and the carboxyl group 23·4° from the aromatic plane. The exocyclic carbon atom and the nitrogen atom are displaced 0·209 and 0·160 Å respectively from the aromatic plane in opposite directions. The carbon atoms C(1) and C(2) are displaced respectively 0·013 and 0·020 Å from the least-squares plane; the displacements of these atoms are, as expected, small and in the same directions as the atoms C(7) and N respectively (Table 4). The C(1)–C(7) and C(2)–N bonds are displaced sideways as well (Fig. 2).

The dimensions of the carboxyl group and the nitro group in *o*-nitrobenzoic acid show only minor differences from those in *p*-nitrobenzoic acid (Fig. 2).

The projections of the structure along the *b* and *a* axes are shown in Figs. 3 and 4 respectively, in which the principal intermolecular distances are also shown. The *o*-nitrobenzoic acid dimers are held in a stable structure by normal van der Waals interactions. The hydrogen bonds forming dimers are 2·645 Å in length (2·653 Å in *p*-nitrobenzoic acid).

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The Crystal Structure of D-Galactono-γ-lactone

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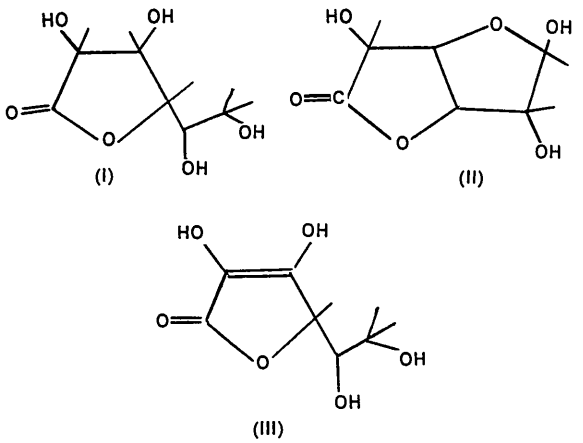
The crystal structure of D-galactono-γ-lactone, $C_6H_{10}O_6$, has four molecules in a cell, $a=6{\cdot}746$, $b=10{\cdot}67$, $c=10{\cdot}98$ Å, with space group $P2_12_12_1$. The structure was determined from three-dimensional photographic data by means of a resolved centrosymmetric projection and the Harker sections of the Patterson synthesis. The molecule consists of a 1,4 lactone ring to which is attached a $CHOH.CH_2OH$ chain. While the lactone group, C–C–O–C, is planar, the fifth atom of the lactone ring is 0·64 Å out of this plane, forming the puckered furan-type configuration very similar to that found in furanose sugars. The C–O bond adjacent to the carbonyl is 0·10 Å shorter than the other C–O bonds, which do not differ significantly from a mean of 1·421 Å. The intermolecular hydrogen bonding forms a three-dimensional network which includes all the hydroxyl groups as donors and acceptors, but excludes the two oxygen atoms of the lactone group.

Introduction

This crystal structure determination of D-galactono-γ-lactone forms part of a program aimed at providing data pertaining to the effects of intermolecular and intramolecular environments on the stereochemistry of groups of atoms commonly found in the carbohydrates and their derivatives. In this structure (I), and that of D-glucurono-γ-lactone (II) described in the following paper (Kim, Jeffrey, Rosenstein & Corfield, 1967), the lactone group C–C(=O)–O–C is observed in a 1,4 lactone ring, which in the former is attached to a chain and in the latter is fused to a furanose ring. Therefore, the postulate that this group is generally planar, by reason of the valence-bond resonance form $C{-}C{=}O^{+}{-}C$ / O^{-} (*cf.*

Mathieson, 1963), can be examined in detail in these rings, one of which is much more likely to be strained than the other. This same group occurs in what is also likely to be a strained ring system in L-ascorbic acid (III), which Hvoslef (1964, 1966) is studying by X-ray and neutron diffraction.



Crystal data

The crystal data on D-galactono-1,4-lactone, $C_6H_{10}O_6$, obtained from Koch-Light Laboratories Ltd., are as follows:

$a=6{\cdot}746\pm0{\cdot}005$, $b=10{\cdot}67\pm0{\cdot}01$, $c=10{\cdot}98\pm0{\cdot}01$ Å; Space group $P2_12_12_1$ (no. 19) from systematic extinctions: $h00$ with h odd, $0k0$ with k odd, $00l$ with l odd; $D_m=1{\cdot}57\pm0{\cdot}02$ g.cm^{-3}, $Z=4$, M.W. 178·14, $D_x=1{\cdot}56$ g.cm^{-3}.

The cell dimensions were measured from NaCl-calibrated zero-layer Weissenberg photographs, and the density was determined by flotation.

Experimental

Prismatic crystals of $0{\cdot}2\times0{\cdot}2\times0{\cdot}2$ mm^3 were used to record all layers up to $\mu=40°$ about the three principal axes on multiple-film Weissenberg photographs with Cu $K\alpha$ radiation. The intensities were visually estimated with the use of a standard intensity scale, and were correlated and reduced to structure amplitudes with an IBM 7090 program (Shiono, 1966), based on the method of Hamilton, Rollett & Sparks (1965). Of the 1066 reflections within the Cu $K\alpha$ sphere, 1006 were recorded, including 34 which were too weak to be estimated and were given half the minimum observed value.

The structure determination and refinement

Since the structure is centrosymmetric in projection, the IBM 1620 sign-correlation procedure of Beurskens (1964) was applied to the equatorial data taken about the shortest axis. The signs of 44 out of 136 $0kl$ structure factors were found, of which only one, of lowest probability, proved to be incorrect. The resulting E map, shown in Fig. 1(*a*), was readily interpreted, once the five-membered ring was recognized. The initial ($0kl$) structure factor calculation gave an $R=0{\cdot}36$, which was reduced to 0·20 on refinement of the projection by full-matrix least squares, with Shiono's IBM 7090 version (1966) of the Busing, Martin & Levy program (1962). A model of the structure, based on the known projection [*cf.* Fig. 1(*b*)], and assuming reasonable inter- and intra-molecular distances, showed that the essentially planar lactone ring was approximately parallel to the yz plane. An x coordinate for these atoms was derived from the Harker sections at $(u,\frac{1}{2},w)$ and $(u,v,\frac{1}{2})$ calculated on the IBM 1620 computer, and a set of x coordinates for the remaining atoms was obtained from the model. Full-matrix least-squares refinement of the resulting set of three-dimensional positional parameters, together with individual isotropic temperature factors, reduced the R index to 0·27, but there the refinement terminated owing to an incorrect x parameter of the terminal oxygen, O(6). The correction of this parameter, which corresponded to a rotation around the C(5)–C(6) bond, permitted the isotropic refinement to proceed to an $R=0{\cdot}14$. The introduction of anisotropic temperature factors then further improved the agreement to $R=0{\cdot}09$. A three-dimensional difference synthesis calculated with the Zalkin Fourier program (Shiono, 1966), using all the reflections, was then inspected for hydrogen atoms, and peaks varying from 0·8 to 0·3 e.Å^{-3} were found in stereochemically reasonable positions, significantly above the background variations, which were about $\pm0{\cdot}05$ e.Å^{-3}. The positional parameters of these atoms were refined by a cycle of least squares holding other parameters fixed and using as hydrogen thermal parameters those of the atoms to which they are bonded. A final cycle of refinement with the hydrogen parameters fixed gave an R value of 0·078, including all reflections. The final parameters are given in Tables 1, 2 and 3, and the corresponding structure factors in Table 4. Throughout the analysis, the atomic scattering factors used were those of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for carbon and oxygen and of McWeeny (1951) for hydrogen.

