РОССИЙСКАЯ АКАДЕМИЯ НАУК
ЦЕНТР ФОТОХИМИИ

V Всероссийская конференция с международным участием по органической химии

Molecular meccano of photoactive supramolecular devices and machines based on unsaturated and macrocyclic compounds

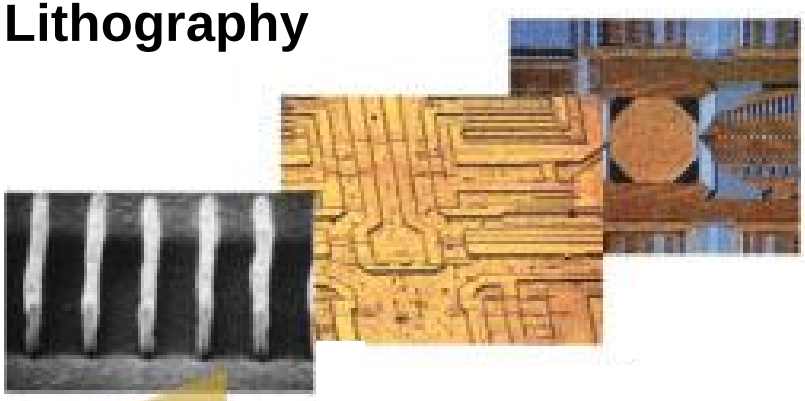
Prof. Sergey P. Gromov

http://suprachem.photonics.ru
http://www.chem.msu.ru/rus/lab/organic/supra-nano.html

NANOTECHNOLOGY “BOTTOM-UP”

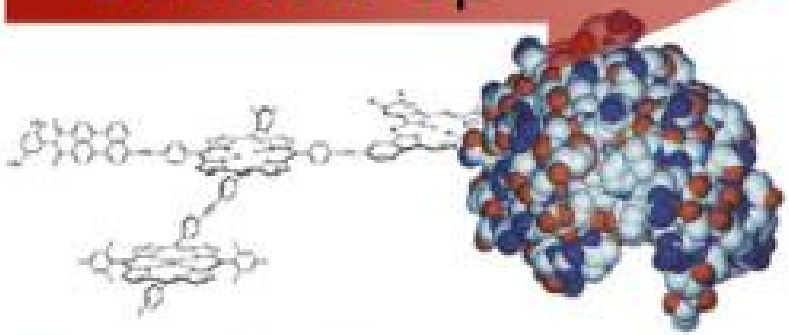
STRATEGIES OF CREATION OF NANOSIZED ARCHITECTURES

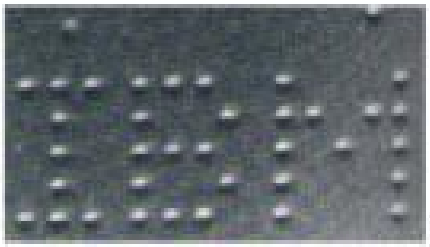
Lithography



Bottom-Up

Top-Down





Organic synthesis
Supramolecular Self-Assembly

HIERARCHY OF STRUCTURAL ORGANIZATION OF MATTER

Atoms a b c

Molecules A (a-a) B (a-b) C (a-c)

covalent bonds

Supramolecular systems A…..A A…..B Supramolecules

A…..B…..C
⋮ ⋮
C…..A

Supramolecular ensembles

noncovalent bonds
(intermolecular)

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TYPES OF INTERMOLECULAR BONDS

Coordination bonds

Ion-ion interactions

Ion-dipole interactions

Hydrogen bonds

Dipole-dipole interactions

Stacking interactions

Hydrophobic interactions

SUPRAMOLECULAR DEVICES AND MACHINES

Supramolecular devices are structurally organized and functionally integrated chemical systems.

Systems that function as a result of mechanical motion of components relative to each other are called *supramolecular machines*.

J.-M. Lehn

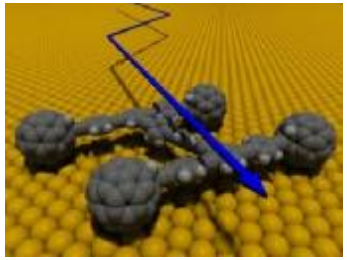
They can be used:

"to design machines for energy and motion generation, conversion, and transmission at nanolevels, to devise a nanotool for the monitoring and diagnostics of nanoquantities of materials and substances.

Critical technologies of the RF

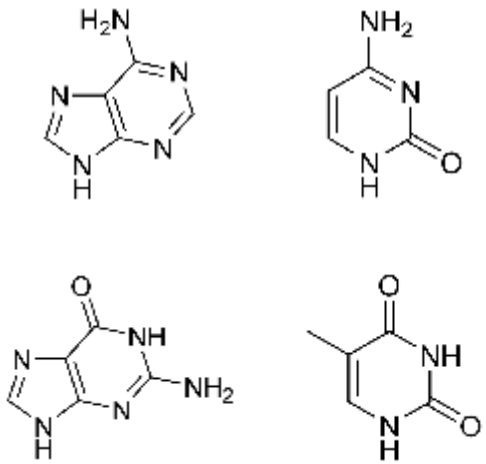
Means for control of supramolecular devices and machines

- **Photoswitching -** $h\nu$
- **Electrochemical switching -** e^-
- **Chemical switching -** H^+, M^{n+}
- **Thermal switching -** Δ

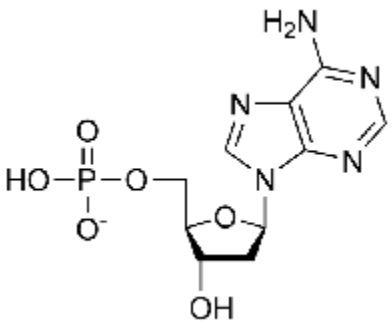


MOLECULAR MECCANO IN LIVING NATURE

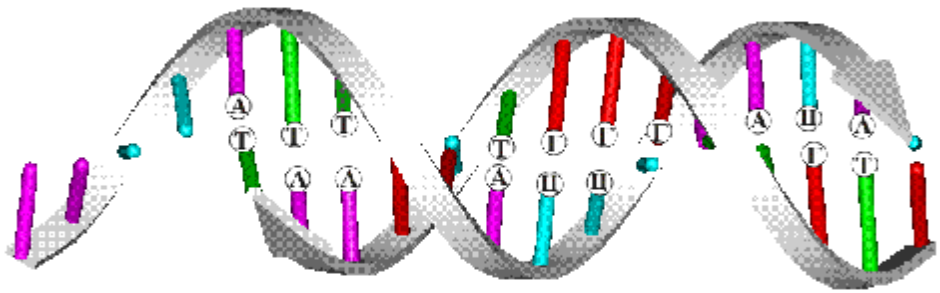
Nucleic acids



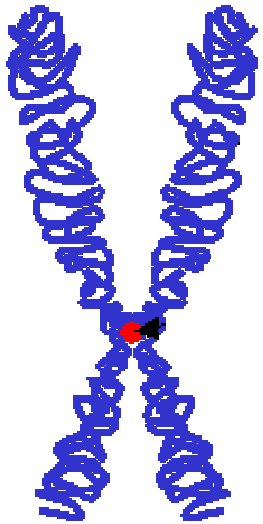
basic nitrogens



nucleotides

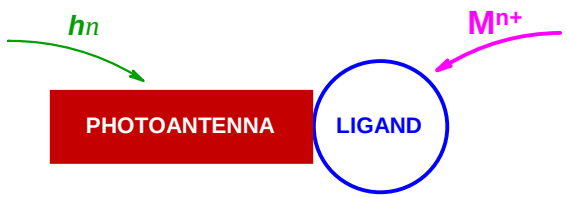


nucleic acids

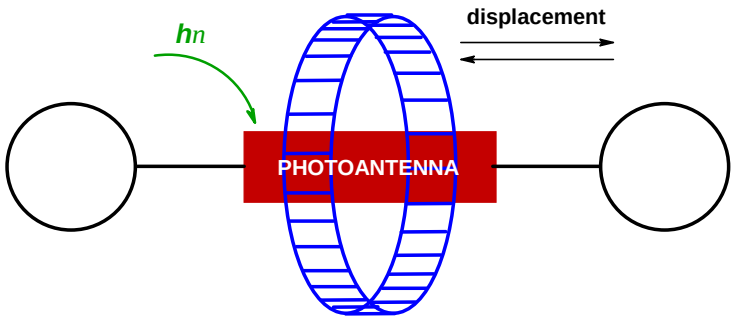


chromosomes

MOLECULAR MECCANO
OF PHOTOACTIVE SUPRAMOLECULAR DEVICES AND MACHINES
IN NANOTECHNOLOGY

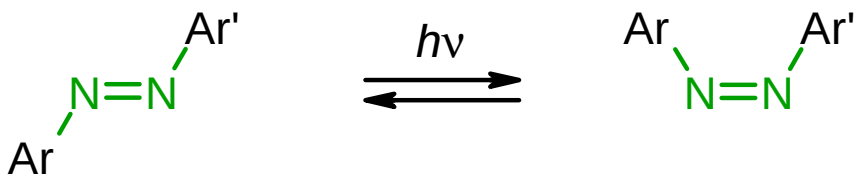


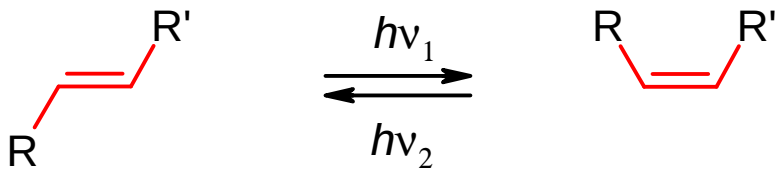
photoswitchable molecular device



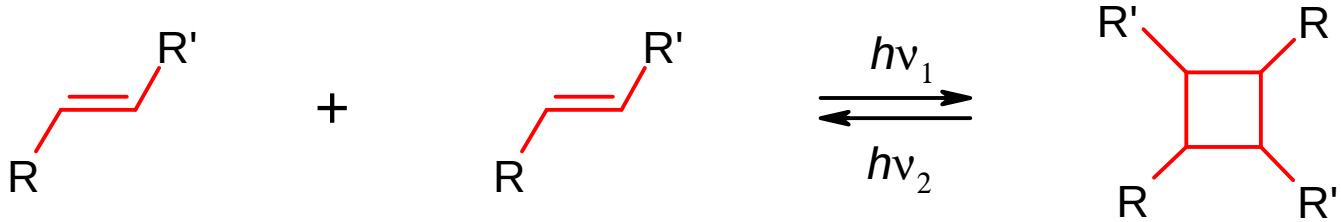
photocontrolled molecular machine

PHOTOANTENNAS OF SUPRAMOLECULAR DEVICES AND MACHINES BASED ON UNSATURATED COMPOUNDS





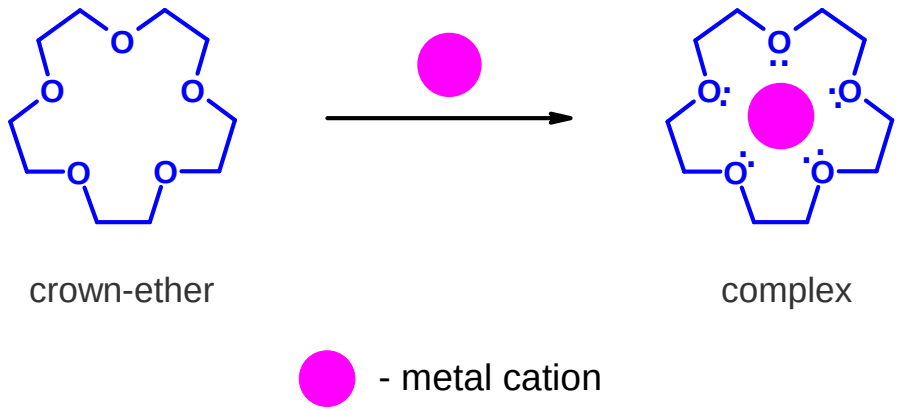
trans-isomer *cis*-isomer

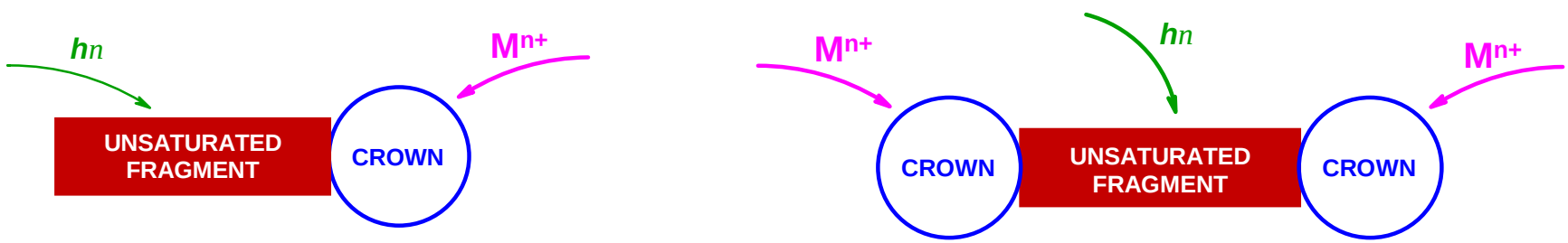


cyclobutane

Gromov S. P. *Russ. Chem. Bull*. **2008**, *57*, 1325 (review);
Gromov S. P. *Rev. J. Chem.* **2011**, *1*, 1 (review);
Ushakov E. N., Gromov S. P. *Russ. Chem. Rev.* **2015**, *84,* 787 (review).

PHOTOSWITCHABLE SUPRAMOLECULAR DEVICES BASED ON UNSATURATED AND CROWN COMPOUNDS





Gromov S. P. *Russ. Chem. Bull*. **2008**, *57*, 1325 (review);
Ushakov E. N., Alfimov M. V., Gromov S. P. *Russ. Chem. Rev.* **2008**, *77*, 39 (review);
Alfimov M. V., Fedorova O. A., Gromov S. P. *J. Photochem. Photobiol., A* **2003**, *158*, 183 (review).

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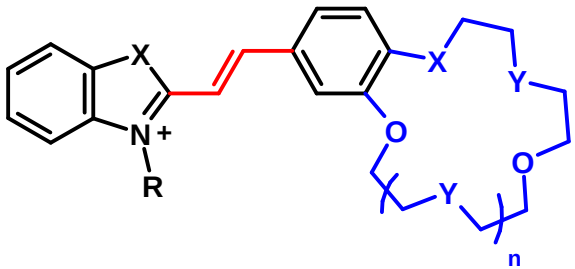
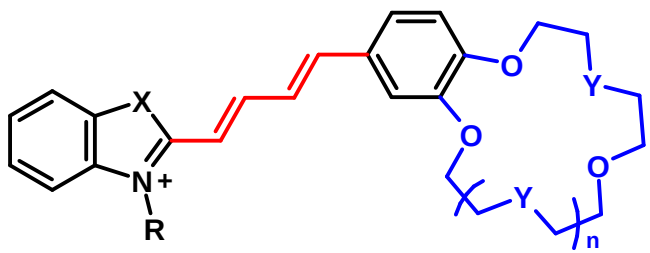
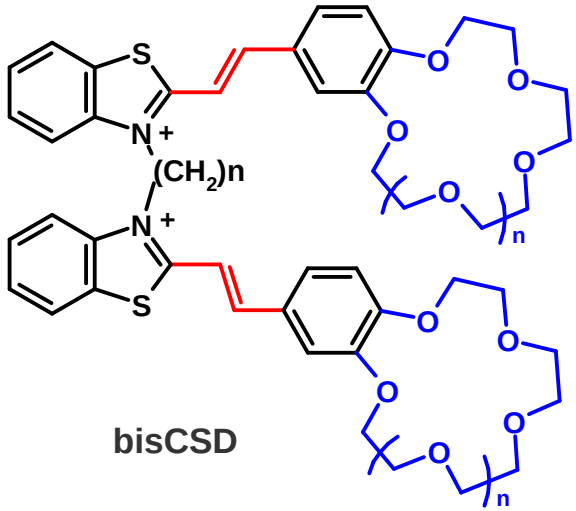
Crown-containing unsaturated compounds



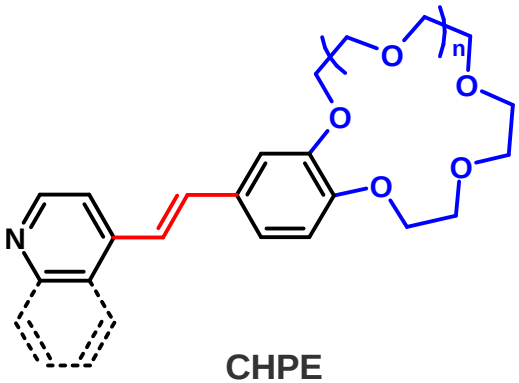
CSD



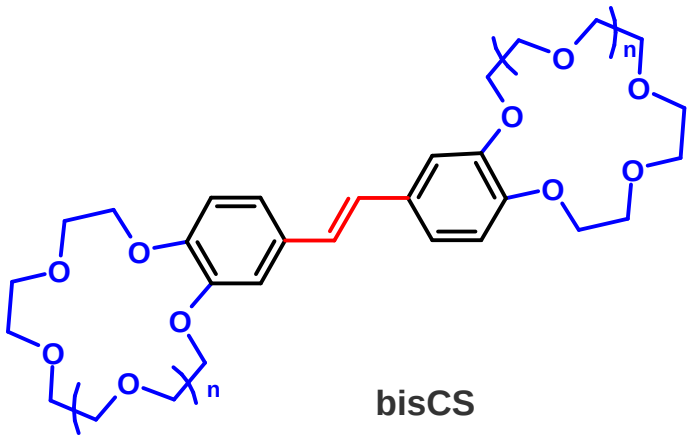
CBD



bisCSD



CHPE



bisCS

n = 1, 2

Gromov S. P., Alfimov M. V. *Russ. Chem. Bull*. **1997**, *46,* 611 (review);
Gromov S. P. *Russ. Chem. Bull*. **2008**, *57*, 1299 (review).

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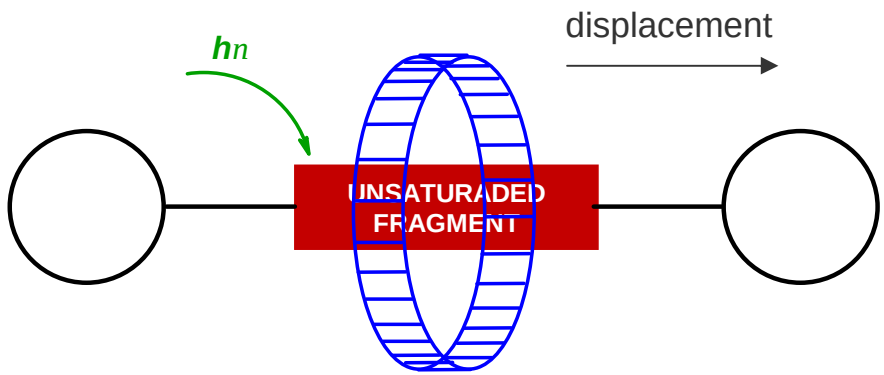
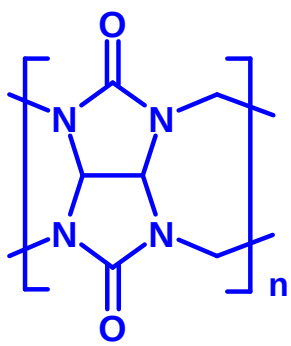
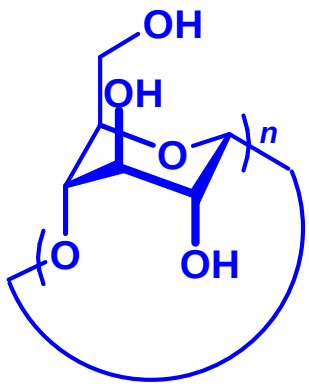
Photocontrolled supramolecular machines based on unsaturated compounds, cucurbiturils and cyclodextrins



photocontrolled supramolecular machine



n = 6-8

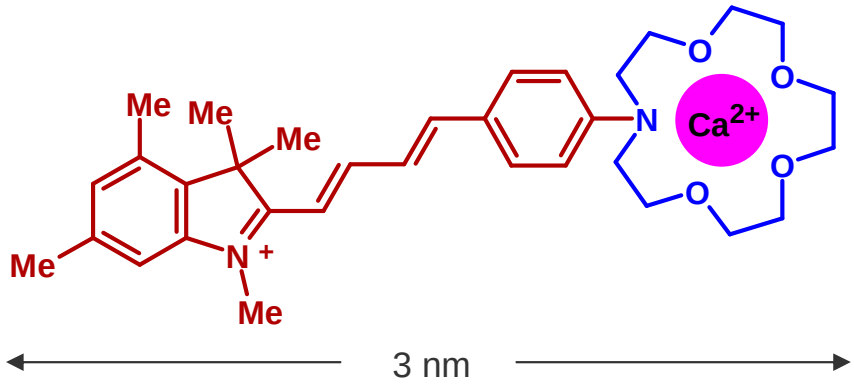


cucurbiturils

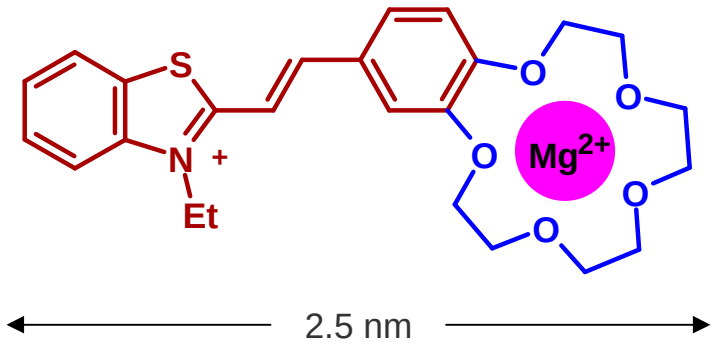
cyclodextrins

Gromov S. P. *Russ. Chem. Bull*. **2008**, *57*, 1325 (review);
Gromov S. P. *Rev. J. Chem.* **2011**, *1*, 1 (review)
Ushakov E. N., Gromov S. P. *Russ. Chem. Rev.* **2015**, *84*, 787 (review).

