given a very small perturbation with variation of counting time and any optimization produced by the use of (1) will be minimal. Table 1 gives the limiting values of I_j for various values of C.

Table 1. *Limiting values of I_j for various values of C*

C	0·10	0·08	0·06	0·04	0·02	0·01
10^4C^2	100	64	36	16	4	1
I_j	25	39	69	156	625	2500

It is of interest to compute the order of magnitude of I_j in inequality (4). The G index for a structure is defined as

$$G^2 = \frac{\sum_j \{|F_{0,j}| - |F_{C,j}|\}^2}{\sum_j |F_{0,j}|^2}$$

which, assuming normal distribution with the variance of $|F_{0,j}|$ as

$$\sigma^2\{|F_{0,j}|\} = \sigma_1^2\{|F_{0,j}|\} + \sigma_2^2\{|F_{0,j}|\}$$
$$\doteq \sigma_2^2\{|F_{0,j}|\} \text{ if } \sigma_1^2\{|F_{0,j}|\} \ll \sigma_2^2\{|F_{0,j}|\},$$

leads to

$$G^2 = C^2$$

or

$$G = C.$$

The G index and R index are numerically very similar and for a completed structure, using high values of I_j, might be of the order of 0·04.

It follows for this case that the analysis of Shoemaker (1968) is impoitant for counts of less than about 150, if negligible background counts are assumed. If the background counts are not negligible then it is easy to compute the effect on I_j. However, it should be noted that there is something suspect in limiting the counts in order to minimize the variances of the parameters. It would be better virtually to eliminate the $\sigma_1^2\{|F_{0,j}|\}$ term by increasing the counts, particularly as with $I_j = 150$ most of the time on the X-ray diffractometer would be spent setting the circles rather than measuring reflexions. This might not be so for a neutron diffractometer. It is extremely unlikely, however, that the optimization process described by Shoemaker will have any use in X-ray diffractometry aimed at normal stereochemical work ($R \simeq 0.05$, $I \simeq 150$), but may well be of practical use when it is possible to obtain diffractometers and crystals capable of producing much lower reliability indices ($R \simeq 0.01$, $I \simeq 2500$).

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The Crystal Structure of Benzo[1,2:4,5]dicyclobutene

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The structure of benzo[1,2:4,5]dicyclobutene, $C_{10}H_6$, has been determined by interpretation of the Patterson projections and refined by least-squares techniques in three dimensions. The molecule is planar and the benzene ring has been distorted from its usual geometry.

Introduction

The chemical structure of benzo[1,2:4,5]dicyclobutene is shown in Fig. 1. The benzenoid system must be highly strained as a result of fusion on opposite sides to cyclobutene rings and this strain is clearly manifested in its ultraviolet spectrum which shows both bathochromic shift and greatly enhanced extinction relative to its open analogue durene (Cava, Deana & Muth, 1960) An X-ray crystallographic investigation was undertaken to give a detailed picture of the type of strain present in the molecule.
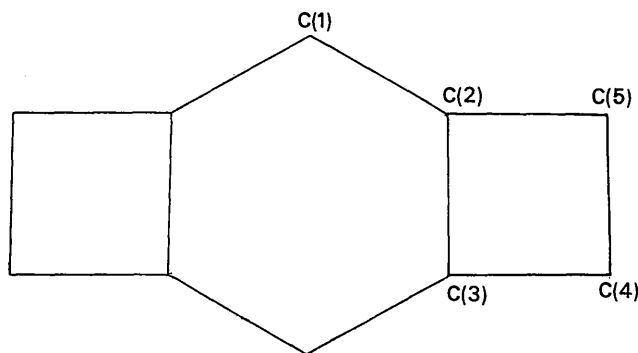
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Fig. 1. The chemical structure of benzo[1,2:4,5]dicyclobutene showing the numbering of the atoms.

Experimental

The crystals used were supplied by Professor M.P. Cava. They were approximately cubic in shape and were very fragile and cut very easily. They appeared to be highly mosaic and were covered with many smaller fragments of crystal which were difficult to remove and it was not expected that intensity data of high accuracy would be obtained. The crystals had to be enclosed in beryllium glass tubes since the substance sublimes slowly at room temperatures.

