CYCLODEXTRIN NEWS

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Ronald Breslow

(1931–2017)

Professor Ronald Breslow (86) passed away in October. His passing is a huge loss for the world of chemistry including cyclodextrin chemistry.

Breslow completed his B.A. (1952), M.A. (1953) and Ph.D. (1955) at Harvard University. He started to work at Columbia University in 1956, where he became a University Professor in 1992 and worked there till his retirement. He served as President of the American Chemical Society in 1996. On his honor, the Ronald Breslow award was established in 2001 (on the occasion of his 70th birthday) to recognize outstanding contributions to the field of biomimetic chemistry. His interest was shared between biology and chemistry (being a member of both chemistry and biology department). He wrote about the history of biomimetic chemistry (which is the history of his life) in an easily understandable style [1].

He made fundamental research on various fields of synthetic chemistry including cyclodextrin modifications. He used the cyclodextrin derivatives in supramolecular chemistry, molecular catalysis and biomimetic chemistry. He has published 75 cyclodextrin-related papers including 2 book chapters and a book. His cyclodextrin-related publications were started with a paper on selective aromatic substitution within a cyclodextrin complex in 1969 [2] and finished with a review chapter in a book entitled "Molecular Encapsulation: Organic Reactions in Constrained Systems" in 2010 [3].

Prof. Crini writes on him in his famous paper on the history of cyclodextrins [4]:

"Breslow, at the University of Colombia in 1970, synthesized an artificial enzyme (a CD modified with a coenzyme) that mimicked the biochemical reactions catalyzed by naturally occurring transaminases [5, 6]. A year later, the same lab managed to realize the dreams of supramolecular chemists: they used a modified CD to catalyze a chemical reaction (a Diels–Alder cycloaddition), which no natural enzyme and no common catalyst could [7]."

Ronald Breslow found cyclodextrins extremely attractive components of artificial enzymes. Enzymes have a hydrophilic exterior and a hydrophobic interior. This structure makes possible hydrophobic substrates to bind the hydrophobic binding site allowing the reaction to occur away from water. Cyclodextrins possessing hydrophilic exterior and hydrophobic cavity behave as enzyme analogs. Their large number of hydroxyl groups can either react with the substrate included in the cavity or can be used to attach other catalytic groups [8].

This concept was used to imitate various enzymes as summarized in several reviews [3, 8–24]. A few examples of the huge library of his cyclodextrin-based enzyme mimics are shown below.

Ribonuclease model was developed using bisimidazoyl-CD derivatives (6A,6B and 6A,6C and 6A,6D isomers) mimicking the two histidines at the active site of the enzyme. Catechol monophosphate, as a model compound of the cyclic intermediate in RNA hydrolysis was hydrolyzed by the beta- and gamma-CD derivatives with an enhanced rate with the 6A,6B isomer of BCD having the two imidazoyl groups in adjacent glucose units (Fig. 1).

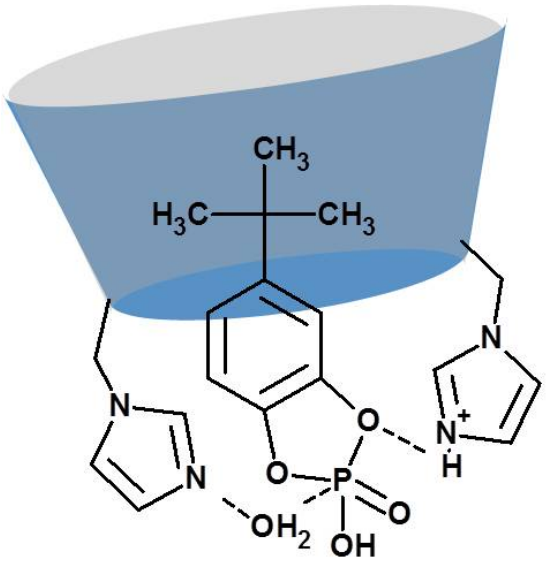

Fig. 1 Intermediate of hydrolysis of cyclic phosphate ester of 4-tert-butylcatechol to noncyclic phosphates by BCD carrying catalytic groups, in this case two imidazole moieties

Cyclodextrin dimers and trimers were found to bind to certain peptides and proteins with much higher affinity than beta-cyclodextrin. A series of dimers and trimers were prepared with various spacer groups (various length and hydrophobicity) to find the best binding agents for polypeptides and proteins. See two examples in Fig. 2 and 3. A dimer with a short disulfide linker between the primary carbons of the two cyclodextrins binds preferably the dipeptide Trp-Trp. Trimers were found to bind larger peptides more effectively than the dimers.

