

METHORICS OF THE PRECIPITATION PROCESSES. XXIV. FLOCCULATION PHE-NOMENA OF NON-IONIC SURFACE-ACTIVE AGENTS ON SILVER IODIDE SOLS

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An explanation concerning the mechanism of sensibilization, flocculation and stabilization action of non-ionic surface-active agents T-X-305 and T-X-705 on the negatively and positively charged silver iodide stable sols *in statu nascendi* was attempted by means of tyndallometric and microscopic investigations at 293, 313 and 333 K.

The flocculation process is explained as a stereospecific acsorption interaction of NSAA with colloid, in which the primary particles of Agl showing the crystalline structure, become coated and surrounded with NSAA molecules and micelles and form the aggregates of definite size during the stabilization and flocculation processes. The maximum aggregation degree is observed in the region of sensibilization and in the flocculation maximum. The particles are disaggregated during the stabilization process.

The discontinuities in $t_c - c_{in}$ curves show the different stability of each step in flocculation/deflocculation processes as a stereospecific phenomena and may be seen by comparing the corresponding phenomena followed by different measurements and calculations presented in Fig. 1.

The problem of flocculation is discussed according to the concepts of B. Težak (1) and V.K. La Mer (2).

Colloids

sols, silver iodide, interaction of with non-ionic surface--active agents,

Flocculation action of non-ionic surfaceactive agents on,

Precipitation phenomena of,

Silver iodide stable sols of,

Surfactants flocculation phenomena of on silver iodide sols,



REFERENCES

B. Težak, *Discuss. Faraday Soc.* 42 (1955) 175.
 V. K. La Mer *Discuss. Faraday Soc.* 42 (1966) 248.

Fig. 1

Presentation of the precipitation processes in the flocculation maximum of positively charged AgI sol $(c_{AgI} = 1.0 \times 10^{-3} \text{ mol dm}^{-3}, \text{ excess } c_{AgNO_3} = 1.0 \times 10^{-3} \text{ mol dm}^{-3}$): a) by measuring tyndellometric values and

 a) by measuring tyndallometric values and electrophoretic mobility (ultramicroscopic electrophoresis);

b) by determination of critical times and presentating the aggregation processes in sensibilization, flocculation and stabilization region by light micrographs (total magnification 480 x), and by plotting the particle sizes versus concentration of T-X-705;

c) the sizes of secondary aggregates by counting particles in the field of light microscope, and

d) the sizes of primary aggregates calculated by DO, method describe the maximum corresponding to the tyndallometric maximum and t_c minimum.

Abstractor: Đ. Težak



SEMIEMPIRICAL STUDIES OF INNER-CORE ENERGY LEVELS. PART 7. ESCA SHIFTS OF GERMANIUM IN MOLECULAR SYSTEMS

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X-ray photoelectron spectroscopy shifts of the $3p_{3/2}$ energy levels of germanium in different chemical moieties were studied by using the semiempirical SCC-MO method. The calculations were performed within the framework of the electrostatic potential method in the point-charge approximation (1). Relaxation energies were explicitly taken into account by employing the equivalent core and transition potential methods (2). It was found out that the relaxation energy plays a decisive role in determining shifts along the series GeH₄— Ge(CH₃)₄. The results obtained by the equivalent core and transition potential methods are similar being in the same time in good agreement with experiment. Performance of various semiempirical schemes in evaluating ESCA shifts is briefly discussed.

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 O. Goscinski, G. G. Howat, and T. Abey, *J.Phys.* B 8 (1975) 11.

