CYCLODEXTRIN NEWS

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3rd EUROPEAN CONFERENCE ON CYCLODEXTRINS

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Novel cyclodextrin derivatives presented at the 3rd European Conference on Cyclodextrins

This year's European Conference on Cyclodextrins organized by Prof. Erem Bilensoy, Hacettepe University, Ankara brought 50 oral presentations and 65 posters. Most of the presented works covered the main theme of the congress: "multifunctional excipient potential of cyclodextrins in pharmaceutical, cosmetic and biomedical industries".

The large number of the presented works dealing with novel cyclodextrin derivatives clearly shows that recently there has been a renewed interest in functionalization of cyclodextrins in order to extend the utilization of these derivatives as tools in catalysis, drug delivery, formulation and stabilization of active molecules and cosmetic ingredients.

This short paper gives a brief review of the presented novel cyclodextrin derivatives, summarizes their synthetic methodologies and the field of their applications. Extended abstracts will be published in the Journal of Inclusion Phenomena and Macrocyclic Chemistry after peer review. The references given here are the papers of the cited authors on similar topics presented at the conference.

NOVEL CYCLODEXTRIN POLYMERS AND NANOSPONGES

Phosphorus-containing cyclodextrin polymers

presented by Prof. Catherine Amiel

A very attractive new cyclodextrin (CD) based polymeric system presented by Prof. Amiel combines the advantages of cyclodextrin polymers (formation of inclusion complexes with apolar guests) and phosphorus-containing polymers (biocompatibility, calcium affinity). These polymeric systems are promising candidates to be used in biomedical applications that jointly require calcium delivery and transport of lipophilic bioactive molecules. A non-toxic cyclic sodium trimetaphosphate (STMP) was used as a cross-linking agent under basic conditions. The molecular weight of the obtained polymers was higher than 10^4 g.mol^{-1}, and it depended on the NaOH/βCD and NaOH/STMP weight ratios. The efficiency of these systems to form inclusion complexes with apolar probes (1-adamantyl acetic acid) and their affinity towards Ca^{2+} cations were demonstrated by isothermal titration calorimetry [1].

"Click" synthesis of novel well-defined cyclodextrin polymers

presented by Thorbjorn Terndrup Nielsen

Nielsen presented a reliable and versatile synthetic methodology, which leads to well-defined dextran-CD polymers. Their synthesis is based on a copper (I) catalyzed "click" reaction between alkyne modified dextran backbones and monoazido-CDs. Besides the well-studied βCD polymers Nielsen also presented the synthetic approach towards polymers based on α- and γCD. As a counterpart polymer, adamantyl grafted dextran backbones were synthesized with different ratios of pendant groups. By mixing the host and guest modified dextrans in water, stable nanoassemblies were formed. The size of these supramolecular structures can be varied by adjusting CD and adamantyl substitution degree of the dextrans, by the total concentration and by the composition of the mixtures. The loading properties of these assemblies towards hydrophobic drugs were evaluated by isothermaI titration calorimetry and the results showed that these systems have potential as drug vehicles [2,3].

New glutathione bio-responsive cyclodextrin nanosponges

presented by Prof. Francesco Trotta

The synthesis and characterization of novel glutathione responsive cyclodextrin nanosponges (GSH-NSs) were presented by Prof. Trotta. Series of nanosponges were prepared by easy one-step synthesis, reacting different types of cyclodextrins, cross-linking agents and variable amounts of 2-hydroxyethyl disulfide in order to incorporate disulfide bridges into the nanosponge. The quantity of the disulfide linkages was determined by the Ellman's reaction; the encapsulation efficiency was investigated using doxorubicine as model drug and the glutathione responsiveness was tested *in vitro* on cancer cells with different glutathione content.

