CCA-336

541.18.041:546.791

Original Scientific Paper

Precipitation and Hydrolysis of Uranium(VI) in Aqueous Solutions. II. Uranyl Nitrate — Sodium Carbonate — Alkaline Earth Chlorides*

*H. Füredi*** *and B. Težak*

*Department of Physical Chemistry, Institute »Ruđer Bošković« and Laboratory of Physical Chemistry, Faculty of Science, Zagreb, Croatia, Yugoslavia****

Received April 17, 1964

The precipitation systems: uranyl nitrate ($2\times10^{-3}N$) — sodium carbonate — Me chlorides (Me = Ba, Sr, Ca, Mg) were investigated and compared. The corresponding precipitation diagrams were constructed by plotting the percentage of uranium precipitated as a function of the concentrations of sodium carbonate and alkaline earth chloride.

Solid phase formation starts in all systems at sodium carbonate concentration of $1.6\times10^{-3}N$ and $p\text{H}\sim 5$, when a critical concentration of the particular alkaline earth chloride, necessary for precipitation, is exceeded. A precipitation maximum with more than 80% of uranium precipitated occurs at $3\times10^{-3}N$ of sodium carbonate and $p\text{H}\sim 6$. At high concentrations of both sodium carbonate and alkaline earth chloride uranium is coprecipitated with the corresponding alkaline earth carbonate. The coprecipitation regions are shifted to higher concentrations of the precipitating components in the normal order of alkaline earth ions. A region with less than 15% of uranium precipitated separates the regions of precipitation and coprecipitation of uranium in the presence of strontium, calcium and magnesium chloride. With barium chloride in solution, this minimum is replaced by a »transitional« precipitation region with 55 — 95% of uranium precipitated. When sodium carbonate is present in concentrations higher than the concentrations of alkaline earth chloride soluble complex, uranyl carbonates prevail in all systems.

INTRODUCTION

Investigations were carried out in our laboratory in order to determine the influence of the change in concentrations of various inorganic and organic electrolytes on the hydrolysis of metal ions and their precipitation from aqueous solutions[1-8]. The chemical behaviour of uranium(VI) in carbonate solutions seems to be of general interest[14].

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[*] Presented in part at the National Symposium on Nuclear Fuels, Radovljica, Yugoslavia, April 20 — 25, 1961, and at the Yugoslav-Italian Symposium on Uranium Technology and Metallurgy, Herceg Novi, September 20 — 28, 1961.

[**] Part of the Ph. D. Thesis of H. Füredi, Faculty of Science, University of Zagreb, 1963.

[***] Contribution No. 109. from the Laboratory of Physical Chemistry, Faculty of Science, Zagreb.

The ability of the uranyl ion to form stable, soluble carbonate complexes[9-16] has been widely used in carbonate leaching of uranium ores[17-21]. The carbonate circuit has been recommended especially for uranium bearing limestones and other high lime ores.[20] However, with the exception of our results previously published[2,3], no data could be found on the interaction of carbonate solutions with those containing uranium(VI) and alkaline earth ions. Investigations were therefore carried out in order to obtain an overall picture of the condititons of precipitation and dissolution of uranium in the precipitation systems uranyl nitrate — sodium carbonate — alkaline earth chlorides.

The results of these investigations are a contribution to our experience with complicated multicomponent precipitation systems. Besides, they can probably give valuable information on the optimal conditions of carbonate leaching of uranium in the presence of alkaline earths.

In this paper the influence of changes in concentration of two of the precipitating components (sodium carbonate and alkaline earth chlorides) at a constant concentration of the third (uranyl nitrate) on the precipitation of uranium, are shown by means of precipitation bodies[3,22] and the corresponding contour diagrams (groundplans[7,8,23,24]). General characteristics of the precipitation systems are discussed and compared.

EXPERIMENTAL

All solutions were prepared with analytical grade chemicals and bidistilled water. The concentrations of standard solutions of alkaline, alkaline earth, and yttrium chlorides were determined by the ion exchange (Dowex — 50 X-8, 50/100 mesh) technique[25]. The equivalent quantity of H^+ ions was titrated with 0.1 N NaOH. The standard solution of uranium (2N uranyl nitrate) was analysed gravimetrically with 8-oxyquinoline[26]. The precipitation systems were prepared by mixing equal volumes of solutions of the precipitating components in the usual experimental technique[3,27]. The concentrations are expressed as the final values after mixing. For the determination of the percentage of uranium precipitated the systems were prepared in polypropylene centrifuge tubes and after mixing allowed to stand overnight at room temperature. Afterwards they were centrifuged in a Servall Superspeed centrifuge for 30 minutes at 15.000 r. p. m. An aliquot of the clear mother liquor with a few drops of $HClO_4$ was evaporated to dryness and the uranium content determined spectrophotometrically[28].

