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Crystal Structure of *trans*-3,10-Dibromocyclodecane-1,2-dione

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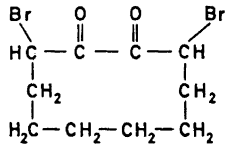
The crystals belong to the monoclinic system and the space group is $P2_1$. The unit cell, containing two molecules, has the following parameters: $a=8.52_2$ Å, $b=6.41_9$ Å, $c=11.04_0$ Å, $\beta=105.2_2°$. The structure was solved by the heavy atom method and refined to $R=6.3$ % ($R_W=5.1$ %) for 873 reflections observed by a four circle diffractometer. The conformation of the ring is similar to that previously observed in several cyclodecane derivatives, with the carbonyl groups so situated as to minimize the number of short transannular H···H contacts. Distances and angles of the ring skeleton are normal (mean values 1.51_8 Å and 116.6°). The bromines are situated at semiaxial positions. The C–C=O angles at the bromine sides are opened to 125° and 126°, respectively, probably due to repulsions between oxygen and bromine. Transannular distances between opposite pairs of methylene and carbonyl carbon atoms are 3.10 Å and 3.13 Å. The oxygens are *outside* the plane through the four carbon atoms of the (C–C(=O)–C(=O)–C)-group by 0.49 Å and 0.39 Å.

Several X-ray investigations of cyclodecane derivatives[1] have led to the conformation shown in Fig. 1 as the energetically stable one. The ring skeleton possesses $2/m$ symmetry to a good approximation and thus only three types of carbon atoms (I, II, III) can be distinguished.

By bromination of 1,2-di(trimethylsiloxy)-cyclodecene in chloroform at 25°C, equal amounts of two stereoisomers of 3,10-dibromo-cyclodecane-1,2-dione are obtained.[2]



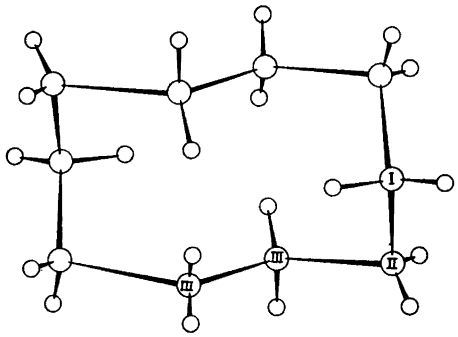
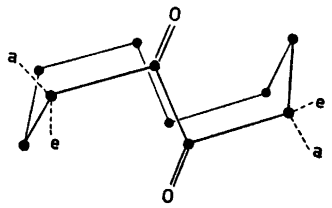


Fig. 1. Energetically favoured conformation of cyclodecane.

Following the arguments given by Dunitz,[3] it appears probable that the carbonyl groups occupy positions corresponding to the shortest transannular H···H contacts in cyclodecane. Since transannular H···H distances at III are 0.10 – 0.15 Å shorter than corresponding contacts at I and II, one would expect the carbonyls to be situated at III.

Under this assumption there are three possibilities for 3,10-substitution (all of which are extra-annular): (e,e), (e,a), and (a,a), where e and a means



semi-equatorial and semi-axial, respectively.

Infrared spectra (KBr and CCl_4) suggest the lowest melting isomer (m.p. 93 – 95°) to be (e,a), while the conformational problem of the other (m.p. 94 – 96°) is unsettled.

Bromination of cyclodecane-1,2-dione in chloroform yields the lower melting compound only, and no trace of a third isomer has been observed.[2]

The present single crystal X-ray structure determination of the higher melting isomer has been carried out in order to determine the carbonyl positions and to settle the conformational problem.

The crystals are monoclinic with two molecules in the unit cell. Cell dimensions, determined by a manual four circle diffractometer, with estimated standard deviations [4,*] are: $a = 6.522(5)$ Å, $b = 6.419(4)$ Å, $c = 11.040(6)$ Å, $\beta = 105.22(2)°$. $0k0$-reflections are systematically absent for $k = 2n+1$. Of the two possible space groups, $P2_1$ and $P2_1/m$, the latter requires that the molecules retain a mirror plane in the crystals.

* All programs used are included in this reference.

