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The Crystal Structure of the L-1-Monoglyceride of 11-Bromoundecanoic Acid

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The optically active 1-monoglyceride of 11-bromoundecanoic acid crystallizes in a monoclinic form with unit-cell dimensions $a = 5{\cdot}78 \pm 0{\cdot}02$, $b = 7{\cdot}43 \pm 0{\cdot}03$, $c = 37{\cdot}6 \pm 0{\cdot}1$ Å, $\beta = 92{\cdot}0 \pm 0{\cdot}2°$. The space group is $P2_1$ and the unit cell contains four molecules. The molecules are arranged 'head-to-head' in layers with parallel hydrocarbon chains. Only the hydroxyl groups participate in the hydrogen bond system.

Introduction

The general features of the molecular packing in fats are known from crystal structure determinations of mono-, di-, and triglycerides (Vand & Bell, 1951; Jensen & Mabis, 1963; Larsson, 1963*a*, 1964*a*, *b*, *c*). Monoglycerides are often used as emulsifiers in technical processing of fats and their function is related to their ability to form mesomorphic phases. The liquid crystalline structures of monoglycerides are also of biological interest, *e.g.* for the understanding of fat absorption into the mucosal cell. In order to obtain information on the molecular arrangement in these complex systems an X-ray investigation of mesomorphic monoglyceride phases has been started. It is only possible, however, to determine the main features of the molecular arrangement. The detailed structure of the polar region, which has a relatively high degree of order, can thus not be derived directly. Many possibilities of inter- as well as intramolecular hydrogen bonding must be considered and it is felt that discussions on hydrogen bonding in the mesomorphic phases should be based on knowledge of the different structures occurring in the crystalline state.

An optical antipode was chosen for this study as both the racemic form and the symmetric 2-isomer have been analysed earlier (Larsson, 1964*b*, *c*). In order to simplify the structure analysis a monoglyceride containing an ω-bromine atom was used, as compounds where the terminating methyl groups are replaced by bromine atoms often crystallize isotypically with the corresponding unsubstituted compounds (Larsson, 1963*b*). This bromo monoglyceride was, however, not isotypic with the stable form of the unsubstituted compound but the chain packing and the general 'head-to-head' arrangement of the molecules are the same in the two forms.

Preparation of crystals

L-1-Mono-11-bromoundecanoin was synthesized according to the general method for preparation of optically active glycerides developed by Baer & Fischer (1945), The specific rotation $[\alpha]_D$ in dry pyridine (*c*, 6·4) was $-3{\cdot}6°$. Crystals for X-ray work were grown from petroleum spirit, b.p. 20–40°C as thin plates with (001) faces dominating and with edges parallel to the *a* and *b* axes. The crystals were biaxial positive and frequently twinned on the (001) planes. Only one crystal form was obtained from solvents and as the compound was found to isomerize very rapidly near the melting point (52·9–53·3°C) the general phase behaviour could not be studied. The crystal form obtained is a β' form according to the glyceride classification suggested by the present author (Larsson, 1964*c*).

X-ray data

Rotation and Weissenberg photographs of the (*Hkl*) and (*hKl*) zones were taken with Cu $K\alpha$ radiation using a calibrated camera. The following data were obtained:

Molecular formula $C_{14}H_{27}O_4Br$
Molecular weight 339·3
Unit cell, monoclinic,
$a = 5{\cdot}78 \pm 0{\cdot}02$, $b = 7{\cdot}43 \pm 0{\cdot}03$, $c = 37{\cdot}6 \pm 0{\cdot}1$ Å,
$\beta = 92{\cdot}0 \pm 0{\cdot}2°$
Four molecules per unit cell
Density calculated 1·396 g.cm^{-3}
Density measured 1·385 g.cm^{-3}
Absent reflexions $0k0$ when k odd
Space group $P2_1$ (taking the optical activity into account).

The reflexion intensities were estimated visually by the multiple-film technique and corrected for the polarization and Lorentz factors but not for absorption (very small crystals were used). Absolute values were later obtained by comparison with calculated structure factors.

Structure determination

The positions of the two bromine atoms in the asymmetric unit could readily be derived from the Patterson function which was sharpened to correspond to point atoms at rest (Abrahamsson & Maslen, 1962). A bromine-phased three-dimensional electron density calculation showed all the carbon atoms of the hydrocarbon chains clearly, whereas only low and broad ridges were found in the region of the cell containing the polar groups. The chains appeared to be pseudo-symmetrically related and their symmetry is $P2_1/a$ pro-

vided that there are no deviations from the common orthorhombic chain packing $O\perp$. The positions of the atoms of acyl chains were refined by a few cycles of structure factor and electron density calculations. The difficulties in obtaining positions for the rest of the atoms were due to the $P2_1/a$ symmetry of the maps and a further complication was that the positions corresponding to the true structure were so close to the superimposed false positions that only two atoms in the polar region gave resolved peaks. Their coordinates were derived by trial and error, making use of the known stereochemistry, and refined by Fourier methods. Block-diagonal least-squares refinement was then started with anisotropic vibration parameters for the bromine atoms and isotropic temperature factors for carbon and oxygen atoms. Owing to the large number of parameters in comparison with the number of observed independent reflexions, and as no pronounced anisotropic movements of carbon and oxygen atoms were indicated from the Fourier maps, only isotropic temperature factors were used for these atoms. The hydrogen atoms of the hydrocarbon chains were included in the structure factor calculation with positions according to the known data from normal paraffins (C–H bond length 1·09 Å and H–C–H bond angle 109·5°) and with the same temperature factors as the corresponding hydrogen-carrying carbon atom. The weighting scheme applied in the refinement was

$$w=\frac{1}{1+[(|F_o|-8|F_{\min}|)/5|F_{\min}|]^2}.$$

The structure refined to an R value of 0·13 for the 680 observed reflexions, and the shifts in the atomic coordinates were then about one third of the standard deviations. The calculations were performed on the Datasaab D21 computer with programs designed by Abrahamsson, Aleby, Larsson, Nilsson, Selin & Westerdahl (1965). The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962).

Discussion

The three-dimensional electron-density series is illustrated in Fig. 1, where also the numbering of the atoms is shown (the second molecule in the asymmetric unit has primed numbers). The final atomic parameters and their standard deviations are given in Tables 1, 2(*a*) and 2(*b*). Observed and calculated structure factors are listed in Table 3.