Turbidimetric measurements were performed in standard test tubes with a Zeiss tyndallometer in connection with a Pulfrich photometer. The solutions were kept at constant temperature of 20 ± 0.1^{0} C. The measurements of optical density were carried out with a Hilger Watts spectrophotometer »Uvispek«. The pH was determined with a glass and a calomel electrode using a Pye pH-meter.

RESULTS

The percentage of uranium precipitated was determined in a series of experiments and plotted (z — axis) as a function of variable concentrations of barium chloride ($1N — 3 \times 10^{-4}N$) strontium chloride ($1N — 3 \times 10^{-4}N$), calcium chloride ($1N — 6 \times 10^{-4}N$) or magnesium chloride ($1N — 2 \times 10^{-3}N$) (x — axis) and sodium carbonate ($1N — 1 \times 10^{-3}N$) (y — axis).

Three-dimensional precipitation diagrams were thus obtained, two of them made as perspex bodies. On the corresponding contour diagrams (groundplans) the results are shown in the plane of concentrations of alkaline earth chloride (apscissa) and sodium carbonate (ordinate). The precipitation regions, numbered from 1 to 10, indicate a 10% difference in uranium precipitated. The regions with more than 85% of uranium precipitated are shaded.

Fig. 1 is a photograph of the model of the precipitation body of the system with barium chloride in solution. It was constructed as a correction of the precipitation body previously reported[3], both the old and the new results being used.

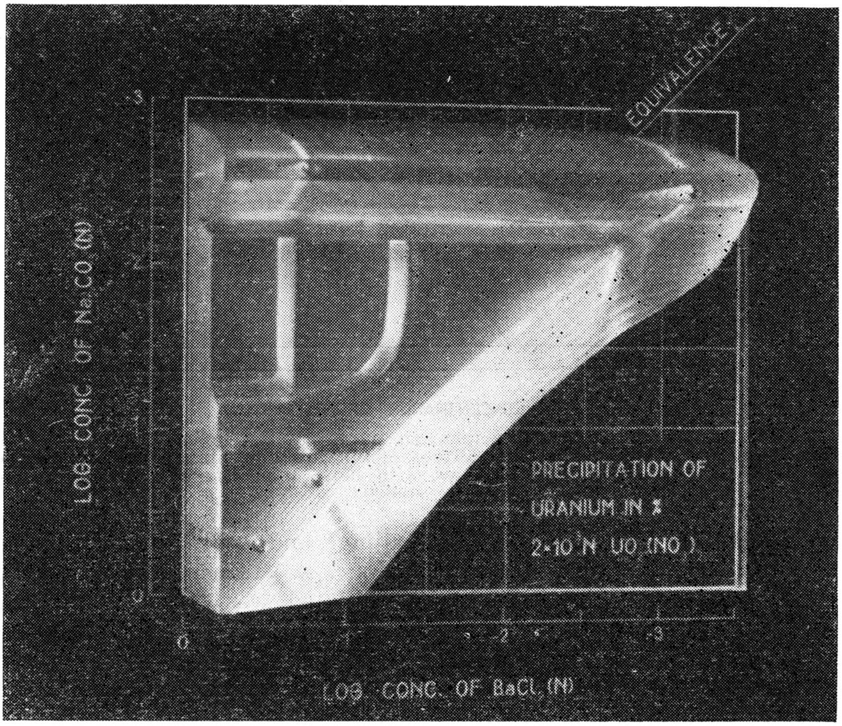

Fig. 1 A photograph of the precipitation body of the system uranyl nitrate ($2 \times 10^{-3}N$) — sodium carbonate-barium chloride. The percentage of precipitated uranium (z — axis) is represented as a function of the concentration of barium chloride (x — axis) and sodium carbonate (y — axis).

In Fig. 2 the corresponding contour diagram of the same system is shown.

The boundary regions, where the percentage of uranium precipitated rapidly decreased and therefore poor reproducibility of the results was obtained, are roughly numbered from 2 to 8 (15 to 85% of uranium precipitated).

In general, two precipitation regions could be observed in the precipitation system, separated by a minimum in the uranium precipitation (25 to 75% of uranium precipitated) at about $6 \times 10^{-3}N$ of sodium carbonate. One of the precipitation maxima occured at 3 to $4 \times 10^{-3}N$ of sodium carbonate and was very sensitive to changes in sodium carbonate concentration. For this reason it might be called the »concentration maximum«. When the concentrations exceeded $1 \times 10^{-1}N$, more than 95% of uranium was precipitated. At such high concentrations of sodium carbonate and barium chloride copreci-

