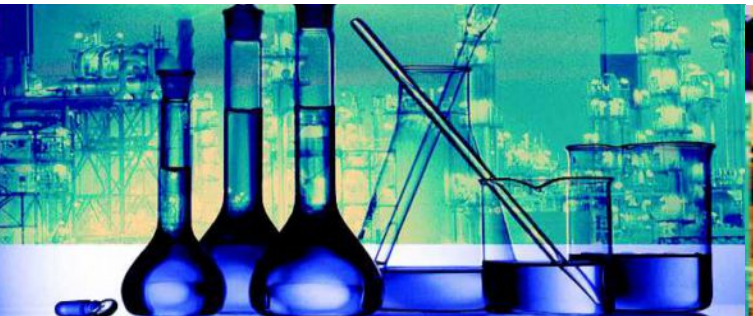
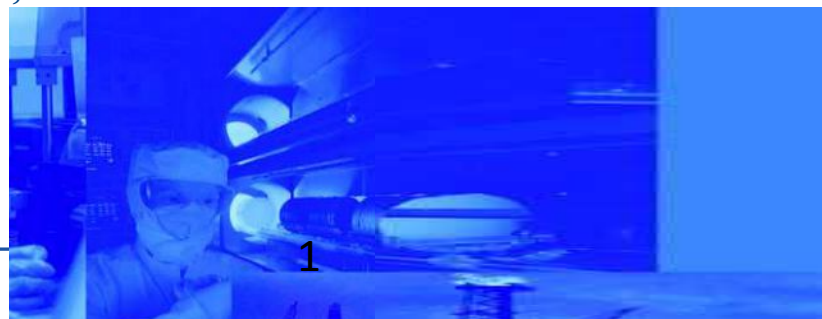
6th workshop
" Combined analysis examples using X-Ray Scattering"

Henry Pillière, Inel
Christophe Fontugne, Inel





Inel SAS, France

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 op 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*

Inel SAS, France

Inel SAS - Head Quarter

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Key figures

Creation : 1974

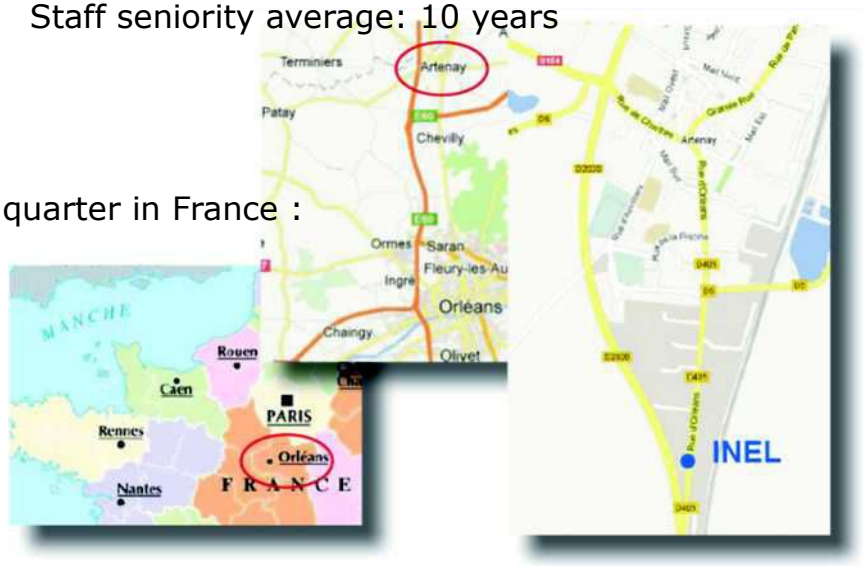
Staff 2015 : 20 employees 80% technically qualified (PhD, Engineers and technicians)

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

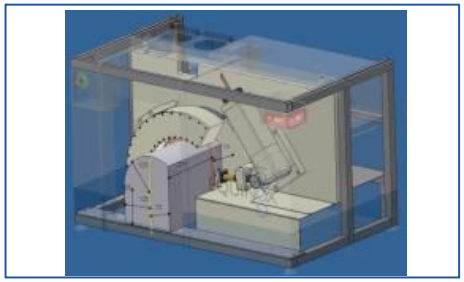


Skills and expertise

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

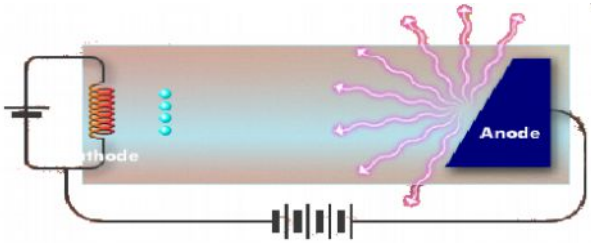
INEL participates in the European and local scale to various projects of R&D to develop Technology.

- 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

Diffusion

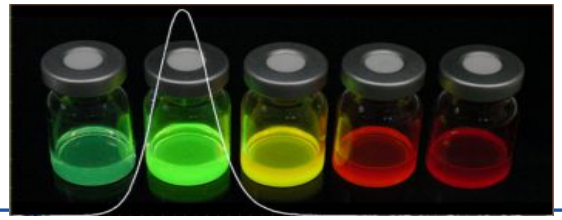


Absorption

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

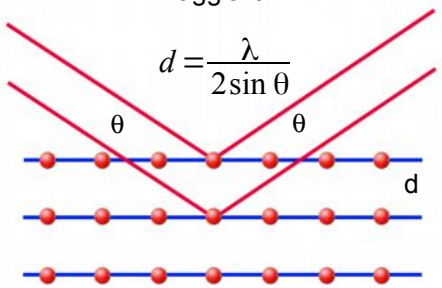
Diffraction

fluorescence



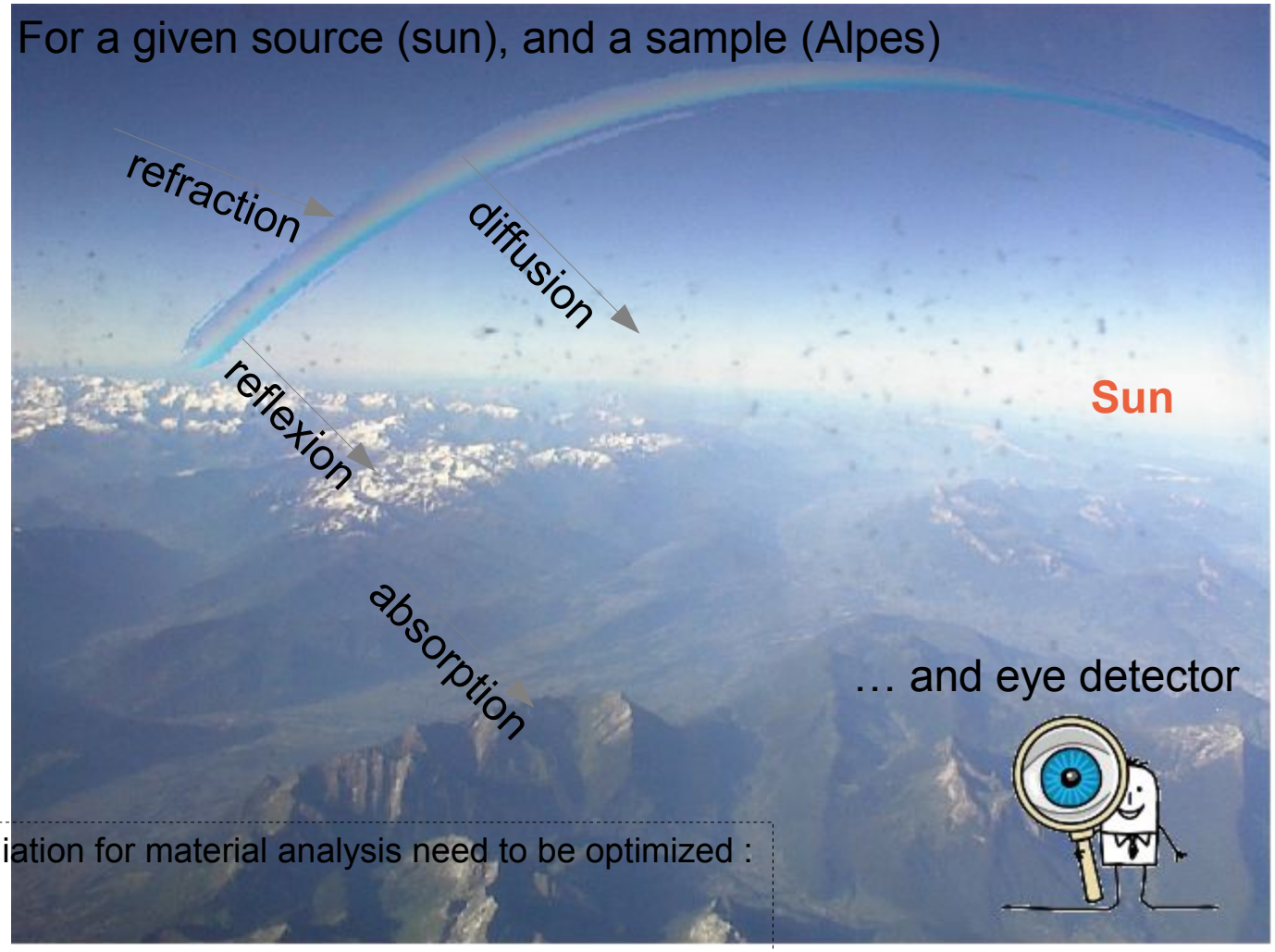
Bragg's law

$$d = \frac{\lambda}{2\sin\theta}$$



Interaction wave-matter

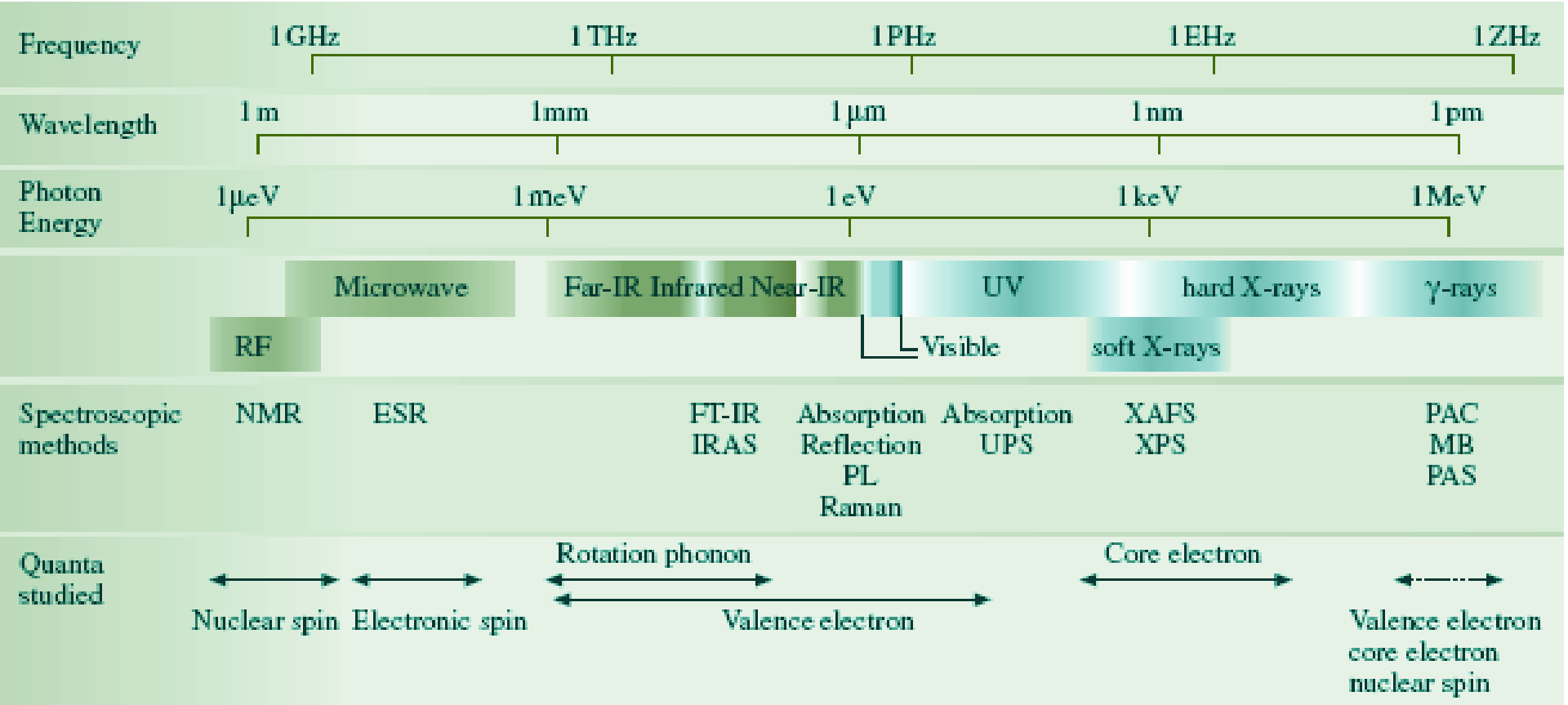
Who can see the error?



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

Interaction wave-matter

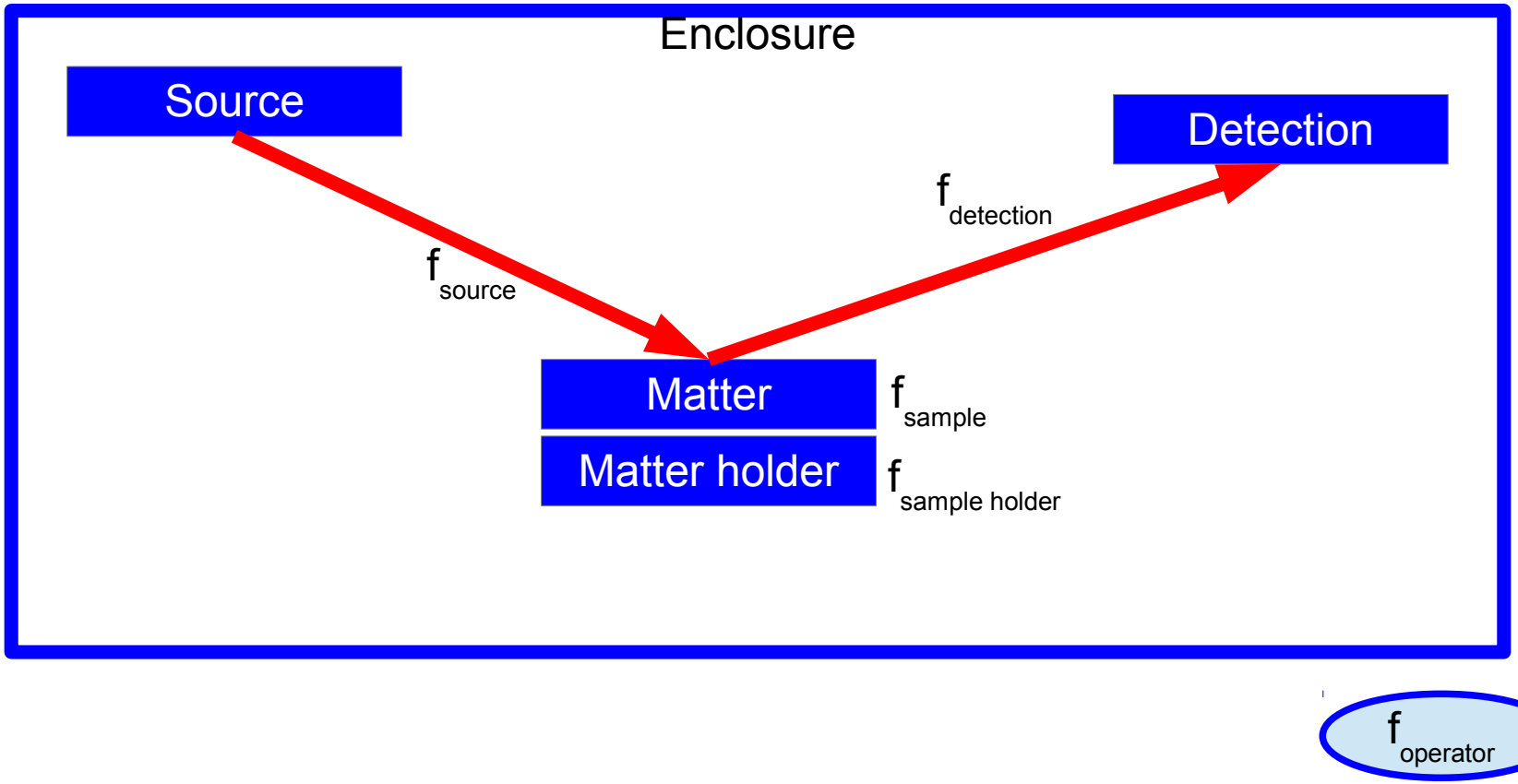
The energy of photons used for optical spectroscopic measurements of various quanta



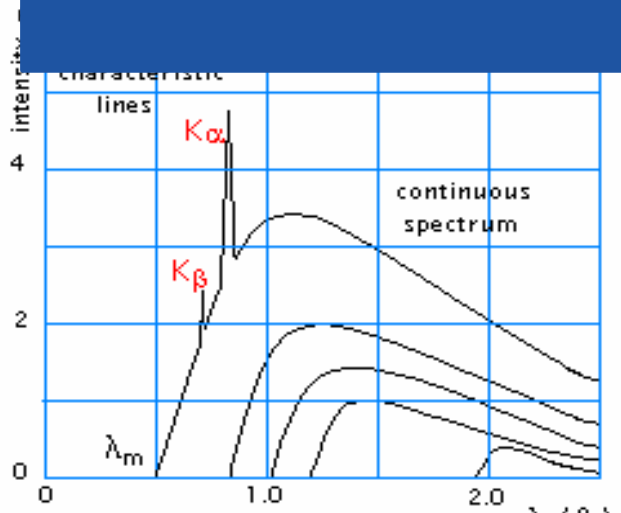
EHz : exahertz (10^{18}) - ZHz : zettahertz (10^{21}) - YHz : yottahertz (10^{24})

Building an instrument

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



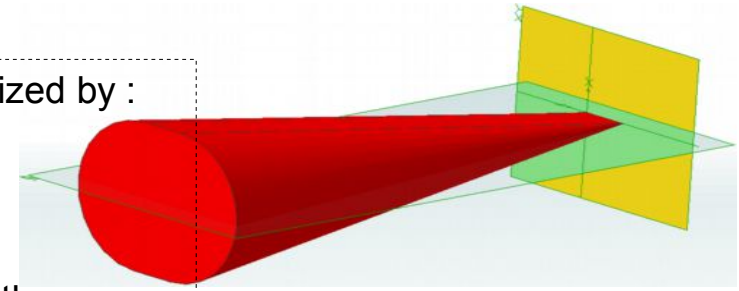
Function source



Molybdenum spectrum vs the applied voltage

A light emission characterized by :

- a spectral range
- a solid angle
- intensity
- dimension and shape of the source

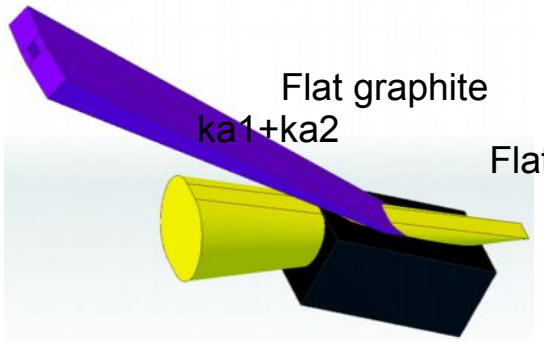


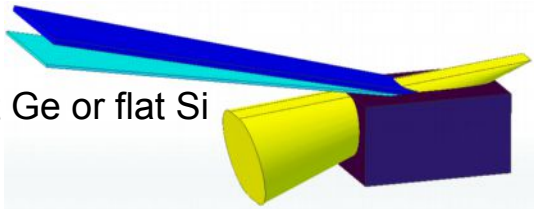
Optimizing the characteristics of a source allows to focus on a given interaction

	fluorescence	imaging	diffraction	reflection	diffusion
Spectral range	large	large	Monochromatic (excepted Laue)	monochromatic	monochromatic
Solid angle	Few degrees	Large (60°)	Small to parallel or focusing	Very small	Very small or focusing
Source size	Small or large	Small for resolution improvment	small	small	small
Source shape	point/linear	point	Point or linear	linear	Point or linear

This is achieved by using appropriate optics (1D, 2D, monochromator, mirror, collimator, slits ...)

