However if the imaginary parts of the anomalous scattering from the heavy atoms are also considered, the inverse reflexions $h0l$ and $\bar{h}0\bar{l}$ with $l \neq 0$ will not be of equal intensity. The relationships, for the present case, will be $|F|_{h0l} = |F|_{\bar{h}0l} = |F|_+$ and $|F|_{h0\bar{l}} = |F|_{\bar{h}0\bar{l}} = |F|_-$, where $|F|_+$ and $|F|_-$ can be appreciably different. Such differences can be expected to be very pronounced for reflexions in which the contributions of the Ti and O atoms to the structure factor oppose that of Ba. This gave another criterion for selecting the reflexions, namely h even and l odd. Calculations were made for these reflexions using the structural parameters of one model, No. 5 (Evans, 1961). The normal and anomalous scattering factors for the neutral atoms were taken from *International Tables for X-ray Crystallography* (1962).

The calculated values of the percentage intensity differences for such selected reflexions are listed in Table 1 for cases where they exceed about 5%. It was noticed that for the particular model employed in the calculations, $|F|_+$ was less than $|F|_-$ in all the h even, l odd reflexions. The differences are quite significant in a large number of reflexions as seen from Table 1. It seems therefore necessary to take into account the imaginary part of the scattering from the Ba and Ti atoms for the structure analysis following the methods that have been discussed (Patterson, 1963, Ibers & Hamilton, 1964).

In these calculations an overall isotropic temperature effect was assumed for the structure factors and so the intensity differences in the table are independent of temperature. Such a procedure was adopted in comparing the experimental and calculated intensity differences in the structure of L-tyrosine hydrochloride to obtain the imaginary part of the scattering for chlorine (Parthasarathy, 1962). It was there observed that while there was good agreement in the sign of the intensity differences, there were appreciable discrepancies in the magnitudes. In any general case, it can be shown that if anisotropic individual temperature factors are introduced, they would affect the intensity differences, to increase them for some reflexions and decrease them for others, depending on the structural and thermal parameters. Even if it is found that the measured differences are very small, it is clear that the parameters should account for such an observation.

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Table 1. *Percentage intensity differences for $h0l$, $\bar{h}0\bar{l}$ (h even, l odd) calculated with allowance for anomalous dispersion, but using an overall isotropic B coefficient*

Reflection $h0l$	Percentage difference $\frac{2(\lvert F\rvert_-^2 - \lvert F\rvert_+^2)}{\lvert F\rvert_-^2 + \lvert F\rvert_+^2}$ %
003	8·4
005	10·3
007	16·0
009	21·7
203	6·9
403	7·0
603	7·5
803	10·0
1003	10·5
205	11·3
405	12·3
605	13·6
805	14·0
207	15·1
407	17·0
607	20·0
807	24·0
209	22·0
409	22·6

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The crystal structure of $MnSO_4$*.

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The structures of most of the anhydrous sulphates of divalent 3d transition metals have been established. In the case of $MnSO_4$, the space group and lattice parameters have been reported (Rentzeperis, 1958; Coing-Boyat, 1959; Pistorius, 1960) but not the atomic positions. $MnSO_4$ crystallizes in the orthorhombic space group *Cmcm* (D_{2h}^{17}) with $a = 5{\cdot}267$, $b = 8{\cdot}046$, $c = 6{\cdot}848$ Å, and is isostructural with $NiSO_4$ (Dimaras, 1957; Poljak, 1958) and $MgSO_4$ (Rentzeperis & Soldatos, 1958), and also with $CrVO_4$ and several other chromates of divalent metals. However, in the course of an investigation of the magnetic structure of $MnSO_4$ (Will *et al.*, 1965), it was found that agreement between observed and calculated nuclear intensities in the neutron diffraction powder pattern using the published atomic parameters for $MgSO_4$ or $NiSO_4$ was very poor. We have therefore refined the crystal structure using both X-ray and neutron diffraction data, and the results are reported in this note.*

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* During revision of the manuscript, we became aware of an earlier X-ray study of $MnSO_4$ (Rentzeperis, 1958), where the isotypism of $MnSO_4$ with $MgSO_4$ and $NiSO_4$ is pointed out. The proposed parameters are derived without refinement from $MgSO_4$ by shifting only the sulfur atoms. They are not of sufficient accuracy to describe the structure and leave large discrepancies between calculated and observed intensities of the low angle reflections.

