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The Crystal Structure of Ryanodol *p*-Bromobenzyl Ether*

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The crystals of ryanodol-*p*-bromobenzyl ether, $C_{27}H_{37}O_8Br$, belong to the space group $P2_12_12_1$ and the unit-cell dimensions are: $a = 17{\cdot}65 \pm 0{\cdot}03$, $b = 14{\cdot}43 \pm 0{\cdot}02$ and $c = 10{\cdot}11 \pm 0{\cdot}02$ Å. The structure was solved by the heavy-atom method from three-dimensional X-ray intensity data collected photographically. The atomic parameters were refined by block-diagonal least squares to the R value of 0·090. The results confirm the structure and relative stereochemistry independently deduced by chemical means by K. Wiesner, except for the configuration at one of the eleven asymmetric centres. Only one hydrogen bond (intermolecular) was found. All other hydrogen atoms of OH groups make intra- and intermolecular contacts with oxygen atoms which do not deviate from the sum of the van der Waals radii.

Introduction

The insecticidal principle ryanodine has been isolated from the stems and roots of *Ryania speciosa* Vahl. by Folkers and his co-workers (Rogers, Koniuszy, Shavel & Folkers, 1948).

A chemical investigation of ryanodine was undertaken by K. Wiesner and his collaborators in 1951, and they established that this compound is an ester of pyrrole-α-carboxylic acid and of the fully saturated polyalcohol ryanodol, $C_{20}H_{32}O_8$. Fig. 1 shows the schematic formula as established by this X-ray investigation and the relationship between ryanodine, ryanodol and ryanodol *p*-bromobenzyl ether. The extensive degradation studies led first to the elucidation of the structure and the determination of the absolute configuration of anhydroryanodine, the dehydration product of ryanodine (Babin, Forrest, Valenta & Wiesner, 1962; Wiesner, 1963) and later to the proposals for the structures of ryanodol and ryanodine (Wiesner, Valenta & Findlay, 1967; Wiesner, 1968).

This paper reports the crystal structure determination of ryanodol *p*-bromobenzyl ether, $C_{27}H_{37}O_8Br$, and confirms the molecular structures for this and related compounds proposed by Wiesner *et al.*, except for their assignment of configuration at the carbon atom carrying the isopropyl group.

The absolute configuration of ryanodol *p*-bromobenzyl ether has not been determined by X-rays, but the skeleton in Fig. 1 and throughout this report conforms with the absolute configuration derived by Wiesner (1963) for anhydroryanodine.

A preliminary account of this work was published earlier (Srivastava & Przybylska, 1968).

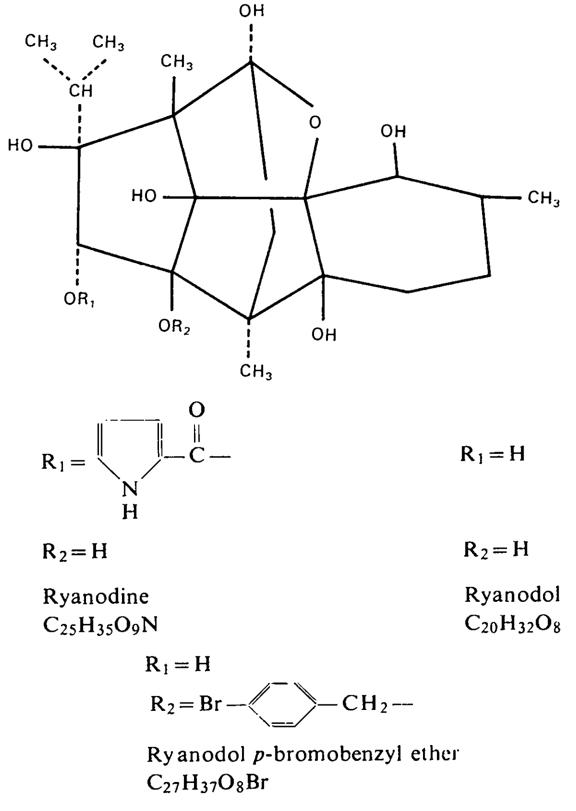

Fig. 1. Schematic formula of ryanodol *p*-bromobenzyl ether and its relationship to ryanodine and ryanodol.

Crystal data

$C_{27}H_{37}O_8Br$, M.W. 569·5

Orthorhombic; the systematic absences determined the space group as $P2_12_12_1$ (D_2^4). There are four molecules per unit cell of dimensions: $a = 17{\cdot}65 \pm 0{\cdot}03$, $b = 14{\cdot}43 \pm 0{\cdot}02$, $c = 10{\cdot}11 \pm 0{\cdot}02$ Å.

$D_m = 1{\cdot}463$ g.cm^{-3} (by flotation in a mixture of carbon tetrachloride and toluene) and $D_c = 1\cdot462$ g.cm^{-3}. $F(000) = 1192$. $\mu = 28\cdot1$ cm^{-1}. Results of the chemical analysis obtained from Dr Wiesner:

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Calculated: C, 56·94; H, 6·55; O, 22·48; Br, 14·05%.
Found: C, 56·79; H, 6·34; O, 22·73; Br, 14·01%.

The crystals from Dr Wiesner were obtained by evaporating slowly a solution of the compound in a mixture of methanol and chloroform. Various attempts in our laboratory to increase the crystals to the size suitable for X-ray work proved unsuccessful, until Dr W. Mechlinski used a minimum amount of ethyl acetate to dissolve the crystals and then added petroleum spirit until the solution became slightly opaque.

The lattice constants were determined from precession photographs (Cu $K\alpha = 1\cdot5418$ Å). Sets of multiple-film, equi-inclination Weissenberg photographs were taken for the crystal rotating about the *b* axis (layer *h*0*l* through *h*4*l*) and about the *c* axis (layer *hk*0 through *hk*8). A total of 2406 independent reflexions were measured by visual comparison with the standard intensity strip prepared with a crystal of the same compound. The reflexions, which were measured twice, were averaged and the absolute scale and overall temperature factors were evaluated by Wilson's (1942) method. The crystals were cut so that they were cylindrical in shape. Their diameters ranged between 0·2 and 0·3 mm and absorption corrections were not applied.

Solution of the structure and its refinement

The position of the bromine atom was derived from the Harker vectors of a three-dimensional Patterson synthesis which was sharpened by the $1/f^2_{\mathrm{Br}}$ function. The first electron density maps calculated with the phases based only on the contribution of the heavy atom revealed the location of ten light atoms, which were treated as carbon atoms. Two subsequent Fourier cycles gave eight and nine additional atoms bringing the total of light atoms to 27 and the R value to 0·32. The remaining eight atoms of the molecule were obtained from three-dimensional difference maps. The refinement of the parameters by least-squares was then undertaken and two additional $\varrho_o - \varrho_c$ syntheses were carried out in order to distinguish the oxygen atoms from carbon atoms. A value for $R = 0\cdot17$ was obtained when the molecular structure was solved. Several cycles of block-diagonal least-squares, with the introduction of anisotropic refinement for all atoms, led to a considerable improvement between the calculated and observed structure factors, giving a reliability index of 0·115.

At this stage the hydrogen atoms were located. First the positions of sixteen hydrogen atoms, those attached to the benzene ring and to di- and tri-substituted carbon atoms, were computed. They were included in the calculation of structure factors used for the evaluation of the last difference synthesis. The remaining twenty-one hydrogen atoms were located on these maps without any difficulty and only the position of a hydrogen atom attached to O(30) had to be adjusted. It was found to be too close to the oxygen atom, and it was moved by about 0·3 Å along the O–H line. In some methyl groups one hydrogen atom did not show up as clearly as the other two, but it was possible to derive their coordinates using the positions of the acceptable hydrogen atoms.

Four additional least-squares cycles were carried out until the shifts for two-thirds of the parameters were less than $\frac{1}{8}$ of their corresponding standard deviations and for the remaining coordinates the shifts were larger, but did not exceed $\frac{1}{3}$ of their e.s.d.'s.

The final value of $R = 0\cdot090$ was obtained excluding the unobserved reflexions. The atomic coordinates and their estimated standard deviations of the non-hydrogen atoms are given in Table 1 and those of the hydrogen atoms are presented in Table 2. The positions of the hydrogen atoms were not refined and the temperature factor $B = 5\cdot0$ Å^2 was used in the calculation of their contribution. The final vibrational parameters and the estimated standard deviations of the non-hydrogen atoms are listed in Table 3, together with the values of the principal radii of the vibration ellipsoids.

Table 1. *Final fractional coordinates and their estimated standard deviations*

	x/a	y/b	z/c
Br	−0·1042 (1)	0·6888 (1)	−0·3019 (1)
C(1)	0·1794 (5)	0·2413 (6)	0·2842 (10)
C(2)	0·0943 (5)	0·2586 (5)	0·2418 (9)
C(3)	0·0803 (4)	0·3643 (5)	0·2567 (9)
C(4)	0·0457 (5)	0·3956 (5)	0·3896 (9)
C(5)	0·0663 (4)	0·5003 (5)	0·3924 (9)
C(6)	0·0533 (6)	0·5574 (6)	0·5126 (9)
C(7)	0·0839 (6)	0·6554 (6)	0·4868 (11)
C(8)	0·1649 (5)	0·6599 (6)	0·4359 (11)
C(9)	0·1836 (4)	0·5891 (5)	0·3253 (8)
C(10)	0·1517 (4)	0·4955 (5)	0·3604 (8)
O(11)	0·1901 (3)	0·4630 (4)	0·4770 (6)
C(12)	0·1788 (5)	0·3630 (5)	0·4787 (9)
C(13)	0·2061 (5)	0·3376 (5)	0·3363 (10)
C(14)	0·1580 (4)	0·4114 (6)	0·2617 (8)
C(15)	0·0940 (5)	0·3479 (6)	0·5005 (9)
C(16)	0·1899 (6)	0·1531 (6)	0·3721 (12)
C(17)	0·1680 (7)	0·0663 (6)	0·2952 (15)
C(18)	0·2698 (8)	0·1418 (9)	0·4233 (15)
O(19)	0·2201 (4)	0·2266 (5)	0·1638 (7)
O(20)	0·0387 (4)	0·2066 (4)	0·3161 (7)
O(21)	0·0292 (3)	0·3963 (4)	0·1569 (6)
C(22)	0·0403 (6)	0·3750 (6)	0·0206 (8)
C(23)	0·0052 (5)	0·4500 (6)	−0·0580 (9)
C(24)	−0·0574 (7)	0·4334 (7)	−0·1352 (11)
C(25)	−0·0916 (7)	0·5049 (9)	−0·2069 (13)
C(26)	−0·0602 (6)	0·5914 (8)	−0·2006 (11)
C(27)	0·0024 (6)	0·6085 (7)	−0·1254 (11)
C(28)	0·0345 (6)	0·5374 (6)	−0·0571 (11)
C(29)	−0·0377 (5)	0·3801 (7)	0·4120 (10)
O(30)	0·0281 (3)	0·5483 (4)	0·2877 (6)
C(31)	0·1825 (7)	0·7584 (6)	0·3865 (13)
O(32)	0·2640 (3)	0·5830 (4)	0·3062 (7)
O(33)	0·2236 (3)	0·3258 (4)	0·5747 (6)
C(34)	0·2922 (5)	0·3535 (7)	0·3284 (10)
O(35)	0·1857 (3)	0·4369 (4)	0·1343 (6)