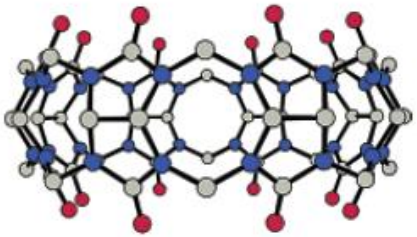
COMPONENT SIZES
IN PHOTOACTIVE SUPRAMOLECULAR DEVICES AND MACHINES



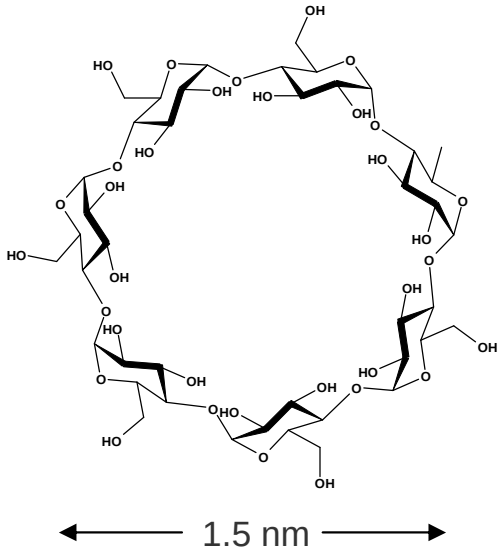
Complex of butadienyl dye



Complex of styryl dye



Cucurbit[8]uril



β-Cyclodextrin

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Self-assembly
of photoswitchable supramolecular devices
with participation of metal cations

Part I

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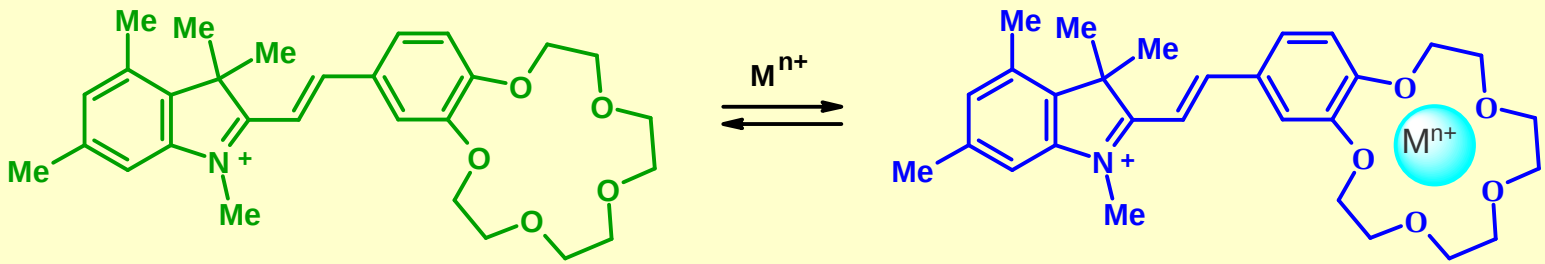
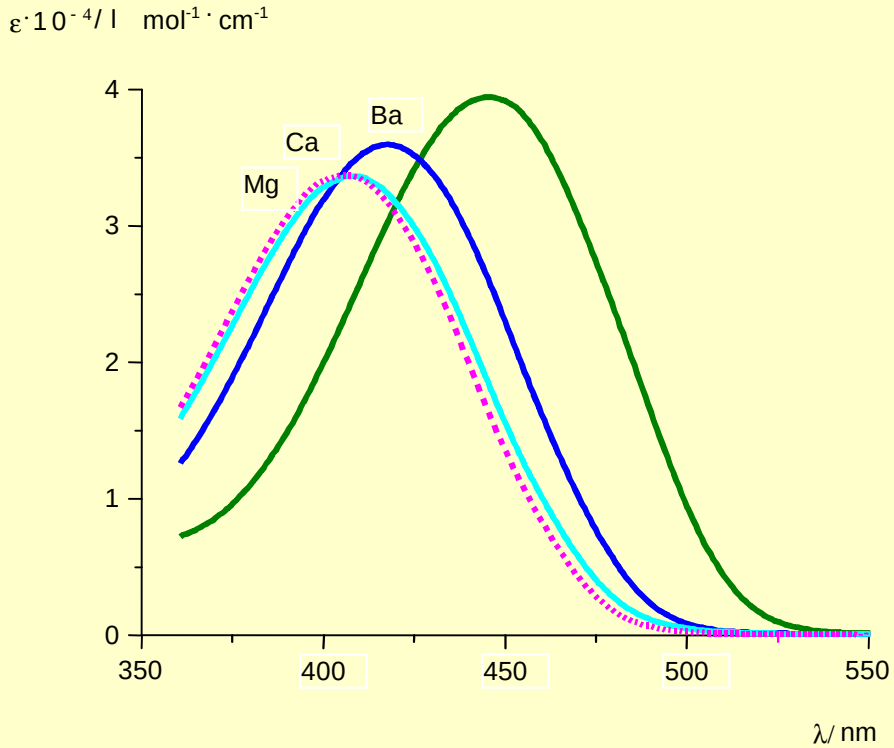
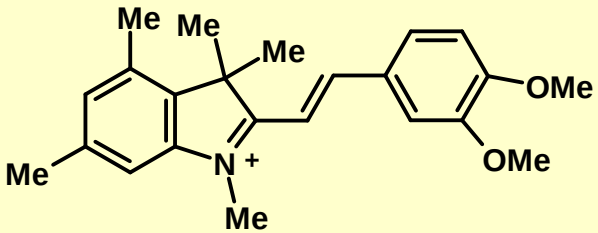
Complex formation



trans-isomer





Dokl. Chem. **1990**, *314*, 279;
Ushakov E. N., Gromov S. P. et al. *Macroheterocycles.* **2010**, *3*, 189 (review)
J. Org. Chem. **2013**, *78*, 9834.

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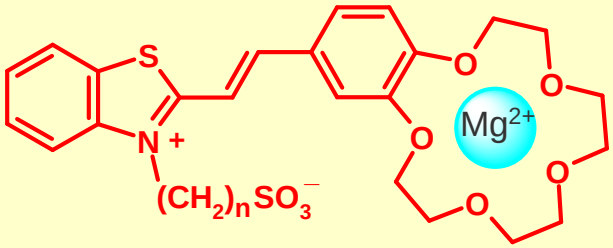
Photoswitchable supramolecular devices

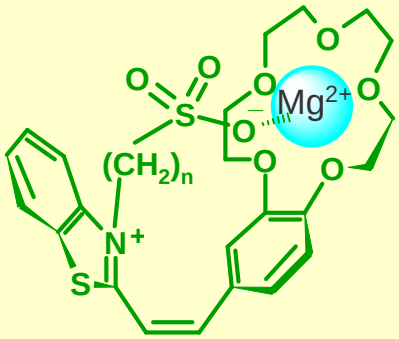


(*trans*-**L**)·Mg^{2+}

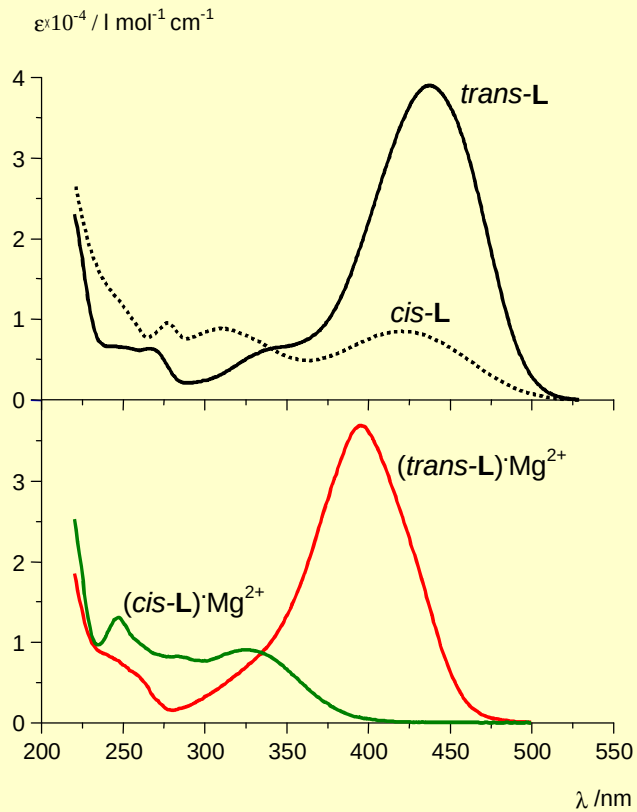
$h\nu_1$ (436 nm) →

← $h\nu_2$

j_{tc} ~ 0.5



anion-"capped"
(*cis*-**L**)·Mg^{2+}



Dokl. Chem. **1991**, 317, 99;
Chem. Phys. Lett. **1991**, *185*, 455;
J. Am. Chem. Soc. **1992**, *114*, 6381;
J. Am. Chem. Soc. **1999**, *121*, 4992.

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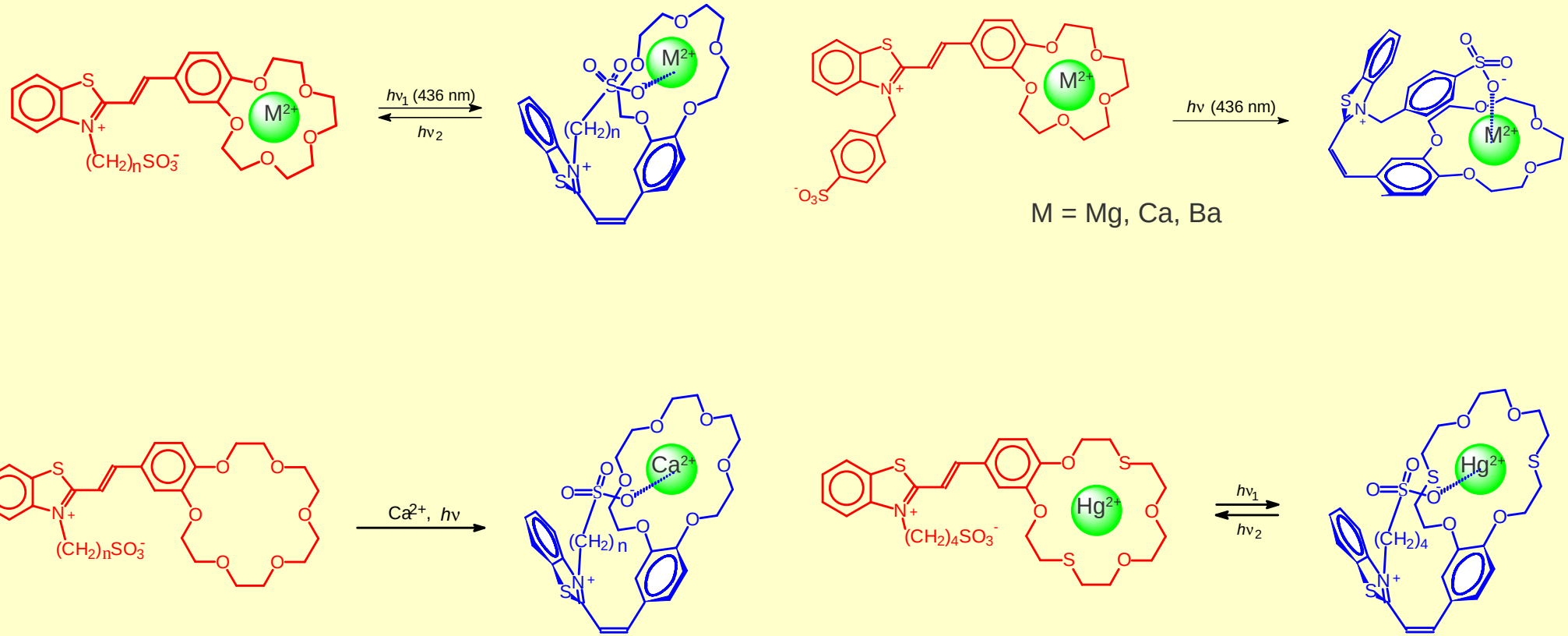
Anion-“capped” complexes



Russ. Chem. Bull. **1998**, *47*, 97;
J. Chem. Soc., Perkin Trans. 2. **1999**, 601;
J. Am. Chem. Soc. **1992**, *114*, 6381;
J. Am. Chem. Soc. **1999**, *121*, 4992.

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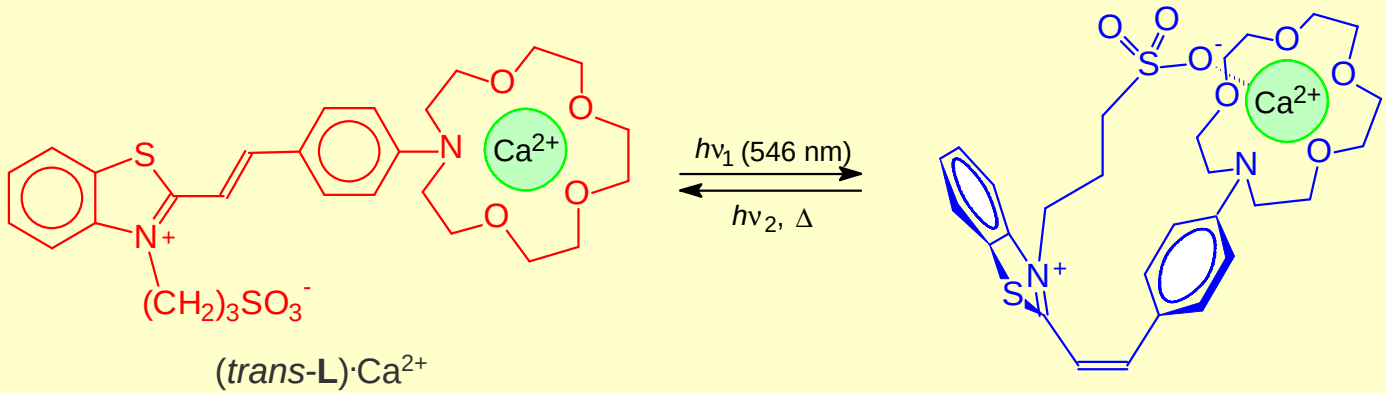
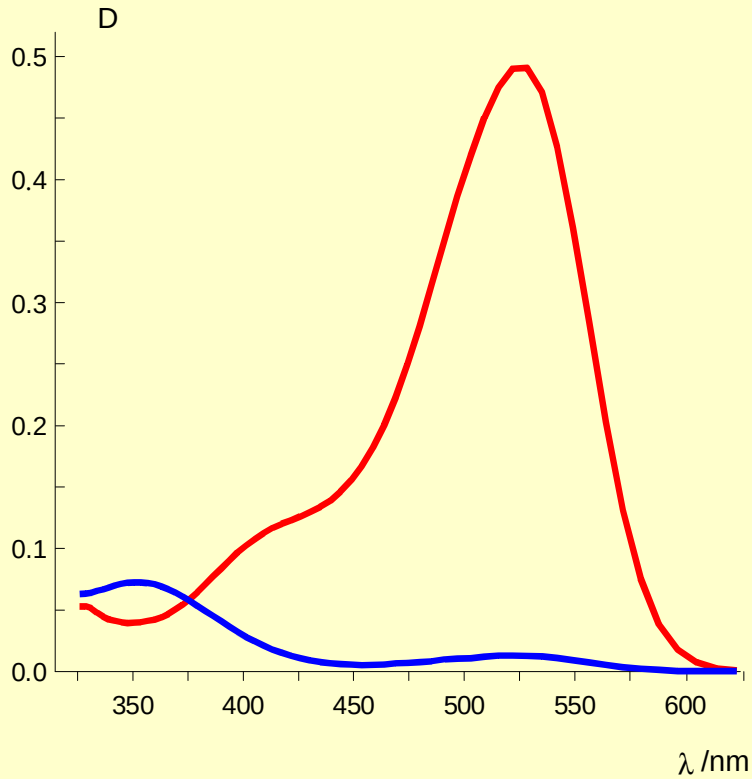
Formation of anion-“capped” complex



(*trans*-**L**)·Ca^{2+}

anion-“capped”
(*cis*-**L**)·Ca^{2+}



Russ. Chem. Bull. **1997**, *46*, 1143.

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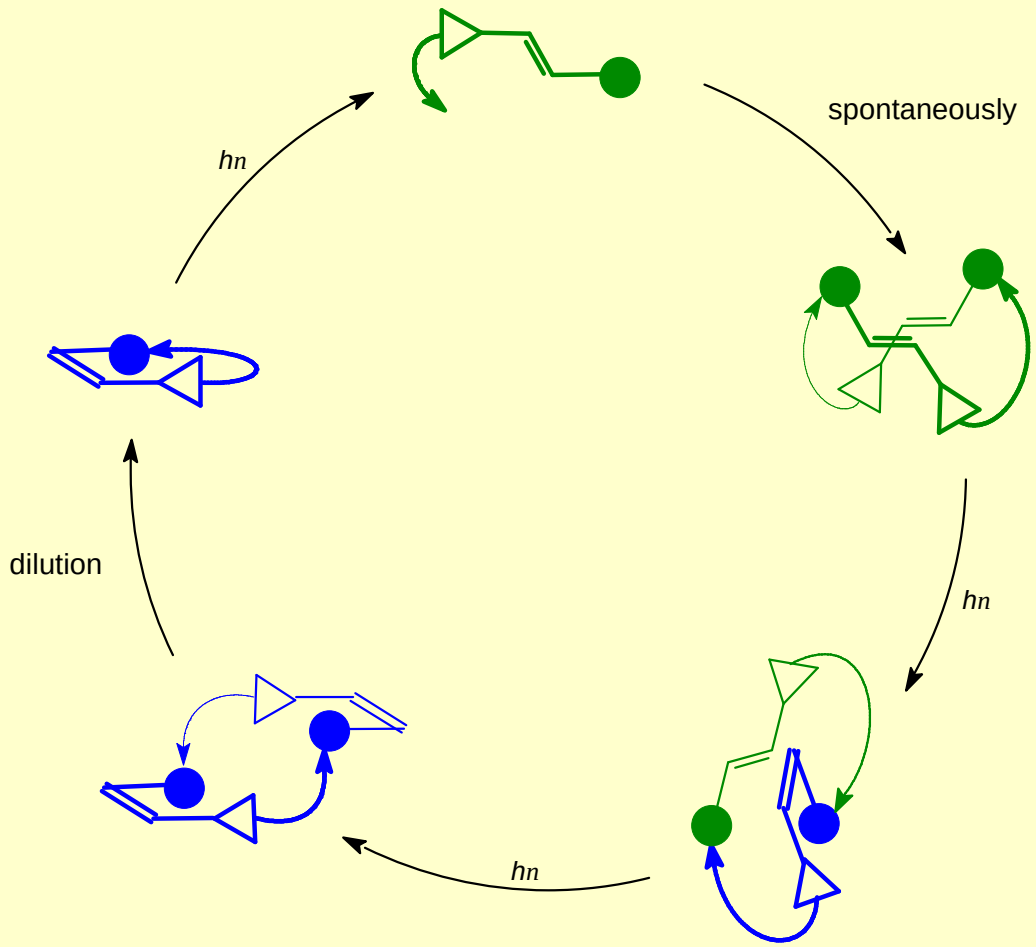
PHOTOCHEMISTRY CENTER

Photocycle of crown-containing styryl dyes



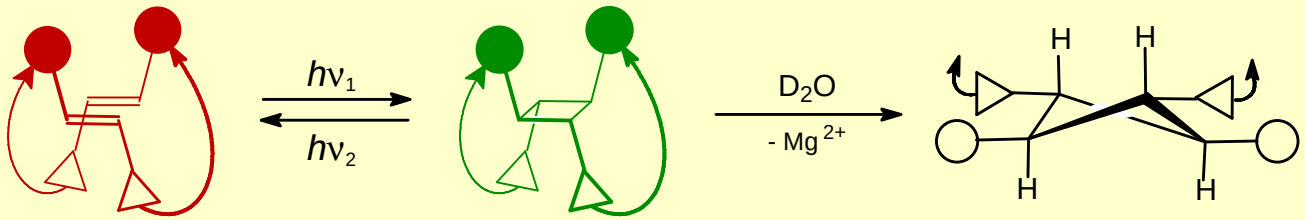
● - is the benzocrown compounds moiety with M^{2+} (Mg, Ca, Hg, Pb);

▷ - is the benzothiazolium moiety; ↷ - $(CH_2)_nSO_3^-$

Gromov S. P. *Russ. Chem. Bull*. **2008**, *57*, 1325 (review).

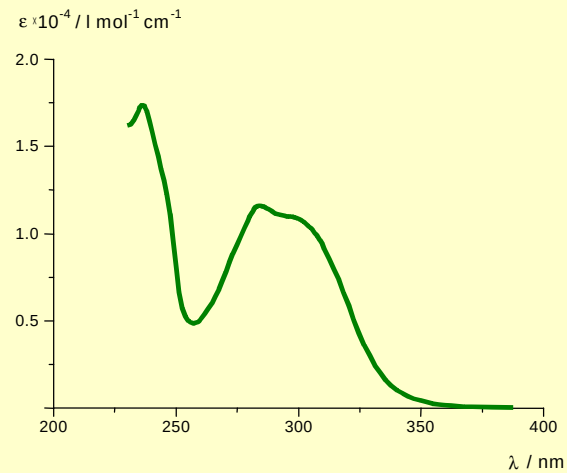
THE RUSSIAN ACADEMY OF SCIENCES
PHOTOCHEMISTRY CENTER

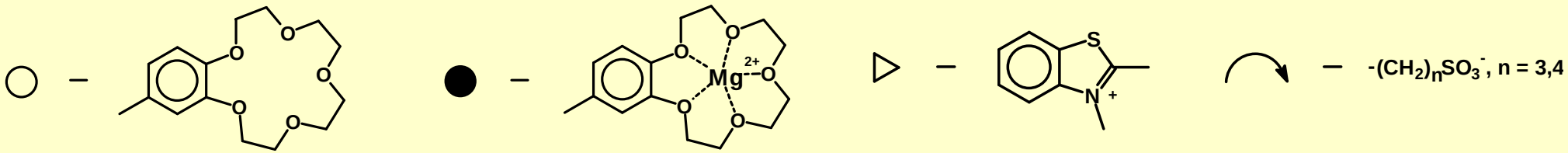
Photoswitchable supramolecular devices



[2+2] Photocycloaddition of CSD

C_L, /mol · l^{-1}	$5 \cdot 10^{-6}$	$2.4 \cdot 10^{-5}$	$4.5 \cdot 10^{-5}$	$2.1 \cdot 10^{-4}$	$2 \cdot 10^{-3}$
F	0.0022	0.0043	0.0052	0.0051	0.0055





J. Am. Chem. Soc. **1992**, *114*, 6381;
Russ. Chem. Bull. **1993**, *42*, 1385;
J. Chem. Soc., Perkin Trans. 2. **1999**, 601;
J. Org. Chem. **2003**, *68*, 6115.

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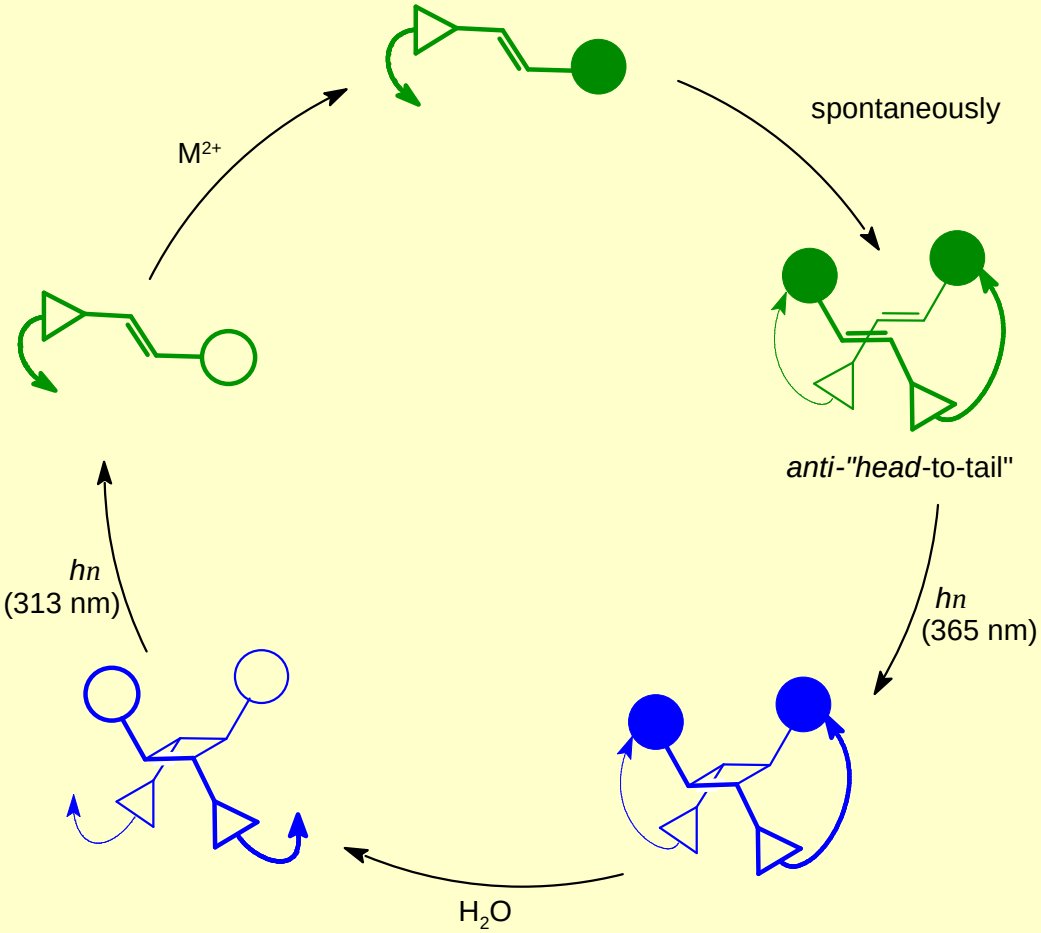
Photocycle of crown-containing styryl dyes



○ is the benzocrown compounds moiety; ● is the benzocrown compounds moiety with M^{2+} (Mg, Ca, Hg, Pb);

▷ is the benzothiazolium moiety; ⌒ - $(CH_2)_nSO_3^-$

Gromov S. P. *Rev. J. Chem.* **2011**, *1*, 1 (review).

