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M. Smolik[a]
[a] Institute of Chemistry, Inorganic Technology and Electrochemistry, Silesian University of Technology, Gliwice, Poland

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DISTRIBUTION OF MICROAMOUNTS OF M^{2+} DURING $NISO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ CRYSTALLIZATION

M. Smolik

Institute of Chemistry, Inorganic Technology and
Electrochemistry, Silesian University of Technology,
44-100 Gliwice, Poland
E-mail: msmolik@zeus.polsl.gliwice.pl

ABSTRACT

Distribution coefficients (*D*) of trace amounts of Co^{2+}, Zn^{2+}, Cu^{2+}, Cd^{2+}, Mn^{2+}, Mg^{2+}, Fe^{2+}, and Ca^{2+} have been found during crystallization of $NiSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ at 25°C and 23°C. These values are affected mainly by solubilities of the corresponding double salts. Most of the microcomponent *D* values could be estimated from solubilities of their double salts. Some of the values are in good agreement with experimental data.

INTRODUCTION

The distribution (cocrystallization) coefficient *D* of a microcomponent is a measure of the effectiveness of crystallization as a purification process. However, the respective data in the literature are scarce. Hence, the dependence of *D* values on various physicochemical and crystallochemical factors, such as ionic charges and radii, similarities of crystal structure and unit-cell dimensions, ability to form solid solutions, and solubilities of corresponding salts, were determined to predict the *D* values for the various crystallization systems that have not been investigated to date.

The effect of these factors on the *D* values may differ for various crystallization systems, and very often these effects overlap as shown by investigations on cocrystallization of microcomponents with selected sulfates (1–4). Therefore, to estimate the influence of some factors, the others should be maintained at constant levels. In the case of the double salts $M^{II}SO_4{\cdot}M^{I}_2SO_4{\cdot}6H_2O$, where $M^{II}(Ni^{2+})$ and $M^{I}(NH_4^+, K^+, Rb^+, Cs^+$, and $Tl^+)$, as well as $M^{I}(NH_4^+)$ and $M^{II}(Co^{2+}, Zn^{2+}, Cu^{2+}, Cd^{2+}, Mn^{2+}, Mg^{2+}$, and $Fe^{2+})$, the effect of structure can be eliminated because all the listed double salts are monoclinic (space group $P2_1/a$) and have almost identical unit-cell dimensions: $a = 9225 \pm 0.062$ Å ($\pm 0.67\%$); $b = 12\,506 \pm 0.098$ Å ($\pm 0.79\%$); $c = 6242 \pm 0.034$ Å ($\pm 0.54\%$); $\beta = 106.58° \pm 0.40°$ ($\pm 0.38\%$) (5). Knowledge of these dimensions permitted us to examine the effects of remaining factors (ionic radius, solubility in water) on the *D* values.

Such an effect was established during the cocrystallization of trace amounts of M^+ ($Na^+, K^+, Rb^+, Ag^+, Cs^+, Tl^+$) with $NiSO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$ (6). The cocrystallization of M^{2+} with the same double salt proceeds in an interesting manner.

The investigations have concerned the cocrystallization of $NiSO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$ with a number of double salts: $CoSO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$ ($D_{Co} = 0.32 \pm 0.02$ at 20°C) (7); $FeSO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$ ($D_{Fe} = 0.14$ at 0° and 20°C) (7); $ZnSO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$ ($D_{Zn} = 0.24 \pm 0.02$ at 25°C) (8–9); $CuSO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$ ($D_{Cu} = 0.066 \pm 0.008$ at 25°C (8–9) or $D_{Cu} = 0.16 \pm 0.01$ at 20°C (10)); $MgSO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$ ($D_{Mg} = 0.114 \pm 0.006$ at 25°C (11) or $D_{Mg} = 0.125 \pm 0.003$ at 40°C (12)); and $MnSO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$ ($D_{Mn} = 0.037 \pm 0.001$) (12). However, the data involve principally the cocrystallization of macroquantities of $M^{II}SO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$ salts ($>0.5\%$) with $NiSO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$. In only one case did a researcher examine the cocrystallization of M^{2+} microquantities (720 ppm Cu^{2+}) (10). The present investigation was aimed to determine the *D* values for microquantities of these ions and for Cd^{2+} as well as Ca^{2+} and to analyze their dependencies on the above mentioned factors.