NEW APPROACHES IN MONOSUBSTITUTION OF CYCLODEXTRINS

Synthesis and properties of regioisomers of monosubstituted cyclodextrin derivatives

presented by Prof. Jindrich Jindrich and co-workers

Group of Jindrich Jindrich is interested in the preparation of so-called "single-isomers" of cyclodextrin derivatives. They developed a synthetic methodology for preparation of different sets of regioisomers of monosubstituted cyclodextrins. in some cases, they achieved even a regiospecific monosubstitution [4], while in the cases where separation of the isomers was difficult, acetylation and separation of the peracetates was used.

Bláhová from this group presented a gram-scale synthesis of 2^{I}-*O*-, 3^{I}-*O*- and 6^{I}-*O*- allyl, formylmethyl, and carboxymethyl derivatives of γCD. All of the mentioned derivatives will serve as precursors for further preparation of regioselectively monosubstituted γCD [5].

Benkovics in collaboration with CycloLab Cyclodextrin Research & Development Laboratory, Ltd. prepared full sets of regioisomers of monocinnamyl α and βCD [6]. These monosubstituted derivatives have a potential to self-organizing to supramolecular structures, which was proved and studied with various methods including NMR, mass spectroscopies as well as light scattering. The results indicate that the position of the cinnamyl group on the CD rim, the solvent and the temperature have a great influence on the supramolecular complex formation. The obtained supramolecular structures will be used as pseudostationary phases in electrophoretic separation methods.

Popr in his poster communication presented a synthesis of complete sets of CD derivatives (α, β, γ) with permanent positive charge(s). in all cases, a multigram-scale reaction sequence was developed starting with selective monotosylation, followed by reaction with amine nucleophile and final methylation of the amine. Because of the permanent positive charge, well-defined charge localization, good solubility and relatively easy access to the cavities, these "single-isomer" cyclodextrins are ideal candidates to be used in capillary zone electrophoresis for chiral separations.

Jindrich in his presentation gave an overview of the developed regioselective preparations and pointed out the importance of the application of pure regioisomers instead of the randomly substituted ones in analytical and supramolecular chemistry [6, 7].

Sensing and delivery systems based on 2-*O* functionalized βCD

presented by Prof. Antonio Vargas-Berenguel

2-*O* monopropargylated βCD was used as a building block for the synthesis of bile salt sensors and drug delivery systems. From this monosubstituted compound face-to-face 2–2′ βCD dimers were prepared with different length and type of the linker between the CD units [8, 9].

Addition of a ferrocene moiety via "click" reaction to the monopropargylated βCD provided a cyclodextrin based redox sensor [10].

In addition the mono-2-*O*-propargyl βCD was used also for the synthesis of βCD coated gold nanoparticles.

The supramolecular behavior, the sensing properties and the drug delivery capability of these newly prepared CD derivatives were evaluated.

NEW APPROACHES IN MULTISUBSTITUTION OF CYCLODEXTRINS

Multisubstituted cyclodextrin polyvalent systems

presented by Chang-Chun Ling

Ling and co-workers's research is focused on a DIBAL-H mediated selective deprotection of cyclodextrins which in many cases results both high chemo- and regioselectivity to liberate free hydroxyl groups in a well-defined positions. This method was already described for selective de-*O*-benzylation and de-*O*-methylation on per-6-*O*-benzyl or per-6-*O*-methyl substituted CD derivatives.

Ling in his presentation showed that this method can be successfully extended to de-*O*-silylations on per-6-*O*-tert-butyldimethylsilyl or on the more sterically hindered per-6-*O*-thexyldimethylsilyl-substituted CD derivatives. The reductive desilylation also gives the required selectivity to afford 6^A and 6^A, 6^D -*O*-didesilylated products in good to excellent yields. Because of the easy access to per-6-*O*-silylated CD substrates and reproducibility, the DIBAL-H-mediated desilylation method can be very useful in CD chemistry, especially in the regioselective disubstititution of CD substrates [11].