Figs. 2 and 3 show the molecular arrangement. The molecules are packed 'head-to-head' in layers of double molecular length. The hydrocarbon chains are tilted 63·3° towards the end group plane and packed according to the common orthorhombic chain packing ($O\perp$). The dimensions of the subcell (calculated from average positions of the hydrocarbon chain atoms) are

$$a=5\cdot16,\quad b=7\cdot43,\quad c=2\cdot47\text{ Å}.$$

Table 1. *Atomic coordinates with their standard deviations* $\times 10^4$ *given in brackets*

	x/a	$\sigma(x/a)$	y/b	$\sigma(y/b)$	z/c	$\sigma(z/c)$
Br(1)	1·5369	(17)	0·4508	(15)	0·0350	(3)
C(1)	1·2667	(110)	0·5350	(127)	0·0609	(16)
C(2)	1·2801	(111)	0·4539	(128)	0·0956	(16)
C(3)	1·0768	(111)	0·5502	(133)	0·1171	(16)
C(4)	1·0858	(112)	0·4512	(128)	0·1551	(17)
C(5)	0·8951	(101)	0·5416	(125)	0·1755	(15)
C(6)	0·8848	(134)	0·4469	(150)	0·2119	(20)
C(7)	0·7050	(115)	0·5391	(138)	0·2336	(17)
C(8)	0·7141	(130)	0·4576	(144)	0·2726	(19)
C(9)	0·5139	(108)	0·5481	(131)	0·2916	(16)
C(10)	0·5386	(113)	0·4344	(134)	0·3278	(17)
C(11)	0·3461	(106)	0·4961	(112)	0·3501	(16)
C(12)	0·1521	(97)	0·4254	(112)	0·4038	(14)
C(13)	0·1928	(128)	0·5624	(149)	0·4306	(19)
C(14)	−0·0130	(141)	0·5564	(145)	0·4556	(20)
O(1)	0·1709	(80)	0·5858	(86)	0·3408	(12)
O(2)	0·3498	(83)	0·4272	(92)	0·3824	(13)
O(3)	0·3898	(90)	0·4965	(93)	0·4539	(14)
O(4)	0·0293	(82)	0·7186	(80)	0·4790	(12)
Br(1′)	2·0477	(16)	1·0448	(16)	0·0334	(2)
C(1′)	1·7842	(108)	0·9291	(122)	0·0580	(16)
C(2′)	1·7807	(116)	1·0417	(134)	0·0917	(17)
C(3′)	1·5703	(132)	0·9536	(150)	0·1165	(20)
C(4′)	1·5882	(99)	1·0364	(115)	0·1522	(15)
C(5′)	1·3946	(110)	0·9561	(133)	0·1734	(16)
C(6′)	1·4075	(104)	1·0309	(120)	0·2105	(16)
C(7′)	1·2124	(120)	0·9592	(137)	0·2316	(18)
C(8′)	1·2215	(109)	1·0283	(124)	0·2710	(16)
C(9′)	1·0179	(105)	0·9586	(119)	0·2908	(16)
C(10′)	1·0313	(116)	1·0490	(137)	0·3270	(17)
C(11′)	0·8378	(128)	0·9956	(135)	0·3476	(20)
C(12′)	0·6626	(122)	0·9784	(130)	0·4031	(18)
C(13′)	0·6841	(110)	1·1136	(108)	0·4334	(16)
C(14′)	0·4853	(107)	1·0562	(119)	0·4544	(15)
O(1′)	0·6888	(92)	0·8942	(91)	0·3384	(14)
O(2′)	0·8511	(84)	1·0612	(96)	0·3818	(12)
O(3′)	0·8745	(81)	1·0268	(90)	0·4551	(12)
O(4′)	0·4575	(86)	1·1626	(84)	0·4847	(12)

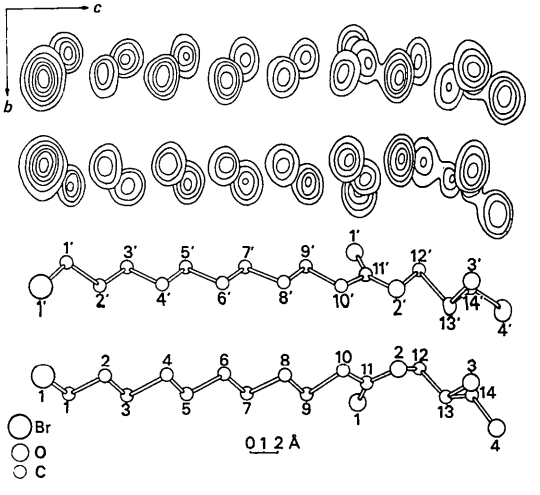

Fig. 1. Superimposed sections of the three-dimensional electron-density distribution through the atomic centres parallel to (100). The corresponding projection of the two molecules in the asymmetric unit is also shown. Contours are given at intervals of 1 e.Å^{-3} for carbon and oxygen atoms and 5 e.Å^{-3} for bromine atoms, starting with 2 e.Å^{-3}.

Table 2(*a*). *Final values of the anisotropic vibration parameters* U_{ij} (Å^2) *for the bromine atoms with their standard deviations* $\times 10^4$ *given in brackets*

	U_{11}	$\sigma(U_{11})$	U_{22}	$\sigma(U_{22})$	U_{33}	$\sigma(U_{33})$	U_{23}	$\sigma(U_{23})$	U_{13}	$\sigma(U_{13})$	U_{12}	$\sigma(U_{12})$
Br(1)	0·0991	(65)	0·0676	(71)	0·0809	(55)	−0·0273	(88)	0·0226	(49)	−0·0625	(97)
Br(1′)	0·1022	(65)	0·0837	(79)	0·0671	(48)	−0·0338	(93)	0·0258	(46)	−0·0685	(103)

Table 2(*b*). *Final values of the isotropic temperature factors, with their standard deviations* $\times 10$ *given in brackets*