With $2\theta_{max} = 55°$ and Mo$K\alpha$-radiation, about 1400 reflections were measured by an automatic four circle diffractometer. 873 were recorded as observed using an observed – unobserved cutoff at $2.3 \cdot \sigma(I)$. The intensities were corrected for absorption effects (crystal size 0.26 mm × 0.21 mm × 0.08 mm).

Table 1. Fractional atomic coordinates for bromine, oxygen, and carbon atoms with estimated standard deviations (multiplied by 10^5).[a] Isotropic temperature factors for oxygen and carbon.

Atom	X	Y	Z	B (Å^2)
Br_1	14692	50000	−04911	
	19	0	14	
Br_2	89072	26033	39488	
	19	47	14	
O_1	55740	14676	25160	4.0
	126	192	87	2
O_2	51056	53282	03631	3.9
	116	199	73	2
C_1	28180	35802	09394	3.4
	192	298	125	3
C_2	46248	41867	10789	3.2
	175	243	123	3
C_3	58956	31814	21746	2.8
	166	242	119	3
C_4	74697	43088	26496	3.4
	176	261	129	3
C_5	73786	65339	30707	4.0
	223	296	149	4
C_6	67089	68628	42127	3.9
	236	318	154	4
C_7	49289	61591	40709	3.2
	209	285	141	3
C_8	36693	71554	30156	3.5
	199	279	136	3
C_9	21035	58886	26180	3.9
	205	270	157	4
C_{10}	21932	38006	20914	4.0
	216	313	161	4

[a] For numbering of atoms, see Fig. 1.

Table 2. (a) Anisotropic thermal vibration parameters for the bromine atoms with estimated standard deviations (multiplied by 10^5).

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Br_1	1453	4305	960	447	−72	715
	32	78	17	103	35	71
Br_2	1109	2870	1196	686	225	935
	27	57	19	91	34	66

(b) 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	$(\overline{u^2})^{\frac{1}{2}}$ (Å)	B (Å^2)
	.001	.146	.031	.307	7.42
Br_1	.072	.042	−.054	.268	5.67
	.098	−.033	.070	.194	2.97
	.001	.083	.077	.288	6.54
Br_2	.059	.114	−.031	.238	4.49
	.106	−.065	.044	.179	2.54

The three-dimensional Patterson map was (as expected) consistent with space group $P2_1$. The structure was solved by the heavy atom method and refined by full-matrix least squares technique, hydrogen positions being calculated by assuming tetrahedral C–H bonds of length 1.03 Å. With a common B-value of 5.0 Å^2, hydrogen parameters were included in the structure factor calculations, but not refined. Anisotropic temperature factors were introduced for the bromine atoms, and the weights in least squares were calculated from the standard deviations in intensities, $\sigma(I)$, taken as

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

where C_T is the total number of counts and C_N the net count (peak minus background). The conventional R-value arrived at was 6.3 % (weighted value $R_W = 5.1$ %) for 873 observed reflections. The form factors used were those of Hanson *et al.*[5] Final fractional coordinates and thermal parameters with estimated standard deviations are given in Tables 1 and 2. A comparison between observed and calculated structure factors is presented in Table 3.

Bond distances and angles may be found in Fig. 2 which shows the molecule viewed along [010]. The standard deviations, ranging from 0.015 Å to 0.026 Å and from 1.0° to 1.6° for distances and angles, respectively, were estimated from the correlation matrix corresponding to the last least squares refinement cycle.

Fig. 2 shows that the conformation of the molecule is very similar to the normal conformation of the cyclodecane ring with the trigonal atoms in type III positions, and the bromines substituted in (a,a) at the II positions.

