being over all overlapping reflections contributing to an observed peak and *j* being the multiplicity of each reflection. The parameter values thus obtained are listed in Table 1 in parentheses. In the case of the oxygen atoms, the standard deviations are seen to be rather smaller, reflecting the relatively much larger neutron scattering amplitude of oxygen, and the overall agreement with the X-ray values is excellent. The appropriate *R* factor (on the basis of G^2 rather than *F*) is 0·072, and the observed and calculated intensities are given in Table 2.

The crystal structure of $MnSO_4$ is therefore as shown in Fig. 1. The sulfur atoms are at the center of slightly distorted oxygen tetrahedra, which are oriented so that one of the symmetry planes containing the sulfur and two oxygen atoms is parallel to (100), and the manganese ions occupy roughly octahedral sites. Table 3 lists some of the individual bond distances and angles. In these calculations, the *y* parameter of sulfur was taken from the X-ray determination, and the *z* parameter of O(1) from the neutron determination. All other values were averaged. The standard errors to be assigned to the interatomic distances lie between 0·02 to 0·03 Å.

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Refinement of cyclobutane structures.

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Octachlorocyclobutane and the centrosymmetric isomer of 1,2,3,4-tetraphenylcyclobutane (TPCB) are two of a small number of cyclobutane derivatives that have been studied by X-ray diffraction. The crystal structure of TPCB was reported by Dunitz (1949) and that of C_4Cl_8 by Owen & Hoard (1951). In each compound the reported C–C bond lengths in the cyclobutane ring are longer than the 1·54 Å usually accepted as the 'normal' C–C single bond length. The reported values are 1·555 ± 0·02 and 1·585 ± 0·02 Å for TPCB, and 1·58 and 1·60 Å for C_4Cl_8. Of the cyclobutane derivatives that have been studied by X-ray diffraction these two are unique in that the cyclobutane ring is not part of a condensed polycyclic system nor is there endo- or exocyclic unsaturation associated with the cyclobutane ring. Neither structure has been refined to any great extent owing to lack of computational facilities. This communication describes least-squares refinements of these structures, using the original data, carried out in an attempt to improve the reliability of the C–C bond lengths. The refinements have resulted in only small changes for these bonds; to 1·573 ± 0·015 and 1·566 ± 0·015 Å in TPCB, and

Table 1. *Atomic coordinates for* TPCB

Atom*	*x*	*y*	*z*
C(1)	0·0503	0·1416	0·0153
C(2)	0·1602	0·1533	0·0963
C(3)	0·2219	−0·0254	0·1859
C(4)	0·3241	−0·0123	0·2559
C(5)	0·3641	0·1767	0·2376
C(6)	0·3028	0·3586	0·1484
C(7)	0·2029	0·3458	0·0802
C(8)	−0·0031	0·0751	0·0796
C(9)	0·0539	0·0391	0·2313
C(10)	0·0381	−0·1526	0·2846
C(11)	0·0810	−0·1654	0·4218
C(12)	0·1426	0·0104	0·5081
C(13)	0·1612	0·1994	0·4566
C(14)	0·1157	0·2143	0·3179

* Numbering of atoms as in Dunitz (1949).

Table 2. *Anisotropic thermal parameters for* TPCB

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	0·0043	0·027	0·0066	0·0004	0·0033	0·0005
C(2)	0·0042	0·028	0·0066	−0·0022	0·0032	−0·0007
C(3)	0·0047	0·028	0·0072	−0·0013	0·0035	0·0002
C(4)	0·0057	0·039	0·0087	−0·0014	0·0049	−0·0016
C(5)	0·0045	0·038	0·0094	−0·0023	0·0043	−0·0031
C(6)	0·0057	0·032	0·0096	−0·0033	0·0049	−0·0027
C(7)	0·0060	0·028	0·0106	−0·0017	0·0059	0·0000
C(8)	0·0043	0·024	0·0070	−0·0004	0·0037	−0·0005
C(9)	0·0038	0·033	0·0061	0·0010	0·0030	0·0010
C(10)	0·0054	0·034	0·0078	0·0009	0·0043	0·0025
C(11)	0·0062	0·037	0·0082	0·0002	0·0044	0·0007
C(12)	0·0052	0·041	0·0076	0·0010	0·0038	−0·0001
C(13)	0·0055	0·042	0·0076	−0·0013	0·0040	−0·0023
C(14)	0·0051	0·034	0·0082	−0·0025	0·0042	−0·0034

to 1·57 ± 0·03 and 1·58 ± 0·03 Å in C_4Cl_8. These values may be compared with the electron diffraction result of 1·548 ± 0·003 Å for cyclobutane itself (Almenningen, Bastiansen & Skancke, 1961).

