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The Crystal Structure of 1,5,9,13-Tetraoxacyclohexadecane

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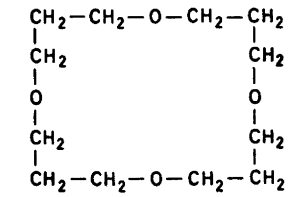
The crystals are monoclinic with space group $P2_1/c$ and four molecules in the unit cell. The cell dimensions are $a = 8.62_3$ Å, $b = 9.87_6$ Å, $c = 17.42_2$ Å and $\beta = 111.0_0°$. The phase problem was solved by a computer procedure based on direct methods. The weighted R-value arrived at for 1046 observed reflections is $R_w = 4.7$ %, the reflections being measured on a four-circle diffractometer. Averaged bond distances and angles are: C–C: 1.50_7 Å, C–O: 1.41_5 Å, C–C–C: $112._9°$, C–C–O: $108._3°$, C–O–C: $113._3°$. The molecule has the "square" ring conformation and the symmetry is very close to D_{2d}. C–C and C–O dihedral angles are somewhat larger than 60° and somewhat smaller than 180°, respectively. The effect may probably be explained by *intra*-molecular repulsions between hydrogen and oxygen atoms.

16-Membered rings have been extensively studied by Dale and Borgen.[1] A marked tendency for the saturated ring skeleton to follow the diamond lattice is observed. Ring substitution by hetero atoms (oxygen) may reduce the *gauche* interactions at corner positions of the "square" ring conformation. The IR-spectra in solid state and solution, respectively, of 1,5,9,13-tetraoxacyclohexadecane as well as of 1,5,9,13-tetraoxa-3,7,11,15-octamethylcyclohexadecane show that these compounds are conformationally homogeneous. Since the methyl groups in the latter compound must occupy corner positions, the observed similarities between the two suggest that also the former compound has the four oxygen atoms, each situated midway between corner atoms.

In order to obtain more detailed information, the crystal structure determination of 1,5,9,13-tetraoxacyclohexadecane has been carried out.



The crystals are monoclinic with space group $P2_1/c$. The cell dimensions, determined by a manual four circle diffractometer, with estimated standard deviations [2*] are: $a = 8.623(2)$ Å, $b = 9.876(4)$ Å, $c = 17.422(4)$ Å, $\beta = 111.00(4)°$. The unit cell contains four molecules $\varrho_{calc} = 1.11$ g · cm^{-3}, $\varrho_{obs} = 1.09$ g · cm^{-3}.

With $2\theta_{max} = 50°$ and Mo$K\alpha$-radiation, about 2100 reflections were measured by an automatic four-circle diffractometer with a highly orientated graphite crystal monochromator. 1046 reflections were recorded as observed using an observed-unobserved cutoff at 2.0 $\sigma(I)$. No corrections for absorption or secondary extinction effects have been carried out.

The phase problem was solved three-dimensionally by a computer procedure based on direct methods. 237 signs were determined (all of which turned out to correct), and the corresponding Fourier map contained 16 peaks with heights ranging from 3 e Å^{-3} to 5 e Å^{-3} which clearly showed the molecule.

Table 1. Fractional atomic coordinates and anisotropic thermal vibration parameters with estimated standard deviations (multiplied by 10^5).

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
O_1	04315	29440	06028	2367	1399	415	−285	909	193
	34	26	15	66	45	15	89	55	40
O_2	−21420	33624	19035	2663	1224	436	−144	734	108
	32	26	15	66	43	14	85	52	39
O_3	14319	37890	36327	2254	1439	581	136	718	−180
	34	28	15	66	47	16	92	54	42
O_4	38129	40749	23397	2743	1467	384	420	716	85
	30	28	14	66	46	14	86	54	40
C_1	−08244	20710	06478	3005	1385	472	−886	732	− 98
	64	50	27	127	74	25	147	91	76
C_2	−24123	28720	04316	2549	1910	432	−1221	438	143
	63	56	28	113	86	24	173	84	73
C_3	−23172	39699	10449	2151	1330	539	−102	568	294
	59	47	27	102	70	26	162	80	66
C_4	−18883	43138	24495	2125	1261	574	− 30	713	−146
	59	44	29	104	67	36	159	82	68
C_5	−14867	35430	32441	2773	1300	517	−285	1147	−222
	61	48	28	114	70	25	155	86	70
C_6	01449	28161	34844	3291	1347	409	0	752	− 33
	65	49	28	134	71	23	154	88	75
C_7	29940	32056	37449	2775	1862	451	1149	811	536
	64	54	29	124	86	24	156	88	81
C_8	41871	43225	37466	2360	2264	444	702	508	−183
	59	59	28	128	95	24	165	86	82
C_9	37216	50378	29290	2381	1540	485	−339	535	−116
	61	49	28	114	78	25	165	87	63
C_{10}	32781	46050	15290	2232	1293	516	−72	791	165
	61	46	27	109	68	24	149	79	63
C_{11}	32830	34617	09529	2461	1780	480	686	985	208
	59	53	27	117	83	24	158	83	72
C_{12}	20341	23714	09105	2640	1470	506	346	1034	36
	62	50	28	117	76	24	143	88	71

* All programs used are included in this reference.

Table 2. Fractional atomic coordinates for hydrogen atoms with estimated standard deviations (H_{mn} are bonded to C_m).

Atom	x	y	z	B (Å²)
$H_{1,1}$	−.0485	.1639	.1206	5.6
	37	32	21	1.0
$H_{1,2}$	−.0982	.1259	.0266	6.2
	39	36	19	1.0
$H_{2,1}$	−.3329	.2263	.0416	6.0
	41	34	20	1.0
$H_{2,2}$	−.2612	.3286	−.0147	6.5
	39	34	22	1.0
$H_{3,1}$	−.3391	.4586	.0830	4.4
	39	32	18	0.9
$H_{3,2}$	−.1344	.4613	.1126	5.5
	39	33	18	1.0
$H_{4,1}$	−.2957	.4852	.2339	5.8
	43	34	19	1.0
$H_{4,2}$	−.1019	.4980	.2449	7.6
	45	38	21	1.2
$H_{5,1}$	−.2401	.2893	.3200	7.1
	44	37	21	1.1
$H_{5,2}$	−.1470	.4242	.3669	6.3
	40	36	20	1.0
$H_{6,1}$	.0103	.2178	.2999	4.5
	35	30	18	0.9
$H_{6,2}$	.0378	.2241	.3985	6.3
	39	33	22	1.0
$H_{7,1}$	.2827	.2487	.3256	6.3
	39	34	22	1.0
$H_{7,2}$	.3406	.2779	.4265	7.2
	46	37	22	1.1
$H_{8,1}$	.5286	.3990	.3894	8.1
	45	39	23	1.2
$H_{8,2}$	.4188	.5040	.4146	8.0
	47	37	23	1.2
$H_{9,1}$	.4476	.5797	.2931	8.7
	44	41	21	1.3
$H_{9,2}$	.2514	.5429	.2731	5.7
	44	31	18	1.0
$H_{10,1}$	.4062	.5385	.1524	5.8
	40	34	18	1.0
$H_{10,2}$	.2160	.5047	.1387	4.9
	40	31	18	0.9
$H_{11,1}$	.4358	.3050	.1119	7.7
	48	38	23	1.2
$H_{11,2}$	.3011	.3825	.0392	6.3
	39	34	22	1.0
$H_{12,1}$	.2203	.1989	.1504	5.1
	37	31	19	0.9
$H_{12,2}$	.2172	.1564	.0549	8.0
	43	38	23	1.1

