DCC-41 (Univ. Zagreb)

Croat. Chem. Acta CCACAA 45 (4) B1—B2 (1973)

Synthesis of Glucuronic Esters of Amino Acids

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1-*O*-Acylaminoacyl- and aminoacyl- derivs. of fully methylated glucuronic acid were synthesized and completely characterized by phys. and chem. methods.

The cyclic structure and anomeric configuration of methyl 2,3,5-tri-*O*-methyl-D-glucofuranuronate — the sugar component of these esters — was deduced from NMR spectra, GLC analysis, and the cryst. derivs.

Fully protected glucuronic esters of amino acids were obtained as anomeric mixts. in high yields by two methods involving direct participation of imidazole: (a) accelerated active ester (AAE) synthesis and (b) dicyclohexylcarbodi-imide (DCC) condensation. In both reactions the intermediate *N*-acylimidazole, formed by the nucleophilic attack of imidazole on the activated amino acid ester and the *O*-acylurea, respectively, participates directly in the transfer of the acyl group to the sugar component. The anomeric sepn. of methyl 1-*O*-acylaminoacyl-2,3,4-tri-*O*-methyl-D-glucofuranuronates was achieved by column chromatography on silica gel. Both methods proceeded without racemization of the aglycone moiety.

Catalytic hydrogenation of β-anomers of some 1-*O*-(*N*-benzyloxy-carbonyl-aminoacyl)-2,3,5-tri-*O*-methyl-D-glucofuranuronates gave the corresponding 1-*O*-aminoacyl derivs. which were isolated as monooxalate or as internal salts.

For comparison purposes, some compds. from the class of glucose-amino acid 6-esters were synthesized by the same methods.

The thesis was partly published in: *Tetrahedron Lett.* (1970) 2983; *Croat. Chem. Acta* **44** (1972) 229; *Carbohyd. Res.* (in press).

Examiners: Dr. D. Keglević, Dr. N. Pravdić, and Prof. D. Sunko.

Oral examination: December 27, 1972.

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

(80 pages, 4 tables, 12 figures, 101 references, original in Croatian).

G. ROGLIĆ

DCC-41 (Univ. Zagreb)

1. Syntheses of Glucuronic Esters of Amino Acids

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Accelerated active ester method
Amino acids, glucuronic esters with,
Dicyclohexylcarbodi-imide method with imidazole
Glucuronic esters
Glucofuranuronic acid, methyl ester, 1-*O*-(acylaminoacyl)-2,3,5-tri-*O*-methyl-α- and β-D-,
Glucopyranuronic acid, methyl ester, 1-O-(*N*-acyl-*DL*-alanyl)-2,3,5-tri-*O*-methyl-α- and β-D-
Glucopiranoside, 6-*O*-glycyl-β-D-, methyl,
—, 2,3,4-tri-*O*-acetyl-6-*O*-(*N*-acetylglycyl)-β-*D*-, methyl,
—, 2,3,4-tri-*O*-benzyl-6-*O*-(*N*-benzyloxycarbonyl-aminoacyl)-β-*D*-, methyl,
Sugar-amino acid linkage

MCC-50 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta CCACAA 45 (4) B3—B4 (1973)

Radiometric Investigation of Ca-Phosphates

N. Filipović-Vinceković

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Radiometric procedures for fast determination of the solubility limits, precipitation kinetics, and precipitate equilibration of Ca-phosphates with the supernatant have been developed. The results of radiometric analysis show the formation of variously soluble Ca-phosphates. Solubility limits correspond to four morphological limits at the precipitation diagram established by Füredi and co-workers.

Analysis of the data on ^{45}Ca and ^{32}P radionuclide distribution in systems at 25 °C show that the precipitates of Ca-phosphates pass through two steps: after formation of the metastable colloidal defective apatite acidic Ca-phosphate will be formed as a product corresponding to the second precipitation step. Depending on the initial conditions, OCP or DCP will be formed as a second product. By aging in the supernatant »amorphous« and unstable Ca-phosphates grow to the stable Ca-phosphate composition *via* Ostwald ripening and structural changes.

Examiners: Dr. R. Despotović, Dr. H. Füredi, and Prof. B. Težak

Oral examination: March 10, 1972

Thesis deposited at the Faculty of Science, University of Zagreb.

(128 pages, 21 table, 38 figures, 131 reference)

R. DESPOTOVIĆ

MCC-50 (Univ. Zagreb)

1. Radiometric Investigation of of Ca-phosphates

I. Filipović-Vincekovič N.

II. Institute »Ruđer Bošković«, 41000 Zagreb, Croatia, Yugoslavia

Ca-phosphates
Crystal growth
Exchange, heterogeneous of ^{45}Ca and ^{32}P with Ca-phosphates
Ostwald ripening
Precipitation

MCC-51 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta CCACAA 45 (4) B5—B6 (1973)

Radiometric Investigation of Stable AgI—I^- Sols

B. Subotić

»Ruđer Bošković« Institute, 41000 Zagreb, Croatia, Yugoslavia

Heterogeneous exchange in the AgI-I system was investigated by means of the radioactive tracer technique. As a selected problem, the phenomenon of the acceleration of the exchange caused by the addition of coagulating electrolyte was examined. The results show strong dependence of the exchange acceleration on the concentration of coagulation electrolytes up to the critical coagulation concentration. The analysis of this phenomenon shows the influence of the coagulation electrolyte on the »methoric mass« of silver iodide, indicating that in silver iodide suspensions three fractions of colloid subsystems could be obtained: embryonal silver iodide, fraction of the metaphase mass, and methoric silver iodide. During the aging the embryos disappear by transforming into the solid phase, the silver iodide particles grow and the methoric mass disappears too turning into the solid phase.

Examiners: Dr. R. Despotović, Prof. M. Mirnik, Prof. R. Wolf

Oral examination: March 10, 1972

Thesis deposited at the Faculty of Science, University of Zagreb.

