LCC analytical platform

FIT24

Y. Coppel et L. Vendier

→ 08/07/2024

Le LCC en chiffres : le personnel

Direction :

Azzedine Bousseksou, DU
Anne-Marie Caminade et **Eric Manoury** (DUA)
Christian Lorber, SDU, Administration

Le LCC (06/2019) :

41	chercheurs (40 CNRS dont 4 DREM et 1 INSERM)
37	enseignants-chercheurs (29 UPS dont 1 PREM, 8 INPT dont 1 PREM)
44	ITA CNRS
9	BIATSS (4 UPS, 5 INPT dont 4 quotité LCC 50 %)
16	post-docs
60	doctorants
~ 40	stagiaires divers (1er semestre)

En moyenne, plus de 270 personnes présentes au LCC …

« Ouverture du LCC dans les deux sens ! »

Coût du LCC ~ 17 M€/an
(masse salariale 16 M€/an, fonctionnement & infra ~ 1 M€/an)

Le LCC : la recherche

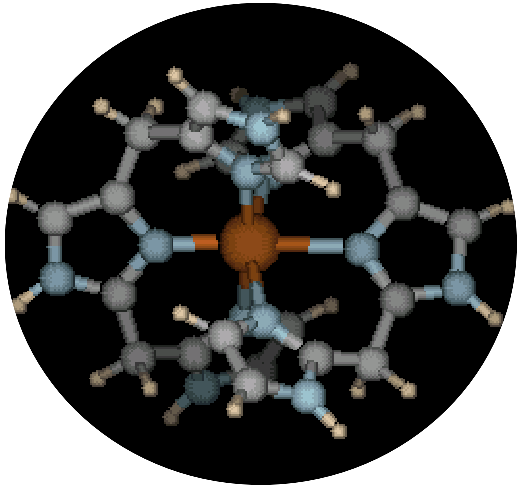
Coord. Chem. Rev. 308 (2016)

La recherche développée au LCC possède une unité thématique qui s'articule autour de la chimie des métaux et des hétéro-éléments et que l'on peut définir comme :

- Chimie de coordination

- Chimie organométallique

- Chimie des Hétéro-éléments



- Chimie et santé

- Chimie et matériaux

- Chimie et catalyse

Les domaines d'application sont la catalyse, la synthèse organique, la synthèse macromoléculaire, les matériaux moléculaires, les nanosciences, la chimie bio-inorganique, les médicaments, …

LCC : Organisation, ~ 270 membres

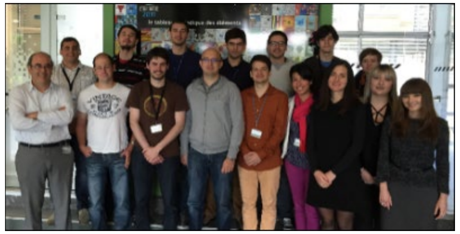
13 équipes de Recherche

LCC : Organisation

www.lcc-toulouse.fr





Chemistry & Catalysis ***(4 Teams)***

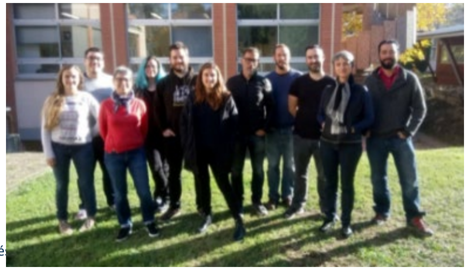
Ingénierie Moléculaire des Pré-Catalyseurs : **V. CESAR**
Catalyse et Chimie Fine : **P. SERP, J. VOLKMAN**
Ligands, Architectures Complexes et Catalyse : **E. DEDIER & E. MANOURY**
Activation de Petites Molécules : **S. BONTEMPS & A. SIMONNEAU**


Chemistry & Materials ***(6 teams)***

Ingénierie des Nanoparticules Métalliques : **K. PHILIPPOT**
Dendrimères et Hétérochimie : **A.-M. CAMINADE**
Matériaux Moléculaires Commutables : **A. BOUSSEKSOU**
Molécules et Composites pour l'optique: **I. MALFANT**
Matériaux Moléculaires et Supramoléculaires : **J.-P. SUTTER**
Nanochimie, Organisation et Capteurs : **M. KAHN**

Chemistry & Health ***(3 Teams)***

Alzheimer et Amyloïdes : **C. HUREAU**
Chimie et Biologie Médicinale pour l'Oncologie : **H. GORNITZKA & O. CUVILLIER**
Nouvelles Molécules Antipaludiques et Approches Pharmacologiques : **F. BENOIT-VICAL**




LCC analytical platform

Operating mode

Autonomous operation under the responsibility of ITA/BIATSS staff
Internal user committees
Platform associated with the Institut de chimie de Toulouse (UAR 2599)

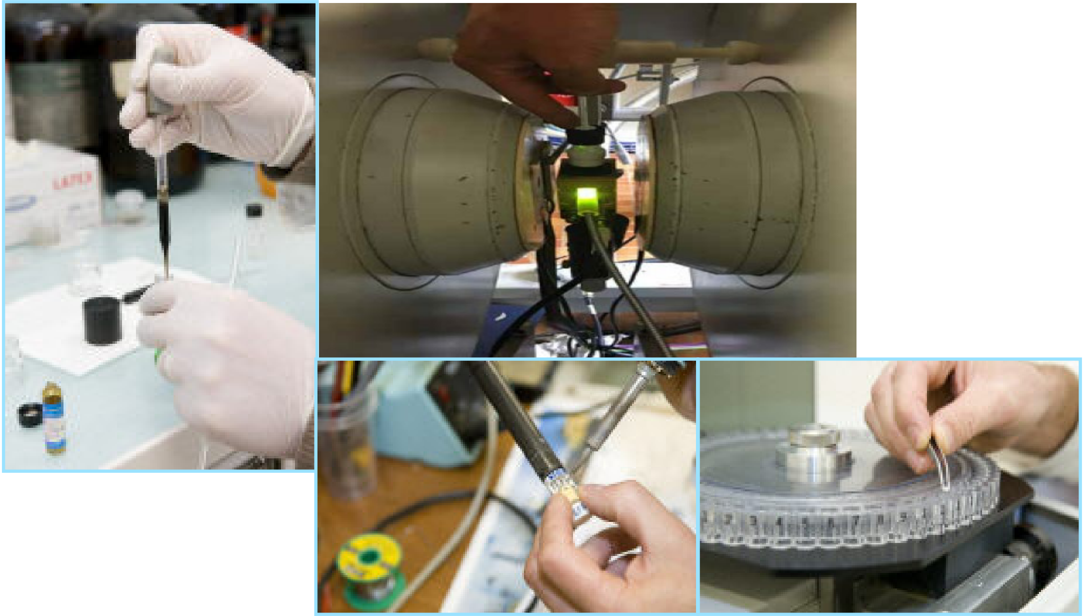
Accessibility to other laboratories

Open to external laboratories (private or public)
Fee schedule for analysis on each device
One full-cost certified platform: NMR