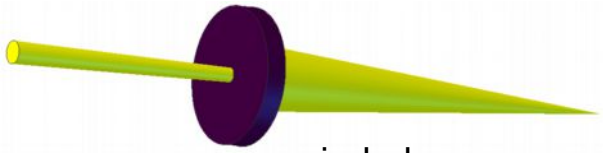
Function source

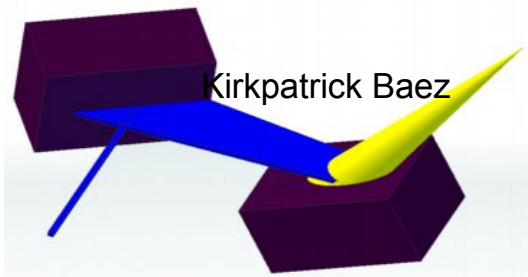




"Monochromaticity"

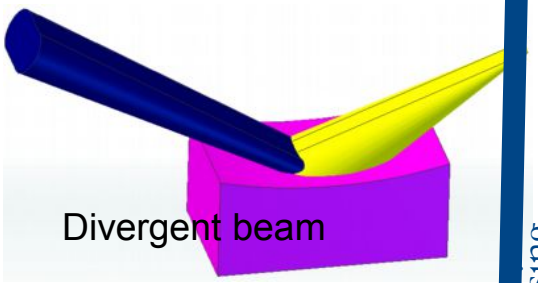
Divergence

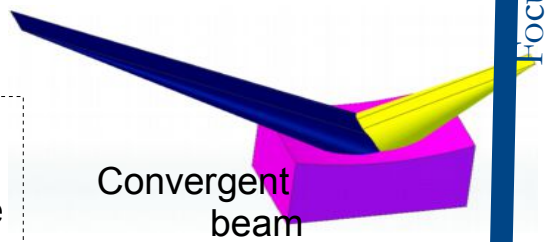




An optic is characterized by :

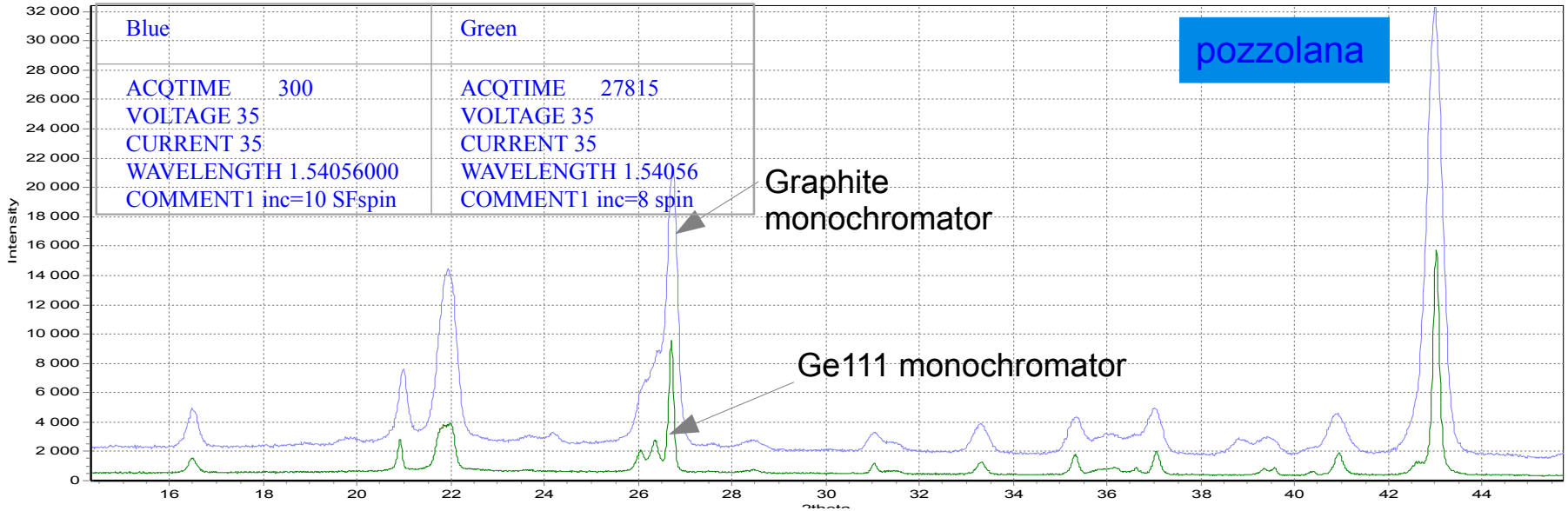
- mosaicity → spectral range
- a capture angle → beam size
- divergence → resolution

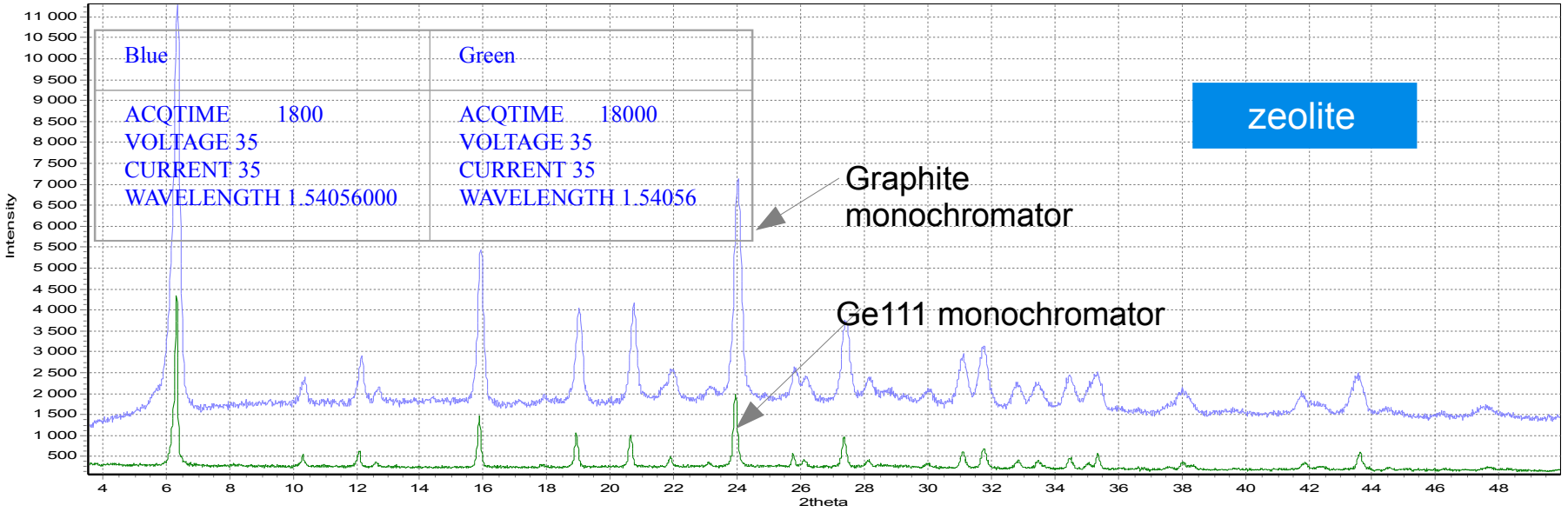




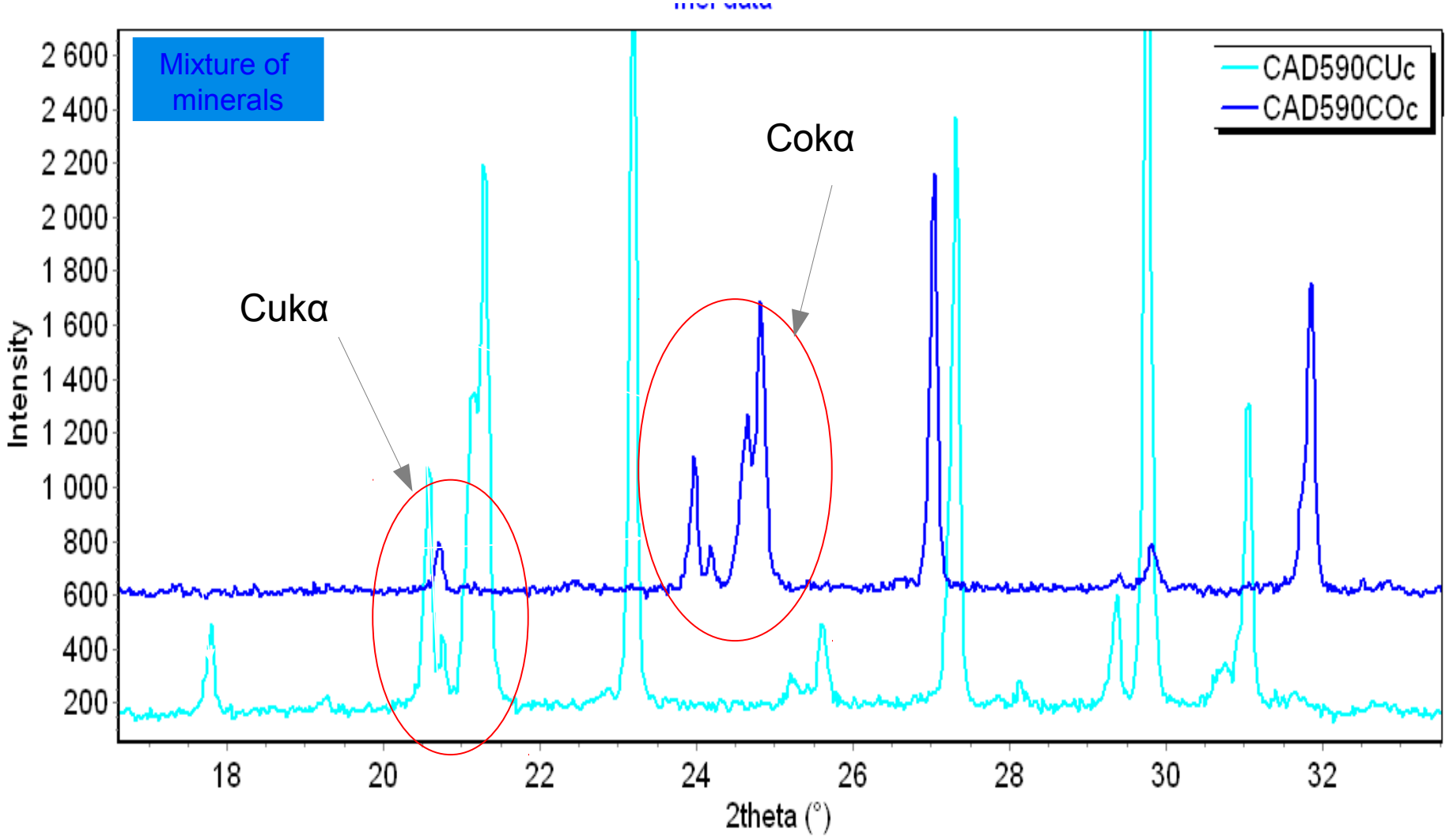
Focusing

Effect of optic: comparison between high resolution and high flux



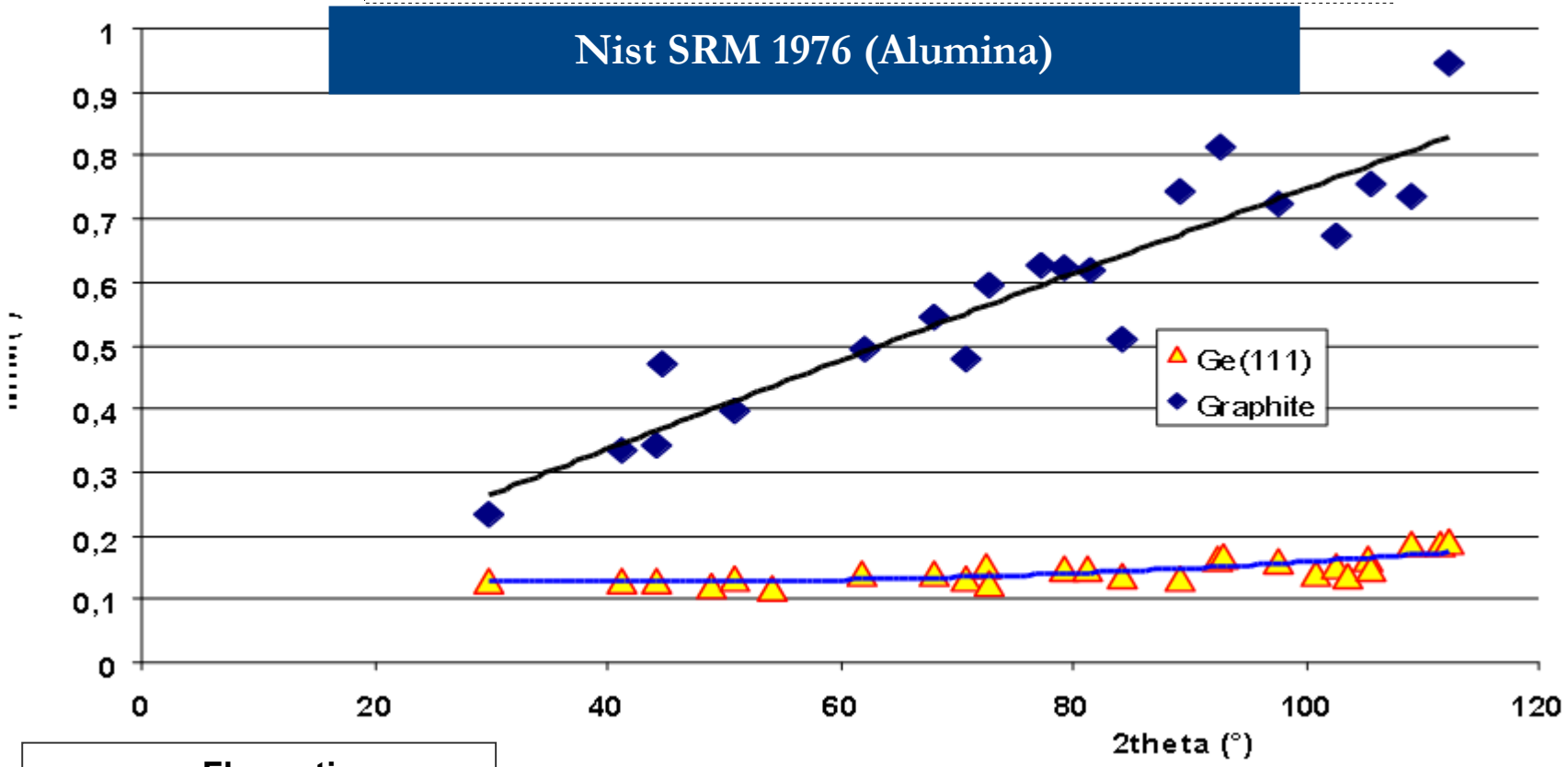


Effect of wavelength



Effect of optic

With graphite, ka_1/ka_2 doublet is considered as a single peak.



