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H4.SMR/1503 - 06

**WORKSHOP ON NUCLEAR DATA FOR SCIENCE AND  
TECHNOLOGY: MATERIALS ANALYSIS**

( 19 - 30 May 2003)

**Prompt gamma activation analysis  
Part 2: Applications**

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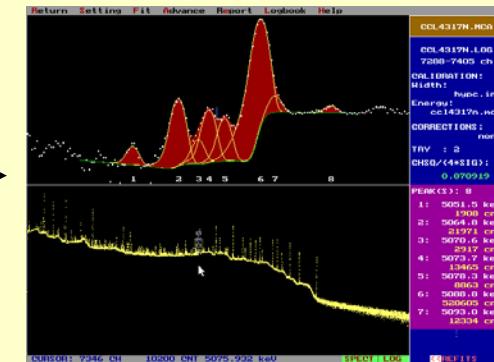
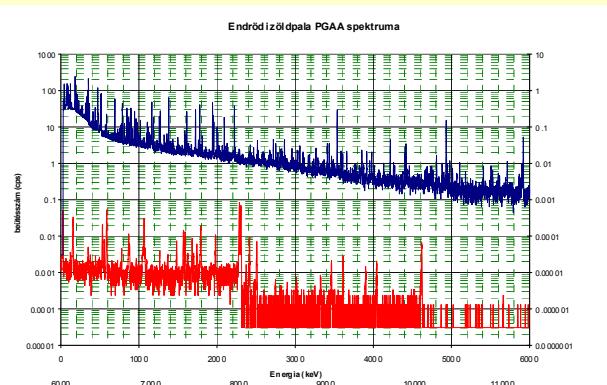
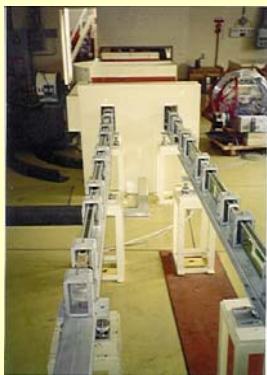
Workshop on Nuclear Data for Science and Technology:  
Materials Analysis  
ICTP Trieste, 19-30 May 2003

# Prompt gamma activation analysis Part 2: Applications

Gábor L. Molnár

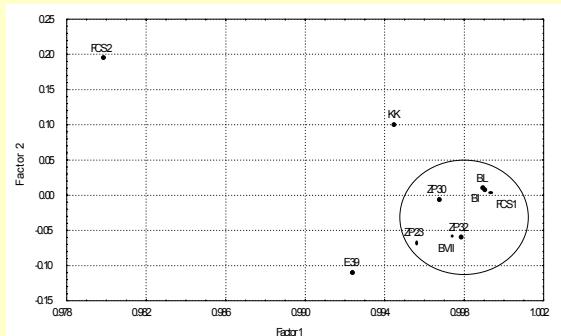
Institute of Isotope and Surface Chemistry  
Chemical Research Centre  
Budapest, Hungary

# Main steps of PGAA analysis



Spectru C:\HY PC\PECTRA\ARCHEO\ZOLDPALA\VFV41103C.MCA

Live Tim 3290.48



# Main results in methodology

- Data library transportable to other labs
- evaluation based on several peaks
  - least squares fit to component's mass
- complete analysis
  - panorama analysis
- analytical precision for the important elements
  - relative uncertainty: 1–2%

# DETERMINATION OF CHEMICAL COMPOSITION

$$A_E = m \cdot S \cdot t$$

$$S = \frac{N_A}{M} \cdot \theta \cdot \sigma_0 \cdot I_\gamma \cdot \Phi_0 \cdot \varepsilon(E_\gamma)$$

m : Mass of the element

S : Sensitivity

$A_E$  : Peak area

$N_A$  : Avogadro-number

M : Molar weight

$\theta$  : Isotopic abundance

$\sigma_0$  : Neutron capture cross-section

$I_\gamma$  : Gamma-yield

$\Phi_0$  : Neutron flux

$\varepsilon(E_\gamma)$  : Detector efficiency

# Introducing the flux- independent

$$k_{0,C}(X) = \frac{(\theta \cdot \sigma_0 \cdot I_\gamma / M)_X}{(\theta \cdot \sigma_0 \cdot I_\gamma / M)_C}$$

$$\frac{m_X}{m_Y} = \frac{A_X}{A_Y} \cdot \frac{S_{\gamma,Y}}{S_{\gamma,X}} = \frac{A_X}{A_Y} \cdot \frac{k_{0,C}(Y)}{k_{0,C}(X)} \cdot \frac{\epsilon_{\gamma,Y}}{\epsilon_{\gamma,X}}$$

gives the mass ratio of arbitrary elements

$$c_x = m_x / \sum m_i$$

gives the mass fraction (concentration)

## IN THE PAST

- Used for supplementary analysis only
  - to provide additional information on the sample
- Only in case of selected elements
  - (B, H, Cd...)
- Every lab analyzed
  - using its own data library
  - based on its own measurements
  - 1 or 2 lines per element

# NOW

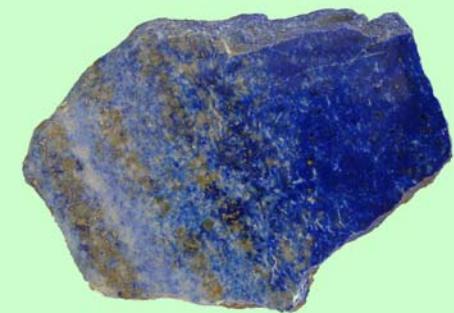
- Multielement analysis
  - to provide full information on the sample
- Analysis based on  $k_0 / \sigma_\gamma$  library
  - no need for element standards
- Several labs worldwide
  - NIST, JAERI, SNU/KAERI, BARC etc.

# **APPLICATIONS**

# **Selected Applications**

- **Rocks and minerals** (Geology, Archaeology)
- **Ceramics** (Archaeology)
- **Glasses** (Archaeology, Industry)
- **Metals** (Materials Research, Archaeology)
- **Chemistry**
- **Nuclear Materials** (Safeguards, Transmutation)

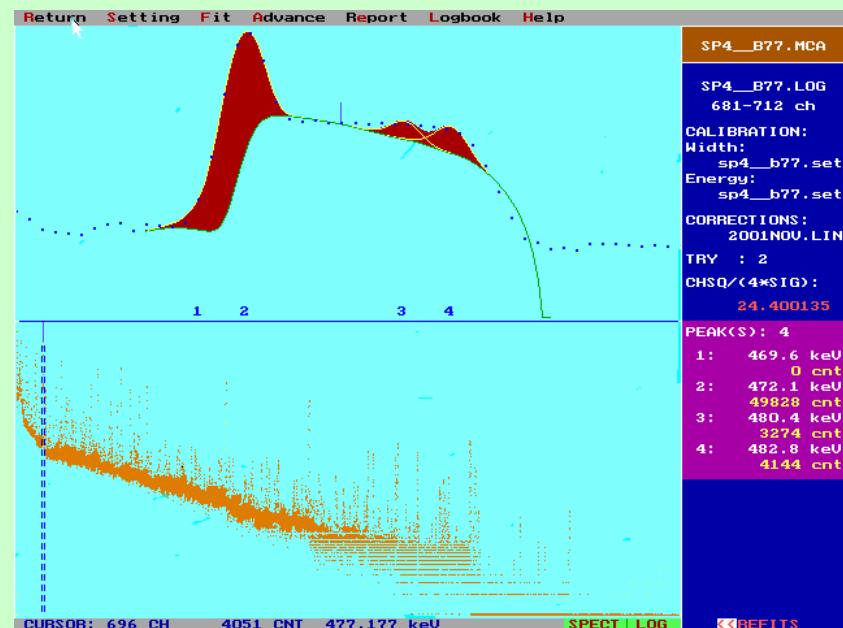
# ROCKS AND MINERALS



# BORON IN MIOCENE VOLCANIC ROCKS

(Dept. of Petrography, Eötvös University )

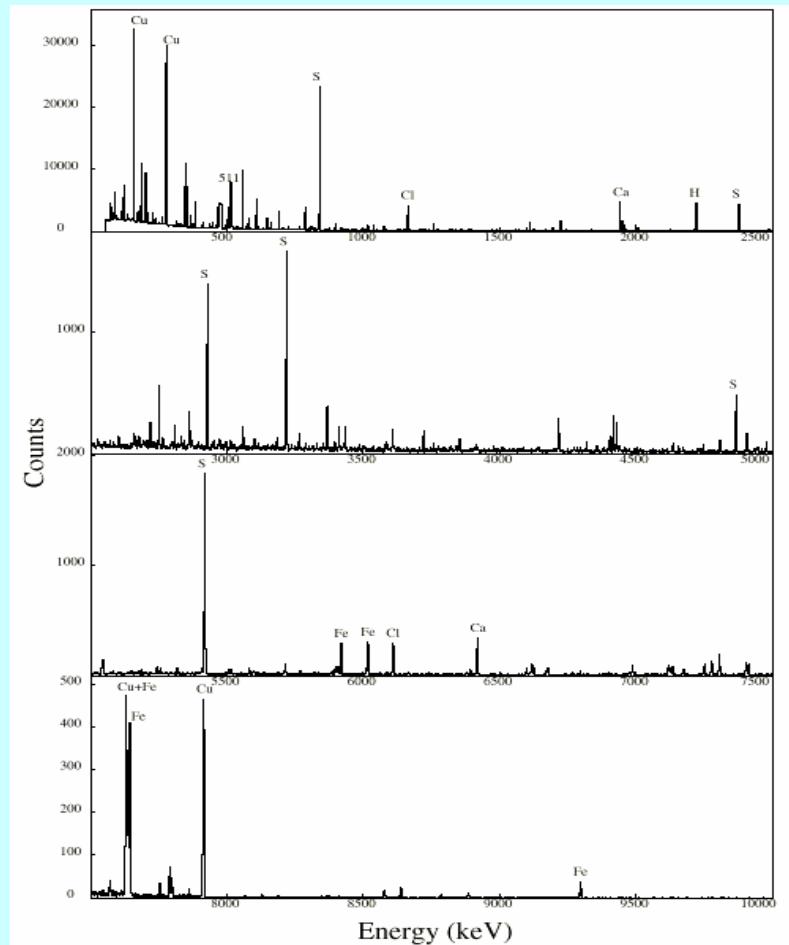
- B is a fluid-mobile trace element / conc. of 10-25 ppm
- Representative samples from Northern Pannonian Basin
- Major components were detected with high accuracy
- Systematic variation in B conc. as function of distance from subduction zone



# DEEP SEE VENTS ON THE PACIFIC FLOOR

*(Lawrence Berkeley National Laboratory)*

- Deep see vents have been found on the ocean floor near faults
- The overheated water dissolves different minerals
- The investigated samples contain sulfates of Cu and Fe



## Experimental Results With PGAA

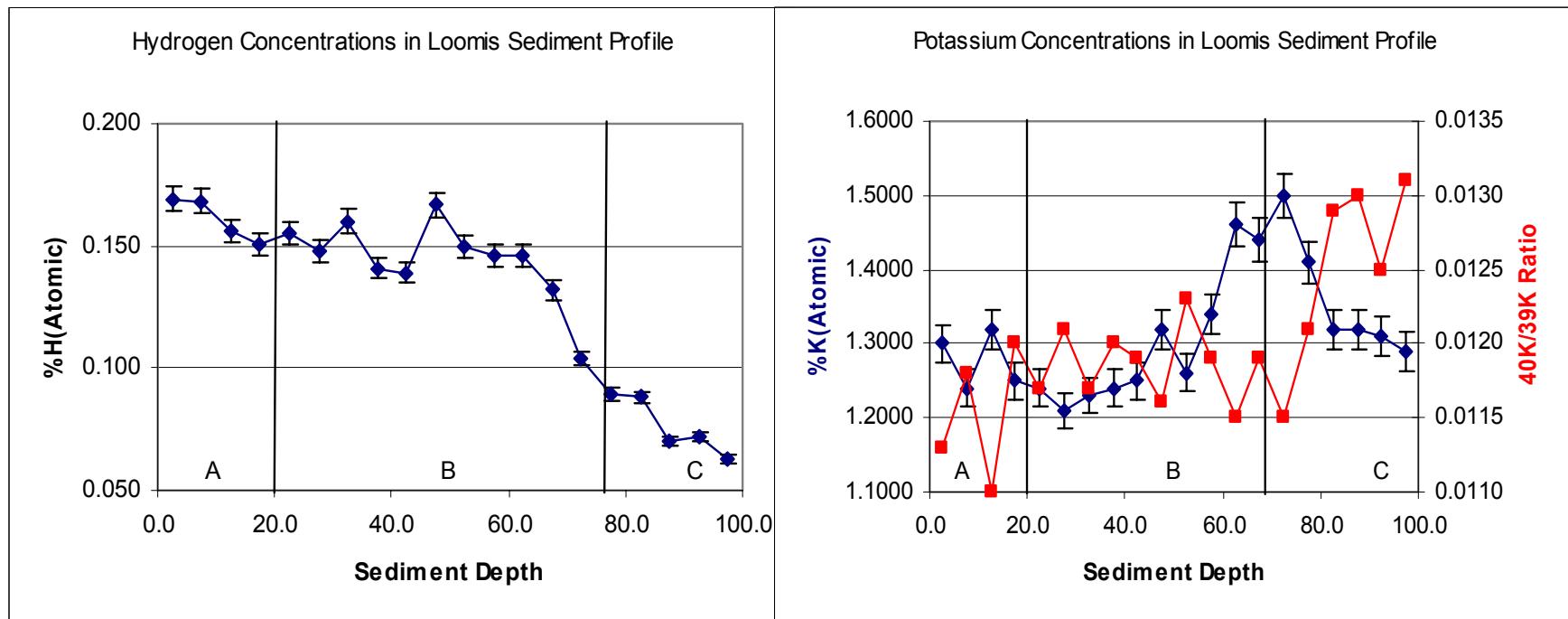
- Analysis of Deep Sea Vents (% by weight)