Services

Self-service after training (mostly for internal users)
Service contracts or analysis by quotation
Analyses, interpretation of results, scientific collaborations
Formations (CNRSFormation entreprise, doctoral school, …)

www.lcc-toulouse.fr



01

Chemical composition

Elemental Analysis CHN/S

Staff

1 CNRS Technician (Isabelle Borget)

Equipement

Flash combustion analyser Perkin-Elmer 2400 Serie II:
- combustion temperature >1800°C
- operates in two modes : CHN or CHNS
- helium Carrier Gas
- sample : powder and non volatile liquid (mass required : 10 mg)

Applications

Elemental analyzer for organic materials and organic part in inorganic materials:

- certifies the chemical composition and purity of synthetic products
- validates their manufacturing process
- gives the organic element content of natural substances and unknown materials for research or quality controls
- determines weight percent (wt%) of Carbon, Hydrogen, Nitrogen routinely / and Sulfur once a year

Accuracy of ± 0.3 % and precision of± 0.2 % (duplicate determination)

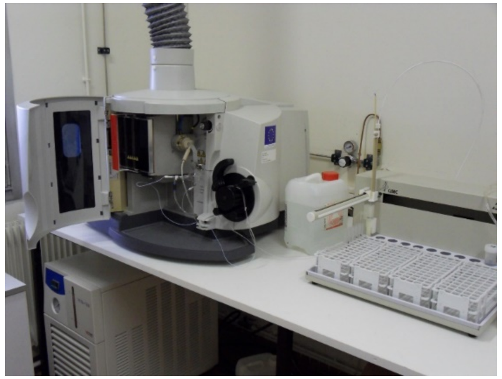
ICP –AES (Inductively Coupled Plasma – Atomic Emission Spectrometry)

Staff

CNRS engineers (Alain Moreau)

Equipement

ICP-AES Thermo Icap 6300 Duo

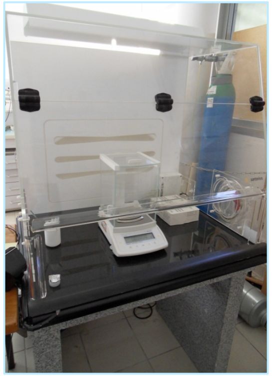


Applications

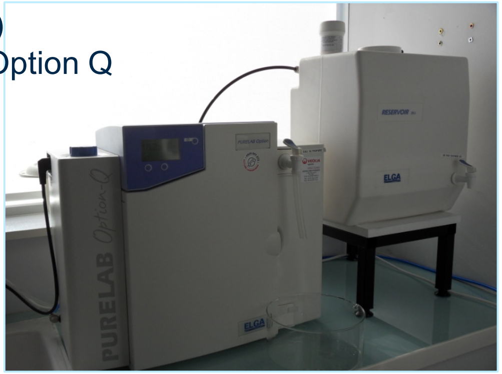
Analysis of solid samples or aqueous solutions

Determination of the mass concentration of one or more elements in a sample.

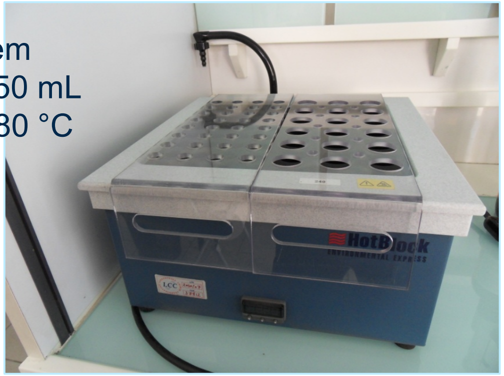
Analysed elements: Ag, Al, Au, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, Ir, K, Li, Mg, Mn, Na, Ni, P, Pb, Pd, Pt, Ru, Rh, Sr, Zn



Ultrapure water
(18.2 MΩ)
Veolia - Purelab Option Q



« Wet » mineralisation
(HNO_3, HCl, H_2SO_4)
Hotblock Digestion System
50 positions - tubes 15 and 50 mL
programmable from 25 to 180 °C



Mass spectrometry: LC-MS and GC-MS

Staff

1 CNRS Technician (Isabelle Borget)

Equipement

LC Dionex Optima 3000 - MS ThermoScientific LCQ Fleet (ion trap)
- ESI ionization mode
- Auto sampler, with or without column (not supplied)
- Direct infusion

Shimadzu QP2010 Ultra
- Column GC ZB-5MS: slighty polar, T_{max} = 330°C
- Wide range of analytes
- Electron impact ionization (EI)
- Direct injection (DI)

Services/Applications

Self-service use

LC-MS: m/z ratio (mass to charge) ; MS+ and MS- detection; UV-Vis detection
ionizable and/or chromophore compounds
GC-MS: Reaction/catalysis analysis ; simple organic compounds MS characterization ; spectra Library

02

Molecular structure analysis

Nuclear Magnetic Resonance

Staff

4 CNRS engineers (Christian Bijani, Antoine Bonnet, Yannick Coppel*, David Paryl)

Spectrometers

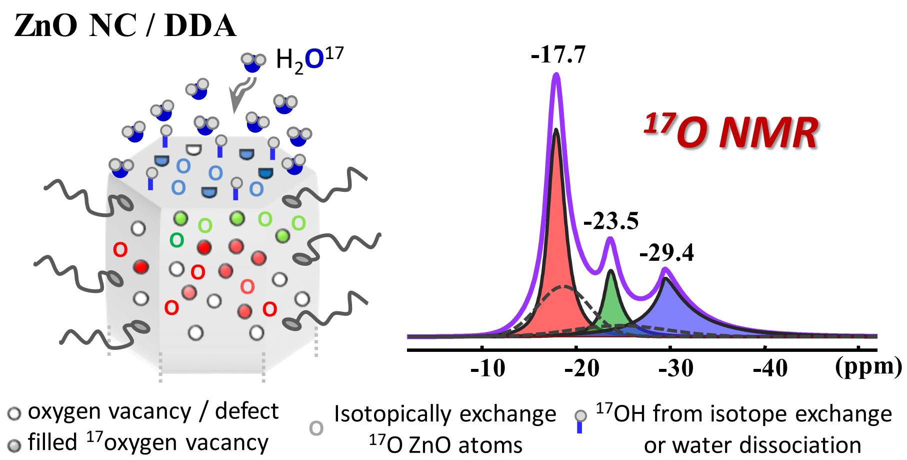
6 NMR spectrometers (Bruker) from 7.0 to 14.1 T (300 to 600 MHz – ^{1}H)

