ACTA CHEMICA SCANDINAVICA 18 (1964) 923–931

Crystal Structure Determination of Cyclohexane-1,4-dione from Three-dimensional Data Obtained at Room Temperature

P. GROTH and O. HASSEL

Universitetets Kjemiske Institutt, Oslo 3, Norway

Cyclohexane-1,4-dione belongs to the monoclinic space group $P2_1$. The unit cell containing *two* molecules has the parameters (at room temperature):

$a = 6.91$ Å; $b = 6.34$ Å; $c = 6.72$ Å; $\beta = 99.88°$

The structure was solved starting with the [010] projection and the structure factor signs determined using Harker–Kasper inequalities and by statistical use of Sayre's sign relationship.

The molecules have nearly two-fold symmetry and the conformation is that of a "twisted boat" form, the angle between the two C–O bonds being 154°.

The finding that the cyclohexane-1,4-dione molecule exhibits an electric dipole moment in solution and that the molecules cannot therefore all be centrosymmetric in solutions [1] was discussed at a meeting of the Faraday Society in 1934.[2] This result made it appear worth while to determine the crystal structure of the solid compound. However, the space group $P2_1$ and the number of molecules in the unit cell ($Z = 2$)[3] made a determination based on the older, conventional methods rather difficult. For this reason structure determinations have only recently been carried out.[4,5] The results of the two independent investigations are in good agreement and show that both at room temperature and at −140°C the molecule has a symmetry corresponding at least very nearly to that of a twofold axis (C_2) and that the molecular conformation may be described in terms of a "twisted boat" form with an angle between the two C–O bonds somewhat greater than 150°, an angle which is in good agreement with that to be expected from the dipole moment found in solution (1.3 D) if we may assume that all the molecules retain this conformation in the liquid phase.

The results of the three-dimensional structure determination carried out at room temperature are in good agreement with the results obtained from the two- and from the three-dimensional analysis carried out at −140°C.[5,6]

The lattice parameters determined by employing a Guinier camera are at room temperature:

$a = 6.913$ Å; $b = 6.338$ Å; $c = 6.720$ Å; $\beta = 99.88°$.

The solution of the phase problem will be described briefly because the method used differs from that followed by the Dutch investigators in their low temperature work. Starting with the projection along the b axis which has the plane group $P2$ and considering the fact that the number N of "heavy" atoms present in the unit cell is 16, the value of $1/N^{½}$ is 0.25, it was decided to apply direct methods to this projection. Employing statistical methods the structure factors were put on an absolute scale, and the temperature factor

Table 1. Final atomic coordinates and their standard deviations.

	x	σx	y	σy	z	σz
O_1	.60279	(.00053)	.17284	(.00122)	.53473	(.00062)
O_2	−.09176	(.00062)	−.00031	(.00124)	.08949	(.00078)
C_1	.44225	(.00069)	.17400	(.00138)	.42584	(.00076)
C_2	.25624	(.00073)	.17338	(.00153)	.51396	(.00072)
C_3	.07264	(.00070)	.22121	(.00127)	.35238	(.00078)
C_4	.06210	(.00076)	.07903	(.00129)	.17321	(.00082)
C_5	.25200	(.00079)	.02836	(.00140)	.10256	(.00080)
C_6	.42195	(.00070)	.17733	(.00159)	.19821	(.00082)

Table 2. Anisotropic thermal vibration parameters.

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
O_1	.01667	.02991	.02708	−.00310	−.00392	−.00563
O_2	.01888	.04900	.03473	−.01728	.00430	−.02166
C_1	.01741	.01487	.02192	−.00317	.00437	.00142
C_2	.01864	.02813	.01839	−.00335	.00781	−.00350
C_3	.01598	.02338	.02193	.00345	.00672	−.00643
C_4	.01752	.02116	.02270	.00032	.00325	−.00383
C_5	.01949	.02796	.02026	.00271	.00863	−.00095
C_6	.01596	.02879	.02554	.00296	.01323	.00910

Table 3. Interatomic distances and angles.

