6th workshop
“Combined analysis examples using X-Ray Scattering”

Henry Pillière, Inel
Christophe Fontugne, Inel
Presentation of Inel (very quick)

Interaction X-ray and matter (no maths)

Instrumental function: maybe I should have taken item 27458b instead of 28563c from option list?

XRD setup: some instruments and the information they give

- Laboratory systems
- Industrial (on-line)

Some real XRD examples:

- Dust analysis
- Phase transition at (not very) high temperature
- Thin layer: diffraction, reflectometry, stress
- Micro-diffraction
- SAXS
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Key figures
Creation : 1974
Staff 2015 : 20 employees 80% technically qualified (PhD, Engineers and technicians)
Staff seniority average: 10 years

Resources
All technical and human resources are situated in our headquarter in France :

• Research & Development, technical assistance
• Informatics tools development & assistance
• Mechanical parts design & metrology
• X-Ray diffraction systems assembling
• Installation & After Sales Services for instrumentation
• Components and electronic cards design & integration
**Skills and expertise**

**Instrumentation**
Inel designs, manufactures and provides analytical instrumentation:
- X-Ray diffraction instrumentation
- X-Ray radiography instrumentation (CND)
- Extreme Ultraviolet sources instrumentation

**Engineering**
Your needs are specific or evolved and your equipment doesn’t fit your needs anymore? Our mission:
- Project consultant & management
- Feasibility studies
- New equipment design & installation

**Scientific Studies & Projects**
INEL participates in the European and local scale to various projects of R&D to develop Technology.
- European ENVIROMONITOR project coordination: real time automated instrument development using XRD for on-site quantitative analysis on breathable spray particles, including nanoparticles
- Scientific state projects, Thesis financing
- Works coordination & supervision
- Patents creation & operation
Wave-matter interaction

- **Reflection**
- **Absorption**
- **Diffusion**
- **Diffraction**

**X-rays Production**: classically by excitation of external electronic level with electron beam

**Bragg's law**

\[ d = \frac{\lambda}{2\sin \theta} \]
For a given source (sun), and a sample (Alpes)

Instrumentations using radiation for material analysis need to be optimized:
- source characteristics
- detection characteristics
- sample environment
- mechanical design

Who can see the error?
The energy of photons used for optical spectroscopic measurements of various quanta

EHz : exahertz \((10^{18})\) - ZHz : zettahertz \((10^{21})\) - YHz : yottahertz \((10^{24})\)
Building an instrument

Instrument using a wave for probing matter is defined by several functions:

- Source
- Detection
- Matter
- Matter holder
- Enclosure

- $f_{\text{source}}$
- $f_{\text{detection}}$
- $f_{\text{sample}}$
- $f_{\text{sample holder}}$
- $f_{\text{operator}}$
Function source

A light emission characterized by:
- a spectral range
- a solid angle
- intensity
- dimension and shape of the source

Optimizing the characteristics of a source allows to focus on a given interaction:

<table>
<thead>
<tr>
<th></th>
<th>fluorescence</th>
<th>imaging</th>
<th>diffraction</th>
<th>reflection</th>
<th>diffusion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spectral range</td>
<td>large</td>
<td>large</td>
<td>Monochromatic (excepted Laue)</td>
<td>monochromatic</td>
<td>monochromatic</td>
</tr>
<tr>
<td>Solid angle</td>
<td>Few degrees</td>
<td>Large (60°)</td>
<td>Small to parallel or focusing</td>
<td>Very small</td>
<td>Very small or focusing</td>
</tr>
<tr>
<td>Source size</td>
<td>Small or large</td>
<td>Small for resolution improvement</td>
<td>small</td>
<td>small</td>
<td>small</td>
</tr>
<tr>
<td>Source shape</td>
<td>point/linear</td>
<td>point</td>
<td>Point or linear</td>
<td>linear</td>
<td>Point or linear</td>
</tr>
</tbody>
</table>

This is achieved by using appropriate optics (1D, 2D, monochromator, mirror, collimator, slits …)
An optic is characterized by:
- mosaicity → spectral range
- a capture angle → beam size
- divergence → resolution

