Table 1 (*cont.*)

	Mn			Fe			Co			Ni			Cu		
h k l	d_o	d_c	I_o	d_o	d_c	I_o	d_o	d_c	I_o	d_o	d_c	I_o	d_o	d_c	I_o
1 8 1	1·666	1·657	*ms*				1·495	1·510	*w*	1·490	1·509	*w*	1·639	1·646	*w*
2 8 1	1·610	1·601	*m*				1·464	1·473	*w*	1·462	1·471	*w*	1·610	1·600	*w*
6 3 1	1·589	1·593	*m*	1·605	1·603	*w*									
6 4 1	1·536	1·536	*m*	1·539	1·535	*m*									
4 8 0				1·495	1·500	*w*	1·440	1·434	*w*	1·439	1·433	*w*			
210 0	1·464	1·461	*m*										1·480	1·462	*m*
4 8 1	1·445	1·440	*vw*										1·440	1·439	*vw*
8 2 0	1·409	1·409	*w*	1·405	1·406	*w*	1·400	1·390	*w*	1·397	1·391	*w*	1·416	1·406	*vw*
5 1 2	1·394	1·394	*m*										1·372	1·389	*m*
1 7 2				1·361	1·363	*m*	1·322	1·330	*m*	1·318	1·327	*m*			
6 7 1													1·325	1·325	*m*
8 2 1	1·316	1·312	*s*	1·310	1·317	*s*	1·297	1·299	*s*	1·297	1·298	*s*	1·304	1·305	*s*
6 0 8				1·295	1·285	*w*									
9 1 1	1·195	1·195	*w*				1·177	1·180	*vw*	1·163	1·156	*vw*			
5 7 2	1·170	1·174	*vw*	1·181	1·180	*vw*							1·167	1·170	*vw*
012 0							1·109	1·109	*vwv*	1·106	1·108	*vvw*			
6 7 2				1·117	1·117	*vw*	1·099	1·096	*w*	1·095	1·095	*w*			
10 4 0	1·094	1·097	*vw*	1·091	1·091	*vw*	1·073	1·070	*vw*	1·059	1·069	*vw*	1·095	1·095	*w*
9 6 1	1·079	1·080	*m*	1·075	1·075	*w*	1·052	1·051	*w*	1·050	1·050	*w*	1·083	1·081	*m*
10 5 0	1·072	1·072	*w*				1·038	1·040	*w*	1·033	1·039	*w*	1·064	1·069	*w*
9 1 2	1·034	1·032	*m*	1·054	1·054	*m*							1·029	1·028	*m*
7 8 2	1·014	1·014	*w*				0·990	0·990	*w*	0·989	0·989	*w*	1·011	1·012	*w*
6 1 3	0·998	1·000	*m*	1·046	1·046	*w*									
5 6 3	0·966	0·967	*m*	0·992	0·996	*m*	0·992	0·992	*m*	0·989	0·989	*w*	0·966	0·965	*m*
12 0 0	0·955	0·955	*m*	0·960	0·958	*m*	0·938	0·940	*m*	0·937	0·940	*m*	0·954	0·953	*m*
10 4 2	0·930	0·932	*w*	0·938	0·940	*w*		0·935			0·933		0·929	0·930	*m*
2 3 4				0·920	0·921	*s*	0·918	0·918	*s*	0·918	0·918	*s*			

Table 2. *Crystallographic constants of* MNb_2S_4

M	*a* (Å)	*b* (Å)	*c* (Å)	X-ray density (g.cm^{-3})
Mn	11·470 ± 0·003	15·096 ± 0·007	3·536 ± 0·001	4·01
Fe	11·481 ± 0·002	14·071 ± 0·003	3·762 ± 0·001	4·05
Co	11·300 ± 0·004	13·315 ± 0·006	3·822 ± 0·002	4·25
Ni	11·299 ± 0·004	13·305 ± 0·004	3·805 ± 0·001	4·35
Cu	11·438 ± 0·005	15·121 ± 0·007	3·524 ± 0·001	4·12

were observed, indicating that the studied materials were single phase.

Magnetic measurements on all of these compounds, as well as Mössbauer effect studies of ^{57}Fe in $FeNb_2S_4$ (Eibschütz, Hermon & Shtrikman, 1966), are also consistent with the existence of one single phase in the compounds reported here.

Lattice constants were calculated by Cohen's (1935) analytical least-squares method (Eibschütz, 1965), and are given in Table 2.

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A method of allowing for thermal anisotropy in evaluating Wilson plots and normalized structure factors.

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A method has been developed for allowing for thermal anisotropy in calculating Wilson plots and evaluating normalized structure factors. The data are first brought to an approximately correct scale by the normal Wilson plot process, and a second series of plots is then made against six products of the Miller indices. The limitations of the method are discussed.

The reliability of direct methods of phase determination (Karle & Karle, 1966) depends to some extent on the accuracy of the calculation of the normalized structure factors, or E values, from the magnitudes of the observed structure factors F. The scaling is normally carried out by a Wilson plot procedure (Wilson, 1942) in which the logarithm of the reciprocal of the mean value of $|F|^2/\sum_{j=1}^{N} f_j^2$, N being the number of atoms in the unit cell and f_j the scattering factor of the jth atom, is evaluated as a function of $\sin^2\theta$, θ being the Bragg angle. In this process it is assumed that the mean temperature-factor coefficient of the atoms in the structure is isotropic, but often this is by no means true, and an improved method of calculating the E values is necessary. Wilson's method may be extended quite simply as follows. The data are first scaled to give an approximate set of normalized structure factors E' in the usual manner. The logarithm of the reciprocal of the mean value of $|E'|^2$ is then evaluated as a series of functions of the products of the Miller indices h^2, k^2, l^2, kl, hl and hk respectively and correction factors are determined from each as from a Wilson plot. In order to obtain satisfactory statistics for the functions of the cross products kl, hl and hk it may be necessary to take the mean values for ranges of the product. The slopes of the plots can readily be expressed in terms of the differences between the mean anisotropic and the mean isotropic thermal parameters if desired. The prescaling to E' values gives all the observations approximately equal weight, which is advisable for statistical reasons. In extreme cases a further improvement can be made by repeating the procedure after it has been once applied.

The thermal anisotropy corrections determined are the mean values for the whole unit cell, and do not compensate for all the anisotropy where the corresponding principal axes of the mean thermal motions within each of the asymmetric units of the structure are not parallel. Only those components of the mean anisotropic thermal motions which are parallel will be eliminated. Consequently the correction factor functions of kl, hl and hk for orthorhombic and higher symmetries will normally be negligibly different from unity. This will also hold for the correction factor functions of kl and hk for monoclinic symmetry, assuming that b is the unique axis.

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