	Å	σ(Å)			σ(°)
O_1-C_1	1.220	0.0057	$C_1-C_2-C_3$	112.04°	0.32
O_2-C_4	1.223	0.0075	$C_2-C_3-C_4$	111.51	0.43
C_1-C_2	1.505	0.0074	$C_3-C_4-C_5$	117 46	0.50
C_2-C_3	1.552	0.0067	$C_4-C_5-C_6$	112.21	0.44
C_3-C_4	1.496	0.0092	$C_5-C_6-C_1$	109.98	0.50
C_4-C_5	1.504	0.0083	$C_6-C_1-C_2$	117.26	0.38
C_5-C_6	1.557	0.0098			
C_6-C_1	1.512	0.0075	$O_1-C_1-C_2$	120.94	0.89
			$O_1-C_1-C_6$	121.61	0.95
			$O_2-C_4-C_3$	122.44	0.95
			$O_2-C_4-C_5$	120.28	1.01

and unitary structure factors were computed. Only 18 unitary structure factors (U_H) were found to be greater than 0.3. Two signs — $s(205)$ and $s(501)$ — were arbitrarily put equal to +. Three unknown signs x, y, and z, corresponding to the reflexions (304), (30$\bar{2}$) and (003) were then introduced, and by applying the Harker-Kasper inequalities 14 additional signs could be expressed in terms of x, y, and z. From the list now containing 19 signs the statistical use of Sayre's relation yielded seven additional signs.

Corresponding to the 8 possible permutations of x, y, and z, eight Fourier maps were computed. The combination $x = -$, $y = +$, $z = -$ turned out to produce the most satisfactory Fourier map and to give a projection of the molecule which could easily be interpreted. After the last refinement it could be stated that all 26 signs had indeed been correctly determined.

The y-parameters for all the heavy atoms could now easily be derived from the sharpened 0kl-Patterson map. The final parameters resulting from the two-dimensional analysis were used in the first computation of the three-dimensional structure factors. The resulting R-value was 13.9 %. 4 cycles of the least-squares calculations were then carried out on the Ferranti MERCURY computer using a program written by Keilhau and Seip. Anisotropic thermal vibration parameters were refined for all the heavy atoms. The hydrogen atoms were not refined. The R-value finally arrived at was 6.9 %.

A comparison of the structural results with those obtained in the investigation carried out at a much lower temperature show that the agreement is very good, so good indeed that the results may be said to be identical within the probable limits of errors.

The finding that the molecular conformation is not based on the "chair" form of the cyclohexane ring but rather on a "twisted boat" configuration appears rather particular. It is easily seen that the configuration encountered in the solid is well suited for bringing the hydrogen atoms of neighbouring methylene group into a favourable mutual distance, but this might also have been the case, had the molecule been found to have the "chair" form. For this reason we would expect that the energy difference between the twisted boat and the chair conformation is comparatively small, and it would appear tempting to determine the molecular structure of the dione both in the gas phase and in some solid addition compounds which it might be expected to form with suitable acceptor molecules. Electron diffraction diagrams were taken in this laboratory a few months ago, and we hope that the result of their analysis may soon be available. As to addition compounds, we have prepared 1:1 compounds both with mercuric chloride and with diiodo-acetylene. The crystal structure of the former compound has been investigated and a short communication published.[7] In the mercuric chloride compound the symmetry of the cyclohexane-1,4-dione molecule is that corresponding to a two-fold axis and the conformation is again that of a "twisted boat", but the angle between the two C—O bonds is about 20° greater than in the crystal of the dione itself. In the addition compound formed with diiodoacetylene, however, space group ($P2_1/c$) and the number of molecules in the unit cell ($Z = 2$) would indicate that the molecule has a center of symmetry. A two-dimensional analysis of the structure is not incompatible with the assumption that the dione molecule is present in the "chair" conformation, but minor details in

Table 4. Observed and calculated structure factors and phase angles.

