of atoms in the two-dimensional projections generally used and the high symmetry of the 'ideal' structures require that deviations from special positions be supported by unambiguous evidence. The methods currently employed for assessing the accuracy of the electron-density distributions, atomic parameters and the significance of differences in bond lengths at each stage of the refinement (Lipson & Cochran, 1953; Jeffrey & Cruickshank, 1953) are hence of particular importance in the analysis of layer silicates. Where no recourse to these criteria has been made, plausible but unwarranted structural features have been proposed.

As an obvious example of the effects of inadequate data, the recent analysis of a chlorite by Steinfink (1958) may be cited. In this analysis, 51 positional parameters, as well as hidden parameters involved in loading the atomic scattering curves, are claimed to have been derived from 28 $hk0$ and 50 $0kl$ structure amplitudes, with an accuracy, implied by the later discussion, of considerably better than $\pm 0{\cdot}05$ Å, although no actual limits are quoted. The suspect nature of the atomic parameters and inter-atomic distances derived from these data is immediately apparent from a comparison of the tabulated atomic parameters (Table 2 of the paper) with the contoured electron-density maps (Figs. 1 and 2 of the paper). In our Fig. 1, we have placed in juxtaposition extended reproductions of Steinfink's ($0kl$) and ($hk0$) electron-density projections. Inspection reveals the following, more obvious, points of criticism:

(i) The displacement of atoms OH_2 and OH_4 (Fig. 1(a)) from 'ideal' positions is justified by Steinfink on the basis of the asymmetry of their peak distributions. Atom O_2, however, which shows equally marked asymmetry of distribution, is assigned an 'ideal' position.

(ii) No account is taken of the ridge extending from O_5 in the z-direction, while a displacement of the y-parameter of this atom is held to be significant. The latter displacement involves a shift of $\varDelta y = 0{\cdot}333 - 0{\cdot}328 \equiv 0{\cdot}046$ Å.

(iii) The selected y parameter of atom OH_1, as indicated in the ($hk0$) projection at A (Fig. 1(b)) is clearly inconsistent with the observed peak distribution in the more reliable ($0kl$) projection* (Fig. 1(a)). In this connection, it should be pointed out that the projection corresponding to the ($hk0$) contour map is inherently extremely complex due to overlap. With typical peak heights of individual octahedral, tetrahedral and oxygen sites of the order of 46, 50 and 20 e.Å^{-2} respectively (derived from Mathieson, 1958), it is evident that this projection is so crowded by the heavier scattering units that exact deductions regarding oxygen positions cannot be expected from 28 structure amplitudes.

These considerations lead us to the conclusion that the probable errors in this analysis are much greater than would be required to substantiate a real deviation from the 'ideal' structure and hence, to support the subsequent detailed argument put forward by Steinfink.

In view of the increasing interest presently being shown in the analysis of layer silicates, we are of the opinion that a general plea for caution in the interpretation of minor details in such analyses is called for, more particularly in the interests of those not fully conversant with the methods of structure analysis and hence unable to judge the strength of the evidence presented.

References

JEFFREY, G. A. & CRUICKSHANK, D. W. J. (1953). *Quart. Rev.* **7**, 335.
LIPSON, H. & COCHRAN, W. (1953). *The Crystalline State*, Vol. III. London: Bell.
MATHIESON, A. McL. (1958). *Amer. Min.* **43**, 216.
STEINFINK, H. (1958). *Acta Cryst.* **11**, 191.

---

* Although neither projection is based on adequate data, y parameters derived from the ($0kl$) projection with 50 terms will be more trustworthy than those from the ($hk0$) projection with 28 terms.

Anhydrous *bis*-salicylaldehydato-nickel.

By F. K. C. LYLE, B. MOROSIN and E. C. LINGAFELTER, *Department of Chemistry, University of Washington, Seattle 5, Washington, U.S.A.*

We have previously reported the isomorphism of anhydrous *bis*-salicylaldehydato–nickel and –zinc as possible evidence for a tetrahedral configuration for 4-coordinated nickel (II) (Curtiss, Lyle, Lingafelter, 1952). Because of the considerable likelihood of the existence of a 'polymerized' octahedral coordination, it would be desirable to have a complete determination of the crystal structure. We have succeeded in obtaining a few very small single crystals of the nickel compound and report at this time the cell dimensions and space group.

Crystals were obtained by two different techniques. The first batch was prepared by the slow evaporation at about 60 °C. of a saturated solution of nickel stearate in benzene to which stearic acid and salicylaldehyde had been added. The second batch was prepared by the slow

evaporation of a chloroform solution formed by refluxing a mixture of dry chloroform and *bis*-salicylaldehydato–nickel. Neither batch was large enough to allow chemical analyses to be made. However, powder patterns of both batches are identical with that from a dehydrated sample of *bis*-salicylaldehydato–nickel (Curtiss, Lyle & Lingafelter, 1952).

The crystals are monoclinic, with 12 molecules in a cell of dimensions:

$$a_0 = 15{\cdot}34, \quad b_0 = 12{\cdot}46, \quad c_0 = 19{\cdot}44 \text{ Å}; \quad \beta = 97^\circ 8'.$$

Density, observed 1·61 g.cm.$^{-3}$; calculated 1·63 g.cm.$^{-3}$.

The data were collected from rotation and equi-inclination Weissenberg photographs, using Cu $K\alpha$ radiation ($\lambda = 1{\cdot}5418$ Å).

Systematic absence of (hkl) reflections for $k+l$ odd and of ($h0l$) for h odd indicate the space group to be Aa or $A2/a$.

The presence of 12 molecules indicates that the asymmetric unit probably contains 3 molecules, analogous to that found for bis-acetylacetonato–nickel (Bullen, 1956).

