

MATNANTECH-CEEX 031 (2005-2008)
MATERIALS WITH LUMINESCENT PROPERTIES FOR
OPTOELECTRONIC DEVICES OPTOLUM

Project director: dr. Elisabeth-Jeanne Popovici

CONSORTIUM STRUCTURE

Coordinator :

CO-P1 Institute for Research in Chemistry, Cluj-Napoca, **ICCR**

Parteneri:

P2- National Institute for R&D for Microelectronics, Bucuresti, **IMT** (dr. Ileana Cernica)

P3- Babes Bolyai University Cluj-Napoca, **UBB** (prof.dr. Luminita Silaghi-Dumitrescu);

UBB-1 Faculty of Chemistry and Chemical Engineering, (prof.dr. Luminita Silaghi- Dumitrescu)

UBB-2 Electronique Microscopy Center (dr. Constantin Caciun)

P4- Institute of Physical Chemistry “ I.G. Murgulescu “ of the Romanian Academy , Bucuresti **-ICF**
(dr. Marilena Vasilescu)

P5- National Institute of R&D for Isotopical and Molecular Technologies, Cluj-Napoca, **INCDTIM**
(dr. Emil Indrea)

P6- SC NEONPRODUCT srl Cluj-Napoca-**NEON** (ing. Ioan Nemeth)

GENERAL OBJECTIVE

- ❑ Synthesis and characterisation of some luminescent materials (OPTOLUM) for optoelectronic devices and/or luminous (advertising) signs
- ❑ Obtaining of some new mixed coordination and organo-metallic compounds containing elements from 13th (Ga, In) and 15th (P, As) Groups utilisable as precursors for the preparation of thin films from A³B⁵ class
- ❑ Manufacture of some optoelectronic devices for lightning.

PROJECT PLAN

1st Stage. Scientifical and technical fundamentation of the project . Preliminary attempts for material synthesis (dec.2005). Partners: ICCRR; IMT; UBB-1; ICF

2nd Stage. Studies referring to the synthesis by wet-chemical route of synthesis intermediates and of some luminescent materials for optoelectronic devices (DOE) (iun.2006). Partners: ICCRR, UBB-2, INCDTIM