Using Cu $K\alpha$ radiation ($\lambda = 1\cdot5418$ Å), equi-inclination Weissenberg photographs of the zero, first, second and third layers about the a,b and c axes were
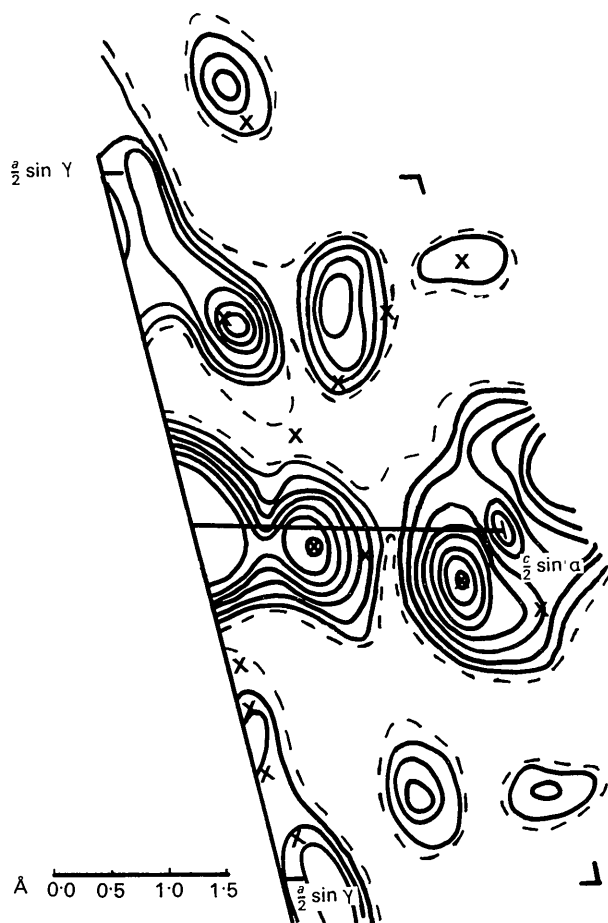

Fig. 2. The projection of the Patterson function on (010) with the positions of the interatomic peaks marked.

taken. Multiple-film techniques were employed and the relative intensities of the film were measured by visual estimation with the aid of a carefully prepared calibration strip. Lorentz and polarization factors were applied and the intensities were put on a common scale by applying scale factors between films and layers. 640 out of the 735 reflexions within the copper sphere were recorded with non-zero intensity.

Space group and cell dimensions

The cell dimensions were measured from zero-layer Weissenberg photographs by analytical extrapolation and were found to be

$$a = 5\cdot47 \pm 0\cdot02, \quad b = 5\cdot51 \pm 0\cdot02, \quad c = 6\cdot06 \pm 0\cdot02 \text{ Å}.$$
$$\alpha = 96\cdot0 \pm 0\cdot2°, \quad \beta = 106\cdot0 \pm 0\cdot2°, \quad \gamma = 96\cdot2 \pm 0\cdot2°.$$

The experimentally obtained density was ($1\cdot26 \pm 0\cdot04$) g.cm^{-3} compared with a calculated value of ($1\cdot27 \pm 0\cdot02$) g.cm^{-3} assuming one molecular per unit cell.

No systematic absences were detected and all the crystallographic angles were different from ninety degrees, showing the space group to be triclinic. Since there is only one centrosymmetrical molecule per unit cell the space group must be $P\bar{1}$ with the origin of the unit cell coinciding with the centre of symmetry of the molecule.

Structure determination

The structure was determined by interpretation of the projections of the Patterson function onto (001) and (010). Since the origin and the centre of the molecule coincide, peaks due to vectors between atoms related by a centre of symmetry, C(i)–C(i'), should appear in Patterson space at coordinates which are twice those of the atoms in real space; vectors between neighbouring atoms in the benzene ring, should produce peaks in Patterson space at the relative coordinates of the atoms in real space; and so on. All these peaks were identified in the projections of the Patterson function. Fig. 2 shows the projection on (010) with the positions of the interatomic peaks superimposed.

The R values for the two zones were 0·40 and 0·36, the R index being defined as $\Sigma||F_o| - |F_c||/\Sigma |F_o|$ and only the structure amplitudes of observed reflexions being included. Both zones were then refined by a series of structure factor calculations and difference-Fourier summations, the atomic scattering factors used being taken from International Tables for X-ray Crystallog-

Table. 1. Final fractional coordinates, standard deviations and isotropic temperature factors for benzo[1,2:4,5]dicyclobutene

	x/a	$\sigma(x)/a$	y/b	$\sigma(y)/b$	z/c	$\sigma(z)/c$	B
C(1)	0·2725	0·0020	0·0780	0·0014	0·1375	0·0016	3·75 Å^2
C(2)	0·1045	0·0015	−0·1000	0·0016	0·1855	0·0013	3·61
C(3)	−0·1450	0·0017	−0·1730	0·0015	0·0575	0·0015	3·30
C(4)	−0·1975	0·0015	−0·3590	0·0015	0·2070	0·0012	4·75
C(5)	0·0935	0·0018	−0·2820	0·0014	0·3565	0·0015	4·85