Discussion of the structure

The interatomic distances and angles are given in Tables 5 and 6, employing the following notation:

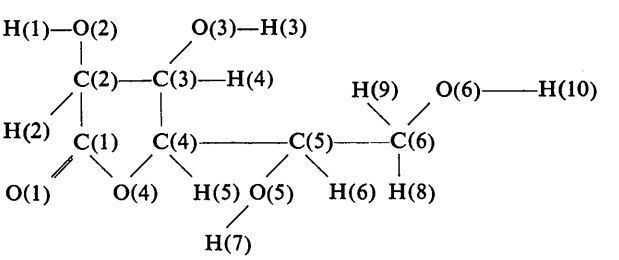


Table 1. *Fractional atomic coordinates in galactono-γ-lactone*

The estimated standard deviations in parentheses refer to the last decimal positions of respective values.

	x	y	z		x	y	z
C(1)	0·2709 (7)	0·2345 (4)	0·0277 (4)	H(1) [O(2)]	0·381 (14)	−0·021 (7)	−0·101 (7)
C(2)	0·2728 (7)	0·0930 (4)	0·0451 (4)	H(2) [C(2)]	0·133 (14)	0·064 (8)	0·021 (7)
C(3)	0·3347 (7)	0·0803 (4)	0·1780 (3)	H(3) [O(3)]	0·339 (14)	−0·096 (7)	0·220 (7)
C(4)	0·2253 (7)	0·1963 (4)	0·2323 (4)	H(4) [C(3)]	0·499 (12)	0·084 (7)	0·199 (7)
C(5)	0·3122 (7)	0·2452 (4)	0·3537 (4)	H(5)]C(4)]	0·079 (12)	0·169 (6)	0·259 (7)
C(6)	0·1959 (8)	0·3606 (4)	0·3916 (4)	H(6) [C(5)]	0·286 (14)	0·193 (7)	0·415 (7)
O(1)	0·2989 (6)	0·2932 (3)	−0·0638 (3)	H(7) [O(5)]	0·637 (14)	0·206 (7)	0·359 (7)
O(2)	0·4060 (5)	0·0303 (3)	−0·0355 (3)	H(8) [C(6)]	0·254 (14)	0·435 (7)	0·340 (7)
O(3)	0·2627 (6)	−0·0281 (3)	0·2382 (3)	H(9) [C(6)]	0·053 (14)	0·331 (7)	0·418 (7)
O(4)	0·2535 (6)	0·2915 (3)	0·1378 (3)	H(10)[O(6)]	0·323 (17)	0·466 (9)	0·519 (8)
O(5)	0·5259 (5)	0·2725 (3)	0·3440 (3)				
O(6)	0·2300 (8)	0·3926 (4)	0·5181 (3)				

Table 2. *Anisotropic thermal parameters in galactono-γ-lactone*

The temperature factor expression used was $\exp[-10^{-4}(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})]$, and the estimated standard deviations in parentheses refer to the last digit of respective values.

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	76 (9)	26 (4)	21 (3)	4 (5)	−13 (5)	1 (3)
C(2)	72 (9)	24 (3)	14 (3)	−1 (5)	0 (5)	−1 (2)
C(3)	82 (9)	15 (3)	11 (3)	−1 (5)	−3 (4)	−1 (2)
C(4)	61 (8)	14 (3)	24 (3)	8 (4)	−9 (5)	0 (2)
C(5)	77 (9)	12 (3)	22 (3)	−6 (5)	0 (5)	−1 (3)
C(6)	79 (10)	34 (4)	29 (3)	3 (6)	7 (5)	−9 (3)
O(1)	127 (8)	38 (3)	24 (2)	4 (4)	−4 (4)	9 (2)
O(2)	100 (7)	32 (3)	18 (2)	9 (4)	−8 (4)	−5 (2)
O(3)	110 (7)	16 (2)	23 (2)	−3 (4)	16 (4)	0 (2)
O(4)	120 (8)	19 (2)	23 (2)	13 (4)	−13 (4)	0 (2)
O(5)	60 (7)	16 (2)	30 (3)	−6 (3)	−9 (4)	8 (2)
O(6)	171 (10)	41 (3)	34 (3)	−28 (5)	20 (5)	−3 (2)

Table 3. *Principal axes of thermal ellipsoids in galactono-γ-lactone*

The root-mean-square displacement U_i corresponds to the ith principal axis of the ellipsoid and $\theta_{ia},\theta_{ib},\theta_{ic}$ are angles between the ith axis and the crystallographic axes a,b,c.

	i	U_i	θ_{ia}	θ_{ib}	θ_{ic}
C(1)	1	0·097 Å	54·83°	105·19°	39·25°
	2	0·124	91·23	162·12	107·83
	3	0·141	144·80	99·18	56·37
C(2)	1	0·093	88·76	84·26	5·87
	2	0·119	101·65	166·91	84·12
	3	0·125	11·72	101·72	90·04
C(3)	1	0·081	84·95	82·99	8·64
	2	0·094	90·77	172·85	82·90
	3	0·133	5·10	91·38	94·90
C(4)	1	0·079	117·34	29·44	100·07
	2	0·110	128·80	117·21	129·12
	3	0·133	129·01	100·38	40·90
C(5)	1	0·082	77·74	13·52	84·37
	2	0·116	87·09	84·88	174·11
	3	0·131	12·60	102·48	88·26
C(6)	1	0·111	119·17	57·00	46·95
	2	0·135	149·61	114·90	106·27
	3	0·156	97·74	43·49	132·45
O(1)	1	0·108	82·71	118·55	29·64
	2	0·159	81·22	148·91	119·55
	3	0·166	11·44	78·74	92·04
O(2)	1	0·100	80·50	74·87	17·98
	2	0·128	53·80	142·99	83·29
	3	0·158	37·81	57·15	106·60
O(3)	1	0·097	82·58	12·43	99·92
	2	0·107	66·22	102·14	152·94
	3	0·163	25·04	92·60	65·11
O(4)	1	0·097	110·07	28·51	109·38
	2	0·114	100·75	114·20	153·22
	3	0·169	22·99	75·84	107·74
O(5)	1	0·086	81·37	21·57	109·61
	2	0·106	154·84	90·42	115·15
	3	0·149	113·44	68·43	32·79
O(6)	1	0·129	120·96	123·07	48·59
	2	0·144	97·45	135·46	133·57
	3	0·211	32·05	116·13	72·78

The C–C bond lengths vary from 1·501 to 1·546 Å and are not significantly different from the mean value of 1·525 Å. The C–O bond lengths other than those in the lactone ring vary from 1·405 to 1·447 with a mean value of 1·430 Å. There is a suggestion that the $-CH_2OH$ (1·447 Å) may be longer than the $>CH-OH$ (mean 1·411 Å), but the difference is barely significant and requires verification from other structures. In the lactone ring, the two C–O bond lengths, C(1)–O(4) and O(4)–C(4) differ by 0·106 Å, which is nine times 2σ.

Table 4. *Observed and calculated structure factors*

Columns are: index, $|F_{obs}|$, $|F_{cal}|$, A_{cal}, B_{cal}. * indicates unobserved reflections.

