Università di Sassari

SCUOLA DI DOTTORATO IN SCIENZE DELLA NATURA E DELLE SUE RISORSE
CICLO XXVI

NEW GAMMA-RAY SPECTROMETRY METHODS FOR ESTIMATING K, U, TH CONCENTRATIONS IN ROCKS OF THE SARDINIA BATHOLITH

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Scientific motivations of this PhD thesis

- Study of the correlation between the spatial distribution of natural radionuclides (^{40}K, ^{238}U and ^{232}Th) and principal geological formations in order to reveal new features in the geological structure of N-E Sardinia

- Realize the first radiometric map of the natural radionuclides concentration in the rocks of N-E Sardinia using advanced geostatistical methods

- Estimate the radiogenic contribution to the local heat flow in the N-E Sardinia

- Study the correlation between SiO_2 content and K, U and Th abundances in the main lithotypes of N-E Sardinia

- Design and realize a portable collimated lightweight gamma-ray spectrometer for refined in-situ measurements

- Calibrate the spectrometer validating the results with data obtained in laboratory with HPGe detectors

Open questions

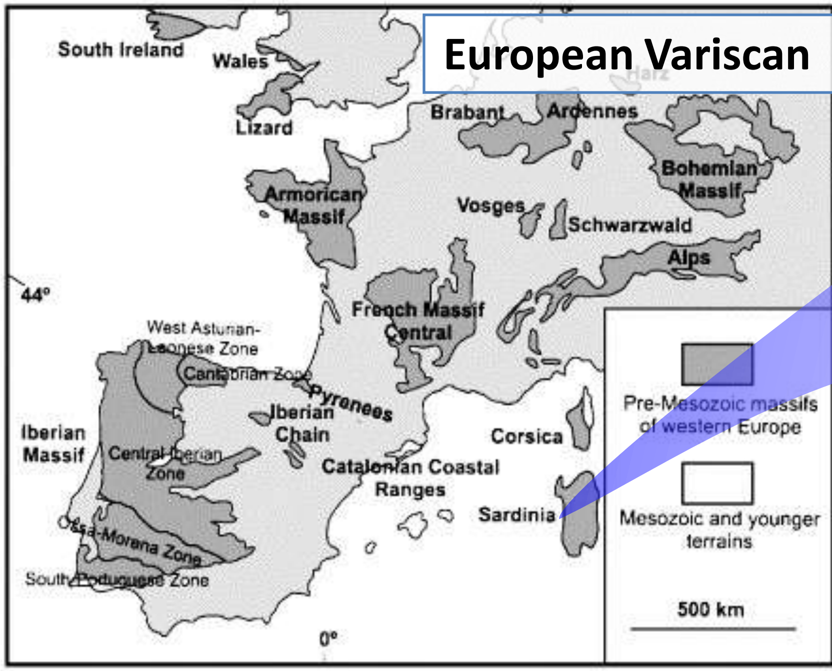
What is the distribution of K, U and Th in European Variscan?

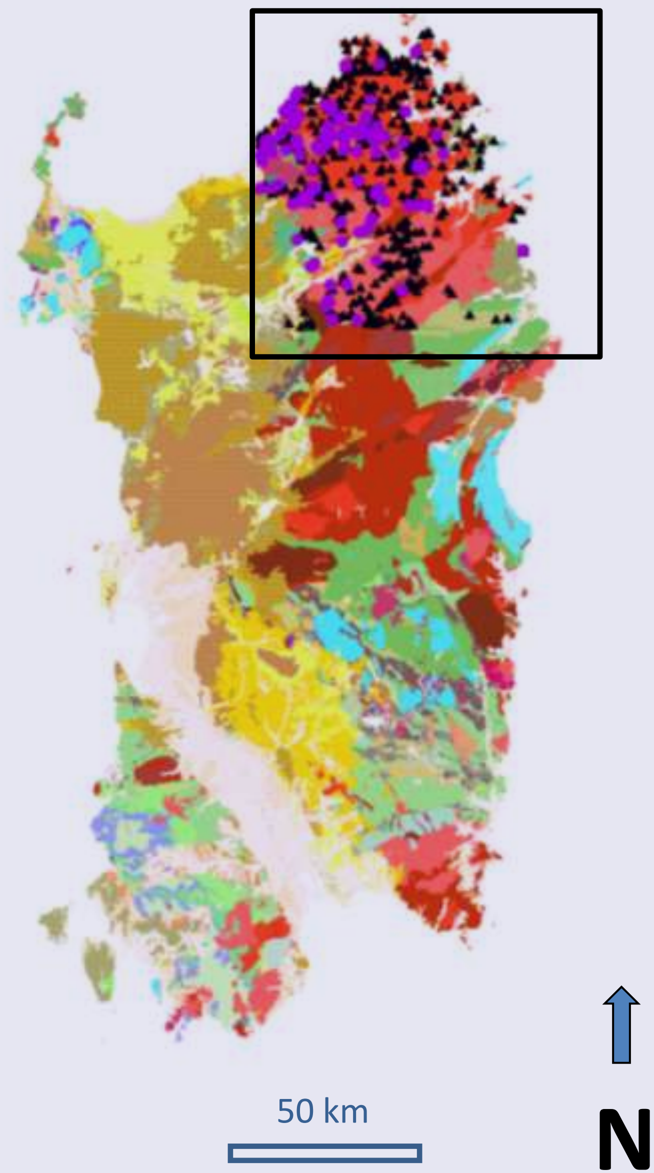
How much U, Th and K is in the deep crustal layers in the N-E Sardinia?

What is the contribution of radiogenic heat to the Sardinia thermal budget?

Can we characterize the radionuclides content in a outcrop with portable detector?

Geological overview and sampling distribution





γ-ray spectrometry survey → ● **rock samples** / ▲ **In-situ measurements**

The Varsican orogeny of the Corsica-Sardinia belt was formed during *Late Carboniferous* and *Permian* age was followed by magmatic activity and sedimentation.

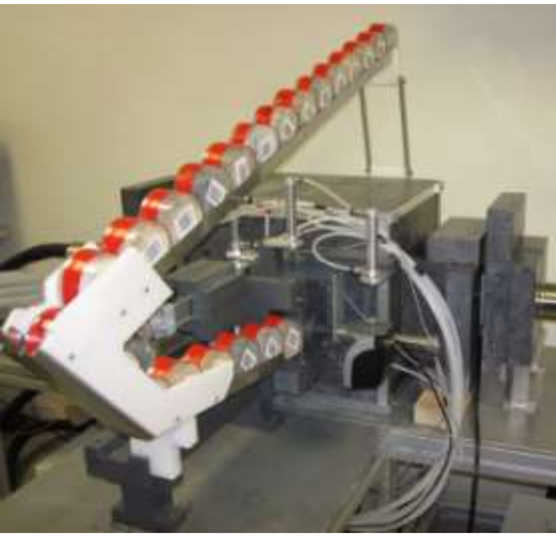
Study area survey – in average 1 sample for 6 km^2:

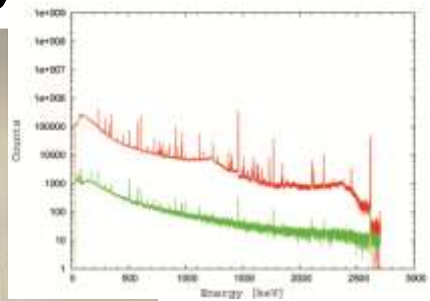
N-E Sardinia	Geological formations	Area (km^2)	In-situ ▲	In lab. ●
Basement	12	4165	545	167

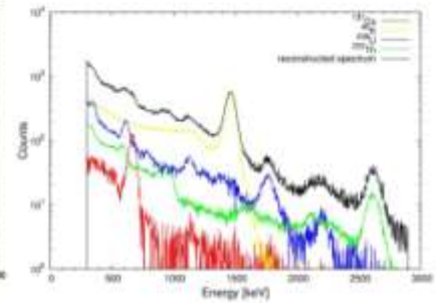
Gamma-ray detectors used

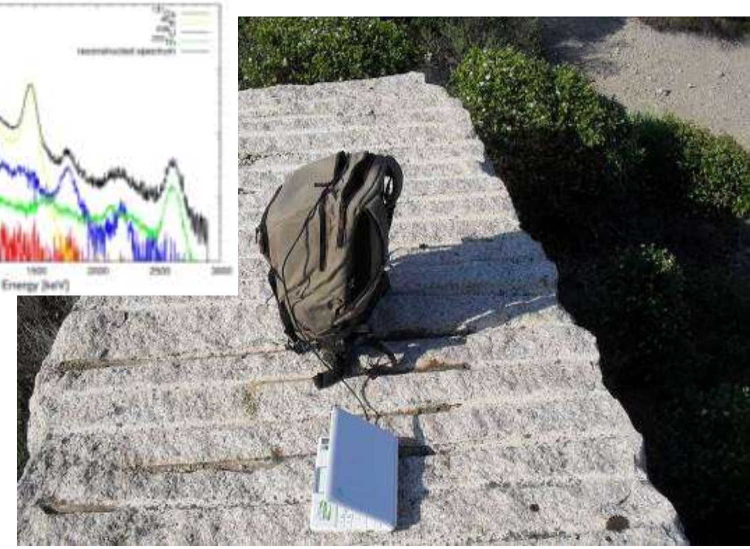
Rock samples are crushed and left undisturbed for 4 weeks in sealed containers prior to be measured by MCA_Rad system.

In-situ measurements are performed for 5 min acquisition time by placing the NaI(Tl) detector on rock outcrops.









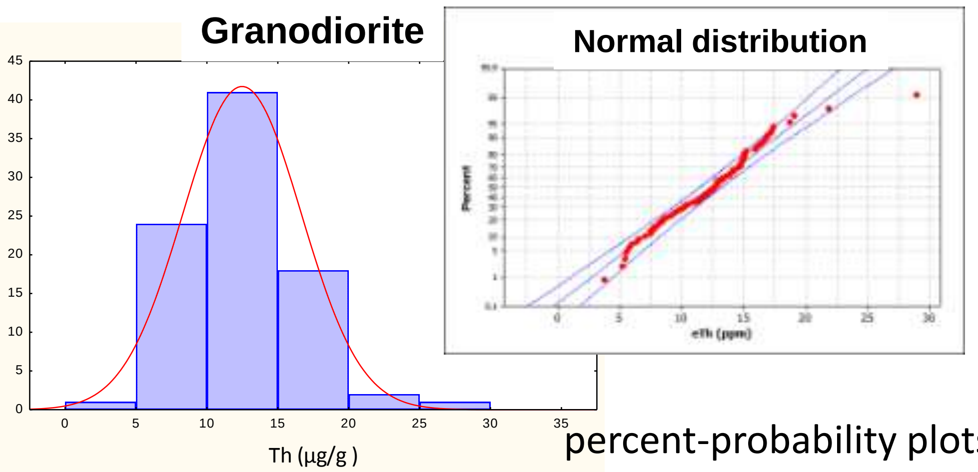
- 2 HPGe detectors with rel. eff. of 60% each
- Energy resolution 0.2% at 1.33 MeV (^{60}Co)
- Electromechanic cooler ~ -190°C
- Shielding configuration: 10 cm Cu-Pb
- Automatic sample changer: 24 samples

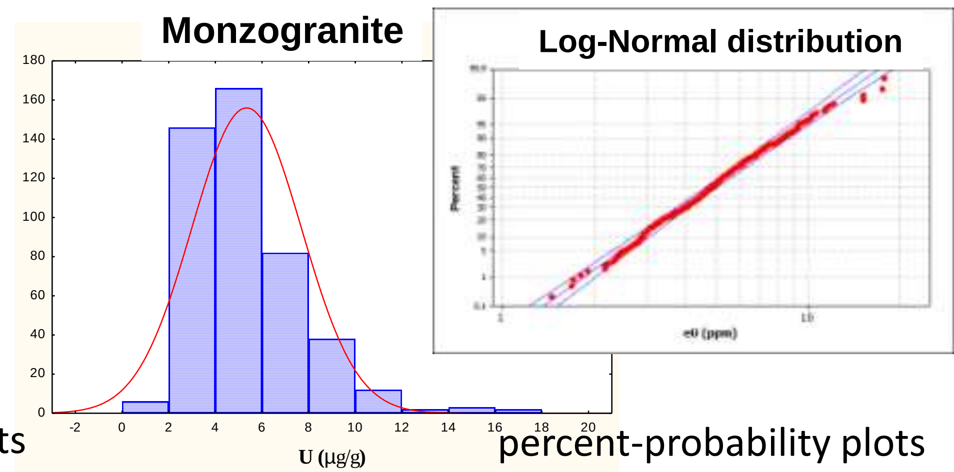
Sample volume: 200 cc
Acquisition live time: 1 h

- 1L NaI(Tl) detector with cubic shape crystal
- Energy resolution: 5.3 % at 1.33 MeV (^{60}Co)
- Environment temperature
- No shielding
- Quick measurement (5 min)

- USB GPS antenna
- USB Temp & Humidity sensor

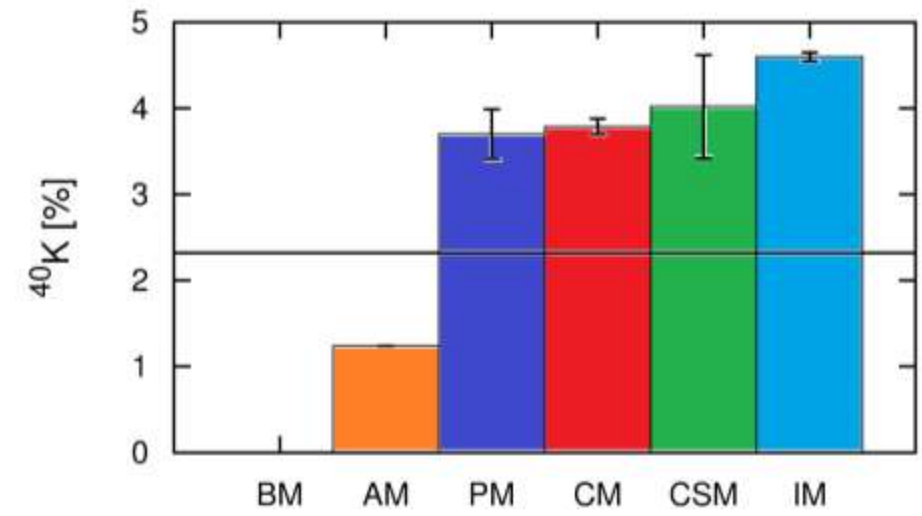
Radioelement abundances in N-E Sardinia rock lithotypes