Molecular orbital

- semiempirical SCC, of compounds containing germanium, photoelectron spectra in relation to,
- Photoelectric emission X-ray, of compounds containing germanium,

Abstractor: N. Trinajstić



THE INVESTIGATION OF THE STABILITY OF 2-OXOPROPANOATE COMPLEXES OF COBALT(II), NICKEL(II), COPPER(II), ZINC(II), CADMIUM(II), AND LEAD(II) IN AQUEOUS SOLUTIONS

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Stability constants of 2-oxopropanoate complexes of Co^{+2} , Ni^{+2} , Cu^{+2} , Zn^{+2} , $\text{Cd}^{\pm 2}$ and Pb^{+2} were determined in aqueous solution at (298.2 \pm 0.1) K and the ionic strength $l = 2 \mod \text{cm}^{-3}$ (adjusted with NaClO_4) by using two independent methods: spectrophotometry (1) and potentiometry with glass and quinhydrone electrodes (2,3). Ligand and metal-ion concentrations were $c_{\text{L}} \leq 100 \mod \text{m}^{-3}$, $c_{\text{M}} \leq 40 \mod \text{m}^{-3}$; the absorbancies were measured at 520 nm, 670 nm and 760 nm. The experimental data were evaluated by using a weighted non-linear least-squares algorithm (1,2) which yielded the refined values of cumulative stability constants defined as

$$\beta_n = \frac{[\mathsf{ML}_n]}{[\mathsf{M}][\mathsf{L}]^n} \cdot (\mathsf{mol} \, \mathsf{dm}^{-3})^n \tag{1}$$

The agreement of β values obtained potentiometrically and spectrophotometrically was reasonably good as can be judged from the values displayed in Table I.

Formation constants and Stability constants of metal 2-oxopropanoate complexes,

Potentiometry

metal complexes, stability constants determination,

Spectrometry

metal complexes, stability constants determination by,

Stability

of metal 2-oxopropanoate complexes,

8			
Metal ion	Method ^{a)}	lg β_1	$\lg \beta_2$
	GE	0.60	0.44
Co ⁺²	QE	0.61	0.59
	Sp.	0.71	1.31
	GE	0.73	0.81
Ni ⁺²	QE	0.72	0.83
	Sp.	0.82	0.83
	GE	1.35	2.05
Çu ⁺²	QE	1.41	2.03
	Sp.	1.39	
	05	0.70	0.54
7-+2	GE	0.72	0.54
211	QE	0.76	0.80
	GE	0.69	1.07
Cd ⁺²	QE	0.71	1.19
	GE	1 50	2.25
Pb ⁺²	OF	1.50	2.25
. ~	Safe Bar	1.01	2.30

Stability Constants of 2-Oxopropanoate Complexes $l = 2 \mod dm^{-3} (NaClO_4)$

a)GE: glass electrode potentiometry, QE: quinhydrone electrode potentiometry, Sp.: spectrophotometry

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- 3. I. Kruhak, B. Grabarić, I. Filipović, and I. Piljac, Croat. Chem. Acta 48 (1976) 119.

Abstractor: VI. Simeon



THE INFLUENCE OF SODIUM AND IODIDE IONS ON THE TRANSFORMATION OF METAPHASIC SILVER IODIDE INTO THE CRYSTALLINE SILVER IODIDE

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The influence of concentration of constitutive (AgI,I) and nonconstitutive (Na⁺) components on the kinetics and mechanism of the transformation of metaphasic silver iodide into crystalline silver iodide is studied. The metaphase fraction of silver iodide was determined as a function of the ageing time t_A . The second order kinetics of particle aggregation is assumed (1) and the following equation is used for interpretation of data:

$$1/(*f_m - p) = c_0 \cdot k_\pi \cdot t_A / (1 - b) + 1 / (1 - b)$$
(1)

where ${}^{*}f_{m}$ is the fraction of metaphase, b is the fraction of metaphasic silver iodide in secondary particles, and k_{π} is the ratio between the number of primary particles at $t_{A} = 0$ and the molar concentration of silver iodide in the system (c_{o}) , multiplied by second order rate constant of aggregation. The p value is a difference between b and a certain constant factor which depends on the sol concentration and on the exchange time, but is independent on the ageing time of the sol. Equation (1) is linear with respect to t_{A} and can be used for determination of b and k_{π} values from the experimental data.

