

CCA-1351

YU ISSN 0011-1643

UDC 546.42:541.48

Original Scientific Paper

Precipitation and Characterization of Strontium Phosphates

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Received February 15, 1982

Spontaneous precipitation of strontium phosphates was studied over a wide concentration range of strontium chloride ($2 \cdot 10^{-4}$ — $8 \cdot 10^{-1}$ mol dm⁻³) and of sodium phosphate ($4 \cdot 10^{-4}$ — $3 \cdot 10^{-1}$ mol dm⁻³) solutions. In all systems, initial pH was 7.4 and sodium chloride concentration was 0.15 mol dm⁻³. The precipitates aged for 1 day and 30 days at 298 K and 310 K were characterized by light microscopy and X-ray powder diffraction. Solid phases SrHPO₄ (α and β modifications), Sr₁₀(PO₄)₆(OH)₂, H₃Sr₆(PO₄)₅ · 2H₂O, and Na [Sr (H₂O)₉] PO₄ were determined. According to the precipitation boundary after 30 days (equilibrium conditions) the solubility product of Na [Sr (H₂O)₉] PO₄ is determined as follows: $\log ([Na^+] [Sr^{2+}] [PO_4^{3-}]) = -9.98 \pm 0.11$ (at $I = 0.25$ mol dm⁻³, 298 K).

INTRODUCTION

The investigation of the system strontium chloride — sodium phosphate — sodium chloride (0.15 mol dm⁻³), reported in this paper is a part of a general study on the spontaneous precipitation of the alkaline earth phosphates. An early phase rule study of the system SrO—P₂O₅—H₂O at 298 K in the acid region indicated the existence of two solid phases, Sr(H₂PO₄)₂ and SrHPO₄.¹ In the system SrCl₂—KH₂PO₄—H₂O ($6 < \text{pH} < 10$), SrHPO₄ and Sr₁₀(PO₄)₆(OH)₂ were detected.² SrHPO₄ may be precipitated from solutions in two different crystalline modifications (α and β), the formation of which depends on several conditions, temperature being the most critical.³

This paper deals with spontaneous precipitation in the system SrCl₂—H₃PO₄—NaOH—H₂O occurring under physiological conditions ($\text{NaCl} = 0.15$ mol dm⁻³, $\text{pH}_i = 7.4$) at 298 K and 310 K.

Investigation of precipitation phenomena of strontium phosphates is important in biological mineralization due to the isomorphous replacement of calcium by strontium in hydroxyapatite⁴ and the similar biological metabolism of these elements.⁵ The role of strontium in pathological calcification has been reported by several authors.⁶⁻⁹ It can produce rickets in bone⁶ and increase the bone abnormalities caused by scurvy.¹⁰ Its effect on caries has also been observed.¹¹ Low concentrations of strontium make easier the transformation of carbonate-containing hydroxyapatite (a constituent of bones and tooth enamel) into β -tricalcium phosphate by heat treatment.¹²

EXPERIMENTAL

Solutions were prepared by dissolving analytical grade chemicals in triply distilled water. Stock solutions of SrCl_2 and NaCl were standardized by ion exchange (Dowex 50-W resin). The concentration of H_3PO_4 was determined by titration, using thymolphthalein as indicator.¹³ The precipitates were prepared by mixing a SrCl_2 solution with an equal volume of a sodium phosphate solution ($\text{H}_3\text{PO}_4 + \text{NaOH}$, adjusted to a chosen initial pH, and NaCl to give a constant concentration of 0.30 mol dm^{-3}). The systems were aged for 1 day and 30 days at temperatures of 298 K and 310 K. The pH was measured with a GK 2302C electrode using a Radiometer Mo 26 pH-meter.

The morphology of the precipitates was examined in white and polarized light in a Orthoplan microscope supplied with an automatic Orthomat-W camera (Leitz, Wetzlar). The X-ray powder patterns of selected samples were recorded by means of a Philips diffractometer with a proportional counter, using graphite monochromated $\text{Cu K } \alpha$ radiation. The d-spacings were compared with the values reported by Ropp et al.¹⁴ and Powder Diffraction File.¹⁵ The composition of $\text{Na}[\text{Sr}(\text{H}_2\text{O})_9]\text{PO}_4$ was determined by precipitating strontium with KH_2PO_4 , drying at 393 K and weighing as SrHPO_4 .¹³ Phosphorus was precipitated with ammonium molybdate and after drying at 476–673 K, it was weighed as $(\text{NH}_4)_3\text{PMo}_{12}\text{O}_{40}$. Sodium was determined by atomic absorption spectroscopy and water by thermogravimetric analysis.

