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A one-dimensional integrating method for estimating the intensities on upper-level equi-inclination Weissenberg photographs.

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The difficulty of estimating visually the intensities of reflexions recorded on upper-level Weissenberg photographs, due to the variations in the size of the spots and the inadequacy of the eye as an integrating device, has long been recognized. Methods for estimating and correcting the intensities have been proposed by Kaan & Cole (1949) and Scouloudi (1953); these are not entirely satisfactory, particularly when the axis of rotation does not coincide with a symmetry axis of the crystal. Phillips (1954) has proposed a method whereby the extended spots alone are corrected, using analytical expressions for the dimensions of the spots.

The two-dimensional integrating method of Wiebenga (1947) and Wiebenga & Smits (1950) is not available in most laboratories and, in extreme cases, may not be sufficiently flexible to cover the wide range of spot lengths encountered in upper-level photographs. In equi-inclination photographs the distortion of the spots is restricted to a direction parallel to the camera axis, and integration in this direction only would make the visual estimation of the intensities easier and more reliable. This can be achieved by using a multiple-exposure technique. Between successive exposures the camera is translated parallel to its axis, independent of the carriage, by a small distance. The incremental translation should be a reasonably small fraction of the length of the shortest spot (say one-fifth) and the total translation should be greater than the length of the longest spot. The density estimated at the centre of each integrated spot is then directly related to the required integrated intensity and can be estimated visually with comparative ease. Most cameras are equipped with a vernier scale which enables the translations to be made with sufficient accuracy since the minimum translation likely to be required is about 0·1 mm. Fig. 1 shows two photographs of the *hkl*8 reflexions of $CaS_2O_6.4H_2O$, $\mu = 21{\cdot}7°$, for

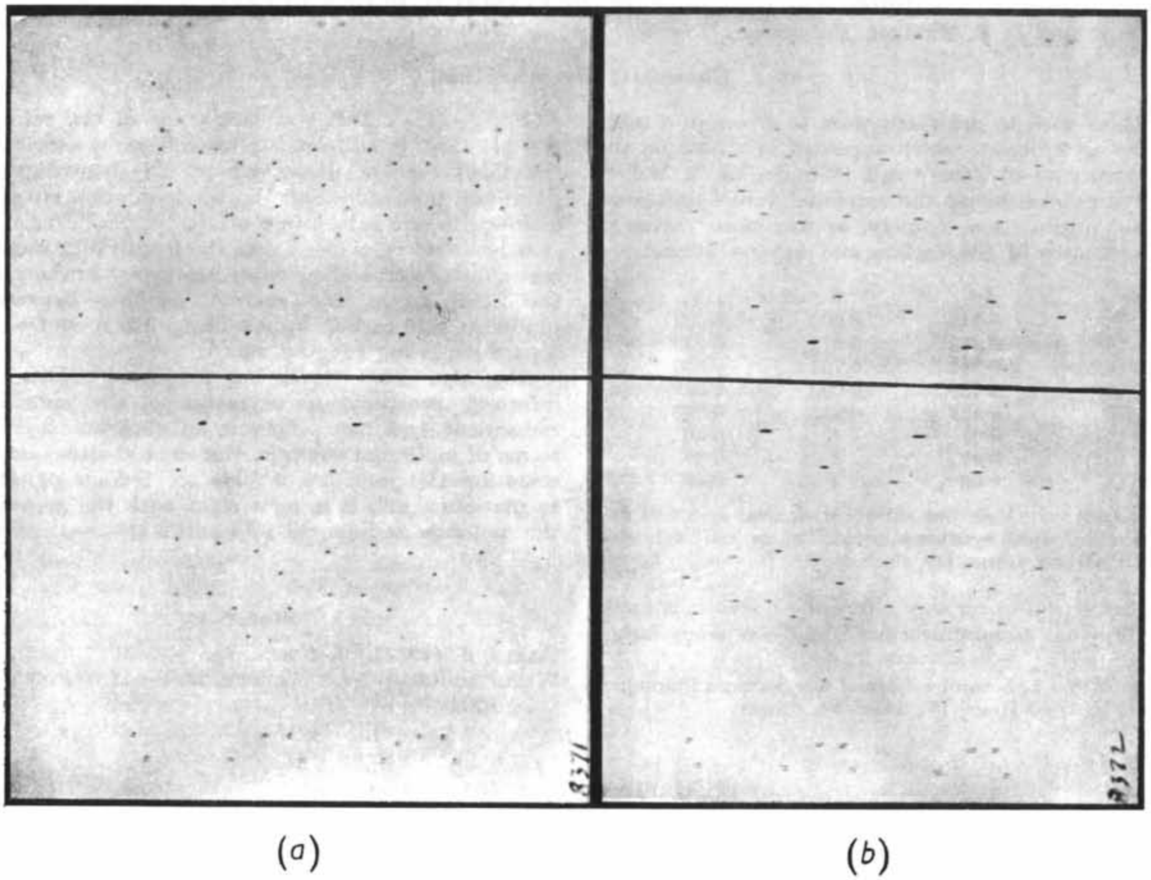

Fig. 1. (*a*) Ordinary upper-level equi-inclination Weissenberg photograph of the *hkl*8 reflexions of $CaS_2O_6.4H_2O$, $\mu = 21{\cdot}7°$. Exposure 2·0 hr. (*b*) Multiple photograph of the same level consisting of 16 exposures of 10 min. each with incremental translation 0·1 mm. (The axis of rotation is a symmetry axis.)