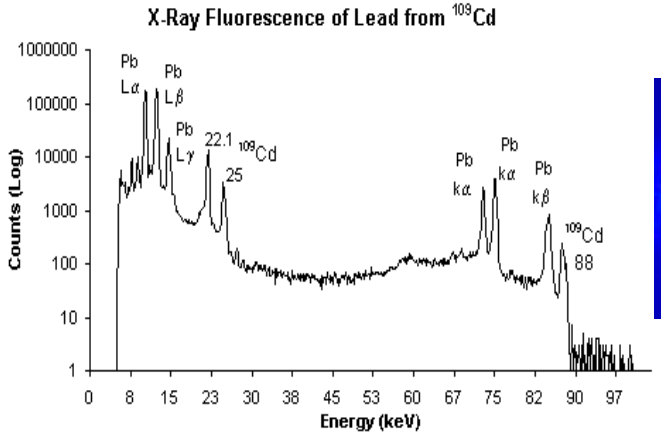
Flux ratio	
Ge(111)	1
Graphite	5
parab. Mirror	15
Elliptical mirror	20

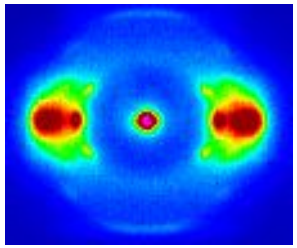
Function detection

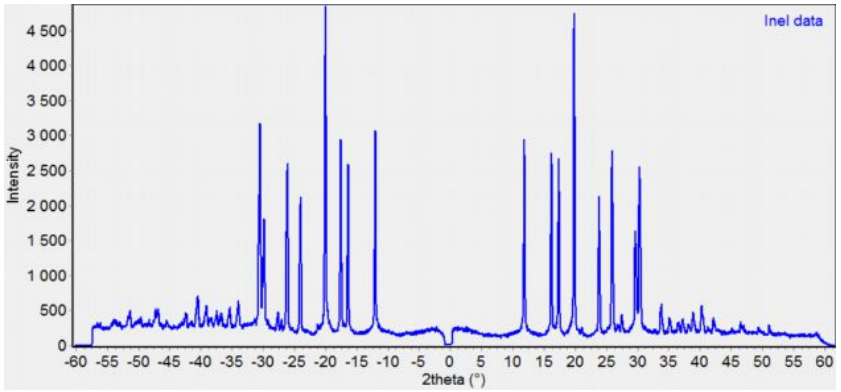
A detector is characterized by :
- spacial resolution
- dynamic range
- energy resolution
- dimension

Optimizing the characteristics of a detector allows to improve the measurement

	fluorescence	imaging	diffraction	reflection	diffusion
Spacial resolution	none	good	good	none	medium
Dynamic range	3	4~5	3~5	6~8	3~4
Energy resolution	~200eV	None Filtering possible	None or 1KeV	None or 1KeV	None or 1KeV
dimension	0D	2D (1D)	0D, 1D, (2D)	0D(1D)	1D-2D





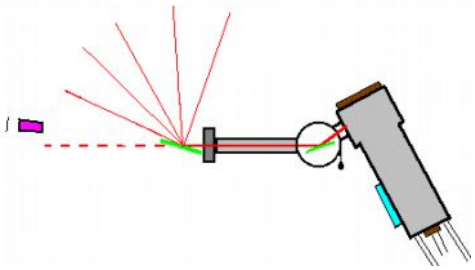


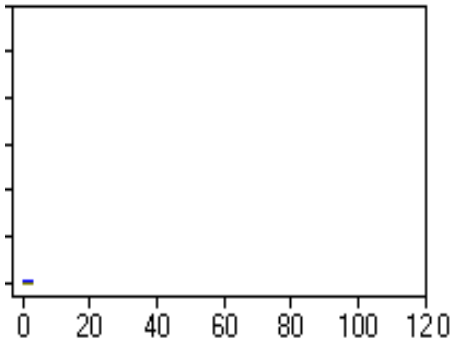
Function detection

0D Detection :

Acquisition is done Stepwise

2θ and statistics are time dependent

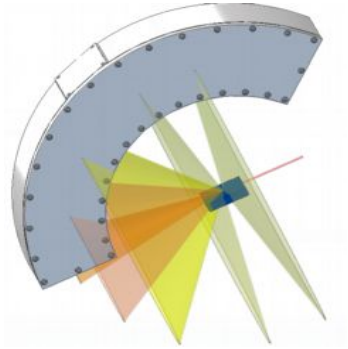


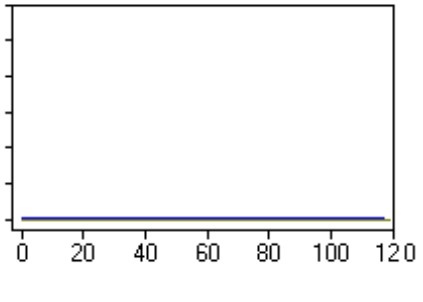


1D Detection :

Acquisition is done in snapshots

Statistics is time dependent



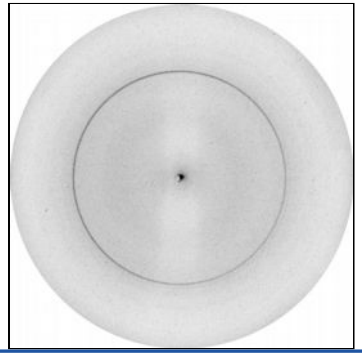


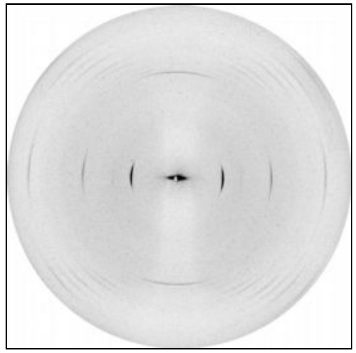
2D Detection :

Acquisition is done in snapshots

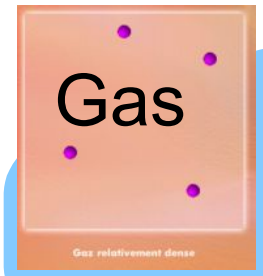
Statistics is time dependent

Texture information but point beam required



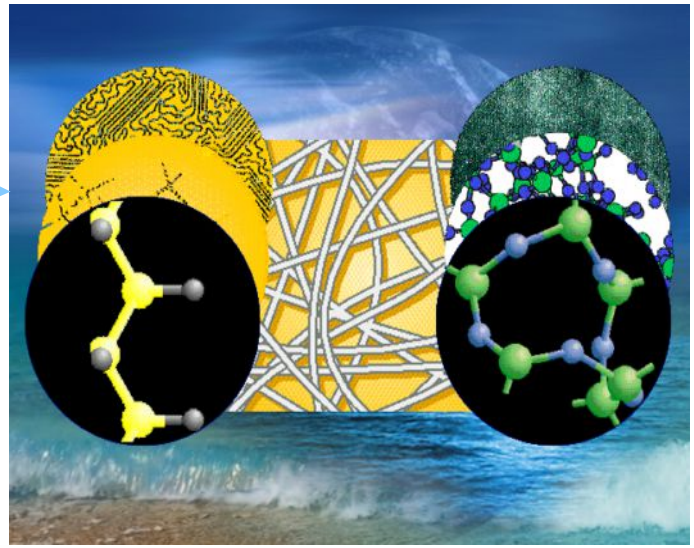


Function matter

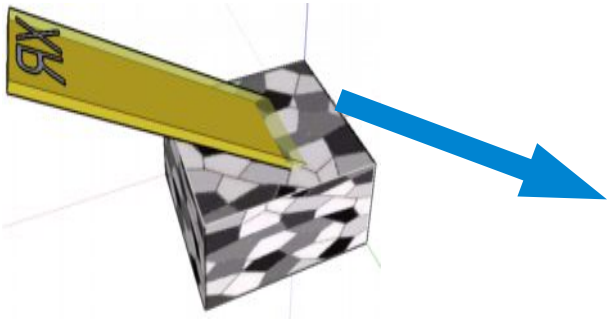


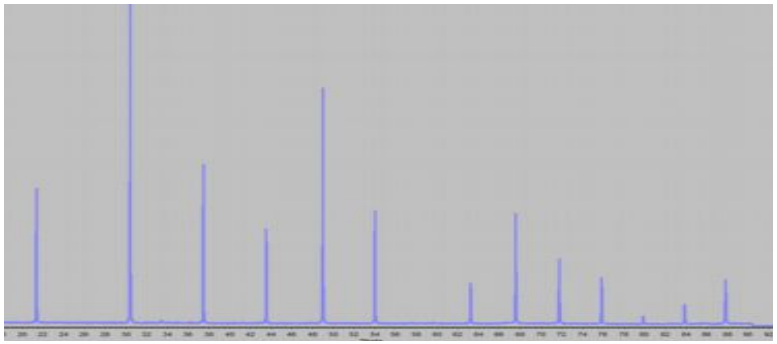
States of matter

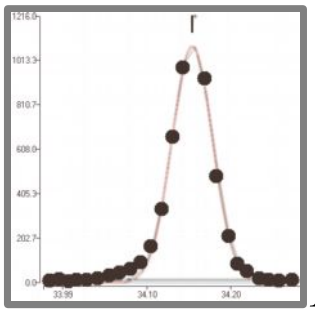
whatever the state,
XRD allows to evidence and measure
structural parameters in matter



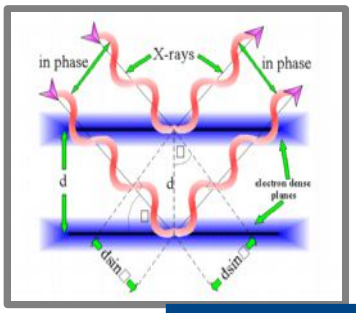
Informations obtained by XRD







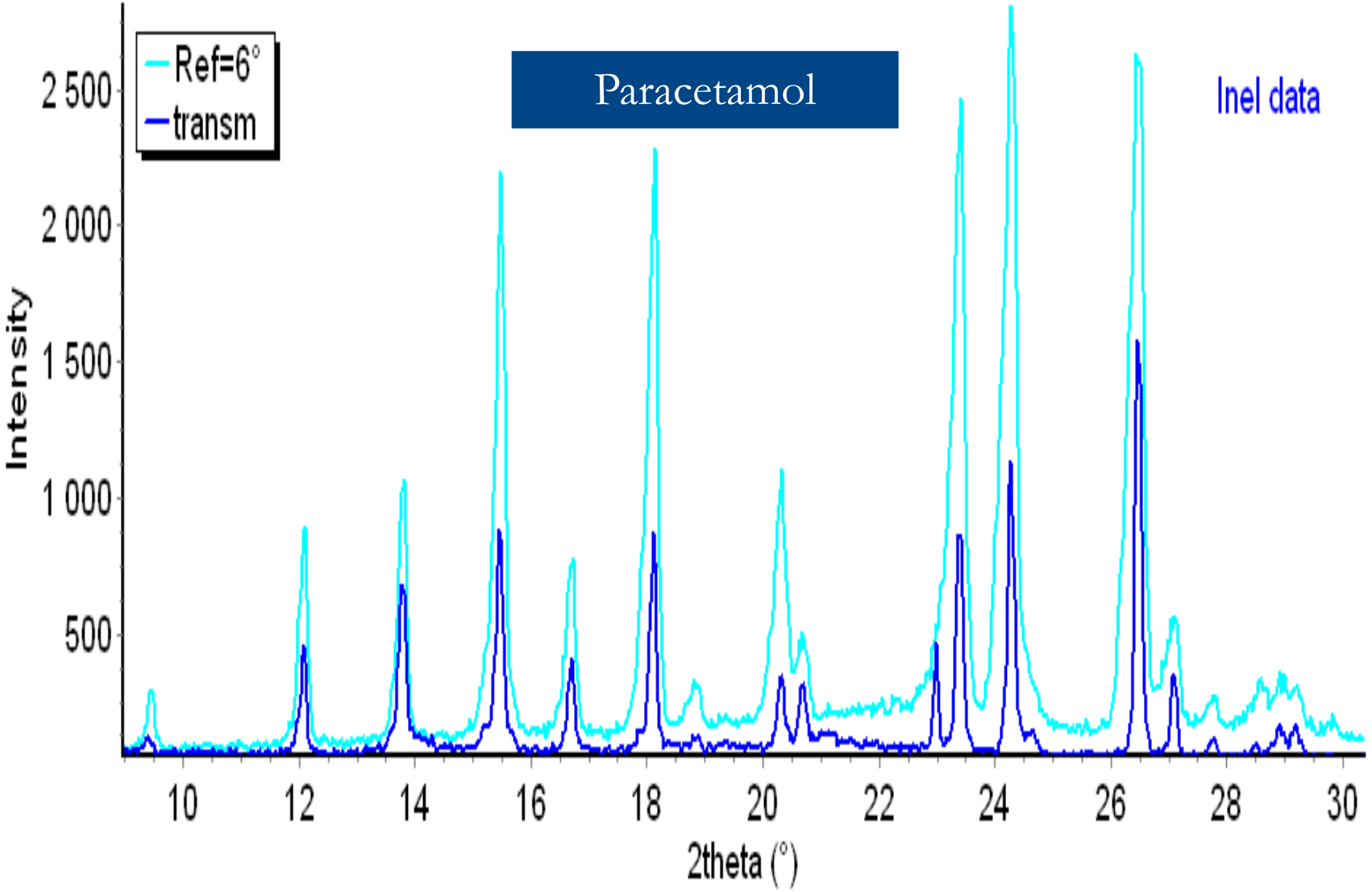
$$I = I_0 . P . L . F^2 . m . A . e^{-2M} \frac{.1}{V^2}$$



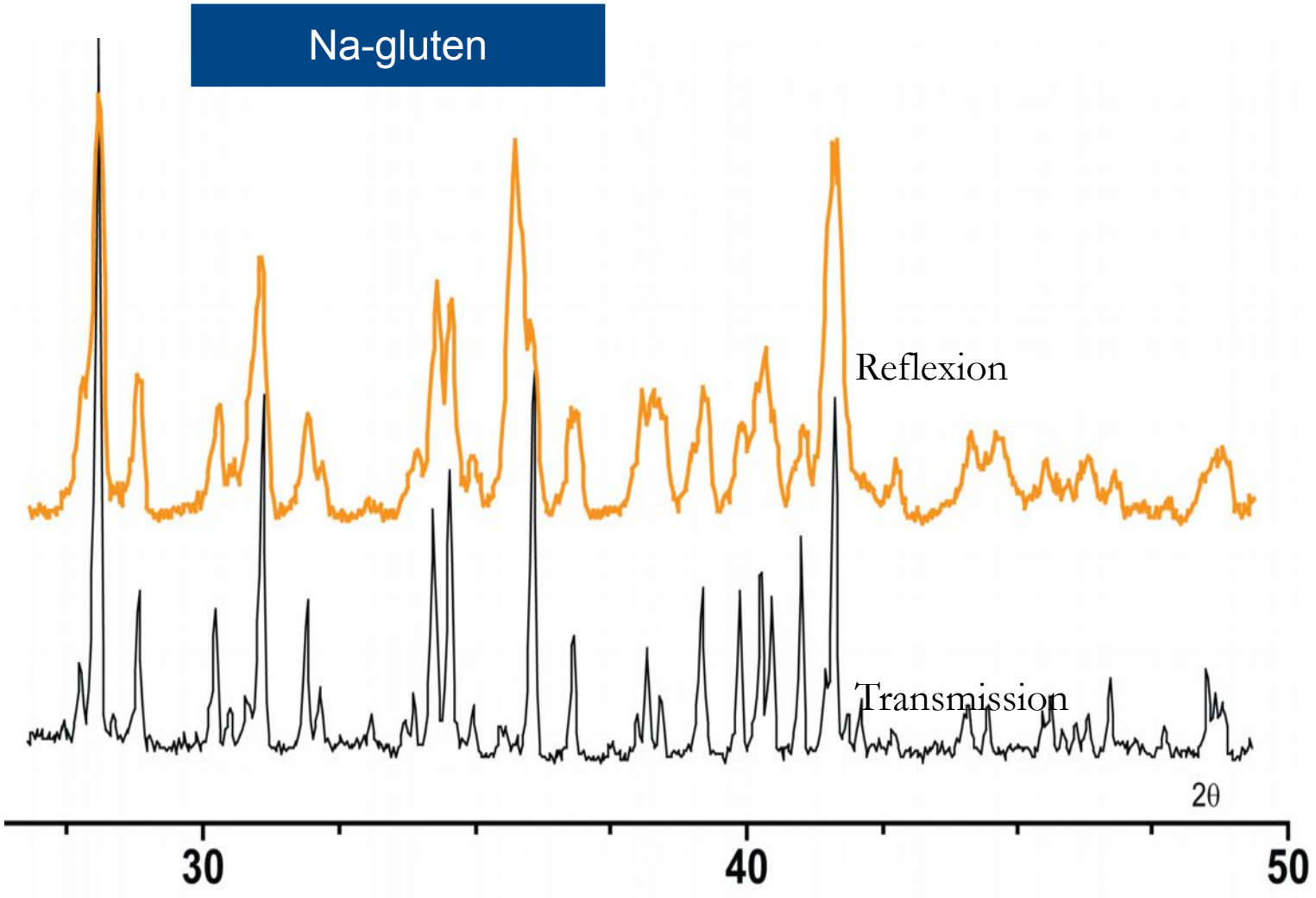
$\lambda = 2d_{hkl} \sin(\theta_{hkl})$

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



Function sample holder



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

X-ray diffraction setup

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, …

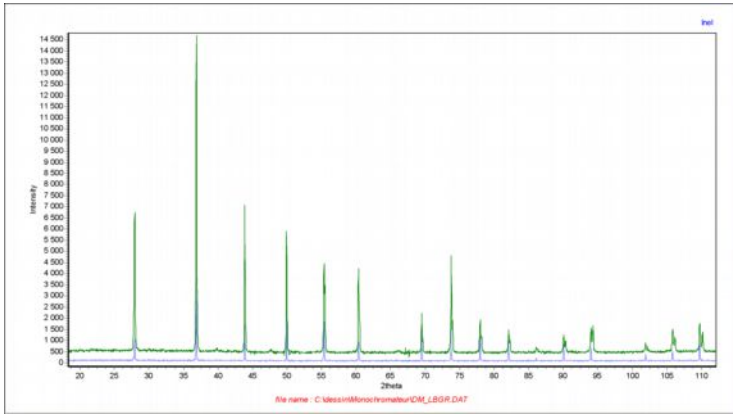
Cristallography

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

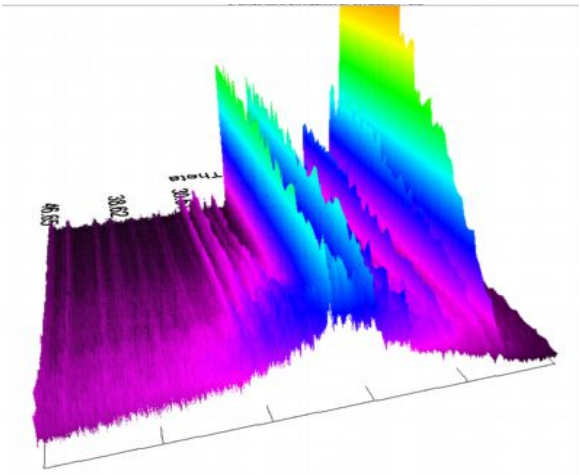
Temperature / Pressure
Phase transitions