RESULTS AND DISCUSSION

Spontaneous precipitation of strontium phosphates was carried out over concentration ranges of strontium chloride from $2 \cdot 10^{-4}$ to $8.0 \cdot 10^{-1} \text{ mol dm}^{-3}$ and total phosphates from $4 \cdot 10^{-4}$ to $3.0 \cdot 10^{-1} \text{ mol dm}^{-3}$. The concentration of sodium chloride was 0.15 mol dm^{-3} in all systems.

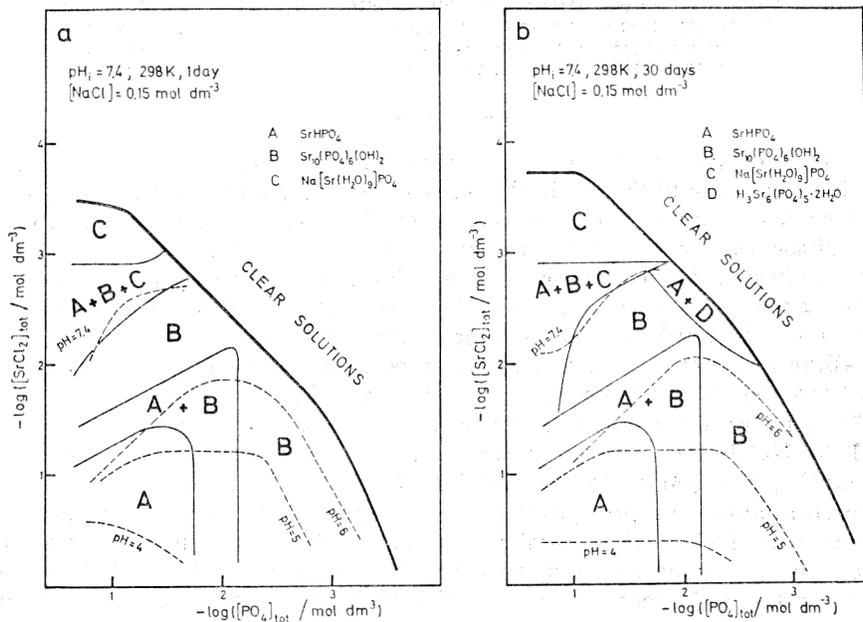


Figure 1. Precipitation diagrams of the system strontium chloride–sodium phosphate–sodium chloride–water at $\text{pH}_i = 7.4$, 298 K:

(a) aged 1 day

(b) aged 30 days.

The precipitation and phase boundaries are assigned by full lines and iso-pH curves by dashed lines.

The precipitation diagrams of strontium phosphates, 1 day and 30 days after mixing the components are presented in Figure 1. The precipitation and phase boundaries were detected microscopically. The solid phases were identified by X-ray powder diffraction patterns.

The precipitation region of pure $\text{Sr}_{10}(\text{PO}_4)_6(\text{OH})_2$ appears in excess of strontium chloride (resulting pH range $4 < \text{pH} < 7$) and also in excess of phosphate (resulting pH range $6.5 < \text{pH} < 7.4$). The precipitates of $\text{Sr}_{10}(\text{PO}_4)_6(\text{OH})_2$ are submicroscopic sized crystals of crystal aggregates (microphotograph I).

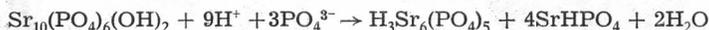
The mixture of two modifications (α and β) of SrHPO_4 precipitates at high reactant concentrations ($3 < \text{pH} < 4$). The α -modification prevails in excess of phosphate. SrHPO_4 crystallizes in the prismatic shape (microphotograph II) or as spherulitic agglomerates (microphotograph III).