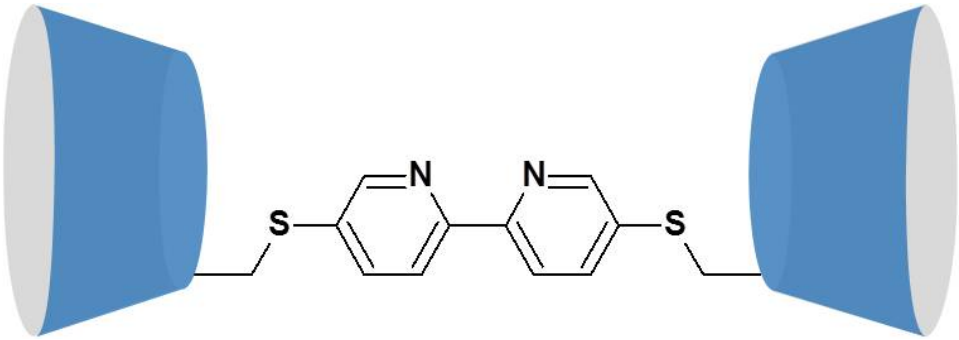

Fig. 2 Cyclodextrin dimers for binding ditopic substrates (clamshell structure). The linker containing bipyridyl group can form complexes with metal ions and these metal complexes are effective catalysts in hydrolyzing esters with two hydrophobic moieties

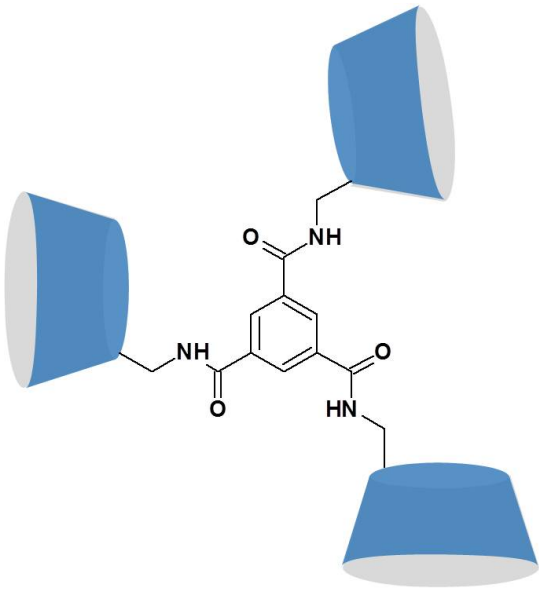

Fig. 3 Cyclodextrin trimer for tritopic binding substrates, such as trimeric aminoacid (phenylalanine and/or triptophane) amides to mimic binding of peptides and proteins

Manganese porphyrin carrying four cyclodextrins attached to fluorinated phenyl groups (Fig. 4), Cytochrome P-450 mimic, was used as a catalyst in the hydroxylation of androstane-3,17-diol. The steroid was converted to diester carrying tert-butyl groups for binding into cyclodextrins prior to hydroxylation regioselectively at the C-6 position of the steroid using iodosobenzene as oxidizing agent. Without the tert-butylphenyl binding groups no oxidation of the steroid occurred under the same conditions.