	ALVIN 917-R4	ALVIN 1457-1R-C	ALVIN 1461-2R
O	45.9*	41(6), 44.9*	45.1*
S	20.0 (0.2)	0.151 (0.005)	0.16 (0.01)
Ca	11.3 (0.2)	7.22 (0.11)	7.25 (0.13)
Fe	9.28 (0.11)	9.65 (0.08)	9.37 (0.09)
Cu	7.67 (0.07)	---	---
Al	---	7.10 (0.07)	7.06 (0.12)
Mg	1.8 (0.2)	3.98 (0.11)	3.6 (0.2)
Zn	1.36 (0.05)	---	---
P	---	0.85 (0.18)	1.6 (0.2)
Ni	1.17 (0.003)	0.022 (0.002)	---
Ti	---	1.097 (0.008)	1.060 (0.010)
Si	0.55 (0.05)	22.6 (0.3)	22.3 (0.3)
H	0.368 (0.004)	0.0290 (0.0005)	0.027 (0.001)
K	0.27 (0.06)	0.138 (0.004)	0.16 (0.01)
Cl	0.194 (0.002)	0.0566 (0.0005)	0.0188 (0.0005)
Mn	---	0.154 (0.002)	0.161 (0.004)
Na	0.140 (0.014)	1.97 (0.04)	1.96 (0.05)
V	---	0.042 (0.002)	0.046 (0.003)
Co	0.0066 (0.0011)	0.0045 (0.0003)	0.0058 (0.0009)
Sc	---	0.0039 (0.0002)	0.0058 (0.0005)
Cd	0.00352 (0.00005)	---	0.00024 (0.00003)
B	0.00220 (0.00002)	0.000659 (0.000007)	0.000658 (0.000008)
Dy	---	0.00099 (0.00008)	0.00111 (0.00014)
Gd	0.000050 (0.000006)	0.000524 (0.000007)	0.000556 (0.000010)
Sm	0.00033 (0.00003)	0.000330 (0.000005)	0.000340 (0.000007)

## Experimental Results With PGAA

- Analysis of a Sediment Profile from an Archaeological site



Si,H,Al,Na,K,Ca,Fe,Cl,B,Sm,Gd,  
Mn,Cu observed with PGAA

$^{40}\text{K}$ ,U,Th, $^{137}\text{Cs}$  from low-background  
counting;  $^{39}\text{K}$  from PGAA

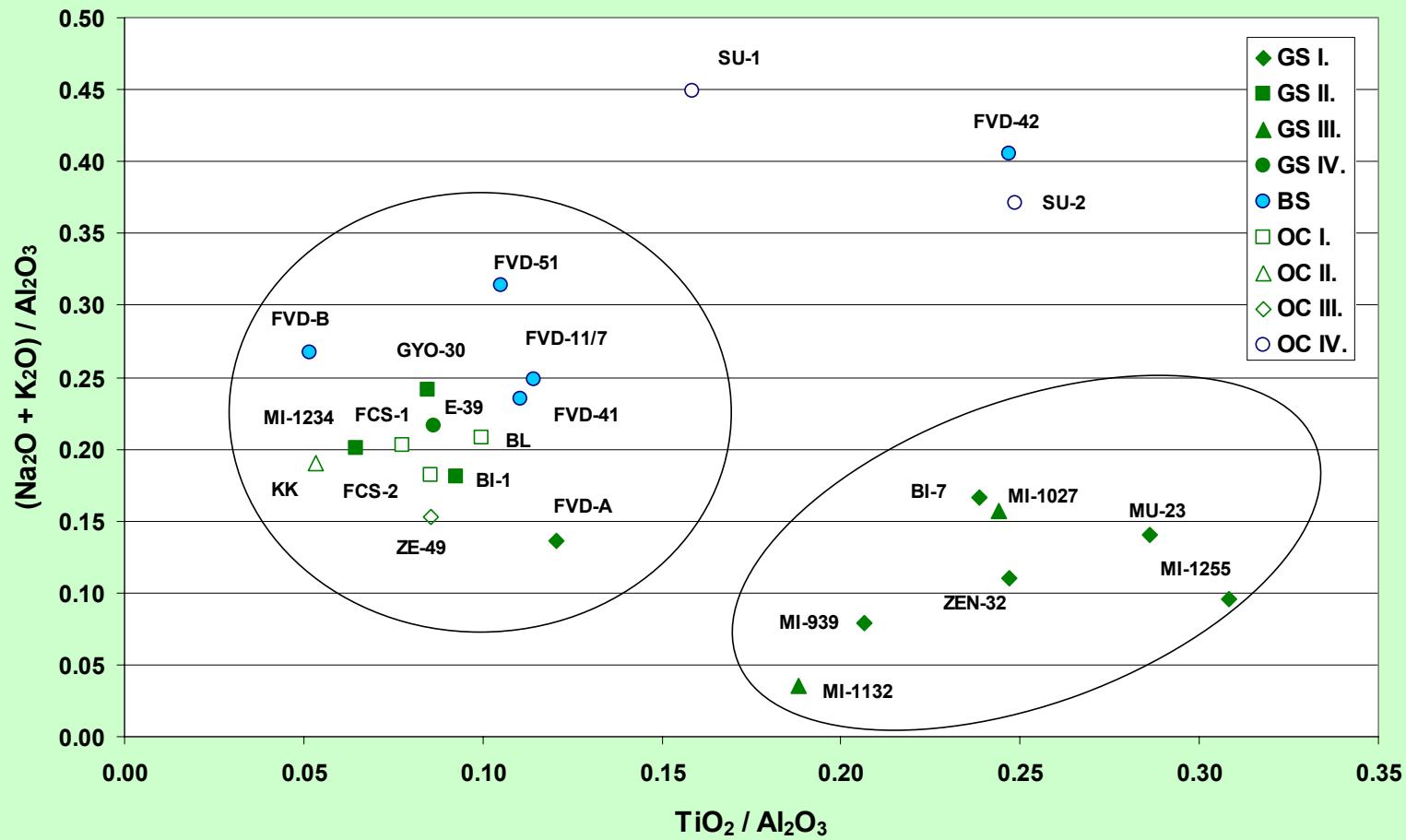
# **NEOLITHIC POLISHED STONE TOOLS - GREENSCHIST AND BLUESCHIST**

*(Dept. of Petrography, Eötvös University )*

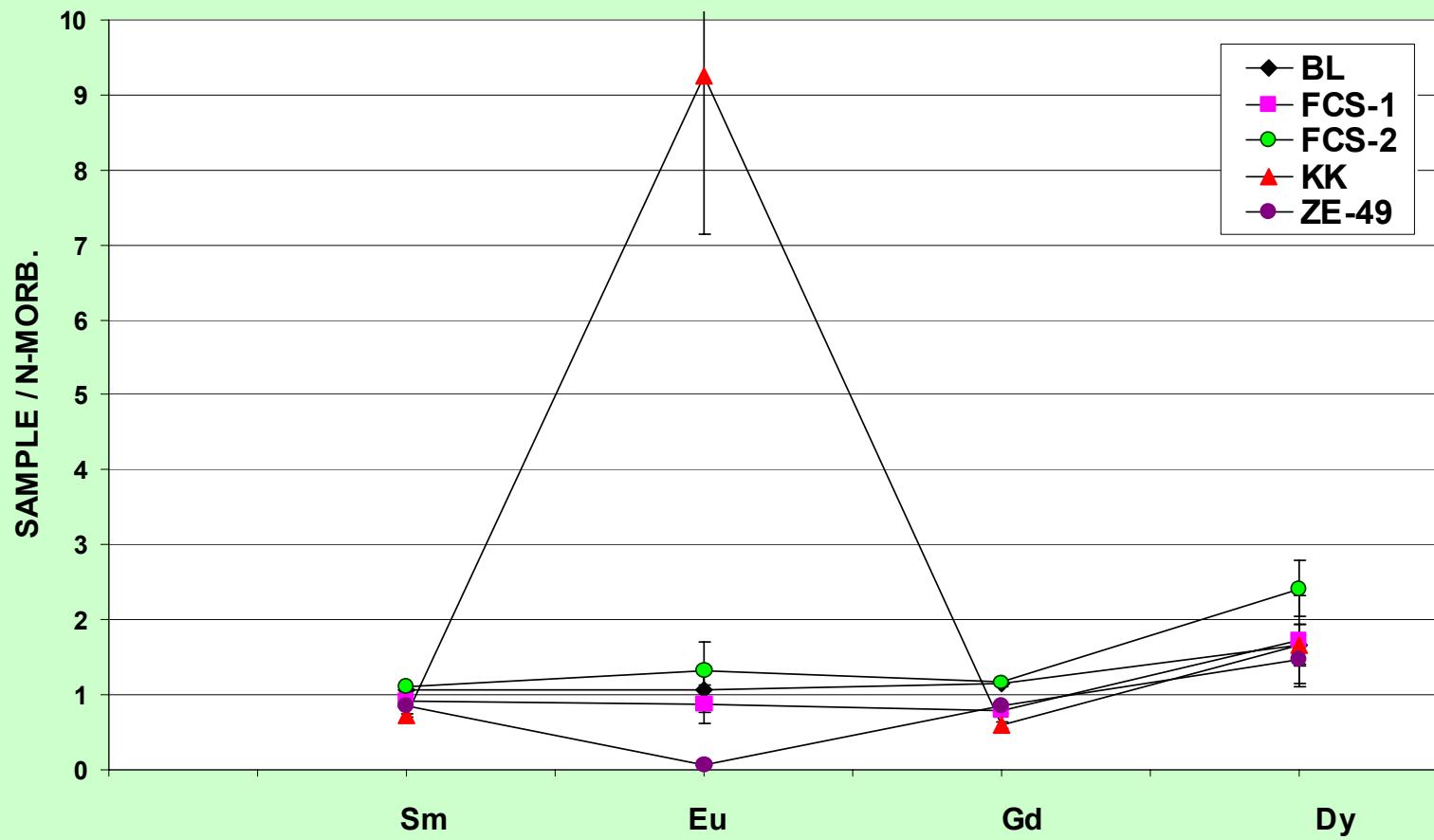


- **17 archaeological objects** from the Carpathian basin  
6-8 thousand-year-old settlements
- **7 rocks** from quarries (Eastern-Alps, Western-Carpathian)
- **Aim:** Provenance study of stone tools
- **Analysis:** Petrography, **PGAA**  
Major ( $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$   $\text{Fe}_2\text{O}_3$ ,  $\text{MnO}$ ,  $\text{CaO}$ ,  $\text{MgO}$ ,  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$ )  
and traces (**B**, **Sc**, **V**, **Co**, **Cr**, **Sm**, **Eu**, **Gd**, **Dy**)

# CLASSIFICATION OF GREENSCHIST AND BLUESCHIST OBJECTS



# REE-PATTERN OF SAMPLES FROM QUARRIES (Eu-ANOMALY)



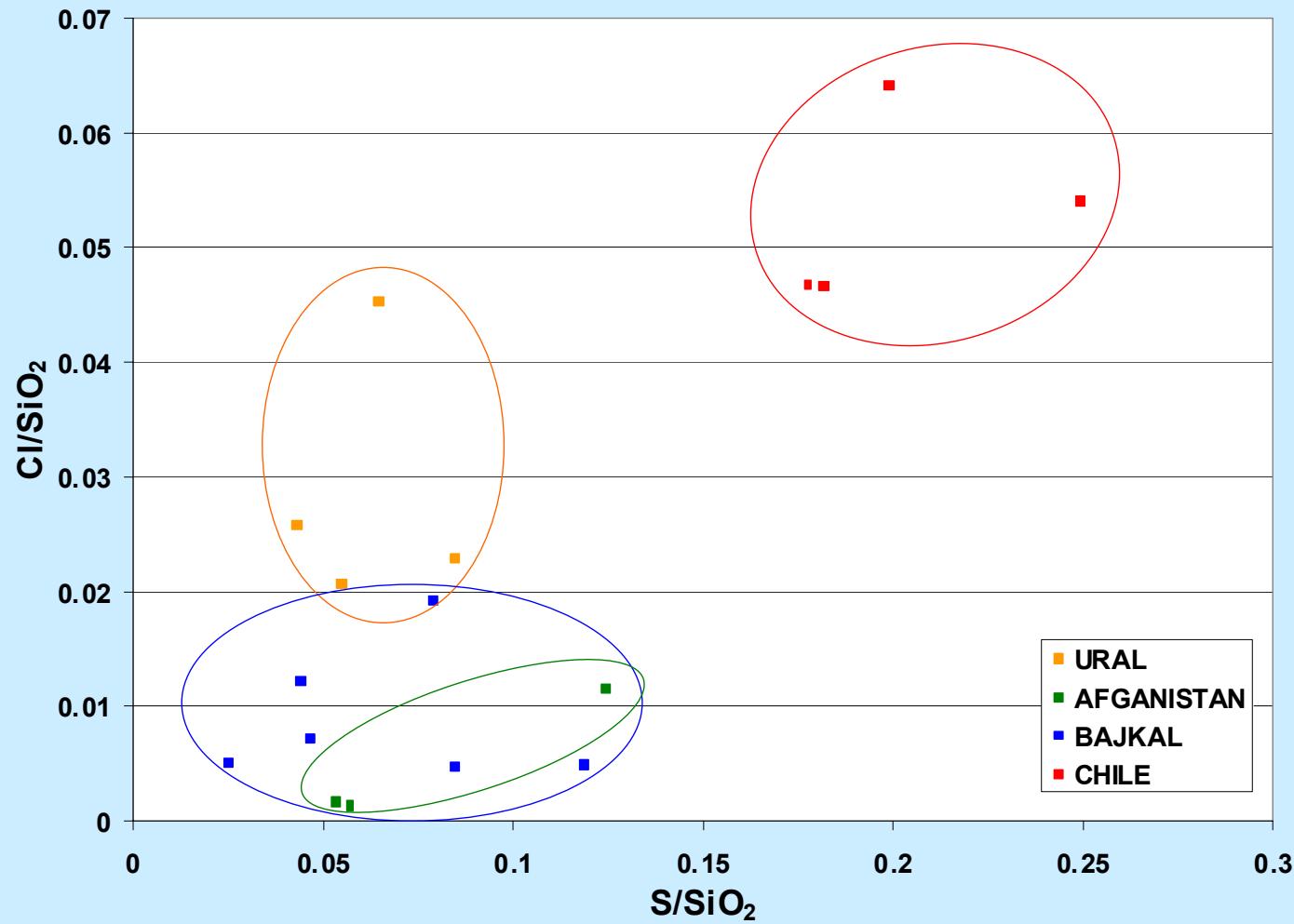
# LAPIS-LAZULI

*(University of Tübingen)*



- Semi-precious stone
- Few quarries known over the World  
(Ural, Chile, Afghanistan, Lake Baikal)
- Mineral: Lazurite /  $(\text{Na}, \text{Ca})_{7-8}(\text{Al}, \text{Si})_{12}\text{O}_{24}[(\text{SO}_4)\text{Cl}_2(\text{OH})_2]$
- PGAA: H, Na, Mg, Al, Si, K, Ca, Ti, Mn, Fe, S, Cl