Table 2. *Fractional coordinates of the hydrogen atoms*

The number of the parent atom is obtained by omitting the last digit. The atoms marked with an asterisk were found experimentally.

	x/*a*	*y*/*b*	*z*/*c*
H(21)	0·086	0·237	0·142
H(61)	0·082	0·526	0·595
H(62)	−0·006	0·561	0·534
H(71)	0·084	0·694	0·578
H(72)	0·050	0·689	0·415
H(81)	0·202	0·647	0·518
H(91)	0·158	0·613	0·236
H(151)	0·080	0·375	0·595
H(152)	0·084	0·274	0·499
H(161)	0·157	0·160	0·460
H(171)*	0·172	0·007	0·356
H(172)*	0·207	0·057	0·210
H(173)*	0·110	0·072	0·258
H(181)*	0·285	0·199	0·481
H(182)*	0·309	0·135	0·342
H(183)*	0·272	0·080	0·487
H(191)*	0·217	0·270	0·085
H(201)*	0·085	0·155	0·303
H(221)	0·013	0·311	−0·002
H(222)	0·099	0·370	0·000
H(241)	−0·081	0·363	−0·144
H(251)	−0·144	0·490	−0·262
H(271)	0·026	0·677	−0·123
H(281)	0·087	0·551	−0·002
H(291)*	−0·059	0·319	0·363
H(292)*	−0·070	0·439	0·374
H(293)*	−0·048	0·371	0·518
H(301)*	−0·028	0·547	0·287
H(311)*	0·180	0·809	0·468
H(312)*	0·238	0·760	0·338
H(313)*	0·143	0·779	0·309
H(321)*	0·293	0·557	0·382
H(331)*	0·215	0·360	0·659
H(341)*	0·308	0·423	0·297
H(342)*	0·317	0·303	0·259
H(343)*	0·317	0·341	0·427
H(351)*	0·240	0·444	0·142

The scattering factors for the non-hydrogen atoms were those of Hanson, Herman, Lea & Skillman (1964) and for the hydrogen atoms those of Stewart, Davidson & Simpson (1965).

The observed and calculated structure factors are listed in Table 4. The structure factors for the unobserved reflexions were calculated, but they were excluded from the refinement procedure and from general sums. The agreement summary is given in Table 5. Two

Table 3. *Vibration tensor components and their e.s.d.'s* (Å$^2 \times 10^3$)

Temperature factor $= \exp \{-2\pi^2(U_{11}a^{*2}h^2 + U_{22}b^{*2}k^2 + U_{33}c^{*2}l^2 + 2U_{23}b^*c^*kl + 2U_{13}a^*c^*hl + 2U_{12}a^*b^*hk)\}$.

B_i (Å^2) are the principal radii of the vibration ellipsoids.

	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{13}$	$2U_{12}$	B_1	B_2	B_3
Br	113 (1)	95 (1)	64 (1)	32 (1)	8 (2)	77 (2)	11·51	5·76	4·20
C(1)	50 (4)	47 (4)	59 (6)	−22 (8)	11 (8)	4 (7)	5·23	4·01	3·02
C(2)	51 (4)	35 (3)	38 (4)	−19 (6)	−6 (7)	−15 (6)	4·24	3·56	1·94
C(3)	45 (4)	41 (4)	35 (4)	−6 (7)	5 (6)	−6 (6)	3·78	3·15	2·69
C(4)	45 (4)	40 (3)	44 (5)	3 (7)	15 (7)	3 (6)	4·10	3·16	2·88
C(5)	42 (4)	39 (3)	42 (5)	14 (7)	0 (7)	14 (6)	3·97	3·30	2·43
C(6)	67 (5)	45 (4)	43 (5)	−15 (8)	14 (9)	5 (8)	5·42	4·07	2·77
C(7)	69 (6)	43 (4)	55 (6)	−13 (8)	−5 (9)	16 (8)	5·72	4·40	3·09
C(8)	52 (4)	42 (4)	63 (6)	−12 (8)	−10 (9)	1 (7)	5·26	3·95	3·22
C(9)	48 (4)	35 (3)	38 (5)	−12 (6)	2 (7)	0 (6)	3·82	3·36	2·40
C(10)	46 (4)	38 (3)	33 (4)	3 (7)	−2 (7)	3 (6)	3·69	3·02	2·59
O(11)	48 (3)	37 (2)	43 (3)	−2 (5)	−16 (5)	2 (4)	4·27	2·94	2·90
C(12)	49 (4)	37 (3)	39 (5)	−10 (7)	−8 (7)	−3 (6)	4·00	3·37	2·57
C(13)	45 (4)	38 (3)	57 (6)	6 (7)	3 (8)	−4 (6)	4·54	3·60	2·95
C(14)	42 (3)	47 (4)	33 (4)	−16 (7)	4 (6)	−19 (6)	4·47	2·87	2·28
C(15)	56 (4)	44 (4)	37 (4)	2 (7)	−1 (7)	−1 (7)	4·43	3·51	2·89
C(16)	60 (5)	45 (4)	81 (7)	9 (9)	9 (10)	6 (8)	6·50	4·65	3·47
C(17)	80 (7)	37 (4)	115 (10)	2 (11)	−29 (15)	6 (9)	9·51	5·97	2·94
C(18)	92 (8)	68 (6)	93 (9)	−23 (14)	−45 (15)	39 (13)	9·80	5·74	4·53
O(19)	68 (4)	67 (4)	57 (4)	−37 (7)	27 (7)	2 (7)	6·73	5·41	3·05
O(20)	59 (3)	49 (3)	58 (4)	−11 (6)	11 (6)	−24 (5)	5·63	4·23	3·23
O(21)	51 (3)	50 (3)	39 (3)	4 (5)	−5 (5)	−2 (5)	4·10	3·90	3·01
C(22)	73 (5)	42 (4)	29 (4)	−10 (7)	−11 (8)	−20 (8)	6·00	3·31	2·06
C(23)	56 (5)	50 (4)	37 (5)	−2 (8)	−14 (8)	−16 (7)	4·96	3·70	2·70
C(24)	88 (7)	62 (5)	54 (6)	7 (10)	−40 (11)	−43 (11)	8·43	4·33	3·33
C(25)	76 (6)	82 (7)	67 (7)	22 (13)	−45 (12)	−18 (12)	8·17	5·74	3·83
C(26)	67 (6)	85 (7)	39 (5)	20 (11)	11 (9)	27 (10)	7·42	4·72	2·91
C(27)	66 (6)	63 (5)	56 (6)	37 (10)	−5 (10)	−26 (9)	6·74	4·79	3·05
C(28)	71 (6)	48 (4)	53 (6)	13 (9)	−1 (10)	−16 (9)	5·82	4·46	3·32
C(29)	43 (4)	63 (5)	52 (6)	−10 (9)	6 (8)	−4 (7)	5·19	3·96	3·31
O(30)	51 (3)	47 (3)	45 (3)	−4 (5)	−12 (5)	15 (5)	4·69	3·48	3·15
C(31)	85 (7)	37 (4)	80 (8)	−22 (9)	5 (12)	11 (9)	6·81	6·51	2·67
O(32)	53 (3)	52 (3)	56 (4)	17 (6)	12 (6)	3 (5)	5·17	4·03	3·50
O(33)	58 (3)	47 (3)	47 (3)	12 (5)	−25 (6)	1 (5)	5·25	3·88	2·83
C(34)	44 (4)	57 (5)	60 (6)	−7 (9)	−11 (8)	2 (7)	5·05	4·37	3·35
O(35)	56 (3)	50 (3)	40 (3)	−12 (5)	26 (5)	−5 (5)	5·11	3·86	2·51

Table 4. *Observed and calculated structure factors* (× 10)

Reflexions too weak to be measured are marked with an asterisk.

