

DCC-29 (Univ. Zagreb)

Croat. Chem. Acta 42 (1970)

The Interaction of Some Organic and Inorganic Ions with Silver Iodide Sols *in statu nascendi*

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The pptn. phenomena were investigated by tyndallometric and fluorometric measurements in the systems *in statu nascendi* with the following components: silver nitrate, sodium iodide, sodium fluoresceinate, rhodamine B, triton T-X-305, T-X-205 or T-X-100, potassium, barium and lanthanum nitrate.

The system sodium fluoresceinate — rhodamine B shows isoelectric pptn. and formation of an *equivalent body*.

By systematic measurements in various concn. regions in the system consisting of silver-, halide- and fluoresceinate ions the conception of Fajans' indicators was improved. That conception involves: stereoadsorption of fluoresceinate, flocculation and reversal of charge of silver iodide, pptn. of silver fluoresceinate both on the surface and independently in solution, *i. e.* copptn. of various ionic species.

The amt. of fluoresceinate adsorbed on the silver iodide surface is calcd. from fluorometric measurements.

The influence of the presence of nonionic surface-active agents on the pptn. effects of silver iodide is examd. tyndallometrically. T-X-305, T-X-205 or T-X-100 shows similar pptn. phenomena as the org. ions of dyes (fluoresceinate or rhodamine B), and as small inorg. ions of high valency. Three pptn. regions can be distinguished: stabilization, flocculation and concn. region without any effects. The flocculation limits for positive and negative silver iodide sol were found to be in the region from 10^{-7} to 10^{-6} of triton.

The interaction of silver iodide colloidal particles with surface-active agents was investigated in the presence of inorg. cations (Na^+ , Ba^{2+} , La^{3+}) and fluoresceinate or rhodamine B. The results show that the stereoadsorption of triton is a primary process and that there is an opportunity of ion-pair formation only on the free places on the surface of colloidal particles.

In the pptn. systems consisting of silver nitrate, sodium iodide, sodium fluoresceinate (or rhodamine B) and triton, the stabilizing action of triton is quite pronounced; the critical protective values are found to be in order of 10^{-4} M.

The thesis was partly published in: 1. *Croat. Chem. Acta* 36 (1964) 59; 2. *Ibid.* 36 (1964) 133.

Examiners: Prof. M. Mirnik, Prof. C. Djordjević, Dr. O. Weber.
Oral examination: May 25, 1965.

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DCC-29

1. The Interaction of Some Organic and Inorganic Ions with Silver Iodide Sols *in statu nascendi*

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Adsorption phenomena

Barium nitrate

Lanthanum nitrate

Potassium nitrate

Precipitation phenomena

Rhodamine B

Silver nitrate

Sodium fluoresceinate

Sodium iodide

Triton T-X-100, T-X-205, T-X-305

MCC-34 (Univ. of Zagreb)

Master of Science Thesis

Croat. Chem. Acta 42 (1970)

**Investigations into the Kinetics of Electrochemical-Chemical
Reactions of Uranium Carbonate Complexes by Cyclic
Chronopotentiometry**

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The method and technique of chronopotentiometry is applied to the study of complex electrochem. and chem. reactions mechanisms. The work contains a development of the method into cyclic chronopotentiometry, by application of computational methods to the estn. of second order rate coeff. of a chem. reaction following electron transfer.

It has been found that the range of applicability is for rate coeff. between 1 and $10^5 \text{ l mol}^{-1} \text{ sec}^{-1}$. In the optimum range between 10 and 50 the error of a detn. of a rate coeff. is less than 10%. A computer program written in Fortran II is presented, which simulates digitally the complex reaction kinetics encountered in some ionic systems *e.g.* U(IV), (V) and (VI) carbonate soln.

The rate coeff. of the disproportionation reaction of U(V) in 1 M NaHCO_3 at $\text{pH} = 8.5$ is $9.9 \text{ l mol}^{-1} \text{ sec}^{-1}$. Several expts. indicate that the rate coeff. for this reaction increases with a decrease either in the concn. of free carbonate or the ionic strength of the soln. In addition it has been found, contrary to some indications in the literature, that the U(V) — tri-carbonate complex is oxidized either directly (*i.e.* without prior dissociation) at the mercury cathode, or the rate coeff. of the preceeding dissociation is larger than about 35 sec^{-1} .

Part of the work (contained) was reported at the 1st Yugoslav Symposium on Electrochemistry, Belgrade 1968. A part was published in Croat. Chem. Acta 42 (1970) 21.

Examiners: Prof. I. Filipović, Prof. M. Mirnik, and Dr V. Pravdić.

Oral Examination: June 4, 1969.