Aging of silver iodide,

Crystallization of silver iodide,

Silver iodide metastable, transformation of into crystalline form,

1.2 o [Ag] = 0.002 mol dm-3 1.1 • [Ag] = 0.5[Na*] 1.0 log k 0.9 0.8 0.7 0.6 2.6 28 1 1.2 1.4 1.6 1.8 2.0 2.2 2.4 -log {[Na*]/mol dm-3}

The dependence of values of log k_π on the concentration (log scale) of Na⁺ ions present in the liquid phase. The values marked by empty circles correspond to systems with constant concentration of silver iodide ([Ag1] = 0.002 mol dm⁻³) and the values marked by full circles correspond to the systems with various AgI concentrations.

It is observed that k_{π} values within certain concentration range of constitutive components (as shown in the figure) is only affected by the concentration of Na⁺ ions present in the system.

Abstractor: I. Ružić

Fig. 1



THE CRYSTAL AND MOLECULAR STRUCTURE OF 3-(PYRIDYL-2')-IMIDAZO--[1,5-a] PYRIDINE

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In view of the continuing interest in the chemistry of azoloazines with bridgehead nitrogen, the crystal structure of 3-(pyridyl-2')-imidazo-[1,5-*a*]pyridine was determined from MoK*a* diffractometer data. The crystal data at 293(1) K are: $C_{12}H_9N_3$, M.W. 195.23, orthorhombic, $P_{2,1}2_{1,2}$, *a* = 0.5107(1), *b* = 1.3076(2), *c* = 1.4343(3) 'nm, V = 0.9579 nm₃³, D_m = 1.37(5) (by flotation), D_x = 1.354 Mg.m⁻³, Z = 4.

The structure was solved by direct methods (MULTAN 78) (1) and fully refined (SHELX 76) (2) to R and $R_{\rm W}$ values of 0.085 and 0.080 for 641 contributing reflexions. The crystal structure consists of discrete molecules. Most of the bond lengths and angles are within normal ranges for aromatic heterocyclic compounds (3). The interplanar angle between the mean planes through the pyridine and imidazo-pyridine parts of the molecule is 3.8°; therefore, the molecule as a whole is planar within 0.007 nm owing to two weak C-H...N interactions.

Crystal structure of 3-(pyridyl-2')imidazo-- [1,5-a] pyridine,



A schematic view of the molecule with the atomic numbering.

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Abstractor: I. Leban



AQUATION OF (DIMETHYL SULPHOXIDE)PENTAAMMINECOBALT(III) IN AQUE-OUS CETYLTRIMETHYLAMMONIUM BROMIDE SOLUTIONS

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The rate of aquation of $[Co(NH_3)_5(DMSO)]Br_3$ was measured in solutions containing the surfactant cetyltrimethylammonium bromide (CTAB) over the concentration range 9.55 x 10⁻⁴ mol dm⁻³ to 0.450 mol dm⁻³. This large change in CTAB concentration affected the rate very little and no significant amounts of $Co(NH_3)_5Br^{2+}$ were formed. On the basis of a comparison with the results of the anation in pure solvents (1,2) (DMSO and water) it was concluded that, if Br⁻ counterions were concentrated in the microregions around the positively charged CTAB micelles then there was no corresponding increase in the formation of $Co(NH_3)_5(DMSO)^{3+}\cdot Br^{-}$ ion-pairs in this region with subsequent $Co(NH_3)_5Br^{2+}$ formation.

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Aquation

of (dimethyl sulfoxide)pentaamminecobalt(III),

Cobalt, compounds

complexes, rate of aquation of,

Surfactants

cetyltrimethylammonium bromide, in aquation of cobalt(III) complexes,

Abstractor: M. Biruš



A CHIRAL PHOSPHINE (CRC-PHOS) DERIVED FROM (+)-CAMPHANIC ACID. A NEW LIGAND FOR HOMOGENEOUS ASYMMETRIC HYDROGENATION

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Camphanic acid methyl ester (1) was reduced with LAH to 2, which was tosylated in 3 (Scheme). Tosylate 3 was reacted with diphenylphosphine lithium in THF at -10 $^{\circ}$ C affording 4 (53.4 % after chromatography on silica with *n*-hexane/acetone 7:3).



