diffraction intensity, even if the sample is assumed to be composed of small crystallites so isolated that the interference among them may be neglected, the $\bar{v}$ function should be taken into account as the correction factor.

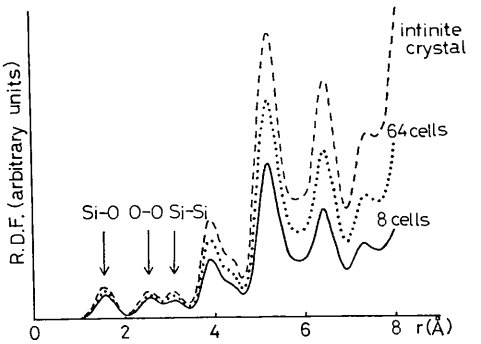

Fig. 2. The radial distribution function $4\pi r^2 D(r)$ for high-cristobalite crystals.

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Extinction in Sodium Fluoride

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Abstract

X-ray intensity measurements for a single crystal of sodium fluoride obtained at four wavelengths by Howard & Jones [*Acta Cryst.* (1977), A**33**, 776–783] have been re-analysed using both the Cooper–Rouse and Becker–Coppens extinction formalisms in order to study the wavelength dependence of the extinction in this crystal, since the original analysis was unable to account for the wavelength dependence of the intensities of the strong reflections. The results indicate that the crystal is intermediate between type I and type II in nature and are consistent with the wavelength dependence predicted by the theories, although the extinction is not large enough at the shorter wavelengths to provide a useful test of the validity of the theoretical wavelength dependence. The analysis also demonstrates some of the possible consequences of the use of unbalanced weighting schemes and of inappropriate models for the scattering factors.

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Introduction

In a recent study of the electron distribution in sodium fluoride, Howard & Jones (1977) carried out a series of accurate X-ray diffraction measurements on a single crystal of sodium fluoride at four different wavelengths, using Ag $K\alpha$, Mo $K\alpha$, Cu $K\alpha$ and Co $K\alpha$ radiations. These authors analysed their results using six sets of theoretical scattering factors and concluded that the best model for the electron distribution in sodium fluoride was given by the model 2 scattering factors of Aikala & Mansikka (1972) which take into account the overlap of the wave functions within the crystal environment as well as incorporating a contraction of the $2p$ orbital of the F^- ion compared with that in the free ion (the AM2 model).

In analysing their results Howard & Jones included a correction for extinction using the Zachariasen (1967) model, but also considered possible amendment of the angle dependence in line with that predicted by Becker

& Coppens (1974) for type II crystals, *i.e.* crystals in which the extinction is determined by the domain radius rather than the mosaic spread (see Zachariasen, 1967). An attempt was made to compare the observed wavelength dependence of the strongest reflections with the theoretical predictions for type I and type II secondary extinction for both the Zachariasen (1967) and the Becker & Coppens (1974) theories, but it was concluded that none of these predictions of the wavelength dependence was clearly applicable in this case. In view of similar conclusions about the wavelength dependence of extinction effects in other simple materials (Cooper & Rouse, 1976) one of these authors (Howard) suggested privately that further analysis of their data using various extinction models might be worthwhile. It is the purpose of this paper to present the results obtained from this analysis.

Method of analysis

Detailed experimental results were kindly supplied by Dr C. J. Howard and these were analysed using the Harwell *TAILS* computer program. This program allows for corrections for extinction to be based on either the Cooper–Rouse formalism (Cooper & Rouse, 1970) or the Becker–Coppens formalism (Becker & Coppens, 1974).

The program determines those values of the variable parameters which minimize the quantity

$$S = \sum_i w_i (I_{oi} - I_{ci})^2, \qquad (1)$$

where I_{oi} is the observed intensity, I_{ci} is the corresponding calculated intensity and w_i is the weight of the observation.