# CLASSIFICATION OF LAPIS-LAZULI RAW MATERIAL



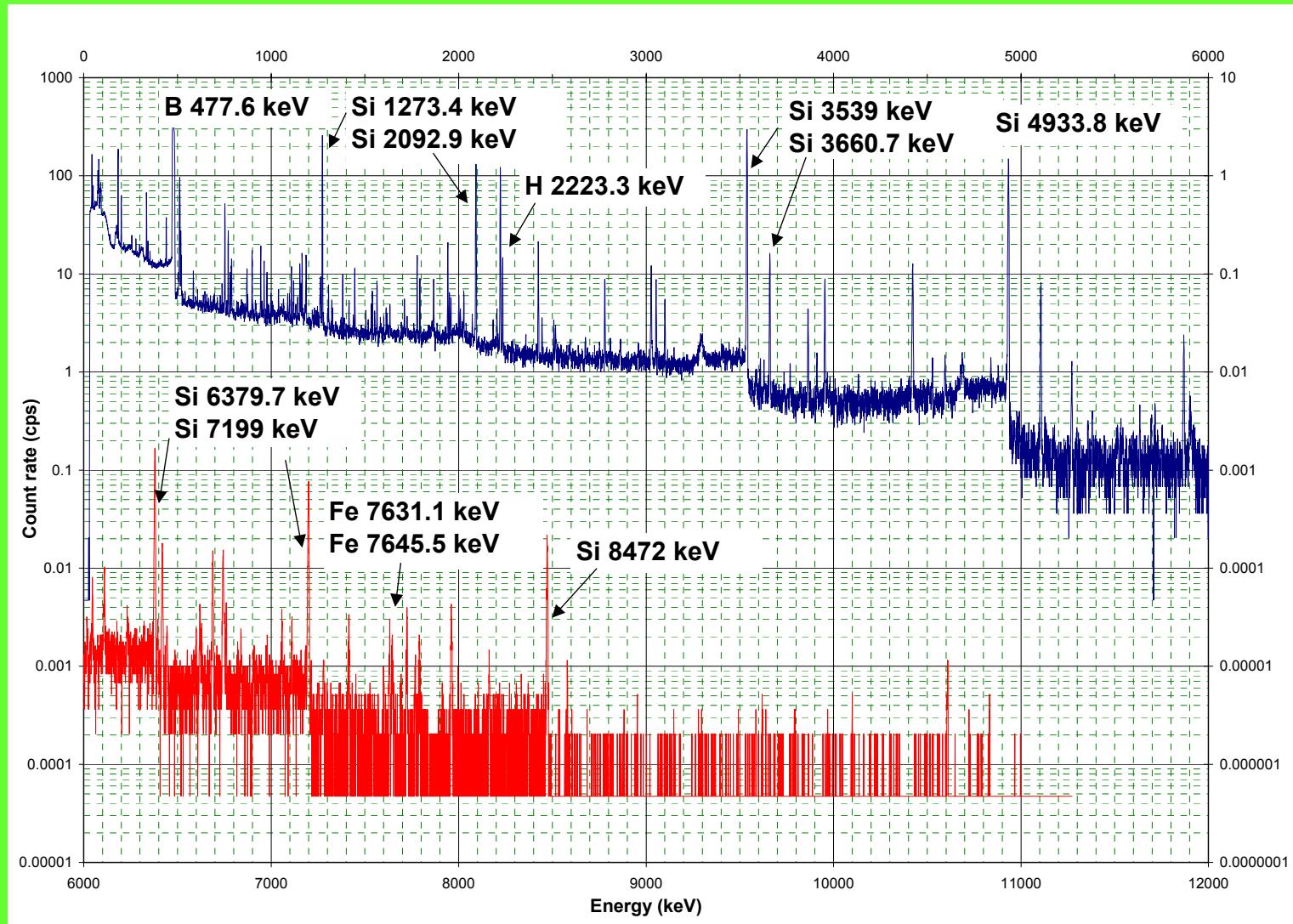
# **CHIPPED STONE TOOLS MADE OF GREY FLINT**

*(Hungarian National Museum)*

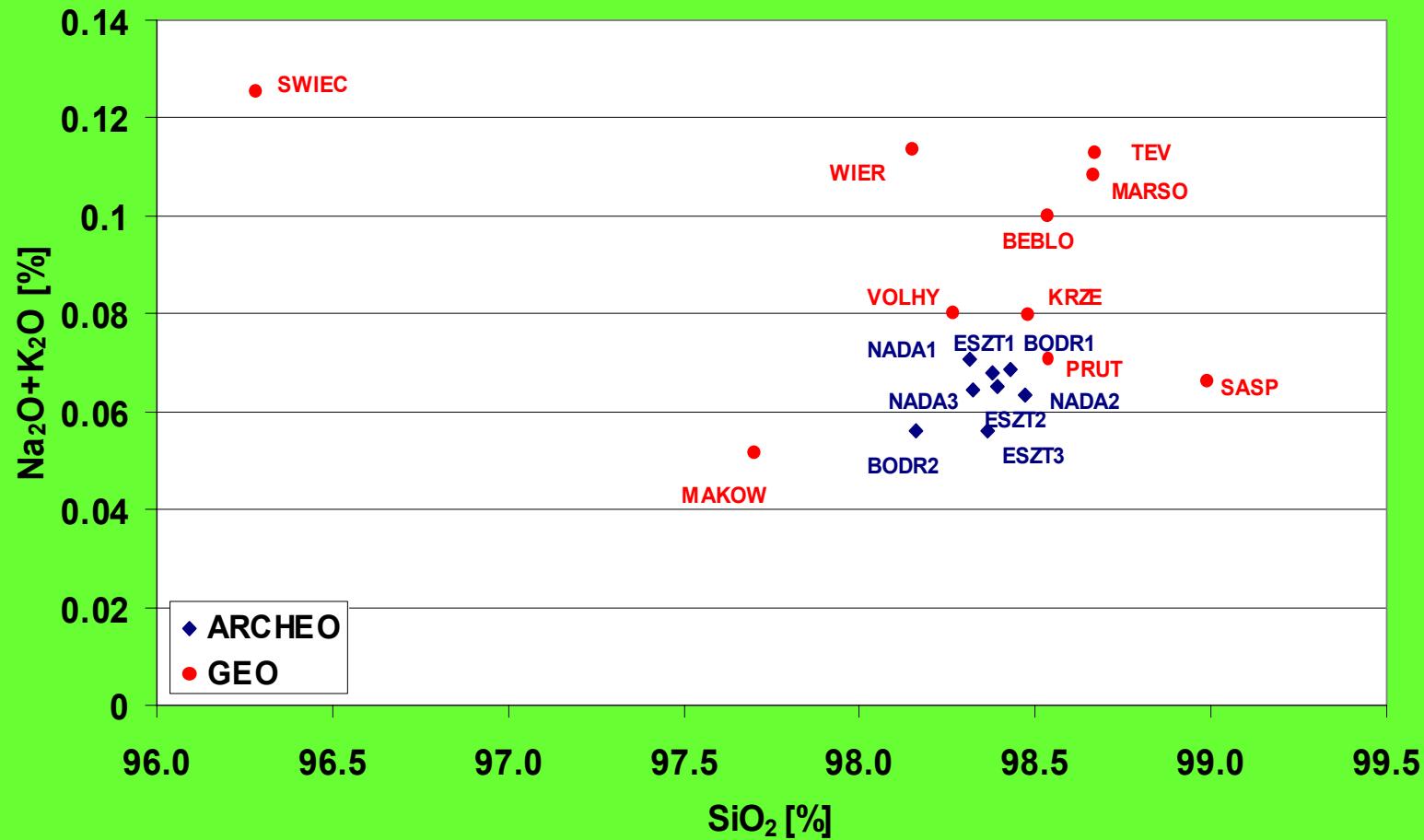


- **AIM:** Provenance study of 8 archaeological objects from the Carpathian basin (Upper Paleolithic - **16-30 thousand years** )
- **GEOLOGICAL REFERENCE:** Lithotheca of the HNM (Quarries of Poland, Czech, Hungary, Ukraine, Romania)
- **ANALYTICS:** >95% **SiO<sub>2</sub>**; Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, CaO, Na<sub>2</sub>O, K<sub>2</sub>O, MgO, MnO, H<sub>2</sub>O, Cl, B, Sm, Eu, Gd, Sc, Cd

# PGAA spectrum of a grey flintstone



# CLASSIFICATION OF GREY FLINT SAMPLES



# CERAMICS



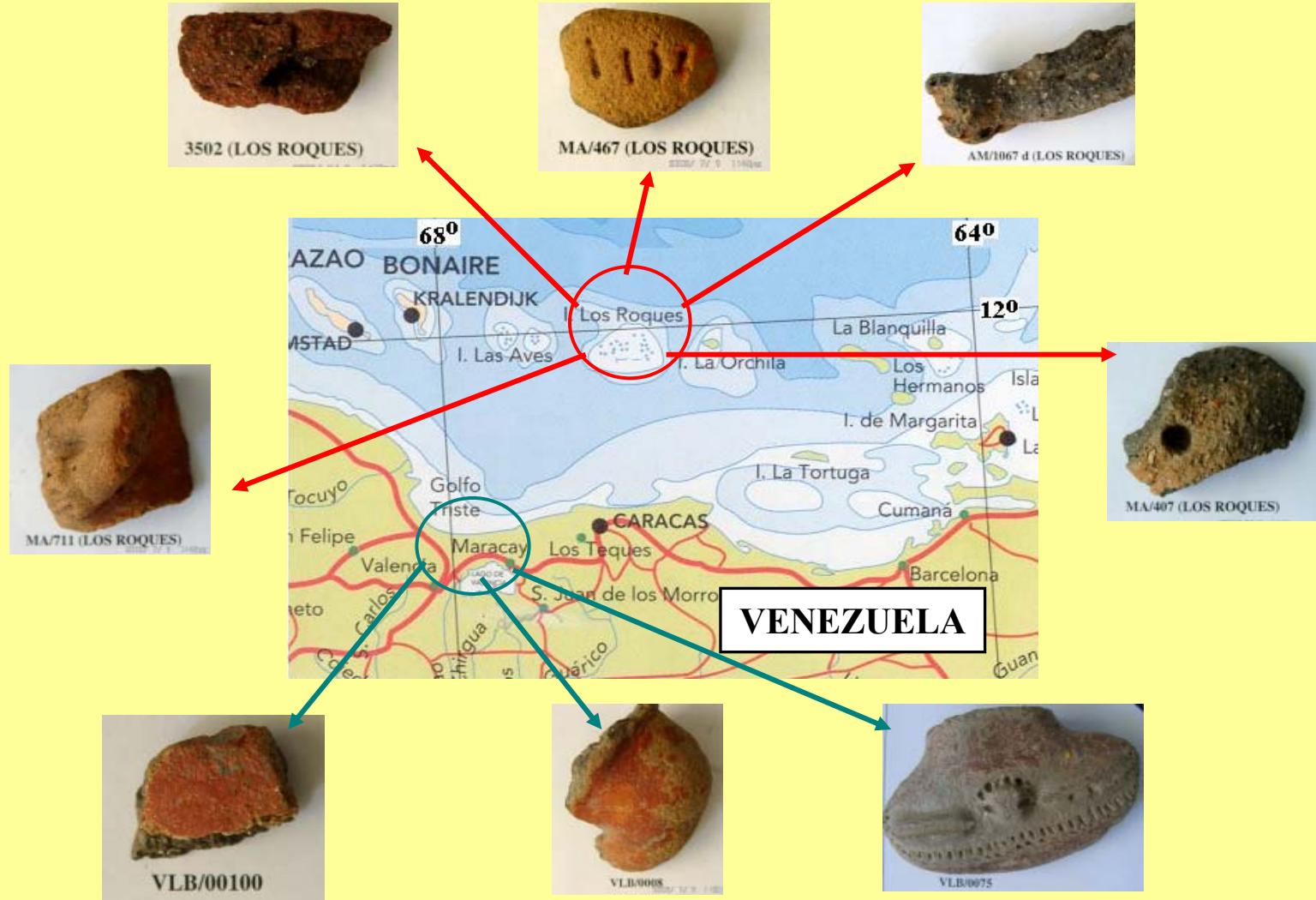
# PRE-COLUMBIAN POTTERY FROM VENEZUELA

(*Simón Bolívar University, Caracas*)