K= 0 H= 0

2	298	299	299	0
4	184	197	197	0
6	196	196	198	0
8	158	147	147	0
10	306	359	359-	0
12	45	37	37-	0
14	80	86	86-	0

K= 1 H= 0

1	284	228	0	228
2	253	289	0	289
3	278	374	0	374
4	277	314	0	310-
5	28	35	0	35
6	19	16	0	16
7	253	283	0	283-
8	191	201	0	201-
9	90	78	0	78-
10	99	98	0	98-
11	133	125	0	125-
12	99	85	0	85-
13	76	66	0	66-

K= 2 H= 0

0	598	517	517-	0
1	28	27	27-	0
2	37	46	46	0
3	28	23	23-	0
4	587	648	648	0
5	347	366	366-	0
6	140	151	151-	0
7	285	298	298-	0
8	55	51	50-	0
9	19	24	24	0
10	130	128	128	0
11	9	9	9	0
12	11	14	14-	0
13	48	36	36-	0

K= 3 H= 0

1	119	140	0	140-
2	244	237	0	237-
3	362	391	0	391-
4	285	329	0	329-
5	54	49	0	49
6	200	193	0	193-
7	84	83	0	83
8	35	35	0	35-
9	258	292	0	292-
10	31*	19	0	19
11	34	24	0	24
12	39	29	0	29-
13	72	49	0	49-

K= 4 H= 0

0	560	551	551	0
1	50	38	38	0
2	244	196	196-	0
3	197	195	195-	0
4	74	61	61-	0
5	562	553	553	0
6	30	29	29	0
7	117	119	119-	0
8	73	67	67	0
9	18	16	16	0
10	232	265	265-	0
11	42	50	50-	0
12	28	18	18	0
13	15*	8	8-	0

K= 5 H= 0

1	310	320	0	320
2	143	137	0	137-
3	323	367	0	367
4	85	76	0	76
5	33	4	0	4
6	168	178	0	178
7	60	57	0	57-
8	127	110	0	110-
9	22*	5	0	5
10	36	24	0	24
11	59	49	0	49
12	87	81	0	81-
13	174	196	0	196-

K= 6 H= 0

0	304	350	350-	0
1	187	162	162	0
2	143	136	136-	0
3	39	44	44-	0
4	114	100	100	0
5	106	92	92-	0
6	328	348	348-	0
7	135	118	118	0
8	100	93	93	0
9	228	243	243	0
10	65	80	80	0
11	4	2	2	0
12	4	10	10-	0

K= 7 H= 0

1	214	242	0	242
2	100	101	0	101-
3	32	23	0	23-
4	61	44	0	44-
5	28	30	0	30
6	271	298	0	298-
7	172	165	0	165
8	124	125	0	125-
9	28	22	0	22-
10	87	98	0	98
11	61	53	0	53-

K= 8 H= 0

0	58	55	55	0
1	163	163	163	0
2	121	122	122	0
3	65	63	63	0
4	30	28	28-	0
5	468	463	463	0
6	112	115	115	0
7	126	119	119	0
8	82	94	94	0
9	62	69	69	0
10	22*	15	15	0
11	58	60	60	0

K= 9 H= 0

1	146	158	0	158-
2	47	50	0	50
3	269	313	0	313
4	67	54	0	54
5	48	33	0	33
6	84	86	0	86
7	46	35	0	35
8	34	30	0	30
9	4	10	0	10
10	54	47	0	47-

K=10 H= 0

0	129	148	148	0
1	224	255	255	0
2	36	36	36	0
3	48	51	51-	0
4	84	85	85	0
5	45	38	38-	0
6	24	33	33-	0
7	19*	5	5	0
8	27	29	29-	0
9	82	80	80	0

K=11 H= 0

1	95	100	0	100
2	23	22	0	22-
3	80	72	0	72-
4	20*	5	0	5-
5	99	101	0	101-
6	84	78	0	78
7	23	16	0	16*
8	3	17	0	17

K=12 H= 0

0	90	95	95-	0
1	34	36	36-	0
2	18*	4	4-	0
3	53	52	52-	0
4	26	26	26-	0
5	57	59	59	0
6	115	121	121	0

K=13 H= 0

1	14*	9	0	9-
2	105	113	0	113
3	84	81	0	81
4	28	18	0	18

K= 0 H= 1

1	261	277	0	277-
2	130	107	107-	0
3	188	168	0	168
4	307	316	316	0
5	402	420	0	420
6	267	264	264-	0
7	65	53	0	53-
8	140	148	148-	0
9	18	10	0	10-
10	228	235	235-	0
11	112	99	0	99
12	19*	12	12-	0
13	25	23	0	23-
14	92	111	111-	0

K= 1 H= 1

0	380	382	0	382
1	589	626	235-	580-
2	474	487	290-	391-
3	69	66	41-	51-
4	316	350	77	341
5	323	331	134-	303
6	167	152	29	149-
7	85	76	7	75
8	143	146	130-	67-
9	195	212	80	196-
10	146	154	23	152-
11	57	66	33	57-
12	102	99	99	1
13	68	51	50	5

K= 2 H= 1

0	170	145	0	145-
1	527	545	507-	200
2	126	111	54-	97-
3	302	318	240-	208
4	43	48	47-	7-
5	268	274	240-	132-
6	98	102	87-	54-
7	183	170	56-	160
8	140	120	118	21
9	166	163	44-	157
10	169	159	156	33-
11	54	45	45-	0
12	77	77	74	23
13	108	104	104	6

K= 3 H= 1

0	577	644	0	644
1	287	299	227-	195
2	325	391	353	168
3	230	233	170-	160-
4	258	252	244-	61-
5	39	39	4	39
6	132	134	115-	70-
7	93	86	59	63-
8	107	98	62	75
9	274	324	120	324
10	117	112	98-	54-
11	89	88	33	82
12	62	65	62-	17-
13	65	58	50	29

K= 4 H= 1

0	320	364	0	364-
1	533	546	524-	153-
2	65	47	34	31-
3	34	41	35-	21
4	154	160	160	12-
5	220	248	21	247
6	207	193	172	87-
7	153	131	116-	61
8	189	188	176	68
9	216	241	239	32-
10	106	116	86-	77
11	59	65	10	64
12	20*	3	2-	1
13	73	70	70	3

K= 5 H= 1

0	56	51	0	51
1	121	118	101-	60
2	240	230	212-	89-
3	115	104	17-	103-
4	393	402	16-	402
5	188	200	29-	198
6	40	37	9	35-
7	245	229	107-	202-
8	250	261	110-	237
9	37	51	1-	50-
10	97	88	34-	81
11	40	32	28	14-
12	83	71	36	60

K= 6 H= 1

0	103	102	0	102
1	331	370	309	204
2	173	162	123	105-
3	129	122	106-	60
4	222	218	184	117-
5	139	149	136-	60-
6	176	184	184	1
7	125	121	49	110
8	143	147	145	24
9	62	56	2	56
10	25*	17	5	16-
11	57	52	48-	20-
12	178	177	167	58

K= 7 H= 1

0	40	28	0	28-
1	164	159	37	155
2	40	29	18	23
3	174	168	156-	60
4	157	144	122-	76-
5	129	114	26	111
6	141	134	125-	49-
7	122	104	68	78
8	9	18	8-	16
9	110	102	39-	94
10	90	83	44-	70-
11	54	51	27	43-
12	39	42	42-	4-

K= 8 H= 1

0	145	138	0	138-
1	64	49	18-	45-
2	134	141	128	59-
3	117	114	114	8-
4	178	177	172-	39
5	215	228	228	8
6	144	150	148-	24-
7	62	61	56-	22-
8	28	40	40-	5
9	63	57	37	42-
10	71	66	17	64
11	46	43	4	43

K= 9 H= 1

0	102	93	0	93-
1	180	180	23-	179
2	88	81	71-	38-
3	182	211	77	196-
4	56	48	39	29
5	111	106	13-	105
6	48	46	44-	13-
7	51	55	55-	0
8	113	105	102-	25-
9	108	81	28	76
10	14	14	7	12-

K=10 H= 1

0	42	44	0	44
1	22*	20	11-	16
2	184	194	168-	98-
3	175	184	184-	13-
4	108	104	65	81-
5	28	21	6	20-
6	83	74	48	56
7	45	46	45-	5-
8	74	69	62-	29-
9	43	47	46-	5