Solid $\text{Na}[\text{Sr}(\text{H}_2\text{O})_9]\text{PO}_4$ precipitates at $[\text{PO}_4]_{\text{tot}} \gg [\text{Sr}]_{\text{tot}}$, as tetrahedral crystals (microphotograph IV).¹⁶ Chemical analysis of this compound revealed the following values:

	% Sr	% P	% Na	loss % weight
Found:	23.65 ± 0.30	8.49 ± 0.15	6.27 ± 0.05	43.8 ± 1.5
Calculated:	23.83	8.42	6.25	44.09

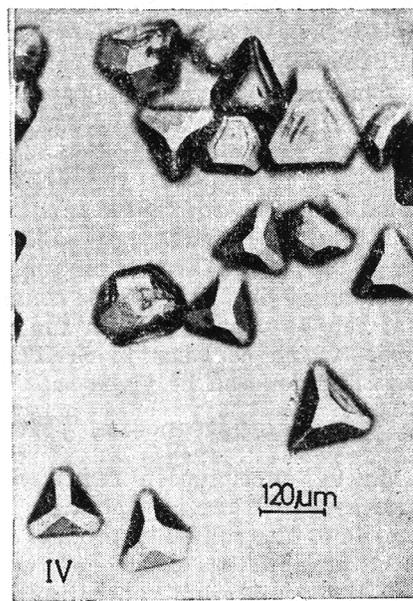
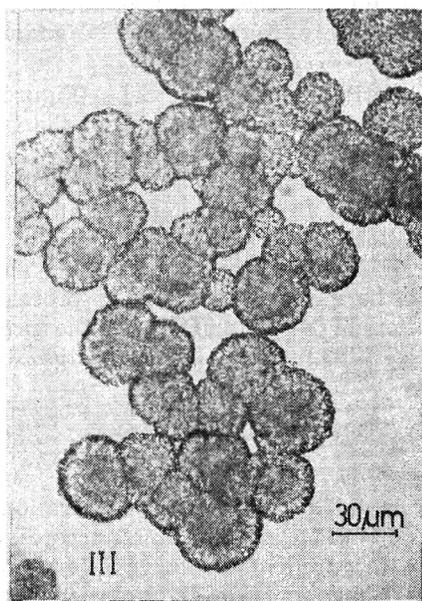
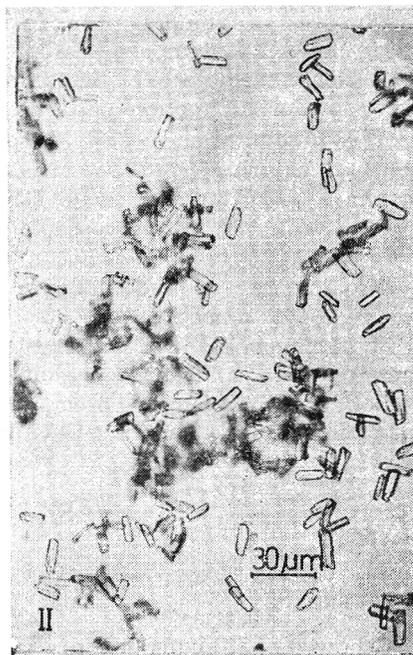
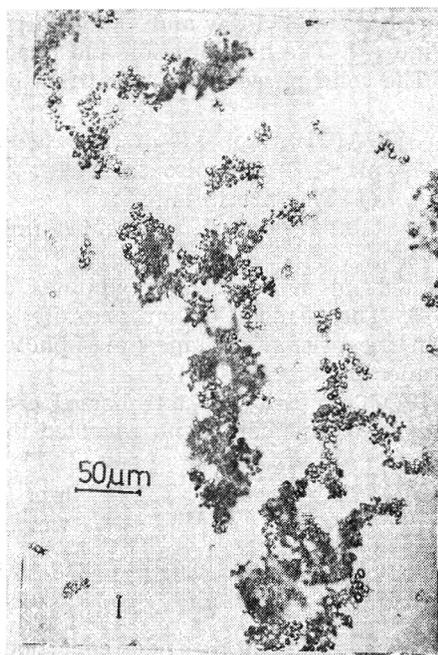
TGA showed continuous dehydration from 313 K up to 390 K. Under conditions close to the precipitation boundary $\text{Na}[\text{Sr}(\text{H}_2\text{O})_9]\text{PO}_4$ precipitates alone ($7.6 > \text{pH} > 7.4$). At higher $[\text{Sr}]_{\text{tot}}$ it appears in the mixture with SrHPO_4 and/or $\text{Sr}_{10}(\text{PO}_4)_6(\text{OH})_2$.

