LETTERS TO THE EDITOR

THE CRYSTAL STRUCTURE OF MONOFUMARATO TETRAAQUO NICKEL (II), DIHYDRATE

THE title compound $[Ni(C_4H_2O_4), 4H_2O], 2H_2O$ was investigated to unravel the scheme of hydrogen bonds in the crystal as also to study the oxygen ligands around the metal.

Crystal data—Monoclinic with $a = 9.00$, $b = 10.77$, $c = 5.14$ Å, $\beta = 98.0°$, space group $P2_1/c$, $D_{obs} = 1.86$ gm/cm³, $D_{cal} = 1.89$ gm/cm³, $Z = 2$. 510 reflexions were collected by Weissenberg photography using single crystals and intensities estimated visually, usual corrections were applied and statistical method used to get the $|F|^2$ values on approximately absolute scale.

Structure determination and comments.—As the space group $P2_1/c$ requires four general formula units in the crystal, both the metal atoms as well as the fumarate anions must occupy centres of symmetry. The three water molecules in the asymmetric unit will occupy general positions. The structure was solved from a three-dimensional Patterson map and refined by successive [001] and [010] Fourier projections. At the present stage of analysis, the R-factors are $R(hko) = 0.15$, $R(hol) = 0.16$, $R(okl) = 0.16$, $R(hkl) = 0.18$.
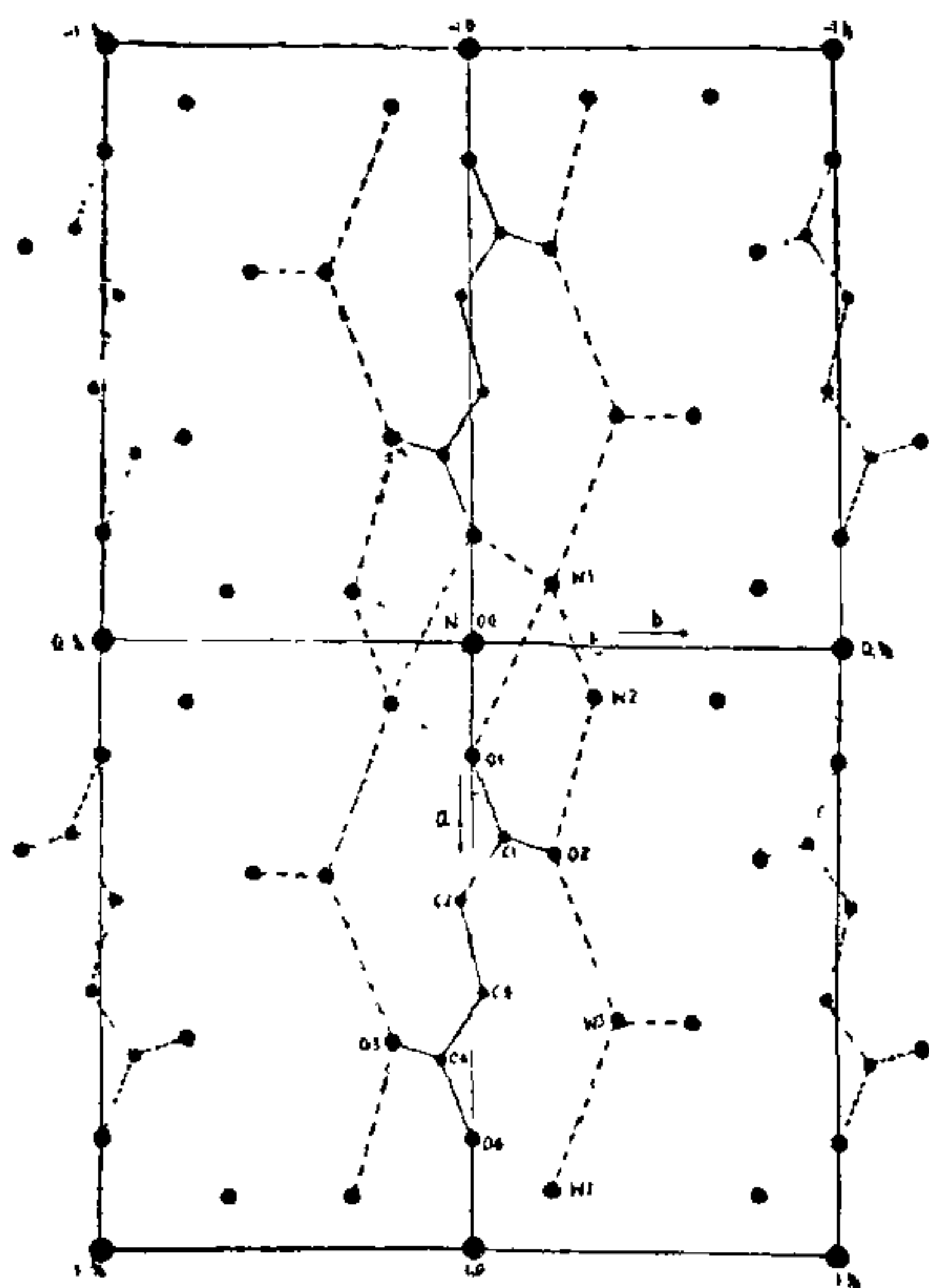

FIG. 1

A view of the crystal structure looking down [001] is shown in Fig. 1 with hydrogen bonds shown by dashes and dots and metal-oxygen linkages being shown by dotted lines.

In the crystal, the geometry of the fumarate anion is normal. There is six-fold coordination around the metal, Ni – O distances ranging from 2.02 to 2.06 Å. The structure is held together mainly by ionic and hydrogen bond linkages and also by van der Waals contacts. Chains of fumarate anions are linked *via* water molecules parallel to [100]. The hydrogen bonds *via* water molecules range from 2.63 to 2.91 Å.

As considerable interest lies in locating the hydrogen atoms; further work to refine the structure is in progress.

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FRACTIONAL CRYSTALLIZATION OF ALUMS—IV

Introduction

WHEN a pair of isomorphous salts is rapidly crystallized from an aqueous solution, the resulting solid solution changes in composition continuously. A study of the distribution of the salts in the resulting system is of considerable theoretical and practical interest.

Abu-Elamayem[1] studied the equilibrium between the solid and liquid phases by the relation

$$Y^2 W (K - 1) - Y \{1 + (K - 1)(W + a\} + K = 0 \tag{1}$$

where a and Y represent the weight fraction of the less soluble isomorphous salt in the original mixture and crystals separated respectively, W being the weight fraction of the two salts which are separated as crystals and K is the distribution coefficient.

For nonequilibrium condition, the following equation was also proposed :[1,2]

$$- \frac{dY}{dW} = \frac{1}{W} \left[\frac{1}{1 + \frac{1}{K} \left(\frac{1 - W}{a - WY} - 1 \right)^{1/m}} - Y \right] \tag{2}$$

where m is an empirical constant.

In our previously published investigations[1-3] on distribution during crystallization of systems containing two inorganic salts, it was confirmed that the experi-