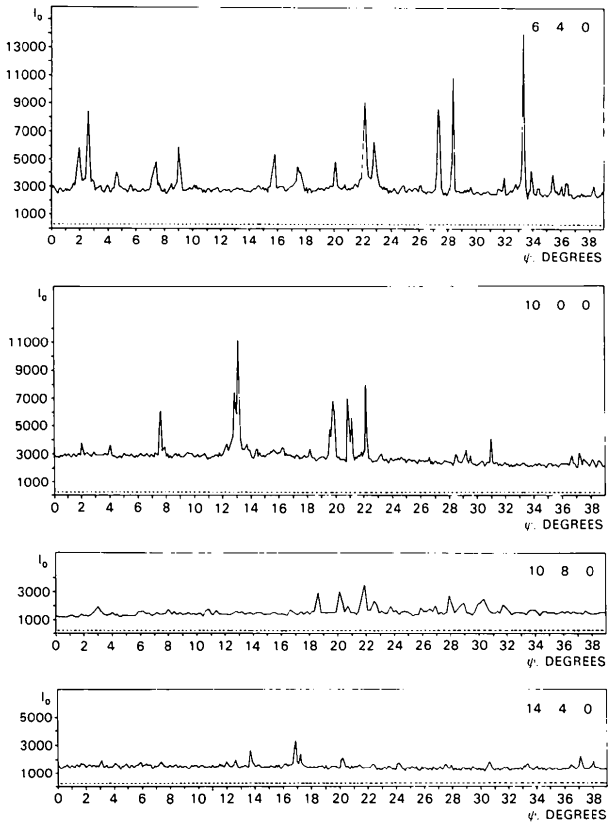

Fig. 1. Intensities of four reflections of $CuIn_5S_8$, taken with the θ–2θ scan technique at various angles, ψ, around their scattering vectors, with $\Delta\psi = 0{\cdot}1°$. The continuous curve represents the integrated intensity I_0, corrected for the background, and the dotted line indicates the $3\sigma(I)$ value.

arises from the fact that these reflections are affected by the Renninger effect. Since low-angle reflections (*e.g.* 200) are affected more in X-ray diffraction than in other techniques, we have examined some high-angle forbidden reflections.

Debye–Scherrer powder films of $CuIn_5S_8$ and $AgIn_5S_8$ showed that both have cubic spinel-type structures. For precise determination of lattice constants a Philips camera with the asymmetric Straumanis film mounting was used. Four reflections with $\theta > 65°$ gave the following final cell constants: $AgIn_5S_8$: a = 10·8268 (5) Å; $CuIn_5S_8$: a = 10·6858 (3) Å (the Nelson–Riley function was plotted). Single crystals were mounted on a four-circle Syntex $P2_1$ diffractometer; Mo $K\alpha$ (λ = 0·71073 Å) radiation was used (μ = 11·54 and 12·21 mm^{-1} respectively). The crystal was rotated about the scattering vector of each reflection (ψ angle) and a θ–2θ scan collection carried out at fixed intervals (0·1°) of ψ. Two background values were measured at 1° below and 1° above the $K\alpha_1$ and $K\alpha_2$ peaks respectively.

The copper compound shows the presence of a considerable number of forbidden reflections, even at relatively high θ values. Four of these, chosen for their remarkable intensities, are plotted in Fig. 1. There are many Renninger peaks, separated by approximately flat regions, where the intensity variations are of the order of three or four times $\sigma(I)$.

The diagrams show, from top to bottom, two plots for each reflection: the intensity I_o corrected for the background, and the $3\sigma(I)$ level.

On the basis of these results $CuIn_5S_8$ can be assigned to space group $F\bar{4}3m$. However, these results could be explained by a non-random distribution of copper and indium ions in the two tetrahedral sites 4(*a*) and 4(*c*), so a complete structural determination of this compound has been undertaken.

No forbidden reflections were detected for the silver compound, so we believe that, on the basis of the X-ray diffraction analysis, it must be assigned to the conventional space group $Fd3m$.

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Reference

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Radiation leakage around X-ray tube shields

The Union's Commission on Crystallographic Apparatus recommends that the radiation level around X-ray tube shields should be carefully checked, because considerable leakage has been detected in some laboratories. Particular care should be taken when high-energy tubes are used and when tubes made by one manufacturer are enclosed in shields made by a different manufacturer.

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