K=11 H= 1

0	69	51	0	51
1	90	91	46	78
2	48	50	29-	40-
3	56	51	10-	49
4	91	85	40-	75-
5	121	111	28	107
6	93	85	67-	53-
7	108	107	44	97
8	53	49	19-	45-

K=12 H= 1

0	56	59	0	59-
1	60	58	32-	48-
2	23	22	6	21-
3	49	48	34-	34-
4	122	116	115-	11
5	52	46	43-	17-
6	117	139	124-	62-

K=13 H= 1

0	58	54	0	54
1	56	54	19-	50
2	116	108	3-	108
3	99	105	81	67-
4	55	55	9-	54-

K= 0 H= 2

0	1524	1426	1426-	0
1	636	620	0	620-
2	190	185	185-	0
3	120	99	0	99-
4	295	290	290-	0
5	94	90	0	90-
6	161	161	161	0
7	48	43	0	43
8	172	176	176-	0
9	91	96	0	96
10	124	121	121	0
11	14	15	0	15-
12	42	46	46	0
13	130	102	0	102

K= 1 H= 2

0	170	137	137	0
1	441	408	158	376-
2	238	251	180	175-
3	251	214	81-	198
4	80	59	9-	59-
5	157	156	153-	27-
6	202	183	27	181-
7	87	65	65-	8
8	136	136	112-	78
9	178	188	101-	159
10	89	72	68-	25-
11	151	160	65-	146
12	57	54	23-	49
13	43	47	15	44-

K= 2 H= 2

0	257	245	245	0
1	124	109	39	102-
2	336	342	342	9-
3	167	143	142	12
4	147	138	133-	35-
5	298	292	271	110-
6	88	84	25-	80
7	272	297	294	43-
8	50	45	0	45
9	73	64	63	12
10	109	94	29-	90-
11	56	49	5-	49
12	115	114	114-	5
13	48	41	24	33

K= 3 H= 2

0	36	21	21-	0
1	446	484	61-	480
2	152	131	89-	97-
3	301	290	290-	8
4	183	175	41	170
5	160	141	88-	110
6	117	110	83	71
7	165	142	103-	97
8	173	171	104-	136-
9	106	93	25-	90
10	71	67	16-	65-
11	75	76	38-	66-
12	28	28	1	28
13	72	77	5	77

K= 4 H= 2

0	24	11	11	0
1	193	171	164-	47-
2	247	244	242	28
3	275	282	226	168-
4	234	212	47-	206-
5	203	188	184-	37-
6	199	189	164	95-
7	125	127	115	53
8	85	78	72-	28-
9	55	51	22	45
10	89	89	80	40-
11	125	122	118	33
12	32	30	9-	29
13	18	20	14-	15

K= 5 H= 2

0	269	283	283	0
1	219	221	79-	206-
2	232	215	126	174
3	48	43	41	15
4	179	176	5-	176-
5	58	34	13	32
6	268	292	45	288-
7	96	92	40-	83-
8	135	128	12-	127
9	54	47	15-	45
10	86	79	21-	76-
11	56	78	47-	62-
12	49	44	43-	12-

K= 6 H= 2

0	121	107	107	0
1	34	23	23	1
2	275	277	276	9
3	130	135	132	30-
4	70	72	70	16
5	119	108	40	100
6	164	158	141	70-
7	68	63	60	21-
8	113	109	57-	93-
9	101	92	83-	39-
10	73	73	21-	70-
11	61	70	27-	64
12	62	72	72-	3

K= 7 H= 2

0	39	31	31	0
1	82	78	77-	14
2	192	200	150	132-
3	49	50	48-	14
4	186	183	51	155
5	53	37	2-	37
6	250	263	12	263
7	177	161	145-	70-
8	42	38	26-	27-
9	81	75	10	75-
10	44	48	12	46-
11	56	59	53	26-

K= 8 H= 2

0	20*	21	21	0
1	201	232	232-	11
2	171	162	129-	98-
3	79	66	3-	66-
4	129	123	37-	117-
5	196	188	188-	0
6	119	137	42-	130-
7	58	57	56-	8
8	76	85	60-	61-
9	118	106	71-	79-
10	54	52	50-	13-

K= 9 H= 2

0	21*	0	0	0
1	178	183	43	178
2	122	126	63	109
3	119	121	44	113-
4	146	147	76	126-
5	29	18	10	15-
6	111	117	39	111-
7	66	63	28	57-
8	102	103	64	80
9	35	29	25	13
10	59	51	47-	19

K=10 H= 2

0	168	180	180-	0
1	82	76	61-	45-
2	107	105	45-	95-
3	105	112	110	23
4	77	79	72-	32
5	50	33	32-	8-
6	33	28	7	27-
7	135	138	136	26-
8	87	78	26	74-
9	59	59	19	55

K=11 H= 2

0	27	17	17-	0
1	37	35	25-	25-
2	113	120	99	66-
3	127	122	10-	121
4	82	96	26	92
5	101	111	24	108
6	59	53	51-	15
7	79	71	68	18-

K=12 H= 2

0	59	57	57	0
1	63	66	48-	45
2	72	65	59-	26
3	62	51	48	19
4	14*	13	8	10-
5	99	94	76	55-

K=13 H= 2

0	73	68	68	0
1	84	97	81	52

Table 4 (*cont.*)