The precipitation boundary of $\text{Sr}_{10}(\text{PO}_4)_6(\text{OH})_2$ in excess of $[\text{Sr}]_{\text{tot}}$ (Figures 1a, b; $[\text{SrCl}_2]_{\text{tot}} > 1 \cdot 10^{-2} \text{ mol dm}^{-3}$, $[\text{PO}_4]_{\text{tot}} < 2 \cdot 10^{-3} \text{ mol dm}^{-3}$) does not change with time. In the concentration region of $[\text{SrCl}_2]_{\text{tot}} < 1 \cdot 10^{-2} \text{ mol dm}^{-3}$, $2 \cdot 10^{-3} \text{ mol dm}^{-3} < [\text{PO}_4]_{\text{tot}} < 1 \cdot 10^{-2} \text{ mol dm}^{-3}$ the precipitation boundary at 30 days is shifted to lower reactant concentrations (Figures 1a, b). Systems exhibiting clear solutions after 1 day, on being aged for 30 days showed precipitates containing β - SrHPO_4 or a mixture of β - SrHPO_4 and $\text{H}_3\text{Sr}_6(\text{PO}_4)_5 \cdot 2\text{H}_2\text{O}$.¹⁵ The formation of SrHPO_4 at these conditions can be either a spontaneous process, or it can be connected with the precipitation of $\text{Sr}_{10}(\text{PO}_4)_6(\text{OH})_2$ as the precursor, which converts to SrHPO_4 and $\text{H}_3\text{Sr}_6(\text{PO}_4)_5 \cdot 2\text{H}_2\text{O}$. The reaction of transformation can be given as:



In the precipitation region characterized by crystallization of $\text{Na}[\text{Sr}(\text{H}_2\text{O})_9]\text{PO}_4$ the precipitation boundary is shifted to lower concentrations with time. The solubility product of $\text{Na}[\text{Sr}(\text{H}_2\text{O})_9]\text{PO}_4$ is determined according to the precipitation boundary after 30 days (Figure 1b). In Table I (systems 1—6) the equilibrium concentrations of all components in the solutions are listed: $[\text{Sr}]_{\text{eq}}$, $[\text{PO}_4]_{\text{eq}}$, $[\text{Na}]_{\text{eq}}$, pH values, and ionic strengths.

The concentration of $[\text{Sr}]_{\text{eq}}$ is the sum of $[\text{Sr}^{2+}]$ and $[\text{SrHPO}_4^0]$, while $[\text{SrH}_2\text{PO}_4^+]$ is negligible at corresponding $[\text{PO}_4]_{\text{eq}}$ and pH. The concentration of $[\text{PO}_4]_{\text{eq}}$ is the sum of free phosphates (HPO_4^{2-} ions prevail, H_3PO_4 being negligible). Phosphates incorporated in the strontium complex (SrHPO_4^0) can be neglected at $[\text{Sr}]_{\text{eq}} \ll [\text{PO}_4]_{\text{eq}}$. The concentration of $[\text{Na}]_{\text{eq}}$ is equal to the



Microphotographs I–IV of strontium phosphate precipitates: I – $\text{Sr}_{10}(\text{PO}_4)_6(\text{OH})_2$, II and III SrHPO_4 , IV – $\text{Na}[\text{Sr}(\text{H}_2\text{O})_9]\text{PO}_4$. The experimental conditions for all systems shown were: pH_i 7.4, $T = 298 \text{ K}$, $t = 30$ days. The initial concentrations of strontium chloride and sodium phosphate, and the final pH for particular systems were:

I – $[\text{SrCl}_2]_{\text{tot}} = 4 \cdot 10^{-3} \text{ mol dm}^{-3}$,
 $[\text{PO}_4]_{\text{tot}} = 6 \cdot 10^{-3} \text{ mol dm}^{-3}$, pH 6.52

II – $[\text{SrCl}_2]_{\text{tot}} = 4 \cdot 10^{-2} \text{ mol dm}^{-3}$,
 $[\text{PO}_4]_{\text{tot}} = 3 \cdot 10^{-2} \text{ mol dm}^{-3}$, pH 5.28

III – $[\text{SrCl}_2]_{\text{tot}} = 1 \cdot 10^{-3} \text{ mol dm}^{-3}$,
 $[\text{PO}_4]_{\text{tot}} = 1 \cdot 10^{-1} \text{ mol dm}^{-3}$, pH 7.35

IV – $[\text{SrCl}_2]_{\text{tot}} = 1 \cdot 10^{-3} \text{ mol dm}^{-3}$,
 $[\text{PO}_4]_{\text{tot}} = 8 \cdot 10^{-2} \text{ mol dm}^{-3}$, pH 7.55