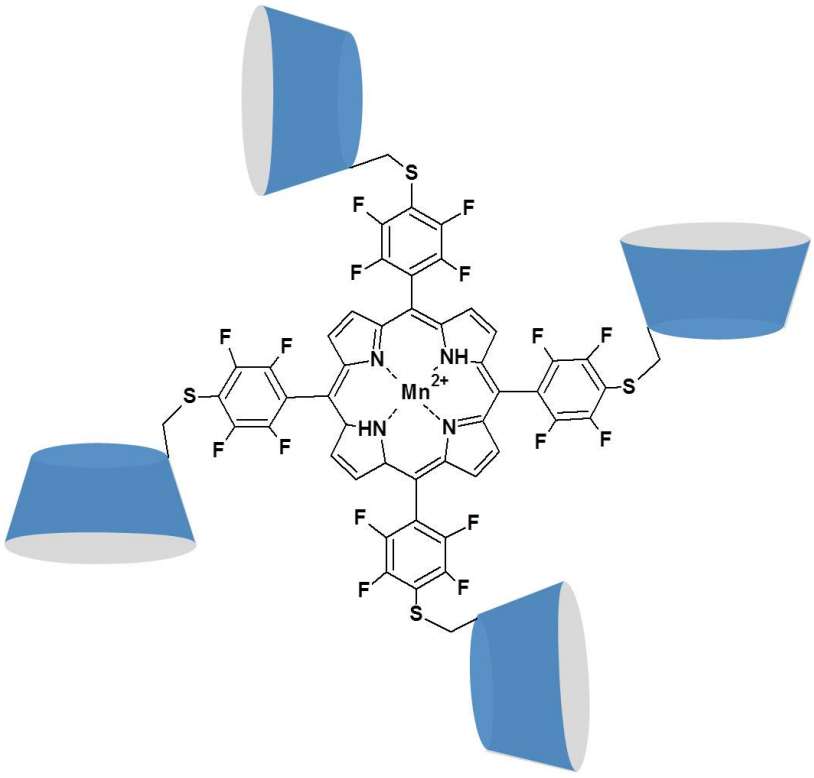

Fig. 4 Manganese porphyrin carrying four CDs attached to fluorinated phenyl groups

Prof. Breslow inspired by biology recognized the importance of geometric control imposed by defined binding to the catalyst and the potential of cyclodextrins in such processes. The wealth of fundamental knowledge he collected in this field largely contributes to the development of artificial enzymes as well as cyclodextrin chemistry.

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Cyclodextrin News Retrospective

We wrote 10, 20 and 30 years ago

10 years ago, the editorial of Cyclodextrin News dealt with the application of differently methylated BCD derivatives (and their cholesterol complexes) in controlled manipulation of membrane cholesterol content. The reported major findings were the followings:

- Decreasing the degree of methylation resulted in decreased solubilization of cholesterol,

- Comparing different purity grades of DIMEB it was observed that the isomer purity had hardly any influence on cholesterol solubilization and the effects on cell viability were also very similar,

- Ionic, "Second generation" CD derivatives obtained by further derivatization of methylated CDs showed much lower cholesterol solubilizing effect than their parent methylated CDs, nevertheless higher than the related ionic CD derivatives.

"TUNING CYCLODEXTRINS TO MODIFY THEIR CHOLESTEROL BINDING" Cyclodextrin News, 2007 Vol. 21, No. 12.

20 years ago, the editorial of the December issue of Cyclodextrin News was a cordial invitation to the 9th International Symposium on Cyclodextrins, University of Santiago de Compostela.

30 years ago, the editiorial was a short report on the Symposium of "Possibllities for Application of CDs in the Pharmaceutical industry" organized in West-Berlin on 20-22 September 1987 by the Pharmaceutical Institute of the Free University in Berlin. Some topics represented there are still actively studied research areas, it is surprising how actual questions were discussed three decades earlier, such as:

K.H. Frömming: ''General aspects of CD-applications in the pharmaceutical industry"

J. Szejtli: "Groups of drugs to be complexed by CDs: possibilities and limits"

R. Kupferschmied. R. Schmied: "Examples for Application of Silicabonded CDs in Drug Analysis"

T. Tóth: "Quality control of CDs"

T. Nagai: "Application forms and application ways of drug formulations with CD inclusion compounds"

E. Smolkova-Keulemansova: "Use of cyclodextrins in chromatography"

Bibliography & Keywords of Selected Publications of the Month

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Molecular structure of cyclomaltodextrinase derived from amylolytic lactic acid bacterium Enterococcus faecium K-1 and properties of recombinant enzymes expressed in Escherichia coli and Lactobacillus plantarum