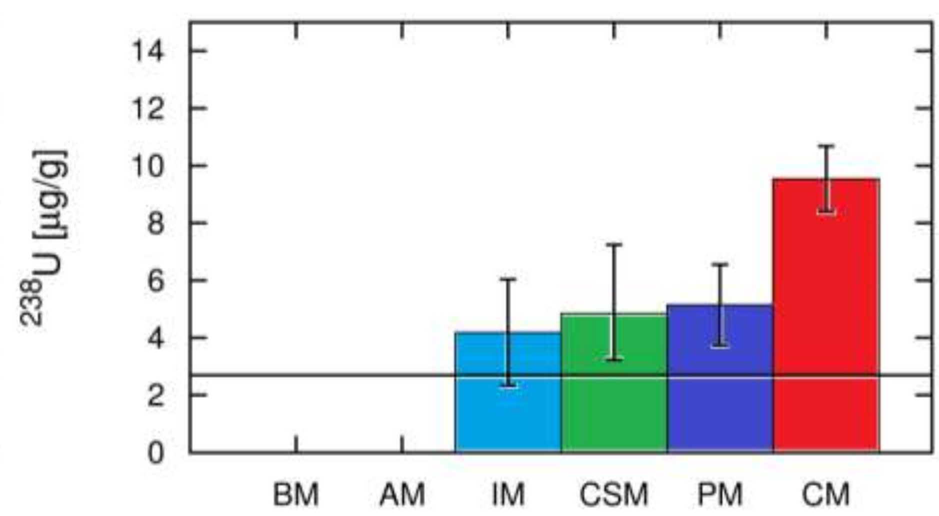


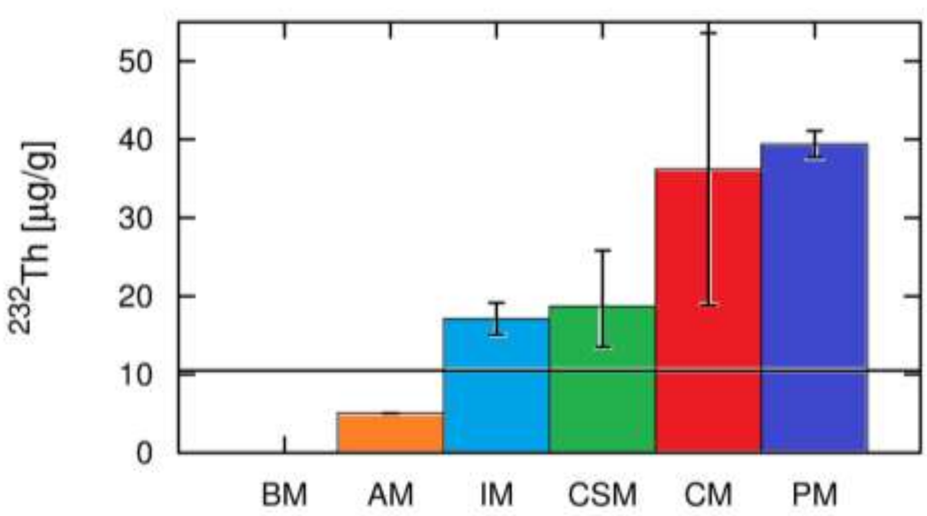


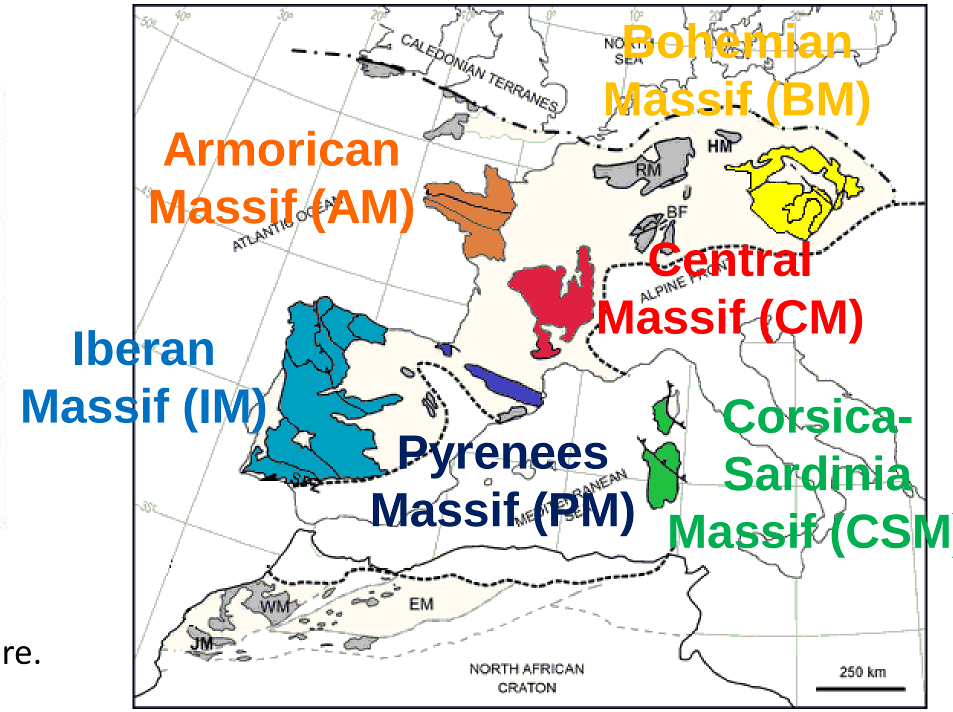
rock-type	No.	K (%)	U (µg/g)	Th (µg/g)	Th/U
monzogranite	446	**4.02 ± 0.60**	**$4.85^{+2.40}_{-1.62}$**	**$18.70^{+7.11}_{-5.17}$**	$3.85^{+2.63}_{-1.56}$
granodiorite	87	2.92 ± 0.87	$2.97^{+1.10}_{-0.81}$	12.41 ± 4.16	**$4.07^{+2.29}_{-1.63}$**
migmatite	25	3.42 ± 0.60	$4.55^{+1.40}_{-1.07}$	13.10 ± 4.76	**$2.81^{+1.48}_{-1.14}$**
granite	21	3.77 ± 0.94	$4.18^{+4.08}_{-2.07}$	16.13 ± 7.07	$3.62^{+4.51}_{-2.16}$
leucogranite	16	**4.09 ± 0.74**	$4.24^{+4.17}_{-2.11}$	**19.99 ± 8.67**	**$4.43^{+5.45}_{-2.63}$**
leuco-monzogranite	16	**4.43 ± 0.50**	**7.37 ± 2.74**	**23.09 ± 8.31**	$3.12^{+2.28}_{-1.31}$
orthogneiss	13	$3.31^{+0.93}_{-0.73}$	$4.40^{+2.42}_{-1.56}$	$12.78^{+5.96}_{-4.02}$	$2.91^{+2.26}_{-1.28}$
amphibolite	12	**1.56 ± 0.70**	**2.11 ± 0.71**	**$8.01^{+2.90}_{-2.14}$**	$3.89^{+2.57}_{-1.39}$
eclogite	11	**$0.22^{+0.27}_{-0.12}$**	**$0.31^{+0.34}_{-0.16}$**	**$0.37^{+1.78}_{-0.31}$**	**$1.22^{+6.79}_{-1.04}$**
sienogranite	11	**4.52 ± 0.51**	**$5.58^{+4.27}_{-2.40}$**	**26.31 ± 5.46**	**$4.64^{+3.80}_{-2.14}$**
alcaline-granite	10	3.96 ± 0.19	4.16 ± 1.33	$19.85^{+5.19}_{-4.16}$	**$4.86^{+2.71}_{-1.52}$**

Monzogranite in the European Variscan



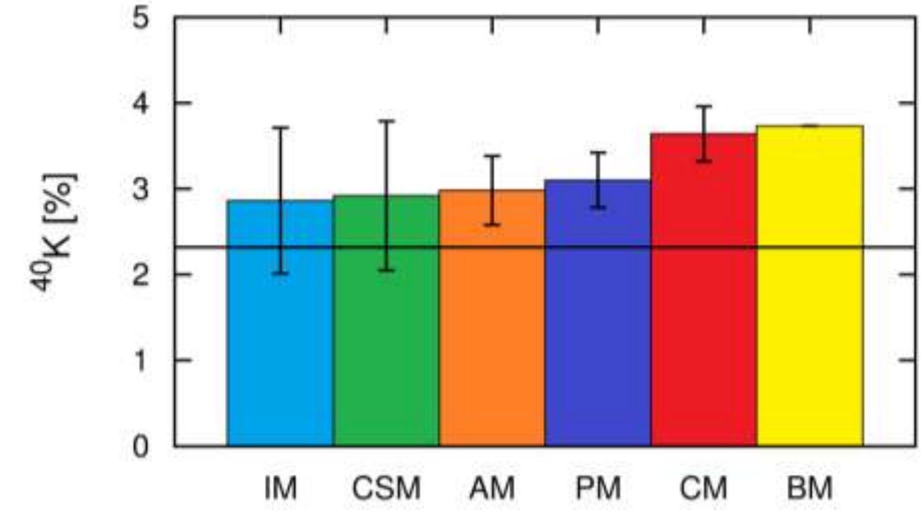


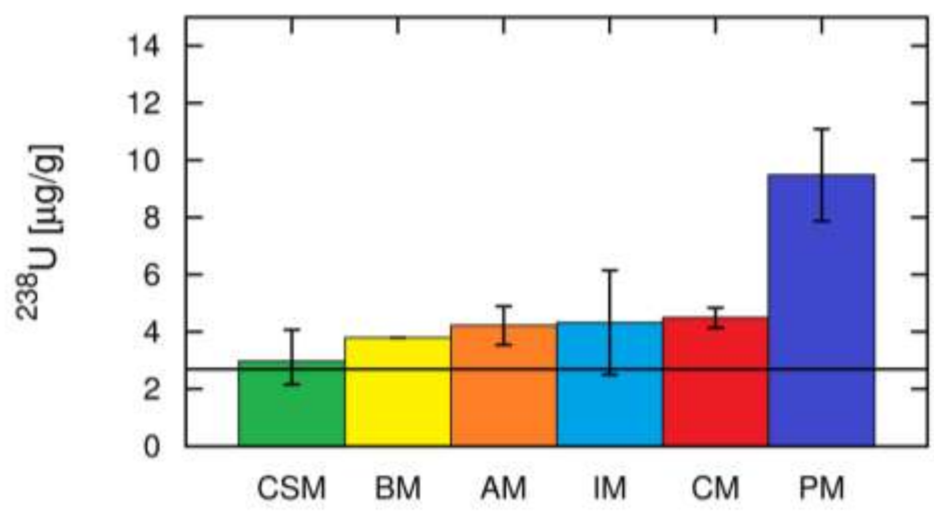


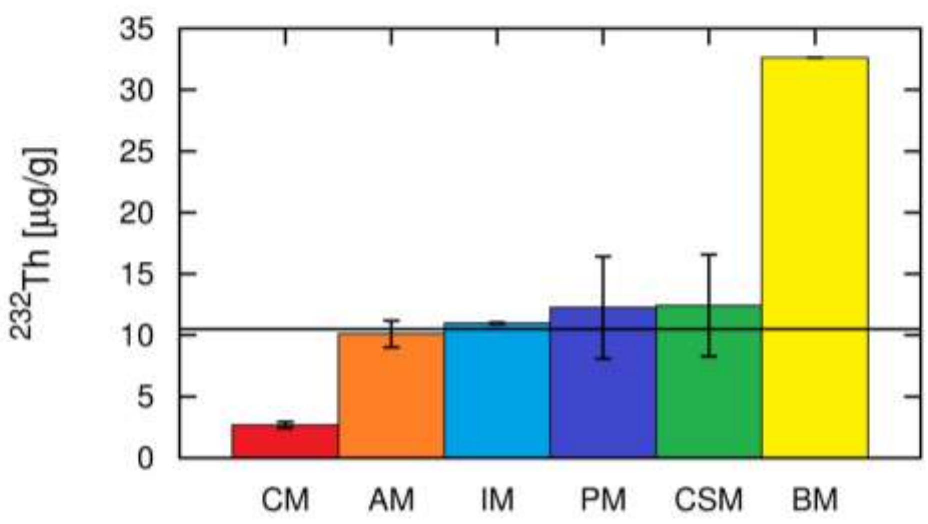


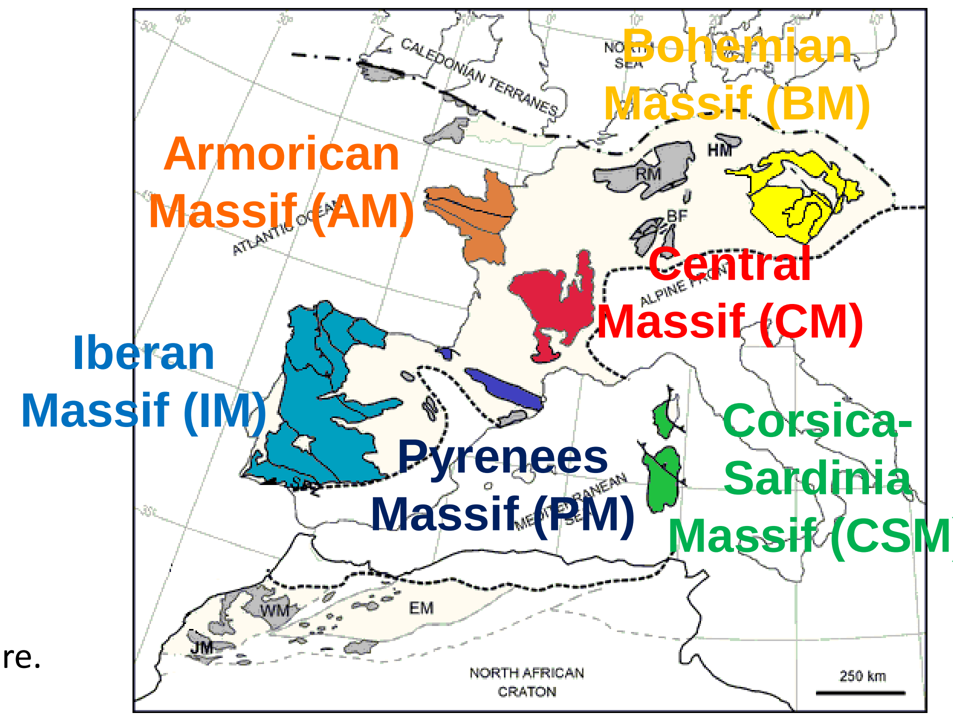
The data for BM, AM, IM, CM and PM are taken from literature.
The data for CSM are from this study.

Granodiorite in the European Variscan









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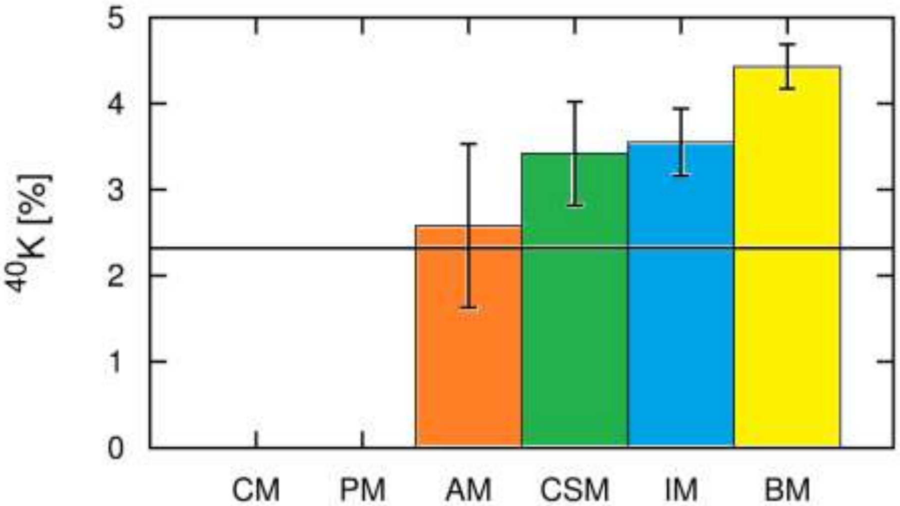
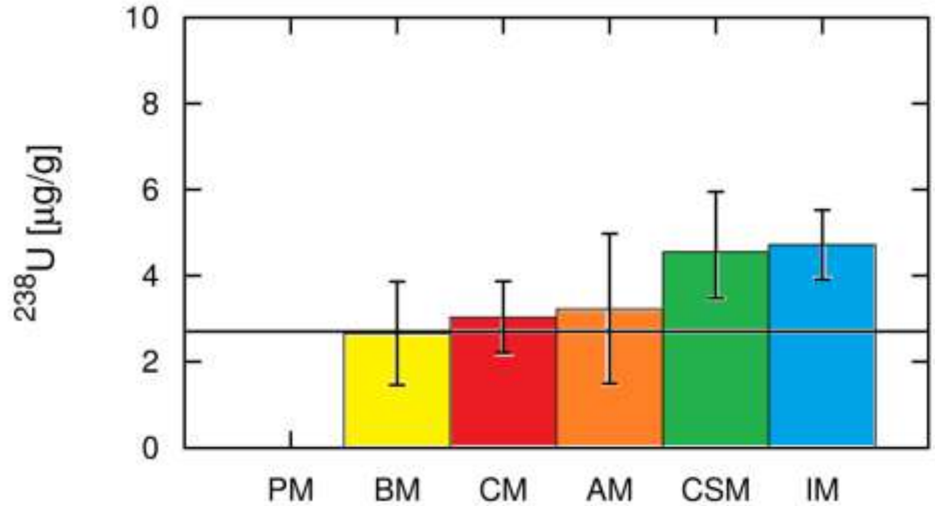
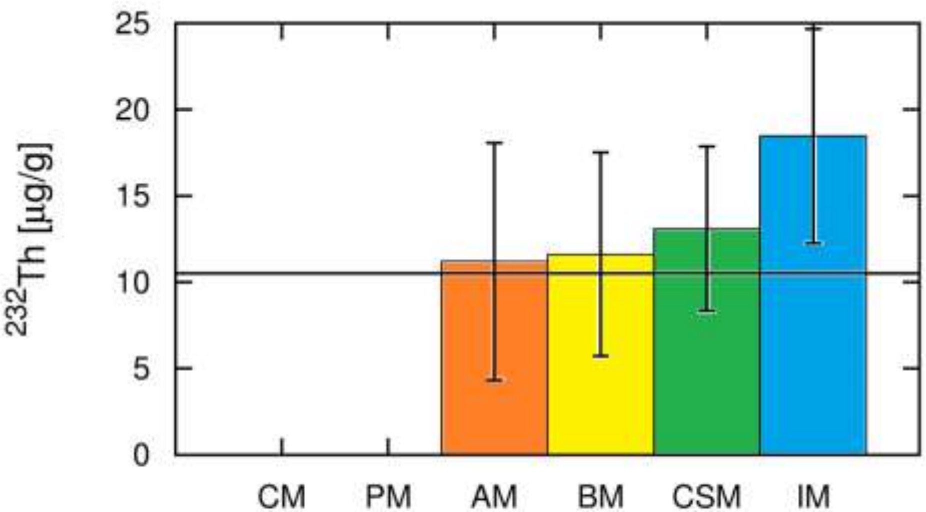
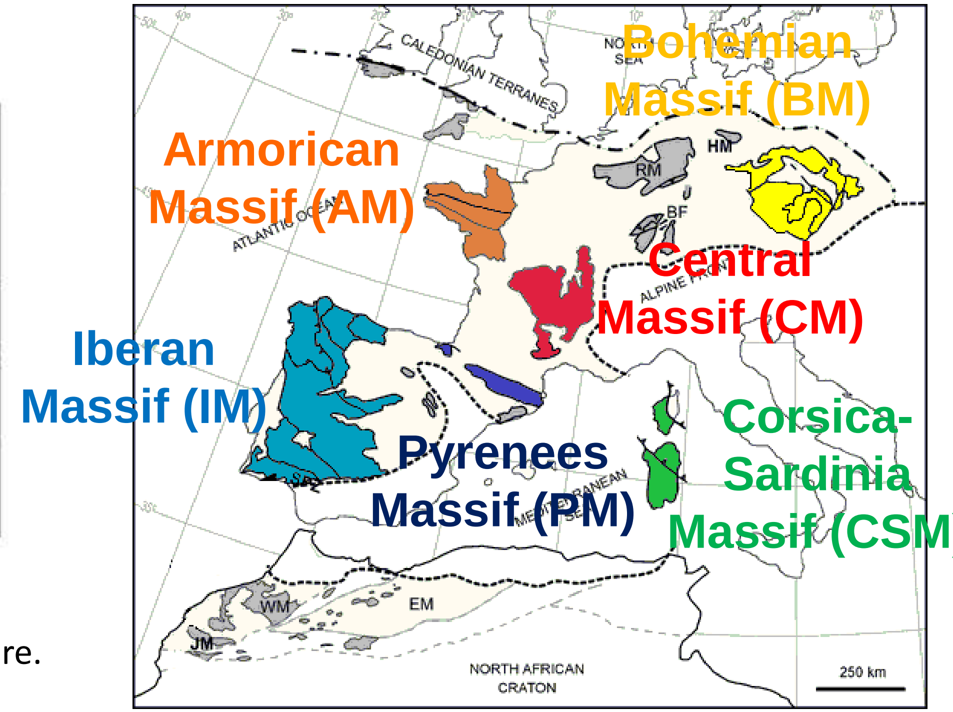
The data for CSM are from this study.