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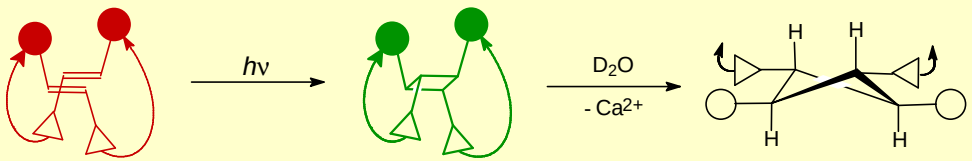
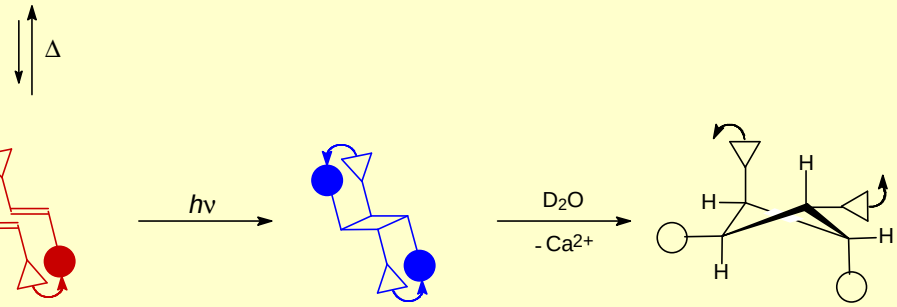
[2+2]-Photocycloaddition of CSD



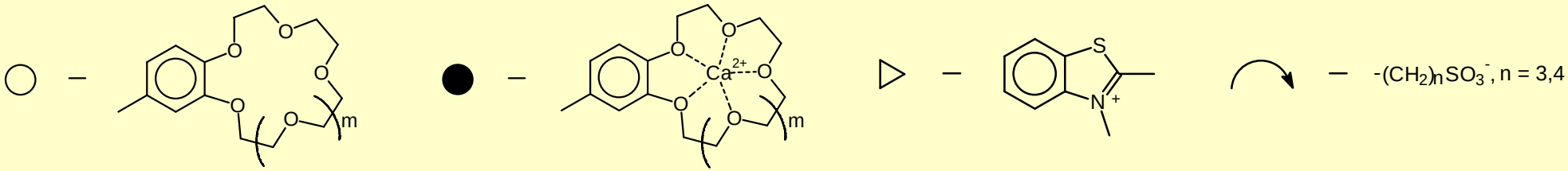
anti-[(*trans*-**L**)·Ca^{2+}



sin-[(*trans*-**L**)·Ca^{2+}

n = 3, m = 2

CSD	n = 3 m = 1	n = 4 m = 1	n = 3 m = 2	n = 4 m = 2
F	0.001	0.01	0.0004	0.06



Russ. Chem. Bull. **1996**, *45*, 654.

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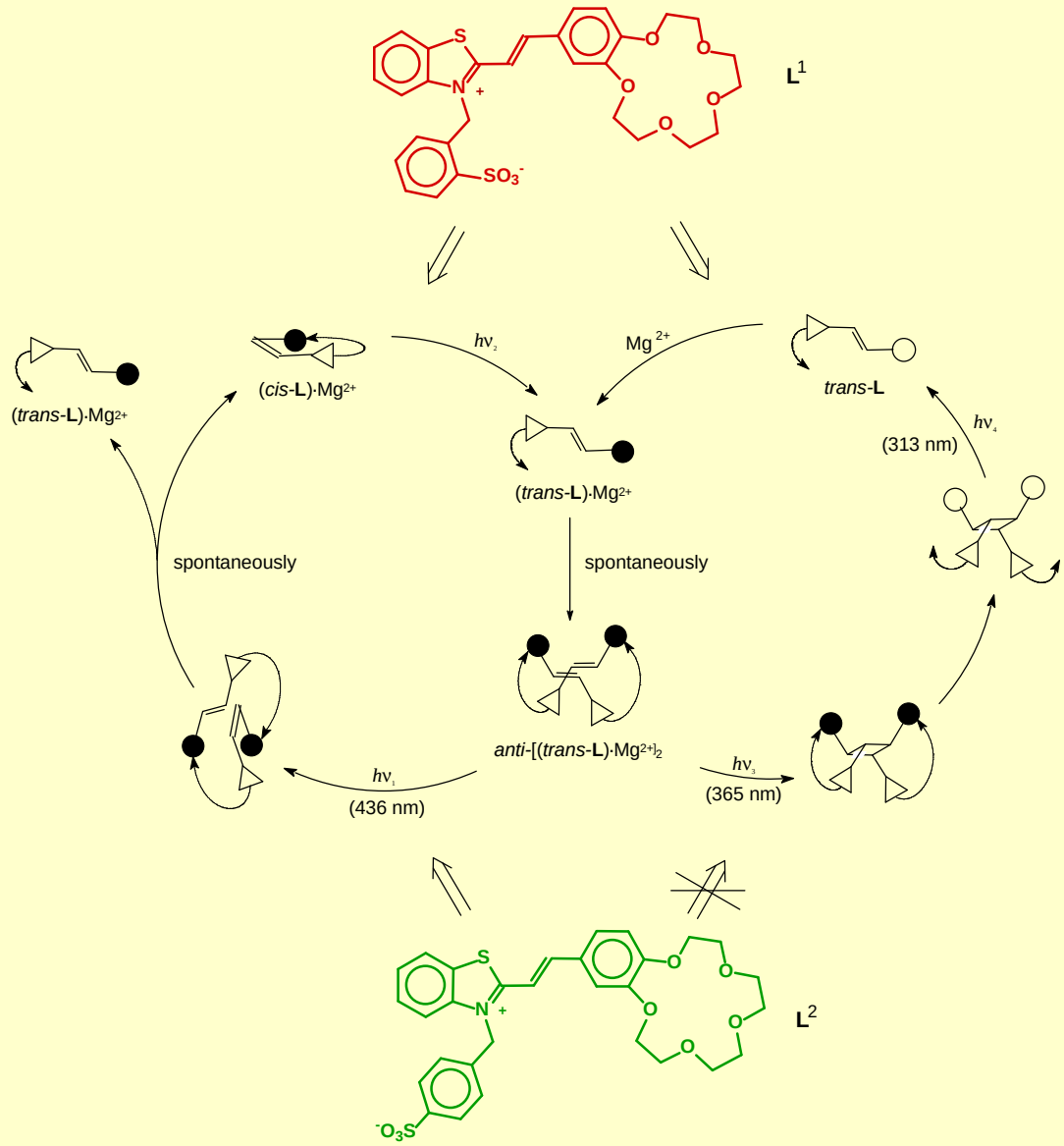
PHOTOCHEMISTRY CENTER

[2+2] PHOTOCYCLOADDITION OF MULTIPHOTOCHROMIC CSD



CSD	R, A°	F	$\log K_1$
L[1]	6.7	0.018	7.3
L[2]	9.9	---	10.0

Russ. Chem. Bull. **1998**, *47*, 97; *J. Chem. Soc., Perkin Trans. 2.* **1999**, 601.

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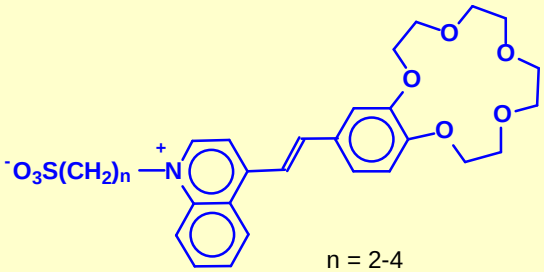
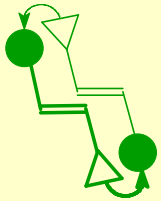
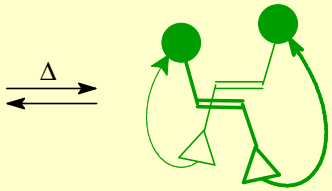
^{1}H NMR SPECTRA



n = 2-4

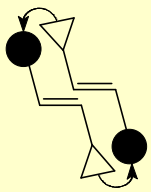


syn-"head-to-tail"

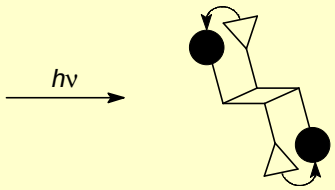


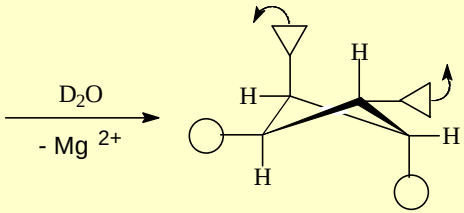
anti-"head-to-tail"

n = 3



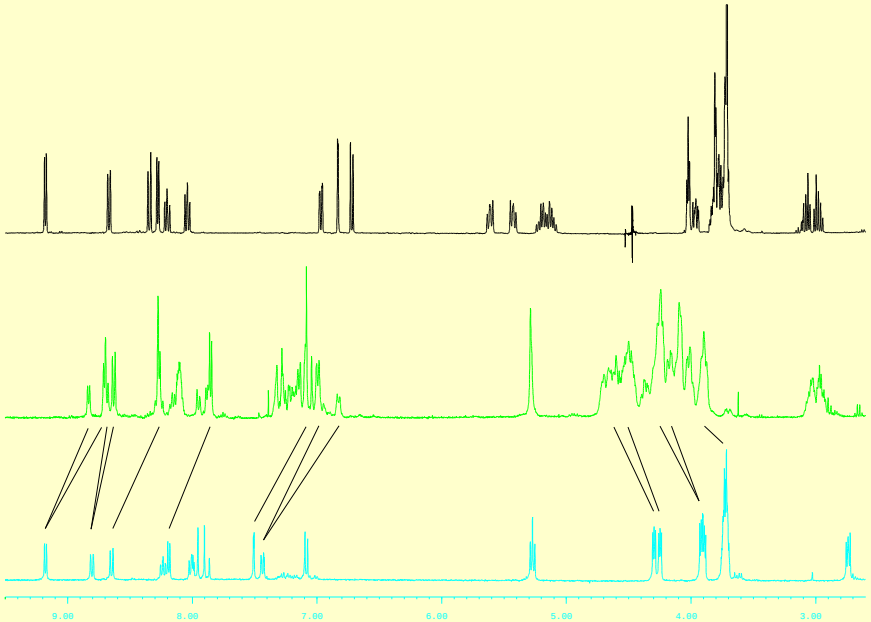
syn-"head-to-tail"





$F_{PCA} = 0.20$

$F_{retro\text{-}PCA} = 0.02$



Russ. Chem. Bull. **1995**, *44*, 2131;
J. Org. Chem., **2003**, *68*, 6115.

Bruker AMX-400, in CD_3CN

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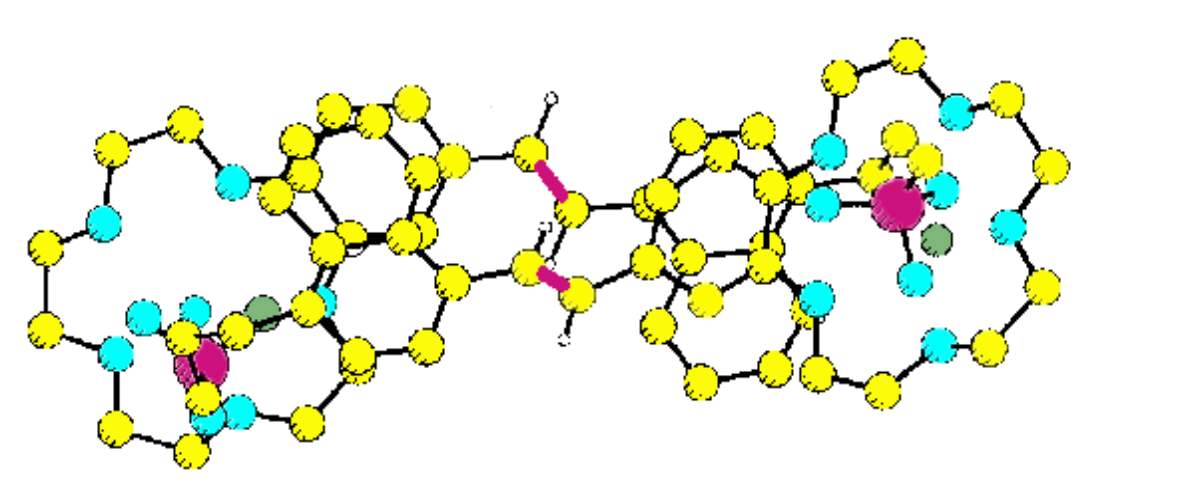
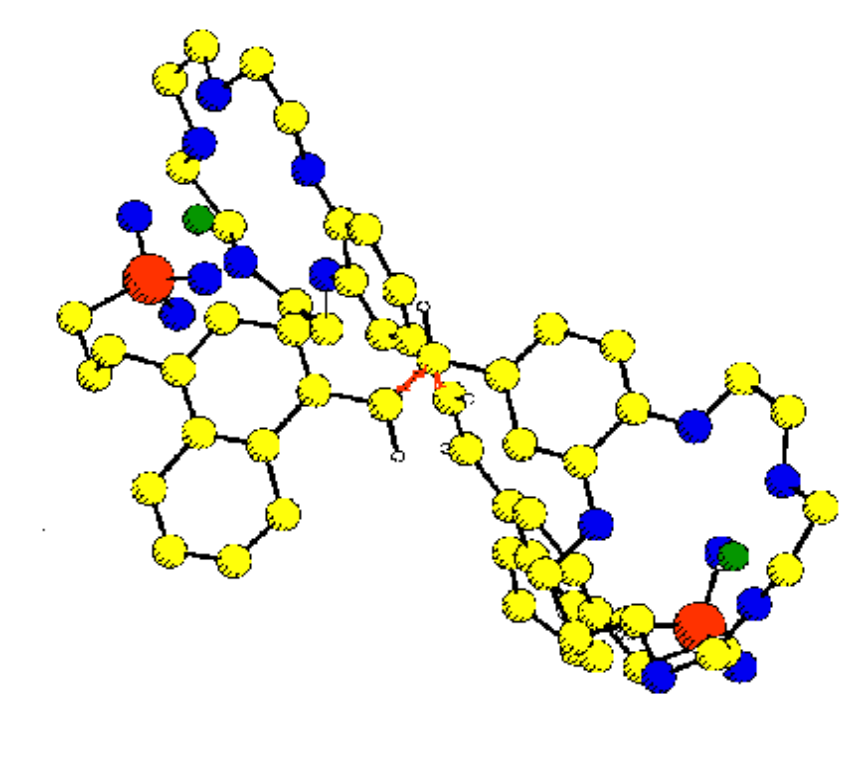
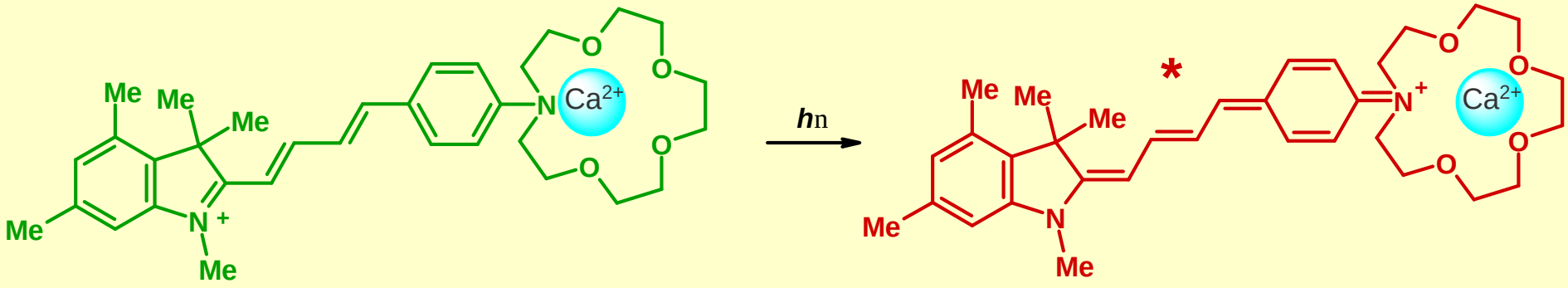
DIMERIC COMPLEXES



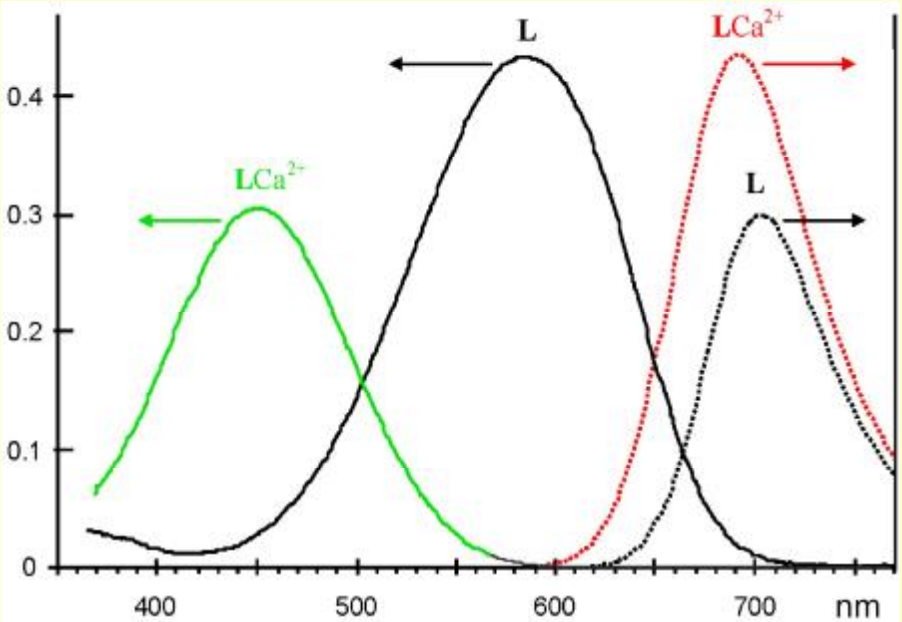


Russ. Chem. Bull. **1998**, *47*, 2117.

Photoswitchable supramolecular device



Photoinduced recoordination reaction



Russ. Chem. Bull. **1999**, *48*, 525;
J. Fluor. **1999**, *9*, 33;
Rusalov M. V., Gromov S. P. et al. *Russ. Chem. Rev.* **2010**, *79*, 1193 (review).

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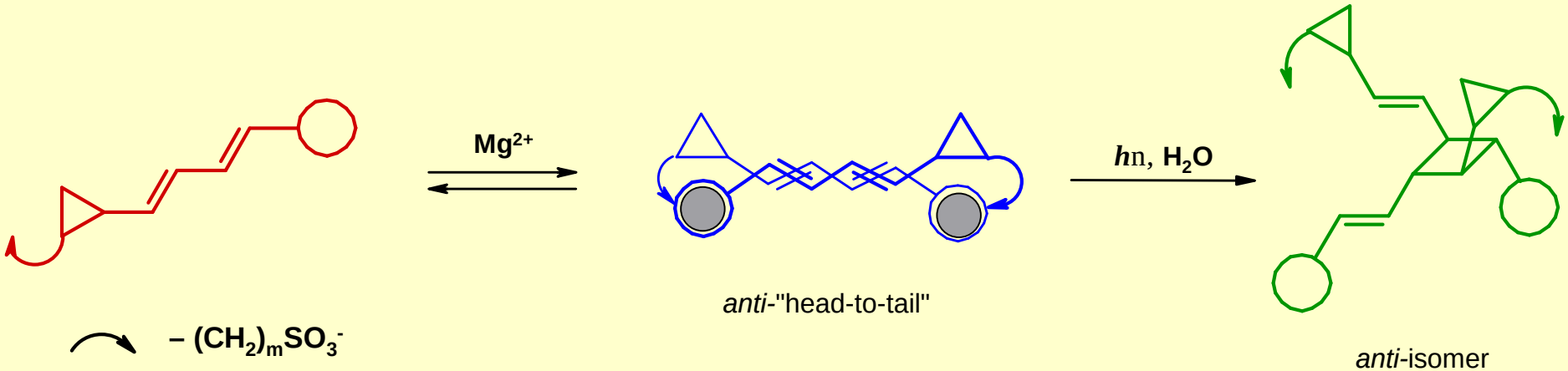
PHOTOCHEMISTRY CENTER

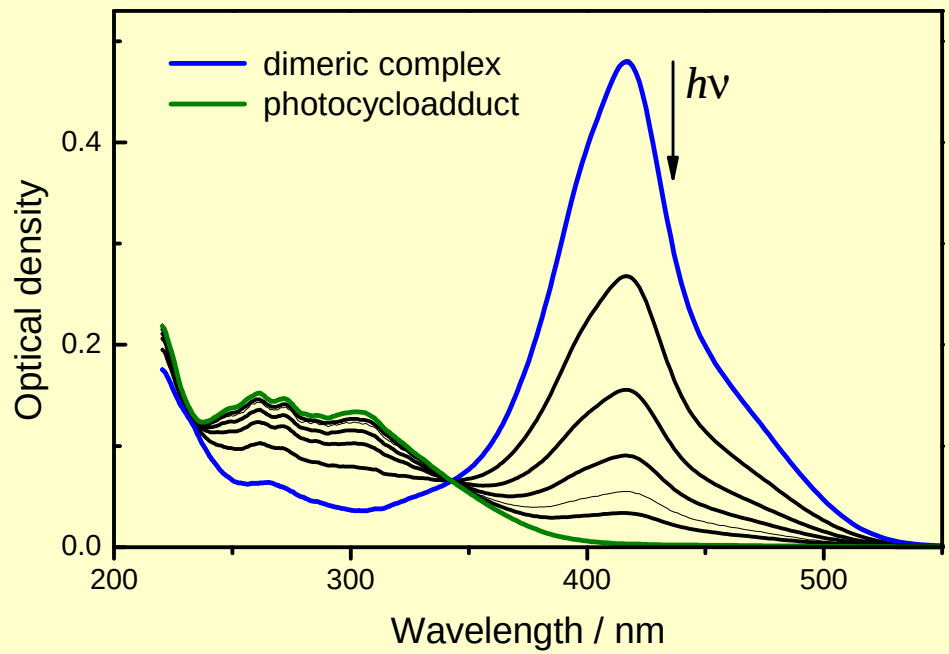
[2+2] PHOTOCYCLOADDITION OF CBD



anti-"head-to-tail"

anti-isomer

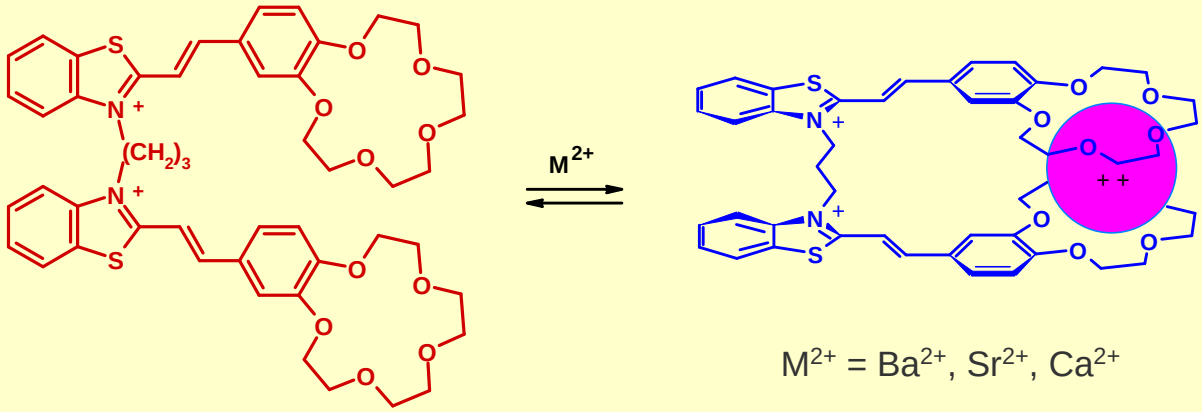
F_{PCA} = 0.35



Helv. Chim. Acta **2002**, *85*, 60;
Photochem. Photobio. Sci., **2011**, *10*, 15.