The centrosymmetric isomer of 1,2,3,4-tetraphenylcyclobutane crystallizes in space group $P2_1/a$; 2 molecules per cell; $a = 17·02 \pm 0·05$, $b = 5·775 \pm 0·02$, $c = 12·35 \pm 0·05$ Å, $\beta = 127 \pm 1°$. The least-squares refinement was carried out with the full-matrix program of Gantzel, Sparks & Trueblood on an IBM 7094 computer. The atomic parameters and 1620 observed structure factors of Dunitz (1949) were used in the refinement. The function minimized was $\Sigma w(|F_o|-|F_c|)^2/\Sigma w|F_o|^2$, where the weights, w, were assigned according to Hughes (1941), with $F_{\min}$ taken as 3·0 on the scale of Dunitz. Atomic scattering factors for neutral carbon were obtained from *International Tables for X-ray Crystallography* (1962). Anisotropic temperature factors were allowed to vary in the form $\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)]$. Hydrogen atoms were not included in the calculations. About 6 cycles of least-squares reduced the conventional unweighted R index from 0·21 (Dunitz, 1949) to 0·14 for the non-zero reflections with the results shown in Tables 1 and 2. The refined values of the bond lengths and angles are given in Tables 3 and 4. The standard deviation of a C–C bond is about 0·015 Å, estimated from the agreement of the 12 C–C bonds in the phenyl groups. Similarly, the standard deviation of a bond angle is about 1·0°. The average value for the aromatic C–C bond length is 1·394 ± 0·004. When corrected for thermal motion with one atom assumed to ride on another (Busing & Levy, 1964) the average value is 1·399 ± 0·004. These values are in agreement with the average value of 1·395 ± 0·003 reported by Sutton (1958) for aromatic C–C bonds. The cyclobutane ring is square within experimental error with C–C bond lengths of 1·573 and 1·566 Å, each ± 0·015 Å. Thermal motion corrections to the bond lengths within the cyclobutane ring are negligible.

The unit cell of C_4Cl_8 contains two molecules; space group $P2_1/m$; $a = 8·00$, $b = 10·64$, $c = 6·28$ Å, $\beta = 107° 45'$. The atomic coordinates of Owen & Hoard (1951) and the 612 observed structure factors listed by Owen (1950) were used in the least-squares calculations. The refinement procedure was essentially the same as described above with anisotropic temperature factors for chlorine and isotropic for carbon. $F_{\min}$ was chosen as 3·0 on the scale of Owen (1950). A total of 6 cycles of least-squares reduced R from 0·17 to 0·12 for the non-zero reflections with the results given in Tables 5 and 6. The standard deviations of the bond lengths listed in Table 7 are about 0·03 Å, calculated from the least-squares e.s.d.'s of the atomic parameters. The average C–Cl bond length is 1·74 ± 0·01 Å. Corrected for thermal motion as described above this value becomes 1·75 ± 0·01 Å, somewhat shorter than the average value of 1·767 ± 0·002 reported by Sutton (1958) for a paraffinic C–Cl bond. The cyclobutane ring is nonplanar with a dihedral angle* of about 19°. The C–C bond lengths are equal within experimental error with values of 1·57 and 1·58 Å, each ± 0·03 Å. Thermal motion corrections to these bond lengths are negligible.

Table 3. *Bond lengths in* TPCB

C(1)–C(8)	1·573 Å	C(4)–C(5)	1·375 Å
C(1)–C(8′)	1·566	C(5)–C(6)	1·422
		C(6)–C(7)	1·373
		C(9)–C(10)	1·395
C(1)–C(2)	1·500	C(9)–C(14)	1·385
C(8)–C(9)	1·520	C(10)–C(11)	1·386
		C(11)–C(12)	1·386
		C(12)–C(13)	1·392
C(2)–C(3)	1·412	C(13)–C(14)	1·395
C(2)–C(7)	1·406		
C(3)–C(4)	1·403		

Table 4. *Bond angles in* TPCB

C(3)–C(2)–C(7)	119·0 Å	C(8)–C(1)–C(8′)	89·4 Å
C(2)–C(3)–C(4)	120·3	C(1)–C(8)–C(1′)	90·6
C(3)–C(4)–C(5)	119·6		
C(4)–C(5)–C(6)	120·6	C(1)–C(2)–C(7)	119·0
C(5)–C(6)–C(7)	119·7	C(1)–C(2)–C(3)	122·0
C(2)–C(7)–C(6)	120·8	C(8)–C(9)–C(10)	122·0
C(10)–C(9)–C(14)	119·2	C(8)–C(9)–C(14)	118·5
C(9)–C(10)–C(11)	120·7		
C(10)–C(11)–C(12)	120·0	C(2)–C(1)–C(8)	122·8
C(11)–C(12)–C(13)	119·9	C(2)–C(1)–C(8′)	114·1
C(12)–C(13)–C(14)	119·9	C(1)–C(8)–C(9)	121·5
C(9)–C(14)–C(13)	120·3	C(1′)–C(8)–C(9)	118·2

Table 5. *Atomic parameters in* C_4Cl_8

Atom*	x	y	z	B(Å^2)
Cl(1)	0·2407	0·0896	−0·0090	
Cl(4)	0·2392	0·0173	0·4251	
Cl(5)	0·4615	0·2500	0·6411	
Cl(6)	0·0205	0·2500	0·4662	
Cl(7)	−0·0837	0·2500	−0·0050	
Cl(8)	0·5640	0·2500	0·2468	
C(1)	0·096	0·2500	0·240	3·2
C(2)	0·239	0·147	0·251	2·4
C(3)	0·386	0·250	0·345	2·9

* Numbering of atoms as in Owen & Hoard (1951).