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comparison. It appears that the uniform central part of the integrated spot is quite long, indicating that the original reflexions had a flat-topped distribution and that the total translation could have been reduced. Table 1 gives the visually estimated intensities of the reflexions on both halves of the two films. (The c axis of calcium dithionate tetrahydrate is an axis of symmetry.)

Table 1

	Non-integrated		Integrated	
θ (°)	Top	Bottom	Top	Bottom
16	56	22	20	20
20	40	20	14	12
30	6	4	2	3
32	28	16	9	10
34	22	12	7	7
40	28	22	10	16
42	11	7	4	3
44	18	11	5	5
50	11	8	4	5
70	20	14	8	7
71	8	6	4	3

Although the method can be used directly by adjusting the camera between exposures it would not be difficult to devise a simple mechanism which could be fitted to existing cameras to give the camera the required incremental translation at the end of each traverse.

The proposal of Whittaker (1953) to uncouple the oscillation and translation motions of the Weissenberg goniometer, and allow the camera to oscillate (through a small distance) parallel to the axis independent of the crystal oscillation, would achieve the same result in providing a linear integration of the spots. The integrated spots would be short streaks perpendicular to the single layer line and some of the advantages of the Weissenberg photograph would be lost.

I am grateful to Dr D. C. Phillips for giving me access to the manuscript of his theoretical work on this subject, prior to publication, which led to this method.

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Re-investigation of the crystal structure of CsI_3.

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A recent X-ray examination of Cs_2I_8 showed this compound to contain I_3^- ions in weak interaction with I_2 molecules (Havinga, Boswijk & Wiebenga, 1954). It was thought interesting to compare the dimensions of the I_3^- ion constituent in Cs_2I_8 with those of the I_3^- ion in tri-iodides. It appeared, however, that no data on the tri-iodides were available which could be expected to be sufficiently accurate for this purpose. For CsI_3 only unit-cell dimensions and space group have been determined (Bozorth & Pauling, 1925). The structure of NH_4I_3, isomorphous with CsI_3, was investigated by Mooney (1935). The I_3^- ion was reported to be linear to within 3°; the two I–I distances in the ion were found to be different (2·82 and 3·10 Å). According to the author herself, however, this difference might be fictitious, the analysis being based only on qualitatively estimated intensities, making use of trial-and-error methods. It was therefore decided to re-investigate the crystal structure of one of the tri-iodides. CsI_3 was chosen because, with this compound, an accurate determination of both anion and cation coordinates should be possible.

Making use of NaCl powder diagrams superimposed on rotation photographs, the lattice constants were determined:

$$a = 6{\cdot}86,\ b = 9{\cdot}98,\ c = 11{\cdot}09\ \text{Å}\ (\lambda(\text{Cu}\ K\alpha) = 1{\cdot}5418\ \text{Å})\ .$$

Values for these unit-cell dimensions, derived independently from various photographs made in different cameras, showed a standard deviation of approximately 0·01 Å. The lattice constants are in fair agreement with those reported by Bozorth & Pauling (1925).

The characteristic absences ($h0l$ for l odd and $hk0$ for

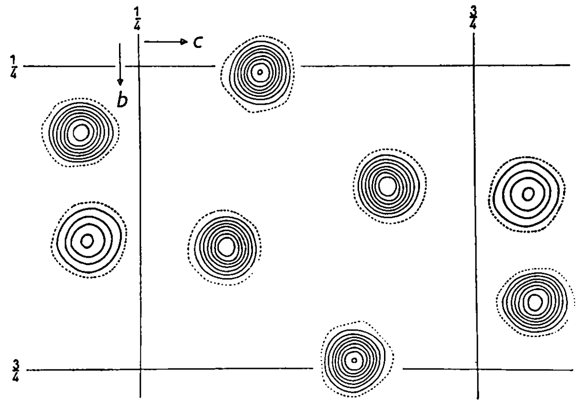

Fig. 1. Electron-density projection along [100]. Contour intervals: 10 e.Å^{-2} (with 10-electron line dotted) except on cesium ions (heavy lines), where the interval is 20 e.Å^{-2}.