“Monochromaticity”
Effect of optic: comparison between high resolution and high flux

**Blue**
- ACQTIME: 300
- VOLTAGE: 35
- CURRENT: 35
- WAVELENGTH: 1.54056000
- COMMENT1: inc=10 SFspin

**Green**
- ACQTIME: 27815
- VOLTAGE: 35
- CURRENT: 35
- WAVELENGTH: 1.54056
- COMMENT1: inc=8 spin

**Graphite monochromator**

**Ge111 monochromator**

**Blue**
- ACQTIME: 1800
- VOLTAGE: 35
- CURRENT: 35
- WAVELENGTH: 1.54056000

**Green**
- ACQTIME: 18000
- VOLTAGE: 35
- CURRENT: 35
- WAVELENGTH: 1.54056

**Graphite monochromator**

**Ge111 monochromator**

**pozzolana**

**zeolite**
X-Ray Diffraction for the 21st Century

Effect of wavelength

Mixture of minerals

Cukα

Cokα
Effect of optic

With graphite, $\frac{ka_1}{ka_2}$ doublet is considered as a single peak.

Nist SRM 1976 (Alumina)

<table>
<thead>
<tr>
<th>Flux ratio</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ge(111)</td>
<td>1</td>
</tr>
<tr>
<td>Graphite</td>
<td>5</td>
</tr>
<tr>
<td>parab. Mirror</td>
<td>15</td>
</tr>
<tr>
<td>Elliptical mirror</td>
<td>20</td>
</tr>
</tbody>
</table>
Function detection

A detector is characterized by:
- spacial resolution
- dynamic range
- energy resolution
- dimension

Optimizing the characteristics of a detector allows to improve the measurement

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<th>reflection</th>
<th>diffusion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spacial resolution</td>
<td>none</td>
<td>good</td>
<td>good</td>
<td>none</td>
<td>medium</td>
</tr>
<tr>
<td>Dynamic range</td>
<td>3</td>
<td>4~5</td>
<td>3~5</td>
<td>6~8</td>
<td>3~4</td>
</tr>
<tr>
<td>Energy resolution</td>
<td>~200eV</td>
<td>None</td>
<td>None</td>
<td>None or 1KeV</td>
<td>None or 1KeV</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Filtering possible</td>
<td>None or 1KeV</td>
<td></td>
<td></td>
</tr>
<tr>
<td>dimension</td>
<td>0D</td>
<td>2D (1D)</td>
<td>0D, 1D, (2D)</td>
<td>0D(1D)</td>
<td>1D-2D</td>
</tr>
</tbody>
</table>
Function detection

0D Detection:
Acquisition is done stepwise
$2\theta$ and statistics are time dependent

1D Detection:
Acquisition is done in snapshots
Statistics is time dependent

2D Detection:
Acquisition is done in snapshots
Statistics is time dependent
Texture information but point beam required
X-Ray Diffraction for the 21st Century

**Function matter**

Whatever the state, XRD allows to evidence and measure structural parameters in matter.
Informations obtained by XRD

\[ \lambda = 2d_{hkl} \sin(\theta_{hkl}) \]

- Phase identification
- Phases quantification
- Particles size, micro strains
- Structure, organization of electronic density levels (cell parameters, valence, atomic occupation, ...)
- Structural anisotropy (stress, texture, thin film characterization ...)
- Distribution of structures
- Phase transition, kinetics study
Function sample holder

Paracetamol

Inel data
X-Ray Diffraction for the 21st Century

Function sample holder

Na-gluten

Reflexion

Transmission
**Instrumental function for XRD**

**Some basis:**

- Instrumental function is governed by all components of the XRD instrument:
  - source characteristics
  - optics and collimation
  - detection device
  - sample environment

- XRD components should be compatible to each other
  - Example: 1D optic is not recommended with a 2D detector (equatorial aberration)

- The good knowledge of the instrumental function allows to estimate as well the quality of the result
  - Example: absorption correction or LP correction are not the same in Bragg Brentano or in Debye Scherrer

- The instrument must be adapted to the requested measurement
  - Example: performing transmission measurement with Bragg-Brentano XRD is not appropriate

- Instrumental conditions must be correctly chosen (reproducibility of results)
  - Example: choose of the appropriate wavelength vs sample

- Use of appropriate standards
  - Example: in reflexion, eccentricity is influenced by transparency. Using standard with same absorption can correct this
Understanding how to get the result

- appropriate instrumental configuration

- appropriate sample conditionning

- appropriate calibrations / corrections
**X-ray diffraction setup: powder diffraction applications**

**Identification**
- Police (narcotics, explosives, pigments, ...)
- Museum (work of art, ...)
- Pharmacy, cosmetic, mining, geology, ...