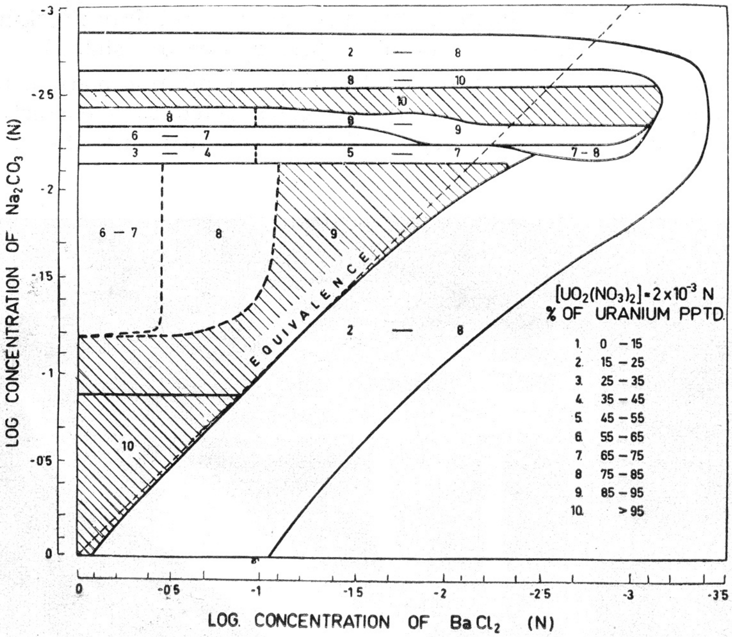

Fig. 2 The contour diagram of the precipitation system uranyl nitrate ($2 \times 10^{-3}N$) — sodium carbonate — barium chloride. The 10% differences in uranium precipitated are marked by figures 0 to 10. The isoprecipitating regions with more than 85% of uranium precipitated are shaded.

pitation processes were obviously prevailing[3] and consequently this region might be called the »coprecipitation region«. A »transitional« precipitation region with 55 to 95% of uranium precipitated was found to be imposed between the »coprecipitation region« and the minimum of uranium precipitation at $6 \times 10^{-3}N$ of sodium carbonate. In this region as well as in the precipitation minimum mentioned the percentage of uranium precipitated decreased with increasing concentration of barium chloride.

In Fig. 3 a photograph of the precipitation body of the system with strontium chloride in solution is shown. Fig. 4 represents the corresponding contour diagram of the same system.

As in the system with barium chloride, the main region of uranium precipitation was located at concentrations of sodium carbonate higher than 1.2 to $1.6 \times 10^{-3}N$ and with strontium chloride in excess (to the left of the the equivalence, Fig. 4). A region with less than 15% of uranium precipitated was observed when sodium carbonate was present in concentrations in excess of strontium chloride (to the right of the equivalence, Fig. 4). No solid phase was formed below approximately $1.2 \times 10^{-3}N$ of sodium carbonate and pH about 5.

The precipitation and the coprecipitation regions were much more separated from one another than was the case before (Figs. 1,2).

The »concentration maximum« appeared in the same range of sodium carbonate concentrations as in the presence of barium chloride, but the minimum of uranium precipitation extended from $6 \times 10^{-3}N$ to $1.2 \times 10^{-2}N$ of sodium carbonate. Regardless of the concentration of strontium chloride, less than 15% of uranium was precipitated in this region. At concentrations of both sodium carbonate and strontium chloride higher than $8 \times 10^{-2}N$, more than 95% of uranium was coprecipitated with strontium carbonate.

In Fig. 5 the contour diagram of the precipitation system with calcium chloride is shown. While the »concentration maximum« is similar in features to that observed with barium and strontium chloride in solution, the region of complex solubility of uranium is extended further but the coprecipitation region is much smaller. Poor reproducibility of the results, in the coprecipitation region is indicated in Fig. 5 by dotted contour lines.