H	FO	FC
K= 0, L= 0		
2	409	450
4	347	278
6	159	242
8	1056	1108
10	485	543
12	0*	27
14	460	451
16	223	216
18	0*	6
20	0*	20
22	63	70
K= 1, L= 0		
1	1352	1449
2	1800	2131
3	212	289
4	397	489
5	584	469
6	0*	15
7	1103	1171
8	751	847
9	52	78
10	266	258
11	0*	18
12	365	354
13	157	112
14	145	157
15	82	82
16	168	161
17	85	95
18	78	72
19	0*	36
20	49	42
K= 2, L= 0		
0	49	152
1	201	219
2	803	845
3	363	320
4	671	745
5	705	832
6	832	890
7	480	431
8	273	284
9	212	191
10	648	620
11	359	374
12	70	60
13	180	158
14	128	111
15	83	75
16	191	196
17	0*	44
18	65	73
19	92	95
20	0*	10
21	52	45
22	46	45
K= 3, L= 0		
1	331	312
2	67	137
3	656	718
4	939	920
5	17	24
6	0*	86
7	80	122
8	412	439
9	340	244
10	216	227
11	313	301
12	103	91
13	76	85
14	67	49
15	257	254
16	0*	13
17	67	13
18	114	103
19	0*	16
20	0*	2
21	52	46
22	39	38
K= 4, L= 0		
0	969	910
1	914	931
2	92	70
3	631	664
4	477	486
5	408	470
6	85	66
7	0*	45
8	0*	35
9	285	277
10	132	118
11	247	247
12	161	143
13	175	184
14	49	67
15	132	124
16	0*	8
17	49	50
18	0*	20
19	60	43
20	0*	9
21	49	60
K= 5, L= 0		
1	842	892
2	219	132
3	621	676
4	946	886
5	128	99
6	89	100
7	217	261
8	100	81
9	39	39
10	255	246
11	354	357
12	97	91
13	404	358
14	172	135
15	115	101
16	0*	26
17	110	102
18	0*	8
19	67	55
20	0*	5
21	46	42
K= 6, L= 0		
0	275	207
1	1278	1291
2	140	122
3	665	566
4	771	818
5	573	585
6	585	547
7	319	260
8	277	216
9	0*	0
10	415	392
11	246	228
12	90	85
13	211	204
14	139	131
15	403	424
16	113	93
17	130	137
18	0*	21
19	0*	20
20	30	26
K= 7, L= 0		
1	472	437
2	355	331
3	682	641
4	618	577
5	183	122
6	445	455
7	65	81
8	598	555
9	336	344
10	0*	33
11	114	89
12	273	261
13	0*	40
14	132	146
15	158	138
16	39	28
17	89	101
K= 8, L= 0		
0	409	452
1	119	83
2	0*	1
3	196	188
4	125	134
5	141	115
6	167	147
7	322	302
8	200	230
9	135	140
10	225	199
11	235	225
12	95	98
13	121	84
14	144	124
15	95	108
16	0*	14
17	0*	43
18	80	76
K= 9, L= 0		
1	308	297
2	284	311
3	304	324
4	325	310
5	318	276
6	70	41
7	206	169
8	89	75
9	192	167
10	317	309
11	95	102
12	55	39
13	0*	51
14	155	131
15	89	85
16	95	93
17	0*	10
18	80	78
19	46	39
K= 10, L= 0		
0	237	182
1	356	334
2	430	407
3	149	155
4	144	147
5	371	332
6	275	262
7	115	118
8	217	241
9	114	111
10	195	193
11	163	163
12	0*	24
13	158	184
14	0*	20
15	0*	35
16	80	76
17	0*	32
18	52	50
K= 11, L= 0		
1	0*	16
2	130	126
3	535	516
4	421	379
5	0*	20
6	113	100
7	165	160
8	78	76
9	191	168
10	0*	23
11	214	209
12	0*	10
13	97	100
14	0*	11
15	70	66
16	0*	28
17	0*	8
18	46	61
K= 12, L= 0		
0	173	154
1	158	137
2	484	425
3	0*	72
4	256	237
5	361	332
6	0*	21
7	0*	31
8	0*	1
9	191	194
10	0*	5
11	240	223
12	80	76
13	0*	12
14	76	67
15	35	5
16	25	23
17	43	50
K= 13, L= 0		
1	145	131
2	35	51
3	237	198
4	85	98
5	49	58
6	0*	3
7	308	287
8	0*	53
9	153	115
10	206	217
11	67	64
12	70	84
13	60	42
14	49	33
15	67	58
K= 14, L= 0		
0	0*	4
1	87	70
2	103	102
3	49	42
4	0*	48
5	155	135
6	74	79
7	0*	20
8	127	140
9	92	100
10	92	84
11	55	58
12	87	106
13	0*	12
14	90	98
K= 15, L= 0		
1	80	72
2	0*	55
3	72	70
4	0*	25
5	85	92
6	83	77
7	0*	1
8	103	93
9	0*	9
10	0*	6
11	63	69
12	58	63
13	58	73
K= 16, L= 0		
0	0*	61
1	0*	31
2	94	93
3	0*	16
4	95	99
5	63	28
6	74	56
K= 17, L= 0		
1	67	71
2	110	95
3	49	46
4	49	49
5	104	106
6	90	82
K= 18, L= 0		
0	0*	11
1	52	59
2	58	72
3	0*	29
4	60	62
K= 0, L= 1		
1	612	589
2	982	1154
3	511	543
4	439	495
5	1491	1683
6	267	254
7	189	205
8	57	66
9	1182	1201
10	823	779
11	0*	42
12	143	126
13	167	159
14	113	91
15	64	65
16	0*	6
17	161	158
18	64	39
19	143	135
20	0*	2
21	52	37
K= 1, L= 1		
0	0*	15
1	364	406
2	1145	1457
3	971	1055
4	777	869
5	419	444
6	109	108
7	1089	1115
8	224	217
9	199	157
10	220	190
11	73	69
12	257	226
13	187	169
14	70	52
15	131	113
16	52	31
17	72	40
18	0*	33
19	70	49
20	0*	14
21	52	35
22	40	30
K= 2, L= 1		
0	899	1048
1	237	286
2	910	976
3	351	338
4	742	777
5	546	578
6	280	281
7	621	600
8	204	172
9	314	331
10	410	381
11	230	211
12	154	135
13	151	127
14	101	101
15	93	70
16	73	63
17	57	51
18	28	19
19	46	33
20	101	80
21	52	46
22	57	60
K= 3, L= 1		
0	258	296
1	539	557
2	527	664
3	543	643
4	441	504
5	554	557
6	804	792
7	307	298
8	474	454
9	435	452
10	135	150
11	96	77
12	265	255
13	133	120
14	64	54
15	216	208
16	123	105
17	0*	27
18	0*	21
19	28	32
20	52	41
21	57	52
K= 4, L= 1		
0	787	865
1	495	530
2	96	116
3	301	300
4	684	699
5	363	360
6	572	592
7	347	346
8	582	505
9	229	242
10	500	490
11	246	228
12	77	70
13	116	87
14	170	164
15	66	53
16	33	32
17	110	111
18	0*	12
19	61	53
20	49	20
K= 5, L= 1		
0	811	800
1	490	530
2	584	580
3	372	399
4	659	640
5	381	426
6	869	892
7	204	222
8	751	729
9	279	262
10	130	119
11	70	44
12	167	154
13	145	142
14	175	155
15	129	117
16	0*	33
17	118	114
K= 6, L= 1		
0	229	202
1	728	718
2	589	527
3	606	637
4	966	896
5	215	210
6	470	467
7	87	66
8	238	180
9	240	267
10	388	366
11	226	229
12	192	200
13	292	259
14	224	188
15	236	222
16	105	91
17	0*	24
18	0*	18
19	57	41
20	59	57
K= 7, L= 1		
0	131	70
1	388	443
2	596	536
3	761	735
4	297	299
5	136	126
6	85	86
7	384	390
8	193	190
9	136	128
10	124	112
11	310	266
12	303	270
13	255	241
14	0*	58
15	72	61
16	0*	45
17	140	115
18	117	94
19	46	39
K= 8, L= 1		
0	61	49
1	600	563
2	563	488
3	229	214
4	154	147
5	537	540
6	314	313
7	194	204
8	114	108
9	258	240
10	185	198
11	196	187
12	218	209
13	0*	10
14	0*	33
15	131	112
16	73	74
17	54	42
18	57	58
19	43	32
K= 9, L= 1		
0	426	398
1	535	522
2	162	162
3	213	201
4	140	148
5	106	89
6	0*	19
7	520	481
8	75	89
9	385	363
10	266	251
11	124	94
12	90	114
13	122	103
14	221	156
15	73	71
16	64	54
17	0*	19
18	49	48
K= 10, L= 1		
0	0*	26
1	282	266
2	223	220
3	255	261
4	188	203
5	314	277
6	0*	12
7	259	244
8	167	166
9	294	252
10	151	160
11	357	331
12	215	190
13	70	47
14	73	54
15	80	82
16	0*	15
17	59	57
18	49	42
K= 11, L= 1		
0	200	185
1	289	219
2	413	403
3	102	85
4	0*	50
5	129	127
6	165	154
7	113	107
8	93	89
9	339	311
10	117	104
11	0*	36
12	88	84
13	82	67
14	66	70
15	0*	30
16	93	82
K= 12, L= 1		
0	415	385
1	139	121
2	279	236
3	242	235
4	91	95
5	0*	12
6	87	91
7	0*	9
8	108	109
9	79	87
10	0*	21
11	206	203
12	59	48
13	61	56
14	123	120
15	33	35
16	52	46
K= 13, L= 1		
0	0*	44
1	157	180
2	143	116
3	118	111
4	90	102
5	0*	39
6	0*	43
7	72	60
8	132	107
9	0*	48
10	99	88
11	40	25
12	123	124
13	118	110
14	57	74
15	57	70
K= 14, L= 1		
0	91	57
1	46	56
2	123	125
3	64	83
4	167	178
5	100	116
6	142	115
7	80	89
8	75	74
9	79	84
10	104	95
11	0*	47
12	59	61
13	57	61
14	54	56
K= 15, L= 1		
0	87	83
1	79	68
2	75	59
3	77	86
4	59	39
5	59	48
6	0*	30
7	0*	44
8	122	103
9	57	44
10	80	75
11	85	88
K= 16, L= 1		
0	57	32
1	66	61
2	70	68
3	66	61
4	54	43
5	57	40
6	0*	16
7	0*	14
8	0*	33
9	72	66
10	43	32
K= 17, L= 1		
0	0*	36
1	0*	14
2	0*	19
3	0*	50
4	52	75
5	43	45
6	40	30
7	40	37
K= 0, L= 2		
0	1196	1306
1	1414	1477
2	133	84
3	244	277
4	940	888
5	622	578
6	404	462
7	151	148
8	333	311
9	100	125
10	299	256
11	112	101
12	32	38
13	201	200
14	155	151
15	118	96
16	177	170
17	0*	4
18	0*	22
19	20	26
20	0*	1
21	68	66
22	48	59
K= 1, L= 2		
0	972	966
1	628	694
2	1857	1597
3	1182	1064
4	804	754
5	1021	1001
6	228	218
7	127	82
8	472	461
9	429	411
10	253	230
11	105	125
12	134	146
13	224	229
14	226	228
15	176	154
16	110	99
17	32	40
18	58	65
19	51	36
20	55	55
21	45	52
22	28	17
K= 2, L= 2		
0	850	719
1	1095	944
2	1350	1155
3	717	638
4	317	338
5	240	171
6	362	374
7	120	139
8	248	187
9	468	432
10	405	391
11	223	202
12	384	400
13	357	369
14	127	122
15	219	199
16	65	64
17	64	69
18	42	30
19	53	52
20	66	59
21	44	40
22	44	52
K= 3, L= 2		
0	1292	1152
1	573	568
2	591	508
3	880	816
4	370	342
5	59	52
6	429	425
7	517	521
8	348	299
9	107	58
10	85	96
11	302	314
12	454	446
13	284	277
14	95	99
15	61	54
16	49	38
17	144	151
18	41	22
19	48	63
20	47	54
K= 4, L= 2		
0	611	558
1	594	599
2	540	501
3	777	649
4	425	395
5	656	652
6	487	419
7	468	446
8	401	392
9	542	490
10	173	153
11	176	178
12	125	109
13	68	75
14	162	155
15	214	207
16	169	180
17	0*	35
18	52	55
19	74	69
20	0*	24
21	28	25
K= 5, L= 2		
0	616	625
1	995	953
2	264	234
3	555	542
4	88	97
5	167	154
6	355	354
7	394	384
8	95	103
9	122	87
10	174	168
11	204	197
12	128	127
13	360	361
14	177	176
15	0*	27
16	105	105
17	83	79
18	77	80
19	45	60
20	36	42
21	0*	20
K= 6, L= 2		
0	643	610
1	780	687
2	532	508
3	573	522
4	395	427
5	77	64
6	173	195
7	81	75
8	236	272
9	275	301
10	181	192
11	284	294
12	233	239
13	94	91
14	51	44
15	59	61
16	101	108
17	51	48
18	0*	13
19	0*	25
20	42	48
21	44	65
K= 7, L= 2		
0	317	325
1	405	435
2	267	283
3	180	219
4	466	475
5	225	231
6	364	387