Methylene hydrogen positions were calculated assuming tetrahedral C−H bonds of length 1.03 Å. The parameters were refined by full-matrix least squares technique. The weights in least squares were calculated form the standard deviations in intensities, $\sigma(I)$, taken as

$$\sigma(I) = [C_T + (0.02C_N)^2]^{\frac{1}{2}}$$

Table 3. Observed and calculated structure factors (on 10 times absolute scale).

h	k	l	F_o	F_c
0	0	2	420	- 431
0	0	4	865	- 884
0	0	6	283	- 283
0	0	8	49	47
0	0	10	226	- 225
0	0	16	47	39
0	1	2	388	- 385
0	1	3	191	- 190
0	1	4	860	- 884
0	1	5	40	19
0	1	6	684	- 680
0	1	7	198	201
0	1	8	281	286
0	1	10	185	189
0	1	11	67	- 57
0	1	13	90	- 94
0	1	15	140	140
0	1	16	80	83
0	2	0	521	- 505
0	2	1	1409	1505
0	2	2	839	- 847
0	2	3	335	332
0	2	4	317	321
0	2	5	290	- 291
0	2	7	197	- 194
0	2	8	409	410
0	2	9	172	171
0	2	10	59	64
0	2	11	39	- 39
0	2	15	80	- 84
0	3	2	170	170
0	3	3	270	265
0	3	4	446	451
0	3	5	189	189
0	3	6	37	- 18
0	3	7	425	- 423
0	3	8	144	140
0	3	9	42	39
0	3	10	76	- 91
0	3	11	118	115
0	3	13	75	- 84
0	3	14	141	- 137
0	4	0	75	- 75
0	4	1	68	66
0	4	2	196	198
0	4	3	186	- 183
0	4	4	114	117
0	4	5	131	130
0	4	6	58	- 47
0	4	7	344	- 345
0	4	8	191	- 190
0	4	9	143	141
0	4	10	70	- 69
0	4	14	77	92
0	4	16	45	- 40
0	5	1	66	- 65
0	5	2	82	- 79
0	5	3	78	- 79
0	5	4	42	- 50
0	5	5	40	4
0	5	6	39	- 11
0	5	7	100	- 104
0	5	9	163	167
0	5	10	62	- 58
0	5	12	62	72
0	5	13	64	61
0	6	1	39	- 35
0	6	4	76	- 79
0	6	5	47	35
0	6	6	185	184
0	6	7	86	90
0	6	8	173	- 177
0	6	9	108	106
0	6	10	80	83
0	6	11	61	- 63
0	7	2	92	- 93
0	7	4	87	77
0	7	5	89	83
0	7	7	63	- 62
0	7	8	98	- 98
0	7	10	75	76
0	7	11	69	- 64
0	7	12	45	30
0	8	5	89	- 93
0	8	7	48	57
0	8	9	99	- 95
0	8	10	69	69
0	8	13	54	39
0	9	1	42	33
0	9	3	96	- 89
0	10	0	48	31
0	10	3	49	33
0	0	-18	43	- 25
0	0	-16	60	42
0	0	-14	244	246
0	0	-10	389	- 394
0	0	-8	418	- 412
0	0	-6	118	113
0	0	-4	110	- 113
0	0	-2	79	- 81
0	0	0	404	406
0	0	2	792	788
0	0	4	250	- 246
0	0	6	103	97
0	0	8	186	- 178
0	0	10	35	- 34
0	0	12	108	104
0	0	14	129	- 131
1	1	-18	73	69
1	1	-15	153	- 154
1	1	-14	239	- 242
1	1	-13	259	266
1	1	-12	37	12
1	1	-11	57	- 66
1	1	-10	129	- 132
1	1	-9	237	- 238
1	1	-8	423	- 421
1	1	-7	213	214
1	1	-6	561	548
1	1	-5	221	220
1	1	-4	1103	1126
1	1	-3	251	- 248
1	1	-2	55	- 59
1	1	-1	205	- 199
1	1	0	85	- 85
1	1	1	276	273
1	1	2	93	- 93
1	1	3	70	63
1	1	4	112	- 111
1	1	5	125	- 127
1	1	6	72	60
1	1	8	231	- 228
1	1	9	75	78
1	1	10	78	74
2	1	-16	46	- 40
2	1	-15	61	- 49
2	1	-14	125	- 119
2	1	-13	133	142
2	1	-12	76	- 64
2	1	-11	82	89
2	1	-10	122	131
2	1	-8	557	549
2	1	-7	514	- 514
2	1	-6	57	59
2	1	-5	376	379
2	1	-4	83	81
2	1	-3	290	290
2	1	-2	37	31
2	1	-1	303	- 297
2	1	0	932	- 934
2	1	1	276	- 276
2	1	2	32	- 23
2	1	3	220	216
2	1	4	223	218
2	1	8	125	- 126
2	1	9	57	- 38
2	1	10	89	90
2	1	11	41	- 39
2	1	12	80	65
2	1	13	88	90
3	1	-18	46	- 33
3	1	-17	69	55
3	1	-15	50	30
3	1	-14	67	- 61
3	1	-13	91	- 92
3	1	-12	202	198
3	1	-10	218	216
3	1	-9	74	72
3	1	-8	41	39
3	1	-7	463	- 459
3	1	-6	428	- 422
3	1	-5	185	186
3	1	-4	223	- 229
3	1	-3	480	477
3	1	-2	191	- 187
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3	1	0	36	43
3	1	1	69	69
3	1	2	83	83
3	1	4	59	61
3	1	5	161	- 160
3	1	6	46	58
3	1	7	311	306