Novel multisubstituted cyclodextrins as contrast agents in MRI

presented by Anais Biscotti

Functionalized cyclodextrins (CDs) are interesting scaffolds for contrast agents (CA) used in magnetic resonance imaging. For the further improvement of the sensitivity of this medical diagnostic tool is necessary to fully understand the role of the CDs in the efficiency of the CA.

Biscotti and co-workers presented an innovative synthetic strategy of the preparation of two new CD based CAs. in their complexes the metal ion (Gd(III), Eu(III) and La(III)) was heptacoordinated by 2,3-dimethylated βCD or native βCD.

The metal complexation occurs thanks to seven acetate ligands grafted on the βCD primary face. The per-6-*O* acetylation of the primary face of 2,3-dimethylated βCD was achieved according to Zavada's procedure but this methodology did not give selective per-6-*O* functionalization of the native βCD. For the protection of the secondary face, allylic functionalization was chosen which had never been used before as a temporary protective group for CD synthesis. The per-2,3-*O*-diallylated βCD was obtained with 45% yield from 6-*O*-persilylated βCD. The quantitative desilylation of the primary face led to a key intermediate, which was selectively deprotected and was soluble in dichloromethane, therefore suitable for Zavada's procedure. The allylic protecting group turned to be easily removable which means that this new methodology could be useful in order to reach new multisubstituted CD structures.

The aim of this work was not only to prepare these two structurally similar CAs but also to compare the obtained MRI results and to evaluate the influence of hydrogen bonding interactions on the second coordination sphere (secondary side of the βCD) of the CA [12].

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1,3-Dipolar Cycloaddition, Click Chemistry, Contrast Agent, Drug Delivery, Gadolinium Imaging Agent, Microwave Chem., Molecular Dynamics, Molecular Dynamics Simulations

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Preparation, Amino, Ureido, Thioureido, Per-6-Substituted Beta-Cyclodextrin

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Alpha-Cyclodextrin, Arbutin, Biocatalysis, Citric Acid, Controlled Study, Cyclodextrin Glucanotransferase, Cyclomaltodextrin Glucanotransferase Glycosylation, Maltodextrin, Thermoanaerobacter, Thermoanaerobacter Sp, Transfer Efficiency, Transglycosylation

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Cyclomaltodextrin Glucanotransferase, DNA Sequence, Escherichia Coli, Gene Encoding, Gene Expression Regulation, Recombinant Enzymes, RNA Transcription

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GLYCAM04 AMBER, Inclusion Complexes, Large-Ring Cyclodextrins, MD, PCA

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Smart Materials, Hydrogel, Morphology Transformation, Vesicle

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Animal Experiment, Animal Tissue, Area Under the Curve, Bioavailability, Complex Formation, Controlled Study, Diffusion Chamber, Dog, Gamma Cyclodextrin, Intestine Mucosa Permeability, Maximum Plasma Concentration, Time to Maximum Plasma Concentration

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Insulin, Langmuir Monolayers

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Complexation, Cyclosophoraose, Hydroxypropyl Cyclosophoraose, Alpha-Naphthoflavone

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Aggregates, Atomic Force Microscopy, Electrochem. Oxidation, Ethylene Glycol, Film Growth, Oligo(Ethylene Glycol), Organometallics, Polyacrylic Acids, Polypseudorotaxanes, X-Ray Diffraction Measurements

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Cyclodextrin, Macromolecular Architecture, Reversible-Deactivation Radical Polymerization, Supramolecular Chemistry

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Biocompatibility, Chain Length, Living Polymerization, Raft Polymerization, Thermoresponsive Polymer

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Aggregation Number, Chemical Shift, Inclusion Complex, Ionic Liquids, Packing Parameter

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Environmental Conditions, Inorganic-Organic Hybrid Polymers, Polyethylene Oxides, Polyhedral Oligomeric Silsesquioxanes, Spherical Aggregates

