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On the Crystal Structure of the Ozonide 3-Carbomethoxy-5-anisyl-1,2,4-trioxacyclopentane

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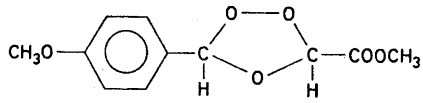
The crystals belong to the orthorhombic system and the space group is *Pbca*. The unit cell, containing eight molecules, has the following parameters:

$$a = 25.39_0 \text{ Å}, \; b = 10.96_6 \text{ Å}, \; c = 8.17_4 \text{ Å}$$

The phase problem was solved by direct methods, and full-matrix least squares refinement gave an R-value of 7.1 % for 1183 observed reflections. The structure is disordered, containing D-and L-molecules with different conformations at the same crystal site. The disorder is observable for the ozonide ring only, and the relative amounts of the two types of molecules are 65 % and 35 %. The molecules have the *trans* configuration. No definite conclusions concerning the conformational problem of the ozonide ring can be drawn.

By ozonation of *trans* *p*-methoxy cinnamic acid in methylene chloride at 78°C, two stereoisomeric ozonides are obtained by fractional crystallization.[1] These are the *cis* and *trans* isomers of 3-carbomethoxy-5-anisyl-1,2,4-trioxacyclopentane:



An electron diffraction investigation of gaseous 1,2,4-trioxacyclopentane [2] (the only ozonide structure analysis reported) indicates a large degree of puckering for a five-membered ring, with maximum torsional angle of 49.4° for C_s geometry and 49.1° for C_2 geometry, respectively. Although the model with C_2 symmetry is favoured by calculations of conformational energies, the conformational problem of the ozonide ring is not completely settled by this work.

In order to confirm the assignment of configuration (being based on PMR, melting points, solubilities, *etc.*[1]), and to obtain detailed structural information

about the five-membered ring, an X-ray crystal structure determination of the *trans* isomer has been carried out.

3-Carbomethoxy-5-anisyl-1,2,4-trioxacyclopentane belongs to the orthorhombic system, the systematically absent reflections leading to the space group *Pbca*. The cell parameters were derived from *h0l*-and *hk*0-Weissenberg photographs calibrated with BaF_2-powder diagrams. 2θ-values of 18 reflections were used in a least squares refinement[3]* of lattice parameters, and the following results were obtained:

$$a = 25.390(4)\ \text{Å},\ b = 10.966(4)\ \text{Å},\ c = 8.174(4)\ \text{Å}$$

The number of molecules in the unit cell is eight ($\varrho_{calc} = 1.40$ gcm^{-3}, $\varrho_{obs} = 1.38$ gcm^{-3}) Intensity data were obtained (at room temperature) by photometric measurements of equiinclination integrated Weissenberg diagrams corresponding to *h0l*,....., *h5l* and *hk*0,....., *hk*5. Due to instability, four crystals were used. 1183 independent reflections were strong enough to be measured. No corrections have been made for absorption or secondary extinction effects.

The phase problem was solved by direct methods.[3,4] The intensities were statistically [5] put on absolute scale and the unitary structure factors calculated. Signs for three large unitary structure factors were chosen in order to specify the origin. Although the sign determination process ran smoothly in terms of two symbols, none of the four corresponding Fourier maps (based on 350 reflections) could be interpreted.

However, the data reduction output had revealed that: (1) Wilson ratio and $N(Z)$-plot gave poor indication of centro symmetry, (2) $\langle U^2 \rangle$ greatly exceeded $1/N$, N being the number of approximately equally heavy atoms in the unit cell, and (3) the overall temperature factor, $B = 2.8$ Å^2, was suspiciously small. The unitary structure factors were therefore recalculated with a more reasonable temperature factor, $B = 4.0$ Å^2, and an absolute scale satisfying $\langle U^2 \rangle = 1/N$. 356 signs were determined uniquely and the corresponding Fourier map contained 17 peaks with heights ranging from 4.2 e.Å^{-3} to 7.6 e.Å^{-3}, and one peak of 3.4 e.Å^{-3}. All other density maxima were smaller than 1.5 e.Å^{-3}.

Since distances and angles between the 17 largest peaks roughly corresponded to the expected molecular configuration, peak number 18 was discarded, and a least squares refinement carried out. However, the R-value arrived at after six sycles of anisotropic refinement was as large as 15.1 %. Analysis of the thermal vibration parameters showed extremely large amplitudes for the oxygen atoms of the ozonide ring, and some of the distances of this ring were unreasonable (Fig. 1).