h	k	l	F_{obs}	F_{calc}	α	h	k	l	F_{obs}	F_{calc}	α
0	0	1	11.9	11.7	180	3	0	7	3.9	4.3	180
0	0	2	16.7	17.5	0	4	0	−8	1.5	1.5	0
0	0	3	20.1	20.5	180	4	0	−7	< 0.9	0.1	0
0	0	4	5.4	4.8	180	4	0	−6	1.3	1.2	0
0	0	5	2.9	3.3	180	4	0	−5	8.7	8.4	180
0	0	6	1.8	1.2	0	4	0	−4	3.9	3.5	0
0	0	7	2.4	2.4	180	4	0	−3	5.7	5.4	180
0	0	8	< 0.7	0.5	180	4	0	−2	1.6	0.4	180
1	0	−8	< 0.7	0.4	0	4	0	−1	1.4	1.0	180
1	0	−7	1.7	1.9	180	4	0	0	2.8	3.4	180
1	0	−6	1.3	1.5	0	4	0	1	2.0	1.7	0
1	0	−5	4.8	4.4	180	4	0	2	< 1.0	0.7	0
1	0	−4	13.0	12.7	180	4	0	3	4.8	4.2	0
1	0	−3	12.6	12.6	0	4	0	4	2.9	2.8	180
1	0	−2	5.9	6.4	180	4	0	5	3.2	3.4	0
1	0	−1	32.5	37.3	0	4	0	6	1.2	1.4	180
1	0	0	1.0	0.2	180	4	0	7	< 0.3	0.4	180
1	0	1	7.0	7.0	0	5	0	−7	< 0.9	0.3	0
1	0	2	10.0	10.2	180	5	0	−6	1.5	1.3	0
1	0	3	7.6	7.2	0	5	0	−5	< 1.2	0.1	180
1	0	4	3.8	3.6	180	5	0	−4	2.5	1.9	0
1	0	5	0.8	1.0	180	5	0	−3	2.8	2.3	180
1	0	6	2.4	2.0	0	5	0	−2	2.4	2.3	0
1	0	7	1.2	1.4	180	5	0	−1	1.8	1.2	0
1	0	8	1.2	1.2	0	5	0	0	< 1.0	0.5	180
2	0	−8	1.8	1.6	180	5	0	1	12.3	12.3	180
2	0	−7	1.3	1.2	0	5	0	2	< 1.0	0.6	0
2	0	−6	2.6	2.6	0	5	0	3	4.6	4.8	180
2	0	−5	1.0	0.2	180	5	0	4	5.0	4.9	0
2	0	−4	1.6	1.7	0	5	0	5	2.3	2.5	0
2	0	−3	9.5	10.3	180	5	0	6	< 0.6	0.6	180
2	0	−2	16.3	15.4	180	6	0	−7	2.9	2.9	0
2	0	−1	9.3	9.2	180	6	0	−6	0.9	1.0	180
2	0	0	11.7	11.9	0	6	0	−5	< 1.0	0.3	0
2	0	1	5.1	4.5	0	6	0	−4	1.7	1.8	0
2	0	2	2.9	2.1	180	6	0	−3	5.0	5.4	0
2	0	3	10.0	9.9	0	6	0	−2	0.8	1.6	180
2	0	4	6.5	6.4	180	6	0	−1	3.4	2.8	0
2	0	5	11.6	11.8	0	6	0	0	14.9	15.4	180
2	0	6	2.4	2.0	180	6	0	1	< 1.2	0.3	180
2	0	7	0.8	1.0	0	6	0	2	4.0	3.6	180
3	0	−8	< 0.7	0.1	0	6	0	3	3.7	3.5	0
3	0	−7	4.8	4.6	0	6	0	4	0.9	1.0	0
3	0	−6	1.5	3.5	0	6	0	5	1.4	1.5	0
3	0	−5	< 1.0	0.5	180	7	0	−6	1.2	0.7	180
3	0	−4	12.8	13.0	180	7	0	−5	1.4	1.4	0
3	0	−3	5.3	4.8	180	7	0	−4	1.7	2.2	0
3	0	−2	17.5	17.3	180	7	0	−3	1.8	2.0	180
3	0	−1	10.6	10.0	0	7	0	−2	< 1.0	0.7	0
3	0	0	3.9	3.6	0	7	0	−1	1.8	1.8	180
3	0	1	13.2	12.9	180	7	0	0	< 1.0	0.5	180
3	0	2	15.2	14.5	0	7	0	1	2.0	1.8	180
3	0	3	< 1.0	0.7	180	7	0	2	0.9	0.7	0
3	0	4	11.7	11.4	0	7	0	3	1.2	1.3	180
3	0	5	1.4	1.1	180	7	0	4	0.9	1.3	0
3	0	6	< 1.0	1.0	0	8	0	−5	2.5	2.4	180