C(1)	4·1 (1) Å^2
C(2)	4·1 (2)
C(3)	4·3 (1)
C(4)	4·1 (1)
C(5)	3·5 (1)
C(6)	6·7 (2)
C(7)	4·7 (2)
C(8)	6·3 (2)
C(9)	3·9 (1)
C(10)	4·4 (2)
C(11)	3·9 (2)
C(12)	2·3 (1)
C(13)	6·0 (2)
C(14)	7·3 (2)
O(1)	5·6 (1)
O(2)	6·2 (1)
O(3)	8·2 (2)
O(4)	5·6 (1)
C(1′)	3·3 (1)
C(2′)	4·7 (2)
C(3′)	6·6 (2)
C(4′)	3·0 (1)
C(5′)	4·2 (2)
C(6′)	3·7 (1)
C(7′)	5·4 (2)
C(8′)	4·3 (2)
C(9′)	3·7 (1)
C(10′)	4·9 (2)
C(11′)	6·4 (2)
C(12′)	5·5 (2)
C(13′)	3·5 (2)
C(14′)	3·6 (1)
O(1′)	7·1 (1)
O(2′)	6·6 (1)
O(3′)	6·7 (1)
O(4′)	5·6 (1)

There are no significant irregularities in the chain packing. All the carbon atoms of the acyl chains lie within 0·1 Å from their best least-squares planes. Bond distances and angles for the two molecules in the asymmetric unit are given in Table 4. The average value of the carbon–carbon distance is 1·535 Å and the average bond angle in the hydrocarbon chains is 107·1°, and the standard deviations for these values are 0·06 Å and 3·3° respectively in close agreement with the corresponding standard deviations estimated from the least-squares refinement. The planes through the carboxyl groups are twisted 5° in one molecule and 19° in the other from the corresponding planes through the zigzag chains.

All the short oxygen–oxygen contacts which can correspond to hydrogen bonds are listed in Table 5. The intramolecular distances between the two free hydroxyl groups of the molecules are rather short but are not interpreted as hydrogen bonds as their directions are far from the possible directions of the attached hydrogen atom and the two free electron-pairs around each oxygen atom (assuming sp^3 hybridization). There are two lone-pair electrons available for each hydrogen atom, and it would then be possible for both electron

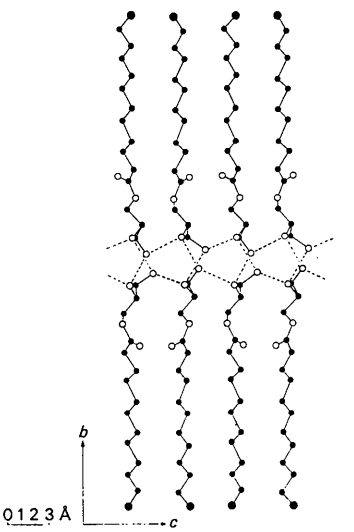

Fig. 2. Projection along the *a* axis showing the molecular arrangement. The small filled and open circles are carbon and oxygen atoms respectively and the large filled circles are bromine atoms.

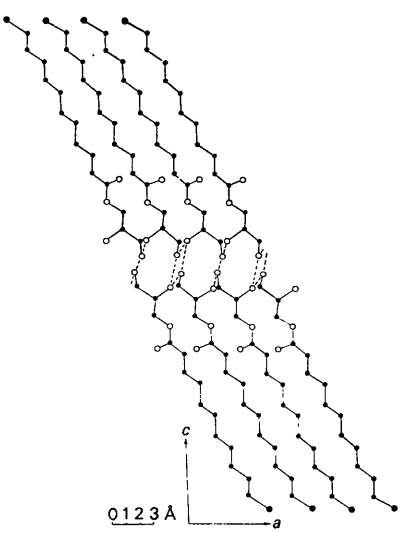

Fig. 3. Projection along the *b* axis showing the molecular arrangement. The small filled and open circles are carbon and oxygen atoms respectively and the large filled circles are bromine atoms.

Table 3. *Observed and calculated structure factors* ($\times 100$) *with phase angles* (FI) *given in fractions of* 360°

