INLAND WATERS BRANCH
DEPARTMENT OF ENERGY, MINES AND RESOURCES

Prediction of Saturation Precipitation of Low Solubility Inorganic Salts from Subsurface Waters under Conditions of Total Concentration, Temperature and Pressure

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TECHNICAL BULLETIN NO.30

CANADA

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OTTAWA, CANADA, 1970

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Abstract

Graphs are presented that enable the determination of the degree of (under- or over-) saturation of aqueous solutions with respect to $BaSO_4$, $CaSO_4$, $SrSO_4$, BaF_2, CaF_2 and MgF_2 under a variety of conditions of temperature, pressure and total-salt concentration. Also presented are examples of the influence of temperature changes, dilution, evaporation, addition of non-common salt, and mixing, on the degree of saturation of the above solutions.

Prediction of Saturation Precipitation of Low Solubility Inorganic Salts from Subsurface Waters under Conditions of Total Concentration, Temperature and Pressure

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INTRODUCTION

Inorganic salts of low solubility in aqueous solutions may cause problems under a variety of circumstances during the production, handling, use and disposal of subsurface waters. Changes in temperature, pressure, gas content and total-salt concentration may result in increased or decreased solubility, giving rise to dissolution and corrosion, or to the formation of precipitates. Precipitates or "scale" may be formed in casing and tubing, either in wells or above ground, and also in the subsurface formations that produce or accept the water in question.

In principle, it is possible to use a chemical analysis to predict the behaviour of the low solubility salts in a water under changing conditions. Enough data are available on solubilities at atmospheric pressure and low temperatures to enable a reasonable confidence in such predictions, especially if the total salt concentration in the water is low. For solutions with an ionic strength not exceeding 0.1 molal (moles per 1000 grams of H_2O), standard thermodynamic calculations, making use of the Debye-Hückel equation, may give good results (for example, Hanshaw *et al.*, 1965; van Everdingen, 1969). However, data are insufficient for practical treatment of these problems at temperatures that differ appreciably from 25°C. For high total-salt concentrations, the results of the Debye-Hückel equation for the calculation of activity coefficients become increasingly unreliable, chiefly because of the increasing formation of complex ions in such concentrated solutions.

The above considerations have led to the construction of a set of solubility graphs that should enable a rapid graphical determination of the approximate degree of saturation of a solution with respect to a number of inorganic salts of low solubility. The procedures described should be regarded as an attempt to improve on the method described by Stiff and Davis (1952) for the prediction of calcium-sulfate precipitation, at the same time extending the method to other slightly soluble sulfates and fluorides.

The graphs presented here (Figures 3-5) were derived from solubility data available in the literature. Although the range of application is limited in the case of some of the salts, it is felt that even these graphs of limited applicability may be useful for rapid approximations, until more data become available.

In the following sections a description of the theoretical background for the graphs and conversion scales is presented to enable evaluation of the method. Next, the practical use of the graphs and scales is explained, and a number of possible applications are given. A few examples will be discussed to illustrate the method.

THEORETICAL BACKGROUND AND BASIC DATA

The method described here for the prediction of probable precipitation of salts of low solubility from an aqueous solution is based on the use of molal solubility products K'_{sp}, derived from published solubility data. The molal solubility product K'_{sp}, for a salt in a saturated aqueous solution is given by

$$K'_{sp} = [\text{cation}]_S^c \cdot [\text{anion}]_S^d \qquad .....(1)$$

where $[\text{cation}]_S$ and $[\text{anion}]_S$ are the molal concentrations (moles per 1000 gram H_2O) of the cation and the anion of the particular salt in a saturated solution of that salt at a particular temperature, and c and d are the number of cations and anions, respectively, formed by the dissociation of one molecule of the salt (for a salt like CaF_2, c = 1 and d = 2).

For a solution containing both the cation and the anion of a salt of low solubility the ion-concentration product Q' is given by

$$Q' = [\text{cation}]^c \cdot [\text{anion}]^d \qquad .....(2)$$

If the solution is undersaturated with respect to the salt studied, the value of Q' will be smaller than K'_{sp}; if the solution is saturated, Q' equals K'_{sp}; if the solution is supersaturated under the conditions studied, Q' will be larger than K'_{sp}.

For more convenient graphical interpretations, the decadic logarithms of K'_{sp} and Q' are used in the method described here. On the solubility graphs (Figures 3-5), values of

$$\log K'_{sp} = c \log [\text{cations}]_S + d \log [\text{anion}]_S \qquad .....(3)$$

were plotted versus temperature (in degrees centigrade) for various values of total-salt concentration (expressed as ionic strength in moles/1000 grams H_2O). Most of the curves for ionic strength above 0.5 molal are based on solubilities of the various salts in aqueous NaCl solutions.

Data for the solubility of gypsum for the region between 10° and 100°C were taken from Linke (1958) and Power *et al.* (1966).

Data for the solubility of anhydrite between 50° and 300°C, for pressures of 1, 100 and 500 bars, and for NaCl concentrations up to 6 molal, were derived from Dickson *et al.* (1963), Marshall and Slusher (1968), and Blount and Dickson (1969).

Solubility data for $BaSO_4$ between 25° and 95°C, for NaCl concentrations up to 4.5 molal were taken from Templeton (1960); those for $BaSO_4$ in H_2O between 0° and 100°C, from Linke (1958).

