

Abstracts

for

**The 6th Scientific Session of Undergraduate,
Master and PhD Students, Iasi, Romania**

June, 26, 2015

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P1. Synthesis by Autocombustion of AB_2O_4 Spinel Compounds, where $A = Ni$, $B = Cr, Al$ and Fe , Using Fructose

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The synthesis of $NiCr_2O_4$, $NiAl_2O_4$ and $NiFe_2O_4$ compounds was achieved by sol-gel autocombustion method.

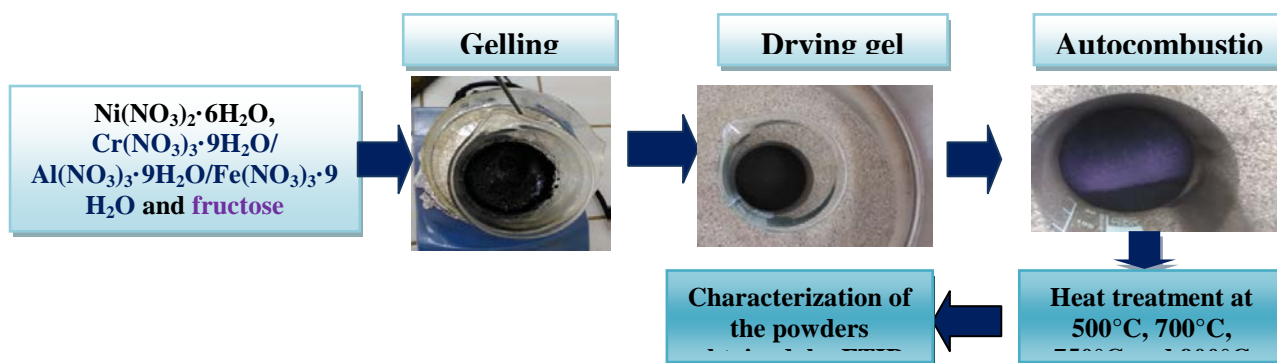
In this paper fructose was used for the first time as a complexing/fuel agent. FTIR spectroscopy was employed as a method of tracking the solid phase chemical reaction. The samples obtained were characterized by XRD, and the fuel agent was subjected to thermal analysis.

Combinations of polymetallic oxide with spinel structure are widely used in present technologies.

Currently, nickel chromite ($NiCr_2O_4$) is used in the processes of oxidative dehydrogenation of propane in the catalytic material and as a gas sensor.

Nickel aluminate ($NiAl_2O_4$) is used in ceramic pigments, coatings, catalysts and as anode electrode material for internal reforming solid oxide fuel cells (IR-SOFC), and nickel ferrite ($NiFe_2O_4$) is used to obtain ferrofluids, catalysts, devices that use microwave technology, gas sensors and magnetic materials.

Synthesis and analysis of samples was conducted through the following work protocol:



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P2. Synthesis and NMR Characterization of New *bis*-Pyridine-Imidazolium Salt

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A thorough literature survey has revealed that dihydroxyacetophenone derivatives are one of the most used classes of building blocks in supramolecular chemistry. Also, imidazolium salts are compounds with biological properties: antibacterial, anti-inflammatory, anticancer. Besides, pyridine is intensively used as scaffold for drug development.

Having these considerations in view, our goal was to synthesize and characterize a new *bis*-pyridine-imidazolium salt. The synthesis was done in three steps: (I) *O*-alkylation of 3,5-dihydroxyacetophenone with *bis*-chloromethyl pyridine, giving a halogenated derivative with increased reactivity; (II) *N*-alkylation of imidazole with the compound obtained in the first step; (III) quaternization reaction of *bis*-pyridine-imidazole derivative with *p*-nitrophenacyl bromide.