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

h	k	l	F_o	$\lvert F_c \rvert$
0	0	1	151	145
0	0	2	236	223
0	0	3	684	674
0	0	4	54	14
0	0	5	373	384
0	0	6	538	555
0	0	7	123	112
0	0	8	78	66
0	0	9	324	305
0	0	11	204	183
0	0	12	96	73
0	1	1	571	545
0	1	2	338	338
0	1	3	434	425
0	1	4	461	470
0	1	5	250	253
0	1	6	405	420
0	1	7	421	423
0	1	8	117	105
0	1	9	104	106
0	1	11	95	101
0	1	12	95	66
0	1	13	93	69
0	2	0	129	121
0	2	1	1045	1056
0	2	2	440	426
0	2	3	241	244
0	2	4	473	495
0	2	5	299	311
0	2	6	130	117
0	2	7	176	173
0	2	8	212	200
0	2	9	72	71
0	2	10	265	256
0	2	13	110	85
0	3	1	315	323
0	3	2	423	430
0	3	3	500	503
0	3	4	192	198
0	3	5	235	238
0	3	6	285	291
0	3	7	185	194
0	3	8	175	172
0	3	9	93	97
0	3	12	109	94
0	4	0	346	363
0	4	2	358	355
0	4	3	286	287
0	4	4	152	144
0	4	5	251	253
0	4	6	79	67
0	4	7	72	59
0	4	8	76	39
0	4	9	193	181
0	4	11	96	89
0	5	1	74	80
0	5	2	302	297
0	5	3	203	185
0	5	4	181	181
0	5	5	102	111
0	5	6	97	85
0	5	7	114	103
0	5	8	101	98
0	5	9	85	60
0	6	1	182	191
0	6	4	148	128
0	6	5	92	73
0	6	8	107	89
1	0	-9	284	275
1	0	-8	338	329
1	0	-7	136	136
1	0	-6	476	489
1	0	-5	56	67
1	0	-4	439	447
1	0	-3	356	353
1	0	-2	784	747
1	0	-1	509	466
1	0	0	189	175
1	0	1	303	297
1	0	2	76	78
1	0	3	862	877
1	0	4	126	119
1	0	5	573	592
1	0	6	296	299
1	0	7	52	62
1	0	8	237	220
1	0	9	196	192
1	0	12	157	124
1	1	-13	90	71
1	1	-12	109	101
1	1	-11	91	58
1	1	-10	69	70
1	1	-9	204	214
1	1	-8	324	329
1	1	-7	100	87
1	1	-6	150	154
1	1	-5	294	300
1	1	-4	514	526
1	1	-3	975	959
1	1	-2	955	938
1	1	-1	808	763
1	1	0	704	629
1	1	1	686	669
1	1	2	509	502
1	1	3	421	424
1	1	4	218	219
1	1	5	390	399
1	1	6	405	411
1	1	7	354	360
1	1	8	223	223
1	1	9	160	146
1	1	10	150	147
1	1	11	124	117
1	2	-10	154	143
1	2	-9	106	96
1	2	-8	200	203
1	2	-7	387	404
1	2	-6	103	89
1	2	-5	530	553
1	2	-4	111	123
1	2	-3	138	147
1	2	-2	627	602
1	2	-1	558	558
1	2	0	247	231
1	2	1	408	398
1	2	2	466	484
1	2	3	41	29
1	2	4	777	812
1	2	5	125	132
1	2	6	151	160
1	2	7	199	210
1	2	8	155	151
1	2	9	96	98
1	2	10	150	127
1	2	11	110	96
1	3	-11	130	121
1	3	-10	97	75
1	3	-9	91	81
1	3	-8	63	48
1	3	-7	239	247
1	3	-6	103	109
1	3	-5	286	300
1	3	-4	363	375
1	3	-3	411	425
1	3	-2	407	415
1	3	-1	273	264
1	3	0	373	379
1	3	1	388	388
1	3	2	338	344
1	3	3	158	159
1	3	4	228	224
1	3	5	242	238
1	3	6	224	224
1	3	7	182	167
1	3	8	245	232
1	3	9	148	138
1	3	10	149	147
1	3	11	117	92
1	4	-11	83	41
1	4	-9	92	113
1	4	-6	404	445
1	4	-4	199	190
1	4	-3	199	191
1	4	-2	114	121
1	4	-1	265	268
1	4	0	436	410
1	4	1	167	171
1	4	2	125	127
1	4	3	427	425
1	4	4	129	132
1	4	5	253	263
1	4	7	142	117
1	4	8	104	108
1	4	9	103	96
1	4	10	88	65
1	5	-8	120	96
1	5	-6	102	117
1	5	-5	103	84
1	5	-4	191	191
1	5	-3	136	153
1	5	-2	201	209
1	5	-1	184	160
1	5	0	221	206
1	5	1	229	245
1	5	2	131	125
1	5	3	112	126
1	5	4	77	81
1	5	5	142	140
1	5	6	139	142
1	5	7	177	155
1	5	9	111	106
1	6	-7	112	91
1	6	-5	166	162
1	6	-2	80	72
1	6	-1	136	142