Migmatite in the European Variscan









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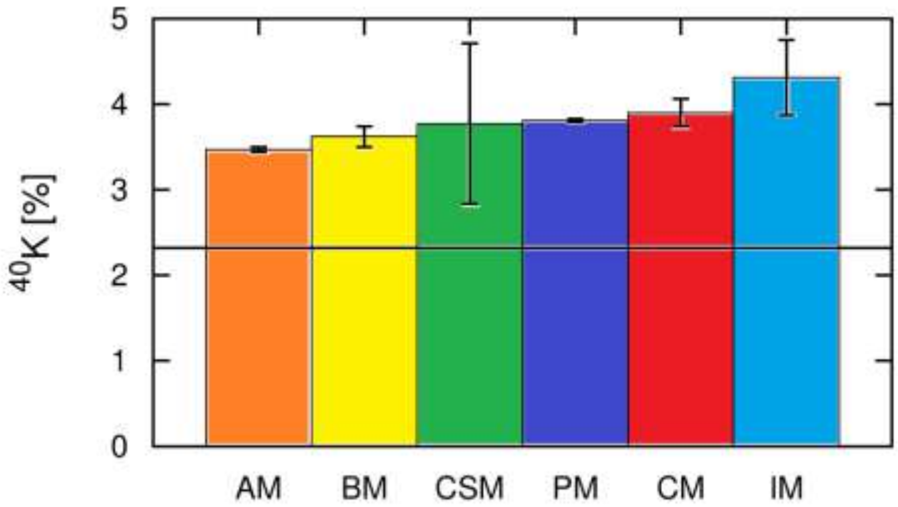
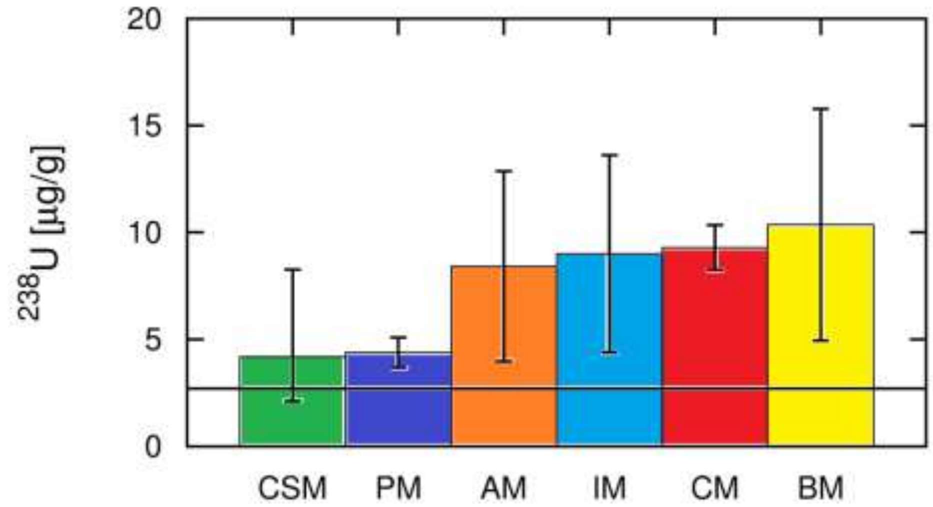
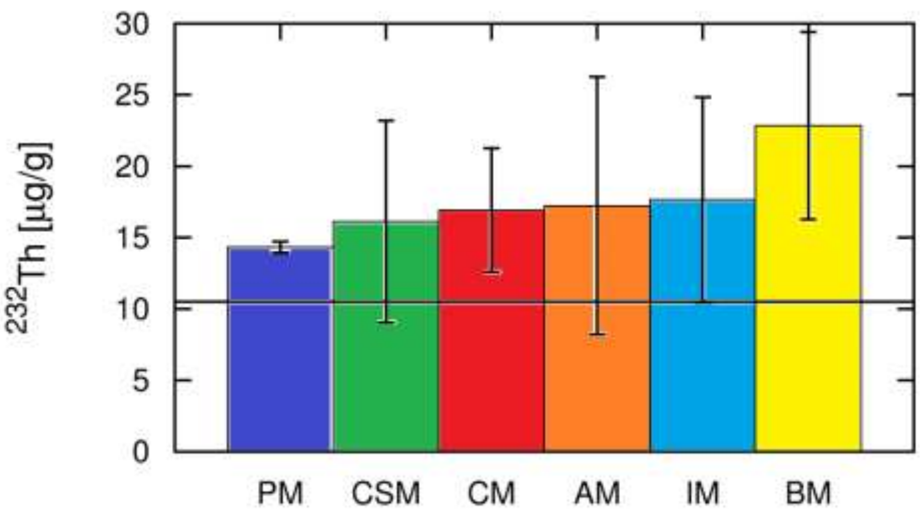
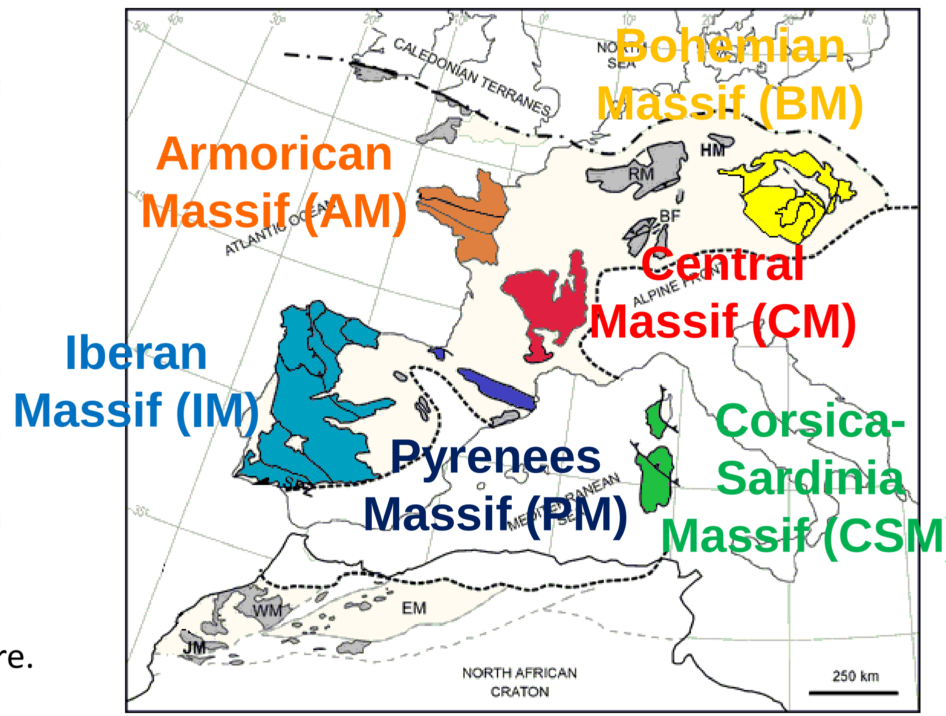
Granite in the European Variscan



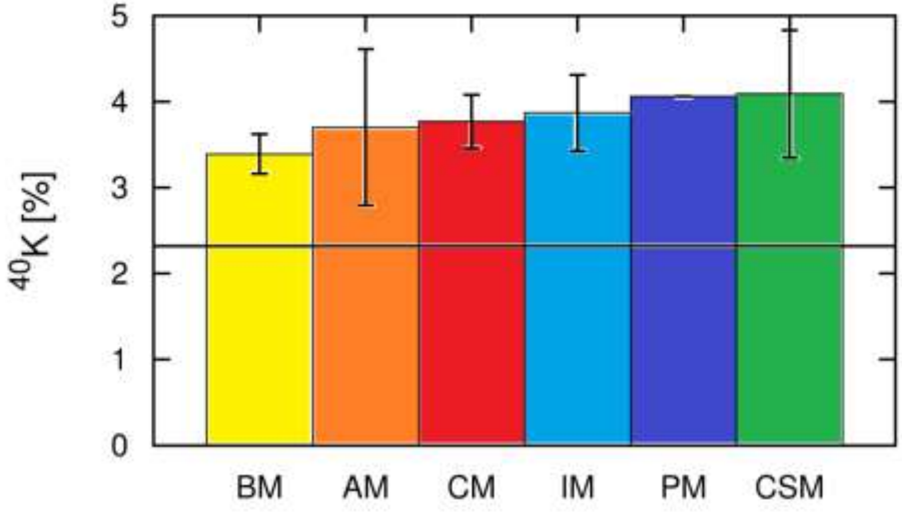


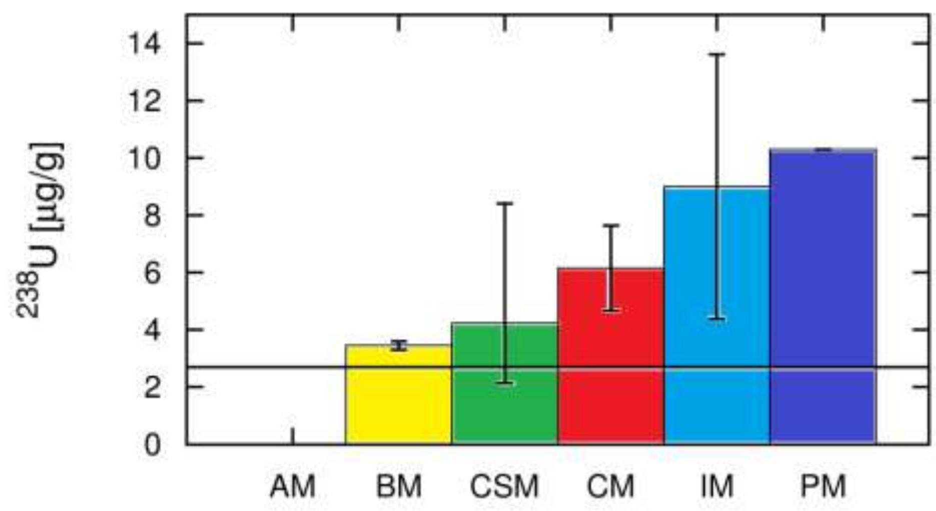


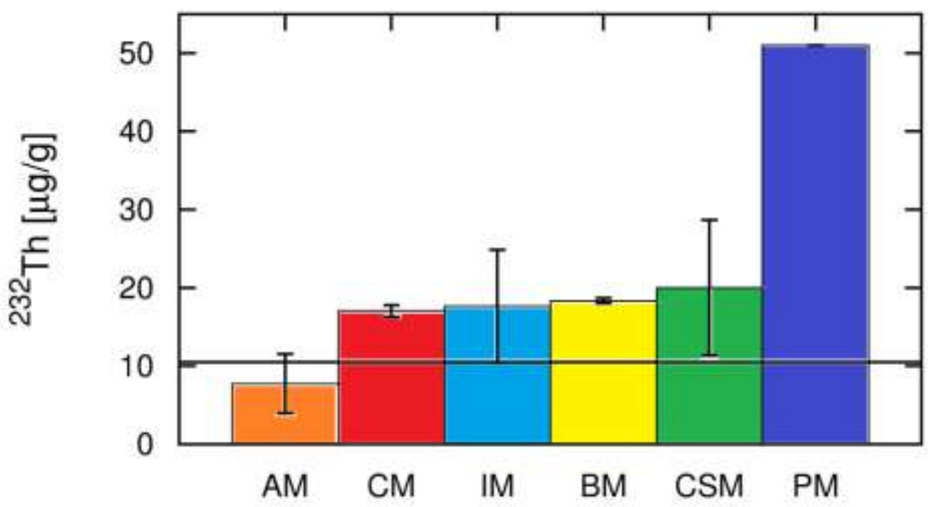


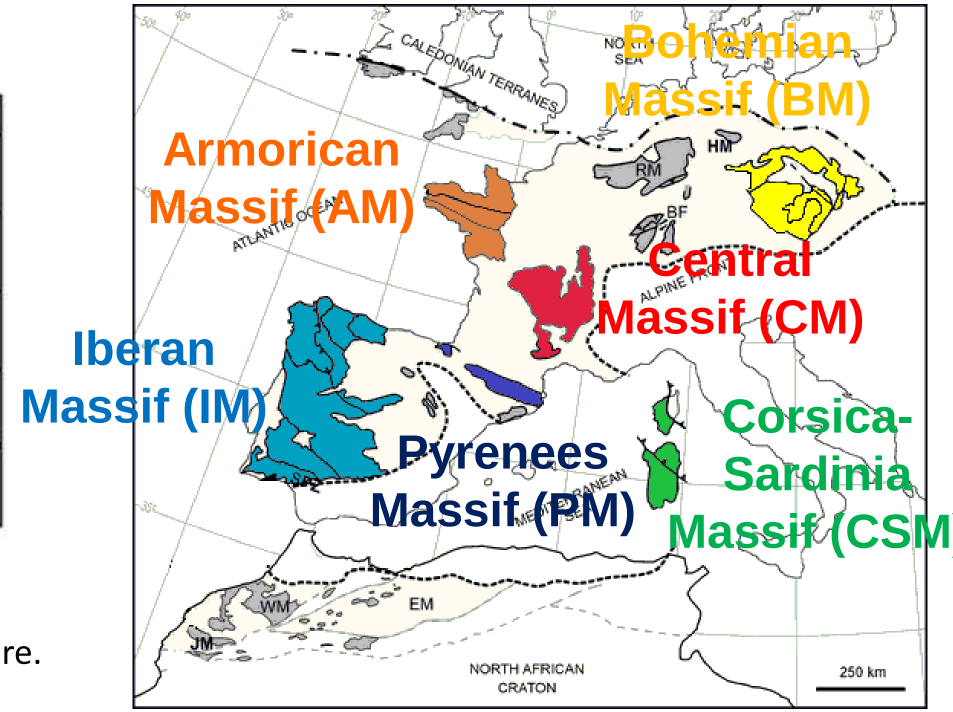
The data for BM, AM, IM, CM and PM are taken from literature.
The data for CSM are from this study.

Leucogranite in the European Variscan









The data for BM, AM, IM, CM and PM are taken from literature.
The data for CSM are from this study.

Geostatistical analysis: new insights on geological knowledge of N-E Sardinia

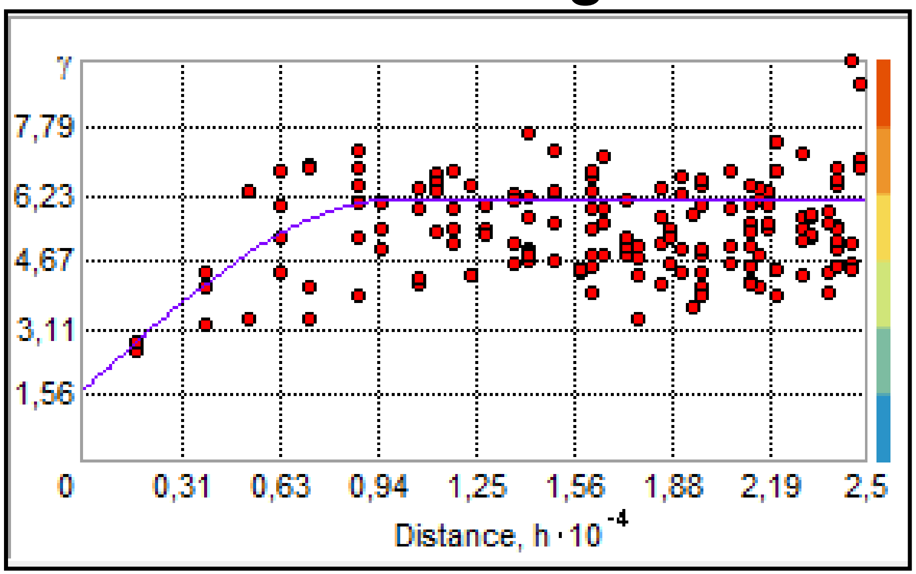
Variable	^{40}K (%)	^{238}U (µg/g)	^{232}Th (µg/g)
Counts[a]	701	701	701
Minimum	0.1	0.5	1.5
Maximum	6.0	17.9	45.3
Mean	3.8	4.9	18.0

Ordinary Kriging is a geostatistical estimator that infers the value of the variable in unobserved locations from **input data points**.

$$\gamma(h) = \frac{1}{2N(h)} \sum_{n=1}^{N(h)} \left[z(x_n) - z(x_n + h) \right]^2$$

Computation and modeling of **Experimental Semi-Variogram** (ESV) with 8 lags (h) of 4000 m for ^{40}K, 10 lags of 2500 m for ^{238}U and 8 lags of 3000 m for ^{232}Th.