Table 2. *Measured and calculated values of the observed structure factors*

h	k	F_o	F_c
l = 0			
1	0	114	121
2	0	44	−43
3	0	37	−33
4	0	10	−13
5	0	8	−10
6	0	3	−3
0	1	62	68
1	1	23	−24
2	1	2	−2
3	1	27	31
4	1	8	11
5	1	8	−6
6	1	3	−3
$\bar{1}$	1	58	66
$\bar{2}$	1	17	−18
$\bar{3}$	1	31	−31
$\bar{4}$	1	25	−23
$\bar{5}$	1	14	−16
$\bar{6}$	1	2	−2
0	2	30	−26
1	2	29	−31
2	2	2	−3
3	2	20	16
4	2	4	4
5	2	2	0
6	2	2	2
$\bar{1}$	2	25	−25
$\bar{2}$	2	25	−26
$\bar{3}$	2	4	6
$\bar{4}$	2	20	16
$\bar{5}$	2	2	−1
$\bar{6}$	2	4	−6
0	3	2	2
1	3	25	−21
2	3	2	2
3	3	15	15
4	3	6	5
5	3	2	−2
$\bar{1}$	3	14	18
$\bar{2}$	3	13	−11
$\bar{3}$	2	9	−11
$\bar{4}$	3	7	10
$\bar{5}$	3	7	9
$\bar{6}$	3	2	−1
0	4	24	−19
1	4	2	−2
2	4	28	24
3	4	22	18
4	4	2	0
5	4	4	−4
$\bar{1}$	4	7	−9
$\bar{2}$	4	8	−10
$\bar{3}$	4	14	−11
$\bar{4}$	4	2	1
$\bar{5}$	4	5	6
$\bar{6}$	4	2	2
0	5	8	−9
1	5	2	2
2	5	7	7
3	5	2	0
4	5	2	−2
$\bar{1}$	5	15	−12
$\bar{2}$	5	12	−12
$\bar{3}$	5	8	−8
$\bar{4}$	5	6	5
$\bar{5}$	5	9	9
0	6	2	−3
1	6	5	−6
2	6	4	−5
$\bar{1}$	6	4	6
$\bar{2}$	6	4	6
$\bar{3}$	6	2	−2
0	7	2	−3
$\bar{1}$	7	4	3
$\bar{2}$	7	5	7
l = 1			
0	0	77	85
1	0	8	−9
2	0	40	−41
3	0	35	−31
4	0	18	−22
5	0	6	−6
6	0	7	7
$\bar{1}$	0	40	45
$\bar{2}$	0	28	−24
$\bar{3}$	0	10	7
$\bar{4}$	0	26	24
$\bar{5}$	0	6	5
$\bar{6}$	0	4	−5
0	1	129	138
1	1	28	−28
2	1	45	−41
3	1	17	−16
4	1	9	−12
5	1	5	−6
6	1	2	2
$\bar{1}$	1	198	202
$\bar{2}$	1	39	44
$\bar{3}$	1	31	−31
$\bar{4}$	1	19	−17
$\bar{5}$	1	8	−8
$\bar{6}$	1	8	−7
0	2	8	−7
1	2	8	−10
2	2	24	25
3	2	20	16
4	2	4	−5
5	2	4	−5
$\bar{1}$	2	47	47
$\bar{2}$	2	24	27
$\bar{3}$	2	16	−14
$\bar{4}$	2	20	−17
$\bar{5}$	2	13	−14
$\bar{6}$	2	6	−9
0	3	22	−19
1	3	2	−3
2	3	17	14
3	3	5	6
4	3	2	−1
5	3	2	1
$\bar{1}$	3	11	−10
$\bar{2}$	3	13	−12
$\bar{3}$	3	14	−11
$\bar{4}$	3	5	6
$\bar{5}$	3	6	7
$\bar{6}$	3	2	−2
0	4	11	−12
1	4	2	−2
2	4	9	11
3	4	4	6
4	4	2	−1
$\bar{1}$	4	7	4
$\bar{2}$	4	5	6
$\bar{3}$	4	8	−8
$\bar{4}$	4	2	−4
$\bar{5}$	4	6	7
$\bar{6}$	4	4	4
0	5	5	−5
1	5	15	13
2	5	11	14
3	5	2	2
$\bar{1}$	5	9	−10
$\bar{2}$	5	4	−4
$\bar{3}$	5	6	−6
$\bar{4}$	5	4	−5
$\bar{5}$	5	2	2
0	6	2	0
1	6	4	5
$\bar{1}$	6	4	−6
$\bar{2}$	6	4	−6
$\bar{3}$	6	4	6
0	$\bar{1}$	35	−34
1	$\bar{1}$	42	−46
2	$\bar{1}$	4	1
3	$\bar{1}$	24	27
4	$\bar{1}$	7	5
5	$\bar{1}$	9	−9
6	$\bar{1}$	2	−1
$\bar{1}$	$\bar{1}$	35	−38
$\bar{2}$	$\bar{1}$	33	−30
$\bar{3}$	$\bar{1}$	6	8
$\bar{4}$	$\bar{1}$	17	14
$\bar{5}$	$\bar{1}$	2	2
$\bar{6}$	$\bar{1}$	2	0
0	$\bar{2}$	40	36
1	$\bar{2}$	9	−8
2	$\bar{2}$	21	24
3	$\bar{2}$	13	11
4	$\bar{2}$	17	16
5	$\bar{2}$	2	0
6	$\bar{2}$	4	−4
$\bar{1}$	$\bar{2}$	9	−8
$\bar{2}$	$\bar{2}$	29	26
$\bar{3}$	$\bar{2}$	4	6
$\bar{4}$	$\bar{2}$	17	15
$\bar{5}$	$\bar{2}$	2	3
