HART, M. (1965). *Appl. Phys. Lett.* **7**, 96-98.
HART, M. (1966). *Z. Phys.* **189**, 269-291.
IOLIN, E. (1987). Preprint LAFI-102. Physics Institute of the Latvian Academy of Sciences, Salaspils, USSR.
IOLIN, E. & ENTIN, I. (1983). *Sov. Phys. JETP*, **58**, 985-989.
IOLIN, E., RAITMAN, E., KUVALDIN, B. & ZOLOTOYABKO, E. (1988). *Sov. Phys. JETP*, **67**, 989-997.
IOLIN, E., ZOLOTOYABKO, E., RAITMAN, E. & KUVALDIN, B. (1989). In Twelfth Eur. Crystallogr. Meet., Moscow. Collected Abstr., Vol. 3, p. 54.
IOLIN, E., ZOLOTOYABKO, E., RAITMAN, E., KUVALDIN, B. & GAVRILOV, V. (1986). *Sov. Phys. JETP*, **64**, 1267-1271.
KATO, N. (1964). *J. Phys. Soc. Jpn*, **19**, 67-77.
KATO, N. & ANDO, Y. (1966). *J. Phys. Soc. Jpn*, **21**, 964-968.
KOHLER, R., MOHLING, W. & PEIBST, H. (1974). *Phys. Status Solidi B*, **61**, 173-180.
LUKAS, P. & KULDA, J. (1989). *Phys. Status Solidi B*, **156**, 41-48.
OKKERSE, B. & PENNING, P. (1963). *Philips Res. Rep.* **18**, 82-94.
PENNING, P. & POLDER, D. (1961). *Philips Res. Rep.* **16**, 419-440.
TEWORTE, R. & BONSE, U. (1984). *Phys. Rev. B*, **29**, 2102-2108.

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Band-Structure Calculations and Structure-Factor Estimates of Cu – their Complementarity

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Abstract

Rather than an uncritical comparison of experimental and theoretical values, the various sets of structure-factor values of copper metal derived from experimental diffraction procedures are mutally compared as also are the various sets of theoretical values derived from band-structure calculations. This approach reveals the presence of outlier sets in each group and allows recognition of their condition before any attempt is made to intercompare the groups. Within the experimental group, the γ-ray values do not appear to sustain the absolute status originally claimed for them. Within the theoretical group, an inadequacy in defining the core contribution is indicated. The latter conclusion suggests that it is an inappropriate operation to make direct comparison between diffraction-sourced experimental values of structure factors and theoretical values from band-structure calculations. Instead, the latter should be used on a *complementary* basis with the full $(\sin\theta)/\lambda$ range of experimental values to establish the best core contribution. The minor valence-bond contribution to scattering, which is largely restricted to the low $(\sin\theta)/\lambda$ region, is most sensitively defined by reference to band-structure prediction of photo-emission spectral distribution. Attention is drawn to the possible significance of the form-factor curve *versus* $(\sin\theta)/\lambda$ being dependent on the unit-cell dimension.

Introduction

When there are many published sets of structure-factor values which have been determined by various experimental means and also those derived by theoretical calculations, comparison procedures to determine 'best' values require careful consideration. In many cases, experimental and theoretical values have been compared quite arbitrarily so that individual features intrinsic to experimental details or data reduction, on the one hand, or theoretical approximations and underlying assumptions, on the other, go unrecognized.

A more logical approach is to consider first the two groups separately. This provides a preliminary insight into each area, with respect to the spread of values, indications of consistency and trends with time. Only after such a procedure may it be appropriate to make a comparison between the two areas.

Indeed it becomes obvious on applying this approach to the published evidence for Cu that even a direct comparison of the two areas may not be wholly appropriate. Rather, from consideration of the bases of the numerical results from the two sources, it appears more likely that band-structure calculations and structure-factor values from diffraction techniques provide *complementary* (rather than comparable) views of the details of the total charge-density distributions in solids.