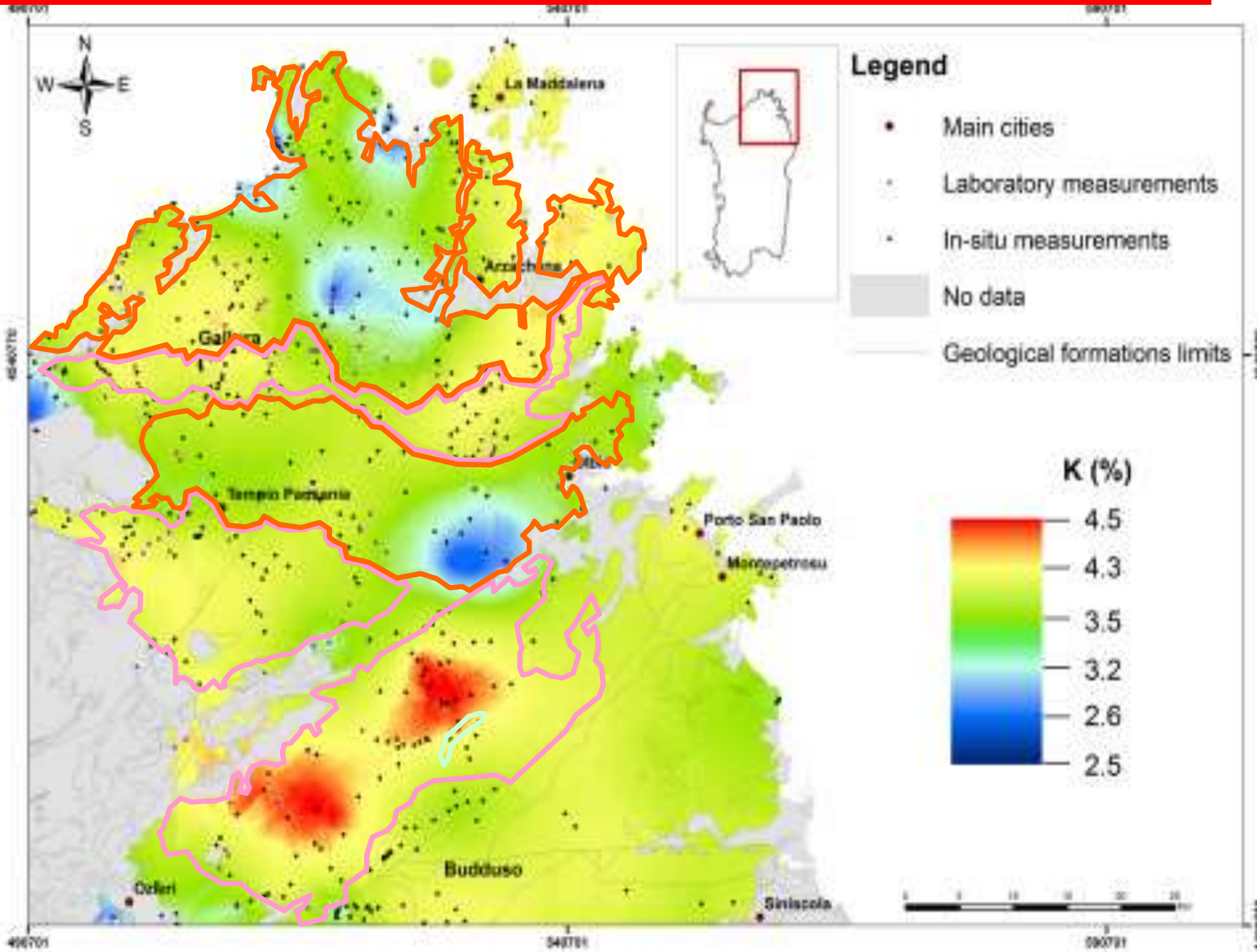
E.g. of ESV model for U (µg/g)

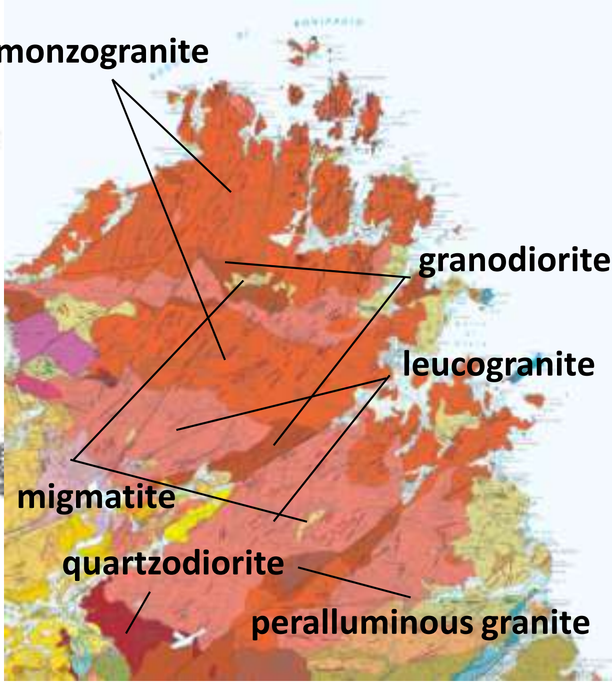


Model description		
Structure	**Range (m)**	**Sill**
Nugget effect	-	1.6 (µg/g) ^{2}
Spherical model	10000	4.5 (µg/g) ^{2}

Cross-validation	
Mean Std. Err.	**Variance Std. Err.**
0.2 10^{-4}	1.17

Map of K(%) abundance



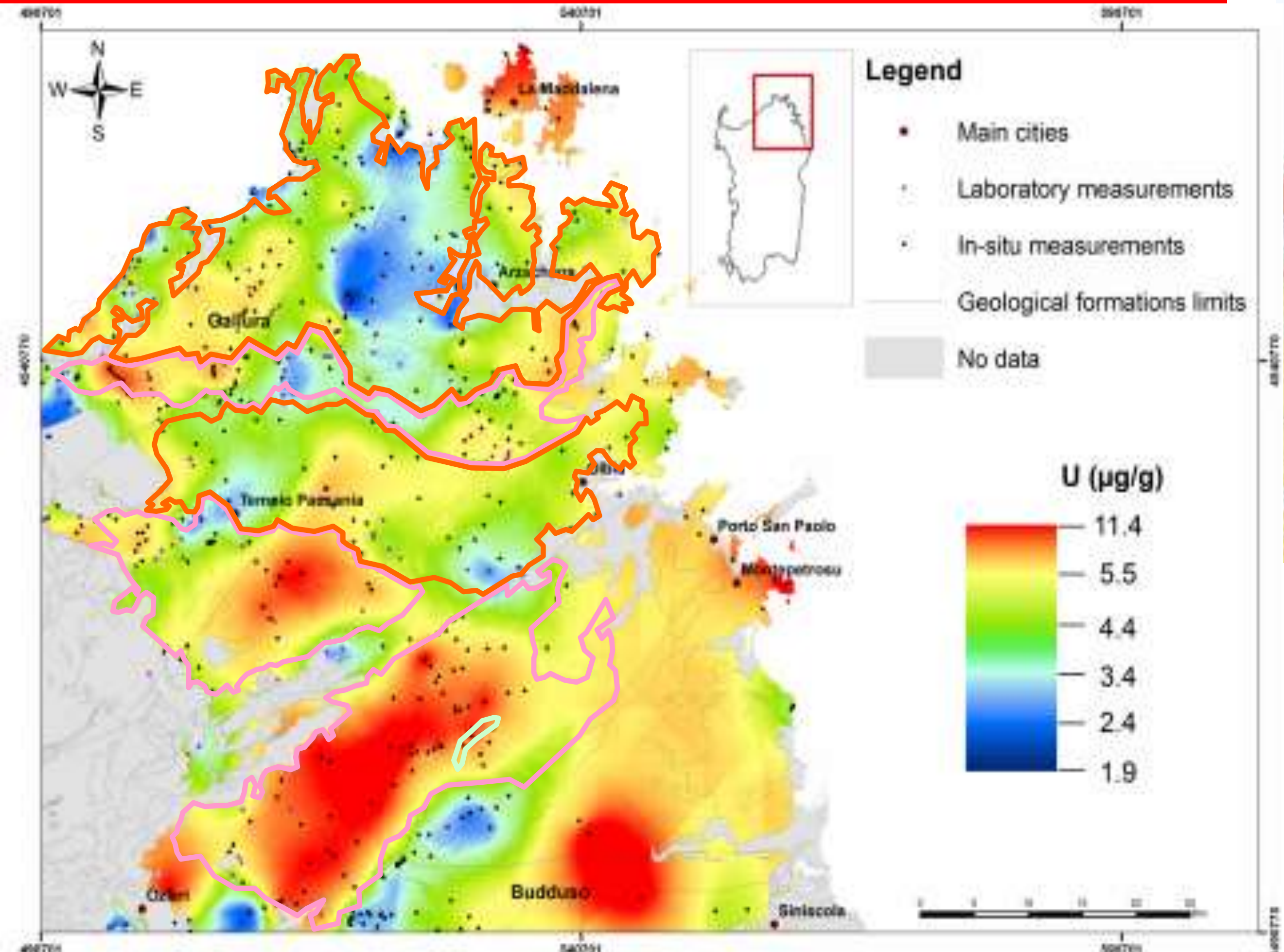


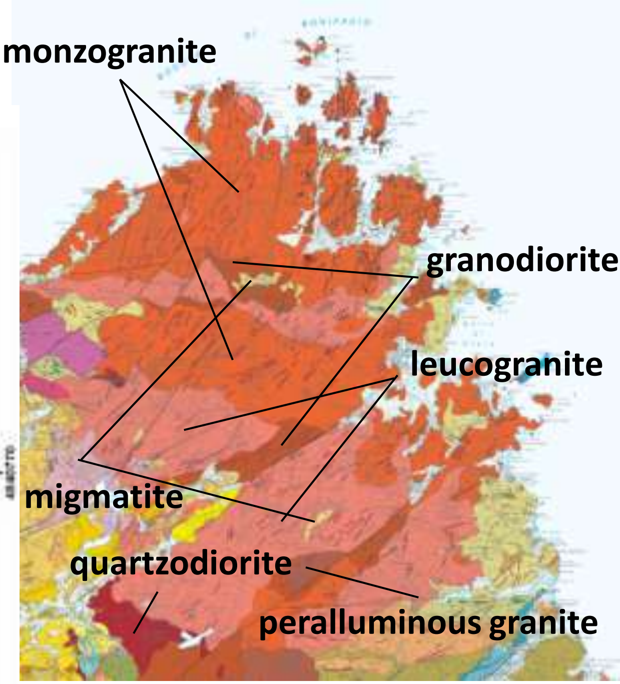
- The main geological structures are confirmed by the map of K (%) abundance.

- Leucogranites are relatively homogeneous and they show the highest K abundances.
- Evidences of two anomalies of high K abundance give new clues for interpreting the geological map, e.g. presence of alkali-rich leucogranites.

- Monzogranites show relatively high dis-homogeneity of K(%) abundance.
- The pattern of the distribution of K abundance in Gallura seems to be affected by the presence of dykes spacing from basic (NE) and acid (NW)

Map of U (µg/g) abundance



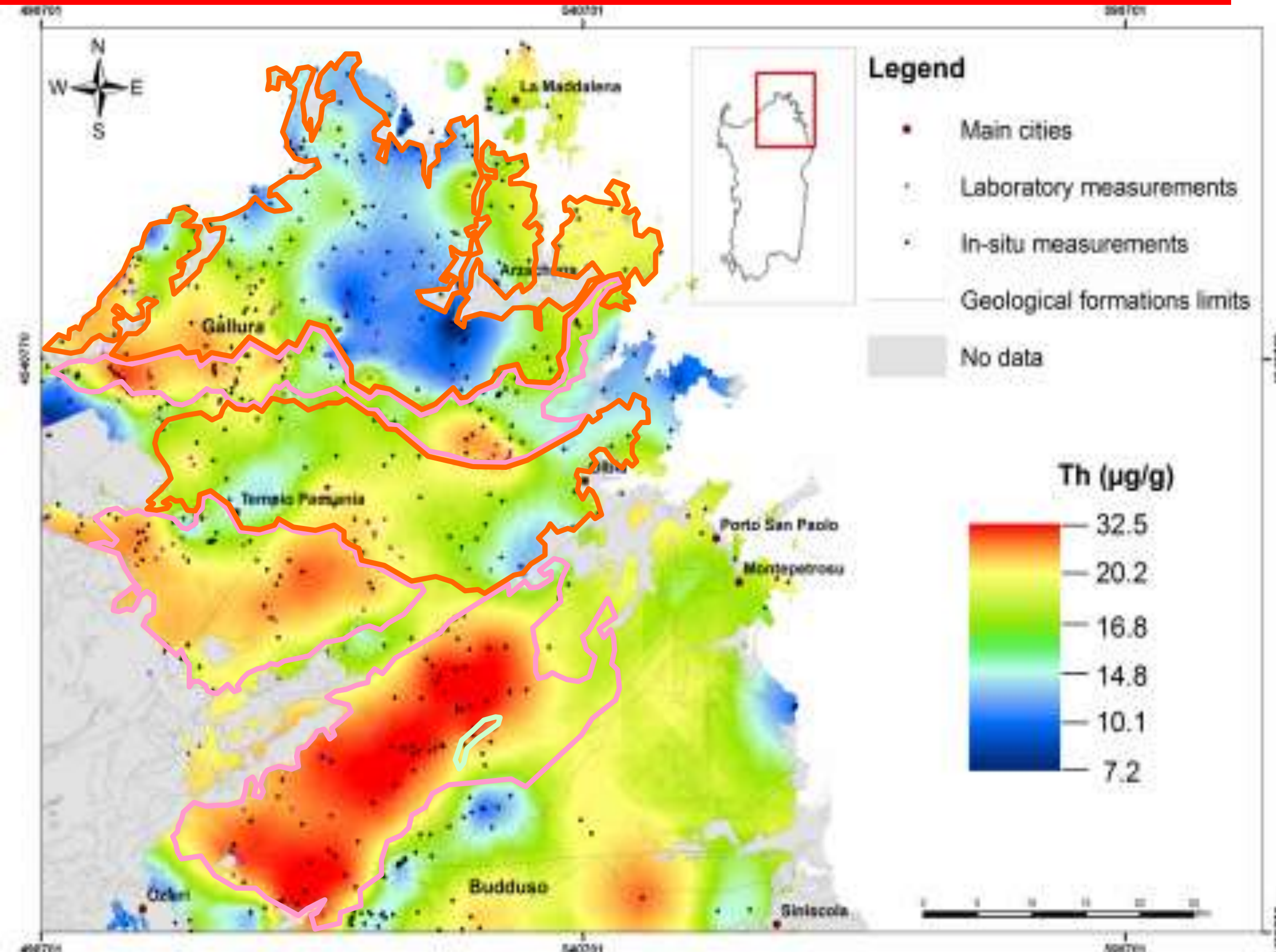


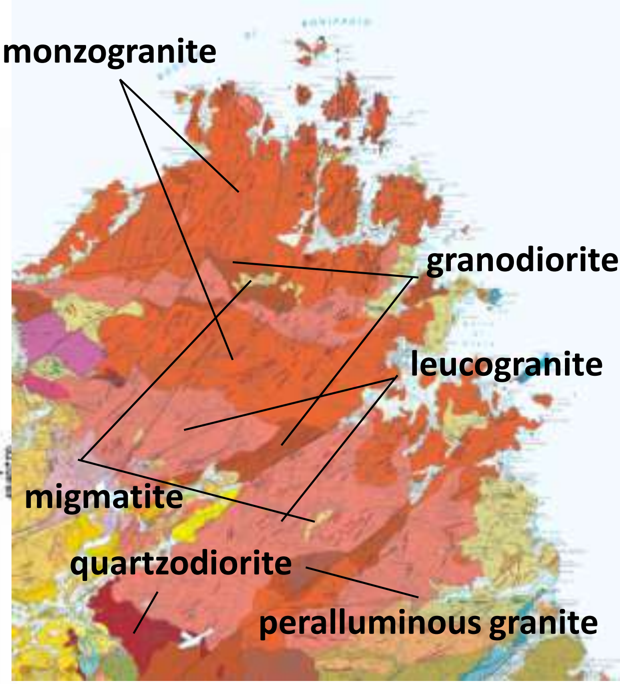
- The main geological structures are confirmed by the map of U (µg/g) abundance.

- Leucogranites are relatively homogeneous and they show the highest U abundances.
- There are no evidences of anomalies in the U abundances distribution.
- Evidences of relatively high dis-homogeneity is shown in the N part.

- Monzogranites show relatively high dis-homogeneity of U (µg/g) abundance.
- Again the pattern of the distribution of U abundance in Gallura seems to be affected by the presence of dykes spacing from basic (NE) and acid (NW)

Map of Th (µg/g) abundance



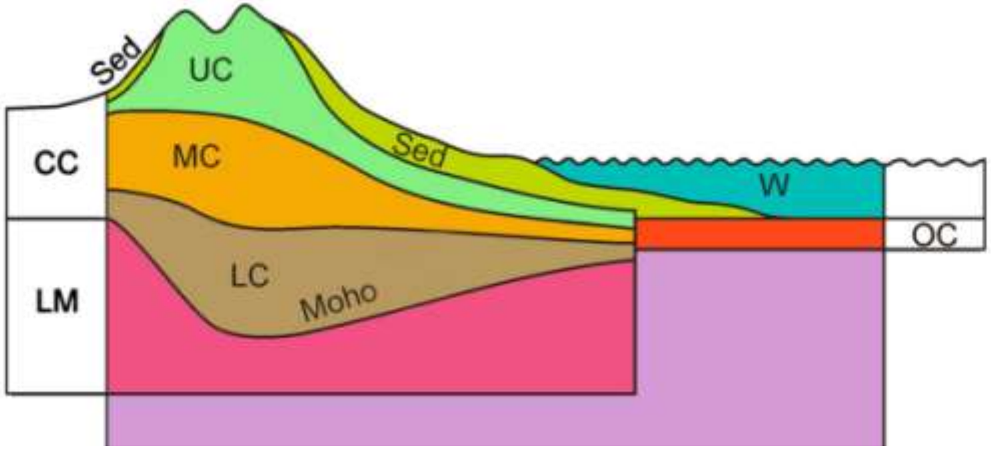


- The main geological structures are confirmed by the map of Th (µg/g) abundance.