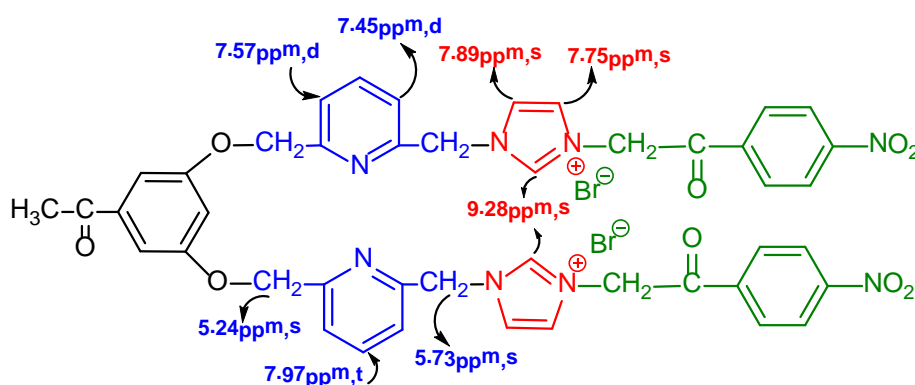


Figure 1. The structure of *bis*-pyridine-imidazolium salt with the most important signals from ¹H NMR spectrum

The NMR experiments (¹H NMR, ¹³C NMR, 2D-correlations) were used to prove the structure of the newly synthesized salt. The NMR spectra have been recorded on a Bruker Avance III 500 spectrometer. In ¹H and ¹³C spectra, chemical shifts are reported in δ units (ppm) relative to the residual peak of solvent (ref: DMSO, ¹H: 2.50 ppm; ¹³C: 39.52 ppm).

Acknowledgements: This work was supported by PN-II-DE-PCE-2011-3-0038, no.268/05.10.2011. We also thank to the POSCCE-O 2.2.1, SMIS-CSNR 13984-901, No. 257/28.09.2010 Project, CERNESIM, for the NMR experiments.

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P3. Chromatographic Fingerprints as a First Step in the Long Way of Deciphering the Very Complex Nature of Herbal Materials

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Chromatography is a recent recommended technique for generating herbal fingerprints. Moreover, the World Health Organization accepts chromatographic fingerprints as a tool for identification and quality control assessments of herbal medicines. Gas chromatography tandem mass spectrometry (GC-MS) has been used in the present work in order to decipher the mystery induced by the complex chemical composition of a selected herbal, i.e. *Datura*. To our knowledge, these are the first attempts for the identification of the chemical constituents of *Datura* (leaves, flowers, and seeds) based on the evaluation of entire chromatographic fingerprint profiles. The first pool of the interest samples has been collected from a countryside region in October 2014. Preparative steps involved: grinding, sieving, weighing, extracting (6 solvents have been tested) by using an ultrasonic water bath, usually applied cleanup and drying procedure, filtering and GC-MS analysis of the extracts. GC analysis was conducted on a DB-5ms (Agilent) capillary column of 30 m, 0.25 mm i.d., 0.25 microns film thickness, and the following conditions were used: He constant flow 1 mL/min, inlet temperature 280 °C, injection volume 0.005 mL (splitless), MS transfer line temperature 280 °C, temperature program 100 °C for 1 min, then 15 °C/min ramp to 180 °C (held for 1.3 min), followed by 5 °C/min ramp to 300 °C (held for 37 min). Figure 1 shows a GC-MS total ion chromatogram of a seeds matrix extract.

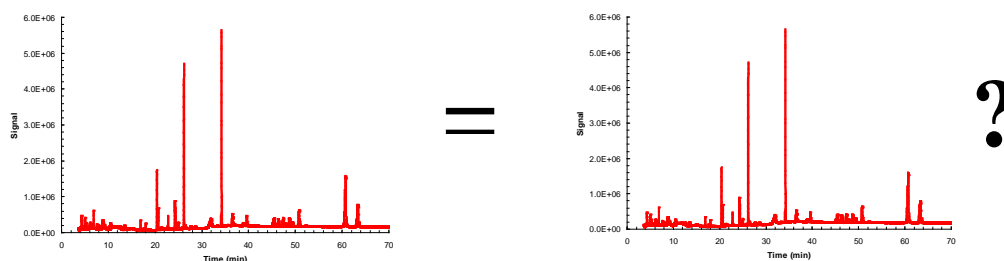


Figure 1: A GC-MS chromatographic fingerprint signal from a *Datura* seeds extract.

