



PRESENTATION OF CEA MARCOULE LABORATORY DEPARTMENT

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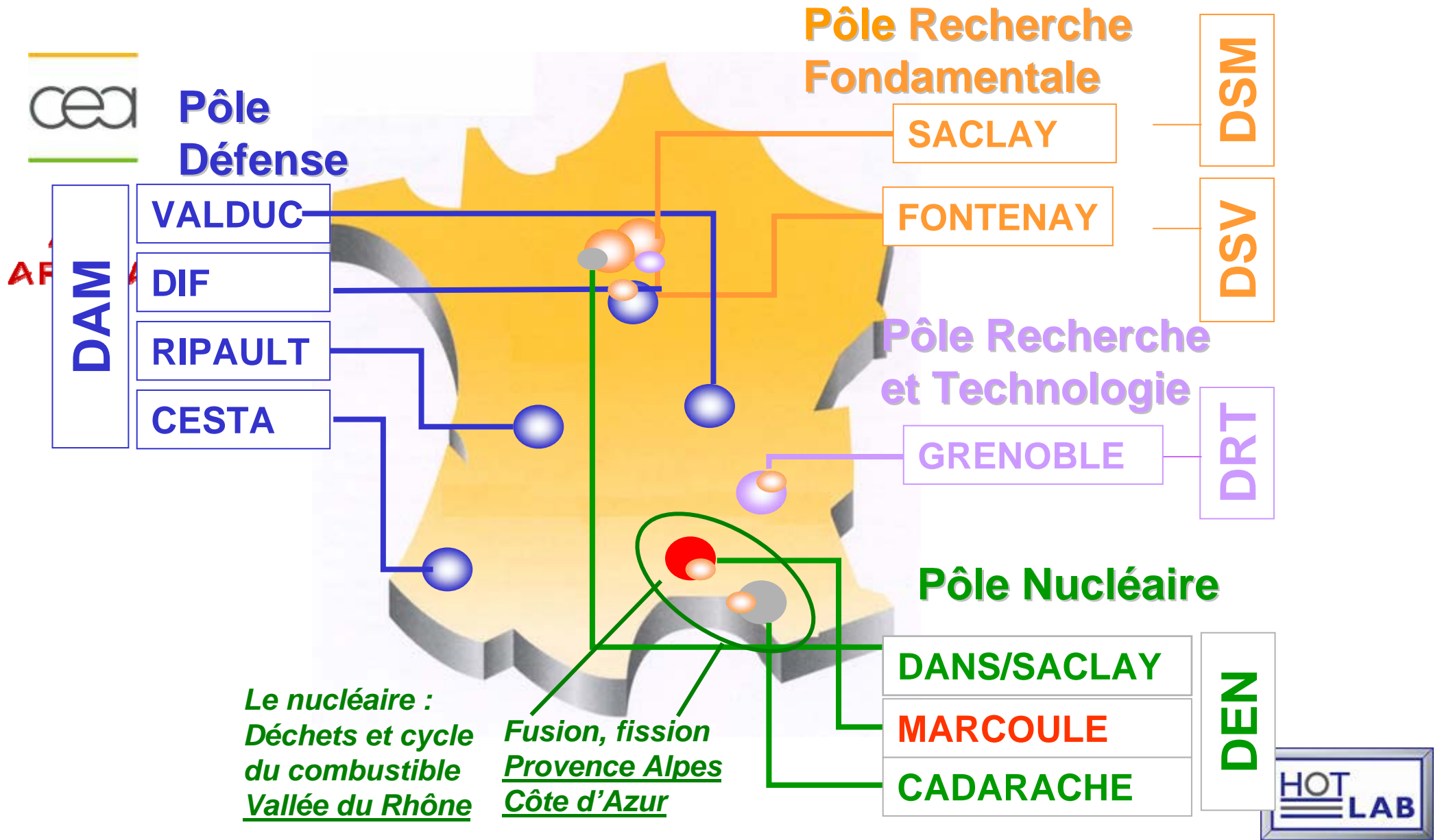
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CEA, DEN, Marcoule,

