	6-
3	4*	6	3	5
4	38	38	22	32
5	4*	2	1-	1
6	19	20	7	19-
7	21	20	17	11-
H= -4 K= 7				
1	62	59	0	59
2	14	13	6	11
3	24	23	9	21-
4	30	30	28	12-
5	25	25	4	25-
H= -4 K= 8				
1	3*	5	5-	3-
2	33	35	33-	11-
H= 5 K= 0				
0	4*	6	6-	0
1	4*	3	3-	0
2	4*	7	7	0
3	48	48	48	0
4	71	71	71	0
5	53	52	52	0
6	4*	2	2-	0
H= 5 K= 1				
0	81	81	11-	80-
1	59	59	10	58-
2	25	24	17-	16-
3	34	35	35-	4-
4	59	60	60-	6
5	45	46	18-	42
6	65	67	4	67
H= 5 K= 2				
0	44	45	7	45
1	23	22	21	5
2	29	27	9	26
3	11	12	5-	11-
4	22	21	21-	0
5	18	18	11-	14-
H= 5 K= 3				
0	66	65	27	59-
1	81	80	18	78-
2	54	54	8	53-
3	24	25	23-	9-
4	15	15	12-	9
5	17	18	8-	16-
H= 5 K= 4				
0	26	26	20	17
1	19	17	3-	17-
2	53	54	14-	53-
3	43	45	45-	4-
H= -5 K= 0				
1	32	32	32-	0
2	41	39	39-	0
3	27	28	28-	0
4	4*	3	3-	0
5	4*	6	6-	0
6	46	46	46-	0
H= -5 K= 1				
1	61	62	25	56
2	17	16	16-	2-
3	52	53	49-	20-
4	47	47	43-	19
5	33	34	5-	33
6	46	48	29	38
H= -5 K= 2				
1	35	36	5	35
2	11	11	10-	5
3	48	50	2-	50-
4	75	79	5	78-
5	36	38	6	37-
6	28	29	24	16-
H= -5 K= 3				
1	28	29	28-	9
2	22	23	21-	9
3	48	49	37-	32
4	39	39	31-	24
5	24	25	20-	15
H= -5 K= 4				
1	32	33	12	32
2	24	28	15	23
3	11	9	1-	9-

with a thin film of Krylon plastic spray to retard decomposition. The $\theta/2\theta$ scanning mode and variable scan-width was used with Cu $K\alpha$ radiation (λ= 1·5418 Å); 1161 reflections were measured, of which 37 were less than three standard deviations above background. Absorption corrections were applied in the reduction to structure amplitudes, using an IBM 1130 program (Craven, 1963) based on the procedure of Busing & Levy (1957).

Structure determination and refinement

The phases of 204 largest normalized structure amplitudes were obtained by application of the symbolic addition and tangent formula (Karle & Hauptman, 1956; Karle & Karle, 1966). An IBM 7090 version of the Hall (1968) program was used. The $|E|$ values and starting phases for the tangent refinement were 106, 3·47, π; 105, 3·07, π; $\bar{1}14$, 2·25, π; $\bar{1}06$, 2·92, 0; $\bar{3}54$, 2·90, π; $\bar{2}31$, 2·83, $\pi/2$, of which the first three were arbitrarily assigned.

A three-dimensional E map with 201 terms showed twelve peaks which were consistent with the carbon and oxygen atoms of a stereochemically reasonable model for the glucitol molecule. A more complete Fourier synthesis revealed the pyridine molecule. The carbon and oxygen parameters were refined by a block-diagonal least-squares calculation on an IBM.1130 computer (Shiono, 1968) to an R value of 0·05. The hydrogen atoms were located on difference syntheses at R values of 0·10 and 0·07. All parameters except the thermal parameters of the hydrogen atoms were refined by full-matrix least squares on an IBM 7090 computer (Shiono, 1966) to a final R of 0·042. The H thermal parameters used were those of the atoms to which they are attached. The function minimized was

