CYCLODEXTRIN NEWS

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Boldog Karácsonyi Ünnepeket és Sikerekben Gazdag Új Esztendőt!

Merry Christmas and a Happy New Year!

Frohe Weihnachten und ein Glückliches Neues Jahr!

Joyeux Noël et Bonne Année!

CycloLab Ltd.

"All's Well That Ends Well"*: The year 2016

This year has been indeed a busy one, full with important events (most relevant was obviously the 18th International Cyclodextrin Symposium in Gainesville, Florida between May 18 – 21). This year has also brought to our scientific community in late autumn a really good news: *The Nobel Committee for Chemistry at the Royal Swedish Academy of Sciences announced the decision that the Nobel prize in Chemistry in 2016, will be shared with three outstanding scientists, the Dutch Bernard Feringa of the University of Groningen, the French Jean-Pierre Sauvage of the University of Strasbourg and the Scottish Sir Fraser Stoddart at Weinberg College of Arts and Sciences of Northwestern University.*

The laurates receive the highest recognition in science for their groundbreaking work on nanoscale structures, molecular machines that may soon find applications in drug delivery, in smart materials and even in creating artificial life. Though all the above awardees are highly reputed chemists, Sir Fraser Stoddart, the renowned supramolecular chemist has been playing significant role in in cyclodextrin science and technology and is therefore a closer acquaintance for many of the readers of Cyclodextrin News. The Editorial article of December issue of CD News intends to celebrate the decision of the Royal Swedish Academy and attempts to take a closer view at Professor Stoddart's most significant achievements in cyclodextrin chemistry and related fields. By Thomson Scientific, Sir Fraser Stoddart is ranked as the number 3. most-cited scientist in chemistry for the 10-year period (between January 1996 and August 2006).

*(Shakespeare)

He published more than 770 communications, papers and reviews, and has delivered over 700 invited lectures around the world. Stoddart is one of the few chemists who created a new field of chemistry over the past 30 years by introducing besides the well-established chemical bonds an additional bond, where no electrons are shared or distributed, where only physical interactions occur: this is what he calls *MECHANICAL BOND*. Stoddart pioneered the use of molecular recognition and self-assembly to create mechanically bound or interlocked systems attempting to bridge chemistry and life sciences with them.

Stoddart as a carbohydrate chemist: a solid background to work with cyclodextrins

Not many of us would be aware that Fraser Stoddart has a great deal of knowledge in carbohydrate chemistry, too and can be considered as a glycoscientist. Below is shown the cover of his great monograph written on the stereochemistry of carbohydrates [1]. (One of the most complicated stereo-chemical systems and arrangements of organic compounds.)

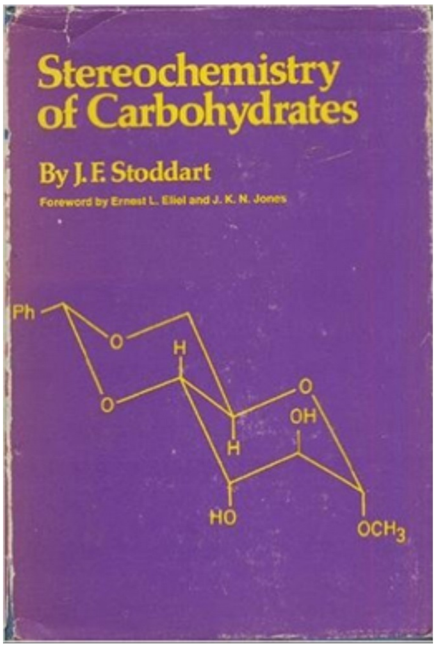

Figure 1. Front cover of Stoddart's book on Stereochemistry of Carbohydrates [1].

Stoddart and the cyclodextrins

Early cyclodextrin-related publications by Stoddart dealt with CD-enabled synthetic enzymes. His review paper on chemical /artificial enzymes discussed the alpha-CD and crown ether-based enzyme models and supramolecular catalysis [2].