Our analysis revealed the existence of various chemicals such as phenols, sugars, alkaloids, squalenes, tocopherols and sterols. It seems that sterols abundances might be related to the defense mechanism developed by the plant in order to fight with various pathogens.

Acknowledgements: *CERNESIM* is gratefully acknowledged for the infrastructure used to achieve related experimental tasks.

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P4. The Students' Perceptions Regarding the Act of Teaching-Learning and Evaluation

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The variables were constructed and applied by us, as researchers, based on examples of good practice and follow opinions regarding the act of teaching-learning and evaluation for perceptual categories like: the influence of the laboratory over the interest of a specific subject, the usefulness of laboratory work for understanding the course content, the importance of the difficulty of the subjects for a more accessible theory.

It is desirable to find correlations between the frontal, team and individual work. The frontal activity is favored because of its low cost, but what can a professor do when a few students do not understand the information presented to them, but the rest understand it? For this reason, the methods of team work are complementing the frontal activity. Unfortunately, the costs are higher, on a longer period of time, and fewer information is presented.

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P5. New Photoactive Urethane Carbohydrates Employed in Grid Structures Fabrication by Two-photon Polymerization (2PP)

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Currently, polymeric materials are significant components of every field of human activities, being a part of our daily life, this highlighting the "plastic-dependence" of the modern society. For example, the carbohydrate-bearing polymers, especially synthetic polymers containing pendant sugar moieties (known as glycopolymers), have found applications mostly in biomedicine and as biomaterials. Therefore, there have been many studies on the preparation of glycopolymers with a large diversity of architectures, using different approaches like conventional and controlled radical polymerization, living anionic polymerization, cyanoxyl mediated polymerization, ring opening polymerization and post-polymerization modification. A topic of interest, but less investigated consists in employing glycomonomers in the photocuring process. The photopolymerization reaction induced by UV or laser radiation represents an environmentally friendly procedure without pollutant emission such as volatile organic compounds (VOC), which can be conducted at room temperature without any degradation of sensitive molecules, and can be easily controlled by simply turn a light on and off.

Taking into account that one of the greatest challenges of our time is to yield materials through non-polluting technologies, the purpose of this work is to enlarge the family of photoactive carbohydrates employing an eco-friendly synthesis method which may be easily applied for large-scale production. Hence, in the present work, we present the synthesis and characterization of photopolymerizable methacrylates bearing a monosaccharide (glucofuranose, galactopyranose, mannitol) derivative. Following their photobehavior at the exposure to UV light by FTIR measurements, a good photoreactivity of the monomers in photopolymerization reactions (especially in the presence of Irg2959) was observed, which may be improved by the addition of urethane dimethacrylates with PEG units as comonomers. Besides, for creating the grid structures through two-photon polymerization experiments Irg819 is recommended to be used as photoinitiator. The design of predetermined molecular architectures with controlled porosity may be exploited in tissue engineering applications.

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P6. Cyanide Determination in Environmental Samples

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Cyanide represents any chemical compound which contains at least one cyano group (C≡N), comprised of one carbon atom in triple bound to a nitrogen atom through a triple bond. Alkali metal cyanides are known as poisons, the most commonly used being the potassium cyanide (KCN). Ingestion of a very small amount of cyanide (200 mg) – solid or solution -, as well as exposure to air containing 270 ppm of cyanide can rapidly lead to death.

The major source of cyanide emission into the air is represented by exhaust gases, followed by plastic materials fabrication, various materials combustion (coal, plastic, wood, biomass), steel production, public waste incineration, oil refining [1]. In Romania, biomass incineration is quite frequent.