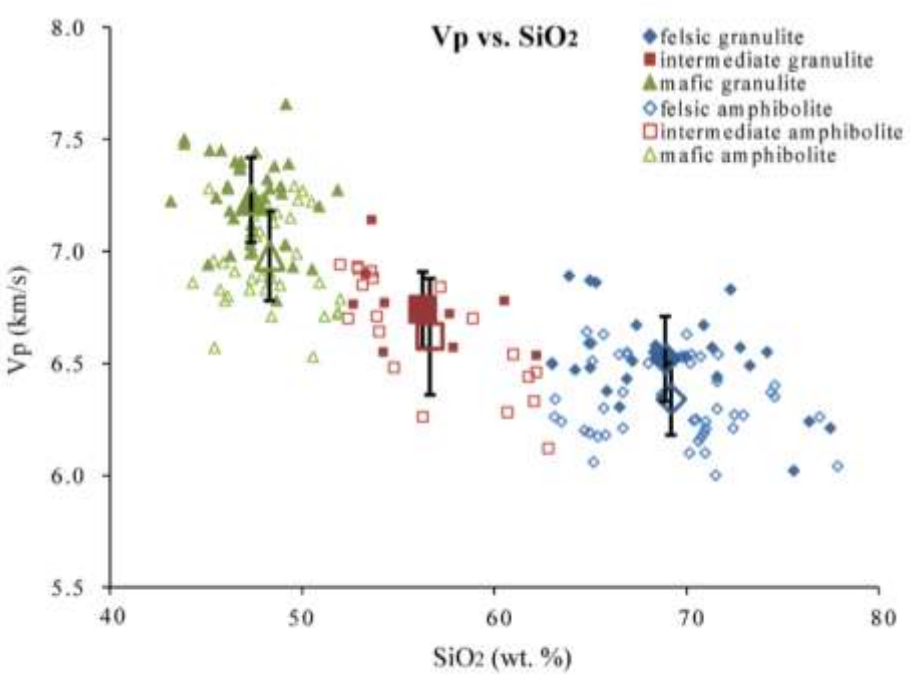
- Leucogranites of Buddusò show the highest abundances of Th (µg/g).
- There are no evidences of anomalies for the Th abundances.
- Evidences of relatively high dis-homogeneity is shown in the N part.

- Monzogranites show relatively high dis-homogeneity of Th (µg/g) abundance.
- Again the pattern of the distribution of Th abundance in Gallura seems to be affected by the presence of dykes spacing from basic (NE) and acid (NW)

How much U, Th and K is in the deep crust?



- Felsic and mafic rocks can be distinguished on the basis of P and S waves velocities
- Ultrasonic velocity measurements of deep crustal rocks provide a link between seismic velocity and lithology.



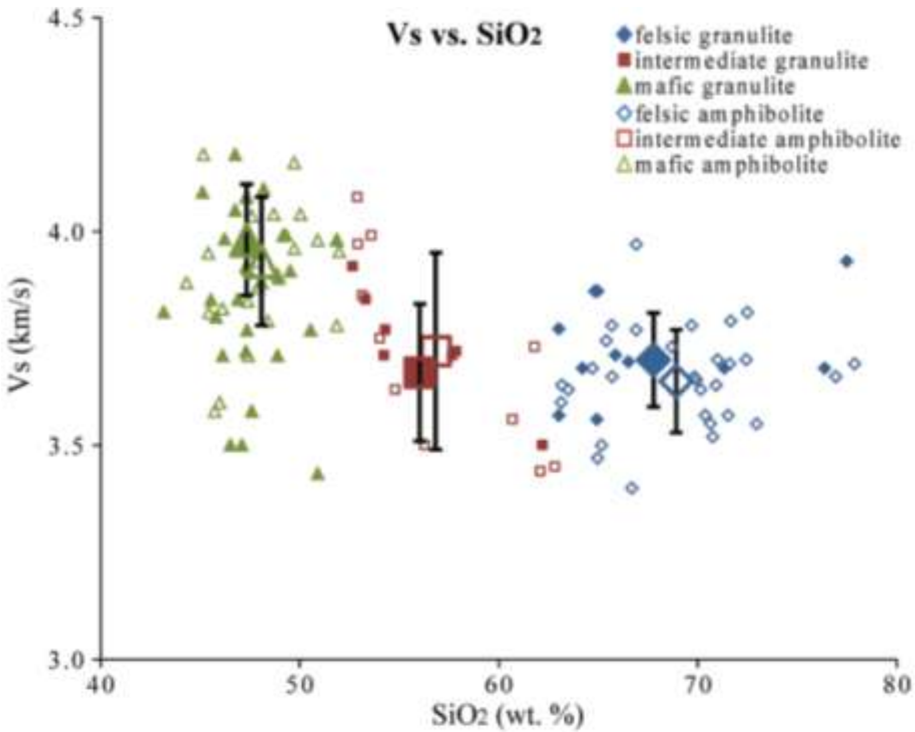
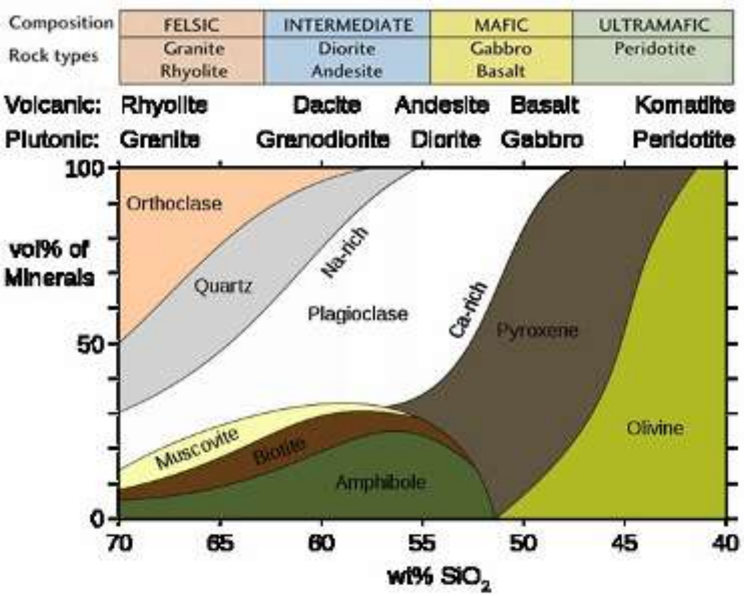


Figure from Huang Y et al. 2013 – Geochemistry, Geophysics, Geosystems – 2013.

SiO_2 content in Sardinia rock lithotypes

This study based on bibliographic data on elemental composition SiO_2 of the most representative rocks of the Corsica-Sardinia Batholiths.

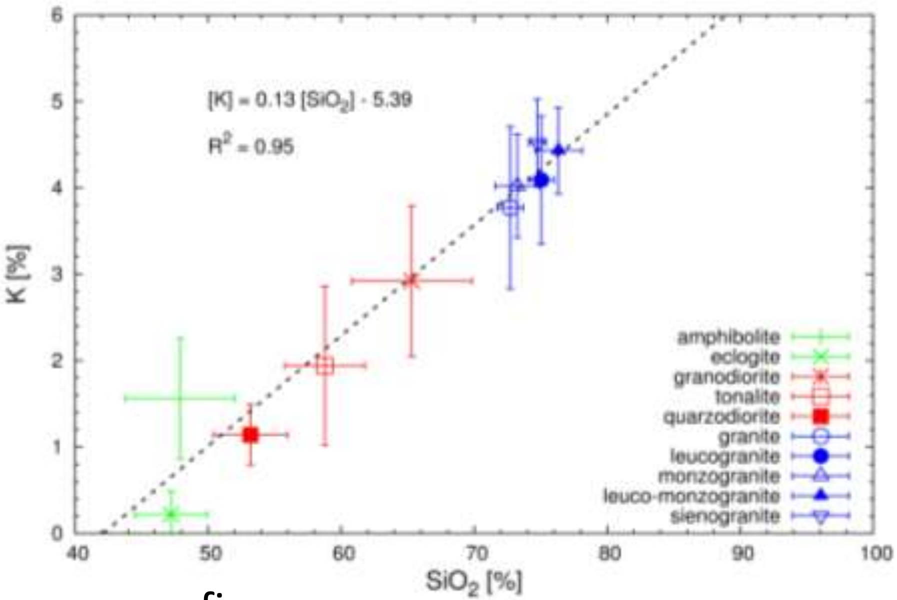
The basic classification scheme for igneous rocks on their mineralogy

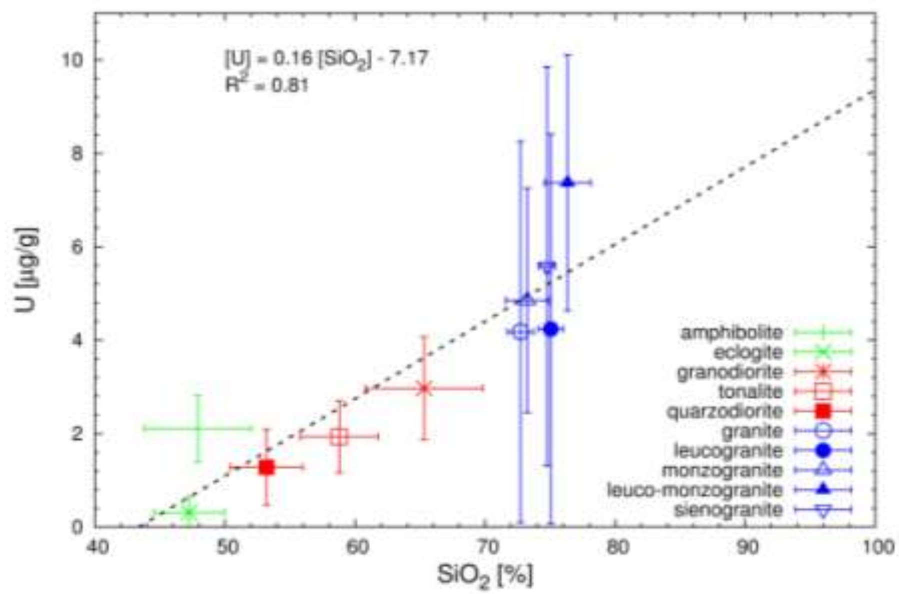


Rock type		$SiO_2 \pm \sigma$ (wt. %)
Amphibolite	Mafic (45-52 %)	47.9 ± 4.1
Eclogite		47.2 ± 2.7
Granodiorite	Intermediate (52-63%)	65.3 ± 4.5
Tonalite		58.8 ± 3.0
Quarzodiorite		53.2 ± 2.8
Granite	Felsic (> 63%)	72.7 ± 1.0
Leucogranite		75.1 ± 1.0
Monzogranite		73.2 ± 1.6
leuco-monzogranito		76.3 ± 1.8
Sienogranito		74.7 ± 0.6

The average (± 1 σ standard deviation) SiO2 (in % wt.) content in the rocks of Sardinia obtained from bibliographic studies.

Correlation between SiO_2 and K, U, Th abundances

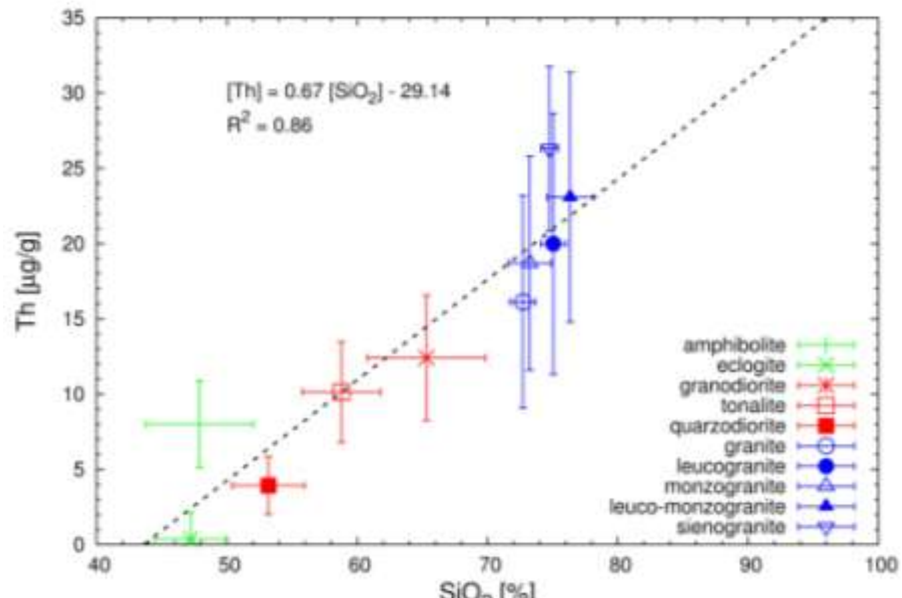




mafic

intermediate

felsic



- I calculate a linear correlation between K, U and Th abundances and SiO_2 content.
- A good correlation ($r^2 \sim 0.9$) between SiO_2 and K, U and Th abundances is confirmed.

Preliminary study of radiogenic heat generation

Specific heat power (a) generated from U and Th decay chains and ^{40}K decay

Decay	**Natural isotopic abundance**	**a (W/kg)**
$^{40}K \rightarrow {}^{40}Ca + e^- + \bar{\nu}$ (89%)	1.17×10^{-4}	2.55×10^{-9}
$^{40}K + e^- \rightarrow {}^{40}Ar + \nu$ (11%)	1.17×10^{-4}	0.78×10^{-9}
	total K	0.33×10^{-9}
$^{238}U \rightarrow {}^{206}Pb + 8\,{}^{4}He + 6e^- + 6\bar{\nu}$	0.9927	9.45×10^{-5}
$^{235}U \rightarrow {}^{207}Pb + 7\,{}^{4}He + 4e^- + 4\bar{\nu}$	0.0072	0.40×10^{-5}
	total U	9.85×10^{-5}
$^{232}Th \rightarrow {}^{208}Pb + 6\,{}^{4}He + 4e^- + 4\bar{\nu}$	1.0000	2.64×10^{-5}

The **radiogenic heat production rate (A)** generated by rock types can be calculated as:

$$A\,(\mu W / m^3) = \rho\left(9.85C_U + 2.64C_{Th} + 3.33C_K\right)\times10^{-5}$$

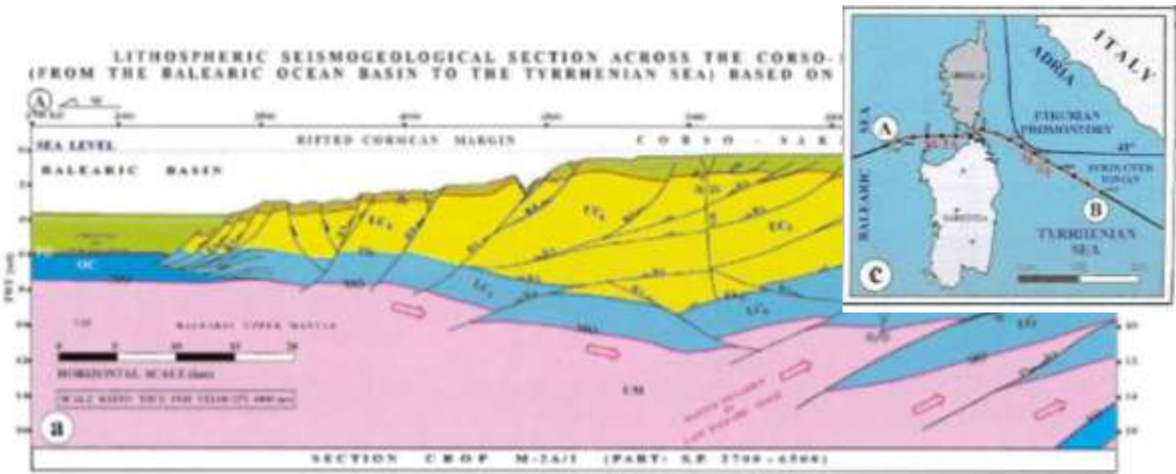
$\mathbf{C_U}$, $\mathbf{C_{Th}}$ (in µg/g) and $\mathbf{C_K}$ (in %) are the abundances of U, Th and ^{40}K

ρ is the rock density (in kg/m³).