In early 1980s he published a review on the host-guest chemistry using over 145 references on the historical background and nomenclature of major host compounds such as crown-ethers, cryptands, cavitands, cyclodextrins and their binary clathrates, summarizing the actual knowledge accumulated in that field by 1983 [3].

Soon a great deal of works has been dedicated to the cyclodextrin complexes of different organoplatinum compounds aiming at a more efficient anticancer drug delivery system. As professor of University of Sheffield, Stoddart and his co-worker, Dr. Alston studied the mode of inclusion of cyclobutane-1,1-dicarboxylatodiamineplatinum-(II) guest into the alpha-cyclodextrin host in aqueous solution. This work has resulted not only in a number of great papers but also in a European patent assigned to Johnson Matthey company [4].

During the second half of 1980-ies, Stoddart's group worked on the **Second Sphere Coordination** adducts of transition metal complexes with different parent- and methylated alpha- and beta-CDs. The first seminal Angewandte paper appeared in 1988 [5].

Working on the utility of supramolecular systems and molecular recognition Stoddart developed sensitive sensors suitable for organic volatiles. A benzene vapor detector has been developed, by coating the piezoelectric quartz crystal with chemically modified CDs. The 2,6-per-O-(tert-butyldimethylsilyl)-alpha-CD was found to be the best for the 0.08-400 mg dm^{-3} range benzene vapor in air. Besides this derivative, 2,6-per-O-alkyl-alpha-CDs were found also suitable for sensing organic gases. The detectors are selective towards benzene vapor over methane, propane, butane, pentane, ethane, ammonia, nitrobenzene and toluene, this last one gives rise to the most serious interference [6].

Stoddart and international cyclodextrin symposia

The CD community had the pleasure listening to Prof. Stoddart's first seminal lecture, in Lancaster, at the 3rd International Symposium on Cyclodextrins and Inclusion Phenomena organized by Prof. Eric Davis in July 1986. His talk was about synthesis and spectroscopic characterization of two chemically modified cyclodextrins (2,6-per-O-allyl-alpha-CD and the 2,6-per-O-(t-Bu-dimethylsilyl)-alpha-CD).

The next CD symposium held in Munich, in 1988 has witnessed the talk of Stoddart's group on chemically modified cyclodextrins as second sphere ligands for transition metal complexes. He introduced to the audience not only the binary traditional host-guest complexes, but also his unique ditopic molecular receptors called "molecular oysters" for the first time. The original drawing of this structure by Stoddart is shown in Figure 2 [7].

He presented brilliant lectures at the 5th (Paris), 8th (Budapest), 15th (Vienna) and 16th (Tianjin) Symposia as well on various syntheses including those of metal organic frameworks using CDs [8–11].

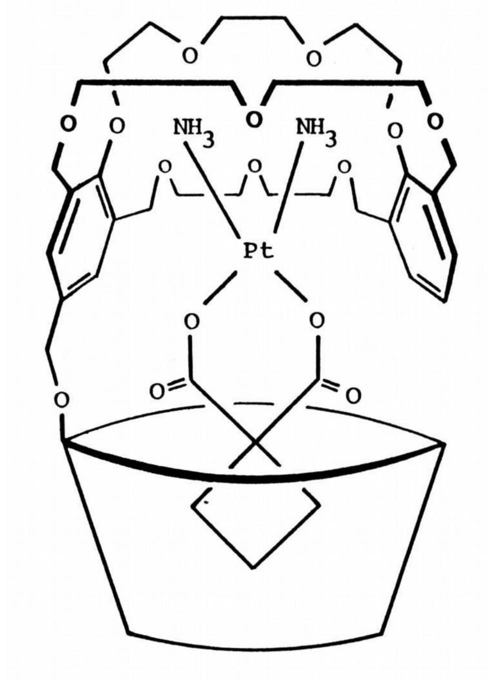

Figure 2. Structure of ditopic molecular receptors or "molecular oyster" based on macrobicyclic polyether combination with carboplatinum guest entrapped.

