lating strips running parallel to the $a+b$, c planes. These strips are joined by AsO_4 tetrahedra which share edges with MgO_8 groups in one strip and corners with $Mg(2)O_6$ groups in the next. Similar sheets are generated normal to those in Fig. 1 by the symmetry of the crystal and provide the three-dimensional bonding. Two of the three Mg atoms have distorted octahedral environments of oxygen atoms with average Mg(1)–O and Mg(2)–O distances of 2·074 and 2·099 Å respectively. The third Mg atom lies at a site of $\bar{4}$ symmetry, with fractional occupancy of $\frac{1}{2}$, and is bonded to four oxygen atoms at 2·240 Å and an additional four at 2·722 Å. From Brown & Shannon's (1973) relationship between bond length, R, and bond strength S_i:

$$\ln S_i = 5·0 \ln \frac{2·098}{R} - 0·0996,$$

it is found that a total bond strength of 0·956 is contributed by the four nearest oxygen atoms in agreement with a value of 1 expected for the fractionally occupied Mg^{2+}. The longer bond lengths contribute an additional 0·408 to the $\sum S_i$ value, however.

As(1) lies on a twofold axis and As(2) lies at a site with $\bar{4}$ symmetry. In the former case the two unique sets of As–O bond lengths differ significantly and in both cases the AsO_4 groups show substantial angular distortion with bond angles ranging from about 104 to 121°. In the case of $As(2)O_4$ this arises from a compression along the $\bar{4}$ axis. The $As(1)O_4$ group, on the other hand, shares three of its edges with three different Mg polyhedra [the O(2)–O(2') edge is shared with an $Mg(1)O_6$ group while the O(2)–O(3) and O(2')–O(3') edges are shared with two different $Mg(3)O_n$ polyhedra] and the three smallest angles of the $As(1)O_4$ group correspond to the three angles subtended by these shared edges.

Robijn (1967) has postulated, on the basis of chemical analyses of various samples of $Mg_3As_2O_8$, that a range of stable non-stoichiometric compositions might exist. The structure of $Mg_3As_2O_8$ suggests that two additional Mg ions could be accommodated in the unit cell by filling the fractionally occupied Mg(3) site. A composition $Mg_{20}As_{11·2}O_{48}$ can thus be obtained by the removal of arsenic ions in order to maintain electrical neutrality. Refinement of the population parameters of the arsenic and magnesium ion sites in the present case did not indicate significant deviations from the composition $Mg_3As_2O_8$.

The X-ray powder pattern of $Mg_3As_2O_8$ has been reported independently by three groups of workers (Ide, Kawai & Yamazaki, 1962; Robijn, 1967; Travni-çek, Kröger, Botden & Zalm, 1952). One of these attempted to index the pattern on a hexagonal cell (Robijn, 1967). The powder pattern of the sample used in this study agrees well with those previously reported, indicating that the hexagonal assignment was incorrect.

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Refinement of the Crystal Structure of Caesium Dichloroiodide

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Abstract. $CsICl_2$, trigonal, space group $R\bar{3}m$ with $a = 5·469$ (2) Å, $\alpha = 70·67$ (3)°, $Z = 1$. The atomic positions have been determined by least-squares refinement of counter intensities, the final R being 0·031 for 256 reflexions. The I–Cl bond length is 2·548 Å.

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Experimental. The orange-yellow crystals commonly develop the forms {100}, {110}, and {1$\bar{1}$0}, referred to rhombohedral axes, and are elongated along [111]. The material slowly loses halogen and the crystals were mounted with grease in thin-walled glass capillaries.

The cell constants were obtained from Weissenberg photographs superimposed with NaCl reflexions. The intensities for the 756 reflexions in one quadrant of

reciprocal space with $(\sin \theta)/\lambda \leq 0.808$ Å^{-1} were collected on a Nonius AD3 diffractometer with a $\theta/2\theta$ scan technique and Zr-filtered Mo radiation. A crystal of approximate dimensions $0.05 \times 0.02 \times 0.02$ cm, aligned along [111], was used. Corrections were made for intensity changes in the primary beam (by use of reference intensities) and for Lorentz and polarization effects. An absorption correction [μ(Mo $K\alpha$) = 129.6 cm^{-1}] was applied by the method of Busing & Levy (1957). Symmetry-equivalent reflexions were averaged to give 256 significant independent intensities.

The least-squares refinement was performed with the X-RAY 70 system of programs (1970), the function minimized being $\sum w(F_o - F_c)^2$. Scattering factors for the atoms were taken from *International Tables for X-ray Crystallography* (1962). The atomic positions determined by Wyckoff (1920) were used as starting parameters. Four cycles of full-matrix least-squares refinement with isotropic and six with anisotropic temperature factors reduced R to 0.031. Refinement of an extinction parameter (Zachariasen, 1968) indicates a reduction in intensity of approximately 35% for the two strongest low-order reflexions 01$\bar{1}$ and 121. The maximum parameter shift in the final cycle was 0.07σ. In the final cycles $\langle (F_o - F_c)^2 \rangle$ appeared to be approximately independent of $|F_o|$ and $(\sin \theta)/\lambda$ and, in consequence, unit weights were used.

The final atomic parameters, with estimated standard deviations, are given in Table 1.*

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* A list of observed and calculated structure factors has been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30186 (4 pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Atomic parameters with estimated standard deviations*

	Site symmetry	x/a	U_{11}^*	U_{12}
Cs	$\bar{3}m$	0.0†	0.0378 (4)	−0.0104 (2)
I	$\bar{3}m$	0.5†	0.0274 (4)	−0.0075 (2)
Cl	$3m$	0.2913 (2)	0.0380 (8)	−0.0131 (5)

* Temperature factor in the form: $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}kha^*b^* + 2U_{23}klb^*c^* + 2U_{13}hla^*c^*)]$, $U_{11} = U_{22} = U_{33}$ and $U_{12} = U_{23} = U_{13}$.
† Parameters fixed by symmetry

Discussion. We were interested in determining the structure of the ICl$_3$F$^-$ ion as no information exists on the geometry of this ion or any pentahalide ions containing three different halogen atoms. Attempts to prepare the caesium salt as described by Booth, Swinehart & Morris (1932) failed, and instead we obtained CsICl$_2$ whose structure is described by Wyckoff (1920). It is noticeable, however, that the I–Cl bond length (2.25 Å) is considerably shorter than in other compounds containing the ICl$_2^-$ ion (Visser & Vos, 1964; Rømming, 1958) and shorter than predicted by valence theory (Wiebenga & Kracht, 1969). It was therefore decided to refine the structure.

The Cs$^+$ and ICl$_2^-$ ions lie on special positions. The anion lies along the $\bar{3}$ axis, the iodine atom coinciding with the inversion centre at $(\frac{1}{2},\frac{1}{2},\frac{1}{2})$. The cation lies on the $\bar{3}$ axis at the origin. The I–Cl bond length of 2.548 (2) Å determined in this work is in good agreement with the value of 2.55 Å found in N(CH$_3$)$_4$ICl$_2$ (Visser & Vos, 1964), being some 0.23 Å longer than the sum of covalent radii for iodine and chlorine.

The distance (3.957 Å) between the iodine atom in one cell and the chlorine atoms in adjacent cells may be compared with the sum of van der Waals radii (Pauling, 1960) for the atoms (3.95 Å). The remaining non-bonded contacts, Cs$\cdots$I (4.181 Å), Cs$\cdots$Cl (3.558, 3.688 Å) and Cl$\cdots$Cl (3.794 Å), are rather longer than the sum of van der Waals radii.

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