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Table 1. *List of theoretical structure-factor values for* Cu *derived from various band-structure calculations*

Values in square brackets are nominally appropriate to low-temperature, unbracketed to room temperature. The differences are based on the results of MacDonald *et al.* (1982).

h	*k*	*l*	1967 Arlinghaus	1968 Snow	1971 Wakoh & Yamashita	1980 Bagayoko *et al.*	1982 MacDonald *et al.*	1984 Ekardt *et al.*	Mean of last five
				[21.55]	[21.64]	[21.68]	[21.65]	[21.87]	[21.68]
1	1	1	21.54	21.63	21.72	21.76	21.73	21.95	21.76
				[20.33]	[20.39]	[20.35]	[20.32]	[20.61]	[20.42]
2	0	0	20.25	20.40	20.46	20.42	20.39	20.68	20.49
					[16.56]	[16.62]		[16.83]	[16.67]
2	2	0	16.39		16.63	16.67		16.90	16.74
					[14.58]	[14.70]		[14.88]	[14.72]
3	1	1	14.43		14.64	14.76		14.94	14.78
				[14.10]	[14.04]	[14.17]	[14.19]	[14.32]	[14.16]
2	2	2	13.90	14.16	14.10	14.23	14.25	14.38	14.22
					[12.28]	[12.42]		[12.56]	[12.42]
4	0	0	12.19		12.34	12.48		12.62	12.48
					[11.30]	[11.41]		[11.55]	[11.42]
3	3	1	11.25		11.35	11.46		11.60	11.47
					[11.03]	[11.13]		[11.27]	[11.14]
4	2	0	10.98		11.07	11.17		11.31	11.18
						[10.16]		[10.28]	[10.22]
4	2	2	10.05			10.20		10.32	10.26
						[9.58]	[9.63]	[9.69]	[9.63]
3	3	3	9.51			9.61	9.66	9.72	9.66
						[9.58]	[9.64]	[9.70]	[9.64]
5	1	1	9.51			9.61	9.67	9.73	9.67
			Room temperature $a = 3.6147$ Å non-self-consistent	Room temperature $\alpha = 2/3$	Temperature ?	Low temperature $a = 3.6032$ Å Schneider *et al.*	Low and room temperatures $a = 6.809$a.u. $a = 6.831$a.u.	Room temperature $a = 3.615$ Å	

Theoretical values

In Table 1, we have listed the more recent theoretical values of structure factors of Cu in copper metal derived from band-structure calculations. Six original sources are listed: Arlinghaus (1967); Snow (1968); Wakoh & Yamashita (1971); Bagayoko, Laurent, Singhal & Callaway (1980); MacDonald, Daams, Vosko & Koelling (1982); Ekardt, Fritsche & Noffke (1984). It should be noted that Arlinghaus recorded only the valence-electron contribution in his paper. The values tabulated in column 2 are from Schneider, Hansen & Kretschmer (1982) who added a core contribution to produce the listed structure factors. The values listed under Snow in the table came from Wood (1967), see also Temkin, Henrich & Raccah (1972). The values tabulated under Bagayoko *et al.* (1980) did not appear in the original paper but were supplied to Schneider privately and are listed in Schneider, Hansen & Kretschmer (1981).

As far as possible, we have identified the cell dimensions used by the author(s). Only MacDonald *et al.* (1982) have carried out calculations corresponding to two temperatures, low and room temperature, with the cell dimensions 6.809 a.u. (3.6032 Å) and 6.831 a.u. (3.6148 Å). The resultant differences in structure-factor values demonstrate that they are due not only to the effect of unit-cell dimensional change but also to the resultant alteration in the inner potential distribution. Apart from those of Arlinghaus, the calculations are all self-consistent. In the case of Snow, calculations for three values of the parameter α are recorded in Wood (1967), but only those for $\alpha = 2/3$ are presented here, the others being regarded as less appropriate according to general experience [see the review of Courths & Hüfner (1984)]. The calculations by Ekardt, Fritsche & Noffke (1984) were carried out for $a_0 = 3.615$ Å and correspond to room temperature. We have therefore, guided by the data of MacDonald *et al.* and assuming that the effect of small variations will be of comparable magnitude in the case of other calculations, modified the data identified as room temperature to correspond with a low-temperature calculation, shown bracketed in Table 1. It appears that the work of Ekardt, Fritsche & Noffke (1982) is, at least up to 1984 [see the review by Courths & Hüfner (1984)], the most detailed available, its predictions showing an excellent match with experimental measurements of various energy aspects, such as photo-emission spectra, and it appears to be regarded as a reference work in the field. Hence there appears to be no good reason to exclude it from serious consideration, for purely subjective reasons [as Tabbernor, Fox & Fisher (1990) did], despite its differences from previous studies.