**Quantification**
- Environment (quartz, asbestos, dust analysis ...)
- Mining industry, ...

**Cristallography**
- Analysis of new phases (pharmacy, electronic, ...)

**Microstructure**
- Information about crystalline defects:
  - Physico chemistry: reactivity (catalysis), oxidation
  - Mechanical: joints of grain, fragility area
  - Conducting: atomic replacement, doping
X-ray diffraction setup : in-situ applications

**Temperature / Pressure**
Phase transitions

**Electrochemistry**
Following the phase transition : load, unload
from a battery (ageing, reversibility, functioning)

**Reactivity**
Search match on metastable components during a chemistry reaction
Cement hydration / Gas absorption in zeolithe

**Industrial control on line**
Cement quality control,
phosphate industries
Solar panel control

**Following the product state in the time**
Product evolution with the temperature and the humidity
X-Ray Diffraction for the 21st Century

**X-ray diffraction setup on bulk material**

**Texture, crystalline orientation**
- Phase in a rock, information about the area tectonic
- Deposition on a substrate, consequence for electronic properties
- On metal, determination of the oxidation resistance
  - OR acceptance of a particular cover
- Fiber materials (C,PET),

**Residual stress (metallurgy)**
- Stress state measurements of a mechanic piece
  - (Young module and stress vector)

**Micro-diffraction**
- Homogeneity analysis of a bulk sample

**Grazing diffraction**
- Thickness measurement of a thin film
- Deposition identification (irradiated zirconia)
EQUINOX, a comprehensive real time XRD range

Inel XRD systems:

- **EQUINOX 100**: Stand alone benchtop XRD
- **EQUINOX 1000**: Benchtop XRD
- **EQUINOX 2000**: Routine XRD
- **EQUINOX 3000**: Powder high resolution XRD
- **EQUINOX 4000**: Microdiffraction/mapping XRD
- **EQUINOX 5000**: High resolution XRD
- **EQUINOX 6000**: 4 circles texture/stress XRD
- **SWAXS**: SAXS WAXS diffractometer
- **Single cristal**: Laue and single cristal diffractometer

- Real time detection by PSD detector
- Simultaneous data acquisition up to 120°/2θ
- Very high resolution, up to 0.05°/2θ on peak profiles
- Complete 2θ measurement time of just 1 second
- Multi axis goniometer with real time detection
- Micro diffraction capability down to 10 µm
- Monochromatic optic
- Sample holders
- Reflection / Transmission mode
- Variable temperature furnaces (up to 2700°C)
- Data treatment softwares
A unique detection mode

Curved detectors principle
The EQUINOX diffractometers use the curved detectors principle, namely real time acquisition across a wide acquisition range.
• No motorization required: neither on sample nor on detector incidence
• Asymmetric acquisition mode: for a fix $\theta$ sample incidence you can see all diffraction peaks on $2\theta$ on the detector

3 detector types

<table>
<thead>
<tr>
<th>Detection angular range</th>
<th>Curvature (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CPS 590</td>
<td>90° 2θ</td>
</tr>
<tr>
<td></td>
<td>R = 500</td>
</tr>
<tr>
<td>CPS 120</td>
<td>120° 2θ</td>
</tr>
<tr>
<td></td>
<td>R = 250</td>
</tr>
<tr>
<td>CPS 180</td>
<td>180° 2θ</td>
</tr>
<tr>
<td></td>
<td>R = 180</td>
</tr>
</tbody>
</table>

The detector choice depends on the requirements of the measure to be made:
• The more the detector is far from the sample, the better the resolution will be
• The more the detector is near the sample, the faster the acquisition will be
EQUINOX 100, stand alone benchtop XRD

A stand alone benchtop X-ray diffractometer
Designed for crystalline phases analysis (qualitative, quantitative, structural…) on powder or bulks. It is an ideal instrument for academic and QA/QC laboratories that need a small and easy to use equipment.