7	55	58
8	224	227
9	218	241
10	75	79
11	206	232
12	88	97
13	97	111
14	89	95
15	79	79
16	91	102
17	0*	31
18	51	60
19	32	33
20	34	49
K= 8, L= 2		
0	353	393
1	95	107
2	126	136
3	374	389
4	203	241
5	199	200
6	101	82
7	293	304
8	131	147
9	209	223
10	90	75
11	237	263
12	149	162
13	152	154
14	184	192
15	0*	26
16	67	62
17	83	85
18	23	26
19	34	41
20	23	30
K= 9, L= 2		
0	264	270
1	266	320
2	400	433
3	137	139
4	154	159
5	289	298
6	90	90
7	150	137
8	49	58
9	150	149
10	49	68
11	52	79
12	118	113
13	66	56
14	0*	23
15	104	123
16	87	92
17	0*	23
18	66	81
19	42	56
K= 10, L= 2		
0	551	550
1	45	13
2	281	334
3	217	250
4	218	261
5	280	320
6	129	119
7	156	181
8	141	128
9	160	167
10	85	82
11	130	142
12	107	112
13	0*	18
14	84	81
15	28	35
16	49	65
17	34	40
18	28	43
K= 11, L= 2		
0	30	37
1	97	95
2	100	116
3	271	283
4	223	238
5	105	81
6	84	80
7	302	341
8	154	186
9	63	65
10	174	184
11	94	99
12	95	102
14	44	47
15	69	88
16	61	70
17	32	28
K= 12, L= 2		
C	184	202
1	182	225
2	137	129
3	52	49
4	74	94
5	243	291
6	167	165
7	114	116
8	133	122
9	83	87
10	107	143
11	62	61
12	44	58
13	32	35
14	78	88
15	23	30
16	55	72
K= 13, L= 2		
0	124	138
1	112	138
2	93	92
3	89	96
4	100	115
5	52	53
6	173	185
7	65	63
8	54	59
9	56	64
10	41	40
11	69	95
12	71	77
13	32	42
14	34	42
15	28	36
K= 14, L= 2		
0	0*	19
1	153	167
2	101	103
3	44	60
4	112	135
5	51	48
6	52	59
7	0*	38
8	63	74
9	47	39
10	47	51
11	51	65
12	23	18
13	20	21
14	37	46
K= 15, L= 2		
0	75	60
1	30	20
2	48	45
3	89	95
4	41	40
5	75	88
6	85	95
7	41	24
8	51	65
9	0*	29
10	37	41
11	0*	19
12	65	78
K= 16, L= 2		
0	62	71
1	44	46
2	37	10
3	65	66
4	122	136
5	54	54
6	60	74
7	74	95
8	30	16
9	0*	34
10	85	103
K= 17, L= 2		
0	0*	30
1	63	60
2	138	119
3	60	56
4	32	25
5	45	37
6	45	46
7	32	30
8	41	61
K= 18, L= 2		
0	76	63
1	47	45
2	25	32
3	45	46
K= 0, L= 3		
1	174	180
2	691	684
3	673	647
4	219	188
5	536	502
6	454	415
7	242	225
8	1167	1013
9	213	198
10	195	162
11	111	111
12	281	281
13	300	303
14	140	132
15	0*	3
16	68	65
17	0*	45
18	0*	23
19	0*	12
20	0*	12
21	42	38
K= 1, L= 3		
0	610	737
1	296	252
2	370	395
3	316	357
4	819	771
5	472	489
6	558	578
7	509	521
8	412	420
9	166	178
10	373	398
11	234	234
12	176	169
14	258	239
15	121	116
16	78	77
17	64	72
18	64	62
19	64	65
20	0*	43
21	55	60
K= 2, L= 3		
0	351	322
1	333	329
2	965	898
3	584	606
4	865	871
5	42	36
6	690	721
7	679	734
8	422	430
9	155	156
10	244	225
11	156	159
12	224	190
13	251	235
14	305	287
15	109	116
16	69	46
17	124	131
18	116	111
19	59	48
20	64	69
21	95	93
K= 3, L= 3		
0	100	43
1	466	388
2	674	623
3	434	475
4	375	395
5	644	637
6	467	446
7	496	458
8	448	485
9	345	369
10	132	137
11	101	110
12	49	62
13	208	197
14	227	237
15	144	163
16	72	71
17	68	76
18	136	138
19	64	55
20	74	83
21	42	46
K= 4, L= 3		
0	1627	1261
1	1501	1154
2	470	355
3	357	360
4	135	116
5	187	201
6	265	278
7	287	336
8	197	202
9	304	300
10	216	209
11	378	362
12	90	92
13	361	350
14	219	237
15	104	120
16	80	84
17	0*	13
18	0*	20
19	0*	7
20	0*	27
21	36	27
K= 5, L= 3		
0	768	620
1	627	609
2	555	566
3	151	164
4	468	580
5	460	494
6	109	125
7	188	156
8	517	573
9	365	368
10	224	213
11	68	66
12	194	209
13	102	111
14	91	83
15	90	91
16	92	94
17	0*	20
18	102	110
19	51	48
20	42	41
21	30	39
K= 6, L= 3		
0	63	20
1	540	462
2	548	535
3	412	382
4	211	201
5	295	318
6	367	394
7	520	544
8	221	229
9	164	160
10	191	195
11	248	247
12	225	207
13	81	67
14	0*	35
15	0*	36
16	95	102
17	63	51
18	55	69
19	55	72
20	51	59
K= 7, L= 3		
0	288	317
1	267	239
2	319	280
3	465	444
4	408	414
5	274	287
6	191	172
7	127	120
8	115	121
9	332	312
10	181	187
11	344	355
12	0*	33
13	152	167
14	146	154
15	78	76
16	0*	30
17	68	64
18	51	44
19	47	49
20	44	62
K= 8, L= 3		
0	207	158
1	143	105
2	512	514
3	272	260
4	182	192
5	396	387
6	231	237
7	26	38
8	235	252
9	273	272
10	63	55
11	246	246
12	78	84
13	78	80
14	0*	15
15	0*	30
16	100	93
K= 9, L= 3		
0	365	334
1	129	114
2	66	87
3	428	431
4	210	189
5	61	61
6	259	262
7	339	351
8	112	122
9	272	286
10	265	288
11	0*	25
12	0*	42
13	143	147
14	63	59
15	26	35
16	36	30
K= 10, L= 3		
0	372	306
1	444	398
2	139	100
3	383	361
4	117	115
5	252	271
6	131	158
7	143	149
8	78	79
9	78	96
10	131	148
11	140	148
12	131	158
13	39	46
14	112	105
15	82	72
K= 11, L= 3		
0	75	67
1	234	230
2	116	107
3	449	434
4	225	227
5	133	134
6	109	117
7	21	35
8	144	132
9	75	90
10	132	139
11	193	189
12	59	56
13	129	152
14	0*	44
15	0*	34
16	68	71
17	44	57
K= 12, L= 3		
0	39	30
1	125	117
2	131	149
3	177	172
4	249	255
5	178	198
6	42	53
7	115	127
8	49	57
9	63	60
10	195	216
11	42	48
12	30	23
13	149	143
14	89	79
15	74	70
16	33	36
K= 13, L= 3		
0	57	71
1	215	218
2	135	125
3	68	71
4	69	67
5	143	161
6	0*	12
7	71	67
8	63	71
9	69	84
10	61	44
11	90	98
12	69	69
13	53	55
14	47	61
15	69	89
K= 14, L= 3		
0	63	64
1	33	23
2	63	79
3	111	112
4	64	53
5	71	79
6	106	108
7	77	87
8	66	67
9	53	45
10	81	78
11	42	43
12	26	29
13	72	75
K= 15, L= 3		
0	71	84
1	57	39
2	0*	29
3	81	89
4	96	98
5	74	78
6	47	57
7	0*	17
8	0*	34
9	47	50
10	47	50
11	47	57
12	39	45
K= 16, L= 3		
0	59	61
1	42	55
2	57	65
3	36	29
4	0*	20
5	0*	26
6	0*	16
7	0*	26
8	47	57
K= 17, L= 3		
0	47	33
1	33	50
2	36	30
3	0*	29
4	0*	20
5	0*	30
6	0*	32
7	30	58
K= 0, L= 4		
0	246	213
1	912	781
2	466	363
3	408	406
4	826	760
5	53	51
6	466	422
7	475	476
8	101	97
9	12	11
10	153	158
11	299	296
12	303	295
13	141	135
14	139	151
15	53	42
16	84	93
17	107	116
18	50	45
19	43	42
K= 1, L= 4		
0	104	103
1	671	680
2	864	742
3	524	500
4	381	431
5	559	609
6	460	419
7	144	155
8	84	97
9	296	299
10	284	274
11	93	100
12	138	139
13	63	61
14	189	176
15	208	204
16	62	69
17	21	38
18	0*	8
19	46	46
20	0*	22
21	28	28
K= 2, L= 4		
0	159	176
1	458	457
2	654	657
3	772	794
4	528	487
5	232	237
6	287	301
7	278	276
8	63	72
9	233	234
10	55	51
11	347	368
12	436	451
13	159	156
14	83	85
15	76	80
16	0*	20
17	0*	31
18	0*	8
19	53	40
20	0*	13
21	17	19
K= 3, L= 4		
0	528	549
1	945	842
2	794	753
3	387	333
4	963	910
5	138	129
6	182	189
7	61	46
8	208	221
9	190	193
10	104	106
11	353	339
12	182	208
13	264	281
14	301	315
15	53	63
16	79	86
17	41	49
18	0*	11
19	0*	23
20	45	46
21	25	35
K= 4, L= 4		
0	30	28
1	429	418
2	431	469
3	354	354
4	754	746
5	141	171
6	580	561
7	238	209
8	371	367
9	259	282
10	194	209
11	173	171
12	198	191
13	153	148
14	109	116
15	0*	13
16	117	118
17	0*	4
18	59	59
19	0*	33
20	30	31
K= 5, L= 4		
0	848	723
1	434	476
2	227	238
3	323	328
4	499	484
5	126	86
6	359	354
7	179	191
8	98	89
9	332	319
10	183	196
11	171	162
12	83	76
13	0*	17
14	190	172
15	66	71
16	0*	59
17	0*	57
18	0*	32
19	0*	32
20	30	34
K= 6, L= 4		
0	101	76
1	418	395
2	535	540
3	277	304
4	469	417
5	136	132
6	119	105
7	279	288
8	0*	3
9	0*	39
10	184	167
11	343	357
12	218	226
13	118	132
14	97	111
15	28	31
16	48	53
17	61	69
18	54	60
K= 7, L= 4		
0	259	203
1	566	509
2	242	224
3	169	196
4	158	170
5	330	334
6	139	144
7	134	148
8	61	71
9	275	282
10	200	220
11	115	127
12	0*	7
13	205	226
14	37	50
15	133	146
16	100	96
17	43	43
18	0*	30
19	48	53
K= 8, L= 4		
0	189	185
1	468	437
2	175	156
3	318	319
4	177	161
5	154	110
6	98	107
7	371	358
8	182	180
9	106	102
10	159	159
11	139	149
12	113	124
13	183	180
14	59	65
15	0*	27
16	0*	19
17	86	82
18	63	63
19	30	46
K= 9, L= 4		
0	69	56
1	257	248
2	145	143
3	335	349
4	295	298
5	266	266
6	187	169
7	116	96
8	127	136
9	264	262
10	120	131
11	93	85
12	133	127
13	0*	22
14	21	25
15	90	94
16	51	62
17	0*	24
18	39	55
K= 10, L= 4		
0	181	241
1	250	253
2	345	360
3	106	113
4	173	164
5	64	76
6	192	186
7	217	195
8	59	58
9	89	68
10	84	99
11	57	51
12	117	107
13	140	136
14	51	60
15	46	46
16	39	44
17	37	45
K= 11, L= 4		
0	21	25
1	240	274
2	147	154
3	250	271
4	151	160
5	247	221
6	195	186
7	94	98
8	117	133
9	76	84
10	72	48
11	69	57
12	92	110
13	0*	19
14	51	51
15	0*	3
16	45	48
K= 12, L= 4		
0	177	211
1	129	157
2	113	109
3	309	269
4	123	120
5	164	161
6	138	143
7	61	53
8	159	161
9	67	77
10	0*	29
11	0*	46
12	101	87
13	28	36
14	0*	2
15	48	58
K= 13, L= 4		
0	233	261
1	0*	28
2	0*	4
3	143	148
4	66	91
5	55	57
6	114	110
7	123	128
8	48	53
9	103	114
10	93	92
11	0*	24
12	54	68
13	41	45
14	33	28
K= 14, L= 4		
0	0*	8
1	59	64
2	149	159
3	0*	40
4	55	51
5	50	50
6	46	48
7	121	128
8	77	74
9	25	30
K= 15, L= 4		
0	46	36
1	61	70
2	0*	40
3	99	105
4	28	35
5	114	117
6	43	38
7	53	53
8	41	35
9	81	89
10	41	42
K= 16, L= 4		
0	0*	1
1	63	61
2	0*	22
3	101	86
4	45	59
5	35	47
6	45	52
7	0*	22
8	39	40
K= 17, L= 4		
0	25	29
1	88	94
2	87	93
3	0*	31
4	45	55
K= 0, L= 5		
1	0*	56
2	228	243
3	86	85
4	206	202
5	446	485
6	479	494
7	109	96
8	576	616
9	128	115