Data for the solubility of $SrSO_4$ and for BaF_2, CaF_2, MgF_2 and SrF_2 were taken from Linke (1958). Where conflicting data were found, usually the lower of the solubilities has been adopted for the present method.

In addition to indicating the degree of saturation of a salt in water under fixed conditions, the graphs also permit a rapid check of the effects of a change in solution temperature; the effects of evaporation at constant temperature and the addition of distilled water (both of which change the

total ionic strength of the solution as well as the ion-product for the salt of interest); the effect of adding a salt that has no ions in common with the salt under study (a change in total ionic strength only); and the effects of mixing two solutions that each contain one or both of the ions making up a slightly soluble salt.

GRAPHICAL DETERMINATION OF SATURATION DEGREES

For the proper application of the method, it is necessary to convert concentrations given in the analysis in ppm (parts per million = grams/10^6 grams solution), epm (equivalents per million = gram-equivalents/10^6 grams solution) and mg/l (milligrams per 1000 cm^3 solution) into molal concentrations, by using one of the equations listed in the Appendix.

The logarithms of the molal concentrations for both cation and anion are combined to derive log Q' from

$$\log Q' = c \log [\text{cation}] + d \log [\text{anion}] \qquad .....(4)$$

The value of log Q' for the salt in question is entered on the ordinate of the solubility graph for the salt in question (see example in Figure 1).

Next the ionic strength, I, of the water is calculated from the molal concentrations, m_i, of all the individual ions, from

$$I = 1/2\ m_a z_a^2 + m_b z_b^2 + m_c z_c^2 + ... + m_x z_x^2 \qquad .....(5)$$

where z is the valency of the ions.

The saturation degree of the solution with respect to the salt in question can now be determined. First a horizontal line is drawn from the log Q' point on the ordinate in Figure 1 until it intersects the vertical line corresponding to the temperature, T_w, of the solution. The point of intersection may lie above or below the curve representing the solubility of the salt for the ionic strength, I, of the solution. If the intersect of log Q' and T_w falls below the curve for I, then the solution is undersaturated; if the intersect falls above the curve for I, then the solution is supersaturated with respect to the salt under study.

The saturation temperature, T_s, for the solution in question is found by extending the horizontal line representing log Q' until it intersects the curve for the ionic strength, I, of the solution; the reading directly below this second intersection is the required temperature, T_s.

A numerical value for the relative amount of under- or oversaturation can be found by extending the vertical line representing the temperature, T_w, of the solution to where it intersects the curve representing the ionic strength, I, of the solution. A horizontal line from this point of intersection to the ordinate will give the value of log K'_{sp} for the T_w and ionic strength of the solution. Subtracting,

$$\log S = \log Q' - \log K'_{sp} \qquad .....(6)$$

gives the degree of undersaturation (log S<0) or supersaturation (log S>0).

FURTHER APPLICATIONS

The procedure outlined above makes it possible to determine the effect of a change in solution temperature on the degree of saturation (Figure 1). The horizontal line from log Q' on the ordinate is in that case intersected with the vertical representing the new solution temperature, T_1. The position of the new point of intersection is compared in relation to the curve for I as before.

The solubility graphs also make it possible to assess the effect of adding salts that have no ions in common with the salt under study, for example, addition of NaCl to a solution in which the saturation degree for $BaSO_4$ is being investigated. To do this, the position of the point of intersection (log Q', T_w) is analyzed, not in relation to the curve representing the original ionic strength, I, of the solution, but in relation to the curve representing the new ionic strength, I_1, which results from adding the extra salt. The ionic strength for the new total-salt concentration must be calculated using equation (5) and the new individual ion concentrations.

Furthermore, it is possible to determine the effects of dilution with distilled water on the one hand, and evaporation at constant temperature on the other. Both the ionic strength, I, of the solution and the individual ion concentrations of the salt under study will be affected by either of these modifications. It is necessary to introduce a factor, called f_d in this report, which indicates the fractional decrease in concentration (f_d smaller than 1.0) or the fractional increase in concentration (f_d larger than 1.0) that results from dilution or evaporation, respectively.

After dilution or evaporation, the new ionic strength, I_2, of the solution is found by multiplying the original ionic strength, I, and the factor, f_d, in the formula

$$I_2 = I \times f_d \qquad \ldots\ldots(7)$$

The modified individual ion concentrations are found by multiplying the original concentrations and f_d,

$$\text{for example, } [\text{cation}]_2 = [\text{cation}] \times f_d \qquad \ldots\ldots(8)$$

And for the logarithm of the new ion-concentration product log Q_2', we find from equations (4) and (8):

$$\log Q_2' = c \log [\text{cation}] + d \log [\text{anion}] + (c + d) \log f_d$$

$$= \log Q' + (c + d) \log f_d \qquad \ldots\ldots(9)$$

Values for (c + d) log f_d are given in Table 1 for various values of f_d between 0.1 (equal to 10 times dilution) and 5.0 (equal to 5 times increase in concentration), for (c + d) = 2 and (c + d) = 3. For intermediate values of f_d not given in Table I, the values of (c + d) log f_d can easily be calculated.