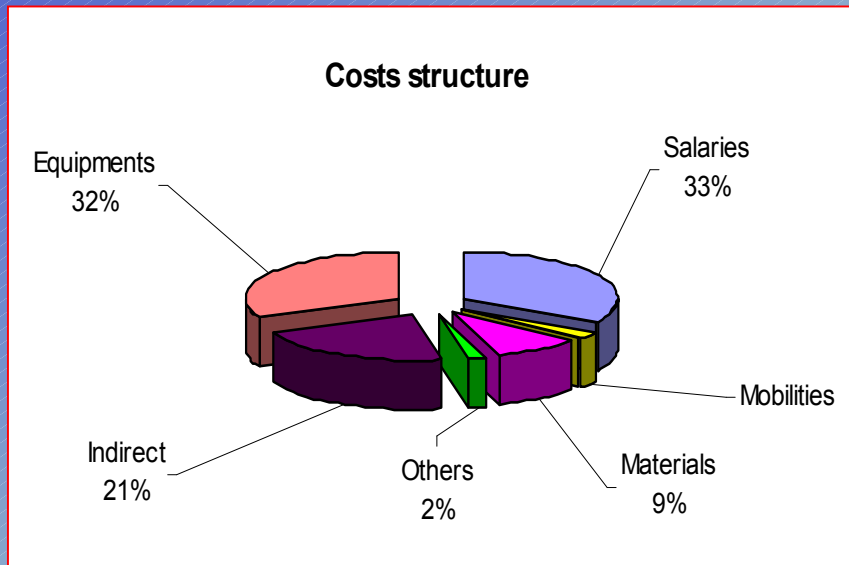
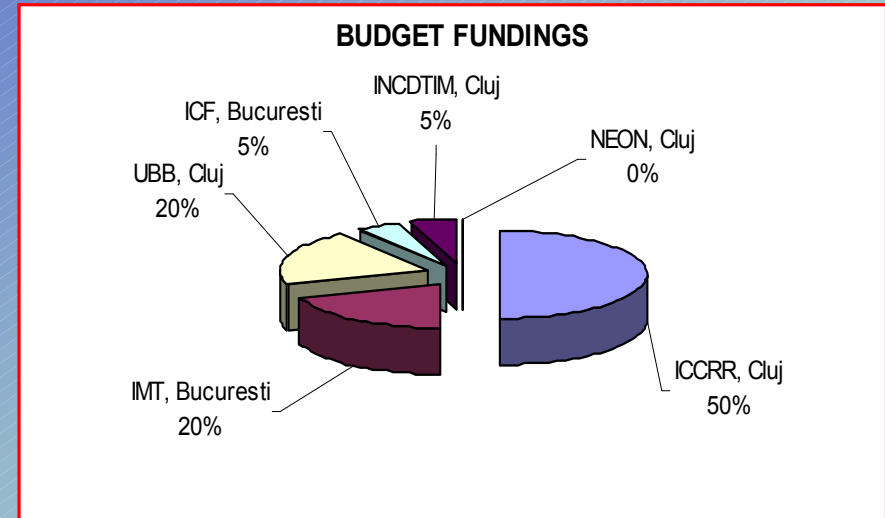
3rd Stage. Studies referring to the synthesis of some materials type OPTOLUM-1 and elaboration of the laboratory procedure for the preparation of the luminescent material; Elaboration of the LED technological (dec.2006). Partners: ICCRR, IMT; UBB-1, ICF; NEON

4th Stage. Studies referring to the preparation of some luminescent materials type OPTOLUM-2 and OPTOLUM-3 (sept.2007). Partners: ICCRR, UBB-2, INCDTIM; NEON

5th Stage. Elaboration and experimentation of the technology of preparation of PFLM-YAG with emission in the yellow spectral domain (apr.2008); Partners: ICCRR; IMT; UBB; INCDTIM; ICF; NEON

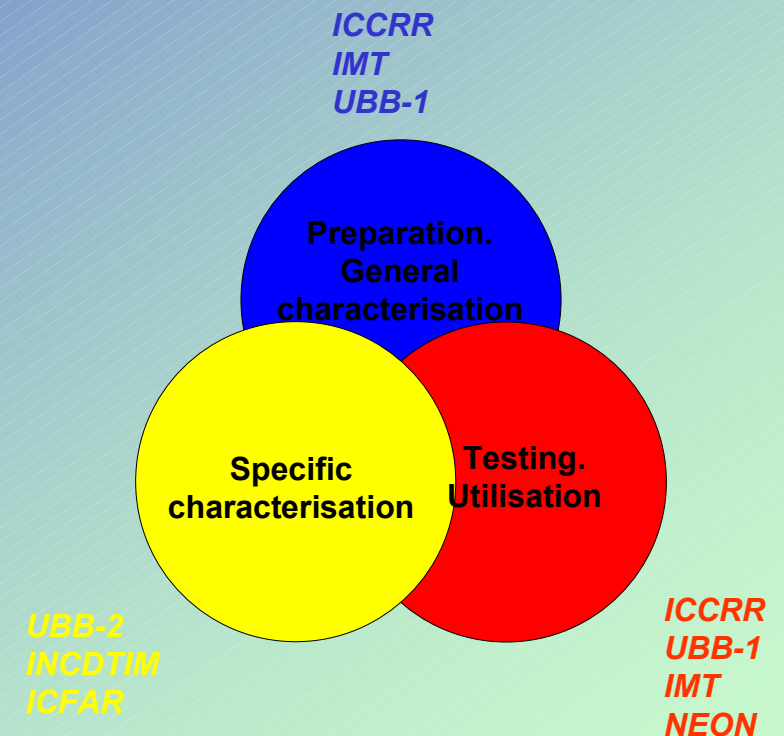
Where luminescent materials are based on the oxide systems: Y₂O₃-Al₂O₃-Ce₂O₃ (OPTOLUM-1); Y₂O₃-Al₂O₃-Ce₂O₃-M₂O₃, with M=Gd/Tb (OPTOLUM-2) and Y₂O₃-Ta₂O₅ (OPTOLUM-3);