When checking the 356 signs determined against those corresponding to $R = 15.1$ %, no discrepancies were found. A Fourier synthesis based on all data, with phases corresponding to $R = 15.1$ %, contained a peak at the same position as peak number 18 mentioned above (shaded in Fig. 1). Furthermore, the electron density corresponding to O_4 (Fig. 1) was only about 2/3 of the values for the other oxygen atoms. The height of the 18th peak was about 1/3 of an oxygen peak.

* All programs used are included in this reference.

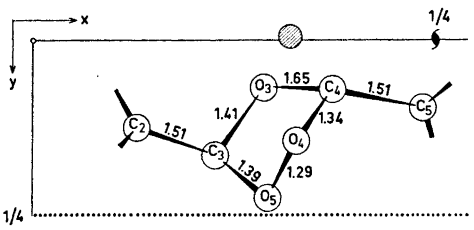


Fig. 1. Schematical drawing (viewed along [001]) showing the bond distances of the ozonide ring obtained by least squares refinement without introducing disorder.

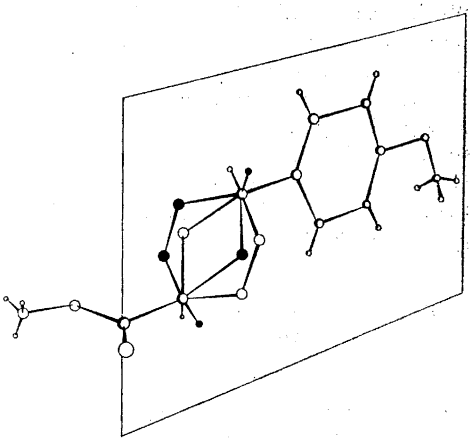


Fig. 2. Schematical drawing showing the assumed disorder. The plane (1) of Table 4 is shown.

These findings suggest a disordered structure (Fig. 2). Bond distances and angles, as well as the thermal parameter analysis and the shapes of the electron density peaks, indicate that the disorder is observable for the oxygen atoms of the ozonide ring only.

From model considerations it seems reasonable to describe the disorder in terms of D-and L-molecules with *different conformations* (Fig. 3) occupying the same crystal site.

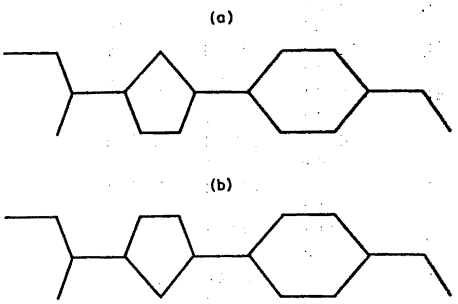


Fig. 3. Schematical drawing indicating (a) the conformation of the molecule with 65 % weight. (b) The conformation of the molecule with 35 % weight.

By introducing partial atoms for oxygens as indicated in Fig. 2, with starting weights 0.6 and 0.4 for the two types, respectively, three cycles of least squares refinement (positional and anisotropic thermal parameters as well as the weights being refined), gave a final R-factor of 7.1 %. Fig. 4 shows that some of the interatomic distances for the five-membered ring are unreasonable. However, the considerable reduction of the R-value, together with the facts that the temperature factors for the disordered atoms are

moderate (Table 2), and that their multiplicity factors have refined to a consistent set of values of about 65 % and 35 %, justify the assumption of a disordered structure.

Several attempts were made to force the disordered atoms into more acceptable positions, using models with C – O distances and O – O distances in the range 1.41 Å – 1.43 Å and 1.46 Å – 1.48 Å, respectively. In all cases the *R*-factor increased to 12 – 14 %, and when the models were subjected to least squares refinement the final parameters corresponded closely to those given in Tables 1 and 2, with *R*-values of about 7 %.