(163 pages, 32 figures, 2 tables, 106 references, original in Croatian)

R. DESPOTOVIĆ

MCC-51 (Univ. Zagreb)

1. Radiometric Investigation of Stable AgI—I^- Sols

I. Subotić B.

II. Institute »Ruđer Bošković«, 41000 Zagreb, Croatia, Yugoslavia

Analysis, radiometric
Colloid, stability of AgI,
—, subsystems of AgI
—, formation and
—, aging of AgI
Exchange, heterogeneous
Ostwald ripening
Silver Iodide, formation of sols,
—, radiometric analysis of
—, colloidal

MCC-52 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta CCACAA 45 (4) B7—B8 (1973)

Numerical and Analytical Investigation of Complicated Molecular Integrals

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Two methods of evaluation of cimplicated molecular integrals over Slater-type orbitals are presented.

Three center nuclear-attraction integrals are calculated using the analytical method given by Bosanac and Randić which is derived using the Neumann expansion and the expansion of Legendre functions of fist and second kinds. Well known A_n, A_{-n} and B_n functions are appearing as basic quantities. The final formula involves double infinite summation. After some trial calculations the requirement of accuracy has been put to $\varepsilon = 10^{-7}$. This leads to between three and at the most eleven terms in outer l-summation. In such a way we obtained absolute accuracies of 10^{-5} to 10^{-6}. Numerical results are obtained for linear and nonlinear molecules with the limitation m = O. The different combinations of Slater-type orbitals from (1 s) to (4 s) orbitals are included and the corresponding coefficients $a_{n,j}$ are tabulated (for some cases $m \neq 0$ too).

The formula for the general integrals with $m \neq 0$ is also given.

Multicenter electron repulsion integrals were calculated using a numerical Fourier transform method based on a formulation first given by Bonham, Peacher, and Cox and developed by Harris and Monkhorst. Spherical expansion of plane waves was introduced and this enabled an insight into the behavior and the derivation of a method for accurate calculation of Fourier transforms of two-center charge distributions. This enabled the calculation of three-center exchange integrals. Using the free choice of the origin of transforms coplanar four-center integrals were calculated. In both cases only (1 s) orbitals were used.

The convergence properties of the method were discussed. Absolute accuracies of 10^{-5} to 10^{-6} were obtained. Integrals with diffuse and not too asymmetrical charge distributions were computed to four or five significant figures. When highly asymmetrical or compact charge distributions arise the choice of the origins of transforms was found to be very important. These conclusions are expected to hold for general *(ns)* orbitals as well.

The formula for general non-planar four-center integrals is given too and some possible further applications are mentioned.

A part of this work was published in *Intern. J. Quantum Chem.* 7 (1973) 233.

Examiners: Dr. N. Trinajstić, Dr. E. Coffou and Dr. J. N. Herak

Oral examination: June 10, 1972.

Thesis deposited at the University of Zagreb.

(85 pages, 13 tables, 60 references, original in Croatian)

A. GRAOVAC

MCC-52 (Univ. Zagreb)

1. Numerical and Analytical Investigation of Complicated Molecular Integrals

I. Graovac A.

II. Institute »Ruđer Bošković«, 41000 Zagreb, Croatia, Yugoslavia

Molecular integrals, Slater-type orbitals

Multicenter electron repulsion integrals, *(ns)* orbitals, numerical Fourier-transform method

Three center, nuclear-attraction integrals, A_n, A_{-n} and B_n functions

MCC-53 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta CCACAA 45 (4) B9—B10 (1973)

Characterization of the Negative Concentration Maximum of Silver-Bromide by Macromolecules and by Change of Temperature

V. Crnički

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The precipitation phenomena in the region of negative concentration maximum of silver bromide were investigated at 20 and 60 °C by the *in statu nascendi* method using a Zeiss tyndallometer in connection with a Pulfrich photometer.

The influence of surface active agents triton T-X-305 and gelatin on the sol in the region of negative concentration maximum was also examined. With triton T-X-305 both flocculation and stabilization effects were observed while gelatin only stabilized the sols. All the effects were found to be dependent on the concentration of excess potassium bromide.

The temperature dependence of the complex solubility limit of negative silver bromide sol was also studied. By cooling the saturated solution from 90 to 20 °C silver bromide precipitated in form of larger crystals. When adding water or solutions of electrolytes or macromolecules to the cooled systems, sols were formed showing H. O. T. S. during their formation.

Examiners: Prof. B. Težak, Prof. R. Wolf, and Dr. H. Füredi-Milhofer

Oral examination: October 30, 1972.

Thesis deposited at Faculty of Science, University of Zagreb.

(92 pages, 33 figures, 66 references, original in Croatian)

V. CRNIČKI

MCC-53 (Univ. Zagreb)

1. Characterization of the Negative Concentration Maximum of Silver Bromide by Macromolecules and by Change of Temperature

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Flocculation, of AgBr sols
Silver bromide sol
 —, temperature effect on
Stabilization of AgBr sols
Surface active agents
 —, effects on AgBr sols

MCC-54 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta CCACAA 45 (4) B11—B12 (1973)

The Role of Ionic Interactions in the Coagulation Process

N. Kallay

Institute of Physical Chemistry, University of Zagreb, 41000 Zagreb, Croatia, *Yugoslavia*

The influence of several electrolytes and electrolyte mixtures on the rate of coagulation of the negative silver iodide sol was examined. The methods proposed by Težak and by Reerink and Overbeek were used.

A linear relationship between the log of the electrolyte concentration and log of the rate constant of coagulation was found in the case of KNO_3, $Mg(NO_3)_2$, $La(NO_3)_3$.