The experimental data were provided in the form of F_o^2 values obtained after correction of the individual measured intensities for absorption and Lorentz–polarization factors as well as values of F_o^2 averaged over equivalent reflections and corrected for thermal diffuse scattering ($\overline{F_o^2}$). For convenience the 'intensities' in equation (1) were therefore taken as:

$$I_o = \overline{F_o^2} \operatorname{cosec} 2\theta \qquad (2)$$

and

$$I_c = sy|F_c|^2 \operatorname{cosec} 2\theta, \qquad (3)$$

where s is the scale factor and y the extinction factor which has the form given in earlier papers (for example, Sakata, Cooper, Rouse & Willis, 1978). The calculated structure factors F_c included corrections for dispersion using the values given in *International Tables for X-ray Crystallography* (1974) for Ag $K\alpha$, Mo $K\alpha$ and Cu $K\alpha$ radiations and by Cromer (1976) for Co $K\alpha$ radiation. Values for the weights were derived from the standard deviations of $\overline{F_o^2}$.

Since the observed measurements were averaged over extensive sets of equivalent reflections they were analysed using an isotropic model for the extinction, approximating the crystal shape to that of a sphere of equal volume; this procedure has been used before in similar analyses (for example, Cooper & Rouse, 1973) and has proved to be a valid approximation provided that the extinction is not too severe. However, this approximation has to be taken into account in considering the results of the analysis.

As pointed out by Howard & Jones the choice of scattering factors is determined primarily by the very weak odd-index reflections and so is not a major consideration in the study of extinction effects. However, it is nevertheless possible for extinction effects to interact with the analysis of the electron distribution and we have therefore used two sets of scattering factors in order to investigate this. For convenience we have used the poly-detor ion (PD) values, as given in *International Tables for X-ray Crystallography* (1974), and the AM2 ion values given by Aikala & Mansikka (1972).

In order to investigate the wavelength dependence of the extinction effects we initially analysed the data obtained at each wavelength separately. Because of the difficulty of determining accurate values for both extinction parameters at the same time (see Cooper & Rouse, 1976) and the uncertainty in the choice of mosaic-spread function in the Becker–Coppens formalism the following models were used in the initial analysis: (1) Cooper–Rouse, (2) Becker–Coppens, type I, Gaussian (secondary); (3) Becker–Coppens, type I, Lorentzian (secondary); (4) Becker–Coppens, type II, (secondary); (5) Becker–Coppens, mixed-type Lorentzian (secondary); (6) Becker–Coppens, primary; (7) Becker–Coppens, general (mixed-type Lorentzian plus primary).

For each model the only parameters to be refined were the temperature factors B_{Na} and B_F and the extinction parameters r and/or g (r = domain radius, g = mosaic-spread parameter). As shown by Howard & Jones (1977) the difference between B_{Na} and B_F is not significant for the PD and AM2 electron distribution models; analysis with both B_{Na} and B_F varied gave values which were the same within the sum of their standard deviations. Consequently these parameters were constrained to be equal and a common value B refined. Because of the limited data available for the two longer wavelengths (Cu $K\alpha$ and Co $K\alpha$) the value of $\bar{B}$ was determined from the data measured with the two shorter wavelengths (Ag $K\alpha$ and Mo $K\alpha$) in each case. This parameter was then fixed at the value so obtained before subsequent analysis of all sets of data to determine the extinction parameters appropriate to each model.

Summary of results

During the course of this analysis it became clear that the sensitivity of the lowest-angle odd-index reflection (111), which had been measured very precisely, on the difference between the scattering factors for the two ions was severely distorting the analysis for the longer-wavelength data, because of the small number of reflections measured at these wavelengths and the relatively large weight given to the 111 reflection. Since the least-squares procedure will attempt to minimize any weighted differences resulting from inadequacies of the scattering factors this can result in incorrect values being obtained for the refined parameters, in this case the extinction parameters. As a consequence of this it was not possible to obtain a self-consistent set of extinction parameters for any one model using weights for all reflections based on the standard deviations of the observed data. The analysis was therefore repeated with the weight given to the 111 reflection at all wavelengths reduced to such an extent that it did not influence the determination of the extinction parameters.