FUNDINGS. COSTS STRUCTURE



PROGRAMMED RESULTS

- ❑ **Technology:** Technological process for the manufacture of a photoluminescent pigment (*PFLM-LED*) for *light emitting diodes*
- ❑ **Market/evaluation study:** *“Distribution o and demands of advertising signs containing LED^s and/or gas discharge tubes manufactured with blue-yellow-green*
- ❑ **Procedure:** Preparation of some organo-metallic compounds, precursors for *A³B⁵ thin films*;
- ❑ **Product:** Photoluminescent pigment with yellow/green emission (*PFLM-YAG*) for the manufacture of *light emitting diodes/ gas discharge tubes for advertising lightning* ;
- ❑ **Product:** Optoelectronic device based on LED^s with *PFLM-YAG*;
- ❑ **Technology:** Process/metodology for the manufacture of some light emitting diodes
- ❑ **Method:** *“Evaluation of the real photoluminescence characteristics (corrected emission /excitation spectra)”*
- ❑ *Increasing of the scientific/technical visibility of the research groups (results dissemination)*
 - Scientific works – Papers, Communications
 - Workshop- *“Materials with special optical properties”*
 - Technical results- Posters. Exhibitions.
 - Patents(s)



PROJECT PROGRESS

Up-to-now results

- There was achieved the information basis needed for the project progress.
- Basic-applicative researches were performed in the field of the synthesis of luminescent materials based on yttrium aluminate with garnet structure (*Annex #1*);
- Studies were performed aiming to the synthesis of some new organo-metallic compounds with groups 13th and 15th elements to be used as precursors for the preparation of A³B⁵ thin films (*Annex #2*)
- The technology for the manufacture of LED matrices was elaborated;
- There were synthesised and characterised some luminescent materials two of the research directions, i.e. cerium activated yttrium aluminate (PFLM-YAG:Ce) and yttrium tantalate (PRLM –YTa); materials are in intermediate performance stage;
- Material basis of partners was consolidated by: S-a consolidat baza materiala a partenerilor:
 - a) *R&D Equipments* : **Thermal Analyser*: TG-DTG-SDTA system and DSC calorimeter (Mettler-Toledo); Spectrofluorimeter JASKO 6500 (*Annex #3*)
 - b) *Laboratory instruments/ devices*;
 - c) *PC equipments, soft-wares*
- Scientific results were disseminated as papers (submitted) or communication (*Annex #4*)

Studies referring to the synthesis of somer luminescent materials based on yttrium aluminate doped with cerium, with garnet structure (OPTOLUM-1 class)

Elisabeth-Jeanne Popovici, Amalia Hristea, Florica Imre, Rodica Puscas, E. Indrea, C. Craciun

Introduction

Aim of the study

Synthesis and characterisation of one luminescent pigment (PFLM) for optoelectronic devices (OED) type LED or gas discharge tubes for advertising signs.