3	1	8	74	- 78
3	1	9	190	- 189
3	1	10	102	100
3	1	14	48	- 27
4	1	-17	44	- 3
4	1	-13	41	- 31
4	1	-12	64	70
4	1	-11	133	- 122
4	1	-10	81	- 76
4	1	-9	35	25
4	1	-8	67	53
4	1	-7	167	169
4	1	-6	529	- 533
4	1	-5	167	163
4	1	-4	65	58
4	1	-3	146	- 142
4	1	-2	301	312
4	1	-1	233	233
4	1	0	65	62
4	1	1	100	- 103
4	1	2	89	- 87
4	1	3	135	- 134
4	1	4	71	68
4	1	5	221	218
4	1	6	88	94
4	1	8	36	- 37
4	1	9	103	- 104
5	1	-12	67	- 62
5	1	-10	45	- 36
5	1	-8	138	139
5	1	-7	192	192
5	1	-6	247	- 242
5	1	-5	84	- 90
5	1	-4	161	161
5	1	-3	86	- 94
5	1	-2	173	170
5	1	-1	63	- 62
5	1	0	79	- 69
5	1	1	68	- 70
5	1	3	90	92
5	1	5	94	- 89
5	1	6	152	- 152
5	1	7	51	46
5	1	8	152	153
5	1	9	91	- 89
5	1	11	80	66
5	1	12	82	- 77
6	1	-8	42	52
6	1	-6	73	- 88
6	1	-5	154	- 160
6	1	-4	157	157
6	1	-3	46	- 35
6	1	-2	86	- 74
6	1	3	86	- 88
6	1	4	69	- 69
6	1	5	65	62
6	1	6	70	75
6	1	7	56	52
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7	1	-7	102	104
7	1	-5	93	- 98
7	1	-4	55	61
7	1	0	76	79
7	1	1	55	49
7	1	2	148	- 140
7	1	3	66	64
7	1	4	74	77
7	1	7	67	- 70
7	1	11	51	42
8	1	-13	47	- 15
8	1	-8	42	54
8	1	-7	52	56
8	1	-6	50	59
8	1	-5	105	- 110
8	1	-4	53	- 54
8	1	-3	53	53
8	1	1	56	57
8	1	3	82	- 80
8	1	4	65	- 72
8	1	6	45	33
8	1	8	77	59
8	1	9	48	- 18
9	1	-8	46	- 32
9	1	-2	97	- 93
9	1	1	73	66
9	1	3	124	- 112
9	1	4	64	57
9	1	6	60	24
9	1	7	77	76
0	0	-16	65	69
0	0	-14	47	48
0	0	-12	66	80
0	0	-10	255	- 253
0	0	-8	129	- 132
0	0	-6	85	89
0	0	-4	178	175
0	0	-2	30	23
0	0	0	779	- 804
0	0	2	322	323
0	0	4	58	68
0	0	6	113	- 115
0	0	12	107	- 112
0	0	14	190	195
1	1	-18	109	97
1	1	-16	63	67
1	1	-15	70	- 73
1	1	-14	206	- 210
1	1	-13	66	66
1	1	-12	79	- 75
1	1	-11	53	66
1	1	-10	69	74
1	1	-9	45	45
1	1	-7	109	- 109
1	1	-4	183	180
1	1	-3	531	- 526
1	1	-2	604	- 603
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1	1	0	478	480
1	1	1	477	- 481
1	1	2	78	84
1	1	3	79	77
1	1	4	69	- 78
1	1	5	100	- 95
1	1	6	71	- 67
1	1	7	124	128
1	1	12	110	112
1	1	13	49	- 50
2	1	-17	47	28
2	1	-14	167	- 177
2	1	-11	137	137
2	1	-10	177	178
2	1	-9	226	- 226
2	1	-8	113	- 108
2	1	-6	143	142
2	1	-5	132	- 131
2	1	-4	88	- 87
2	1	-3	317	320
2	1	-2	93	90
2	1	-1	371	374
2	1	1	479	- 481
2	1	2	99	101
2	1	3	131	125
2	1	5	63	62
2	1	8	49	52
2	1	9	77	- 76
2	1	10	85	95
2	1	12	68	- 71
2	1	13	117	- 114
2	1	15	81	85
3	1	-18	47	- 32
3	1	-15	108	112
3	1	-13	139	- 138
3	1	-12	79	85
3	1	-11	162	- 160
3	1	-10	43	- 40
3	1	-9	164	168
3	1	-8	215	216
3	1	-7	44	53
3	1	-6	169	- 169
3	1	-5	93	- 94
3	1	-4	34	43
3	1	-3	37	18
3	1	-2	333	- 336
3	1	-1	51	51
3	1	0	423	426
3	1	1	74	- 74
3	1	2	111	- 108
3	1	4	176	175
3	1	5	153	- 155
3	1	6	169	- 169
3	1	7	42	26
3	1	11	54	- 45
3	1	13	121	113
3	1	14	81	- 69
4	1	-17	54	- 58
4	1	-16	47	50
4	1	-15	48	63
4	1	-13	86	- 91
4	1	-10	115	122
4	1	-9	102	107
4	1	-8	204	- 205
4	1	-6	50	67
4	1	-4	105	- 105
4	1	-3	33	20
4	1	-2	193	194
4	1	-1	124	- 126
4	1	0	215	209
4	1	1	80	- 76
4	1	2	238	- 235
4	1	4	95	- 96
4	1	5	101	- 107
4	1	7	122	123
4	1	8	89	94
4	1	10	67	- 63
4	1	11	134	137
4	1	13	63	- 55
5	1	-11	75	78
5	1	-10	235	- 235
5	1	-9	126	- 127
5	1	-8	140	143
5	1	-7	157	155
5	1	-6	106	108
5	1	-5	111	- 107
5	1	-2	160	- 158
5	1	-1	75	- 73
5	1	1	35	- 39
5	1	2	49	47

Table 3. Continued.