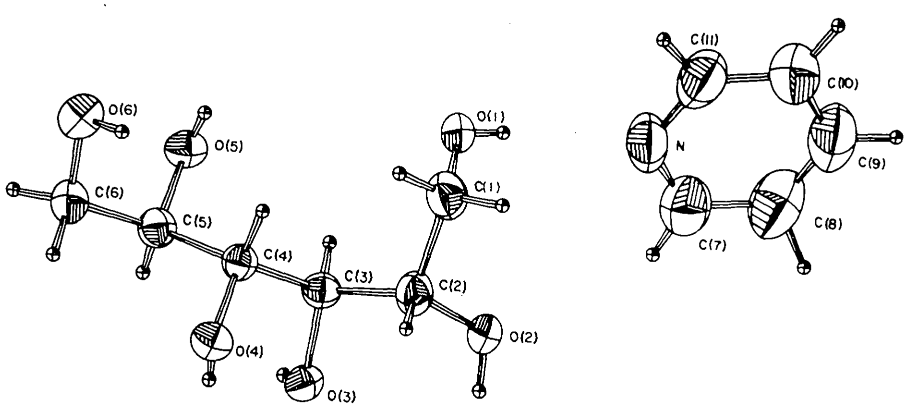


Fig. 1. *ORTEP* plot of one asymmetric unit of the glucitol–pyridine complex, showing atomic notation and thermal ellipsoids.

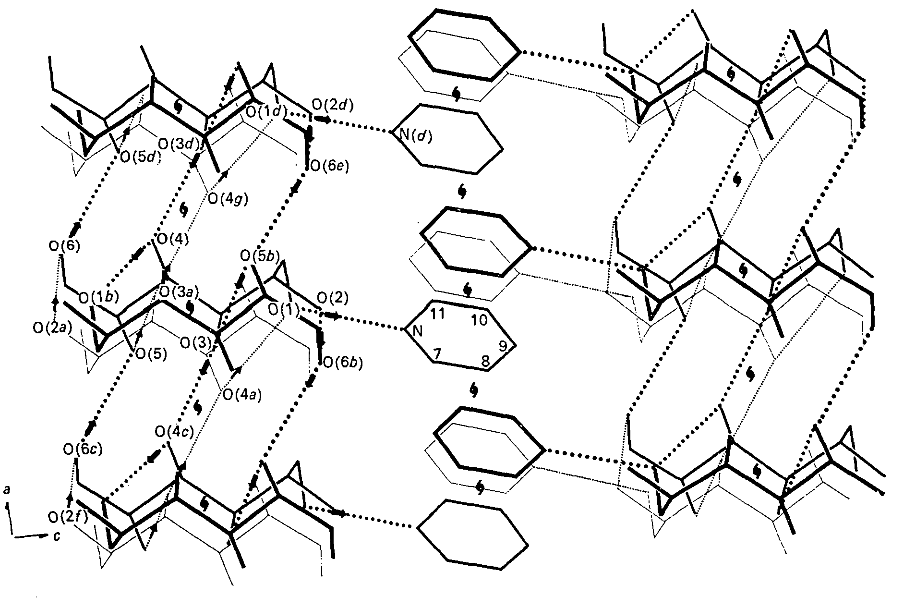


Fig. 2. View of the crystal structure of glucitol–pyridine down the b axis. Dotted lines are hydrogen-bonds; arrows indicate donor direction.

