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2016 Nobel Prize in Chemistry for Molecular Machines

The Royal Swedish Academy of Sciences has decided to award **Jean-Pierre Sauvage**, **Sir James Fraser Stoddart** and **Bernard (Ben) L. Feringa** the Nobel Prize in Chemistry 2016 "for the design and synthesis of molecular machines". This is a high recognition for supramolecular chemistry utilizing building blocks of host-guest complexes including also cyclodextrin complexes.

The molecular machines mimic the movements of machines in a highly miniaturized scale. They need external fueling which is usually light or other energy. The first approach was topological entanglement (interlocked molecular assemblies). The building blocks are not covalently bound together but are entangled through loops and stoppers. *Catenanes* consisting of two interlocked rings and *rotaxanes* based on a ring threaded over an axle with stoppers are the main groups (Fig. 1).

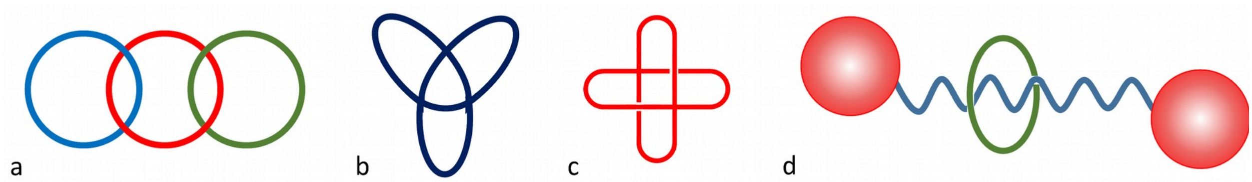

Fig. 1. Examples of molecules with topological entanglement: a) [3]catenane, b) trefoil knot, c) Solomon link (redrawn after [1]) synthesized by Jean-Pierre Sauvage et al. (CNRS, Louis Pasteur University, Strasbourg, France) in the eighties [2] and d) scheme of rotaxane

Dramatic, reversible changes in the catenanes' molecular shape were observed upon decomplexation and recomplexation of the metal coordination entities with Cu(I) as first examples of *translational isomerism* [3].

The group of Sir James Fraser Stoddart (University of Sheffield, UK) synthesized paraquat cyclophane structure threaded around an axle containing two hydroquinol units [4] (Fig. 2). The resulting rotaxane cyclophane ring could be shown to act as a *molecular shuttle*, able to move between the two hydroquinol stations on the axle. The trigger of the motion is electrochemical oxidation-reduction or pH change.

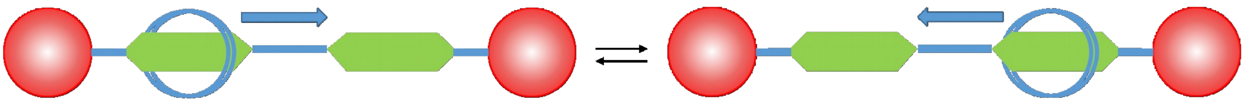

Fig. 2. Translational isomerism, molecular shuttle: the macrocycle can move between two positions

In parallel with the development of interlocked structures, systems based on isomerizable unsaturated bonds able to rotate unidirectionally in a controlled manner were synthesized. One of the first approaches published by Feringa's group (University of Groningen, the Netherlands) is illustrated in Fig. 3 [5]. Taking together 4 such motors into one structure resembling to a 4-wheeled car, this group constructed the prototype of a 'nanocar' (four-wheeled molecule) [6].

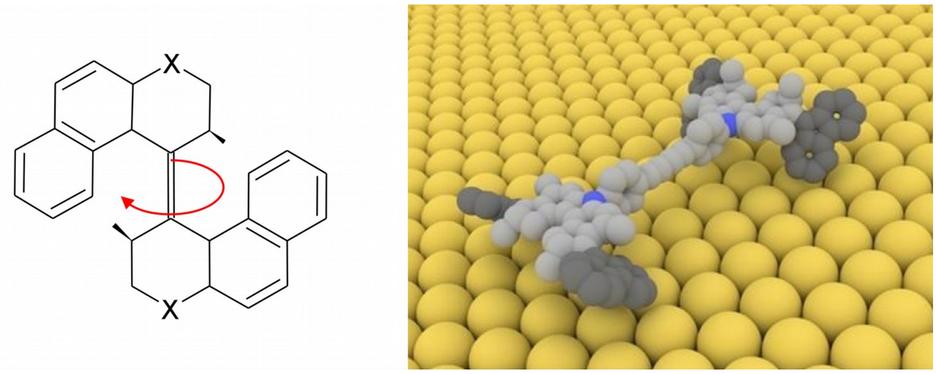

Fig. 3. Unidirectional molecular rotary motor (the cis–trans isomerization of the double bond on the effect of UV-light is the driving force) and the 'nanocar' equipped with 4 such rotors [6, 7]