The coagulation concentrations of electrolytes KNO_3, K_2SO_4, KOH (total concn. of K^+ ions) was found to be the same but the coag. concn. of K_3PO_4 was greater. The coag. concns. of acids (total concn. of H^+ ions) decrease in the folowing order: CH_3COOH, H_3PO_4, H_2SO_4, HNO_3, but the coag. activities of H^+ ions were found to be practically the same. The results were explained in terms of ion pairing between counter and co-ions.

The observed antagonistic effects in the case of electrolyte pairs $Mg(NO_3)_2$—KNO_3 and $Mg(NO_3)_2$—HNO_3 were explained with general ionic interactions in the bulk of solution, and quantitatively interpreted using the Debye-Hückel theory. Constant coag. activity of Mg^{2+} ions was found in spite of the increase of the KNO_3 and HNO_3 concentrations.

The observed antagonistic effects in the case of electrolyte pairs: $Mg(NO_3)_2$—K_2SO_4, $Mg(NO_3)_2$—H_2SO_4, $Mg(NO_3)_2$—H_3PO_4, $Mg(NO_3)_2$—$Na_3C_6H_5O_7$, $La(NO_3)_3$—K_2SO_4 were explained by ion pairing between counter- and co-ions. On the basis of experimental results the dissociation constants of ion pairs $MgSO_4$ and $LaSO_4^+$ were calculated. The values were found to be in agreement with the literature results obtained by conventional methods.

The method for determination of ion-pair stability constants by measuring the rate of coagulation of lyophobic colloids was developed.

A part of this work was published in *Croat. Chem. Acta* 45 (1973) 169.

Examiners: Prof. B. Težak, Prof. R. Wolf, and Dr. Z. Pučar.

Oral examination: October 30, 1972.

Thesis deposited at the Faculty of Science, University of Zagreb.

(70 pages, 4 tables, 16 figures, 29 references, original in Croatian)

N. KALLAY

MCC-54 (Univ. Zagreb)

1. The Role of Ionic Interactions in the Coagulation Process

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Activity of ions
 —, effect on coagulation rate
Coagulation
 —, in electrolyte mixtures
 —, in strong electrolytes
 —, in weak electrolytes
 —, rate of
Ion pairs
 —, determination of
 —, stability constants of
 — —, $MgSO_4$
 — —, $LaSO_4^+$

MCC-55 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta CCACAA 45 (4) B13—B14 (1973)

Chelating Properties of Proline and Some Prolyl Peptides

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Acid dissociation constants of proline, L-prolylglycine, and glycil-L-proline were determined by a potentiometric titration method at 20 °C in 0.37 mol dm^{-3} $NaNO_3$. The composition of the complexes formed by the above mentioned ligands with bivalent ions of nickel, copper, zinc, cadmium, and lead was also ascertained and their stability constants determined. The complexes of investigated dipeptides were found to be more stable than would be predicted on the ground of theoretical considerations. This could be explained by assuming an additional coordination site — presumably the peptide nitrogen — to be active in complex formation although not taking part in protonation equilibria. The copper(II)-prolylglycine sistem proved to be more involved than the others so that only rough estimates of the complex formation constants could be obtained.

The work was reported at the *Third Yugoslav Congres of Pure and Applied Chemistry,* Ljubljana 1972.

Examiners: Prof. O. A. Weber, Prof. B. Težak, and Dr. V. Katović.

Oral examination: December 15, 1972.

Thesis deposited at the University Library, Zagreb, Feculty of Science, Univ. of Zagreb, and Institute for Medical Research, Yugoslav Academy of Sciences and Arts, Zagreb.

(71 pages, 6 tables, 9 figures, 46 references, original in Croatian).

B. KUŽNAR

MCC-55 (Univ. Zagreb)

1. Chelating Properties of Proline and Some Prolyl Peptides

I. Kužnar, B.

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Cadmium(II)
Chelate Compounds
Copper(II)
Glycyl-L-proline
Lead(II)
Nickel(II)
Proline
Prolylglycine
Zinc(II)

MCC-56 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta CCACAA 45 (4) B15—B16 (1973)

Electrometric Determination of the Constitutive Ion Adsorption on the Silver Halides

S. Musić

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The adsorption of I^- ions on negative AgI sols was determined potentiometrically using the Ag/Ag_2S electrode for the measurement of I^- and Ag^+ ion activities. For the fresh sols *in statu nascendi* the linear decrease of the adsorbed amount with pI (pAg) and the change of the molar adsorption capacity calculated by the formula $(100 \times \Delta \text{ ads. } I^-)/([AgI] \times \Delta \text{ pI})$ at pI = pAg = 8, were reproduced. Molar adsportion capacity decreases with the increase of the concentration of NaI and $AgNO_3$ solutions, with the age of the sols and decreases too in the critical region of the concentration of counter ions from maximal to an approximately ten times lower value. The concentrations of counter ions in which the capacity is decreased to 50% of its maximal value depend on the valency of the counter ion and follow the linear formulation of the Schulze-Hardy rule. It was also demonstrated that the variation of the adsorbed amount of I^- ions on dried »isoelectric« AgI equals the change in the amount of counter ions within the limits of experimental error. The decrease of the molar adsorption capacity in the flocculation region was demonstrated for Triton X 305, Na-dodecyl sulfate, *n*-miristylamine nitrate, *n*-laurylamine nitrate.

Examiners: Prof. M. Mirnik, Dr. R. Despotović and Prof. R. H. Wolf.

Oral examination: December 20, 1972.

Thesis deposited at the University of Zagreb.