Cyanide is also notoriously used in mining, for gold extraction, since it has the capacity to separate gold from other minerals. It is becoming necessary to replace or forbid using cyanide in mining operations, especially in areas where sensitive ecosystems are being threatened [2]. Furthermore, as a result of cyanide mining, there are numerous accidents recorded, caused by cyanide spills and leaks. Hence, there is a need of detecting and determining the cyanide content from environmental samples (especially waters) with the help of improved, faster and more effective methods, compared to the standard ones. Cyanide toxicity effects are the most significant upon water organisms. Thus, contaminated waters and fish lead to contaminating human beings.

New detection methods were emphasized, noticing the research preference towards spectrophotometric and spectrofluorimetric methods during the last years.

Acknowledgement: *LT gratefully acknowledges financial support from the contract POSDRU/159/1.5/S/137750.*

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P7. Clinical Diagnostics of Neuronal Ceroid Lipofuscinoses on Dry Blood Spots

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The loss of enzyme activity is a characteristic feature of lysosomal storage diseases (LSDs), a group of ca. 70 metabolic disorders such as mucopolysaccharidoses, sphingolipidoses, and neuronal ceroid lipofuscinoses. Reduced enzymatic activity causes substrate accumulation in lysosomes, which can lead to severe disease symptoms and finally death. For several LSDs treatment has become available by enzyme replacement therapy (ERT), however, successful ERT is critical to start early which renders clinical diagnostics of key importance. Neuronal ceroid lipofuscinoses (NCLs) are a group of neurodegenerative diseases in childhood, characterized by vision loss, dementia, epilepsy and physical decline and early death of patients with incidence rate of about 1:30000 live birth.

Here we describe specific and sensitive diagnostics on dry blood spots (DBS) for neuronal ceroid lipofuscinoses, by simultaneous fluorimetric and MS-MRM analysis (Figure 1).

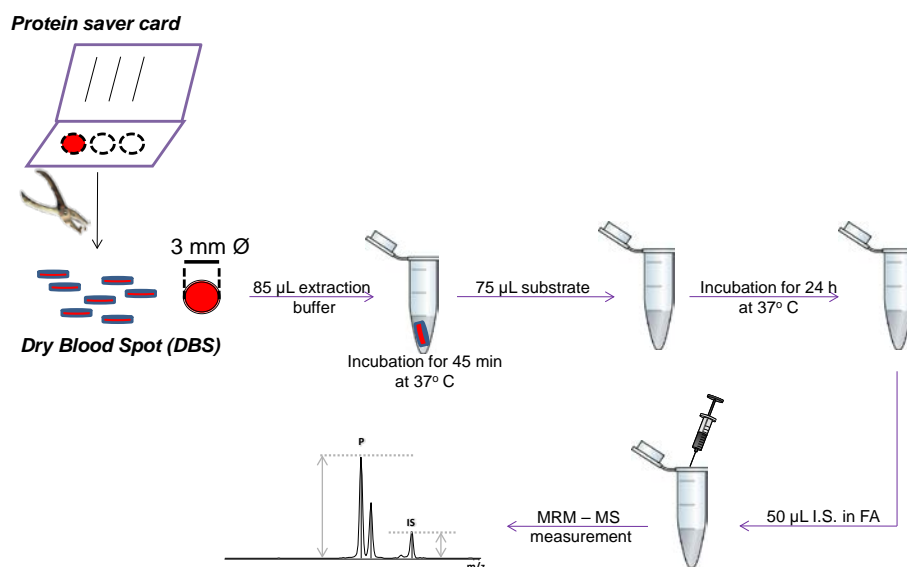


Figure 1. Schematic representation of MRM-MS diagnostics of NCLs

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P8. Coordinative compounds of copper with salicylic acid derivatives and 2,2'-bipyridyl. Obtaining and characterization.

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The coordinative compounds of Cu(II) with mixed ligands were synthesized by the co-precipitation method. Ligands and solvents were used without further purification. In the first stage, complexes of copper with salicylic acid derivatives were synthesized, then the mixed ligand complexes of Cu(Hsal-X)₂ (X = (3,5) t-butyl, (5)/(3,5) NO₂, (3) CH₃, (3) OCH₃, (4)/(6) OH) with 2,2'-bipyridyl were obtained.