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(79 pages, 13 tables, 16 figures, 76 references, 2 computer programs, original in Croatian)

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MCC-34 (Univ. of Zagreb)

1. Investigations into the Kinetics of Electrochemical — Chemical Reactions of Uranium Carbonate Complexes by Cyclic Chronopotentiometry

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Carbonate
Chronopotentiometry
—, cyclic
Computer program
Digital simulation
Disproportionation
Electrochemistry
Electron transfer
Kinetics
—, second order chemical
Oxidation
Reduction
Rate coefficient
Uranium

MCC-35 (Univ. Zagreb)

Master of Science Thesis

Croat. Chem. Acta 42 (1970)

Binding of ^{65}Zn to Serum Proteins *in vitro*

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The thesis deals with the binding of zinc to serum proteins *in vitro* depending on the ageing of the system at $+4^{\circ}\text{C}$. For the separation of serum proteins the following methods were used: one-dimensional paper electrophoresis and agar gel electrophoresis, microzone electrophoresis on acetate cellulose, two-dimensional electrochromatography and continuous electrophoresis on filter paper. Expts. were performed in a barbituric buffer at pH 8.6 and 7 and in phosphate buffer at pH 7.

Normal human serum labelled *in vitro* with carrier free $^{65}\text{Zn}/10^{-7}\text{ M}$) immediately binds almost all ^{65}Zn to a protein which migrates electrophoretically in the albumin region. The 24-hour incubation of serum labelled with the ^{65}Zn at 4°C does not cause any significant changes. During a further 3 to 8 day ageing period the amount of ^{65}Zn bound to alpha and beta-globulins is increasing. The binding of ^{65}Zn to globulins can be ascribed to the exchange of radiozinc with the inactive zinc which is physiol. present in these fractions.

It is clearly shown that one-dimensional electrophoresis on a supporting medium cannot yield a correct result regarding the binding of radiozinc to serum albumins because of the radioactive rest-proteins on the supporting medium. Two dimensional electrochromatography is an extremely suitable method for such investigations, because electromigration zones of particular fractions of serum proteins do not overlap during the sepn. and thus prevents the radioactive contamination of inactive serum proteins fractions what is not the case when one-dimensional electrophoresis on a support is applied.

The ^{65}Zn which is not bound to serum proteins and has a strong chromatographic effect migrates under the electrochromatographic conditions partly in the form of a complex with barbituric acid.

By continuous electrophoresis it was found that 90 per cent ^{65}Zn in the albumin region was bound to a protein which was called »zincophilin«. The rest of ^{65}Zn was bound to alpha and beta-globulins, and only 0.2 per cent to gamma-globulins. After 14-day ageing of the system 80 per cent ^{65}Zn in the albumin region was bound to »zincophilin«, 16 per cent to alpha and beta-globulins and 2 per cent to gamma-globulins. From the electrophoregram stained for lipids and the corresponding radioautogram it is possible to conclude that lipids play an important role in the binding of ^{65}Zn to electrophoretic serum fractions.

Parts of this thesis were published and reported: *Regional Meeting of Chemists*, Zagreb, Croatia 1969 and *1st Yugoslav Symposium on the Chemistry and Technology of Macromolecules*, Feb. 1969, Prosc. B-2/39, *Arh. hig. rada* No 1, Vol. 21 (1970) 23.

Examiners: Dr. Z. Pučar, Prof. B. Težak, and Prof. M. Herak.

Oral examination: July 9, 1970.

Degree conferred: October 31, 1970.

Thesis deposited at the University Library, Zagreb. Institute for Medical Research, Yugoslav Academy of Sciences and Arts, and »Ruder Bošković« Institute, Zagreb.

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MCC-35

1. Binding of ^{65}Zn to Serum
Proteins in Vitro

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Electrochromatography

Electrophoresis

alpha-Globulins

beta-Globulins

gamma-Globulins

Lipids

Radiozinc

MCC-36 (Univ. Zagreb)

Master of Science Thesis

Croat. Chem. Acta 42 (1970)

Redox Processes, Hydrolysis and Precipitation of Uranium in Aqueous Solutions

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The redn. and oxidn. processes of uranium(VI), (V), (IV) and (III) have been studied in perchlorate aq. solns. using the dropping, hanging mercury drop electrode and thin layer technique. The expts. have been carried out in the acidic media ($0 < \text{pH} < 2.7$) at different concn. of uranium and const. ionic strength $\mu = 1$. In certain conditions the observed data show that the uranium(V) redn. process is influenced by its chem. reaction with uranium(III), resulting in the formation of uranium(IV). Due to this reaction the character of the polarographic and chronovoltammetric curves is changed. The disappearance of uranium(III) oxidn. peak on chronovoltammetric curves obtained with hanging mercury drop electrode can be also explained by this reaction. The kinetic parameters of the uranium(V) to (IV) redn. were detd. as: $k_{f,h} = (4.0 \pm 0.4) \times 10^{-8}$ cm./sec. and $\alpha = 0.46 \pm 0.01$. The mechanism of uranium(V) to uranium(III) redn. process is discussed and proposed.