h	k	l	F_{obs}	F_{calc}	α	h	k	l	F_{obs}	F_{calc}	α
8	0	−4	0.8	0.7	0	3	1	2	14.5	13.0	41
8	0	−3	< 0.9	0.0	180	3	1	3	2.5	2.3	171
8	0	−2	4.9	4.8	0	3	1	4	4.0	4.0	11
8	0	−1	1.3	1.1	0	3	1	5	6.5	5.9	123
8	0	0	1.7	1.6	180	3	1	6	2.4	2.5	100
8	0	1	0.9	1.2	180	3	1	7	1.1	1.1	102
8	0	2	< 0.6	0.6	0	4	1	−8	1.2	1.0	143
0	1	1	35.2	33.4	132	4	1	−7	1.5	1.2	59
0	1	2	22.2	23.3	93	4	1	−6	5.5	5.3	136
0	1	3	10.1	9.3	85	4	1	−5	4.2	4.1	11
0	1	4	1.9	2.0	70	4	1	−4	1.5	1.3	55
0	1	5	5.6	5.4	30	4	1	−3	3.2	2.6	96
0	1	6	4.1	4.2	170	4	1	−2	2.7	2.2	148
0	1	7	1.5	1.6	67	4	1	−1	3.2	2.7	71
0	1	8	< 0.6	0.7	168	4	1	0	3.5	3.0	146
1	1	−8	1.4	1.6	149	4	1	1	4.9	4.0	36
1	1	−7	2.8	2.8	21	4	1	2	2.5	2.3	92
1	1	−6	2.8	2.5	154	4	1	3	2.7	2.7	162
1	1	−5	2.1	1.9	178	4	1	4	1.5	1.7	0
1	1	−4	5.5	4.9	38	4	1	5	1.3	1.0	37
1	1	−3	12.6	11.6	67	4	1	6	1.2	1.4	135
1	1	−2	12.3	11.9	121	5	1	−7	1.6	1.0	32
1	1	−1	8.7	8.5	48	5	1	−6	0.9	0.6	144
1	1	0	19.6	21.2	144	5	1	−5	< 0.9	0.4	145
1	1	1	10.2	9.4	78	5	1	−4	1.4	0.9	179
1	1	2	13.3	12.1	45	5	1	−3	< 0.9	0.4	4
1	1	3	2.4	2.4	119	5	1	−2	4.0	3.8	58
1	1	4	2.2	1.6	175	5	1	−1	4.9	4.5	30
1	1	5	2.0	1.8	26	5	1	0	12.5	12.5	144
1	1	6	1.0	0.8	168	5	1	1	5.2	4.9	31
1	1	7	< 0.9	0.3	89	5	1	2	4.2	4.3	158
1	1	8	< 0.5	0.5	3	5	1	3	7.3	7.3	56
2	1	−8	1.5	1.4	156	5	1	4	1.7	1.5	71
2	1	−7	3.0	2.7	36	5	1	5	< 0.7	0.4	101
2	1	−6	1.5	1.9	140	5	1	6	1.7	1.7	146
2	1	−5	3.0	2.7	28	6	1	−7	1.2	1.1	65
2	1	−4	8.2	7.3	119	6	1	−6	3.7	3.2	164
2	1	−3	2.4	3.0	57	6	1	−5	2.4	2.4	20
2	1	−2	10.5	9.5	177	6	1	−4	1.9	1.9	7
2	1	−1	24.4	26.2	36	6	1	−3	2.5	3.0	114
2	1	0	9.0	8.5	146	6	1	−2	4.3	4.2	56
2	1	1	5.2	5.0	133	6	1	−1	10.7	10.2	135
2	1	2	2.0	1.8	101	6	1	0	2.2	2.1	1
2	1	3	3.1	2.6	130	6	1	1	1.6	1.4	76
2	1	4	3.7	3.5	22	6	1	2	6.2	6.2	65
2	1	5	3.7	3.4	22	6	1	3	2.7	2.7	136
2	1	6	5.0	4.4	131	6	1	4	1.7	1.9	49
2	1	7	2.4	1.9	101	6	1	5	1.7	1.9	150
3	1	−8	3.0	2.2	46	7	1	−6	0.7	0.9	12
3	1	−7	1.2	1.2	113	7	1	−5	0.9	0.8	20
3	1	−6	2.4	2.4	63	7	1	−4	1.3	1.6	159
3	1	−5	13.3	12.7	132	7	1	−3	0.9	1.0	126
3	1	−4	8.7	8.7	8	7	1	−2	1.5	1.8	69
3	1	−3	8.2	7.9	155	7	1	−1	1.1	1.2	25
3	1	−2	21.3	20.3	41	7	1	0	1.4	1.4	129
3	1	−1	9.7	9.4	146	7	1	1	1.9	1.8	45
3	1	0	13.2	12.5	106	7	1	2	1.9	2.0	159
3	1	1	7.7	7.1	145	7	1	3	0.6	0.8	59

