united nations educational, scientific and cultural organization
international atomic energy agency

the **abdus salam** international centre for theoretical physics

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

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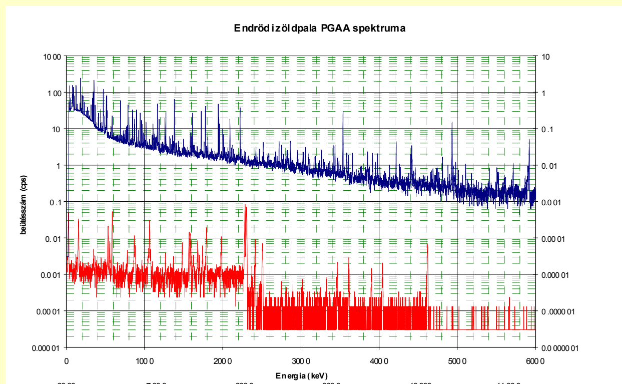
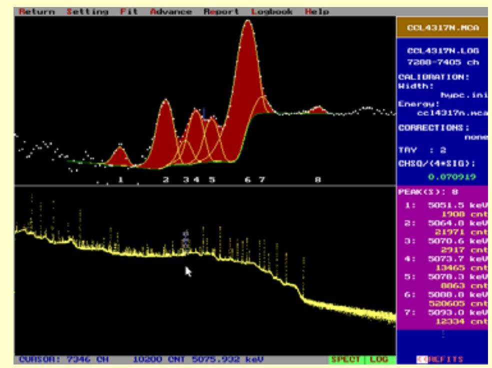
Main steps of PGAA analysis





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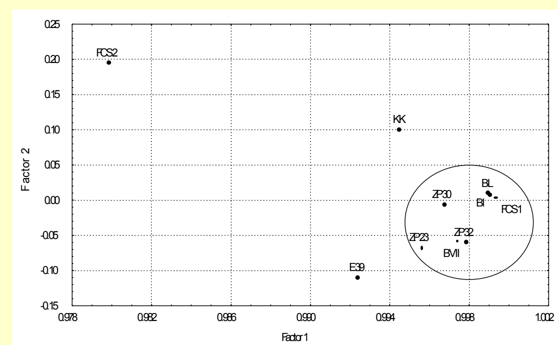
Z	El	M	m	unc%	m(bkg)	unc%	m(net)	n(ox)	m(ox)	unc%	c% atom	c% el/el	c% el/ox	c% ox/ox	unc%
1	H	1.00794	0.0729	1.2	0.00018	3.0	0.07272	0.5	0.6499	1.2	0.027	0.904	0.484	4.328	1.2
5	B	10.811	6.7E-05	1.1	1E-08	0.0	6.7E-05	1.5	0.00022	1.2	3E-04	8E-04	4E-04	0.001	1.2
11	Na	22.9898	0.39095	2.5	0	0.0	0.39095	0.5	0.52699	2.5	3.36	4.859	2.604	3.51	2.5
12	Mg	24.305	0.93332	3.6	0	0.0	0.93332	1	1.5477	3.6	8.48	11.6	6.216	10.31	3.6
13	Al	26.9815	1.21162	1.6	0.00273	5.0	1.20889	1.5	2.28416	1.6	12.19	15.03	8.051	15.21	1.6
14	Si	28.0855	3.09038	1.5	0	0.0	3.09038	2	6.61136	1.5	32.45	38.41	20.58	44.03	1.5
16	S	32.066	0.03361	5.8	0	0.0	0.03361	3	0.08391	5.8	0.403	0.418	0.224	0.559	5.8
17	Cl	35.4527	0.0015	7.0	1.8E-05	20.0	0.00149	0	0.00149	7.1	0.02	0.018	0.01	0.01	7.1
19	K	39.0983	0.04832	12.4	0	0.0	0.04832	0.5	0.05821	12.4	0.706	0.601	0.322	0.388	12.4
20	Ca	40.078	0.75737	2.0	0	0.0	0.75737	1	1.05972	2.0	11.35	9.414	5.044	7.058	2.0
21	Sc	44.9559	0.00063	14.5	0	0.0	0.00063	1.5	0.00097	14.5	0.011	0.008	0.004	0.006	14.5
22	Ti	47.867	0.1515	1.0	0	0.0	0.1515	2	0.25277	1.0	2.711	1.883	1.009	1.683	1.0
23	V	50.9415	0.00471	6.8	0	0.0	0.00471	2.5	0.00841	6.8	0.09	0.059	0.031	0.056	6.8
24	Cr	51.9961	0.00984	7.9	0	0.0	0.00984	1.5	0.01438	7.9	0.191	0.122	0.066	0.096	7.9
25	Mn	54.938	0.01509	2.4	0	0.0	0.01509	1	0.01948	2.4	0.31	0.188	0.101	0.13	2.4
26	Fe	55.845	1.3222	1.2	0.00144	5.0	1.32076	1.5	1.88835	1.2	27.57	16.42	8.796	12.58	1.2
27	Co	58.9332	0.00531	4.0	0	0.0	0.00531	1	0.00676	4.0	0.117	0.066	0.035	0.045	4.0
62	Sm	150.36	5.4E-05	2.1	0	0.0	5.4E-05	1.5	6.3E-05	2.1	0.003	7E-04	4E-04	4E-04	2.1
64	Gd	157.25	7.6E-05	2.0	0	0.0	7.6E-05	1.5	8.7E-05	2.0	0.004	9E-04	5E-04	6E-04	2.0
66	Dy	162.5	0.00014	18.3	0	0.0	0.00014	1.5	0.00017	18.3	0.009	0.002	1E-03	0.001	18.3
							8.04523		15.0151	0.806	100	100	53.58	100	
					- O calculated				6.96985	46.42	%				
					mass w/o O				8.04523						



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

θ : Isotopic abundance

σ_0 : Neutron capture cross-section

I_γ : Gamma-yield

Φ_0 : Neutron flux

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

Introducing

the flux-independent

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

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

gives the mass ratio of arbitrary elements

$$c_x = m_x / \Sigma m_i$$

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 / σ_γ library
  - no need for element standards
- Several labs worldwide
  - NIST, JAERI, SNU/KAERI, BARC etc.

APPLICATIONS

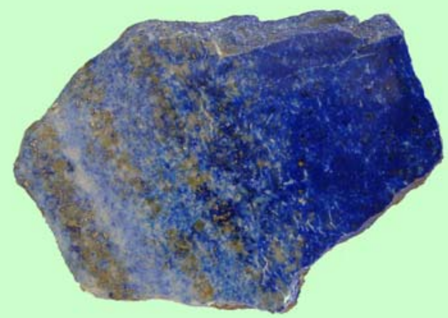
Selected Applications

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

ROCKS AND MINERALS



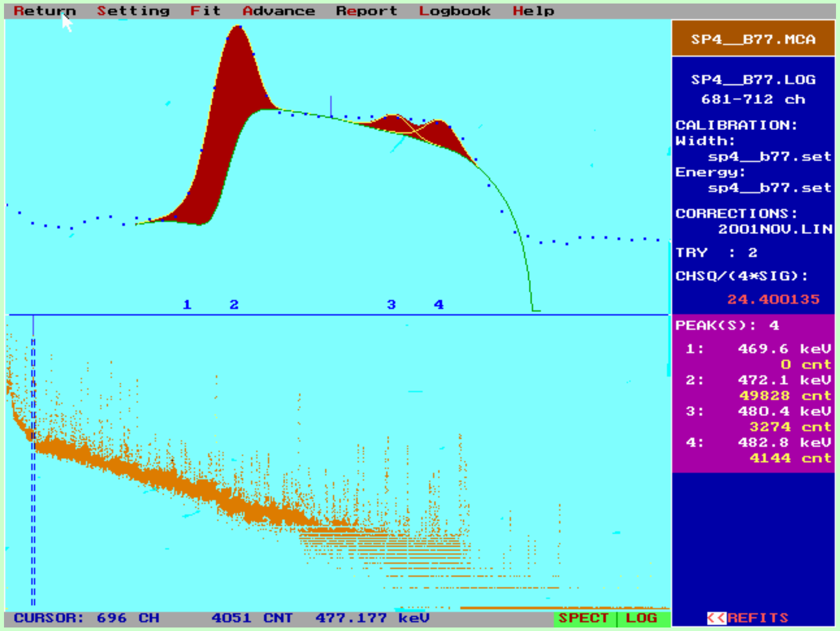


BORON IN MIOCENE VOLCANIC ROCKS

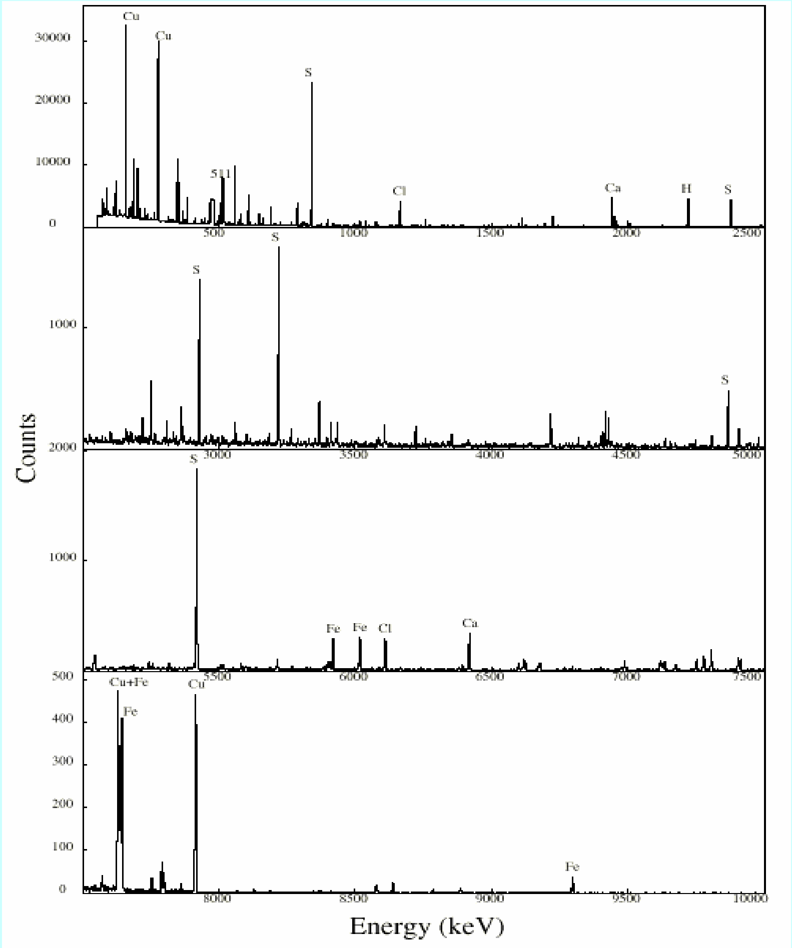
(Dept. of Petrography, Eötvös University)