TABLE I

Equilibrium Concentration Determined According to Precipitation Boundary and Calculated K_s Values for $\text{Na}[\text{Sr}(\text{H}_2\text{O})_9]\text{PO}_4(\text{s})$

	$-\log [\text{Sr}]_{\text{eq}}$	$-\log [\text{PO}_4]_{\text{eq}}$	$-\log [\text{Na}]_{\text{eq}}$	pH	$I/\text{mol dm}^{-3}$	$\log K_s$
1	3.11	1.82	0.75	7.50	0.20	-9.97
2	3.16	1.70	0.73	7.47	0.21	-9.95
3	3.35	1.52	0.68	7.49	0.24	-9.96
4	3.50	1.40	0.65	7.42	0.26	-10.09
5	3.55	1.30	0.61	7.52	0.29	-9.94
6	3.70	1.22	0.58	7.54	0.30	-10.01

concentration of $[\text{Na}^+]$. Ionic strengths varied in systems 1–6 (Table I) from 0.20 mol dm^{-3} to 0.30 mol dm^{-3} . The association constants of phosphoric acid have been given in the literature^{17,18} for ionic strengths $I = 0.1 \text{ mol dm}^{-3}$ and $I = 0.4 \text{ mol dm}^{-3}$ (Table II eqs. 1 and 2). The constant of strontium phosphate complex is determined¹⁸ at $I = 0.2 \text{ mol dm}^{-3}$ (Table II, eq. 3). The values of association constants at $I = 0.2$ and 0.3 mol dm^{-3} calculated from the values of constants at $I = 0.1$ and 0.4 mol dm^{-3} (using Davies equation¹⁹) are close (see deviations of constants at $I = 0.25 \pm 0.05$ in Table II). Therefore the set of constants corrected to $I = 0.25 \text{ mol dm}^{-3}$ (mean value of ionic strengths in systems 1–6) is used (Table II, eqs. 1–3). The solubility product of $\text{Na}[\text{Sr}(\text{H}_2\text{O})_9]\text{PO}_4$ ($K_s = [\text{Na}^+][\text{Sr}^{2+}][\text{PO}_4^{3-}]$) is expressed as:

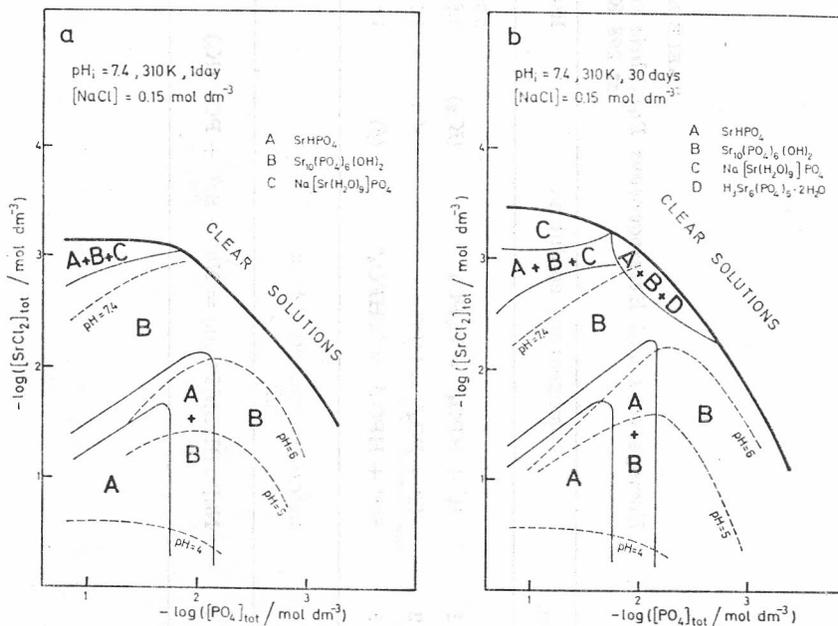


Figure 2. Precipitation diagrams of the system strontium chloride–sodium phosphate–sodium chloride–water at $\text{pH}_1 = 7.4, 310 \text{ K}$:

- (a) aged 1 day
- (b) aged 30 days.

The precipitation and phase boundaries are assigned by full lines and iso-pH curves by dashed lines.