(58 pages, 15 figures, 55 references, original in Croatian)

R. DESPOTOVIĆ

MCC-56 (Univ. Zagreb)

1. Electrometric Determination of the Constitutive Ion Adsorption on the Silver Halides

I. Musić S.

II. Institute »Ruđer Bošković«, 41001 Zagreb, Croatia, Yugoslavia

Adsorption capacity
AgI suspension, aging of
Potentiometry, determination
  of adsorption capacity by
Schulze-Hardy rule
Surfactants,
  influence on adsorption capacity

MCC-57 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta CCACAA 45 (4) B17—B18 (1973)

Investigation of the Process of Heterogeneous Exchange in the Systems AgI—I/Fluorescein and AgI—I/tetrachloro-tetraiodo-fluorescein

Z. Selir

Institute »Ruđer Bošković«, 41000 Zagreb, Croatia, Yugoslavia

The purpose of this work was to establish whether it is possible to apply consequently the Trauber principle to the organic molecules when their molecular mass increases by introducing small and heavy inorganic substituents. Sodium fluoresceinate (FLN) has been chosen as a model for the organic substance and sodium tetrachloro-tetraiodo-fluorescienate (BRB) has been used as a derivative Silver iodide sol, prepared *in statu nascendi* has been taken as the colloid system. General conditions were as follows: 0.01 M NaI, 0.01 M AgI, FLN or BRB from 1×10^{-6} to 3×10^{-3} M. The experiments were carried out using various methods: tensometry, potentiometry, conductometry, turbidimetry, ultramicroscopic electrophoresis, X-ray diffractometry, microphotography, radiometric determination of the adsorption capacity and radiometric following of the process of heterogeneous exchange.

The results show that FLN and BRB behave differently under the same conditions of investigation: in relation to FLN, BRB behaves as an organic molecule with a large number of carbon atoms. Although these two substances have the same carbon skeleton, they have different inorganic substituents. From the analysis of the cited experiments and from the process of heterogeneous exchange it was concluded that the Trauber rule can also be extended to the effects caused by the increase of the mass of the organic molecule by introduction of heavier inorganic substituents.

Examiners: Dr. R. Despotović, Prof. M. Mirnik and Prof. R. H. Wolf.

Oral examination: December 22, 1972

Thesis deposited at the University of Zagreb.

(82 pages, 2 tables, 23 figures, 60 references, original in Croatian)

R. DESPOTOVIĆ

MCC-57 (Univ. Zagreb)

1. Investigation of the Process of Heterogeneous Exchange in the Systems AgI—I^-/Fluorescein and AgI—I^-/Tetrachloro-tetraiodo-fluorescein

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Exchange, heterogeneous
^{131}I
Fluorescein
  —, tetrachloro-tetraiodo
Silver iodide sol
  autodiffusion
  recrystallization
Ostwald ripening
Traube rule

MCC-58 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta CCACAA 45 (4) B19—B20 (1973)

Characterization of the Precipitation System of Silver Chloride in the Solutions of Some Electrolytes and Macromolecules

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The precipitation of silver chloride was investigated in the presence of nonionic surface-active agents of the OPE-series (Triton X-305), amphoteric polyelectrolyte gelatin (isoelectric point at pH = 4.7) and some electrolytes as: KNO_3, $Ba(NO_3)_2$, $La(NO_3)_3$. The concn. range between 10^{-3} and 10^{-4} mol dm^{-3} AgCl was examined.

The precipitation was followed by tyndallometric measurements and the sign of the particle charge was confirmed by microscopic electrophoresis. The effect of added substances upon the shape and size of AgCl crystallites formed by cooling of supersaturated solns was also investigated by light and electron microscopy.

Stabilization and flocculation effects of triton and gelatin were obtained on both positively and negatively charged silver chloride sols *in statu nascendi* at 20 °C. Flocculation has been less expresed on negatively charged sols, which are more stable than the positively charged sols.

AgCl crystallites of the approximately equal shape and size were prepared by cooling the complex solns from 90 °C to 20 °C in the presence of triton or gelatin in greater concn. than c. s. c. or in the presence of electrolyte in greater concn than c.c.c. At their flocculating or coagulating concns, however, crystallites of various size and shape were obtained.

In dependence of the concentrational relationships of AgCl and of the added substances, studied systems show various colloid and crystalline characteristics.

Examiners: Prof. B. Težak, Prof. R. Wolf and Dr. R. Despotović.

Oral examination: January 12, 1973.

Thesis deposited at the Faculty of Science, University of Zagreb.

(104 pages, 43 figures, 53 references, original in Croatian)

N. STUBIČAR

MCC-58 (Univ. Zagreb)

1. Characterization of the Precipitation System of Silver Chloride in the Solutions of Some Electrolytes and Macromolecules

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II. Institute of Physical Chemistry, University of Zagreb, 41000 Zagreb, Croatia, Yugoslavia

Flocculation
  by nonionic surface-active agents
    (Triton X-305)
  by amphoteric polyelectrolyte gelatin
Monodisperse sol(s)
  of silver chloride
Silver chloride sol(s)
  precipitation of
  flocculation of
  crystallization of
Stabilization
  by gelatin
  by nonionic surface-active agents
    (Triton X-305)

MCC-59 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta CCACAA 45 (4) B21—B22 (1973)

Adsorption of Tri-*n*-butyl Phosphate from Aqueous Solutions on the Mercury Electrode and its Influence ou the Electrode Processes of some Metal Ions

D. Krznarić

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The adsorption of tri-*n*-butyl phosphate (TBP) on the mercury electrodes from aqueous solutions, and its influence on the electrode processes of Tl(I), Cu(II), Cd(II), Pb(II), In(III), and U(VI) was investigated by means of d. c. polarography, Kalousek commutator, and double potential-step chronocoulometry.

Four different methods for the determination of TBP concentration, in aqueous solutions, are proposed. Two of the methods employ Kalousek commutator, while the other two consist of different modes of application of double potential-step chronocoulometry. These methods enable the determination of TBP concentration in aqueous solution from 5×10^{-6} M up to 2×10^{-3} M.

The investigated metal ions can be divided into four different groups according to their electrode processes in the presence of TBP. One type of behaviour is represented by the Tl(I) ion, the next one by Cu(II), Cd(II), and Pb(II) ions, the third by In(III), and the fourth one by the U(VI) ion.