Both linear and rotary motion was achieved on molecular scale providing artificial molecular machines built up from shuttles and switches (motors and pumps) where supplies of energy in the form of chemical fuel, electrochemical potential and light activation become a minimum requirement for them to function away from equilibrium [8].

Activity of Nobel laureates with cyclodextrins

According to Scopus all the three Nobel-Prize laureates have abundantly published their findings (Table 1). Although all of them mentioned CDs in their reviews or introduction of their research papers, only Stoddart was active in the development of novel structures including CD as building block.

Table 1 Publications statistics (Scopus, accessed on 24 November 2016)

	No. of papers	No. of papers mentioning CDs	CDs in the title, abstract or keywords
Stoddart, J.F.	904	279	59
Sauvage, J.P.	533	73	0
Feringa, B.J.	739	26	0

Stoddart was born in 1942 and studied at Edinburgh University. Later on he made research at various universities in the US, UK and Canada. He was made a Knight Bachelor by Her Majesty Queen Elizabeth II. in 2007 for his services in chemistry and molecular nanotechnology.

Sir J.F. Stoddart was identified as one of the most-cited chemists in 1995–2005 period with over 11000 citations [9]. In October 2016 he had 83941 citations, 35439 since 2011 (Google Scholar, accessed on 20 November, 2016) with 7 papers of over 1000 citations. The three most cited papers are:

- Artificial molecular machines (Angew. Chemie [10], No. of citations: 2102)
- Self-assembly in natural and unnatural systems (Angew. Chemie [11], No. of citations: 1919)
- Electronically configurable molecular-based logic gates (Science [4], No. of citations: 1823)

Stoddart started to use the following expressions in his papers: switchable molecular devices [12], artificial molecular pumps [13], supramolecular devices, mechanically linked polymers, molecular elevators [14], molecular computers, molecular electronics [15], molecular logic gates [16], etc.

His CD-related activity covered also other topics, such as gold recovery, catalysis, metal-organic frameworks, sensors, etc.

Molecular machines with CDs

Preparation of interlocked systems

The very first attempt to thread CD on an axle molecule was a catenane prepared by the group of Friedrich Cramer (Fig. 4) preceding the works of the present Nobel laureates.

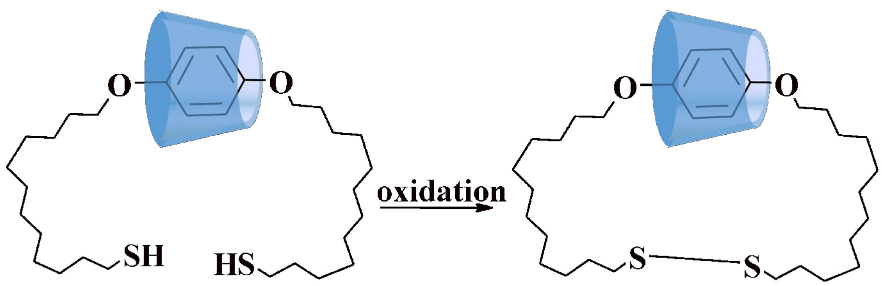

Fig. 4. Scheme of the very first catenane using αCD prepared by Cramer's group published in 1958 [17]

The very first *rotaxane* containing CD was prepared by Hiroshi Ogino (Tohoku University, Japan) utilizing the non-covalent interaction between α,ω-diaminoalkenes and CD using $CoCl(ethylenediamine)_2$ bulky groups as stoppers [18].

The first *polyrotaxanes* containing several CDs threaded on an axle molecule was published in the same year (in 1992) by Harada et al. and Wenz et al. (Fig. 5) [19, 20]. Harada used diamine-terminated polyethylene glycol and aCD to get a molecular necklace attaching dinitrofluorobenzene groups as stoppers, while Wenz stringed αCD rings on polyiminooligomethylene chains and terminated the chains with nicotinoyl groups. However, these systems did not show controllable motion.