- Real time detection across 110°/2θ (CPS180)
- Simplified goniometric deck with no motorization
- No external water cooling
- Works on standard power supply (110V-20A/230V-16A)
- Friendly-user instrument driving & data treatment software

Applications: qualitative & quantitative analysis, phases identification, structure determination, crystallites orientation...

Inel data

lactose

reflection

transmission
EQUINOX 1000, benchtop XRD

A fast and powerful benchtop XRD with small dimensions, ideal for all your X-ray diffraction applications on powder. It is a very easy using instrument. No diffractometer alignment is required and the operating protocol is saved by the software. XRD experiments are available to anyone in a few moments.

- Real time detection across 110°/2θ (CPS180)
- Monochromatic optics
- 3500 Watts generator
- External water cooling
- Working wavelength : Copper or Cobalt

Applications:
Qualitative & quantitative analysis, phases identification, structure determination, crystallites orientation...
Quality control in detergent

On the graph below, are represented diffractograms recorded for each sample. We can clearly evidence an increase of STPP 1 with the index (red arrows).

Quantitative analysis has been performed by using the Rietveld method. Good reproducibility, and good agreement with other techniques (chemistry).
EQUINOX 2000, routine XRD

A routine diffractometer

with the EQUINOX 1000 performances and a bigger sample space, the EQUINOX 2000 enlarges your applications capabilities. You can now realize variable temperature and/or atmosphere measurements.

• Fast results
• Thermodiffraction (phases transitions, unstable compounds)
• Large sample space
• Real time detection across 110°/2θ (CPS180)
• Monochromatic optics
• 3500 Watts generator
• Working wavelength : Copper or Cobalt
• Environmental chambers (option)
• 30 positions autosampler (option)

Applications

Qualitative & quantitative analysis, phases identification, structure determination, crystallites orientation, phases transitions under variable environments, ...

Data collection with sample changer
EQUINOX 3000, powder high resolution XRD

A fast, powerful and multi-purpose XRD
You are now able to realize variable temperature and/or atmosphere measurements, or even add a motorized sample holder to perform thin layer analysis.

- Evolutive depending on your needs
- Real time detection
  - CPS120 across 120°/2θ
  - CPS590 across 90°/2θ (High resolution)
- Monochromatic optics
- 30 positions autosampler (option)
- Environmental chambers (option)

Applications
Qualitative & quantitative analysis, phases identification, structure determination, crystallites orientation, phases transitions under variable environments, thin layer analysis, uniaxial stress analysis...

Thermal diffraction: 5sec/pattern
**Phase transition at low 2θ**

**Source**: Copper

**Generator**: 3,5 kW

**Optique**: Parabolic mirror

**Incidence**: Transmission (1mm capillary)

**Sample holder**: non spinning furnace type FURCAP

**Detector**: CPS120

**power**: 35 kV – 35 mA

**Acquisition time**: 3min

**setting**: MPD

**Transition at 70°C**

*figure 1: temperature profile: from RT to 70°C step 10°C and every 5°C while cooling down.*
**EQUINOX 4000, DRX microdiffraction / cartographie**

**EQUINOX 4000** is designed for all your microdiffraction or mapping applications. The instrument uses X, Y and Z translation stages with strong travel.

- Real time detector (CPS 120)
- \( \theta / 2\theta \) goniometer with large stages X, Y and Z
- Working wavelength: Copper or Cobalt

**Applications**

Microdiffraction, mapping...

*Microdiffraction on the edge of an aluminum plate*

**Experimental:**  
Beam size: 20mic, Power: 1200W  
Acquisition: 120sec/pattern
EQUINOX 5000, high resolution XRD

A high resolution diffractometer
for all your powder analysis under variable temperature and/or atmosphere that require a very high resolution.