$\bar{6}$	$\bar{2}$	2	−2
0	$\bar{3}$	20	−18
1	$\bar{3}$	14	−14
2	$\bar{3}$	22	−21
3	$\bar{3}$	2	−4
4	$\bar{3}$	11	9
5	$\bar{3}$	4	4
6	$\bar{3}$	2	0
$\bar{1}$	$\bar{3}$	28	−29
$\bar{2}$	$\bar{3}$	4	6
$\bar{3}$	$\bar{3}$	37	32
$\bar{4}$	$\bar{3}$	20	17
$\bar{5}$	$\bar{3}$	2	−2
0	$\bar{4}$	21	−20
1	$\bar{4}$	24	−21
2	$\bar{4}$	19	−17
3	$\bar{4}$	4	4
4	$\bar{4}$	20	17
5	$\bar{4}$	6	8
$\bar{1}$	$\bar{4}$	10	−12
$\bar{2}$	$\bar{4}$	4	5
$\bar{3}$	$\bar{4}$	6	9
$\bar{4}$	$\bar{4}$	2	−1
$\bar{5}$	$\bar{4}$	2	−2
0	$\bar{5}$	8	9
1	$\bar{5}$	11	13
2	$\bar{5}$	2	0
3	$\bar{5}$	2	−4
4	$\bar{5}$	4	5
$\bar{1}$	$\bar{5}$	6	−7
$\bar{2}$	$\bar{5}$	7	−9
$\bar{3}$	$\bar{5}$	6	−7
$\bar{4}$	$\bar{5}$	4	−5
0	$\bar{6}$	4	3
1	$\bar{6}$	16	14
2	$\bar{6}$	10	9
3	$\bar{6}$	4	−2
$\bar{1}$	$\bar{6}$	4	−5
$\bar{2}$	$\bar{6}$	2	−2
0	$\bar{7}$	2	−1
1	$\bar{7}$	2	3
l = 2			
0	0	42	−43
1	0	7	−10
2	0	28	29
3	0	14	11
4	0	6	−9
5	0	2	−3
$\bar{1}$	0	26	−23
$\bar{2}$	0	30	−27
$\bar{3}$	0	20	−17
$\bar{4}$	0	8	9
$\bar{5}$	0	8	9
0	1	4	6
1	1	30	−31
2	1	31	−31
3	1	21	−23
4	1	7	−9
5	1	4	6
$\bar{1}$	1	57	52
$\bar{2}$	1	15	13
$\bar{3}$	1	16	−13
$\bar{4}$	1	6	8
$\bar{5}$	1	15	14
$\bar{6}$	1	2	1
0	2	6	−4
1	2	33	−32
2	2	20	16
3	2	12	−11
4	2	6	−7
$\bar{1}$	2	82	84
$\bar{2}$	2	85	90
$\bar{3}$	2	13	10
$\bar{4}$	2	16	−18
$\bar{5}$	2	6	−8
$\bar{6}$	2	5	−5
0	3	10	−10
1	3	10	12
2	3	16	14
3	3	4	−2
4	3	4	−5
$\bar{1}$	3	2	3
$\bar{2}$	3	25	24
$\bar{3}$	3	11	9
$\bar{4}$	3	8	−8
$\bar{5}$	3	8	−9
$\bar{6}$	3	6	−7
0	4	4	−4
1	4	6	8
2	4	6	6
3	4	2	0
4	4	2	1
$\bar{1}$	4	10	9
$\bar{2}$	4	2	3
$\bar{3}$	4	4	6
$\bar{4}$	4	2	−4
$\bar{5}$	4	4	4
$\bar{6}$	4	2	33
0	5	2	−3
1	5	6	7
2	5	5	5
$\bar{1}$	5	6	−6
2	5	2	2
$\bar{3}$	5	2	1
$\bar{4}$	5	4	−5
0	6	6	5
1	6	9	9
$\bar{1}$	6	4	−4
$\bar{2}$	6	4	−5
$\bar{3}$	6	2	−2
0	$\bar{1}$	33	4
1	$\bar{1}$	35	−34
2	$\bar{1}$	4	6
3	$\bar{1}$	27	23
4	$\bar{1}$	2	3
5	$\bar{1}$	4	−6
6	$\bar{1}$	2	−2
$\bar{1}$	$\bar{1}$	27	33
$\bar{2}$	$\bar{1}$	20	−18
$\bar{3}$	$\bar{1}$	20	−18
$\bar{4}$	$\bar{1}$	8	10
$\bar{5}$	$\bar{1}$	9	11
$\bar{6}$	$\bar{1}$	2	1
0	$\bar{2}$	20	−17
1	$\bar{2}$	26	−30
2	$\bar{2}$	13	−11
3	$\bar{2}$	20	−24
4	$\bar{2}$	6	7
5	$\bar{2}$	2	1
$\bar{1}$	$\bar{2}$	33	−29
$\bar{2}$	$\bar{2}$	31	−29
$\bar{3}$	$\bar{2}$	16	14
$\bar{4}$	$\bar{2}$	29	31
$\bar{5}$	$\bar{2}$	10	11
$\bar{6}$	$\bar{2}$	4	3
0	$\bar{3}$	35	−31
1	$\bar{3}$	34	−31
2	$\bar{3}$	2	0
3	$\bar{3}$	22	24
4	$\bar{3}$	13	15
5	$\bar{3}$	2	1
$\bar{1}$	$\bar{3}$	30	26
$\bar{2}$	$\bar{3}$	12	−11
$\bar{3}$	$\bar{3}$	6	9
$\bar{4}$	$\bar{3}$	6	7
$\bar{5}$	$\bar{3}$	2	−3
$\bar{6}$	$\bar{3}$	2	−1
0	$\bar{4}$	20	24
1	$\bar{4}$	4	5
2	$\bar{4}$	12	−10