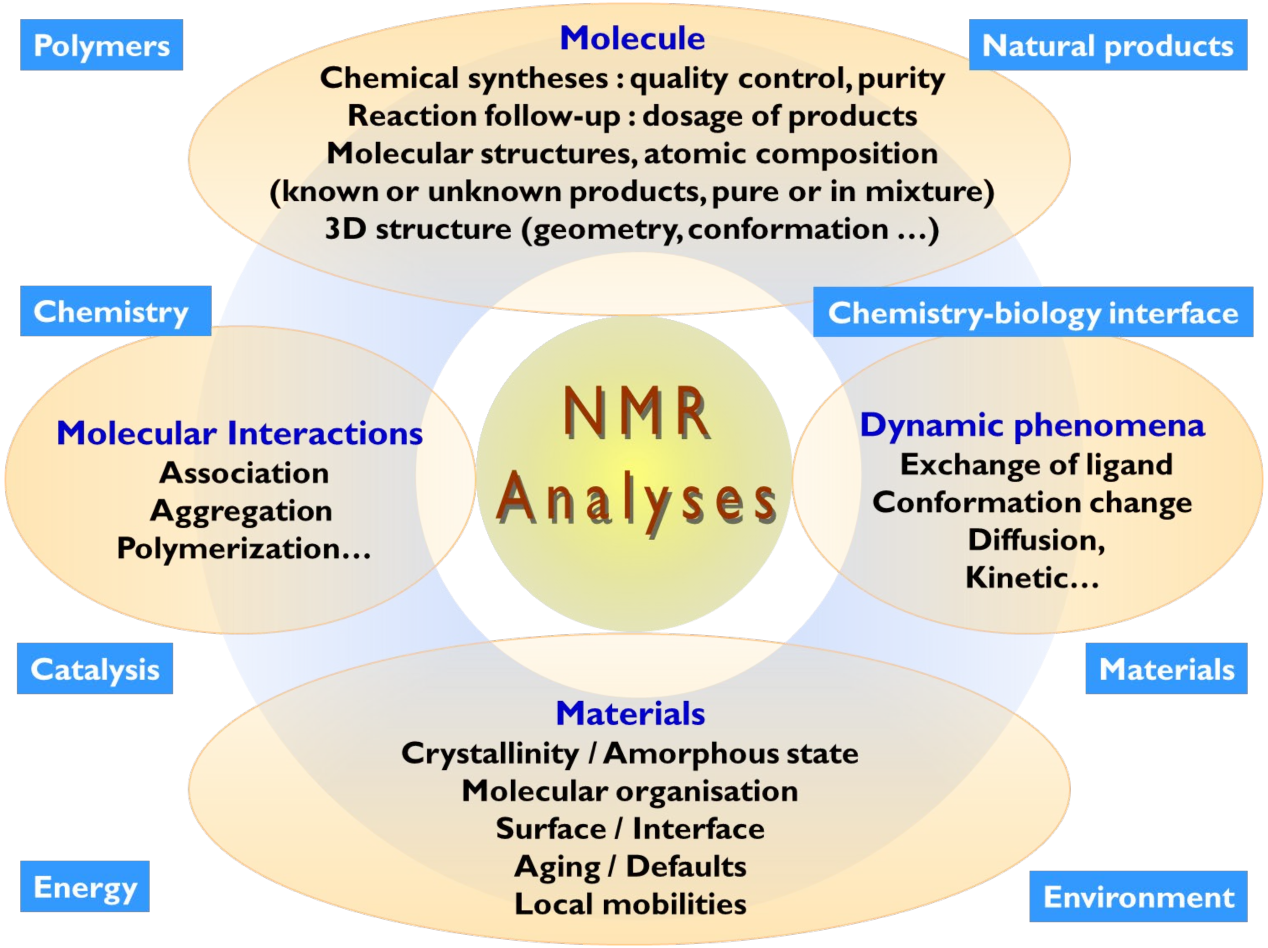
- **For sample in gas, liquid, gel and solid phases**
- **5 and 10 mm NMR tubes and 1.3 to 4 mm NMR rotors**
- **Variable temperature capabilities (± 100°C)**
- **Hyperpolarisation with parahydrogen (NEW !!)**

Nuclear Magnetic Resonance

Applications

- **Organic, organometallic and inorganic molecules**
- **Special know-how for non-standard nuclei:**
  ^{2}H, ^{7}Li, ^{11}B, ^{17}O, ^{27}Al, ^{29}Si, ^{51}V, ^{103}Rh, ^{109}Ag, ^{119}Sn, ^{195}Pt …
- **Study of dynamic phenomena**
- **Catalytic studies**
- **Molecular materials and polymers**





03

Material Analysis

Atomic Force Microscopy and Raman Spectroscopy

Staff

1 CNRS Technician (Marine Tassé)

Equipements

XPLORA Raman spectrometer (Horiba):
- Laser sources: 532, 638 and 785 nm.
- Temperature: 80 K to 800K
- High-pressure: up to 40 kBars

INVIA Raman spectrometer (Renishaw):
- 532 nm laser source
- Measurements for sensitive samples
- Fast Raman mapping

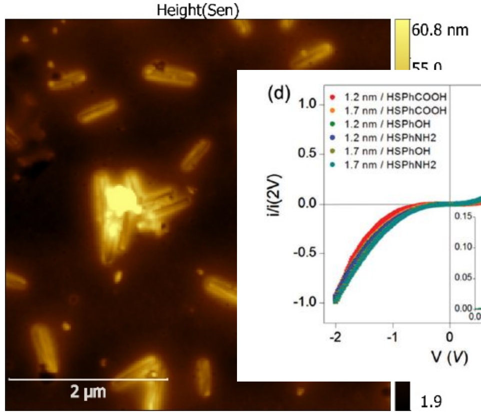
Smart SPM 1000 AFM microscope (Horiba):
- Air topography measurements
- Various electrical measurements (KFM, C-AFM, EFM).