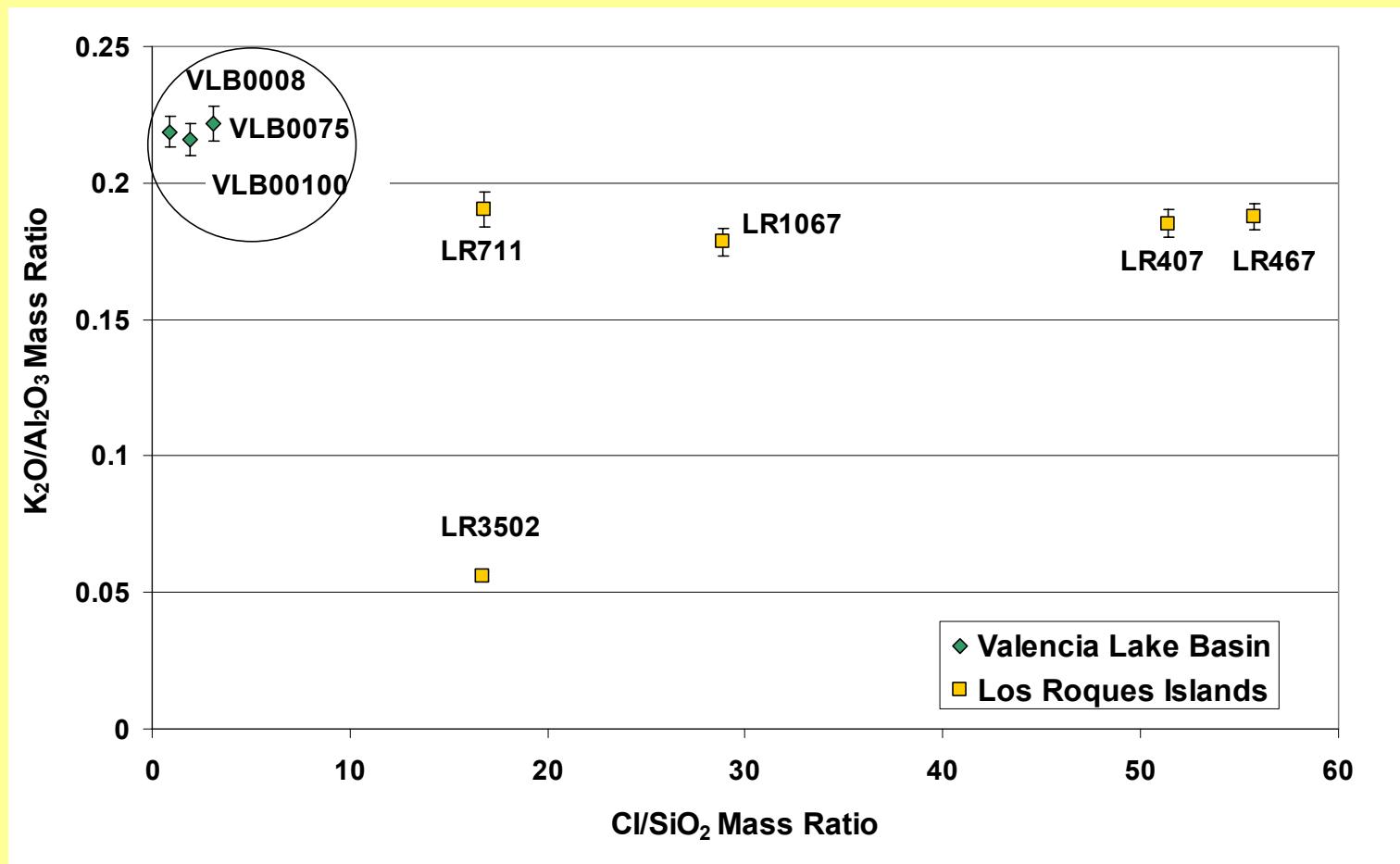


- Fragments of pottery figurines from 12<sup>nd</sup>-15<sup>th</sup> Century
- **Provenance:** Valencia Lake Basin and the Los Roques Islands
- **Question:** Did the occupants use the same raw material?
- **Analytic:** Major and trace components  
(H, Na, Mg, Al, Si, K, Ca, Ti, Mn, Fe  
B, S, Cl, Sc, V, Cr, Ba, Sm, Eu, Gd, Dy)

# PROVENANCE OF VENEZUELAN POTTERY



# CLASSIFICATION OF VENEZUELAN POTTERY



# GLASS



# HISTORICAL GLASS

(Inst. of Nuclear Chemistry and Technology, Warsaw)

WHITE	Conc. [%]		BLUE	Conc. [%]		GREEN	Conc. [%]
$\text{SiO}_2$	59.7		$\text{SiO}_2$	71.8		$\text{SiO}_2$	71.2
$\text{K}_2\text{O}$	8.33		$\text{K}_2\text{O}$	17.6		$\text{K}_2\text{O}$	17.4
$\text{PbO}$	25.1		$\text{PbO}$	<2		$\text{PbO}$	3.1
$\text{CaO}$	1.3		$\text{CaO}$	2.98		$\text{CaO}$	2.27
$\text{Al}_2\text{O}_3$	0.58		$\text{Al}_2\text{O}_3$	<0.3		$\text{Al}_2\text{O}_3$	<0.3
$\text{Na}_2\text{O}$	3.1		$\text{Na}_2\text{O}$	1.79		$\text{Na}_2\text{O}$	1.12
$\text{MgO}$	0.5		$\text{MgO}$	<0.2		$\text{MgO}$	<0.2
$\text{P}_2\text{O}_5$	<2		$\text{P}_2\text{O}_5$	2.16		$\text{P}_2\text{O}_5$	<2
$\text{MnO}$	0.07		$\text{MnO}$	0.018		$\text{MnO}$	0.030
$\text{Fe}_2\text{O}_3$	0.081		$\text{Fe}_2\text{O}_3$	<0.05		$\text{Fe}_2\text{O}_3$	<0.05
$\text{CuO}$	<0.1		$\text{CuO}$	0.83		$\text{CuO}$	0.77
$\text{TiO}_2$	0.037		$\text{TiO}_2$	<0.01		$\text{TiO}_2$	<0.01
B	0.00137		B	0.00296		B	0.00570



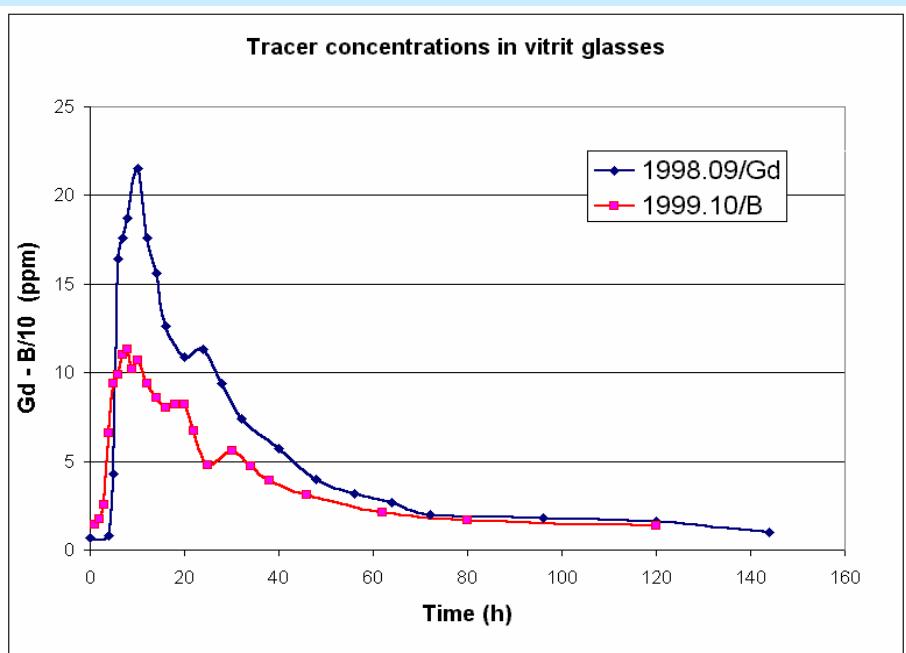
Good agreement  
with EPMA !

- W: High Pb, colourless K-glass / Piece of goblet from 17<sup>th</sup> C., England
- B and G: K-glass, colourants are Cu (U), opaque from  $\text{P}_2\text{O}_5$  Wastes from 19<sup>th</sup> C. Silesian glasswork

# INACTIVE TRACING OF A GLASS FURNACE

(*GE Lighting Tungsram*)

- Homogenization and flow properties of an industrial melting furnace were investigated
- To avoid high level radioactivity, inactive tracers of  $\text{Gd}_2\text{O}_3$  and  $\text{H}_3\text{BO}_3$  were added in 10 ppm concentration
- Samples were taken regularly at the outlet and measured with PGAA
- Properties were found to be close to ideal case



# METALS



# ROMAN BRONZE FIBULAE AND COINS

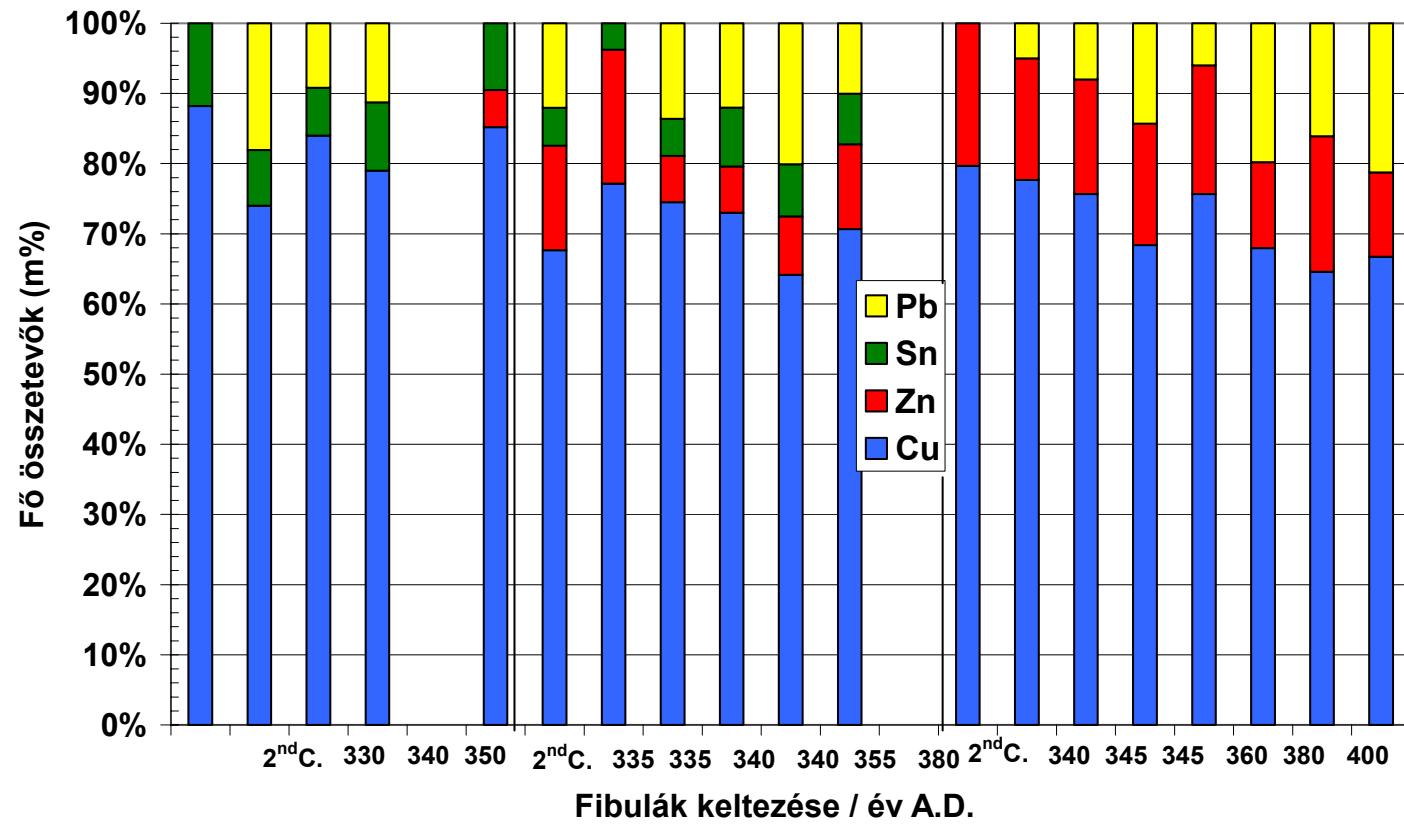
*(Archaeological Institute of the HAS)*



- Cemetery of Hegyeshalom (H): 2<sup>nd</sup> and 4<sup>th</sup> century AD  
Excavated objects: **26 fibulae and 383 coins**
- The graves can be dated with a 10-20 years accuracy  
The inner chronology of fibulae can not be determined according to typology
- **Aim:** Classification of objects, information on workshop, trade, etc. - according to composition
- **Problem:** Recycling of raw material



# CLASSIFICATION OF FIBULAE BASED ON MAJOR COMPONENTS



# LARGE ROMAN BRONZE OBJECTS

*(University of Bielefeld, Hungarian National Museum)*



Most of them are pure tin-bronze (Cu:Sn ratio 9:1), some with high Pb or Zn

With a collimated beam major components of any parts of jugs, helmets, shields, etc. can be determined