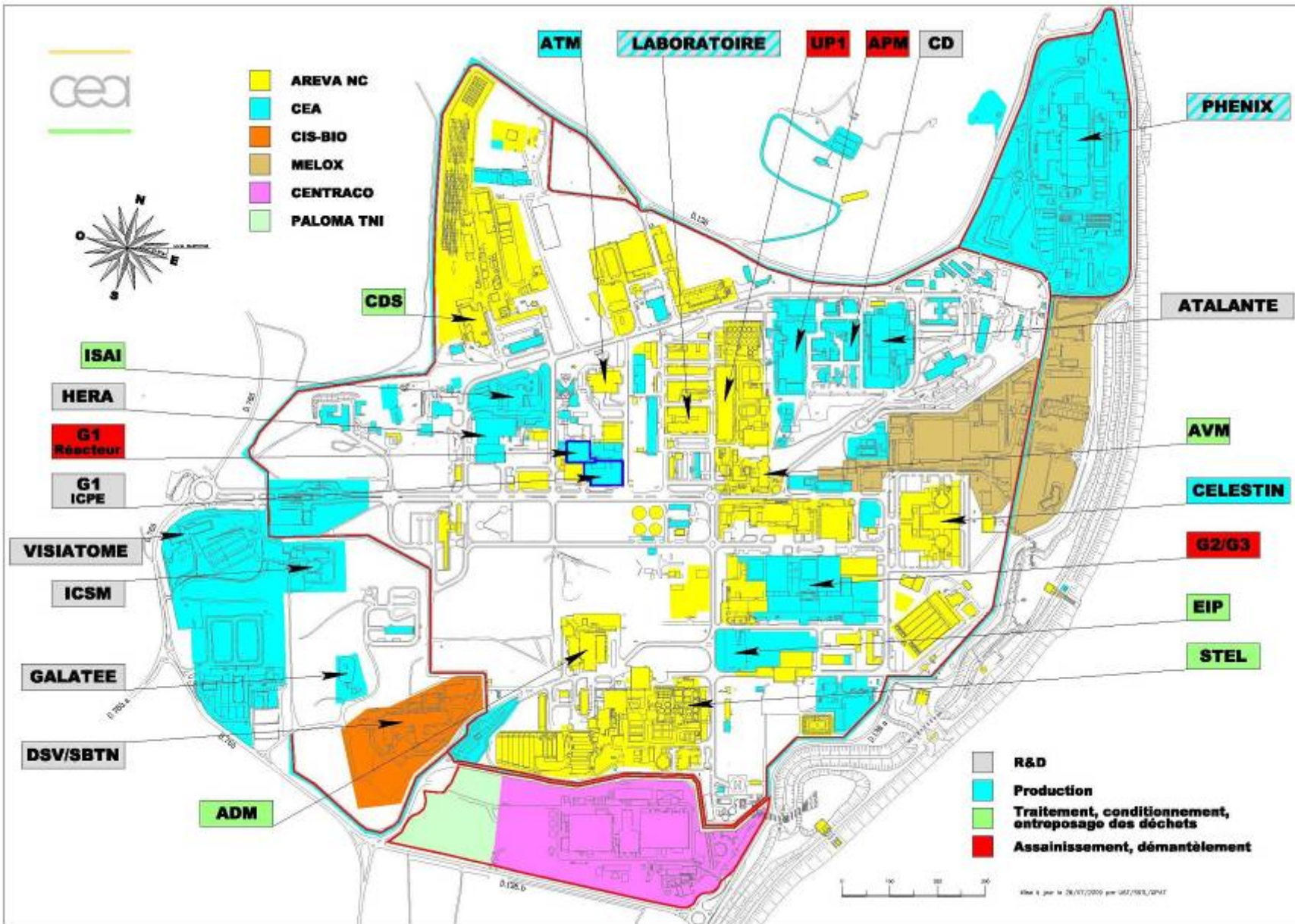
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CEA MARCOULE RESEARCH CENTER



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HISTORICAL CONTEXT



- **MARCOULE: Birthplace of Plutonium Production 1956**
 - ➔ **Usine de Plutonium (UP1)**
- **Laboratory Department operational in 1958:**
 - **Initially:**
 - **Analytical support for production (chemical, radiological)**
 - on line process control (via pneumatic transport)
 - **Quality control on raw materials and finished products**
 - **Effluent**
 - **Waste treatment**
 - **1976 : Creation of COGEMA**
 - **Lab operated by COGEMA**
 - **1997: shut down of UP1**