The coordinative compounds of Cu(II) with salicylic acid and its derivatives were prepared by reaction between metal and ligand under stirring on water bath. Next, Cu(Hsal-X)₂ were dissolved in DMF, mixed with a methanolic solution of 2,2'-bipyridyl and left overnight to crystallize the new Cu(II) complexes.

The obtained compounds were analyzed by single-crystal X-ray diffraction, UV-Vis and IR spectroscopy. The X-ray diffraction results of examined samples indicate that they crystallize in monoclinic or triclinic systems. For the sample with (3-OCH₃-sal) ligand two types of crystals were obtained: one with orthorhombic structure and the other with monoclinic structure (figure 1).

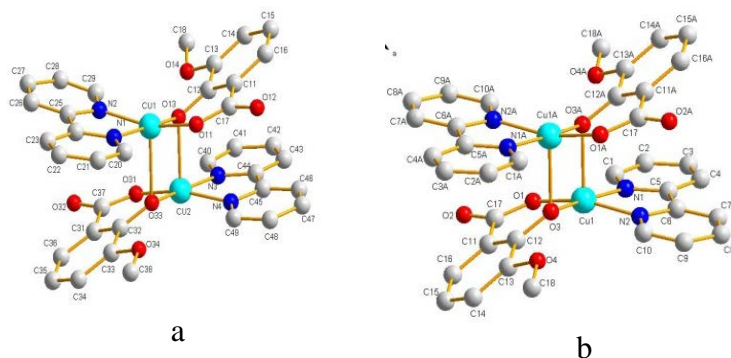


Figure 1. Schematic representation of {[Cu(3-OCH₃-sal)(bpy)]} molecular structure in orthorhombic (a) and monoclinic (b) modifications

The IR spectra revealed the shift of characteristic frequency bands of ligands and disappearance of the signal of OH carboxylic group.

Acknowledgement: I. Bulimestru gratefully acknowledges to Erasmus Mundus IANUS II Program for financial support.

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P9. Synthesis and characterization of copper coordinative compounds with 1,10-phenanthroline and salicylic acid derivatives

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A series of coordinative compounds of copper (II) with 1,10-phenanthroline and salicylic acid derivatives were synthesized and analyzed by UV-Vis spectroscopy, IR spectrophotometry and X-ray diffraction. UV-Vis spectra were obtained on solid samples using a Camspec 501M spectrophotometer, in MgO pellets. IR spectra were determined by ATR (attenuated total reflectance)-FTIR technique with Bruker α spectrophotometer. Characterization of compounds by X-ray diffraction was carried out with a single crystal X-ray diffractometer Supernova 250.

Synthesis of mixed complexes of Cu(II) was done in two stages: in the first stage, [Cu(Hsal)₂] coordinative compounds were obtained by precipitation of Cu(II) (from acetate solution) with various salicylic acid derivatives; the second stage was carried out to obtain the complexes of [Cu(Hsal)₂] with 1,10-phenanthroline by mixing of the two components in DMF-methanolic solution. The corresponding compounds were crystallized from the solution mixture at room temperature.

The FTIR spectra of compounds revealed the disappearance of the characteristic frequency band associated to the protonated carboxylic group after ligands coordination to the metallic center. From electronic spectra analysis could be observed the shifting of ligands specific transitions to lower energies do to a retrodative π -bond formation. The samples studied by single crystal X-ray diffraction have a triclinic structure, some of them being in monomeric (figure 1) form.

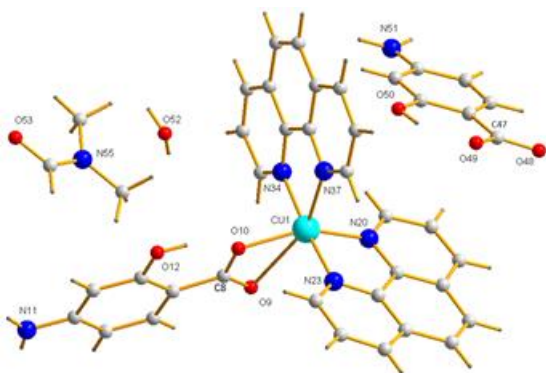


Figure 1. Molecular structure of [Cu(4-NH₂-Hsal)(phen)₂](4-NH₂-Hsal)·DMF·H₂O.