Heat production rate A (μW/m³)

Rock-type	No.	K (%)	U (μg/g)	Th (μg/g)	ρ (kg/m³)	A (μW/m³)
monzogranite	446	4.02 ± 0.60	$4.85^{+2.40}_{-1.62}$	$18.70^{+7.11}_{-5.17}$	2750	**$3.12^{+0.83}_{-0.63}$**
granodiorite	87	2.92 ± 0.87	$2.97^{+1.10}_{-0.81}$	12.41 ± 4.16	2750	$2.51^{+0.71}_{-0.55}$
migmatite	25	3.42 ± 0.60	$4.55^{+1.40}_{-1.07}$	13.10 ± 4.76	2750	$2.52^{+0.51}_{-0.46}$
granite	21	3.77 ± 0.94	$4.18^{+4.08}_{-2.07}$	16.13 ± 7.07	2750	$2.73^{+1.16}_{-0.81}$
leucogranite	16	4.09 ± 0.74	$4.24^{+4.17}_{-2.11}$	19.99 ± 8.67	2750	**$3.07^{+1.24}_{-0.91}$**
leuco-monzogranite	16	4.43 ± 0.50	7.37 ± 2.74	23.09 ± 8.31	2750	**$4.08^{+0.97}_{-0.95}$**
orthogneiss	13	$3.31^{+0.93}_{-0.73}$	$4.40^{+2.42}_{-1.56}$	$12.78^{+5.96}_{-4.02}$	2750	$2.51^{+0.77}_{-0.56}$
amphibolite	12	1.56 ± 0.70	2.11 ± 0.71	$8.01^{+2.90}_{-2.14}$	3300	**$1.57^{+0.34}_{-0.32}$**
eclogite	11	$0.22^{+0.27}_{-0.12}$	$0.31^{+0.34}_{-0.16}$	$0.37^{+1.78}_{-0.31}$	3300	**$0.20^{+0.22}_{-0.09}$**
sienogranite	11	4.52 ± 0.51	$5.58^{+4.27}_{-2.40}$	26.31 ± 5.46	2750	**$3.87^{+1.20}_{-0.78}$**
alcaline-granite	10	3.96 ± 0.19	4.16 ± 1.33	$19.85^{+5.19}_{-4.16}$	2750	$2.95^{+0.52}_{-0.48}$

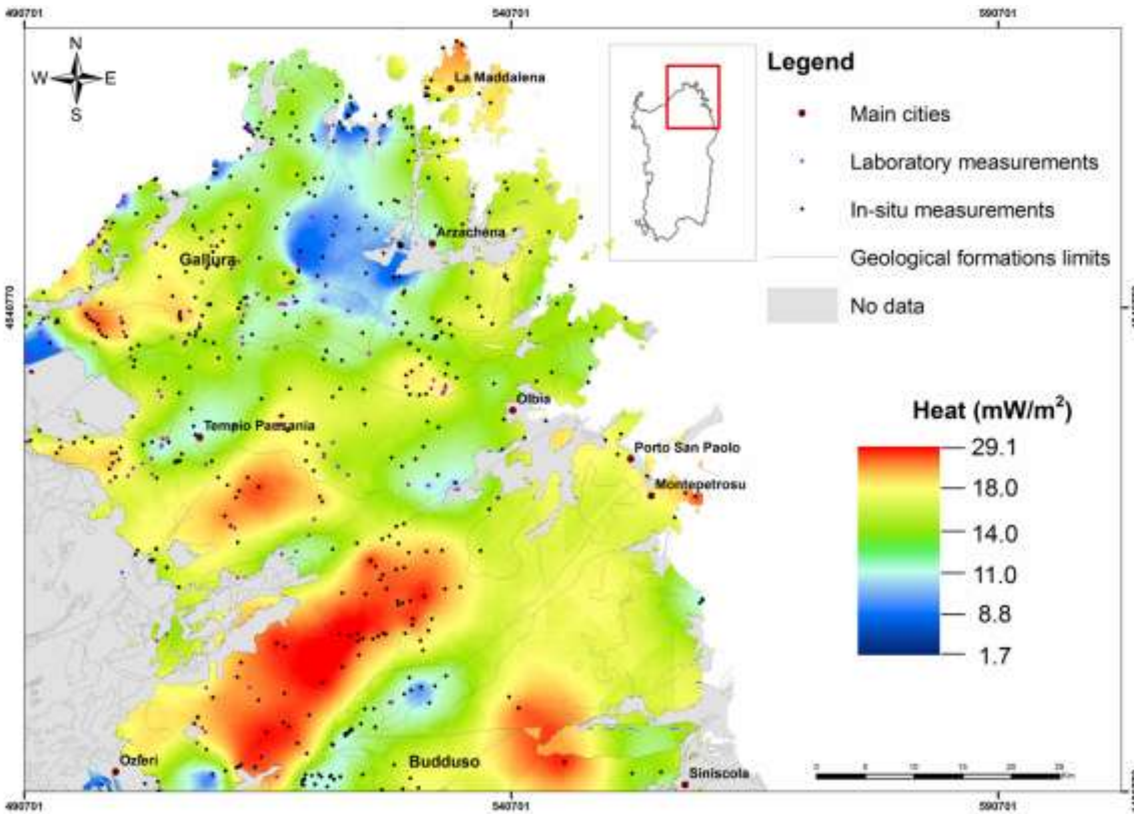
A geophysical model is built on the base of seismic data, which constrain the thickness of Upper Crust.

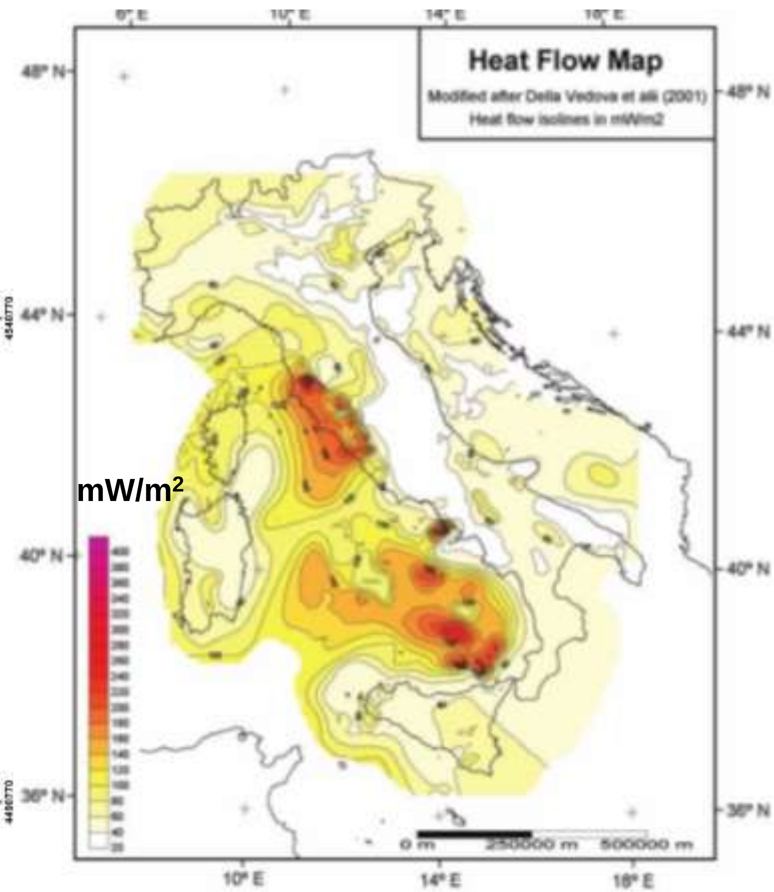


Preliminary study of radiogenic heat generation in UC

In a first approximation, we calculated the surface heat flow rate generated from the upper crust due to the presence of the isotope of ^{40}K, U and Th assuming a constant thickness:

$$H_{rad}\ (mW/m^2) = A \times h$$



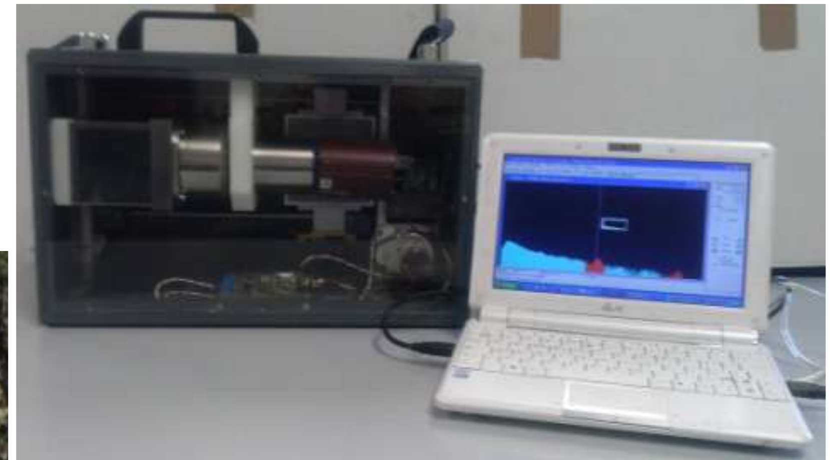


The surface heat production rate varies between 5.4 ± 2.5 mW/m^2 (quarzodiorite) to 18.4 ± 6.8 mW/m^2 (leucogranite) with an average of 14.9 ± 5.6 mW/m^2 (upper crust only).

Similar studies in this are performed in Corsica indicate that the heat conducted through the whole crust from the underlying mantle is on average is 33 mW/m^2 (M. Verdoya et al.1998.Tectonophysics.1998**)**

PART II

A new portable instrument for detailed in-situ gamma-ray survey



Scientific and technological motivations

Desired features for a customized instument

- **In-situ accurate identification and quantification of radionuclides**
- **Portable lightweight collimated instrument**
- **Quick response measurement on few cm^2 field of view**

APPLICATIONS:
Natural radioactivity survey (e.g. decorative stones quarries)
Geophysical survey (e.g identification of uranium and thorium ore veins)
Homeland security (e.g. orphan source identification)

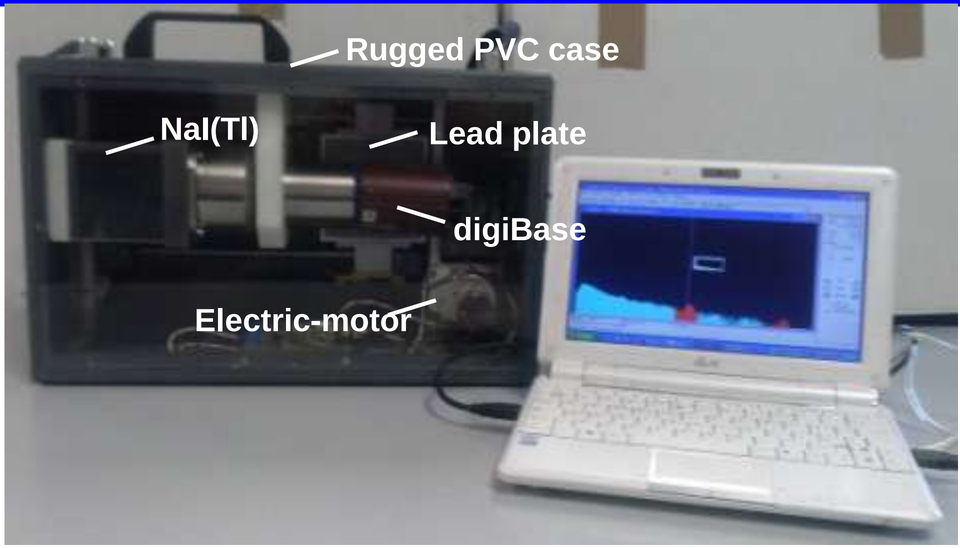
A new portable instrument: Cava-Rad System

PARAMETERS:

Physical parameters
Dimensions (L 43.0 cm x H 27.0 cm x W 13.5 cm)
Weight 8.0 kg
Environmental parameters
Temperature -10 to +50 °C
Humidity 85%

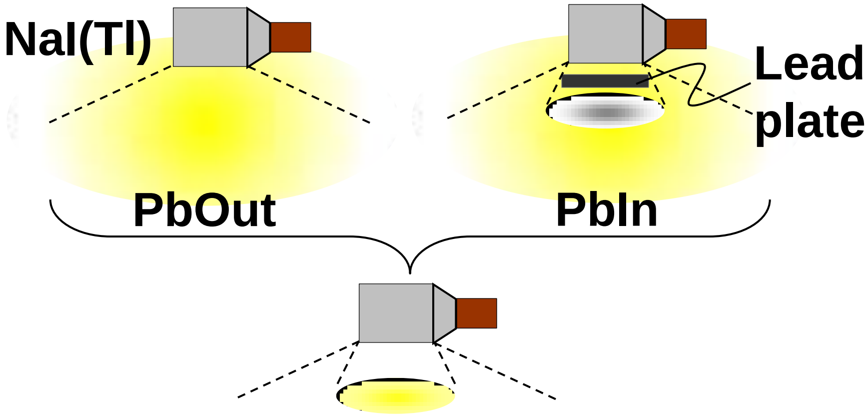
Gamma-ray detector
NaI(Tl) scintillation detector of 0.3 L volume
Energy resolution
7.3 % at 662 keV (^{137}Cs)
5.2 % at 1172 and 1332 keV (^{60}Co)
Physical parameters
Dimensions (L 7.62 cm x H 7.62 cm x W 7.62 cm)
Weight 2.0 kg

Collimation configuration
Lead plate
Physical parameters
Dimensions (L 9.0 cm x H 9.0 cm x W 3.0 cm)
Weight 3.0 kg

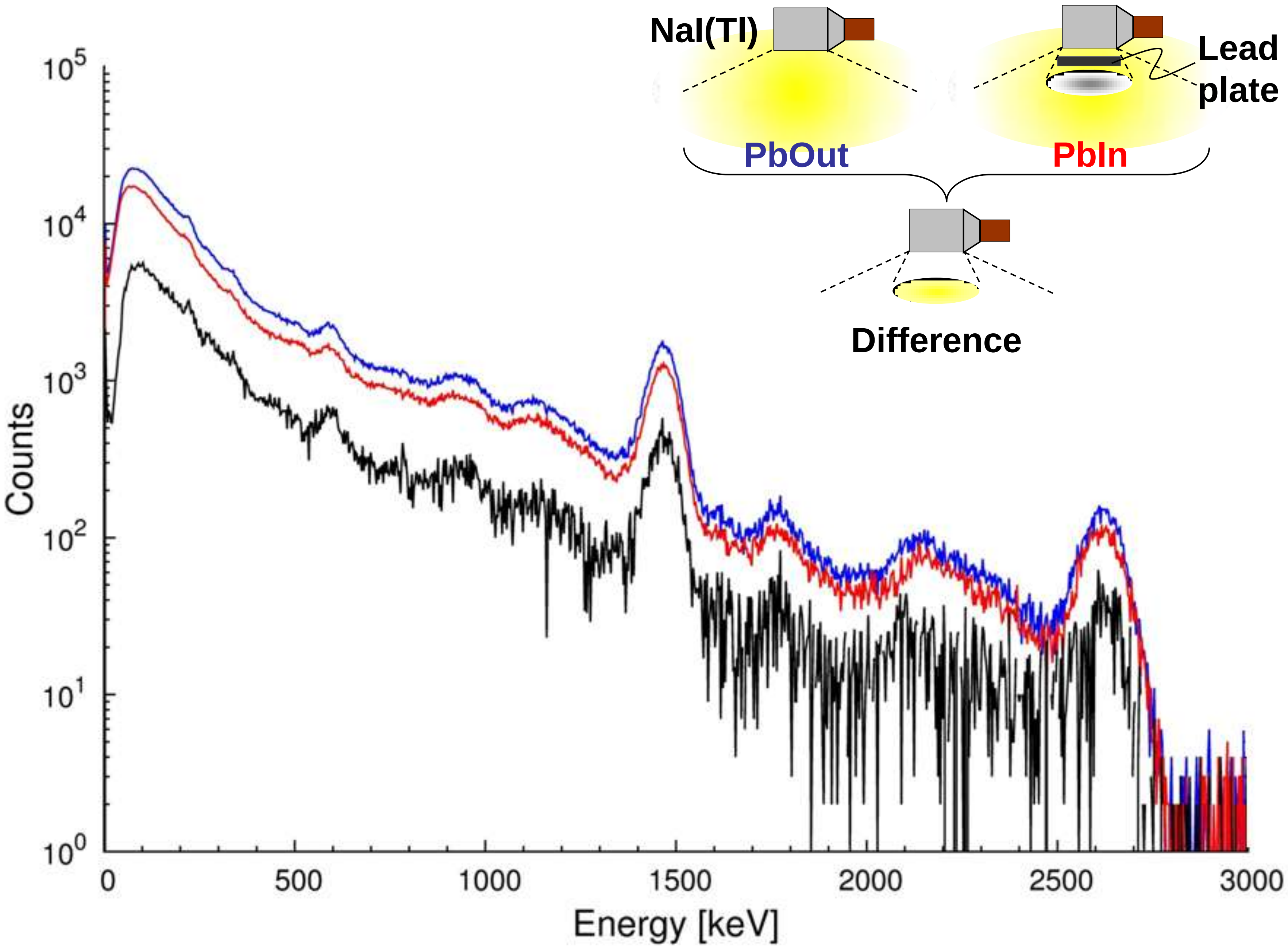


HOW DOES IT WORK?

Cava-rad system uses the "lead plate" method in order to obtain the collimation effect and perform measurements on a restricted area

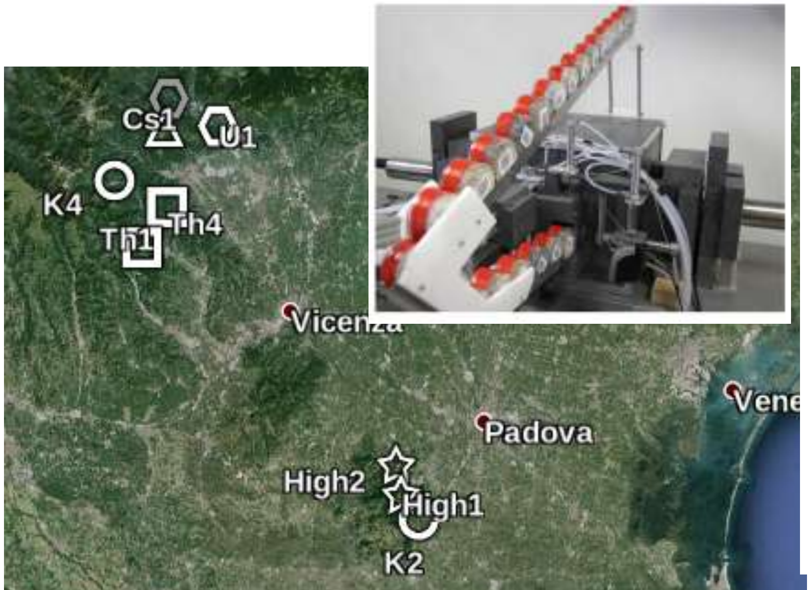


Typical Cava-Rad spectra



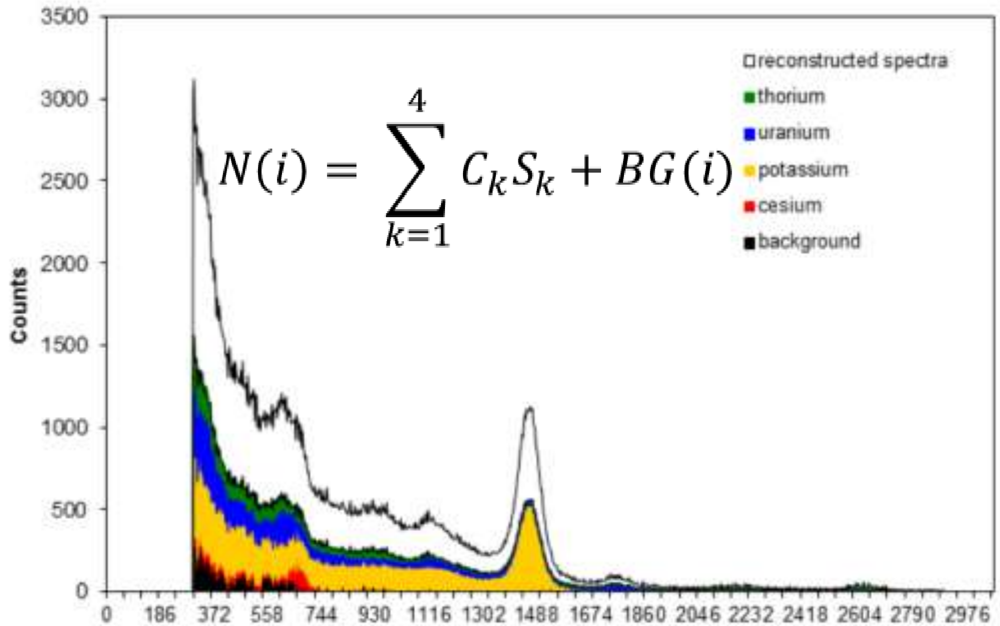
Calibration of Cava-Rad system

The radioactivity content of reference sites used for calibration of Cava_Rad is measured by HPGe