h	k	l	Fo	Fc
5	5	4	52	50
5	5	6	88	- 96
5	5	12	58	- 63
6	6	-16	46	- 35
6	6	-15	44	36
6	6	-12	44	- 54
6	6	-11	51	- 67
6	6	-10	67	68
6	6	-9	46	62
6	6	-7	39	- 23
6	6	-6	49	- 60
6	6	-5	72	- 72
6	6	-4	53	- 47
6	6	-3	82	- 78
6	6	-2	70	76
6	6	-1	58	58
6	6	0	61	- 66
6	6	1	175	168
6	6	2	66	56
6	6	3	92	91
6	6	4	124	124
6	6	5	126	- 125
6	6	6	179	- 178
6	6	8	50	47
6	6	10	45	- 50
6	6	11	49	- 42
6	6	12	48	48
7	7	-11	44	50
7	7	-9	138	- 136
7	7	-8	69	- 56
7	7	-7	62	57
7	7	-6	47	- 26
7	7	-5	75	62
7	7	-2	55	39
7	7	0	90	88
7	7	3	41	- 39
7	7	4	230	- 238
7	7	6	105	103
7	7	7	144	- 142
7	7	8	62	55
7	7	9	95	95
7	7	11	50	37
8	8	-11	58	- 54
8	8	-10	58	- 66
8	8	-7	74	60
8	8	-6	118	119
8	8	-2	61	- 74
8	8	1	100	- 99
8	8	2	50	- 32
8	8	4	62	67
8	8	5	77	77
8	8	9	53	47
9	9	-10	59	33
9	9	-6	59	43
9	9	-4	54	- 53
9	9	-1	61	- 55
9	9	0	42	- 27
9	9	1	45	41
9	9	2	59	53
9	9	3	73	66
9	9	5	71	- 61
9	9	6	61	55
0	0	-18	52	72
0	0	-16	228	- 223
0	0	-14	157	161
0	0	-12	48	- 40
0	0	-10	100	- 102
0	0	-8	32	- 23
0	0	-6	112	111
0	0	-4	29	- 24
0	0	-2	119	119
0	0	0	153	154
0	0	2	86	87
0	0	4	54	66
0	0	6	249	- 256
0	0	12	53	- 44
1	1	-15	72	75
1	1	-14	61	55
1	1	-13	70	- 78
1	1	-11	58	49
1	1	-10	118	113
1	1	-9	41	35
1	1	-6	33	48
1	1	-3	90	90
1	1	-2	83	80
1	1	-1	708	- 704
1	1	0	352	- 346
1	1	1	262	260
1	1	2	56	64
1	1	3	127	124
1	1	4	154	- 154
1	1	5	122	125
1	1	6	72	- 67
1	1	7	131	- 128
1	1	9	58	59
1	1	10	57	46
1	1	11	41	42
1	1	12	73	60
1	1	13	58	- 58
2	2	-18	51	- 3
2	2	-17	112	116
2	2	-16	43	- 41
2	2	-15	134	- 135
2	2	-14	102	105
2	2	-13	52	36
2	2	-11	59	60
2	2	-10	83	78
2	2	-8	239	- 238
2	2	-7	119	- 120
2	2	-5	164	- 160
2	2	-4	113	110
2	2	-1	192	- 192
2	2	-2	273	- 274
2	2	-1	401	405
2	2	0	36	- 45
2	2	1	160	162
2	2	3	67	69
2	2	4	33	10
2	2	5	201	204
2	2	6	115	117
2	2	7	216	- 218
2	2	10	82	81
2	2	14	65	- 60
3	3	-14	69	86
3	3	-12	106	- 104
3	3	-11	94	- 98
3	3	-9	58	26
3	3	-8	176	- 182
3	3	-7	58	- 56
3	3	-6	48	48
3	3	-5	81	72
3	3	-4	53	- 57
3	3	-3	263	255
3	3	-2	559	558
3	3	-1	272	264
3	3	0	271	- 275
3	3	1	43	- 41
3	3	2	128	- 126
3	3	3	119	- 119
3	3	4	197	202
3	3	5	100	- 87
3	3	6	209	213
3	3	7	193	- 193
3	3	8	164	- 169
3	3	10	55	- 50
3	3	11	73	57
4	4	-15	70	- 68
4	4	-12	80	- 73
4	4	-10	43	- 42
4	4	-9	222	216
4	4	-8	94	94
4	4	-7	160	- 163
4	4	-6	251	256
4	4	-5	171	167
4	4	-3	223	220
4	4	-2	279	- 285
4	4	-1	263	- 261
4	4	0	247	252
4	4	1	311	- 305
4	4	3	107	- 109
4	4	4	171	- 171
4	4	5	37	- 26
4	4	6	188	184
4	4	7	103	102
4	4	8	127	- 133
4	4	9	62	67
4	4	10	66	- 68
4	4	13	58	50
4	4	14	47	44
5	5	-13	63	62
5	5	-10	78	81
5	5	-9	171	175
5	5	-4	123	- 124
5	5	-3	166	- 168
5	5	-2	47	- 93
5	5	3	92	- 62
5	5	5	156	154
5	5	6	39	13
5	5	7	212	211
5	5	8	95	- 85
6	6	-10	49	- 30
6	6	-9	118	- 123
6	6	-8	123	128
6	6	-7	149	- 143
6	6	-6	186	- 191
6	6	-5	100	115
6	6	-4	54	- 73
6	6	-3	50	- 63
6	6	-2	110	113
6	6	0	74	70
6	6	1	215	212
6	6	2	39	6
6	6	3	183	183
6	6	5	144	- 138
6	6	6	46	- 54
6	6	8	44	- 8
6	6	10	69	61
6	6	11	73	- 70
7	7	-12	42	- 18
7	7	-11	53	- 60
7	7	-10	74	- 70
7	7	-8	65	67
7	7	-7	110	- 105
7	7	-6	49	- 36
7	7	-5	139	130
7	7	-3	128	123
7	7	-1	52	51
7	7	1	65	64
7	7	3	48	- 33
7	7	4	78	- 83
7	7	5	78	- 62
7	7	8	65	54
8	8	-7	117	119