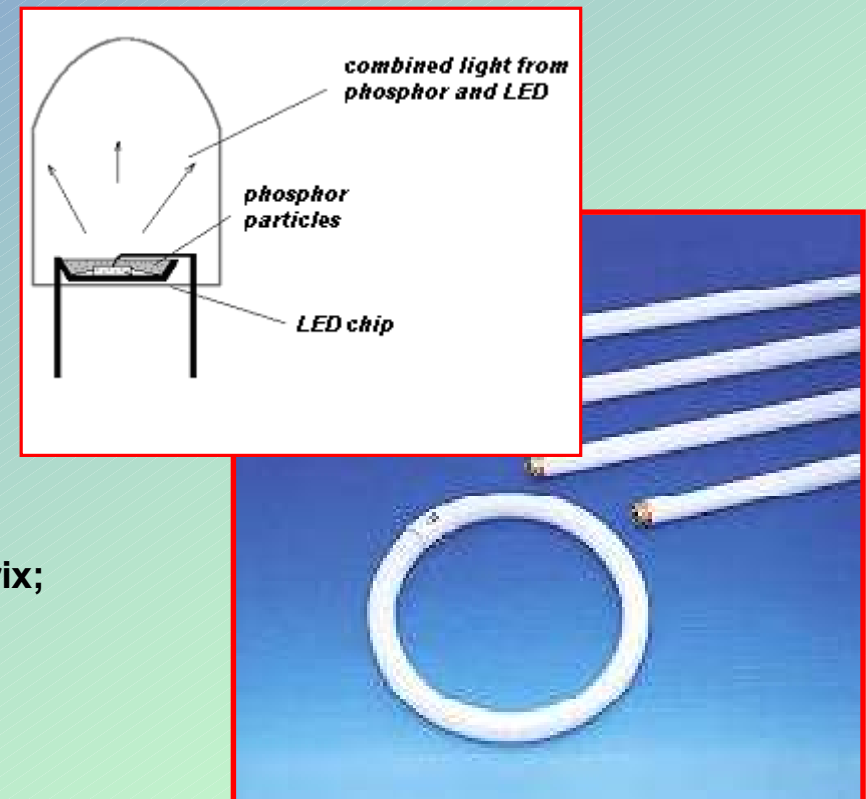
PFLM demands for use in OED

□ Luminescence characteristics:

- Colour purity (RGB);
- High brightness/ emission intensity;
- High efficiency;
- PL/CL/RL stability on long term;
- Compatibility with the chip optical characteristics;

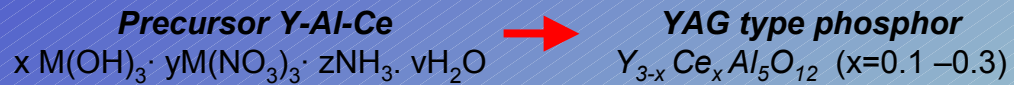
□ Powder characteristics:

- High degree of crystalline purity;
- High crystallinity;
- Homogeneous distribution of activator into the matrix;
- Spherical shape for powder particles;
- Reduced particle sizes;
- High disperdability;
- Low porosity etc.