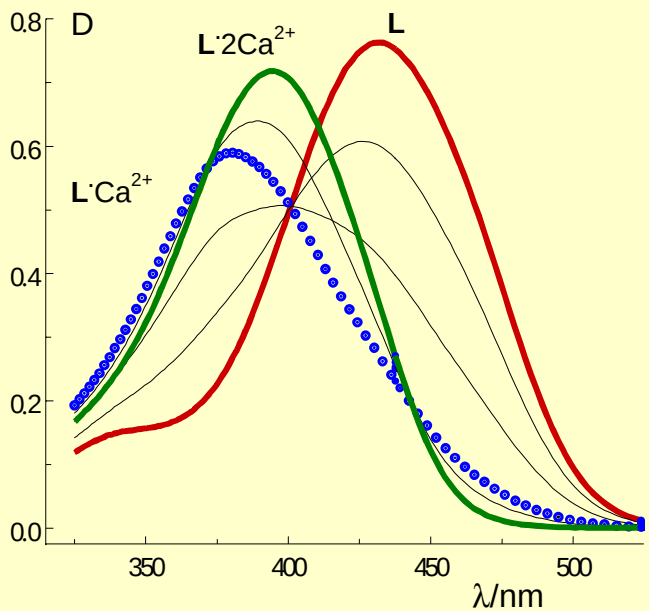
THE RUSSIAN ACADEMY OF SCIENCES
PHOTOCHEMISTRY CENTER

Self-assembly of sandwich complexes



trans,trans-isomer

$M^{2+} = Ba^{2+}, Sr^{2+}, Ca^{2+}$



Complex	$\log K_1$	λ_{LM}, nm	$\lambda_L - \lambda_{LM}$, nm
L·Ba^{2+}	8.0	390	42
Monomer · Ba^{2+}	4.39	402	28

J. Chem. Soc., Perkin Trans. 2. **1999**, 1323;
J. Phys. Chem. A. **1999**, 103, 11188;
RF patent 2389745 **2010**.

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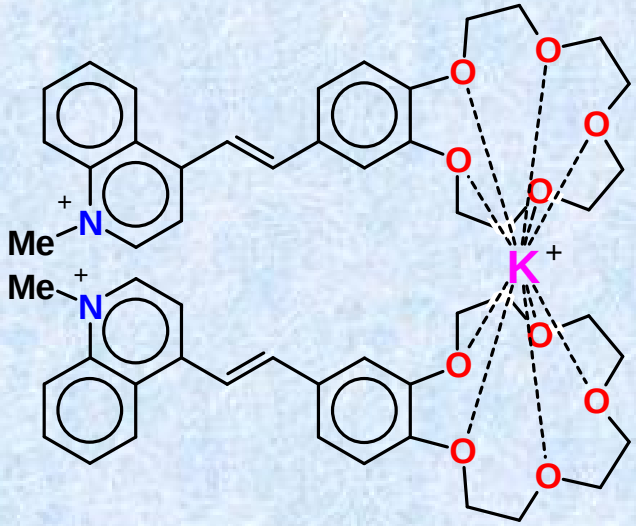
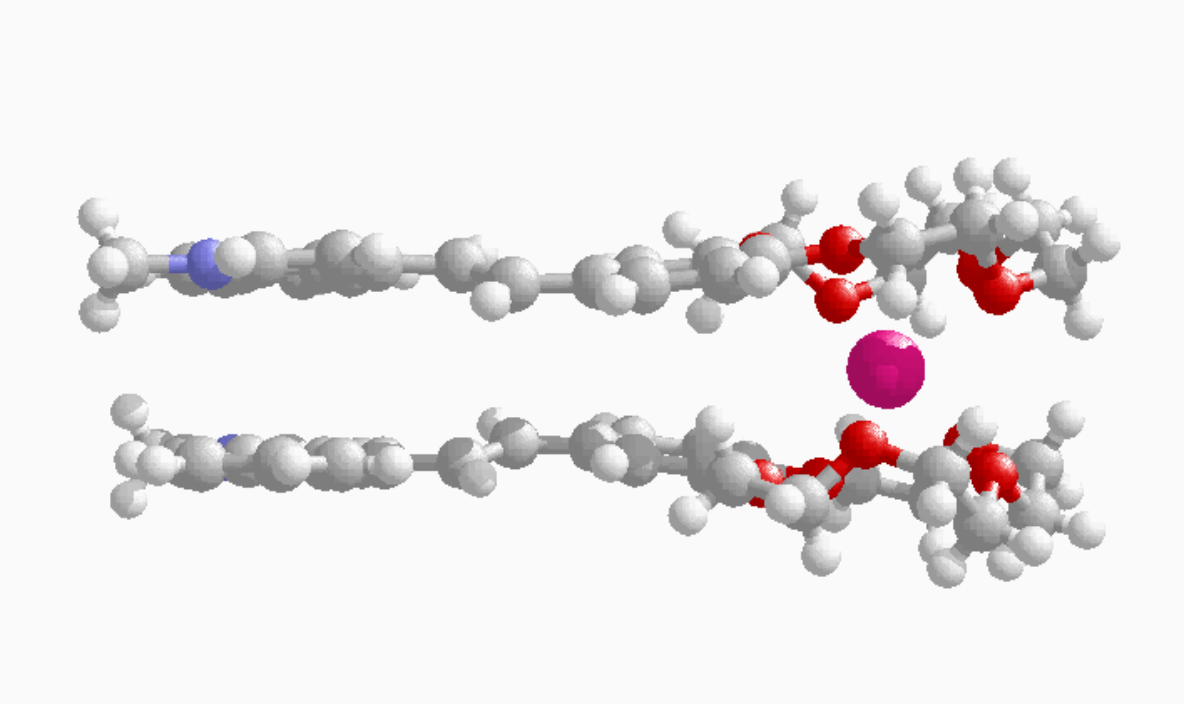
PHOTOCHEMISTRY CENTER

X-ray structure determination of sandwich complex





Crystallography Reports **2003**, *48*, 613.

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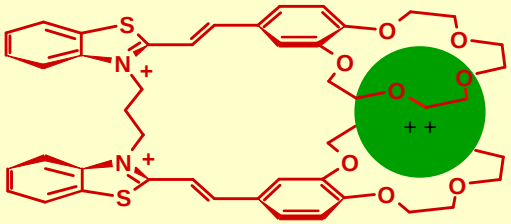
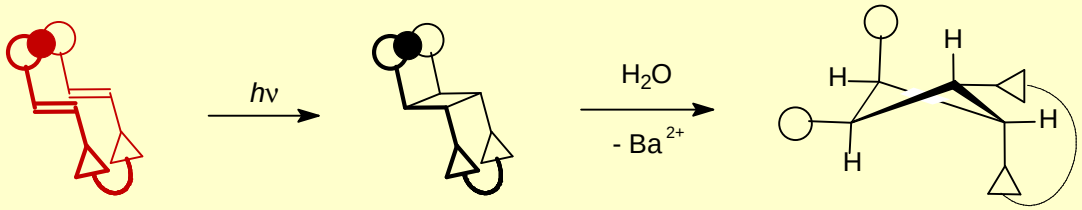
PHOTOCHEMISTRY CENTER

Intramolecular [2+2] photocycloaddition of bisCSD

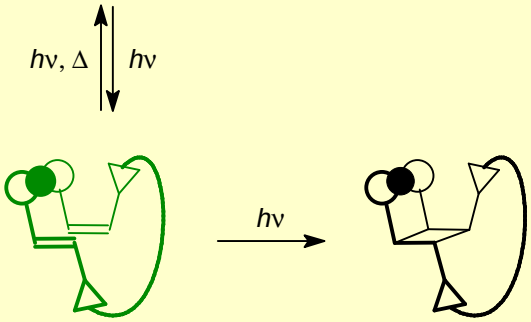


(*trans,trans*-**L**)·Ba^{2+}

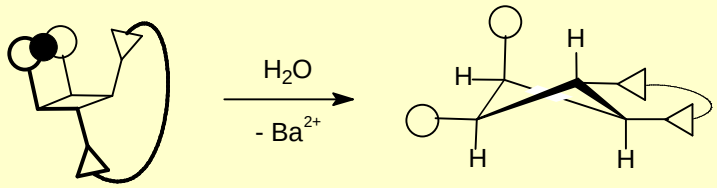


(*trans,trans*-**L**)·Ba^{2+}

syn-isomer



(*trans,cis*-**L**)·Ba^{2+}



$F_{PCA} = 0.001$
$F_{retro\text{-}PCA} = 0.3$

J. Chem. Soc., Perkin Trans. 2. **1999**, 1323.

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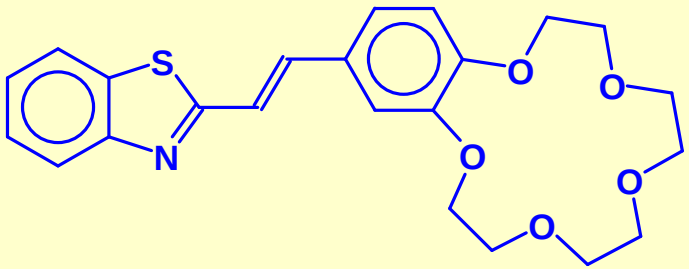
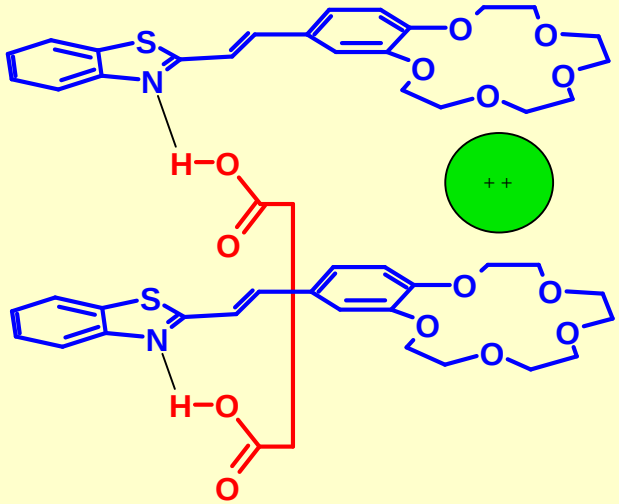
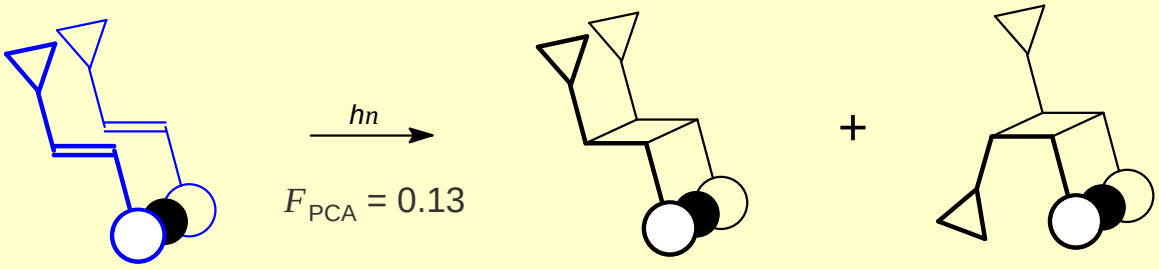
PHOTOCHEMISTRY CENTER

[2+2]-Photocycloaddition of hetarylphenylethenes





$(CH_2)_n(COOH)_2$; n = 1-4



syn-"head-to-head" — log K_{11} = 4.74

syn-isomer — log K_{11} = 7.69

log K_{11} = 7.21

● - Ba^{2+}, Sr^{2+}

Russ. Chem. Bull. **2005**, *54*, 1569.

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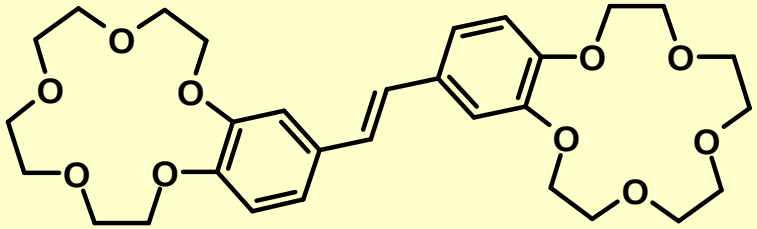
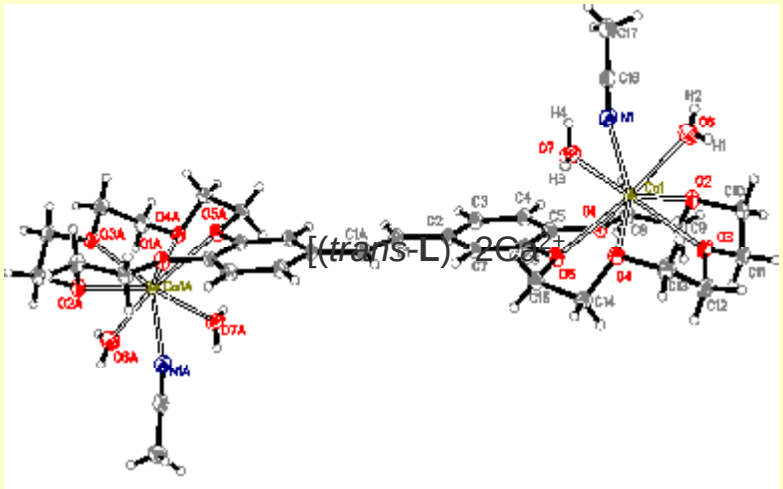
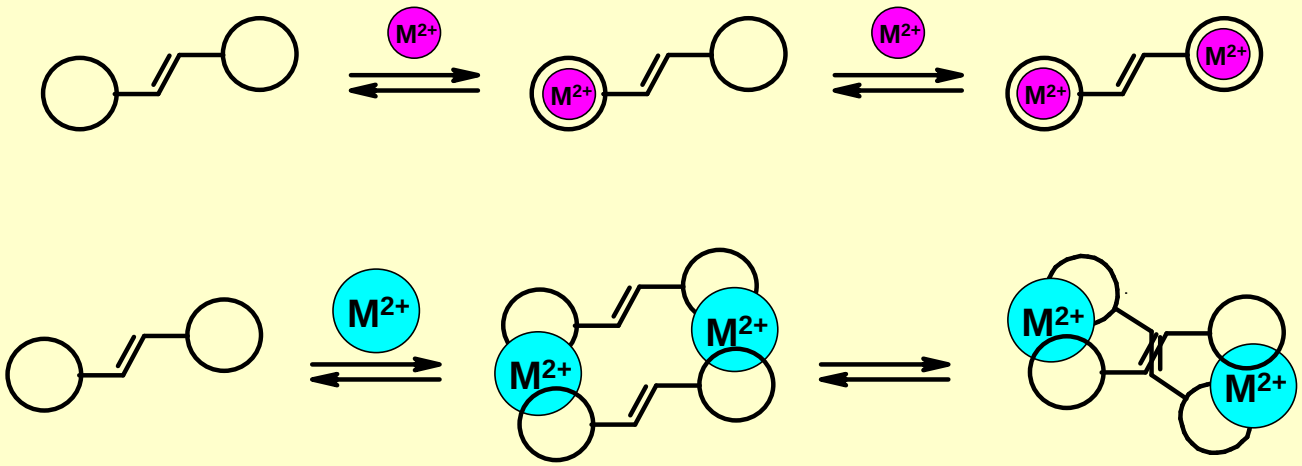
Complex formation of bisCS



trans-**L**





M^{2+} = Mg^{2+}, Ca^{2+} M^{2+} = Sr^{2+}, Ba^{2+}

New. J. Chem. **2011**, *35*, 724.

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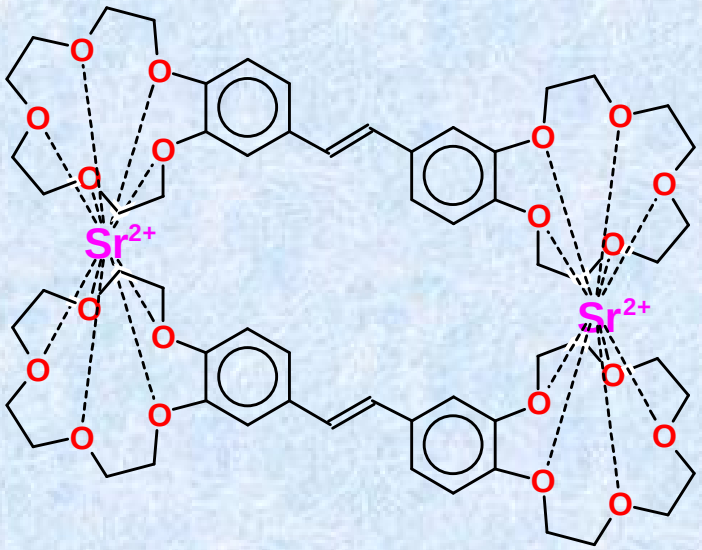
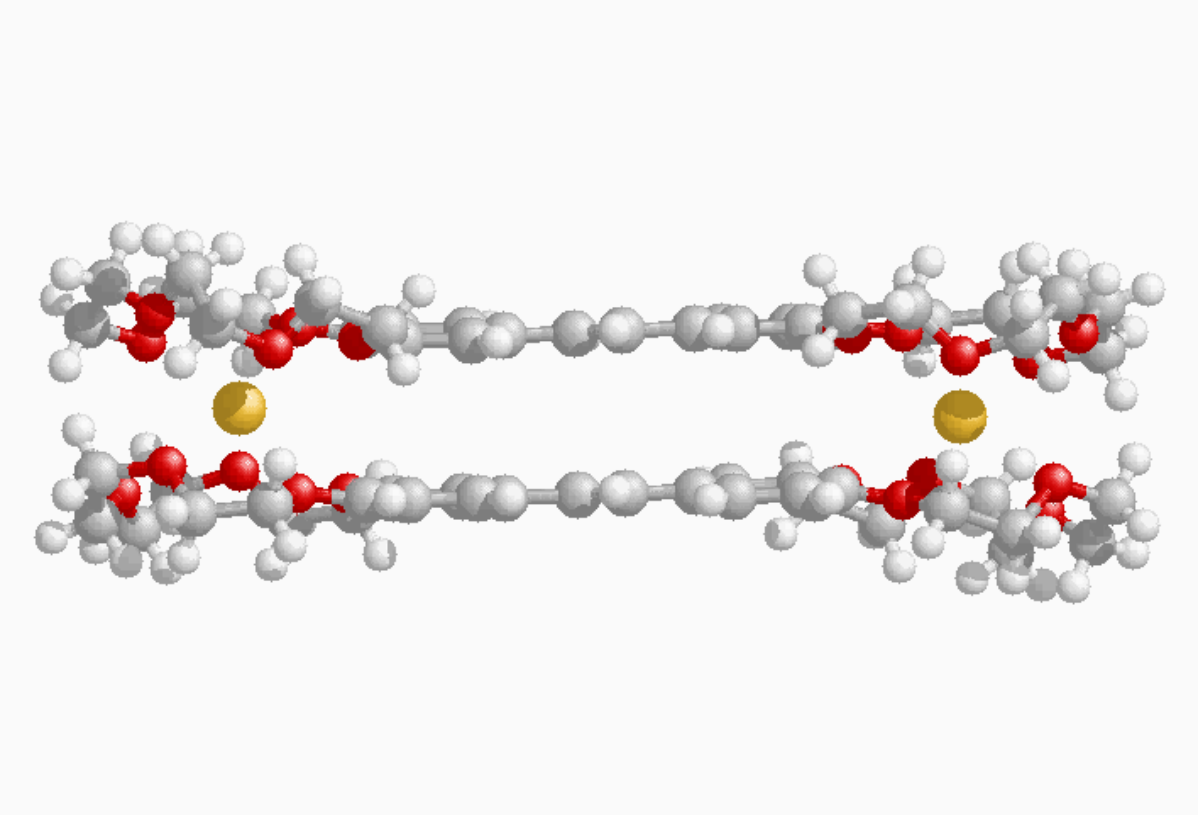
X-ray structure determination of double sandwich complex



2(*trans*-**L**) · $2Sr^{2+}$



New. J. Chem. **2011**, *35*, 724.