H	K	L	FOBS	FCALC	FI
0	0	6	2327	3090	1.0000
0	0	7	1503	1147	0.5000
0	0	8	4856	5291	0.5000
0	0	10	7638	9706	0.5000
0	0	11	4449	4987	0.5000
0	0	12	8567	9490	0.5000
0	0	13	6386	7790	0.5000
0	0	14	9148	10357	0.5000
0	0	15	5244	7100	0.5000
0	0	16	13225	13421	0.5000
0	0	17	4921	5731	0.5000
0	0	18	3326	1854	1.0000
0	0	19	4724	5383	0.5000
0	0	20	2427	2502	0.5000
0	0	22	1870	1491	0.5000
0	0	24	2521	2358	1.0000
0	0	25	1676	1851	1.0000
0	0	26	2988	2949	0.0000
0	0	27	3587	3592	0.0000
0	0	28	2223	2348	0.0000
0	0	29	3713	4006	0.0000
0	0	30	2318	2531	0.0000
0	0	31	3162	3049	0.0000
0	0	32	1949	2154	0.0000
0	0	33	3233	3255	0.0000
0	0	34	1977	2514	0.0000
0	0	35	1397	1716	0.0000
0	0	36	1389	1595	0.5000
0	0	41	1232	1413	0.5000
0	0	43	1576	1570	0.5000
0	0	46	963	905	0.5000
1	0	-28	1006	645	0.5000
1	0	-27	984	225	0.5000
1	0	-25	1322	829	0.5000
1	0	-24	908	875	0.5000
1	0	-23	1248	468	0.5000
1	0	-22	1483	1537	0.5000
1	0	-20	1136	1052	0.5000
1	0	-18	751	1255	0.5000
1	0	-17	725	841	1.0000
1	0	-16	1212	1594	0.5000
1	0	-15	1653	1285	1.0000
1	0	-13	625	971	1.0000
1	0	-11	1635	1464	1.0000
1	0	-9	1738	979	1.0000
1	0	-7	846	313	0.9999
1	0	-5	899	614	1.0000
1	0	-3	592	903	1.0000
1	0	20	1631	1440	0.5000
2	0	-35	1946	1532	1.0000
2	0	-33	4758	3406	1.0000
2	0	-31	5211	3933	1.0000
2	0	-28	8619	7012	1.0000
2	0	-27	5142	4491	1.0000
2	0	-23	3496	2717	0.5000
2	0	-22	2869	2528	0.5000
2	0	-21	3957	4134	0.5000
2	0	-20	6181	5539	0.5000
2	0	-19	5403	4988	0.5000
2	0	-18	7204	7147	0.5000
2	0	-17	6678	6308	0.5000
2	0	-16	7790	7589	0.5000
2	0	-15	3374	3738	0.5000
2	0	-14	11281	10479	0.5000
2	0	-12	9790	8841	0.5000
2	0	-11	5017	5732	0.5000
2	0	-10	8453	7856	1.0000
2	0	-9	2770	1869	1.0000
2	0	-8	5719	6259	1.0000
2	0	-7	5211	5554	1.0000
2	0	-6	9369	9771	1.0000
2	0	-5	2566	2852	1.0000
2	0	-4	13464	13581	1.0000
2	0	-3	3098	3507	1.0000
2	0	-2	10486	10215	1.0000
2	0	-1	5958	6355	1.0000
2	0	0	7390	7108	1.0000
2	0	1	2602	3471	1.0000
2	0	2	7025	6283	1.0000
2	0	4	1113	1209	1.0000
2	0	5	1599	1795	0.5000
2	0	6	7274	7227	1.0000
2	0	8	9162	8052	0.5000
2	0	9	5038	5074	0.5000
2	0	10	9996	9515	0.5000
2	0	11	3583	4220	0.5000
2	0	12	8558	8263	0.5000
2	0	13	5409	6207	0.5000
2	0	14	3878	4181	0.5000
2	0	15	5884	6735	0.5000
2	0	16	2511	2514	0.5000
2	0	17	2579	2056	0.5000
2	0	18	1555	2219	0.5000
2	0	23	1115	1367	1.0000
2	0	24	6059	6501	0.0000
2	0	25	4431	5041	0.0000
2	0	27	4387	3922	0.0000
2	0	29	3327	2635	0.0000
2	0	31	1977	1772	0.0000
3	0	-24	1476	1587	0.5000
3	0	-23	1029	808	0.5000
3	0	-15	1268	766	1.0000
3	0	-13	2121	1385	1.0000
3	0	-11	1676	1675	1.0000
3	0	-9	1151	1160	1.0000
3	0	-8	804	262	1.0000
3	0	-7	1946	1564	1.0000
4	0	-37	1422	1281	1.0000
4	0	-36	1067	1322	1.0000
4	0	-35	1586	1518	1.0000
4	0	-34	1657	1863	1.0000
4	0	-33	1216	1257	1.0000
4	0	-32	1775	1558	1.0000
4	0	-24	1977	1978	0.5000
4	0	-23	2796	3053	0.5000
4	0	-20	3330	2760	0.5000
4	0	-19	3009	3082	0.5000
4	0	-18	4438	3543	0.5000
4	0	-17	1911	2233	0.5000
4	0	-16	3227	2133	0.5000
4	0	-15	1878	2344	0.5000
4	0	-11	1281	1514	1.0000
4	0	-10	1270	1196	1.0000
4	0	-9	3035	2644	1.0000
4	0	-8	3013	2446	1.0000
4	0	-7	2994	2984	1.0000
4	0	-6	3541	2260	1.0000
4	0	-4	8226	7655	1.0000
4	0	-3	4165	5051	1.0000
4	0	-2	4914	4534	1.0000
4	0	-1	2440	2596	1.0000
4	0	0	1220	1617	1.0000
4	0	1	1728	1530	1.0000
4	0	4	1747	912	0.5000
4	0	5	2484	2586	0.5000
4	0	6	3010	2504	0.5000
4	0	7	1780	2269	0.5000
4	0	8	3054	3118	0.5000
4	0	9	3079	2971	0.5000
4	0	10	1824	1699	0.5000
4	0	11	3134	3014	0.5000
4	0	13	1326	1043	0.5000
0	1	6	3298	3704	0.5133
0	1	7	1950	1407	0.4451
0	1	8	4190	4374	0.4974
0	1	9	2520	2174	0.4621
0	1	10	1956	1985	0.4642
0	1	11	4129	3259	0.4575
0	1	12	1373	1386	0.5352
0	1	13	2036	1257	0.4342
0	1	14	2681	2255	0.4704
0	1	15	642	710	0.2858
0	1	16	3541	2584	0.4232
0	1	17	984	994	0.0269
0	1	18	6938	4877	0.9750
0	1	19	3002	1902	0.0177
0	1	20	1555	1511	0.9722
0	1	21	2546	1981	0.9782
0	1	22	2039	1819	0.9584
0	1	23	860	759	0.9693
0	1	24	1983	2028	0.9497
0	1	26	939	873	0.8790
0	1	34	1066	715	0.5023
1	1	-38	1015	1520	0.7331
1	1	-37	2541	2372	0.7585
1	1	-35	2602	2450	0.7523
1	1	-34	1066	1476	0.7003
1	1	-32	4615	4244	0.7367
1	1	-30	2737	2163	0.2776
1	1	-29	1762	2024	0.3046
1	1	-28	3865	3040	0.2496
