# The Renninger effect — an example and its implication. By J. C. Speakman, *Chemistry Department,*

*The University, Glasgow, W.2, Scotland*

The Renninger effect (James, 1950) occurs when the surface of the Ewald sphere passes simultaneously through two reciprocal lattice points ($h_1$ and $h_2$). Because the sphere necessarily passes through the origin as well, a third point ($h_R$) is also involved, with $h_R = h_1 + h_2$. These three points, or their antipodes, are in a mutually equivalent relationship; there are three positions of the sphere, in each of which its surface passes through one pair of points. The situation is simply illustrated for the two-dimensional case by Fig. 1 ($a$), which is a modification (and slight emendation) of Fig. 31 of Lipson & Cochran (1953). In their example two strong reflexions (here indexed as $02\bar{1}$ and $0\bar{7}2$) are supposed to give rise to the double reflexion $0\bar{5}1$. The Ewald circle in the settings $A$, $B$ and $C$ passes respectively through the pairs $0\bar{7}2/0\bar{2}1$, $0\bar{5}1/02\bar{1}$ and $05\bar{1}/07\bar{2}$.

This implies that the corresponding pairs of spots on a Weissenberg photograph must lie each on a vertical line, as is represented in Fig. 1 ($b$). This principle immediately suggests a convenient way of searching for a pair of strong reflexions to account for a supposed Renninger reflexion. It can be extended to three dimensions by superposing Weissenberg diagrams of different layers, in a correct lateral relationship.

In the special case of an orthogonal net, which has $mm$ symmetry, all three points ($h_1$, $h_2$ and $h_R$, or their equivalents) lie on a single Ewald circle, and hence all three spots lie on a single vertical line in the Weissenberg photograph. We have recently come across a striking example of this in rubidium hydrogen bisglycollate (Golič & Speakman, 1965), which crystallizes in the monoclinic system with $a = 4.052$, $b = 17.91$, $c = 10.52$ Å, $\beta = 98.4°$. The space group is $P2_1/n$, and this was the basis for a successful structure analysis. Yet on photographs taken about the $a$ axis there appeared several weak, or very weak, reflexions that violated the space-group conditions: the most prominent of these were 050

and 0,13,0. That they were Renninger spots was suggested by their absence from $c$-axial photographs, and corroborated by the fact that each lay in a vertical line with a pair of strong reflexions in the $0kl$ Weissenberg photograph: 050 could be attributed to two pairs $09\bar{1}/0\bar{4}1$ and $0,11,\bar{2}/0\bar{6}2$, since the five spots 050, $09\bar{1}$, $0\bar{4}1$, $0,11,\bar{2}$ and $0\bar{6}2$ were vertically collinear: whilst 0,13,0 could be attributed to $091/0\bar{4}\bar{1}$, with the three spots 0,13,0, 091 and 041 collinear.

This interpretation can be tested analytically by calculating the $\omega$ values for the two sets of reflexions. Using the symbols defined in *International Tables for X-ray Crystallography* (1959), we have

$$\omega = \tfrac{1}{2}\varUpsilon + \varphi = \theta + \sin^{-1}(1c^*/2\sin\theta),$$

and

$$4\sin^2\theta = (kb^*)^2 + (1c^*)^2,$$

for an orthogonal net.

For this bisglycollate crystal, with Cu-radiation, the reciprocal-lattice parameters are $b^* = 0.086085$ and $c^* =$

Table 1. *Analytical confirmation of Renninger spots at* 050
*and* 0,13,0. (*Rubidium hydrogen bisglycollate*).

| Reflexion | $\theta$ | $\varphi$ | $\omega$ |
|---|---|---|---|
| $09\bar{1}$ | $+23°\ 14'$ | $-10°\ 51'$ | $12°\ 23'$ |
| $0\bar{4}1$ | $-10°\ 48'$ | $+23°\ 18'$ | $12°\ 30'$ |
| $0,11,\bar{2}$ | $+29°\ 47'$ | $-17°\ 23'$ | $12°\ 24'$ |
| $0\bar{6}2$ | $-12°\ 19'$ | $+29°\ 51'$ | $12°\ 32'$ |
| | | Mean | $12°\ 27'$ |
| 050 | $+12°\ 26'$ | — | $12°\ 26'$ |
| 091 | $+23°\ 14'$ | $+10°\ 51'$ | $34°\ 5'$ |
| 041 | $+10°\ 48'$ | $+23°\ 18'$ | $34°\ 6'$ |
| | | Mean | $34°\ 6'$ |
| 0,13,0 | $+34°\ 1'$ | — | $34°\ 1'$ |