EXPERIMENTAL

Reagents and Solutions

Nickel (II) and ammonium sulfate hexahydrate, p.a., was purified by crystallization twice. Standard solutions of Mg(II), Cd(II), Zn(II), Co(II), Cu(II), Fe(II), and Mn(II) as sulfates and Ca(II) as a nitrate were used. Ammonia solution

(14 mol/L) was obtained by saturating distilled water with ammonia gas. Sodium versenate p.a. (POCh, Gliwice) was placed in a 0.1-mol/L water solution. Murexide ind. (POCh, Gliwice). Hydrazine sulfate p.a. (POCh, Gliwice) was added to a 2%-water solution.

Apparatus

An atomic absorption spectrometer model 3300 manufactured by Perkin Elmer was applied.

Analytical methods

Determination of the Macrocomponent Ion

Nickel (II) was determined by complexometric titration with sodium versenate in ammonia solution in the presence of murexide (13).

Determination of Microcomponents

Microcomponents (Co^{2+}, Zn^{2+}, Cu^{2+}, Cd^{2+}, Mn^{2+}, Mg^{2+}, Fe^{2+}, and Ca^{2+}) were determined by means of direct atomic absorption spectrometry (AAS) from solutions containing 1.00 g (or less) of $NiSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ in 25 mL. Absorbance values of the samples and a set of standards, in which the salt concentrations were identical to those of the standard, were measured under identical conditions. Fe^{2+} was also determined by means of AAS after oxidation and preliminary concentration and separation from an Ni^{2+} matrix by extraction of its thiocyanate complexes with methylisobuthylketone (KMIB) (14)

Procedure

After crystallization the crystals were separated from solution by filtration through a Büchner funnel with a sintered glass disk that was weighed, washed with saturated purified $NiSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ solution, and dissolved in water. The degree of nickel (II) and ammonium sulfate crystallization was determined from the nickel (II) content in both solutions.

The necessary sample volumes for determining the microcomponents, based on results obtained from the Ni(II) determinations, were calculated. The microcomponent concentration was equal to 1.00 g (or less) of $NiSO_4 \cdot (NH_4)_2 SO_4 \cdot 6H_2O$ in 25 mL.

Relative concentrations of microcomponents (ppm) in relation to $NiSO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$ in the mother solution, a'_r, and in the washed crystal, a'_k, determined by direct atomic absorption, permitted the calculation of homogeneous D values (Henderson - Kraèek, Khlopin) from the following relation:

$$D = \frac{a'_k}{a'_r}$$

D values were determined by 1 of the following 2 methods, which enabled homogeneous distribution of microcomponents during the crystallization (15): 1) isothermal decreasing of supersaturation during vigorous stirring and 2) long-time stirring of crushed crystals in their saturated solution.

With the decreased-supersaturation method, the effect of the initial microcomponent concentration on D values was established at 25 ± 1°C. The equilibrium distribution coefficients, D_{eq}, were determined by the second method of stirring crushed crystals. The equilibrium was reached when a ratio of the microcomponent in crystal and in solution exceeded the expected value of its equilibrium coefficient

$$D^{o}_{max} = \frac{a'_{ko}\text{ (``contaminated'' crystal)}}{a'_{ro}\text{ (``purified'' solution)}}$$

or from a ratio lower than the expected value

$$D^{o}_{min} = \frac{a'_{ko}\text{ (``contaminated'' crystal)}}{a'_{ro}\text{ (``purified'' solution)}}$$

When the values of D^{o}_{max} and D^{o}_{min} were selected, the highest and the lowest values of D obtained during crystallization of $NiSO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$ by the decreased-saturation method (no. 1) were taken into consideration. The experiments were conducted as follows:

1. Determining the effect of initial concentration of the microcomponents M^{2+} (Fe^{2+}, Mg^{2+}, Co^{2+}, Mn^{2+}, Zn^{2+}, Cd^{2+}, Ca^{2+}, and Cu^{2+}) on their distribution coefficients when crystallizing $NiSO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$ at 25 ± 1°C:

The supersaturated[1] nickel (II) and ammonium sulfate solutions containing different initial concentrations of microcomponents (a'_o), after addition of hydrazine sulfate solution to avoid oxidation of Fe(II) to Fe(III), were placed in beakers, covered with a watch glass, and stirred at approximately 300 rpm for 6 hours at 25 ± 1°C. The crystals obtained were filtered and washed with pure saturated nickel (II) and ammonium sulfate solution. The effects of the initial concentration on the D values and their average values (D_{av}) in the investigated concentration range are shown in Fig. 1.

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[1] The degree of supersaturation was such that the degree of crystallization, α = (macrocomponent in the solid)/(macrocomponent in the system), amounted to 0.2–0.6. Results from the preliminary investigations show that α (in this range) does not affect D values.