Glycoside hydrolase αCD, βCD, Pullulan, Starch

International Journal of Biological Macromolecules, 2017,- ;
DOI:https://doi.org/10.1016/j.ijbiomac.2017.09.060

Malanga, M.; Fejős, I.; Varga, E.; Benkovics, G.; Darcsi, A.; Szemán, J.; Béni, S.

Synthesis, analytical characterization and capillary electrophoretic use of the single-isomer heptakis-(6-O-sulfobutyl)-beta-cyclodextrin

Five-steps synthesis, First example of single-isomer sulfobutylated cyclodextrin, Chiral separation, Enantiomer migration order

Journal of Chromatography A, 2017, 1514, 127 - 133;
DOI:https://doi.org/10.1016/j.chroma.2017.07.069

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γ-Cyclodextrin

Aggregation behavior, ADME

International Journal of Pharmaceutics, 2017, 516, 278 - 292;
DOI:https://doi.org/10.1016/j.ijpharm.2016.10.062

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Solubilizing steroidal drugs by β-cyclodextrin derivatives

β-cyclodextrin thioethers, heptakis-6-methylsulfanyl-6-deoxy-2-(2-(2-(2-methoxyethoxy)ethoxy)ethyl)]-β-CD, heptakis-6-thioglyceryl-6-deoxy-β-CD, Gender selectivity, Testosterone, Estradiol

International Journal of Pharmaceutics, 2017, 531, 559 - 567;
DOI:https://doi.org/10.1016/j.ijpharm.2017.07.046

Lucio, D.; Irache, J. M.; Font, M.; Martinez-Ohariz M. C.

Nanoaggregation of inclusion complexes of glibenclamide with cyclodextrins

Nanoaggregates of CD and complexes, Hydrogen bond network

International Journal of Pharmaceutics, 2017, 519, 263 - 271;
DOI:https://doi.org/10.1016/j.ijpharm.2017.01.028

Banafsheh Rastegari and Hamid Reza Karbalaei-Heidari and Sedigheh Zeinali and Heather Sheardown

The enzyme-sensitive release of prodigiosin grafted β-cyclodextrin and chitosan magnetic nanoparticles as an anticancer drug delivery system: Synthesis, characterization and cytotoxicity studies

Fe_3O_4 nanoparticles, Carboxymethyl chitosan, βCD as carriers, Prodigiosin as the model anti-tumor drug

Colloids and Surfaces B: Biointerfaces, 2017, 158, 589 - 601;

DOI:https://doi.org/10.1016/j.colsurfb.2017.07.044

Taotao Xu and Junhua Li and Jun Cao and Wenxia Gao and Li Li and Bin He

The effect of α-cyclodextrin on poly(pseudo)rotaxane nanoparticles self-assembled by protoporphyrin modified poly(ethylene glycol) for anticancer drug delivery

Poly(ethylene glycol) (PEG) modified with protoporphyrin (PpIX), Spherical shape, Doxorubicin, Cell internalization efficiency

Carbohydrate Polymers, 2017, 174, 789 - 797;
DOI:https://doi.org/10.1016/j.carbpol.2017.07.012

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Surface-deacetylated chitin nanofibers reinforced with a sulfobutyl ether β-cyclodextrin gel loaded with prednisolone as potential therapy for inflammatory bowel disease

Oral administration to rats, Controlled release, Colitis, Elastic gel, Release properties

Carbohydrate Polymers, 2017, 174, 1087 - 1094;
DOI:https://doi.org/10.1016/j.carbpol.2017.07.028

Gao, Y.; Li, G.; Zhou, Z.; Guo, L.; Liu, X.

