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

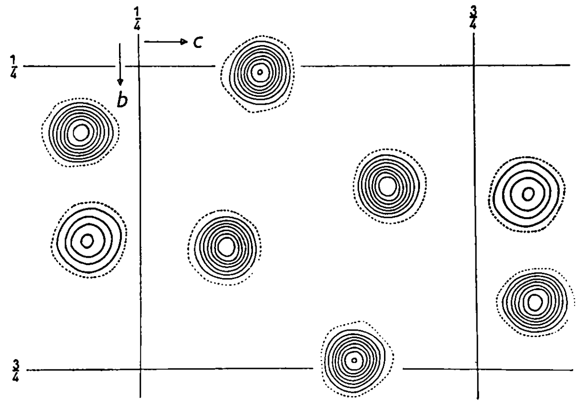

Fig. 1. Electron-density projection along [100]. Contour intervals: 10 e.Å^{-2} (with 10-electron line dotted) except on cesium ions (heavy lines), where the interval is 20 e.Å^{-2}.

$h+k$ odd) indicate the space group *Pmcn*. Since, even on photographs of very long exposure, no exceptions to these absences were observed, and since the unit cell contains only 4 molecules, all atoms must have x coordinates $x = \frac{1}{4}$ and $\frac{3}{4}$.

This means that the remaining coordinates can be obtained from the [001] projection only.

Ordinary and integrated Weissenberg photographs were made about the a axis of a roughly isodimensional crystal, approximately 0·0075 cm. in diameter, using both Mo and Cu radiation. The intensities of 98 independent $0kl$ reflexions were obtained and corrected for absorption and Lorentz- and polarization factors. Starting from the coordinates as given by Mooney for NH_4I_3, the [100] projection was refined by a series of consecutive Fourier syntheses. The final contour map is shown in Fig. 1. The coordinates were corrected for the finite-series effect by the 'back-shift' method. They are listed below.

	y	z
Cs	0·4640	0·8259
I_1	0·3598	0·1641
I_2	0·5500	0·3778
I_3	0·7367	0·5697

With these coordinates the value of $R = \Sigma\{|F_o| - |F_c|\} \div \Sigma|F_o|$ is 0·105, the summations being taken over all observed reflexions. In the calculated structure factors an isotropic temperature factor $\exp[-1{\cdot}00\,(\sin\theta/\lambda)^2]$ has been introduced.*

The dimensions of the I_3^- ion may be read from Table 1; those for NH_4I_3 and Cs_2I_8 have been included for comparison. The bond lengths and angles observed in the early analysis of NH_4I_3 are in surprisingly good agreement with our results.

Table 1. *Dimensions of* I_3^- *ion*

	NH_4I_3	CsI_3	Cs_2I_8†
I_1–I_2 (Å)	2·82	$2{\cdot}83_0(0{\cdot}01_5)$	$2{\cdot}82_5(0{\cdot}01_8)$
I_2–I_3 (Å)	3·10	$3{\cdot}03_5(0{\cdot}01_5)$	$3{\cdot}00_1(0{\cdot}01_8)$
I_1–I_2–I_3 (°)	177	176·3(0·5)	176·8(0·8)

Values in parentheses represent the standard deviations calculated with Cruickshank's formula (Cruickshank, 1949); an estimated allowance for imperfect correction for finite series was included.

From the data given in Table 1 it is beyond doubt that in CsI_3 and Cs_2I_8 the I_3^- ion has an asymmetric, non-linear structure. It is difficult to judge, however, whether the rather small deviation from linearity is a property of the 'free' ion itself or may be explained by the influence of intermolecular forces in the crystals.

Fig. 2 gives a survey of the interatomic distances. The Cs–I distances indicated amount to 3·92 Å on an average; they are comparable with those observed in CsI (3·95 Å) and Cs_2I_8 (3·85–4·07 Å). The short intermolecular I–I distances (4·34 Å on an average) may be compared with those in NH_4I_3 (4·28 Å).

Prof. E. H. Wiebenga suggested the subject of this investigation; his help in preparing the manuscript is gratefully acknowledged. Our thanks are also due to Theodorus Niemeijer N. V. for putting their I. B. M. equipment at our disposal and to the Netherlands Organization for Pure Research (Z. W. O.) for their indirect support.

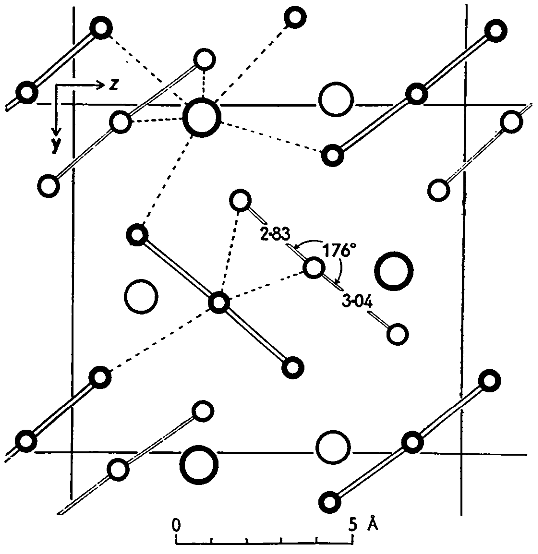

Fig. 2. Projection of the CsI_3 structure on (100). Large and small circles represent cesium and iodine respectively. Heavy lines and circles indicate atoms and bonds in the plane of the drawing, the others in planes 3·43 Å above and below. Intermolecular distances are indicated by dotted lines.

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* A table of observed and calculated structure factors has been deposited as Document No. 4431, with the ADI Auxiliary Publications Project, Photoduplication Service, Library of Congress, Washington 25, D.C., U.S.A. A copy may be secured by citing the Document number and by remitting $1·25 for photoprints, or $1·25 for 35 mm. microfilm. Advance payment is required. Make cheques or money orders payable to: Chief, Photoduplication Service, Library of Congress.

† The values listed for Cs_2I_8 are slightly different from those given in the article on the structure of this compound (Havinga, Boswijk & Wiebenga, 1954) because a more accurate correction for the finite-series effect was made after the publication of this paper.