h	k	l	F_{obs}	F_{calc}	a	h	k	l	F_{obs}	F_{calc}	a
7	1	4	< 0.3	0.9	12	3	2	−1	13.5	12.5	145
8	1	−5	< 0.3	0.6	137	3	2	0	1.7	1.7	143
8	1	−4	3.0	2.7	59	3	2	1	14.7	14.2	41
8	1	−3	1.0	0.7	100	3	2	2	3.2	2.7	98
8	1	−2	2.9	2.7	16	3	2	3	8.6	8.6	29
8	1	−1	4.8	4.9	144	3	2	4	7.6	7.4	82
8	1	0	1.2	1.3	51	3	2	5	2.8	2.7	34
8	1	1	0.7	0.8	44	3	2	6	1.9	2.1	127
8	1	2	< 0.5	0.3	131	3	2	7	2.2	2.3	89
0	2	0	37.6	39.4	99	4	2	−7	1.3	1.2	28
0	2	1	38.7	35.6	24	4	2	−6	0.8	1.1	144
0	2	2	10.1	9.6	101	4	2	−5	5.7	5.5	68
0	2	3	12.8	12.4	91	4	2	−4	7.8	8.1	161
0	2	4	6.6	6.1	160	4	2	−3	2.8	2.8	142
0	2	5	3.6	3.4	163	4	2	−2	4.3	3.8	100
0	2	6	3.2	3.6	153	4	2	−1	< 0.7	0.5	82
0	2	7	1.3	1.2	42	4	2	0	2.5	2.3	27
0	2	8	0.7	0.8	98	4	2	1	1.7	1.9	138
1	2	−8	1.7	1.5	156	4	2	2	2.9	2.9	100
1	2	−7	0.8	0.7	103	4	2	3	2.2	1.7	64
1	2	−6	3.1	3.4	146	4	2	4	4.6	4.7	31
1	2	−5	0.8	0.8	140	4	2	5	1.9	2.2	127
1	2	−4	6.2	5.9	72	4	2	6	< 0.6	0.6	63
1	2	−3	13.9	13.3	150	5	2	−7	< 0.6	0.4	180
1	2	−2	11.0	11.1	34	5	2	−6	1.1	1.0	129
1	2	−1	16.6	17.4	82	5	2	−5	< 0.9	0.7	52
1	2	0	19.1	21.4	29	5	2	−4	< 0.9	0.9	108
1	2	1	9.5	9.1	29	5	2	−3	4.3	4.1	13
1	2	2	6.1	6.1	80	5	2	−2	3.2	3.3	53
1	2	3	8.8	8.1	166	5	2	−1	3.6	3.7	145
1	2	4	< 0.9	0.5	3	5	2	0	6.1	6.1	149
1	2	5	1.1	0.8	10	5	2	1	7.0	6.7	99
1	2	6	1.7	1.7	100	5	2	2	5.2	5.3	179
1	2	7	1.3	1.4	25	5	2	3	4.4	4.5	46
1	2	8	1.0	1.0	151	5	2	4	2.7	2.6	102
2	2	−8	1.5	1.3	53	5	2	5	1.0	1.0	51
2	2	−7	1.5	1.3	26	6	2	−6	2.2	2.5	30
2	2	−6	1.9	1.5	24	6	2	−5	2.4	2.3	25
2	2	−5	1.8	1.6	25	6	2	−4	1.5	1.5	31
2	2	−4	4.7	4.9	166	6	2	−3	3.2	3.8	83
2	2	−3	7.5	7.0	146	6	2	−2	1.1	1.4	14
2	2	−2	1.9	1.7	164	6	2	−1	6.6	6.6	150
2	2	−1	3.3	2.7	69	6	2	0	7.2	7.1	81
2	2	0	9.8	9.4	136	6	2	1	6.7	6.9	167
2	2	1	2.5	2.1	22	6	2	2	2.5	2.5	115
2	2	2	9.1	8.7	73	6	2	3	2.7	2.7	91
2	2	3	2.6	2.3	69	6	2	4	1.9	2.2	8
2	2	4	10.6	11.0	27	6	2	5	< 0.3	0.8	4
2	2	5	8.5	8.2	107	7	2	−5	1.1	0.8	153
2	2	6	2.5	2.4	26	7	2	−4	1.3	1.2	149
2	2	7	2.1	2.0	131	7	2	−3	2.4	2.7	22
3	2	−8	2.1	2.1	6	7	2	−2	< 0.9	0.5	105
3	2	−7	2.5	2.4	62	7	2	−1	0.8	1.1	128
3	2	−6	1.5	1.2	52	7	2	0	2.3	2.5	148
3	2	−5	< 0.9	0.6	89	7	2	1	1.7	1.6	149
3	2	−4	7.5	7.5	102	7	2	2	1.1	1.2	124
3	2	−3	10.2	10.2	172	7	2	3	1.9	2.1	15
3	2	−2	3.6	3.6	145	8	2	−4	0.6	0.4	127