RAMAN-XPLORA-HORIBA

AFM-SmartSPM1000-AIST-NT

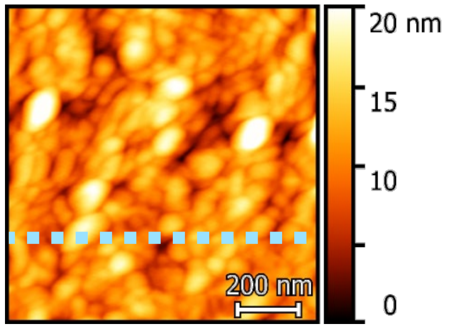
RAMAN-INVIA-REINISHAW

Atomic Force Microscopy and Spectroscopy Raman

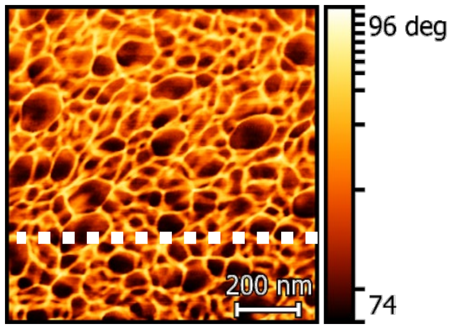


Conductive C-AFM- I(V) curves

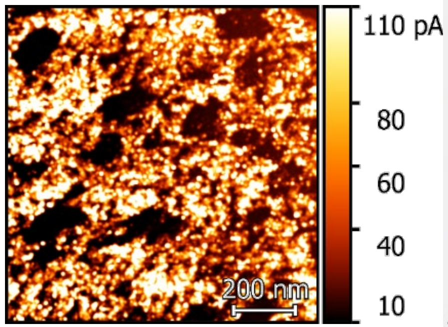
Topography AFM

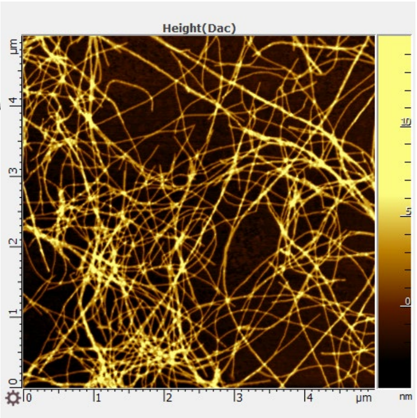


Phase, EFM mode

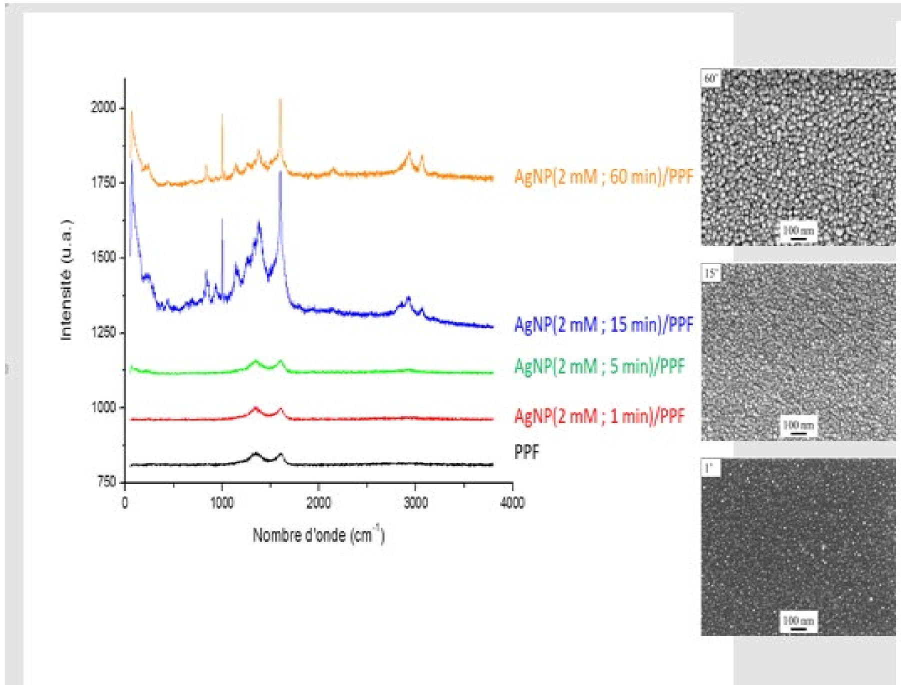


Current-C-AFM



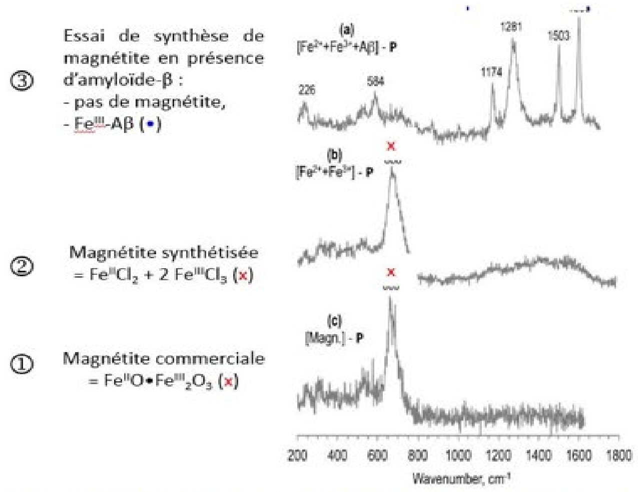


Biological Fibers



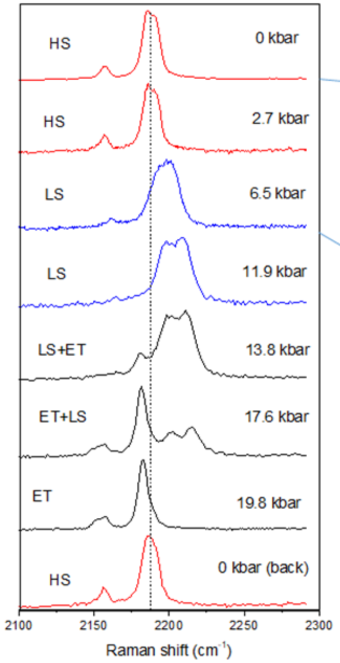
-Variation de l'organisation de particules Plasmoniques-Maxime Puyo-Equipe T-

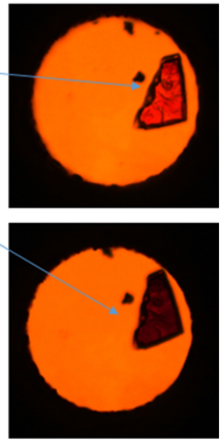
La magnétite n'est pas impliquée dans la maladie d'Alzheimer



La spectroscopie Raman montre que la magnétite ne se forme pas en présence d'amyloïde-β

Gumpelmayer, M., Nguyen, M., Molnar, G., Bousseksou, A., Meunier, B., & Robert, A. (2018). Magnetite Fe_3O_4 has no intrinsic peroxidase activity, and is probably not involved in Alzheimer's oxidative stress. *Angew. Chem., Int. Ed.*, *57*, 14758 –14763





Electronic transitions under pressure in an Fe(II) complex Livia Getzner Team P

Electron Microscopy

Staff

1 UPS engineer (Vincent Collière)

Microscopes

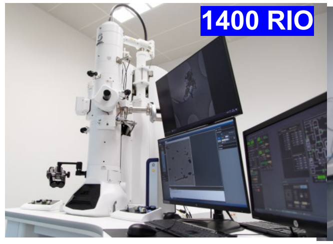
Use of 7 electron microscopes of the Raimond Castaing microcharaterization center (UAR 3623):

- 5 transmission electron microscopes (TEM 100 to 200kV).
- 2 field emission scanning electron microscopes (SEM).