- **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



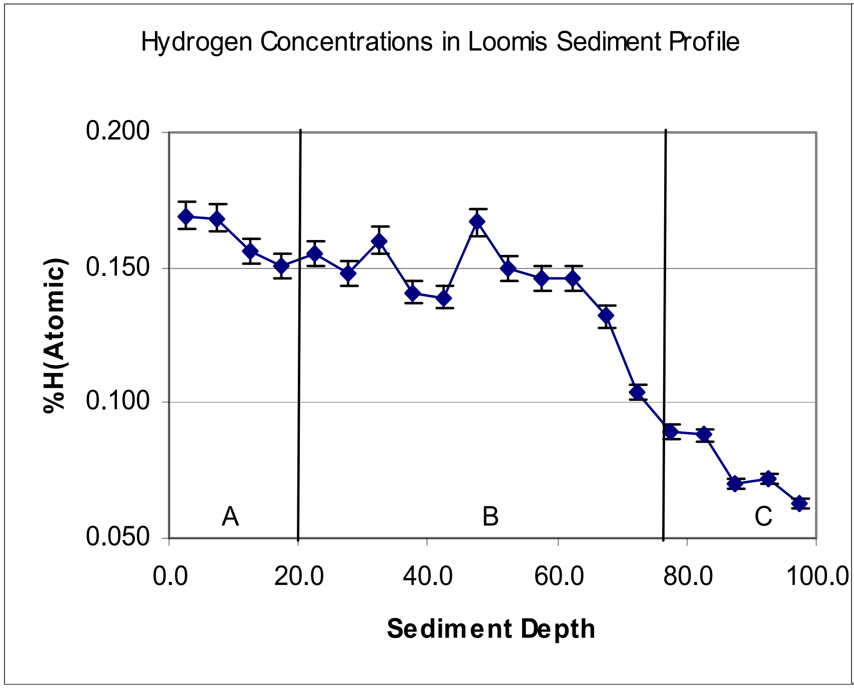
Experimental Results With PGAA

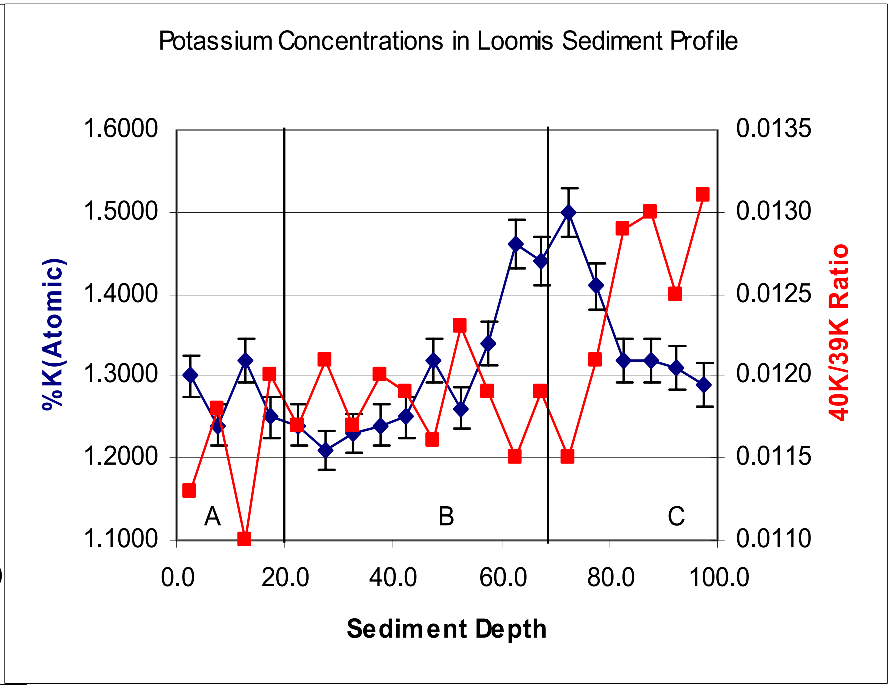
- **Analysis of Deep Sea Vents (% by weight)**

	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

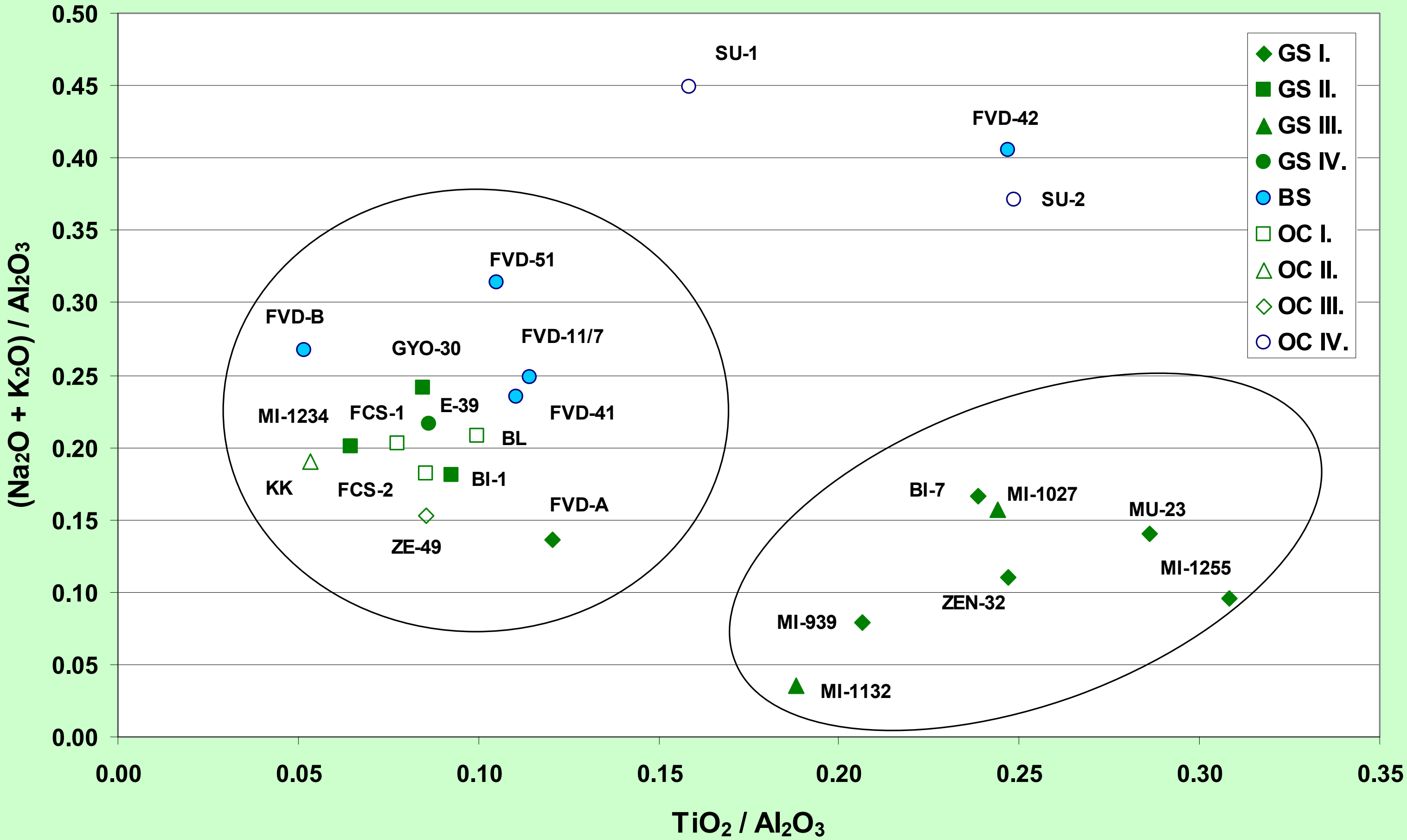
^{40}K,U,Th,^{137}Cs from low-background counting; ^{39}K from PGAA

NEOLITHIC POLISHED STONE TOOLS - GREENSCHIST AND BLUESCHIST

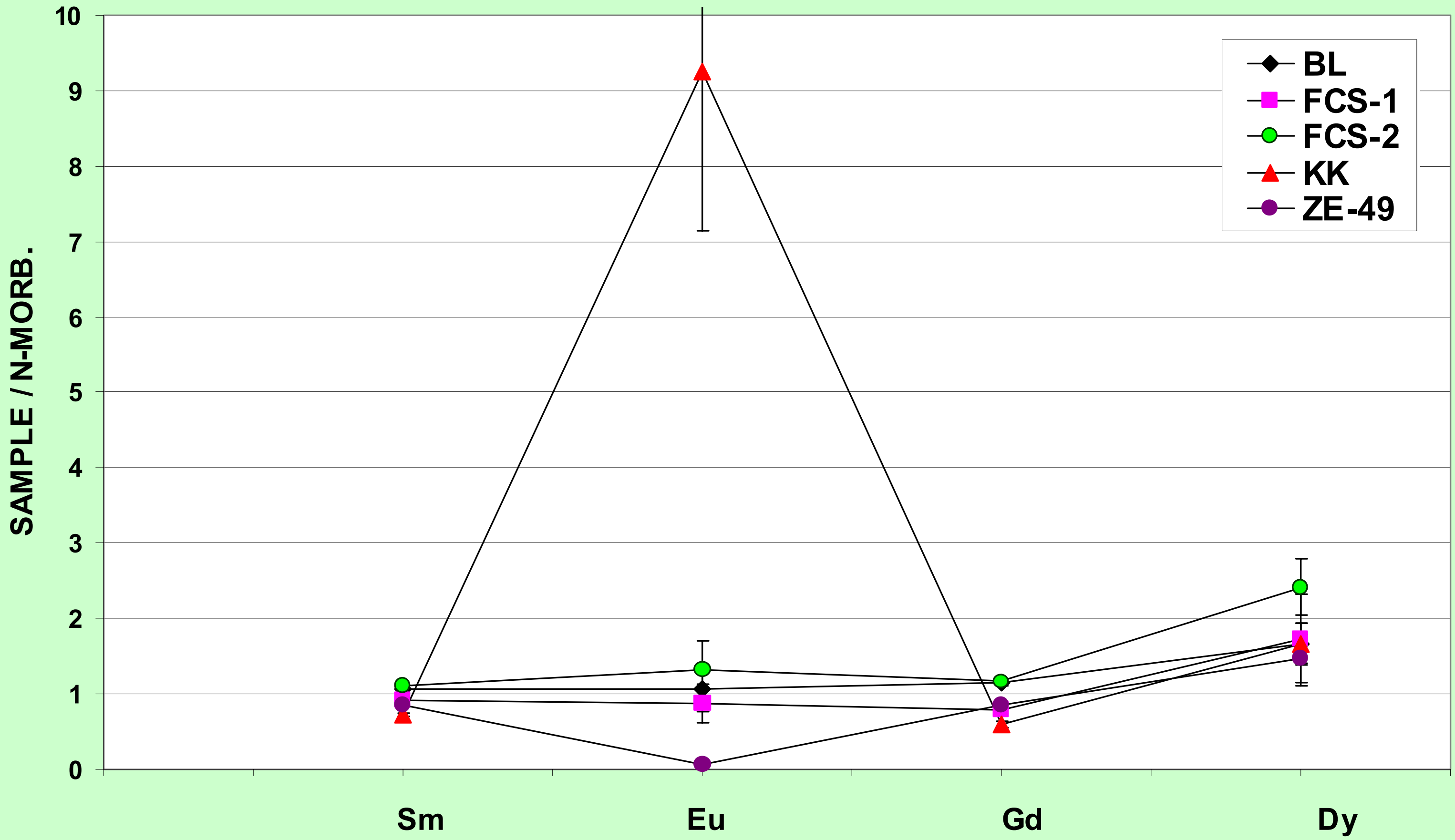
(Dept. of Petrography, Eötvös University)

- **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 (SiO_2, Al_2O_3, TiO_2 Fe_2O_3, MnO, CaO, MgO, Na_2O, K_2O)
  and traces (B, Sc, V, Co, Cr, Sm, Eu, Gd, Dy)