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

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
O_1	04117	03015	17327	200	1307	1630	−145	235	−656
	14	34	44	6	41	65	26	32	92
O_2	04660	17069	36834	292	148	2309	−270	591	−1724
	17	38	51	9	49	84	34	43	113
O_3	14516	07034	11442	192	1114	1225	−23	234	−303
	53	154	119	24	92	188	56	107	317
O_4	16551	14066	36859	235	1390	1340	−87	140	−604
	25	69	84	12	75	100	49	59	179
O_5	14219	23897	26868	228	1240	2090	−238	165	0
	46	102	210	23	116	156	66	108	284
O_3'	15888	19290	26815	170	1045	1577	−177	8	−395
	79	174	349	34	208	268	102	175	491
O_4'	15934	−00633	18171	149	922	2330	−374	34	−659
	41	109	167	17	106	258	82	106	299
O_5'	13806	07620	04991	259	1594	976	−14	409	−377
	118	351	195	40	205	296	138	186	564
O_6	39812	14838	05614	161	1106	1913	28	−173	322
	12	32	43	6	36	65	23	31	89
C_1	−00807	−01838	24397	172	1560	2665	−106	244	183
	22	61	88	11	74	135	44	57	171
C_2	06349	12190	24994	206	790	1393	83	61	−627
	20	44	60	9	41	83	32	45	115
C_3	11363	16570	15964	207	1014	1389	−65	95	−30
	20	49	61	10	50	86	39	46	127
C_4	18660	06769	25479	204	1321	1478	108	115	490
	22	57	71	10	60	94	45	53	147
C_5	24141	09488	19623	189	847	1042	20	2	301
	18	44	55	9	45	73	34	41	105
C_6	25417	19462	09932	164	849	1223	57	58	612
	18	43	56	8	43	74	32	40	106
C_7	30581	21624	04779	165	743	1388	59	−153	124
	18	40	60	7	41	80	30	41	109
C_8	34514	13691	09805	156	735	1132	−16	−133	−152
	17	39	55	8	42	70	30	37	103
C_9	33314	03548	19309	188	729	1746	101	−249	222
	21	41	66	9	44	96	32	51	113
C_{10}	28183	01543	24364	217	751	1261	−24	−111	323
	20	44	62	10	43	80	32	44	111
C_{11}	41408	25523	−03539	190	1244	2213	0	181	1119
	20	56	75	9	57	112	42	55	146

[a] For numbering of atoms, see Fig. 4.

Table 2. 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	$(\bar{u}^2)^{\frac{1}{2}}$ Å	B (Å^2)
	.018	−.070	.054	.307	7.44
O_1	.031	.053	.024	.247	4.80
	−.016	.024	.107	.212	3.56
	.022	−.054	.071	.384	11.64
O_2	.031	.056	−.017	.274	5.91
	−.011	.049	.098	.204	3.27
	.023	−.066	.047	.270	5.75
O_3	.028	.061	.023	.253	5.07
	−.015	0.15	.111	.187	2.75
	.018	−.076	.038	.305	7.35
O_4	.035	.043	.000	.271	5.79
	−.006	.025	.116	.201	3.18
	.028	−.060	.033	.306	7.39
O_5	.004	.043	.107	.268	5.65
	.028	.054	−.048	.236	4.39
	.020	−.073	.041	.274	5.95
O_3'	.024	.006	−.096	.232	4.24
	.024	.055	.064	.208	3.43
	.015	−.053	.088	.303	7.26
O_4'	−.023	.041	.083	.259	5.31
	.028	.062	.020	.156	1.93
	.016	−.081	.029	.316	7.88
O_5'	.034	.041	.031	.301	7.13
	−.013	.010	.115	.156	1.93
	−.009	.060	.087	.273	5.89
O_6	.020	.064	−.059	.253	5.03
	.033	−.023	.062	.215	3.66
	.000	.079	.061	.312	7.69
C_1	.015	−.042	.099	.306	7.40
	.037	.017	−.039	.224	3.96
	.037	.028	−.008	.262	5.43
C_2	.010	−.059	.087	.248	4.84
	−.007	.063	.086	.179	2.54
	.034	−.041	.024	.266	5.61
C_3	.017	.082	.015	.244	4.70
	.009	.002	−.119	.214	3.62
	.016	.079	.039	.297	6.95
C_4	.036	−.039	.006	.252	5.00
	.007	.024	−.116	.214	3.61
	.039	.015	.006	.249	4.88
C_5	−.007	.084	.042	.232	4.25
	.000	−.031	.115	.181	2.59

Table 2. Continued.