Supersaturation and subsequent precipitation of a salt of low solubility may result from the mixing of two aqueous solutions, for example, in brine disposal, artificial recharge and water drive projects when one of the solutions contains the cation (or both ions) and the other solution contains the anion (or both ions) of the salt of low solubility. The behaviour of low-solubility salts in various mixtures of two solutions can be

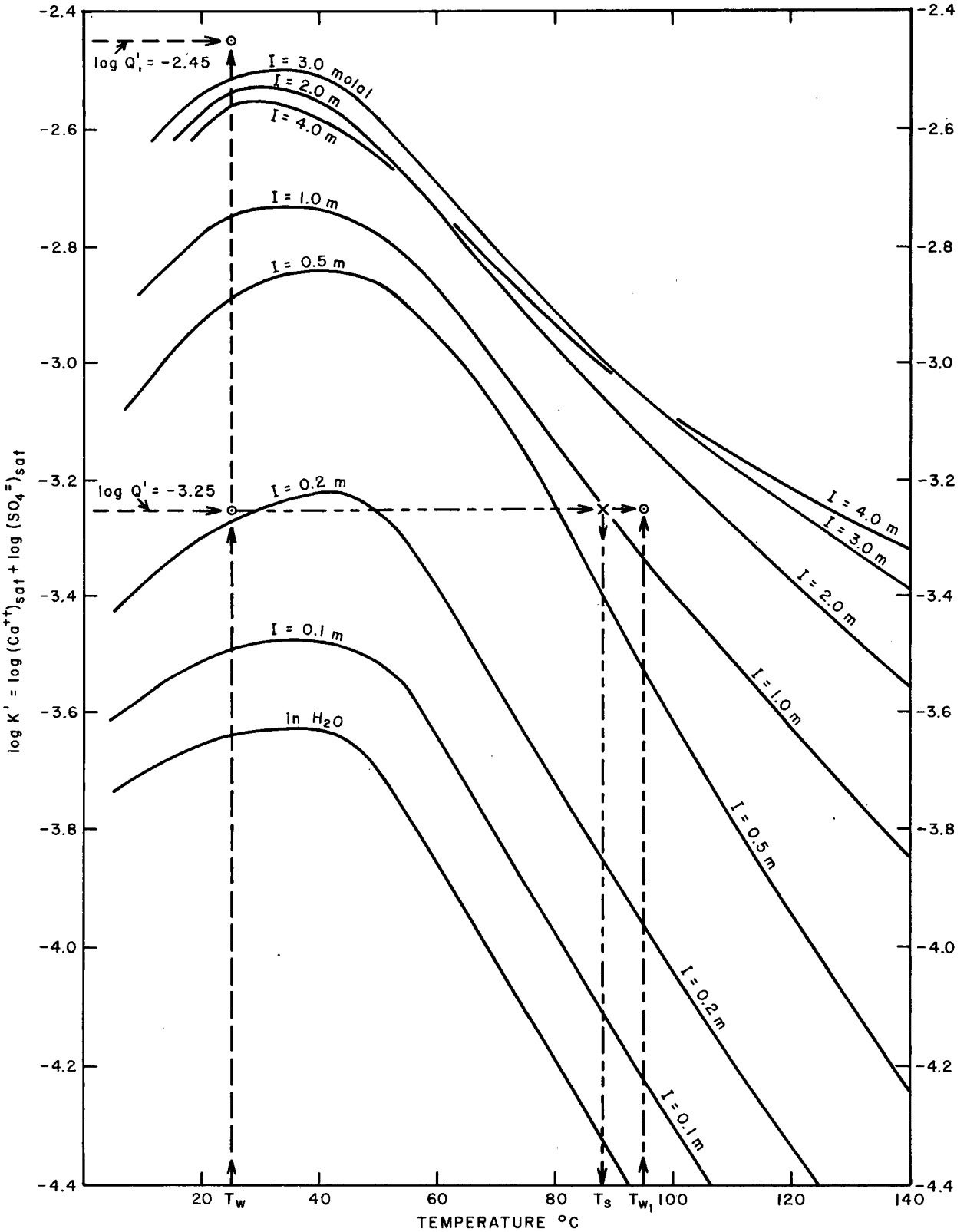

Figure 1. Log K'_{CaSO_4} vs. temperature, in water and NaCl solutions of increasing ionic strength. (Example only, not to be used for actual determinations).

TABLE 1

Values of (c+d) log f_d for various values of f_d.

	(c+d) = 2 (Sulfates of Ba^{++}, Ca^{++} and Sr^{++})	(c+d) = 3 (Fluorides of Ba^{++}, Ca^{++} and Mg^{++})
f_d	(c+d) log f_d	(c+d) log f_d
Dilution		
0.9	-0.0915	-0.1374
0.8	-0.1938	-0.2907
0.75	-0.2500	-0.3747
0.7	-0.3098	-0.4647
0.6	-0.4437	-0.6654
0.5	-0.6021	-0.9030
0.4	-0.7959	-1.1937
0.3	-1.0458	-1.5687
0.25	-1.2041	-1.8063
0.2	-1.3979	-2.0970
0.1	-2.0000	-3.0000
Evaporation		
1.1	+0.0828	+0.1242
1.2	+0.1584	+0.2376
1.3	+0.2279	+0.3417
1.4	+0.3923	+0.4383
1.5	+0.3522	+0.5283
1.6	+0.4082	+0.6123
1.7	+0.4609	+0.6912
1.8	+0.5105	+0.7659
1.9	+0.5575	+0.8364
2.0	+0.6021	+0.9030
2.5	+0.7959	+1.1937
3.0	+0.9542	+1.4313
3.5	+1.0881	+1.6323
4.0	+1.2041	+1.8063
4.5	+1.3064	+1.9596
5.0	+1.3979	+2.0970

predicted in principle through the above graphical method, with the use of an additional graphic determination (Figure 2). In Figure 2 the ionic strength of solution A, and the molal concentrations for the ions of interest in solution A, are plotted along the left hand edge (100% A); those for solution B are plotted along the right hand edge (100% B). To increase the accuracy for low concentrations, the list of scale factors on Figure 2 is provided to enable the use of different graphical scales for different components. One should be careful, however, to use the same scale (same scale factor) for the same component on both sides of the mixing graph. For example ionic strength was plotted in Figure 2 using scale 2; $[Ca^{++}]$ and $[SO_4^{=}]$ were plotted using scale 3.