The Presence of TBP does not influence the electrode processes of Tl(I), while the electrode processes of In(III), in chloride medium, are almost completely inhibited by the layer of TBP on the electrode. From the current-time curves of Pb(II), Cd(II), and Cu(II) it is evident that the inhibition of the electrode processes, on the covered surface, increases with the acidity of the solution and also with the increase of the bulk concentration of TBP. Contrary to this, the change of the concentration of TBP does not affect the reduction process of the uranyl ion. This can be attributed to the fact that the uranyl ion is not reduced to the amalgam like the other investigated metal ions. It is therefore supposed that the possible effect of the formed amalgam should be considered.

No adsorption of the reactants was found by chronocoulometric measurements for the electrode processes of the mentioned metal ions in the presence of TBP. It was concluded therefore that the complex formation between metal ions and TBP does not occur on the electrode surface prior to the electrode process. The adsorbed TBP most probably affects chemical reactions, such as dehydration, which could be involved in the electrochemical processes of Cd(II), Pb(II), Cu(II), and In(III) ions.

Part of this work was reported at the *3rd Yugoslav Congress on Pure and Applied Chemistry*, Ljubljana 1972. A part of the thesis was published in: *J. Electroanal. Chem.* 44 (1973) 401—410.

Examiners: Dr. M. Branica, Prof. I. Filipović, and Prof. B. Težak.

Oral examination: January 29, 1973.

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

(115 pages, 32 figures, 81 references, original in Croatian)

D. KRZNARIĆ

MCC-59 (Univ. Zagreb)

1. Adsorption of Tri-*n*-Butyl Phosphate from Aqueous Solutions on Mercury Electrode and Its Influence on the Electrode Processes of some Metal Ions

I. Krznarić D.

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Adsorption, of tri-*n*-butyl phosphate on mercury electrodes
Metal ions, influence of the adsorption of tri-*n*-butyl phosphate on the electrode processes of
Tri-*n*-butyl phosphate,
  adsorption of, on mercury electrodes from aqueous solutions,

MCC-60 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta CCACAA 45 (4) B23—B24 (1973)

Graph Theory and Molecular Orbitals

I. Gutman

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Graph theory (GT) has been proved to be a suitable mathematical technique for molecular orbital (MO) studies of conjugated hydrocarbons. Necessary notions of GT including Sachs theorem are presented. The commutation relations between the Hückel Hamiltonian and the graph adjacency matrix (A) is discussed. It is shown that the MO's and MO energies are identical with the eigenvectors and eigenvalues of A, respectively.

The pairing theorem is analysed from several points of view. It is shown that the number of zero eigenvalues (NZE) of A determines the stability of conjugated hydrocarbons. Nine new theorems for calculating and/or estimating the NZE's are presented.

The total π-electron energy (E) is shown to be a function of A. It is found that the Hückel $(4m + 2)$-rule can be generalized to all conjugated hydrocarbons and a new »loop rule« has been derived. Using the loop rule approximate expressions for E and general rules governing the dependence of E on molecular structure are obtained.

A part of this work was published in *Theor. Chim. Acta* **26** (1972) 67, *Croat. Chem. Acta* **44** (1972) 365, and *Chem. Phys. Lett.* **17** (1972) 535.

Examiners: Dr. N. Trinajstić, Dr. D. Cvetković, and Dr. Z. Maksić.

Oral examination: February 9, 1973.

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

(47 pages, 37 references, original in Croatian)

I. GUTMAN

MCC-60 (Univ. Zagreb)

1. Graph Theory and Molecular Orbitals

I. Gutman I.

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Conjugated hydrocarbons
Graph theory
Hückel
  —, rule
  —, theory
Molecular orbitals
Topology

MCC-61 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta CCACAA 45 (4) B25—B26 (1973)

Secondary Deuterium Isotope Effecats in Solvolysis of Cyclopentyl *p*-Bromobenzenesulfonate. Stereochemistry of *E1* and S_N1 Reactions

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Secondary α- and β-isotope effects in solvolysis of cyclopentyl-I-*d*, *cis*-cyclopentyl-2-d, *trans*-cyclopentyl-2-d, and cyclopentyl-2,2,5,5-d_4 brosylates were measured in different ethanol—water (E—W) and trifluoroethanol—water (TFE) mixtures. The magnitude of the effects indicates that in the E—W mixtures the products are derived by rate determining subtitution and elimination on the reversibly formed intimate ion pair. However, the change to TFE—W mixtures causes a change in mechanism; in these solvents the formation of the solvent separated ion pair is rate determining and the products are formed in fast subsequent attack on this intermediate. Consistent with this interpretation of the isotope effects, the elimination in E—W mixtures was found to be stereospecifically *trans*, while in TFE—W mixtures it is nonstereospecific. It was also determined that the configuration of the cyclopentanol formed in both types of solvent was inverted in comparison to the starting material. A detailed analysis provides an estimation of the isotope effects in each of the individual steps in the reaction.

A part of this work was published in *J. Amer. Chem. Soc.* **95** (1973) 7722.

Examiners: Dr. K. Humski, Dr. S. Borčić and Dr. A. Deljac.

Oral examination: June 19, 1973.

Thesis deposited at the University of Zagreb.

(81 pages, 10 figures, 20 tables, 74 references, original in Croatian)

V. SENDIJAREVIĆ

MCC-61 (Univ. Zagreb)

1. Secondary Deuterium Isotope Effects in Solvolysis of Cyclopentyl *p*-Bromobenzenesulfonate. Stereochemistry of *E1* and S_N1 Reactions

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Cyclopentyl derivatives, solvolysis of
Isotope effects, deuterium, secondary
Solvolysis
Stereochemistry, of *E1* and S_N1 reactions

MCC-62 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta CCACAA 45 (4) B27—B28 (1973)

Solvolysis of Menthyl and Neomenthyl Tosylates. Conformation and Secondary Deuterium Isotope Effects

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Transition states geometries in solvolyses of menthyl and neomenthyl tosylates were studied by means of secondary α- and β-deuterium isotope effects and product analyses.