Table 4 (*cont.*)

H	FO	FC
K=	0, L=	5
10	267	297
11	188	191
12	100	104
13	0*	45
14	0*	22
15	0*	29
16	61	69
17	0*	7
18	0*	35
19	0*	26
20	74	77
K=	1, L=	5
0	621	715
1	218	249
2	234	280
3	318	328
4	183	220
5	236	255
6	533	590
7	239	238
8	450	463
9	177	175
10	246	229
11	359	367
12	75	89
13	152	204
14	100	94
15	64	66
16	94	90
17	44	39
18	33	36
19	30	32
K=	2, L=	5
0	151	154
1	359	377
2	520	616
3	163	162
4	251	247
5	572	595
6	154	140
7	384	441
8	347	363
9	331	333
10	128	120
11	281	282
12	289	291
13	131	144
14	60	62
15	0*	12
16	65	61
17	0*	19
18	44	41
K=	3, L=	5
0	130	97
1	403	396
2	355	348
3	497	482
4	580	605
5	900	884
6	209	220
7	122	132
8	114	120
9	309	344
10	371	312
11	392	391
12	169	177
13	103	94
14	97	109
15	0*	23
16	0*	9
17	0*	36
18	44	41
19	38	37
K=	4, L=	5
0	80	85
1	492	614
2	249	280
3	561	631
4	126	142
5	190	151
6	143	158
7	394	392
8	194	161
9	61	71
10	229	240
11	151	136
12	61	72
13	208	202
14	67	87
15	0*	55
16	58	51
17	76	76
18	52	44
19	38	49
K=	5, L=	5
0	383	386
1	332	390
2	122	131
3	138	136
4	199	229
5	353	382
6	233	235
7	173	173
8	112	71
9	315	296
10	122	120
11	53	43
12	163	160
13	141	131
14	100	95
15	111	128
16	67	84
17	42	42
18	0*	29
19	65	42
20	42	50
K=	6, L=	5
0	138	100
1	151	161
2	370	380
3	250	237
4	101	110
5	89	92
6	135	143
7	353	330
8	60	60
9	246	227
10	53	35
11	109	105
12	150	140
13	140	124
14	79	91
15	0*	25
16	53	62
17	0*	18
18	0*	32
19	60	50
K=	7, L=	5
0	269	265
1	336	288
2	108	78
3	118	100
4	349	345
5	166	141
6	246	225
7	141	135
8	133	146
9	132	113
10	262	221
11	128	109
12	128	101
13	0*	36
14	60	63
15	0*	29
16	0*	29
17	0*	29
18	109	87
19	46	43
K=	8, L=	5
0	213	205
1	304	288
2	329	290
3	90	76
4	101	77
5	129	103
6	365	337
7	221	190
8	443	388
9	55	59
10	69	81
11	116	124
12	133	114
13	46	28
14	123	117
15	125	137
16	55	93
17	0*	41
18	67	65
K=	9, L=	5
0	443	396
1	478	458
2	224	203
3	206	168
4	245	229
5	200	199
6	187	156
7	95	73
8	191	186
9	81	78
10	313	267
11	64	63
12	84	82
13	159	183
14	92	101
K=	10, L=	5
0	267	255
1	185	157
2	406	316
3	264	245
4	216	181
5	190	168
6	102	82
7	106	86
8	158	145
9	128	93
10	125	107
11	128	130
12	117	79
13	67	52
14	84	76
15	0*	66
16	44	40
K=	11, L=	5
0	240	187
1	111	96
2	219	193
3	158	155
4	128	99
5	200	180
6	140	120
7	146	143
8	120	113
9	166	154
10	87	73
11	48	38
12	74	69
13	87	90
14	55	43
15	56	35
K=	12, L=	5
0	62	68
1	0*	48
2	147	119
3	229	197
4	62	61
5	126	115
6	149	133
7	151	139
8	137	104
9	44	38
10	108	101
11	0*	17
12	0*	18
13	70	53
K=	13, L=	5
0	72	74
1	125	93
2	58	52
3	70	68
4	55	53
5	105	82
6	0*	34
7	0*	8
8	70	60
9	92	95
10	48	36
11	55	67
K=	14, L=	5
0	56	45
1	133	86
2	122	100
3	121	92
4	53	43
5	30	21
6	61	67
7	85	76
8	42	34
9	86	82
10	40	46
11	55	66
K=	15, L=	5
0	120	101
1	44	32
2	185	145
3	0*	40
4	86	79
5	61	54
6	52	50
7	0*	25
8	38	44
9	42	43
K=	16, L=	5
0	107	99
1	46	41
2	101	94
3	99	62
4	91	58
5	33	30
6	58	51
7	46	52
K=	0, L=	6
0	646	687
1	691	724
2	102	93
3	305	321
4	0*	9
5	117	84
6	47	49
7	60	57
8	122	108
9	96	82
10	241	205
11	295	249
12	187	182
13	313	311
14	127	124
15	70	75
16	0*	26
17	0*	4
18	40	39
19	0*	2
20	26	33
K=	1, L=	6
0	177	172
1	466	459
2	334	349
3	344	364
4	275	298
5	200	233
6	270	269
7	195	191
8	168	157
9	250	253
10	219	210
11	282	274
12	153	136
13	81	78
14	76	89
15	90	83
16	75	81
17	56	59
18	90	94
19	42	58
20	26	43
K=	2, L=	6
0	79	83
1	221	229
2	309	324
3	550	578
4	317	293
5	192	186
6	310	308
7	418	400
8	144	129
9	170	155
10	182	172
11	97	63
12	142	137
13	65	75
14	60	63
15	37	35
16	73	64
17	79	77
18	97	95
K=	3, L=	6
0	675	638
1	402	390
2	303	298
3	171	169
4	90	88
5	378	312
6	209	225
7	232	226
8	241	252
9	75	92
10	284	281
11	95	91
12	143	133
13	136	145
14	179	176
15	62	69
16	54	53
17	54	66
18	0*	5
19	37	29
K=	4, L=	6
0	223	208
1	218	215
2	381	666
3	50	38
4	131	150
5	148	170
6	131	126
7	266	255
8	321	345
9	243	241
10	163	165
11	171	163
12	101	91
13	70	65
14	58	64
15	91	80
16	62	85
17	0*	30
18	0*	32
19	37	57
K=	5, L=	6
0	673	565
1	315	306
2	183	187
3	343	282
4	371	364
5	458	462
6	293	297
7	130	111
8	139	121
9	119	126
10	82	80
11	203	206
12	97	102
13	72	69
14	56	64
15	47	57
16	86	93
17	45	54
K=	6, L=	6
0	67	72
1	203	163
2	339	303
3	315	304
4	464	449
5	75	70
6	106	111
7	228	223
8	92	89
9	91	94
10	185	176
11	107	128
12	135	138
13	0*	7
14	107	125
15	78	80
16	70	64
17	42	45
18	34	33
K=	7, L=	6
0	106	98
1	245	219
2	307	280
3	90	90
4	253	230
5	347	330
6	263	246
7	83	64
8	128	116
9	106	114
10	216	225
11	70	72
12	133	131
13	108	128
14	47	59
15	78	80
16	45	54
17	89	88
K=	8, L=	6
0	231	209
1	353	344
2	159	136
3	439	406
4	163	144
5	124	112
6	185	181
7	58	60
8	132	135
9	177	164
10	154	158
11	111	108
12	114	113
13	109	102
14	58	71
15	42	35
16	37	37
17	30	36
K=	9, L=	6
0	283	228
1	252	230
2	115	109
3	58	31
4	257	261
5	272	232
6	178	169
7	0*	28
8	177	166
9	209	237
10	0*	23
11	50	41
12	34	44
13	75	69
14	42	34
15	111	110
16	34	45
K=	10, L=	6
0	58	55
1	109	84
2	113	99
3	107	72
4	83	78
5	0*	42
6	165	182
7	235	223
8	112	130
9	56	48
10	0*	24
11	56	70
12	75	70
13	52	47
14	45	54
K=	11, L=	6
0	242	246
1	136	116
2	94	87
3	101	85
4	173	164
5	130	113
6	111	128
7	56	49
8	58	67
9	89	77
10	92	98
11	83	69
12	0*	30
13	26	31
14	30	42
K=	12, L=	6
0	0*	1
1	52	57
2	128	117
3	132	121
4	65	56
5	83	77
6	64	51
7	65	61
8	101	106
9	50	62
10	47	53
K=	13, L=	6
0	97	86
1	50	51
2	0*	14
3	0*	14
4	85	95
5	0*	10
6	98	103
7	50	66
8	42	40
9	72	81
10	65	60
11	42	48
K=	14, L=	6
0	78	75
1	45	27
2	45	39
3	73	78
4	102	104
5	47	51
6	0*	27
7	0*	23
8	40	44
K=	15, L=	6
0	121	103
1	50	56
2	50	44
3	76	85
K=	16, L=	6
0	58	70
K=	0, L=	7
1	390	349
2	322	318
3	77	76
4	247	214
5	549	514
6	402	328
7	87	88
8	300	265
9	105	90
10	0*	29
11	53	37
12	101	85
13	57	54
14	53	24
15	68	47
16	0*	37
17	0*	28
18	44	41
19	39	58
K=	1, L=	7
0	106	122
1	172	168
2	121	136
