



ICP-MS facility for radio-pure material screening

S. Nisi

LNGS Chemistry Department

stefano.nisi@lngs.infn.it

IBS – INFN One-day workshop on underground physics

LNGS-May 30, 2019

Outline

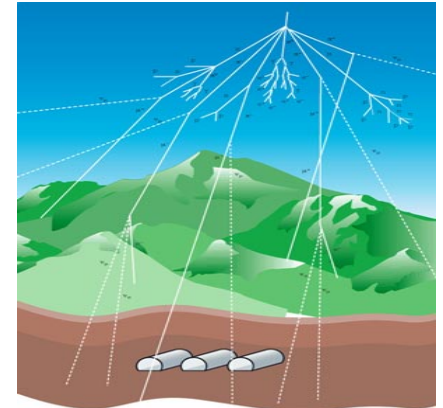
- ✓ Gran Sasso National Laboratory
- ✓ Low Radioactive Techniques (LRTs) at LNGS
- ✓ ICP-MS: instrumentation and performances
- ✓ Interdisciplinary application examples
- ✓ Conclusion

Gran Sasso National Laboratory

Detection of extremely weak events



1400 m of rock (3600 mwe)
Cosmic ray flux reduction: 10^6
Neutron flux reduction: 10^4



- The underground facility provides the necessary **low radioactive background**
- Selection of **highly radio-pure materials**

K Pb Th U

Neutron Attivation Analysis, γ -Ray Spectroscopy, ICP-Mass Spectrometry

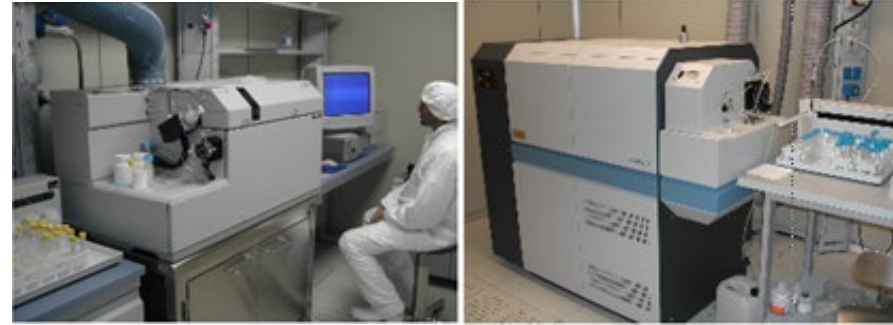
Ultra-low level radioactivity counting facilities

STELLA SubTERRanean Low Level Assay



- **Y spectrometry High-Purity Ge Detectors (HPGE)**
- α spectrometry Silicon PIPS detectors
- Liquid scintillation counters
- + Sample treatment free (GRS)
- Sensitivity depend on the sample mass (Kg)
- Long measurement time is requested to achieve high sensitivity (weeks)

ICP-Mass Spectrometry

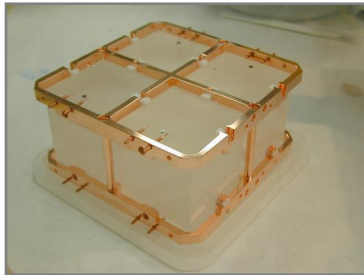


- Quadrupole and double focusing ICPMS
- ISO 6 Clean room
- Reagents purification systems
- Sample treatment device
- + Small sample (g)
- + Relatively quick measurement
- Sample treatment is mandatory and delicate

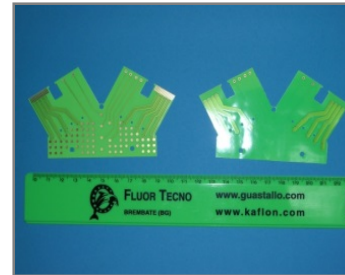
R&MS are often applied both to check for secular equilibrium of decay chain

ICPMS low radioactivity measurements

≈ 200 samples/year (complex matrices)
hundreds samples/year (reagents and water)



Cu, TeO₂ and reagents
-CUORE-



Printed Circuit Board (PCB)
-GERDA-



Metals and alloys
-GERDA, XENON, DARK SIDE



Al-Mylar insulating foils
-XENON, DARK SIDE-

Our 'routine' ICP-MS detection limits:

Sample	Liquid	Metals (solid)		Plastic (solid)
		Dissolution and dilution	Analyte separation	
Element	[pg g ⁻¹]	[pg g ⁻¹]	[pg g ⁻¹]	[pg g ⁻¹]
K	50	5×10 ⁴	-	5×10 ⁴
Pb	0.5	500	-	100
Th	0.05	50	1.0	10
U	0.05	50	0.5	10

U in Cu AVG 3Meas SD
ppt 2.36 0.05



30 ± 0,6 μBq/Kg

Detection limit = 0.2×10⁻¹² g/g



2,4 μBq/Kg

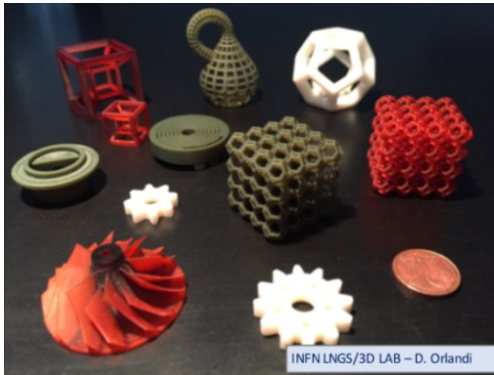
LRTs performances comparison

		ICPMS LNGS	ULL GRS LNGS	ULLGS+NAA Milano-Pavia
		Primordial parents	Y emitters	Primordial parents
		Surface/bulk	Bulk	Surface/bulk
Destructive		Yes	No	Yes
DL	[10^{-12} g/g]	Th=0.5 U=0.5	Th= 10-20 U= 10-20	Th(^{233}Pa)= 0.5 U(^{239}Np)= 3-5
Sample size	[g]	0.1-10	1-10000	200
Sample treatment		Contamination risk not negligible	Almost free	Hot sample handling Low cont risk
Analysis Time		Days	Weeks	Days-week