The data in Table 1 are tabulated in chronological order because one might expect that predictions would become progressively more exact. As Mac-

Donald *et al.* (1982) observe and Courths & Hüfner (1984) concur 'As the fundamental variable in density functional theory, the charge density is certainly one feature (besides the band structure) that should be yielded correctly by the computation.' Hence, if this is indeed so, one would expect a trend in the band-structure calculations towards a limit set of structure-factor values. This appears to be contradicted by the results of Ekardt, Fritsche & Noffke (1984) which differ markedly from the four previous self-consistent sets. The difference between the Ekardt *et al.* values and the average of these four sets [see Fig. 1(*a*)] shows a gradual falling-off, a trend compatible with the inner core effect, *cf.* the corresponding plot of (say) 1.5% of the Cu^{2+} (Ar core) scattering curve, Fig. 1(*b*), or 1% of the Ga (Cu core) scattering curve, Fig. 1(*c*) (*International Tables for X-ray Crystallography*, 1974). In other words, a minor modification of the inner-core contribution (or equivalent modification of the pseudo-potential) of Ekardt *et al.* would bring the values more into line with the other four sets of calculations but would not significantly affect the photo-emission spectral distribution. This does not of course imply that the results of any one of the five self-consistent sets are superior to the others, but merely that they differ mainly in respect of the inner-core contributions.

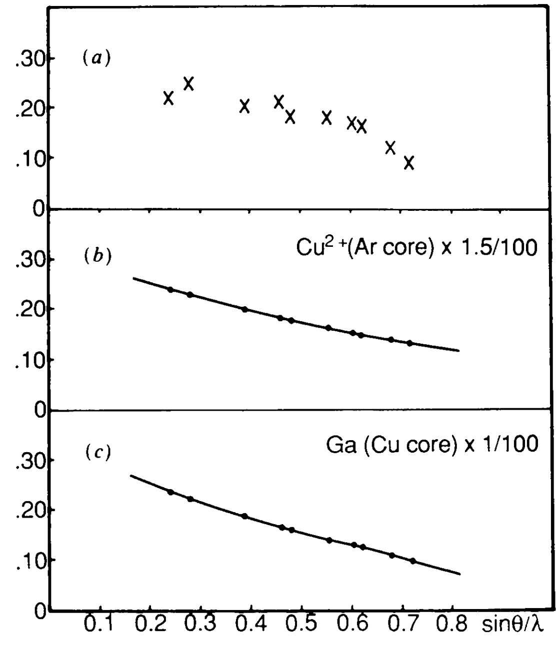

Fig. 1. Plots against (sin θ)/λ ($Å^{-1}$) at the reflections for Cu up to 333/511. (*a*) The differences between the values of Ekardt, Fritsche & Noffke (1984) and the average of the four earlier sources, Snow (1968), Wakoh & Yamashita (1971), Bagayoko *et al.* (1980) and MacDonald *et al.* (1982); (*b*) 1.5% of the argon core of Cu^{2+} from *International Tables for X-ray Crystallography* (1974); (*c*) 1% of the Cu core of Ga from *International Tables for X-ray Crystallography* (1974).