- $\theta/\theta$ or $\theta/2\theta$ 2 circles goniometer
- High resolution detection (CPS 590)
- Environmental chambers (option)

Applications
Qualitative & quantitative analysis, phases identification, structure determination, crystallites orientation, phases transitions under variable environments, thin layer analysis, uniaxial stress analysis...

Coating on stainless steel

Thin film analysis
**EQUINOX 6000, 4 circles texture/stress XRD**

**4 circles X-ray diffractometer**
Ideal for all your 4 circles goniometer X-ray diffraction applications.

- Robust and multi-purpose goniometer
- Heavy loads accepted on every movements
- Large sample space
- Real time detection (CPS590)
- 4 circles goniometer ($\theta$, $2\theta$, $\chi$, $\phi$)
- Collimating monochromatic optics
- 3500 Watts generator
- Variable working wavelength
- Temperature chambers (option)

**Applications**
Texture, stress, thin layer, qualitative & quantitative analysis, phases identification, structure determination, crystallites orientation, phases transitions under variable environments, microdiffraction...
Sample characteristics:
- Diameter max = 128 mm
- Thickness max = 10 mm
- Weight max = 1 kg

Extension:
- Possibility to set the furnace: DHS 1100 AP

Sample holders
- Goniometer head
- For bulk (max 30x30x8 mm)
- For powder
Helix pomatia (Burgundy land snail: Outer com. crossed lamellar layer), From D. Chateignier – CRISMAT (Caen)

Experimental
Generator: 3.5 kW (power 40kV – 30mA )
Monochromator: graphite flat monochromator.
Slits : 1 x 1mm
Sample holder: goniometer head, no spinning
Detector: CPS120
Acquisition time: 5 sec/pattern

Simultaneous pole figures
Absorption and defocusing corrections
Chi from 0° to 80° step 5°, phi from 0° to 355° step 5°
SWING, texture/stress XRD

A decoupled goniometer
- No Chi motion on the sample goniometer
- A lot of space for the sample
- Diffraction plane rotates around the beam axis
To better answer its customers’ needs, Inel designed a flexible SAXS line to make textured samples analysis or small angles scattering. This technique is used to determine particles structure at nanometric range (size, form, distribution...). Studied materials can be solid, liquid or gas. This non-destructive method is accurate and requires usually only a minimal sample preparation. It can be used as well for research as for QA/QC.

- Evolutive mounting
- Variable sample/detector distance
- Specific supports and chambers available
- 1D or 2D detection with dedicated software

**Applications**
Colloids, metals, cement, clays, oil, polymer, plastics, proteins, pharmaceutical industry...

- 3500 Watts generator
- Diffraction bench with monochromatic optics
- Variable working wavelength
Laue back reflection diffractometer

A fast tool for crystal orientation

- Easy sample positioning
- Fast acquisition
- Fast orientation determination

Sample holder can be customized according to crystal

Si220 orientation
Inel XRD flexibility
Combining X-ray diffraction with other techniques:

• One sample and several datas
• Real time analysis
• XRD comes to sample
• XRD for on line industrial control

Inel portable XRD systems:

EQUIRAM Combination of XRD and Raman spectrometer
XRD/DRIFT Combination of XRD and DRIFT spectrometer
STRESS/WAXS Combination of WAXS and stress analysis
SWAXS SAXS WAXS diffractometer
COSMA On-line production control XRD
PRECIX Robotics for XRD residual stress measures
XRD/DRIFT combination

Phase transitions observation

- Development of a laboratory system, combining XRD and DRIFT, to perform in-situ measurement
- Development of an adapted environmental cell pressure-temperature
- Concept of instrumented system, with an appropriate expert software
- XRD in transmission with Mo radiation
- IR spectrometer in reflection (DRIFT)
- Sample cell with pressure/temperature, and gas mixing.
**XRD/DRIFT combination**

DRIFT spectra recorded during thermal decomposition of calcium oxalate (50 mg, 5 K min⁻¹).

X-ray diffraction patterns recorded during the thermal decomposition of calcium oxalate (50 mg, 5 K min⁻¹)

Blue traces represent stable crystallographic phases and orange diffraction traces indicate intermediate crystallographic structures.