Supramolecular assembly of poly(β-cyclodextrin) block copolymer and benzimidazole-poly(ε-caprolactone) based on host-guest recognition for drug delivery

Atom transfer radical polymerization (ATRP), Core-shell structure, Benzimidazole modified poly(ε-caprolactone), Complex micelles, Doxorubicin

Colloids and Surfaces B: Biointerfaces, 2017, - ;
DOI:https://doi.org/10.1016/j.colsurfb.2017.09.047

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Methyl-β-cyclodextrin, HPβCD, Depth of photosensitizer penetration, Nanoshuttle mechanism, Diagnostic, Photodynamic therapy

International Journal of Pharmaceutics, 2017, 529, 568-575;
DOI:https://doi.org/10.1016/j.ijpharm.2017.07.037

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Lecithin, Ethanol, βCD, Kneaded complex, Carrageenan induced paw edema, Transdermal delivery

Journal of Drug Delivery Science and Technology, 2017, 40, 95 - 104;
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Dexamethasone, Mucoadhesive film
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Mycobacterial infections, DIMEB, pH-controlled release
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Novel dissolving microneedles for enhanced transdermal delivery of levonorgestrel: *In vitro* and *in vivo* characterization

Contraception drug, Chitosan, Beta-sodium glycerophosphate, HPβCD, Enhanced bioavailability
International Journal of Pharmaceutics, 2017, 534, 378 - 386;
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Acid-labile acetylated βCD, ROS-sensitive Ox-βCD, Rapamycin, Intraperitoneal delivery in apolipoprotein E-deficient mice, Responsive to mildly acidic or abnormally high ROS microenvironments
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HPβCD, Side chain length of the parabens, Methyl- and butylparaben, Heptylparaben
International Journal of Pharmaceutics, 2017, 529, 442 - 450;
DOI:https://doi.org/10.1016/j.ijpharm.2017.07.026

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Surfactant free, Three-month toxicological testing in rabbits
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Docetaxel, Target folate receptors of tumor cells, Suppressed tumor growth
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Target prostate cancer cells, Protection from serum nucleases
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HPβCD, Excisional wounds in the skin of rats, Carbopol 940® base gel, Migration of keratinocytes, Revascularization
Injury, 2017, 48, 2417 - 2425;
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Preparation and characterization of novel chitosan and β-cyclodextrin polymer sponges for wound dressing applications

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Pore-forming toxins, Lethal toxin of Bacillus anthracis.
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Intrathecal 2-hydroxypropyl-β-cyclodextrin decreases neurological disease progression in Niemann-Pick disease, type C1: a non-randomised, open-label, phase 1-2 trial

Patients, non-randomly, Sequentially assigned in cohorts, Dose finding study, Biomarker, Neuronal cholesterol homoeostasis, Hearing loss, Slowed disease progression
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Role of 6-O-A-maltosyl-β-cyclodextrin in lysosomal cholesterol deprivation in Npc1-deficient Chinese hamster ovary cells

Metabolization by lysosomal A-glucosidase to 6-O-A-D-glucosyl-β-cyclodextrin, Release of unesterified cholesterol
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Journal of Controlled Release, 2018, 269, 148 - 158;
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Cellular uptake, Ternary, Hepatocyte-specific siRNA delivery system
International Journal of Biological Macromolecules, 2017, - ;
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β-Cyclodextrin induces the differentiation of resident cardiac stem cells to cardiomyocytes through autophagy

Enhanced myogenic transition, Improving intracellular cholesterol levels, Affecting cholesterol efflux
Biochimica et Biophysica Acta (BBA) - Molecular Cell Research, 2017, 1864, 1425 - 1434;
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Rutenberg, R.; Bernstein, S.; Fallik, E.; Paster, N.; Poverenov, E.

The improvement of propionic acid safety and use during the preservation of stored grains

Antimicrobial formulations in agricultural products, Improved storability and quality, Propionic acid, Carboxymethyl cellulose, βCD, Controlled release
Crop Protection, 2017, - ;
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Sponge like microparticles for drug delivery and cosmeto-textile use: Formulation and human skin penetration

HPβCD complexes, Indomethacin, Tocopherol

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