1	6	1	142	135
1	6	2	132	141
1	6	4	126	119
1	6	7	103	73
1	6	8	92	56
2	0	-12	124	109
2	0	-11	107	100
2	0	-10	105	94
2	0	-9	58	35
2	0	-8	187	180
2	0	-6	441	450
2	0	-5	61	45
2	0	-4	585	580
2	0	-3	1054	1037
2	0	-2	436	422
2	0	-1	559	508
2	0	0	179	156
2	0	1	230	215
2	0	2	38	26
2	0	3	836	874
2	0	4	181	194
2	0	5	349	351
2	0	6	387	397
2	0	8	378	365
2	0	12	106	81
2	1	-11	140	127
2	1	-10	230	220
2	1	-9	218	217
2	1	-8	285	288
2	1	-7	270	284
2	1	-6	319	330
2	1	-5	347	368
2	1	-4	503	512
2	1	-3	208	214
2	1	-2	304	293
2	1	-1	796	720
2	1	0	795	697
2	1	1	691	677
2	1	2	651	670
2	1	3	650	669
2	1	4	471	478
2	1	5	332	343
2	1	6	204	203
2	1	7	127	121
2	1	8	126	124
2	1	9	186	177
2	1	10	147	126
2	1	11	95	78
2	2	-11	106	106
2	2	-10	63	36
2	2	-8	65	67
2	2	-7	327	343
2	2	-6	125	127
2	2	-5	433	450
2	2	-4	390	419
2	2	-3	72	77
2	2	-2	636	645
2	2	-1	273	257
2	2	0	148	137
2	2	1	112	124
2	2	2	682	709
2	2	3	147	141
2	2	4	325	353
2	2	5	284	299
2	2	6	154	150
2	2	7	367	365
2	2	9	152	147
2	2	10	87	77
2	2	11	97	78
2	3	-11	98	71
2	3	-10	132	112
2	3	-9	210	207
2	3	-8	172	168
2	3	-7	101	106
2	3	-6	169	169
2	3	-5	350	356
2	3	-4	328	341
2	3	-3	263	260
2	3	-2	166	154
2	3	-1	273	260
2	3	0	516	489
2	3	1	517	534
2	3	2	388	415
2	3	4	278	292
2	3	5	234	241
2	3	6	251	248
2	3	8	109	82
2	3	9	142	143
2	3	10	87	80
2	3	11	112	84
2	4	-8	177	183
2	4	-6	216	231
2	4	-5	145	135
2	4	-4	132	136
2	4	-3	274	279
2	4	-2	157	160
2	4	-1	349	327
2	4	0	97	83
2	4	1	260	262
2	4	2	190	197
2	4	3	311	314
2	4	4	95	81
2	4	5	179	165
2	4	6	266	251
2	4	8	180	157
2	5	-9	109	93
2	5	-8	93	71
2	5	-7	127	115
2	5	-6	103	79
2	5	-5	134	139
2	5	-4	126	135
2	5	-3	165	158
2	5	-2	83	70
2	5	-1	214	186
2	5	0	235	221
2	5	1	269	257
2	5	2	128	123
2	5	4	82	92
2	5	5	215	205
2	5	6	99	83
2	5	7	113	76
2	5	8	84	97
2	6	-7	99	102
2	6	-5	85	66
2	6	-3	109	104
2	6	-2	226	214
2	6	2	109	98
2	6	3	77	75
2	6	4	193	188
2	6	5	106	86
2	6	7	99	108
2	7	-3	88	55
3	0	-12	168	134
3	0	-11	92	84
3	0	-10	99	113
3	0	-9	235	220
3	0	-8	102	91
3	0	-7	352	355
3	0	-6	240	239
3	0	-5	480	476
3	0	-4	103	114
3	0	-3	771	762
3	0	-2	265	264
3	0	-1	925	822
3	0	0	996	901
3	0	2	557	591
3	0	3	77	73
3	0	4	120	119
3	0	5	52	51
3	0	6	508	526
3	0	8	196	163
3	0	9	216	203
3	0	10	94	72
3	0	11	172	137
3	1	-11	114	112
3	1	-10	131	126
3	1	-9	115	113
3	1	-8	94	93
3	1	-7	176	183
3	1	-6	305	327
3	1	-5	455	472
3	1	-4	242	253
3	1	-3	74	85
3	1	-2	271	255
3	1	-1	687	606
3	1	0	787	713
3	1	1	207	211
3	1	2	372	385
3	1	3	395	408
3	1	4	455	470
3	1	5	284	295
3	1	6	192	195
3	1	7	166	171
3	1	8	150	125
3	1	9	90	95
3	1	10	133	125
3	2	-13	91	90
3	2	-11	148	136
3	2	-10	97	119
3	2	-9	80	88
3	2	-8	256	255
3	2	-7	225	226
3	2	-6	135	127
3	2	-5	54	72
3	2	-4	524	554
3	2	-3	59	54
3	2	-2	662	659
3	2	-1	339	318
3	2	0	412	376
3	2	1	709	735
3	2	2	84	80
3	2	3	351	380
3	2	5	217	225
3	2	7	339	328
3	2	8	105	126
3	2	9	153	141
3	2	10	166	143
3	3	-10	101	83
3	3	-9	77	78