8	8	-5	67	68
8	8	-4	104	107
8	8	-3	79	- 85
8	8	-2	93	- 91
8	8	0	56	- 52
8	8	1	104	- 97
8	8	4	53	59
8	8	5	46	32
8	8	8	46	1
9	9	-6	63	- 68
9	9	-3	63	- 57
9	9	2	62	- 43
9	9	3	49	45
9	9	4	61	53
4	0	-14	106	- 100
4	0	-8	106	- 107
4	0	-6	70	72
4	0	-4	90	- 91
4	0	-2	437	436
4	0	0	73	- 74
4	0	2	94	93
4	0	8	102	- 111
4	1	-16	55	- 67
4	1	-15	50	- 51
4	1	-12	64	52
4	1	-11	45	39
4	1	-9	86	92
4	1	-8	198	198
4	1	-7	99	- 104
4	1	-6	46	- 51
4	1	-5	46	- 51
4	1	-4	111	109
4	1	-3	102	97
4	1	-2	275	273
4	1	-1	356	- 362
4	1	0	263	- 262
4	1	2	89	- 86
4	1	3	129	123
4	1	6	62	- 51
4	1	10	42	33
4	1	11	42	- 46
4	1	12	56	- 41
4	1	13	80	76
4	1	14	46	51
4	2	-20	49	- 22
4	2	-16	53	53
4	2	-15	48	36
4	2	-12	38	21
4	2	-10	128	119
4	2	-9	83	85
4	2	-8	34	28
4	2	-7	201	- 200
4	2	-6	265	- 260
4	2	-5	200	- 194
4	2	-3	212	- 215
4	2	-2	284	283
4	2	-1	411	416
4	2	0	381	- 387
4	2	1	97	98
4	2	2	79	- 75
4	2	6	84	83
4	2	9	80	- 74
4	3	-17	73	70
4	3	-16	88	87
4	3	-15	61	- 70
4	3	-12	82	- 78
4	3	-10	85	- 74
4	3	-9	294	- 293
4	3	-8	96	86
4	3	-7	82	78
4	3	-6	130	- 131
4	3	-5	66	70
4	3	-4	138	- 138
4	3	-3	221	219
4	3	-2	217	218
4	3	-1	371	368
4	3	1	57	- 51
4	3	2	70	- 66
4	3	3	185	- 183
4	3	5	45	- 30
4	3	6	179	179
4	3	12	61	- 62
4	4	-13	68	- 65
4	4	-12	43	- 31
4	4	-11	165	- 168
4	4	-10	115	- 113
4	4	-9	200	196
4	4	-8	184	180
4	4	-7	294	299
4	4	-6	101	98
4	4	-5	41	48
4	4	-4	74	- 78
4	4	-3	103	- 101
4	4	-2	144	- 145
4	4	-1	92	87
4	4	0	145	141
4	4	1	208	- 204
4	4	2	74	81
4	4	3	108	- 111
4	4	5	40	- 40
4	4	7	96	91
4	4	10	75	- 68
4	5	-14	68	- 55
4	5	-11	56	95
4	5	-10	187	195
4	5	-9	42	36
4	5	-8	131	- 132
4	5	-7	148	150
4	5	-6	51	- 31
4	5	-5	133	- 139
4	5	-4	53	46
4	5	-2	45	- 94
4	5	-1	105	- 103
4	5	1	85	93
4	5	2	111	107
4	5	3	47	42
4	5	4	70	- 160
4	5	5	101	- 106
4	5	10	64	44
4	6	-16	48	41
4	6	-14	69	51
4	6	-13	86	78
4	6	-12	51	41
4	6	-10	151	- 153
4	6	-9	145	- 150
4	6	-7	119	- 119
4	6	-4	56	44
4	6	1	149	152
4	6	2	45	- 30
4	6	3	59	47
4	6	4	52	39
4	6	6	44	- 31
4	7	-13	66	- 71
4	7	-12	51	- 49
4	7	-11	82	- 79
4	7	-7	68	- 52
4	7	-6	41	63
4	7	-4	44	31
4	7	-3	168	165
4	7	0	78	- 89
4	7	2	62	61
4	7	3	74	75
4	7	4	63	68
4	7	5	104	- 108
4	7	6	73	- 74
4	8	-13	44	8
4	8	-7	73	69
4	8	-6	52	- 46
4	8	0	55	- 59
4	8	1	48	- 32
4	8	3	112	- 109
4	8	5	52	40
4	9	-7	43	12
4	9	-6	48	- 33
4	9	-3	54	- 53
5	0	-16	88	90
5	0	-14	164	- 165
5	0	-12	38	- 36
5	0	-10	54	50
5	0	-6	39	- 41
5	0	-4	187	185
5	0	-2	326	- 332
5	0	0	248	250
5	0	2	76	- 74
5	0	6	43	- 44
5	0	12	45	34
5	1	-17	102	- 93
5	1	-16	76	- 80
5	1	-15	80	78
5	1	-12	40	27
5	1	-11	59	56
5	1	-10	66	56
5	1	-5	105	- 102
5	1	-4	81	- 86
5	1	-2	87	85
5	1	-1	117	122
5	1	0	85	87
5	1	1	103	- 108
5	1	2	66	- 75
5	1	7	47	37
5	1	8	47	52
5	2	-19	46	- 19
5	2	-16	76	70
5	2	-15	165	168
5	2	-11	42	- 48
5	2	-9	41	- 44
5	2	-8	80	83
5	2	-6	61	- 65
5	2	-5	175	- 172
5	2	-3	207	207
5	2	-2	55	- 57
5	2	-1	169	- 169
5	2	1	89	85
5	2	4	71	81
5	2	6	91	- 96
5	2	11	46	- 43
5	3	-18	47	32
5	3	-17	61	50
5	3	-16	58	- 50
5	3	-15	49	52
5	3	-14	61	70
5	3	-13	87	- 83
5	3	-12	70	- 59
5	3	-11	90	- 89
5	3	-10	37	- 7
5	3	-9	85	- 78
5	3	-7	91	100
5	3	-6	45	40
5	3	-5	94	98
5	3	-4	163	- 163
5	3	-3	158	- 162
5	3	-2	66	- 53