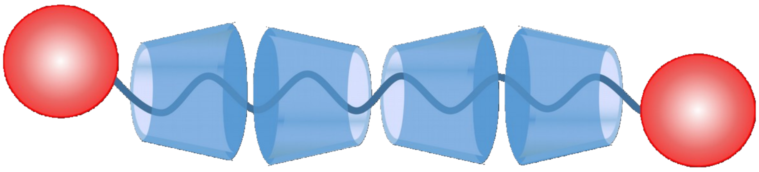

Fig. 5. Scheme of polyrotaxanes

Stimuli-responsive molecular devices and molecular machines with CDs

Typical photoswitchable molecular devices based on *cis–trans* photoisomerization of azobenzene moiety (Fig. 6) were prepared by several groups [21, 22]. Light-driven rotaxane molecular shuttles and nanovalves containing αCD and azobenzene unit were constructed. The *cis–trans* photoisomerization of azobenzene moiety induces reversible motion of the CD ring on the effect of UV (hv) and visible (hv^1) irradiation (Fig. 6 and 7) [23].

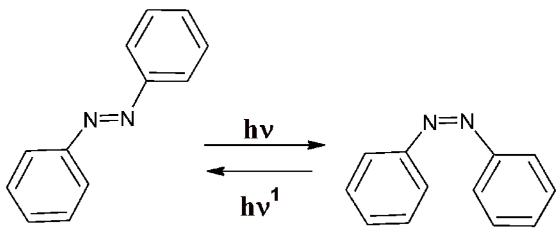

Fig. 6. Photoisomerization of azobenzene

CD selectively binds to *trans*-azobenzene, which is less hydrophilic than the *cis* isomer. Upon irradiation the *trans*-azobenzene is transformed into the *cis* form causing αCD to unthread. Thermal relaxation allows *cis*-azobenzene to transform back to *trans* isomer and αCD to rebind.

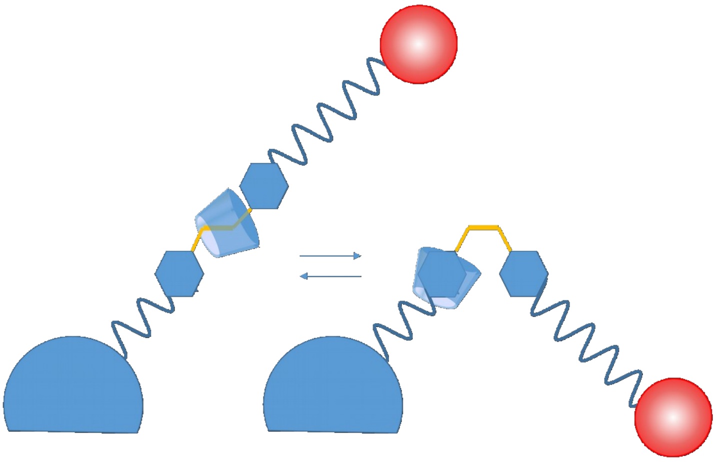

Fig. 7. Nanovalves based on cis–trans photoisomerization of azobenzene attached to mesopores of silica nanoparticles [23]

Various other stimuli-responsive systems have been published. For instance, pH-responsive materials (Fig. 8) [24], redox-switchable molecular machines [25], etc. Some recent reviews give detailed overview on such systems [26–30].