Table 3. Continued.

h	k	l	F_o	$\lvert F_c \rvert$
3	3	-8	94	108
3	3	-7	191	198
3	3	-6	332	344
3	3	-5	124	133
3	3	-4	215	220
3	3	-3	195	197
3	3	-2	236	239
3	3	-1	385	361
3	3	0	418	394
3	3	1	121	128
3	3	2	299	300
3	3	3	311	322
3	3	4	220	229
3	3	5	272	281
3	3	6	102	107
3	3	7	111	77
3	3	8	146	141
3	3	9	140	124
3	4	-9	127	125
3	4	-7	112	119
3	4	-6	84	89
3	4	-5	114	118
3	4	-3	453	450
3	4	-2	81	48
3	4	-1	274	246
3	4	0	279	268
3	4	1	153	157
3	4	2	394	413
3	4	3	114	101
3	4	4	87	75
3	4	6	188	174
3	4	7	78	97
3	4	8	142	150
3	5	-9	86	42
3	5	-7	90	93
3	5	-5	230	224
3	5	-4	106	115
3	5	-3	71	51
3	5	-2	168	168
3	5	-1	179	176
3	5	1	202	192
3	5	3	192	172
3	5	4	198	205
3	5	5	78	88
3	5	6	110	99
3	6	-8	117	85
3	6	-4	153	145
3	6	-2	190	174
3	6	-1	113	90
3	6	1	166	151
3	6	2	101	97
3	6	3	113	89
3	6	5	98	90
3	6	7	101	75
3	7	-4	118	66
3	7	3	106	70
4	0	-13	105	59
4	0	-11	78	77
4	0	-9	280	284
4	0	-8	59	43
4	0	-7	289	280
4	0	-6	236	242
4	0	-5	114	109
4	0	-4	538	539
4	0	-3	347	348
4	0	-1	69	47
4	0	0	531	504
4	0	1	89	88
4	0	2	606	625
4	0	3	220	220
4	0	5	433	448
4	0	6	76	44
4	0	7	68	61
4	0	8	81	73
4	0	9	146	120
4	0	11	157	131
4	1	-13	83	49
4	1	-11	86	84
4	1	-10	81	79
4	1	-8	170	166
4	1	-7	199	200
4	1	-6	213	220
4	1	-5	342	351
4	1	-4	429	440
4	1	-3	468	452
4	1	-2	616	587
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4	1	1	108	120
4	1	2	287	307
4	1	3	349	360
4	1	4	363	394
4	1	5	254	264
4	1	6	305	318
4	1	7	218	211
4	1	8	196	194
4	1	9	154	129
4	2	-11	91	45
4	2	-10	158	156
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4	2	-5	355	364
4	2	-4	197	204
4	2	-3	319	334
4	2	-2	243	241
4	2	-1	443	431
4	2	0	240	234
4	2	1	558	563
4	2	2	92	97
4	2	3	251	262
4	2	4	287	306
4	2	5	111	114
4	2	6	237	242
4	2	7	89	67
4	2	10	149	137
4	3	-7	198	217
4	3	-6	238	261
4	3	-5	192	198
4	3	-4	169	170
4	3	-3	243	250
4	3	-2	290	270
4	3	-1	407	400
4	3	0	91	117
4	3	1	116	117
4	3	2	139	161
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4	3	4	166	163
4	3	5	238	241
4	3	6	153	144
4	3	7	202	205
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4	4	-6	186	170
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4	4	2	206	221
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4	5	-4	99	90
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4	5	2	86	111
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4	5	6	102	95
4	5	7	108	98
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4	6	2	104	60
4	6	4	108	112
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5	0	-9	164	139
5	0	-6	451	460
5	0	-4	489	494
5	0	-3	316	315
5	0	-2	181	174
5	0	-1	290	289
5	0	0	449	438
5	0	1	132	134
5	0	2	395	407
5	0	3	586	612
5	0	4	133	136
5	0	5	377	377
5	0	6	99	95
5	0	7	96	102
5	0	8	126	124
5	0	9	108	88
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5	1	-12	95	87
5	1	-11	91	91
5	1	-10	135	125
5	1	-9	133	130
5	1	-8	213	209
5	1	-7	199	199
5	1	-6	108	115
5	1	-5	83	79
5	1	-4	203	198
5	1	-3	369	372
5	1	-2	391	373
5	1	-1	303	303
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5	1	1	346	336
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5	1	3	346	360
5	1	4	114	108
5	1	5	67	72
5	1	7	178	175
5	1	8	114	123
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5	2	-13	90	77
5	2	-10	78	98
5	2	-9	82	63
5	2	-8	137	133
5	2	-7	204	213
5	2	-5	375	390
5	2	-4	81	88
5	2	-3	155	152
5	2	-2	294	284
5	2	-1	262	259
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5	2	1	351	350
5	2	2	207	206
5	2	4	435	454