Uranium(VI) in 1 M NaClO₄ (pH = 2), 1 M Na₂CO₃ and 1.1 M LiOH solns. have been reduced to uranium(V) on the mercury electrode at -0.6, -1.2, and -1.1 V (vs. SCE), resp., and the uranium(V) pptd. The pptn. was carried out in the case of perchlorate and carbonate solns. by the addn. of NaOH, while in LiOH soln. the uranium(V) ppts. during the electrolysis. The O/U ratio of the ppt. was 2.5, with no significant alkaline metal content, showing the formation of hydrated uranium oxides.

Electrochemical redn. with simultaneous electrodiffusion of 0.2 to 1 M uranium(VI) chloride and nitrate solns. have been investigated to find out the condition for obtaining stable UO₂-sols. Mercury, amalgamated copper and tantal electrodes have been used. Mercury electrode was suitable to obtain up to 0.4 M UO₂-sols. When the reduced uranium concn. was higher than 0.4 M, the mercury electrode during the stirring was transformed into droplets of 0.1 to 2 mm. diameter. Such droplets were stable for few weeks. The amalgamated copper and the tantal electrodes were not suitable, because of the copper contamination in the first case, while low current densities have been obtained with the tantal electrode.

A new anal. procedure for the polarographic detn. of the O/U ratio in uranium oxides of samples dissolved in orthophosphoric and sulfuric acid is proposed. The concn. of uranium(VI) is detd. polarographically. The total uranium present in the soln. is oxidized with ceric ions to the hexavalent state, and then again polarographically detd. The O/U ratio is calcd. from the ratio of the limiting currents and diln. The method can be applied to very small samples (0.1 to 50 mg.) to all O/U ratios from uranium dioxide up to trioxide and with the coeff. of the variation of about 0.6%.

Part of this thesis is published in *J. Polarog. Soc.* 14 (1968) 3.

Examiners: Dr M. Branica, Prof. B. Težak, and Prof. M. Herak.

Oral examination: July 7, 1970.

Thesis deposited at the University Library, Zagreb and Institute »Ruđer Bošković« Zagreb.

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L. SIPOS

MCC-36

1. Redox Processes, Hydrolysis and Precipitation of Uranium in Aqueous Solutions

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Uranium in aqueous solutions
Uranium, redox processes of
Uranium, hydrolysis of
Uranium, precipitation of

MCC-37 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta 42 (1970)

Polarography of Cadmium in Electrolyte Solutions in the Presence of Ethylenediaminetetraacetic Acid

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Polarographic redn. of cadmium(II) in the presence of EDTA was investigated in chloride solns. at pH = 8.

The wave height of Cd-EDTA redn. is strongly influenced by the concn., charge, and kind of supporting electrolyte cation. The effect increases in the following order:



The electrode process, which is preceded by the chem. reaction, is an irreversible one with a n value 0.96.

Increasing the concn. of supporting electrolyte the limiting current becomes less dependent on the preceding reaction and the half-wave potential shifts toward more positive values.

The conclusion, based on kinetical parameters calculation, ψ_2 potential and influence of supporting electrolyte, is that the chem. reaction takes place in the vicinity of the mercury drop surface and precedes the electrode reaction.

The existence of CdY^{2-} ion at pH = 8 was confirmed and this, electroinactive ion is transformed into an active form CdYMe (where Me denotes mentioned cations) by recombination of CdY^{2-} with these cations on the electrode surface. The former corresponds to the existence of the electroactive ion CdYH^- which is predominant at lower pH ($2 < \text{pH} < 5$).

Examiners: Dr. M. Branica, Prof. B. Težak and Prof. M. Herak.

Oral examination: November 9, 1970.

Thesis deposited at the University Library and Institute »Ruder Bošković«, Zagreb.

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B. RASPOR

MCC-37

1. Polarography of Cadmium in Electrolyte Solutions in the Presence of Ethylenediaminetetraacetic Acid

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Cadmium, polarography of EDTA

Polarography of cadmium complexes

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BCC-740

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KRKA Pharmaceutical and Chemical Works, Institute for Antibiotics, Pharmacy and Technology, Novo Mesto, Yugoslavia

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D. Korunčev i D. Kolbah

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BCC-757

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Institute of Colloid and Surface Science and Department of Chemistry,
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Katalytische Hydrierung von Purinen*Ann. Chem.* 729 (1969) 73.

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Clarkson College of Technology, Potsdam, N. Y.

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BCC-770

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BCC-774

H. Hüther, L. Klasinc, F. Kajfež, and V. Šunjić

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An NMR-Study of 1-Substituted 2-Methyl-4(5)-nitroimidazoles*J. Mol. Structure* **4** (1969) 108.

BCC-775

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Institut za antibiotike, farmaciju i tehnologiju, »Krka«, tvornica farmaceutskih proizvoda, Novo Mesto

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BCC-778

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BCC-779

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