It is therefore necessary to recognize that while band-structure calculations in respect of photo-emission matters are sensitive to the fine detail of the potential distribution due to the valence electrons (and hence of the low-angle diffraction region), they are not so sensitive to the potential due to the inner-electron core (which dominates out to the high-angle diffraction scattering region), see Fig. 2 in Wood (1967). Theoretical structure-factor values derived from band-structure calculations are therefore not to be regarded as intrinsically absolute and so cannot properly be used as an absolute reference with which to test experimental structure factors fully. Rather, it is the case that experimental estimates of structure factors which are adequately established as absolute may be utilized in a *complementary* fashion to provide a test of the appropriateness of that component of the potential associated with the inner core.

So, given that we have no good reason to select at this stage any particular one of the five self-consistent band-structure sets as superior to the others in respect of their predictive capability for structure-factor values, we have perforce simply estimated the mean values of the five sets. These are given in the last column of Table 1.

Experimental values

In Table 2, we have listed experimental structure factors derived by procedures which are claimed to yield 'absolute' values, *i.e.* they do not depend on establishing their scale level by reference to any set of theoretical structure factors. This criterion largely excludes data derived from powder diffractometry methods.

Columns 2 to 5 correspond to values derived by measurement of absolute reflectivity in the Laue mode from imperfect crystals of constant thickness using γ-radiation of wavelength *ca* 0.03 Å. Those in column 2, reported by Schnieder (1976, 1977) and identified as Schneider I, were taken at room temperature, while those in column 4, reported by Schnieder, Hansen & Kretschmer (1981) and identified as Schneider II, were measured at 50 K. These two show close compatibility and have not subsequently been altered or modified in print by the author(s) so one may accept that they are based on the author's (authors') well considered decisions. The values for 220 given in columns 3 and 5 were derived from the experimental profiles published in Schneider I and II respectively; they resulted from re-interpretation by Mackenzie & Mathieson (1979) and (1984) respectively, the work having been done to demonstrate the role of extrapolation to achieve 'extinction-free' values of structure factors (Mathieson, 1979).

Columns 6 to 8 correspond to data from X-ray measurements with 'perfect' crystals and the application of dynamical theory, either in curve fitting to

Table 2. *List of experimental structure-factor values for* Cu, *nominally absolute, derived from diffraction experiments with γ-rays, X-rays and electrons*

			γ-rays				X-rays			Electron diffraction		
h	*k*	*l*	1976/7 Schneider I	1979 Mackenzie & Mathieson	1981 Schneider *et al.* II	1984 Mackenzie & Mathieson	1964 Jennings *et al.*	1979 Nittono *et al.*	1982 Takama & Sato	1980 Smart & Humphries	1988 Fox & Fisher	Mean of X-rays and electron diffraction
1	1	1	[21.22(5)]		21.51(5)		21.52(10)		21.80(6)	21.786	21.72(4)	21.71
2	0	0			20.22(4)				20.28(11)	20.454	20.45(4)	20.39
2	2	0	16.46(7)	16.76	16.45(5)	16.76		16.75(8)	16.75(8)	16.696	16.68(8)	16.72
3	1	1			14.54(4)				14.74(4)		14.76(7)	14.75
2	2	2	13.98(8)		14.07(5)		14.10(10)		14.36(6)			14.19
4	0	0			12.29(6)				12.46(6)			12.46
3	3	1			[11.30]							
4	2	0			11.02(6)							
4	2	2			10.08(6)							
3	3	3	9.53(5)		9.49(6)		9.41(10)					
5	1	1			9.53(6)							
			Room temperature		50 K				Room temperature			

Bragg reflections from extended-face crystals or in *Pendellösung* measurements. These are from Jennings, Chipman & DeMarco (1964), Nittono, Yamagishi & Nagakura (1979) and Takama & Sato (1982) respectively. Columns 9 and 10 refer to structure factors derived by electron diffraction measurements on essentially selected perfect areas of crystals, using the critical-voltage technique. The data are from Smart & Humphreys (1980) and Fox & Fisher [(1988), see Tabbernor, Fox & Fisher (1990)] respectively.