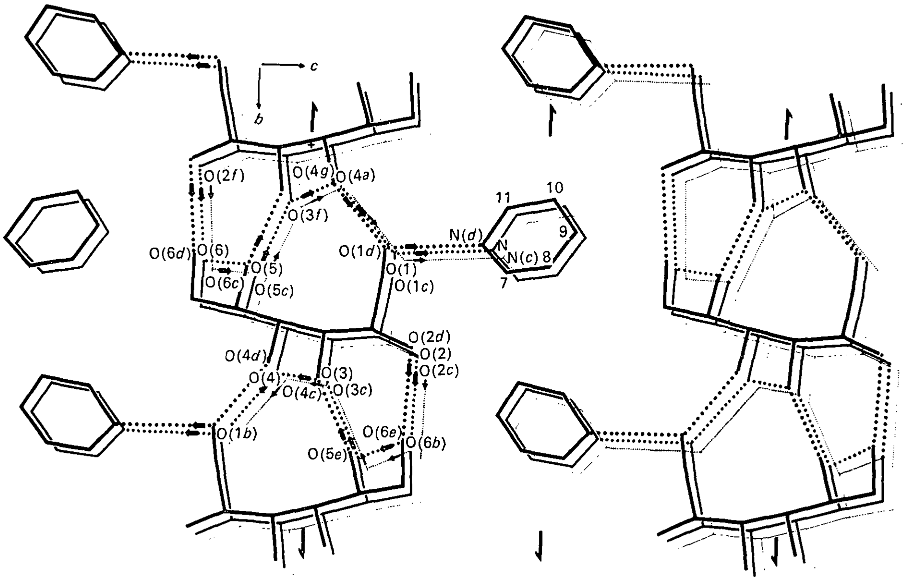


Fig. 3. View of the crystal structure of glucitol–pyridine down the *a* axis. Dotted lines are hydrogen-bonds; arrows indicate donor direction.

$\omega_i(|F_o|-|KF_c|)^2$ where $\omega_i=|F_o|^{-2}$ for $|F_o|\geq|4F_{\text{min}}|$ and

$$|4F_{\text{min}}|^{-2} \text{ for } |F_o|<|4F_{\text{min}}| \; : \; |F_{\text{min}}| \text{ was } 9{\cdot}64.$$

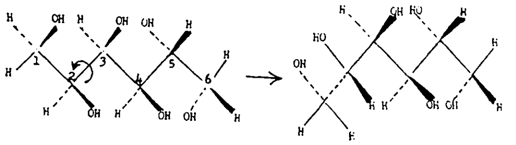
The final parameters are given in Table 1 and the corresponding structure factors in Table 2. The atomic notation is shown in Fig. 1. The atomic scattering factors were those by Cromer & Waber (1965) for C, N and O and by Stewart, Davidson & Simpson (1965) for H.

Discussion

The crystal structure is illustrated in Figs. 2 and 3. It consists of layers of glucitol molecules separated by layers of pyridine molecules, the packing of which shows a marked similarity to that in the structure of the $C_6H_5N.ICl$ complex (Hassel & Rømming, 1956, 1957), and in the hydrochloride, $C_6H_5N.HCl$ (Rérat, 1962). The other pyridine complex structure which has been studied, $C_6H_5N.BF_3$, shows no obvious similarity in packing (Zvonkova, 1956). The layers are parallel to (001) and one molecule thick. The glucitol molecules are hydrogen-bonded within their layers with a parallel alignment of the molecules, which is characteristic of the crystal structures of the alditols. The pyridine molecules are aligned so that their rings are stacked parallel in columns in the **a** direction and alternately inclined in a herringbone arrangement in the **b** direction. The closest approaches between pyridine atoms are H(C7)···H(C10), 2·79 Å, and C(8)···C(11), 3·60 Å. Other shortest van der Waals separations in the structure are between the glucitol and pyridine atoms and are O(6)···C(9) = 3·32, O(2)···C(10) = 3·44, O(6)···C(8) = 3·60 Å.

The glucitol and pyridine layers are associated by a O–H···N hydrogen bond of 2·814 Å to form a very simple type of hydrogen-bonded complex consisting of alternate layers of the two species of molecules. As in the case of the glucose–urea (*cf.* Snyder, Rosenstein, Kim & Jeffrey, 1970), the stability of the crystalline complex implies the formation of a system of intermolecular hydrogen bonds and van der Waals interactions with a lattice energy greater than that of the separate structures of like molecules. It is a general rule in the alditol crystal structures, that the number of intermolecular hydrogen bonds per molecule is equal to the number of hydroxyl groups, since each oxygen atom is involved in two hydrogen bonds, one as donor and one as acceptor. In this structure, each oxygen atom forms a donor and acts as an acceptor except O(2)H, which forms only a donor bond to O(6) on an adjacent molecule. This is compensated by the bond from O(1)H to the pyridine nitrogen atom. Therefore, instead of six O–H···O bonds per molecule, as in D-glucitol for example, there are in the complex, five O–H···O and one O–H···N bonds. The O···O hydrogen bond distances have values (between 2·744 and 2·906 Å Table 3) which extend over a wider range about a greater mean value than in the alditols, and are presumably correspondingly weaker. These hydrogen bonds form finite chains, which start with O(2), linking six glucitol molecules in the donor–acceptor sequence, and terminate at the pyridine nitrogen atom, *i.e.* O(2) → O(6) → O(5) → O(3) → O(4) → O(1) → N. In the alditols, the hydrogen bonding generally forms infinite chains or closed loops. The pyridine molecules in the layers pack in a herringbone arrangement similar to that in benzene (Cox, Cruickshank & Smith, 1958) and other small aromatic molecules. That this normal type of van der Waals packing can be accommodated between the layers of hydrogen-bonded glucitol molecules so as to permit the formation of O–H···N bonds would seem to be the most simple geometrical explanation for the particular stability of this 1:1 complex.