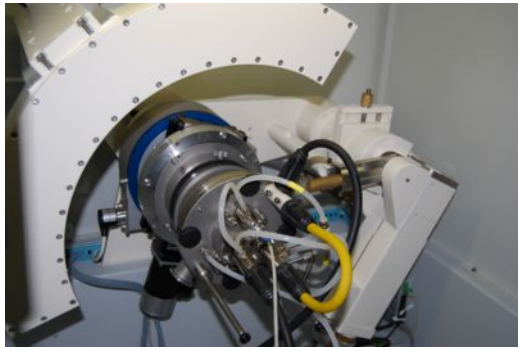
Electrochemistry
Following the phase transition : load, unload
from a battery (ageing, reversibility, functioning)

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

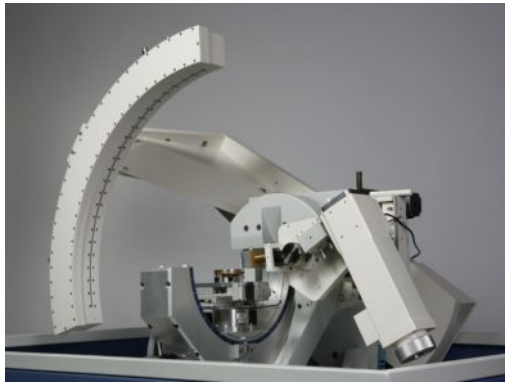
Industrial control on line
Cement quality control,
phosphate industries
Solar panel control

Following the product state in the time
Product evolution with the temperature and the humidity





X-ray diffraction 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)

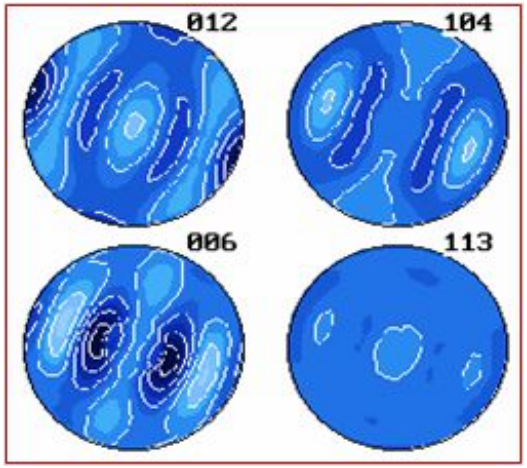
Micro-diffraction

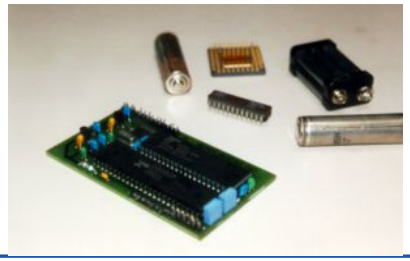
Homogeneity analysis of a bulk sample

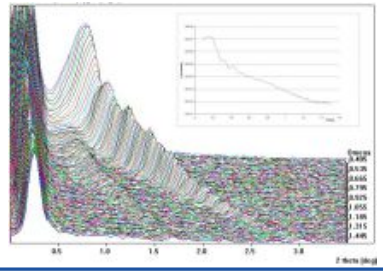
Grazing diffraction

Thickness measurement of a thin film

Deposition identification (irradiated zirconia)







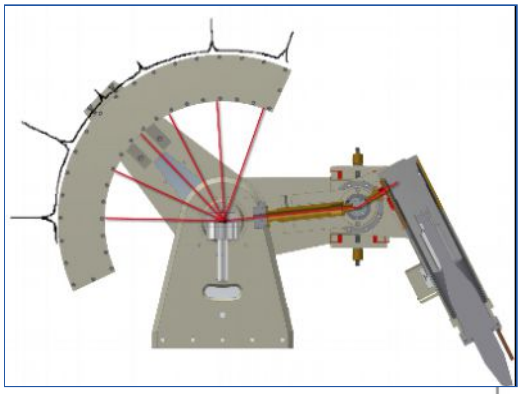
EQUINOX, a comprehensive real time XRD range

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	Laüe 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

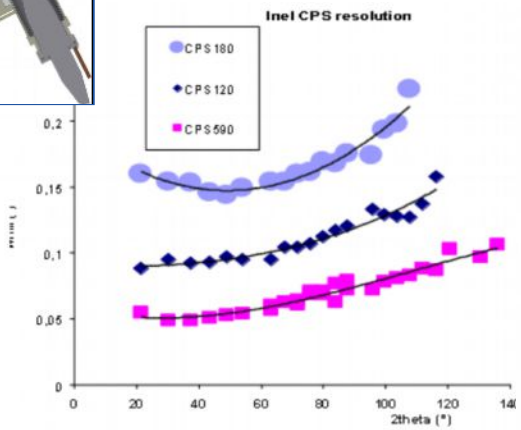
A unique detection mode

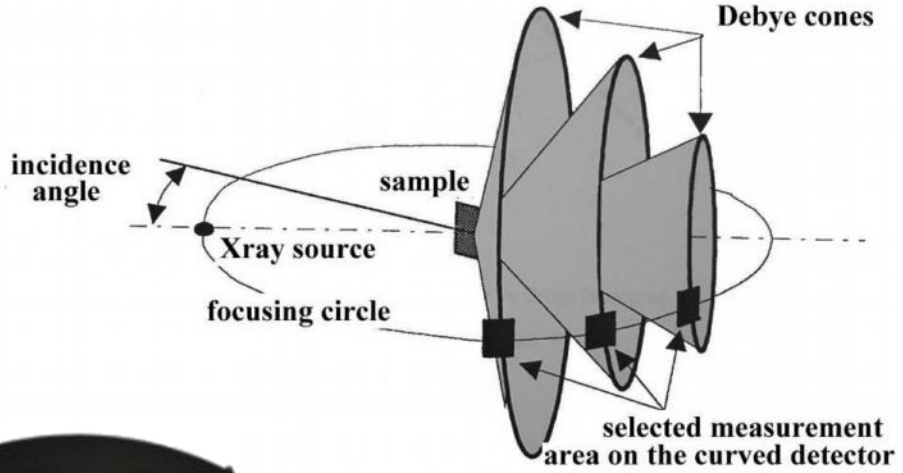


Curved detectors principle

The EQUINOX diffractometers use the curved detectors principle, namely real time acquisition across a wide acquisition range.

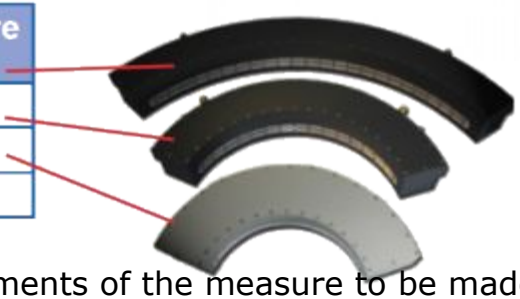
- No motorization required: neither on sample nor on detector incidence
- Asymmetric acquisition mode: for a fix θ sample incidence you can see all diffraction peaks on 2θ on the detector





3 detector types

	Detection angular range	Curvature (mm)
CPS 590	90° 2θ	R = 500
CPS 120	120° 2θ	R = 250
CPS 180	180° 2θ	R = 180



Analysis speed
& resolution
No maintenance

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

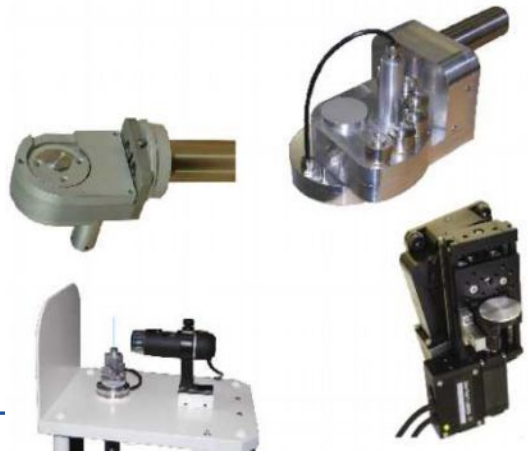
A stand alone benchtop X-ray diffractometer

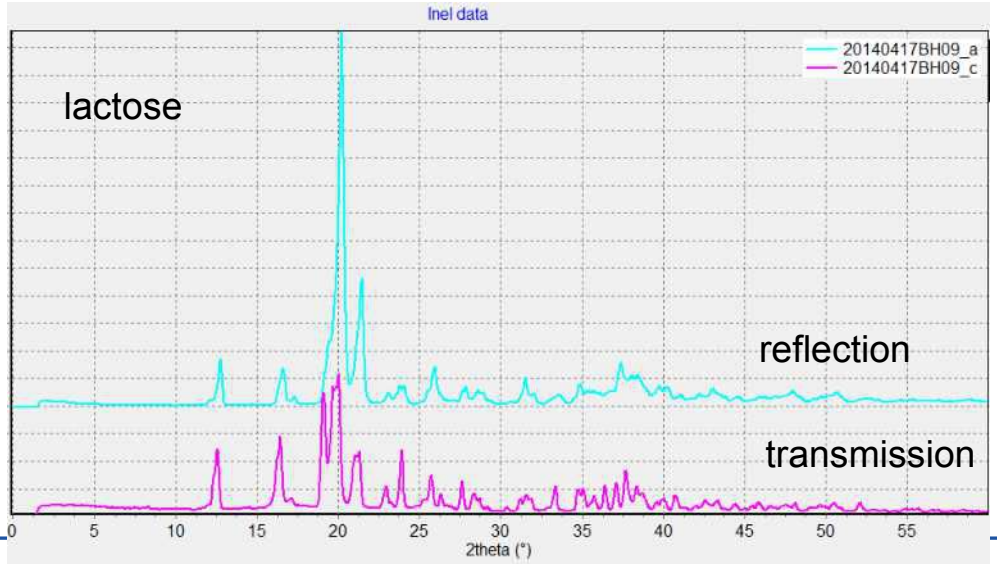
Designed for crystalline phases analysis (qualitative, quantitative, structural…) on powder or bulks.

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...



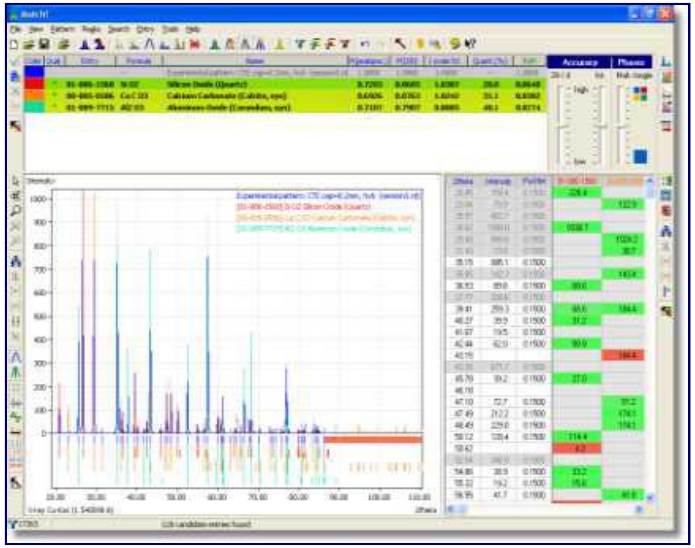


EQUINOX 1000, benchtop XRD

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.



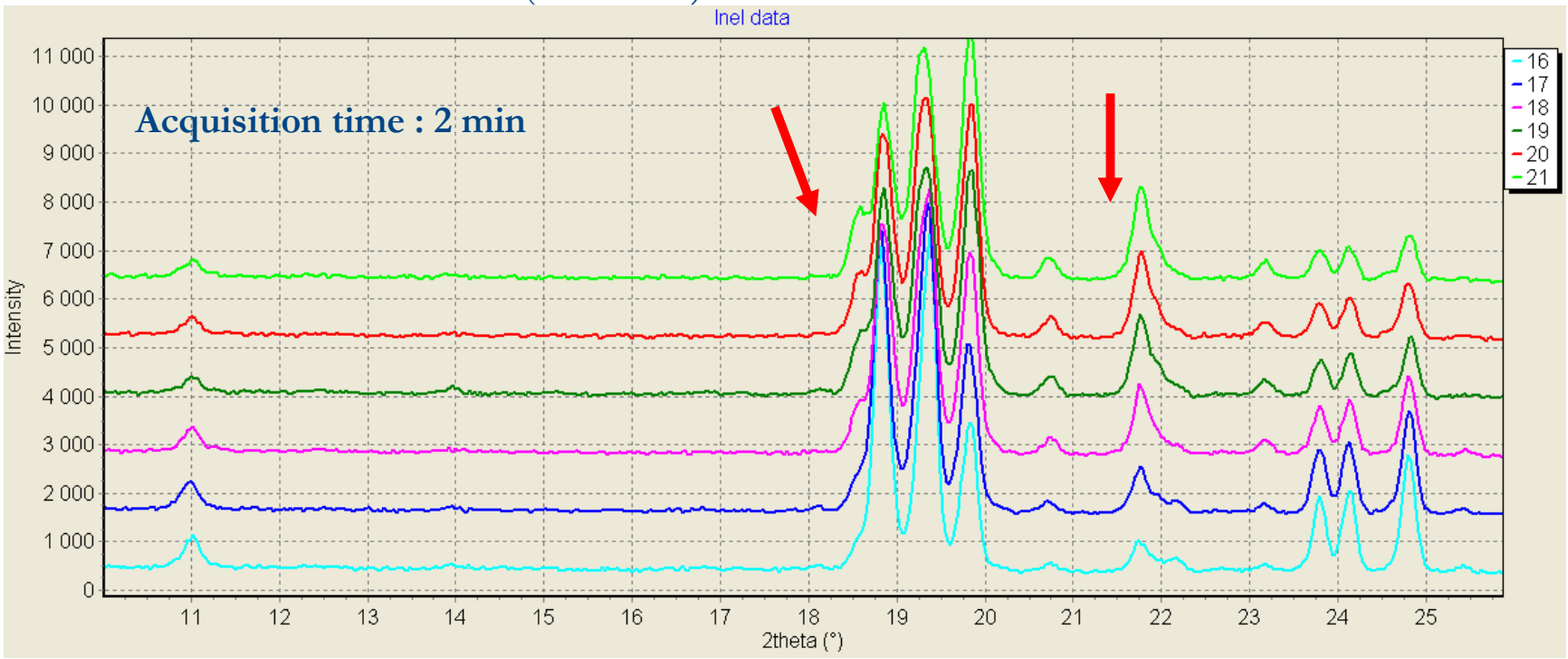
- Real time detection across 110°/2θ (CPS180)
- Monochromatic optics
- 3500 Watts generator
- External water cooling
- Working wavelength : Copper or Cobalt

Applications:

Qualitative & quantitative analysis, phases identification, structure determination, crystallites orientation...

Quality control in detergent

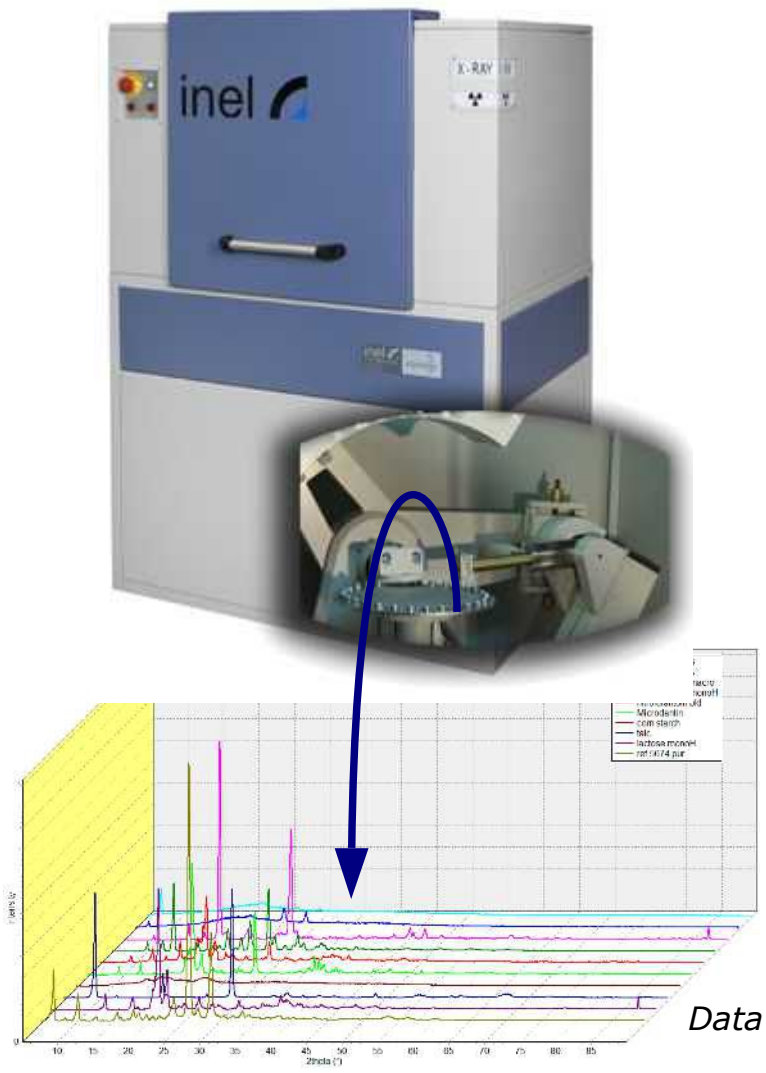
On the graph below, are represented diffractograms recorded for each sample. We can clearly evidence an increase of STPP 1 with the index (red arrows).