TABLE II
Homogeneous and Heterogeneous Equilibria in the Strontium — Phosphate System
 (at 298 K)

eq.	homogeneous equilibria	literature value log K	corrected value
1	$H^+ + HPO_4^{2-} = H_2PO_4^-$ (K_{12})	6.72 (I = 0.1) ¹⁸ 6.62 (I = 0.4) ¹⁷	log K (I = 0.25 ± 0.05 mol dm ⁻³) 6.65 ± 0.02
2	$H^+ + PO_4^{3-} = HPO_4^{2-}$ (K_1)	11.74 (I = 0.1) ¹⁸	11.59 ± 0.04
3	$Sr^{2+} + HPO_4^{2-} = SrHPO_4^0$ (β)	1.52 (I = 0.2) ¹⁸	1.46 ± 0.06
heterogeneous equilibria			
4	$Na[Sr(H_2O)_9]PO_4(s) = Na^+ + Sr^{2+} + PO_4^{3-} (K_8)$		determined value log K (I = 0.25 ± 0.05 mol dm ⁻³) —9.98 ± 0.11

$$K_s = \frac{[\text{Na}]_{\text{eq}} [\text{Sr}]_{\text{eq}} [\text{PO}_4]_{\text{eq}} (K_{12} K_1 [\text{H}^+]^2 + K_1 [\text{H}^+] + 1)^{-1}}{1 + \beta [\text{PO}_4]_{\text{eq}} (1 + K_{12} [\text{H}^+] + K_1^{-1} [\text{H}^+]^{-1})^{-1}}$$

The solubility products calculated for each system are presented in Table I. The mean value of $\log K_s$ is given in Table II (eq. 4).

The diagrams of precipitation systems aged one day and 30 days at 310 K are shown in Figure 2. Solutions which were clear after one day (Figure 2a). ($2 \cdot 10^{-3} \text{ mol dm}^{-3} < [\text{PO}_4]_{\text{tot}} < 2 \cdot 10^{-2} \text{ mol dm}^{-3}$), when aged for 30 days (Figure 2b) contained mixed precipitates: α - SrHPO_4 and $\text{Sr}_{10}(\text{PO}_4)_6(\text{OH})_2$, α - SrHPO_4 and $\text{H}_3\text{Sr}_6(\text{PO}_4)_5 \cdot 2\text{H}_2\text{O}$. In the high excess of phosphates ($[\text{PO}_4]_{\text{tot}} > 2 \cdot 10^{-2} \text{ mol dm}^{-3}$) pure $\text{Na}[\text{Sr}(\text{H}_2\text{O})_9]\text{PO}_4$ was formed during the same period of time. The phase boundaries of SrHPO_4 (mixture of α and β modifications) and of $\text{Sr}_{10}(\text{PO}_4)_6(\text{OH})_2$ are also shown in Figure 2.

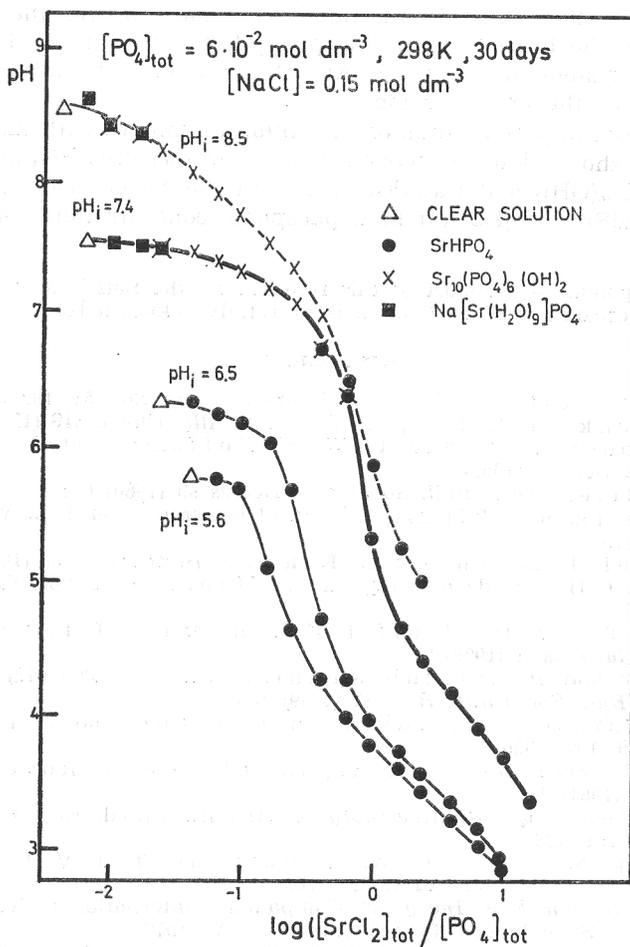


Figure 3. Relation between final pH and reactant concentrations during precipitation at initial pH of 5.6, 6.5, 7.4, and 8.5. The symbols indicate the various compositions of precipitated solid phases.