CLASSIFICATION OF GREENSCHIST AND BLUESCHIST OBJECTS



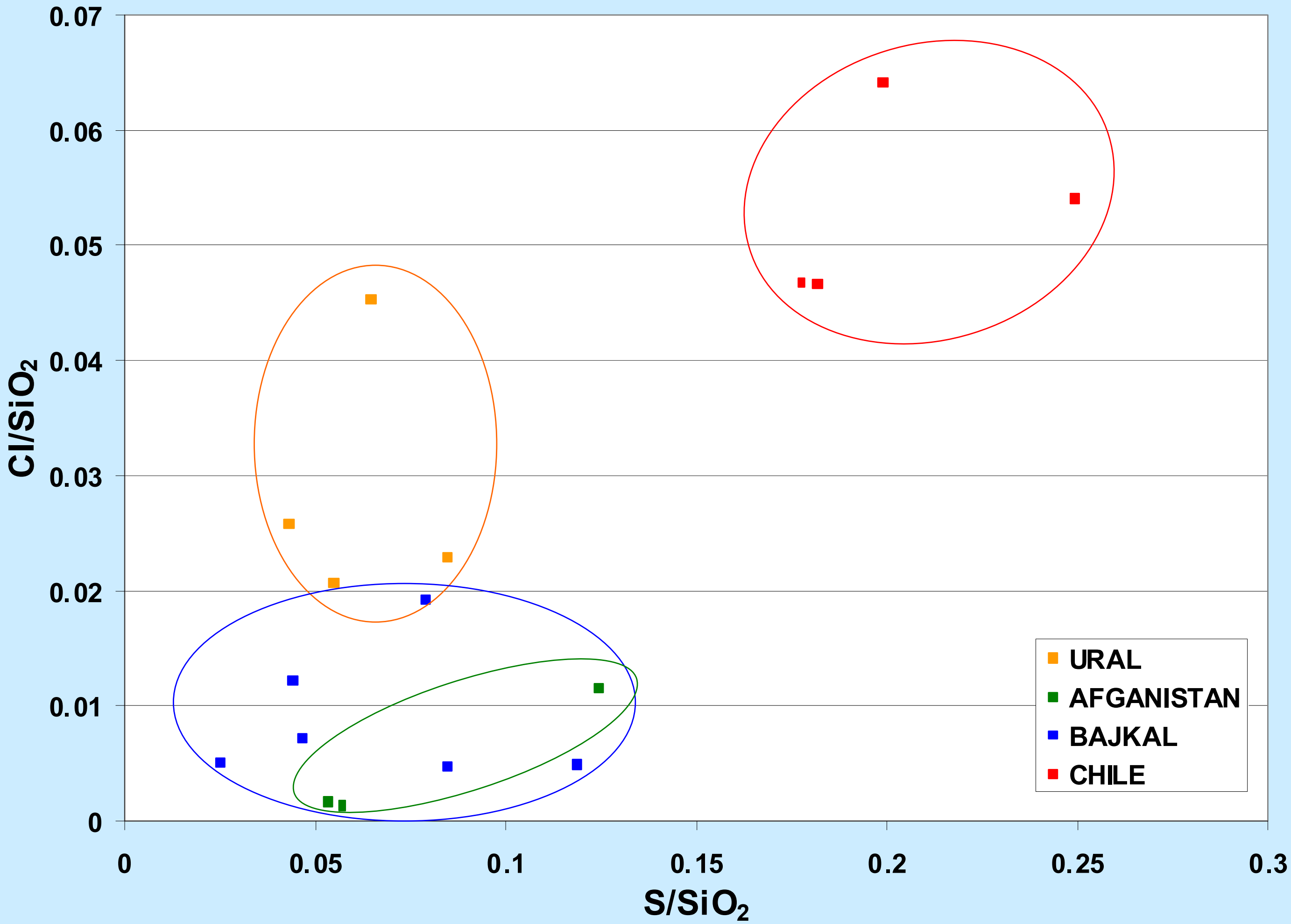
REE-PATTERN OF SAMPLES FROM QUARRIES (Eu-ANOMALY)



LAPIS-LAZULI
(University of Tübingen)

- Semi-precious stone
- Few quarries known over the World (Ural, Chile, Afghanistan, Lake Baikal)
- **Mineral**: Lazurite / $(Na,Ca)_{7\text{-}8}(Al,Si)_{12}O_{24}[(SO_4)Cl_2(OH)_2]$
- **PGAA**: H, Na, Mg, Al, Si, K, Ca, Ti, Mn, Fe, S, Cl

CLASSIFICATION OF LAPIS-LAZULI RAW MATERIAL

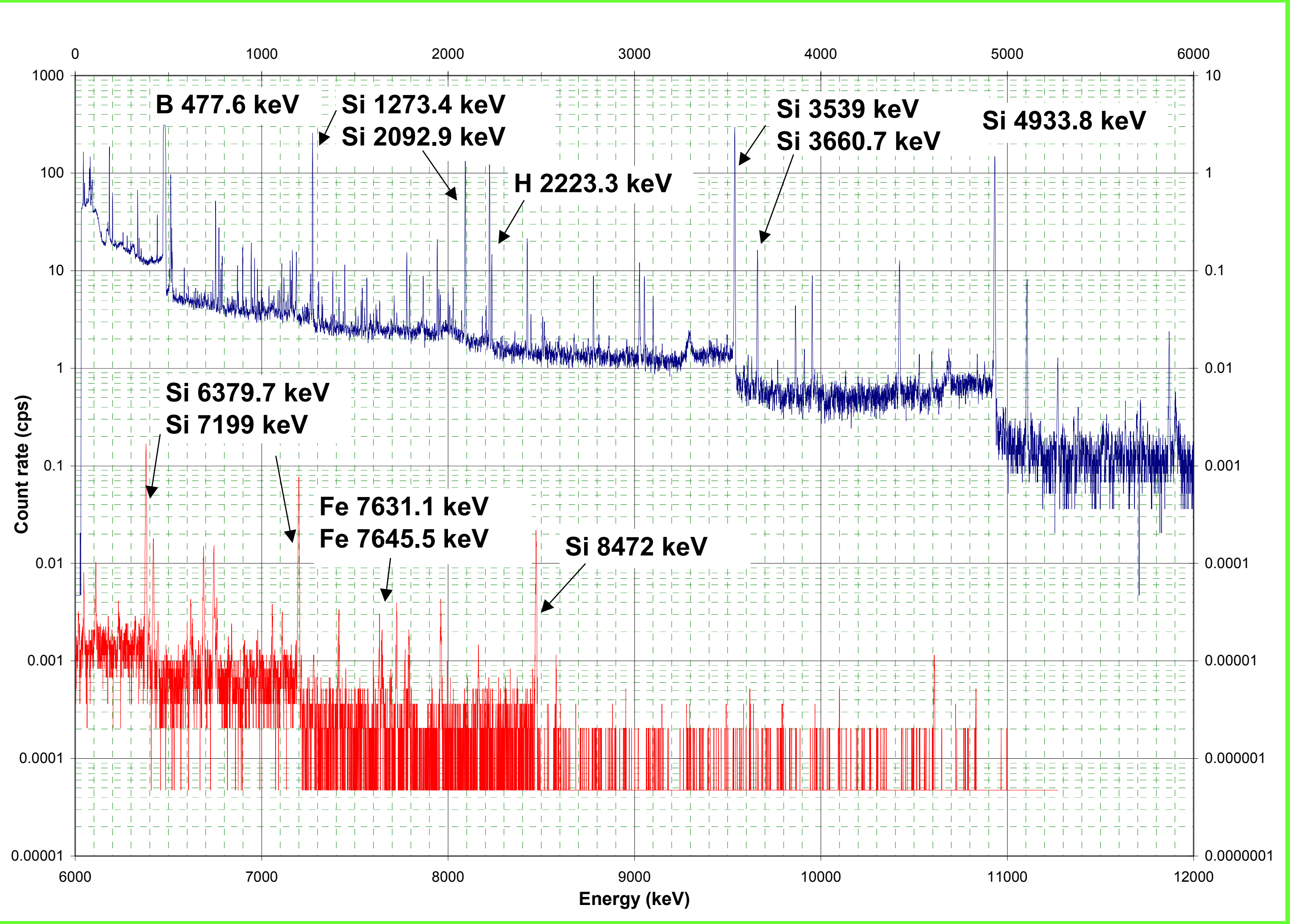


CHIPPED STONE TOOLS MADE OF GREY FLINT

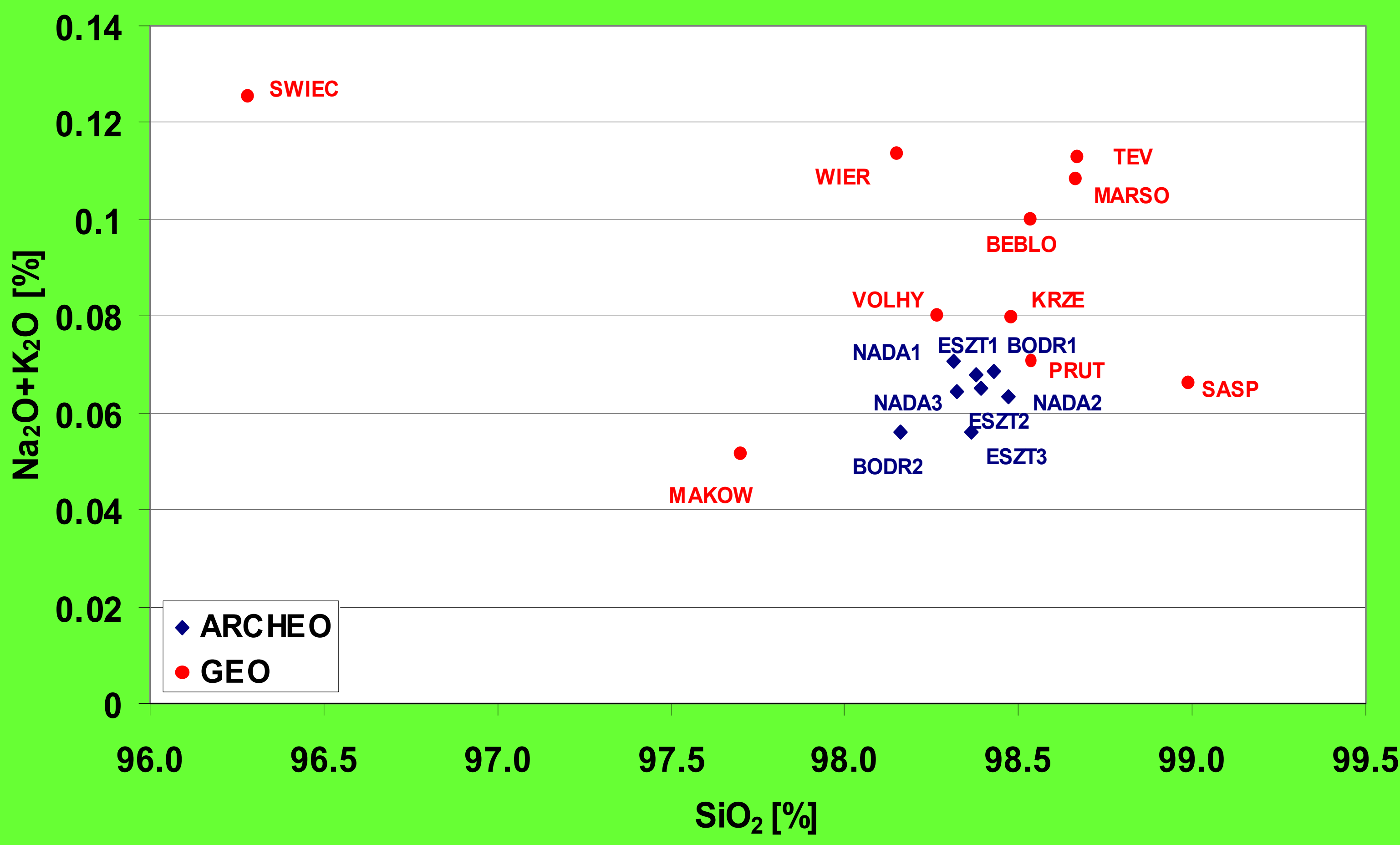
(Hungarian National Museum)