Sample	16	17	18	19	20	21
STPP I	17,67%	25,9%	40,9%	51,35%	51,17%	60,2%
STPP II	82,33%	70,7%	56,93%	45,23%	46,57%	37,93%
Other	0,0%	3,3%	2,17%	3,43%	2,27%	1,87%

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, …

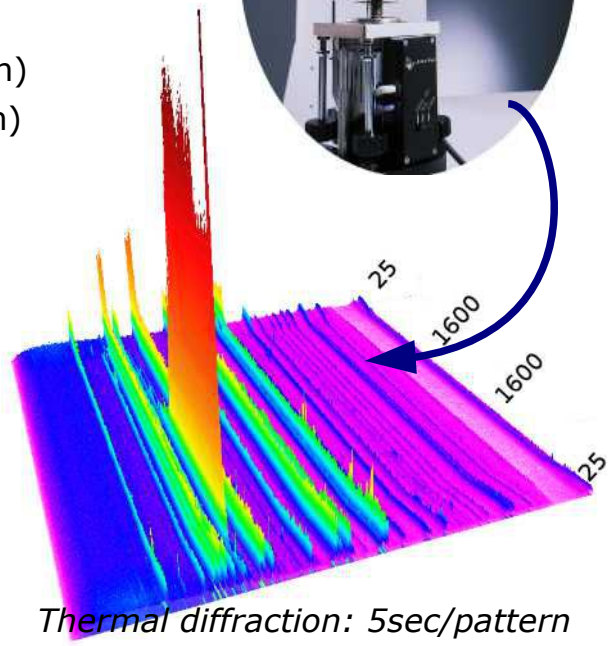
Data collection with sample changer

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)

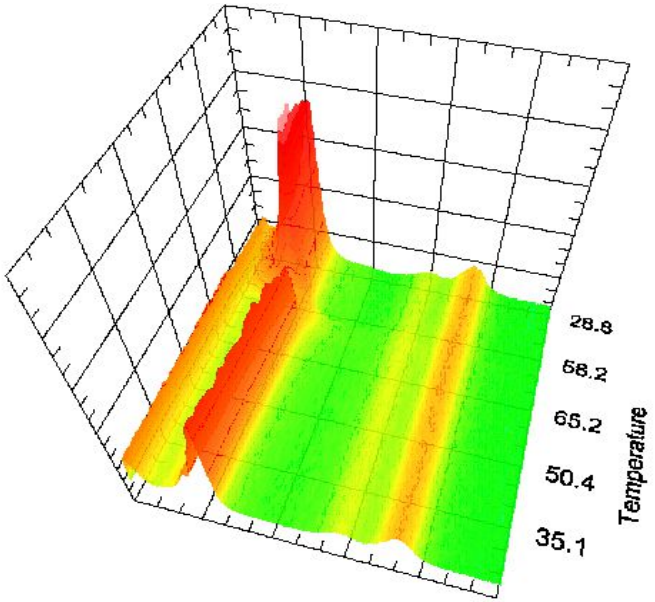


Thermal diffraction: 5sec/pattern

Applications

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

Phase transition at low 2θ



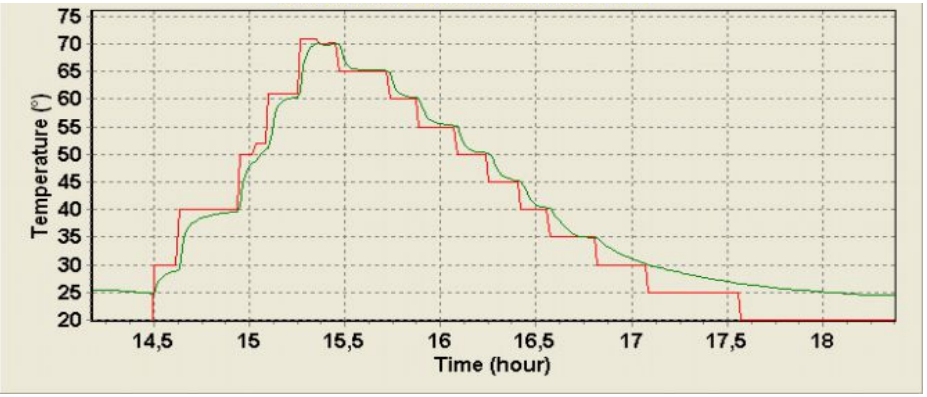
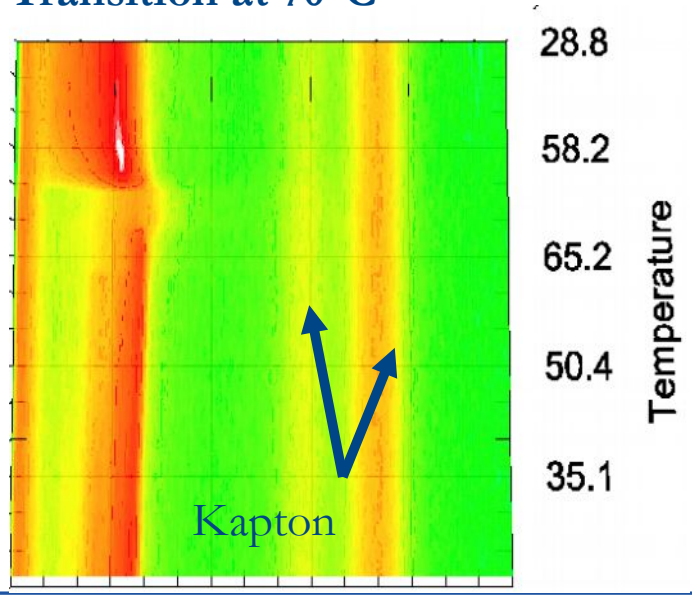


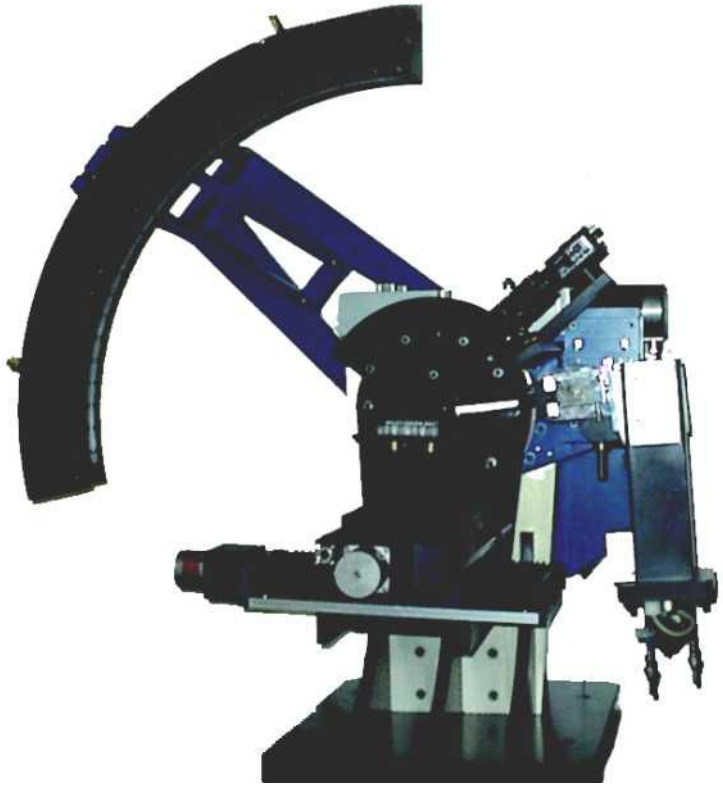
figure 1 : temperature profile : from RT to 70°C step 10°C and every 5°C while cooling down.

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



EQUINOX 4000, DRX microdiffraction / cartographie

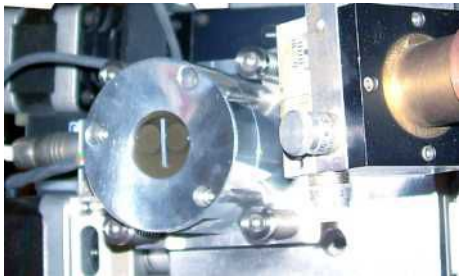


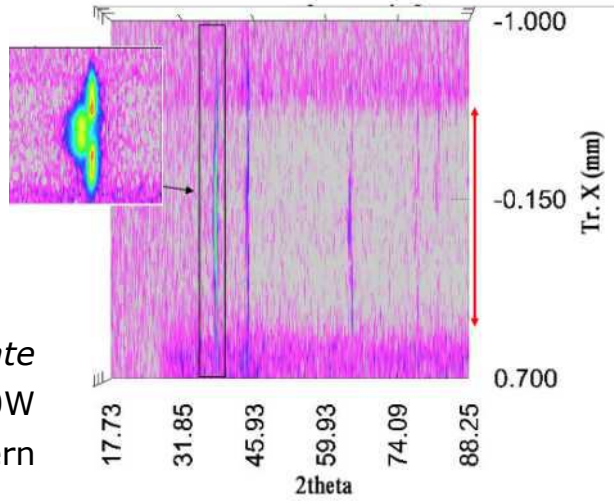
EQUINOX 4000 is designed for all your microdiffraction or mapping applications. The instrument uses X, Y and Z translation stages with strong travel.

- Real time detector (CPS 120)
- θ / 2θ 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

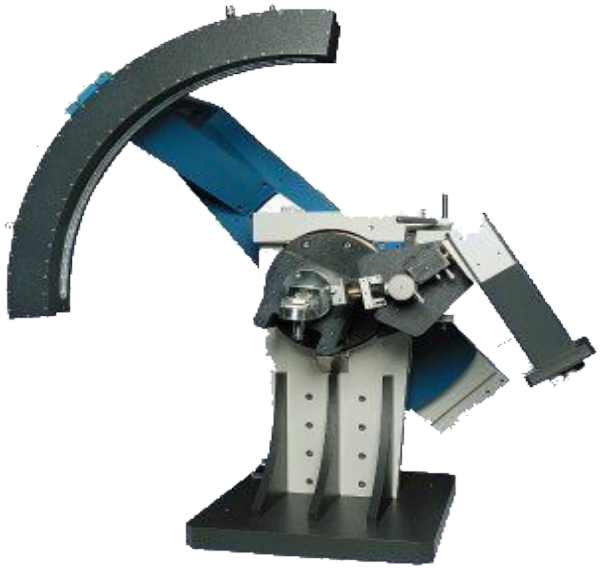
Acquisition : 120sec/pattern

EQUINOX 5000, high resolution XRD

A high resolution diffractometer

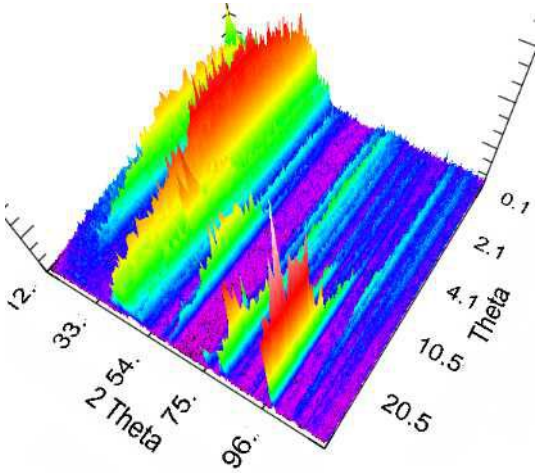
for all your powder analysis under variable temperature and/or atmosphere that require a very high resolution.

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



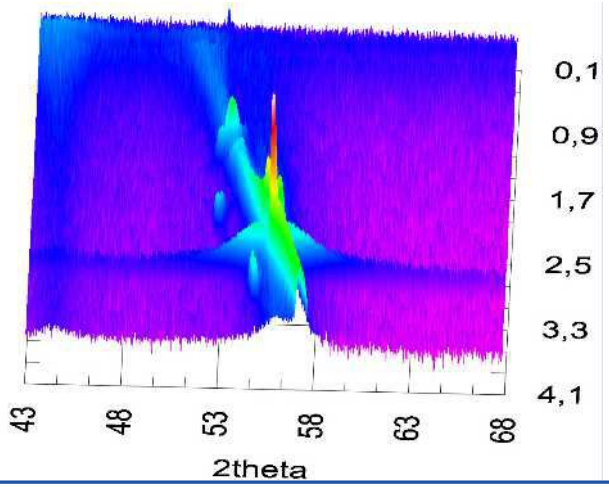
Applications

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 (θ, 2θ, χ, ϕ)
- 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…

EQUINOX 6000, sample holder

Sample characteristics :

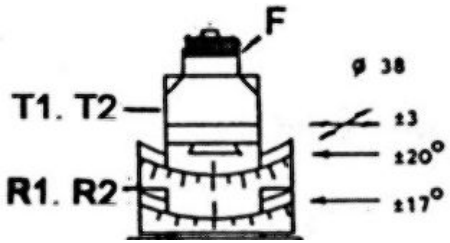
- Diameter max =128mm
- Thickness max = 10mm
- Weight max = 1kg

Extension :

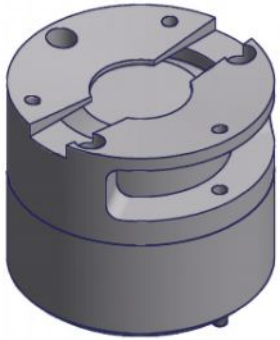
- Possibility to set the furnace : DHS 1100 AP

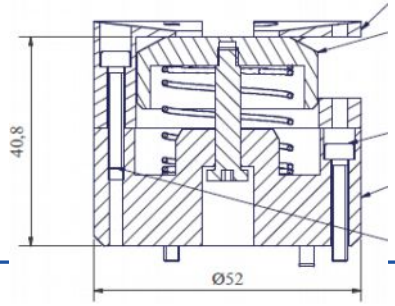
Sample holders

Goniometer head

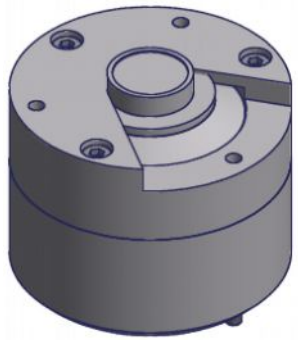


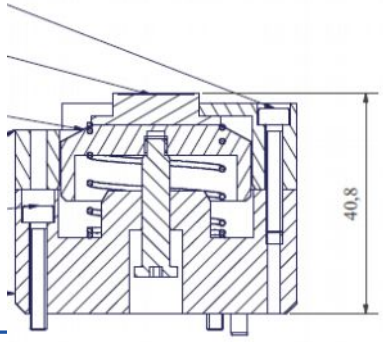
For bulk (max30x30x8mm)



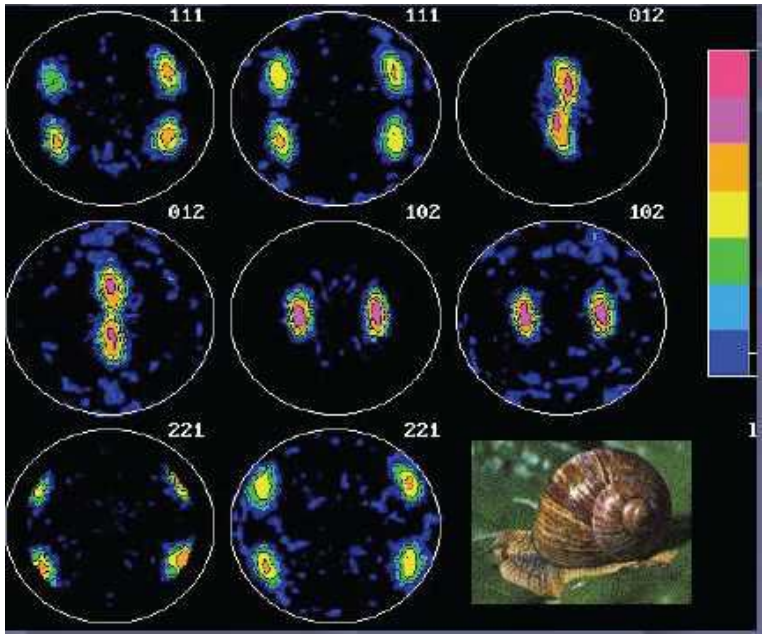


For powder





EQUINOX 6000, 4 circles texture/stress XRD



Helix pomatia (Burgundy land snail: Outer com. crossed lamellar layer), From D. Chateignier – CRISMAT (Caen)

