Acta Cryst. (1957). **10**, 380

Comments on the structure of $KBrF_4$.*

By Stanley Siegel, *Chemistry Division, Argonne National Laboratory, Lemont, Illinois, U.S.A.*

(*Received* 12 *February* 1957)

Sly & Marsh (1957) have suggested an alternative structure for the compound $KBrF_4$ (Siegel, 1956). These authors propose a square configuration for the BrF_4^- ion, whereas I report a tetrahedral arrangement.

I agree with Sly & Marsh that the planar configuration is the one to be expected. However, it is not possible to distinguish between the two cases with certainty even if one places considerable reliability on the few isolated discrepancies.

In order to determine the fluorine positions, it is necessary to consider reflections *hkl* with *l* odd. Unfortunately, only a limited number of such reflections are observed on the powder pattern, and these generally suffer interference. Only a few reflections of this class (211, 213, 215) are observed in a single-crystal oscillation pattern, but interferences arise here from powder lines of decomposition products. Contrary to a statement by Sly & Marsh concerning the ability to resolve intensities from the powder pattern, the fact remains that it has been possible, with only a few exceptions, to evaluate intensities with reasonable consistency from line profiles in the microphotometer tracings. The agreement between observed and calculated $\sin^2\theta$ values certainly indicates some measure of resolution.

The 215 reflection, when corrected for the effects of 224, has an $I_o^{\frac{1}{2}}$ value of 33. Since this value includes the intensity from a non-resolvable neighboring impurity maximum, the true $I_o^{\frac{1}{2}}$ value must be less than 33 and is so indicated. However, it is doubtful that the interfering line has a high enough intensity to make up the difference between my observed and calculated intensities. Hence, the value given by Sly & Marsh appears to be reasonable. The 415 reflection is distinct. My calculated value of $I_c^{\frac{1}{2}}$ (1·2 and not 12 as given) is obviously low. The planar configuration also gives a low calculated value, but it is certainly a more reasonable one.

The 413 reflection is poorly resolved from its neighbors. However, the intensity is sufficiently high to permit its position and intensity to be determined. The tetrahedral case accounts for this reflection. However, the intensity calculated for the planar configuration is low, and, for the intensity scale used, the line would show near-zero intensity on the film. The only other discrepancy arises in the 114 reflection. Here, the planar configuration also gives a low calculated intensity. Data at the larger angles are not too reliable for evaluation. Finally, if we restrict ourselves only to the 215 and 415 reflections, major changes in the *z* coordinate of the tetrahedral fluorines will be required in order to improve intensities. Such shifts would, of course, modify the entire set of calculated intensities. Presumably, only minor changes in coordinates would be required for the planar fluorines in order to effect better agreement for the two reflections.

The discrepancy warrants a review of the structure under conditions of improved resolution. Accordingly, a new structure determination has been initiated.

References

Siegel, S. (1956). *Acta Cryst.* **9**, 493.
Sly, W. G. & Marsh, R. E. (1957). *Acta Cryst.* **10**, 378.

---

* Based on work performed under the auspices of the U.S. Atomic Energy Commission.

Acta Cryst. (1957). **10**, 380

Comments on Pauling's paper on effective metallic radii for use in the β-wolfram structure.

By S. Geller, *Bell Telephone Laboratories Incorporated, Murray Hill, New Jersey, U.S.A.*

(*Received* 16 *March* 1957)

The author's determination of a set of effective coordination number (12) radii for compounds A_3B having the β-wolfram structure (Geller, 1956) was recently criticized by Pauling (1957), who states that by the application of his resonating-valence-bond theory he obtains different radii which not only lead to better agreement with the measured cell edges but also are nearly the same as his *R*(L12) metallic radii formulated in 1947.

In the following we discuss Pauling's criticism under three headings:

(1) Has it been demonstrated that the resonating-valence-bond theory applies to compounds with the β-W structure?
(2) If not, has a new and justifiable technique been used to obtain a set of metallic radii?
(3) Are the new results, judged merely as an empirical correlation, an improvement on those of this author?

(1) The set of radii for the 32 compounds A_3B was obtained by Pauling by weighting the contributions to the interatomic distances in accordance with the relative numbers of atoms of each kind in the compound, i.e. 3:1. As he points out, the resonating-valence-bond theory requires a recognition of the coordination and valence of the atoms and the strength of bonds. However, because the 3:1 weighting alone suffices to give a seemingly good set of radii, these considerations, ordinarily a basic