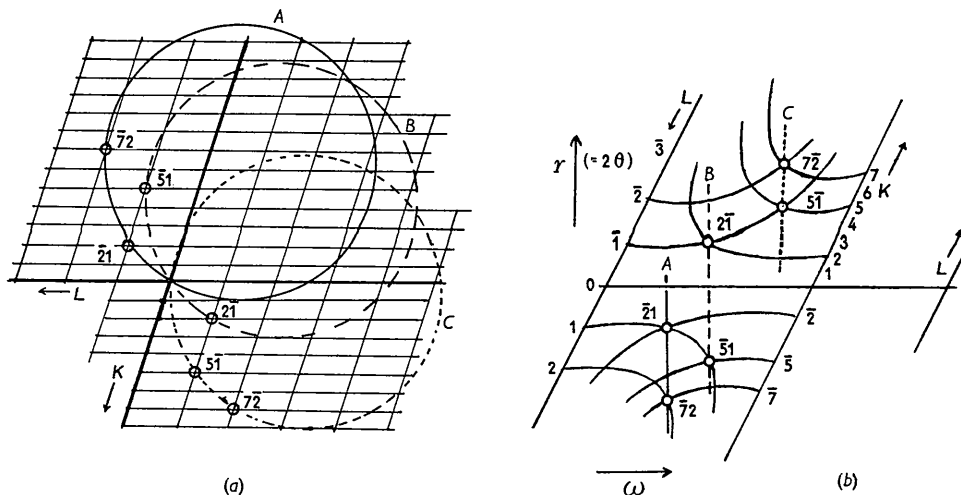

Fig. 1. Relationship of double reflexion $0\bar{5}1$ to reflexions $02\bar{1}$, $07\bar{2}$ which give rise to it.

0·148262, and we take $\theta$, or sin $\theta$, to be negative for a spot on the lower half of the film. The results are set out in Table 1.

On our photographs the spots extend over about 1° in the $\omega$ direction. This extension must mainly be due to crystal size. We do not know the true mosaic spread. Table 1 suggests that conjunction within 5′ in the $\omega$ values is adequate to cause double reflexion, which seems a reasonable conclusion.

If a tolerance of this order applies generally, double reflexions must be fairly common. With a cell of moderate size, $\sim 10^3$ reflexions may occur in the region covered, and the chance must be statistically high that two with at least moderate intensities will have $\omega$ values within 5′ of each other. The consequent double reflexion will be obvious only in the comparatively rare case where it leads to a breach of the space-group absences. In general it will merely add to the intensity of a normal reflexion.

As was pointed out by Lipson & Cochran (see also Yakel & Fankuchen, 1962), this is yet another source of error in the measurement of intensities. It may deserve consideration when an accuracy better than 5% is being sought.

### References

Golič, L. & Speakman, J. C. (1965). *J. Chem. Soc.* To be published.
*International Tables for X-ray Crystallography* (1959). Vol. II, Chapter 4. Birmingham: Kynoch Press.
James, R. W. (1950). *The Optical Principles of the Diffraction of X-rays.* London: Bell.
Lipson, H. & Cochran, W. (1953). *The Determination of Crystal Structures.* London: Bell.
Yakel, H. L. & Faunkuchen, I. (1962). *Acta Cryst.* **15**, 1188.

---

# A new polytype of zinc sulfide crystals. By Maria Farkas-Jahnke, *Research Institute for Technical Physics of the Hungarian Academy of Sciences, Budapest, Hungary*

As is well known, zinc sulphide is liable to form polytype modifications similarly to silicon carbide, during the growing process, in addition to the wurzite and sphalerite structures. This problem has been investigated by several authors (Frondel & Palache, 1947; Strock & Brophy, 1955; Buck & Strock, 1955; Smith, 1955; Evans & McKnight, 1959; Ramsdell, 1947; Verma, 1957), and polytypes containing 2, 3, 4, 6, 8, 9, 10,12, 15, 21, 54, … layers have been already found among natural and artificially made zinc sulphide crystals. Investigating the structure of zinc sulphide single crystals, grown from the vapour phase, we have found, besides the known polytypes, one crystal containing a new polytype modification of 24 layers. The lattice parameters of the new modification are:

$$a_0 = 3,853, \quad c_0 = 75,067 \text{ Å}; \quad a_0/c_0 = 1:19,483 ,$$

with cell contents $Zn_{24}S_{24}$. In the same crystal there are several kinds of the numerous possible 24-layer structures, as seen in the 25° oscillation patterns in Fig. 1. The X-ray diffraction patterns were made in a cylindrical camera of 57·3 mm diameter, with unfiltered Fe $K$ radiation.

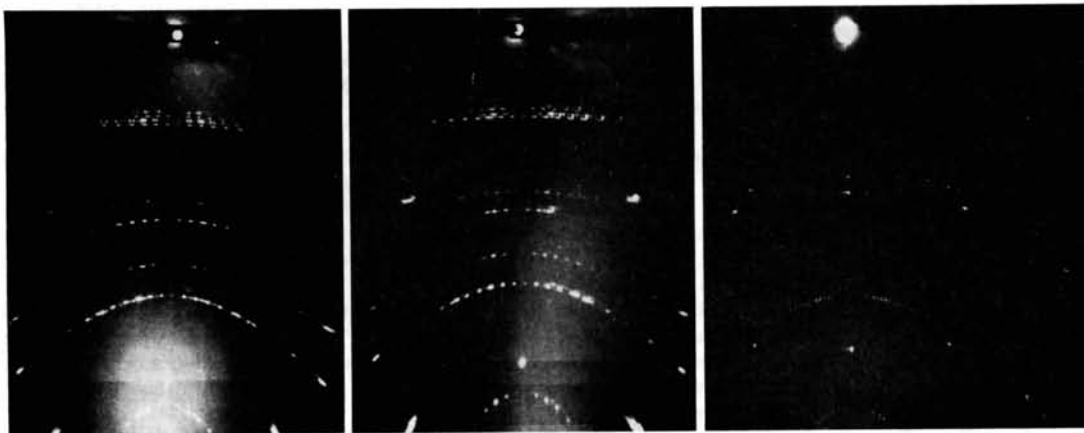These interesting structures will be discussed later in detail.

Fig. 1. 25° oscillation patterns taken from different parts of the 24-layer ZnS polytype crystal.