5	2	6	224	216
5	2	7	142	126
5	2	9	116	100
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5	3	-10	118	89
5	3	-9	167	147
5	3	-8	128	140
5	3	-7	135	138
5	3	-6	170	168
5	3	-4	96	113
5	3	-3	346	347
5	3	-2	299	306
5	3	-1	232	235
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5	3	2	273	290
5	3	3	127	162
5	3	4	76	110
5	3	6	169	181
5	3	7	83	84
5	3	8	136	120
5	4	-9	102	110
5	4	-6	186	178
5	4	-5	100	56
5	4	-4	181	178
5	4	-3	137	116
5	4	-2	92	87
5	4	-1	193	193
5	4	0	197	194
5	4	3	187	187
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5	4	8	114	102
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5	5	-7	88	81
5	5	-4	102	114
5	5	-3	91	84
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5	5	-1	110	111
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5	5	2	153	161
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5	5	7	108	98
5	6	-5	156	127
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5	7	-3	123	48
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6	0	-12	108	89
6	0	-10	133	117
6	0	-8	65	53
6	0	-7	65	50
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6	0	-5	111	115
6	0	-4	150	153
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6	0	-2	91	58
6	0	-1	588	595
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6	0	3	113	127
6	0	4	89	36
6	0	5	203	194
6	0	6	172	167
6	0	8	263	241
6	1	-9	112	129
6	1	-8	166	159
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6	1	-3	193	193
6	1	-2	120	125
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6	1	4	144	140
6	1	5	200	191
6	1	6	179	156
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6	2	-11	154	127
6	2	-9	74	39
6	2	-7	118	119
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6	2	0	316	336
6	2	1	193	199
6	2	2	118	110
6	2	3	183	191
6	2	4	215	213
6	2	5	97	75
6	2	6	101	90
6	2	7	214	208
6	3	-9	87	109
6	3	-8	154	147
6	3	-6	75	74
6	3	-5	80	83
6	3	-4	230	219
6	3	-3	153	158
6	3	0	234	218
6	3	1	162	151
6	3	2	217	220
6	3	3	139	123
6	3	4	133	147
6	3	5	138	141
6	3	6	140	128
6	4	-10	108	70
6	4	-7	96	76
6	4	-6	153	137
6	4	-5	103	76
6	4	-4	171	161
6	4	-3	284	264
6	4	-1	248	269
6	4	1	108	99
6	4	2	111	94
6	4	3	165	174
6	4	4	87	103
6	5	-8	90	64
6	5	-3	93	92
6	5	1	169	146
6	5	2	118	85
6	5	4	115	95
6	6	-2	115	124
7	0	-11	82	40
7	0	-10	98	89
7	0	-9	88	88
7	0	-7	288	277
7	0	-5	112	104
7	0	-3	239	235
7	0	-2	94	102
7	0	-1	342	355
7	0	0	186	186
7	0	2	352	340
7	0	4	119	126
7	1	-10	77	69
7	1	-9	82	72
7	1	-8	113	119
7	1	-7	112	112
7	1	-6	161	167
7	1	-5	220	225
7	1	-4	255	244
7	1	-3	167	159
7	1	-2	130	111
7	1	-1	276	288
7	1	0	250	259
7	1	1	122	132
7	1	2	108	88
7	1	3	139	136
7	1	4	201	190
7	1	5	248	233
7	1	6	212	181
7	2	-8	150	159
7	2	-6	169	166
7	2	-4	120	105
7	2	-3	115	120
7	2	-2	270	262
7	2	-1	86	75
7	2	0	119	107
7	2	1	221	248
7	2	3	239	242
7	2	6	138	104
7	3	-7	81	80
7	3	-6	118	126
7	3	-5	191	192
7	3	-4	165	171
7	3	-3	117	126
7	3	-2	145	154
7	3	-1	148	164
7	3	0	182	176
7	3	1	118	134
7	3	2	87	99
7	3	3	131	108
7	3	4	153	155
7	3	5	150	154
7	4	-10	93	54
7	4	-9	91	50
7	4	-7	135	119
7	4	-6	99	56
7	4	-3	116	124
7	4	-2	101	85
7	4	-1	103	108
7	4	2	182	190
7	5	+5	107	87
7	5	-3	99	84
7	5	0	142	114
8	0	+9	123	129
8	0	-7	131	126
8	0	-4	232	232
8	0	-3	219	215
8	0	-2	122	109
8	0	0	219	229
8	0	2	266	270
8	0	5	191	176
8	1	-10	95	74
8	1	-9	107	89
8	1	-7	113	145
8	1	-6	130	134
8	1	-3	139	149
8	1	-2	245	250
8	1	-1	189	179
8	1	0	167	161
8	1	1	139	136
8	1	2	120	119
8	1	3	181	159