A closer step to cyclodextrin technology

In 1989, a special issue of Carbohydrate Research was dedicated to the cyclodextrins. In this special issue of the journal, Stoddart contributed by writing an article and the introductory Foreword [12].

His foreword was titled: "*A century of cyclodextrins".* Sir Fraser reviewed this field by selecting and listing 56 most relevant references published during the nearly 100 years. Moreover, in this overview Stoddart commented on the status of global CD research including Eastern European cyclodextrin research and technology by concluding: *"The commercial interest in CDs is growing at a phenomenal rate, particularly in Japan and Eastern Europe, where Szejtli's group impact has been enormous."* (We all felt honored that Hungarian CD research was admired by him that much.)

It is noteworthy that at that time Szejtli and Stoddart had multiple correspondence on the concept of creating "hybrid hosts" namely, crown-ether-cyclodextrin covalent combinations for multiple mode of molecular recognition. Below please see a copy of correspondence of Szejtli to Stoddart with Szejtli's hand drawing of such a possible "hybrid" host compound complexing an ionic guest.

Dear Professor Stoddart,

Thank you very much for sending me your manuscript on the Transition Metal Complexes Cyclodextrins (Submitted to the Rec. Trav. Chim. Pays-Bas). I enjoyed much also your lecture at the Munich Symposium, but because of the very crowded program I could not "digest" all details. Now, spending my holydays at the Balaton-lake finally had enough time to read this excellent work with due attention. I should like to mention another specific type of CD-crown ether metal ion combinations. Some years ago we prepared a crown ether-appended-CD, which very effectively complexed the Na-p-nitrophenolate:

I am waiting with great interest the paper on the modified CD-based piezoelectric chemical sensors for benzene vapour. My laboratory›s activity is focused on the industrial aspects of cyclodextrins, not only pharmaceutical applications, but virtually any potential uses of CDs. Please find enclosed our new brochure on CHINOIN›s CD-products.

Figure 3. Szejtli's letter to Sir Fraser Stodart with the suggested structure of sodium p-nitro-phenolate complex with a crown-ether-CD combination, in 1988.
(source: CycloLab's Archive)

Stoddart's dynamic rotaxanes and catenanes for molecular pirouettes and shuttles

One of the most significant fundamental contributions of Stoddart, which was recognized by the 2016 Nobel prize is his pioneering work on special dynamic host-guest complexes, called rotaxanes and catenanes. Since 1991 he has prepared numerous rotaxane and molecularly interlocked systems and studied their molecular dynamics in detail. Soon a review paper was published on the CD-based rotaxanes where Stoddart referred to earlier synthesized rotaxane prototypes. This early rotaxane was made by Ogino, in early 1980-ies and comprised from a terminal-diamino-alkane alpha-CD inclusion complex fixed by using cobalt salt at both ends of amino functions [13].

In his own research Stoddart threaded different molecular rings (among others cyclodextrins, too) as host compounds on a thin, linear molecular axles (as guest compounds) and demonstrated that the host ring was able to move back and forth along the axle. On the ground of his studies on rotaxanes, Stoddart further combined supramolecular systems to create molecular "elevators" or lifts, later even molecular muscles and nano-electronic devices.