Some difficulty was encountered in preparing a suitably pure sample of the anhydrous material. Dehydration at various temperatures in air or argon yielded a product contaminated with small amounts of unidentified impurities. A satisfactory specimen was finally prepared by heating 'Baker analyzed' $MnSO_4 . H_2O$ for several days at 180 °C *in vacuo*. The weight loss was within 0·2 % of the theoretical value and the lattice parameters based on Cu $K\alpha$ radiation ($\lambda = 1{\cdot}542$ Å), were found to be $a = 5{\cdot}264$, $b = 8{\cdot}040$, $c = 6{\cdot}846$ Å, with $\sigma = 0{\cdot}003$ Å, in good agreement with published values. X-ray diffraction data for intensity measurements were collected from a powdered sample at room temperature on a Norelco diffractometer using filtered Cu $K\alpha$ radiation and a scintillation counter detector. In a two-day period the data were quite reproducible, indicating that hydration effects were negligible during this time. The peaks were integrated with a planimeter and corrected for Lorentz and polarization effects.

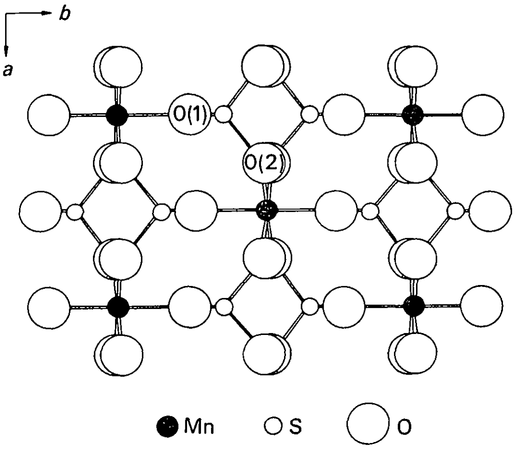

Fig. 1. The crystal structure of $MnSO_4$ in projection on (001).

Table 1. *Atomic parameters and temperature factors derived from X-ray and neutron diffraction*

Neutron diffraction data in parenthesis

Atom	Position	x/a	y/b	z/c	B
Mn	$4(a)\ (0,0,0)$	0	0	0	0·7
S	$4(c)\ (0,y,\frac{1}{4})$	0	0·361 ± 0·002 (0·355 ± 0·01)	$\frac{1}{4}$	0·7
O(1)	$8(f)\ (0,y,z)$	0	0·256 ± 0·003 (0·254 ± 0·002)	0·084 ± 0·006 (0·071 ± 0·003)	3·3
O(2)	$8(g)\ (x,y,\frac{1}{4})$	0·231 ± 0·006 (0·229 ± 0·005)	0·459 ± 0·003 (0·460 ± 0·003)	$\frac{1}{4}$	2·9

Table 2. *Comparison of observed and calculated structure factors and intensities*

All data are on a relative scale. For the X-ray data, $I_c = 1{\cdot}197 \times 10^{-5} \cdot \mathrm{Lp}\ \Sigma j F_c^2$, where the summation is over all reflections, of multiplicity j, contributing to the observed peak. The neutron diffraction data were calculated with $b_{\mathrm{Mn}} = -0{\cdot}36$, $b_{\mathrm{S}} = 0{\cdot}31$, $b_{\mathrm{O}} = 0{\cdot}577 \times 10^{-12}$ cm.

		X-ray diffraction data				Neutron diffraction data		
$H\,K\,L$	d_c	F_o	F_c	I_o	I_c	I_c		I_o
1 1 0	4·402	88·8	80·9	23	19·5	784		804
0 2 0	4·021	128·9	135·2	20·1	22·5	214		210
1 1 1	3·703	136·7	135·6	75·7	76·2	1229		1190
0 2 1	3·467	159·9	155·2	45·2	43·6	713	1225	1144
0 0 2	3·424		19·7			512		
1 1 2	2·702	221·2	218·9	100·0	100·0	4		n.o.
2 0 0	2·630		242·4			0		n.o.
0 2 2	2·607		55·6	30·5	31·8	2106		2102
1 3 0	2·388	218·6	220·8	37·0	38·5	0		n.o.
1 3 1	2·255		12·9	n.o.	0·2	61	2413	2482
2 2 0	2·201		23·5	n.o.	0·3	2472		
2 2 1	2·095		49·5	18·9	16·2	28	501	527
2 0 2	2·086		152·3			473		
1 1 3	2·026		3·8	7·1	6·2	428		
0 4 0	2·011	165·7	153·5			205		
0 2 3	1·985	100·7	103·5	5·1	5·5	176	1078	1442
1 3 2	1·959	67·6	73·1	4·4	5·3	122		
0 4 1	1·929	23·9	22·8	0·5	0·3	147		
2 2 2	1·851	163·4	160·0	22·7	22·2	0		n.o.
0 4 2	1·734	186·7	185·7	12·6	12·7	0		n.o.
0 0 4	1·712		216·7	13·3	11·3	96	102	50
3 1 0	1·713		90·2			6		
3 1 1	1·662	68·3	73·1	3·0	3·5	168	894	790
1 3 3	1·650	83·2	86·7	4·4	4·9	726		
2 4 0	1·597		71·6	2·7	4·2	1		
1 1 4	1·595		66·3			186		
2 2 3	1·584		40·9	5·6	8·7	43		
0 2 4	1·575		163·3			108		
2 4 1	1·555	59·2	63·4	1·9	2·3	547	1280	1123
1 5 0	1·538		124·3	8·7	14·4	75		
3 1 2	1·532		137·0			151		
0 4 3	1·509		21·2	n.o.	0·1	169		