HISTORICAL CONTEXT



- **1998: new orientations needed Lab analytical capacities**
 - ➔ **Support to dismantling activities and waste management (deategorization) of MARCOULE center**
- **2005: Lab operated by AREVA NC under CEA responsibility**
- **2008: Starting of the Lab renovation to respect the new safety rules fire and earthquake resistance**
- **Arrival of CEA teams and opening of the activities towards analytical support of R & D and industrial expectations**
- **2011: Safety authority agreement for the Laboratory**



RESSOURCES



- **STAFF: 109 persons,**
 - **AREVA NC :**
 - **Central management entity (management, safety, security, quality, environment unit): 7 persons**
 - **General Service (Utilities, Radioprotection): 19 persons**
 - **Nuclear Methods and Measurement Design Laboratory: 10 persons**
 - **Analytical Chemistry Laboratory: 31 persons**
 - **Industrial Chemistry and Projects Laboratory: 15 persons**
 - **Radioactive Measurement Laboratory: 15 persons**
 - **CEA :**
 - **Metallography and Chemical Analysis Laboratory: 10 persons**
 - **Advanced Decontamination Process Laboratory: 3 persons**



ANALYTICAL CAPACITIES

The CEA logo consists of the lowercase letters 'cea' in a stylized, rounded font, positioned between two horizontal lines: a yellow one on top and a green one on the bottom.The AREVA logo features a large, red, stylized letter 'A' above the word 'AREVA' in a bold, red, sans-serif font.

- **Synergies between CEA and AREVA analytical teams grouped at a same location**
- **2008 DATA**
 - 5000 samples
 - 23550 analytical determinations
 - 95000 working hours
 - 3160 study hours
 - working 3X8 and 2X8 hour shifts



FACILITIES

The logo for CEA (Commissariat à l'Énergie Atomique) consists of the letters 'cea' in a stylized, lowercase font, enclosed within a square frame.The AREVA logo features a large, stylized red letter 'A' above the word 'AREVA' in a bold, red, sans-serif font.

- **AREA: 5 interconnected building wings 4200 m²**
 - **Unrestricted access zone : 1700 m²**
 - locker room,
 - storage area,
 - yards
 - offices
 - lab

 - **Restricted access zone : 2500 m² of lab**
 - 80 fume cup boards
 - 40 glove boxes
 - 35 shielded cells

