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CONTENTS

Transition Metals Complexes with Helical 2,2'-Bipyridine Ligands	
<i>Martin Kos</i>	5
Phosphorus Derivatives of Helicenes	
<i>Ing. Tomáš Beránek</i>	7
Synthesis and Functionalization of Aza[5]phenacenes	
<i>Ing. Lubomír Váňa</i>	8
From Cationic Carbosilane Dendrimers and Glycodendrimers towards Multifunctional Dendritic Wedges	
<i>Ing. Monika Müllerová</i>	10
Effect of Conditions on TiO ₂ Aerogel Crystallization Using Modified Supercritical Carbon Dioxide	
<i>Ing. Marie Cerhová</i>	12
Analyzing the Experimental Thermodynamic Data for Ionic Liquids and their Study as Thermal Energy Storage Materials	
<i>Maja Čanji, MSc.</i>	14
Single-Step Purification of Raw Biogas to Biomethane Quality by Hollow Fiber Membranes without any Pre-Treatment – Innovation in Biogas Upgrading	
<i>Ing. Michal Žák</i>	16
Magnesium Silicide and Germanide from Waste as a Source of CVD Precursors and Ultrapure Semiconductors	
<i>Ing. Jakub Bumba</i>	18
Hydrodynamic Behavior of Large Bubbles in Slot Channels	
<i>Kingsley Ezeji, MSc.</i>	20

Transition Metals Complexes with Helical 2,2'-Bipyridine Ligands

Student: Martin Kos
Supervisor: Dr. Vladimír Církva

2,2'-Bipyridine (bipy) derivatives are widely used N,N' -bidentate ligands in coordination chemistry, giving access to a great variety of complexes.¹ The luminescence properties of d^6 transition metal pyridyl complexes have been increasingly studied for the development of new metal-based luminescent materials and sensing probes.² Many of these complexes exhibit room temperature phosphorescence from triplet metal-to-ligand and therefore find applications as electroswitchable emissive systems,³ a cellular imaging agents,⁴ chromophores for photoredox chemistry,⁵ *etc.*

It would therefore be of great interest to develop chiral analogues in order to benefit from the chiral version of emission, namely circularly polarized luminescence, which may potentially be used in cryptography or for 3D-displays.⁶

Herein we report synthesis of series of novel chiral complexes of various metals (Cu, Re, *etc.*) with helical ligands containing bipyridine moiety (Fig. 1). Photochemical and photophysical properties of prepared complexes will be studied in both racemic and optical pure form.

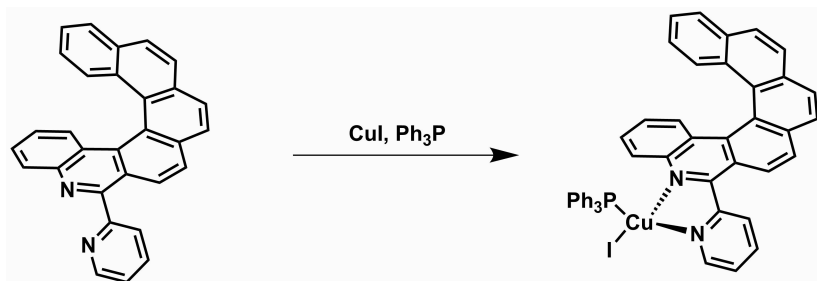


Figure 1

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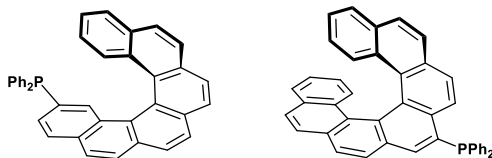
Phosphorus Derivatives of Helicenes

Student: Ing. Tomáš Beránek

Supervisor: Ing. Jan Sýkora, Ph. D.

Supervising Expert: RNDr. Jaroslav Žádný, Ph. D.

Helicenes possess a helical conjugated polyaromatic system consisting of *ortho*-fused benzene rings as a fundamental property of this fascinating compounds. Due to this, they also exhibit interesting physicochemical properties including P-type conductivity and enormous optical activity. Helicene chemistry is being considered as an expanding and modern field of research, leading to various applications¹ in supramolecular chemistry, nanoscience, chemical-biology, polymers, or materials science. Surprisingly, coordination chemistry of helically chiral phosphines and their use as chirality inductors is still strongly under-developed. Although several pilot experiments have already been carried out in this area, a larger study of the preparation of metal complexes bearing helical ligands is still missing.² In this work, synthetic pathways providing suitable phosphorous derivatives of helicenes and their transition metal complexes in both the racemic and the chiral forms were investigated in asymmetric Suzuki reaction.