h	k	l	F_{obs}	F_{calc}	α	h	k	l	F_{obs}	F_{calc}	α
8	2	−3	2.5	2.2	5	4	3	−7	1.6	1.8	136
8	2	−2	2.7	2.3	97	4	3	−6	0.9	0.9	97
8	2	−1	0.6	0.7	22	4	3	−5	4.6	4.8	111
8	2	0	1.5	1.5	31	4	3	−4	< 0.9	0.4	90
8	2	1	0.8	0.9	115	4	3	−3	5.0	4.9	0
0	3	1	2.7	1.9	137	4	3	−2	4.7	4.6	71
0	3	2	12.2	11.2	134	4	3	−1	1.8	1.9	148
0	3	3	5.9	6.1	91	4	3	0	1.8	2.0	153
0	3	4	4.1	3.9	65	4	3	1	3.4	3.7	173
0	3	5	1.8	2.6	94	4	3	2	1.8	1.5	48
0	3	6	3.9	4.0	79	4	3	3	3.6	3.7	37
0	3	7	0.5	0.5	180	4	3	4	0.9	0.8	109
1	3	−8	0.9	1.1	33	4	3	5	1.5	1.5	103
1	3	−7	1.6	1.5	85	4	3	6	< 0.4	0.3	61
1	3	−6	2.3	2.4	74	5	3	−7	0.9	0.8	155
1	3	−5	2.1	2.2	49	5	3	−6	< 0.7	0.2	38
1	3	−4	2.2	2.3	168	5	3	−5	1.4	1.4	112
1	3	−3	1.3	1.1	19	5	3	−4	2.2	2.1	40
1	3	−2	9.0	8.3	52	5	3	−3	3.0	3.3	29
1	3	−1	4.6	4.5	119	5	3	−2	2.6	2.5	128
1	3	0	3.4	3.2	115	5	3	−1	7.4	7.3	116
1	3	1	9.0	8.7	125	5	3	0	4.9	4.9	106
1	3	2	8.2	8.0	128	5	3	1	3.8	4.0	135
1	3	3	4.5	4.6	28	5	3	2	5.1	5.3	71
1	3	4	4.3	4.3	32	5	3	3	2.3	2.3	36
1	3	5	< 0.9	0.1	174	5	3	4	1.8	1.7	91
1	3	6	< 0.9	0.3	60	5	3	5	0.6	0.7	36
1	3	7	1.4	1.2	167	6	3	−6	2.9	2.8	80
2	3	−8	2.2	2.2	47	6	3	−5	1.6	1.6	120
2	3	−7	0.6	0.7	82	6	3	−4	2.2	2.5	76
2	3	−6	< 0.9	0.8	69	6	3	−3	1.5	1.5	30
2	3	−5	4.4	4.6	106	6	3	−2	5.0	4.8	117
2	3	−4	2.8	3.0	146	6	3	−1	2.7	2.6	109
2	3	−3	2.9	3.2	52	6	3	0	3.8	3.8	111
2	3	−2	8.8	8.6	58	6	3	1	1.7	2.0	121
2	3	−1	6.5	6.3	127	6	3	2	2.3	2.3	4
2	3	0	6.1	5.9	100	6	3	3	3.6	4.0	72
2	3	1	4.0	3.9	116	6	3	4	0.6	0.7	151
2	3	2	4.9	4.7	91	7	3	−5	1.1	1.1	150
2	3	3	5.8	5.9	20	7	3	−4	1.4	1.5	22
2	3	4	3.0	3.0	50	7	3	−3	0.9	0.9	3
2	3	5	3.4	3.4	109	7	3	−2	1.1	1.4	152
2	3	6	0.6	0.3	120	7	3	−1	2.5	2.5	92
2	3	7	2.1	1.9	152	7	3	0	0.7	0.8	109
3	3	−7	0.9	0.9	149	7	3	1	< 0.7	0.1	10
3	3	−6	4.2	4.1	108	7	3	2	2.0	2.3	51
3	3	−5	3.8	3.7	104	8	3	−3	1.3	1.3	69
3	3	−4	9.7	9.5	106	8	3	−2	2.2	2.1	116
3	3	−3	6.1	5.7	47	8	3	−1	2.3	2.3	73
3	3	−2	6.7	6.3	76	8	3	0	1.3	1.4	114
3	3	−1	11.0	10.6	90	0	4	0	10.9	10.5	80
3	3	0	6.2	6.4	81	0	4	1	12.9	14.7	69
3	3	1	8.6	8.4	98	0	4	2	6.3	5.7	91
3	3	2	2.8	2.5	97	0	4	3	5.3	5.1	34
3	3	3	4.9	4.7	20	0	4	4	5.3	4.5	137
3	3	4	3.9	3.9	93	0	4	5	2.2	2.4	172
3	3	5	0.7	0.6	121	0	4	6	1.4	1.3	157
3	3	6	2.5	2.3	117	0	4	7	1.4	1.4	149