Table 3. Continued.

h	k	l	F_o	F_c
3	3	-1	79	81
3	3	0	146	150
3	3	1	94	91
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3	3	4	43	- 34
3	3	6	54	- 49
3	3	7	44	- 54
4	4	-17	54	- 38
4	4	-16	92	- 90
4	4	-13	74	- 52
4	4	-12	54	44
4	4	-9	82	- 79
4	4	-8	122	- 124
4	4	-7	249	255
4	4	-6	117	111
4	4	-4	76	- 64
4	4	-3	43	28
4	4	-2	106	95
4	4	-1	96	- 96
4	4	4	45	- 31
4	4	5	69	77
4	4	6	54	- 50
4	4	7	88	- 88
4	4	8	57	60
5	5	-17	57	- 42
5	5	-15	62	- 57
5	5	-14	56	43
5	5	-13	83	90
5	5	-10	42	- 17
5	5	-8	98	- 145
5	5	-7	62	54
5	5	-6	147	149
5	5	-3	130	- 142
5	5	-2	91	- 93
5	5	-1	64	- 60
5	5	2	59	49
6	6	-11	186	182
6	6	-10	83	98
6	6	-8	156	- 158
6	6	-7	143	- 155
6	6	-6	115	114
6	6	-5	44	- 46
6	6	-2	42	- 0
6	6	0	98	- 88
6	6	6	63	61
6	6	7	57	- 55
7	7	-13	47	- 27
7	7	-12	45	32
7	7	-7	107	- 101
7	7	-6	50	48
7	7	-3	83	82
7	7	-1	43	45
7	7	1	58	- 48
7	7	8	52	- 36
8	8	-13	45	- 23
8	8	-12	53	- 31
8	8	-11	50	- 49
8	8	-10	71	61
8	8	-9	85	96
8	8	-4	59	- 49
8	8	-2	50	54
8	8	1	53	- 56
0	0	-16	46	- 39
0	0	-12	60	- 60
0	0	-10	69	67
0	0	-4	78	70
0	0	-2	68	- 61
0	0	2	86	- 97
0	0	4	131	- 122
0	0	6	76	72
1	1	-19	49	- 21
1	1	-17	44	34
1	1	-10	50	45
1	1	-6	71	64
1	1	-5	58	- 60
1	1	-4	59	- 63
1	1	-3	76	73
1	1	-2	145	- 146
1	1	3	55	- 53
1	1	5	65	63
1	1	6	70	64
1	1	8	63	54
2	2	-11	92	- 84
2	2	-10	60	- 55
2	2	-9	49	36
2	2	-2	39	- 32
2	2	-1	53	- 54
2	2	1	60	67
2	2	2	54	59
2	2	4	76	71
2	2	8	65	- 54
3	3	-12	46	- 34
3	3	-11	57	- 55
3	3	-9	54	56
3	3	-8	46	33
3	3	-7	79	- 71
3	3	-6	42	- 21
3	3	-5	59	59
3	3	-4	47	- 59
3	3	-3	76	83
3	3	-2	85	85
3	3	-1	121	- 125
3	3	0	78	82
3	3	1	48	21
3	3	3	58	52
3	3	4	70	- 67
3	3	5	71	- 57
3	3	7	51	- 48
4	4	-16	65	- 45
4	4	-15	50	- 57
4	4	-12	86	77
4	4	-11	82	78
4	4	-10	91	- 86
4	4	-7	48	- 39
4	4	-5	53	51
4	4	-4	69	67
4	4	3	72	- 73
4	4	4	41	- 33
4	4	5	58	55
4	4	-8	54	56
4	4	4	78	78
4	4	-14	49	- 46
6	6	-11	72	65
6	6	-10	69	72
6	6	-8	57	34
6	6	-6	81	- 78
6	6	4	47	- 64
6	6	-6	46	- 40
7	7	0	43	32
7	7	2	53	- 59
8	8	-9	47	32
8	8	0	45	17
0	0	-18	59	- 52
0	0	-12	43	- 43
0	0	-10	118	- 113
0	0	-8	103	113
0	0	-6	139	139
0	0	-2	103	104
0	0	0	78	- 54
0	0	2	81	- 82
0	0	4	103	- 107
1	1	-17	66	54
1	1	-16	56	29
1	1	-8	53	47
1	1	-7	50	32
1	1	-6	58	56
1	1	-5	43	25
1	1	-4	86	76
1	1	-3	197	- 195
1	1	-2	200	- 208
1	1	-1	92	90
1	1	0	96	- 92
1	1	2	53	63
1	1	4	48	31
1	1	5	56	- 58
1	1	7	52	41
1	1	8	44	25
2	2	-17	47	- 37
2	2	-16	50	42
2	2	-11	48	33
2	2	-10	40	29
2	2	-9	97	- 91
2	2	-6	106	- 106
2	2	-4	109	- 117
2	2	-3	63	- 59
2	2	-2	66	64
2	2	-1	58	51
2	2	1	47	57
2	2	2	45	46
2	2	4	43	43
2	2	5	52	- 64
3	3	-10	113	- 111
3	3	-6	47	- 16
3	3	-4	119	112
3	3	-2	137	148
3	3	-2	52	- 45
3	3	0	74	69
3	3	1	77	- 66
3	3	5	57	- 44
3	3	6	50	- 53
4	4	-12	45	- 24
4	4	-11	41	- 9
4	4	5	44	- 18
5	5	-11	67	56
5	5	-9	88	- 92
5	5	-8	51	51
5	5	-4	46	- 25
5	5	-1	60	- 57
5	5	0	42	5
5	5	3	43	16
7	7	-11	45	40
7	7	-10	45	42
7	7	-7	50	51
8	8	-6	53	17
0	0	-16	78	- 66
0	0	-10	67	- 57
0	0	-6	155	158
0	0	-4	42	43
1	1	-17	53	- 41
1	1	-16	64	- 61
1	1	-10	106	99
1	1	-9	53	- 46
1	1	-6	41	- 2
1	1	0	64	- 60
2	2	-12	49	19
2	2	-11	52	45
2	2	-10	74	71
2	2	-8	65	- 72
2	2	-7	68	- 55
2	2	-6	79	- 74
2	2	-5	42	42
2	2	-2	44	- 7
3	3	-9	45	36
3	3	-8	94	- 93
3	3	-5	43	- 4
3	3	0	43	- 5
4	4	-14	47	- 30
4	4	-12	50	- 48
4	4	-10	51	46
4	4	-9	63	66
4	4	-3	44	11
4	4	1	49	- 23
5	5	-7	47	- 49
6	6	-5	45	- 22
6	6	0	45	- 4
0	0	-4	54	- 46
1	1	-3	70	63
1	1	-1	47	- 33
2	2	-1	47	- 8

where C_T is the total number of counts and C_N the net count (peak minus background). Anisotropic temperature factors were introduced for oxygen and carbon atoms. The weighted R-value arrived at was 4.7 % (conventional R-value 6.7 %) for 1046 observed reflections. The atomic form factors were those of Hanson *et al.*[3] except for hydrogen.[4] A final difference Fourier map, calculated with the phases determined by the parameters corresponding to R_w = 4.7 % contained no larger density fluctuations than ± 0.3 e Å^{-3}.

Final fractional coordinated and thermal parameters with estimated standard deviations are given in Tables 1 and 2. The expression for anisotropic vibration is:

$$\exp - (B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{13}hl + B_{23}kl)$$

A comparison between observed and calculated structure factors is presented in Table 3.