Table 2 (*cont.*)

		X-ray diffraction data				Neutron diffraction data		
H K L	d_c	F_o	F_c	I_o	I_c	I_c		I_o
1 5 1	1·501		36·0	n.o.	0·6	34		
3 3 0	1·467	181·3	175·8	7·9	7·6	27	1098	1376
2 4 2	1·447	208·0	203·0	20·1	19·5	503		
2 0 4	1·435		110·4	2·0	2·8	300		
3 3 1	1·435		4·0			155		
1 5 2	1·403	78·8	77·4	2·4	2·6	67		
1 3 4	1·391	138·8	149·4	8·1	9·6	12		
3 1 3	1·370		9·8	n.o.	0·0			
2 2 4	1·351		75·8	n.o.	3·9			
3 3 2	1·349		40·6					
0 6 0	1·340		96·6					
0 6 1	1·315		15·4	4·2	6·9			
4 0 0	1·316		216·7					
2 4 3	1·309		43·1					
1 1 5	1·308		61·9					
0 4 4	1·303		41·7					
0 2 5	1·296		75·6	0·5	1·0			
1 5 3	1·275	98·5	80·6	3·4	2·3			
3 3 3	1·234		49·4	3·7	2·6			
4 2 1	1·230		70·4					
4 0 2	1·228		24·1					
3 5 2	1·120		59·1	n.o.	0·9			
3 3 4	1·114	121·9	131·7	4·0	4·8			
1 7 1	1·108		17·8	4·0	6·5			
1 1 6	1·105		117·7					
4 4 0	1·101		61·8					
0 2 6	1·098		89·8					
4 2 3	1·096		60·6					
3 1 5	1·069		47·4	3·0	5·0			
1 7 2	1·067		129·1					
2 6 3	1·058		69·4	6·3	11·0			
0 6 4	1·055		110·6					
3 5 3	1·052		73·5					
4 4 2	1·048		122·6					
2 0 6	1·047		27·6					
5 1 0	1·044		32·7					
4 0 4	1·043		133·5					
2 4 5	1·040		35·1					

Table 3. *Interatomic distances and angles*

		SO_4–tetrahedron			
O(1)–S	1·49 Å	O(1)–O(1′)*	2·45 Å	O(1)–S–O(1′)	110·4°
O(2)–S	1·45	O(1)–O(2)	2·38	O(1)–S–O(2)	108·2
		O(2)–O(2′)	2·42	O(2)–S–O(2′)	113·6
		MnO_6–octahedron			
O(1)–Mn	2·11 Å	O(1)–O(2)	3·13 Å	O(1)–Mn–O(2)	92·0°
O(2)–Mn	2·25	O(1)–O(2′)	3·03	O(2)–Mn–O(2′)	78·4
		O(2)–O(2′)	2·84		

* The primed figures distinguish equivalent positions.

A structure refinement based on the non-overlapping peaks was carried out by full-matrix least-squares methods using the program of Busing & Levy (1959) in a version modified by Ibers & Hamilton (1964) to accommodate corrections for anomalous dispersion. Atomic scattering factors for Mn^{2+}, S, and O, including dispersion corrections for Mn and S, were taken from *International Tables for X-ray Crystallography* (1962). The function minimized was $\Sigma(|F_{\rm obs}|-|F_{\rm calc}|)^2$, each observation being given unit weight. In the later cycles refinement was extended to include individual isotropic temperature factors, and an agreement index of $R=0\cdot033$ was ultimately reached. The final parameters and their estimated standard deviations are listed in Table 1. The positions are shifted with respect to those of isostructural NiSO$_4$ by between 0·01 Å and 0·4 Å. Table 2 lists observed and calculated structure factors and integrated relative intensities for all peaks.

Neutron diffraction data were obtained at 77 °K with thermal neutrons of wavelength $1\cdot03_3$ Å. Owing to the much lower resolution of the neutron spectrometer compared with the X-ray diffractometer, only four peaks were resolved. Consequently, structure analysis was performed by means of a least-squares program (Hamilton, 1964) in which $\Sigma w(I_o/{\rm Lp}-G_c^2)^2$ was the function minimized with a weighting scheme based upon the standard deviations of the observed intensities. Here, $G_c^2=\Sigma jF_c^2$, the summation 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