Experimental part

Sample preparation

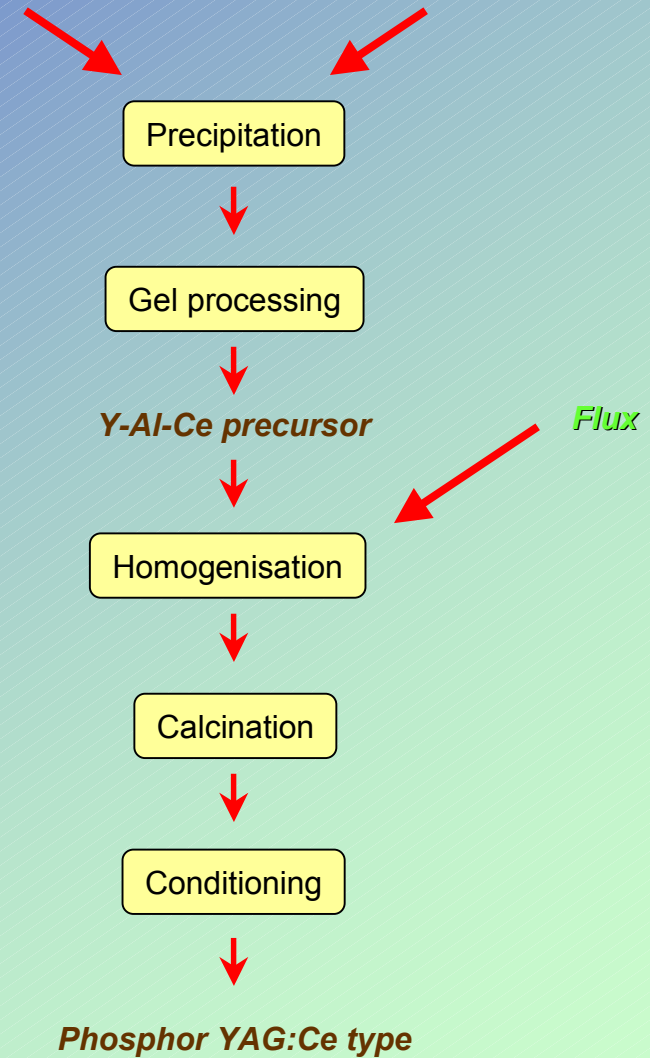


Sample characterisation

- **Precursor:** FTIR; SEM, TEM; TD-DTG-DTA
- **Phosphor:** XRD; FTIR; SEM, TEM; PL