Atom	e_x	e_y	e_z	$(\overline{u^2})^{\frac{1}{2}}$ Å	B (Å^2)
	.017	.067	.063	.253	5.04
C_6	−.035	.033	.029	.226	4.03
	.000	.052	−.101	.178	2.50
	.033	.014	−.063	.244	4.69
C_7	.008	.074	.068	.220	3.81
	.020	−.052	.080	.196	3.03
	.036	.004	−.050	.233	4.27
C_8	.007	−.086	.034	.214	3.63
	.015	.029	.106	.184	2.68
	.030	.006	−.081	.271	5.80
C_9	.019	.059	.074	.233	4.28
	.018	−.069	.055	.192	2.92
	−0.38	.011	.027	.269	5.72
C_{10}	.009	.072	.069	.225	3.99
	.004	−.055	.097	.191	2.87
	.007	.063	.087	.319	8.04
C_{11}	.035	−.038	.024	.254	5.08
	.017	.054	−.083	.216	3.68

Although neither the temperature factors nor the shapes of the electron density peaks of C_3 and C_4 indicate observable disorder for these atoms, introduction of partial atoms was attempted. No reduction of the R-value was obtained, and the resulting distances after least squares refinement were still unreasonable.

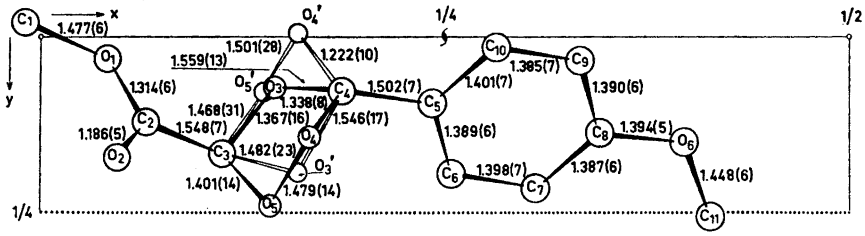


Fig. 4. Schematical drawing of the molecule (viewed along [001]) showing bond distances.

Fig. 4 and Table 3 show that the bond distances and angles of the rest of the molecule are normal. It may be seen from Table 2 that O_2 has a large thermal motion which possibly could be interpreted as observable disorder at this position, However, introduction of partial atoms, followed by least squares refinement, led to the obviously wrong result of two C=O distances of 1.09 Å and 1.32 Å, respectively.

Table 3. Bond angles and dihedral angles with estimated standard deviations.

Angle	(°)	Angle	(°)
$C_1-O_1-C_2$	117.0(4)	$C_5-C_4-O_3$	112.8(6)
$O_1-C_2-O_2$	125.4(5)	$C_5-C_4-O_4$	118.3(6)
$O_1-C_2-C_3$	111.4(4)	$C_3-O_3-C_4$	102.1(8)
$O_2-C_2-C_3$	123.1(5)	$O_3-C_4-O_4$	103.3(7)
$C_4-C_5-C_6$	123.7(4)	$C_4-O_4-O_5$	102.2(7)
$C_4-C_5-C_{10}$	117.9(4)	$O_4-O_5-C_3$	98.0(8)
$C_5-C_6-C_7$	121.6(4)	$O_5-C_3-O_3$	107.9(7)
$C_6-C_7-C_8$	118.7(4)	$C_2-C_3-O_5'$	115.7(1.4)
$C_7-C_8-C_9$	120.6(4)	$C_2-C_3-O_3'$	114.3(1.0)
$C_8-C_9-C_{10}$	120.0(4)	$C_5-C_4-O_4'$	120.0(7)
$C_9-C_{10}-C_5$	120.5(4)	$C_5-C_4-O_3'$	105.7(9)
$C_{10}-C_5-C_6$	118.5(4)	$C_3-O_3'-C_4$	97.6(1.2)
$C_9-C_8-O_6$	114.9(4)	$O_3'-C_4-O_4'$	111.4(1.1)
$C_7-C_8-O_6$	124.4(4)	$C_4-O_4'-O_5'$	98.8(1.5)
$C_8-O_6-C_{11}$	118.0(4)	$O_4'-O_5'-C_3$	96.6(1.1)
$C_2-C_3-O_3$	111.9(7)	$O_5'-C_3-O_3'$	100.0(1.2)
$C_2-C_3-O_5$	107.6(7)		

Dihedral angle	(°)
$C_3-O_3-C_4-O_4$	12.9(1.0)
$C_3-O_3'-C_4-O_4'$	−8.3(1.7)
$O_3-C_4-O_4-O_5$	−38.8(9)
$O_3'-C_4-O_4'-O_5'$	40.5(1.7)
$C_4-O_4-O_5-C_3$	51.2(8)
$C_4-O_4'-O_4'-C_3$	−58.0(1.7)
$O_4-O_5-C_3-O_3$	−43.3(1.0)
$O_4'-O_5'-C_3-O_3'$	51.7(1.9)
$O_5-C_3-O_3-C_4$	20.4(1.1)
$O_5'-C_3-O_3'-C_4$	−29.1(1.8)

The situation arrived at is indeed unsatisfactory. According to the formal standard deviations in C−O and O−O distances (based on the correlation matrix of the last cycle of least squares refinement) the results show *highly significant* deviations from expected bond lengths. The deviations are, however, so huge that the e.s.d.'s have to be regarded as unrealistically small.