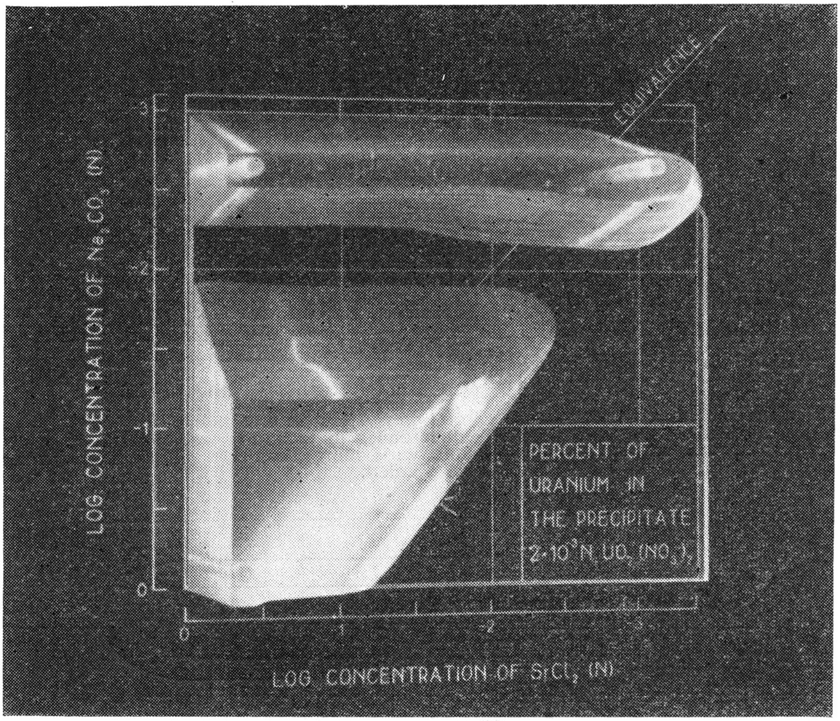

Fig. 3 A photograph of the precipitation body of the system uranyl nitrate ($2 \times 10^{-3}N$) — sodium carbonate — strontium chloride. The percentage of precipitated uranium (z — axis) is given as a function of the concentration of strontium chloride (x — axis) and sodium carbonate (y — axis).

Fig. 6 shows the contour diagram of the precipitation system with magnesium chloride.

The coprecipitation region has almost vanished giving way to the region of complex solubility. Even at very high concentrations of the precipitating components ($8 \times 10^{-1}N$) only about 60% of uranium was coprecipitated.

One of the characteristics common to the systems investigated is the strong dependence of the precipitation of uranium on the changes in the concentration of sodium carbonate and only a slight influence of changes in concentrations of alkaline earths. Thus the main features of each precipitation

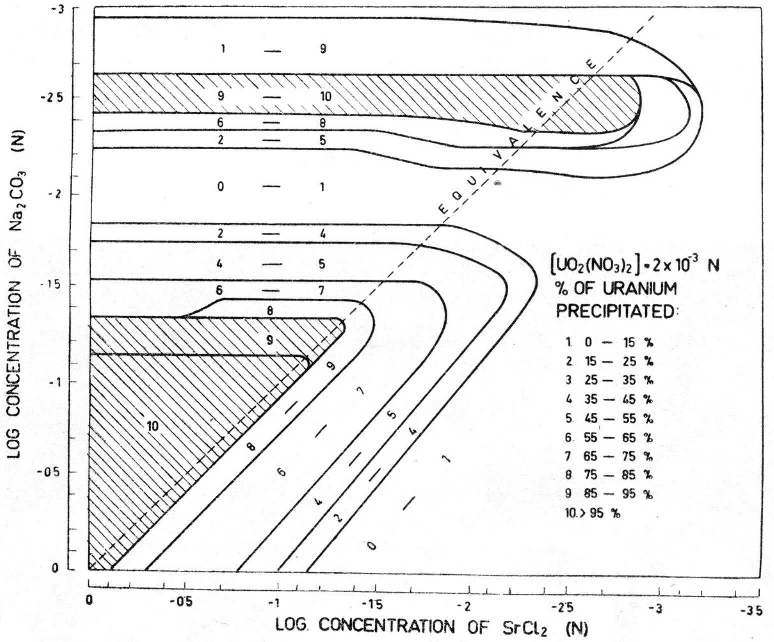

Fig. 4 The contour diagram of the precipitation system uranyl nitrate ($2 \times 10^{-3} N$) — sodium carbonate — strontium chloride. The 10% differences in uranium precipitated are marked by figures 0 to 10. The isoprecipitating regions with more than 85% of uranium precipitated are shaded.

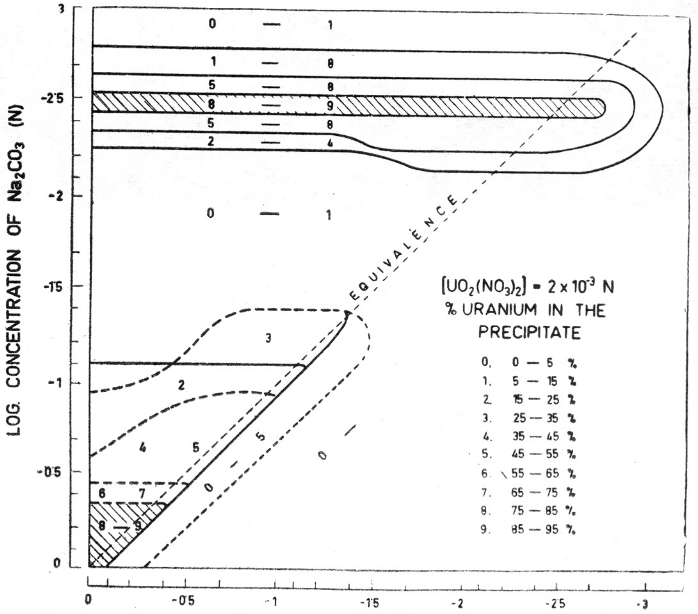

Fig. 5 The contour diagram of the precipitation system uranyl nitrate ($2 \times 10^{-3} N$) — sodium carbonate — calcium chloride. The 10% differences in uranium precipitated are marked by figures 0 to 9. The isoprecipitating regions with more than 85% of uranium precipitated are shaded. The regions with poor reproducibility of the results are designated by dotted contours.

system can be compared by means of properly chosen cross-sections of the precipitation bodies, as shown in Fig. 7.