COSMA, on-line XRD control

On-line real time XRD measures
The industrial production performance (cement, lime, sand, aluminosilicates...) depends on the materials mineralogy. FCT-ACTech (Australia), CSIRO Minerals (Australia) and Inel (France) developed together a unique instrument that is able to perform mineralogy real time control on production line.

• Continuous powder flow extract from production line, so no more laboratory analysis required
• Real time mineralogy on crystalline materials
• Every minute results update for a step by step production quality follow-up

Applications
Powder manufacturers from a global prospective (cement, lime, sand, aluminosilicates...)

For further details please contact FCT-ACTech at:
info@fctinternational.com

C₃S, C₂S, C₃A, C₄AF, CaO, Ca(OH)₂, CaCO₃, CaSO₄·2H₂O, CaSO₄·½H₂O, Other minerals

High energy X-ray diffraction

- Development of a high energy X-ray diffractometer (200-400 keV) for the quality control of large crystal
- Misalignment determination
- Sample monitoring and image acquisition
- Possible extrapolation of the instrument for stress and texture analysis

From 100 to 200 keV beam
The technical performance was to setup an X-ray source based on a standard tube and a position and energy sensitive detector, and also the robot positioning metrology to perform residual stress measures at pole coordinates.

The robot here doesn’t maintain the part, it’s only the goniometer. The sample stays in a fix position, so users have no limitation regarding sample size and weight.
XRD combining

**Interest of combining**

- Sample is a complicate mixture: XRD+XRF, XRD + Raman

- Complementarity to observe phase transition: XRD + IR, XRD + Raman, XRD + DSC

- Relationship between mecanical properties and structure: texture + stress

- Many other combinations ...

**Advantages**

- 1 sample for several measurements, but 1 result

- Eliminating non possibilities (XRF prefiltering before search match)

- ...

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X-Ray Diffraction for the 21st Century
**Stress/WAXS combination**

**In situ XRD system on a traction machine, ASTREE**

Allows to perform residual stress measurements and phase identification

- WAXS CPS detector
- Sample
- X-ray source & Stress detector
XRD/Raman combination

Pharmaceutical and geological applications

Complete identification and characterization using the same sample in the same experimental conditions (P, T...)

≠

identification and characterization using same sample with relatively same conditions

Raman

XRD
Since 2010, wedlock between PSD detector and X-ray minisource

XRD escape from laboratories
Combining X-ray minisource and CPS detector:

• Low consumption XRD
• Robust instrument
• No external water cooling
• Works on standard power supply

Inel portable XRD systems:

EQUINOX 100  Stand alone benchtop XRD
Enviromonitor  Aerosol quantification expert system
XSOLO  Nomadic Stress System
Equinox Trail  Rackable XRD
SOLXPERT  Portable XRD/XRF system for in field applications
EQUINOX 100, stand alone benchtop XRD

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- Works on standard power supply (110V-20A/230V-16A)
- Friendly-user instrument driving & data treatment software

Applications: qualitative & quantitative analysis, phases identification, structure determination, crystallites orientation...

![X-ray Diffraction](image)

- lactose
- reflection
- transmission

56
Xsolo, the nomadic stress system

Bring the analyzer to your samples
Now you will be able to bring the analyzer directly to your sample and measure residual stress and texture in field with the new Inel Xsolo system.

- Whole system ≈ 10kg et 25 cm
- Easy to use
- 2D 24 x 34mm detector
- Anode source Cr 4 Watts
- Angular range: 90 to 170° 2θ
- Pixel size 20μm
- CCD Peltier cooling system
- «Phi» axis motorization (Texture)
- «Psi/Omega» angular sensor
- Working distance: ≈ 22mm

Applications
Welds control in the field: pipelines, bridges, nuclear reactor facilities..., On-site critical industrial parts control: aerospace, automotive..., Determination of critical failures in damaged important structures, Validation of repaired area as «restored» to original specifications, and even more...