3	391	388
4	450	383
5	143	154
6	143	143
7	414	380
8	222	191
9	159	146
10	270	247
11	72	77
12	275	252
13	180	166
14	78	76
15	69	58
K=	2, L=	7
0	117	101
1	91	96
2	175	215
3	252	272
4	283	300
5	212	197
6	225	231
7	134	123
8	218	206
9	151	135
10	190	179
11	253	237
12	262	256
13	84	90
14	63	53
K=	3, L=	7
0	269	236
1	419	361
2	423	389
3	227	212
4	283	272
5	106	100
6	219	213
7	84	78
8	158	149
9	126	136
10	68	56
11	118	126
12	102	85
13	57	45
14	57	55
15	66	76
16	57	54
K=	4, L=	7
0	266	265
1	196	184
2	137	140
3	245	263
4	197	211
5	146	102
6	51	29
7	288	290
8	168	188
9	47	33
10	152	153
11	166	158
12	74	69
13	63	54
14	51	46
K=	5, L=	7
0	142	159
1	85	43
2	222	214
3	166	180
4	207	219
5	259	274
6	256	265
7	136	137
8	288	275
9	151	171
10	59	43
11	77	56
12	53	47
13	0*	5
14	78	76
15	42	34
16	47	54
17	53	73
K=	6, L=	7
0	68	76
1	135	132
2	266	311
3	96	100
4	142	185
5	140	154
6	151	160
7	189	210
8	53	28
9	156	153
10	69	65
11	59	45
12	148	138
13	55	60
14	0*	17
15	49	51
16	0*	16
17	55	54
K=	7, L=	7
0	363	380
1	139	146
2	161	125
3	148	167
4	137	154
5	198	199
6	86	103
7	158	154
8	128	116
9	0*	50
10	150	111
11	147	158
12	0*	28
13	77	76
14	82	94
15	0*	24
16	66	75
17	55	63
K=	8, L=	7
0	326	355
1	84	70
2	217	232
3	71	70
4	192	219
5	110	90
6	130	112
7	114	120
8	167	152
9	136	130
10	33	42
11	33	32
12	121	120
13	75	82
14	66	61
15	71	65
16	30	27
K=	9, L=	7
0	57	51
1	194	222
2	180	146
3	166	188
4	74	72
6	179	195
7	66	59
8	147	165
9	55	43
10	55	50
11	51	61
12	0*	19
13	42	29
14	39	38
15	61	67
K=	10, L=	7
0	102	84
1	157	149
2	119	103
3	57	61
4	77	80
5	142	127
6	167	163
7	64	70
8	59	67
9	55	50
10	72	77
11	68	70
12	51	43
13	55	43
14	44	36
K=	11, L=	7
0	92	87
1	105	98
2	87	96
3	72	63
4	110	104
5	0*	16
6	61	70
7	44	37
8	133	132
9	89	102
10	51	67
11	66	51
12	91	90
13	49	46
K=	12, L=	7
0	84	83
1	148	143
2	51	47
3	82	77
4	78	89
5	0*	14
6	86	76
7	53	52
8	44	55
9	0*	26
10	85	89
11	64	67
12	47	52
K=	13, L=	7
0	0*	0
1	162	167
2	74	77
3	66	62
4	53	47
5	101	98
6	0*	4
7	42	37
8	59	52
9	61	74
10	26	25
K=	14, L=	7
0	89	73
1	93	95
2	0*	34
3	42	50
4	39	41
5	36	44
6	49	51
7	51	47
8	33	32
K=	15, L=	7
0	0*	20
1	30	27
2	89	74
3	0*	27
4	0*	31
5	36	43
K=	0, L=	8
0	175	192
1	70	76
2	82	61
3	330	264
4	113	80
5	195	173
6	175	158
7	0*	19
8	376	320
9	137	137
10	124	94
11	89	33
12	137	118
K=	1, L=	8
0	80	82
1	325	295
2	334	350
3	352	344
4	201	228
5	130	132
6	103	98
7	52	30
8	161	166
9	118	104
10	227	231
11	52	60
12	30	41
13	25	42
14	0*	40
15	83	75
16	55	55
17	46	45
K=	2, L=	8
0	327	303
1	396	428
2	65	64
3	235	221
4	157	190
5	273	273
6	113	118
7	113	119
8	135	122
9	157	175
11	107	99
12	70	79
13	132	132
14	0*	12
15	60	72
K=	3, L=	8
0	278	234
1	125	77
2	260	234
3	519	459
4	152	154
5	163	146
6	214	214
7	237	235
8	58	61
9	70	72
10	131	169
11	125	124
12	135	132
13	114	112
K=	4, L=	8
0	0*	29
1	221	230
2	278	306
3	55	88
4	67	57
5	276	303
6	0*	20
7	0*	32
8	107	119
9	245	267
K=	5, L=	8
0	423	393
1	203	225
2	85	78
3	249	260
4	65	45
5	103	101
6	119	138
7	226	220
8	58	53
9	80	76
10	65	71
11	67	82
12	90	101
13	76	90
14	49	49
K=	6, L=	8
0	187	208
1	250	239
2	221	241
3	131	123
4	151	158
5	58	44
6	65	72
7	140	154
8	80	88
9	63	64
10	74	84
11	67	57
12	74	88
13	52	55
14	60	66
15	58	64
K=	7, L=	8
0	67	56
1	124	143
2	209	254
3	78	66
4	157	171
5	242	272
6	151	149
7	92	90
8	120	142
9	78	90
10	70	82
11	67	88
12	52	48
13	49	41
14	39	31
15	30	29
K=	8, L=	8
0	314	366
1	118	65
2	119	118
3	140	107
4	118	125
5	95	89
6	161	163
7	119	103
8	157	169
9	58	65
10	78	90
11	0*	26
12	52	56
13	46	52
14	39	48
K=	9, L=	8
0	120	86
1	120	104
2	229	241
3	131	131
4	169	172
5	119	133
6	52	45
7	117	107
8	143	134
9	74	78
10	0*	45
11	107	106
12	89	69
13	43	42
14	35	45
K=	10, L=	8
0	158	165
1	70	55
2	0*	50
3	94	93
4	123	133
5	72	84
6	168	186
7	117	119
8	107	104
9	55	47
10	120	128
11	63	71
12	39	40
13	25	32
K=	11, L=	8
0	0*	42
1	114	105
2	114	74
3	132	155
4	0*	34
5	63	71
6	0*	42
7	30	34
8	80	97
9	39	55
10	39	49
11	39	61
K=	12, L=	8
0	52	30
1	63	54
2	106	94
3	104	73
4	0*	11
5	83	91
6	65	63
7	55	56
8	65	73
K=	13, L=	8
0	46	53
1	52	35
2	0*	23
3	0*	39
4	58	78
5	43	36
6	46	52
7	39	55
8	39	67
K=	14, L=	8
0	74	78
1	0*	6
2	0*	45
3	55	26
4	30	71
5	65	46
K=	0, L=	9
1	402	369
2	127	152
3	423	398
4	128	132
5	169	168
6	168	187
7	167	164
8	0*	61
9	167	166
10	0*	8
11	139	131
K=	1, L=	9
0	0*	31
1	414	424
2	78	83
3	310	323
4	175	176
5	0*	41
6	212	209
7	122	92
8	104	112
9	113	91
10	28	33
11	105	97
12	68	60
13	96	82
K=	2, L=	9
0	136	126
1	150	143
2	179	180
3	131	113
4	282	343
5	244	247
6	210	199
7	102	91
8	86	101
9	162	147
10	86	81
11	61	56
12	63	58
13	91	92
K=	3, L=	9
0	200	162
1	157	158
2	255	204
4	96	92
5	221	208
6	240	227
7	293	300
K=	4, L=	9
0	307	345
1	0*	73
2	0*	34
3	141	171
4	160	207
K=	0, L=	10
0	255	232
1	0*	49
2	125	133
3	0*	57
4	157	154
5	0*	40
6	274	245
K=	1, L=	10
0	77	83
1	96	99
2	171	164
3	165	157
4	124	104
5	135	132
6	121	119
7	0*	55
8	108	73
9	0*	27
10	0*	22
11	49	42
12	94	55
13	49	48
K=	2, L=	10
0	147	133
1	205	213
2	86	103
3	43	61
4	137	129
5	81	96
6	35	57
7	0*	14
8	61	70
9	59	64
10	61	68
11	59	58
K=	3, L=	10
0	0*	3
1	121	106
2	76	82
3	193	167
K=	0, L=	11
1	117	100
2	0*	38
3	0*	63
4	0*	26
5	231	221
K=	1, L=	11
0	0*	12
1	130	142
2	108	98
3	143	151
4	109	102
5	51	34
6	50	51
7	171	168
K=	2, L=	11
0	148	135
1	136	126
2	0*	20
3	0*	6
4	33	50
5	75	82
6	0*	10
7	60	74
8	33	27
9	75	84
10	0*	25
11	73	79
K=	3, L=	11
0	0*	4
1	86	85
2	60	74
3	97	94
4	0*	36
5	93	66
6	84	68
7	0*	18
8	64	65
9	83	106
K=	0, L=	12
0	0*	30
1	111	109
2	0*	58
3	0*	15
4	0*	39
5	0*	27
K=	1, L=	12
0	0*	18
1	80	83
2	90	93
3	56	75
4	0*	39
5	0*	32
6	0*	37
7	39	39
8	40	67
K=	2, L=	12
0	56	60
1	125	117
2	0*	38
3	82	84
4	70	76
5	49	50
6	96	85
7	40	40
K=	3, L=	12
0	0*	39
1	142	120
2	0*	16
3	74	66
4	85	90
5	72	55
6	71	73