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Functional Polymers, Hydrogels, Hydrophilic Copolymers, Organometallics, Porous Network Structures, Raft, Stimuli-Responsive, Supramolecular Hydrogels

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Phosphorescence Enhancement, Photoswitchable Phosphorescent Colors

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Chemosensors, Fluorescence Quenching, Hemoglobin, Protein Sensing, Stern-Volmer Equation, Stilbazolium Dye

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3. CDs in Drug Formulation

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Brain-Targeting, Lactoferrin, Nano-Drug Delivery System, Transferrin, Beta-Cyclodextrin Derivatives

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Architectural Parameters, Carbohydrate-Protein Interactions, DNA, Functional Materials, Gene Delivery, Gene Transfer, Lectins, Nano-Devices, Nanoparticles, Non-Specific Interactions, Nucleic Acids, Structure-Activity Relationships

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Cyclodextrin, Gene Delivery, Non-Viral Vector, Polymer, Transfection

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Angiotensinogen, Cardiac Myocytes, Caveolae, Map Kinases

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Beta-Cyclodextrin, Binding Affinity, Cell Adhesion, Cell Membrane, Cholesterol, Hermes Antigen, Hyaluronic Acid, Receptor Upregulation, Sphingolipid, T Lymphocyte

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Cyclodextrins, Gene Delivery, Intestinal Barriers, Non-Viral Vectors, Stability

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Cerebrospinal Fluid, Cholesterol, Dynamic Light Scattering, Niemann-Pick C Disease, Randomly Methylated Beta-Cyclodextrin, HPBCD, SBEBCD

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Actin Cytoskeleton, Focal Adhesion, Lipid Raft, Melanoma Cell Migration

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Imazalil, Thiabendazole, O-Phenylphenol, Coumarines, Carotenoid

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Rooting Process, 1-Naphthaleneacetic Acid

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Food Industry, Large Surface Area, Electrospinning, Electrospun Nanofibers, Eugenol, High Thermal Stability, Polyvinyl Alcohols, Thermodynamic Stability

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Green Synthesis, Microwave Irradiation, Nitrogen Doped Carbon Nanotubes, Water Purification, Beta-Cyclodextrin Polymers

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Inclusion Complex, Lycopene, Micronization, Supercritical Antisolvent, Beta-Cyclodextrin

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In Vitro, Fatty Acid Concentration, Bifidobacteria Number, Sucrose Caramel, Raftilose

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Adsorption, Aluminum, Chlorine Compounds, Clay Minerals, Competitive Adsorption, Controlled Release Formulations, Cyclodextrins, Enhanced Adsorptions, Herbicide, Herbicide Adsorption, Herbicides, Imazaquin, Inclusion Complex, Inner Sphere Complexes, Leaching, Montmorillonite, Pillared Clay, Porosity, Sandy Soil, Weed Control

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Inclusion Complex, Soybean Lecithin, Beta-Cyclodextrin

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3-Arylpropionic Esters, Acrolein, Cinnamaldehyde, Heck Arylation, Palladium Catalysts, Water

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Carbon, Carbon Nanomaterial, Chronic Exposure, Controlled Study, Crustacean, Daphnia Magna, Ecotoxicology, Environmental Impact, Fullerene Derivative, Gamma-Cyclodextrin, Pollution Exposure, Single Walled Nanotube

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Armoracia Rusticana, Cell Inclusion, Dithiothreitol, Enzyme Reconstitution, Escherichia Coli, Fluorescence Analysis, Glutathione Disulfide, Horse-Radish Peroxidase, Porphyrins, Protein Refolding, Recombinant Enzyme

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Bioavailability, Biodegradation, Cyclodextrin, PAHS, Petroleum Hydrocarbons, Tenax

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Adsorption Capacities, Clenbuterol, Environment Science, Molecular Sieves, Nitrogen Adsorption-Desorption, Photoelectrons, Powder X-Ray Diffraction (pXRD), X Ray Photoelectron Spectroscopy