# **SOUTH-ITALIAN BRONZE OBJECTS (I-II. C.)**

*(University of Bielefeld, Hungarian National Museum)*



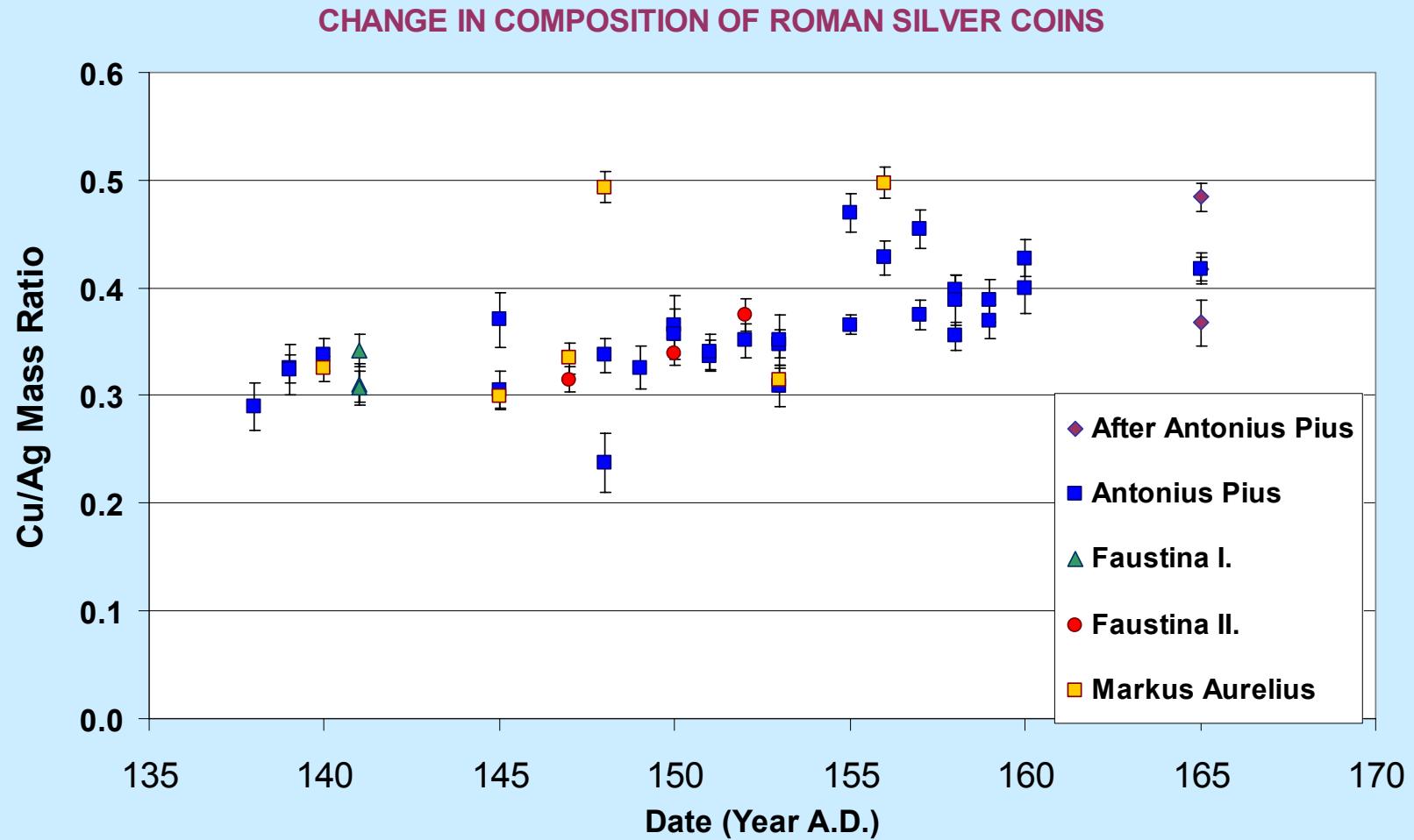
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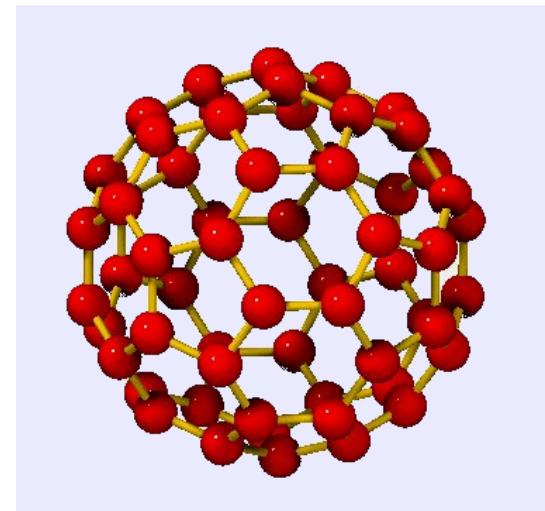
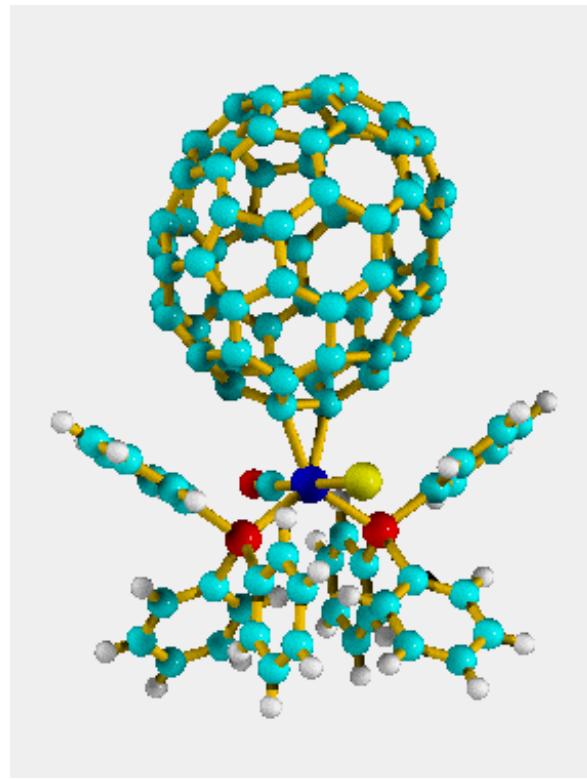
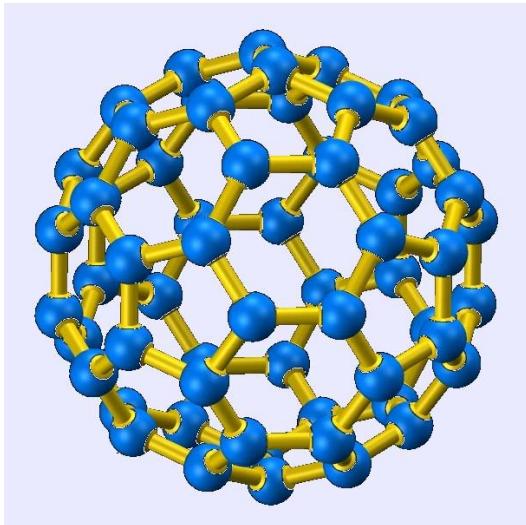


# ROMAN SILVER COINS

(Inst. of Nuclear Chemistry and Technology, Warsaw)



# CHEMISTRY



## Experimental Results With PGAA

- Analysis of Reagent Materials (LBNL + Budapest)

$\text{CaF}_2$  (Reagent grade, Baker and Adamson)

Ca	$54.3 \pm 0.9\%$
F	$44.4 \pm 1.9\%$
Al	$0.66 \pm 0.07\%$
Cl	$0.150 \pm 0.003\%$
Na	$0.040 \pm 0.009\%$

$\text{ZnO}$  (Mallinckrodt)

Zn	100%
Cd	$5.1 \pm 0.3$ ppm

$\text{HfO}_2$  (98%, Aldrich Chemical Co.)

$\text{HfO}_2$  (no detectable impurities)

$\text{TiO}_2$

Ti	96.8%
K	$3.1 \pm 0.2\%$
H	$0.04 \pm 0.01\%$
Eu	$0.022 \pm 0.002\%$
Gd	$5.2 \pm 0.2$ ppm
B	$2.0 \pm 0.2$ ppm

$\text{Gd}_2\text{O}_3$  – no detectable impurities

$\text{Ca(OH)}_2$

Ca	$93.6 \pm 1.9\%$
H	$5.04 \pm 0.005\%$
K	$1.3 \pm 0.2\%$
Cl	$0.028 \pm 0.002$
Eu	$54 \pm 9$ ppm
Gd	$26 \pm 1$ ppm
B	$4.2 \pm 0.3$ ppm

$\text{CeO}_2$

Ce	99.8%
K	$0.077 \pm 0.016\%$
S	$0.074 \pm 0.010\%$
Na	$0.061 \pm 0.009\%$
H	$0.004 \pm 0.001\%$
B	$31 \pm 1$ ppm
Eu	$11 \pm 1$ ppm
Sm	$1.8 \pm 0.1$ ppm
Gd	$1.2 \pm 0.1$ ppm

# SUPPORTED Pt CATALYSTS

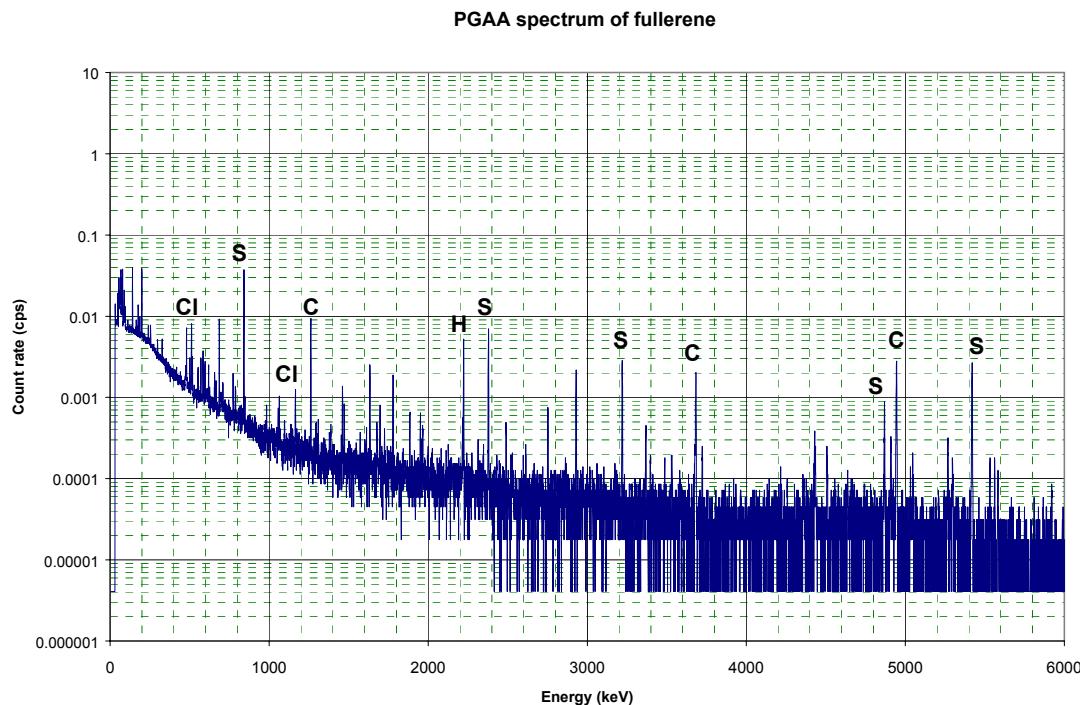
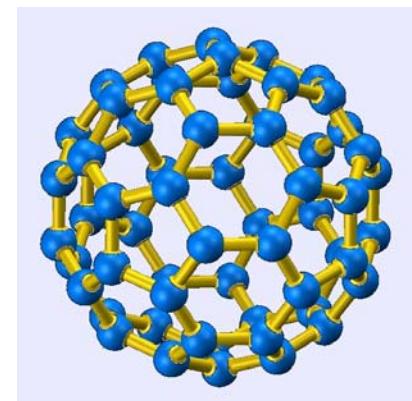
- Non-destructive test of catalysts containing noble metals has a great economical importance
- With PGAA metal content and H was also detectable
- For EUROPT catalysts the PGAA results are in good agreement with other methods

	m/m (%)		mol %	
H	1.16	±	0.025	19.01
Al	46.40	±	0.696	28.27
Cl	1.06	±	0.009	0.49
Pt	0.34	±	$8.8 \cdot 10^{-3}$	0.03
O*	50.66	±	1.840	52.09

Composition of an industrial Pt/Al<sub>2</sub>O<sub>3</sub> catalyst

# SULFUR IN FULLERENE

(Dept. of Inorganic Chemistry, Eötvös University)