The principal axes of the thermal vibration ellipsoids were calculated from the thermal parameters of Table 1. Root mean square amplitudes, the corresponding B-values for the atomic anisotropic thermal vibration along the principal axes, as well as their components along the crystal axes are given in Table 4.

Table 4. The principal axes of the thermal vibration ellipsoids given by the components of a unit vector in fractional coordinates e_x, e_y, e_z; the corresponding r.m.s. amplitudes, and the B-values.

Atom	e_x	e_y	e_z	$(\bar{u}^2)^{1/2}$ Å	B (Å^2)
O_1	−.093	.061	.000	.285	6.43
	.077	.047	.050	.276	6.00
	.031	.066	−.036	.235	4.36
O_2	.118	−.015	.006	.300	7.10
	.035	.077	.040	.253	5.04
	.015	−.064	.046	.232	4.26
O_3	.035	.051	−.041	.295	6.88
	.118	−.005	.038	.270	5.76
	−.014	.087	.026	.256	5.19
O_4	.114	.034	.009	.308	7.51
	−.038	.095	.004	.264	5.51
	.031	−.012	.061	.225	3.98
C_1	.111	−.041	.009	.330	8.58
	.002	−.040	.053	.251	4.92
	.056	.083	.030	.244	4.72
C_2	−.074	.072	.008	.351	9.73
	.078	.071	−.005	.255	5.13
	.062	−.003	.061	.238	4.47
C_3	−.044	.050	.038	.299	7.04
	.115	.033	.032	.261	5.40
	.020	−.082	.036	.238	4.47
C_4	.013	.031	−.052	.285	6.41
	.123	−.014	.026	.264	5.51
	.013	.096	.021	.245	4.75
C_5	.117	−.024	.034	.306	7.41
	.041	.075	−.027	.260	5.34
	−.003	.064	.044	.235	4.37
C_6	.120	.001	.006	.335	8.84
	.005	−.101	.007	.258	5.26
	.033	.011	.061	.232	4.27
C_7	.083	.074	.022	.340	9.11
	−.080	.049	.020	.281	6.26
	.047	−.048	.054	.222	3.88
C_8	.045	.090	−.009	.349	9.62
	−.101	.045	.004	.274	5.93
	.057	.010	.061	.241	4.59
C_9	−.099	.037	.010	.296	6.94
	.009	.086	−.028	.277	6.08
	.074	.039	.054	.247	4.81
C_{10}	−.064	.055	.027	.274	5.91
	.106	.026	.045	.270	5.75
	.009	.081	−.032	.241	4.58
C_{11}	.076	.079	.021	.315	7.84
	.097	−.063	.021	.265	5.54
	.017	.006	−.054	.241	4.58
C_{12}	.115	.037	.026	.300	7.09
	.041	−.092	.023	.265	5.56
	−.024	.022	.051	.248	4.85

The r.m.s. discrepancy between atomic anisotropic vibration tensor components calculated from the thermal parameters of Table 1, and those calculated from the rigidbody parameters obtained by analysis of librational, trans-

lational and screw motion,[5] is as large as 0.0054 Å^2, and does not support the assumption of regarding the molecule as an oscillating rigid body. Accordingly, no librational corrections of the coordinates were carried out.

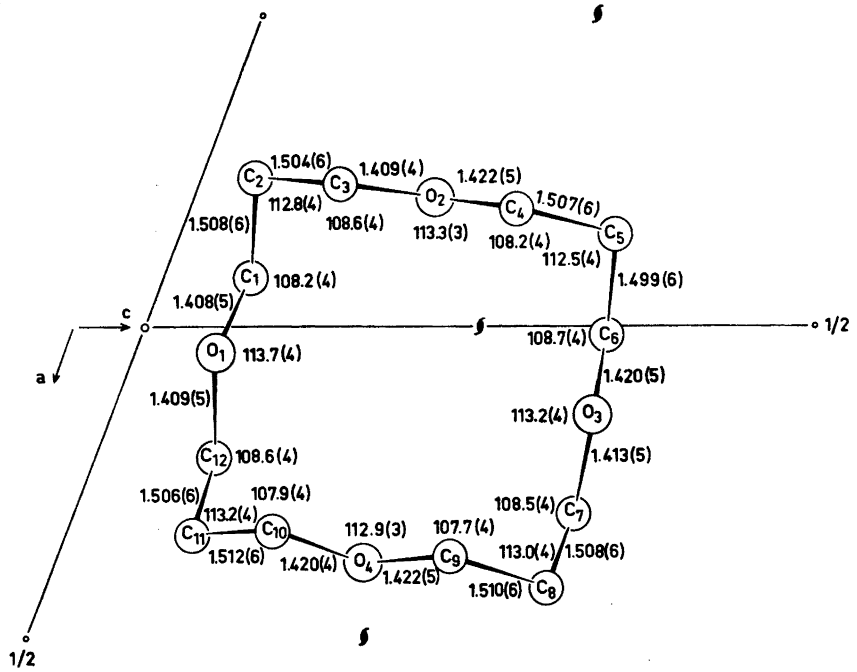

Fig. 1. Schematical drawing of the molecule viewed along [010].

Bond distances and angles may be found in Fig. 1 and Table 5. Dihedral angles are presented in Table 6.

The C–C and C–O distances as well as the C–C–C, C–C–O, and C–O–C angles, respectively, are equal within probable limits of error with mean values C–C: 1.507 Å, C–O: 1.415 Å, C–C–C: 112.9°, C–C–O: 108.3°, and C–O–C: 113.3°. The latter angle is somewhat larger than the micro wave value for dimethyl ether [6] (111.5°), while the C–O distance of 1.417 Å in dimethyl ether agrees closely with the present result. The relatively large C–O–C angles and small C–C–O angles may be due to repulsions between hydrogen atoms of methylene groups in positions adjacent to the oxygen atoms.

Table 6 shows that C–C dihedral angles are somewhat larger than 60° (mean value 64.5°) and C–O somewhat smaller than 180° (mean value 172.6°). This effect may probably be explained by *intra*-molecular hydrogen-oxygen atom repulsions (for example between $H_{1,1}$ and O_2).

The four shortest transannular H–H distances are 3.03 Å, 2.93 Å, 2.93 Å, and 2.90 Å, respectively. This is 0.6 Å to 0.7 Å longer than the van der Waals contact and means that the molecule has a cavity of this size in the middle.