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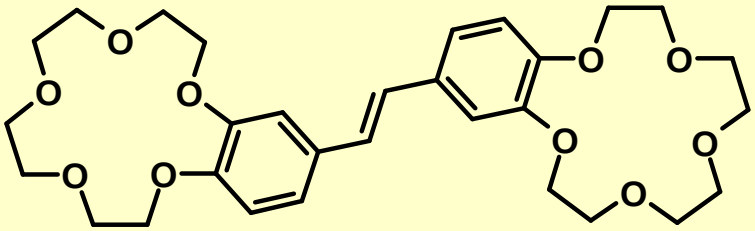
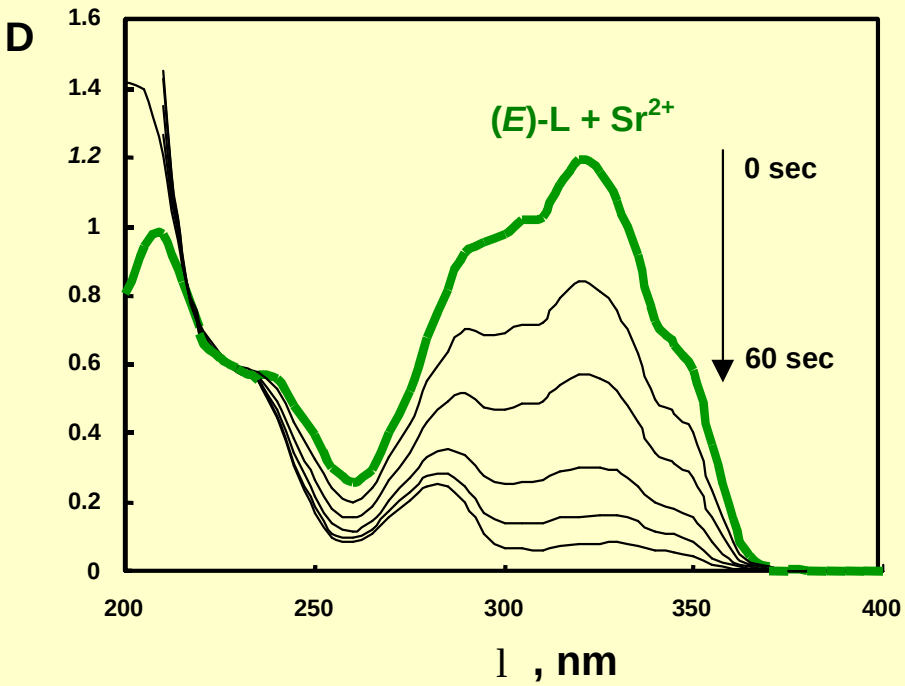
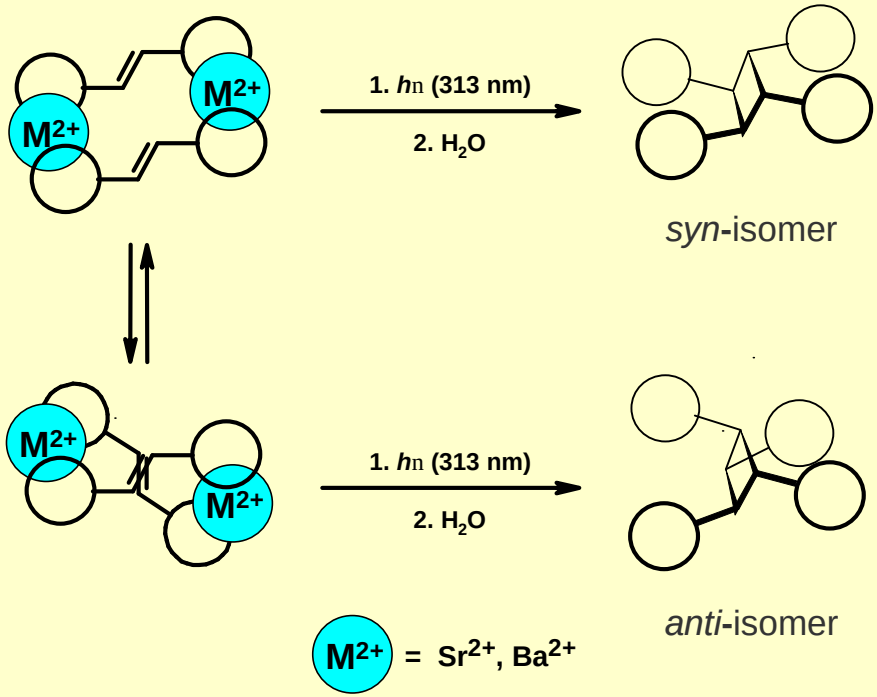
[2+2] Photocycloaddition of bisCS



trans-**L**





New. J. Chem. **2011**, *35*, 724.

THE RUSSIAN ACADEMY OF SCIENCES
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Self-assembly
of photoswitchable supramolecular devices
with participation of hydrogen bonds

Part II

THE RUSSIAN ACADEMY OF SCIENCES
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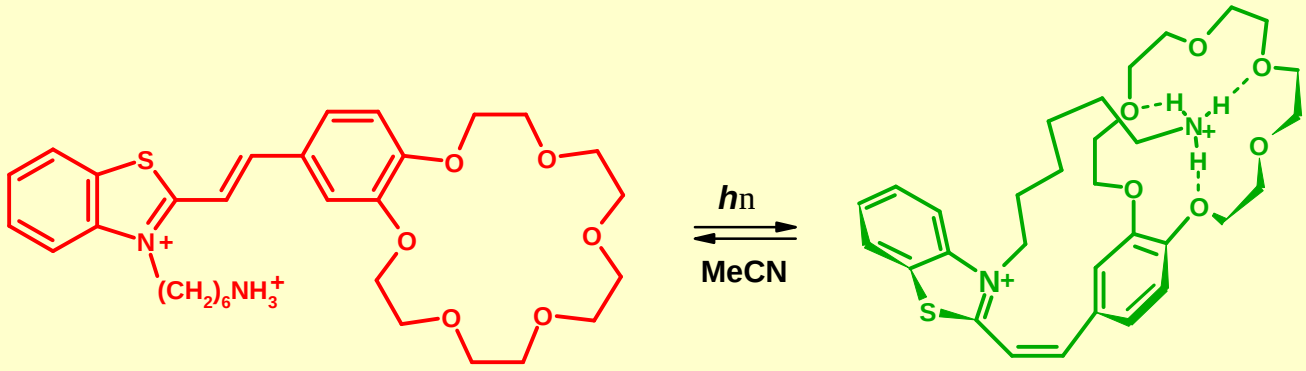
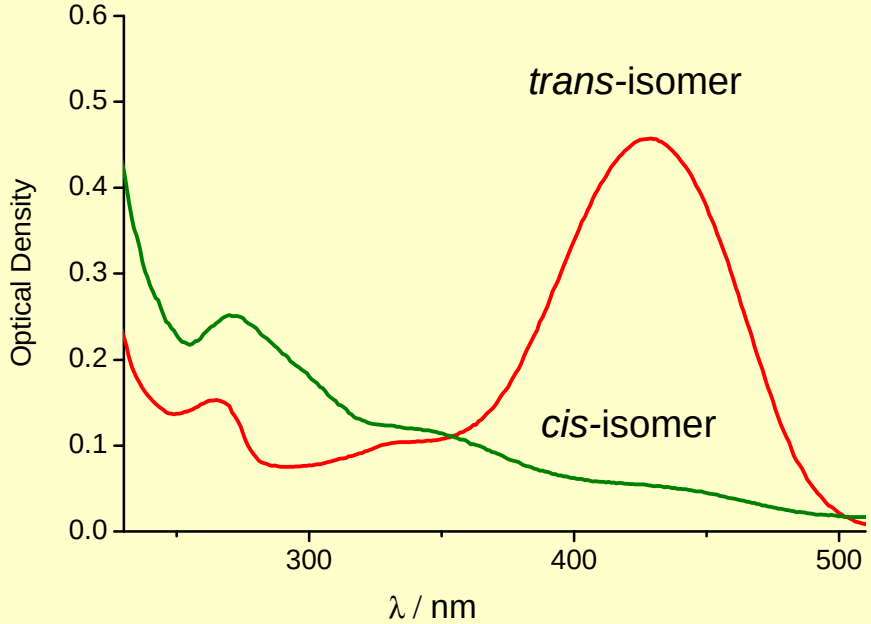
Intramolecular complexation of *cis*-isomers



trans-isomer

cation-“capped” complex of *cis*-isomer



Mendeleev Commun. **2007**,*17*, 264;
J. Mol. Structure. **2009**, *935*, 136.

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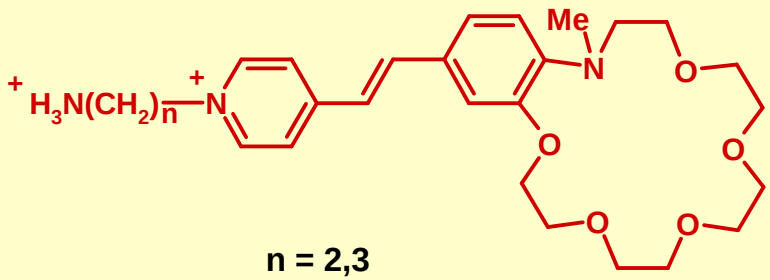
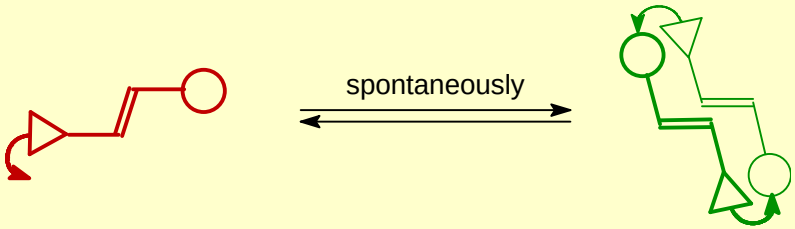
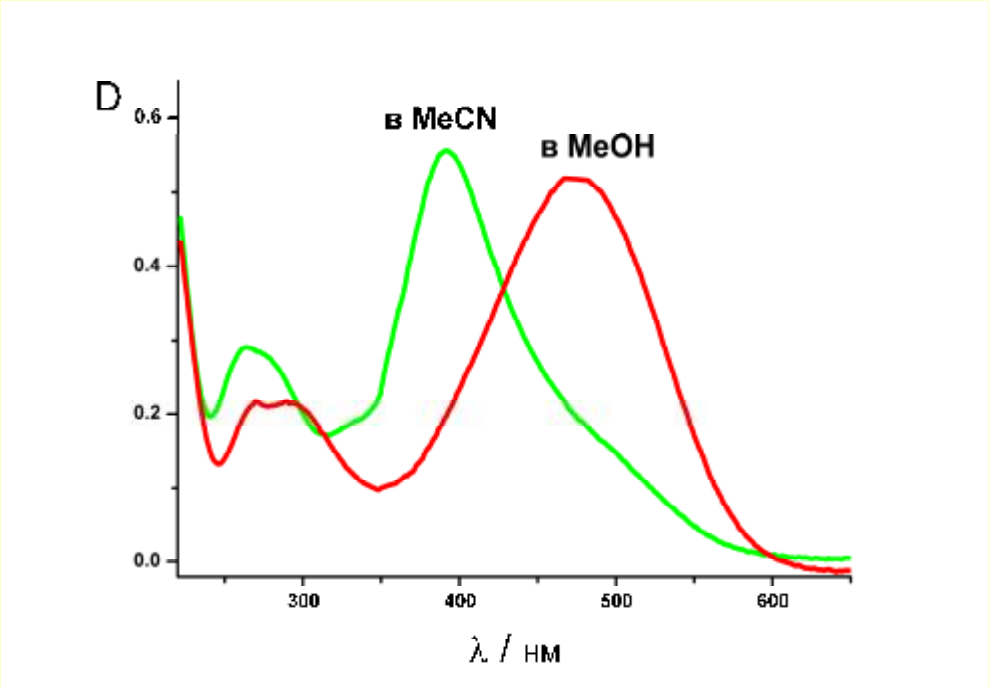
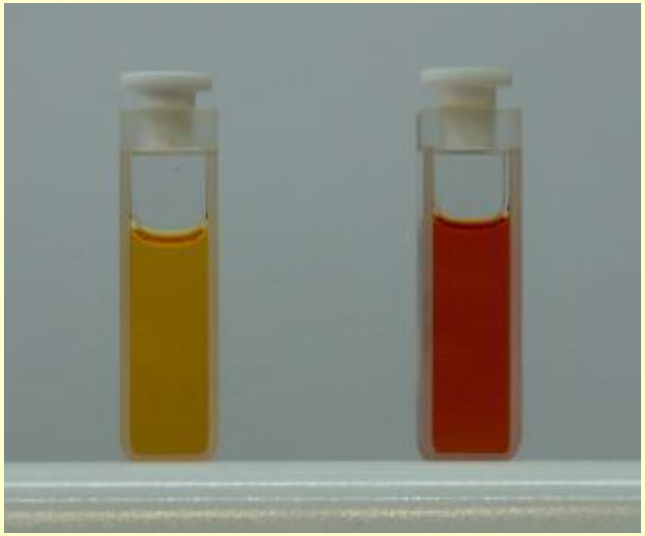
PHOTOCHEMISTRY CENTER

Dimerization of CSD









in MeCN in MeOH

RF patent 2278134 **2006**;
J. Org. Chem. **2014**, *79*, 11416;
J. Phys. Chem. A **2015**, *119*, 13025.

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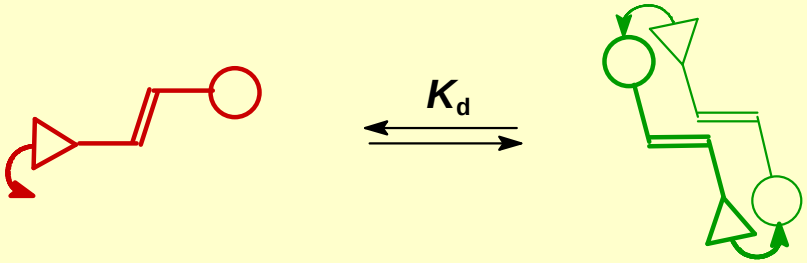
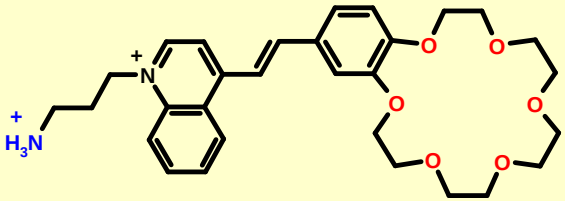
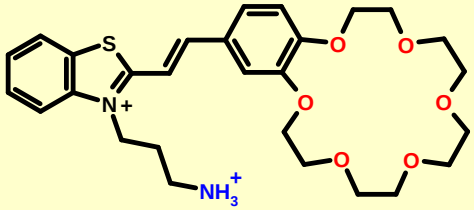
DIMERIZATION



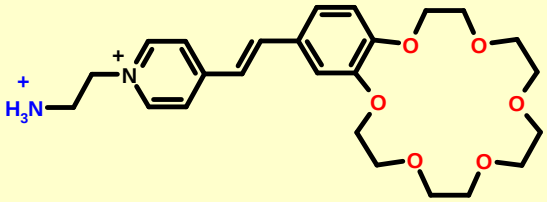
log K_d



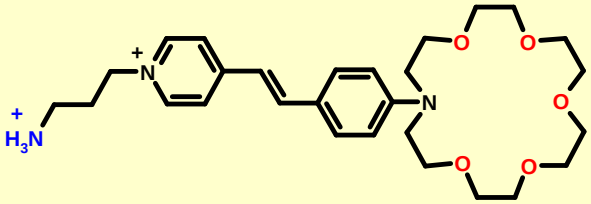
8.03



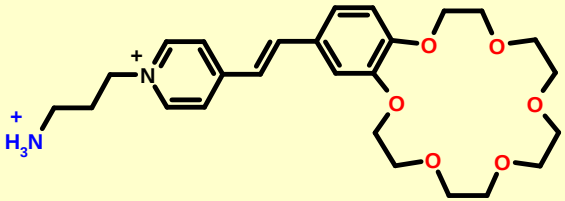
5.87



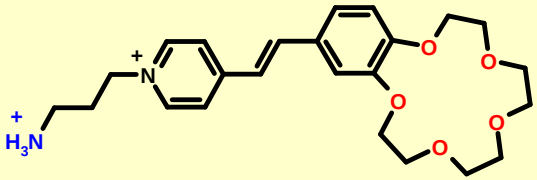
7.90



3.61



7.12



2.44

in CD_3CN

Russ. Chem. Bull. **2009**, *58*, 1211;
J. Org. Chem. **2014**, *79*, 11416;
J. Phys. Chem. A **2015**, *119*, 13025.

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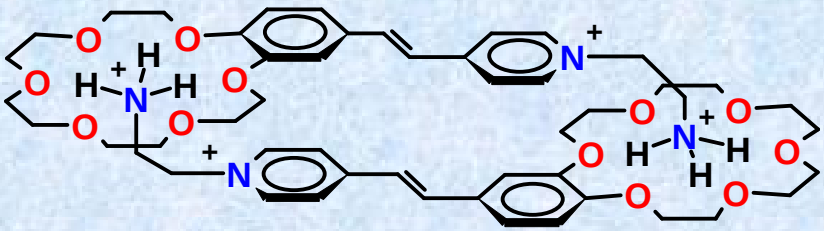
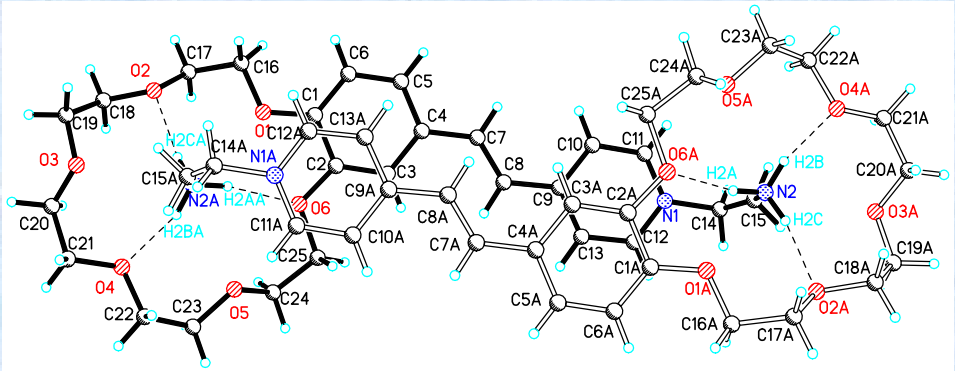
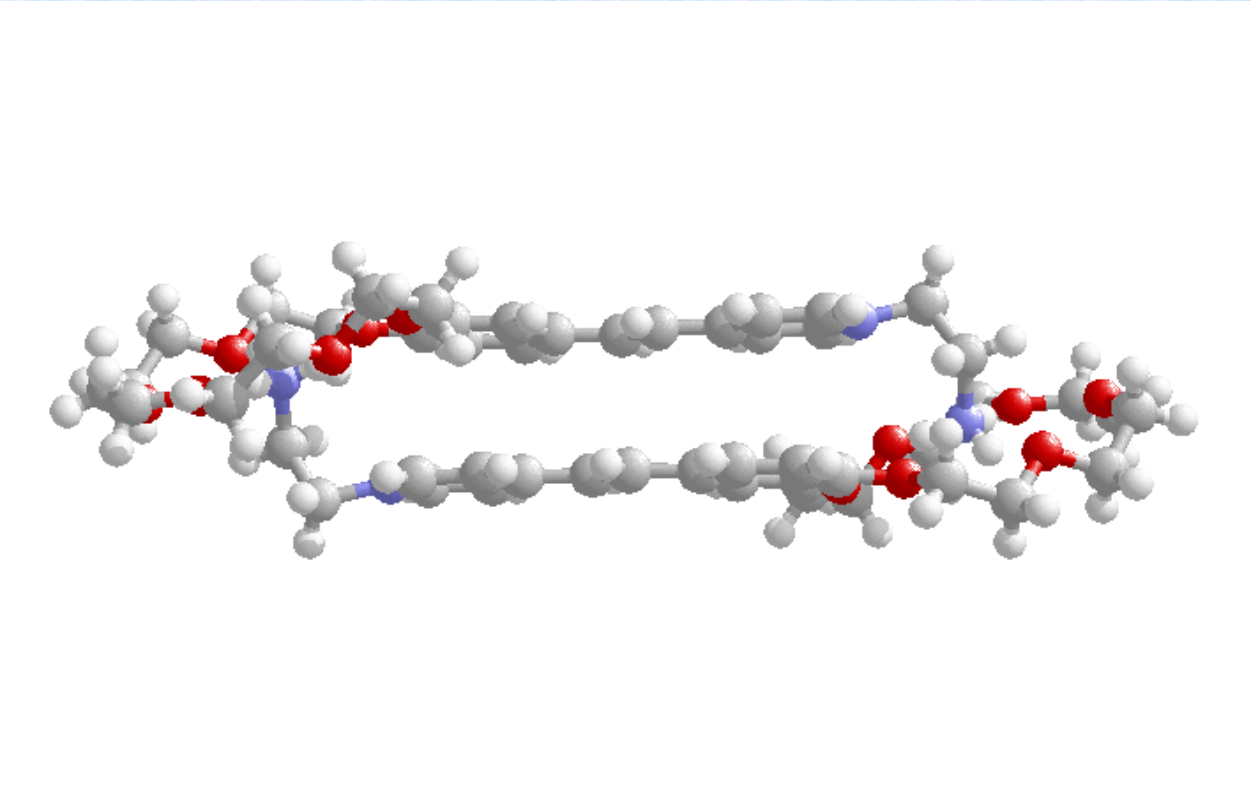
X-ray structure determination of dimeric compex



syn-"head-to-tail" dimeric complex





Russ. Chem. Bull. **2009**, *58*, 1211;
J. Org. Chem. **2014**, *79*, 11416.

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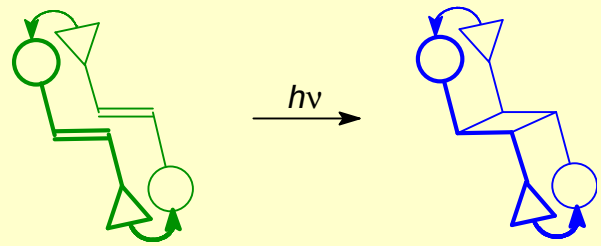
PHOTOCHEMISTRY CENTER

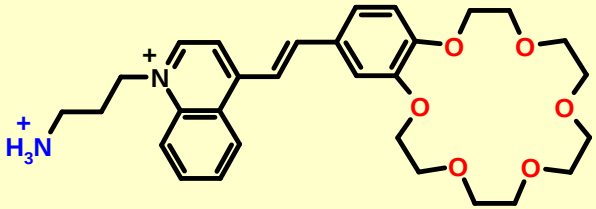
[2+2] Photocycloaddition of CSD



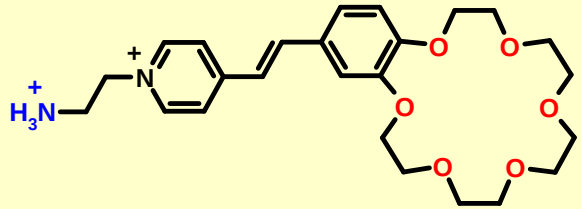
F_{PCA} up to 0.38

syn-"head-to-tail" *syn*-isomer

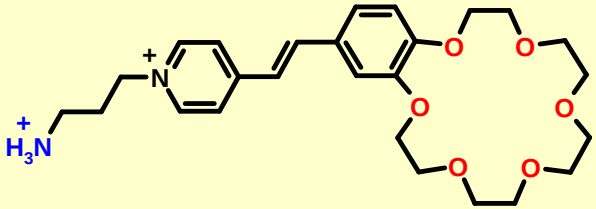
Yield, %



100



33



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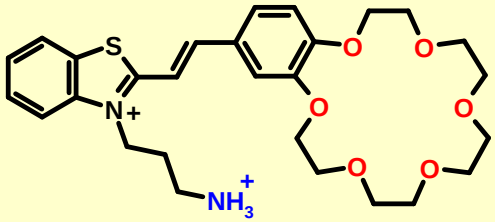

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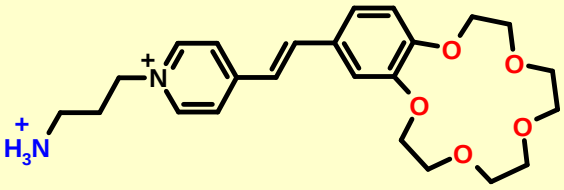
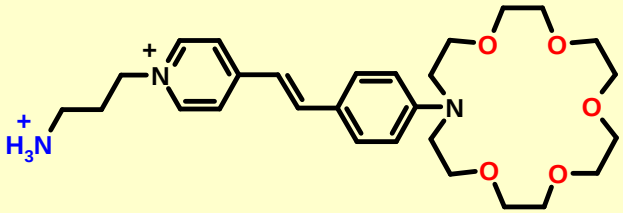


40



0

In MeCN, irradiation time, 4 h

RF patent 2278134 **2006**;
Russ. Chem. Bull. **2009**, *58*, 1211;
J. Org. Chem. **2014**, *79*, 11416;
J. Phys. Chem. A **2015**, *119*, 13025.