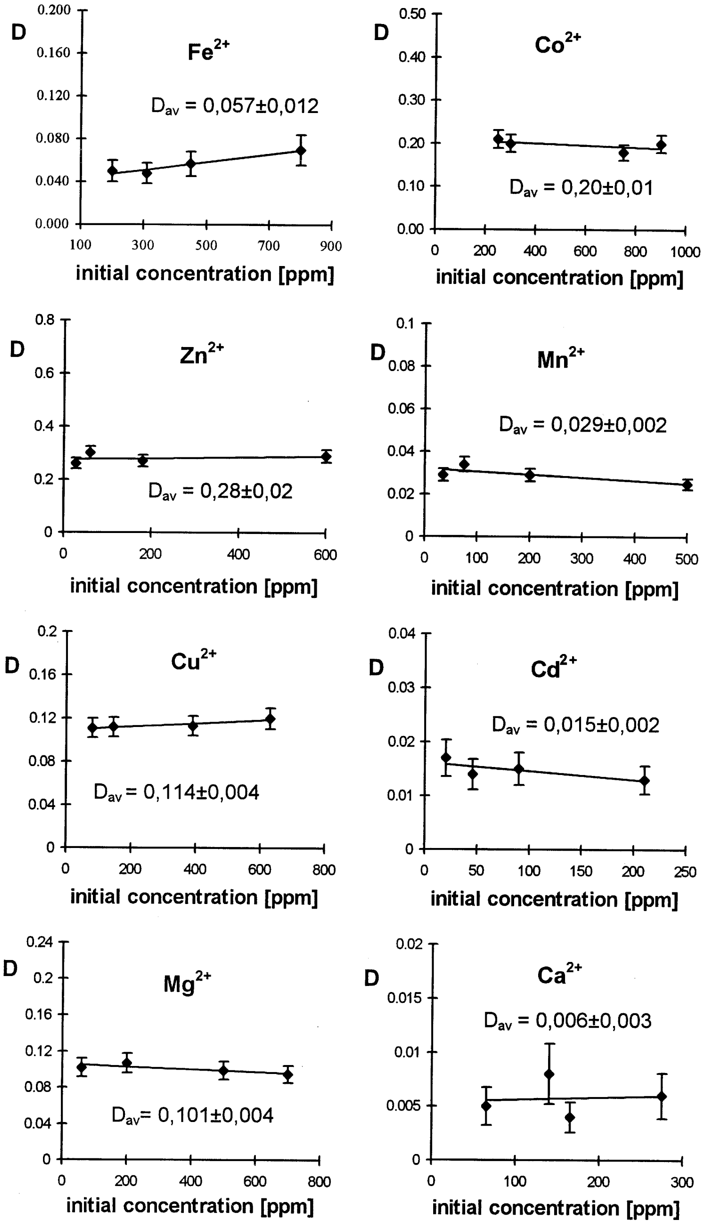

Figure 1. Effect of microcomponents (M^{2+}) concentration on their distribution coefficients, *D*, during $NiSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ crystallization at 25 ± 1°C.

Table 1. Average Distribution Coefficients, D, of Microcomponents M^{2+} During Long-Time Stirring of Crushed Crystals $NiSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ in Their Saturated Solution at 23 ± 1°C.

	$D^0 = \frac{a_{k'o}}{a_{r'o}}$		$\overline{D} + t_{0.05}\frac{s}{\sqrt{n}}$		
Microcomponent	D^o_{max}	D^o_{min}	for D^o_{max}	for D^o_{min}	Average Equilibrium Distribution Coefficient
Fe	0.19	0.035	0.056 ± 0.017	0.045 ± 0.004	0.050 ± 0.01
Co	0.30	0.13	0.20 ± 0.02	0.20 ± 0.03	0.20 ± 0.02
Zn	0.64	0.20	0.25 ± 0.07	0.23± 0.02	0.24 ± 0.03
Mn	0.097	0.011	0.017 ± 0.009	0.013 ± 0.002	0.015 ± 0.004
Cu	0.39	0.054	0.120 ± 0.034	0.085 ± 0.015	0.103 ± 0.020
Cd	0.047	0.006	0.012 ± 0.003	0.009 ± 0.002	0.010 ± 0.002
Mg	0.36	0.039	0.070 ± 0.021	0.051 ± 0.006	0.060 ± 0.008