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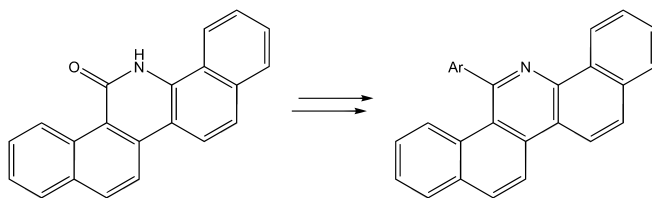
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Synthesis and Functionalization of Aza[5]phenacenes

Student: Ing. Lubomír Váňa
Supervisor: Dr. Ing. Vladimír Církva

The study of organic electronics based on π -electron network molecules has made rapid progress in recent years. Phenacenes with their extended two-dimensional π -conjugated structure are suitable for this purpose as they can provide charge-transporting pathway when arranged into appropriated thin layer in solid state.¹

This work is aimed at the development of new efficient methodology towards the synthesis of various aza[5]phenacenes with δ -lactam rings in its structures. Possibilities of functionalization or derivatization of [5]phenacene with δ -lactam ring are also discussed (Scheme 1). The resulting compounds are subsequently investigated in terms of their material properties, especially for the formation of the thin film structures capable of efficient semiconductivity. In addition, the complexation properties of suitable aza-derivatives as ligands with transition metals or lanthanides can be studied due to their potential application as photoluminescent complexes in OLEDs.²



Ar = phenyl, 2-pyridyl, 2-thienyl, 3-thienyl, etc.

Scheme 1. Functionalization of [5]phenacene with δ -lactam ring

Acknowledgement

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From Cationic Carbosilane Dendrimers and Glycodendrimers towards Multifunctional Dendritic Wedges

Student: Ing. Monika Müllerová
 Supervisor: Ing. Tomáš Strašák, Ph. D.

Carbosilane dendrimers (CS-DDMs) with their excellent stability, inertness, and nonpolar structure have been successfully employed in biomedicine as drug delivery systems (DDS).¹ In our previous work, we synthesized a series of 1st – 3rd generation CS-DDMs bearing cationic (ammonium, phosphonium) moieties at the periphery. As the phosphonium CS-DDMs performed lowered *in vitro* cytotoxicity, high dendriplex stability and elevated transfection efficacy compared to the ammonium ones, but in general as well, they represent a promising alternative to current DDS (vectors) in gene therapy.² In a similar manner, we prepared CS glucose glycodendrimers to perform the first comparative study of the *in vitro* (MTT) and *in vivo* (modified FET) cytotoxicity with remarkable results.³

Interior of CS-DDMs is prepared by standard iterative reaction steps (catalytic hydrosilylation, ω -alkenylation, resp.). Therefore, a scope of possible modifications during the synthesis is limited. Recently, to extend the range of versatility, we developed a novel modular toolbar of multifunctional carbosilane dendritic wedges (CDWs) varying in several ways, such as generation, density of inner branching, or polarity.

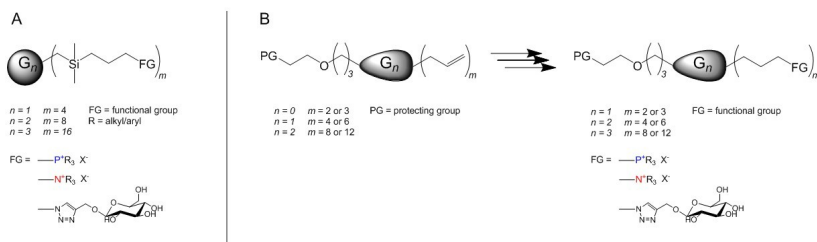


Fig. 1: A) published CS-DDMs with cationic peripheral units and CS glucose glycodendrimers, B) corresponding CDWs and suggested pathway towards their functionalization.

Here we present the CDWs of the 1st-2nd generation with allyl groups at the periphery and a hydroxy/amine terminated linker. The suggested synthesis requires highly selective reactions with excellent yields. Moreover, due to an employment of strong nucleophiles or basic/acidic conditions during the synthesis, it is essential to select convenient protecting groups, which will be briefly discussed.

Thus obtained CDWs can be modified following the same synthetic pathway used for the preparation of the cationic CS-DDMs and glycodendrimers. Moreover, the hydroxy/amine linker group enables an attachment of these CDWs to various substrates (hydrophobic/hydrophilic chains, multivalent cores, fluorescent molecules, *etc.*) to obtain *e.g.* amphiphilic segments of CDWs or Janus-type CDWs.⁴

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Effect of Conditions on TiO₂ Aerogel Crystallization Using Modified Supercritical Carbon Dioxide

Student: Ing. Marie Cerhová

Supervisor: Ing. Marie Sajfrtová, PhD.