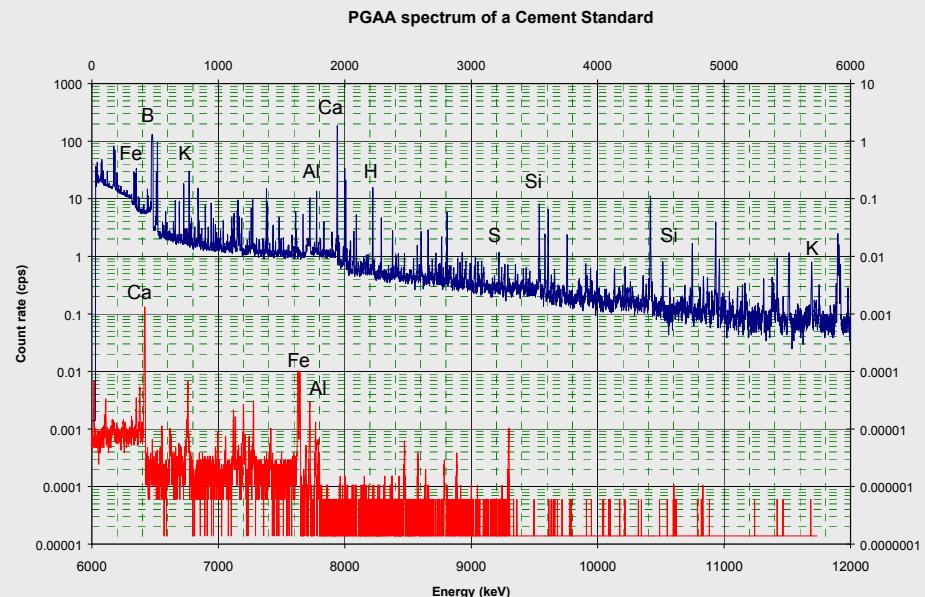


- Sulfur and other impurities were determined with PGAA
- C is an ideal matrix
- S attributed to  $\text{C}_{60}\text{S}_{16}$  (clathrate) during the purification process

# CEMENT AND CLINKER

## (Cemkut Ltd.)

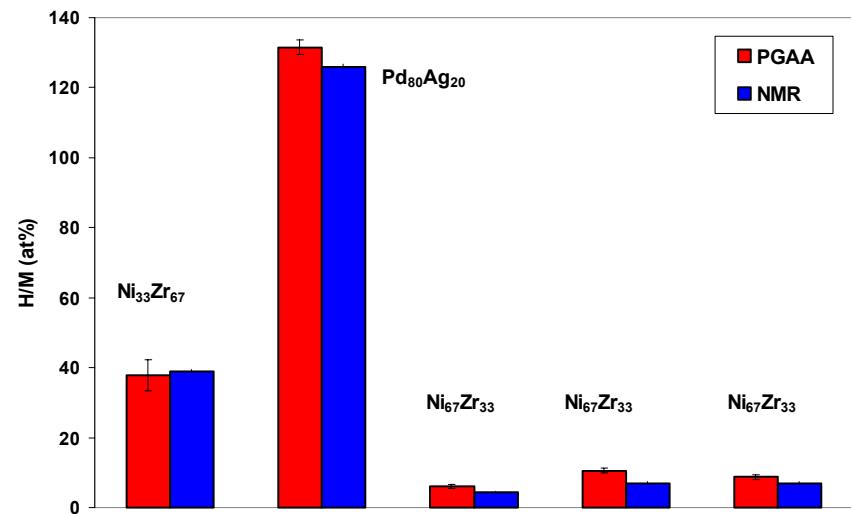
- Major components of industrial cements and clinker were measured
- PGAA gives high accuracy data of Ca, Si, Al and Fe oxides
- The analysis is much shorter (2-5 hours) than the classical quality control



# HIDROGEN IN AMORPHOUS ALLOYS

(Res. Inst. for Solid State Physics, KFKI)

- NiZr and PdAg alloys for H storage cells
- Storage capacity and H dynamics with NMR and PGAA
- PGAA detects significantly higher H than NMR



# **NUCLEAR MATERIALS**

# $^{99}\text{Tc}$ measurements

✓ Tc  
✓ Tc

✓  $\text{NH}_4\text{TcO}_4$

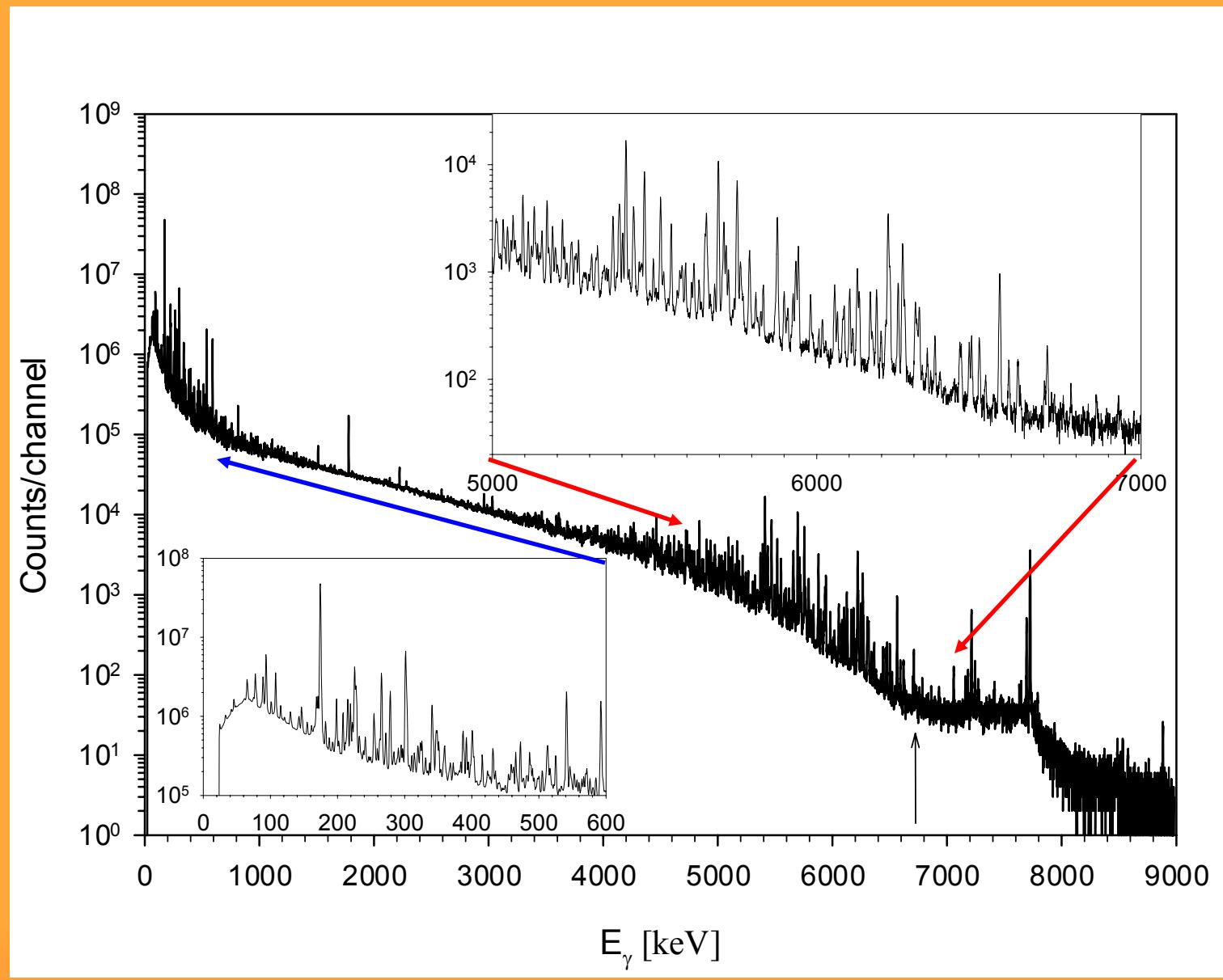
In progress:

Tc

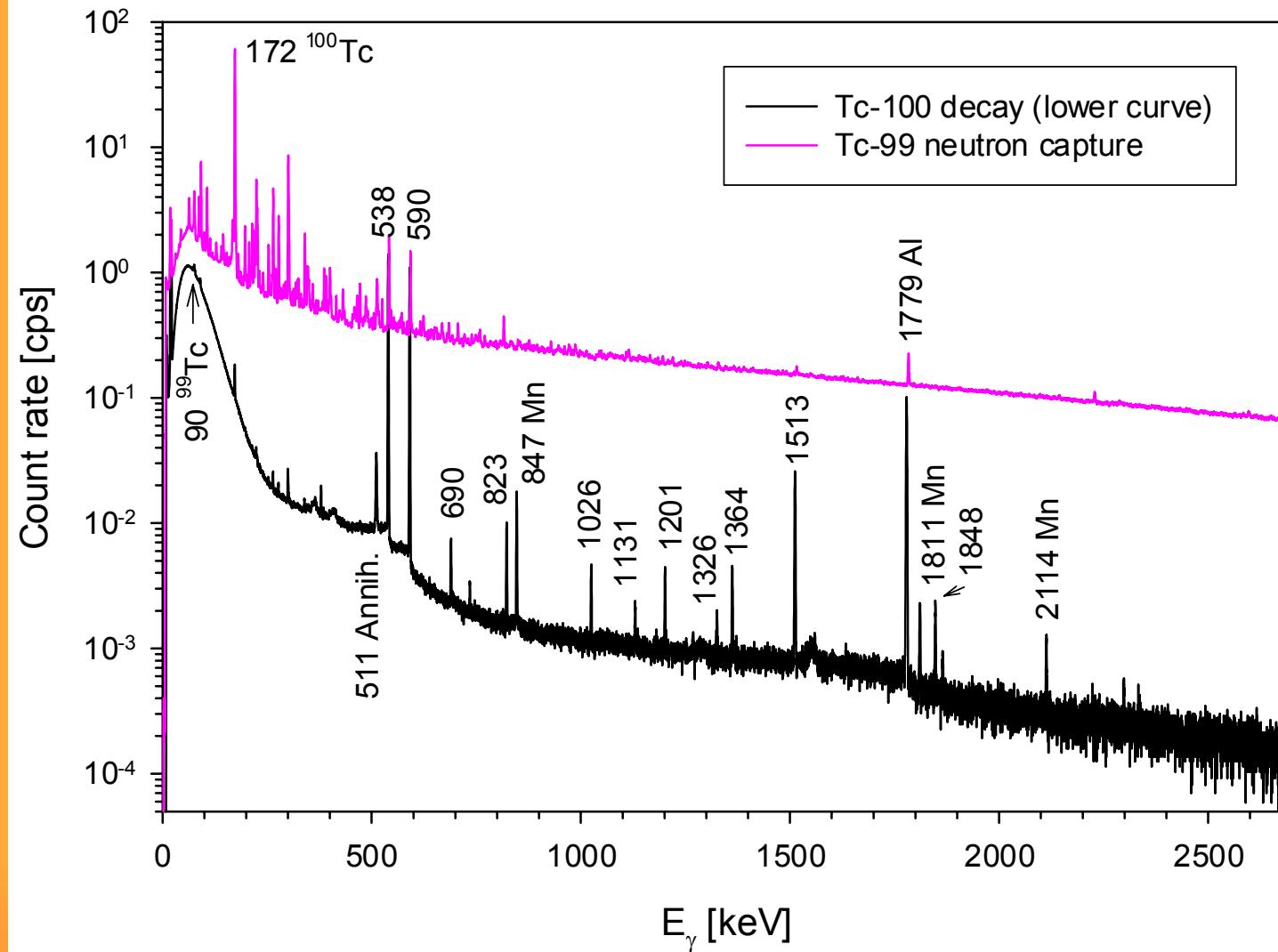
In-beam PGAA: rel.  $\gamma$  intensities  
Chopped beam PGAA:  $^{100}\text{Tc}$   $\beta^-$  decay  
    > rel.  $\gamma$  intensities  
capture and decay lines: partial xsecs  
    > capture cross section

Coincidence: level scheme  
    > capture cross section

# $^{99}\text{Tc}$ 0.5 g sample ( $n,\gamma$ ) spectrum



# $^{99}\text{Tc}$ chopped beam ( $n,\gamma$ ) spectra



# Partial $\gamma$ -ray production cross sections of capture and decay lines for a $^{99}\text{Tc}$ target

$E_\gamma$ (keV)	Origin	$P_\gamma$ ( $\gamma$ /100 captures or decays)	$\sigma_\gamma$ <sup>a</sup> (b)	Sensitivity (cps/mg)
172.1	$^{99}\text{Tc}(n,\gamma)$	$67 \pm 6$	$16.61 \pm 0.15$	3.0
223.4	$^{99}\text{Tc}(n,\gamma)$	$6.1 \pm 0.6$	$1.472 \pm 0.013$	0.24
263.5	$^{99}\text{Tc}(n,\gamma)$	$5.9 \pm 0.5$	$1.425 \pm 0.012$	0.21
539.5	$^{100}\text{Tc} \beta^-$	$6.6 \pm 0.5$ <sup>b</sup>	$1.604 \pm 0.014$	0.14
590.7	$^{100}\text{Tc} \beta^-$	$5.3 \pm 0.5$	$1.296 \pm 0.011$	0.10
89.5	$^{99}\text{Tc} \beta^-$	$(6.5 \pm 1.5) \times 10^{-4}$ <sup>c</sup>		$4.3 \times 10^{-3}$

# Inferred total thermal neutron capture cross section of $^{99}\text{Tc}$

Method	Basis	$\sigma$	Comment
		(b)	
$^{100}\text{Tc} \beta^-$	539 $\gamma$	$24.7 \pm 2.3$	with $P_\gamma$ from Ref. [9]
	591 $\gamma$	$23.9 \pm 1.8$	
	<i>Average</i>	$24.3 \pm 2.2$	unweighted average
$^{99}\text{Tc}(n,\gamma)$	$\Sigma \sigma_\gamma$ g.s.	$21.21 \pm 0.17$	lower limit
		$26.5 \pm 2.6$	missing $\sim 20\%$ added

Literature: EXFOR database       $19 \pm 2$  b  
                                          $24 \pm 4$  b  
                                          $22.9 \pm 2.6$  b

Harada:

# Investigation of fissile materials

- Passive techniques
  - from radioactive radiation of the isotopes
- Active techniques (irradiation with neutrons)
  - NAA
  - PGAA
  - chopped beam PGAA
  - other neutron irradiation techniques