- **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_2**; Al_2O_3, TiO_2, Fe_2O_3, CaO, Na_2O, K_2O, MgO, MnO, H_2O, Cl, B, Sm, Eu, Gd, Sc, Cd

PGAA spectrum of a grey flintstone



CLASSIFICATION OF GREY FLINT SAMPLES



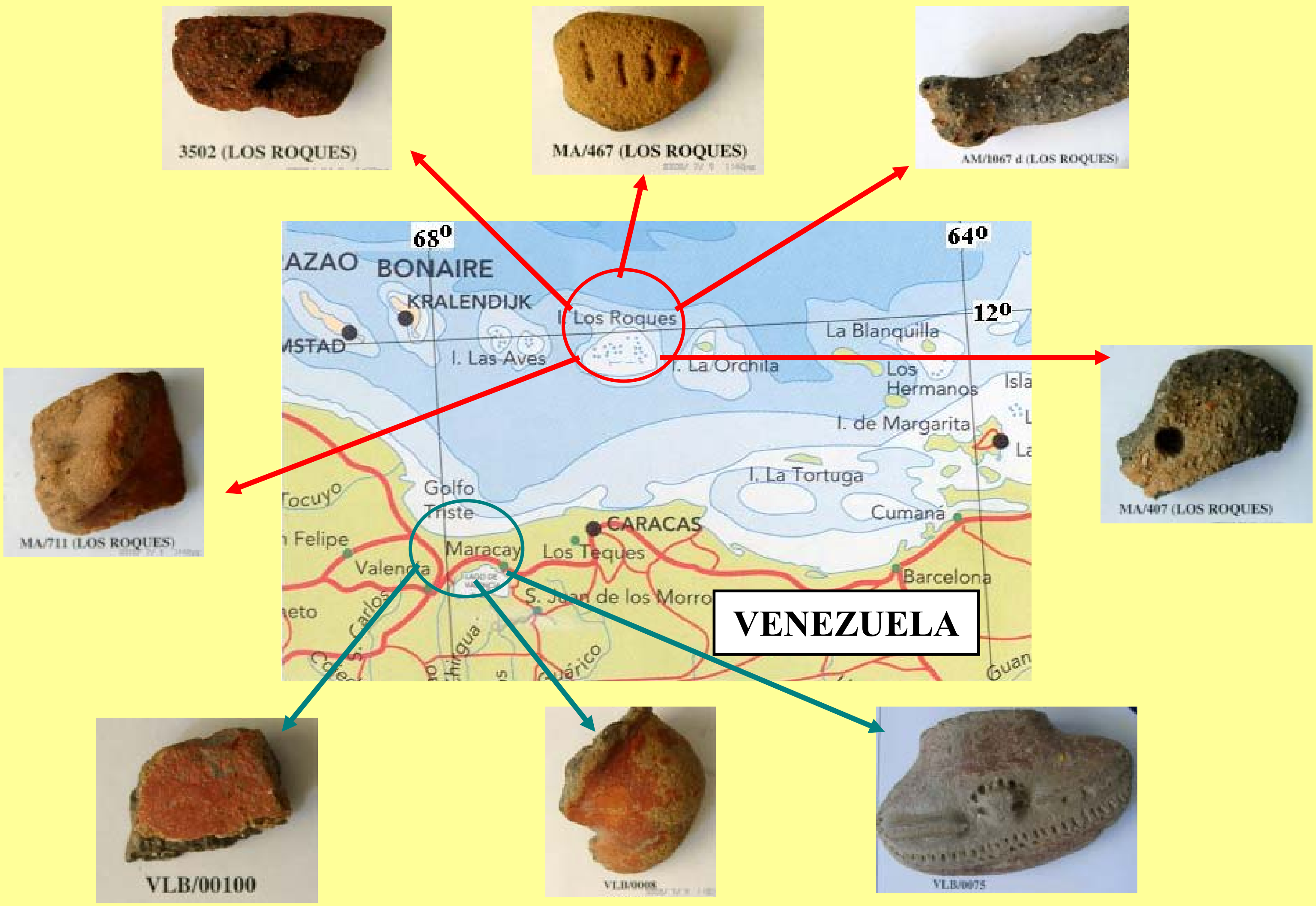
CERAMICS

PRE-COLUMBIAN POTTERY FROM VENEZUELA

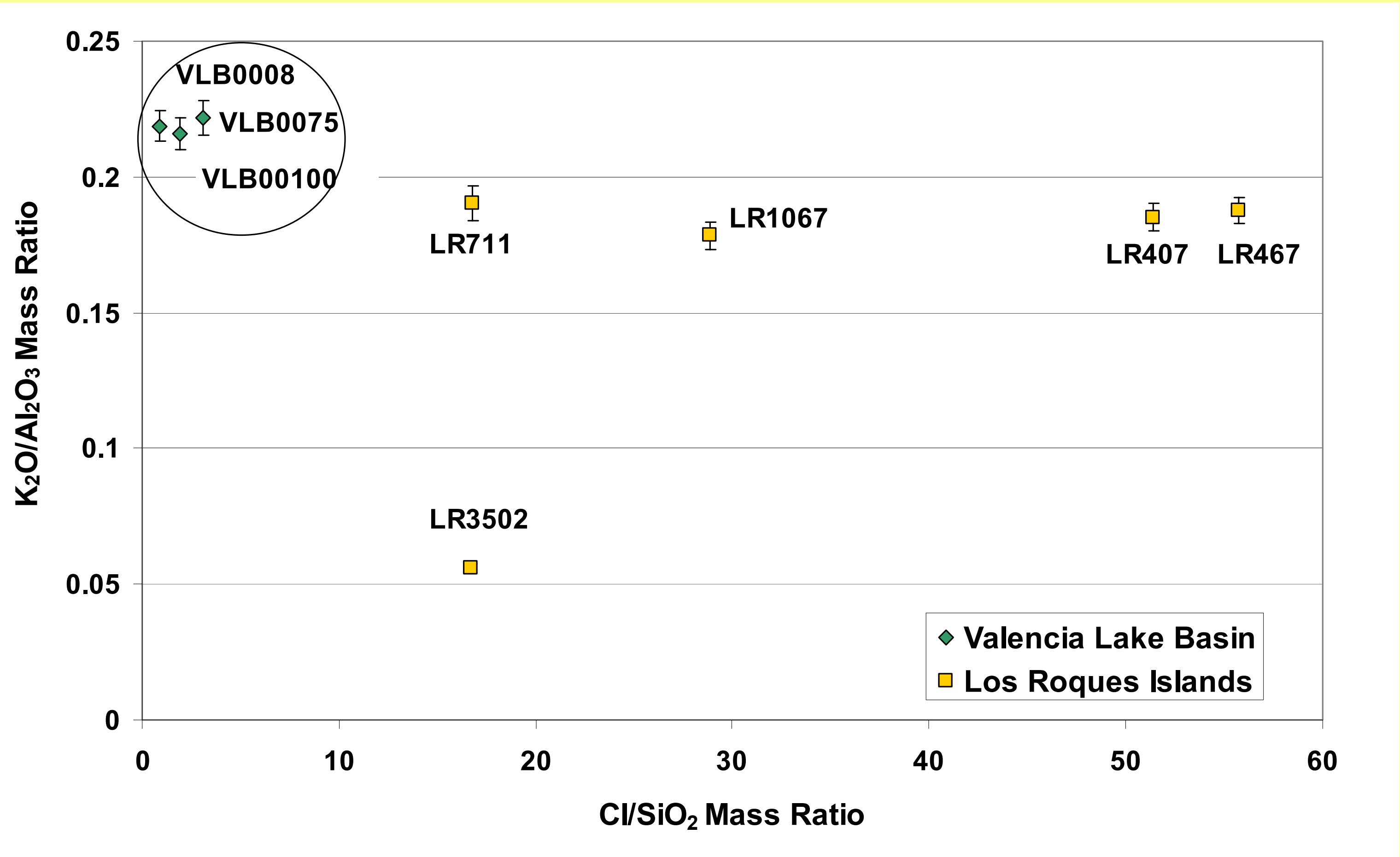
(Simón Bolívar University, Caracas)

- Fragments of pottery figurines from 12^{nd}-15^{th} 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)

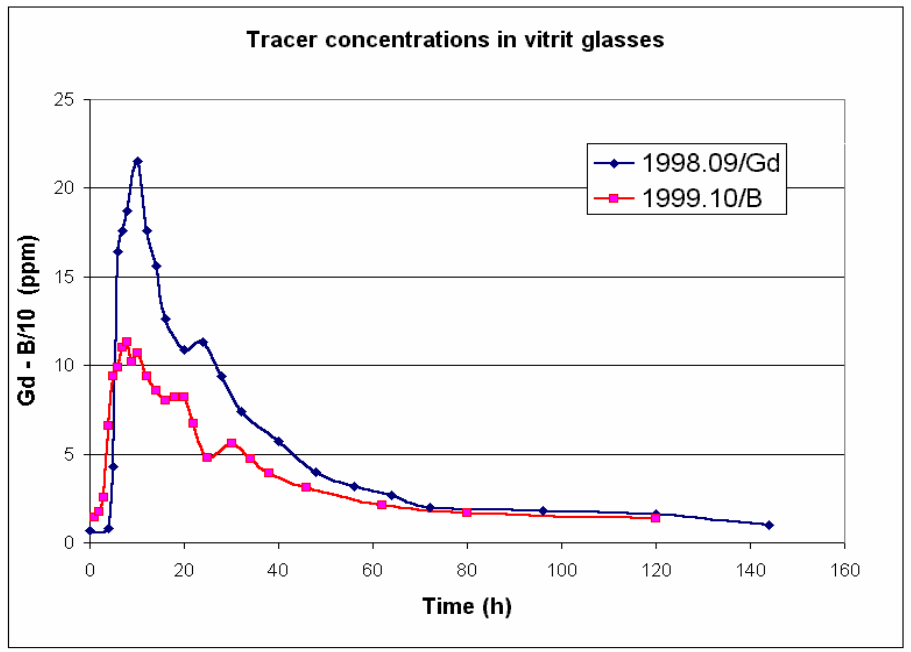
WHITE	Conc. [%]		BLUE	Conc. [%]		GREEN	Conc. [%]
SiO_2	59.7		SiO_2	71.8		SiO_2	71.2
K_2O	8.33		K_2O	17.6		K_2O	17.4
PbO	25.1		PbO	<2		PbO	3.1
CaO	1.3		CaO	2.98		CaO	2.27
Al_2O_3	0.58		Al_2O_3	<0.3		Al_2O_3	<0.3
Na_2O	3.1		Na_2O	1.79		Na_2O	1.12
MgO	0.5		MgO	<0.2		MgO	<0.2
P_2O_5	<2		P_2O_5	2.16		P_2O_5	<2
MnO	0.07		MnO	0.018		MnO	0.030
Fe_2O_3	0.081		Fe_2O_3	<0.05		Fe_2O_3	<0.05
CuO	<0.1		CuO	0.83		CuO	0.77
TiO_2	0.037		TiO_2	<0.01		TiO_2	<0.01
B	0.00137		B	0.00296		B	0.00570

Good agreement with EPMA !