When looking for an explanation of these somewhat confusing results, it should be pointed out that interaction between overlapping atoms subjected to least squares refinement may lead to wrong atomic positional parameters.[8] Furthermore, the intensity data were obtained from four different crystals of relatively poor quality. A comparison of structure amplitudes corresponding to common reflections from different crystals revealed large discrepancies; some of them so large (30 % or more in the F's) that one might suspect the relative amounts of the two types of molecules to vary from one crystal to another. Finally the possibility of wrong assumptions concerning the kind of disorder present may be considered.

The positions of the four hydrogens at the phenyl ring were calculated by assuming sp^2-hydridization with C−H bonds of length 1.03 Å. The methyl hydrogens could not be localized in the difference Fourier map. A comparison between observed and calculated structure factors is presented in Table 5.

Table 4. Least squares planes.

Plane No.	Atoms defining the plane	Deviations (Å)	Other atoms	Deviations (Å)
	C_2	.022	O_3	− .95
	C_3	−.149		
	C_4	.010	O_3'	.88
	C_5	.050		
	C_6	.067	O_4	1.25
(1)	C_7	.020		
	C_8	−.018	O_4'	− .93
	C_9	−.056		
	C_{10}	−.004	O_5	1.12
	O_6	−.060		
	C_{11}	.047	O_5'	−1.36
	C_1	−.008		
	O_1	.003		
(2)	C_2	.022		
	O_2	−.005		
	C_3	−.012		
	C_3	−.057		
	O_3	.060	O_5	− .69
(3)	C_4	−.081		
	O_4	.044		
	C_3	.029		
	O_3'	−.033	O_5'	.82
(4)	C_4	.053		
	O_4'	−.029		
	C_2	.0	O_4	.29
(5)	O_3	.0		
	C_4	.0	O_5	− .47
	C_3	.0	O_4'	− .16
(6)	O_3'	.0		
	C_4	.0	O_5'	.70

The results of the present structure analysis clearly show that the assignment of configuration (*trans*) is correct, but do not allow any definite conclusions concerning the detailed structure of the five-membered ring to be drawn. The dihedral angles of Table 3 and the least squares planes (3)−(6) of Table 4 show that the geometry arrived at for the ring with 35 % weight corresponds roughly to an envelope form, while the other is somewhere between an envelope and the conformation with symmetry C_2.

As pointed out before, bond distances and angles of the rest of the molecule agree within probable limits of error with earlier findings. In dimethyloxalate [6] the C=O distance is found to be 1.19 Å while C−O and C−CH_3 are 1.31 Å and 1.46 Å, respectively. The angle C−O−CH_3 is 118°. For 5-methoxy-2-nitrosophenol [7] the reported values for C−O and O−CH_3 bond distances are 1.34 Å and 1.46 Å, respectively, with estimated standard deviations of about 0.02 Å.

Table 5. Observed and calculated structure factors on ten times absolute scale.