Table 3. Continued

h	k	l	F_o	$\|F_c\|$	h	k	l	F_o	$\|F_c\|$	h	k	l	F_o	$\|F_c\|$	h	k	l	F_o	$\|F_c\|$
8	1	4	142	145	8	3	0	130	122	9	0	1	143	145	9	3	-3	115	98
8	2	-8	135	135	8	3	3	145	142	9	1	-7	94	81	9	3	-2	104	93
8	2	-6	130	100	8	4	-4	112	115	9	1	-6	115	93	9	3	1	101	95
8	2	-5	148	127	8	4	0	125	116	9	1	-3	91	107	9	3	2	144	118
8	2	-4	106	104	8	4	2	142	134	9	1	-2	145	153	9	4	-4	128	124
8	2	-3	198	211	8	4	3	145	72	9	1	-1	110	117	10	0	-5	116	105
8	2	-2	80	102	8	5	-5	115	71	9	1	1	132	126	10	0	-4	89	77
8	2	1	239	242	8	5	-1	101	105	9	1	2	184	161	10	0	-3	90	98
8	2	3	187	166	8	5	0	103	91	9	1	3	112	103	10	0	-1	174	163
8	2	4	174	138	9	0	-6	139	130	9	2	-5	217	201	10	1	-3	124	100
8	3	-6	96	99	9	0	-4	212	215	9	2	-3	113	123	10	2	-3	96	18
8	3	-4	137	115	9	0	-2	106	95	9	2	-2	107	109	10	2	-2	107	125
8	3	-3	122	109	9	0	-1	192	183	9	2	0	147	165	10	2	0	96	133
8	3	-1	160	178	9	0	0	114	89	9	2	1	127	111					

All C – C distances are equal within probable limits of error, the mean value being 1.51_8 Å. Also the C – C – C angles have normal [1] values with an average of 116.6°.

