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The determination of the unit-cell dimensions of non-cubic substances.

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A graphical extrapolation method for the accurate determination of the unit-cell dimensions of cubic substances from high-angle reflexions on a powder photograph was described by Bradley & Jay (1932). More recently Nelson & Riley (1945) and Taylor & Sinclair (1945) have described graphical plots which are linear down to quite small values of θ. These often permit of extension to non-cubic substances, since a number of reflexions from each of the planes 100, 010 and 001 may occur within the range of θ values which is covered. Alternatively, analytical methods, such as described by Cohen (1935), are available for non-cubic substances. The present note describes a graphical method of procedure which can be used with non-cubic substances when only one line in a main zone is suitable for accurate measurement.

The principle of this method is that the substance under investigation is mixed with some cubic substance giving a reasonable number of lines which do not overlap those of the first substance. Extrapolation of the a values calculated for each of the cubic lines permits the determination of a correction factor for any position on the film. This correction can then be applied to the measured spacing for any line of the non-cubic substance, since, using the mixture technique, the effect of absorption and camera eccentricity will be the same, at a given θ value, for both substances. Using a camera calibrated by direct measurement it is not necessary to assume any knowledge of the spacing of the mixture substance.

Table 1 shows the results of measurements made to determine the cell dimensions of BeO, using NaCl as the cubic substance, in a 19 cm. camera. Spacings are given in Ångström units, assuming λ for Cu $K\alpha_1 = 1{\cdot}54050$ A.

Table 1. *Results of measurements on cell dimensions of* BeO

Reflecting plane		θ for	Interplanar spacing	a for NaCl	Correction	Corrected spacing
NaCl	BeO	Cu $K\alpha_1$	in A.	in A.	factor	in A.
440	—	50·638°	0·99624	5·6356	—	—
531	—	53·951°	0·95267	5·6361	—	—
—	$20\bar{2}3$	57·678°	0·91148	—	—	—
620	—	59·791°	0·89129	5·6370	—	—
—	$21\bar{3}0$	60·766°	0·88267	—	1·00042	0·88304
—	$21\bar{3}1$	62·885°	0·86536	—	—	—
622	—	64·998°	0·84989	5·6375	—	—
—	$10\bar{1}5$	69·991°	0·81973	—	1·00022	0·81992
444	—	71·163°	0·81384	5·6384	—	—
117	—	77·293°	0·78959	5·6388	—	—
460	—	80·039°	0·78204	5·6394	—	—
—	$30\bar{3}0$	81·516°	0·77877	—	1·00005	0·77881

The a values for NaCl were plotted against

$$\frac{1}{2}\left(\frac{\cos^2\theta}{\sin\theta}+\frac{\cos^2\theta}{\theta}\right)$$

and from the extrapolated value the correction factors shown were determined in turn for each of the BeO lines $10\bar{1}5$, $21\bar{3}0$ and $30\bar{3}0$. From the last two reflexions independent values of a for BeO were obtained and these values, for four independent pairs of films, are given in the first two lines of Table 2.

Table 2. *Cell dimensions of* BeO

(Values in A.)

Film numbers	1	2	3	4
a from $21\bar{3}0$ and NaCl	2·6977	2·6978	2·6978	2·6975
a from $30\bar{3}0$ and NaCl	2·6979	2·6979	2·6977	2·6977
a from extrapolation of $21\bar{3}0$, $30\bar{3}0$	2·6978	2·6978	2·6977	2·6977
c from $10\bar{1}5$	4·3778	4·3780	4·3780	4·3777

In this particular case it is also possible to do a two-point zone extrapolation of a values from $21\bar{3}0$ and $30\bar{3}0$, and the extrapolated value of a is given in line 3 of Table 2 for comparison. It is pointed out that the large θ value for $30\bar{3}0$ makes this plane largely determine the extrapolation.

The value of the c dimension for BeO, given in Table 2, is determined from the $10\bar{1}5$ reflexion, as there is no suitable $000l$ reflexion. If d is the $10\bar{1}5$ spacing, it can be shown that $\frac{25}{c^2}=\frac{1}{d^2}-\frac{4}{3a^2}$, and the two terms on the right-hand side of this expression are in the ratio of about 8 : 1. Hence the previously determined value of a can be used to obtain what is largely an independent value of c.

All the above measurements were made at a temperature of 21° C., maintained to $\pm\frac{1}{2}$° C. during the exposure.

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