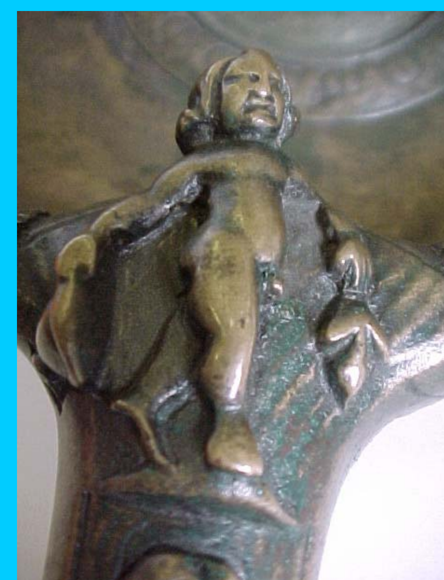
- **W: High Pb, colourless K-glass / Piece of goblet from 17^{th} C., England**
- **B and G: K-glass, colourants are Cu (U), opaque from P_2O_5**
  Wastes from 19^{th} C. Silesian glasswork

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 Gd_2O_3 and H_3BO_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



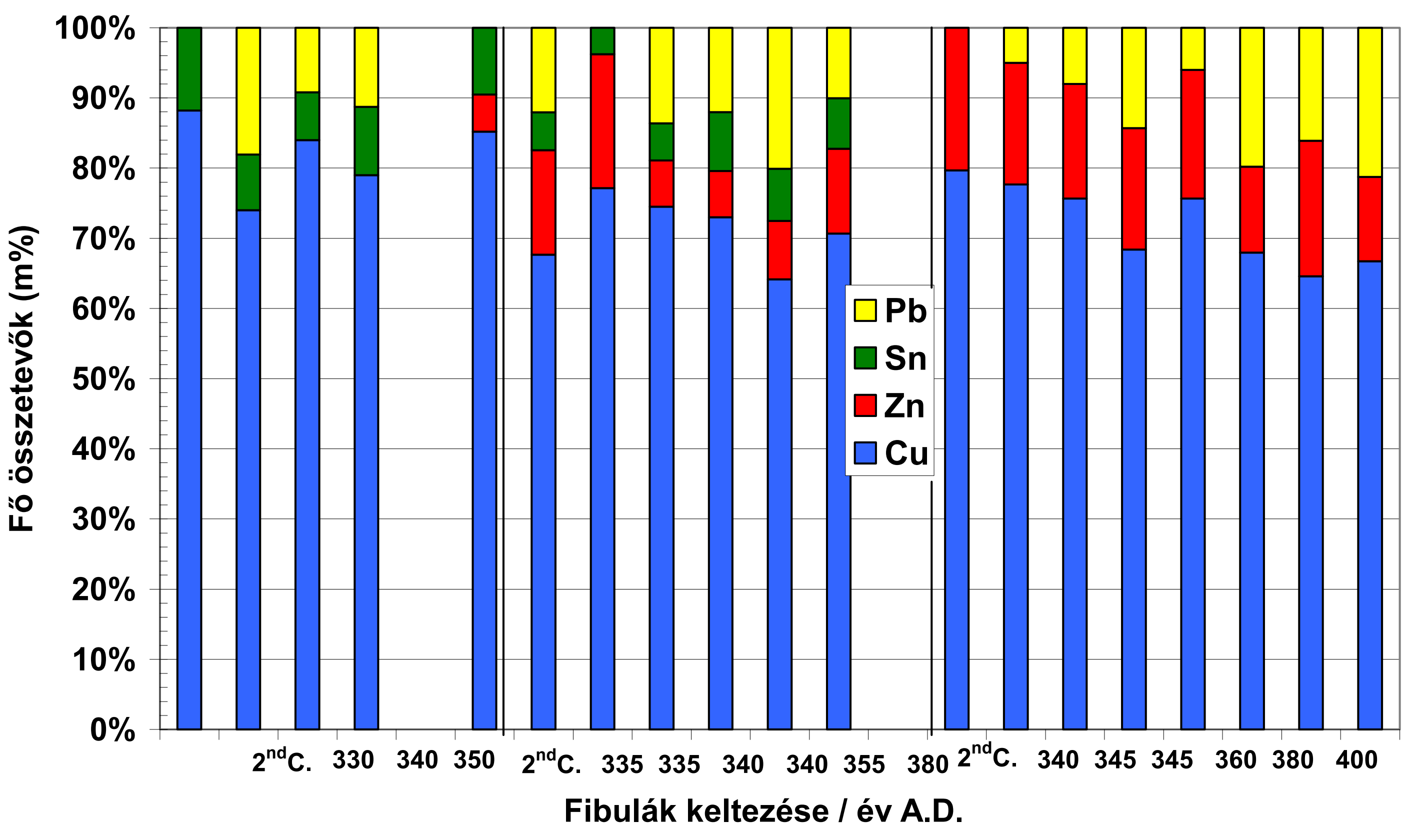
ROMAN BRONZE FIBULAE AND COINS

(Archaeological Institute of the HAS)

- **Cemetery of Hegyeshalom (H)**: 2nd and 4th 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*)

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

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

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

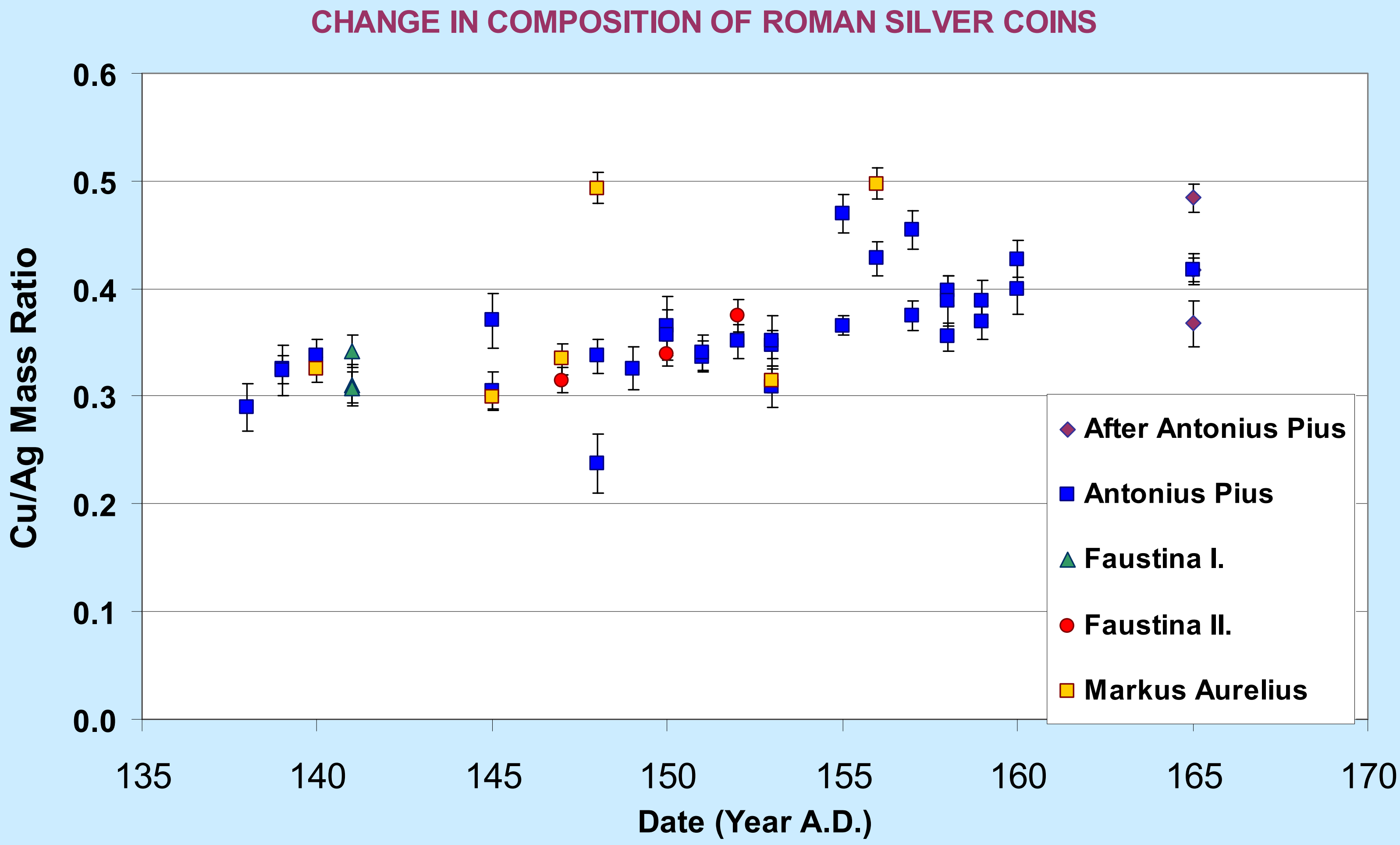
(*University of Bielefeld, Hungarian National Museum*)

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

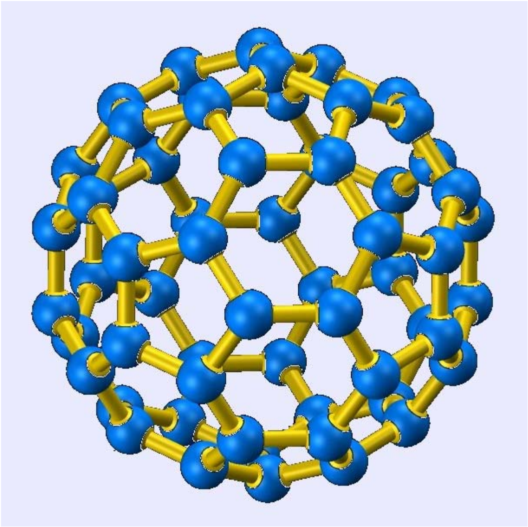
Most of them are pure tin-bronze (Cu:Sn ratio 9:1), some with high Pb or Zn

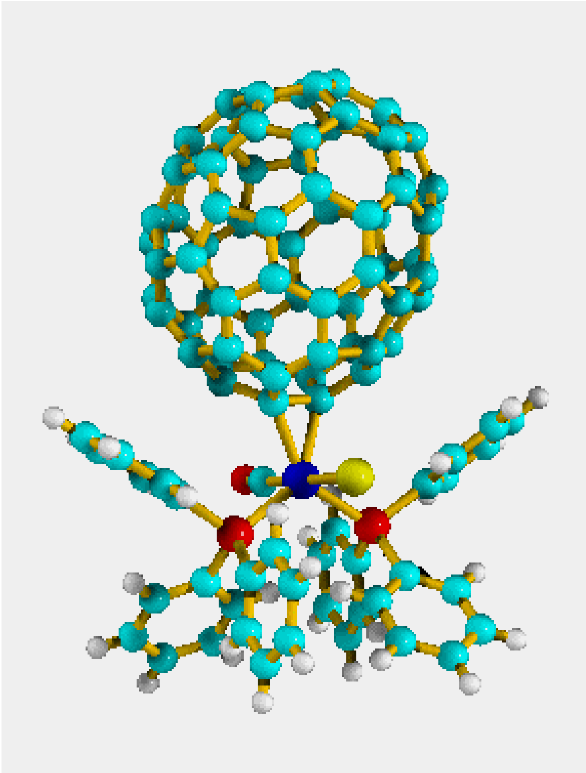
ROMAN SILVER COINS

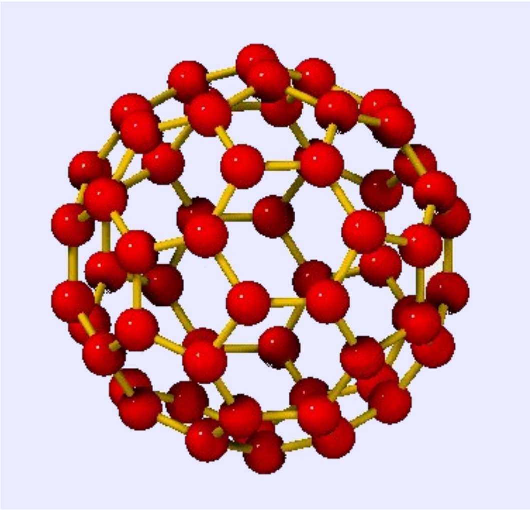
(Inst. of Nuclear Chemistry and Technology, Warsaw)



CHEMISTRY







Experimental Results With PGAA

- **Analysis of Reagent Materials (LBNL + Budapest)**

CaF_2 (Reagent grade, Baker and Adamson)

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

ZnO (Mallinckrodt)

Zn	100%)
Cd	5.1±0.3 ppm

HfO_2 (98%, Aldrich Chemical Co.)