2	22	23	6	22
3	74	8.	21	77-
	K= 0 H= 3			
1	52	44	0	44-
2	147	158	158	0
3	175	154	0	154-
4	152	140	140	0
5	56	41	0	41-
6	301	314	314	0
7	93	100	0	100
8	175	161	161	0
9	54	53	0	53
10	291	324	324	0
11	32	39	0	39
12	5	5	5	0
13	53	47	0	47
	K= 1 H= 3			
0	155	146	0	146
1	319	324	29-	323
2	363	399	67-	393
3	106	96	29-	92
4	142	143	120	78-
5	189	192	95	164-
6	209	207	72	194
7	28	27	25-	9
8	82	67	65-	16-
9	175	195	81-	177
10	53	58	0	58
11	22	2	12	16
12	122	121	112-	45-
13	33	42	36-	21-
	K= 2 H= 3			
0	219	212	0	212
1	229	237	133	196
2	129	131	120	54
3	172	147	145	21-
4	162	165	135	94-
5	127	105	98	38
6	206	218	166	141
7	31	16	16	14
8	43	47	46	7
9	85	78	58	60-
10	63	57	99-	42
11	109	109	17	108-
12	34	31	29-	5
13	38	36	29-	21
	K= 3 H= 3			
0	193	18.	0	180-
1	310	349	239	254-
2	187	192	137-	134
3	157	145	67-	129
4	128	118	24-	116
5	153	147	49-	138
6	40	38	4-	38
7	55	47	15-	44
8	89	10.	41	91-
9	144	166	7	166-
10	73	78	76	16
11	30	28	28-	0
12	69	63	31	55-
	K= 4 H= 3			
0	130	115	0	115
1	102	92	88	28-
2	82	7.	24	66
3	41	13	12-	5-
4	86	83	6	82
5	139	130	126-	29-
6	34	28	11	26-
7	151	159	79	138-
8	117	125	62-	109-
9	96	94	85-	39
10	123	116	115	13-
11	56	61	12	59
12	17	21	21	1
	K= 5 H= 3			
0	70	65	0	65
1	120	113	113-	6
2	149	151	25	149
3	249	242	164	177
4	257	273	80	261-
5	30	28	6	28-
6	104	100	77-	65
7	215	234	57	227
8	147	146	67-	129-
9	169	179	133	120
10	131	122	90	110-
11	42	59	30	51
12	94	99	29-	94-
	K= 6 H= 3			
0	87	91	0	91-
1	298	309	292-	100-
2	62	57	14	55-
3	108	101	75-	67-
4	163	161	149-	60-
5	34	30	20	22-
6	171	175	61-	164
7	100	82	28-	76-
8	56	55	54-	10-
9	43	34	11	32-
10	38	32	21-	24-
11	95	92	85	36-
	K= 7 H= 3			
0	61	36	0	38-
1	96	88	43	76-
2	101	88	47	74
3	55	51	3-	51
4	156	138	21	136
5	26	15	15-	1
6	102	100	92	39-
7	47	44	5	44
8	29	15	14	6-
9	44	38	38-	2-
10	53	55	9-	54
11	87	109	77-	77
	K= 8 H= 3			
0	118	119	0	119-
1	127	136	136-	1-
2	83	84	65-	53
3	159	166	166-	1-
4	120	108	108	1-
5	187	205	189-	77
6	191	187	102	156-
7	45	37	37	1-
8	67	47	46	5-
9	74	66	5	65
10	69	57	6-	56
	K= 9 H= 3			
0	86	81	0	81
1	125	123	68-	102-
2	28	14	12	6-
3	176	169	84	169
4	118	116	94-	69-
5	39	32	0	32-
6	51	52	10-	51
7	125	141	43-	134
8	105	92	82	41
9	56	55	43	33-
	K=10 H= 3			
0	60	52	0	52
1	42	36	24-	27-
2	99	102	99	23
3	9	14	5	12
4	72	67	47-	47-
5	26	20	0	20-
6	61	58	39-	43
7	65	64	63	8
8	73	79	24	75
	K=11 H= 3			
0	89	87	0	87-
1	68	65	65	0
2	33	23	22	3-
3	127	127	125-	16
4	76	64	32	55
5	47	63	27-	56-
6	99	95	94	14
	K=12 H= 3			
0	121	124	0	124-
1	111	111	33-	106
2	87	80	47-	65
3	65	61	18	59
4	100	93	74	56
	K= 0 H= 4			
0	650	599	599	0
1	162	161	0	161
2	67	58	58-	0
3	159	142	0	142
4	75	74	74	0
5	45	39	0	39-
6	28	17	17-	0
7	111	112	0	112-
8	141	154	154	0
9	30	23	0	23
10	153	155	155-	0
11	110	97	0	97
12	17	8	8-	0
	K= 1 H= 4			
0	280	251	251-	0
1	173	165	151-	65
2	103	86	63-	58
3	116	102	100-	22
4	149	141	115	81
5	125	108	99-	45-
6	172	172	117-	126
7	106	66	68-	53-
8	115	116	114	22-
9	39	30	18-	24
10	145	153	132	77
11	130	121	5-	121-
12	69	65	11-	64-
	K= 2 H= 4			
0	328	325	325-	0
1	109	113	63	93-
2	151	132	119-	57-
3	285	296	68-	288-
4	141	146	138	47-
5	232	236	229-	56
6	194	223	161-	154-
7	149	152	106-	109
8	136	147	7	147-
9	59	51	51-	6-
10	128	96	74	61
11	70	60	60-	1-
12	41	41	41	4-
	K= 3 H= 4			
0	361	297	297	0
1	187	184	37-	180-
2	148	148	142	40
3	172	159	139	77-
4	56	49	45-	19
5	106	98	21-	96-
6	55	42	41	7
7	44	41	34-	22-
8	98	94	82	45
9	116	123	90-	84-
10	96	94	60-	73
11	34	24	11	21
	K= 4 H= 4			
0	129	120	120	0
1	166	179	22	177-
2	236	245	242-	40-
3	245	240	204-	127
4	138	89	77-	43
5	155	133	132	17-
6	39	35	7-	34-
7	144	148	94-	114-
8	52	53	50	18-
9	83	85	82-	22
10	93	91	73-	53-
11	43	46	39-	23
	K= 5 H= 4			
0	183	199	199-	0
1	82	70	69	7
2	53	46	18	42
3	138	132	93-	94
4	167	159	157	22-
5	146	144	133-	55-
6	168	176	85-	154
7	34	29	25-	13
8	22	17	15-	8
9	31	30	16-	25
10	53	40	0	40
	K= 6 H= 4			
0	111	105	105-	0
1	134	140	132	47-
2	209	235	192-	136-
3	135	142	12-	142-
4	115	104	15-	102-
5	172	183	138-	121-
6	96	94	90-	26-
7	58	68	25-	64
8	55	54	21	50-
9	102	84	84	9
10	50	58	48	33
	K= 7 H= 4			
0	179	191	191	0
1	47	31	22-	21-
2	49	43	42-	7
3	103	101	37	94-
4	67	49	14	47-
5	51	55	54	9-
6	100	98	46	86-
7	141	138	118	71
8	35	24	4	24-
9	56	51	48-	18
10	97	111	111-	2-
	K= 8 H= 4			
0	43	34	34	0
1	28	29	1	29-
2	132	138	63	123
3	120	117	70-	94
4	23	26	23	12
5	230	239	239	2-
6	78	76	71	26
7	64	61	46-	39-
8	42	33	19-	27
9	105	116	9	116
	K= 9 H= 4			
0	80	86	86	0
1	107	100	17	99-
2	69	70	63	31
3	45	44	36-	24
4	4	8	8-	0
5	55	56	55-	11-
6	61	72	61-	38
7	51	38	0	38
8	78	84	80-	27
	K=10 H= 4			
0	118	126	126	0
1	124	135	97	94
2	34	31	31	0
3	68	58	58-	2-
4	59	56	26	50-
5	47	60	56-	21
6	49	44	9-	43-
	K=11 H= 4			
0	114	108	108	0
1	29	18	10	15-
2	71	74	51-	53-
3	122	115	29	111-
4	63	48	38	30
5	67	74	57	48-
	K=12 H= 4			
0	95	96	96-	0
1	35	42	42-	3-
	K= 0 H= 5			
1	126	117	0	117-
2	189	207	207-	0
3	36	34	0	34-
4	9	8	8-	0
5	44	34	0	34
6	18	24	24-	0
7	196	226	0	226-
8	89	90	90-	0
9	170	200	0	200-
10	174	183	183-	0
11	19	26	0	26-
	K= 1 H= 5			
0	46	47	0	47
1	125	125	125	4-
2	110	112	97-	57-
3	105	97	96	16-
4	127	115	67	92
5	92	85	17-	83-
6	133	139	14	138-
7	60	53	11	52
8	38	42	24-	34
9	76	72	49	52-
10	88	93	53	76-
11	39	44	15-	41-
	K= 2 H= 5			
0	37	39	0	39-
1	73	61	53-	31-
2	177	200	189	66-
3	182	185	185-	16-
4	102	90	85-	31
5	229	259	41-	256-
6	76	75	56-	49-
7	47	39	39	1
8	124	131	130	17-
9	44	39	19-	33
10	80	78	9	77-
11	51	44	44	2
	K= 3 H= 5			
0	69	67	0	67
1	103	91	19-	89
2	216	219	219	5-
3	81	68	53	41-
4	36	19	6-	18
5	102	95	73	61
6	25	8	5	6-
7	73	68	68	4-
8	86	82	63	53
9	81	83	70-	45
10	52	52	44-	27-
	K= 4 H= 5			
0	127	111	0	111-
1	139	144	84-	117-
2	123	115	113-	23
3	70	63	9-	62-
4	150	138	137	13
5	107	108	78	74
6	48	47	46	3
7	99	97	97-	10
8	52	56	21-	52
9	137	124	50	113-
10	37	40	4-	40
	K= 5 H= 5			
0	117	130	0	130
1	141	138	77	114
2	129	130	99-	84-
3	99	82	27	77-
4	167	159	77	139
5	49	37	5-	37-
6	151	162	161	12
7	122	122	122-	10-
8	51	47	45	11
9	80	71	71-	4-
10	34	41	35	20
	K= 6 H= 5			
0	280	276	0	276
1	155	151	88	123
2	64	62	39	47
3	99	96	71-	64
4	91	90	85	27
5	49	43	9	42-
6	68	64	12	63-
7	109	103	78	67
8	107	108	107	16
9	63	60	17-	58
	K= 7 H= 5			
0	22	14	0	14
1	65	65	59	27-
2	71	73	47	55-
3	47	47	44-	14-
4	85	80	25-	76-
5	127	128	14	127
6	61	60	55-	22
7	71	75	72	21-
8	79	89	88	14
	K= 8 H= 5			
0	64	53	0	53
1	62	61	10	60-
2	102	91	47-	78
3	125	141	140	5
4	135	140	2	140
5	61	56	41	38-
6	56	69	31-	61
7	91	96	86-	42-
	K= 9 H= 5			
0	17	10	0	10
1	12	115	12-	114
2	32	20	8	18-
3	34	31	20	24-
4	115	115	115	8
5	60	51	17-	48
6	75	72	71	6
	K=10 H= 5			
0	76	75	0	75
1	52	52	8-	91
2	31	26	26-	2-
3	70	66	61-	23-
4	37	35	30-	17
	K=11 H= 5			
0	42	54	0	54-
	K= 0 H= 6			
0	144	154	154-	0
1	194	199	0	199-
2	98	91	91-	0
3	70	62	0	62-
4	196	216	216-	0
5	20	14	0	14
6	79	77	77	0
7	3	8	0	8-
8	88	83	83-	0
9	48	39	0	39
	K= 1 H= 6			
0	13	0	0	0
1	132	128	59	114-
2	150	155	133	79
3	116	117	64	98
4	21	14	7	12
5	72	66	60	28
6	21	9	6-	7
7	85	89	8	89-
8	118	113	71-	88
9	18	30	29	1
	K= 2 H= 6			
0	85	91	91-	0
1	118	112	35-	106
2	40	35	25-	24-
3	54	46	31	34
4	91	93	33-	87
5	77	67	67	5-
6	91	105	21	103
7	26	22	16-	15
8	77	81	4-	81
9	68	69	30	61-
	K= 3 H= 6			
0	94	84	84-	0
1	82	77	6	77
2	121	113	97-	59-
3	189	206	125-	164-
4	45	48	22-	42
5	83	91	52	74
6	76	82	1	82
7	101	106	30-	101
8	52	52	46-	23
9	50	51	51	3-
	K= 4 H= 6			
0	58	56	56-	0
1	102	97	90-	36
2	65	63	39	49
3	86	88	87	16
4	58	63	52-	36-
5	60	56	49-	27-
6	58	57	56	5-
7	32	27	22-	15
8	29	29	21-	19
	K= 5 H= 6			
0	56	53	53	0
1	102	98	17	97-
2	75	82	67	47
3	80	73	57	45-
4	71	73	25-	69
5	27	14	4-	14-
6	67	61	17-	59-
7	108	105	68	79-
8	76	74	21	71
	K= 6 H= 6			
0	80	81	81-	0
1	115	105	71-	77
2	127	122	122	8
3	115	106	68-	81
4	109	109	31	104
5	91	91	39-	82
6	37	50	47-	14
7	9	14	14-	2-
	K= 7 H= 6			
0	80	73	73-	0
1	18	11	1-	11-
2	71	65	17	63-
3	80	76	58-	48-
4	105	113	69-	89
5	39	29	0	29-
6	68	64	38	51
	K= 8 H= 6			
0	29	22	22	0
1	30	30	23-	19-
2	68	54	51-	16-
3	28	18	18	4-
4	121	126	68-	106-
5	144	160	156-	33-
	K= 9 H= 6			
0	39	45	45-	0
1	77	78	53	58-
2	56	56	46-	33
3	55	48	47	8
	K= 0 H= 7			
1	52	65	0	65-
2	79	72	72	0
3	3	3	0	3
4	68	65	65	0
5	36	33	0	33
6	14	17	17-	0
7	40	39	0	39