The results of this analysis indicated that the crystal is intermediate between types I and II and that Gaussian and Lorentzian mosaic-spread functions are equally acceptable. Consequently the Becker–Coppens type I and type II models were unable to give self-consistent results for all wavelengths. Whilst the primary-extinction model could also provide excellent agreement for the individual wavelengths it gave values of the domain radius which were different for the different wavelengths. However, the remaining models, *i.e.* the Cooper–Rouse model and the Becker–Coppens mixed-type models with and without primary extinction, all gave values for the extinction parameters which were consistent, within their derived accuracy, with the predicted wavelength dependence. There was no significant difference between the agreement obtained for the two Becker–Coppens mixed-type models, but since some primary extinction is to be expected the general model including primary extinction is probably more acceptable.

A final analysis was therefore carried out for the Cooper–Rouse and Becker–Coppens general models with a single wavelength-independent model in each case, *i.e.* using values of the extinction parameters which did not change with the wavelength. The results of this analysis are summarized in Table 1 for the two sets of scattering factors (PD and AM2). The levels of agreement obtained are indicated by the values of the weighted discrepancy index,

$$R_w = \frac{\sum_i w_i |I_{oi} - I_{ci}|}{\sum_i w_i I_{oi}}, \qquad (4)$$

which are given in Table 2(*a*).

Table 1. *Results of the analysis using PD and AM2 scattering factors (SF)*

SF	Model Extinction*	$\bar{B}$ (Å)	$r(\times 10^{-5}$ cm)	g
PD	C–R	0·910 (5)	1·07 (†)	610 (60)
PD	B–C	0·912 (3)	2·50 (†)	980 (60)
AM2	C–R	0·915 (5)	1·00 (†)	620 (60)
AM2	B–C	0·918 (3)	2·30 (†)	1020 (60)

* C–R: Cooper–Rouse; B–C: Becker–Coppens.

† Because of the difficulty in obtaining reliable values for both r and g from data measured over a limited range of wavelengths the reliability of the extinction model has been estimated by fixing the value of r and deriving a value for the standard deviation of g only.

Table 2. *Weighted discrepancy index values* (%)

(*a*) With the 111 reflection suppressed

SF	Model Extinction	Ag $K\alpha$	Mo $K\alpha$	Cu $K\alpha$	Co $K\alpha$
PD	C–R	1·63	1·62	3·13	0·19
PD	B–C	1·65	1·64	3·18	0·62
AM2	C–R	1·19	1·28	1·37	0·23
AM2	B–C	1·19	1·25	1·94	0·60

(*b*) With the 111 reflection included

SF	Model Extinction	Ag $K\alpha$	Mo $K\alpha$	Cu $K\alpha$	Co $K\alpha$
PD	C–R	1·72	2·33	9·40	3·68
PD	B–C	1·73	2·34	9·93	4·19
AM2	C–R	1·18	1·30	1·06	0·93
AM2	B–C	1·18	1·29	1·55	0·59

In order to demonstrate the significance of the 111 reflection with respect to the choice of scattering factors we have also calculated the values of the weighted discrepancy index with the weight of the 111 reflection restored to the value derived from its standard deviation. These are given in Table 2(*b*). It can be seen from a comparison with Table 2(*a*) that this change in fact results in a significant increase in the value of R_w for all wavelengths for the PD model, but makes very little difference for the AM2 model, indicating a clear superiority of the AM2 model. This is also shown by significance tests on the R_w ratio for the two models (see Hamilton, 1965). Even with the 111 reflection suppressed the R_w ratio shows that the AM2 model is significantly better than the PD model at a significance level of 0·5%, for all wavelengths except Co $K\alpha$. However, since only four reflections were measured with Co $K\alpha$ radiation it is not surprising that the two models can only be distinguished at this wavelength if the 111 reflection is included. Inclusion of the 111 reflection results in a significant increase in the R_w ratio (R_{PD}/R_{AM2}) for all wavelengths.