The glucitol molecule has a non-planar carbon-chain conformation, derived from the planar zigzag extended chain arrangement by a rotation of 120° about C(2)–C(3) as shown below:



The equations for the two planes containing the carbon atoms are given in Table 4, and the detailed conformation angles about the C–C bonds are given in Fig. 4. This conformation is consistent with the observation that the carbon-chain conformations in the crystal structures of pentitols and hexitols are those which avoid the parallel alignment of C(n)–O and C(n+2)–O bonds, *i.e.* C(1)–O and C(3)–O, C(2)–O and C(4)–O in this case. Planar zigzag carbon chains are therefore found in DL-arabinitol (Hunter & Rosenstein, 1968), D-mannitol (Berman, Jeffrey & Rosenstein, 1968; Kim, Jeffrey & Rosenstein, 1968), galactitol (Berman & Rosenstein, 1968), and non-planar chains in D-glucitol (Jeffrey & Park, 1970), ribitol (Kim, Jeffrey & Rosenstein, 1969) and xylitol (Kim & Jeffrey, 1969). This general relationship between configuration and conformation in the polyols is discussed in detail by Jeffrey & Kim (1970).

The interatomic distances and angles are given in Table 5. In the glucitol molecule, the mean C–C and C–O distances are 1·530 and 1·425 Å. The spread of ±0·011 and ±0·006 Å, respectively, about these mean values is not significant. The C–C–C and C–C–O angles are 110·8 to 114·0° with a mean of 112·6°, and

Table 3. *Hydrogen bond distances and angles in glucitol–pyridine*

i	j	k	l	D_{jk}	$\angle ijk$	$\angle lkj$
C(1)	O(1) →	N	C(7)	2·814 Å	105·6°	126·5°
		N	C(11)			117·0
C(2)	O(2) →	O(6*b*)	C(6*b*)	2·906	119·9	124·9
C(3)	O(3) →	O(4*c*)	C(4*c*)	2·755	103·2	122·1
C(4)	O(4) →	O(1*b*)	C(1*b*)	2·794	109·7	140·3
C(5)	O(5) →	O(3*a*)	C(3*a*)	2·822	107·7	112·9
C(6)	O(6) →	O(5*d*)	C(5*d*)	2·744	111·5	115·9

Symmetry code

	x	y	z
a	$1-x$	$-\frac{1}{2}+y$	$-z$
b	$1-x$	$\frac{1}{2}+y$	$-z$
c	$-1+x$	y	z
d	$1+x$	y	z
e	$-1+x$	y	$1+z$
f	$1-x$	$-\frac{1}{2}+y$	$1-z$

→: direction of O–H···O bond

Table 4. *Least-squares planes in glucitol–pyridine*

Equation for plane: $Ax+By+Cz=D$, where x,y,z are in Å.

	Atoms in plane		Distance from the best plane	Constant
Glucitol		C(1)	−1·527 Å	$A=$ 0·1098
	C(2)		−0·125	$B=$ 0·9798
	C(3)		0·101	$C=$ −0·1675
	C(4)		0·146	$D=$ 5·3549
	C(5)		−0·092	
	C(6)		−0·030	
	O(2)		0·042	
Glucitol	C(1)		0·000	$A=$ −0·0987
	C(2)		0·000	$B=$ 0·2010
		C(3)	−1·215	$C=$ 0·9751
	O(1)		0·000	$D=$ 2·7437
		O(2)	1·206	
Pyridine	C(7)		0·009	$A=$ 0·7630
	C(8)		−0·004	$B=$ 0·5942
	C(9)		−0·003	$C=$ 0·2505
	C(10)		0·005	$D=$ 4·1809
	C(11)		0·000	
	N		−0·007	

106·9 to 112·9° with a mean of 110·1° respectively. These results are in agreement with those observed in the other polyols.