In Fig. 7 the percentage of uranium precipitated and the pH values were plotted against the concentration of sodium carbonate at constant concentration of uranyl nitrate ($2 \times 10^{-3}N$) and alkaline earth chlorides ($1 \times 10^{-1}N$).

The precipitation of uranium began at about $1.6 \times 10^{-3}N$ of sodium carbonate and *p*H about 5, and the »concentration maximum« took place at $3 \times 10^{-3}N$ of sodium carbonate and *p*H about 6 in all the systems. In the presence of strontium, calcium and magnesium chloride respectively, at $6 \times 10^{-3}N$ of sodium carbonate started the region of sodium carbonate concentrations where less than 15% of uranium was precipitated. This minimum was extended and the coprecipitation of uranium reduced in the following order of alkaline earth ions: $Sr^{2+} < Ca^{2+} < Mg^{2+}$.

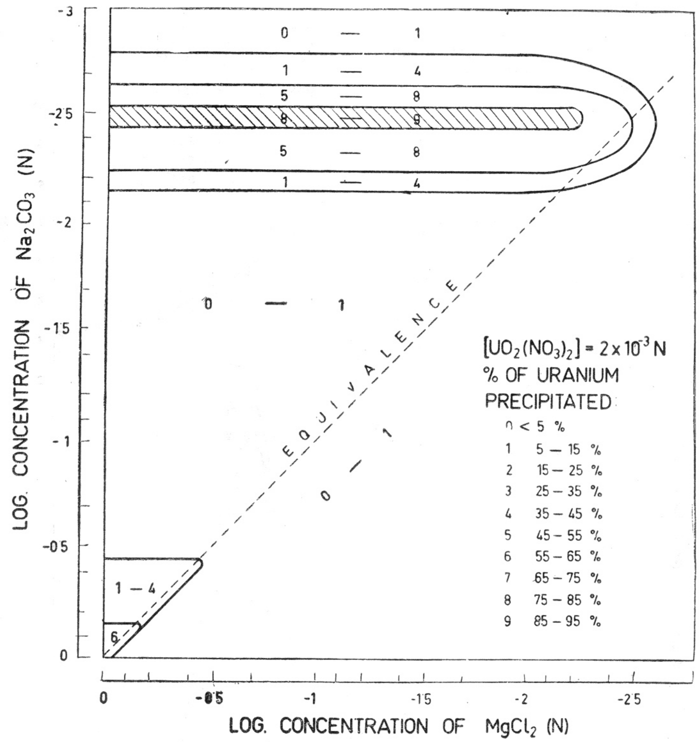

Fig. 6 The contour diagram of the precipitation system uranyl nitrate ($2 \times 10^{-3}N$) — sodium carbonate — magnesium chloride. The 10% differences in uranium precipitated are marked by figures 0 to 9. The isoprecipitating region with more than 85% of uranium precipitated is shaded.

With barium chloride in solution, about 50% of uranium was still precipitated at $6 \times 10^{-3}N$ of sodium carbonate. The »transitional« precipitation region (description of Figs. 1,2) occured at $8 \times 10^{-3}N$ to $6 \times 10^{-2}N$ of sodium carbonate where about 80% of uranium precipitated. At about $1 \times 10^{-1}N$ of sodium carbonate, uranium was completely coprecipitated with barium carbonate.

At concentrations of sodium carbonate in excess of alkaline earth chlorides (higher than 1 × $10^{-1}N$) the amount of uranium precipitated decreased in all the systems.

The pH data of the precipitation systems with barium, strontium and calcium chloride, respectively, fit in a common *p*H curve, which has two inflections separated by a wide range of sodium carbonate concentrations (6 × $10^{-3}N$ — 1 × $10^{-1}N$) at *p*H about 7. In the presence of magnesium chloride no region with constant *p*H value was obtained.