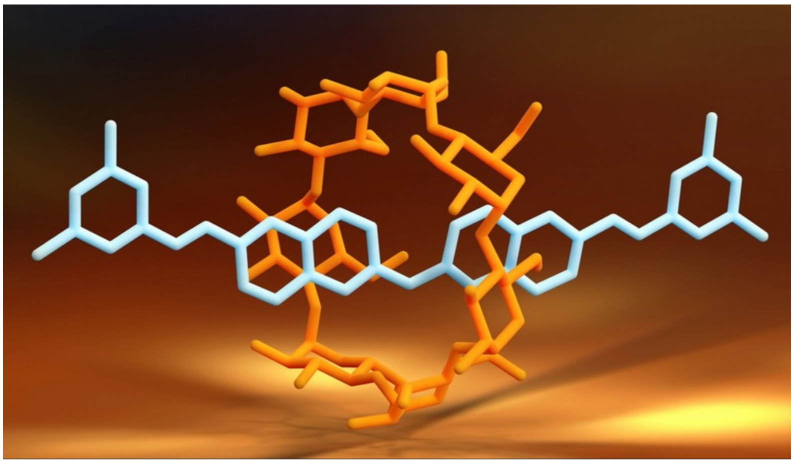

Figure 4. A typical rotaxane structure by Stoddart' group.
(source: Cosmos, News Chemistry 05 October 2016)

Stoddart's interest in molecularly interlocked structures has been maintained in the coming decades and his team published a great body of papers on catenanes, rotaxanes with different molecular constituents. Some of these papers dealt with cyclodextrin-based interlocked systems. In 1998, Chemical Reviews Vol. 98 was dedicated to Cyclodextrin Chemistry. In this issue, Stoddart with his co-workers Drs. Negopodiev and Gattuso contributed with two papers. One reviewed the interlocked molecular systems, the CD-based catenanes and rotaxanes [14], the other dealt with synthetic cyclic oligosaccharides [15].

Fraser Stoddart as an entrepreneur: from metal organic frameworks to novel gold mining technology

Sir Fraser has been convincingly emphasizing the role and significance of serendipity in scientific-technical discoveries. Stoddart's Mechanochemistry Group Lab at Northwestern University spent years with preparing and investigating multicomponent non-covalent superstructures, so-called metal-organic-frameworks (MOF). Their pioneering work has resulted in numerous seminal papers published between 2000-2010. An example is Angewandte Chemie paper on gamma-CD based edible MOF systems. (See Figure 5 below)

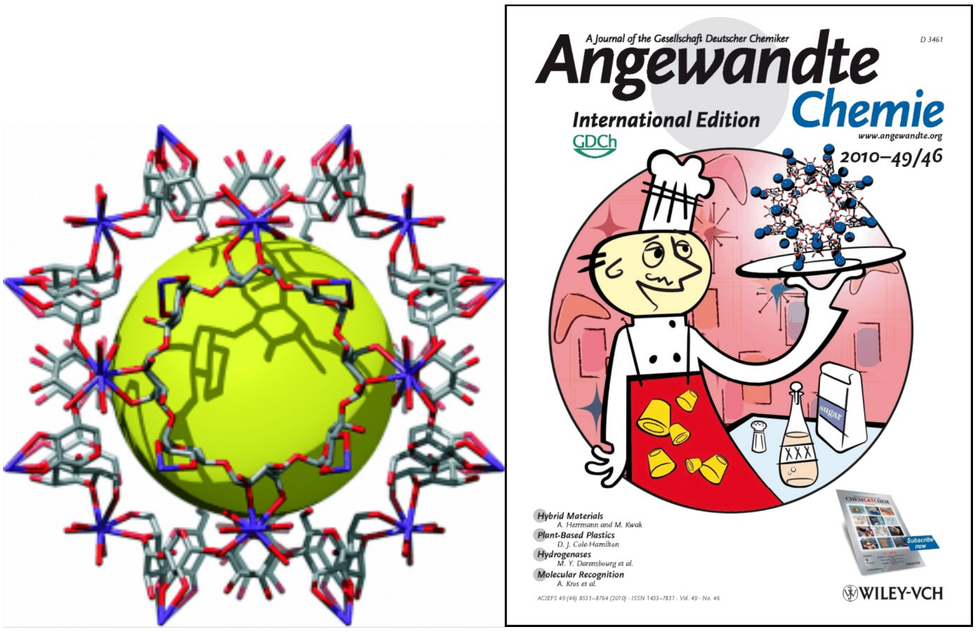

Figure 5. Structure of gammaCD-based nanoporous MOF (left) and the cover of Angew. Chem. with the chef serving superstructure "edible MOF".