There is very little difference in the agreement obtained with the two extinction formalisms, although

the Cooper–Rouse model gives slightly better agreement in most cases. We therefore give the final values of I_o, I_c and y for the Cooper–Rouse model only; these are listed in Tables 3 to 6. Examination of the F_o^2 values for individually measured reflections, as supplied by Dr Howard, indicated some variation in the values for equivalent reflections, consistent with a small degree of anisotropy in the extinction, but this was not of sufficient magnitude to cast doubt on the validity of the isotropic approximation used in the analysis.

Table 3. Ag $K\alpha$ *data* ($\lambda = 0{\cdot}56$ Å)

$h\,k\,l$	I_o	$\sigma(I_o)$	I_c(PD)	I_c(AM2)	y(AM2)
1 1 1	125.8	2.6	99.1	125.8	0.999
2 0 0	12711.0	55.5	12700.9	12603.4	0.923
2 2 0	5506.4	9.9	5553.1	5543.3	0.966
3 1 1	104.2	0.6	102.9	103.4	0.999
2 2 2	3003.9	4.3	2967.8	2963.7	0.982
4 0 0	1765.3	3.8	1769.8	1771.8	0.989
3 3 1	48.7	0.5	50.8	48.6	1.000
4 2 0	1130.6	1.9	1137.3	1139.6	0.993
4 2 2	770.8	1.2	771.6	772.9	0.995
3 3 3	21.4	0.6	23.4	22.5	1.000
5 1 1	22.1	0.4	23.4	22.5	1.000
4 4 0	396.4	1.9	400.8	400.7	0.998
5 3 1	10.8	0.4	11.1	10.8	1.000
4 4 2	304.5	1.0	302.7	303.0	0.998
6 0 0	306.9	1.8	302.7	303.0	0.998
6 2 0	234.4	1.0	233.8	234.0	0.999
5 3 3	5.4	0.5	5.5	5.4	1.000
6 2 2	182.6	1.0	184.2	184.2	0.999
4 4 4	147.3	1.7	147.7	147.5	0.999
5 5 1	1.8	0.4	2.8	2.9	1.000
7 1 1	2.9	0.5	2.8	2.9	1.000
6 4 0	118.7	0.7	120.0	119.9	0.999
6 4 2	99.0	0.6	99.0	98.9	0.999
5 5 3	1.4	0.4	1.5	1.5	1.000
7 3 1	1.1	0.3	1.5	1.5	1.000
8 0 0	70.0	0.9	69.3	69.4	1.000
7 3 3	0.6	0.4	0.8	0.8	1.000
6 4 4	59.1	0.6	58.8	58.9	1.000
8 2 0	58.0	0.6	58.8	58.9	1.000
6 6 0	49.0	1.4	50.3	50.4	1.000
8 2 2	51.3	1.1	50.3	50.4	1.000
6 6 2	43.6	0.9	43.3	43.3	1.000
8 4 0	38.9	0.9	37.5	37.5	1.000
8 4 2	31.5	0.6	32.6	32.5	1.000
6 6 4	28.5	1.0	28.4	28.4	1.000
8 4 4	23.7	0.8	21.7	21.7	1.000
8 6 0	19.5	0.8	19.0	19.0	1.000
10 0 0	19.0	1.6	19.0	19.0	1.000

Table 4. Mo $K\alpha$ *data* ($\lambda = 0{\cdot}71$ Å)