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Table 6. *Anisotropic thermal parameters in* C_4Cl_8

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cl(1)	0·0172	0·0085	0·024	0·0001	0·008	−0·0034
Cl(4)	0·0158	0·0065	0·028	0·0003	0·006	0·0032
Cl(5)	0·0092	0·0099	0·021	0	0·000	0
Cl(6)	0·0081	0·0104	0·027	0	0·004	0
Cl(7)	0·0088	0·0112	0·030	0	−0·002	0
Cl(8)	0·0088	0·0113	0·037	0	0·001	0

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* The dihedral angle is defined as the angle between the normals to the planes of C(1)–C(2)–C(3) and C(1)–C(2)–C(4), assuming all C–C bonds to be of equal length and all C–C–C angles to be 88·4°.

Table 7. *Bond lengths in* C_4Cl_8

C(1)–Cl(7)	1·76 Å	C(2)–Cl(4)	1·76 Å
C(1)–Cl(6)	1·70		
C(3)–Cl(8)	1·72		
C(3)–Cl(5)	1·77	C(1)–C(2)	1·57
C(2)–Cl(1)	1·75	C(2)–C(3)	1·58

Table 8. *Bond angles in* C_4Cl_8

C(2)–C(1)–C(4)	88·4°	Cl(6)–C(1)–Cl(7)	109·1°
C(1)–C(2)–C(3)	88·9	Cl(1)–C(2)–Cl(4)	108·0
C(2)–C(3)–C(4)	87·8	Cl(5)–C(3)–Cl(8)	108·7

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A use for the Zoltai layer-line screen for the precession camera.

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Measurements of intensities of reflexions for structure determination are commonly made on photographs taken with the precession camera. This may be the most convenient method of scaling together different layers photographed with the Weissenberg camera if the crystals are very fine needles or plates. A disadvantage of the use of the precession camera for this purpose is the critical crystal-to-film distance which must be employed, rendering impossible the use of the multi-film pack widely used in Weissenberg photography. This difficulty is commonly overcome by taking a series of accurately timed exposures of a layer. However, this procedure may be difficult to apply if a wide range of intensities is encountered in a layer, and makes considerable demands on the operator and the stability of the X-ray generator.

A method of overcoming this difficulty is to use the double-spot eliminating screen, described by Zoltai (1963). This layer-line screen has a semicircular slit in it, instead of the normal full circle, and in use it rotates in its holder about the axis of the camera. Thus, each reflexion appears on the film only once in each complete precession cycle, and doubling of the spots on the film due to faulty camera adjustment, or misorientation of the crystal, is eliminated (Buerger, 1964).

By using the Zoltai screen, a multi-film pack may be used without any doubling of the spots on the lower films, and the normal methods of visual estimation of intensities may be employed. A standard film holder will take a pack of up to five films without any modification. The record on the lower films is slightly dilated but this effect is negligibly small, and in any case, as Zoltai points out, it is unwise to make accurate measurements of cell dimensions on photographs taken with the new screen.

Since each reflexion occurs only once in the precession cycle, twice the exposure is required with the Zoltai screen to obtain the same intensity as is given by the conventional screen. Thus, a somewhat greater overall exposure time is required for a pack of films than for a series of timed exposures of one film at a time, but a considerable saving of operator's time is effected.

If the crystal being photographed is badly shaped, the use of the Zoltai screen improves the shape of the spots, and thus facilitates visual estimation of intensities. For example, an acicular crystal mounted to rotate about a direction other than the needle axis may produce spots shaped like a cross with a conventional screen, owing to the superposition of two differently oriented elongated spots. The single elongated spot obtained with the Zoltai screen is much easier to measure than the composite spot. Thus, the use of the Zoltai screen may permit satisfactory photography of crystals which would otherwise give unsatisfactory spots.

The double-spot eliminating screen, as originally designed by Zoltai, was primarily intended for photographing zero layers. The screen containing the semicircular slit is caused to rotate in its holder about the camera axis by means of a stationary pin, fixed to the collimator assembly and lying in the camera axis, which locates in one of a series of holes drilled along a radius of the screen. Each hole corresponds to one of the available slit radii. This arrangement makes it difficult to photograph non-zero layers, since (using Buerger's (1944) notation) given a definite value of $\bar{\mu}$, there are only one or two fixed values of s which may be set if the stationary pin is to locate in one of the series of holes. In this laboratory, this difficulty has been overcome, and the screen made very much easier to adjust, by replacing the series of holes by a continuous slot. Then s is continuously variable for a fixed $\bar{\mu}$, and the nomograms commonly used for adjusting the camera may be used with the Zoltai screen in the normal way.

I wish to thank Professor Tibor Zoltai for providing me with the blueprints for the new screen, and Mr M. B. Forsyth and Mr J. A. Ducommun of the Physics and Engineering Laboratory, Department of Scientific and Industrial Research for their expert help in constructing it.

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