Table 4 (*cont.*)

	K= 1 H= 7				5	61	59	37	46	1	28	27	16	21	3	80	81	0	81-		K= 1 H= 8			
0	62	7-	0	70-	6	35	31	7-	30	2	62	51	3-	51	1	44	48	46	11-	0	73	58	58-	0
1	54	47	0	47	7	68	74	17-	72-	3	71	63	60	20-	2	39	39	15	36	1	44	42	17	39
2	39	32	12-	30-		K= 3 H= 7				4	90	82	56-	59	3	59	55	2	55-	2	89	91	86-	29-
3	63	6-	57	18	0	62	68	0	68-	5	65	57	47-	32-	4	73	85	80-	27-	3	55	60	2-	60-
4	80	76	27-	71-	1	125	138	73	117-	6	53	55	18-	52-		K= 7 H= 7					K= 2 H= 8			
5	13	9	3	8	2	18*	11	3-	15		K= 5 H= 7				0	97	99	0	99-	0	23	31	31	0
6	96	94	33	88	3	24	19	15-	11-	0	36	37	0	37-	1	14	3	0	3-	1	18	24	10	22
7	27	34	21-	26	4	35	43	38-	20-	1	33	20	13-	15-	2	63	65	27	59	2	11	9	2	9
	K= 2 H= 7				5	18*	9	9-	0	2	32	37	37	0		K= 6 H= 8				3	41	43	30-	31-
0	45	36	0	38	6	62	55	12	53-	3	57	57	19	54	0	25*	21	21	0		K= 3 H= 8			
1	60	67	35	56	7	56	50	29-	40-	4	121	126	4-	126-	1	17	12	0	12	0	80	91	91	0
2	134	139	61-	112		K= 4 H= 7				5	43	46	36	29-	2	11	22	20-	0	1	45	50	47-	16-
3	106	104	95	42-	0	66	63	0	63		K= 6 H= 7				3	158	190	0	190	2	46*	55	12-	53
4	39	43	42-	4-																				

Table 5. *Intramolecular bond distances and angles involving only carbon and oxygen atoms in galactono-γ-lactone*

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

i	*j*	D_{ij}
C(1)	O(1)	1·198 (5) Å
C(1)	C(2)	1·522 (6)
C(1)	O(4)	1·358 (5)
C(2)	O(2)	1·405 (5)
C(2)	C(3)	1·519 (6)
C(3)	O(3)	1·411 (5)
C(3)	C(4)	1·546 (6)
C(4)	O(4)	1·464 (5)
C(4)	C(5)	1·538 (6)
C(5)	O(5)	1·418 (6)
C(5)	C(6)	1·502 (6)
C(6)	O(6)	1·447 (6)

i	*j*	*k*	Angle *ijk*
O(1)	C(1)	C(2)	128·5 (4)°
O(1)	C(1)	O(4)	121·6 (4)
C(2)	C(1)	O(4)	109·5 (4)
C(1)	C(2)	O(2)	113·5 (4)
C(1)	C(2)	C(3)	102·2 (3)
O(2)	C(2)	C(3)	113·6 (4)
C(2)	C(3)	O(3)	115·9 (3)
O(3)	C(3)	C(4)	108·9 (3)
C(1)	O(4)	C(4)	109·3 (3)
C(2)	C(3)	C(4)	100·3 (3)
C(3)	C(4)	O(4)	103·0 (3)
C(3)	C(4)	C(5)	116·0 (4)
O(4)	C(4)	C(5)	109·4 (3)
C(4)	C(5)	O(5)	111·2 (4)
C(4)	C(5)	C(6)	109·6 (4)
O(5)	C(5)	C(6)	110·0 (4)
C(5)	C(6)	O(6)	112·5 (4)

Table 6. *Intermolecular distances and angles in galactono-γ-lactone*

The estimated standard deviations in parentheses refer to the last decimal positions of respective values.