Additive Manufacturing at LNGS

For several years now the Mechanical Workshop is operating 3D printing devices to realize pieces with **photo-polymeric** and MultiJet Hi-performance **thermoplastic resins**

Carbon PEEK 3D printing is coming soon

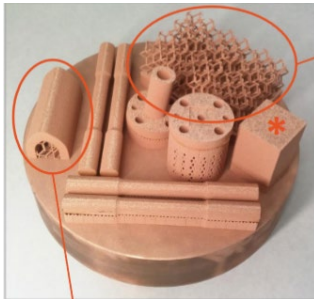


The facility is equipped with a stereoscopic Hi-Res 3D scanning station for quality analysis and reverse engineering

Additive Manufacturing: Future Outlook in Designing Pure Copper Components for LB detectors

AM allows to produce parts:

- complex geometries
- high Resolution
- hollow components
- w/o final traditional machining
- w/o surface cleaning
- mass savings with a factor $\approx 2-3$
- reduction of number of components



LRTs play a fundamental role for production process monitoring

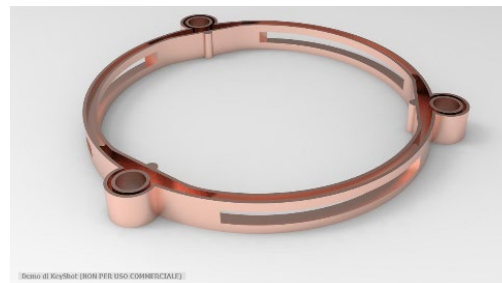
Crystal Holder



Traditional CNC
mass=27g



AM same support
mass=11g



AM new design
mass=9g

M/3 !

Center for Excellence on
Aging (CeSi)



Fondazione
Università
Gabriele d'Annunzio

Validation of a method for the quantitative multi-element profiling of brain tissues

Neurodegenerative
diseases



Changes in brain levels of some elements, among them:

Li, Al, Cr, Co

Ciavardelli et al, “**Characterization of element profile changes induced by long-term dietary supplementation of zinc in the brain and cerebellum of 3xTg-AD mice by alternated cool and normal plasma ICP MS**”, Metallomics, 2012



University of
L'Aquila

Investigation of the geographical origin of saffron through elemental finger-print approach

Three set of samples from different production areas :



Crocus sativus

Medio
Campidano



**Dried stigmas of
Crocus sativus**



Perugia

L'aquila

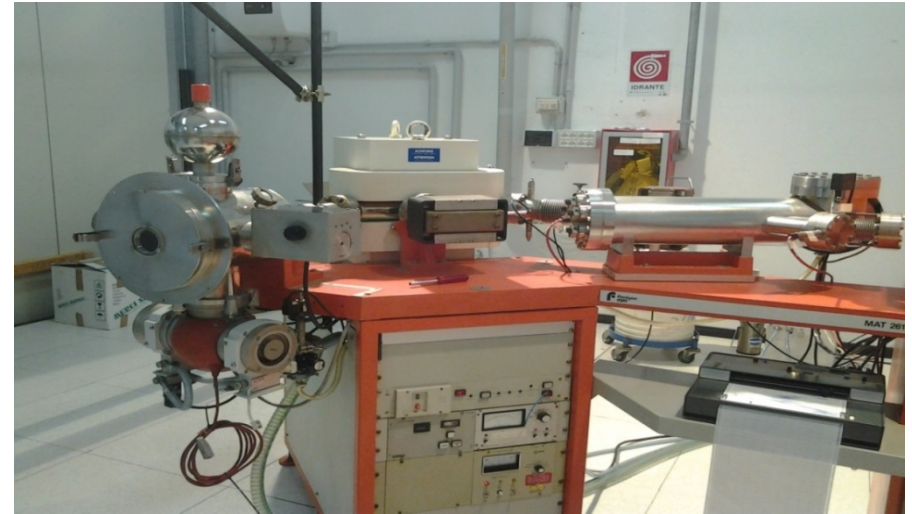
Analysis of the mineral composition of Italian saffron by ICP-MS and classification of geographical origin, [Food Chemistry 157 \(2014\) 485–489](#)

Angelo Antonio D'Archivio , Andrea Giannitto , Angela Incani , Stefano Nisi

Laboratory of Isotopic Mass Spectrometry (LIMS)

MC Thermal Ionization Mass Spectrometry

- Discrimination between isotopic ratio values: $<0.01\%$
- Internal precision of the measurement $>0.005\%$



Applications (Cultural Heritage Network-INFN)

- Archaeometry (Pb, Sr)
- Human science: mobility ancient population study
- Food traceability (Sr)



Conclusion and perspective

- ICPMS is a fundamental technique for the experiments searching for rare and low energy events
- By the application of both ICPMS and RMs it is possible to have more complete and complementary information
- The R&D about new sample preparation procedures allow the achievement of better and better detection limits and reliable analysis
- The experience and the knowhow developed in ultra-trace measurement often make a significant contribute in interdisciplinary activities
- The purchasing of last generation quadrupole based ICPMS equipped with Laser Ablation System is under evaluating (direct measurement of solid sample)