1	1	-27	3087	3341	0.2761
1	1	-26	4571	3425	0.2440
1	1	-25	6634	5196	0.2537
1	1	-24	4782	4317	0.2527
1	1	-23	6453	5035	0.2461
1	1	-22	6087	5110	0.2495
1	1	-21	5900	5270	0.2551
1	1	-20	4666	5441	0.2229
1	1	-19	5530	4784	0.2724
1	1	-18	1496	2078	0.1445
1	1	-17	2286	2076	0.3577
1	1	-16	2206	1859	0.9433
1	1	-15	2128	2146	0.6546
1	1	-14	5539	4999	0.8239
1	1	-13	3120	3472	0.7103
1	1	-12	7201	7440	0.7640
1	1	-11	9767	8624	0.7417
1	1	-10	6286	5863	0.7738
1	1	-9	10981	10295	0.7344
1	1	-8	8588	8326	0.7792
1	1	-7	7754	7771	0.7152
1	1	-6	8600	8182	0.7858
1	1	-5	8583	8162	0.7142
1	1	-4	3839	4239	0.8236
1	1	-3	5170	4996	0.7003
1	1	-2	4575	4340	0.7926
1	1	-1	4229	4305	0.2526
1	1	0	5784	5590	0.7630
1	1	1	6509	7320	0.2349
1	1	2	9658	8501	0.7467
1	1	3	2161	2127	0.1581
1	1	4	27148	28255	0.2511
1	1	5	12353	11966	0.2230
1	1	6	11784	11595	0.2784
1	1	7	12296	10727	0.2090
1	1	8	10730	9947	0.3007
1	1	9	7802	6269	0.1875
1	1	10	7752	7552	0.3101
1	1	11	4548	3697	0.2021
1	1	12	4006	3075	0.3696
1	1	13	1270	1051	0.7149
1	1	14	1978	1870	0.3767
1	1	15	4886	4614	0.7283
1	1	16	2243	2400	0.6576
1	1	17	4407	4052	0.7507
1	1	18	5429	5096	0.6997
1	1	19	5273	4808	0.7733
1	1	20	11015	10323	0.7260
1	1	21	7111	6480	0.7725
1	1	23	3776	3915	0.7928
1	1	24	2894	2610	0.7694
1	1	26	1928	1937	0.8159
1	1	30	1041	736	0.1161
1	1	31	1053	1315	0.2234
1	1	32	1062	1453	0.2106
1	1	33	1507	1026	0.2529
1	1	34	1064	1662	0.2256
1	1	35	1058	1018	0.2602
1	1	37	1454	1304	0.2921
2	1	-28	3102	2203	0.3617
2	1	-27	1441	1156	0.3268
2	1	-25	1388	1366	0.4798
2	1	-24	960	1276	0.4983
2	1	-22	1835	1762	0.5038
2	1	-20	1511	1204	0.5864
2	1	-19	1472	1088	0.6611
2	1	-17	2420	1606	0.7146
2	1	-15	2417	1938	0.8391
2	1	-13	2405	1788	0.9572
2	1	-12	707	919	0.6534
2	1	-10	6091	4934	0.9968
2	1	-8	4739	4283	0.0121
2	1	-6	3215	3102	0.0317
2	1	-7	1592	1682	0.0996
2	1	-6	3500	3843	0.0226
2	1	-5	609	1103	0.2707
2	1	-4	1802	1783	0.0789
2	1	-1	1860	2316	0.4178
2	1	1	3067	3637	0.4534
2	1	2	1455	1315	0.4598
2	1	3	1898	1772	0.4642
2	1	4	3101	2942	0.5150
2	1	5	2693	2290	0.4729
2	1	6	3268	3546	0.5171
2	1	7	5631	5384	0.4962
2	1	8	927	1068	0.9050
2	1	10	2063	1842	0.5279
2	1	11	997	1395	0.0152
2	1	14	763	1078	0.9594
2	1	15	783	1263	0.9632
2	1	17	1168	1353	0.9737
2	1	18	1199	1361	0.9447
2	1	19	870	577	0.9424
2	1	20	893	910	0.0000
2	1	21	915	1235	0.0543
2	1	24	979	1530	0.9181
3	1	-32	1045	807	0.2864
3	1	-30	1503	1550	0.2682
3	1	-29	2384	2604	0.2544
3	1	-27	3970	3934	0.2580
3	1	-25	1044	1097	0.3412
3	1	-24	4260	4115	0.2303
3	1	-23	5587	5163	0.2626
3	1	-22	2463	2621	0.2395
3	1	-21	1980	2237	0.2424
3	1	-20	1686	1648	0.2696
3	1	-17	924	1375	0.7478
3	1	-16	1284	1180	0.7064
3	1	-15	4720	4705	0.7337
3	1	-14	1518	2073	0.7628
3	1	-13	6619	6374	0.7200
3	1	-12	3884	3919	0.7822
3	1	-11	4255	4466	0.7180
3	1	-10	5696	6243	0.7658
3	1	-9	1986	1721	0.7146
3	1	-8	9709	9844	0.7666
3	1	-7	7757	7202	0.7444
3	1	-6	1568	2106	0.8471
3	1	-5	777	248	0.2559
3	1	-3	2661	3086	0.2085
3	1	-2	2296	2916	0.2024
3	1	-1	4187	3906	0.2154
3	1	0	3244	4064	0.2302
3	1	1	6273	5757	0.2256
3	1	2	4071	4636	0.2561
3	1	3	4643	4554	0.2274
3	1	4	5401	5939	0.2691
3	1	5	3428	2918	0.2516
3	1	6	4769	4608	0.2727
3	1	7	3115	2204	0.2534
3	1	8	1996	1964	0.3575
3	1	9	1652	1577	0.7527
3	1	10	5691	5392	0.3041
3	1	11	1206	764	0.8083
3	1	12	1226	1926	0.4711
3	1	13	2160	1382	0.7864
3	1	14	3477	3570	0.6777
3	1	15	2238	1384	0.7702
3	1	16	3603	2986	0.7177
3	1	17	2319	2565	0.7440
3	1	18	3047	2740	0.7539
3	1	19	1697	2063	0.7496
3	1	20	3149	3103	0.7633
3	1	21	1011	1031	0.8111
3	1	22	1449	1743	0.7618
3	1	26	1503	2245	0.7625
3	1	27	1066	1912	0.7955
3	1	28	3369	3207	0.2351
3	1	30	1489	1733	0.2228
3	1	31	1040	1520	0.2388
3	1	33	1000	1355	0.2644
3	1	34	973	950	0.2038
4	1	-24	1065	988	0.5345
4	1	-23	1066	864	0.7155
4	1	-19	1051	1127	0.8247
4	1	-18	1043	1166	0.7530
4	1	-17	1036	891	0.9488
4	1	-16	1027	1278	0.9190
4	1	-15	1019	948	0.8975
4	1	-13	1001	1277	0.9697
4	1	-12	992	1219	0.9865
4	1	-10	976	1719	0.9956
4	1	-7	956	1248	0.1391
4	1	-6	946	1353	0.3736
4	1	-4	943	1201	0.0685
4	1	-3	1330	1624	0.3960
4	1	-1	934	1367	0.4683
4	1	1	940	1259	0.4161
4	1	3	946	1550	0.4819
4	1	14	1026	1008	0.9198
4	1	15	1034	814	0.9341