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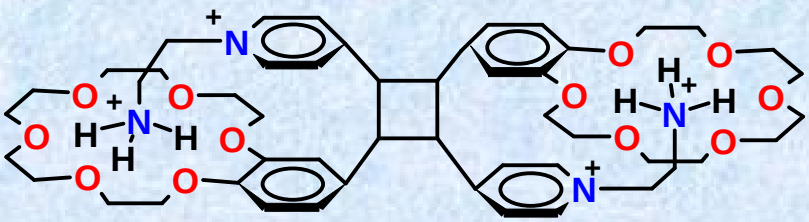
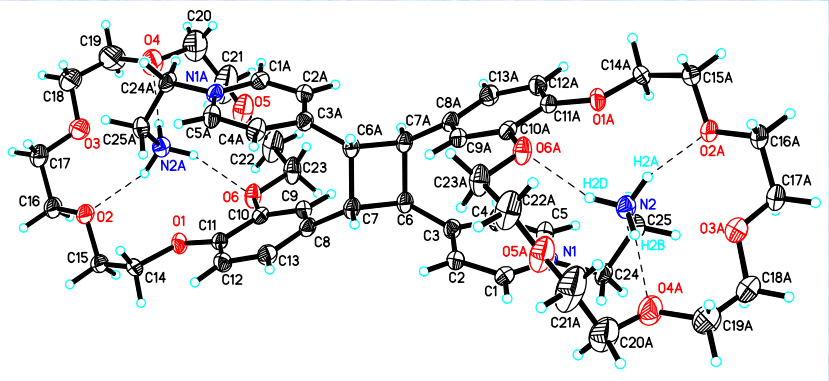
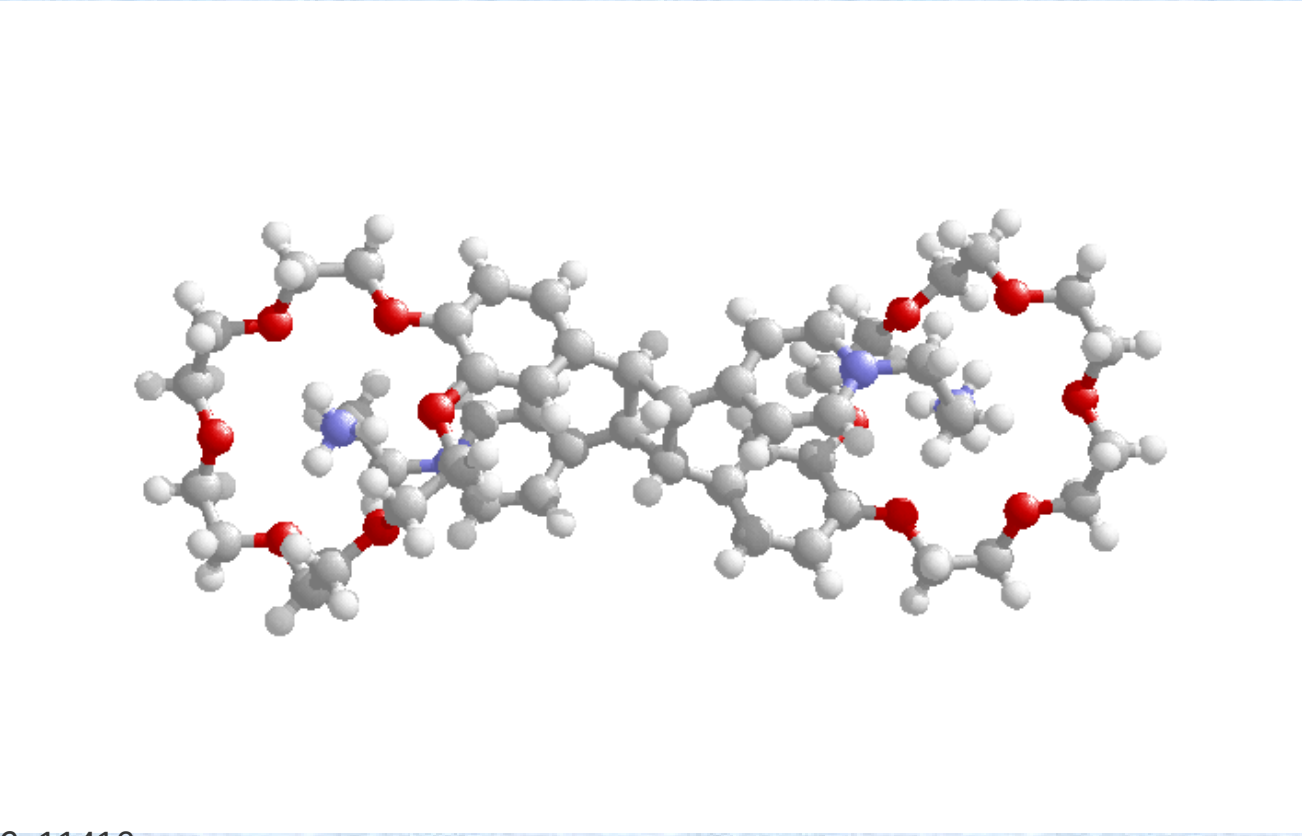
X-ray structure determination of cyclobutane



syn-cyclobutane





J. Org. Chem. **2014**, *79*, 11416.

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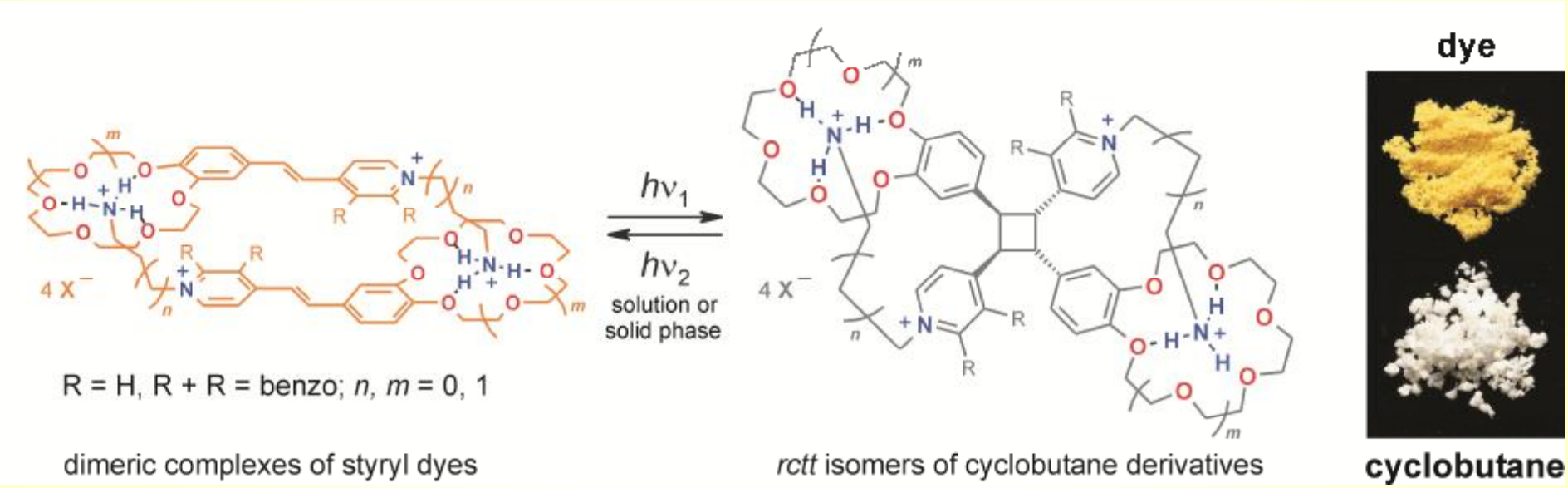
PHOTOCHEMISTRY CENTER

Supramolecular photoswitches based on ammonioalkyl derivatives of crown-ether styryl dyes



(X = ClO_4;
R = H, n = m = 1

Found property provides grounds for believing that the crystals of these photoactive supramolecular systems could be used for data recording and storage.

J. Org. Chem. **2014**, *79*, 11416;
J. Phys. Chem. A **2015**, *119*, 13025.

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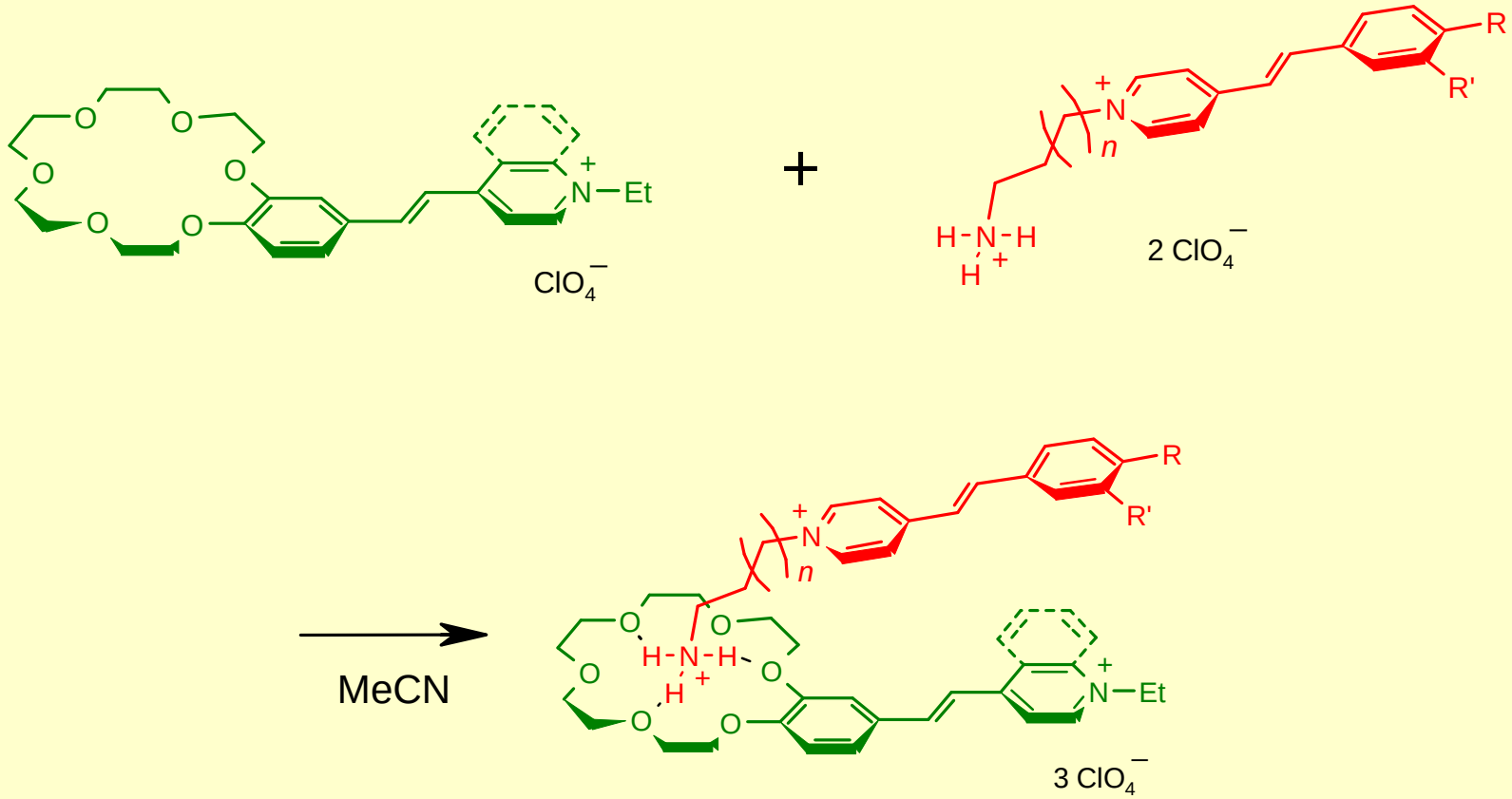
Formation of pseudodimeric complexes



R, R' = H, OMe, SMe, NMe_2, NO_2, Cl

n = 0, 1, 3

log *K* = 3.1 - 3.7

Mendeleev Commun., **2007**, *17*, 29;
Russ. Chem. Bull. **2009**, *58*, 1955;
New. J. Chem. **2016**, *40*, 7542.

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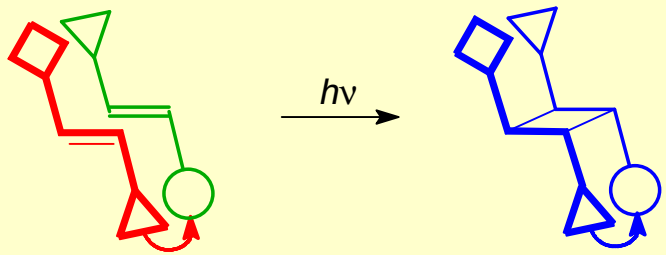
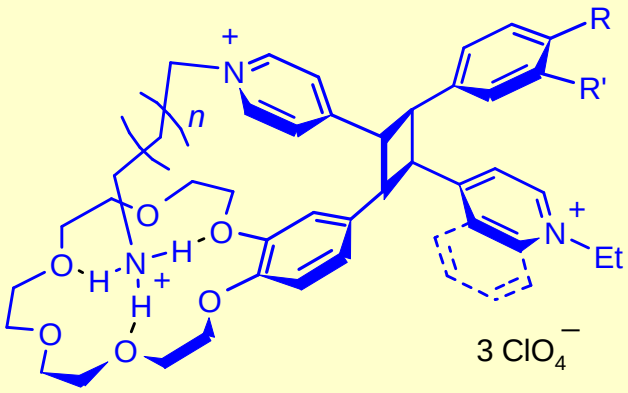
[2+2] cross-Photocycloaddition

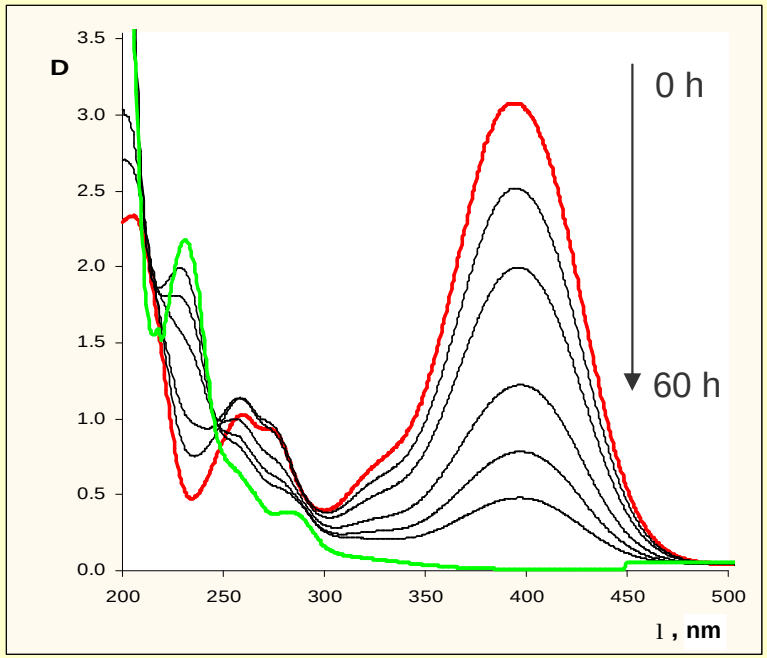


one of the 38 possible isomers

syn-"head-to-tail"



rctt isomers



Mendeleev Commun., **2007**, *17*, 29;
Russ. Chem. Bull. **2009**, *58*, 1955;
RF patent 2383571 **2010;**
New. J. Chem. **2016**, *40*, 7542.

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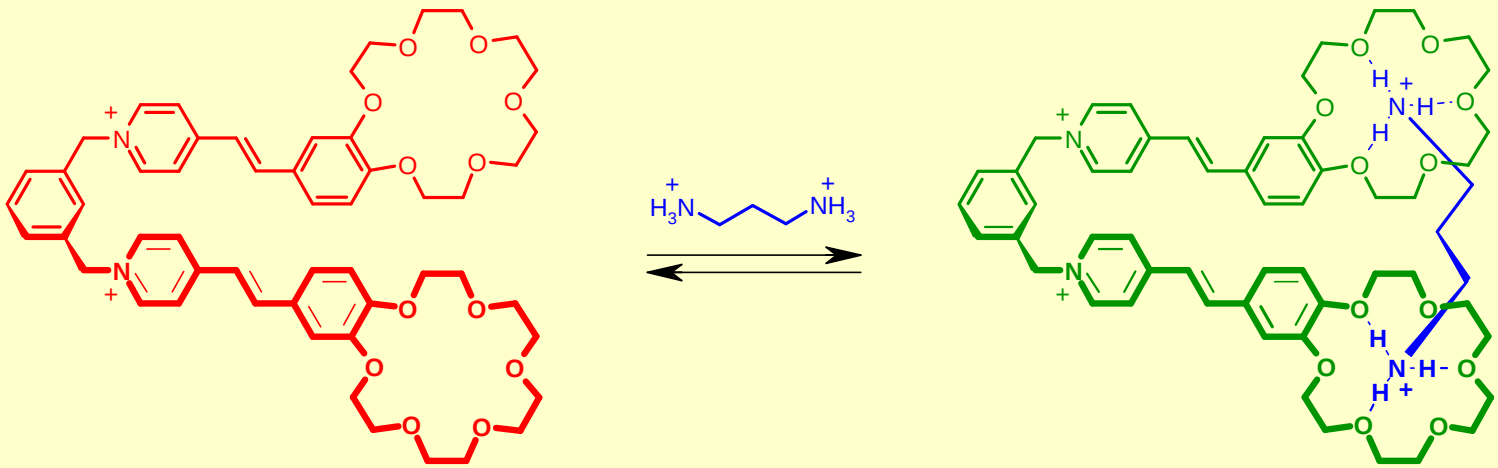
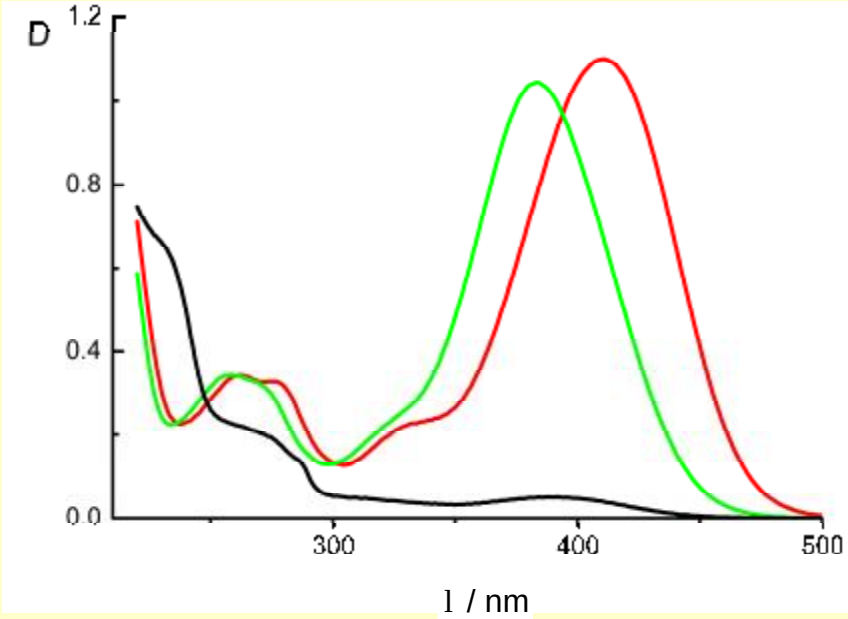
FORMATION OF PSEUDOSANDWICH COMPLEXES





Mendeleev Commun. **2005**, *15*, 173.

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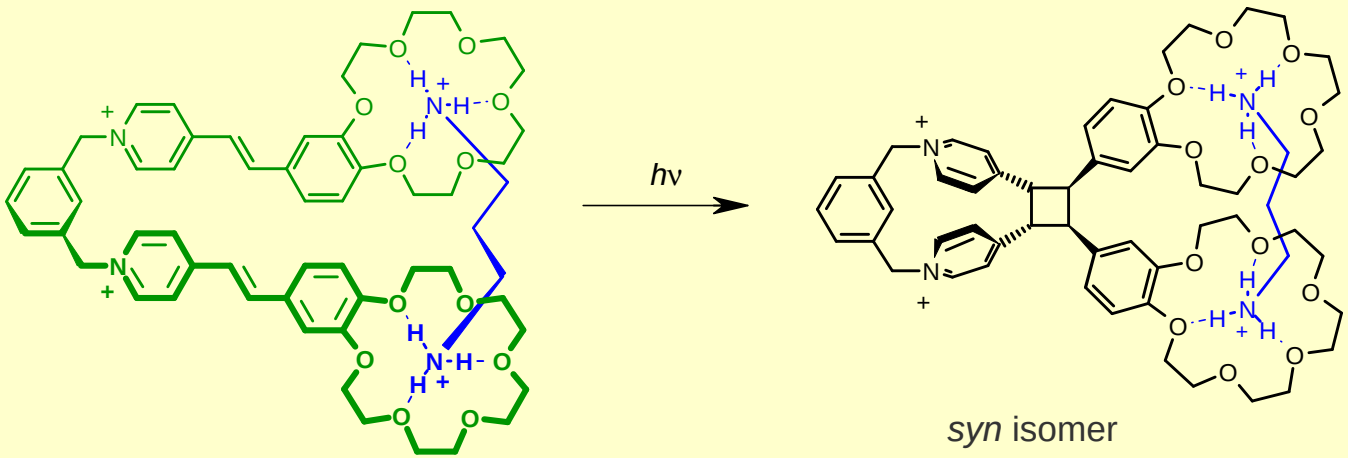
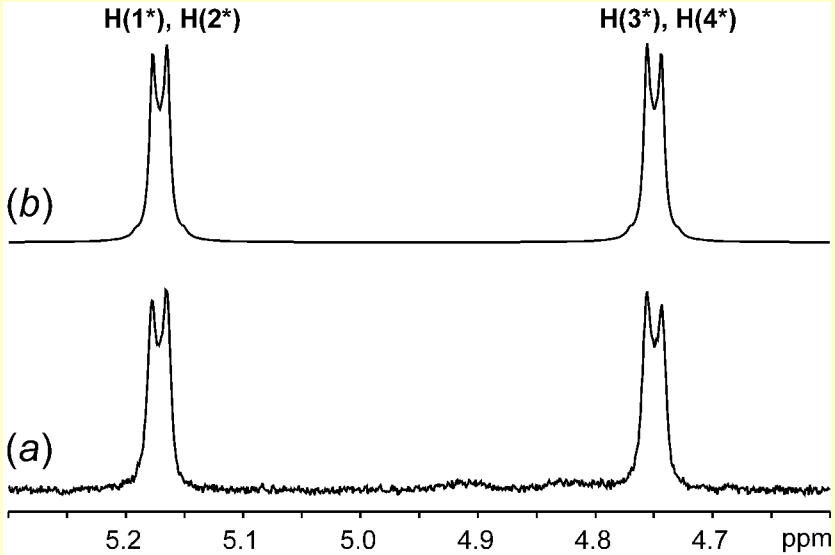
Intramolecular [2+2] photocycloaddition of bisCSD





(*a*) ^{1}H NMR spectrum of the cyclobutane protons and (*b*) its best fit to an AA'BB' spin system.

Mendeleev Commun. **2005**, *15*, 173.