Stoddart simple recipe to make gamma-CD MOF is as follows: "Take a spoonful of sugar (γ-cyclodextrin to be precise), a pinch of salt (most alkali metal salts will suffice), and a swig of alcohol (Everclear fits the bill), and you have a robust, renewable, nanoporous with a Langmuir surface area 1320 $m^2\,g^{-1}$ metal–organic framework for breakfast" [16].

One of Stoddart's PhD student, Liu prepared and studied a number of different cavity size CDs for complexation in aqueous solutions of potassium tetrabromoaurate ($KAuBr_4$) or potassium tetrachloroaurate ($KAuCl_4$). He surprisingly found that among parent CDs alpha-CD forms best a crystalline complex and so isolates gold best. They have discovered an inexpensive and environmentally benign method that uses simple cornstarch—instead of cyanide—to isolate gold from raw materials in a selective manner. Stoddart's team serendipitously discovered the gold isolation process using simple test tube chemistry. Of course, then a series of thorough follow-up investigations provided convincing evidence that the method is indeed useful.

The standard industrial methods for gold recovery use highly poisonous alkali cyanides, which often lead to contamination of the environment. These results were soon published in *Nature Communications* [17]. Suggested superstructure of alpha-CD based self-assembled gold salt, where alpha CD cyclinders are oriented in a head-to-head, tail-to-tail fashion forming a nano-channel filled with an alternating $K(OH_2)_6$ and $AuBr_4$-polyionic chain, is shown below.

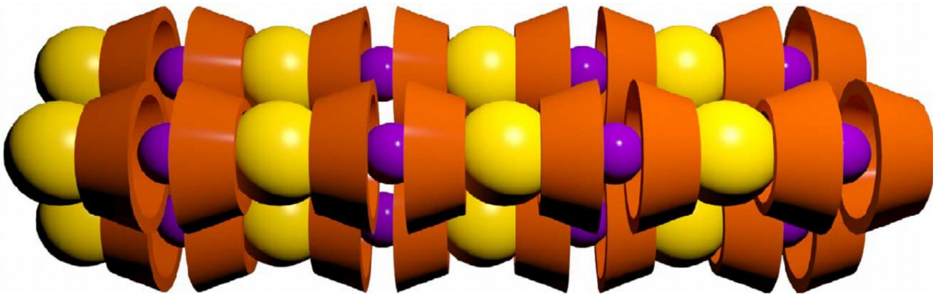

Figure 6. Schematic representation of the spontaneous self-assembly phenomenon upon mixing $KAuBr_4$ and alpha-CD in water, a hydrogen-bonded linear superstructure forms within a minute.

In 2014 Sir Fraser announced the formation of his private cyclodextrin company called **Cycladex Inc.** This firm focuses on the commercialization of selected technologies coming from Stoddart's laboratory at Northwestern University, in particular, utilization of alpha-cyclodextrin complexation for selective and efficient, environment-friendly gold extraction. This novel green chemical method has enormous advantages over the traditional Victorian cyanide process. The current standard gold mining technology uses annually about half a million tons of sodium cyanide, and large spills are unfortunately not seldom. The technology developed by Stoddart at Northwestern University is based on the recognition that alpha-CD will complex and crystallize gold bromide in a simple convenient manner. The cyclodextrin-enabled process provides a high (about 90 %) overall yield, with 99.9% chemical purity. The gold is generated by incineration, and the process is selective in presence of other metals such as Cu, Fe, Cd. The Cycladex company was formed by Sir Fraser Stoddart and his former student, Roger Pettman. The Company successfully applied for grants to finance feasibility phase and scale up and validation of the technology. Soon after the feasibility studies, the process has been used on 9 ores from around the world including Europe, Australia, Kyrgyzstan and the United States. So far more than 150 trials have shown that the system extracts typically 90-95% yield in less than 2 hours, which is faster and more efficient than the Victorian cyanide process. A summary of Cycladex, alpha-cyclodextrin-enabled technology is shown below in Figure 7.