HfO_2 (no detectable impurities)

TiO_2

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

Gd_2O_3 – no detectable impurities

$Ca(OH)_2$

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

CeO_2

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

SUPPORTED Pt CATALYSTS

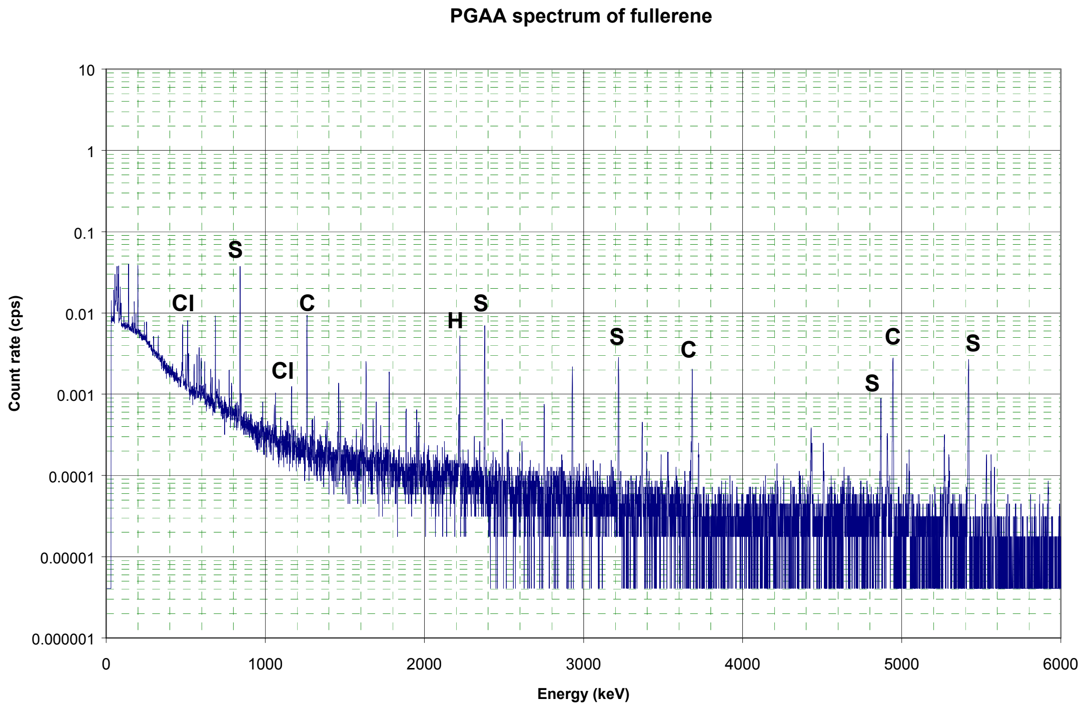
- Non-destructive test of catalysts containing noble metals has a great economical importance
- With PGAA metal content and H was also detectable
- For **EUROPT** catalysts the PGAA results are in good agreement with other methods

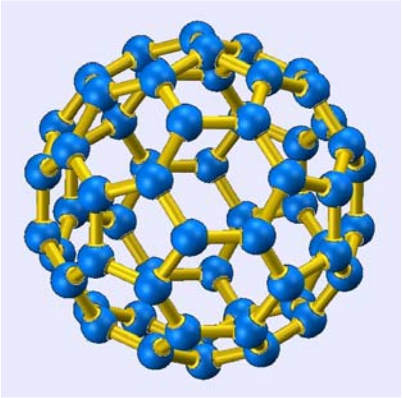
	m/m (%)			mol %
H	1.16	±	0.025	19.01
Al	46.40	±	0.696	28.27
Cl	1.06	±	0.009	0.49
Pt	0.34	±	$8.8 \cdot 10^{-3}$	0.03
O*	50.66	±	1.840	52.09

Composition of an industrial Pt/Al_2O_3 catalyst

SULFUR IN FULLERENE

(Dept. of Inorganic Chemistry, Eötvös University)



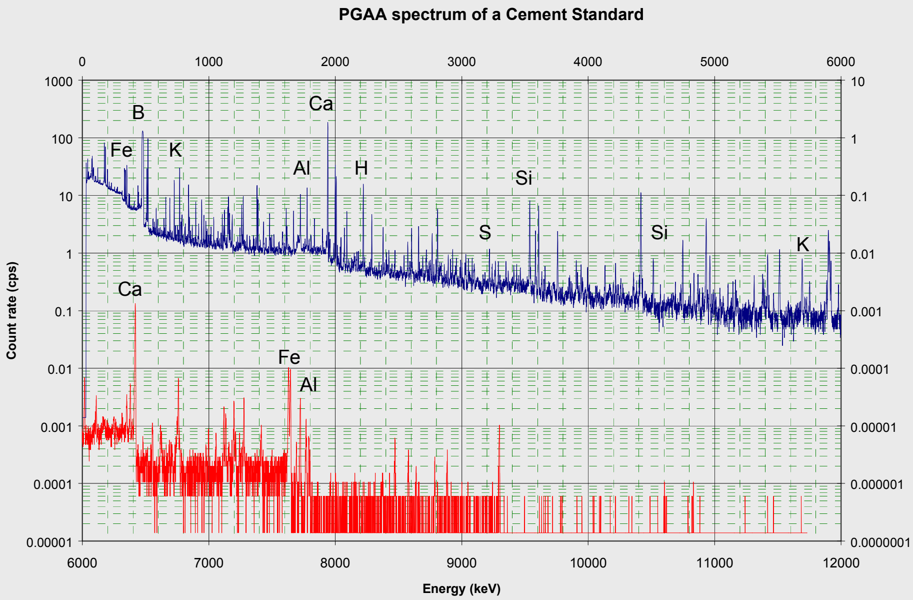


Element	Concentration (%)	Rel. unc (%)	Composition
H	0.012	10	0.08
C	97.1	4.5	60
S	2.88	1.3	0.67
Cl	0.003	20	0.0006

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

CEMENT AND CLINKER
(Cemkut Ltd.)

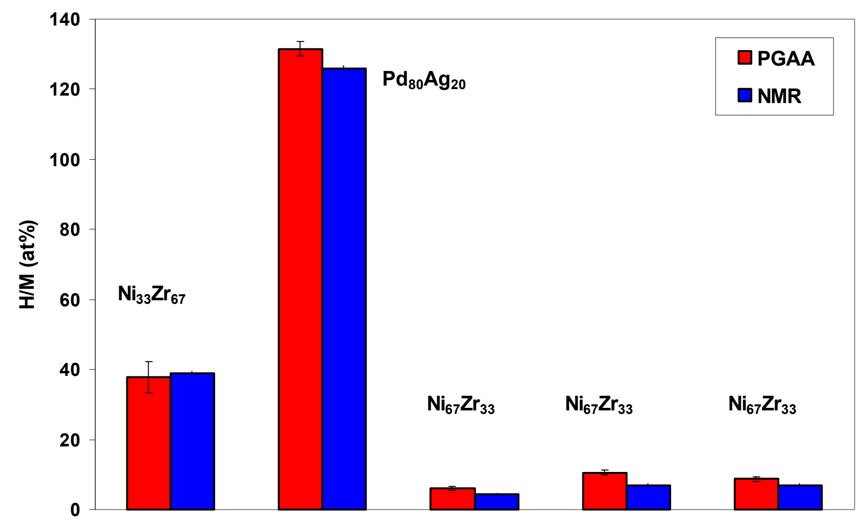
- Major components of industrial cements and clinker were measured
- PGAA gives high accuracy data of Ca, Si, Al and Fe oxides
- The analysis is much shorter (2-5 hours) than the classical quality control