h	k	l	F_o	F_c
2	0	0	907	- 932
4	0	0	885	- 821
6	0	0	652	600
8	0	0	50	- 51
10	0	0	1105	-1127
12	0	0	423	429
14	0	0	314	- 322
16	0	0	262	- 250
18	0	0	126	108
20	0	0	542	519
24	0	0	33	22
26	0	0	83	83
28	0	0	31	27
2	1	0	597	- 601
4	1	0	179	- 250
6	1	0	126	- 135
8	1	0	1236	-1266
10	1	0	478	- 456
12	1	0	822	767
14	1	0	347	341
16	1	0	153	169
18	1	0	143	- 108
20	1	0	112	- 81
22	1	0	67	56
30	1	0	44	- 56
0	2	0	257	243
2	2	0	122	117
4	2	0	837	833
6	2	0	1037	1040
10	2	0	256	- 258
12	2	0	93	- 102
14	2	0	206	166
16	2	0	101	- 121
18	2	0	278	316
20	2	0	164	- 142
22	2	0	102	96
24	2	0	74	57
28	2	0	53	49
2	3	0	695	- 715
4	3	0	205	- 168
6	3	0	780	- 782
8	3	0	182	174
10	3	0	162	154
12	3	0	72	- 48
14	3	0	220	211
16	3	0	110	- 102
18	3	0	234	- 245
20	3	0	314	326
22	3	0	119	- 138
26	3	0	53	- 52
0	4	0	227	201
2	4	0	71	- 90
4	4	0	527	583
6	4	0	485	495
8	4	0	86	- 70
10	4	0	207	220
12	4	0	156	- 134
14	4	0	237	274
16	4	0	128	- 121
18	4	0	177	- 191
20	4	0	90	- 95
22	4	0	205	198
24	4	0	158	154
26	4	0	51	38
2	5	0	125	- 125
4	5	0	136	114
6	5	0	513	444
8	5	0	209	- 241
10	5	0	485	- 481
16	5	0	152	- 170
18	5	0	176	183
20	5	0	211	216
22	5	0	224	- 232
26	5	0	122	122
0	6	0	452	462
2	6	0	348	361
4	6	0	69	85
8	6	0	27	- 20
10	6	0	38	32
12	6	0	83	- 72
14	6	0	257	- 257
16	6	0	199	- 202
18	6	0	432	440
20	6	0	88	74
22	6	0	126	- 126
24	6	0	84	88
2	7	0	58	- 53
4	7	0	443	- 428
6	7	0	88	- 82
8	7	0	180	- 164
10	7	0	74	- 70
12	7	0	219	- 226
14	7	0	113	116
18	7	0	169	147
20	7	0	65	61
0	8	0	143	149
2	8	0	151	141
4	8	0	153	- 131
6	8	0	42	- 50
8	8	0	77	- 90
10	8	0	62	57
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12	4	4	78	53
13	4	4	113	125
14	4	4	134	141
15	4	4	115	131
16	4	4	166	- 167
17	4	4	233	- 271
18	4	4	69	67
1	5	4	278	- 305
3	5	4	378	386
5	5	4	337	328

Table 5. Continued.

h	k	l	F_o	F_c
6	5	4	72	48
7	5	4	128	- 133
8	5	4	48	- 68
9	5	4	116	- 121
11	5	4	107	- 133
12	5	4	156	- 180
13	5	4	125	- 155
14	5	4	100	115
15	5	4	85	97
16	5	4	89	102
18	5	4	76	- 61
19	5	4	92	- 98
0	6	4	260	- 289
3	6	4	84	81
4	6	4	123	127
5	6	4	204	- 206
6	6	4	51	50
7	6	4	214	237
8	6	4	111	109
9	6	4	53	29
10	6	4	120	127
11	6	4	98	87
13	6	4	204	- 241
14	6	4	171	178
16	6	4	94	- 91
1	7	4	117	119
2	7	4	189	186
3	7	4	98	109
5	7	4	170	- 190
6	7	4	107	115
7	7	4	77	- 73
8	7	4	90	58
9	7	4	104	101
10	7	4	109	100
12	7	4	107	- 119
13	7	4	109	- 106
14	7	4	101	- 106
15	7	4	73	52
16	7	4	67	- 52
0	8	4	198	225
2	8	4	113	106
4	8	4	97	98
5	8	4	119	117
6	8	4	214	- 246
8	8	4	106	95
10	8	4	74	- 67
11	8	4	69	- 57
12	8	4	68	- 68
14	8	4	82	76
1	9	4	106	100
3	9	4	69	63
4	9	4	69	- 52
5	9	4	83	- 76
6	9	4	94	85
8	9	4	104	- 101
13	9	4	85	- 85
16	9	4	84	- 76
17	9	4	44	- 47
0	10	4	94	83
6	10	4	89	69
7	10	4	52	- 76
4	11	4	97	- 103
1	12	4	40	45
2	12	4	53	- 54
3	12	4	39	- 26
1	1	5	215	200
2	1	5	245	257
3	1	5	217	190
4	1	5	100	111
6	1	5	132	119
7	1	5	135	- 124
10	1	5	313	307
11	1	5	156	- 145
12	1	5	100	87
13	1	5	46	- 41
14	1	5	140	139
15	1	5	159	- 147
16	1	5	112	- 117
17	1	5	86	86
18	1	5	163	- 180
19	1	5	46	28
20	1	5	49	49
21	1	5	60	57
0	2	5	163	- 141
1	2	5	55	49
2	2	5	137	137
3	2	5	30	27
4	2	5	123	119
5	2	5	356	- 318
6	2	5	167	172
7	2	5	65	- 57
8	2	5	119	128
9	2	5	72	63
10	2	5	187	- 218
11	2	5	73	- 58
12	2	5	59	- 55
13	2	5	44	- 23
14	2	5	103	- 87
15	2	5	185	195
16	2	5	104	- 93
18	2	5	66	71
19	2	5	96	- 101
20	2	5	48	- 40
21	2	5	67	76
24	2	5	118	128
25	2	5	70	- 69