3	$\bar{4}$	9	6
4	$\bar{4}$	10	10
5	$\bar{4}$	2	1
$\bar{1}$	$\bar{4}$	7	8
$\bar{2}$	$\bar{4}$	10	−12
$\bar{3}$	$\bar{4}$	10	−11
$\bar{4}$	$\bar{4}$	2	−1
$\bar{5}$	$\bar{4}$	4	−5
0	$\bar{5}$	24	21
1	$\bar{5}$	24	22
2	$\bar{5}$	2	−1
3	$\bar{5}$	7	−8
4	$\bar{5}$	4	−4
$\bar{1}$	$\bar{5}$	2	0
$\bar{2}$	$\bar{5}$	6	−6
$\bar{3}$	$\bar{5}$	2	−1
$\bar{4}$	$\bar{5}$	2	−3
0	$\bar{6}$	2	3
1	$\bar{6}$	6	6
2	$\bar{6}$	2	1
3	$\bar{6}$	2	−2
$\bar{1}$	$\bar{6}$	2	−3
$\bar{2}$	$\bar{6}$	2	0
$\bar{3}$	$\bar{6}$	2	1
l = 3			
0	0	26	−26
1	0	2	3
2	0	25	23
3	0	10	7
4	0	6	−6
5	0	2	−3
$\bar{1}$	0	13	11
$\bar{2}$	0	16	14
$\bar{3}$	0	19	−17
$\bar{4}$	0	6	−8
$\bar{5}$	0	9	10
$\bar{6}$	0	16	7
0	1	17	−14
1	1	20	18
2	1	17	15
3	1	6	−7
4	1	6	−5
5	1	2	4
$\bar{1}$	1	22	−21
$\bar{2}$	1	11	−10
$\bar{3}$	1	18	−15
$\bar{4}$	1	6	−6
$\bar{5}$	1	4	7
$\bar{6}$	1	4	5
0	2	16	−17
1	2	21	−19
2	2	20	−18
3	2	14	−12
4	2	2	3
$\bar{1}$	2	12	9
$\bar{2}$	2	26	23
$\bar{3}$	2	2	2
$\bar{4}$	2	2	−5
$\bar{5}$	2	6	6
0	3	20	18
1	3	13	11
2	3	6	8
3	3	6	−6
4	3	2	0
$\bar{1}$	3	6	5
$\bar{2}$	3	40	40
$\bar{3}$	3	29	30
$\bar{4}$	3	2	−1
$\bar{5}$	3	7	−8
0	4	4	3
1	4	6	9
2	4	2	0
3	4	4	−4
$\bar{1}$	4	4	−6
$\bar{2}$	4	4	5
$\bar{3}$	4	10	12
$\bar{4}$	4	2	2
0	5	4	4
1	5	4	5
2	5	2	0
$\bar{1}$	5	4	−3
$\bar{2}$	5	4	−4
$\bar{3}$	5	27	−1
$\bar{1}$	6	2	−3
0	$\bar{1}$	29	−27
1	$\bar{1}$	19	−17
2	$\bar{1}$	7	9
3	$\bar{1}$	12	10
4	$\bar{1}$	2	1
5	$\bar{1}$	2	1
$\bar{1}$	$\bar{1}$	18	−14
$\bar{2}$	$\bar{1}$	30	−27
$\bar{3}$	$\bar{1}$	20	−18
$\bar{4}$	$\bar{1}$	17	16
$\bar{5}$	$\bar{1}$	17	20
0	$\bar{2}$	40	−37
1	$\bar{2}$	7	−8
2	$\bar{2}$	26	28
3	$\bar{2}$	20	23
4	$\bar{2}$	4	5
5	$\bar{2}$	2	−1
$\bar{1}$	$\bar{2}$	35	32
$\bar{2}$	$\bar{2}$	26	24
$\bar{3}$	$\bar{2}$	10	−8
$\bar{4}$	$\bar{2}$	11	9
$\bar{5}$	$\bar{2}$	4	4
0	$\bar{3}$	15	13
1	$\bar{3}$	13	−11
2	$\bar{3}$	6	4
3	$\bar{3}$	17	15
4	$\bar{3}$	4	4
5	$\bar{3}$	4	−3
$\bar{1}$	$\bar{3}$	30	30
$\bar{2}$	$\bar{3}$	7	4
$\bar{3}$	$\bar{3}$	12	−14
$\bar{4}$	$\bar{3}$	8	−8
0	$\bar{4}$	30	29
1	$\bar{4}$	2	3
2	$\bar{4}$	11	−12
3	$\bar{4}$	6	−7
$\bar{1}$	$\bar{4}$	20	22
$\bar{2}$	$\bar{4}$	2	−3
$\bar{3}$	$\bar{4}$	4	−6
$\bar{4}$	$\bar{4}$	2	−1
0	$\bar{5}$	4	8
1	$\bar{5}$	2	3
2	$\bar{5}$	2	−3
$\bar{1}$	$\bar{5}$	2	4
$\bar{2}$	$\bar{5}$	2	−3
0	$\bar{6}$	6	6
l = 4			
0	0	16	−15
1	0	4	5
2	0	11	10
3	0	2	2
4	0	2	1
$\bar{1}$	0	18	−16
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0	1	5	−6
1	1	15	15
2	1	12	10
3	1	4	4
4	1	4	−3
$\bar{1}$	1	11	−13
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$\bar{5}$	1	2	−1
0	2	6	7
1	2	12	12
2	2	6	3
3	2	6	−6
$\bar{1}$	2	14	−10
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$\bar{3}$	2	8	−4