(+) - Camphanic acid chiral phosphine derived from,

Hydrogenation

asymmetric of α-acetylamino cinnamic acid,

of *m*-benzoyl-phenylpropenic acid,

enentioselective, with a chiral phosphine (CRC-PHOS),

Stereochemistry

of homogeneous hydrogenation of chiral phosphine complex, Compound 4 (CRC PHOS) gave stable complex with Rh-norbornadiene perchlorate $(Rh(NBD)_2CIO_4)$ as a deep-yellow crystalline powder, which presumably has the stoichiometry $Rh[(CRC PHOS)_2(NBD)_2]CIO_4$, and crystallizes with one molecule of THF. This is concluded from spectral and analytical data. The structure is in accord with the known crystal structures of some oxo-phosphines (2,3). In the presence of this catalytic complex asymmetric hydrogenation of a-acetylamino cinnamic acid, and m-benzoyl-phenylpropenic acid, as the model prochiral substrates, yielded corresponding S-a-substituted carboxylic acids in 3-15% enantiomeric excess (e.e.).

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Abstractor: V. Šunjić



A NEW APPROACH TO SEMISYNTHETIC PENICILLINS BY MIXED ANHYDRIDE METHOD

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An efficient aminoacylation of methyl 6-aminopenicillinate I by mixed high energy phosphate anhydrides is described.

N-Phthaloylglycyldibenzylphosphate anhydride was prepared from phthaloylglycyl chloride and the silver salt of dibenzylphosphate (1). Prepared anhydride was allowed to react with methyl 6-aminopenicillate in the presence of dicyclohexylcarbodiimide to give methyl ester of phthalimidomethylpenicillin (11). The ester II was purified by preparative TLC and the structure confirmed by spectroscopic methods.

Similarly, methyl ester of benzylpenicillin (III) was prepared using phenylacetyldibenzylphosphate as an acylating agent.



Acylation of methyl 6-aminopenicillinate, Antibiotics

semisynthetic penicillins, by mixed anhydride method,

Phosphoric acid

 , N-phthaloylglycyldibenzyl anhydride application of as acylating agent,

synthesis of,

(I) $R = Pht-CH_2$ (II) $R = Ph-CH_2$ REFERENCE

1. J. C. Sheehan and V. S. Frank, *J.Amer.Chem.Soc.* 72 (1950) 1312.

Abstractor: M. V. Proštenik



SYNTHESIS OF SOME NEW THIENYLAZETIDINONES AND THIAZOLIDINONES CONTAINING PYRAZOLINES

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UNCOMPLEX

The synthesis of new azetidinones I-II and thiazo-lidinones III-IV, containing a pyrazoline molety in the para position to the phenyl ring, as potential schistosomidical agents are described.





Azetidine -, 2-one, derivatives synthesis of,

Cyclocondensation reaction of chalcones with hydrazine or phenylhydrazines,





Thus thiophene-2-carboxaldehyde condensed with 4-aminoacetophenone in ethanol gave 4-(thienylideneamino)acetophenone (1). The latter reacts with aromatic aldehydes in the presence of piperidine or aqueous alkali yielding corresponding chalcones V.

Cyclocondensation of V with hydrazine or phenylhydrazines gave *N*-phenylpyrazoline derivatives. Reaction of pyrazolines with chloroacetylchloride or mercaptoacetic acid in dry dioxane furnish title compounds I- IV, respectively.

REFERENCE

1. M. Guia and E. Bagiena, Gazz. Chem. Ital. 51 II (1921) 116.

Abstractor: M. V. Proštenik



QUALITATIVE AND QUANTITATIVE ASPECTS OF USING ECHTBLAUSALZ B TO VISUALIZE 4-HYDROXYCOUMARIN AND SOME OF ITS BIOLOGICALLY ACTIVE DERIVATIVES ON THIN-LAYER CHROMATOGRAMS

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It has been reported previously (1) that several coumarin derivatives can be sharply separated by thin-layer chromatography on silica gel, and detected on chromatograms by exciting their fluorescence. This procedure has been thereafter successfully used with the parent 4-hydroxycoumarin and its 7-hydroxylated derivative, with anticoagulant drugs belonging to the 4-hydroxycoumarin series (dicumarol, Tromexan^R, warfarin), and with two agents producing multiple biological effects – fungicidal, insecticidal, rodenticidal –, 3-(ω -bromoacetyl)--4-hydroxycoumarin and its 7-brominated derivative (2).