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Anthracene, Beta Cyclodextrin, Citric Acid, Cross Linking Agents, Cyclodextrin Polymer, Electrospinning, Gamma Cyclodextrin, Glass Transition Temperature, Waste Water Management

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Catechol, Catecholamine, Inclusion Complex, N-Methylaniline, Oxidation

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Extraction Agents, Polycyclic Aromatic Hydrocarbons (PAH), Soil

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Beta-Cyclodextrin, Chemical Interaction, Chlorine Compounds, Molecular Dynamics, Molecule-Ion Interaction, Supramolecular Organogel

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Cyclodextrin, Ferrocene, Gas Separation, Polyimide, Thermal Annealing

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Carbon Deposition, Ni Particles Size, Ni/Sba-15, Beta-Cyclodextrin

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Antiplasticization, Chitosan, Film Preparation, Glycerol Concentration, Microfluidization, Morphological Properties, Tensile Strength

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Air-Water Interfaces, Cosmetic Formulations, Oil-In-Water Emulsions, Optical Microscopy, Stabilisation Mechanisms, Surfactant-Free Emulsions, Sustainable Materials, Tetradecane-Water Interfaces

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Amino-Cyclodextrins, Desalination, Thin-Film Composite Membrane, Water Permeability, Interfacial Polymerisation

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Advanced Oxidation Processes, Bioassays, HPBCD, PAHs, Recycling, Tween 80

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Crystallization, Nucleating Agent, Plasticizer, Polylactide, Star-Plla, Beta-Cyclodextrin

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Bioaccessibility, Bioavailability, Biodegradation, Model Predictions, Polycyclic Aromatic Hydrocarbon

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Adsorption, Arsenic, Groundwater., Hydrous Ferric Oxide, Beta-Cyclodextrin

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Bisphenol, Flash Photolysis, Kinetics, Phenoxyl Radical, Photoionization, Beta-Cyclodextrin

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Photoinjecting Moiety, Light Harvesting, Charge Transfer Energy

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Alpha-Cyclodextrin, Arachidonate 5-Lipoxygenase, Cell Separation, Head Column Field Amplified Stacking, Hydroxyicosatetraenoic Acid, Icosatetraenoic Acid, Prostaglandin B1, Sensitivity Analysis

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Carboxymethyl-Gamma-Cyclodextrin, Chiral Separation, DMSO, NACE

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Acebutolol, Alimemazine, Alkalinity, Atenolol, Beta Cyclodextrin, Carbinoxamine, Concentration Response, Electrokinetic Chrom., Mathematical Model, Octanol-Water Partition Coefficient, Online Analysis, Quantitative Structure Activity Relation

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Cyclic Nitrones, Epr Spectroscopy, ESR Spectroscopy, Hydroxyl Radical, Spin Adduct Stability, Spin Trapping, Superoxide

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Zwitterionic Form, Capillary Electrophoresis, Enantioselective Synthesis, Lipophilicity, Migration Order, Protonation Constant

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Click Chemistry, In Situ Copolymerization, Mono-(1H-1,2,3-Triazol-4-ylmethyl)-2-Methylacryl-Beta-CD Monomer, Ethylene Dimethacrylate, Enantioselectivity

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Hydrophilic Interaction Liquid Chromatography, Sulfoalkylated Cyclodextrins

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Adenosine Triphosphate, Fluorescence Lifetime, Pyrene Dimer, Splitting Aptamer, Gamma-Cyclodextrin

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Chiral Stationary Phases, Column Robustness, Cosac, Enantiomer Separation, Gas Chromatography, Rosetta

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CT, MRI, NaDyF4, Nanorod, Phosphorescence Imaging, T1 and T2-Weighted

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Capillary Electrophoresis, Cationic Cyclodextrin, Chiral Separation, Enantioselectivity, Hydrogen Bonding

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