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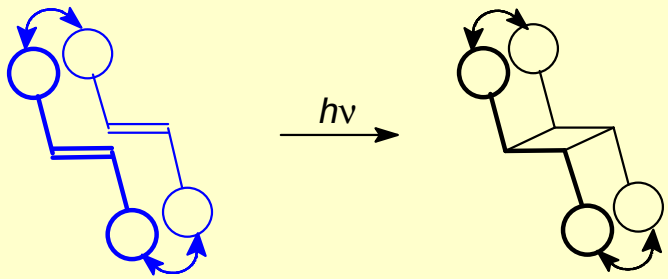
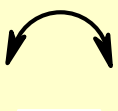
PHOTOCHEMISTRY CENTER

Formation of bispseudosandwich complexes and [2+2] Photocycloaddition

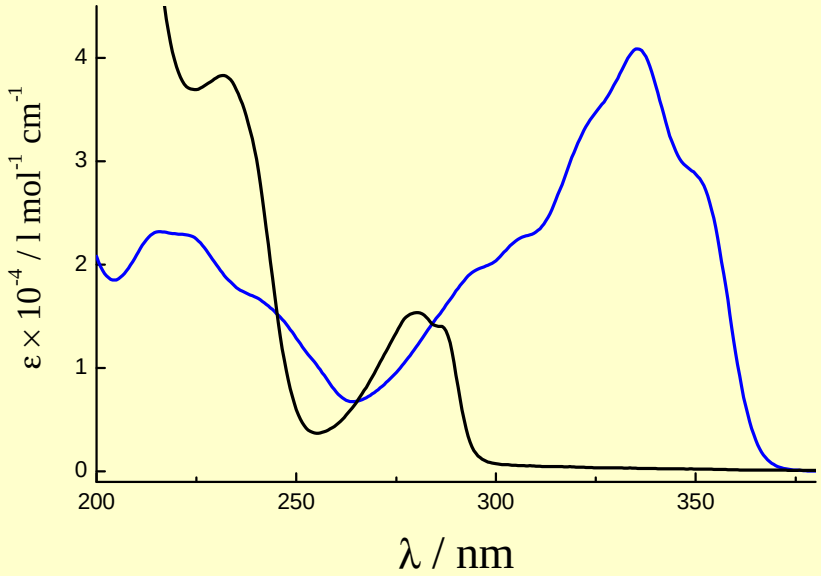


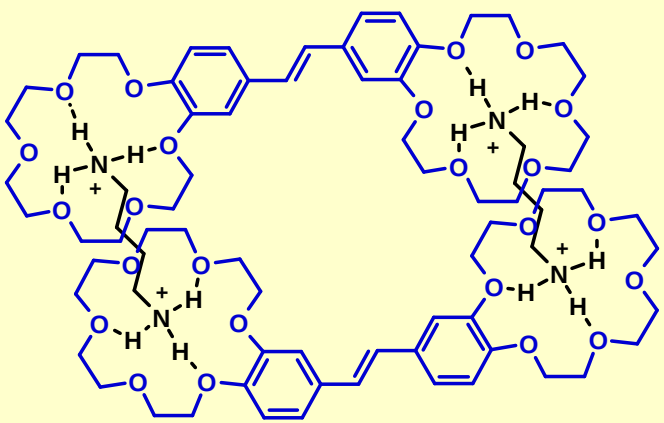
syn-complex *syn-isomer*

F_{PCA} up to 0.27

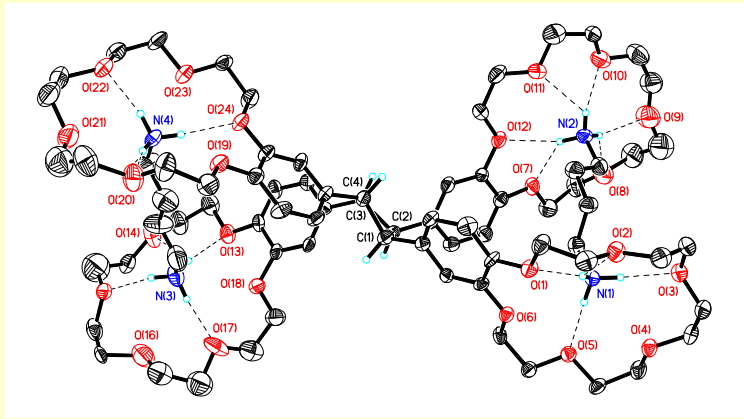


– $H_3N^+(CH_2)_nNH_3^+$ 2 ClO_4^-, n = 2-10, 12





hν



syn-isomer

Russ. Chem. Bull. **2009**, *58*, 108;
New. J. Chem. **2011**, *35*, 724;
J. Photochem. Photobiol. A. **2017**, *340*, 80;

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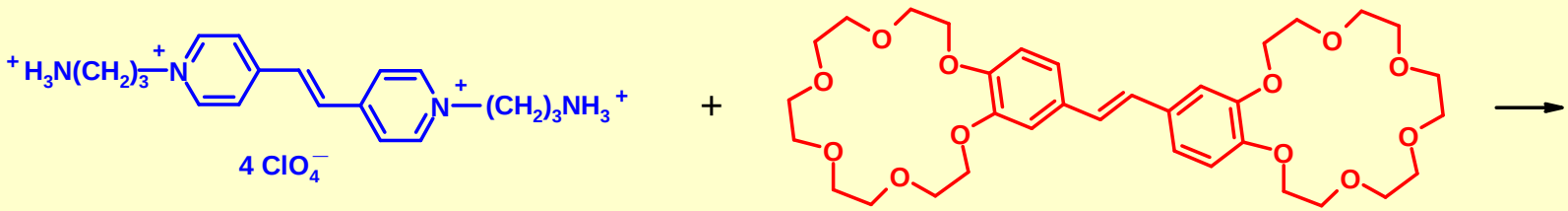
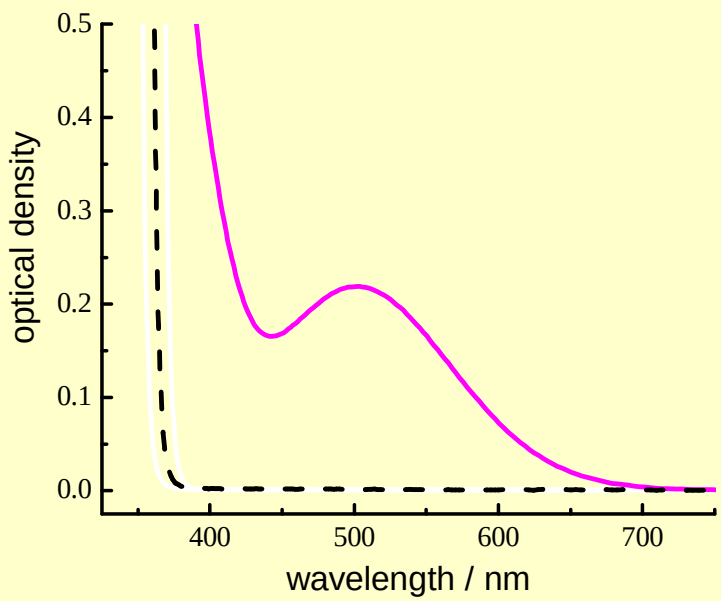
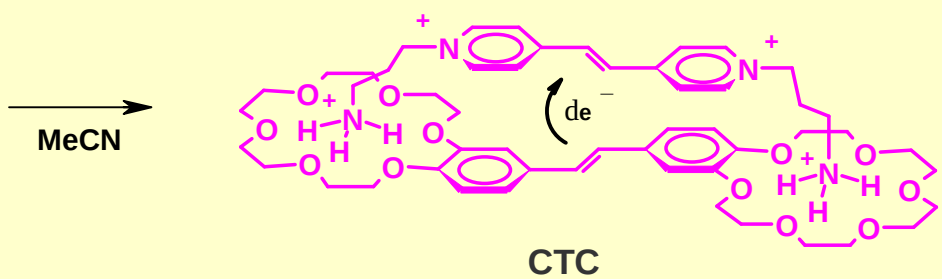
Formation of Charge Transfer Complex of bisCS







log K = 9.08

Org. Lett. **1999**, *1*, 1697 ;
New. J. Chem. **2005**, *29*, 881;
J. Org. Chem. **2011**, *76*, 6768;
Photochem. Photobiol. Sci. **2017**, *16*, 1801.

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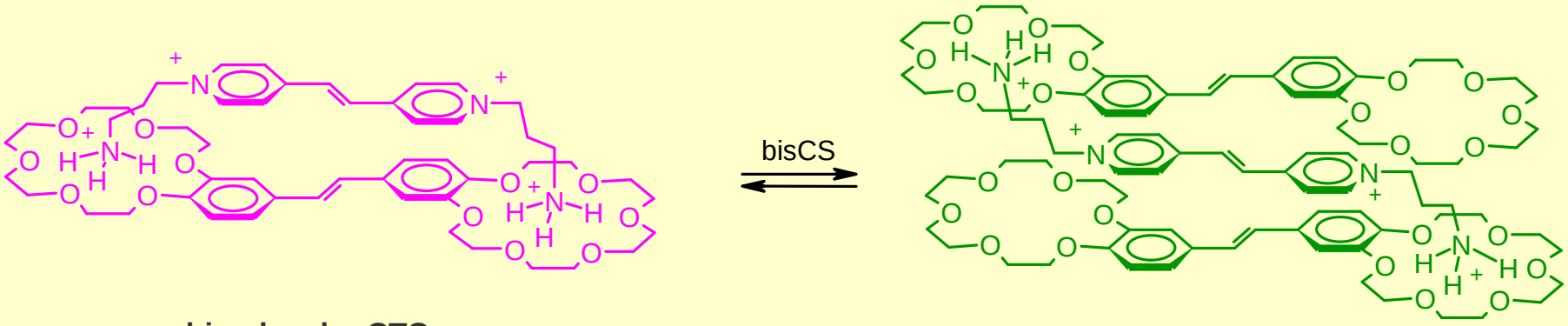
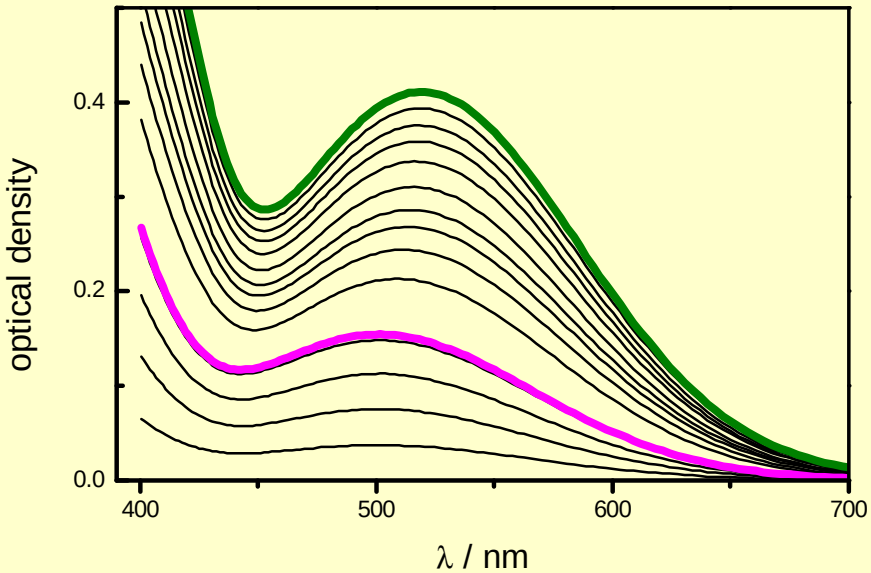
Formation of Charge Transfer Complexes



bimolecular CTC

termolecular CTC



log K = 3.27

J. Phys. Chem. A **2002**, *106*, 2020;
New. J. Chem. **2005**, *29*, 881;
J. Org. Chem. **2011**, *76*, 6768.

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Self-assembly

of photocontrolled supramolecular machines

Part III

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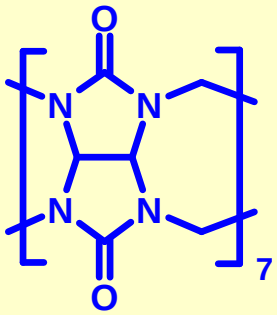
PSEUDOROTAXANE COMPLEXES OF CUCURBITURILS

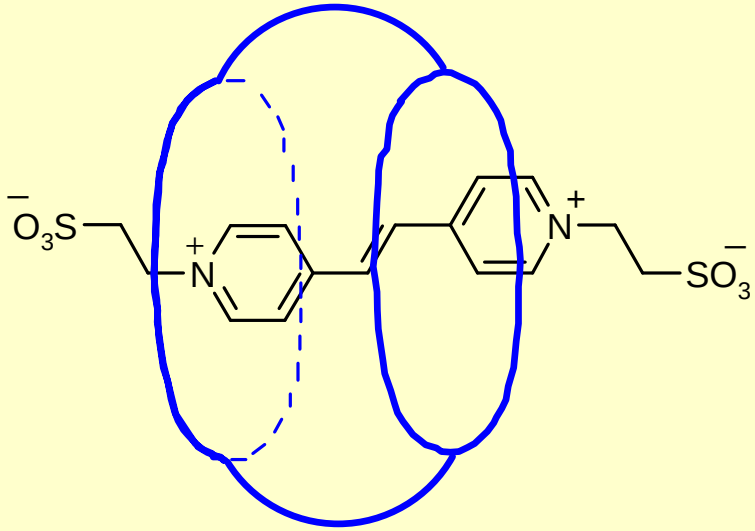


CB[7]

cucurbit[n]urils

trans-**V**

log*K* = 3.5



trans-**V**@CB[7]

Russian Nanotechnologies **2007**, *2*, 56;
J. Mol. Struct. **2011**, *989*, 114;
Chem. Phys. Lett. **2014**, *610-611*, 91;
J. Photochem. Photobio. A. **2018**, *353*, 34.

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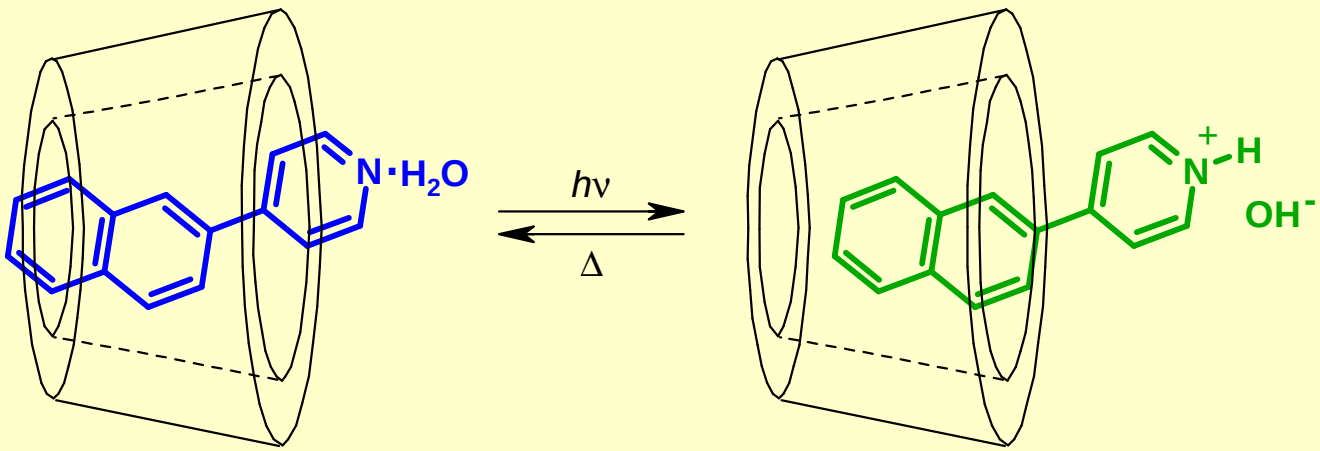
PHOTOCONTROLLED SUPRAMOLECULAR MACHINE



1·H_2O@HP-β-CD

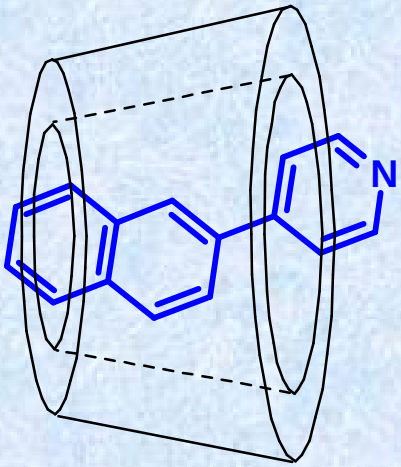
log*K* = 1.9

1H^+OH^-@HP-β-CD

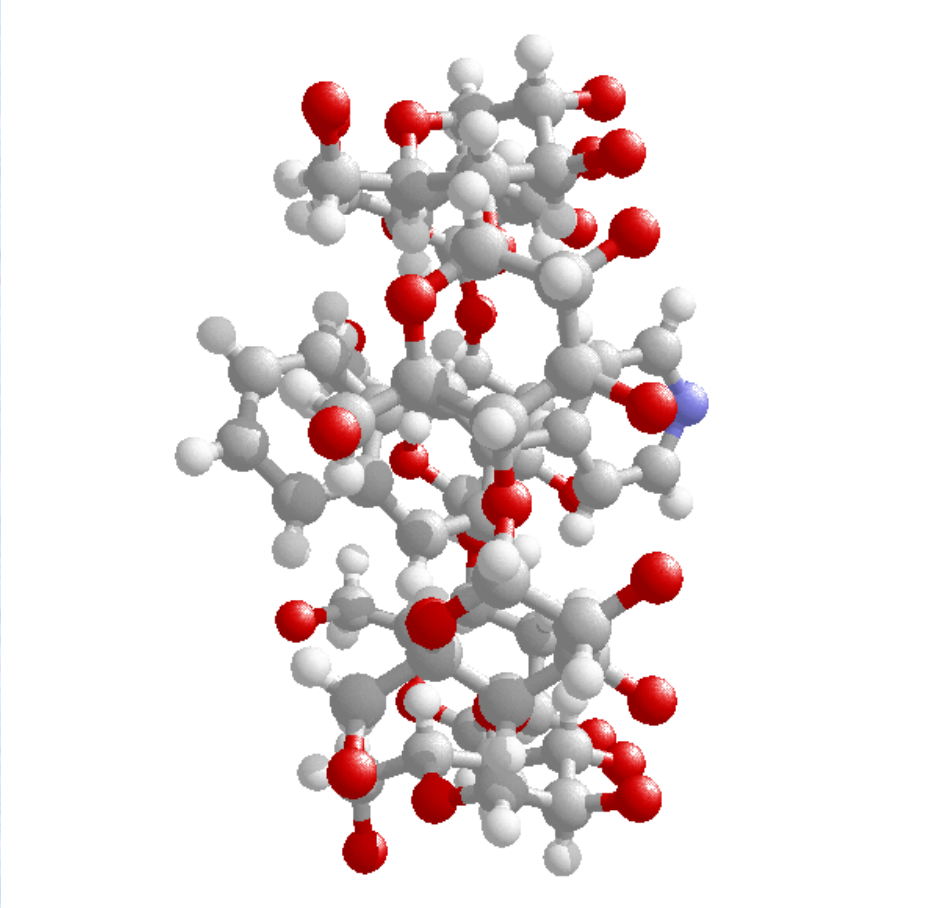
Discovery of the reversible photoinduced mechanical displacement of naphthylpyridine in the β-cyclodextrin cavity allowed us to develop a new type of photocontrolled molecular machines.

Russ. Chem. Bull. **2004**, *53*, 2525;
J. Photochem. Photobiol. A. **2011**, *217*, 87;
Russ. Chem. Bull. **2013**, *62*, 2150.

X-ray structure determination of photocontrolled supramolecular machine



1@β-CD



Russ. Chem. Bull. **2004**, *53*, 2525;
J. Photochem. Photobiol. A. **2011**, *217*, 87;
Russ. Chem. Bull. **2013**, *62*, 2150.

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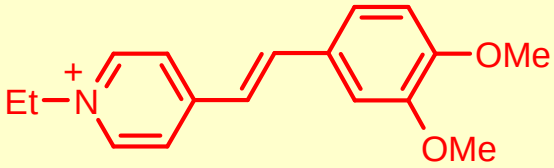
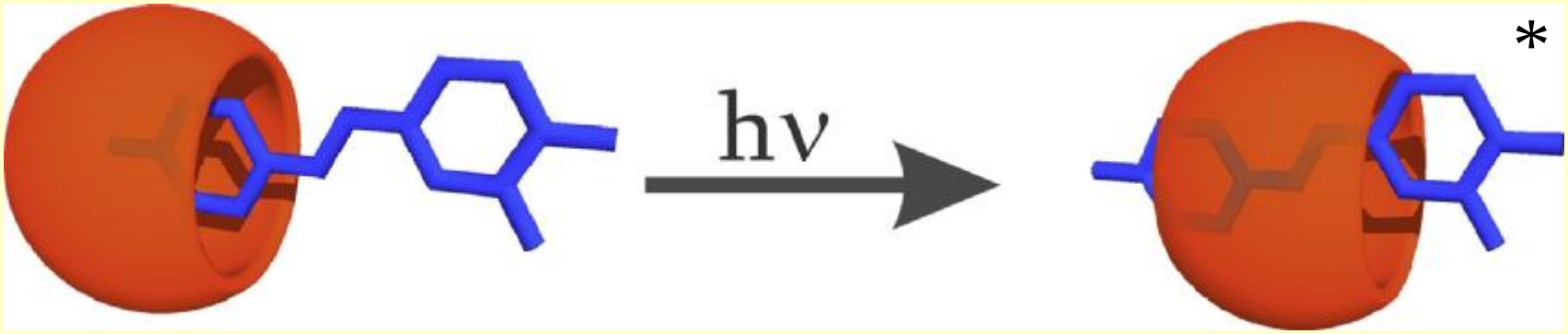
PHOTOCONTROLLED SUPRAMOLECULAR MACHINE



SD



SD@CB[7] **SD**@CB[7] *

Chem. Phys. Lett. **2016**, *647*, 157.