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

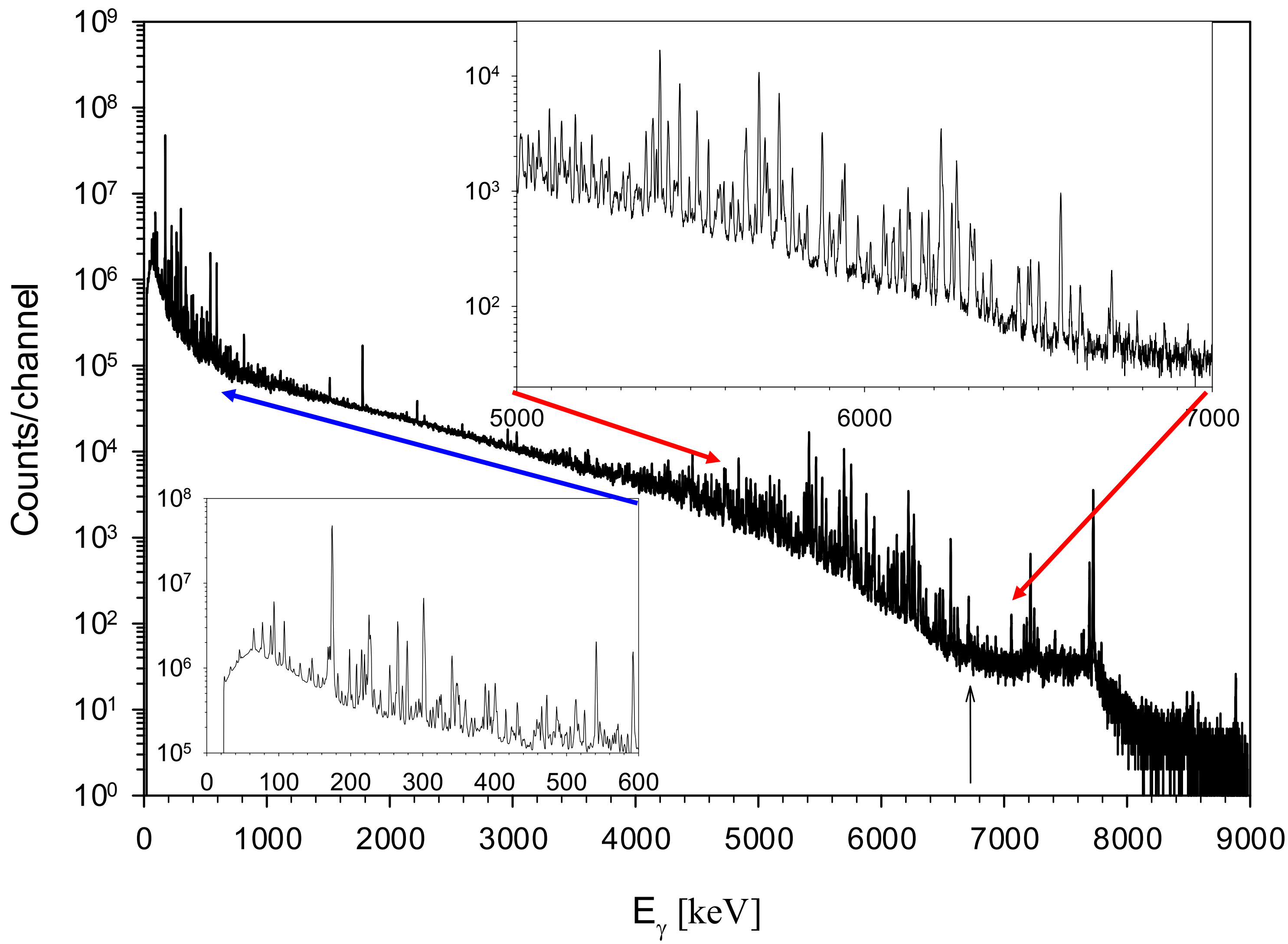


NUCLEAR MATERIALS

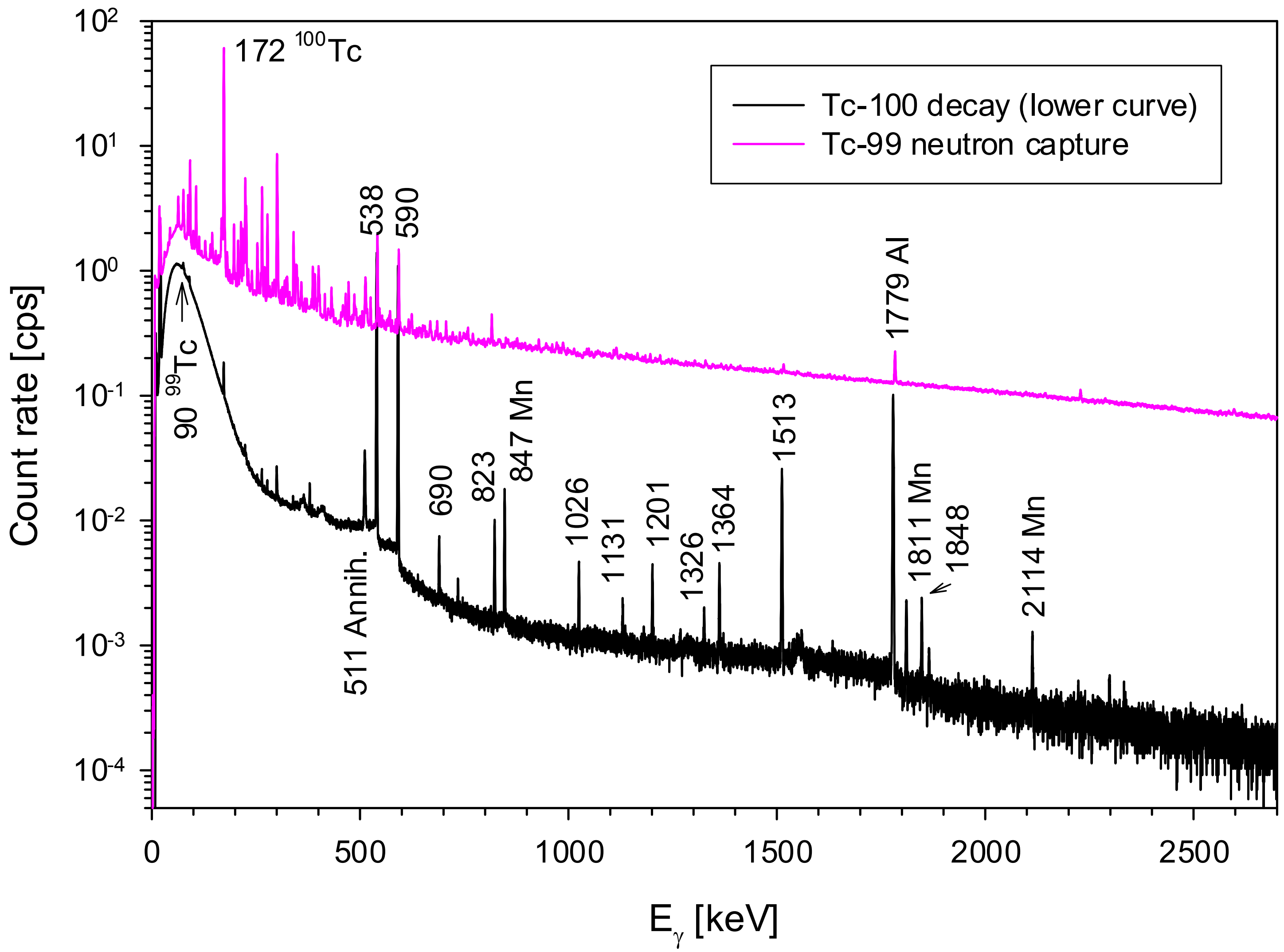
^{99}Tc measurements

✓ Tc	In-beam PGAA: rel. γ intensities
✓ Tc	Chopped beam PGAA: ^{100}Tc β^- decay
	> rel. γ intensities
✓ NH_4TcO_4	capture and decay lines: partial xsecs
	> capture cross section
In progress:	
Tc	Coincidence: level scheme
	> capture cross section

^{99}Tc 0.5 g sample (n,γ) spectrum



^{99}Tc chopped beam (n,γ) spectra



Partial γ-ray production cross sections of capture and decay lines for a ^{99}Tc target

E_γ (keV)	Origin	P_γ (γ/100 captures or decays)	σ_γ [a] (b)	Sensitivity (cps/mg)
172.1	^{99}Tc(n,γ)	67±6	16.61±0.15	3.0
223.4	^{99}Tc(n,γ)	6.1±0.6	1.472±0.013	0.24
263.5	^{99}Tc(n,γ)	5.9±0.5	1.425±0.012	0.21
539.5	^{100}Tc β^-	6.6±0.5 [b]	1.604±0.014	0.14
590.7	^{100}Tc β^-	5.3±0.5	1.296±0.011	0.10
89.5	^{99}Tc β^-	$(6.5\pm1.5)\times10^{-4}$ [c]		4.3×10^{-3}

Inferred total thermal neutron capture cross section of ^{99}Tc

Method	Basis	σ (b)	Comment
^{100}Tc β^-	539 γ	24.7±2.3	with P_γ from Ref. [9]
	591 γ	23.9 ±1.8	
	Average	24.3 ±2.2	unweighted average
^{99}Tc(n,γ)	$\Sigma\sigma_\gamma$ g.s.	21.21±0.17	lower limit
		26.5±2.6	missing ~20% added

Literature: EXFOR database 19±2 b
24 ±4 b

Harada: 22.9 ±2.6 b

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

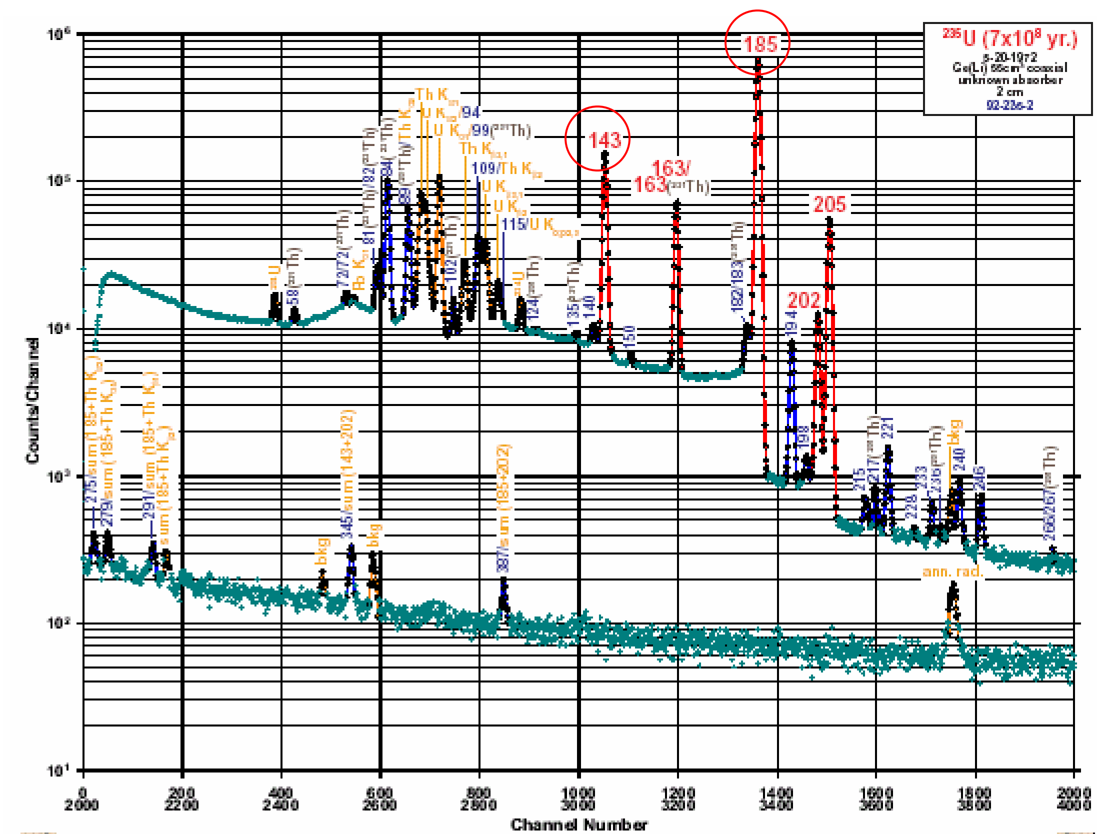
- ^{238}U →
- ^{235}U →
- fission prod. ^{134}Te →

Decay lines

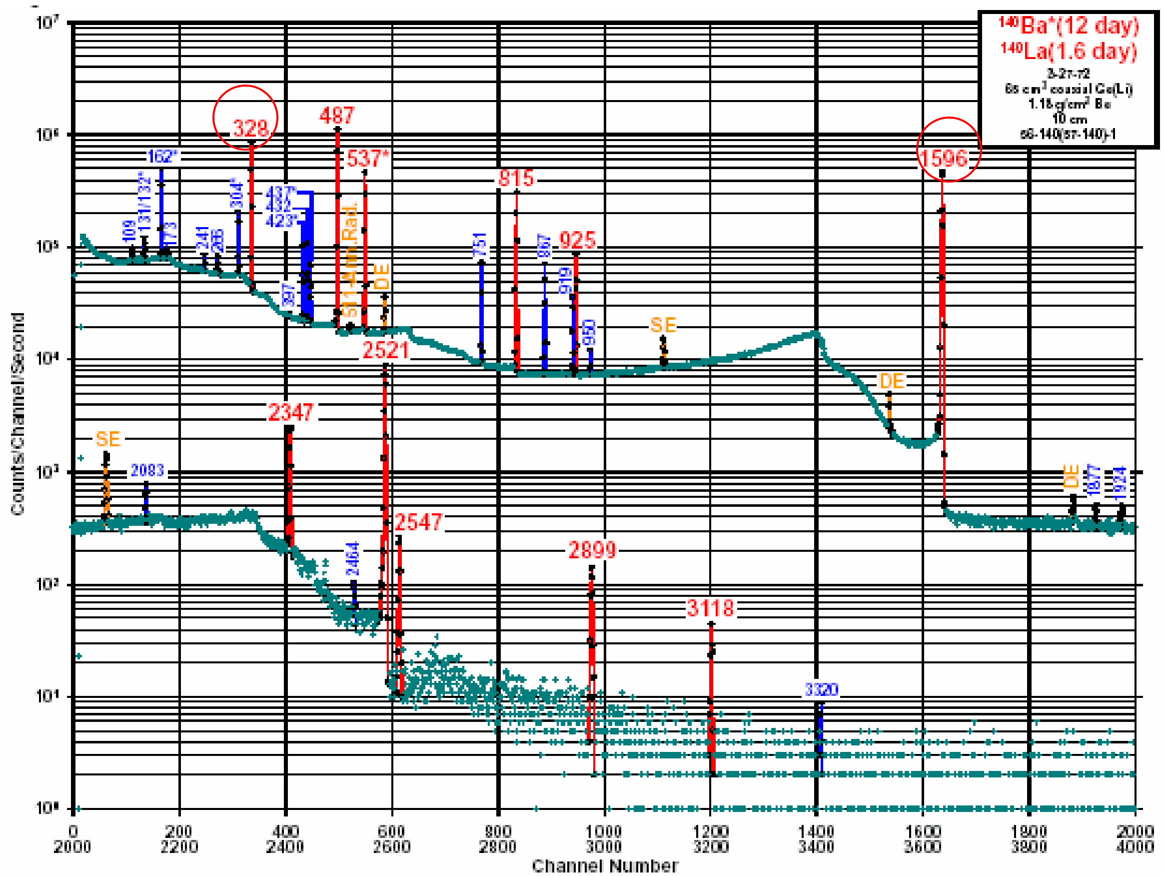
- ^{239}Np, ^{239}Pu
- fission prod. ^{90}Rb (100 s)
- fission prod. ^{140}Ba/^{140}La)

