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An X-ray determination of the thermal parameters for LiF.

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X-ray data obtained from LiF are fitted using scattering factors from *International Tables for X-ray Crystallography* [Vol. III (1968), p. 202. Birmingham: Kynoch Press] with thermal parameters 1·011 and 0·683 Å^2 for lithium and fluoride ions respectively, and assuming no extinction. With scattering factors from Dirac–Slater wave functions the fit to the data is significantly poorer.

In recent determinations of the thermal parameters for LiF, Zachariasen (1968) has obtained results from a crystal affected by type I extinction, while Killean, Lawrence & Sharma (1972) have used a crystal which exhibited extinction of type II. In the course of testing a solid-state detector on the AAEC diffractometer (Khadake, 1973) we have obtained a small crystal of LiF which seems very nearly extinction free.

A piece was cut from the LiF crystal in a Unicam S.30 monochromator and ground into a sphere of radius 0·14 mm. Integrated intensities were collected on the four-circle diffractometer in stationary-counter moving-crystal scans. The radiation selected was molybdenum $K\alpha$ (λ = 0·7107 Å). No attenuators were used, but on-line corrections were made for losses in the counting system, and in any case the count rate never exceeded 10 000 counts/s. Measurements were made of 285 reflexions with values of θ up to 40°, this limit being imposed by the bulk of the detector cryostat.

In view of the small dimensions of the crystal no absorption corrections were made. Nor were corrections made for thermal diffuse scattering. Equivalent reflexions were checked for consistency; the 200 reflexions were found wanting and were not included in the least-squares analysis. The observed structure factors and their standard deviations were derived from the mean and standard error (in the mean) of the intensities of equivalent reflexions. Finally, a least-squares minimization was carried out in which two thermal parameters and the scale factor were determined. The scattering factors used were those for Li^+ and F^- as given in *International Tables for X-ray Crystallography* (1968), while the dispersion corrections were neglected. The results for the thermal parameters (with standard deviations indicated) were

$$B_{Li} = 1{\cdot}011\ (6)\ \text{Å}^2, \quad B_F = 0{\cdot}683\ (3)\ \text{Å}^2,$$

which are in reasonable agreement with previous published values (Witte & Wölfel, 1958; Zachariasen, 1968; Linkoaho & Merisalo, 1970; Killean *et al.*, 1972). The observed and calculated structure factors are shown in Table 1 together with the standard deviations of the observed structure factors. The value of the index R is 0·003.

Table 1. *Observed and calculated structure factors*

hkl	$\sigma(F_o)$	F_o	F_c
200	0·11	29·94	29·73
220	0·03	21·22	21·08
222	0·02	16·18	16·18
400	0·03	13·10	13·05
420	0·01	10·88	10·89
422	0·01	9·30	9·32
440	0·02	7·16	7·18
442	0·01	6·40	6·41
600	0·02	6·41	6·41
620	0·02	5·78	5·79
622	0·01	5·29	5·27
444	0·02	4·82	4·82
640	0·02	4·44	4·43
111	0·03	19·79	19·79
311	0·01	8·85	8·87
331	0·01	5·79	5·81
333	0·03	4·51	4·52
511	0·01	4·51	4·52
531	0·01	3·84	3·83
533	0·01	3·38	3·37
551	0·01	3·04	3·02

In a further refinement an isotropic extinction parameter was included with the other three parameters. This refinement gave the same values (within the statistics) of the thermal parameters, and confirmed that extinction effects were negligible.

Cromer & Waber (1965) have computed alternative scattering factors for Li^+ and F^-. A three-parameter refinement of the data with these scattering factors gave rather different thermal parameters but a relatively poor fit, with

$$B_{Li} = 0{\cdot}90\ (1)\ \text{Å}^2, \quad B_F = 0{\cdot}67\ (1)\ \text{Å}^2$$

and the index R at 0·012. An isotropic extinction parameter refined to an (unacceptable) negative value. The data clearly favour the scattering factors from *International Tables*. However this fact must be interpreted with caution, particularly on account of possible deviations from the purely ionic bonding on which the use of these scattering factors is based.

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