Site	Location	Natural pad for
K2	Galzignano terme	^{40}K
K4	Recoaro	^{40}K
U1	Piovene Rocchette	^{238}U
U3	Arsiero	^{238}U
Th1	Castelvecchio (Altissimo)	^{232}Th
Th4	Corbara (Schio)	^{232}Th
Cs1	Monte Novegno (Schio)	^{137}Cs
H1	Galzignano terme	High conc. of all
H2	Galzignano terme	High conc. of all

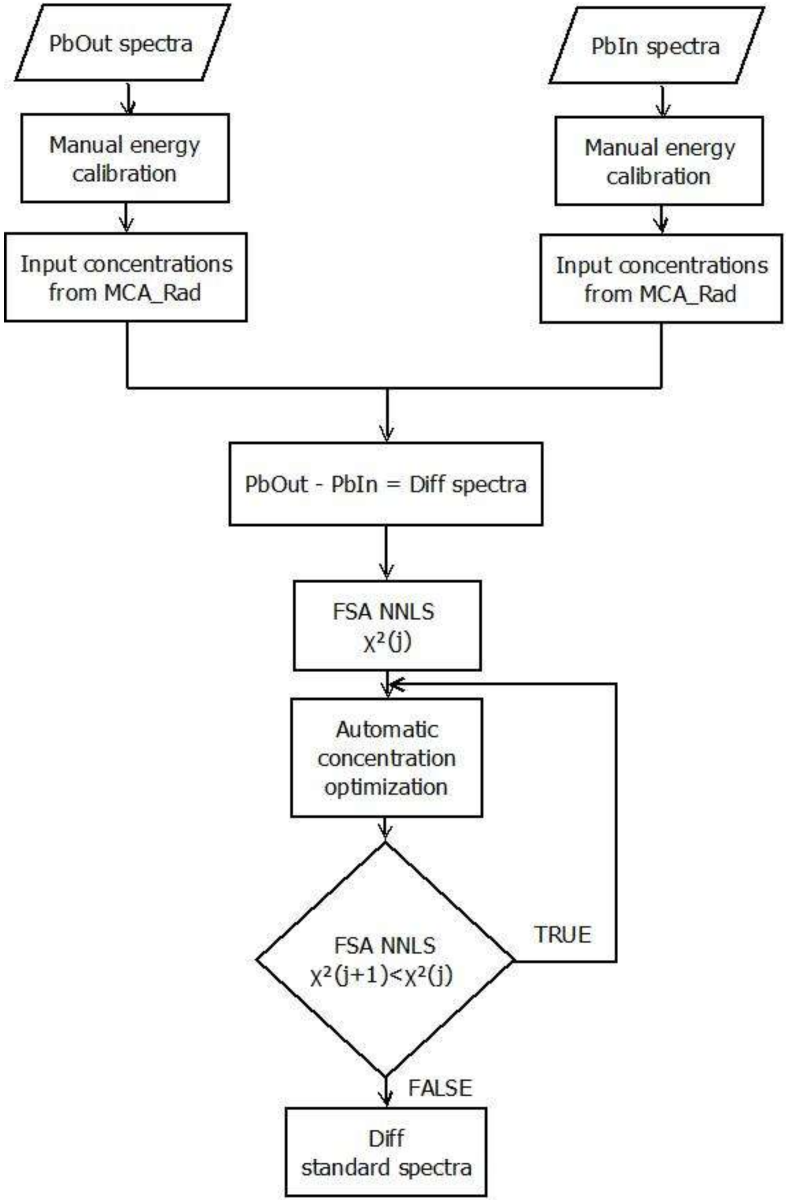
Full Spectrum Analysis with Non Negative Least Squares constraints



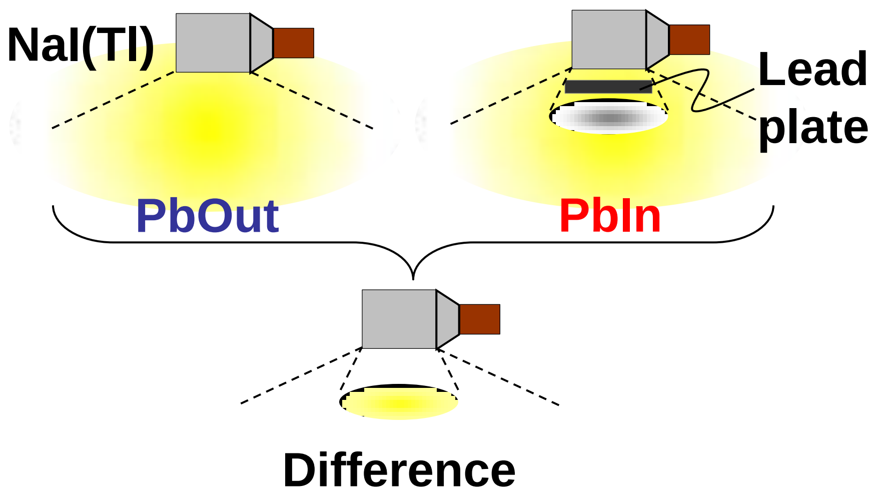
$$\chi^2 = \frac{1}{n-5} \sum_{i=1}^{n} \left[N(i) - \sum_{k=1}^{4} C_k S_k(i) - BG(i) \right]^2 \frac{1}{N(i)}$$

- *N(i)* counts in the channel *i*,
- C_k concentration of the element *k*,
- $S_k(i)$ associated counts to fundamental spectrum of the element *k* in the channel *i*,
- *BG(i)* counts in the channel *i* due to the - background.

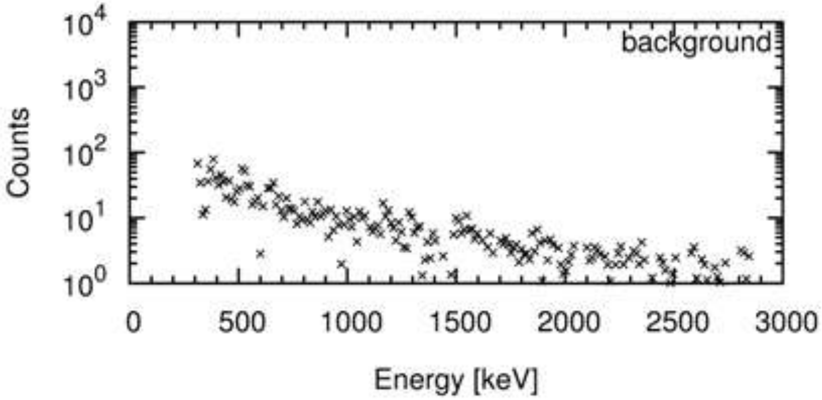
Cava-Rad System: Sensitivity Calibration



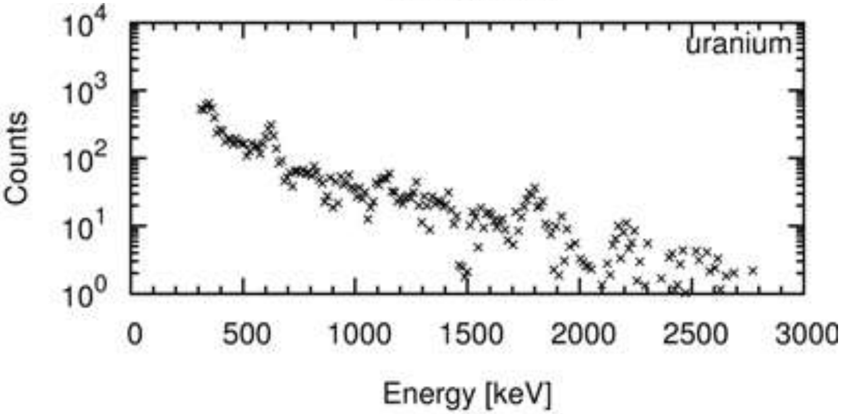
For each site we subtract the energy calibrated spectra, PbOut – PbIn and use the difference spectra as input for the FSA-NNLS algorithm in order to obtain the sensitivity spectra of the differences

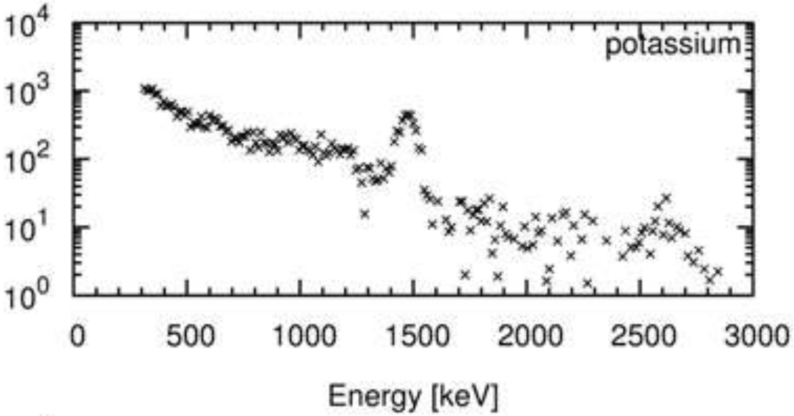


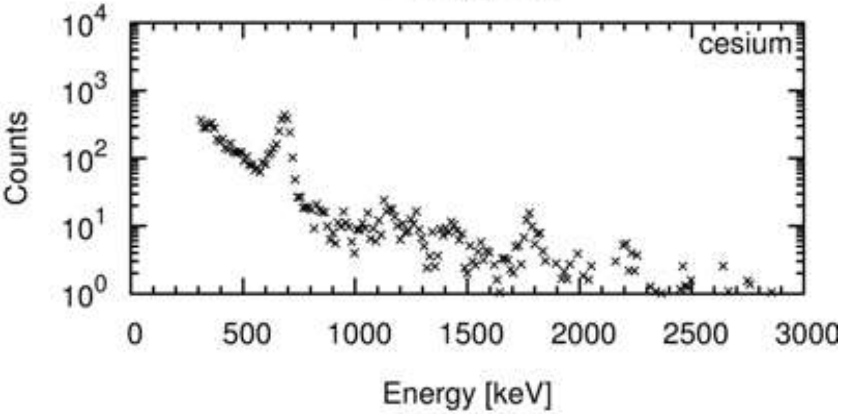
Standard spectra of Cava-Rad system

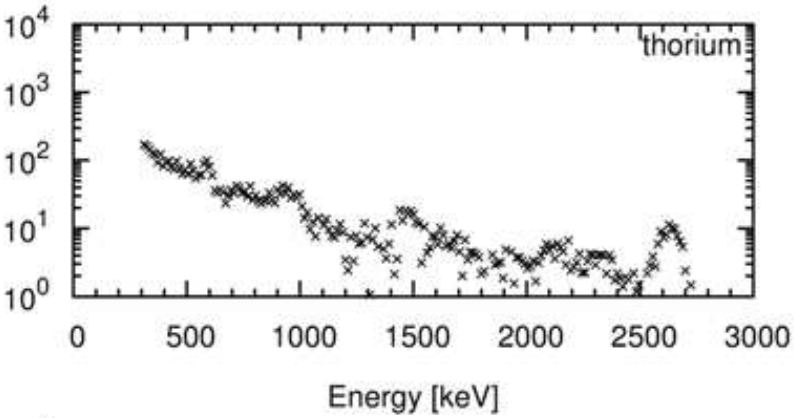


Background: internal BG, cosmic rays and atmospheric radon





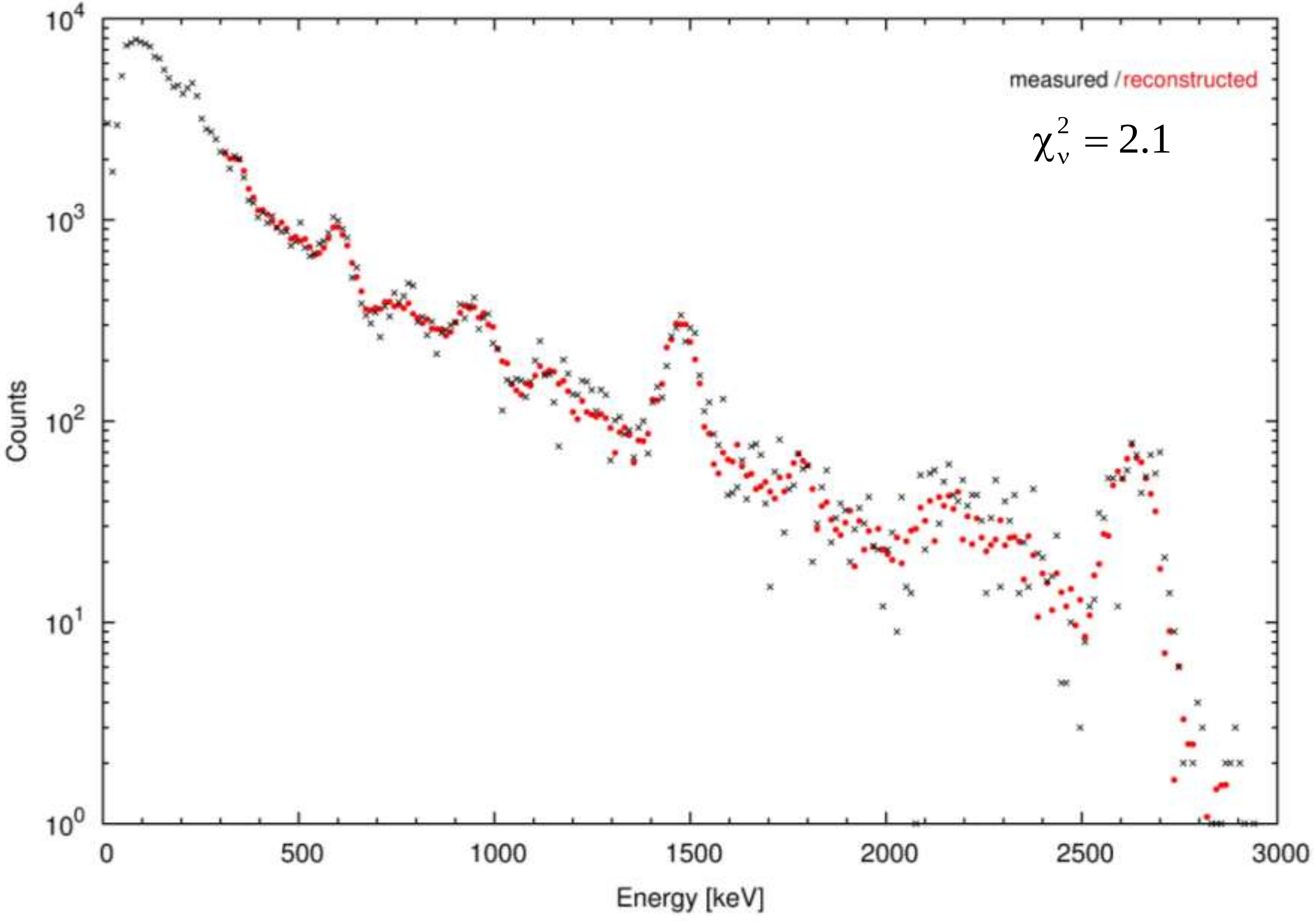




✓ **The obtained spectra show the expected shape**

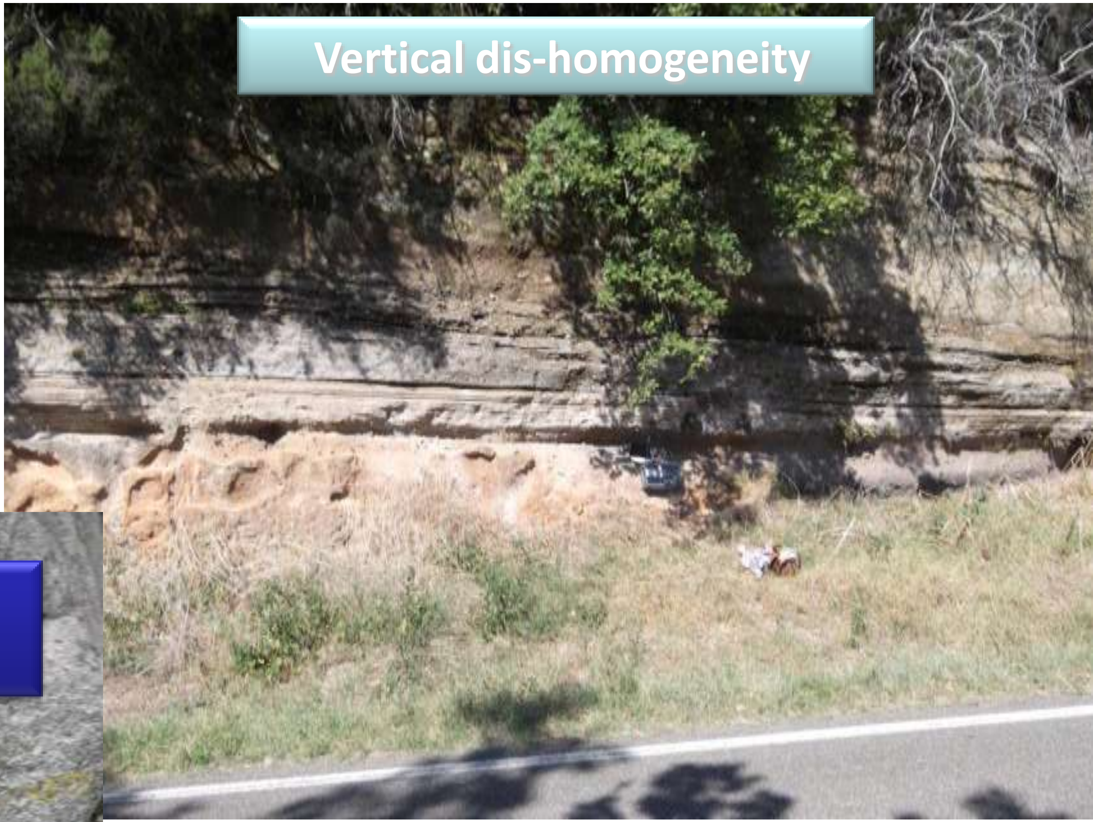
✗ **The standard spectra are affected by low statistics**