$\bar{4}$	2	6	−7
$\bar{5}$	2	2	−1
0	3	12	−11
1	3	12	−11
2	3	9	−9
3	3	2	0
$\bar{1}$	3	6	−7
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$\bar{3}$	3	7	9
$\bar{4}$	3	2	−1
0	4	6	−7
1	4	5	−5
2	4	5	−4
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0	5	5	5
1	5	2	1
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0	$\bar{1}$	17	−14
1	$\bar{1}$	22	22
2	$\bar{1}$	27	26
3	$\bar{1}$	9	7
4	$\bar{1}$	2	−2
$\bar{1}$	$\bar{1}$	31	−29
$\bar{2}$	$\bar{1}$	23	−21
$\bar{3}$	$\bar{1}$	20	−19
0	$\bar{2}$	10	−9
1	$\bar{2}$	2	−1
2	$\bar{2}$	20	17
3	$\bar{2}$	6	−7
4	$\bar{2}$	4	−3
$\bar{1}$	$\bar{2}$	21	19
$\bar{2}$	$\bar{2}$	26	24
$\bar{3}$	$\bar{2}$	1	−2
$\bar{4}$	$\bar{2}$	8	−10
0	$\bar{3}$	14	11
1	$\bar{3}$	10	−12
2	$\bar{3}$	12	−11
3	$\bar{3}$	4	−6
4	$\bar{3}$	4	−4
$\bar{1}$	$\bar{3}$	36	33
$\bar{2}$	$\bar{3}$	22	18
$\bar{3}$	$\bar{3}$	4	−5
$\bar{4}$	$\bar{3}$	2	−4
0	$\bar{4}$	4	5
1	$\bar{4}$	2	−3
2	$\bar{4}$	−2	−3
$\bar{1}$	$\bar{4}$	5	−7
$\bar{2}$	$\bar{4}$	2	0
$\bar{3}$	$\bar{4}$	2	−3
0	$\bar{5}$	4	5
1	$\bar{5}$	2	−2
2	$\bar{5}$	2	0
$\bar{1}$	5	6	7
$\bar{2}$	$\bar{5}$	2	−1
0	$\bar{6}$	4	4
1	$\bar{6}$	2	−1
l = 5			
0	0	10	11
1	0	24	23
2	0	10	9
3	0	2	−1
$\bar{1}$	0	16	−15
$\bar{2}$	0	20	−16
$\bar{3}$	0	10	−11
$\bar{4}$	0	6	−8
$\bar{5}$	0	2	0
0	1	2	2
1	1	4	8
2	1	2	2
3	1	2	0
$\bar{1}$	1	10	−10
$\bar{2}$	1	10	−8
$\bar{3}$	1	5	−6
$\bar{4}$	1	6	−9
0	2	6	8
1	2	10	8
2	2	2	−2
$\bar{1}$	2	4	−6
$\bar{2}$	2	4	−5
$\bar{3}$	2	4	4
0	3	6	7
1	3	2	0
2	3	4	−4
$\bar{1}$	3	2	1
$\bar{2}$	3	6	−5
$\bar{3}$	3	2	−2
0	4	4	−6
1	4	6	−6
$\bar{1}$	4	4	−4
0	$\bar{1}$	2	−4
1	$\bar{1}$	15	12
2	$\bar{1}$	10	9
3	$\bar{1}$	4	−2
$\bar{1}$	$\bar{1}$	4	−4
$\bar{2}$	$\bar{1}$	18	17
$\bar{3}$	$\bar{1}$	16	14
$\bar{4}$	$\bar{1}$	4	−4
0	$\bar{2}$	11	−10
1	$\bar{2}$	11	−12
2	$\bar{2}$	6	−7
$\bar{1}$	$\bar{2}$	18	16
$\bar{2}$	$\bar{2}$	31	28
$\bar{3}$	$\bar{2}$	7	9
0	$\bar{3}$	2	−2
1	$\bar{3}$	2	−3
2	$\bar{3}$	4	−4
$\bar{1}$	$\bar{3}$	4	6
$\bar{2}$	$\bar{3}$	6	7
0	$\bar{4}$	2	−2
1	$\bar{4}$	2	−1
2	$\bar{4}$	2	3
$\bar{1}$	$\bar{4}$	6	7
$\bar{2}$	$\bar{4}$	6	7
0	$\bar{5}$	2	0
1	$\bar{5}$	6	−4
$\bar{1}$	$\bar{5}$	6	5
l = 6			
0	0	9	7
1	0	8	8
2	0	2	−1
$\bar{1}$	0	4	−5
$\bar{2}$	0	2	0
$\bar{3}$	0	11	11
$\bar{4}$	0	6	6
0	1	10	13
1	1	7	8
2	1	2	0
$\bar{1}$	1	4	3
$\bar{2}$	1	8	−9
0	2	6	6
1	2	2	2
$\bar{1}$	2	2	0
$\bar{2}$	2	4	−6
0	3	4	5
$\bar{1}$	3	2	3
0	$\bar{1}$	11	−10
1	$\bar{1}$	8	−6
$\bar{1}$	$\bar{1}$	4	−5
$\bar{2}$	$\bar{1}$	15	13
$\bar{3}$	$\bar{1}$	21	17
0	$\bar{2}$	5	−3
1	$\bar{2}$	2	−3
$\bar{1}$	$\bar{2}$	2	−1
l = 7			
$\bar{1}$	0	7	−6
$\bar{2}$	0	2	−1
$\bar{3}$	0	10	9
$\bar{4}$	0	10	9