h	k	l	F_o	F_c
1	3	5	156	- 155
2	3	5	246	- 250
3	3	5	313	286
5	3	5	42	25
6	3	5	253	231
8	3	5	108	108
9	3	5	46	35
10	3	5	73	71
12	3	5	264	- 300
13	3	5	106	- 115
14	3	5	86	81
15	3	5	76	64
16	3	5	146	157
17	3	5	69	- 71
18	3	5	49	53
19	3	5	60	- 48
1	4	5	227	- 221
3	4	5	197	204
5	4	5	192	- 183
6	4	5	60	51
7	4	5	87	103
8	4	5	55	48
9	4	5	119	- 126
11	4	5	135	141
12	4	5	100	112
13	4	5	202	- 232
14	4	5	50	41
15	4	5	66	61
16	4	5	78	- 87
18	4	5	66	- 66
19	4	5	52	34
20	4	5	95	108
1	5	5	87	86
2	5	5	212	238
3	5	5	115	116
4	5	5	74	51
5	5	5	87	- 89
6	5	5	295	307
7	5	5	46	- 35
8	5	5	58	- 60
10	5	5	73	73
11	5	5	50	- 29
12	5	5	68	49
13	5	5	87	- 72
14	5	5	132	- 156
15	5	5	51	52
17	5	5	51	- 35
0	6	5	45	59
2	6	5	357	358
3	6	5	98	- 101
5	6	5	132	129
6	6	5	168	- 180
7	6	5	194	- 185
8	6	5	60	57
9	6	5	64	54
10	6	5	109	- 120
11	6	5	62	- 59
13	6	5	54	32
17	6	5	81	105
18	6	5	52	53
1	7	5	189	195
2	7	5	196	180
3	7	5	125	- 143
4	7	5	61	- 64
6	7	5	109	- 103
7	7	5	180	176
9	7	5	163	- 201
10	7	5	93	79
11	7	5	56	- 66
12	7	5	51	35
13	7	5	55	23
14	7	5	84	98
16	7	5	64	- 69
18	7	5	56	- 44
0	8	5	159	168
1	8	5	74	- 48
2	8	5	71	62
3	8	5	104	- 128
6	8	5	148	161
7	8	5	128	- 130
8	8	5	77	- 77
13	8	5	46	34
16	8	5	58	57
2	9	5	73	- 67
4	9	5	167	- 182
6	9	5	77	75
7	9	5	47	46
12	9	5	55	56
13	9	5	54	72
16	9	5	52	61
0	10	5	106	- 109
6	10	5	74	74
7	10	5	60	- 48
8	10	5	51	44
0	0	6	337	- 375
1	0	6	234	- 226
2	0	6	252	281
4	0	6	46	36
5	0	6	157	- 155
6	0	6	55	- 61
7	0	6	152	- 158
9	0	6	113	96
11	0	6	75	- 69
12	0	6	130	- 133
13	0	6	46	- 40
14	0	6	203	- 281