Meanwhile the original procedure has been modified by introducing a different mode of visualization. Instead of exciting fluorescence visible colorations are produced by reacting the chromatographically separated coumarin derivatives, *in situ*, with Echtblausalz B (EBB), a double salt containing 4,4'-bis(diazonium)-3,3'-dimethoxybiphenyl dichloride and ZnCl₂.

The modified procedure offers the following advantages: The use of Echtblausalz B distinctly lowers the minimum detectable mass, thereby increasing the detection sensitivities. The only exception has been noticed in the case of 3-(ω -bromoacetyl)--7-bromo-4-hydroxycoumarin. The compounds tested produce Chromatography, thin-layer in separation of 4-hydroxycoumarin derivatives,

Coumarins

 , 4-hydroxy, derivatives of separation of by tlc, visualization of by Echtblausalz B,



different nuances of blue which can be distinguished at a glance enabling thus a fast identification by comparison with colour standards. Quantitative evaluations of the spot area or absorbance measurements after eluting the stains result linearity ranges extending to 30 μ g except for 4-hydroxy- and 4,7-dihydroxycoumarin for which linearities do not exceed 20 μ g of applied compound.

REFERENCES

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Abstractor: Z. Štefanac



Quantitative evaluation of TL chromatograms prepared with some 4-hydroxycoumarin derivatives on 0.50 mm silica gel plates and visualised with Echtblausaltz B. For a description of materials and procedures see leggend of Table I. Increasing amounts of each compound were applied onto the baseline of the same plate and the front of developing solvent was allowed to ascend 15 cm above this line in all runs, Points on the graphs represent averages from three experiments:
, 4-hydroxycoumarin;
, warfarin 3-(3'-oxo-1'-phenylbutyl)-4-hydroxy-coumarin; Ο, dicumarol 3,3'-methylenebis(4-- hydroxycoumarin); Χ, 3-(ωbromoacetyl)-4--hydroxycoumarin; △, 4,7-dihydroxycoumarin. A, Spot area vs. amount applied. B. Absorbance of stain eluates (5 ml of eluent per spot: CHCl, for all compounds except 4,7-dihydroxycoumarin which was eluted with acetone) vs. amount applied; measurements were made at the wavelengths of maximum absorbance as indicated in the graph.



GRAVIMETRIC SEPARATION AND ESTIMATION OF COBALT AND COPPER USING N-p-CHLOROPHENYLCINNAMOHYDROXAMIC ACID

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N-Phenylbenzohydroxamic acid has been reported as a reagent for gravimetric determination of cobalt and copper (1). It has been found that the introduction of electrophilic substituents, like chlorine on *N*-phenyl ring of the hydroxamic acid decreases the stability of metal complexes (2) and consequently increases the selectivity of the reagent.

In this paper *N*-*p*-chlorophenylcinnamohydroxamic acid (*p*-CPCHA) was used as a reagent for gravimetric determination of cobalt and copper. The *p*-CPCHA forms with cobalt and copper a directly weighable complexes having definite composition $(C_{15}H_{11}NO_2Cl)_2M$, M = Co or Cu. The composition of the complexes was determined by elemental analysis and by IR spectra. The melting points of the cobalt and copper *p*-CPCHA complexes are 180 °C and 215 °C, respectively. The optimum pH range for complete precipitation of cobalt and copper was found to be between 5.4 - 6.0 and 3.4 - 3.8, respectively.

The n-CPCHA is comparable to other second and

Cobalt gravimetric determination of,

Copper gravimetric determination of, The other advantages of *p*-CPCHA as the reagent for separation and determination of cobalt and copper are high molecular weight, low conversion factors and higher selectivity.