A comparison of the precipitation and phase boundaries in Figure 1b (at 298 K) and Figure 2b (at 310 K) indicates the influence of temperature on the processes involved. The precipitation boundary of $\text{Sr}_{10}(\text{PO}_4)_6(\text{OH})_2$ at 310 K is at lower reactant concentrations than is the case at 298 K. The precipitation boundary of $\text{Na}[\text{Sr}(\text{H}_2\text{O})_9]\text{PO}_4$ at 310 K is at higher reactant concentrations. The stability region of the latter solid is narrow at 310 K due to the dehydration of this compound starting at 313 K (thermogravimetric data). Lowering the temperature to < 298 K favors the precipitation of $\text{Na}[\text{Sr}(\text{H}_2\text{O})_9]\text{PO}_4$. In the region where mixtures of $\text{Na}[\text{Sr}(\text{H}_2\text{O})_9]\text{PO}_4$, SrHPO_4 and $\text{Sr}_{10}(\text{PO}_4)_6(\text{OH})_2$ were found at 298 K (Figure 1b), only $\text{Na}[\text{Sr}(\text{H}_2\text{O})_9]\text{PO}_4$ precipitated at 291 K. In the concentration region between precipitation boundaries of one day and 30 days, β - SrHPO_4 precipitates at 298 K, and α - SrHPO_4 at 310 K. This result is in accordance with Mooney et al.³

The formation of strontium phosphates at various initial pH values at $[\text{PO}_4]_{\text{tot}} = 6 \cdot 10^{-2}$ mol dm^{-3} is indicated in Figure 3. At the expense of $\text{Na}[\text{Sr}(\text{H}_2\text{O})_9]\text{PO}_4$ the broadening of the precipitation region for $\text{Sr}_{10}(\text{PO}_4)_6(\text{OH})_2$ occurs in the system at $\text{pH}_i = 8.5$. At $\text{pH}_i = 6.5$ and 5.6, only α - SrHPO_4 is precipitated over the entire region.

A comparison of precipitation of strontium phosphates with that of calcium phosphates²⁰⁻²³ shows that analogous solids are formed (SrHPO_4 and $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$, $\text{Sr}_{10}(\text{PO}_4)_6(\text{OH})_2$ and $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$). In the former case an additional solid phase, $\text{Na}[\text{Sr}(\text{H}_2\text{O})_9]\text{PO}_4$ (at high phosphate concentrations and $\text{pH} \approx 7.4$) was found.

Acknowledgment. — Support of this research by the Self-Management Council for Scientific Research of S. R. Croatia is gratefully acknowledged.

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SAŽETAK

Taloženje i karakterizacija stroncij-fosfata

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Proučavan je proces spontanog taloženja stroncij-fosfata u širokom koncentracijskom području stroncij-klorida ($2 \cdot 10^{-4}$ — $8 \cdot 10^{-1}$ mol dm⁻³) i natrij-fosfata ($4 \cdot 10^{-4}$ — $3 \cdot 10^{-1}$ mol dm⁻³). Početni pH u svim sistemima bio je 7.4, a koncentracija natrij-klorida 0,15 mol dm⁻³. Ispitani su talozi stareni 1 dan i 30 dana pri 298 K i 310 K. Karakterizacija taloga načinjena je optičkom mikroskopijom i rendgenskom difrakcijom na praškastim uzorcima. Nađene su slijedeće čvrste faze: SrHPO₄ (α i β modifikacija), Sr₁₀(PO₄)₆(OH)₂, H₃Sr₆(PO₄)₅ · 2H₂O i Na[Sr(H₂O)₉]PO₄. Produkt topljivosti Na[Sr(H₂O)₉]PO₄ određen je iz podataka s taložne granice nakon 30 dana (ravnotežni uvjeti) i iznosi $\log ([Na^+] [Sr^{2+}] [PO_4^{3-}]) = -9.98 \pm 0.11$ ($I = 0.25$ mol dm⁻³, $T = 298$ K).