The angles $C_1 - C_2 = O_2$ (125°) and $C_4 - C_3 = O_1$ (126°) are significantly larger than $C_3 - C_2 = O_3$ (118°) and $C_2 - C_3 = O_1$ (117°), respectively; an effect which may arise from repulsions between oxygen and bromine. The observed distances

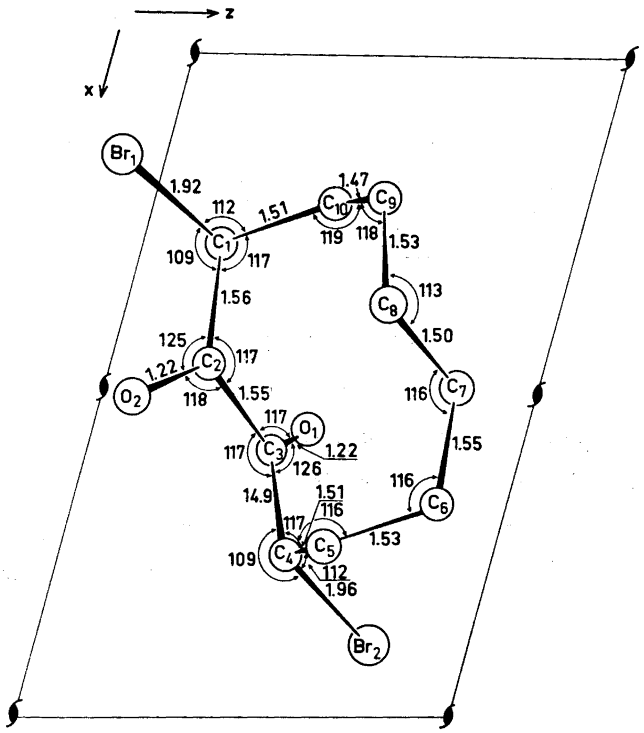

Fig. 2. Schematical drawing of the molecule (viewed along [010]) showing bond distances and angles. Standard deviations are about 0.02 Å and 1° for distances and angles, respectively.

Table 4. Dihedral angles. Standard deviations range from 1° to 2°.

Angle	(°)
$O_2-C_2-C_3-O$	−148
$C_1-C_2-C_3-C_4$	−156
$C_2-C_3-C_4-C_5$	56
$C_3-C_4-C_5-C_6$	65
$C_4-C_5-C_6-C_7$	−62
$C_5-C_6-C_7-C_8$	−58
$C_6-C_7-C_8-C_9$	159
$C_7-C_8-C_9-C_{10}$	−63
$C_8-C_9-C_{10}-C_1$	−58
$C_9-C_{10}-C_1-C_2$	65
$C_{10}-C_1-C_2-C_3$	52

$Br_1\cdots O_2$ (3.00 Å) and $Br_2\cdots O_1$ (2.96 Å) are still considerably shorter than the corresponding van der Waals contact (3.35 Å). Although the distances $Br_1\cdots O_1$ and $Br_2\cdots O_2$ become relatively short with (e,e)-substitution, it appears somewhat surprising that (a,a) is the preferred conformation (as mentioned, the third isomer was not obtained).

A shortening of the distances between opposite pairs of type III atoms, $C_2\cdots C_8$ (3.13 Å) and $C_3\cdots C_7$ (3.10 Å), compared with 3.29 Å in the cyclodecane ring, corresponds to the results obtained for cyclodecane-1,6-dione [1] and 2-oxa-cyclodecane-1,6-dione.[3]

The dihedral angles listed in Table 4 are in good agreement with earlier findings.[1]

The oxygen atoms, O_1 and O_2, are out of the least squares plane through C_1, C_2, C_3, C_4 by 0.49 Å and 0.39 Å, respectively, in *outward* direction.

No unusually short intermolecular contacts are observed.

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