α-Effects of both menthyl and neomentyl tosylates are »normal« indicating that in the transition state(s) there is almost no covalent bonding between the α-carbon and either the leaving group or the nucleophyle.

The value of β-d_3 isotope effect of neomenthyl tosylate, however, is approximatelly twice as high as that of mentyl tosylate.

The neomenthyl tosylate has two hydrogen atoms antiparallel to the leaving group, therefore ideally suited for participation. The only substitution products found in solvolysis of neomentyl tosylate were rearranged alcohol, 1-methyl-4-isopropyl cyclohexane-4-ol, and some amount of alcohol with retained configuration at the reaction center, neomenthol. In olefinic products Δ^3-menthene predominates although exocyclic. $\Delta^{4,8}$-menthene was also formed. Such products composition indicate that a hydrogen bridged cation exists as an intermediate in solvolysis of neomenthyl tosylate. This is in acord with the observed high value of β-deuterium isotope effect.

Menthyl tosylate has no β-hydrogens antiparallel to the leaving group. In a twisted transition state, one of β-hydrogen atoms could assume the antiparallel arrangement favourable for participation. However, the observed β-d_3-isotope effect is quite normal indicating that menthyl tosylate solvolyzes *via* a rigid chair like transition state. The main substitution product in solvolysis (besides ethers) is menthol. This could also be explained by the chair-like transition state. In a twisted, half-chair conformation of transition state the product with predominately inverted configuration at the reaction center would be formed.

It may be concluded that β-deuterium isotope effects in combination with product analysis are an efficient tool for conformational analyses of transition states in solvolyses of cyclohexyl derivatives. Both neomenthyl and menthyl tosylates solvolyze *via* chair like transition states but only the solvolysis of neomenthyl tosylate includes the participation of tertiary β-hydrogen.

Disubstituted cyclohexyl derivatives with equatorial leaving group appear to solvolyze *via* a rigid transition state only if a voluminous alkyl substituent is in the β-position to the leaving group while another alkyl group is substituted at the position 3 or 4.

Examiners: Dr. D. E. Sunko, Dr. Z. Majerski, and Dr. T. Cvitaš

Oral examination: October 31, 1973.

Thesis deposited at the University of Zagreb.

(101 pages, 20 figures, 22 tables, 92 references, original in Croatian)

S. HIRŠL-STARČEVIĆ

MCC-62 (Univ. Zagreb)

1. Solvolysis of Menthyl and Neomenthyl Tosylates. Conformation and Sencondary Deuterium Isotope Effects

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Conformation, of transition states
Cyclohexyl derivatives, solvolysis of
Isotope effects, deuterium, secondary
Participation, neighbouring hydrogen
Solvolysis

MCC-63 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta CCACAA 45 (4) B29—B30 (1973)

Glucosyl Esters of Amino Acids and Dipeptides

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Fully protected D-glucopyranosyl esters of amino acids and dipeptides were synthesized *via* two routes involving direct participation of imidazole in the 1-ester linkage formation: (a) the »accelerated active-ester« method, and (b) the imidazol-promoted dicyclohexylcarbodiimide condensation. The amino function in the aglycon group was protected by the *tert*-butyloxycarbonyl or benzyloxycarbonyl group, and the hydroxyl functions of the sugar moiety were fully benzylated or acetylated. The products were obtained in good yields as anomeric mixtures which were sepd. and fully characterized.

Catalytic hydrogenation of the tetrabenzyl ethers of 1-*O*-*tert*-butyloxycarbonylaminoacyl-β-D-glucopyranoses afforded the corresponding 1-*O*-acylaminoacyl-β-D-glucopyranoses; their structures were confirmed by conversion into the peracetylated derivs. Debenzylation of the α-anomers rezulted in 1 → 2 O-acyl migration; the rearrangement product of 1-*O*-(*tert*-butyloxycarbonyl-L-alanyl)-α-D-glucopyranose was identified by conversion into the corresponding 1,3,4,6-tetra-*O*-acetyl deriv, which was also synthesized by definitive methods. Deprotection of the *tert*-butyloxycarbonyl group with trifluoroacetic acid afforded 1-*O*-glycyl-β-D-glucopyranose in the form of crystalline trifluoroacetate.

Catalytic hydrogenation of the fully benzylated 1-*O*-(*N*-benzyloxycarbonyl-dipeptidyl)-β-D-glucopyranoses (dipeptidyl = glycylglycyl, L-alanylglycyl) gave the corresponding glucosyl esters as the cryst, monooxalate salts. Their structures were confirmed by conversion into the corresponding *N*-acetyl derivatives.

Part of this thesis are published in *Carbohyd. Res.* 22 (1972) 245; *ibid.* 29 (1973) 25.

Examiners: Dr. D. Keglević, Prof. D. Sunko, and Dr. N. Pravdić.

Oral examination: November 7, 1973.

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

(88 pages, 12 figures, 5 tables, 83 references, original in Croatian)

Š. VALENTEKOVIĆ

MCC-63 (Univ. Zagreb)

1. Glucosyl Esters of Amino Acids and Dipeptides

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Accelerated active ester method
Amino acids, glycosyl esters with
Dicyclohexylcarbodi-imide method
Dipeptides, glucosyl esters with
Glucosyl esters
Imidazole
Sugar-amino acid ester linkage

MCC-64 (Univ. Zagreb)
Master of Science Thesis

Croat. Chem. Acta CCACAA 45 (4) B31—32 (1973)

Catalytic Hydrogenation of 2,2'-*Bis*oxalines

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N,N'-Bis(α-hydroxyalkyl)ethylenediamines can be obtained by hydrogenolysis of 2,2'-*bis*oxazolines with Pt/Rh or Adams catalyst. This method was used for the preparation of well known tuberculostatics (+)-*N,N'-bis* (1-hydroxymethylpropyl)-ethylenediamine from (—)-4,4'-diethyl-2,2'-(*bis*oxazoline in almost quantitative yield.