Lines are drawn to connect the A and B points for each of the components and for the ionic strength. The ionic strength and the molal concentrations of the ions of interest can now be read off for any ratio of mixing of the two solutions. When reading the values for any mixture, one should be careful to keep in mind the proper scale to be used. For this purpose the scale used for each component could be marked on the connecting lines in Figure 2. The molal concentrations of the ions of interest, determined for a mixture of two solutions by the use of the mixing graph (Figure 2), should be converted to logarithms. The logarithm of the ion-concentration product, log Q', is found in the usual way from equation (4) and plotted on the solubility graph for the salt in question (example in Figure 1); the saturation degree is determined as described earlier. Through application of this procedure to various mixing ratios of the same two solutions, it is possible to determine those ratios of mixing (if any) that will result in a mixture that is saturated with the salt studied at the temperature used.

EXAMPLES

Degree of Saturation

Column 1 of Table 2 gives the analysis of a saline water, expressed in ppm. Column 2 gives the ionic weights for use in the conversion to molalities. Using equation (10) from the Appendix, the molalities were calculated; the resulting values are given in column 3. Column 4 lists the individual ion valencies to be used for the calculation of the ionic strength, I. The products $m \times z^2$ are given in column 5. Addition of the values in column 5 and division by 2 gives the value of the ionic strength of the solution, I = 1.0 molal.

For the determination of the degree of saturation of this saline water with respect to $CaSO_4$, one converts the molal concentrations $[Ca^{++}]$ and $[SO_4^{=}]$ into the logarithms of these concentrations:

$$[Ca^{++}] = 0.014 = 1.4 \times 10^{-2};\ \log [Ca^{++}] = -1.854$$

$$[SO_4^{=}] = 0.040 - 4.0 \times 10^{-2};\ \log [SO_4^{=}] = -1.398$$

$$\log Q' = -3.252 = -3.25$$

Putting this value for log Q' on the solubility graph (Figure 1) and drawing a horizontal line from the log Q' point and a vertical line from the temperature (T_W = 25°C), one finds that this solution, having an ionic strength of 1.0 molal, is undersaturated. The saturation temperature, T_S, is found by

SCALE NO.	VALUE OF SINGLE SUBDIVISION	MAX. VALUE
1	1×10^{-1}	1.5×10
2	1×10^{-2}	1.5
3	1×10^{-3}	1.5×10^{-1}
4	1×10^{-4}	1.5×10^{-2}
5	1×10^{-5}	1.5×10^{-3}
6	1×10^{-6}	1.5×10^{-4}