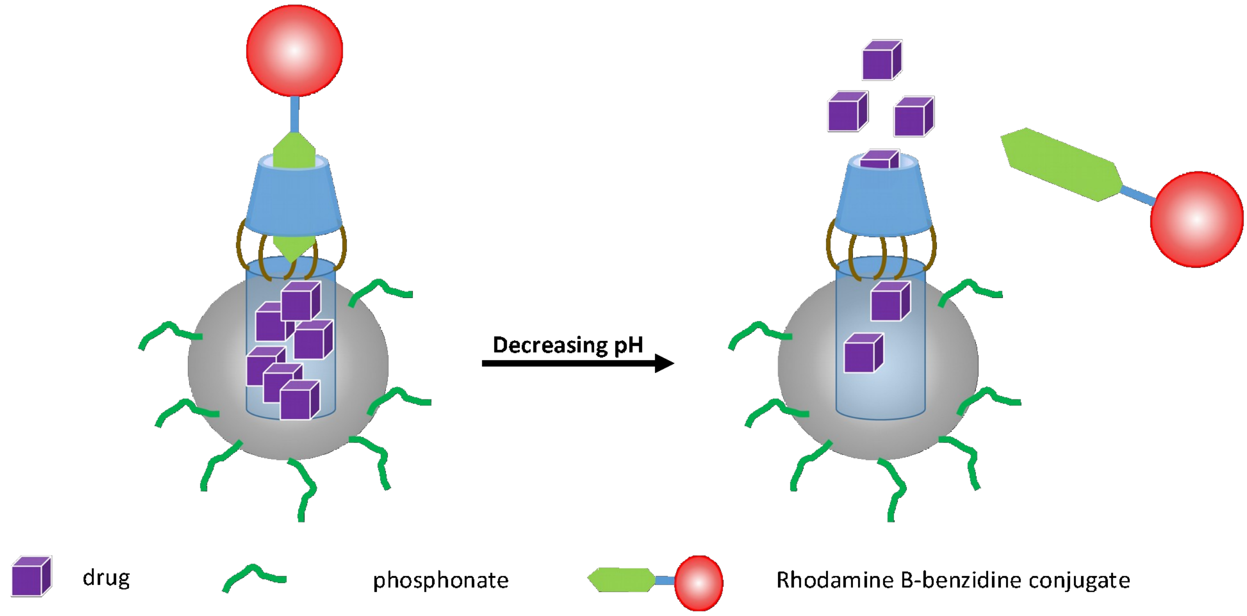

Fig. 8. Molecular pistons used for controlled drug delivery [redrawn after 24]. This invention of Stoddart's team is based on phosphonate-coated silica nanoparticles with a BCD monolayer. Rhodamine B-benzidine conjugate as nanopiston moving in and out of the CD cavity controls the release of the drug from the nanopores of silica. The trigger is the increase or decrease of the pH.

It is a great honor to the entire cyclodextrin society of the world that molecular machines were selected for 2016 Nobel Prize, a field of supramolecular chemistry including advances in cyclodextrin chemistry.

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Phase solubility studies, Sol-gel phase transition

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Curcumin, Magnetic nanoparticles, Cytotoxicity

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Electrospinning, Polyacrylic acid, Modelling, Phase solubility, Antioxidant activity, Photostability

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Sodium lauryl sulfate, Binary solvent mixture, Jouyban-Acree model

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Local anesthetics, Intermolecular hydrogen bonds, Enhanced sensory blockade, Bupivacaine, Drug delivery, Molecular dynamics

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Antidepressant in diabetic patients, Bioavailability, Pancreatic islets, Insulin

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Gatekeeper β-CD, Arg-Gly-Asp, Peptide ligand, Doxorubicin, Magnetically enhanced accumulation, Theranostic nanoplatform for cancer treatment

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Poly-αCD, Poly-βCD, Wound dressing, Tunable release rate, Poly-(ε-caprolactone), Poly(N-vinylpyrrolidone), Hexamethyldisiloxane, Cyclodextrin polymers, Fluconazole

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Extraction of cholesterol from the plasma membrane, Triethoxy(3-isocyanatopropyl)silane, Magnetic manipulation, Maghemite nanoparticles

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5. CDs in Food, Cosmetics and Agrochemicals

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Active packaging, Co-precipitation method, 2-Nonanone, Antimicrobial

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1,4-Phenylene diisocyanate linker, DDT, DDD, DDE, Adsorption

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α-CD, β-CD, γ-CD, RAME-β-CD, Delay in the photodegradation process, Adsorption, Inclusion complex

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Pome fruit, Electrocatalytic oxidation, Voltammetric sensor

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Protein imprinting, Peptide biomarker, Alzheimer disease, α-CD, Natural building blocks, Screen-printed electrodes, Biosensor

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Electrochemical sensor, Screen-printed carbon electrode, Differential pulse voltammetry

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β-CD, Antipsychotic, Experimental design

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Safranine T, SS–β–CD functionalized reduced graphene oxide, "Switch–on" fluorescence response, Graphene

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Highly sensitive and simultaneous electrochemical determination of 2-aminophenol and 4-aminophenol based on poly(l-arginine)-β-cyclodextrin/carbon nanotubes@graphene nanoribbons modified electrode

Core-shell heterostructure

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