The best reaction conditions were found to be Pt/Rh catalyst, methanol, ethanol or ethylacetate as a solvent, 50 atm hydrogen pressure and room temperature.

A series of unknown 2,2'-*bis*oxazolines was prepared but only 2,2'-*bis*oxazolines without an aliphatic or aromatic group in the 2,2'-positions were successfully hydrogenolized.

Examiners: Dr. K. Jakopčič, Dr. I. Butula, and Prof. I. Lovreček

Oral examination: November 27, 1973.

Thesis deposited at the University of Zagreb.

(76 pages, 22 figures, 3 tables, 38 references, original in Croatian)

G. STROHAL

MCC-64 (Univ. Zagreb)

1. Catalytic Hydrogenation of 2,2′-*Bis*oxalines

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(+)-*N*,*N*′-*Bis*(1-hydroxymethylpropyl)-ethylenediamine, preparation of
2,2′-*Bis*oxazolines, hydrogenolysis of
Tuberculostatics

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Institut za organsku kemiju i biokemiju Sveučilišta u Zagrebu i Institut za farmakologiju i toksikologiju Medicinskog fakulteta, Sarajevo

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Bull. Sci. Cons. Acad. Sci. Arts RSF Yougoslavie Sect. A **17** (1972) 147.

BCC-1178

K. Balenović, M. Mihalić, S. Ehrlich, and N. Paulić

Institut za organsku kemiju i biokemiju Sveučilišta u Zagrebu

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N. Blažević, F. Kajfež, V. Šunjić i D. Kolbah

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Institut za organsku kemiju i biokemiju Sveučilišta u Zagrebu

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Bull. Sci. Cons. Acad. Sci. Arts RSF Yougoslavie Sect. A **17** (1972) 148.

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Institute »Ruđer Bošković«, Zagreb (Yugoslavia)

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J. Less-Common Metals **29** (1972) 407.

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M. Fleš

Imunološki institut, Zagreb

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Institute »Ruđer Bošković«, P. O. Box 1016, 41001 Zagreb, Croatia, Yugoslavia

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Ruđer Bošković Institute, Zagreb, Croatia, Yugoslavia

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Zavod za kemiju Farmaceutsko-biokemijskog fakulteta i Institut za organsku i analitičku kemiju Sveučilišta u Zagrebu

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L. Klasinc and H. Guesten

Institut für Radiochemie, Kernforschungszentrum Karlsruhe, Karlsruhe, Germany

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Institut für Physikalische Chemie der Universität Ulm

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Harvard Medical School, Department of Dermatology at the Massachusetts General Hospital, Boston, Massachusetts 02114

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S. Kukolja

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R. Nemeth and E. Matijević

Institute of Colloid and Surface Science and Department of Chemistry, Clarkson College of Technology, Potsdam, New York 13676

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J. Colloid Interface Sci. **41** (1972) 532.

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J. Petričić, D. Kuštrak i J. Kuftinec

Zavod za ispitivanje i kontrolu lijekova, Zagreb, Moše Pijade 160 i Zavod za farmakognoziju, Farmaceutsko-biokemijski fakultet, Zagreb, Marulićev trg 20

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Farm. Glas. **28** (1972) 427.

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Institute »Boris Kidrič«, Vinča, Yugoslavia and Institute »Ruđer Bošković«, Zagreb, Yugoslavia

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M. Randić and A. Rubčić

Faculty of Science and Mathematics, Departments of Chemistry and Physics, University of Zagreb, Zagreb, Croatia (Yugoslavia)

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Department of Chemistry, Faculty of Science and Mathematics, University of Zagreb, Zagreb, Croatia, Yugoslavia and Laboratory of Physical Chemistry, Institute »Boris Kidrič«, Belgrade, Serbia, Yugoslavia

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Ruđer Bošković Institute, Zagreb and Faculty of Pharmacy and Biochemistry, University of Zagreb, Zagreb, Yugoslavia

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Institute Ruđer Bošković, Zagreb, Yugoslavia and College William and Mary, Williamsburg, Virginia 23185, U. S. A.

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Institute of Organic Chemistry and Biochemistry of the University, Strossmayerov trg 14, Zagreb, Croatia, Yugoslavia

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Institute of Organic Chemistry and Biochemistry of the University, Strossmayerov trg 14, YU-41000 Zagreb and Department of Pharmacy and Biochemistry, Zagreb

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Chemistry Department, The University of Tennessee, Knoxville, Tennessee 37916, U. S. A. and Institute »Ruđer Bošković«, 41001 Zagreb, Croatia, Yugoslavia

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N. Bodor, M. J. S. Dewar, and Z. B. Maksić

Department of Chemistry, University of Texas at Austin, Austin, Texas 78712

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BCC-1214

Lj. Bokić and I. Filipović

Institute of Inorganic and Analytical Chemistry, University of Zagreb, and Laboratory of Inorganic Chemistry, Faculty of Technology, Zagreb (Yugoslavia)

Kinetics of Polarographic Reduction of Pentavalent Vanadium in Alkaline Phosphate Buffer Solution

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BCC-1215

M. Bravar, V. Rek and R. Kostelac-Biffl

Institute of Organic Chemical Technology, Faculty of Technology, University of Zagreb, Zagreb, Yugoslavia

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BCC-1216

J. Chevalet and V. Žutić

Center for Marine Research, Ruđer Bošković Institute, P. O. Box 1016, 41000 Zagreb, Croatia (Yugoslavia)

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D. Cvetković, I. Gutman, and N. Trinajstić

Faculty of Electrical Engineering, University of Belgrade, 11001, Belgrade, Serbia, Yugoslavia and Institute Ruđer Bošković, P. O. Box 1016, 41001 Zagreb, Croatia, Yugoslavia

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BCC-1218

B. Ćosović and M. Branica

Center for Marine Research, Ruđer Bošković Institute, Zagreb, Croatia (Yugoslavia)

Study of the Adsorption of Organic Substances at a Mercury Electrode by the Kalousek Commutator Technique

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L. Duić, S. Shrinivasan, and P. N. Sawyer

Electrochemical and Biophysical Laboratories of the Vascular Surgical Services, Department of Surgery, State Univeristy of New York, Downstate Medical Center, Brooklyn, New York 11203

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Electrochemical and Biophysical Laboratories of the Vascular Surgical Services, Department of Surgery, State University of New York, Downstate Medical Center, Brooklyn, N. Y. 11203 (U. S. A.)