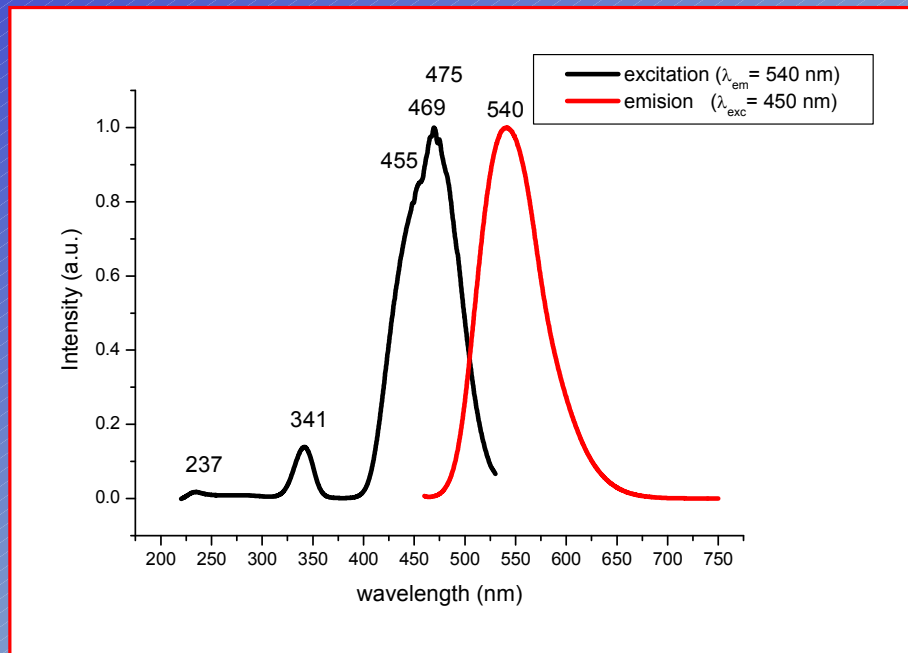
Mixture $M(NO_3)_3$

Precipitation agent

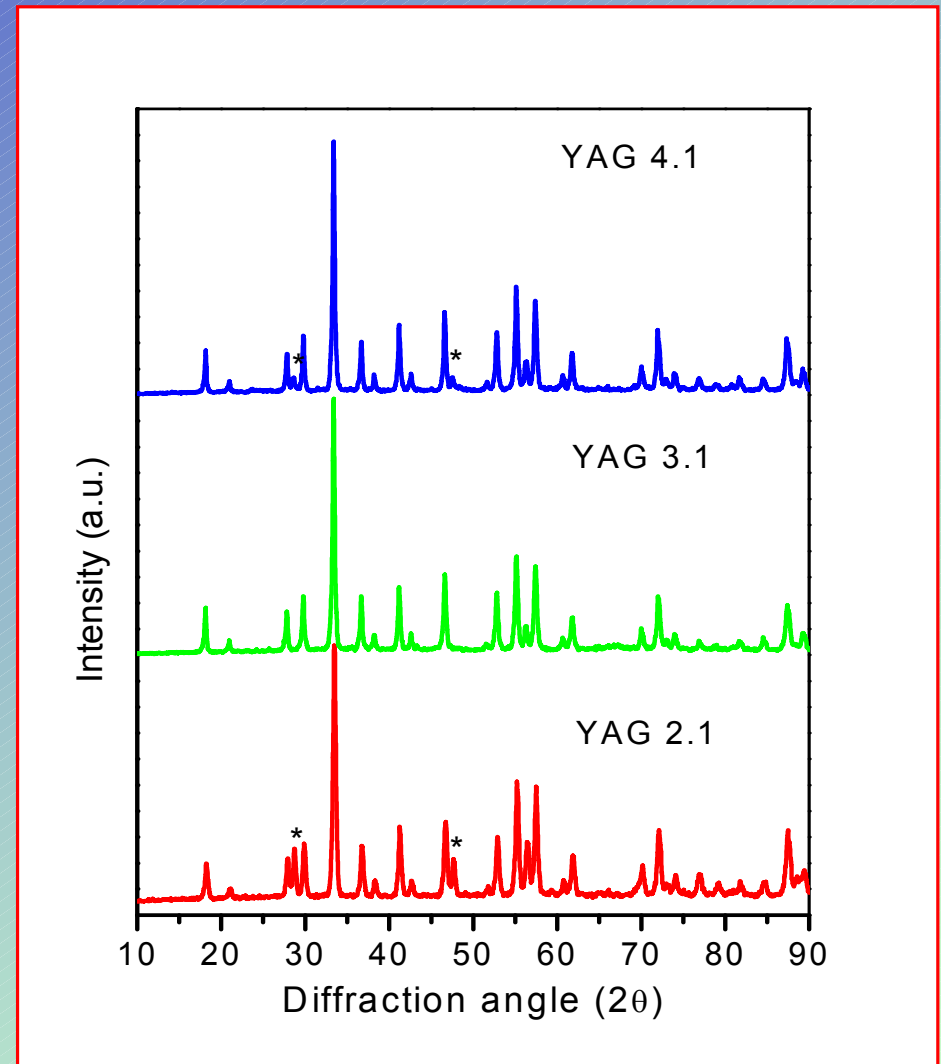


Results

Photoluminescence (left) and crystalline structure (right)

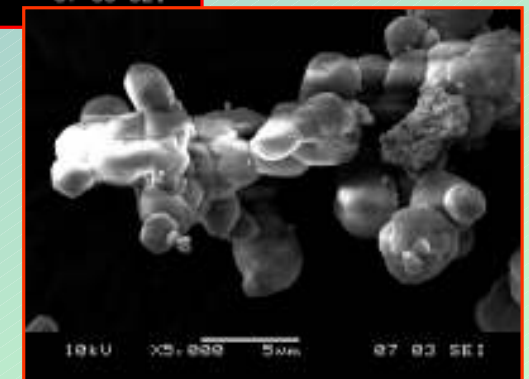
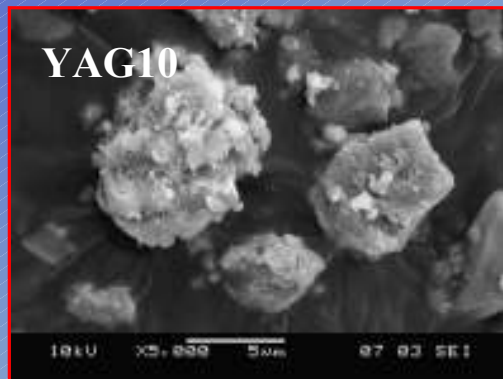


Excitation spectrum (left) and emission spectrum (right) of sample YAG10.1



XRD patterns of phosphors prepared with ammonium carbonate (YAG 2.1), urea (YAG 3.1) and ammonia (YAG 4.1.)