Hydrogen-bond distances (Å) and angles(°)

g	*h*	*i*	*j*	*k*	*D*(*hi*)	*D*(*ij*)	*D*(*hj*)	Angle (*ghj*)	Angle (*hjk*)	Angle (*ihj*)	Angle (*ijh*)
C(2)	O(2)	H(1)	O(3*c*)	C(3*c*)	0·92 (8)	2·06 (8)	2·714 (5)	109·5 (3)	124·7 (3)	38 (5)	16 (2)
C(2)	O(2)	H(10*a*)	O(6*a*)	C(6*a*)	1·89 (10)	0·99 (10)	2·784 (6)	136·7 (3)	108·7 (3)	11 (3)	21 (6)
C(3)	O(3)	H(3)	O(5*a*)	C(5*a*)	0·90 (8)	1·80 (8)	2·686 (5)	108·9 (3)	133·3 (3)	7 (5)	4 (3)
C(5)	O(5)	H(7)	O(6*b*)	C(6*b*)	1·02 (8)	1·81 (8)	2·673 (5)	107·7 (3)	108·2 (3)	26 (4)	14 (3)

Non-bonded distances less than 2·5 Å

i	*j*	*D*(*ij*) (Å)	*i*	*j*	*D*(*ij*) (Å)
C(1)	O(1*e*)	3·311 (6)	C(5)	O(2*f*)	3·481 (5)
C(1)	O(1*d*)	3·096 (6)	C(5)	O(6*b*)	3·386 (7)
C(1)	O(2*e*)	3·448 (5)	C(6)	O(5*g*)	3·414 (6)
C(2)	O(1*e*)	3·307 (6)	O(1)	O(2*d*)	3·348 (5)
C(2)	O(3*c*)	3·448 (5)	O(1)	O(4*e*)	3·185 (5)
C(3)	O(5*a*)	3·415 (5)	O(1)	O(6*h*)	3·476 (6)
C(4)	O(1*d*)	3·326 (6)	O(2)	O(5*a*)	3·490 (5)

Symmetry code

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

Similar differences of about 0·1 Å have been observed in other lactones and are associated with a planar or nearly planar lactone group, –C–C–O–C, (Mathieson

& Taylor, 1961; Fridrichsons & Mathieson, 1962; Kim, Jeffrey, Rosenstein & Corfield, 1967). In this structure, the atoms C(2), C(1), O(1), O(4) and C(4) lie in a plane such that the largest deviation is 0·031 Å for C(1). This deviation is probably not significant. The fifth atom of the lactone ring, C(3) is 0·64 Å out of this plane, forming the puckered furan-type ring invariably found in furanose sugars (*cf.* Spencer, 1959; Sundaralingam, 1965). The internal angles of the lactone ring are similar to those found recently in furanose rings, with a mean value of 104·8° (Furberg, Petersen & Rømming, 1965; Watson, Sutor & Tollin, 1965). It is interesting to note that the two larger angles of 109° occur at the carbonyl carbon and at the ring oxygen.

The conformation about the chain bonds, C(4)–C(5) and C(5)–C(6) is shown in Fig. 2. In both cases it is 'staggered', with O(5) lying between C(3) and O(4) and O(6) lying between O(5) and H(6). Within 3σ, which is about 1·5° for angles involving carbon and oxygen and 15° for those involving hydrogen atoms, the angles about C(4)C(5) approach closely to the ideal staggered arrangement with conformational angles of 60°. As can be seen from Fig. 2, the chain atoms C(3)C(4)-C(5)C(6) are planar and O(6) is twisted out of this plane so that the dihedral angle between C(4)C(5)C(6) and C(5)C(6)O(6) is 16°.

The molecular arrangement is shown in Fig. 3. The hydrogen bonding is entirely intermolecular, linking the molecules into a three-dimensional net as illustrated in Fig. 4. Each of the four hydroxyl groups participates as donor and acceptor. Thus there are four hydrogen bonds per asymmetric unit with O···O distances between 2·673 and 2·784 Å (Table 6). These hydrogen bonds form infinite chains in the donor–acceptor sequence →O(3)→O(5)→O(6)→O(2)→O(3)→ which extends throughout the structure in the general direction of the [010] axis. Two such chains running through the unit cell linking six molecules are shown in projection in Fig. 4.

The two oxygen atoms of the lactone group, O(1) and O(4), are not hydrogen-bond acceptors and do not participate in the hydrogen-bond structure. In consequence the hydrogen-bonding occurs at the ends and on one side of the molecule only (Figs. 3 and 4). The

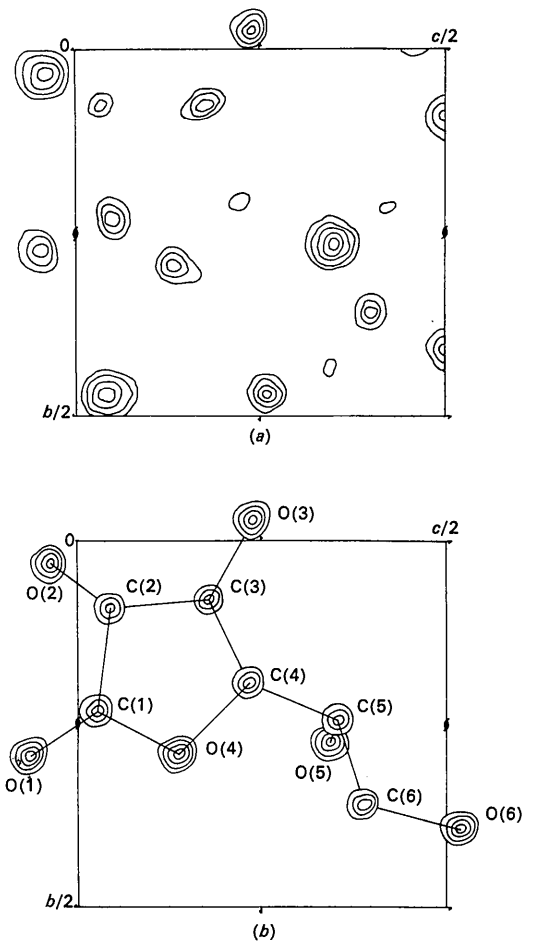


Fig. 1. (*a*) Three-dimensional *E* Fourier synthesis. (*b*) Corresponding final three-dimensional *F* synthesis viewed along [*a*].

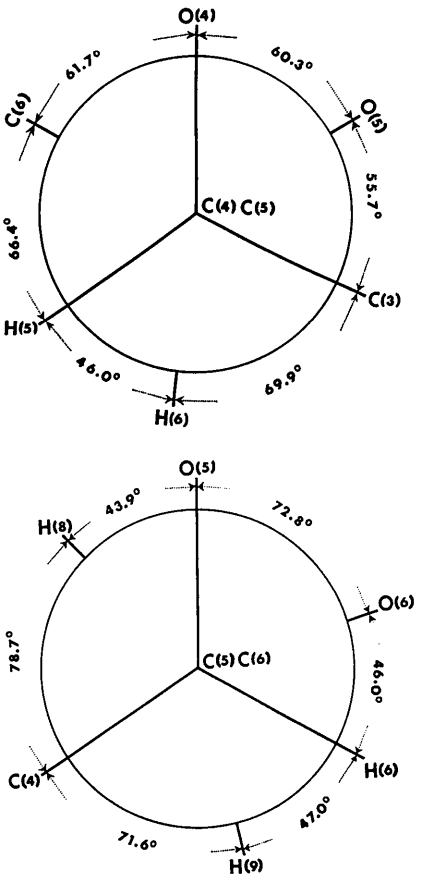
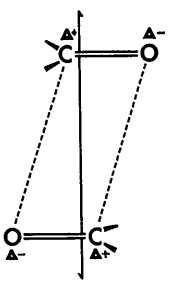


Fig. 2. Newman projections showing conformation about C(4)–C(5) and C(5)–C(6) bonds.