As with Table 1, we have identified the date of publication of the relevant work for consistency. In addition, certain practical details are relevant. Thus, in the case of the γ-ray data, Schneider I was carried out at the Institut Laue–Langevin using a γ-ray rectangular source of 0.2 mm cross section, *i.e.* with relatively high angular resolution. Of the measurements made at 11 different positions of one specimen of Cu only the data from nine were considered relevant by the author. Schneider II was carried out at the Hahn-Meitner-Institut with a rather different source. This was cylindrical, of diameter 2 mm. Because of the decreased resolution, the specimens of Cu had to be correspondingly distorted to a greater extent than in Schnieder I to ensure that the 'mosaic spread' of the specimens was each essentially larger than the angular resolution of the instrument. So the extinction level was necessarily lower than that in Schneider I. Three specimens of copper were used in this latter study.

If one excludes the results from Mackenzie & Mathieson (1979, 1984) from consideration for the moment, it would appear from inspection of Table 2, columns 2 and 4, that the structure factors from γ-ray measurement, as interpreted in Schneider I and II, are outliers when compared with the plateau of results associated with the dynamical techniques. In other words, excluding those outliers, the mean of the values for the dynamical results, recorded in column 11, probably corresponds to the 'best' experimental results, at least up to the present.

The value for 220 derived by Mackenzie & Mathieson appears to fall into the region indicated by the value in column 11 but we have not used it to establish the mean value of 220. Their operations were simply aimed at demonstrating the role of extrapolation and were not intended to establish a numerical value.

Discussion

In the theoretical estimates of structure factors using band-structure calculations, two features warrant comment. Firstly, the various sets of theoretical structure factors for Cu show no indication of homing onto a unique set of numerical values, a feature which would imply that, with refinement, this procedure can yield definitive, *i.e.* absolute, values. Rather, it is clear that the numerical values arrived at are sensitive to the potential selected to represent the inner core. In this respect, Spackman (1986) has shown that, even for Si with experimental data established to an accuracy of the order of 0.1%, for two sets of theoretical structure factors there is a systematic negative discrepancy relative to the experimental data while, in the case of a third theoretical set, the systematic discrepancy is positive. He points to inappropriate core contributions and the consequent impossibility of arriving at strong conclusions based on the match between theory and experiment.

Secondly, it appears rather important that, in future, the numerical contributions to the structure factors for both the valence electrons and the inner core be identified individually in a publication. To establish the most appropriate potential to represent the inner-core contribution, the evidence of the estimates of absolute measures of structure factors from diffraction experiments should be used in a complementary fashion.

It is also important that the unit-cell dimension(s) used should be specified so that the intercomparison of sets is facilitated. Indeed it would appear necessary to perform the calculations for a lower- and higher-temperature cell dimension since MacDonald *et al.* (1982) have demonstrated a difference of the order of 0.3 to 0.4%, which is larger than that due simply to the effect of expansion/contraction applied to the scattering curve assumed invariant. The results of MacDonald *et al.* are of fundamental concern in that they introduce a potential further complication, even if minor, in the extraction of absolute structure-factor values from diffraction measurements carried out at different temperatures. Up to the present, the standard assumption has been that the form factor does not change with temperature. Any change in unit-cell dimensions leads to a small change in $(\sin\theta)/\lambda$ and hence of structure factor, *e.g.* see columns 2 and 10 in Table 4 of Schnieder *et al.* (1981). This assumption allows measurements made at different temperatures to be reduced to an effective 0 K state by back-correction with thermal parameters. [Note that derivation of the thermal parameters from experimental data requires the use of a theoretical temperature-invariant scattering curve.] With a possible variation of scattering curve with unit-cell dimension, the process of extracting physically meaningful structure-factor values is rendered more complicated. This additional feature places even greater importance on absolute measurements and on the attainment of experimental accuracies of the order of 0.1% or better. It also stresses the need for the measurements to be made not merely at one temperature but also at two extremes at least. The theoretical structure-factor values derived from band-structure calculations are of course at 0 K, whatever the nominal temperature suggested by the cell parameters.