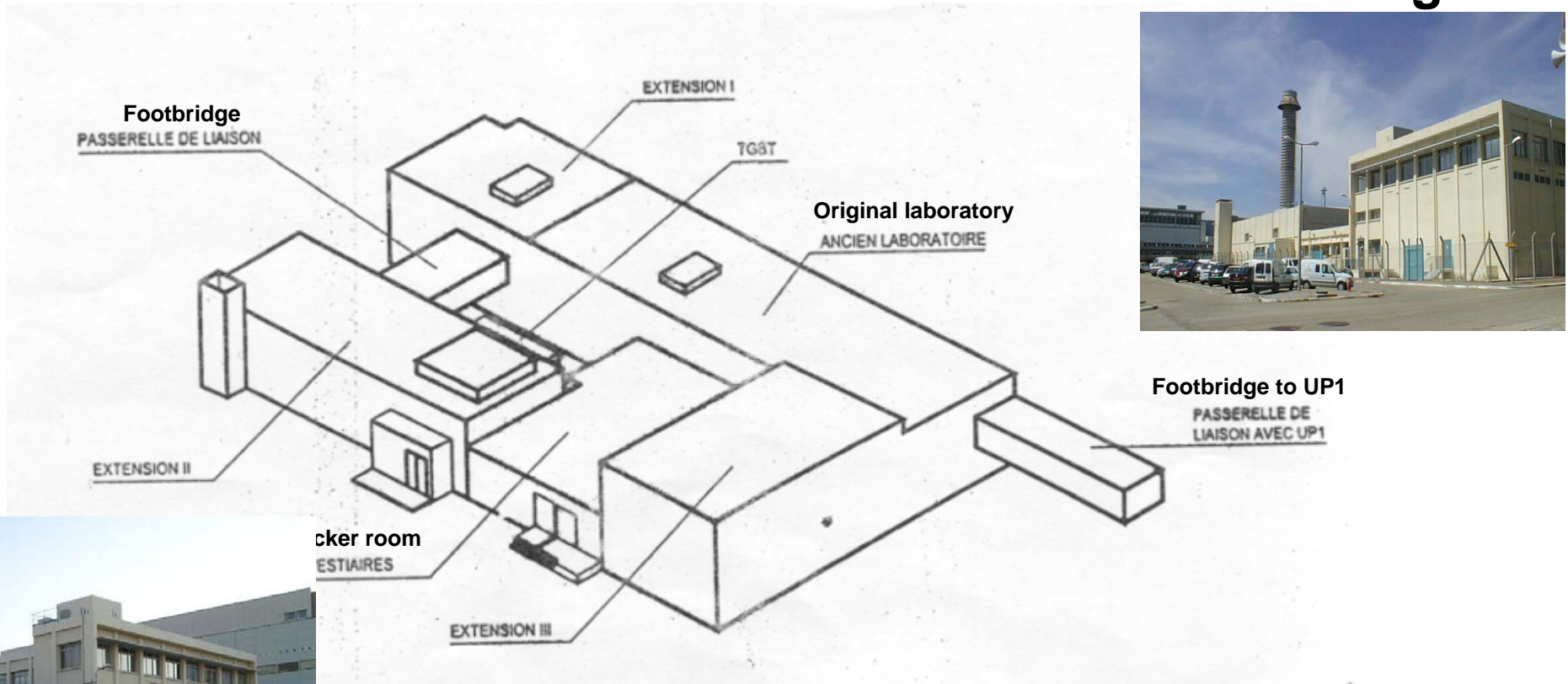


FACILITIES



•1960-1986: increase of analytical capacity

3 extensions added to initial building



BUILDING 109



MISSIONS



- **Studies and Analysis to nuclear customers:**
 - CEA centers, AREVA NC, AREVA NP, MELOX, EDF, COMURHEX, and SOCODEI
- **Analytical part:**
 - **→ wide range of analyses and capacities**
 - Process control to support industrial needs:
 - Process control in continuous operation
 - Regular sampling plans per unit, per sampling point, per campaign, etc.
 - Allowance for process operation (shift schedule, response time, real-time communication with various contacts)
 - Statistical control of the analytical process
 - Inspection of raw materials and finished products



MISSIONS



- **Analytical part:**
 - Characterization and expert examination
 - **complex, specific samples in support to R&D programs.**
 - Fabrication and supply of standards and reference materials
 - Treatment and conditioning of small quantities of exotic waste with high alpha and/or beta contamination
 - In situ nuclear characterization



MISSIONS



- **Studies part (industrial expectations) :**
 - Assistance to plant operators concerning specific process-related problems,
 - Pilot (chemical engineering) testing to adapt new or redesigned processes,
 - Treatment of liquid and solid waste rich in alpha and/or beta emitters,
 - Decontamination of liquid effluents at prototype scale



MISSIONS



- **Research and development parts:**

- Multiple scale profiles (from research to industrial development).
- Analytical development procedure
- Instrumentation development (Canberra)
- Analysis process not predefined and subject to dynamic modification
- International and academic collaborations:
 - **ITU, PSI, SCK/CEN, ORNL**
 - **Universities of Montpellier, Barcelona, Bordeaux, Limoges, Nancy, CNRS**



MISSIONS



- **External recognition and analytical reliability:**
 - **Experts:**
 - **3 senior experts (instrumentation nuclear analysis)**
 - **3 international ISO TC85/WG5 and ENTRAP/WGA experts**
 - **2 national AFNOR/BNEN experts in nuclear waste fields**
 - **Analytical validation procedure by CETAMA (Commission d'ETAbblissement des Methodes d'Analyses) ISO9001 certificated**
 - **Know how shared in 8 analytical working group**
 - **Round robin test to check regularly the lab capabilities or to qualify specific standard materials**



ACTIVITIES: ANALYTICAL CHEMISTRY



Wide range of capabilities in analytical chemistry in support of research, development and dismantling applications:

- Elemental analysis of cations and toxic elements,
- Specific analyses: CN^- , Cl^- , F^- , NO_2^- , NO_3^- , SO_4^{2-} , PO_4^{3-} , $\text{C}_2\text{O}_4^{2-}$, formates, acetates, TBP, DBP, MBP, etc...
- Conventional chemical analysis: density, acidity, pH measurement, etc...
- Preparation and characterization: measurement standards, reactants.
- Pretreatment and preparation of samples before analysis: mineralization, dissolution (bitumen, concrete, etc.).
- Development of analytical procedures.
 - **New XFR quantitative methods applied for multielement nuclear glass.**
- Characterization of the evolution of TBP under a irradiation.
- Waste and decontamination.