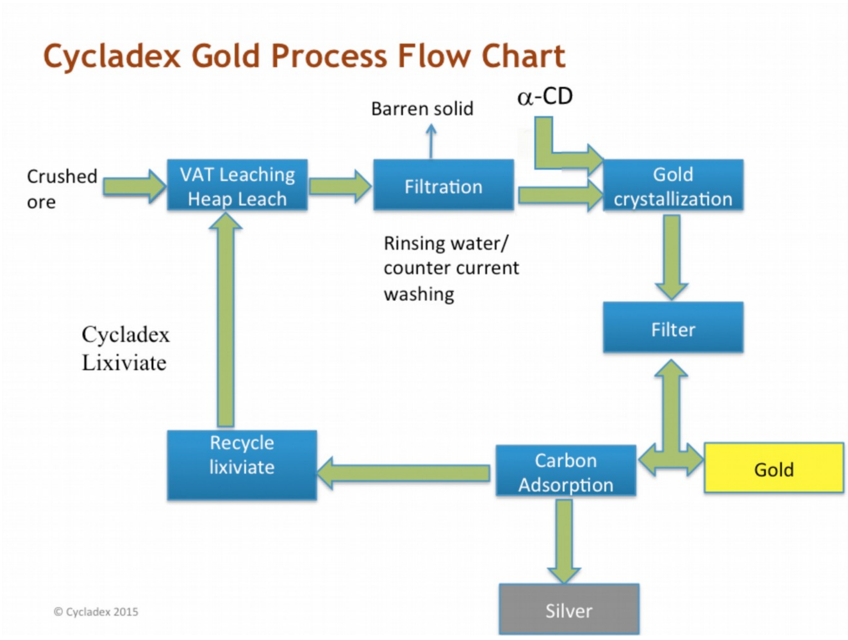

Figure 7. Fow chart of Stoddart's/Cycladex's gold mining process using alpha-CD [18].

Summary

Professor Stoddart was knighted by Her Majesty the Queen for his work in nanotechnology. He also received King Faisal International Prize in Science, the Albert Einstein World Award in Science, the Feynman Prize in Nanotechnology, and the Tetrahedron Prize for Creativity in Organic Chemistry and finally, the Nobel Prize in Chemistry in 2016. Sir Fraser is an exceptional person, scientist, phylantropist, and he is one of the world's most innovative organic chemists, who richly deserves all above high honors. For about half of a century, his research has consistently defined the frontier of science and innovation in his field.

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L-glutamic acid units, Hydrogels, Chiral-twist structures, α-CD, Enhanced photoisomerization-transition efficiency

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Imidazole antifungal agent, Co-precipitation technique, Diffusion-controlled mechanism, BCD, HPBCD

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Inhalation, Nanosized L-leucine crystals, Calu-3 cell monolayer

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Drug delivery, Tissue engineering, Regenerative medicine, Hydrophobic and hydrophilic groups within the cross-linked network

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Calcein, Inclusion of α-cyclodextrins

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Mechanical strength, Chemical stability, α-Cyclodextrin

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β-Cyclodextrin modified camptothecin, Esterification reaction, Click chemistry, Noncovalent interaction between β-cyclodextrin and adamantane, Adamantane modified hyaluronic acid

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Oleic acid, Elaidic acid, Inflammatory cytokines, Methyl-β-cyclodextrin

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Gene delivery, Polyamine-β-cyclodextrin-based cationic polyrotaxanes

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Cationic β-cyclodextrin-polyethyleneimine conjugates, Tumor suppressor microRNA, Tumor targeting

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Microwave irradiation, β-CD, Phase solubility studies, Vacuum oven drying, Freeze-drying, Spray-drying

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Drug coating, Andrographolide, Mannanase, Oligosaccharides, Gamma-cyclodextrin, Beta-cyclodextrin, Pharmaceutical and food industries

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α-CD, Poloxamines, Recycling

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Epichlorohydrin, Direct intercalation, Thermal stability

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