Quality of reconstructed spectrum

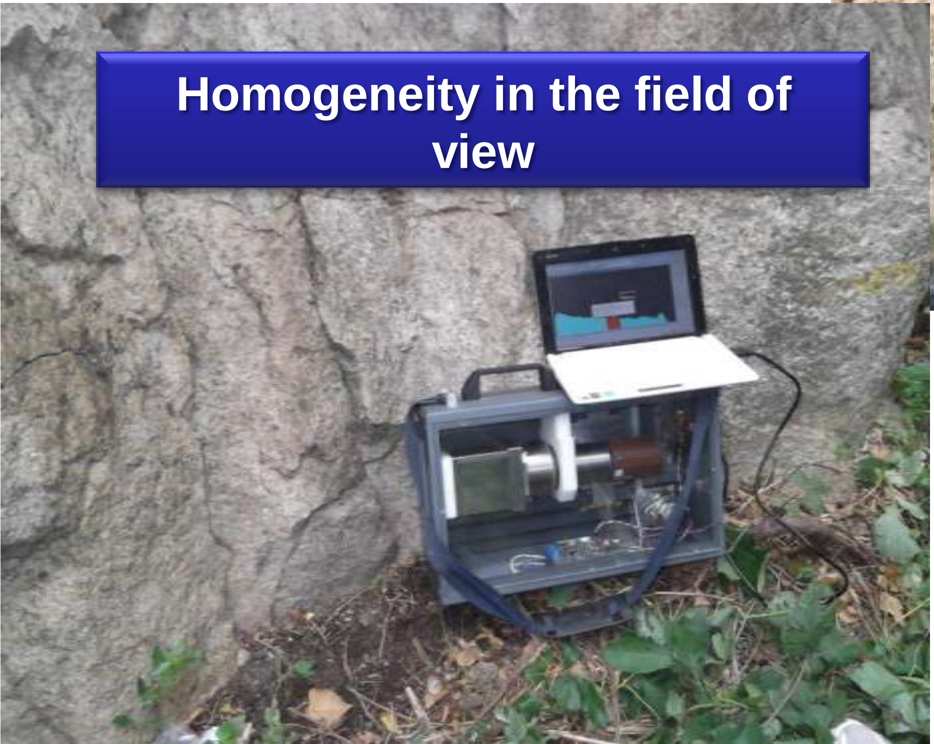


Validation of Cava-Rad results

Monti Vulsini (south Tuscany)
Deposits of different piroclastic rocks due to volcanic activity (~300.000 years ago). High spatial variability of radionuclides abundances.



Euganean Hills (Veneto)
Homogeneous outcrops of acid effusive rocks.

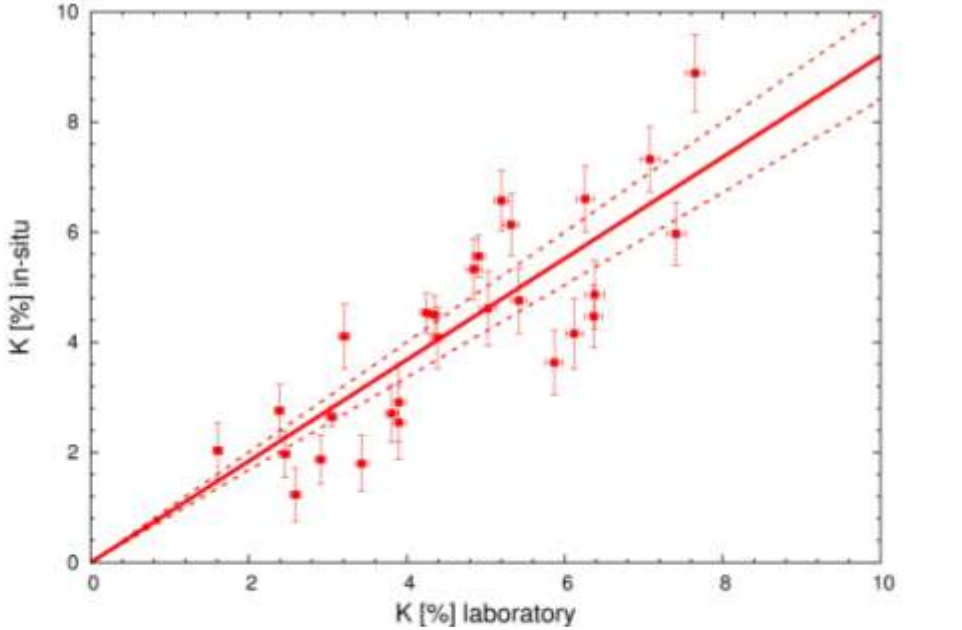


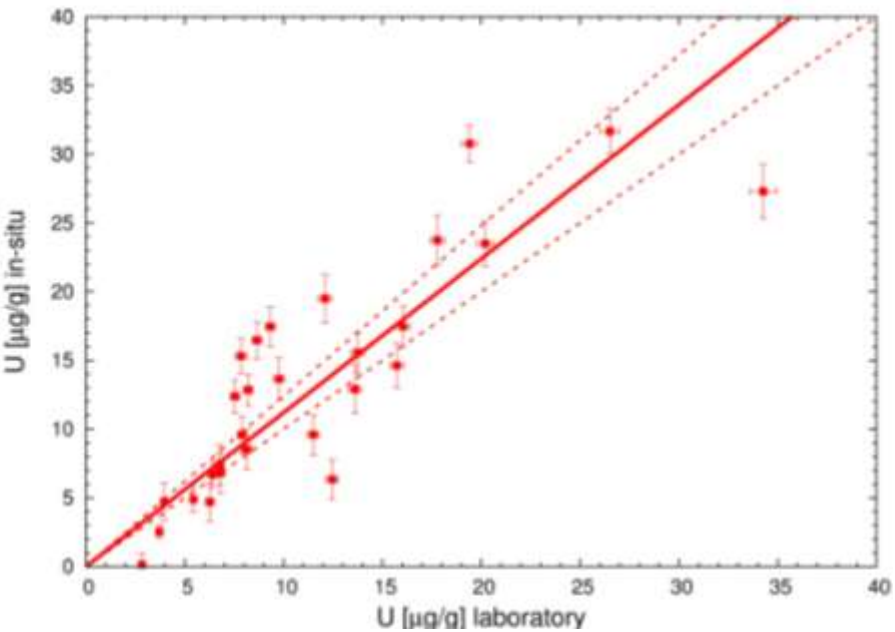
In-situ:
- PbOut and PbIn measurements
- Acquisition time: 5 or 10 minutes

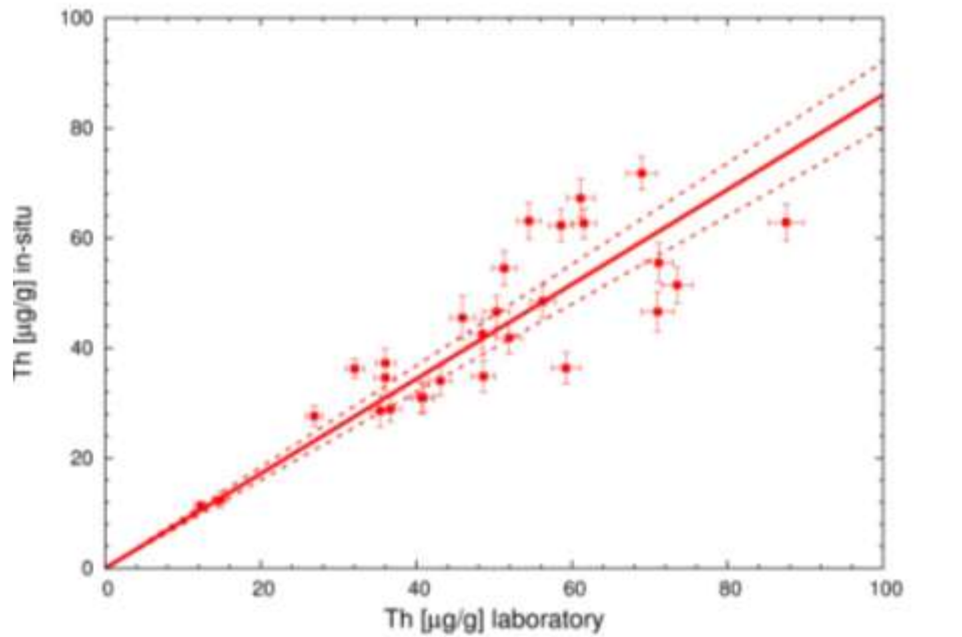
In-laboratory:
- Sample collection, under the detector
- Analysis in laboratory (MCA_Rad)

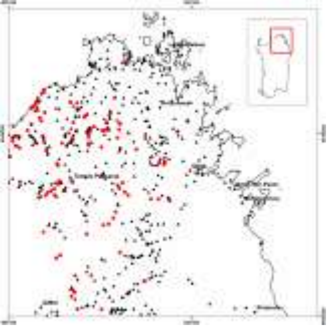
Correlation between in-situ and laboratory measurements

Radionuclide	m ± σ
^{40}K (%)	0.92 ± 0.08
^{238}U (μg/g)	0.82 ± 0.17
^{232}Th (μg/g)	0.67 ± 0.10





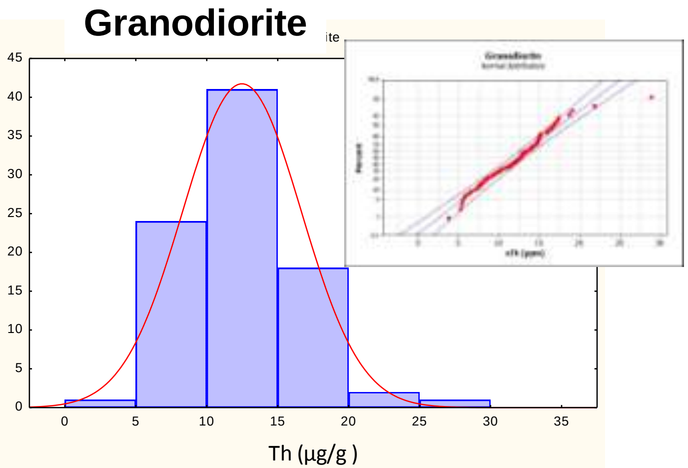




Discussion and conclusions

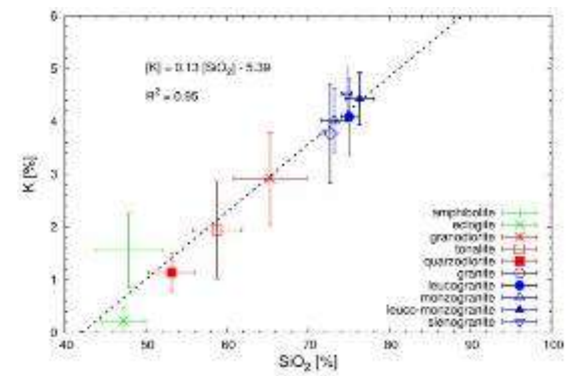
The N-E Sardinia was **investigated with 545 in-situ** measurement using a portable gamma-ray spectrometer **NaI(Tl)** and by collecting **167 rock samples** and measuring them in the laboratory using high-resolution gamma-ray spectrometer **(HPGe).**

Statistical analysis of the data confirmed that K and Th generally show a Gaussian distribution as U show the log-normal distribution.

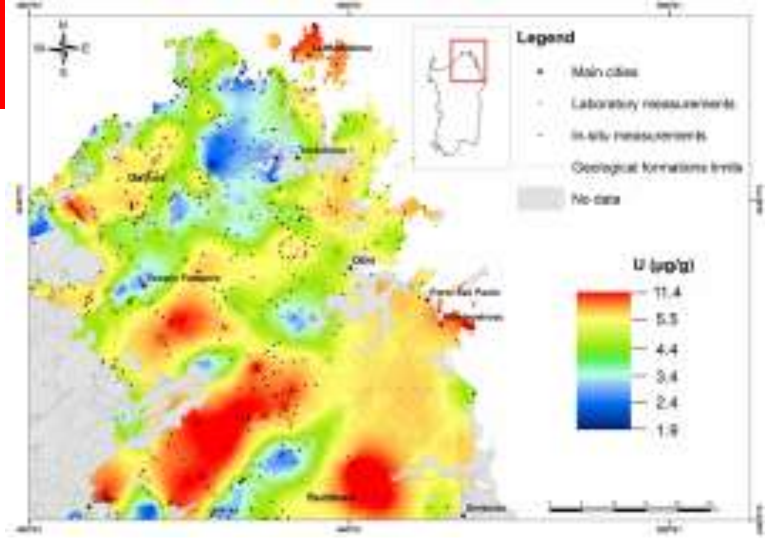


This study shows that the **concentrations** of ^{40}K, U e Th in N-E Sardinia batholite are **comparable** with the values obtained in the European Varsican Batholiths and on average higher than typical concentrations (2.32 % K, 2,7 µg/g U e 10,5 µg/g Th) for the upper continental crust

The concentrations of K, U and Th show an increasing tendency from passing from **mafic to intermediate to felsic rocks**.



Discussion and conclusions



Radiometric maps of K, U and Th concentrations are built using kriging interpolator.

The **radiogenic heat production** rate for various types of rock in the N-E Sardinia Batholith varies from $0,20^{+0,22}_{-0,09}$ μW/m³ (eclogite) di 4.87 ± 1.00 μW/m³ (Monzo-sienogranite). These results are comparable with other study of Europian Varsican Batholith.

In-situ gamma-ray spectrometry with scintillation NaI(Tl) detectors is widely used for assessing natural radionuclides **under homogeneous** spatial variability.

Cava_Rad is **calibrated** with the best approach and a set of **standard spectra** is included in the software ready to use in-situ measurements.

The Cava-Rad system was found to be a quick and practical instrument when applied on geological sites with particular **spatial variability.**

Scientific Publications

1.E. Guastaldi, M. Baldoncini, G.P. Bezzon, C. Broggini, G.P. Buso, A. Caciolli, L. Carmignani, I. Callegari, T. Colonna, K. Dule, G. Fiorentini, **M. Kaçeli Xhixha**, F. Mantovani, G. Massa, R. Menegazzo, L. Mou, C. Rossi Alvarez, V. Strati, G. Xhixha, A. Zanon. *A multivariate spatial interpolation of airborne γ-ray data using the geological constraints.* **Remote Sensing of Environment (2013)**. Doi: http://dx.doi.org/10.1016/j.rse.2013.05.027

2. I. Callegari, G.P. Bezzon, C. Broggini, G.P. Buso, A. Caciolli, L. Carmignani, T. Colonna, G. Fiorentini, E. Guastaldi, **M. Kaçeli Xhixha**, F. Mantovani, G. Massa, R. Menegazzo, L. Mou, A. Pirro, C. Rossi Alvarez , V. Strati, G. Xhixha, A. Zanon. *Total natural radioactivity map of Tuscany (Italy).* **Journal of Maps (2013)**. Doi: http://dx.doi.org/10.1080/17445647.2013.802999

3. A. Puccini, G. Xhixha, S. Cuccuru, G. Oggiano, **M. Kaçeli Xhixha**, F. Mantovani, C. Rossi Alvarez, L. Casini. *Radiological characterization of granitoid outcrops and dimension stones of the Variscan Corsica-Sardinia Batholith.* **Environmental Earth Sciences (2013)**. Doi: http://dx.doi.org/10.1007/s12665-013-2442-8

4. G. Xhixha, G.P. Bezzon, C. Broggini, G.P. Buso, A. Caciolli, I. Callegari, S. De Bianchi, G. Fiorentini, E. Guastaldi, F. Mantovani, G. Massa, R. Menegazzo, L Mou, A. Pasquini, C. Rossi Alvarez, M. Shyti, **M. Kaçeli Xhixha**. *The worldwide NORM production and a fully automated gamma-ray spectrometer for their characterization.* **Journal of Radioanalytical and Nuclear Chemistry (2013)** 295:445–457. Doi: http://dx.doi.org/10.1007/s10967-012-1791-1

5. V. Strati, M. Baldoncini, G. P Bezzon, C. Broggini, G.P Buso, A. Caciolli, I. Callegari, L. Carmignani, T. Colonna, G. Fiorentini, E. Guastaldi, **M. Kaçeli Xhixha**, F. Mantovani, R. Menegazzo, L. Mou , C. Rossi Alvarez, G. Xhixha, A. Zanon. Total natural radioactivity map of Veneto (Italy). **Journal of Maps (2014). Submitted**

6. F. Cfarku, G. Xhixha, E. Bylyku, P. Zdruli, F. Mantovani, F. Përpunja, I. Callegari, E. Guastaldi, **M. Xhixha Kaçeli**, H. Thoma. A preliminary study of gross alpha/beta activity concentrations in drinking waters in some Albanian cities**. Journal of Radioanalytical and Nuclear Chemistry (2014). Submitted**

Grazie a tutti

14.02.2014 Sassari