ACTIVITIES: ANALYTICAL CHEMISTRY



MEANS:

- Monitored zone in conventional working conditions:
 - X-ray fluorescence (x 2).
 - ICP-AES (x 4).
 - ICP-MS (x 2).
 - C/S analyzer, Atomic absorption spectroscopy.
 - GC-MS and μ GC (x2).
 - Optical Asbestos identification.
 - Ignition point, flash point.
 - Liquid chromatography.
 - Ion chromatography.
 - FTIR.



ICP-MS/AES

ACTIVITIES: ANALYTICAL CHEMISTRY

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MEANS:

- **Fume cupboard environment:**
 - ICP-AES.
 - Liquid chromatography.
 - Atomic absorption spectroscopy.
 - UV-visible spectrometry.
 - Mineralization, dissolution.

- **Glove box environment:**
 - ICP-AES.
 - C/S analyzer.
 - UV-visible spectrometry.
 - Particle size analysis.
 - Mineralization, dissolution.
 - Liquid chromatography, GC-FID
 - XRD



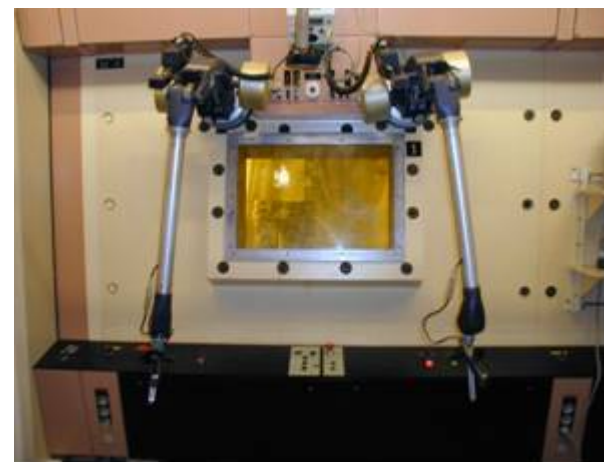
Sample preparation



ACTIVITIES: ANALYTICAL CHEMISTRY

MEANS:

- **Shielded line:**
 - ICP-AES .
 - Liquid chromatography.
 - UV-visible spectroscopy.
 - γ Spectrometry, specific electrodes, etc...
 - Mineralization.
 - Balance, microwave oven, density, etc...



ICP-AES



ACTIVITIES: METALLOGRAPHIC CHARACTERIZATION



Expertise and characterization of materials at local scale (about 1 μm^2)

It supports research, development and industrial studies and projects concerning Gen IV fuel and structural applications, front end:

- **Quality control of materials, fuels (UO_2 , USi , UMo , UCe) and components as received (bulk, deposit, powder): microstructure, adherence, homogeneity, welding.**
- **Examination of materials (steel, ceramic, glass, polymer, composite) after corrosion or aging tests: intergranular corrosion, reactivity mechanism by gas, liquid metal, molten salts or Uranium materials as UC, UN.**
- **Examination of industrial components: failure, corrosion by UF_6 , Cl_2 , CFC, HF.**



ACTIVITIES: METALLOGRAPHIC CHARACTERIZATION

MEANS :

- **Metallographic preparation (cutting, polishing, etc...).**
- **Optical Microscopy: image and image analysis.**
- **SEM-FEG/EDS and SEM-low vacuum/EDS: image and local chemical analysis.**
- **Electron Microprobe (EPMA): quantitative chemical analysis.**
- **X-Ray Diffraction: phase identification.**
- **Macro-microhardness.**



MEB-FEG/EDS





Analytical development and control in support of industrial processes.

- Characterization of active samples:
 - **Sampling** : tools design and assistance to the operator
 - **Macroscopic examination** in shielded cells
 - **Mechanical treatment** (cutting, grinding, sieving...)
 - **Chemical treatment** (acidic dissolution, purification, separation...)
 - **Specific analytical means** (μ calorimetry, DTA/MS-DSC, GC)
 - **Physical characterization** (densities, water rate, graphite rate...)
 - **Explanation and reporting of the results** to the customer

Samples preparation in hot cell



ACTIVITIES: INDUSTRIAL CHEMISTRY



- Back-up to Marcoules's nuclear facilities
 - **Study and validation of new chemical processes**
 - **Recovery and recycling of Pu**
 - **Co-precipitation and reactivity tests for acceptability of big quantities of liquid effluents in Marcoule's Treatment Station**
- Cement encapsulation
 - **Validation of the formulation**
 - **Quantification of gas releases (GC)**
- Destruction of active organic matter :
 - **Supercritical water oxidation process.**
 - **Treatment of Pu-charged resins through silver(II)-electrodissolution and oxalic conversion.**