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Abstractor: B. Grabarić



p-CHLORO SUBSTITUTED CINNAMOHYDROXAMIC ACIDS AS ANALYTICAL REA-GENTS FOR CERIUM. SPECTROPHOTOMETRIC DETERMINATION WITH *N*-PHENYL*p*-CHLOROCINNAMOHYDROXAMIC ACID

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N-Phenyl-*p*-chiorocinnamohydroxamic acid (L) forms a redishbrown complex with Ce(IV) (λ_{max} = 470 nm at pH = 9.0). The composition of the complex was found to be ML₄; it is easily extractable with chloroform (extraction time: (2···3)min) and its colour is very stable (\geq 10 d). Beer's law for the extracted complex is valid in the range 0.2··· 30 ppm of Ce. Most of metals and the most common anions do not interfere with the Ce determagation in \geq 20 µg/25 cm³ solution, provided the following limits are not exceeded: 100 mg (Ag⁺, Be⁺², Mg⁺², Ca⁺², Sr⁺², Cu⁺², Zn⁺², Ni⁺², Pb⁺², Co⁺², Ba⁺², Hg⁺², Mn⁺², Sb⁺³, Fe⁺³); 80 mg (Ta(V), Nb(V), La⁺³ and lamhanides, As(III), In(III), Cr⁺³, Th(IV), NO₂⁻, F⁻, Cl⁻, Br⁻, I⁻, SO₄⁻²); 60 mg (Ga(III), UO₂⁺²); 40 mg (Mo(VI), PO₄⁻³); 30 mg (Ti(IV), Zr(IV), V(V)).

Analytical procedura. Put 2 cm³ of 0.2% reagent solution (in ethanol 4.46 em³ of buffer solution (pH = 9) and 10 cm³ of CHCl₃; add 2 cm³ of cerium containing solution, shake vigorously for (5 ··· 10) min and allow the two layers to separate. Dry the organic phase with anhydrous Na₂SO₄. Repeat the extraction with 1 cm³ of reagent solution and wash the used Na₂SO₄ with CHCl₃. Combine all the extracts and make up to 25 cm³. Measure the absorbance at 470 nm against the reagent blank.

Cerium(IV) spectrophotometric determination of,

Spectrochemical analysis spectrophotometric, determination of cerium,

Abstractor: VI. Simeon

published in

Croatica Chemica Acta 53 (3) 477 - 483 (1980)



APPLICATION OF ANODIC STRIPPING VOLTAMMETRY FOR TRACE METAL SPE-CIATION. II. DIGITAL SIMULATION OF NEOPOLAROGRAM AT THE HANGING MERCURY DROP ELECTRODE

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An introduction to the theoretical and practical characteristics as well as the development of neopolarography is described. This new, modified anodic stripping voltammetry (ASV) method is very convenient for the determination of the ionic form of some metal ions at very low concentrations and meets concentration levels in natural waters (1).

The digital simulation program of neopolarography is given and the experimental results for the neopolarogram at the hanging mercury drop electrode (HMDE) are discussed. Because of high reproducibility of its electroactive surface and hydrodynamic characteristics of the cell, it is a very reliable indicator electrode for neopolarography. If each ASV cycle is performed on a new drop, a well developed neopolarogram is recorded. A limitation of HMDE is its relatively low sensitivity which for neopolarograms (even in stirred solutions) does not exceed 10⁻⁸ (Mdm⁻³) of the depolarizer. In natural waters concentrations of many trace metal ions are below this concentration limit and therefore this method can not be used for direct neopolarographic measurements. However, in polluted waters these concentrations are higher and the HMDE can thus be applied for direct neopolarographic measurements.