h	k	l	F_{obs}	F_{calc}	α	h	k	l	F_{obs}	F_{calc}	α
1	4	−7	1.3	1.0	160	3	4	6	1.6	1.6	147
1	4	−6	1.3	1.3	126	4	4	−7	1.3	1.3	59
1	4	−5	1.7	1.8	128	4	4	−6	1.4	1.1	96
1	4	−4	3.7	3.2	96	4	4	−5	1.8	1.6	103
1	4	−3	6.9	6.5	85	4	4	−4	6.5	6.2	98
1	4	−2	7.0	6.1	73	4	4	−3	1.1	0.9	128
1	4	−1	4.3	3.9	115	4	4	−2	3.9	4.0	161
1	4	0	10.4	11.8	53	4	4	−1	4.4	4.4	81
1	4	1	2.3	2.3	109	4	4	0	2.0	2.00	86
1	4	2	3.6	3.2	37	4	4	1	3.4	3.2	92
1	4	3	5.6	5.9	83	4	4	2	3.3	3.7	32
1	4	4	2.6	2.8	163	4	4	3	1.8	1.9	114
1	4	5	3.1	2.9	89	4	4	4	3.1	3.1	84
1	4	6	0.9	0.8	105	4	4	5	0.7	1.0	81
1	4	7	1.2	1.0	96	5	4	−6	1.4	1.2	106
2	4	−7	0.7	0.7	115	5	4	−5	1.2	1.1	89
2	4	−6	2.5	2.0	54	5	4	−4	2.5	2.4	7
2	4	−5	0.8	1.0	145	5	4	−3	2.5	2.6	84
2	4	−4	2.4	2.7	93	5	4	−2	3.3	3.1	106
2	4	−3	2.0	1.6	27	5	4	−1	< 0.7	0.4	80
2	4	−2	4.3	4.4	170	5	4	0	4.6	4.4	132
2	4	−1	4.7	4.4	107	5	4	1	1.1	1.0	114
2	4	0	4.6	4.5	108	5	4	2	4.4	4.1	94
2	4	1	3.8	3.9	116	5	4	3	2.3	2.3	104
2	4	2	5.1	4.7	11	5	4	4	1.4	1.4	100
2	4	3	3.5	3.3	57	6	4	−5	1.1	1.2	28
2	4	4	7.9	7.7	74	6	4	−4	2.2	2.2	4
2	4	5	2.2	2.1	96	6	4	−3	1.3	1.5	141
2	4	6	2.9	2.9	79	6	4	−2	2.0	2.3	104
3	4	−7	0.6	0.4	51	6	4	−1	3.1	3.0	116
3	4	−6	1.4	1.3	10	6	4	0	3.6	3.6	110
3	4	−5	1.8	1.7	132	6	4	1	4.9	4.8	109
3	4	−4	1.6	1.7	50	6	4	2	0.6	0.7	62
3	4	−3	5.3	5.3	119	6	4	3	2.0	1.9	143
3	4	−2	5.7	5.6	154	7	4	−4	1.4	1.3	64
3	4	−1	4.9	4.4	137	7	4	−3	1.7	1.8	61
3	4	0	0.9	0.8	161	7	4	−2	0.5	0.6	108
3	4	1	4.9	4.5	62	7	4	−1	0.5	0.6	61
3	4	2	6.3	6.1	50	7	4	0	2.5	2.4	128
3	4	3	5.3	5.2	38	7	4	1	0.4	0.6	22
3	4	4	2.3	2.6	122	7	4	2	0.5	0.4	163
3	4	5	3.7	3.6	64						