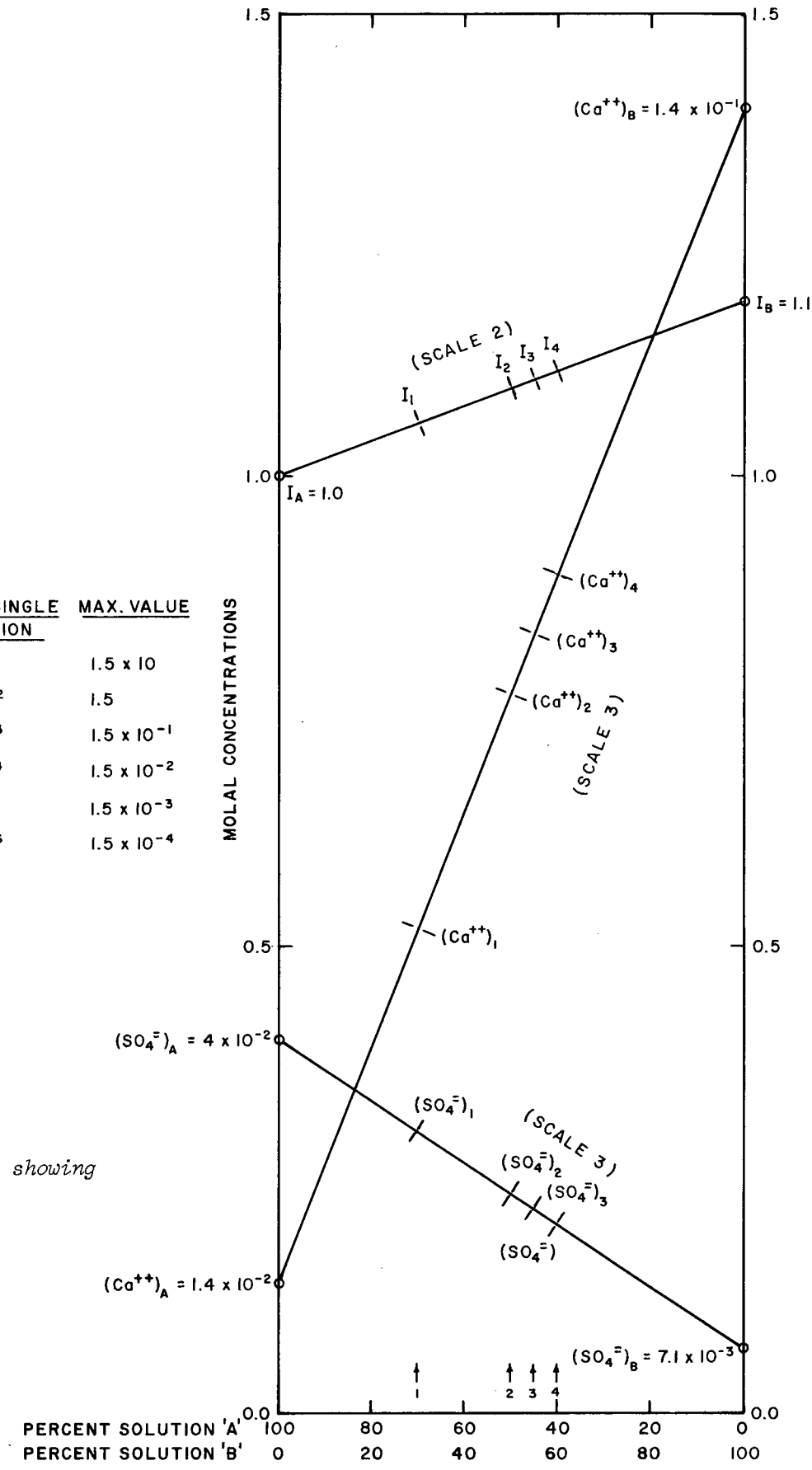

Figure 2. Mixing graph, showing example for $CaSO_4$.

TABLE 2

Chemical analysis of water A.

Ion	Concentration, ppm	Ionic weight W	Molality, moles/10^3g H_2O	z	m x z^2
Na^+	14,880	23.00	0.68	1	0.68
Mg^{++}	1,620	24.32	0.070	2	0.28
Ca^{++}	530	40.08	0.014	2	0.056
K^+	520	39.10	0.014	1	0.014
Cl^-	27,000	35.46	0.80	1	0.80
$SO_4^=$	3,650	96.06	0.040	2	0.16
HCO_3^-	200	61.02	0.0034	1	0.0034
					1.9934
TDS	48,400			I =	1.00

TABLE 3

Chemical analysis of water B.

Ion	Concentration, ppm	Ionic weight W	Molality moles/10^3g H_2O	z	m x z^2
Na^+	14,880	23.00	0.68	1	0.68
Mg^{++}	1,620	24.32	0.070	2	0.28
Ca^{++}	5,300	40.08	0.14	2	0.56
K^+	520	39.10	0.014	1	0.014
Cl^-	27,000	35.46	0.80	1	0.80
$SO_4^=$	648	96.06	0.0071	2	0.0284
HCO_3^-	200	61.02	0.0034	1	0.0034
					2.3658
TDS	50,168			I =	1.18

extending the horizontal line from log Q' until it intersects the curve for I = 1.0 molal; the reading vertically below the intersection point is the required temperature (in the example it is T_s = 88°C). The ionic strength for which this water would be saturated with respect to $CaSO_4$ at 25°C is approximately 0.22 molal.

Influence of a Change in Temperature and of the Addition of a "Non-Common" Salt

If the solution from the foregoing example is heated to T_1 = 95°C, the solubility of $CaSO_4$ decreases sufficiently to make the solution supersaturated with respect to $CaSO_4$. Addition of non-common salt to increase the ionic strength to approximately 1.50 molal would sufficiently increase the solubility of $CaSO_4$ to prevent the solution from becoming supersaturated with respect to $CaSO_4$ at the new temperature, 95°C.

Evaporation

Loss of water through evaporation at constant temperature (T_w = 25°C) will increase the ionic strength and also the individual ion concentrations. Loss of 60 percent of the water ($f_d = \frac{100}{40} = 2.5$) will result in the ionic strength becoming

$$I_1 = f_d \times I = 2.5 \times I = 2.5 \text{ molal}.$$

At the same time, the ion concentrations $[Ca^{++}]$ and $[SO_4^{=}]$ increase, giving a new ion-concentration product in the form

$$\log Q_1' = \log Q' + 2 \log f_d.$$

Taking the proper value for 2 log f_d from Table 1 gives

$$\log Q_1' = -3.25 + 0.80 \rightarrow -2.45$$

Plotting this new value on Figure 1 and drawing the horizontal line to intersect with the 25°C vertical indicates the solution now has become saturated with respect to $CaSO_4$. Neither heating nor the addition of salt will increase the solubility to prevent this supersaturation. Precipitation can only be prevented by adding distilled water or a solution with a lower total-salt concentration that contains no Ca^{++} or $SO_4^{=}$ ions.

Mixing

The degree of saturation of various mixtures of solution A (Table 2) and solution B (Table 3) can be determined as follows (see Figures 2 and 4). Ionic strengths and the $[Ca^{++}]$ and $[SO_4^{=}]$ concentrations are plotted on the left- and right-hand sides of Figure 2 for solutions A and B, respectively.