Table 3 (*cont.*)

H	K	L	FOBS	FCALC	FI
*	1	16	1043	745	0.9920
5	1	-30	810	1139	0.2342
5	1	-29	845	1322	0.2496
5	1	-28	877	1079	0.2488
5	1	-27	905	1549	0.2382
5	1	-26	931	1014	0.2834
5	1	-25	954	941	0.2403
5	1	-19	1041	1860	0.7203
5	1	-17	1054	878	0.7333
5	1	-15	1502	1645	0.7581
5	1	-14	1505	2006	0.7336
5	1	-13	2131	1906	0.7674
5	1	-12	1066	1309	0.7645
5	1	-11	1066	1671	0.7582
5	1	-10	1065	1054	0.8238
5	1	-5	1060	1344	0.2376
5	1	-3	3351	3125	0.2429
5	1	0	3178	2944	0.2453
5	1	1	1060	1408	0.2363
5	1	2	3181	2897	0.2616
5	1	3	1061	1664	0.2210
5	1	4	2124	1965	0.2988
5	1	6	1064	1412	0.3259
5	1	12	1064	1109	0.7197
5	1	14	1058	960	0.7026
5	1	16	1814	2048	0.7301
5	1	17	1040	1380	0.7828
5	1	19	1018	884	0.8009
0	2	7	2157	2024	0.1238
0	2	8	4198	3829	0.5053
0	2	9	3209	2788	0.2676
0	2	10	7885	7443	0.5130
0	2	11	3692	3237	0.3582
0	2	12	8069	7905	0.4972
0	2	13	4816	4923	0.4888
0	2	14	7470	7423	0.4723
0	2	15	6090	5987	0.5299
0	2	16	8924	8836	0.4799
0	2	17	4811	4801	0.5378
0	2	18	2997	1567	0.8702
0	2	19	4516	4707	0.5373
0	2	21	1569	1919	0.5511
0	2	24	2433	2850	0.9292
0	2	26	2869	3083	0.9867
0	2	27	1857	1905	0.9673
0	2	28	2683	2709	0.9931
0	2	29	2366	2543	0.0136
0	2	30	1961	2296	0.9853
0	2	31	1984	2198	0.0368
0	2	32	1414	1843	0.0040
0	2	33	1419	2326	0.0740
0	2	34	1417	2113	0.0309
1	2	-30	1974	1887	0.2192
1	2	-29	1947	1793	0.2691
1	2	-28	1354	1486	0.2636
1	2	-27	1329	1273	0.2861
1	2	-26	1301	1222	0.2353
1	2	-25	1271	834	0.4308
1	2	-24	1238	924	0.2254
1	2	-23	1206	726	0.5957
1	2	-22	1171	739	0.6626
1	2	-20	2701	2837	0.7343
1	2	-19	2616	2146	0.7105
1	2	-18	1461	1478	0.7480
1	2	-17	3737	4182	0.7301
1	2	-16	1929	2236	0.7600
1	2	-15	1611	1882	0.7198
1	2	-14	12331	12055	0.7488
1	2	-13	5983	5630	0.7256
1	2	-12	2878	2223	0.8681
1	2	-11	2395	2623	0.7325
1	2	-10	1877	1408	0.9012
1	2	-9	1801	2155	0.2249
1	2	-8	704	944	0.8486
1	2	-7	3309	3665	0.2073
1	2	-6	3487	3729	0.2100
1	2	-5	2563	3388	0.2117
1	2	-4	5115	4981	0.2352
1	2	-3	5084	6153	0.2132
1	2	-2	3994	4755	0.2591
1	2	-1	4786	5516	0.2288
1	2	0	5710	6849	0.2676
1	2	1	3115	4463	0.2604
1	2	2	5073	5207	0.2824
1	2	3	5906	6808	0.2471
1	2	4	4675	5098	0.6984
1	2	5	633	1309	0.3867
1	2	6	1980	1930	0.3373
1	2	7	4355	4699	0.7227
1	2	8	1760	1054	0.4925
1	2	9	1060	1318	0.6986
1	2	10	6808	5603	0.7202
1	2	11	1626	1049	0.7875
1	2	12	4554	3973	0.7223
1	2	13	4393	4029	0.7519
1	2	14	2234	2196	0.7569
1	2	15	3783	3352	0.7306
1	2	16	2593	2627	0.7955
1	2	17	2029	2227	0.7637
1	2	19	2167	2353	0.7372
1	2	20	2739	2937	0.7502
1	2	21	1153	542	0.8248
1	2	22	1187	1133	0.1272
1	2	23	1220	1584	0.2021
1	2	24	1773	1785	0.2007
1	2	25	1285	486	0.1637
1	2	26	1858	2446	0.2166
1	2	27	1341	1273	0.2088
1	2	29	1385	1824	0.2478
1	2	30	1402	1154	0.2213
2	2	-33	1490	2230	0.0303
2	2	-31	2458	2462	0.0163
2	2	-30	1416	1321	0.9314
2	2	-28	5403	5226	0.4774
2	2	-27	2756	2758	0.9945
2	2	-26	1358	1303	0.9573
2	2	-25	1335	1233	0.6784
2	2	-23	2568	2404	0.5746
2	2	-22	1256	1522	0.4541
2	2	-21	2745	3376	0.5076
2	2	-20	2935	3175	0.4954
2	2	-19	4050	4252	0.4775
2	2	-18	4415	4602	0.5140
2	2	-17	4581	4793	0.4620
2	2	-16	4193	5066	0.5305
2	2	-15	3164	3447	0.4643
2	2	-14	6167	6190	0.5093
2	2	-12	6405	6463	0.4970
2	2	-11	2858	2861	0.5737
2	2	-10	3480	3132	0.0250
2	2	-9	2727	2544	0.9288
2	2	-8	2669	3430	0.9886
2	2	-7	4360	4518	0.9391
2	2	-6	5066	5241	0.0104
2	2	-5	3769	4875	0.9426
2	2	-4	6225	7067	0.0157
2	2	-3	4198	4685	0.9563
2	2	-2	5781	6552	0.0096
2	2	-1	5215	5740	0.9773
2	2	0	3906	4038	0.0759
2	2	1	4476	5625	0.9966
2	2	2	1645	2177	0.1212
2	2	3	3897	3168	0.0291
2	2	4	2062	2124	0.3165
2	2	5	855	910	0.1348
2	2	6	1946	2523	0.1852
2	2	7	1538	1784	0.0387
2	2	8	8414	7587	0.4553
2	2	9	3595	2454	0.4544
2	2	10	8178	6865	0.4836
2	2	11	3375	3808	0.4521
2	2	12	5654	5385	0.5319
2	2	13	5328	4918	0.4630
2	2	14	3490	3594	0.5705
2	2	15	4708	4693	0.4973
2	2	16	1567	2100	0.5988
2	2	17	2543	2775	0.5725
2	2	19	1195	2211	0.6647
2	2	24	5333	5825	0.9645
2	2	25	2713	2999	0.0035
2	2	26	1376	1941	0.9256
2	2	27	2414	2519	0.0289
2	2	29	1415	1695	0.0733
3	2	-24	1394	2001	0.6584
3	2	-23	1380	2061	0.6310
3	2	-22	1364	1584	0.6506
3	2	-21	2331	2723	0.7028