Supervising Expert: Ing. Lenka Matějová, PhD.

Titanium dioxide in the form of aerogels belongs to the most desirable materials due to its crystalline anatase structure exhibiting high photocatalytic activity. TiO₂ is used in the environmental applications such as air or water purification.¹

Crystalline and pure TiO₂ aerogels are prepared by the calcination, which is accompanied by uncontrollable destruction of the porous structure or a decrease in specific surface and porosity.² However, the thermal treatment has recently been successfully replaced by gentle low-thermal supercritical fluid crystallization (SFC) with modified supercritical carbon dioxide (scCO₂).³ In our previous studies,^{3–5} the efficiency of this method was demonstrated for TiO₂ in the forms of thin films and aerogels in which water and ethanol were used as modifiers. The critical effect of water on the TiO₂ crystallization and the beneficial influence of ethanol on the purity and phase composition of TiO₂ were found.

Therefore, this work is focused on a deeper study of TiO₂ aerogel SFC with scCO₂ modified by mixture of ethanol and water in a ratio 1:1. The effect of temperature (40–100 °C), pressure (10–30 MPa) and modifier concentration in scCO₂ (5–15 wt.%) on microstructure, purity and textural properties of TiO₂ aerogel was investigated. Processed monoliths were characterized by XRD analysis, Raman spectroscopy and N₂ physisorption.

Increasing temperature, pressure or modifier concentration in scCO₂ showed the positive effect on the aerogel purity. However, the temperature of 100 °C or the presence of too large amount of mixture (*i.e.* 15 wt.%) resulted into the aerogel destruction. Therefore, an optimal temperature and amount of mixture in scCO₂ in terms of the aerogel crystalline structure presence and sufficient purity was determined to be 80 °C and 10 wt.%. The increasing pressure led to the undesirable decrease of monolith specific surface area.

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Analyzing the Experimental Thermodynamic Data for Ionic Liquids and their Study as Thermal Energy Storage Materials

Student: Maja Čanji, MSc.

Supervisor: Ing. Magdalena Bendová, Ph. D.

The aim of this work is to characterize a series of 1-alkyl-3-methylimidazolium saccharinate ionic liquids (alkyl standing for butyl, hexyl, octyl, and decyl),¹ analyze the experimental data, and assess their potential application in thermal energy storage.²

The characterization of this series of ionic liquids was carried out experimentally by measurements of heat capacity using the differential scanning calorimetry (DSC) and of density by the oscillating-tube densitometry. The experimental data were then analyzed by means of the advanced data analysis methods based on mathematical gnostics.³ Mathematical gnostics is a novel non-statistical approach to data uncertainty. As such it enables us to evaluate the measurement uncertainty of statistically non-significant data sets containing as few as four datapoints and identify unreliable or outlying data.⁴ Also, using a robust regression algorithms along a gnostic influence function, functional dependencies and structure-property patterns can be reliably determined. Modelling based on the COSMO-RS⁵ model was used to predict density and heat capacity⁶ for the studied ILs and compared with the experimental data.

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Single-Step Purification of Raw Biogas to Biomethane Quality by Hollow Fiber Membranes without any Pre-Treatment – Innovation in Biogas Upgrading

Student: Ing. Michal Žák

Supervisor: Ing. Pavel Izák, Ph. D., DSc.

Herein, a radical innovation in biogas production is presented using a single-step membrane purification technology without any necessary pre-treatment steps for contaminant removal. Asymmetric hollow fiber membranes with a thin, selective, non-porous layer made from polyester carbonate on the bore side were successfully used in a single-step raw biogas upgrading process at biogas plant Chořovice in Czech Republic.

A double-stage arrangement operated at 17 °C exhibited the best CO₂/CH₄ separation factor (~23) with 96 vol.% CH₄ purity. This single-step biogas upgrading technology with highly H₂S and water-resistant membrane modules allows reductions in capital expenditures of ~20% and in operational expenditures of ~50% compared to high pressure water scrubbing (HPWS) and ~70% compared to pressure swing absorption (PSA). Due to small energy consumption required merely for biogas compression, our units consumed around only ~2 MJ/Nm³ CH₄. Thus, our approach with significantly improved performance and robustness of the membrane separation process can be seen as a major breakthrough of membrane gas separation for inexpensive biogas production.