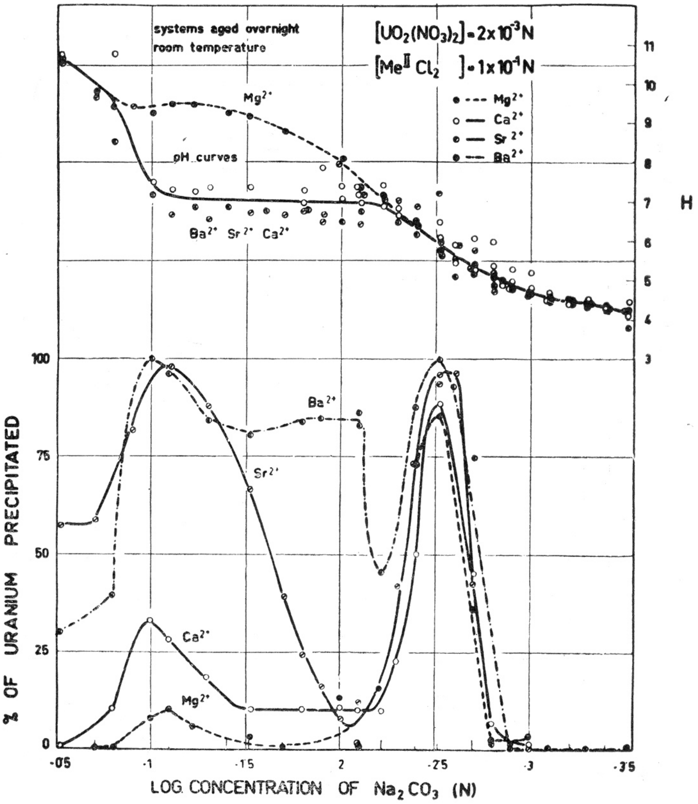

Fig. 7 Characteristic cross-sections of the precipitation body of the systems uranyl nitrate (2 × $10^{-3}N$) — sodium carbonate — alkaline earth chlorides. The percentage of uranium precipitated and the *p*H values are plotted against the concentration of sodium carbonate (2 × $10^{-3}N$) and alkaline earth chlorides (1 × $10^{-1}N$).

As already mentioned (description of Figs. 3 and 4) below 1.2 to 1.6 × $10^{-3}N$ of sodium carbonate and at pH about 5 no solid phase formed. The concentration boundary between the clear and the turbid systems was located by light absorption measurements[23].

In Fig. 8 the results obtained at 1 × $10^{-1}N$ of barium, strontium, calcium and magnesium chloride are shown. The absorbance at 425 mμ was plotted against the concentration of sodium carbonate. The limit between the clear and the turbid systems (marked with signs a and b in Fig. 8) corresponds to the limit of uranium precipitation (Fig. 7), and is independent of the kind

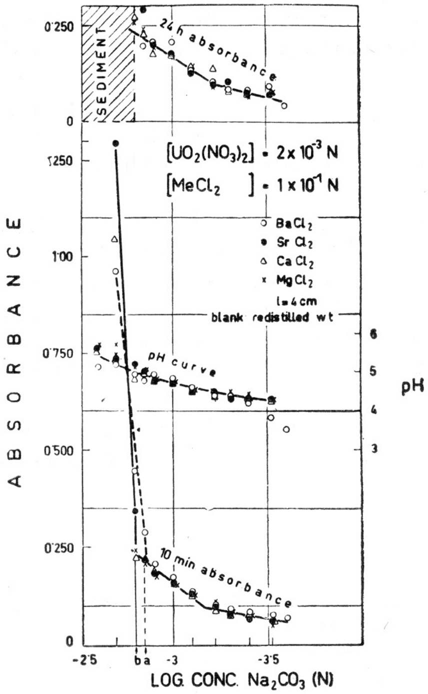

Fig. 8 Absorbance at 425 mμ, measured 10 minutes and 24 hours after mixing the systems, plotted against the concentration of sodium carbonate at constant concentration of uranyl nitrate ($2 \times 10^{-3}N$) and alkaline earth chlorides ($1 \times 10^{-1}N$). Letters a and b denote the concentrations of the last clear systems. The corresponding pH curve is given.

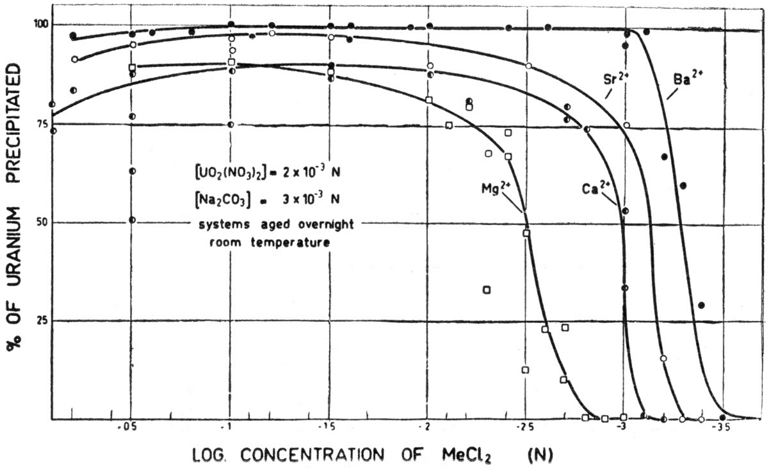

Fig. 9 Characteristic cross-section of the »concentration maximum« of the precipitation systems uranyl nitrate — sodium carbonate — alkaline earth chlorides. The percentage of uranium precipitated is plotted against the concentration of alkaline earth chlorides at constant concentration of uranyl nitrate ($2 \times 10^{-3}N$) and sodium carbonate ($3 \times 10^{-3}N$).

of alkaline earth chloride present in the solution. The precipitation boundary obtained 10 minutes after mixing the systems did not change during 24 hours (Fig. 8).