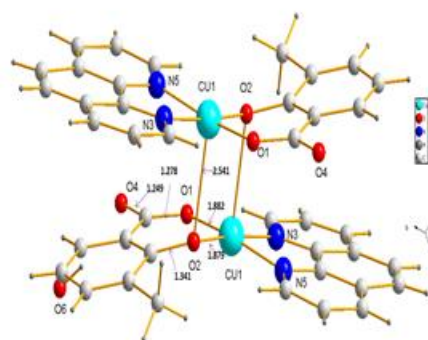


Figure 2. Molecular structure of [Cu(3-CH₃-sal)(phen)]·H₂O.

Acknowledgments: The authors thank to POSCCE-O 2.2.1, SMIS-CSNR 13984-901, Nr. 257/28.09.2010 project, the CERNESIM platform for infrastructure made available in carrying out of this work. Also, I. Bulimestru thanks to Erasmus Mundus IANUS II Program for the financial support.

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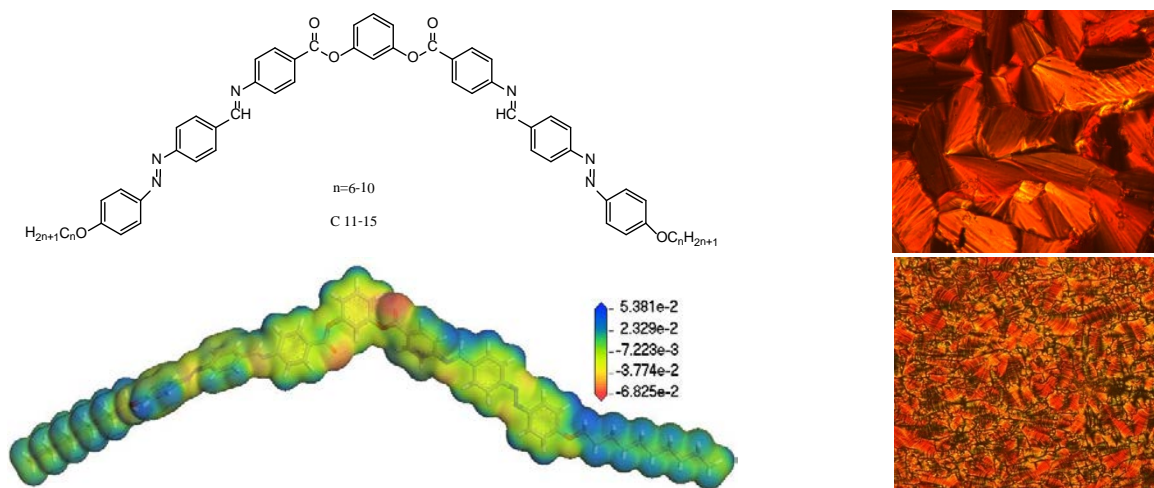
P10. Synthesis and Characterization of some Schiff Bases with Liquid Crystalline Properties

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In the last years, the *bent-core* molecules containing an azo linkage attracted considerable interest and have been investigated for their property of photochromism and photoisomerization upon UV irradiations. Most banana-shaped liquid crystals, reported up to now, correspond to five aromatic rings with a 1,3-phenylene central unit. The first smectic mesophases of banana shaped molecules had thermally sensitive azomethine connecting group between aromatic rings.



New *bent-core* liquid crystals have been synthesized by condensation reactions between chains of 4-((4-alkoxyphenyl)azo)benzaldehydes with the 1,3-phenylene bis(4-aminobenzoate) core. Their structure was varied by changing the length of the terminal chains, considering that the appearance of liquid crystalline behavior depends on the length of the flexible end chain. The synthesized compounds were characterized by ¹H-NMR and ¹³C-NMR, spectroscopy and by POM and DSC for evidence of liquid crystalline properties. The Schiff bases obtained presented enantiotropic behavior, with a broad stability of mesophases both on heating and cooling cycles. Also, they presented a good thermal stability in the existence range of the mesophases as evidenced by thermogravimetric studies.