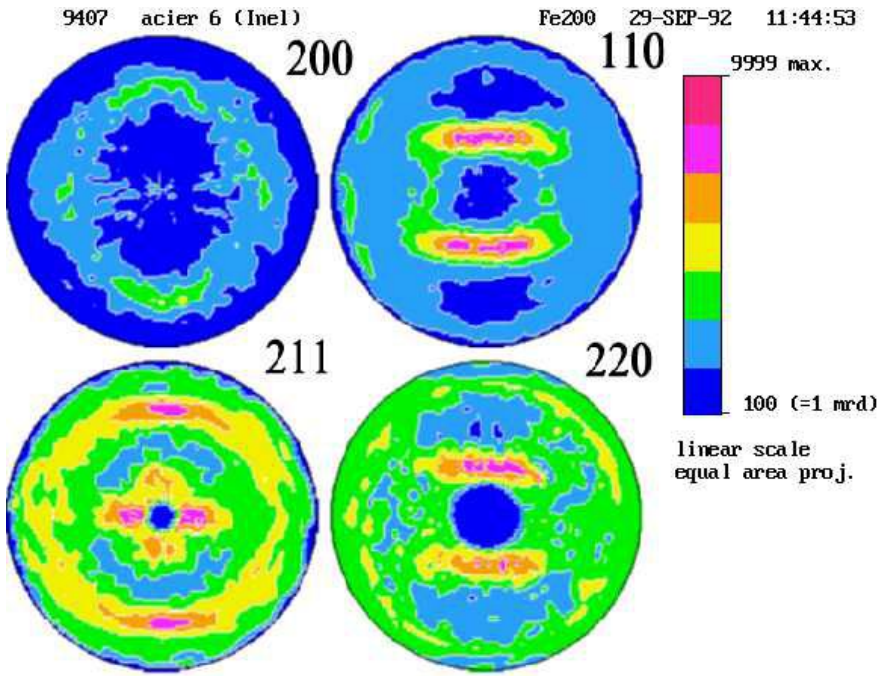
Experimental

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

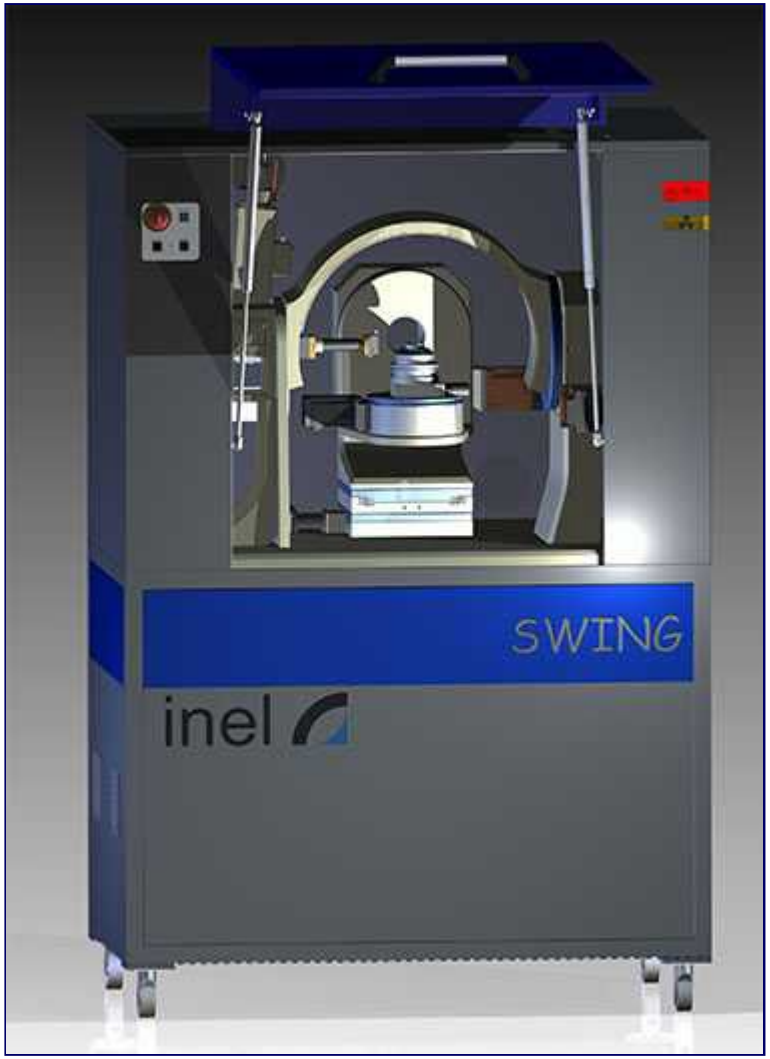
Absorption and defocusing corrections

Chi from 0° to 80° step 5°, phi from 0° to 355° step 5°



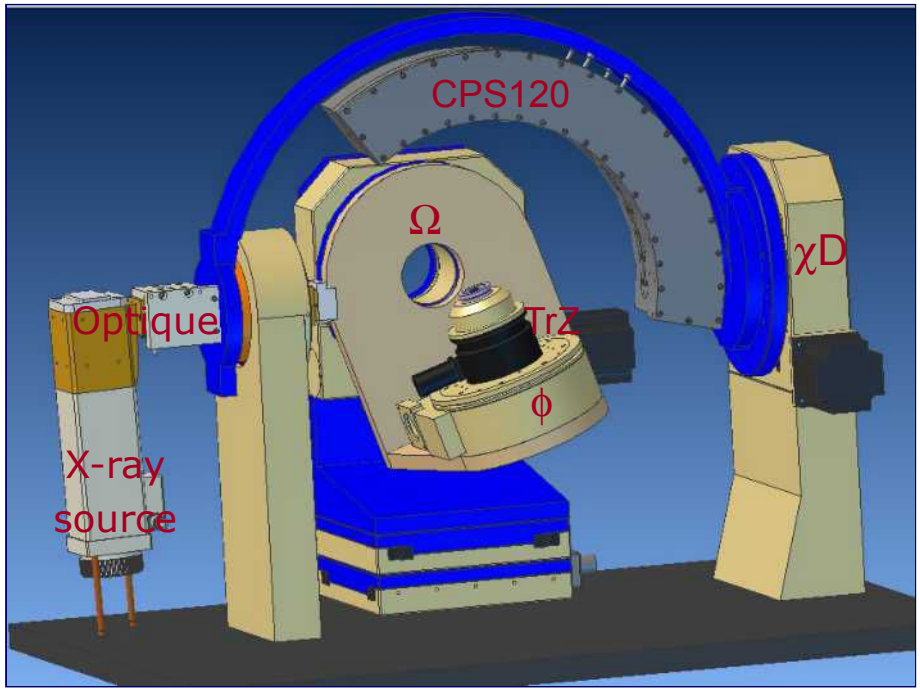
Texture analysis on stainless steel foil

SWING, texture/stress XRD

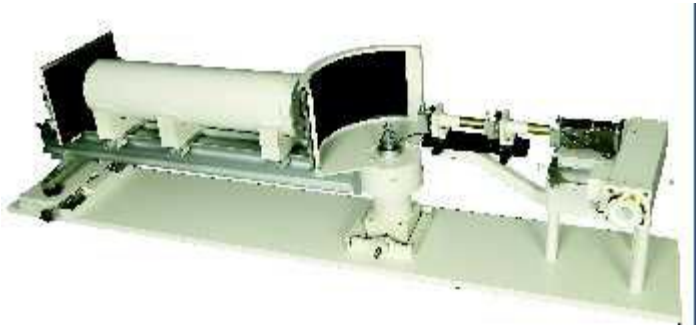


A decoupled goniometer

- No Chi motion on the sample goniometer
- A lot of space for the sample
- Diffraction plane rotates around the beam axis



SWAXS, SAXS WAXS instrumentation



Small Angles X-ray Scattering

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.

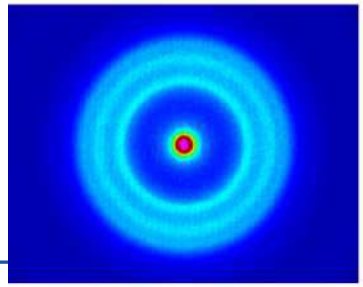
It can be used as well for research as for QA/QC.

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

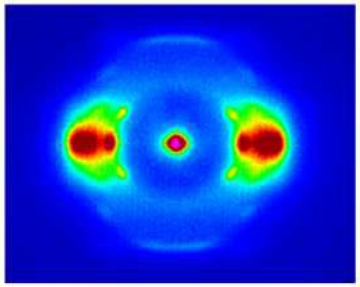
- 3500 Watts generator
- Diffraction bench with monochromatic optics
- Variable working wavelength

Applications

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



PET semicristallin (30%)



PET semicristallin (30%) déformation 1,4

Laüe back reflection diffractometer

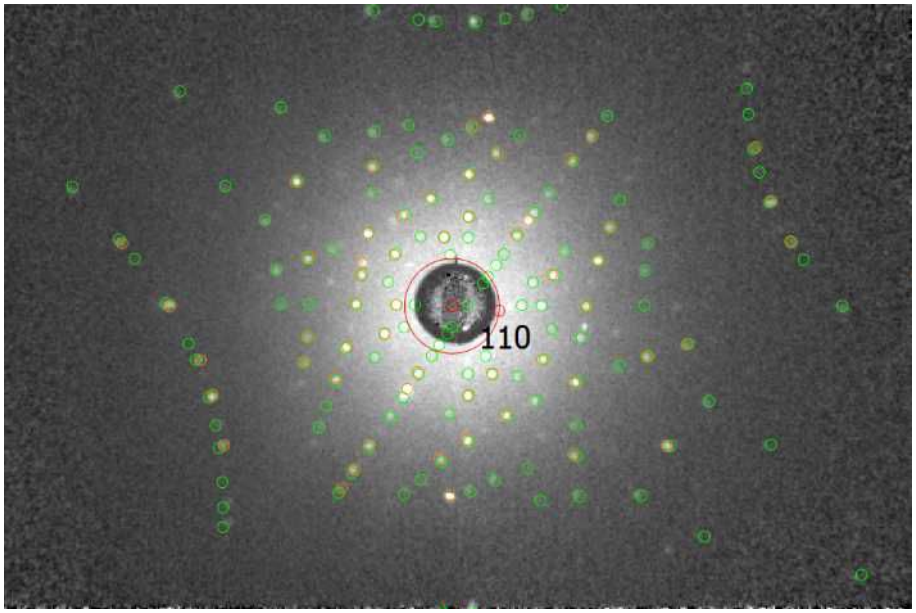
A fast tool for cristal orientation

Easy sample positionning

Fast acquisition

Fast orientation determination

Sample holder can be customized according to cristal



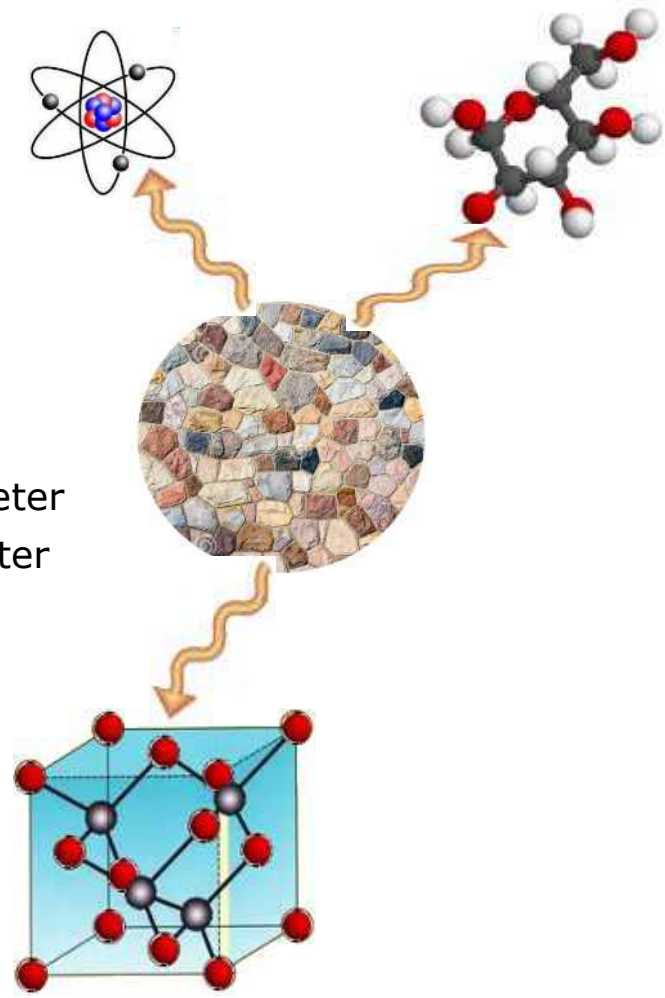
Si220 orientation

Instruments combination

Inel XRD flexibility

Combining X-ray diffraction with other techniques:

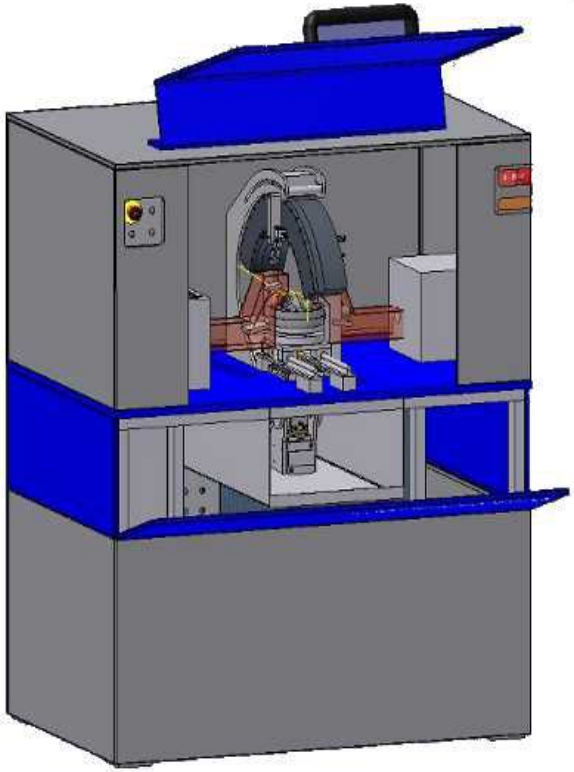
- One sample and several datas
- Real time analysis
- XRD comes to sample
- XRD for on line industrial control



Inel portable XRD systems:

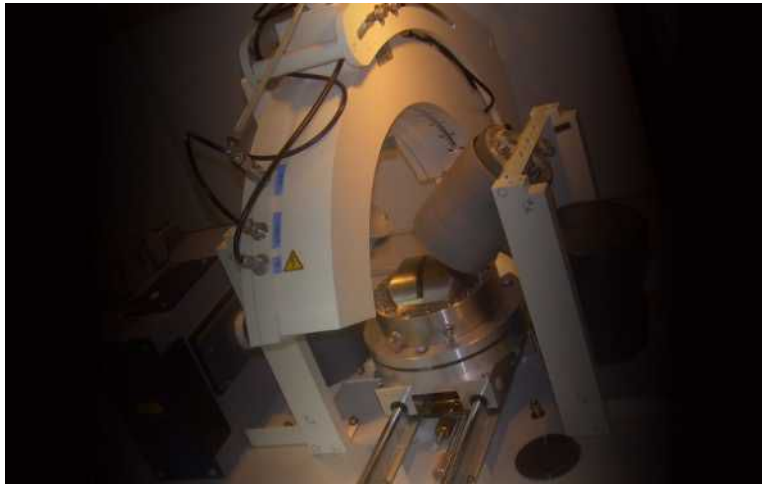
EQUIRAM	Combination of XRD and Raman spectrometer
XRD/DRIFT	Combination of XRD and DRIFT spectrometer
STRESS/WAXS	Combination of WAXS and stress analysis
SWAXS	SAXS WAXS diffractometer
COSMA	On-line production control XRD
PRECIX	Robotics for XRD residual stress measures

XRD/DRIFT combination

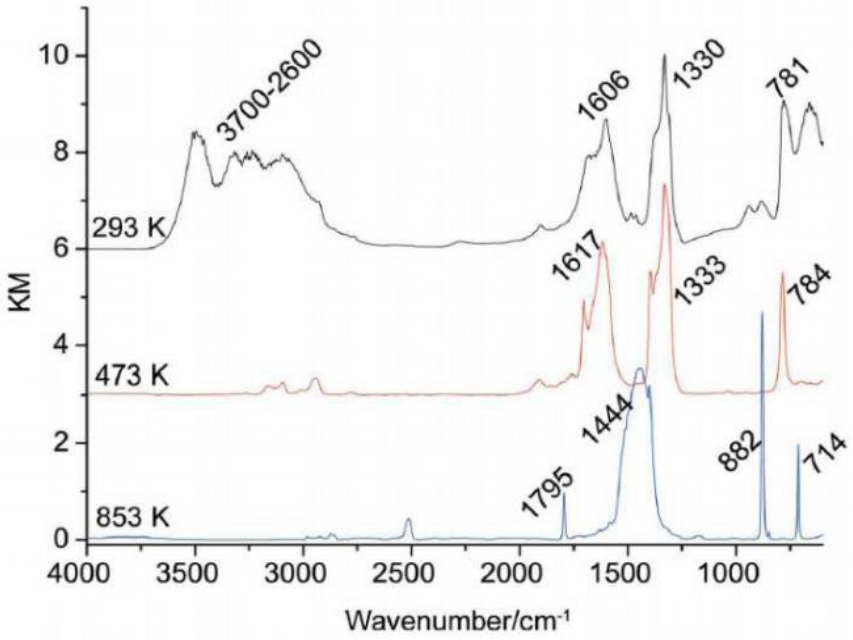


Phase transitions observation

- 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.

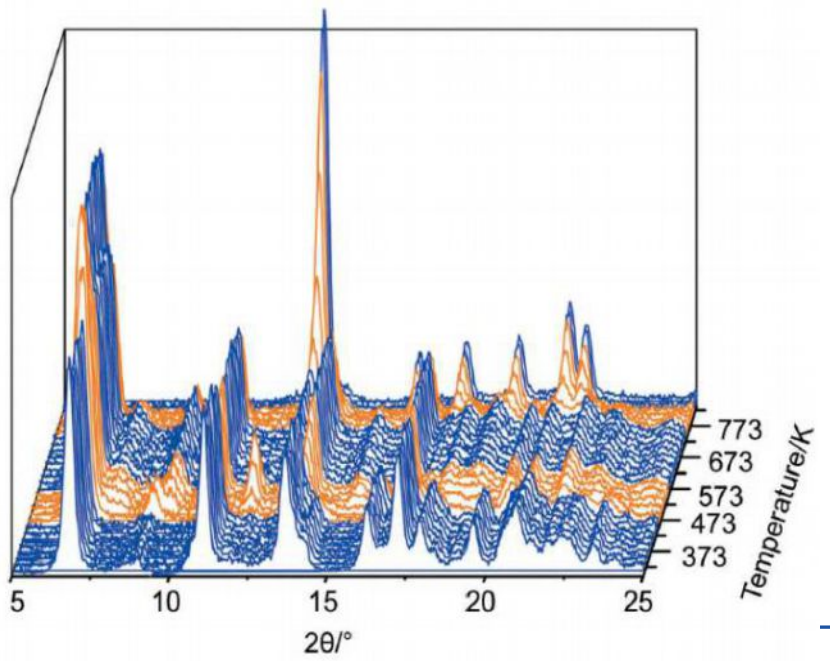


XRD/DRIFT combination



DRIFT spectra recorded during thermal decomposition of calcium oxalate (50 mg, 5 Kmin1).

X-ray diffraction patterns recorded during the thermal decomposition of calcium oxalate (50 mg, 5 K min1) Blue traces represent stable crystallographic phases and orange diffraction traces indicate intermediate crystallographic structures.