Morphology and particle dimension
scanning electronic microscopy



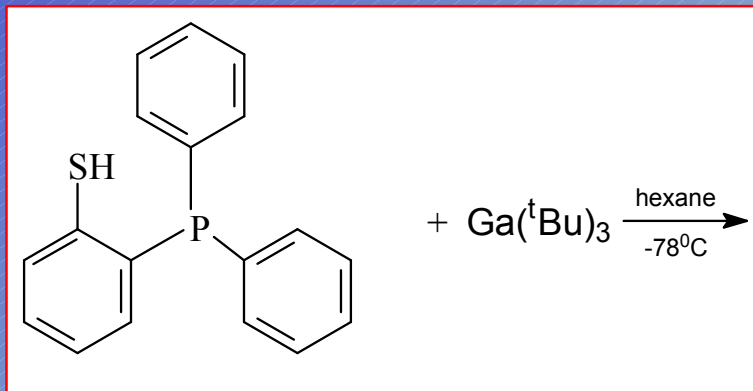
SEM images of the precursor prepared with urea (left) and of the corresponding phosphor samples synthesised in variable thermal regime (right)

Studies referring to the preparation of some new organometallic compounds, precursors for thin films based on A^3B^5

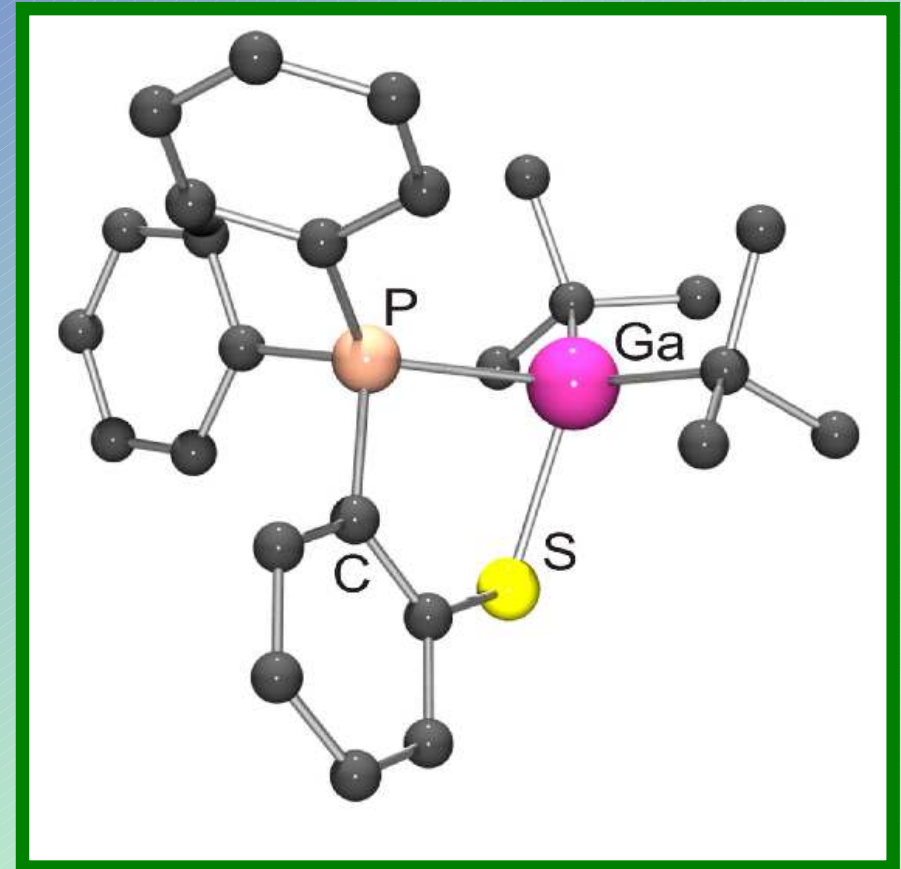
Luminita Silaghi- Dumitrescu, Ana-Maria Valean et al.