In respect of experimental estimates of Cu structure factors, a set of absolute values has evidently not been defined to an accuracy (not merely precision) of better than 0.5%. The feature which is most evident in the experimental arena relates to the difference between the γ-ray results of Schneider and his co-workers and the general plateau of results derived from dynamical procedures. This is a matter of some significance since the γ-ray data have been accepted as the experimental reference in the band-structure field, so much so that, in the review by Courths & Hüfner (1984), they state 'It appears that the calculation of Arlinghaus (1967), using the Chodorow potential, still gives the best agreement with experiment.' Obviously puzzled by this discrepancy between (*a*) the apparent close match with the early non-self-consistency calculation of Arlinghaus and (*b*) the apparent mismatch with the otherwise advanced studies of Ekardt, Fritsche & Noffke, (1984), they make the disconcerting statement that 'this again indicates that agreement with experiment alone does not seem a sufficiently accurate indication of the quality of a calculation'. The present discussion offers a resolution of this apparent discrepancy.

The problem has arisen because the data presented by Schneider and his colleagues were stated to be absolute structure-factor values and have been accepted as such. There appears to be little question but that the actual measures of γ-ray *intensity* diffracted in the Laue mode to establish the profile distribution are absolute since Schneider has frequently stressed the complementarity of the transmitted and diffracted beams, *e.g.* Schneider (1974, 1976) and Schneider, Pattison & Graf (1979). However, between the primary measurements and the derived structure factors, there are several steps which depend on assumptions made within the Darwin extinction theory, see Schneider (1976); in addition, there is a measure of uncertainty whether the Darwin transfer equations are appropriate to the physical state of 'imperfect' single crystals, *i.e.* whether they are to be regarded as fragmented or deformed, see Darwin (1914) and Kulda (1987).

Given the stated absolute status of the published γ-ray results, it does not appear appropriate at this juncture for a scale factor of 1.01 to be introduced by Tabbernor, Fox & Fisher (1990) (even with the private concurrence of Schneider) without a valid explanation for the variation. There are two data sets, Schneider I and II, currently consistent with one another although measured at different temperatures, so if set II is to be rescaled, does this apply also to set I? There are also certain other features which cannot readily be brushed aside. Thus, (i) a 1% scale change in structure-factor values corresponds to a 2% change in the original intensity scale of the profiles and (ii) a change in scale necessitates a re-analysis of thermal parameters.

On an earlier occasion, in establishing a more soundly based operational definition of extinction-free data (Mathieson, 1979), we were concerned to demonstrate the importance of extrapolation to 'zero intensity' and for this purpose Mackenzie & Mathieson (1979) utilized the 11 detailed profile curves published by Schneider (1976, 1977). To ensure authenticity, we carried through his process of analysis again and established that we arrived at essentially the same end points under his assumptions. However, we took the view that his correction procedure for extinction might not be complete and so, using the data for all 11 samples, rather than rejecting two, we extrapolated *both* the uncorrected and the corrected structure-factor values to zero extinction, an operation which yielded a single value for 220 shown in column 3 of Table 2. Later we utilized the data for 220 for one profile (all that was published) in Schneider, Hansen & Kretschmer (1981) to derive another value and, probably fortuitously, it fell on our original extrapolation line.

The result, 16.76, which we had derived originally in 1979 landed in the plateau region established by the dynamical procedures.

We consider that the application of an extrapolation procedure - by using a wedge-shaped single-crystal specimen or by tilting a parallel-sided specimen (Lawrence & Mathieson, 1976) or by estimating the level of extinction (first approximation) from the profile curve (Mackenzie & Mathieson, 1979) - is advisable to establish properly a zero-extinction limit. That there may also be a scale-factor modifier for the γ-ray data is a possibility which cannot be excluded. However, to authenticate this possibility, the applicability of the Darwin transfer relationship requires to be explored more closely, experimentally as well as theoretically.