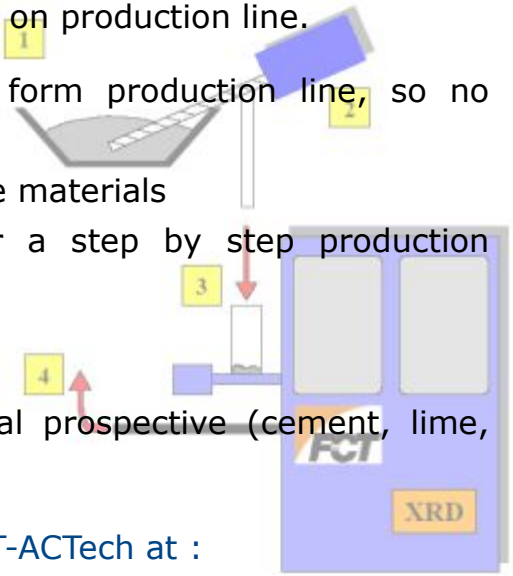
"*An X-ray diffractometer coupled with diffuse reflectance infrared Fourier transform spectroscopy and gas chromatography for in situ and in operando characterization: an innovative analytical laboratory instrument*", L. Braconnier, I. Clemencon, C.Legens, V.Moizan, F.Diehl, H. Pilliere, P. Echegut, D. De Sousa Meneses and Y. Schuurman, J. Appl. Cryst. (2013). 46, 262–266

COSMA, on-line XRD control

"*On-line X-ray diffraction for quantitative phase analysis: Application in the Portland cement industry*", N. Scarlett, I. Madsen, C. Manias, D. Retallack, Powder Diffr. Vol.16, No 2, June 2001

On-line real time XRD measures

The industrial production performance (cement, lime, sand, aluminosilicates...) depends on them 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.



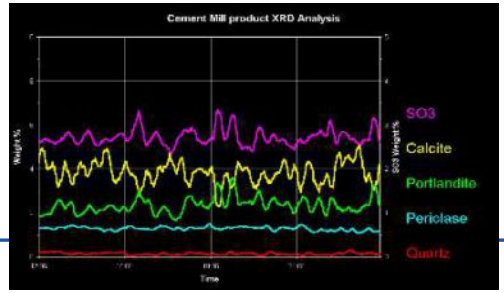
- Continuous powder flow extract form production line, so no more laboratory analysis required
- Real time mineralogy on crystalline materials
- Every minute results update for a step by step production quality follow-up

Applications

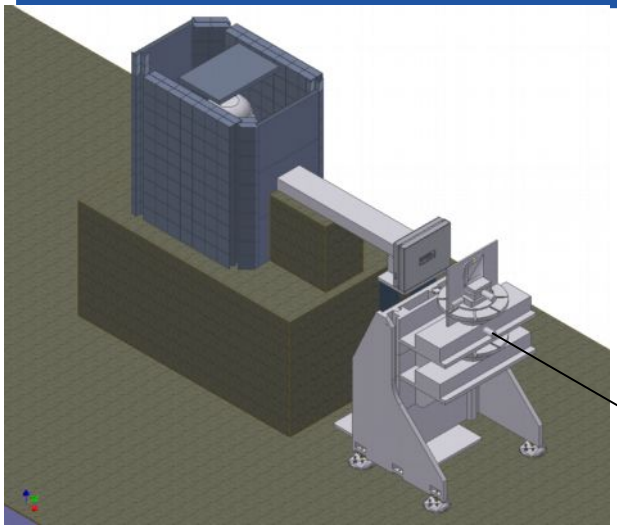
Powder manufacturers from a global prospective (cement, lime, sand, aluminosilicates...)

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

C_3S, C_2S, C_3A, C_4AF, CaO, $Ca(OH)_2$, $CaCO_3$, $CaSO_4.2H2O$, $CaSO_4.{}^1/_2H2O$, Other minerals

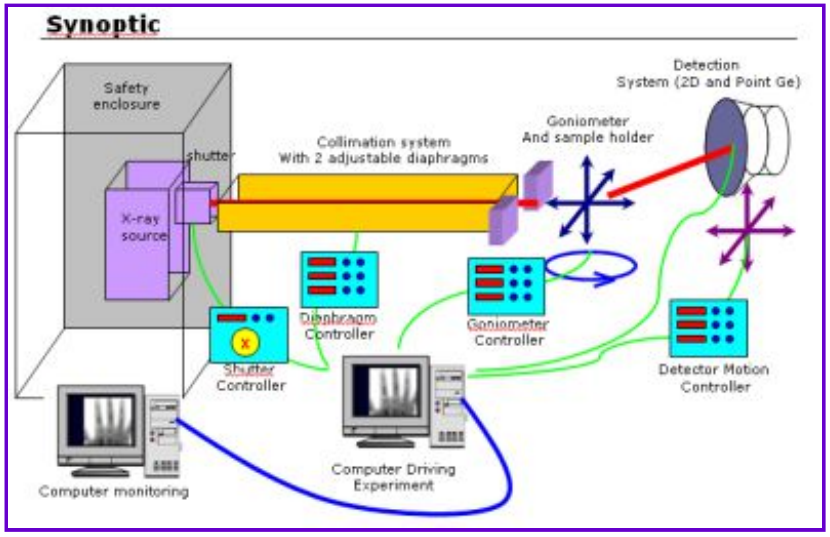


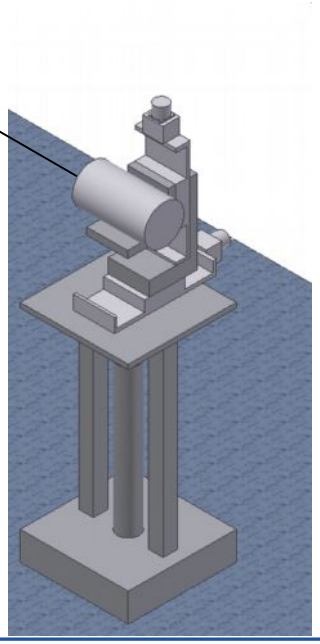
High energy X-ray diffraction



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

From 100 to 200keV beam





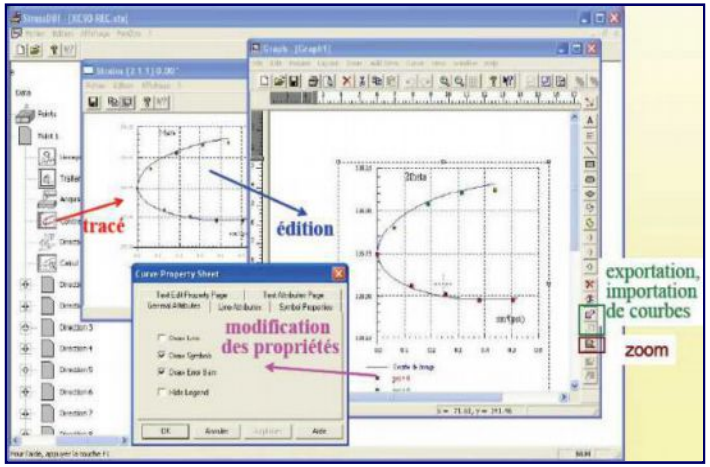
PRECIX, residual stress XRD robot

Robotized residual stress analysis

PRECIX instrument is based on a last generation 6 axis industrial robot.

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.



Software, StressDiff, J.M. Sprauel

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 …

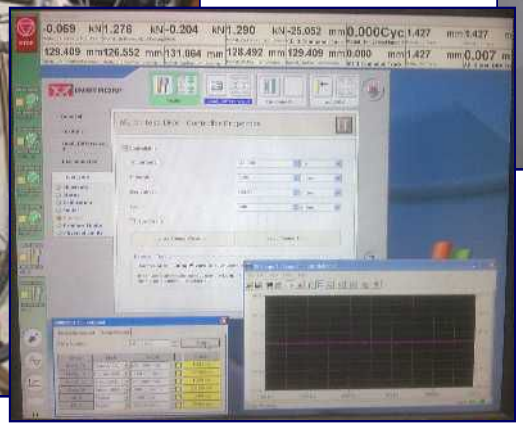
Advantages

- 1 sample for several measurements, but 1 result

- eliminating non possibilities (XRF prefiltering before search match)

- ...

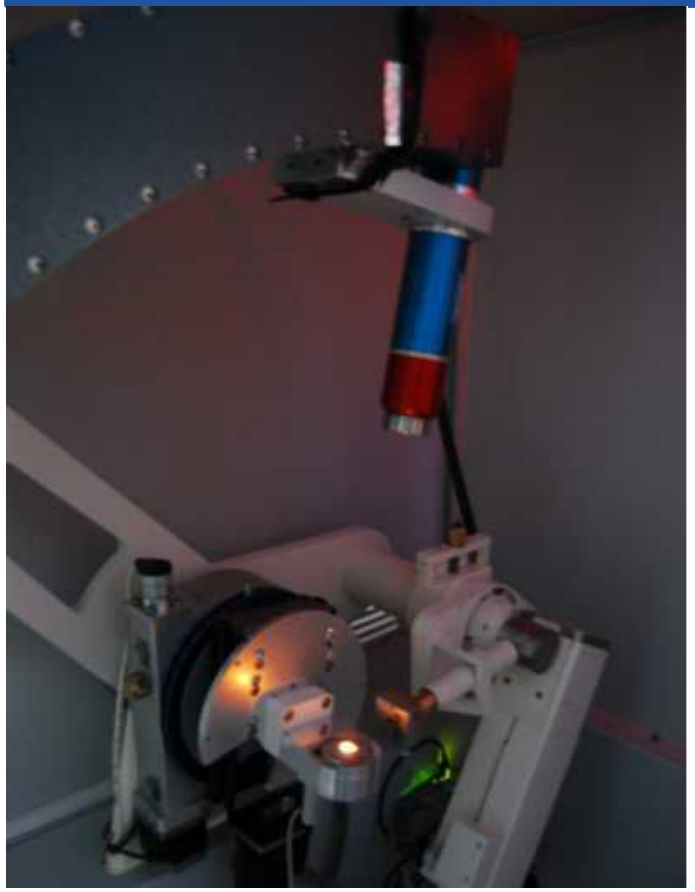
Stress/WAXS combination



In situ XRD system on a traction machine, ASTREE

Allows to perform residual stress measurements and phase identification

XRD/Raman combination



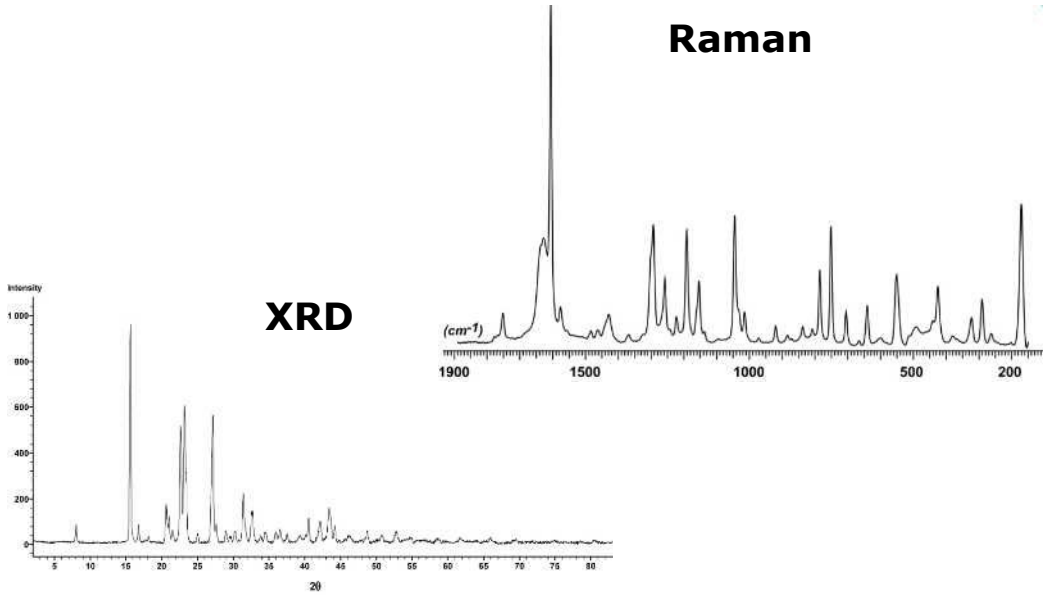
KAISER OPTICAL SYSTEMS, INC.

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

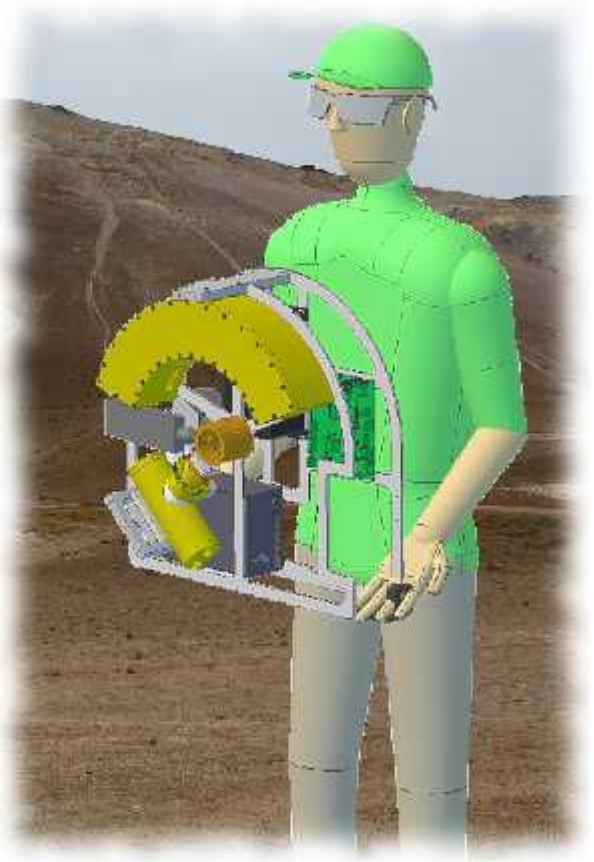


Since 2010, wedlock between PSD detector and X-ray minisource

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

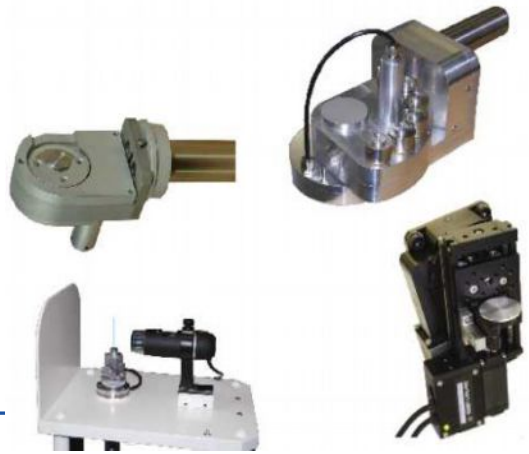
A stand alone benchtop X-ray diffractometer

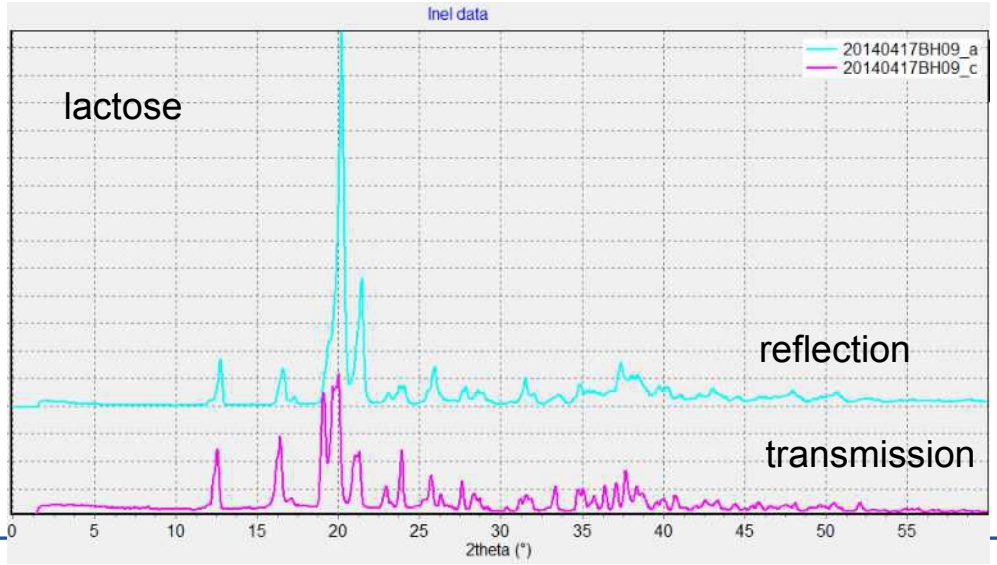
Designed for crystalline phases analysis (qualitative, quantitative, structural…) on powder or bulks.

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...





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.