2. Determination of D_{eq} of the microcomponents M^{2+} (Fe^{2+}, Mn^{2+}, Co^{2+}, Zn^{2+}, Mg^{2+}, Cd^{2+}, and Cu^{2+}) at 23 ± 1°C: Crushed contaminated $NiSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ crystals ($\phi < 0.1$ mm) were introduced into several beakers together with saturated purified nickel (II) and ammonium sulfate solution. Crushed crystals of purified $NiSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ ($\phi < 0.1$ mm) and a contaminated, saturated solution of nickel (II) and ammonium sulfate were introduced to other beakers. Contents of the beakers were stirred for approximately 410 hours with a magnetic stirrer that had been placed in a closed room. Fluctuations of temperature facilitated recrystallization of $NiSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ and helped attain equilibrium and an homogeneous partition of microcomponents in the crystal (16). The mean temperature value was equal to 23 ± 1°C. While stirring was continued, $N_2H_4 \cdot H_2SO_4$ was added in small portions (100 μl of saturated solution) to avoid oxidation of iron (II). The results are presented in Table 1.

RESULTS AND DISCUSSION

The influence of initial microcomponent concentration (within the investigated ranges) on the cocrystallization coefficients of Zn^{2+}, Cu^{2+}, Mg^{2+}, Co^{2+}, and Ca^{2+} is insignificant. With respect to Mn^{2+} and Cd^{2+}, D values decreased slightly, particularly at the lowest initial concentrations, and in the case of Fe^{2+}, D values increased with the increase of initial ion concentration (Fig. 1).

The distribution coefficients of $M^{2+}\{Co^{2+}$, Fe^{2+}, Zn^{2+}, Cu^{2+}, Mg^{2+}, Mn^{2+}, and $Cd^{2+}\}$ for 2 series D^o_{max} and D^o_{min} did not differ from each other, which means that the equilibrium condition was reached for them, and average values for both series were equilibrium values (Table 1). Average D values obtained for different initial concentrations of microcomponents at 25°C (Fig. 1) were very close to the equilibrium coefficients at 23 ± 1°C for Cd^{2+}, Co^{2+}, Fe^{2+}, Cu^{2+}, and Zn^{2+}. Only in the case of Mg^{2+} and Mn^{2+} were they are significantly higher than D_{eq}, which means that they may not be the equilibrium values.[2]

The determined distribution coefficients of microcomponents generally differ from those obtained during cocrystallization of macroquantities of $M^{II}SO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ with $NiSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ (7–12): In the case of Zn^{2+}, the D values for microquantities were very similar to the values for macroquantities; for Co^{2+}, Fe^{2+}, Mn^2, and Mg^{2+} the D values for microquantities are lower than those for macroquantities; and for Cu^{2+}, they are higher than those obtained for macroquantities (9) but lower than those obtained for microquantities (720 ppm) (10).

The dependencies of the cocrystallization coefficients D_{av} of the microcomponents M^{2+} with $(NH_4)_2SO_4 \cdot NiSO_4 \cdot 6H_2O$ on the ionic radius (17) are presented in Fig. 2. The values of D_{av} increase monotonically from r_{Mg2+} to r_{Zn2+},

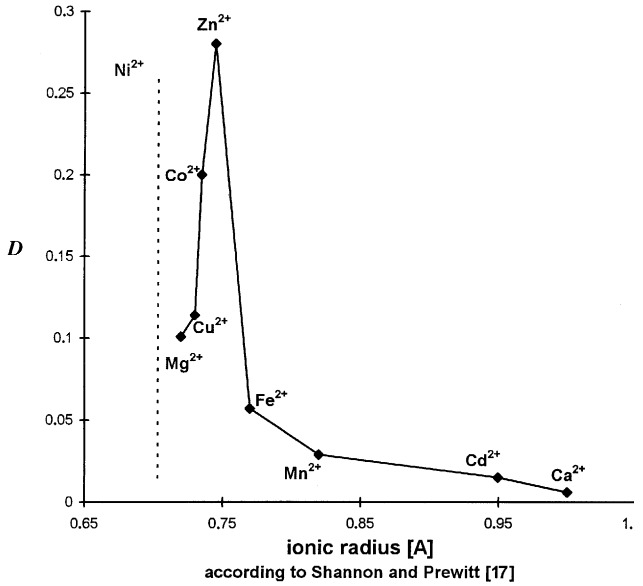

Figure 2. Dependence of the average distribution coefficients, D_{av}, of microamounts of M^{2+} during the crystallization of $NiSO \cdot (NH_4)_2SO_4 \cdot 6H_2O$ at 25 ± 1°C on their ionic radii.

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[2] To a considerable lower degree, a different temperature in both series is responsible for these differences in D. The relationship between D and temperature can be characterized by calculating values of D at 23 and 25°C.

where they achieve the maximum, and with further increase of the ionic radius, D_{av} drops monotonically to minimum values at r_{Ca2+}. However, the maximum of D_{av} does not appear for the r_{M2+} value closest to r_{Ni2+} as often had been found in other crystallization systems (14).