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Institute »Ruđer Bošković«, Zagreb, Croatia, Yugoslavia

Radical Pairs in Irridiaated Single Crystals of 1-methyl Uracil

Mol. Phys. **26** (1973) 605.

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P. McFadyen and E. Matijević

Institute of Colloid and Surface Science and Department of Chemistry, Clarkson College of Technology, Potsdam, New York 13676

Precipitation and Characterization of Colloidal Copper Hydrous Oxide Sols

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Copper Hydrous Oxide Sols of Uniform Particle Shape and Size

J. Colloid Interface Sci. **44** (1973) 95.

BCC-1224

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Institute »Ruđer Bošković«, 41001 Zagreb, Croatia, Yugoslavia

Secondary Dueterium Isotope Effects in the Solvolysis of Cyclobutyl and Cyclopropylcarbinyl Methanesulfonates

J. Org. Chem. **38** (1973) 1881.

BCC-1225

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Institute »Ruđer Bošković«, 41001 Zagreb, Croatia, Yugoslavia and Department of Chemistry, Harvard University, Cambridge, Massachusetts 02138

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Laboratory of General and Inorganic Chemistry, Faculty of Science, University of Zagreb, 41001 Zagreb, Croatia, Yugoslavia

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Inorg. Chem. **12** (1973) 514.

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Institut für Radiochemie Kernforschungszentrum Karlsruhe, Karlsruhe, Germany

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Laboratory for Analytical Chemistry, Faculty of Science and Institute of Inorganic and Analytical Chemistry, University of Zagreb, Yugoslavia

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BCC-1231

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BCC-1232

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Institut Chimie, Université Louis Pasteur, BP 296/R8, 67008 Strasbourg Cedex, France

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BCC-1233

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Laboratory of General and Inorganic Chemistry, Faculty of Science, The University, P. O. Box 153, 41000 Zagreb, Yugoslavia

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BCC-1236

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Rev. Roum. Chim. **18** (1973) 89.

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Acta Crystallogr. Sect. B **29** (1973) 864.

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Institute of Physical Chemistry, Technology Department, University of Zagreb, Zagreb, Yugoslavia and »Pliva«, Pharmaceutical and Chemical Works, Zagreb (Yugoslavia)

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Institute of Electrochemistry and Electrochemical Technology, Faculty of Technology, University of Zagreb, 41000 Zagreb, Savska c. 16/I (Yugoslavia)

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Planta **110** (1973) 57.

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Institute »Ruđer Bošković«, 41000 Zagreb, Yugoslavia and Department of Chemistry, The University of Tennessee, Knoxville, Tennessee 37916

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J. Phys. Chem. **77** (1973) 1520.

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Institute of Colloid and Surface Science and Department of Chemistry, Clarkson College of Technology, Potsdam, New York 13676

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Department of Chemical Technology, Twente University of Technology, Enschede (The Netherlands) and Institute »Ruđer Bošković«, Zagreb, Yugoslavia

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BCC-1251

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Institute »Ruđer Bošković«, Zagreb, Croatia, Yugoslavia

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Department of Chemistry, Faculty of Pharmacy and Biochemistry, University of Zagreb and Institute »Ruđer Bošković«, Zagreb, Croatia, Yugoslavia

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Chemistry Department, State University of New York at Buffalo, Buffalo, N. Y. 14214 (U.S.A.)

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J. Electroanal. Chem. **47** (1973) 329.

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Zavod za ispitivanje i kontrolu lijekova SRH, Zagreb, M. Pijade 160 i Zavod za farmakognoziju Farmaceutsko-biokemijskog fakulteta, Zagreb, Marulićev trg 20

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Farm. Glas. **29** (1973) 235.

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Laboratory of Inorganic Chemistry, Faculty of Technology and Institute of Inorganic and Analytical Chemistry, University of Zagreb, Zagreb, Croatia (Yugoslavia)

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J. Electroanal. Chem. **42** (1973) 433.

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Laboratory for Electrophoresis, Institute »Ruđer Bošković«, Zagreb, Yugoslavia

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J. Inorg. Nucl. Chem. **35** (1973) 1987.

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Institute »Ruđer Bošković«, 41001 Zagreb, P. O. Box 1016, Bijenička 54, Croatia, Yugoslavia

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Electrochemical and Biophysical Laboratories of the Vascular Surgical Services, Department of Surgery, State University of New York, Downstate Medical Center, Brooklyn, New York 11203

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CRC, Compagnia de Ricerca Chimica, Chiasso, Switzerland, and Institute of Organic Chemistry and Biochemistry, University of Zagreb, Yugoslavia

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Tracer Laboratory, Institute »Ruđer Bošković«, 41001 Zagreb, Yugoslavia

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Institute »Ruđer Bošković«, Zagreb, Yugoslavia and College of William and Mary, Williamsburg, Virginia 23185, U.S.A.

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Institute »Ruđer Bošković«, Zagreb, Yugoslavia and College William and Mary, Williamsburg, Virginia 23185, USA

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Faculty of Pharmacy and Biochemistry, University of Zagreb, and Institute »Ruđer Bošković«, Zagreb, Croatia, Yugoslavia

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