Supercritical set up



ACTIVITIES: INDUSTRIAL CHEMISTRY



- Specific work :
 - **Dissolution of a irradiated UNGG fuel element**
 - **Qualification of a nuclearized analytical device (LIBS) for future *in situ* measurements**
 - **Decontamination of debased organic solvents**



ACTIVITIES: RADIOACTIVE MEASUREMENTS



Isotopic analysis, quantification of alpha, beta and gamma emitters;

- **Uranium-plutonium balance.**
- **Isotopic compositions: U, Pu, B and Li.**
- **Determination of beta emitters : ^3H , ^{14}C , ^{36}Cl , ^{55}Fe , ^{59}Ni , ^{63}Ni , ^{90}Sr , ^{94}Nb , ^{99}Tc , ^{129}I , ^{151}Sm .**
- **Fabrication isotopic standards.**
- **Development of specific measurements (e.g. ^{99}Tc in bitumen-encapsulated waste, ^{129}I by ICP-MS).**



ACTIVITIES: RADIOACTIVE MEASUREMENTS

MEANS



- 2 thermal ionization mass spectrometers.
- 2 liquid scintillators
- 6 gamma spectrometers including one with sample changer.
- 7 alpha spectrometers with grid chamber detectors.
- 16 alpha spectrometers with semiconductor detectors.
- 1 ICP MS in fume cupboard.



ICP-MS in fume cupboard



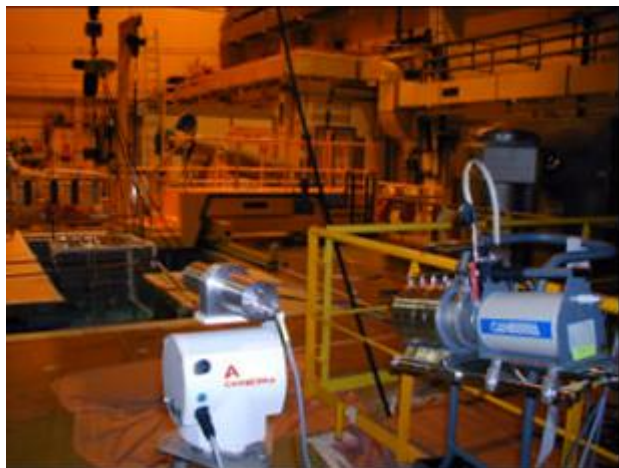
TIMS



ACTIVITIES: NUCLEAR METHODS & CONCEPTION DESIGN

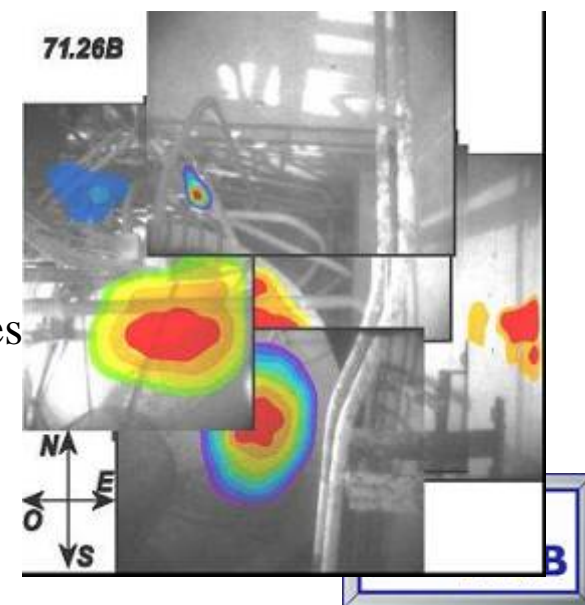


- **In situ radiological characterization of used equipment items (glove boxes, tanks, crushing machine, etc...), decontamination and dismantling monitoring of industrial set up and plants, waste packages.**
- **Development of specific methodology adapted dismantling operations (i.e. UP1)**
- **same entity with various competences and know-how (radiologic and chemical measurements, modelling)**
- **In situ initial radiological survey of complex configurations .**
- **Monitoring of dismantling operations**
- **Local characterization of the waste**