In the pyridine molecule the C–C mean bond length is 1·367 Å, with a spread of −0·008 to +0·015 Å. This distance is 0·02 to 0·03 Å shorter than has been reported in pyridine oxide hydrochloride (Tsoucaris, 1961), pyridine hydrochloride (Rérat, 1962) and pyridinium dicyanomethylide (Bugg & Sass, 1965). The C–N bonds are 1·329 and 1·330 Å, and the C–N–C angle of 116·5° is the smallest within the ring. The ring is planar within ±0·009 Å. The C–H distances varied from 0·88 to 1·16 Å and the O–H distances from 0·68 to 0·89 Å. The angles involving hydrogen atoms, (*i.e.* X–Y–H) varied from 103 to 125°. Those associated with the pyridine ring grouped around 120°, while the remaining were closer to 110°. No other significance was associated with these variations.

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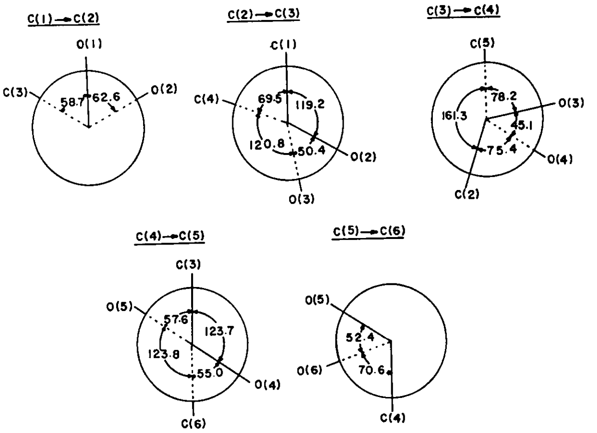

Fig. 4. Conformation angles about the C–C bonds in the glucitol molecule of the glucitol–pyridine complex.

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Table 5. *Intramolecular distances and angles in glucitol–pyridine*

The estimated standard deviations given in parentheses refer to the last decimal positions of respective values. The mean values are calculated from

$$\bar{l}=\sum_i\left(\frac{l_i}{\sigma_i^2}\right)\Big/\sum_i\left(\frac{1}{\sigma_i^2}\right),\ \sigma\ \text{mean}=\left(\frac{\sum_i(\bar{l}-l_i)^2}{N-1}\right)^{\frac{1}{2}}$$

i	*j*	D_{ij}	D_{ij}*
C(1)	C(2)	1·519 (4) Å	1·520 Å
C(2)	C(3)	1·536 (4)	1·537
C(3)	C(4)	1·534 (4)	1·536
C(4)	C(5)	1·535 (4)	1·535
C(5)	C(6)	1·528 (4)	1·530
C(1)	O(1)	1·419 (4)	1·420
C(2)	O(2)	1·420 (3)	1·421
C(3)	O(3)	1·429 (3)	1·431
C(4)	O(4)	1·427 (3)	1·429
C(5)	O(5)	1·430 (3)	1·432
C(6)	O(6)	1·421 (3)	1·422
C(7)	C(8)	1·364 (7)	1·364
C(8)	C(9)	1·359 (6)	1·360
C(9)	C(10)	1·364 (6)	1·365
C(10)	C(11)	1·382 (6)	1·382
C(11)	N	1·329 (6)	1·330
C(7)	N	1·330 (6)	1·331