Table 5. *Agreement summary*

2406 observed reflexions, $|F_o\ \max|=185\cdot7$; $R=0\cdot090$.

Category	Limits			Number
1	$\lvert\Delta F\rvert\leq 1\lvert F_{\rm th}\rvert$	or	$\lvert\Delta F\rvert/\lvert F_o\rvert\leq 2R$	2348
2	$1\lvert F_{\rm th}\rvert<\lvert\Delta F\rvert\leq 2\lvert F_{\rm th}\rvert$	or	$2R<\lvert\Delta F\rvert/\lvert F_o\rvert\leq 3R$	49
3	$2\lvert F_{\rm th}\rvert<\lvert\Delta F\rvert\leq 3\lvert F_{\rm th}\rvert$	or	$3R<\lvert\Delta F\rvert/\lvert F_o\rvert\leq 4R$	7
4	$3\lvert F_{\rm th}\rvert<\lvert\Delta F\rvert$	or	$4R<\lvert\Delta F\rvert/\lvert F_o\rvert$	2
	318 unobserved reflexions, $\lvert F_c\ \max\rvert=7\cdot3$			
1	$\lvert F_c\rvert\leq 1\cdot0\lvert F_{\rm th}\rvert$			291
2	$1\cdot0\lvert F_{\rm th}\rvert<\lvert F_c\rvert\leq 1\cdot5\lvert F_{\rm th}\rvert$			24
3	$1\cdot5\lvert F_{\rm th}\rvert<\lvert F_c\rvert\leq 2\cdot0\lvert F_{\rm th}\rvert$			2
4	$2\cdot0\lvert F_{\rm th}\rvert<\lvert F_c\rvert$			1

$|F_{\rm th}|$ = threshold amplitude = 2·4 to 5·5

reflexions, 210 and 501, which seemed to suffer from extinction errors were also excluded from the refinement.

The weighting scheme for least squares was that described by Mills & Rollett (1961). It was of the form $\sqrt{w}=[1/(1+\{(|F_o|-b)/a\}^2)]^{1/2}$, where a and b were taken as 10·88 and −9·57 respectively.

All the computations were carried out on the IBM/360 system with programs written in Fortran IV by Ahmed, Huber & Pippy (1966).

Discussion of the structure

A perspective view of the molecule showing the atom numbering is given in Fig. 2.

The bond lengths are given in Table 6, and the angles are listed in Table 7. The estimated standard deviations of the bonds and angles, calculated by the expressions of Ahmed & Cruickshank (1953) and from *International Tables for X-ray Crystallography* (1959) respectively, based on the e.s.d.'s of the coordinates in Table 1, were considered to be too low because of the use of block-diagonal rather than full-matrix refinement, and also because thermal corrections were not applied and the unobserved reflexions were omitted. The e.s.d. values were therefore multiplied by two giving a range of 0·02 to 0·03 Å for the bonds and 1·2 to 2·2° for the angles. The highest e.s.d.'s were obtained for the bonds of the isopropyl group and of the benzene ring. On calculating the e.s.d. of a C(sp^3)–C(sp^3) bond from the scatter from the mean, a value of 0·024 Å was obtained. The observed C–C bond length variations are reasonable and their mean value (1·541 Å) agrees well with 1·537 Å given by Sutton (1965). The C(22)–C(23) bond of 1·48 Å is also in agreement with the accepted value of 1·501 Å for the C(sp^3)–C(sp^2) bond (Lide, 1962).

Table 6. *Bond lengths*

C(1)—C(2)	1·58 Å	C(16)–C(17)	1·52 Å
C(1)—C(13)	1·56	C(16)–C(18)	1·51
C(1)—C(16)	1·56	C(22)–C(23)	1·48
C(2)—C(3)	1·55	C(23)–C(24)	1·37
C(3)—C(4)	1·54	C(23)–C(28)	1·36
C(3)—C(14)	1·53	C(24)–C(25)	1·40
C(4)—C(5)	1·56	C(25)–C(26)	1·37
C(4)—C(15)	1·57	C(26)–C(27)	1·36
C(4)—C(29)	1·51	C(27)–C(28)	1·36
C(5)—C(6)	1·49	O(11)–C(10)	1·44
C(5)—C(10)	1·54	O(11)–C(12)	1·46
C(6)—C(7)	1·54	O(19)–C(1)	1·43
C(7)—C(8)	1·52	O(20)–C(2)	1·44
C(8)—C(9)	1·55	O(21)–C(3)	1·43
C(8)—C(31)	1·54	O(21)–C(22)	1·43
C(9)—C(10)	1·51	O(30)–C(5)	1·43
C(10)–C(14)	1·58	O(32)–C(9)	1·44
C(12)–C(13)	1·56	O(33)–C(12)	1·36
C(12)–C(15)	1·53	O(35)–C(14)	1·42
C(13)–C(14)	1·56	Br —C(26)	1·91
C(13)–C(34)	1·54		

The average values for the benzene ring bonds and angles are 1·371 Å and 120·0° respectively and they compare well with those obtained by Abrahamsson & Nilsson (1966) for *p*-bromobenzyl norbormide (1·383 Å and 120·2°).

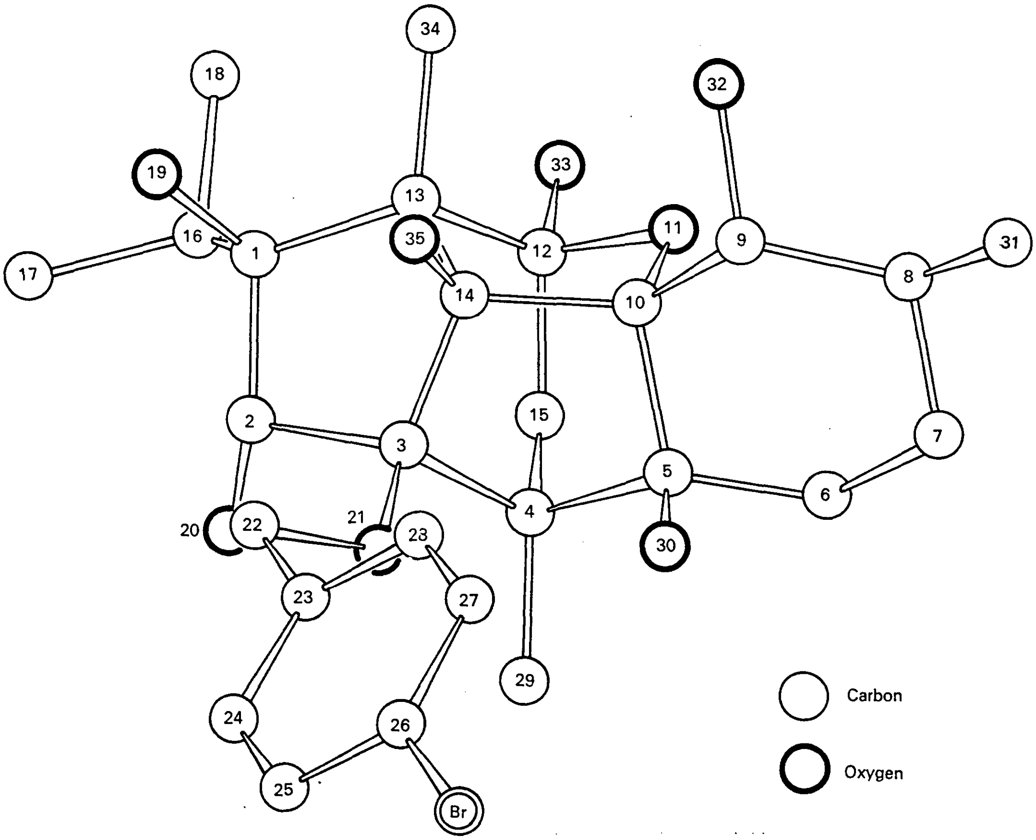

Fig. 2. Perspective view of the molecule of ryanodol *p*-bromobenzyl ether.

The O–C bonds vary from 1·42 to 1·46 Å with the exception of the O(33)–C(12) bond of 1·36 Å. We are grateful to the referee for pointing out that this bond is not significantly shorter than the equational carbon–oxygen bonds observed in the anomeric position in pyranose sugars (Berman, Chu & Jeffrey, 1967).

The C–H bonds of the methyl groups range from 1·05 to 1·11 Å and the average of 30 angles of these groups is 109·5°. They ranged from 104·3 to 113·9°. The O–H bond lengths are listed in Table 8.

All the six-membered rings of the skeleton are in the chair form and the five-membered rings are in the envelope form.

The mean plane of the benzene ring, referred to axes $X'=ax$, $Y'=by$ and $Z'=cz$ is $0{\cdot}5841X'-0{\cdot}2186Y'-0{\cdot}7817Z'+0{\cdot}8995=0$. It was calculated using the procedure described by Blow (1960). The maximum distance of 0·011 Å from that plane was obtained for the C(28) atom; however, χ^2 is 2·7 and $P=40\%$ $(\nu=3)$ and therefore the deviations of the six atoms of

Table 7. *Bond angles*

C(2)—C(1)—C(13)	103·8°	C(9)—C(10)-O(11)	108·0°
C(2)—C(1)—C(16)	113·2	C(9)—C(10)-C(14)	121·0
C(2)—C(1)—O(19)	105·7	O(11)-C(10)-C(14)	103·6
C(13)-C(1)—C(16)	119·8	C(10)-O(11)-C(12)	105·6
C(13)-C(1)—O(19)	105·6	O(11)-C(12)-C(13)	100·3
C(16)-C(1)—O(19)	107·7	O(11)-C(12)-C(15)	106·1
C(1)—C(2)—C(3)	106·2	O(11)-C(12)-O(33)	108·6
C(1)—C(2)—O(20)	115·0	C(13)-C(12)-C(15)	113·7
C(3)—C(2)—O(20)	110·6	C(13)-C(12)-O(33)	112·7
C(2)—C(3)—C(4)	115·8	C(15)-C(12)-O(33)	114·2
C(2)—C(3)—C(14)	107·2	C(1)—C(13)-C(12)	115·3
C(2)—C(3)—O(21)	110·5	C(1)—C(13)-C(14)	106·3
C(4)—C(3)—C(14)	101·3	C(1)—C(13)-C(34)	114·4
C(4)—C(3)—O(21)	105·7	C(12)-C(13)-C(14)	96·8
C(14)-C(3)—O(21)	116·4	C(12)-C(13)-C(34)	108·6
C(3)—C(4)—C(5)	102·0	C(14)-C(13)-C(34)	114·3
C(3)—C(4)—C(15)	106·3	C(3)—C(14)-C(10)	107·3
C(3)—C(4)—C(29)	118·3	C(3)—C(14)-C(13)	101·6
C(5)—C(4)—C(15)	106·7	C(3)—C(14)-O(35)	113·1
C(5)—C(4)—C(29)	111·7	C(10)-C(14)-C(13)	104·9
C(15)-C(4)—C(29)	111·0	C(10)-C(14)-O(35)	113·5
C(4)—C(5)—C(6)	121·2	C(13)-C(14)-O(35)	115·4
C(4)—C(5)—C(10)	100·4	C(4)—C(15)-C(12)	118·1
C(4)—C(5)—O(30)	110·2	C(1)—C(16)-C(17)	120·9
C(6)—C(5)—C(10)	110·4	C(1)—C(16)-C(18)	118·3
C(6)—C(5)—O(30)	105·3	C(17)-C(16)-C(18)	118·8
C(10)-C(5)—O(30)	109·1	C(3)—O(21)-C(22)	119·7
C(5)—C(6)—C(7)	108·4	O(21)-C(22)-C(23)	121·5
C(6)—C(7)—C(8)	115·4	C(22)-C(23)-C(24)	118·8
C(7)—C(8)—C(9)	114·6	C(22)-C(23)-C(28)	122·5
C(7) -C(8) -C(31)	109·9	C(24)-C(23)-C(28)	111·5
C(9)—C(8)—C(31)	109·4	C(23)-C(24)-C(25)	110·4
C(8)—C(9)—C(10)	110·0	C(24)-C(25)-C(26)	113·1
C(8)—C(9)—O(32)	110·5	Br —C(26)-C(25)	108·8
C(10)-C(9)—O(32)	110·3	Br —C(26)-C(27)	121·7
C(5)—C(10)-C(9)	111·9	C(25)-C(26)-C(27)	107·6
C(5)—C(10)-O(11)	107·7	C(26)-C(27)-C(28)	121·0
C(5)—C(10)-C(14)	103·8	C(23)-C(28)-C(27)	121·0