Electrodes

hanging-drop, mercury digital simulation of neopolarograms at,

as indicator electrode for neopolarography,

Polarography

neo-, theoretical and practical characteristics of,

Trace elements determination of by anodic stripping voltammetry,

Voltammetry anodic stripping, in determination of trace metals, REFERENCE

1. M. Branica D. M. Novak, and S. Bubić *Croat. Chem. Acta* 49 (1977) 539.

Abstractor: M. Lovrić



APPLICATION OF ANODIC STRIPPING VOLTAMMETRY FOR TRACE METAL SPE-CIATION. III. SIMULATED AND EXPERIMENTAL NEOPOLAROGRAMS AT ROTATED DISK ELECTRODE

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Neopolarography is an electrochemical method which consists of several ASV cycles at successively varied potentials of preelectrolysis. The digital simulation procedure for neopolarograms at the rotated disk electrode is described and the properties of reversible and nonreversible neopolarograms are discussed. The theory of neopolarography at thin mercury film electrodes is developed. The resulting simulated curves are compared with the experimental neopolarograms of lead in a perchlorate medium. The theoretical predictions are in good agreement with the experimental and simulated results. The neopolarography is well defined analytical technique (1,2) which offers many possibilities for the physico-chemical characterization of trace metals at very low concentration levels. The rotating mercury coated glassy carbon electrode is very useful because of its high sensitivty, easy maintenance and fairly well reproducibility of the results (3).

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Electrodes rotating disk, simulation of neopolarograms at,

Polarography neo-, in characterization of trace metals,

Trace elements determination of by anodic stripping voltammetry,

Voltammetry anodic stripping, in determination of trace metals,

Abstractor: M. Lovrić

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APPLICATION OF ANODIC STRIPPING VOLTAMMETRY FOR TRACE METAL SPE-CIATION. IV. DETERMINATION OF LEAD-CHLORIDE STABILITY CONSTANTS BY ROTATING MERCURY-COATED GLASSY CARBON ELECTRODE

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The stability constants of lead-chloride complexes are determined by the neopolarographic method at the rotating mercury coated glassy carbon electrode (RMCGCE). The experiments were performed using the following Pb²⁺-Cl⁻ system:

 $Pb(NO_3)_2 - NaCIO_4 - HCIO_4 - NaCI - HgCI_2 - H_2O$

1) $pH = 2; I_c = 0.7 \text{ mol dm}^{-3}$

 $1 \times 10^{-4} \le [\text{NaCl}]/\text{mol dm}^{-3} \le 0.7 \text{ mol dm}^{-3}$

2) pH = 2; $I_c = 3.0 \text{ mol dm}^{-3}$

 $1 \times 10^{-3} \leq [\text{NaCl}]/\text{mol dm}^{-3} \leq 3.0 \text{ mol dm}^{-3}$

In all solutions the concentrations of $HgCl_2$ and Pb^{+2} were 2×10^{-5} mol dm⁻³ and 2×10^{-8} mol dm⁻³, respectively. Additional experiments were performed with natural sea water acidified to pH = 2.

Electrodes

- rotating, glassy-carbon, mercurycoated, for lead chloride complexes determination,
- Formation constants and Stability constants
 - of lead chloride complexes, determination of by neopolarography,

Polarography

neo-, in lead determination,

Trace elements

determination of by anodic stripping voltammetry,

Voltammetry

anodic stripping, in determination of trace metals, The apparent stability constants were:

 $K_1 = 4.9$ $K_2 = 2.5$ $K_3 = 0.45$ $(I_c = 0.7 \text{ mol dm}^{-3})$ $K_1 = 4.1$ $K_2 = 4.3$ $K_3 = 2.9$ $(I_c = 3.0 \text{ mol dm}^{-3})$

The results obtained are in agreement with data in the literature (1).