We do not plan to investigate this compound further at this time.

References

BULLEN, G. J. (1956). *Nature, Lond.* **177**, 537.

CURTISS, D. H., LYLE, F. K. C. & LINGAFELTER, E. C. (1952). *Acta Cryst.* **5**, 388.

Maille cristalline et groupe d'espace du sulfate ferreux anhydre, FeSO₄. Par J. COING-BOYAT,

Laboratoire d'Electrostatique et de Physique du Métal, Institut Fourier, Grenoble, France

Une précédente publication (Coing-Boyat, 1959) traite des mailles et des groupes d'espace des deux formes (α et β) du sulfate de cobalt-II et du sulfate de manganèse-II, anhydres. Elle est complétée par la présente étude du sulfate de fer-II anhydre.

FeSO₄ a été préparé en deshydratant le monohydrate FeSO₄, H₂O à 300 °C. dans un courant d'argon et en maintenant ces conditions 20 hr. afin que le sel anhydre cristallise suffisamment. La poudre, blanchâtre, micro-cristalline, ainsi obtenue est hygroscopique; c'est pourquoi, afin de constituer l'échantillon à examiner aux rayons X, elle a été introduite dans un tube de verre d'environ 0,2 mm. de diamètre intérieur, aux parois très minces, scellé ensuite.

L'analogie des diagrammes Debye–Scherrer de FeSO₄ avec ceux de la forme α de CoSO₄ (stable au-dessous de 600 °C.) a donné la clef de leur identification qui a permis la détermination de la maille.

Tableau 1. *Des plans reflecteurs de* FeSO₄

Diagramme D.-S., radiation $K\alpha$ du Fer ($\lambda = 1,9373$ Å)
I: intensité des raies; F: fortes; m: moyennes; f: faibles

hkl	I	d_c (hkl)	d_o (hkl)
110	fm	$4,38_{43}$ Å	$4,38_{60}$ Å
020	f	$3,98_{44}$	$3,98_{86}$
111	F	$3,64_{97}$	$3,64_{82}$
021	m	$3,40_{95}$	$3,40_{82}$
112 ⎫ 200 ⎭	FF	$2,63_{29}$ $2,62_{54}$	$2,62_{95}$
130	mF	$2,37_{05}$	$2,37_{10}$
202	m	$2,05_{29}$	$2,05_{20}$
040	fm	$1,99_{24}$	$1,99_{26}$
132 ⎫ 023 ⎭	fm	$1,92_{39}$ $1,92_{28}$	$1,92_{28}$
222	mF	$1,82_{50}$	$1,82_{40}$
042	m	$1,70_{46}$	$1,70_{48}$
004	fm	$1,64_{64}$	$1,64_{68}$
133	f	$1,61_{06}$	$1,61_{06}$
241 ⎫ 114 ⎭	f	$1,54_{30}$ $1,54_{14}$	$1,54_{12}$
024 ⎫ 312 ⎭	m	$1,52_{18}$ $1,51_{73}$	$1,51_{73}$
330	m	$1,46_{16}$	$1,46_{15}$
242	F	$1,42_{98}$	$1,43_{00}$
152	f	$1,38_{39}$	$1,38_{43}$
134	m	$1,35_{23}$	$1,35_{26}$
400	fm	$1,31_{28}$	$1,31_{26}$

Le Tableau 1 montre la bonne concordance entre, d'une part, les équidistances des plans réflecteurs calculées d'après cette maille, $d_c(hkl)$, et d'autre part, celles déduites des mesures sur diagramme, $d_o(hkl)$.

On constate que, seules, figurent sur les diagrammes de FeSO₄, les raies hkl (et $hk0$, $0kl$) dont $h+k$ est pair, et les raies $h0l$ dont l (et h) sont pairs, ce qui est caractéristique du groupe d'espace centro-symétrique $Cmcm$ auquel appartient CoSO₄ α.

Le sulfate ferreux anhydre cristallise donc dans le système orthorhombique, groupe $Cmcm$ (D_{2h}^{17}), sa structure est du type CrVO₄ (Brandt, 1943); sa maille contient $Z = 4$ unités FeSO₄,

$$a = 5,25_5 \pm 0,005, \quad b = 7,97_5 \pm 0,008, \quad c = 6,59 \pm 0,01 \text{ Å},$$
$$U = 276,2 \text{ Å}^3, \quad D_x = 3,65_3 \text{ g.cm.}^{-3}.$$

Ces résultats diffèrent des données connues jusqu'alors Hammel, 1936):

$$a = 4,82, \quad b = 6,81, \quad c = 8,67 \text{ kX}.$$

Hammel avait admis l'isomorphie de FeSO₄ avec la forme β (hautes températures) de CoSO₄, alors qu'en fait, FeSO₄ est isotype de la forme α de CoSO₄, donc aussi de MgSO₄ (Rentzeperis, 1958) de MnSO₄, de NiSO₄ (Dimaras, 1957) et des chromates de métaux divalents (Brandt, 1943) isomorphes de CrVO₄.

References

BRANDT, K. (1943). *Ark. Kemi, Min., Geol.* A, **17**, No. 6, p. 13.

BRANDT, K. (1955). *Structure Reports*, **9**, 181.

COING-BOYAT, J. (1959). *C. R. Acad. Sci., Paris*, **248**, 2109.

DIMARAS, P. I. (1957). *Acta Cryst.* **10**, 313.

HAMMEL, F. (1936). *C. R. Acad. Sci., Paris*, **202**, 57.

RENTZEPERIS, P. J. & SOLDATOS, C. T. (1958). *Acta Cryst.* **11**, 686.