If one takes a mixture containing 70 percent solution A and 30 percent solution B, the ionic concentrations are found to be:

$$[Ca^{++}]_1 = 5.33 \times 10^{-2} \rightarrow \log [Ca^{++}]_1 = -1.2733$$

$$[SO_4^{=}]_1 = 3.02 \times 10^{-2} \rightarrow \log [SO_4^{=}]_1 = -1.5200$$

$$\log Q_1' = -2.7933; \; I_1 = 1.055$$

Various other mixtures, illustrated in Figure 2 give the following results:

For 50 percent solution A and 50 percent solution B:

$$[Ca^{++}]_2 = 7.68 \times 10^{-2} \rightarrow \log [Ca^{++}]_2 = -1.1146$$

$$[SO_4^{=}]_2 = 2.35 \times 10^{-2} \rightarrow \log [SO_4^{=}]_2 = -1.6289$$

$$\log Q_2' = -2.7435; \quad I_2 = 1.090$$

For 45 percent solution A and 55 percent solution B:

$$[Ca^{++}]_3 = 8.32 \times 10^{-2} \rightarrow \log [Ca^{++}]_3 = -1.0799$$

$$[SO_4^{=}]_3 = 2.20 \times 10^{-2} \rightarrow \log [SO_4^{=}]_3 = -1.6576$$

$$\log Q_3' = -2.7375; \quad I_3 = 1.099$$

For 40 percent solution A and 60 percent solution B:

$$[Ca^{++}]_4 = 8.97 \times 10^{-2} \rightarrow \log [Ca^{++}]_4 = -1.0472$$

$$[SO_4^{=}]_4 = 2.03 \times 10^{-2} \rightarrow \log [SO_4^{=}]_4 = -1.6925$$

$$\log Q_4' = -2.7397; \quad I_4 = 1.108$$

The mixtures in these examples are all undersaturated, even though the 45/55 mixture is very close to saturation. All mixtures investigated here have a higher degree of saturation than either of the original two solutions.

APPENDIX

To convert ppm into molality:

$$m = \frac{C_p}{\left(1000 - \frac{TDS}{1000}\right) \cdot W} \qquad .....(10)$$

where m = molality, moles of solute/1000 grams H_2O

C_p = concentration of ion, in ppm

TDS = total dissolved solids, in ppm

W = gram-formula weight of ion, in grams

To convert epm into molality:

$$m = \frac{C_e}{z\left(1000 - \frac{TDS}{1000}\right)} \qquad .....(11)$$

where C_e = concentration of ion, in epm

z = valency of ion

Ostroff (1965, p. 372) unnecessarily included specific gravity in this conversion.

To convert from mg/liter to molality:

$$m = \frac{C_m}{\left(1000\ d - \frac{TDS}{1000}\right) \cdot W} \qquad .....(12)$$

and from gram/liter to molality:

$$m = \frac{C_g \times 10^3}{(1000\ d - TDS) \cdot W} \qquad .....(13)$$

where C_m = concentration of ion, in mg/liter solution

C_g = concentration of ion, in gram/liter solution

d = density of solution, in grams/cm^3

For the conversion from milligrams- or grams per liter to molality, it is necessary to know the density of the solution. In the case of oil-field waters, a determination of "d" is usually included in the standard analysis. For subsurface waters with total solids concentrations of less than 5000 ppm, only a small error is introduced by disregarding the influence of density on the molal concentration values.

In routine applications the above conversions could easily be processed on a digital computer; in that case it would be logical to include the conversion to logarithms of the molal concentrations, as well as the calculation of the log Q' values. However, if only a small number of samples are to be checked for their degree of saturation, graphical conversion to logarithms could be done by slide rule.

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A report on the hydrogeological conditions to the north of Toronto with a view to determining if the terrestrial water balance of the Lake Ontario Basin is affected by a major seepage from Lake Simcoe.

No. 24 Application of regression analysis in hydrology. N. Tywoniuk and K. Wiebe, 1970.

A description of the applications (and limitations) of regression analysis and a discussion of the distinction between regression - and correlation analysis.

No. 25 Stream gauging techniques for remote areas using portable equipment. M. Church and R. Kellerhals, 1970.

A review of streamflow measuring techniques applicable to rivers with peak flows up to 10,000 cfs.

No. 26 The control of eutrophication. Canada Centre for Inland Waters, Burlington, Ontario; Fisheries Research Board of Canada, Winnipeg; Inland Waters Branch, Ottawa, 1970.

A discussion of the respective roles of phosphorus, nitrogen and carbon as critical elements in limiting the eutrophication process.

No. 27 An automated method for determining mercury in water. P.D. Goulden and B.K. Afghan, 1970.

A report describing a method for determining the mercury content in water containing mercury concentrations as low as 0.05 µg/l.

No. 28. An assessment of the wave agitation in the small boat basin at the Canada Centre for Inland Waters. T.M. Dick, 1970.

A discussion of the results obtained from a model study of wave action in the small boat basin at the Canada Centre for Inland Waters, Burlington, Ontario.

No. 29 Measurement of discharge under ice conditions. P.W. Strilaeff and J.H. Wedel, 1970.

An outline of the difficulties encountered in the measurement of discharge under ice cover, and a discussion of a possible technique for estimating river discharge using a single velocity in a cross-section.

A complete list of titles in the Technical Bulletin Series and copies of any of these publications may be obtained from the Director, Inland Waters Branch, Department of Energy, Mines and Resources, Ottawa, Ontario.