Acknowledgements: C.-I. Ciobanu is thankful to the grant POSDRU/159/1.5/S/137750, Project "Doctoral and Postdoctoral Programs Support for Increased Competitiveness in Exact Sciences Research", for financial support.

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P11. New Coordinative Compounds of Co^{II}, Ni^{II} and Cu^{II} with 4-Nitrophenacyl-Phtalazinium Bromide. Synthesis and Characterization

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Three new coordinative compounds of Co (II), Ni (II) and Cu (II) with 4-nitrophenacyl-phtalazinium bromide (4nphz) have been prepared and studied both in solution and in solid state. The compounds were synthesized in solution by dissolving the ligand and the metal chlorides in acetone, then the stoichiometric mixtures were refluxed to room temperature. The crystallized compounds were obtained by slow evaporation of the solvent. The M(II): 4nphz molar ratio and stability of compounds were determined by spectrophotometric methods. Stability constants increased in $[\text{Ni}(\text{4nphz})_2] < [\text{Co}_2(\text{4nphz})_2\text{Cl}_2] < [\text{Cu}(\text{4nphz})_2]$ series. This behavior was confirmed by electronic spectra, magnetic susceptibility and electrical conductivity parameters.

The solid compounds were also characterized by thermogravimetric measurements, UV-Vis and FT-IR analyzes. Data obtained for the solids are in agreement with those identified from compounds studied in solution. The ligand coordinates to the metal cations by an oxygen atom and a nitrogen atom, suggesting the role of bidentate ligand of 4nphz and distorted square-planar geometry of the coordination center in relation with 4nphz.

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P12. Artifacts Degradation - Pollutant Factor of Soils From Urban Areas

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The term artifacts refers to anthropogenic materials present in the soil mass. These artifacts are represented by : (i) building materials (concrete, brick, ceramics, BCA, chalk, plaster etc.) in various stages of disintegration and physical and chemical alteration; (ii) wood fragments (cellulosic materials), in various stages of decay; (iii) glass and ceramics fragments; (iv) strongly altered ferrous and non-ferrous metal fragments (Fe, Cu, Al, alloys etc.); (v) plastic and textile materials; (vi) various materials: dross, ash, bitumen, pitch.

The physical and chemical degradation and alteration of the artifacts may release a range of chemical compounds with high toxic potential, which can temporarily accumulate in the soil at higher than maximum permissible values. In many cases, the diffusion and dispersion of these compounds is strongly restricted in technosols (as a direct consequence of textural anisotropy and the phenomenon of pedogeochemical segregation) so their persistence to high concentrations cause a number of disruptions of chemical and mineralogical equilibrium. These are translatable by the decrease of the ion exchange and buffering capacity, the destabilization of carbonates, oxyhydroxides of iron or organic-mineral complexes, etc. As an example, stable and highly toxic depositions of organic dyes have been found on the fragments of artifacts and mineral grains, such as titan yellow or dyes based on arsenic or antimony. These dyes are eliminated in the soil by the disintegration and physical and chemical alteration of different construction, textile or plastic materials. These dyes are stable under acid-reducing conditions and they cause a strong hydrophobisation of the soil aggregates by the adsorption on their surface with negative consequences on the air-water regime and physical and chemical characteristics of soils. Under alkaline-reducing or oxidant conditions, these dyes, alongside arsenic and antimony, can decompose and generate organic compounds with smaller molecular masses but with higher bioavailability and toxicity.

Another case, related to the above, is represented by the metallic and non-metallic fragments that occur with high frequency on most technosols. The corrosion of these materials releases high quantities of metallic ions, some of them with toxic potential. In poor drainage conditions the metallic ions can accumulate and in addition to the toxic effects can induce some strong chemical and mineralogical imbalances.

Our study approached the direct correlations between the mentioned phenomena and processes on soils from the urban area of Iași (Romania).

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