The dependence of D_{eq} of the microcomponents M^{2+} on the solubility ratio C_M/C_{Ni} (where C_M and C_{Ni} are molar solubilities in water of $(NH_4)_2SO_4{\cdot}M^{II}$ $SO_4{\cdot}6H_2O$ and $(NH_4)_2SO_4{\cdot}NiSO_4{\cdot}6H_2O$, respectively (18–19)) is presented in a logarithmic system in Fig. 3. This dependence is depicted by a linear relationship with a coefficient of linear correlation close to–1($r_{xy} = -0.9847$) and the slope is

$$\frac{d \log D_M}{d \log \left(\dfrac{C_M}{C_{Ni}}\right)} = -2.66$$

The close dependence of D_{eq} values of these microcomponents on the solubility ratio was used in an attempt to determine molar solubilities C of $NiSO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$ and $M^{II}SO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$ salts (calculated on the basis of the estimated densities of respective saturated solutions)(18–19). The solubility data was used in the following formula derived by Hill et al. (9), which is based on the assumption that the activity coefficients of Ni^{2+} and of a respective M^{2+} in

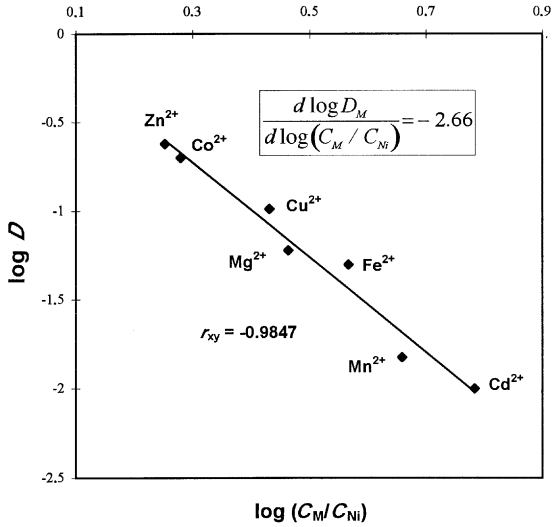

Figure 3. Dependence of the equilibrium distribution coefficients (log D_{eq}) of microamounts of M^{2+} during the crystallization of $NiSO_4{\cdot}(NH_4)_2SO_4{\cdot}6H_2O$ at 23 ± 1°C on the molar solubility ratio (log C_M/C_{Ni}).

Table 2. Comparison of Determined and Calculated Coefficients, *D*, of M^{2+} Microcomponents Cocrystallization with $NiSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$

	D at 23°C	
Microcomponent	Calculated	Determined
Zn^{2+}	0.23	0.24 ± 0.03
Co^{2+}	0.19	0.20 ± 0.02
Cu^{2+}	0.081	0.103 ± 0.020
Mg^{2+}	0.067	0.060 ± 0.008
Fe^{2+}	0.037	0.050 ± 0.010
Mn^{2+}	0.022	0.015 ± 0.004
Cd^{2+}	0.010	0.010 ± 0.002

the solid phase are equal:

$$\log D_M = 2.5(\log C_{Ni} - \log C_M)$$

The values of *D* calculated and determined at 23°C are presented in Table 2. As indicated by the results, many of the *D* values calculated and determined experimentally are in good agreement.

CONCLUSION

D values of the microcomponents M^{2+} during the crystallization of $(NH_4)_2SO_4 \cdot NiSO_4 \cdot 6H_2O$ are not considerably influenced by the ionic radii of the microcomponents (maximum *D* for the values r_{micr} closest to r_{macr} was not found). The run to find the dependence $D = f(r_{micr})$ is more complicated because the solubility influences of respective double salts overlapped, in particular, $ZnSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ and $CoSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ (Fig. 2).

D values of the microcomponents M^{2+} during the crystallization of $(NH_4)_2SO_4 \cdot NiSO_4 \cdot 6H_2O$ evidently depend on the solubility *C* (mol/L) of corresponding salt. The diagram in the system $\log D = f(\log C)$ exhibits a linear character, with the coefficient of linear correlation r_{xy} close to -1 and slope

$$\frac{d \log D_M}{d \log \left(\dfrac{C_M}{C_{Ni}}\right)} = -2.66$$

The value estimation of *D* for these microcomponents is based on the solubility of double salts of the macro- and microcomponents. The *D* values calculated in this way are in many cases in good agreement with the experimental values.

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