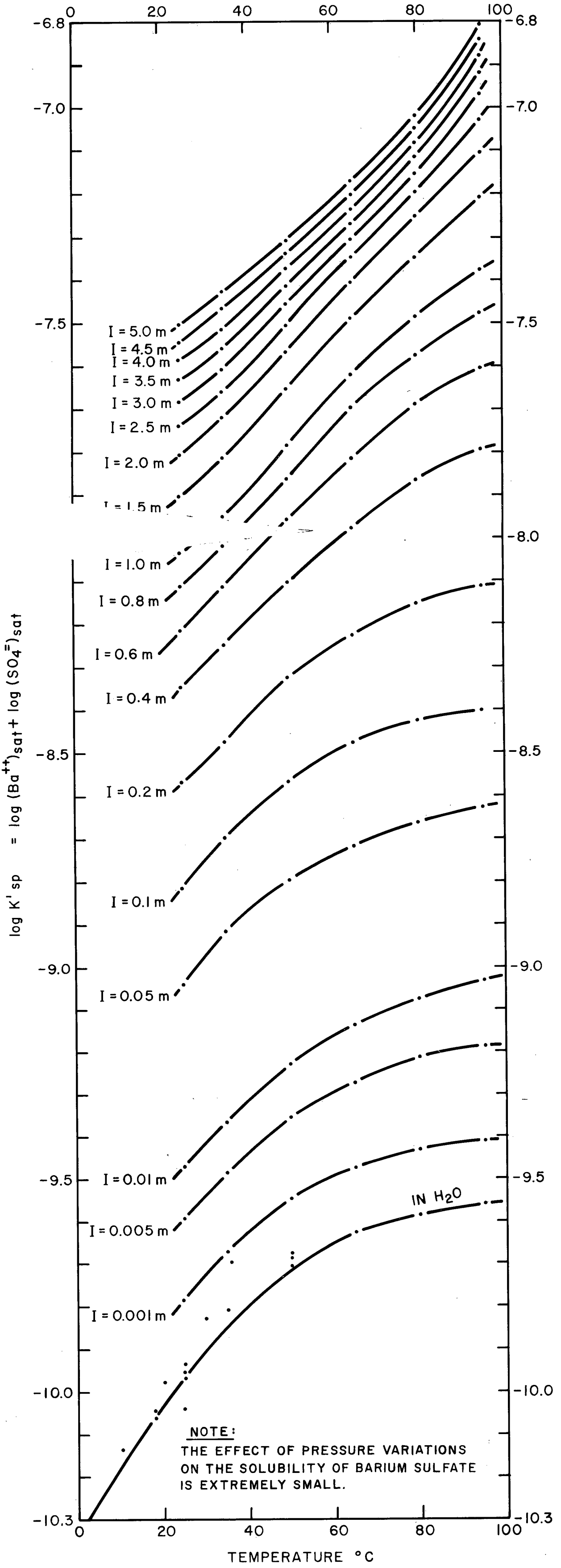

Figure 3. Log K'_{BaSO_4} vs. temperature, in water and NaCl solutions of increasing ionic strength.