Gamma Camera and Gamma probe

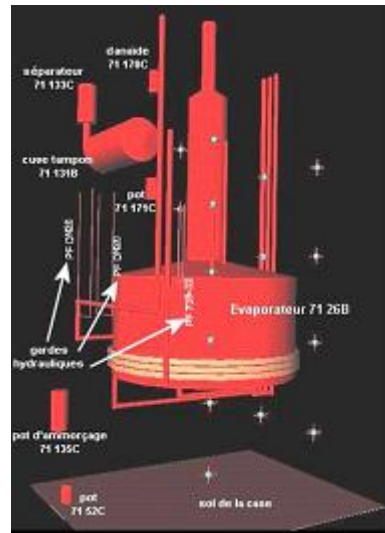
Hot spots in a used evaporator:
superimposed images
of visible image and
gamma image.



ACTIVITIES: NUCLEAR METHODS & CONCEPTION DESIGN



- **Modelling of the residual activity taking into account of the real geometry of the pieces to assess the dismantling strategy in term of costs, schedule and waste specifications**



modelling of the evaporator to evaluate the residual activity

- **Estimation of the Pu mass distribution with a correct uncertainty (1g up to 100g of Pu)**
- **Characterization of historique waste not well characterized**
- **Assessment of waste package management.**



ACTIVITIES: NUCLEAR METHODS & CONCEPTION DESIGN

MEANS



- **Gamma camera (CARTOGAM).**
- **Four portable Ge gamma spectrometry systems (coupled with ISOCS and PASCALYS software).**
- **Six portable Cd-Te gamma spectrometry systems (coupled with PASCALYS).**
- **Nal portable probe for fast hot spot survey**
- **Two dose rate measurement systems for pipes and other configurations.**
- **MERCURAD dose rate modelling software useful for dismantling scenario.**



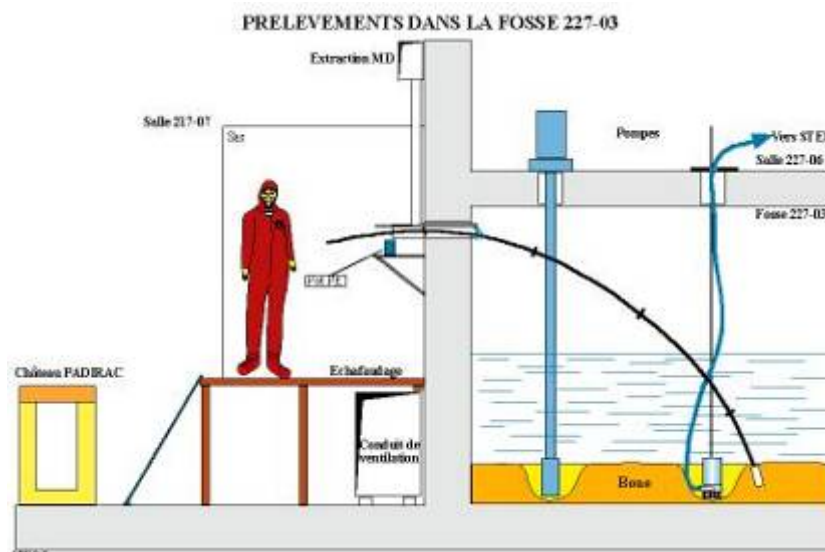
EXAMPLE: DISMANTLING OF UP1/MAR 200

Dismantling of continuous dissolvers in UP1:

- poor historic and documentation → strong of analyses and expertises from the Lab. to feed the safety reports and waste agreements
- Analysis allow to detect the lowest level of fissile mat. Useful to classify waste package
- Development of sample collection system

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HOT LAB

EXAMPLE: DISMANTLING OF UP1/MAR 200

Dismantling of continuous dissolvers in UP1:

- **200 analysed samples (physical, radiological and chemical data) needed for the safety report of elimination route**
- **α , β , γ spectroscopies, chemical analysis of U and Pu, isotopy of Pu, cations, anions**



CONCLUSIONS



- **MARCOULE LABORATORY DEPARTMENT:**

Based on production and dismantling experiments in Marcoule Site,

- ✓ **Huge variety of analytical and radiological techniques**
 - **In situ radiologic measurements**
- ✓ **Large capacities of sample treatment**
- ✓ **Capability of studies in chemical engineering between lab to industrial scale**
- ✓ **Support industrial expectations**
- ✓ **Analytical support for R&D projects**