Table 8. *Examination of the shortest* O···O *contacts*

Intramolecular	∠C–O–H	O–H	H···O	O···O	∠O–H···O
O(19)-H(191)···O(35)	123·4°	1·01 Å	2·52 Å	3·11 Å	116·5°
O(20)-H(201)···O(19)	77·8	1·11	2·96	3·56	114·5
O(33)-H(331)···O(11)	109·1	1·00	2·41	2·29*	71·3
O(35)-H(351)···O(32)	107·1	0·97	2·64	3·06	106·9
Intermolecular					
O(20)-H(201)···O(30)	77·8	1·11	2·68	2·78	83·0
O(30)-H(301)···O(20)	117·9	0·99	2·54	2·78	93·5
O(32)-H(321)···O(35)	115·3	0·99	2·58	3·45	145·6
O(33)-H(331)···O(32)	109·1	1·00	1·74	2·69	159·0
O(35)-H(351)···O(11)	107·1	0·97	2·47	3·07	119·9

* The unusually short O(33)···O(11) distance is due to the fact that the epoxy oxygen atom O(11) is separated only by one carbon atom from O(33) of the hydroxyl group.

the benzene ring from the plane are not significant. The C(22) and Br atoms were found to be situated on the opposite sides of the plane, at distances of 0·03 and 0·04 Å respectively and the C(22)–C(23) and C(26)–Br bonds make angles of only 1·2° with the benzene ring.

The molecules related by the twofold screw axes parallel to the *c* axis form hydrogen-bonded spirals. The packing of the molecules along the *c* axis can be seen in Fig. 3, and Fig. 4 shows three molecules of the spiral. The hydrogen bonding joining these molecules is between the atoms O(33) and O(32), which are 2·69 Å apart. The H(331)$\cdots$O(32) distance was found to be 1·74 Å and $<$O(33)–H(331)$\cdots$O(32) is 159·0°. This is the only hydrogen bond in the structure. All other hydrogen atoms of OH groups were found to make at least one intra- or intermolecular H$\cdots$O contact, which does not deviate significantly from the sum of the van der Waals radii. The hydrogen atoms are directed towards the neighbouring oxygen atoms optimizing the H$\cdots$O interactions. The shortest O$\cdots$O distances are examined in Table 8.

The O$\cdots$C intermolecular approaches are normal, the closest occurring between the molecules of the hydrogen-bonded spiral, and they are: O(33)$\cdots$C(9), 3·26 Å and O(19)$\cdots$C(31), 3·30 Å. The smallest C$\cdots$C distance is 3·56 Å.

The closest contacts of the bromine atom are to the atoms O(33) and C(22). The Br$\cdots$O(33) distance is 3·69 Å with H(331)$\cdots$Br of 3·47 Å and Br$\cdots$C(22) is 3·66 Å, the atoms H(221) and H(222) being situated at 3·10 and 3·30 Å from the bromine atom. All other bromine to carbon distances were found to be above 3·8 Å.

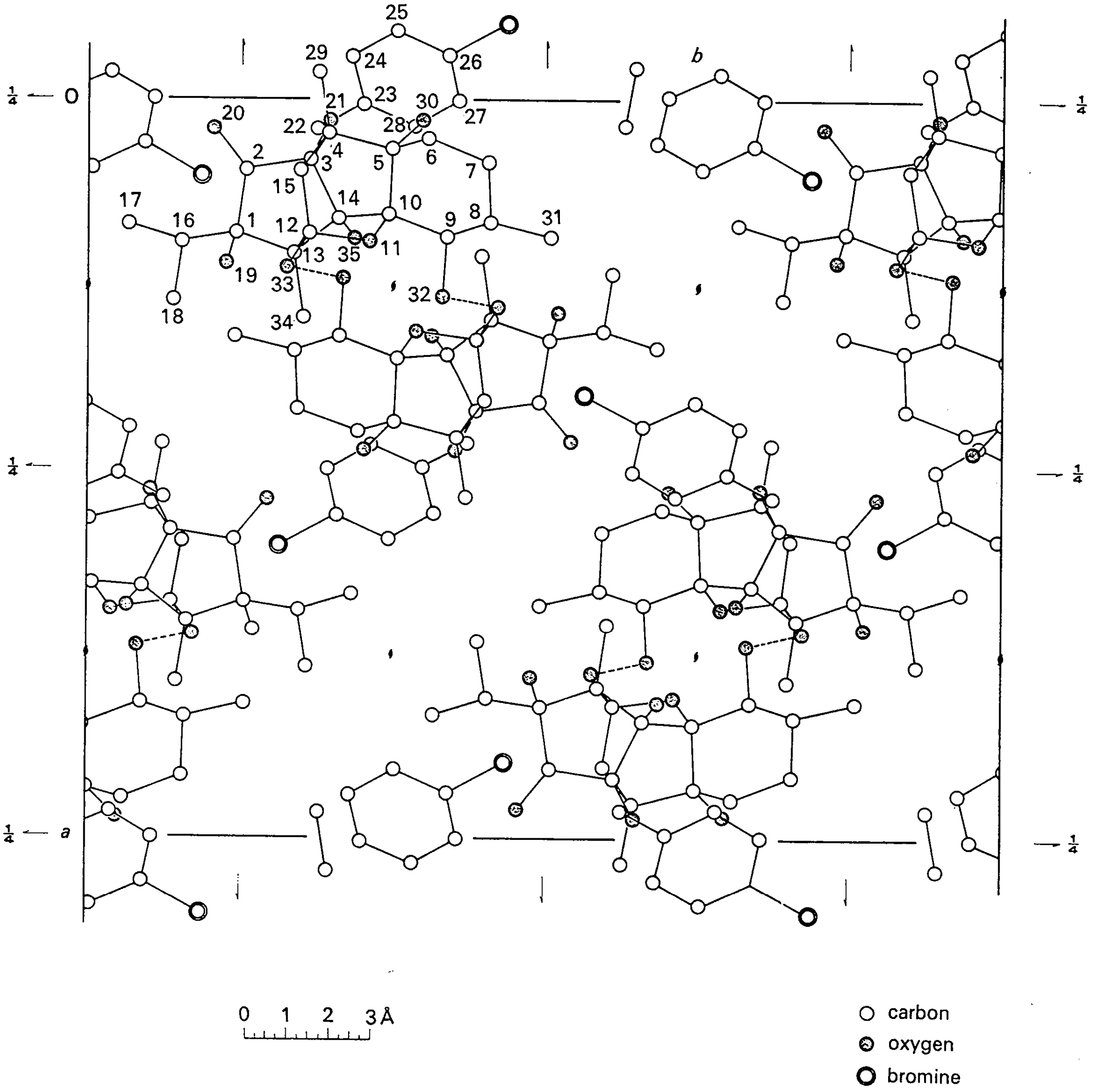

Fig. 3. Packing of the molecules in the (001) projection.

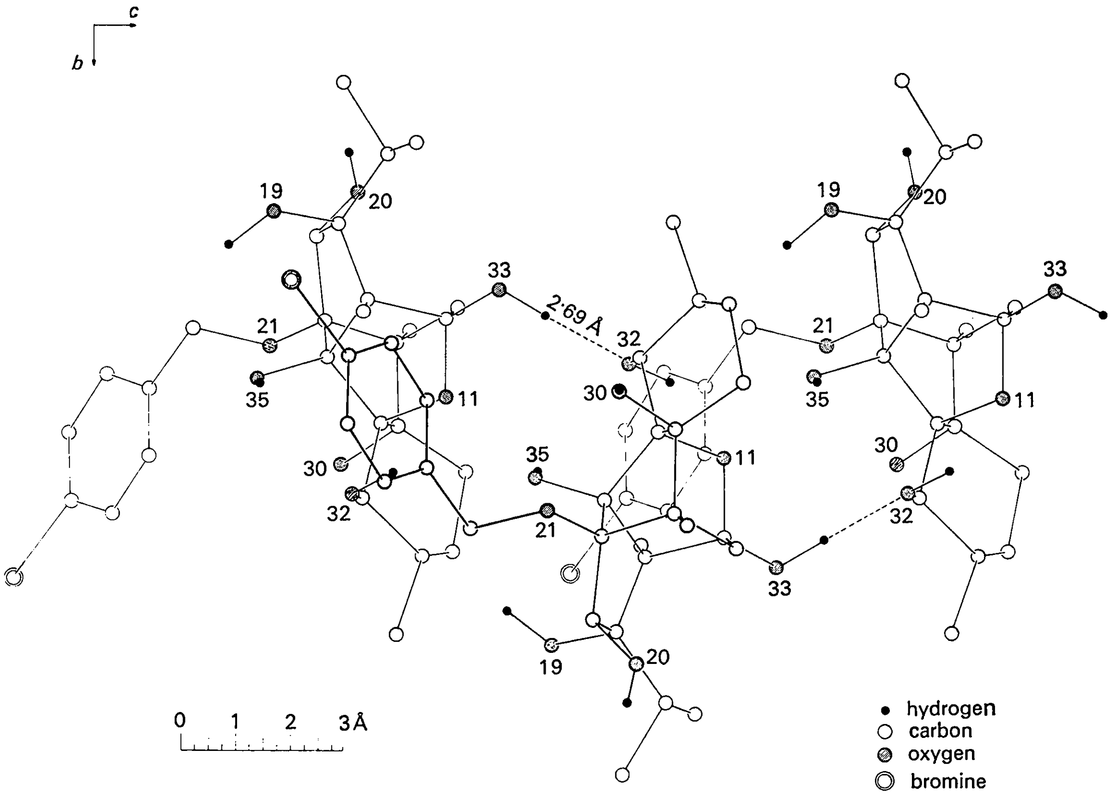

Fig. 4. Projection of three molecules along the *a* axis showing the hydrogen bonds.

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