Services

- Samples preparation
- Characterization of materials synthesized in the LCC
- Results analysis

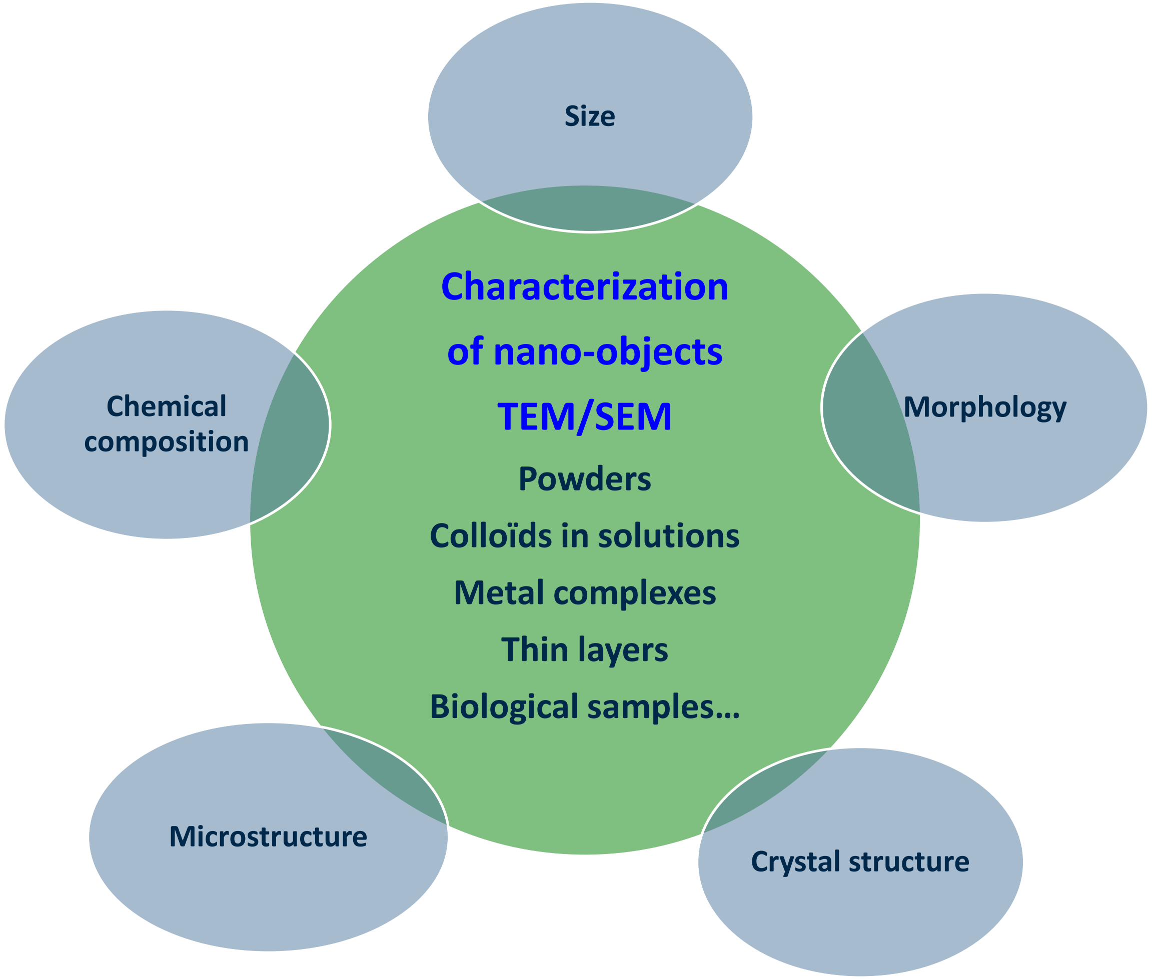
5 Transmission Electron Microscopes



2 Scanning Electron Microscopes

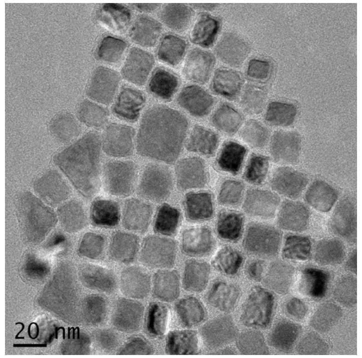
Electron Microscopy

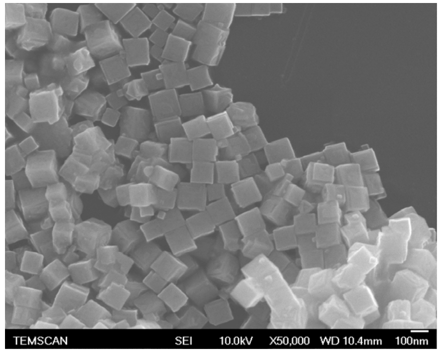
Applications



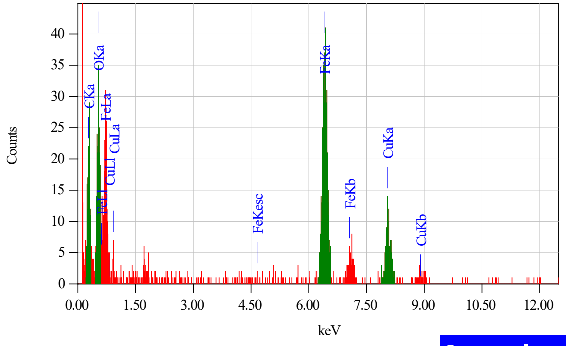
Fe Nanoparticles

Size, Morphology

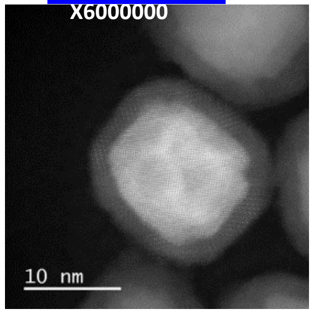




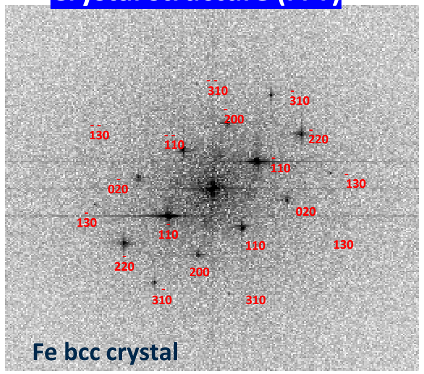
Chemical composition



STEM-HAADF



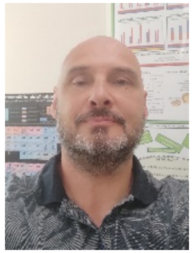
Crystal structure (FFT)