one of the Fourier maps lead to the conclusion that the possibility of a disordered structure based on a less symmetrical conformation cannot be definitively ruled out. For this reason a three-dimensional analysis has recently been started.

It appears interesting that the cyclohexane-1,4-dione molecule has been found to have, both in its solid low-temperature form and in an addition compound, a conformation which is based on the "movable" form of the cyclohexane ring. It may be added that a corresponding result has been derived using electron diffraction technique for the centre ring in the vapour form of one of the stereosiomeric forms of perhydroanthracene, that having a melting point of 50°C. The latter substance representing the *trans-anti-trans* form cannot,

however, be imagined to exist in a conformation in which all cyclohexane rings have the "chair" form.[8]

In Fig. 1 a schematical picture of the dione molecule is reproduced, in Fig. 2 a Fourier map with projection along [010].

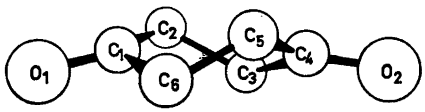

Fig. 1. A schematical picture of the dione molecule.

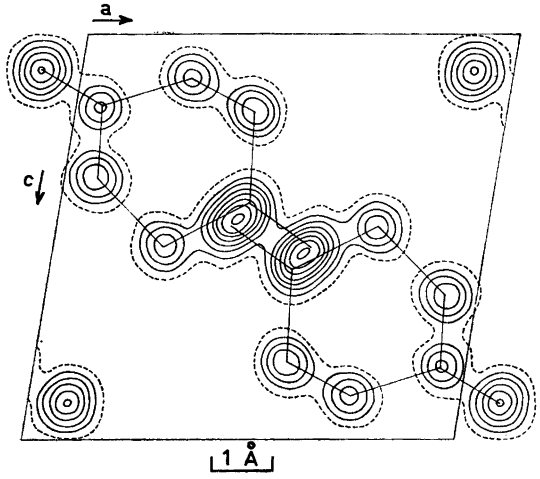

Fig. 2. Electron density map along the *b*-axis.

Table 1 contains the final atomic coordinates and their standard deviations, Table 2 the thermal vibration parameters and Table 3 bond lengths and angles within the molecule (*cf.* Fig. 1).

Table 4 contains observed and calculated structure factors and phase angles.

REFERENCES

1. Hassel, O. and Næshagen, E. *Tidsskr. Kjemi* **10** (1930) 81.
2. Hassel, O. *Trans. Faraday Soc.* **30** (1934) 874.
3. Hassel, O. *Quart. Rev. (London)* **7** (1953) 221.
4. Groth, P. and Hassel, O. *Proc. Chem. Soc.* **1963** 218.
5. Mossel, A., Romers, C. and Havinga, E. *Tetrahedron Letters* **1963** 1247.
6. Mossel, A. *Diss.*, Leiden 1963.
7. Groth, P. and Hassel, O. *Tetrahedron Letters* **1964** 65.
8. Davis, M. and Hassel, O. *Acta Chem. Scand.* **18** (1964) 813.

Received February 25, 1964.