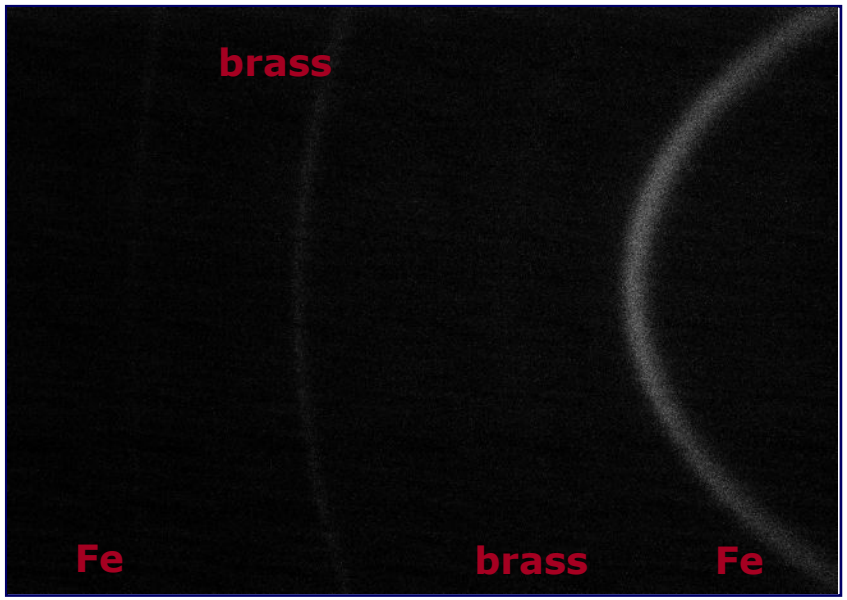
- Whole system ≈ 10kg et 25 cm
- Easy to use
- 2D 24 x 34mm detector
- Anode source Cr 4 Watts
- Angular range: 90 to 170° 2θ
- Pixel size 20µm
- CCD Peltier cooling system
- «Phi» axis motorization (Texture)
- «Psi/Omega» angular sensor
- Working distance: ≈ 22mm

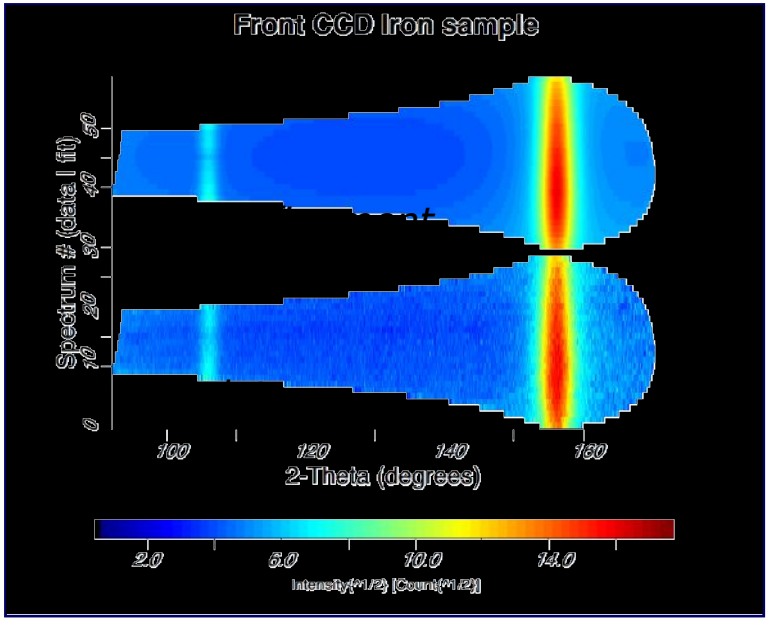
Partnership between
Inel / Université de Trento, Italie

Applications

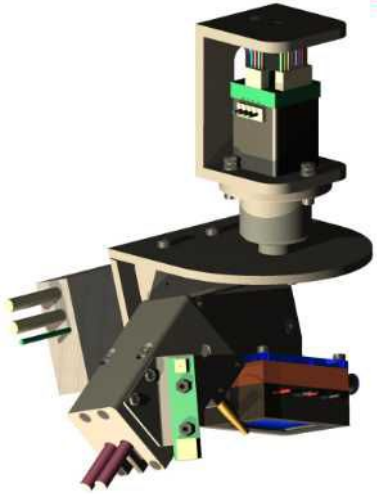
Welds control in the field : pipelines, bridges, nuclear reactor facilities..., On-site critical industrial parts control: aerospace, automotive..., Determination of critical failures in damaged important structures, Validation of repaired area as «restored» to original specifications, and even more...

Xsolo, the nomadic stress system





XR diffraction image from CCD

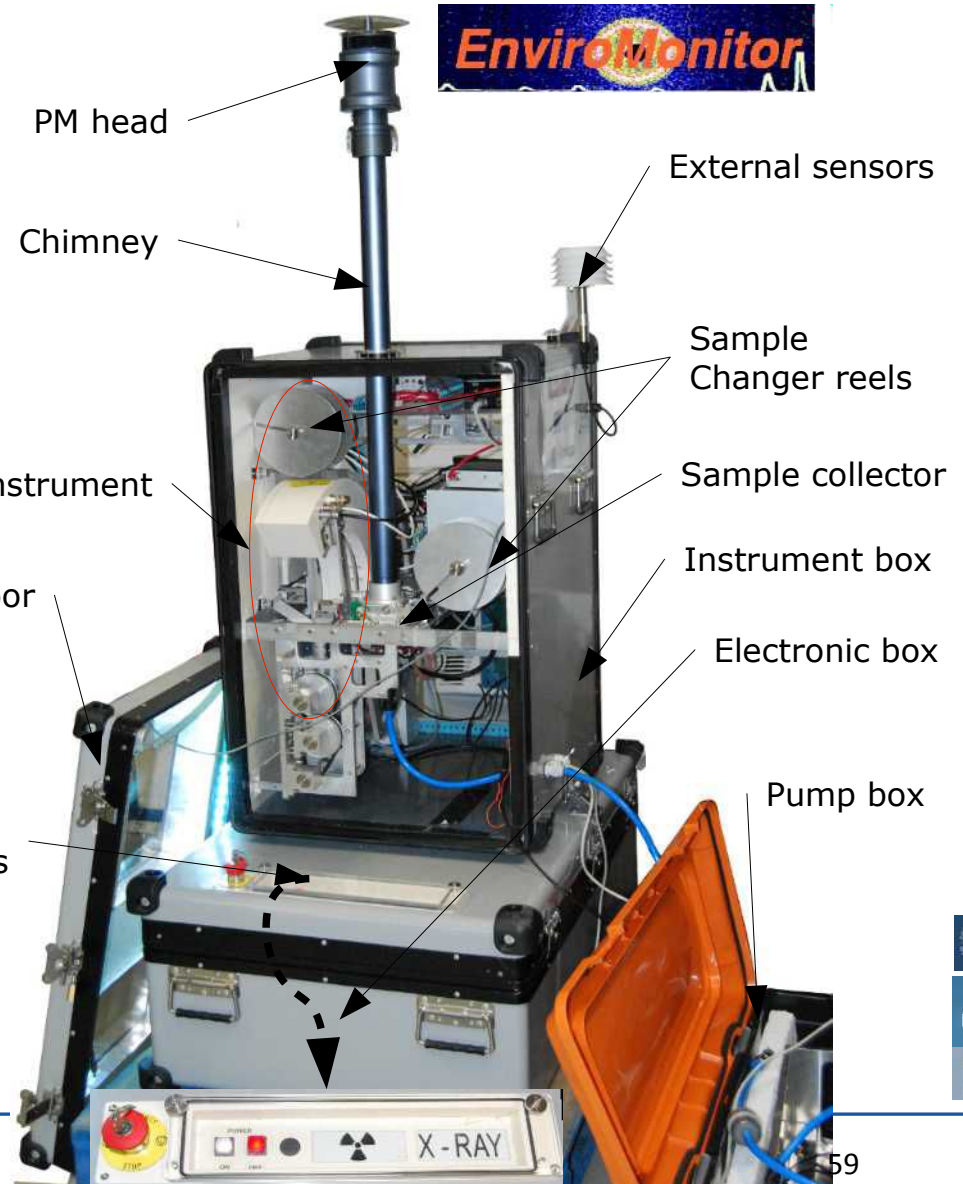


Results after data treatment

σ_{11} (MPa)	σ_{22} (MPa)	σ_{12} (MPa)	Austenite (%vol.)
15+/-2	15+/-2	0	0

"Modeling methodology for stress determination by XRD in polycrystalline materials", S. Dufrenoy, T. Chauveau, R. Brenner, C. Fontugne, B. Bacroix (ECRS 9)

Enviromonitor



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 functionning
- Network servicing

Applications

Dust collection and phase analysis

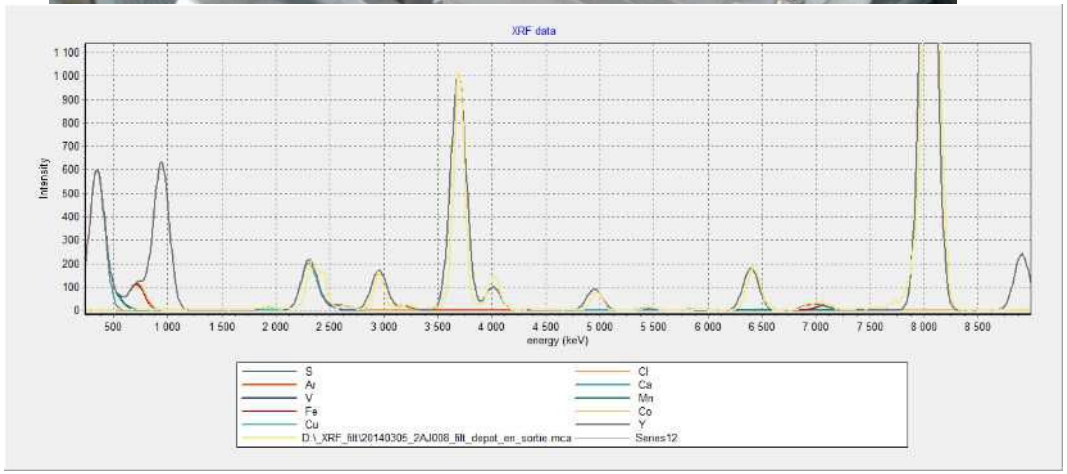
XRD/XRF combination

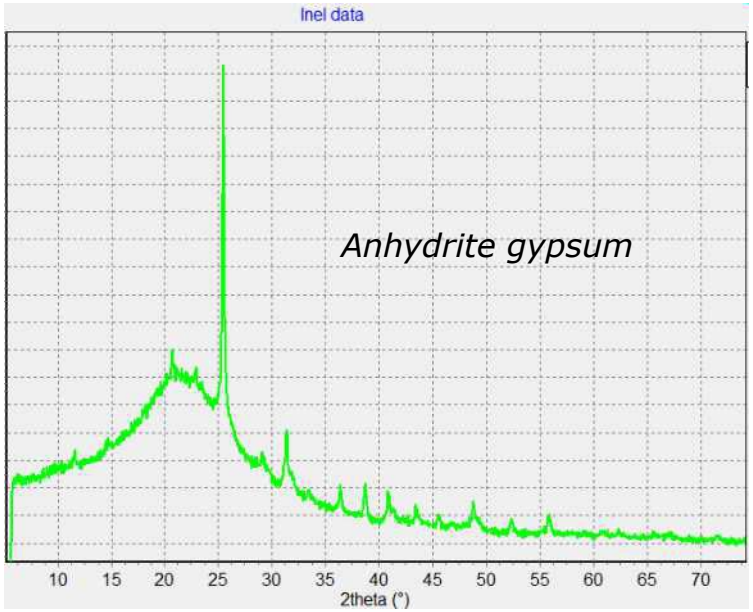
In situ aerosol analysis

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

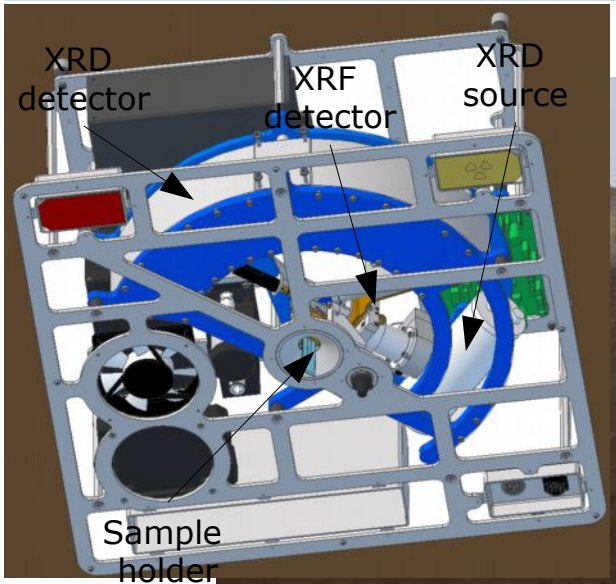
Applications

Dust collection and phase analysis.





SolXpert : XRD/XRF combination in field



In situ analysis with XRD/XRF combination

SolXpert is a system combinaing XRD and XRF. In the field of earth science, it is often difficult to characterize a sample: unknown phases, phase mixture, poor cristallinity. 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...

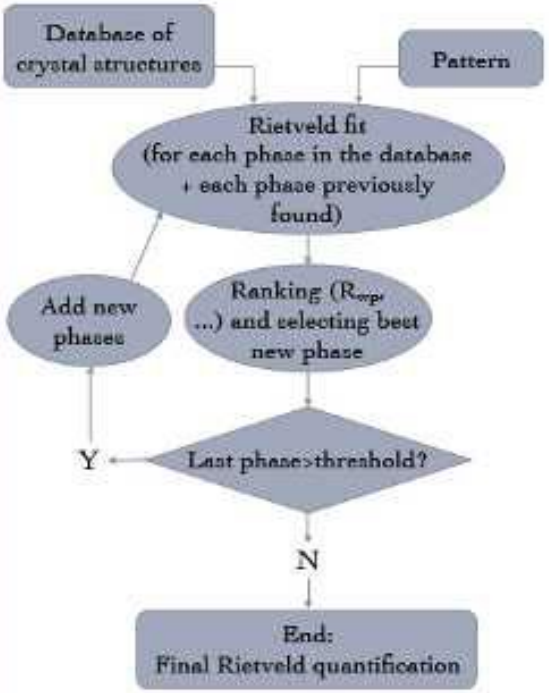
...The size of a computer !

Data treaments

FPSM, a unique software

The FPSM (Full Pattern Search-Match method)

Developed inside the Nanoair project for the portable instrument



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 know 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

A demo version has been setup online at:
http://nanoair.ing.unitn.it:8080/pfpm
search and quantification is limited by the time required (or better server response time) so it should be used restricting the composition as much as possible to speed up computation. A limited number of concurrent connections are supported also INEL SAS can be inquired for the full version.

See also the demo at the Software Payre on tuesday afternoon

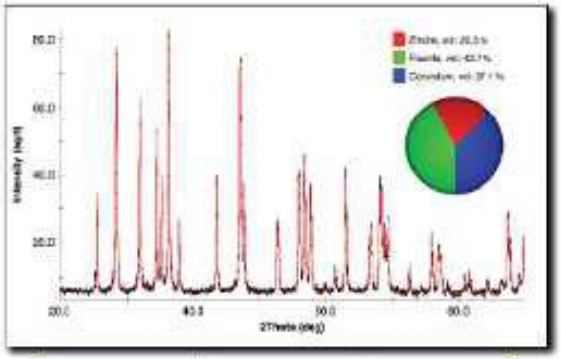
For phases identification and quantification by using Rietveld method
http://cod.iutcaen.unicaen.fr/

Data treaments

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/qaar



Phase (wt%)	FPSM	Round-Robin
ZnO	29.0	29.6
CaF	33.9	34.3
Al_2O_3	37.1	36.1

Total computation time (12 cores, 2.93GHz, COD, inorganic):

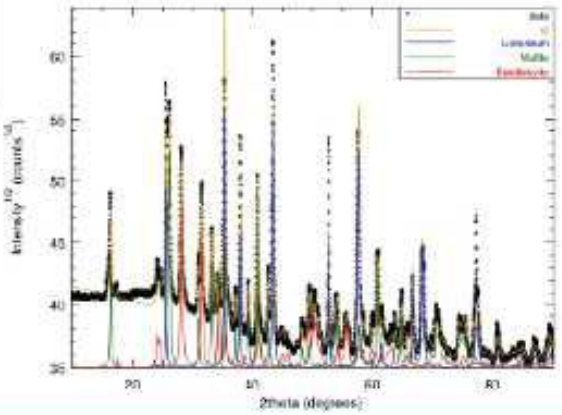
- No composition restriction: 565 secs
- Only Al, Ca, F, Zn, O, Mg, Na, Si, Cl: 19 secs

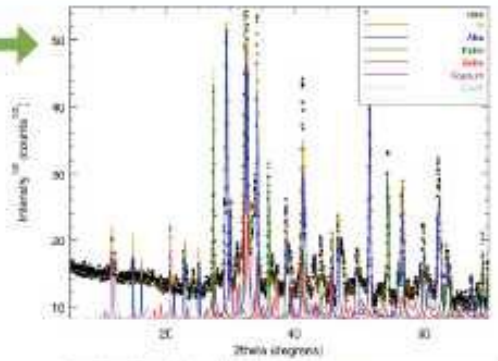
name	vol. (%)	wt. (%)	crystallites (Å)	microstrain
Alite	32.3539	30.1148	2722.86	0.000913623
Rutile	8.3909	11.2199	1382.41	4.9784e-05
Belite	14.6584	14.8578	1014.1	0.00145265
Gypsum	7.28130	5.01503	881.992	0.00179863
C4AF	14.4548	16.3939	485.795	0.00705494
C3A	2.47814	2.27862	1426.2	0.00135916

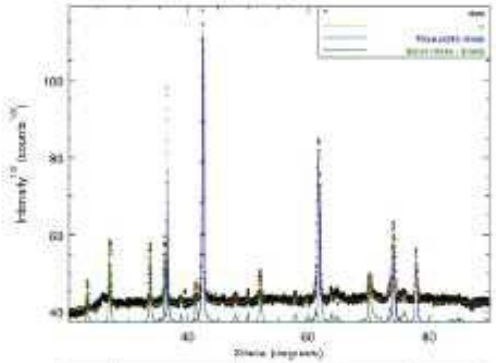
Some additional phases (cement phases) has been added to the database as not present in the COD

Phase ID	name	vol. (%)	wt. (%)	crystallites (Å)	microstrain
9009672	Corundum	41.0726	45.1467	730.664	0.00117145
9005502	Mullite	53.5784	46.23 8	515.483	0.000527227
9005833	Baddeleyite	5.34905	8.62131	371.805	0.00283445

Final Rietveld analysis, Rw: 0.8455532, GoF: 1.79502







Phase ID	name	vol. (%)	wt. (%)	crystallites (Å)	microstrain
1101045	Titanium(II) nitride	50.0729	62.8891	1482.09	0.00122093
1001269	Silicon nitride - β-beta	49.9271	37.1109	860.99	0.00124495

Final Rietveld analysis, Rw: 0.6901521, GoF: 2.9852

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

Région Centre

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

inel

Service, adaptability and innovation always gathered to design today the tools of tomorrow.

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
Cuka1with Ge(111) monochromator
35kV-35mA