Thermal and surface area measurements

Staff

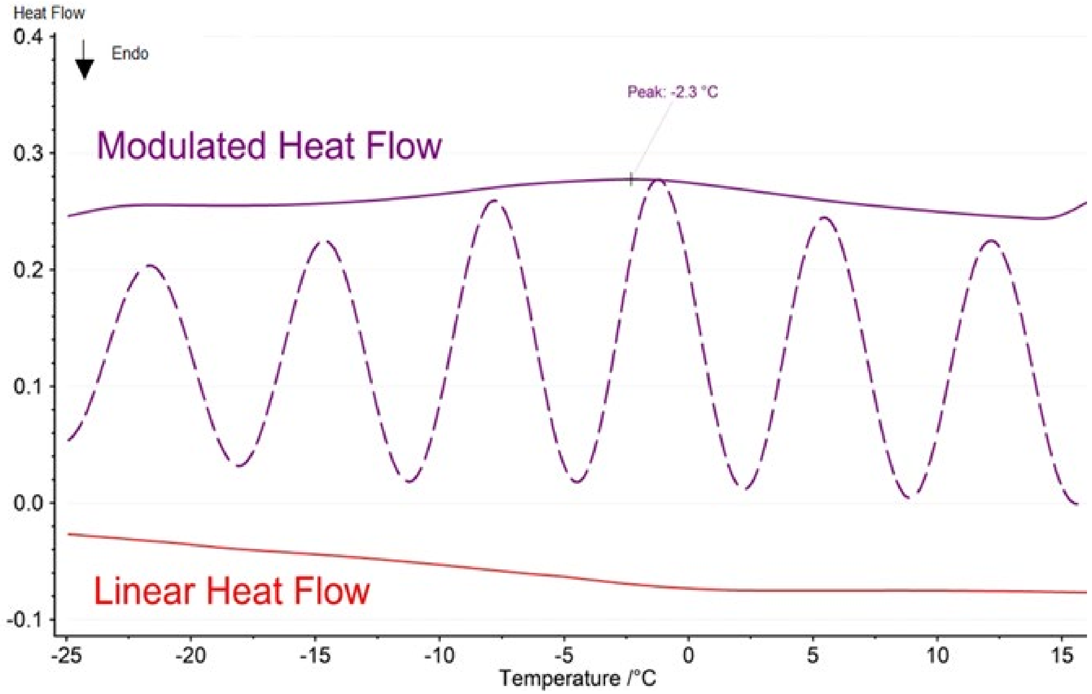
CNRS engineers (Jean-François Meunier, Baptiste Martin)



Apparatus

TGA: *ATG/DSC 3+* Mettler Toledo (20-1100°C)
DSC: *DSC3500 Sirius* NETZSCH (-150°C to 500°C)
BET: ASAP 2020 Micromeritics

- **Suitable for solids and liquids**
- **Several gases for TGA (Ar, Ar+H_2 ,CO_2, Air, N_2)**
- **Temperature modulated DSC experiments (NEW !!)**
- **Physisorption of liquid N_2 and suitable for chemisorption**



TM-DSC thermorgramme

 08/07/2024

Thermal and surface area measurements

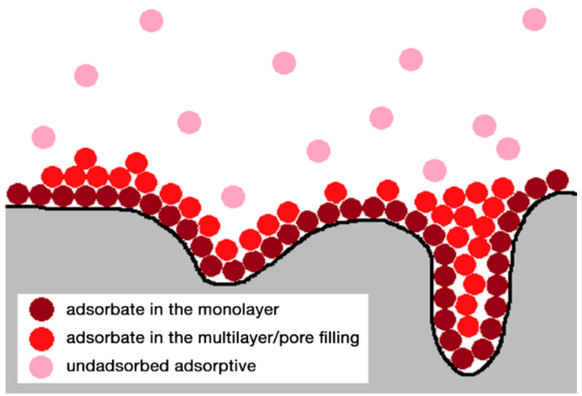
Applications

- **Phase transition in polymers materials, metals, molecular complexes**

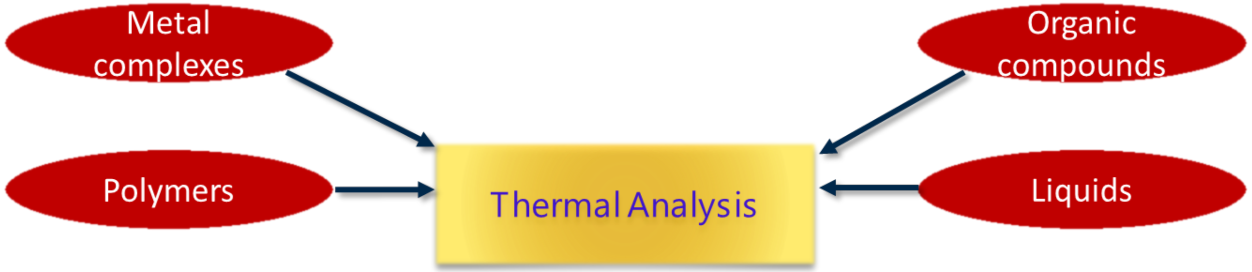
- **Measurement of weak thermal events thanks to TM-DSC**

- **Thermal stability under several atmospheres**
  H_2, CO_2, Air, N_2, CO_2, Ar,

- **Determination of specific surface with several methods**
  BET, Langmuir, t-plot, BJH



Physisorption of N_2 on pores



- Glass transition
- Reversible process
- Phase transition
- Modualtion measurements

DSC

- Thermal Stability
- Degradation Process
- Oxydation Sensitivity
- Solvatation rate