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Abstractor: M. Lovrić



SOME CHARACTERISTICS OF CATHEPSIN B AND *a-N*-BENZOYLARGININE-*β*--NAPHTHYLAMIDE HYDROLASE FROM BOVINE LYMPH NODES

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Cathepsin B and α -N-benzovlarginine- β -naphthylamide (BANA) hydrolase were isolated from bovine lymph nodes by covalent chromatography (1), and several properties of these preparations were studied. Isoelectric focusing showed a strong band with pl = 5.0 to 5.1 for cathepsin B, and two equally strong bands with pl = 7.1 and 7.35 for BANA hydrolase. Amino acid compositions of both enzymes were determined. From CD spectra in the far UV region, the percentages of a-helix and β -structure were estimated by the method of Chen et al. (2); the respective values were 12 and 31% for cathepsin B, and only 18 and 12% for BANA hydrolase. Both enzymes were maximally stable at pH = 6; this was also the pH optimum for hydrolysis of BANA by both enzymes. For the degradation of hemoglobin, pH optima were 4.0 for cathepsin B and 6.5 to 7.0 for BANA hydrolase. Only the latter enzyme, but not cathepsin B, could use leucine-2-naphthylamide as a substrate. Both enzymes were inhibited by 4-chloromercurybenzoate, iodoacetic acid, and iodoacetamide, in accordance with their classification as thiol proteinases. Leupeptin at 10^{-6} mol dm⁻³ inhibited cathepsin B, but not the BANA hydrolase. It was concluded that BANA hydrolase is similar or even identical to cathepsin B from rat liver lysosomes (3).

Cathepsin B

from bovine lymph nodes, properties of,

Hydrolase

 –, α -N-benzoylarginine-βnaphthylamide
 from bovine lymph nodes
 properties of,
 inviting of to asthonain

similarity of to cathepsin H,

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Abstractor: Ž. Kućan



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ERYTHROMYCIN SERIES. X. INHIBITORY ACTIVITY OF SEVERAL NEW ERYTHRO-MYCIN DERIVATIVES IN CELL-FREE AMINO ACID POLYMERIZATION SYSTEMS

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Erythromycin A (1), erythromycin A 9-oxime (11), 9(S)-erythromycylamine (V), and several new derivatives of these compounds (1), were examined for their ability to inhibit the polyadenylic acid-directed synthesis of polylysine and the polycytidylic aciddirected synthesis of polyproline, in cell-free amino acid polymerization systems from *Escherichia coli* (2).

All examined compounds showed similar activities in the two systems, though the concentrations needed to cause 50% inhibition varyed from 0.5 to 1.5 μ mol dm⁻³. The following decreasing order of inhibitory activity was established: methylsuccinate of V (VI), I, V, II, methylsuccinate of II (III), p-toluenesulfonyl-V (VII), p-acetylamino-benzenesulfonyl-V (VIII), and ethylsuccinate of I (IV). The derivative of VII lacking cladinose (IX) showed lower but significant activity. Hence, none of the substitutions in the position 9 of the macrolide ring, present in these compounds, impairs the ability of I to bind the prokaryotic ribosome and inhibit its function, which is the basis for antibacterial activity of erythromycins. Antibiotics

erythromycin derivatives antibacterial activity of, substitution in macrolide ring in relation to,

inhibitory activity of, in amino acid polymerization systems,



I $R_1, R_2 = =0, R_3 = 0, CH_3 (cladinosyl), R_4 = -H$ H₁C 0CH₃

- II $R_1, R_2 = N-OH$, $R_3 = cladinosyl$, $R_4 = -H$
- 111 R₁, R₂==N-O-CO-(CH₂)-COOCH₃, R₃=cladinosyl, R₄=-H
- IV $R_1, R_2 = =0, R_3 = cladinosyl, R_4 = -CO (CH_2)_2 COOC_2H_5$
- V $R_1 = -NH_2$, $R_2 = -H$, $R_3 = cladinosyl$, $R_4 = -H$
- VI $R_1 = -NH CO (CH_2)_2 COOCH_3$, $R_2 = -H$, $R_3 = cladinosyl$, $R_4 = -H$
- VII $R_1 = -NH-SO_2 \langle O \rangle CH_3$, $R_2 = -H$, $R_3 = cladinosyl$, $R_4 = -H$
- VIII $R_1 = -NH SO_2 O NH CO CH_3$, $R_2 = -H$, $R_3 = cladinosyl$, $R_4 = -H$
- 1X $R_1 = -NH SO_2 O CH_3$, $R_2 = -H$, $R_3 = -H$, $R_4 = -H$

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Abstractor: Ž. Kućan

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