In Fig. 9 cross-sections of the »concentration maximum« of all systems investigated are given. The percentage of uranium precipitated is plotted against the concentration of alkaline earth chloride at constant concentration ($3 \times 10^{-3}N$) of sodium carbonate.

When a critical concentration of the particular alkaline earth ion was exceeded, the percentage of uranium precipitated suddenly increased approaching an almost constant value independent of further changes in the concentration of alkaline earth chloride. With calcium and magnesium chloride present, about 80 to 90% of uranium was precipitated, while with strontium and barium chloride in the solution the precipitation of uranium was almost complete (more than 95%).

In this region of sodium carbonate concentrations, the precipitation can be initiated by other electrolytes as well. The »precipitation values« of the

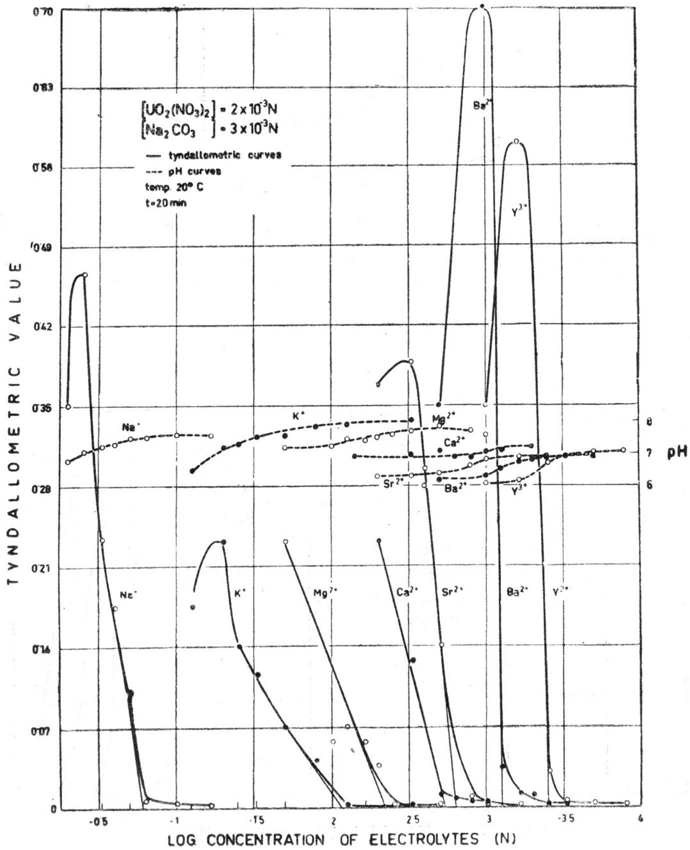

Fig. 10 Concentration tyndallograms measured 10 minutes and pH curves measured 24 hours after mixing of the systems uranyl nitrate $2 \times 10^{-3}N$ sodium carbonate $3 \times 10^{-3}N$, with varied concentrations of yttrium, barium, strontium, calcium, magnesium, potassium and sodium chlorides. The intercepts of the tangents on the steepest part of the tyndallometric curves with the abscissa denote the precipitation values of the electrolytes.

particular cations, indicating the critical concentrations necessary for precipitation, can be obtained by the method used for the evaluation of coagulation values of »neutral« electrolytes[6,27,30,31].

In Fig. 10 the results of the tyndallometric evalution of »precipitation values« of univalent (K^+, Na^+), bivalent (alkaline earths) and trivalent (Y^{3+}) chlorides at constant concentration of sodium carbonate ($3 \times 10^{-3} N$) are presented. The corresponding *p*H curves show an inflection and a decrease of *p*H as a result of precipitation.

DISCUSSION

Owing to its simplicity, the presentation of the precipitation systems by means of precipitation bodies and contour diagrams may be of practical importance. The relations of the concentrations of the precipitating components existing in the particular precipitation regions can be easily obtained from such diagrams. The general characteristics of the precipitation systems uranyl nitrate — sodium carbonate — alkaline earth chlorides will be compared by means of precipitation diagrams presented in this paper (Figs. 1—6).

The almost identical changes in the *p*H values as a consequence of changes in the concentration of sodium carbonate in the systems with barium, strontium and calcium chloride in solution (Fig. 7), indicate similar precipitation processes. The observed region with constant *p*H values is due to the buffering effect of the corresponding sparingly soluble alkaline earth carbonate present. The *p*H curve of the system uranyl nitrate ($2 \times 10^{-3} N$)-magnesium chloride ($1 \times 10^{-1} N$)-sodium carbonate differs from the *p*H curve of the other three precipitation systems investigated by the absence of a region with constant *p*H values. This difference can be attributed to the higher solubility of magnesium carbonate[31] or to the existence of soluble magnesium carbonate complexes[32,33].