Partnership between
Inel / Université de Trento, Italie
X-ray Diffraction for the 21st Century

Xsolo, the nomadic stress system

XR diffraction image from CCD

Results after data treatment

<table>
<thead>
<tr>
<th>$\sigma_{11}$ (MPa)</th>
<th>$\sigma_{22}$ (MPa)</th>
<th>$\sigma_{12}$ (MPa)</th>
<th>Austenite (%vol.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$15+/-2$</td>
<td>$15+/-2$</td>
<td>$0$</td>
<td>$0$</td>
</tr>
</tbody>
</table>

"Modeling methodology for stress determination by XRD in polycrystalline materials", S. Dufrenoy, T. Chauveau, R. Brenner, C. Fontugne, B. Bacroix (ECRS 9)
In situ aerosol analysis
with EnviroMonitor, aerosol are collected on a filter band within certain conditions. The band is able to move to the XRD center. XRD analysis is performed.

- Fully automatic system
- Several days of functioning
- Network servicing

Applications
Dust collection and phase analysis
XRD/XRF combination

In situ aerosol analysis
In EnviroMonitor system, an XRF device was combined with XRD. The combination allows to improve the phase determination.

Applications
Dust collection and phase analysis.

Anhydrite gypsum
SolXpert: XRD/XRF combination in field

In situ analysis with XRD/XRF combination
SolXpert is a system combining XRD and XRF. In the field of earth science, it is often difficult to characterize a sample: unknown phases, phase mixture, poor crystallinity. XRF allows to improve XRD, by limiting the number of possible phases in the search match data treatment.

Applications
Geological and environmental applications
Equinox trail

XRD integration in mobile lab
Mobile lab start to be developed, in order to improve the reactivity in the decision.

• Compact instrumental part
• 19” electronic part

Applications
Environment, geology, police, industry...

...The size of a computer!
Data treatments

FPSM, a unique software

The FPSM (Full Pattern Search-Match method)

Database of crystal structures

Pattern

Rietveld fit (for each phase in the database + each phase previously found)

Add new phases

Ranking ($R_{exp}$) and selecting best new phase

Last phase > threshold?

Y

N

End: Final Rietveld quantification

Pro:
- No user intervention, automatic analysis
- No peaks identification required (works with nano materials/particles)
- Full Rietveld quantitative analysis provided
- Works for neutron and electron diffraction

Cons:
- Only phases with known crystal structure are ready to be used (unknown structures require a list of peaks and calibrated intensities, PONKS)
- Available databases are still uncompleted
- If no elemental composition provided requires > 20 minutes on 12 cores computer
- Good ranking algorithm required for very small phase amount

For phases identification and quantification by using Rietveld method
http://cod.iutcaen.unicaen.fr/
Data treatments

By using the COD database

Testing FPSM using the COD

Sample cpd1h of Round-Robin on quantitative analysis

http://www.iucr.org/resources/commissions/powder-diffraction/projects/qars

Some additional phases (cement phases) have been added to the database as not present in the COD

Phase ID  name  vol. (%)  wt. (%)  crystallites (Å)  microstrain

9005672  Cemcalcite  14.4548  16.3939  165.793  0.0070549

9001583  Cemcalcite  9.47814  2.37802  643.2  0.00175916

Final Rietveld analysis, Rw: 0.455532, Gof: 1.79502
From R&D to product at Inel, building the future

**Academic/inel partnership:**
- SMAM (ISTO/CRISMAT et INEL)
- MatBioReOs (CRMD/INEL)
- SolXpert (INEL et BRGM)
- Thèse CG45 avec le CEMHTI
- plateforme technologique GREMI/INEL

**New inel components:**
- electronic miniaturisation for in-field applications
  - Inel goniometer
  - Dynamix detector (Eurostar)
  - SIAMX optics
  - SQL database

**new applications:**
- XSOLO
- X-TRAIL
- Enviromonitor
- contrôle cimenterie

**Know-how:**
- experience
- creativity
- innovation

**New inel products:**
- Equinox 100
- X-trail (SolXpert)
- XSOLO
- FPSM software
Thank you for your attention.

Questions?
Informations on thin films

One experiment = « a huge of informations »
thin film analysis: Cu coating on Si

Omega from 0.5 to 10.0° step 0.05
2mn acquisition per diffractogram
Cuka1 with Ge(111) monochromator
35kV-35mA

reflectometry
epitaxy
Diffraction -grazing -texture