$h\,k\,l$	I_o	$\sigma(I_o)$	I_c(PD)	I_c(AM2)	y(AM2)
1 1 1	98.3	0.8	78.9	99.9	0.999
2 0 0	9599.3	62.0	9559.8	9483.4	0.886
2 2 0	4294.9	6.8	4298.1	4286.4	0.948
3 1 1	83.0	0.4	82.3	82.5	0.999
2 2 2	2309.6	4.8	2329.4	2323.6	0.972
4 0 0	1378.7	5.5	1402.6	1402.4	0.983
3 3 1	40.4	0.3	41.1	39.3	1.000
4 2 0	903.5	1.6	908.5	909.2	0.989
4 2 2	626.0	1.5	620.8	621.1	0.992
3 3 3	18.8	0.5	19.2	18.5	1.000
5 1 1	18.4	0.3	19.2	18.5	1.000
4 4 0	328.3	1.5	327.0	326.5	0.996
5 3 1	8.9	0.2	9.3	9.0	1.000
4 4 2	247.8	1.1	248.7	248.6	0.997
6 0 0	251.7	2.0	248.7	248.6	0.997
6 2 0	194.0	1.1	193.4	193.4	0.998
5 3 3	4.6	0.2	4.7	4.6	1.000
6 2 2	153.9	0.9	153.5	153.4	0.998
4 4 4	123.5	1.3	124.0	123.7	0.998
5 5 1	2.6	0.4	2.4	2.5	1.000
7 1 1	2.6	0.4	2.4	2.5	1.000
6 4 0	102.5	0.8	101.7	101.4	0.999
6 4 2	86.2	0.6	84.5	84.3	0.999
5 5 3	1.1	0.3	1.3	1.4	1.000
7 3 1	1.3	0.2	1.3	1.4	1.000
8 0 0	62.6	1.2	60.3	60.3	0.999
7 3 3	1.2	0.4	0.8	0.8	1.000
6 4 4	53.5	0.7	51.6	51.7	0.999
8 2 0	52.3	0.6	51.6	51.7	0.999

Table 5. Cu $K\alpha$ *data* ($\lambda = 1{\cdot}54$ Å)

$h\,k\,l$	I_o	$\sigma(I_o)$	I_c(PD)	I_c(AM2)	y(AM2)
1 1 1	51·2	0·3	41·8	50·9	0·995
2 0 0	3738·8	37·0	3694·3	3596·9	0·676
2 2 0	1963·1	9·0	2006·6	1953·4	0·819
3 1 1	46·9	0·3	47·6	46·3	0·995
2 2 2	1174·5	9·8	1234·0	1199·1	0·886
4 0 0	778·8	6·3	832·2	809·5	0·923
3 3 1	25·9	0·3	28·3	26·3	0·997

Table 6. Co $K\alpha$ *data* ($\lambda = 1{\cdot}79$ Å)

$h\,k\,l$	I_o	$\sigma(I_o)$	I_c(PD)	I_c(Am2)	y(AM2)
1 1 1	44·3	0·6	37·3	45·6	0·994
2 0 0	2893·8	74·8	3032·1	2971·4	0·626
2 2 0	1706·8	24·9	1742·0	1707·0	0·776
3 1 1	44·4	0·3	45·1	44·1	0·994

Discussion

The present analysis leads to a number of important conclusions. Perhaps the most significant of these is the observation that an inappropriate model can lead to incorrect values for refined parameters as a result of the least-squares procedure minimizing differences between observed and calculated quantities which arise from the inadequacy of the model and not from incorrect values of the refined parameters. In the present case a choice of inappropriate scattering factors can lead to incorrect values for the extinction parameters, so that inadequacies in the scattering-factor model can be concealed and inconsistent extinction parameters obtained from data measured at different wavelengths. This is particularly true for data sets which contain only a few measurements.