# PGAA / NAA of uranium

## Prompt lines

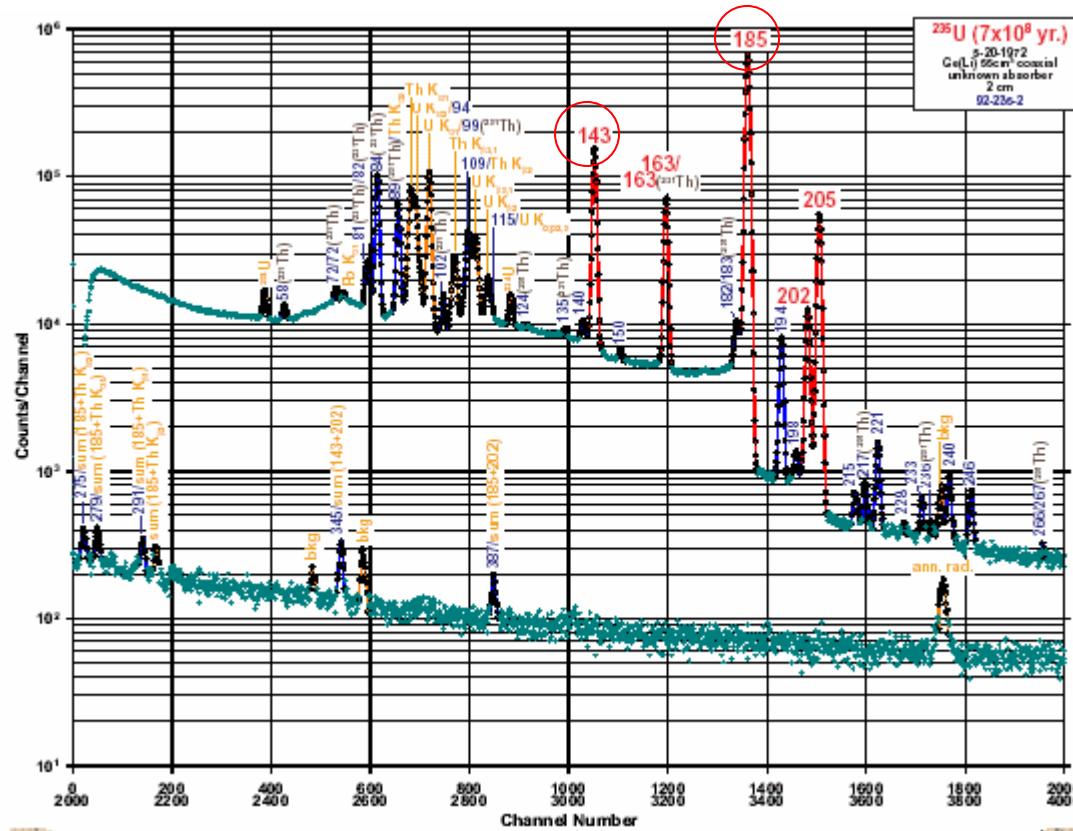
- $^{238}\text{U}$
- $^{235}\text{U}$
- fission prod.  $^{134}\text{Te}$

## Decay lines

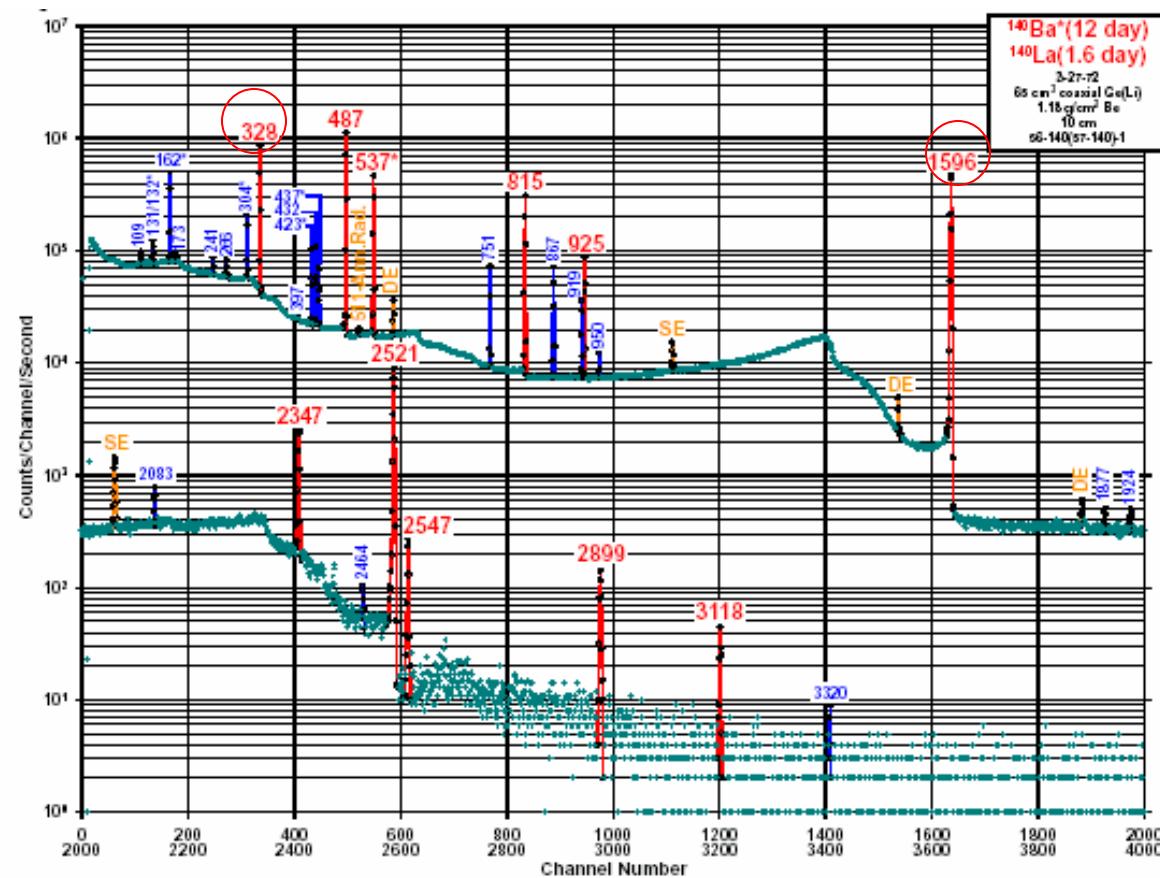
- $^{239}\text{Np}, ^{239}\text{Pu}$
- fission prod.  $^{90}\text{Rb}$  (100 s)
- fission prod.  $^{140}\text{Ba}/^{140}\text{La}$ )

**Natural radioactivity lines:**  $^{235}\text{U}$ ,  $^{238}\text{U}$

# Fissionable isotope U-235 decay spectrum - INEEL Catalog

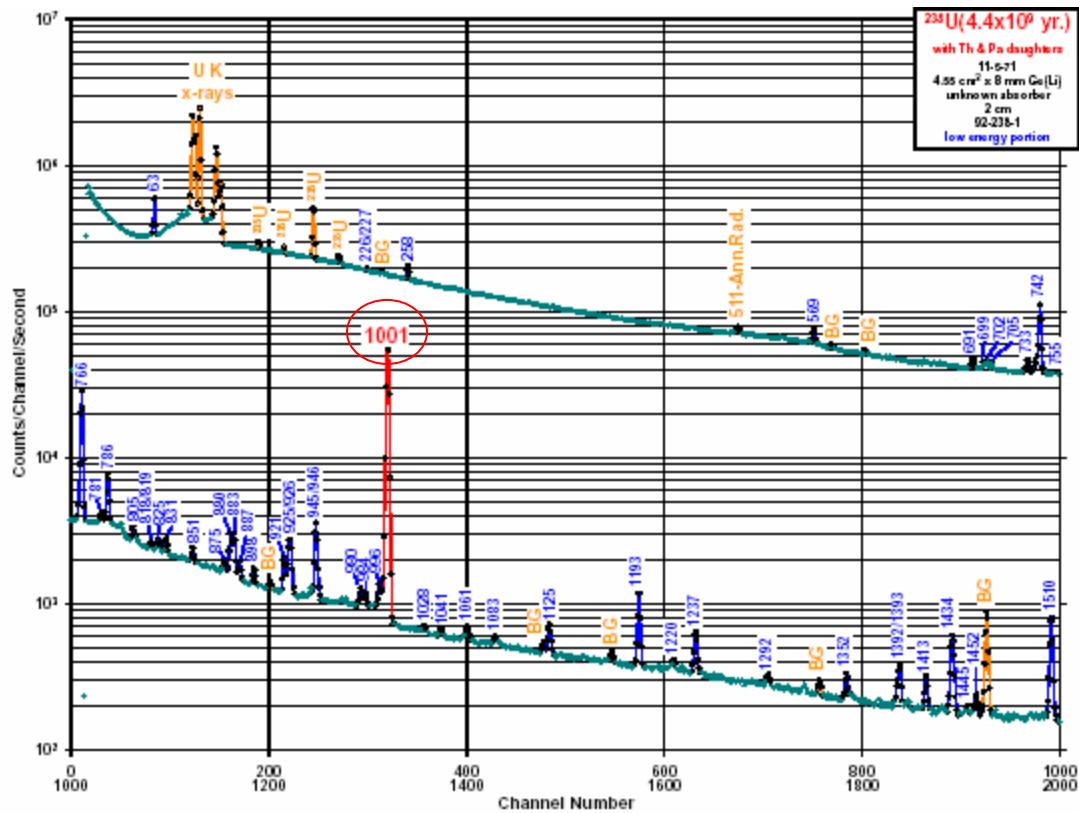


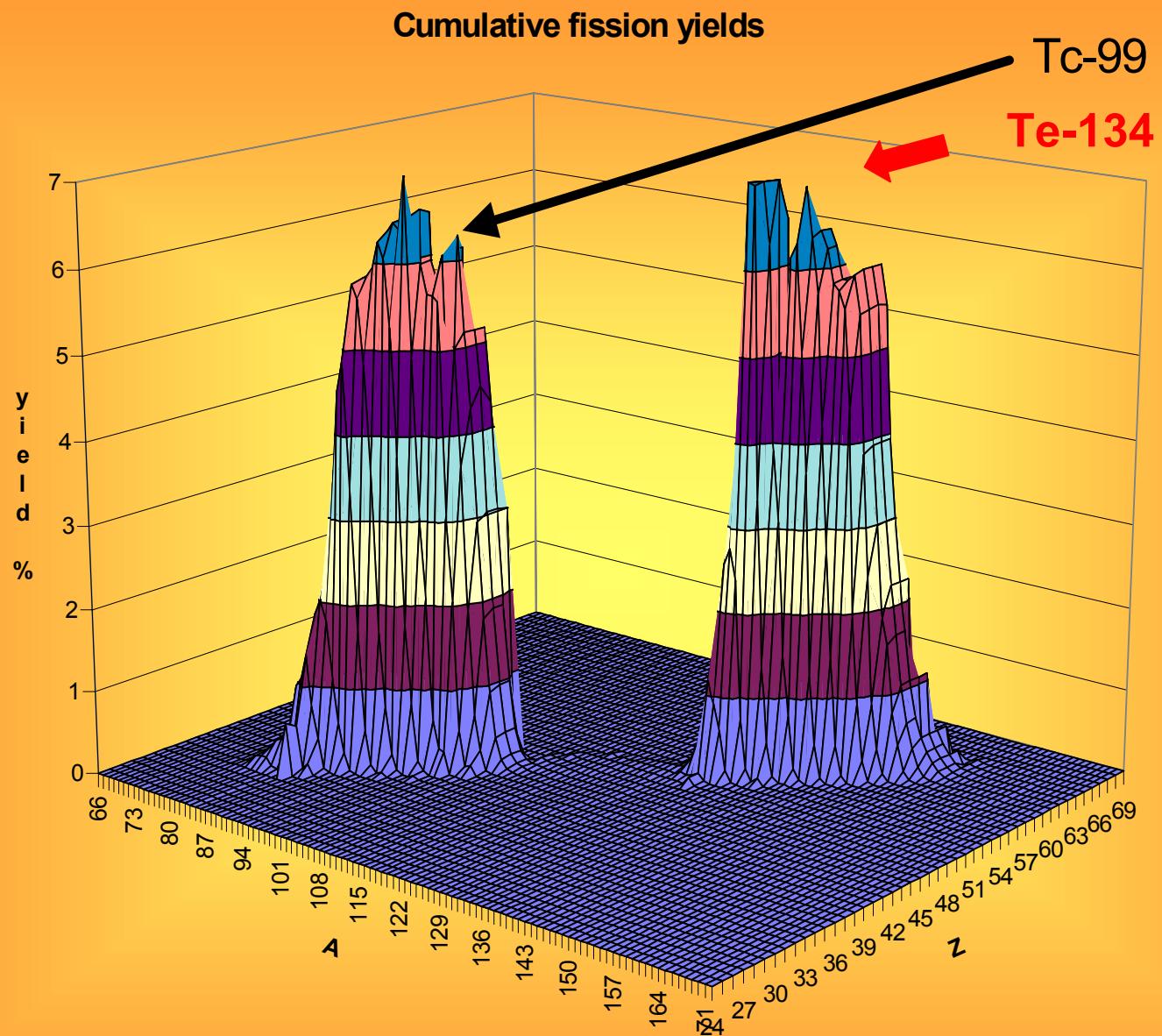
# **U-235** spont. fission > Ba-140 / La-140 spectrum - INEEL Catalog