i	*j*	*k*	*ijk*
C(1)	C(2)	C(3)	112·3 (2)°
C(2)	C(3)	C(4)	113·2 (2)
C(3)	C(4)	C(5)	110·8 (2)
C(4)	C(5)	C(6)	114·0 (2)
O(1)	C(1)	C(2)	112·6 (2)
C(1)	C(2)	O(2)	106·9 (2)
O(2)	C(2)	C(3)	110·5 (2)
C(2)	C(3)	O(3)	108·3 (2)
O(3)	C(3)	C(4)	108·9 (2)
C(3)	C(4)	O(4)	111·2 (2)
O(4)	C(4)	C(5)	110·5 (2)
C(4)	C(5)	O(5)	108·9 (2)
O(5)	C(5)	C(6)	110·4 (2)
C(5)	C(6)	O(6)	112·9 (2)
C(7)	C(8)	C(9)	119·7 (4)
C(8)	C(9)	C(10)	118·8 (4)
C(9)	C(10)	C(11)	118·1 (4)
C(10)	C(11)	N	123·7 (4)
C(11)	N	C(7)	116·5 (4)
N	C(7)	C(8)	123·2 (4)

Glucitol

$\bar{l}_{\text{C–C}}$	= 1·530 Å	σmean = 0·007 Å
$\bar{l}_{\text{C–O}}$	= 1·425	0·005
∠C–C–C	= 112·6°	1·4°
∠C–C–O	= 110·1	1·9

Pyridine

$\bar{l}_{\text{C–C}}$	= 1·367 Å	0·011 Å
∠C–C–C	= 118·9°	0·8°

* Bond distances corrected for libration.

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The Crystal Structure of the Potassium Methoxide Adduct of 4-Methoxy-5,7-Dinitrobenzfurazan, a Meisenheimer Complex

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The crystal and molecular structure of the potassium methoxide adduct of 4-methoxy-5,7-dinitrobenzfurazan, $K^+[(CH_3O)_2C_6H(NO_2)N_2O]^-$, has been determined by X-ray diffraction techniques. The complex crystallizes as red, monoclinic crystals with $a=15{\cdot}152\pm0{\cdot}015$, $b=9{\cdot}307\pm0{\cdot}003$, $c=17{\cdot}780\pm0{\cdot}010$ Å and $\beta=106{\cdot}22\pm0{\cdot}08°$, measured at room temperature. The space group is $P2_1/c$ and with eight molecules per unit cell, $D_c=1{\cdot}712$ g·cm^{-3} compared with $D_m=1{\cdot}718$ g.cm^{-3}. The structure was solved using the symbolic addition procedure and Fourier syntheses. A refinement by full-matrix least-squares methods converged to a final R of 0·068 for the 4013 observed reflections measured using the stationary-crystal stationary-counter technique with molybdenum radiation. The two methoxyl groups are covalently bonded to the same ring carbon atom with an average C–O distance of $1{\cdot}415\pm0{\cdot}006$ Å. The sp^3 hybridized carbon atom in the ring produces distortions throughout the entire molecule. The electron withdrawing power of the coplanar furazan ring has a pronounced effect on the benzene ring system.

Introduction

Meisenheimer complexes, the stable intermediates in aromatic nucleophilic substitution reactions, have been of interest (Fendler, Fendler & Griffin, 1969; Crampton & Gold, 1966; Foster & Fyfe, 1966). The question is whether the intermediate formed by the attack of an alkoxide ion on nitroaryl compounds is a covalently bonded species or a charge-transfer complex. The recent crystal structure determinations of 1,1′-dimethoxy-2,4,6-trinitrobenzene potassium dihydrate by Ueda, Sakabe, Tanaka & Furusaki (1968) and the ethoxide adducts of 2,4,6-trinitrophenetole by Destro, Gramaccioli & Simonetta (1968) have confirmed the covalent nature of the intermediate.

The present study was undertaken in 1965 but was not completed until recently. The benzfurazan ring alters the possible resonance structures and the molecular geometry is distinctly different from the two previous studies. A preliminary account of our results has appeared recently (Messmer & Palenik, 1969).

Experimental

Orange-red platelets of the potassium methoxide adduct of 4-methoxy-5,7-dinitrobenzfurazan (KDMDNBF) were kindly supplied by Dr W. P. Norris. Preliminary Weissenberg photographs indicate that the crystals are monoclinic with the systematic absences of $h0l$ for $l=2n+1$ and $0k0$ for $k=2n+1$, indicating that the space group is $P2_1/c$.

A crystal of dimensions $0{\cdot}23\times0{\cdot}22\times0{\cdot}10$ mm which was dipped in liquid nitrogen to minimize extinction

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