3	2	-20	1876	2616	0.7385
3	2	-19	1306	1588	0.6717
3	2	-18	3150	3352	0.7606
3	2	-17	1264	1706	0.7280
3	2	-16	2488	2219	0.8136
3	2	-15	1223	1653	0.7721
3	2	-14	2084	2280	0.8530
3	2	-12	1165	1908	0.9698
3	2	-10	2264	2804	0.1130
3	2	-9	2233	2051	0.1904
3	2	-8	1103	1890	0.8980
3	2	-7	1890	2082	0.1767
3	2	-6	2161	1713	0.1553
3	2	-5	4152	4203	0.2413
3	2	-4	4995	3980	0.2500
3	2	-3	2596	2067	0.2567
3	2	-2	3952	3819	0.2902
3	2	-1	3654	3386	0.2665
3	2	0	1055	1886	0.4200
3	2	1	3172	3151	0.2882
3	2	2	1061	2141	0.4076
3	2	6	1897	2365	0.6295
3	2	7	1108	1107	0.6348
3	2	8	1122	829	0.6353
3	2	9	1609	1605	0.7059
3	2	10	4162	3878	0.7319
3	2	11	5499	4893	0.7439
3	2	12	1684	1881	0.7523
3	2	13	1712	2472	0.7964
3	2	14	1230	1543	0.7871
3	2	15	1251	1481	0.8214
4	2	-23	2003	2376	0.4911
4	2	-21	1418	766	0.6361
4	2	-20	2002	2221	0.4701
4	2	-19	1996	2388	0.5381
4	2	-18	2810	2358	0.4837
4	2	-17	1397	1908	0.5356
4	2	-16	1388	1488	0.5237
4	2	-15	1379	1543	0.5209
4	2	-10	1329	1312	0.9392
4	2	-9	1320	2207	0.9304
4	2	-8	1312	1569	0.9854
4	2	-7	2610	2887	0.9533
4	2	-6	1299	1277	0.9720
4	2	-4	5156	4930	0.0186
4	2	-3	4454	4427	0.9877
4	2	-2	2568	2644	0.0612
4	2	-1	2566	2700	0.9928
4	2	1	1285	1628	0.0637
4	2	4	1835	1601	0.4007
4	2	6	2621	2517	0.4723
4	2	7	1319	1947	0.3942
4	2	8	2299	2467	0.4907
4	2	9	2316	2176	0.5010
4	2	10	1904	1813	0.4738
4	2	11	1918	1996	0.5512
4	2	12	1367	1652	0.5129
4	2	13	1377	1288	0.5864
0	2	0	30167	32304	0.0084
0	2	1	8567	7874	0.9241
0	2	2	5152	5088	0.1050
0	2	3	9229	8558	0.9356
0	2	4	3133	2941	0.1756
0	2	5	4380	5258	0.9532
0	4	0	5128	7262	0.9882
0	4	1	887	2884	0.9656
0	4	2	1406	1096	0.9586
0	4	7	1458	1751	0.2143
0	4	8	1476	1061	0.6775
0	4	9	2006	2994	0.3002
0	4	10	1516	2029	0.5905
0	4	11	1946	2291	0.2606
0	4	12	1977	2880	0.5098
0	4	13	1230	1172	0.3976
0	4	14	1615	2184	0.4563
0	4	15	1643	1964	0.5286
0	4	16	3172	3360	0.4652
0	5	3	2869	3083	0.4994
0	5	4	3718	3292	0.4882
0	5	5	3342	2756	0.5153
0	5	6	4695	4738	0.4764
0	5	7	3784	3600	0.5213
0	5	8	4177	4001	0.4722
0	5	9	3845	3957	0.5161
0	5	10	3471	3410	0.5007
0	5	11	2477	2804	0.4970
0	5	12	1769	2473	0.5606
0	5	16	2606	2397	0.5282
0	5	17	3222	3402	0.9129
0	5	18	4520	4228	0.9618
0	5	19	4230	3866	0.9509
0	5	20	2694	2144	0.9575
0	5	21	3831	3236	0.9706
0	5	22	1923	2313	0.9708
0	5	23	1927	2329	0.9999
1	4	-17	4391	4110	0.7503
1	4	-16	2494	2330	0.7441
1	4	-15	3471	3655	0.7332
1	4	-14	9352	8986	0.7401
1	4	-13	8234	6348	0.7258
1	4	-6	4344	3569	0.1890
1	4	-5	4030	3900	0.2289
1	4	-4	5241	5122	0.1928
1	4	-3	5213	5095	0.2380
1	4	-2	5609	6073	0.2179
1	4	-1	5794	4712	0.2626
1	4	0	5982	6666	0.2411
1	4	1	5989	5153	0.3008
1	4	2	6006	5518	0.2564
1	4	3	5644	5169	0.2979
1	4	10	5367	5181	0.7009
1	4	11	4021	2482	0.7244
1	4	12	4713	4518	0.7112
1	4	13	4787	4120	0.7582
1	4	14	3846	3540	0.7548
1	4	15	3028	3959	0.7643
0	3	5	3599	5903	0.5092
0	3	6	3326	5974	0.4842
0	3	7	3281	4694	0.5233
0	3	8	4433	5975	0.4801
0	3	9	4127	6019	0.5057
0	3	10	1815	2757	0.5148
0	3	11	4613	6426	0.4838
0	3	18	6203	6773	0.9866
0	3	19	3770	3842	0.9440
0	3	20	2755	3131	0.0039
0	3	21	4021	3968	0.9602
0	3	22	2929	3289	0.9957
1	3	-13	2235	3379	0.7992
1	3	-12	3043	2771	0.6648
1	3	-11	4139	5154	0.8076
1	3	-10	3443	3158	0.6622
1	3	-9	4433	5785	0.7813
1	3	-8	3168	2690	0.7142
1	3	-7	4075	5221	0.7445
1	3	-6	2913	2617	0.7612
1	3	-5	3229	4271	0.7311
1	3	1	2518	3471	0.2300
1	3	2	3572	3127	0.7820
1	3	3	3053	4023	0.2117
1	3	4	9515	8445	0.2301
1	3	5	7054	6658	0.2378
1	3	6	4128	3456	0.2661
1	3	7	5929	6518	0.2213
1	3	8	3229	2518	0.3444
1	3	9	4517	4896	0.2296
2	3	-15	2712	2080	0.9391
2	3	-13	3651	3924	0.9841
2	3	-10	6882	6663	0.9908
2	3	-9	7433	8096	0.9956
2	3	-8	4243	4266	0.0434
2	3	-7	4945	4799	0.0105
2	3	-6	4434	4234	0.0570
2	3	-1	2985	2610	0.4146
2	3	1	5838	5049	0.4471
2	3	2	5392	4014	0.4332
2	3	3	5002	3457	0.4631
2	3	4	6894	5716	0.4766
2	3	5	6103	4589	0.5100
2	3	6	8308	6874	0.4979
2	3	7	9854	8434	0.5123
2	3	9	4154	3194	0.6422
3	3	-8	5698	4680	0.7567
3	3	-7	4850	3119	0.7828
3	3	-4	2756	1065	0.9701
3	3	-1	4697	3459	0.1776
3	3	1	3844	2967	0.1815
3	3	2	3858	2973	0.2412
3	3	3	3878	2533	0.2046
3	3	4	3904	2463	0.2843
3	3	5	3937	2789	0.2712