# Non-fissionable isotope

## U-238 decay spectrum - INEEL Catalog





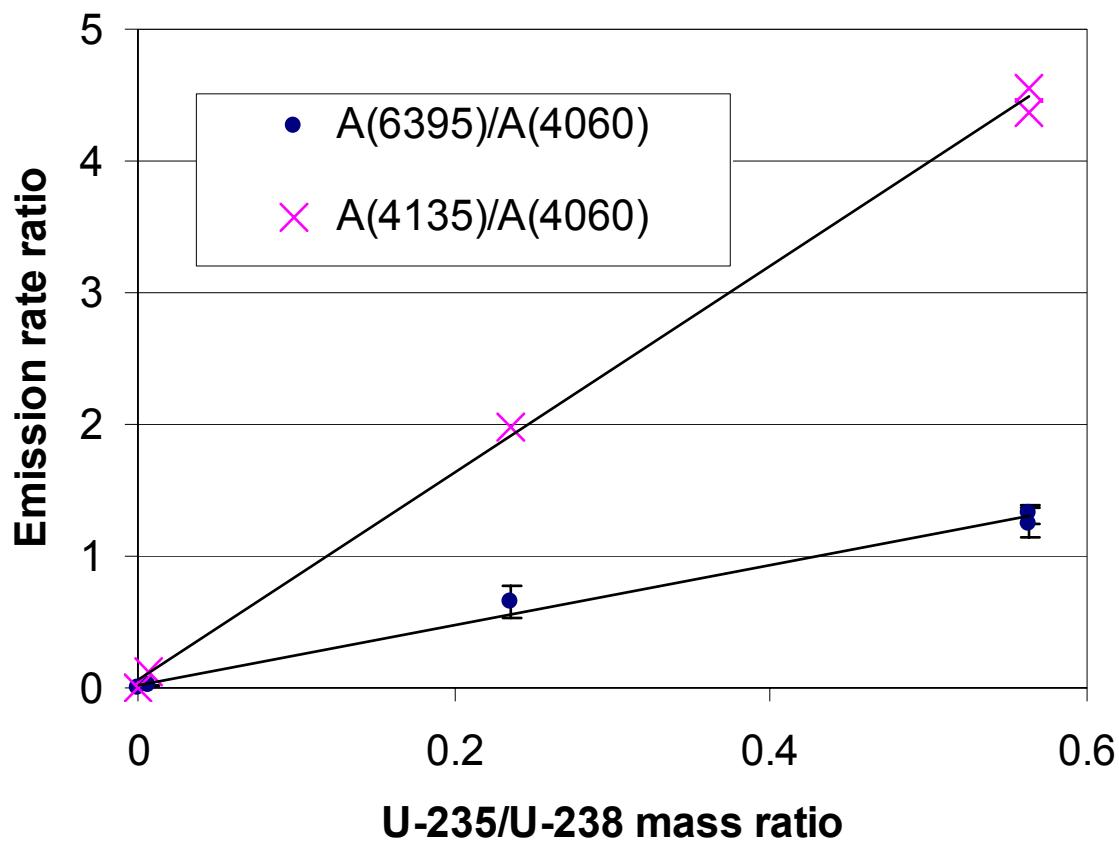
# First uranium measurements

- Nat. uranyl acetate and nitrate
  - PGAA
  - decay regularly after irradiation
- enriched (19% and 36%)  $\text{U}_3\text{O}_8$ 
  - PGAA
  - decay measurements regularly after irrad.

# Main results

- Calibration curve
  - peak area ratio → enrichment
  - 1 hour irradiation, 1 g U,
  - in the region of 0.1–50%
- Partial cross section for the most important lines
  - rel. uncertainty ~10%

# Calibration curve from uranium enrichment measurements



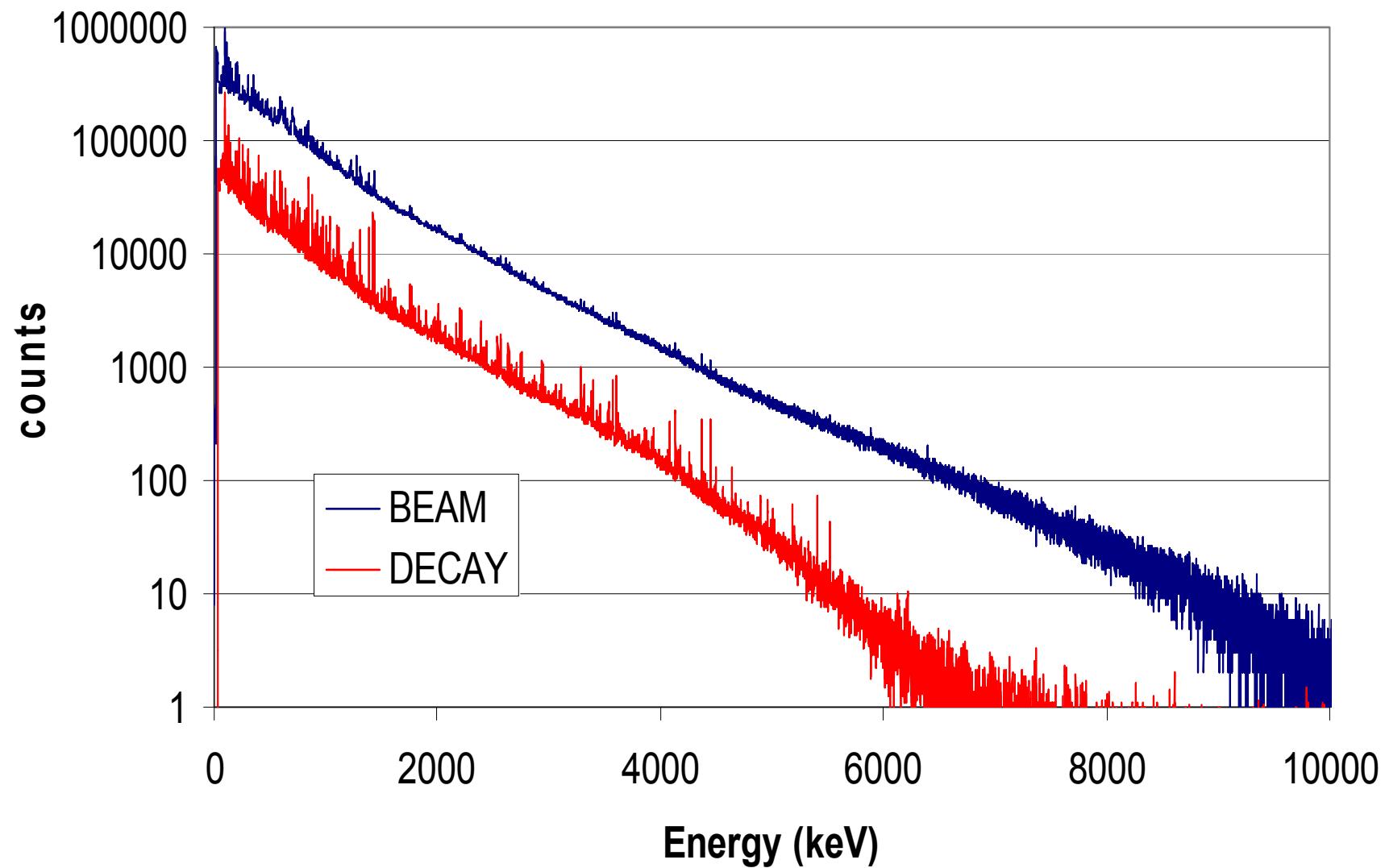
# New uranium measurements

- Nat. uranyl-acetate
  - PGAA
  - chopped beam PGAA
  - decay measurements regularly after irrad.
- enriched (95%)  $\text{U}_3\text{O}_8$ 
  - PGAA
  - chopped beam PGAA
  - decay measurements regularly after irrad.

# Irradiation of 50mg U<sub>3</sub>O<sub>8</sub>

Background	1 cps
natural activity	~5 cps
decay	~90 cps
total in-beam	~700 cps

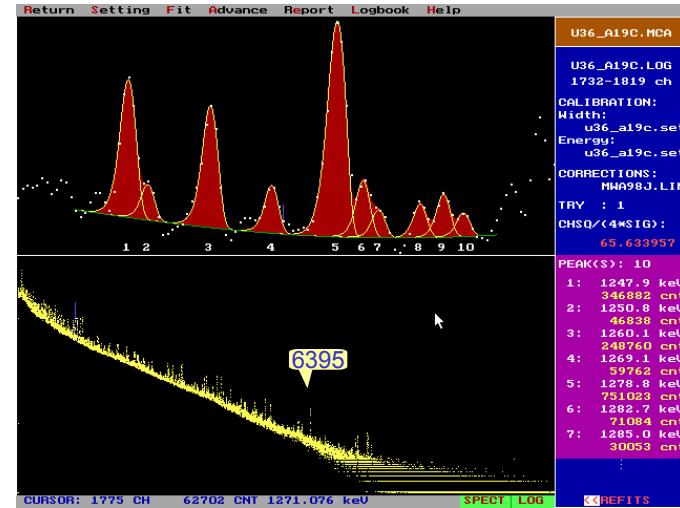
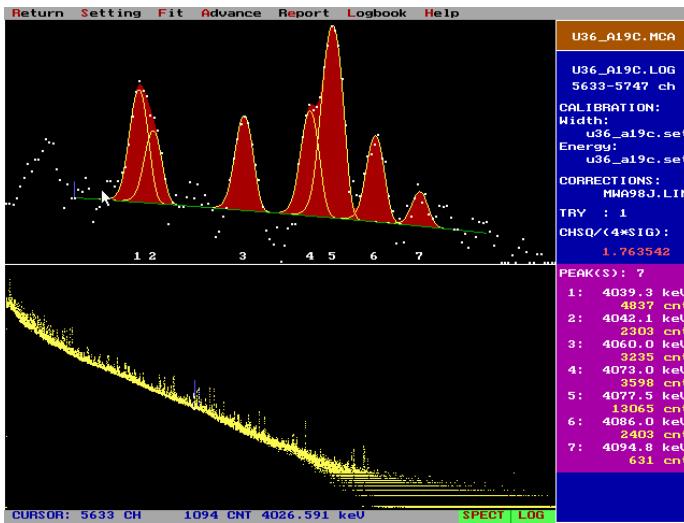
# Uranium spectra



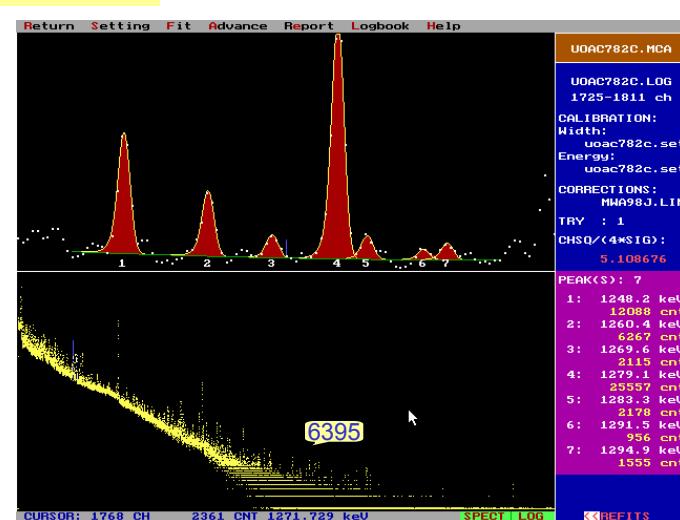
# U-239 capture line - 4060 keV

# Te-134 fission line - 1279 keV

Enriched U (36% U-235)



Natural U (0.72% U-235)



## Summary of PGAA results on U and Tc

Orig. nuclid	Parent nuclide	Type of meas.	Energy (keV)	Half-life	Peak count rate (cps)	Meas. sigma (barn)	Calc. sigma (barn)
<sup>238</sup> U	<sup>239</sup> U	PGAA	4060	0	0.98	<b>0.192(2)</b>	-
	<sup>239</sup> Np	Decay	278	2.36 d	0.074	<b>0.382(6)</b>	0.383(6)
	<sup>234</sup> Th	Nat	92	$4.468 \cdot 10^9$ y	0.045(11)		
<sup>235</sup> U	<sup>236</sup> U	PGAA	6395	0	0.011	<b>0.0038(2)</b>	
	<sup>134</sup> Te	Chopped PGAA	297	0	1.60	<b>0.22(2)</b>	0.22(2)
	<sup>134</sup> Te	Chopped PGAA	1279	0	0.49	<b>0.20(1)</b>	0.22(2)
	<sup>140</sup> Ba	Decay	537	12.75 d	0.0122	<b>0.066(3)</b>	0.064(1)
	<sup>235</sup> U	Nat	186	$7.037 \cdot 10^8$ y	0.073		
<sup>99</sup> Tc	<sup>99</sup> Tc	PGAA	172		3.0	<b>16.61(15)</b>	-
	<sup>99</sup> Tc	PGAA	223		0.24	<b>1.472(13)</b>	-
	<sup>99</sup> Tc	PGAA	263		0.21	<b>1.425(12)</b>	-
	<sup>100</sup> Tc	Decay	539	16 s	0.14	<b>1.604(14)</b>	-
	<sup>100</sup> Tc	Decay	591	16 s	0.10	<b>1.296(11)</b>	-
	<sup>99</sup> Tc	Decay	89	211 000 y	$4.3 \times 10^{-3}$	-	-

<1 %

# **FINAL REMARKS**

- Up to now the capability of PGAA even with thermal and cold neutron beams has been tested for different kinds of samples
- The “PGAA library” (i.e. the sensitivity data) for every elements are compiled
- The non-destructive feature of the method can be exploited by many users - chemists, materials scientists, geologists, archaeologists, etc.
- New methods, such as chopped beam PGAA, coincidence method open new possibilities

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**THANK YOU FOR YOUR COOPERATION!**