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References

Arlinghaus, F. J. (1967). *Phys. Rev.* **153**, 743-750.

Bagayoko, D., Laurent, D. G., Singhal, S. P. & Callaway, J. (1980). *Phys. Lett. A*, **76**, 187-190.

Courths, R. & Hüfner, S. (1984). *Phys. Rep.* **112**, 53-171.

Darwin, C. G. (1914). *Philos. Mag.* **27**, 315-333.

Ekardt, H., Fritsche, L. & Noffke, J. (1984). *J. Phys. F*, **14**, 97-112.

Fox, A. G. & Fisher, R. M. (1988). Unpublished.

International Tables for X-ray Crystallography (1974). Vol. IV, Table 2.2*D*. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht).

Jennings, L. D., Chipman, D. R. & DeMarco, J. J. (1964). *Phys. Rev.* **135**, 1612-1615.

Kulda, J. (1987). *Acta Cryst.* **A43**, 167-173.

Lawrence, J. L. & Mathieson, A. McL. (1976). *Acta Cryst.* **A43**, 1002-1004.

MacDonald, A. H., Daams, J. M., Vosko, S. H. & Koelling, D. D. (1982). *Phys. Rev. B*, **25**, 713-725.

Mackenzie, J. K. & Mathieson, A. McL. (1979). *Acta Cryst.* **A35**, 45-50.

Mackenzie, J. K. & Mathieson, A. McL. (1984). *Aust. J. Phys.* **37**, 651-656.

Mathieson, A. McL. (1979). *Acta Cryst.* **A35**, 50-57.

Nittono, O., Yamagishi, H. & Nagakura, S. (1979). *J. Appl. Cryst.* **12**, 141-146.

Schneider, J. R. (1974). *J. Appl. Cryst.* **7**, 541-546.

Schneider, J. R. (1976). *J. Appl. Cryst.* **9**, 394-402.

Schneider, J. R. (1977). *Acta Cryst.* **A33**, 235-243.

Schneider, J. R., Hansen, N. K. & Kretschmer, H. (1981). *Acta Cryst.* **A37**, 711-722.

Schneider, J. R., Pattison, P. & Graf, H. A. (1979). *Nucl. Instrum. Methods*, **166**, 1-19.

Smart, D. J. & Humphreys, C. J. (1980). *Inst. Phys. Conf. Ser.* No. 52, 211-214.

Snow, E. C. (1968). *Phys. Rev.* **171**, 785-789.

Spackman, M. A. (1986). *Acta Cryst.* **A42**, 271-281.

Tabbernor, M. A., Fox, A. G. & Fisher, R. M. (1990). *Acta Cryst.* **A46**, 165-170.

Takama, T. & Sato, S. (1982). *Philos. Mag.* **B45**, 615-626.

Temkin, R. J., Henrich, V. E. & Raccah, P. M. (1972). *Phys. Rev. B*, **6**, 3572-3581.

Wakoh, S. & Yamashita, J. (1971). *J. Phys. Soc. Jpn*, **30**, 422-427.

Wood, J. H. (1967). In *Energy Bands in Metals and Alloys*, edited by L. H. Bennett & J. T. Waber, pp. 43-63. New York: Gordon & Breach.

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Extinction in Mixed Magnetic and Nuclear Reflections. A Study of the Magnetic Structure of $TbAlO_3$ using Neutron Polarimetry and Integrated Intensity Measurements

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Abstract

The magnetic structure of $TbAlO_3$ single crystals has been studied using zero-field neutron polarimetry and neutron integrated intensity measurements. The results of neither kind of measurements could be understood using a simple model for extinction in the rather good untwinned crystals that were used. To explain the results the Becker-Coppens extinction model [Becker & Coppens (1974). *Acta Cryst.* **A30**, 129-147] has been extended to the case where several magnetic domains occur within one block of the nuclear structure. The consequences for both the integrated intensities and the scattered polarizations have been calculated and it has been shown that the model provides a consistent interpretation of both

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