TGA

04

Physico-chemical properties

Electrochemistry

Staff

2 CNRS engineers (Alix Sournia-Saquet*, Alain Moreau)

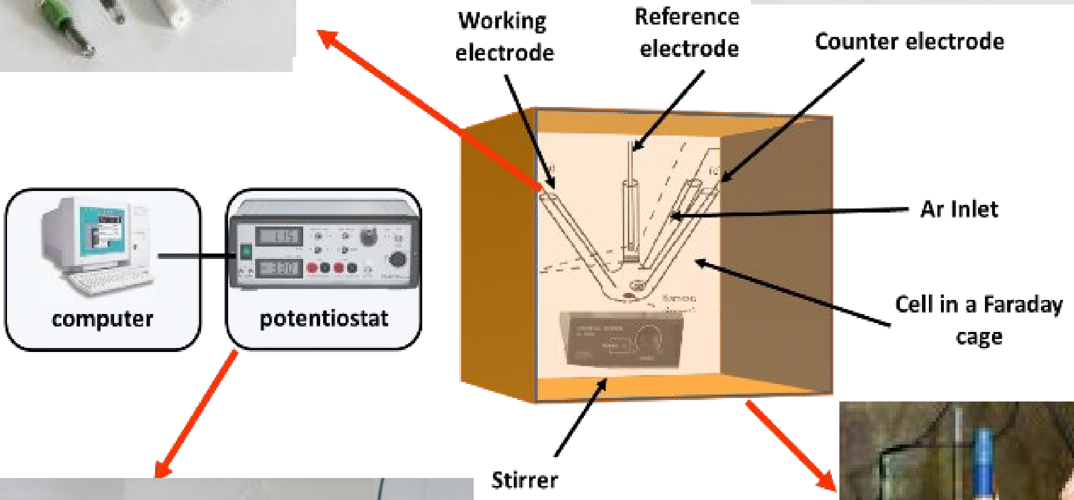
Potentiostat/galvanostat

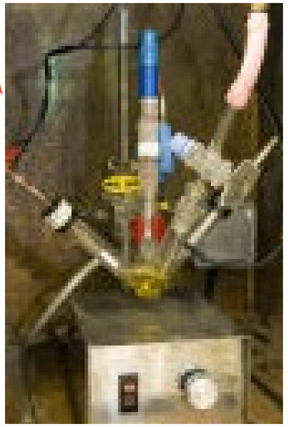
3 potentiostats-galvanostats:
- compliance: 30 -100V
- current 250mA- 6V
- EIS Frequency up to 10 MHz

pH-meter – conductivity meter

Cells: specific cell for inert environment
different volumes: 8 ml to 100 mL

Significant choice of electrodes:
different metals shapes and surfaces
rotating disk electrodes
specific electrodes for the powder

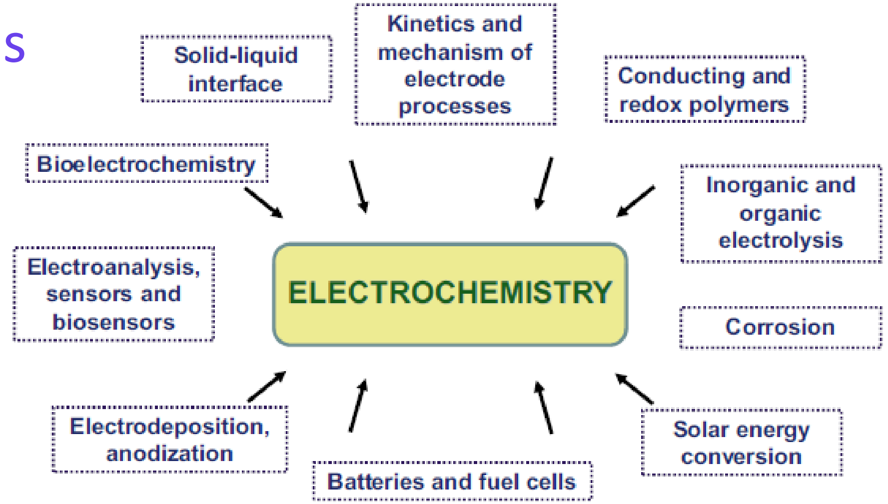




Variable temperature capacabilite: 0-100°C
Polishing machine : to clean the electrode surface
Faraday cage + inverter: to avoid electrical interference

Electrochemistry

Applications



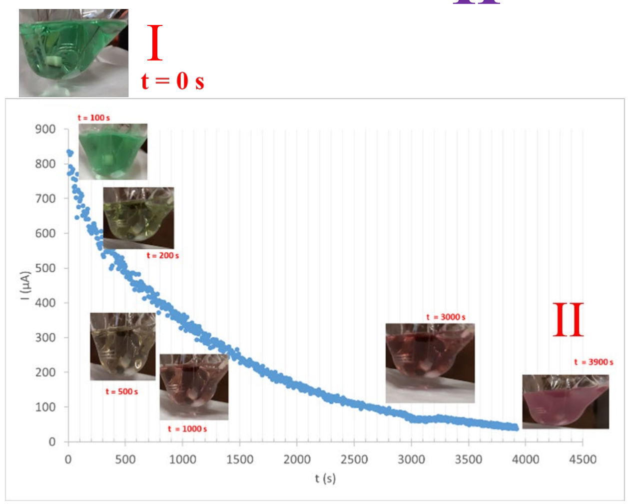
Journal of Solid State Electrochemistry (2024) 28:1007–1013

Experiments and specificities

Potentiometric titration, pHmetry, conductivity
Electrochemical experiments in aqueous or non aqueous solvents with ohmic drop compensation
Voltammetry cyclic or linear sweep voltammetry, normal or differential pulse voltammetry, square wave voltammetry, etc..
Chrono methods: chronomaperometry , chronopotentiometry
Electrochemical studies on insoluble products with cavity microelectrode or carbon powder microelectrode
Electrochemistry in gloves box (NEW)

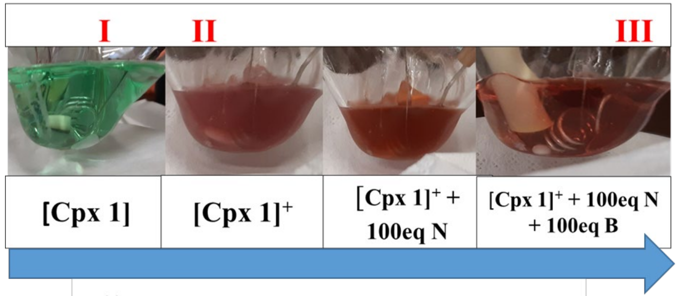
Electrochemical study of complex:
before and after electrolysis and additions

I II III



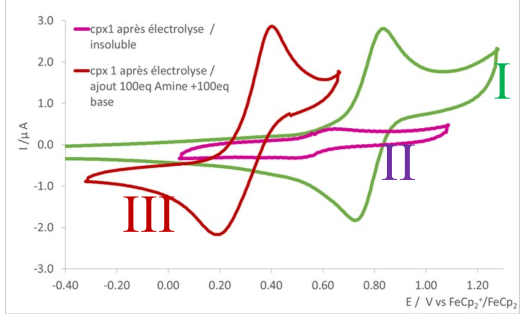
CHRONOAMPEROMETRY:
Electro-oxidation of cpx1

Conversion
[Cpx1] ➡ $[Cpx1]^+$



CYCLIC VOLTAMMETRY

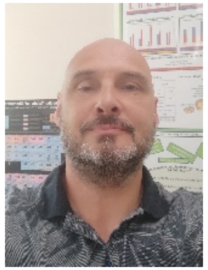
determination of the redox potentials of different species