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Magnesium Silicide and Germanide from Waste as a Source of CVD Precursors and Ultrapure Semiconductors

Student: Ing. Jakub Bumba
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Chemical vapour deposition (CVD) is a technology used for forming solid films of semiconductors, metals or alloys on substrate from precursor in the gas phase. Ultrapure semiconductors mainly silicon, germanium and their SiGe alloys are commonly used for production of electronic components like chips, transistors, optical parts, circuits, *etc.* However, a top purity is required for their application in electronic industry. Silanes ($\text{Si}_n\text{H}_{2n+2}$), germanes ($\text{Ge}_n\text{H}_{2n+2}$) and germysilanes ($\text{Si}_x\text{Ge}_y\text{H}_z$) belong to common CVD precursors applied for creation the thin semiconductor layers. Regrettably, the traditional refining methods for preparation of CVD precursors from waste Si, Ge, or SiGe are energetically and technologically demanding which is significantly reflected in their price. From that reason the newly patented process, in which waste photovoltaic panels and optical lenses served as a raw materials, was tested to be the lucrative source of silicon or germanium for CVD precursors.

The process is based on preparation of magnesium silicide (Mg_2Si), magnesium germanide (Mg_2Ge) or their mixtures directly by thermal synthesis from milled end of life photovoltaic cells and/or broken germanium lenses with waste magnesium chips. Prepared samples were characterized by X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Spectroscopy (EDX) and Raman spectroscopy, which confirmed high conversion to desired products, good crystallinity and homogeneity. The products were then hydrolysed by phosphoric acid solution to form corresponding hydrides. Fourier Transform Infrared Spectroscopy (FTIR) and Gas Chromatography–Mass Spectroscopy (GC-MS) verified that silanes and/or germanes even germysilanes were presented. The fraction distillation of individual hydrides was also successfully tested and obtained hydrides could be used either as a desired CVD precursor or as raw materials for thermal decomposition into ultrapure semiconductors (Si, Ge) and hydrogen.

Acknowledgement

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Hydrodynamic Behavior of Large Bubbles in Slot Channels

*Student: Kingsley Ezeji, MSc.
Supervisor: Ing. Jaroslav Tihon, CSc.*

The vertical movement of gas bubbles in a liquid phase is of enormous significance in a number of industrial operations such as electrolytic cells, filtration devices and heat exchangers. The movement of these gas bubbles provides in these devices vigorous fluid mixing. However, there is very little published data on the hydrodynamic behavior of such bubbles in inclined rectangular channels.

We present the results of experimental investigation on the dynamics of air bubbles rising in stagnant liquid inside an inclined channel. The experiments were conducted in a Plexiglas channel having a rectangular cross-section (240×20 mm). Different channel geometries were then obtained by insertion of suitable plates into this basic channel. The measurements using a high speed video camera were done for channel inclinations ranging from $\alpha = 5^\circ$ (almost horizontal) to 90° (vertical). Subsequent image processing provided information on the bubble shape, size, and rise velocity U_B . The specific impact of channel geometry and inclination on the shape, stability, and velocity of rising bubbles was studied and discussed.

The results obtained in all studied channel configurations suggest that sufficiently large bubbles reach a final rise velocity U_{TB} , which is no more sensitive to further increasing in the bubble size. In vertical channels, this final bubble velocity U_{TB}^v is controlled by the channel perimeter P and a universal velocity scaling based on the Froude number $Fr_v = U_{TB}/(g \cdot P)^{1/2} \cong 0.2^1$ was confirmed to be valid. In horizontal channels, the final translating velocity U_{TB}^h is controlled by the channel height H and the scaling based on the Froude number $Fr_h = U_{TB}/(g \cdot P)^{1/2} \cong 0.5$ is recommended for large tubes and channels² U_{TB}^h values obtained in our experiments by data extrapolation suggest that surface tension plays important role in flat channels and Fr_h decreases with increasing $\Sigma = 4\sigma/\rho g H^2$. In inclined channels, the bubble velocity scaling based on a weighted superposition of U_{TB}^h and U_{TB}^v was proposed³. Our experimental data suggests a relationship in the form $U_{TB} = U_{TB}^v \sin^m \alpha + U_{TB}^h \cos^n \alpha$ where two power parameters (m and n) are dependent on the channel geometry. The first parameter distinct

between flat ($m = 1/2$ for $H < 10$ mm) and tall ($m = 1$ for $H > 20$ mm) channels, whereas the second parameter is sensitive to the aspect ratio of tall channels and thus ranging between $n = 1$ (for narrow channels with $H/W \gg 1$) and $n = 2$ (for wide channels with $H/W \ll 1$).

Our next experiments will be focused on shear stresses induced by rising bubbles on the channel walls. The electrodiffusion measuring technique will be used for this purpose. The results of our preliminary measurements confirmed that the two-strip probe is able provide valuable information on near-wall flow around the bubble, including the detection of near-wall flow reversal under the bubble.

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