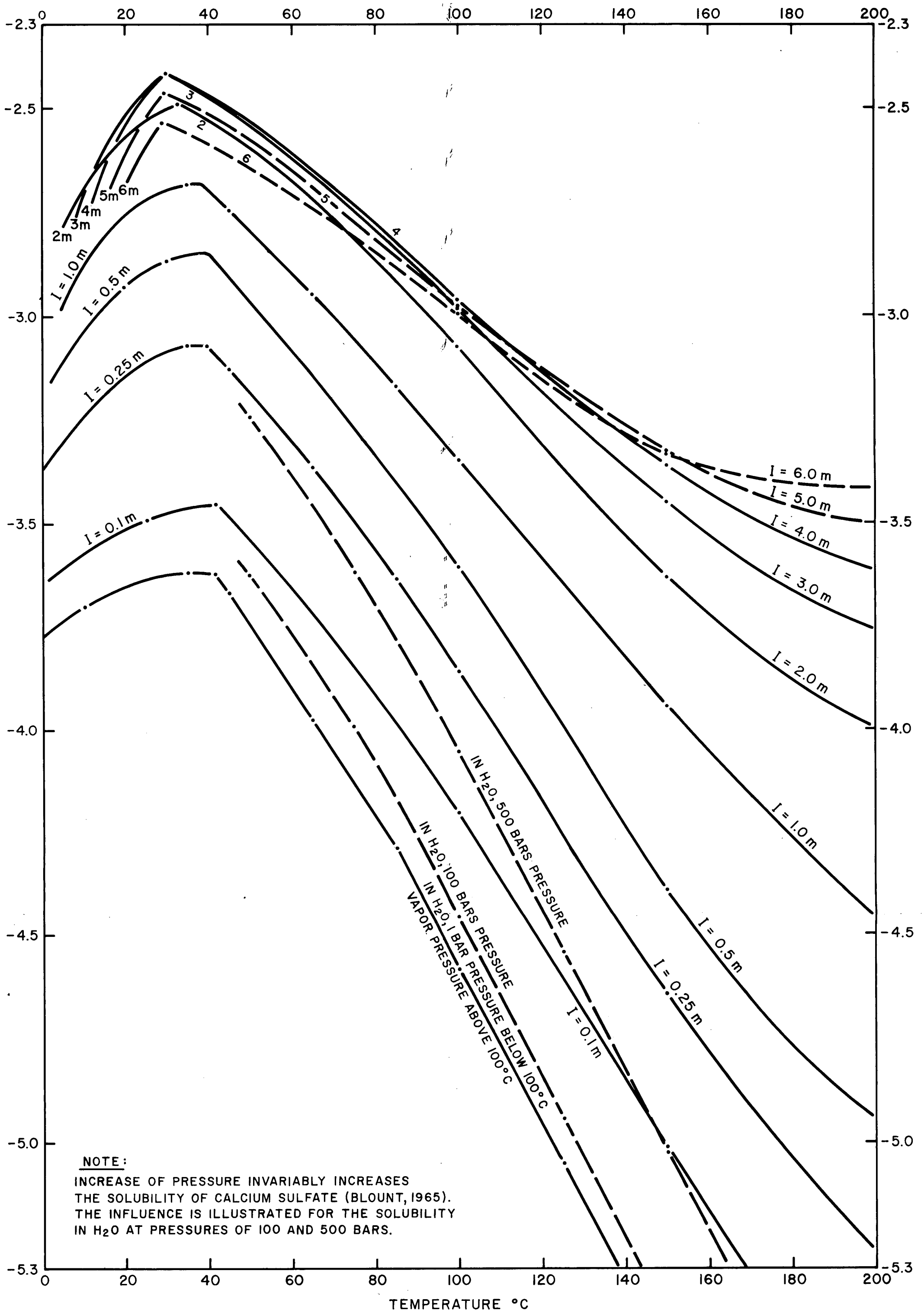

Figure 4. Log K'_{CaSO_4} vs. temperature, in water and NaCl solutions of increasing ionic strength.

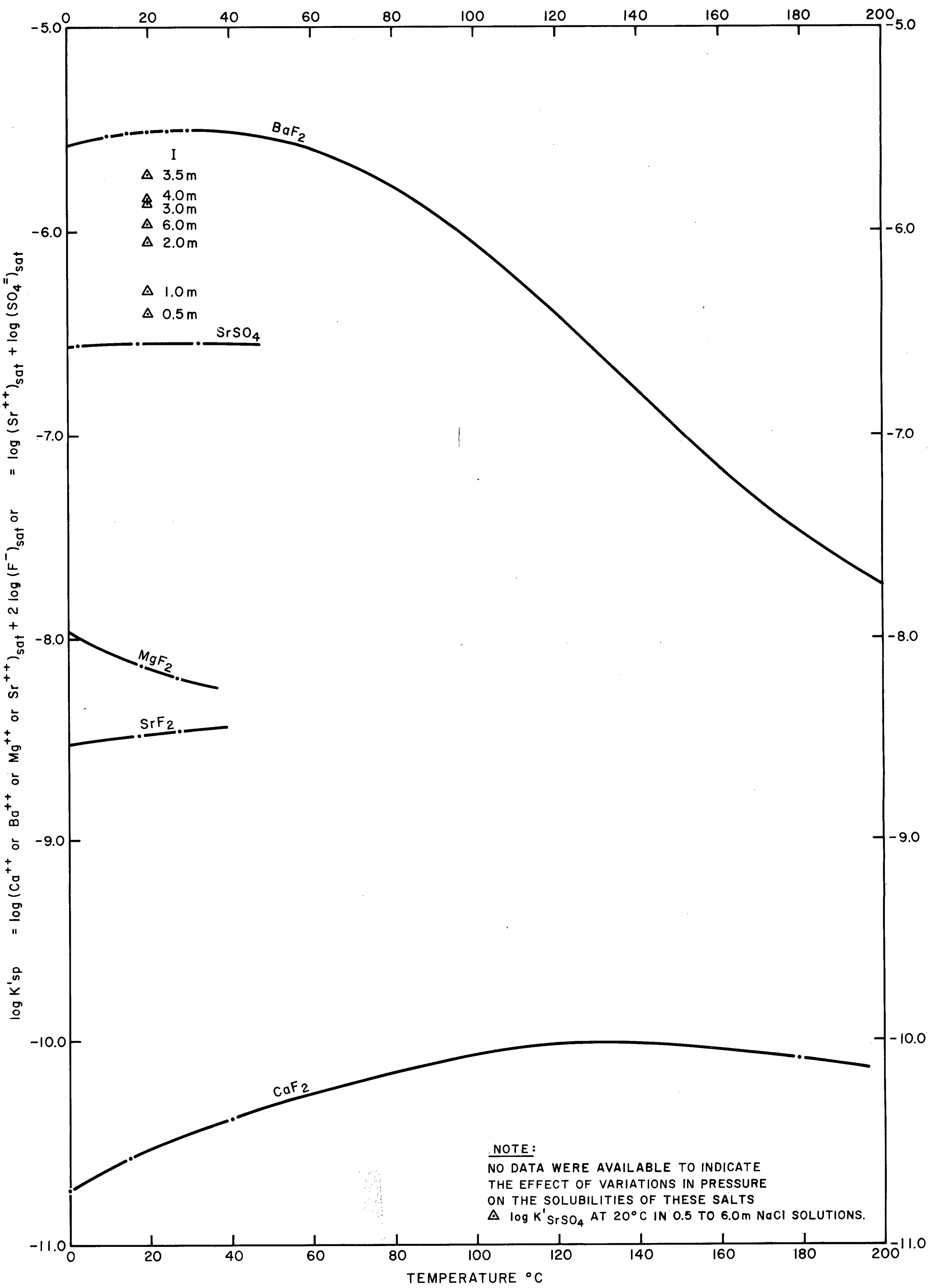

Figure 5. Log K' in water vs. temperature, for BaF_2, CaF_2, MgF_2, SrF_2 and $SrSO_4$.