 08/07/2024

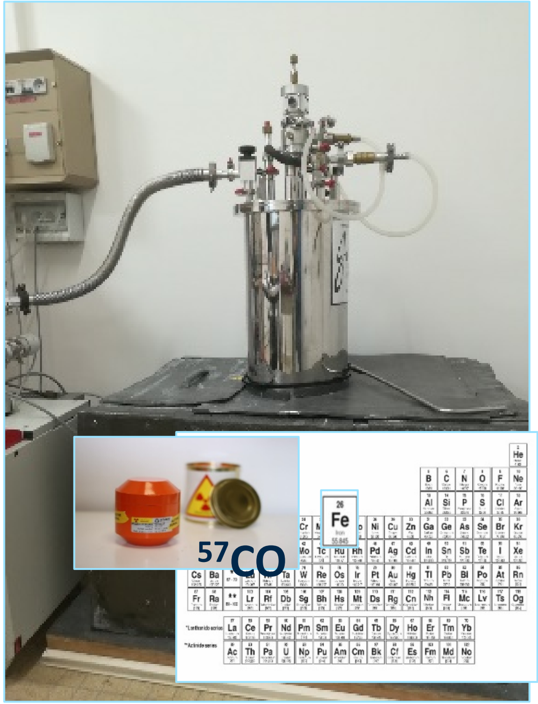
Mössbauer spectroscopy and EPR

Staff



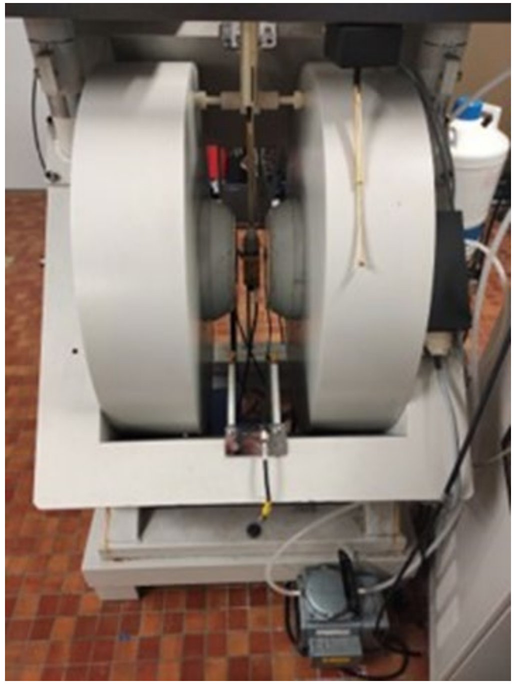
CNRS engineers (Jean-François Meunier, Baptiste Martin)

Scientific equipement



Mössbauer:
Two 4 K – 300 K measuring instruments with ^{57}CO radioactive sources for Iron studies only
Furnace 450 K

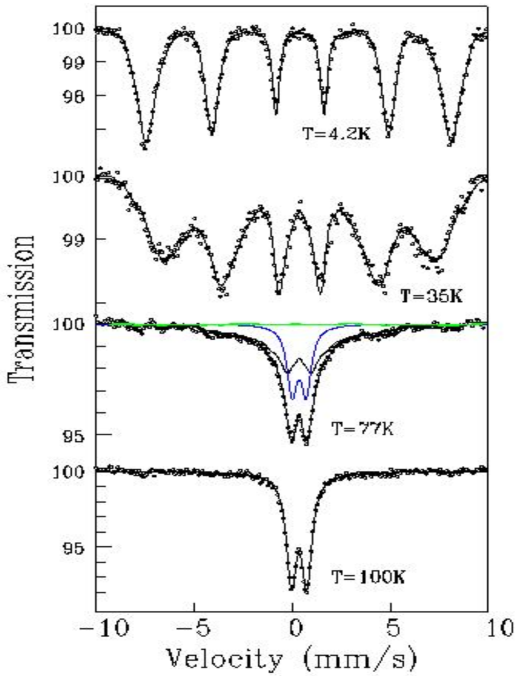
EPR:
- **4 K - 400 K ;**
- **Microwave frequency bridge: 9 GHz,**
- **Powder, solution.**
- **Accessories:**
  - **Magnetic field max.**
  - **Optical fiber for irradiation of samples**
  - **Goniometer for analysis on single-crystal**
  - **Falt Cell for aqueous solution**



Mössbauer spectroscopy and EPR

Mössbauer Applications

- Phases identification, defects, corrosion phenomena
- Discrimination and quantification of Fe sites (Fe0/ FeI/FeII/FeIII)
- Identification of Fe oxide phases
- Characterization of spin transition
- Study of Electronic Relaxation
- Study of magnetic behavior of Iron nanoparticles



Iron nanoparticles Vs temperature

Mössbauer

Mineralogy

Archaeology

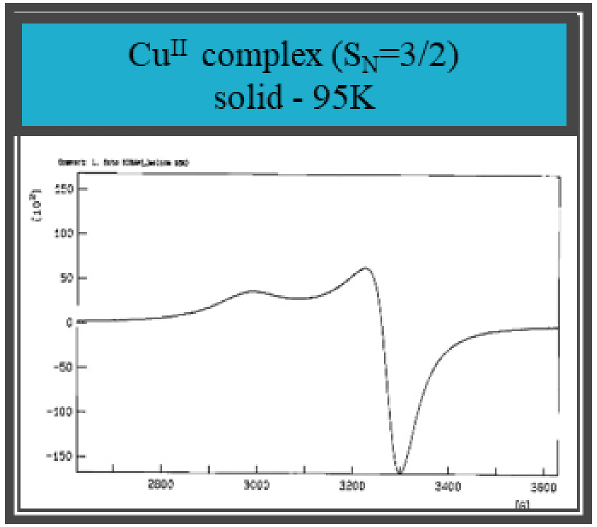
Metalurgy

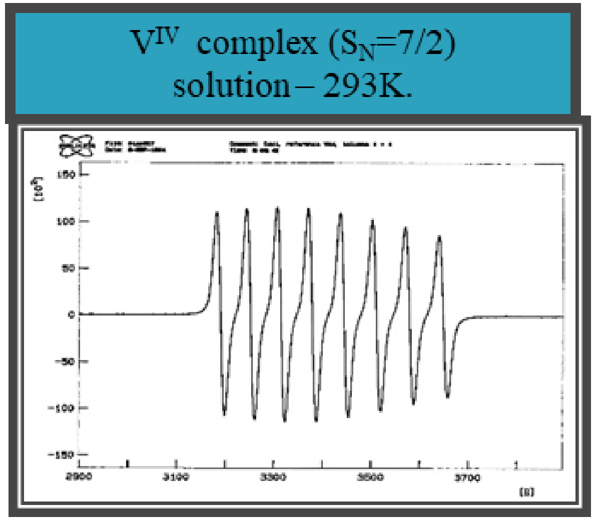
Physic and solid chemistry

Catalysis

EPR Applications

- Identification of organics radicals
- Kinetics of degradation of radical species
- Nature of paramagnetics compounds





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