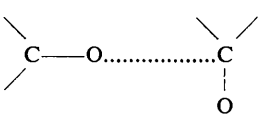
closest intermolecular approaches to these atoms are O(1)···C(1)=3·096 Å and O(1)···O(4)=3·185 Å. These are the shortest non-bonding intermolecular contacts, as shown in Table 6. That involving the carbonyl group is particularly interesting because it corresponds to a dipolar alignment about the screw axes parallel to the *a* axis, as shown in (IV).



(IV)

Similar parallel alignment of oppositely oriented carbonyl groups has been observed in the crystal structures of two pyrimidine derivatives, violuric acid monohydrate (Craven & Mascarenhas, 1964) with C···O= 3·14 and 3·15 Å, and dilituric acid trihydrate (Craven, Martinez-Carrera & Jeffrey, 1964) with C···O=3·21 Å, and in tetrahydroxy-*p*-benzoquinone dihydrate (Klug, 1965) with C···O=3·09 Å. In the quinone structure, it is suggested that these interactions form part of a self-complexing charge transfer system which gives rise to the black color of the crystals.

A related interaction between carbonyl groups on adjacent molecules in crystals has recently been discussed by Bolton (1964). In these cases the molecular packing is generally of the herring-bone type leading to the geometry in (V) and much shorter C···O distances in the range of 2·77 Å (for parabanic acid; Davies & Blum, 1955) to 2·90 Å (for barbituric acid; Bolton, 1963).



(V)

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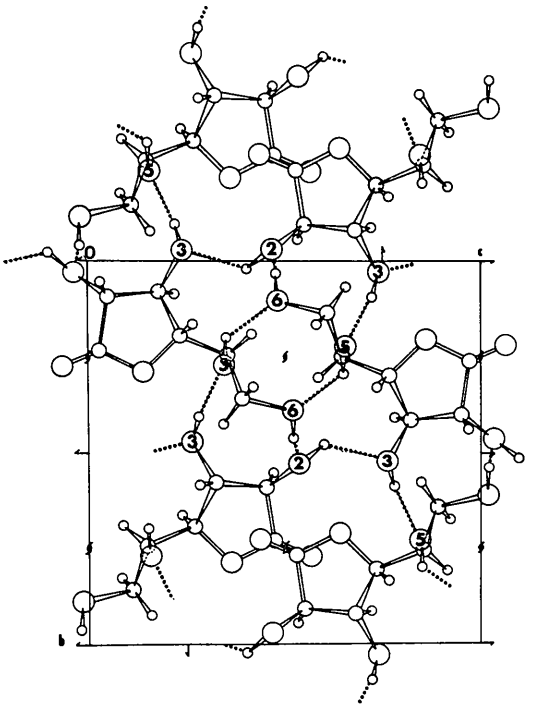

Fig. 3. Molecular arrangement in galactono-γ-lactone. Dotted lines are hydrogen bonds.

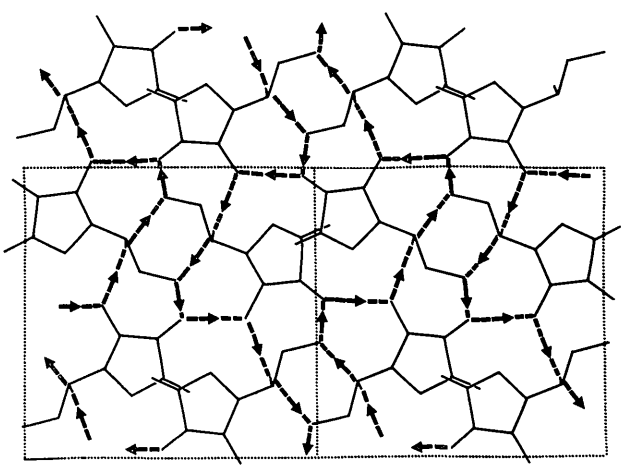

Fig. 4. Hydrogen-bond structure in galactono-γ-lactone, showing chains extending through the structure in the [010] direction.

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The Crystal Structure of β-D-Glucurono-γ-lactone

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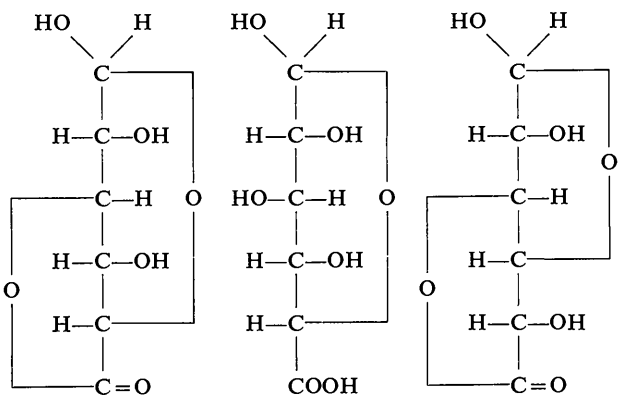
The crystal structure of β-D-glucurono-γ-lactone, $C_6H_8O_6$, has two molecules in a cell with $a=6{\cdot}753$, $b=7{\cdot}488$, $c=6{\cdot}608$ Å, $\beta=93{\cdot}18°$, space group $P2_1$. The structure was solved from the three-dimensional sharpened Patterson function using multiple minimum function and convolution methods on an IBM 1620 computer. Both photographic and automatic diffractometer data were measured. The former gave a terminal R index of 10% and the latter 4%. The molecule was found to have fused five-membered lactone and furanose rings, in agreement with an earlier deduction from chemical evidence. The rings, neither of which is planar, are inclined to each other so that the best planes containing four atoms in each make a dihedral angle of 111·3°. The lactone group is not planar, having a carbon atom 0·26 Å

out of the plane of the C–C–O group, which is planar. The C–O bond adjacent to the carbonyl group is 0·10 Å shorter than the other formal single C–O bonds in the molecule. The molecules are associated in the crystal by a very simple system of hydrogen bonding, which excludes one hydroxyl group and the ring oxygen atoms.

Introduction

Glucurono-γ-lactone (also called glucurone or glycurone) is of biochemical interest. It is converted into L-ascorbic acid in animals (Chatterjee, Chatterjee, Ghosh, Ghosh & Guha, 1960) and in the human body (Baker, Bierman & Plough, 1960), and has been shown to have an antihypnotic action against sodium 5,5-diethylbarbiturate (Tamura, Tsutsumi & Kizu, 1962). The molecule was originally believed to contain a six-membered pyranose and a five-membered lactone ring as shown in (I), since it is derived from glucuronic acid, which has the pyranose structure (II) like the anion in potassium glucuronate (Gurr, 1963; Furberg, Hammer & Mostad, 1963). However, on the basis of chemical experiments with trimethylglucurono-lactone by Reeves (1940) and Smith (1944), Smith proposed the structure (III) containing two five-membered rings, which is confirmed by this structure determination.



Of the eight stereo-isomers that can be formed by different configurations of the secondary alcohol groups at the three positions in this lactone/furanose ring system, only two can be derived from glucuronic acid by ring closure without inversion. These two configurations are 1*n*2*x*5*n* and 1*x*2*x*5*n*, where *endo*, *n*, and *exo*, *x*, refer to substituents directed towards and away, respectively, from the dihedral angle between the two rings. The β form (IV), which has been found in the crystals studied, is the former conformation, with two *endo* substituents; this is contrary to a rule that the stable derivatives of two fused five-membered ring