Aim of the study

Synthesis of some mixed organometallic compounds with Groups 13th (Ga, In) and 15th (P, As) elements



Phosfino-aryl-thiol- ligands $(\text{tBu})_2\{(\text{SC}_6\text{H}_4\text{-2-PPH}_2)\text{-}\kappa\text{S,P}\}$ $\xrightarrow{\text{MOCVD}}$ GaP



CONSOLIDATION OF THE TECHNICAL BACKGROUND OF COORDINATOR ICCRR



**High resolution spectrofluorimeter
JASKO FP-6500 Wavel**



Thermal Analyser (Mettler-Toledo TGA / SDTA850)

RESULTS DISSEMINATION

Annex #4

lucrari comunicate si/sau /publicare:

- “Morpho-structural and luminescent investigations of niobium activated yttrium tantalate powders” (Amalia Hristea, Elisabeth-Jeanne Popovici, Laura Muresan, Rodica Grecu, Anders Johansson and Mats Boman) \Leftrightarrow *Optical Materials (submitted)*
- “The influence of flux nature on $YTaO_4:Nb$ synthesis,, (Amalia Hristea , Elisabeth-Jeanne Popovici, Laura Muresan, Maria Ștefan, Rodica Grecu and Marilena Vasilescu \Leftrightarrow *Studia Universitatis Babes Bolyai Cluj-Napoca(in press)*;
- “Luminescent properties of $YTaO_4:Nb$ thin films prepared by pulsed laser deposition”, Amalia Mesaros, Oscar Alm, Elisabeth-Jeanne Popovici, Maria Ștefan, Laura Muresan, Mats Boman) \Leftrightarrow “*ROMOPTO 2006 (august Sibiu)*
- Reactivity of tetraphenyldiarsineoxide and sulphide, $(Asph)_2E(E=O,S)$. The supramolecular architecture of $[Ph_2AsBr_8]$ formed by bromination of $(Asph_2)_2 S$ (Luminita Silaghi-Dumitrescu et all) \Leftrightarrow *International Conference on Organometallic Chemistry XXII, Zaragoza, Spania (22 iulie-28 iulie 2006)*;
- “Towards novel coupling agents containing unsaturated organogermane fragments”(Gabriela Nemes et.all) \Leftrightarrow *International Conference on Organometallic Chemistry XXII, Zaragoza, Spania (22 iulie-28 iulie 2006)*;
- “FTIR studies on niobium activated yttrium tantalate phosphor (Rodica Grecu, Elisabeth-Jeanne Popovici, Amalia Hristea, Laura Muresan & Emil Indrea \Leftrightarrow *International Conference of the Chemical Societies of the South-East European Countries - ICOSECS 5 (OHRID, Macedonia, Sept.2006)*.
- *Photoluminescence Properties of Niobium and Rare Earth Activated Yttrium Tantalate Powders (Amalia Mesaros, Elisabeth-Jeanne Popovici, Laura Muresan& Rodica Grecu)*
- *Spectroscopical Investigation Of Nanostructured Europium Activated Yttrium Oxide Powders, Laura Muresan, Elisabeth-Jeanne Popovici, Amalia Mesaros, Maria Stefan, Rodica Grecu and Emil Indrea, Advanced Spectroscopies on Biomedical and Nanostructured Systems, NANOSPEC Cluj-N. Sept.2006.;*