Table 5. Bond distances and angles of the hydrogen atoms. Estimated standard deviations are about 0.03 Å and 2°, respectively.

Distance	(Å)
$C_1-H_{1,1}$	1.01
$C_1-H_{1,2}$	1.02
$C_2-H_{2,1}$	0.99
$C_2-H_{2,2}$	1.04
$C_3-H_{3,1}$	1.06
$C_3-H_{3,2}$	1.02
$C_4-H_{4,1}$	1.02
$C_4-H_{4,2}$	1.00
$C_5-H_{5,1}$	1.00
$C_5-H_{5,2}$	1.00
$C_6-H_{6,1}$	1.05
$C_6-H_{6,2}$	1.00
$C_7-H_{7,1}$	1.08
$C_7-H_{7,2}$	0.95
$C_8-H_{8,1}$	0.95
$C_8-H_{8,2}$	0.99
$C_9-H_{9,1}$	0.99
$C_9-H_{9,2}$	1.05
$C_{10}-H_{10,1}$	1.03
$C_{10}-H_{10,2}$	1.01
$C_{11}-H_{11,1}$	0.96
$C_{11}-H_{11,2}$	0.99
$C_{12}-H_{12,1}$	1.06
$C_{12}-H_{12,2}$	1.05

Angle	(°)
$O_1-C_1-H_{1,1}$	111
$O_1-C_1-H_{1,2}$	112
$H_{1,1}-C_1-H_{1,2}$	103
$H_{1,1}-C_1-C_2$	113
$H_{1,2}-C_1-C_2$	111
$C_1-C_2-H_{2,1}$	110
$C_1-C_2-H_{2,2}$	106
$H_{2,1}-C_2-H_{2,2}$	111
$H_{2,1}-C_2-C_3$	107
$H_{2,2}-C_2-C_3$	111
$C_2-C_3-H_{3,1}$	110
$C_2-C_3-H_{3,2}$	112
$H_{3,1}-C_3-H_{3,2}$	105
$H_{3,1}-C_3-O_2$	111
$H_{3,2}-C_3-O_2$	109
$O_2-C_4-H_{4,1}$	109
$O_2-C_4-H_{4,2}$	110
$H_{4,1}-C_4-H_{4,2}$	107
$H_{4,1}-C_4-C_5$	109
$H_{4,2}-C_4-C_5$	114
$C_4-C_5-H_{5,1}$	110
$C_4-C_5-H_{5,1}$	105
$H_{5,1}-C_5-H_{5,2}$	107
$H_{5,1}-C_5-C_6$	110
$H_{5,2}-C_5-C_6$	111
$C_5-C_6-H_{6,1}$	108
$C_5-C_6-H_{6,2}$	113
$H_{6,1}-C_6-H_{6,2}$	108
$H_{6,1}-C_6-O_3$	111
$H_{6,2}-C_6-O_3$	109
$O_3-C_7-H_{7,1}$	108
$O_3-C_7-H_{7,2}$	109
$H_{7,1}-C_7-H_{7,2}$	111
$H_{7,1}-C_7-C_8$	112
$H_{7,2}-C_7-C_8$	108
$C_7-C_8-H_{8,1}$	111
$C_7-C_8-H_{8,2}$	111
$H_{8,1}-C_8-H_{8,2}$	108
$H_{8,1}-C_8-C_9$	109
$H_{8,2}-C_8-C_9$	105
$C_8-C_9-H_{9,1}$	113
$C_8-C_9-H_{9,2}$	112
$H_{9,1}-C_9-H_{9,2}$	107
$H_{9,1}-C_9-O_4$	108
$H_{9,2}-C_9-O_4$	108
$O_4-C_{10}-H_{10,1}$	108
$O_4-C_{10}-H_{10,2}$	110
$H_{10,1}-C_{10}-H_{10,2}$	104
$H_{10,1}-C_{10}-C_{11}$	113
$H_{10,2}-C_{10}-C_{11}$	113
$C_{10}-C_{11}-H_{11,1}$	110
$C_{10}-C_{11}-H_{11,2}$	109
$H_{11,1}-C_{11}-H_{11,2}$	108
$H_{11,1}-C_{11}-C_{12}$	108
$H_{11,2}-C_{11}-C_{12}$	107
$C_{11}-C_{12}-H_{12,1}$	111
$C_{11}-C_{12}-H_{12,2}$	110
$H_{12,1}-C_{12}-H_{12,2}$	108
$H_{12,1}-C_{12}-O_1$	107
$H_{12,2}-C_{12}-O_1$	113

Table 6. Dihedral angles.

Angle	(°)	Angle	(°)
$O_1-C_1-C_2-C_3$	− 65.0 (5)	$O_3-C_7-C_8-C_9$	− 65.4 (6)
$C_1-C_2-C_3-O_2$	− 65.4 (5)	$C_7-C_8-C_9-O_4$	− 63.3 (6)
$C_2-C_3-O_2-C_4$	174.6 (4)	$C_8-C_9-O_4-C_{10}$	175.5 (4)
$C_3-O_2-C_4-C_5$	−172.3 (4)	$C_9-O_4-C_{10}-C_{11}$	−174.9 (4)
$O_2-C_4-C_5-C_6$	66.3 (5)	$O_4-C_{10}-C_{11}-C_{12}$	65.3 (5)
$C_4-C_5-C_6-O_3$	63.6 (5)	$C_{10}-C_{11}-C_{12}-O_1$	62.0 (5)
$C_5-C_6-O_3-C_7$	−171.2 (4)	$C_{11}-C_{12}-O_1-C_1$	−171.2 (4)
$C_6-O_3-C_7-C_8$	170.1 (4)	$C_{12}-O_1-C_1-C_2$	171.0 (4)

Calculations of properly chosen interatomic distances and angles, least squares planes, and moments of inertia show that the symmetry of the molecule is very close to D_{2d} ($2m/\bar{4}$). The four-fold inversion axis through the centre of gravity, with coordinates (0.088, 0.355, 0.209), is roughly parallel to the *b*-axis.

The oxygen atoms are out of the planes through the four nearest carbon atoms by 0.1 to 0.2 Å, in *outward* direction, and the planes are approximately parallel with the $\bar{4}$-axis.

From Fig. 2 it may be realized that the molecules pack with methylene groups fitting into the "holes" in the surface of 2_1-symmetry equivalent molecules. The shortest H – H and H – O *inter*molecular contacts are 2.50 Å and 2.80 Å, respectively, somewhat larger than the corresponding van der Waals contacts (2.4 Å and 2.6 Å).

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