h	k	l	F_o	F_c
15	0	6	219	237
16	0	6	110	- 104
17	0	6	105	114
19	0	6	41	- 29
20	0	6	90	- 96
24	0	6	123	127
25	0	6	68	- 54
26	0	6	61	58
1	1	6	40	- 37
2	1	6	44	- 45
3	1	6	174	- 155
6	1	6	46	38
7	1	6	90	76
8	1	6	71	71
9	1	6	149	131
10	1	6	175	- 171
11	1	6	125	119
12	1	6	126	- 124
13	1	6	125	- 130
14	1	6	63	- 49
15	1	6	85	91
16	1	6	140	143
17	1	6	70	- 71
18	1	6	70	77
19	1	6	45	33
23	1	6	42	- 66
1	2	6	144	- 139
7	2	6	115	122
9	2	6	100	- 103
10	2	6	267	- 286
11	2	6	172	171
12	2	6	81	60
13	2	6	84	- 83
14	2	6	128	- 124
16	2	6	75	- 54
3	3	6	83	80
5	3	6	145	- 133
6	3	6	81	69
10	3	6	147	- 138
11	3	6	102	98
12	3	6	72	58
14	3	6	100	- 100
0	4	6	121	109
1	4	6	78	80
2	4	6	185	187
3	4	6	120	109
16	1	6	140	143
5	4	6	123	116
7	4	6	205	- 200
8	4	6	219	- 219
9	4	6	76	63
10	4	6	209	- 190
11	4	6	73	- 57
12	4	6	67	48
13	4	6	89	110
14	4	6	66	- 46
1	5	6	77	83
2	5	6	226	226
3	5	6	151	- 157
4	5	6	172	- 191
5	5	6	206	234
6	5	6	283	- 273
7	5	6	143	140
8	5	6	88	72
9	5	6	246	- 262
1	1	7	64	69
2	1	7	67	66
3	1	7	108	- 92
4	1	7	50	- 50
5	1	7	70	- 32
6	1	7	67	- 67
10	1	7	267	- 257
0	2	7	134	140
1	2	7	191	179
6	2	7	128	121
8	2	7	165	- 157
9	2	7	79	64
10	2	7	201	- 201
11	2	7	110	- 96
12	2	7	100	82
13	2	7	85	79
14	2	7	118	118
2	3	7	93	- 94
3	3	7	69	69
4	3	7	125	- 131
5	3	7	133	147
6	3	7	247	- 228
9	3	7	153	- 144
11	3	7	74	- 71
3	4	7	67	- 61
4	4	7	137	- 137
5	4	7	105	- 93
6	4	7	76	66
7	4	7	76	- 82
8	4	7	93	- 100
10	4	7	194	235
12	4	7	100	- 106
0	0	8	88	82
1	0	8	77	73
3	0	8	134	- 139
6	0	8	117	112
7	0	8	61	- 54
8	0	8	107	- 100
9	0	8	71	83
10	0	8	59	- 61
11	0	8	195	- 197

h	k	l	F_o	F_c
13	0	8	61	56
14	0	8	123	145
15	0	8	68	59
16	0	8	33	20
18	0	8	63	53
1	1	8	94	- 85
2	1	8	184	- 192
3	1	8	75	82
5	1	8	103	82
6	1	8	49	- 30
7	1	8	63	- 68
8	1	8	51	- 42
10	1	8	96	97
11	1	8	57	26
2	2	8	75	- 56
5	2	8	150	- 143
10	2	8	195	212
11	2	8	70	46
12	2	8	54	- 45
13	2	8	70	- 57
14	2	8	68	70
5	3	8	100	- 83
8	3	8	70	- 61
10	3	8	106	105
13	3	8	89	87
3	4	8	66	40
4	4	8	71	65
5	4	8	78	68
6	4	8	91	- 109
7	4	8	92	- 90
8	4	8	195	207
9	4	8	117	- 110
10	4	8	94	86
11	4	8	63	69
1	1	9	66	- 63
2	1	9	102	- 99
6	1	9	54	49
8	1	9	97	- 108
9	1	9	48	47
10	1	9	101	109
11	1	9	57	47
12	1	9	46	- 24
3	2	9	99	91
8	2	9	127	132
9	2	9	74	- 66
11	2	9	83	89
2	3	9	94	94
3	3	9	59	- 55
6	3	9	54	40
7	3	9	83	84

The angle between the two least squares planes (1) (indicated in Fig. 2) and (2) of Table 4 are about 80°. Owing to the fact that C_2, C_3, O_6, and C_{11} are situated very close to the benzene ring plane, the main difference in shape of D- and L-molecules (caused by the ester group orientation) is eliminated by a 180°-rotation of this group about C_2-C_3. This change of conformation, together with the fact that *inter*molecular distances between oxygen atoms of the ozonide rings are well above the van der Waals distance, make the necessary circumstances favourable for a disordered structure. The situation is analogous to that of dihydrothymine,[9] where the two enantiomorphs (no change of conformation) occupy all sites in the space group *Pbca* in the ratio 2:3.

No short *inter*molecular contacts are observed.

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