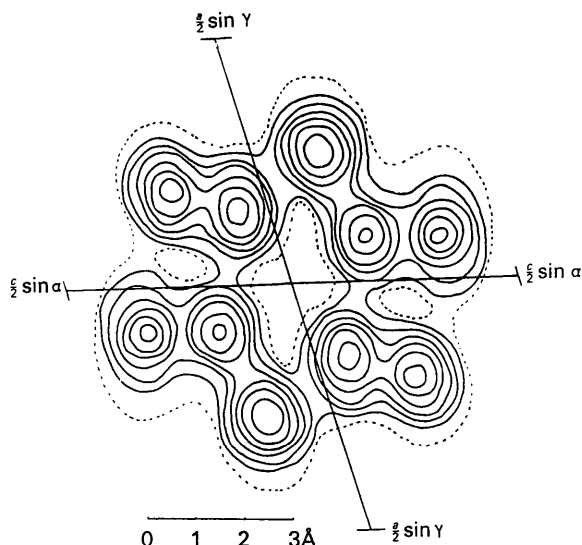
Fig. 3. The projection of the electron density distribution down the b axis. Contours are at intervals of 1 e.$Å^{-2}$, the broken contour being at 1 e.$Å^{-2}$.
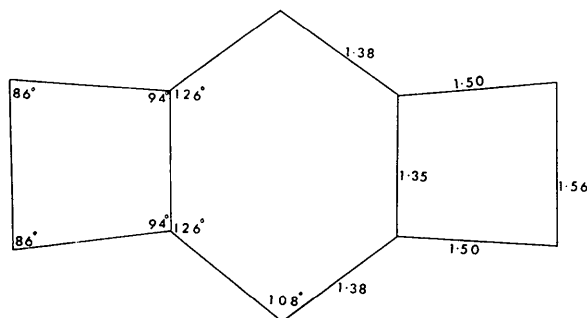