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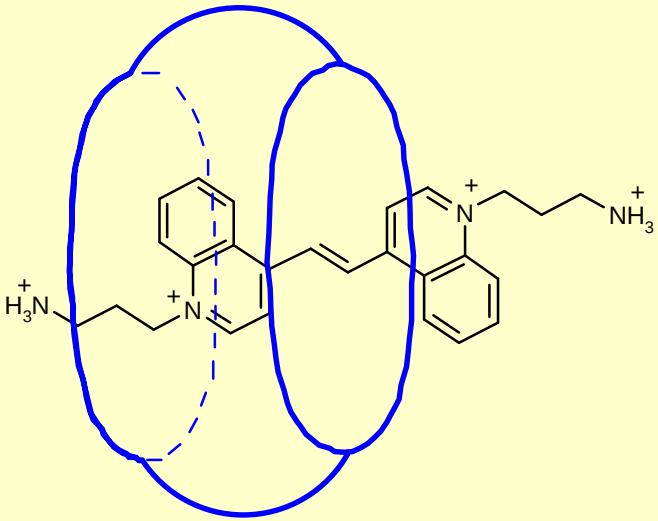
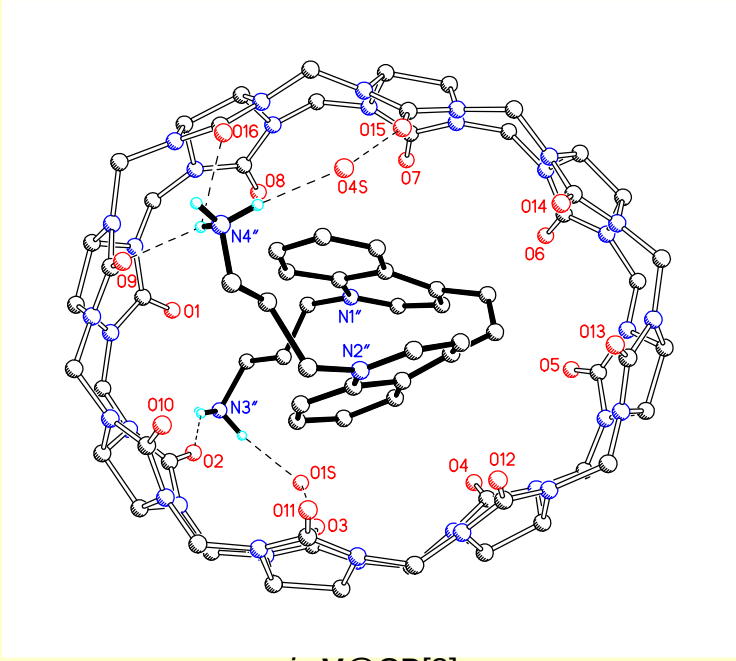
PHOTOCONTROLLED SUPRAMOLECULAR MACHINE



trans-**V**@CB[8]

log *K* = 4.6

$h\nu$



cis-**V**@CB[8]

Pseudorotaxane complexes of cucurbiturils and unsaturated viologen analogues as the design of new-type photocontrolled supramolecular machines

New. J. Chem. **2006**, *30*, 458.

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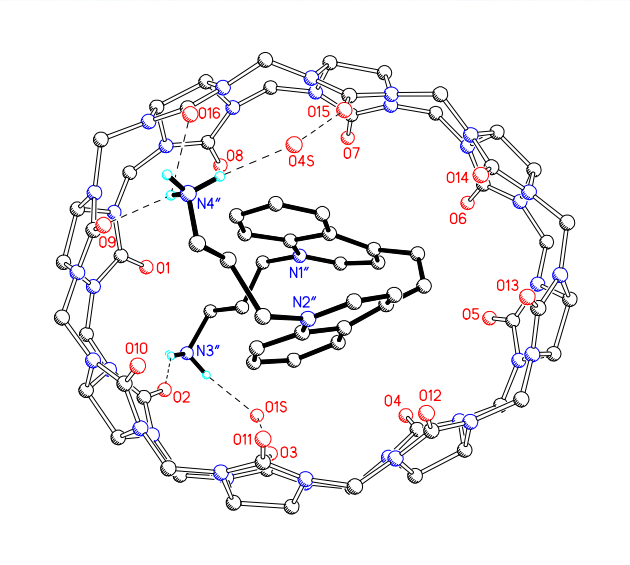
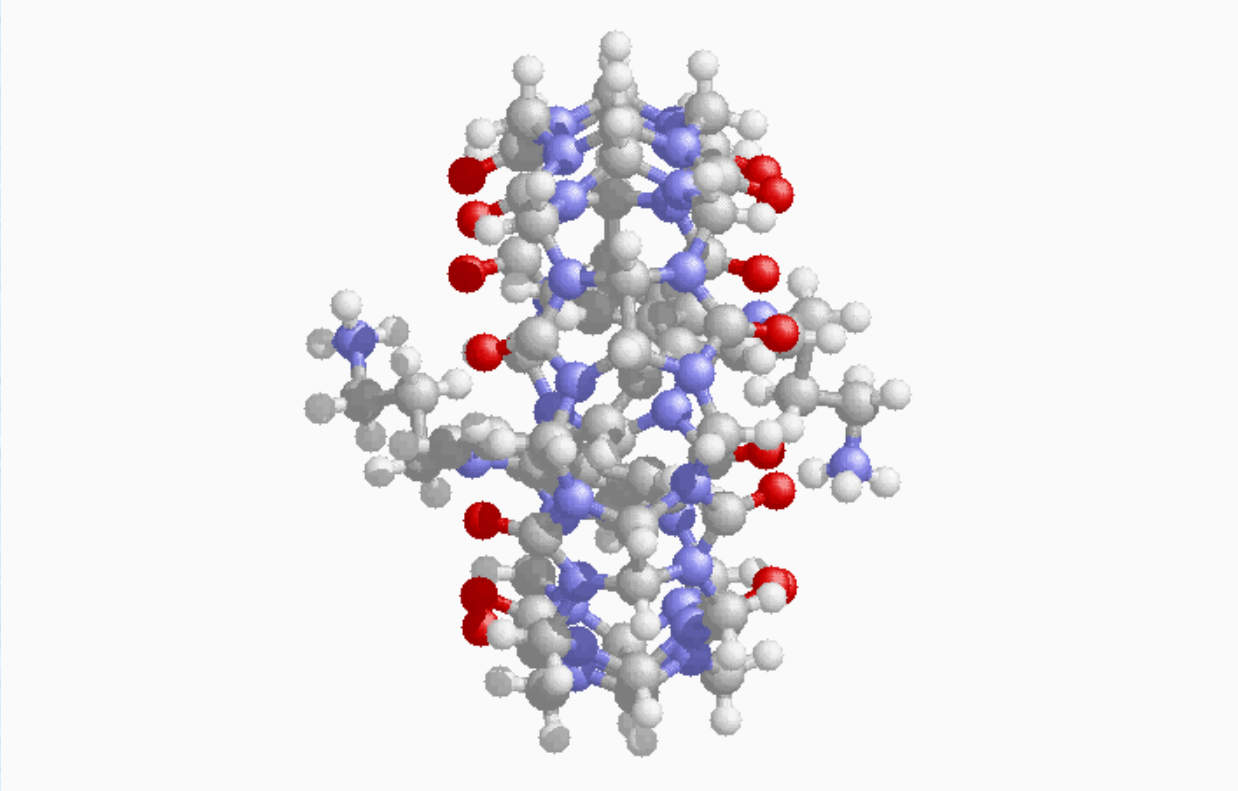
X-ray structure determination of photocontrolled supramolecular machine



cis-**V**@CB[8]



New. J. Chem. **2006**, *30*, 458.

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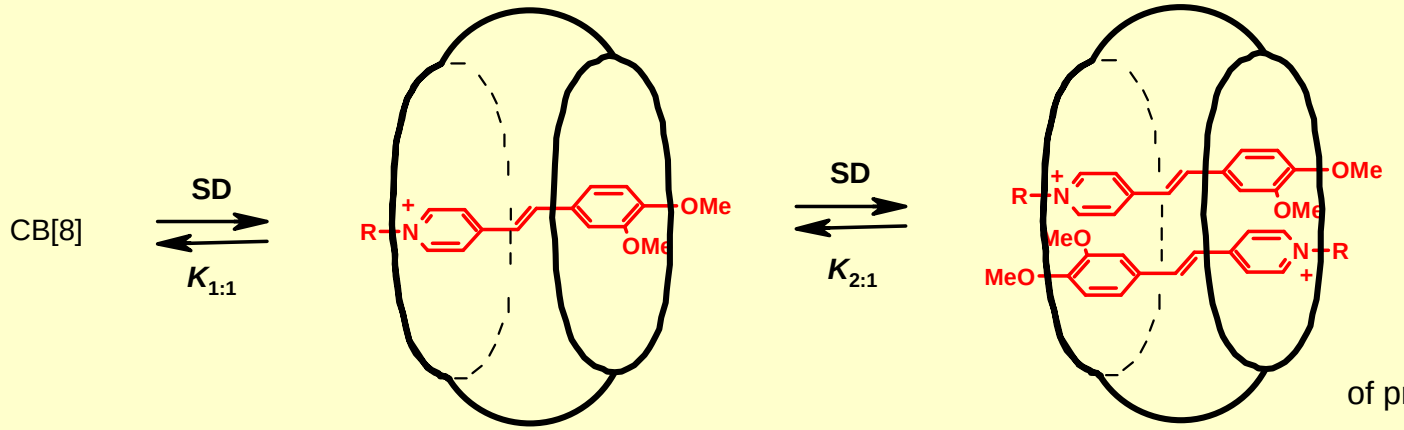
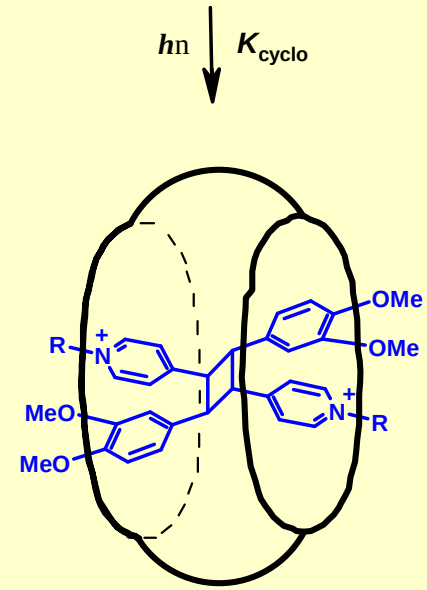
PHOTOCONTROLLED SUPRAMOLECULAR MACHINES



SD@CB[8]

(**SD**)$_2$@CB[8]

Time
of pre-organization
~ 4 ps



cyclobutane@CB[8]

SD	CB[8]		
R	log $K_{1:1}$	log $K_{2:1}$	log K_{cyclo}
Et	4.9	4.1	4.3
$(CH_2)_3NH_3^+$	5.0	4.4	4.8
$(CH_2)_3SO_3^-$	4.0	**2.6**	**3.2**

Eur. J. Org. Chem. **2010**, 2587;
J. Phys. Chem. A. **2011**, *115*, 4505;
J. Photochem. Photobio. A. **2013**, *253*, 52;
Chem. Phys. Lett. **2016**, *647*, 157.

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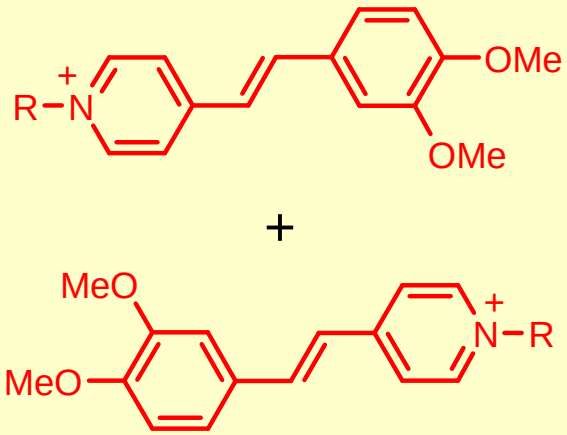
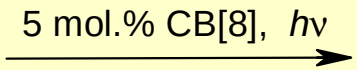
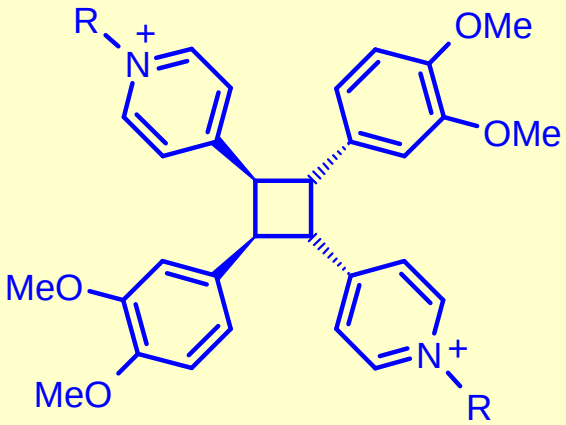
PHOTOCONTROLLED SUPRAMOLECULAR ASSEMBLER BASED ON CUCURBIT[8]URIL



styryl dye (**SD**)





rctt isomer of cyclobutane

Eur. J. Org. Chem., **2010**, 2587.

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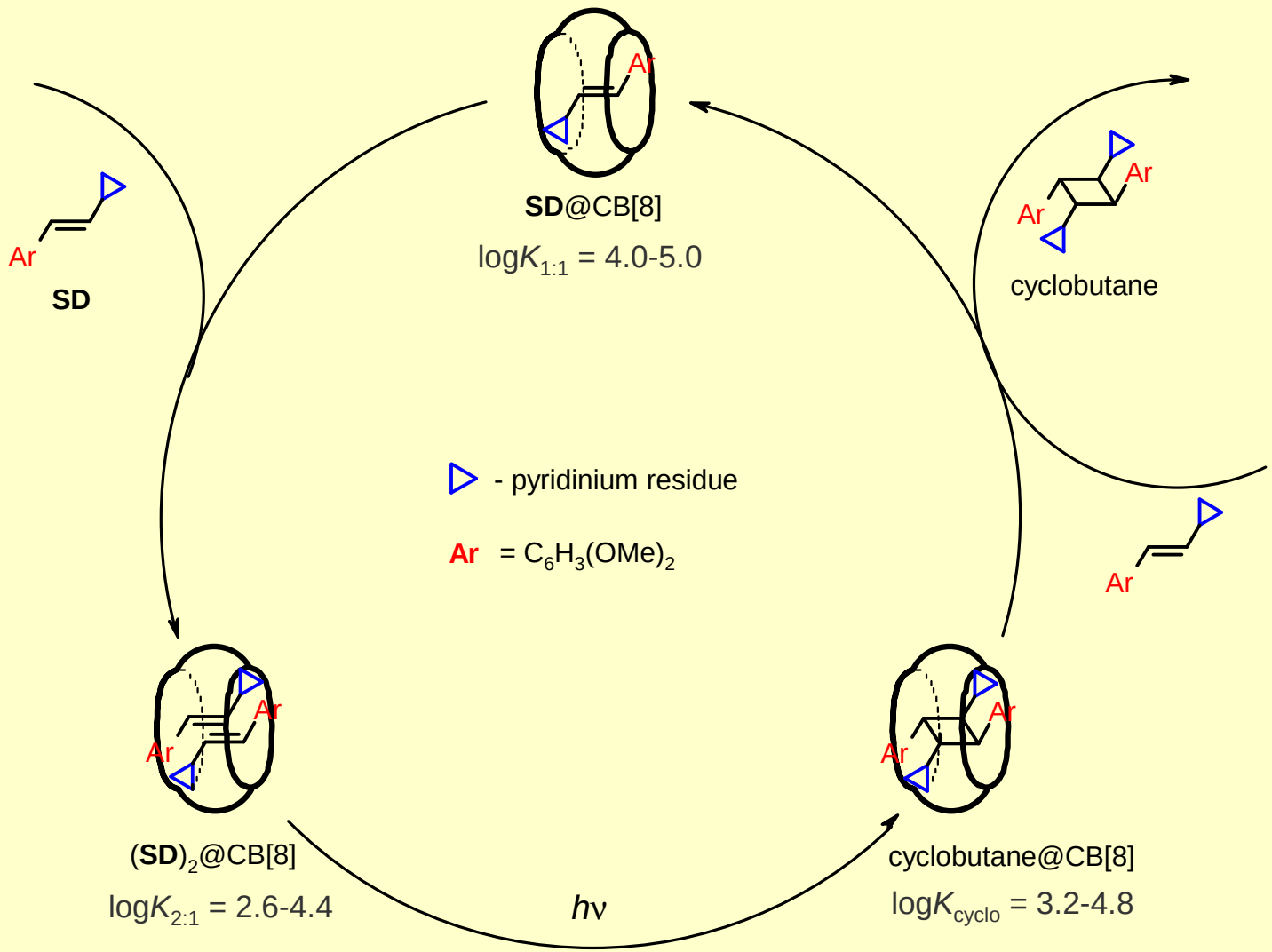
PHOTOCONTROLLED SUPRAMOLECULAR ASSEMBLER BASED ON CUCURBIT[8]URIL



Eur. J. Org. Chem., **2010**, 2587;
J. Phys. Chem. A., **2011**, *115*, 4505;
High Energy Chem., **2014**, *48*, 253;
Chem. Phys. Lett. **2017**, *673*, 99.

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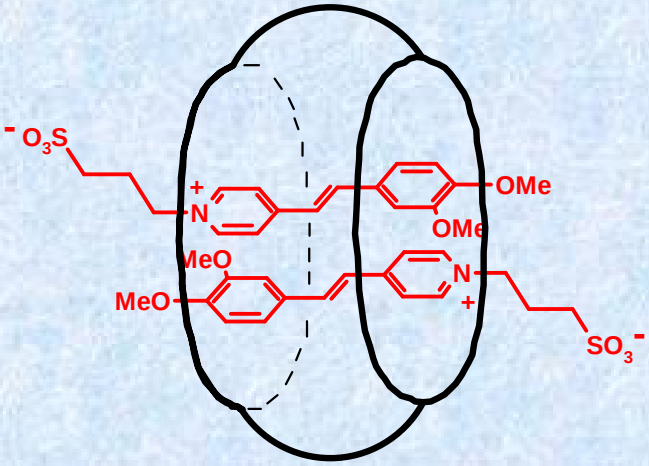
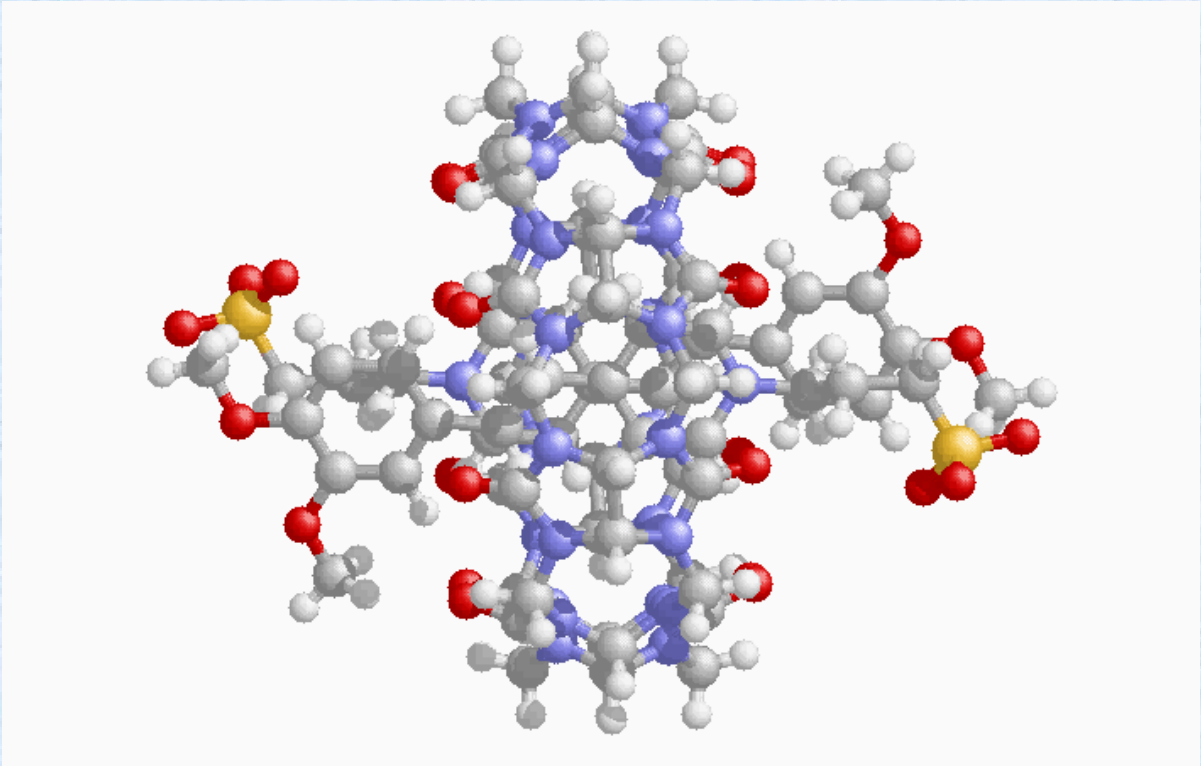
X-ray structure determination of photocontrolled supramolecular assembler



(**SD**)$_2$@CB[8]



Eur. J. Org. Chem., **2010**, 2587.

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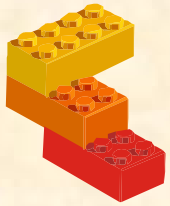
It is possible to implement all main types of photoprocesses:

- Fluorescence, excimer formation
- Photodissociation
- Photoisomerization
- Photocycloaddition
- photoelectrocyclization
- charge-transfer complex formation, electron transfer
- proton transfer
- excitation transfer
- TICT state

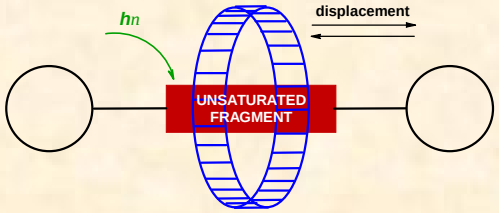
Gromov S. P. *Russ. Chem. Bull*. **2008**, *57*, 1325 (review);
Ushakov E. N., Gromov S. P. et al. *Russ. Chem. Rev.* **2008**, *77*, 39 (review);
Ushakov E. N., Gromov S. P. *Russ. Chem. Rev.* **2015**, *84,* 787 (review).

Molecular meccano
of photoactive supramolecular systems



Unique set of characteristics needed:

- **Accessibility through organic synthesis.**
- **Tendency for spontaneous organization into various supramolecular architectures.**
- **The ability to undergo different types of photochemical transformations depending on the structure.**
- **The feature of high-efficiency molecular photoswitching.**



Gromov S. P. *Russ. Chem. Bull*. **2008**, *57*, 1325 (review);
Gromov S. P. *Rev. J. Chem.* **2011**, *1*, 1 (review).

Applied potential:

new strategy for the design of materials for nanophotonics

Demonstrated by an example of design:

- **Photoswitchable supramolecular devices**
- **Photocontrolled supramolecular machines**
- **Supramolecular photoswitches**
- **Optical chemosensor materials**
- **Data optical recording and storage systems**
- **Photochromic ionophores and photocontrolled membrane transport**
- **Photoswitchable polymeric and LB films**
- **Laser dyes**

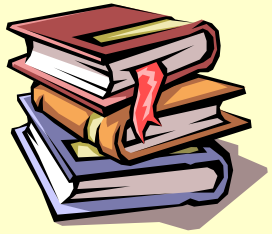
Gromov S. P. *Russ. Chem. Bull*. **2008**, *57*, 1325 (review);
Ushakov E. N., Gromov S. P. et al. *Russ. Chem. Rev.* **2008**, *77*, 39 (review);
Ushakov E. N., Gromov S. P. *Russ. Chem. Rev.* **2015**, *84,* 787 (review).

Publications :

More than 320 publications in scientific journals and patents

Collaboration

- Institute of Problems of Chemical Physics of RAS
- Kurnakov Institute of General and Inorganic Chemistry of RAS
- Lomonosov Moscow State University, Chemical Department
- Institute of Bioorganic Chemistry of RAS
- Lomonosov Moscow State Academy of Fine Chemical Technology
- Zelinsky Institute of Organic Chemistry of RAS
- University of Durham, Great Britain
- Max-Planck-Institut fur Biophysikalische Chemie, Germany
- am Engler-Bunte Institut der Universitat Karlsruhe, Germany
- University of Umea, Sweden
- Bogatsky Physicochemical Institute of NAS, Ukraine
- North Carolina State University, U.S.A.
- The Florida State University, U.S.A.
- Universita' Degli Studi Di Bologna, Italy



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- CRDF (1996 - 2004)
- DFG (1996 - 2004)
- ISF (1993 - 1994)

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State Prize of the Russian Federation (2018)

A. Butlerov prize of Russian Academy of Sciences (2006)

Scientific discovery of the USSR (1980)

Thank You

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http://suprachem.photonics.ru;
http://www.chem.msu.ru/rus/lab/organic/supra-nano.html

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