When the concentration of the »precipitation value« of the corresponding alkaline earth ion was exceeded, a maximum of uranium precipitation, the »concentration maximum« occured in all the systems at the molar ratio 1.5 of sodium carbonate and uranyl nitrate ($3 \times 10^{-3} N$ of sodium carbonate at $2 \times 10^{-3} N$ of uranyl nitrate, Fig. 7) and *p*H about 6. Similar to alkaline earth ions other cations can also initiate precipitation in this region and the »precipitation values« obtained (Fig. 10) nearly agree with the corresponding values determined for the system uranyl nitrate ($2 \times 10^{-3} N$) — potassium hydroxide — neutral electrolytes[6], even though the latter were determined in the *p*H range from 5 to 6. A lowering of the *p*H as a consequence of precipitation was also observed in both cases (Fig. 10;[6]). Consequently the hydrolysis of the uranyl ion and the formation of mixed uranates should be considered as one of the possible mechanisms of the precipitation process in this region.

In the presence of high concentrations of both sodium carbonate and alkaline earth chlorides uranium was coprecipitated with the corresponding alkaline earth carbonate. The coprecipitation regions were shifted to higher concentrations of both precipitating components in the normal order of alkaline earths.

In regions where sodium carbonate was present in concentrations in excess of alkaline earth chlorides as well as in the precipitation minima, separating the »concentration maxima« and the coprecipitation regions in the presence of strontium, calcium and magnesium chloride, soluble complex uranyl di- and tricarbonates were formed and less than 15% of uranium was precipitated.

The precipitation boundary at approximately $1.2 \times 10^{-3} N$ of sodium carbonate and pH about 5 (Fig. 8) was due to the acidity of the solutions. The »transitional« region of the uranium precipitation occurring in the presence of barium chloride only (Figs. 1,2) was probably caused by the formation of sparingly soluble barium uranyl carbonates[34]. In the much simpler precipitation system uranyl nitrate — sodium carbonate[15,35] the solid phase is formed in a narrow concentration range only, with a maximum at the molar ratio 1 : 1 of the precipitating components and at *p*H about 4.5. Comparing this result with the results discussed above, it is evident that alkaline earth ions have a remarkable influence on the precipitation of uranium(VI) from carbonate solutions when present in concentrations higher than the concentration of sodium carbonate. The region of complex solubility of uranium is considerably reduced especially at high concentrations of the precipitating components (coprecipitation regions). This result should be taken into account when good yields of uranium are to be obtained by carbonate leaching procedures.

Acknowledgement. The authors wish to express their thanks to Dr. M. Branica for helpful discussions and to Dr. V. Pravdić for reading and discussing the manuscript. The skilful technical assistance of Miss M. Ivančić is also gratefully acknowledged.

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IZVOD

Taloženje i hidroliza urana (VI) u vodenim otopinama. II. Uranil nitrat — natrijev karbonat — kloridi alkalnih zemalja

H. Füredi i B. Težak

Ispitivani su i uspoređeni taložni sistemi uranil nitrat ($2 \times 10^{-3} N$) — natrijev karbonat — Me klorid (Me = Ba, Sr, Ca, Mg). Odgovarajući taložni dijagrami su konstruirani na taj način, da je postotak istaloženog urana prikazan kao funkcija koncentracija natrijeva karbonata i zemnoalkalijskih klorida.

Taloženje počinje u svim sistemima kod $1.6 \times 10^{-3} N$ natrijeva karbonata i pH ~ 5, kad je koncentracija odgovarajućega zemnoalkalijskoga klorida veća od kritične koncentracije potrebne za taloženje. Kod $3 \times 10^{-3} N$ natrijeva karbonata i *pH* ~ 6 nađen je taložni maksimum sa više od 80% istaloženog urana. Kod visokih koncentracija natrijeva karbonata i zemnoalkalijskih klorida uran koprecipitira s odgovarajućim zemnoalkalijskim karbonatom. Područja koprecipitacije se pomiču normalnim redom zemnoalkalija prema većim koncentracijama taložnih komponenata. Područje taloženja i područje koprecipitacije urana razdvojeni su u prisutnosti stroncijeva, kalcijeva i magnezijeva klorida taložnim minimumom sa manje od 15% istaloženog urana. Umjesto ovoga minimuma, uz barijev klorid pojavljuje se »prelazno« taložno područje sa 55—95% istaloženog urana. Kad su koncentracije natrijeva karbonata veće od koncentracija odgovarajućega zemnoalkalijskoga klorida, u sistemima prevladavaju topljivi kompleksni uranil karbonati.

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Primljeno 17. travnja 1964.