Fig. 4. The bond lengths and bond angles of benzo[1,2:4,5] dicyclobutene.
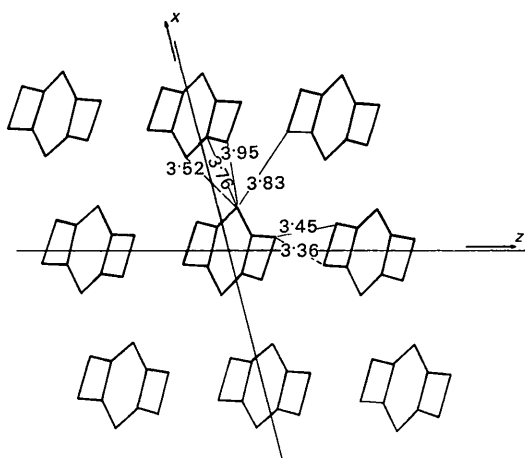

Fig. 5. The arrangement of the molecules in the crystal as viewed down the b axis, showing the shortest intermolecular distances.

raphy Vol. III. R values of 0·19 and 0·18 for the ($hk0$) and ($h0l$) zones resulted. The x coordinates calculated for both projections differed by not more than 0·05 Å. The structure was then refined three-dimensionally by least squares using a Hughes weighting scheme in a program written by J. C. Schoone (Rogers, 1961) for the Stantec Zebra computer, which refines the positional parameters and isotropic temperature factors. A final R index of 0·13 was obtained, after which there was no significant change in either the atomic coordinates or the temperature factors. The high R index was thought to be due to discrepancies in the intensity data caused by the poor quality of the crystals. No hydrogen atoms were detected, probably for the same reason.

The final projection of the electron density down the b axis is shown in Fig. 3, and the final coordinates, standard deviations and isotropic temperature factors are listed in Table 1, the standard deviations being calculated by the method due to Cox & Cruickshank (1948). Table 2 shows the observed and calculated structure factors.

Discussion of the structure

Fig. 4 shows the bond lengths and angles between the bonds for benzo[1,2:4,5]dicyclobutene. Several interesting features are apparent from the X-ray analysis. The angle C(3)–C(1')–C(2') has been compressed from 120° to 108° and the angle C(1)–C(2)–C(3) has been expanded to 126° while the bond C(2)–C(3) has been shortened to 1·35Å compared with the normally accepted distance of 1·395 Å, although this difference is not considered highly significant being only between two and three times the standard deviation of 0·02 Å. Whereas all the saturated C–C bonds in the parent molecule cyclobutene have the same normal single bond length, the bond C(4)–C(5) in benzo[1,2:4,5]dicyclobutene has a larger value of 1·56 Å. The lengthening of these bonds is undoubtedly a result of resistance in the molecule to a further decrease in the already compressed C(2)–C(3)–C(4) bond angles.

The molecule was found to be planar and the mean plane through all the atoms was

$$-0·552x + 0·741y + 0·535z = 0,$$

no atom being more than 0·005 Å from the plane.

The shortest distance between two carbon atoms in different molecules is 3·36 Å between atom C(5) of the molecule whose centre is (0,1,0) and atom C(5') of the molecule whose centre is at (0,0,1). The molecular packing down the y axis is shown in Fig. 5.

References

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Rogers, D. (1961). *Computing Methods and the Phase Problem in X-ray Crystal Analysis*, p.1. London: Pergamon Press.