The analysis also illustrates the importance which the weights given to the measurements can have in this type of study. Since the object of the study was to determine the best model for the electron distribution in sodium fluoride considerable care was taken to measure the weak odd-index reflections, which are extremely sensitive to the difference between the scattering factors for the two ions, with relatively high accuracy. Using weights derived from the standard deviations of the measurements this therefore leads to an unbalanced weighting scheme with the weaker odd-index reflections playing a relatively more important role in the least-squares analysis than the strong even-index reflections. This can in fact lead to artificial fitting to an incorrect model, as discussed above, and so conceal real differ-

ences between models. In the present case this can be overcome by suppressing the effect of the 111 reflection, which is by far the most sensitive to the scattering-factor model, on the deviation of the refined parameters and only taking its correct weight into account in determining the overall fit obtained for each model.

The unbalanced weighting scheme also accounts for the uselessness of unweighted discrepancy indices, which was noted by Howard & Jones (1977), since it is clearly possible for a decrease in the weighted discrepancy index to be accompanied by an increase in the unweighted discrepancy index. Indeed, Ford & Rollett (1970) have shown that there are theoretical grounds for believing that ratios of unweighted 'R factors' will in general be surprisingly more variable than ratios of properly weighted least-squares minimization factors and therefore pointed out the need for weighting in statistical 'R-factor' tests. Significance tests used in the present analysis, based on the weighted discrepancy index,† confirmed the significantly better fit obtained for the AM2 model compared to the PD model. It is also interesting to note that this difference is still significant even when the most sensitive reflection (111) is omitted from the analysis although, as can be seen from Tables 3 to 6, the individual differences are quite small except for the 111 reflection. These results therefore indicate quite conclusively a definite, statistically significant superiority of the AM2 model and the power of the least-squares method when appropriate factors are taken into account.

Although the different data sets were analysed separately in order to determine any deviation of the wavelength dependence of the extinction effects from that expected theoretically, the values of the extinction parameters so obtained were in fact consistent with the wavelength dependence predicted by the models used and it was therefore possible to use the same values for the extinction parameters at each wavelength in the final analysis. This provides further evidence that existing extinction theories are perfectly adequate for this type of experiment if the level of extinction is not too large (in the present case the reduction in intensity due to extinction, even at the longest wavelength, is never more than 40% and is less than 12% at the two shortest wavelengths). For example, the values of the effective domain radius (r^*) obtained for a Cooper–Rouse secondary-extinction model and AM2 scattering factors are 0·32 (3), 0·41 (3), 0·62 (4) and 0·79 (2) × 10^{-5} cm for Ag $K\alpha$, Mo $K\alpha$, Cu $K\alpha$ and Co $K\alpha$ respectively, which are consistent with a linear dependence of $1/r^{*2}$ on $1/\lambda^2$. However, because of the relatively large standard deviations of the shorter-wavelength values they are nevertheless not inconsistent with a curve of the type found for materials such as SrF_2 (Cooper & Rouse, 1976) and these results therefore do not provide a useful test of the exact wavelength dependence of extinction effects.

X-ray measurements on a small spherical crystal of sodium fluoride using Mo $K\alpha$ radiation have also been reported by Sharma (1974*a*,*b*). Since only one wavelength was used it is not possible to deduce the extinction type directly from these measurements but it is interesting to note that, using the original Zachariasen (1967) extinction theory, Sharma obtained a value for r^* of 0·56 (4) × 10^{-5} cm which is in quite close agreement with the values obtained in the present analysis. It is probable therefore that the two crystals have similar extinction properties. Since the present analysis indicates that the Howard & Jones crystal is intermediate between type I and type II, Sharma's conclusion that his crystal is type II, based simply on a similar result inferred for a lithium fluoride crystal, must be in doubt.

I am grateful to Drs C. J. Howard and R. D. G. Jones for suggesting this analysis and for supplying details of their experimental results and a copy of their paper (Howard & Jones, 1977) prior to its publication.

† The use of the statistically more appropriate factor $R'_w = [\sum_i w_i(I_{oi} - I_{ci})^2/\sum_i w_i I_{oi}^2]^{1/2}$ leads, in the present case, to exactly the same conclusions.

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