Table 4. *Bond distances and angles*

Bond	Distance	Bond	Angle
Br(1) –C(1)	1·97 Å	Br(1)—C(1) —C(2)	107·3°
C(1) —C(2)	1·44	C(1) —C(2) —C(3)	104·5
C(2) —C(3)	1·62	C(2) —C(3) —C(4)	103·9
C(3) —C(4)	1·60	C(3) —C(4) —C(5)	104·2
C(4) —C(5)	1·52	C(4) —C(5) —C(6)	107·3
C(5) —C(6)	1·54	C(5) —C(6) —C(7)	108·8
C(6) —C(7)	1·51	C(6) —C(7) —C(8)	109·0
C(7) —C(8)	1·59	C(7) —C(8) —C(9)	105·2
C(8) —C(9)	1·54	C(8) —C(9) —C(10)	96·8
C(9) —C(10)	1·60	C(9) —C(10) –C(11)	105·6
C(10) –C(11)	1·49	C(10) –C(11) –O(1)	128·3
C(11) –O(1)	1·25	C(10) –C(11) –O(2)	114·2
C(11) –O(2)	1·32	O(1) —C(11) –O(2)	116·9
O(2) —C(12)	1·42	C(11) –O(2) —C(12)	122·6
C(12) –C(13)	1·45	O(2) —C(12) –C(13)	105·9
C(13) –C(14)	1·54	C(12) –C(13) –C(14)	107·0
C(13) –O(3)	1·49	C(12) –C(13) –O(3)	106·2
C(14) –O(4)	1·51	O(3) —C(13) –C(14)	102·8
Br(1′)–C(1′)	2·00	C(13) –C(14) –O(4)	102·7
C(1′)—C(2′)	1·52	Br(1′) –C(1′)—C(2′)	110·4
C(2′)—C(3′)	1·69	C(1′)—C(2′)—C(3′)	106·1
C(3′)—C(4′)	1·48	C(2′)—C(3′)—C(4′)	108·1
C(4′)—C(5′)	1·52	C(3′)—C(4′)—C(5′)	106·5
C(5′)—C(6′)	1·50	C(4′)—C(5′)—C(6′)	109·2
C(6′)—C(7′)	1·50	C(5′)—C(6′)—C(7′)	110·2
C(7′)—C(8′)	1·57	C(6′)—C(7′)—C(8′)	112·5
C(8′)—C(9′)	1·51	C(7′)—C(8′)—C(9′)	110·6
C(9′)—C(10′)	1·52	C(8′)—C(9′)—C(10′)	105·9
C(10′)–C(11′)	1·44	C(9′)—C(10′)–C(11′)	110·2
C(11′)–O(1′)	1·19	C(10′)–C(11′)–O(1′)	126·0
C(11′)–O(2′)	1·38	C(10′)–C(11′)–O(2′)	112·8
O(2′) –C(12′)	1·50	O(1′)—C(11′)–O(2′)	120·9
C(12′)–C(13′)	1·52	C(11′)–O(2′) –C(12′)	109·5
C(13′)–C(14′)	1·48	O(2′)—C(12′)–C(13′)	95·0
C(13′)–O(3′)	1·49	O(12′)–C(13′)–C(14′)	99·5
C(14′)–O(4′)	1·40	O(12′)–C(13′)–O(3′)	99·4
		O(3′)—C(13′)–C(14′)	99·0
		C(13′)–C(14′)–O(4′)	112·6

pairs to participate in hydrogen bonds either by disorder of the hydrogen atoms or by a bifurcation of the hydrogen bond. Only one free electron pair of each oxygen atom, however, is hydrogen bonded in this structure, so that there are two hydrogen bonds per oxygen atom as in normal alcohols (Abrahamsson, Larsson & von Sydow, 1960) and 2-monoglycerides (Larsson, 1964*b*). The molecules are linked with neighbouring molecules both over the gap between the molecular layers and laterally within the layers so that an infinite two-dimensional net-work is formed (*cf.* Figs. 2 and 3).

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Table 5. *Short oxygen–oxygen contacts* (< 3·0 Å) *between hydroxyl groups, and the angles they form with corresponding carbon–oxygen bonds*

The values for intermolecular contacts are given in brackets. The suffix 2_1 is used for atoms with $z > 0{\cdot}5$ (possessing 2_1-relation to those given in the asymmetric unit).

O(3) –O(4′)	2·76 Å	O(3) —O(4′) —C(14′)	100·6°
		C(13) –O(3) —O(4′)	129·3
O(3) –O(4′)2_1	2·74	O(3) —O(4′)2_1–C(14′)2_1	112·9
		C(13) –O(3) —O(4′)2_1	124·2
O(3′)–O(4)	2·61	O(3′) –O(4) —C(14)	117·0
		C(13′)–O(3′) —O(4)	143·7
O(3′)–O(4)2_1	2·90	O(3′) –O(4)2_1 –C(14)2_1	97·4
		C(13′)–O(3′) —O(4)2_1	121·3
O(3) –O(4)	(2·85)	C(13) –O(3) —O(4)	(56·8)
		C(14) –O(4) —O(3)	(56·5)
O(3′)–O(4′)	(2·87)	C(13′)–O(3′) —O(4′)	(56·4)
		C(14′)–O(4′) —O(3′)	(50·5)

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