Natural radioactivity lines: ^{235}U, ^{238}U

Fissionable isotope
U-235 decay spectrum - INEEL Catalog

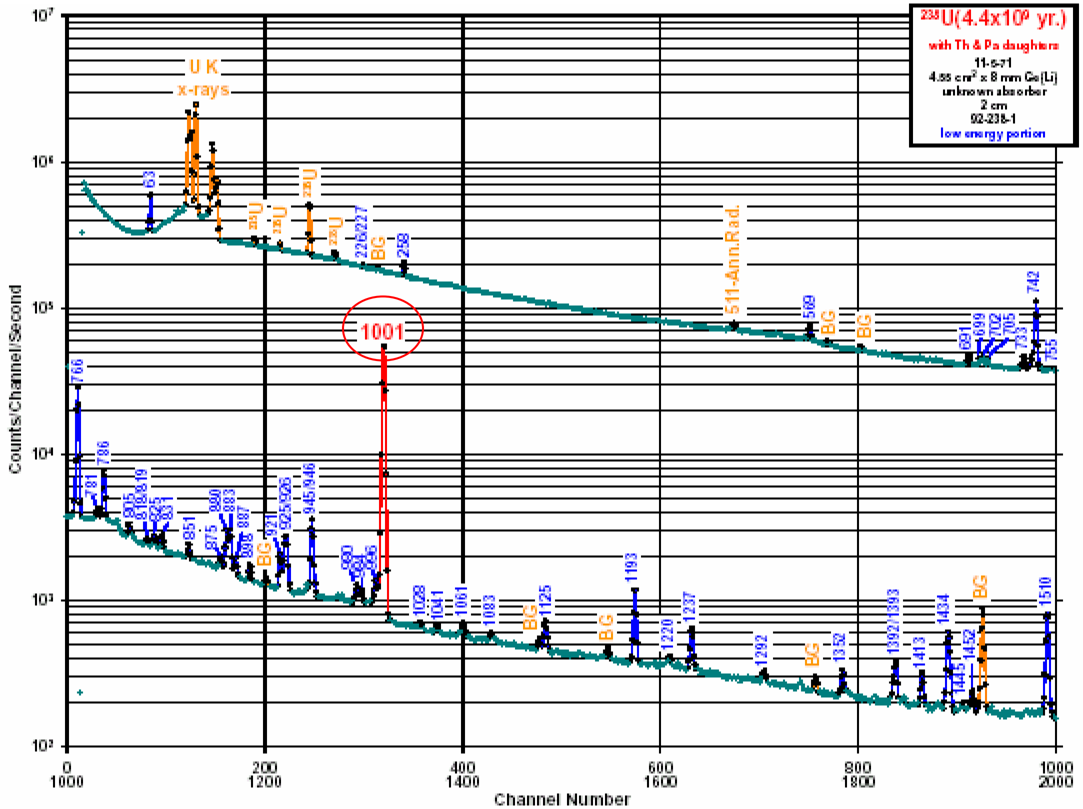


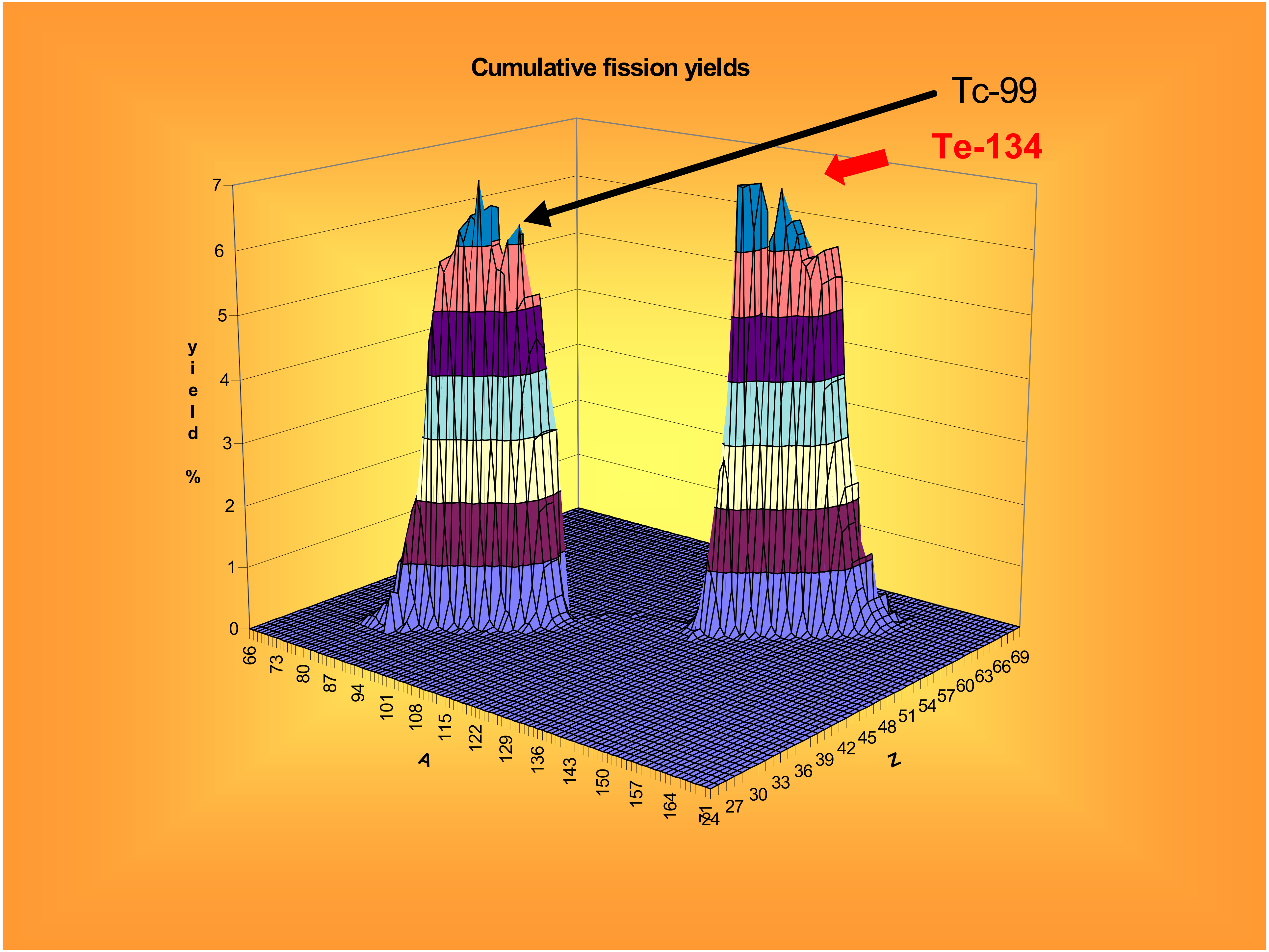
U-235 spont. fission >> Ba-140 / La-140 spectrum - INEEL Catalog



Non-fissionable isotope

U-238 decay spectrum - INEEL Catalog




Cumulative fission yields
Tc-99
Te-134
yield %
A
Z
0
1
2
3
4
5
6
7

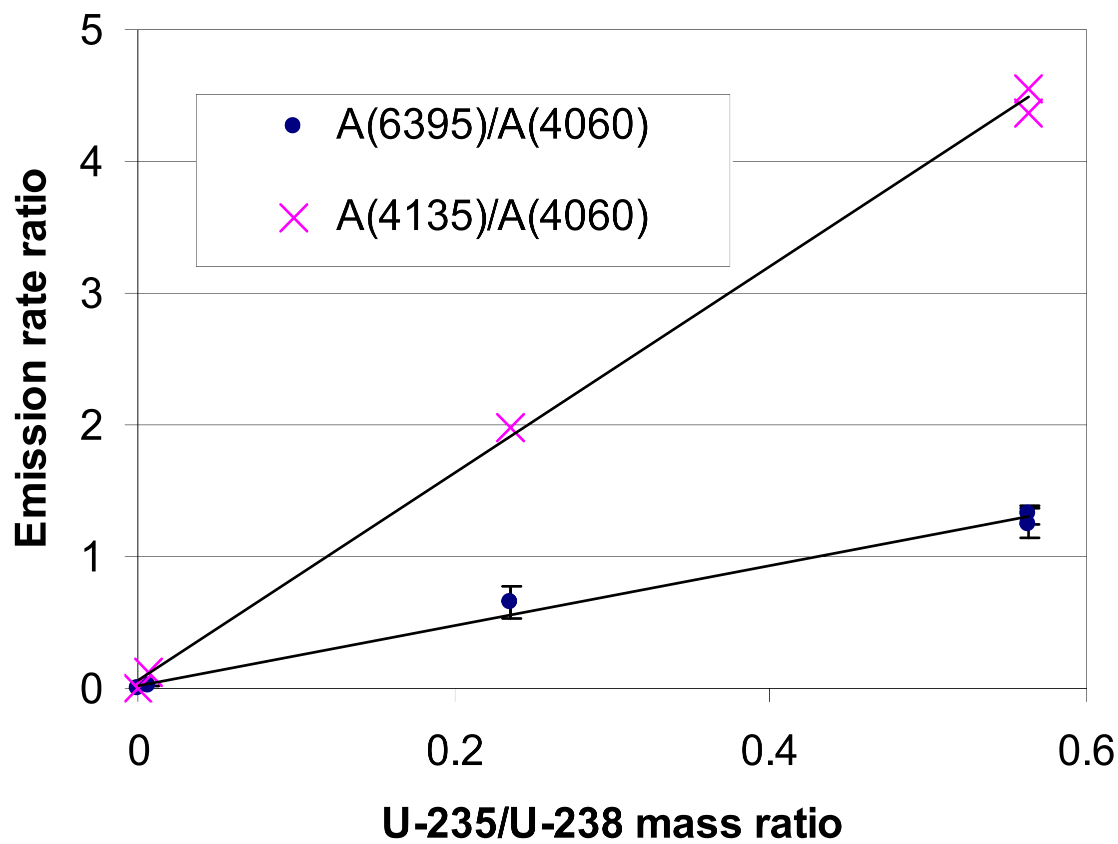
First uranium measurements

- Nat. uranil acetate and nitrate
  - PGAA
  - decay regularly after irradation
- enriched (19% and 36%) U_3O_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

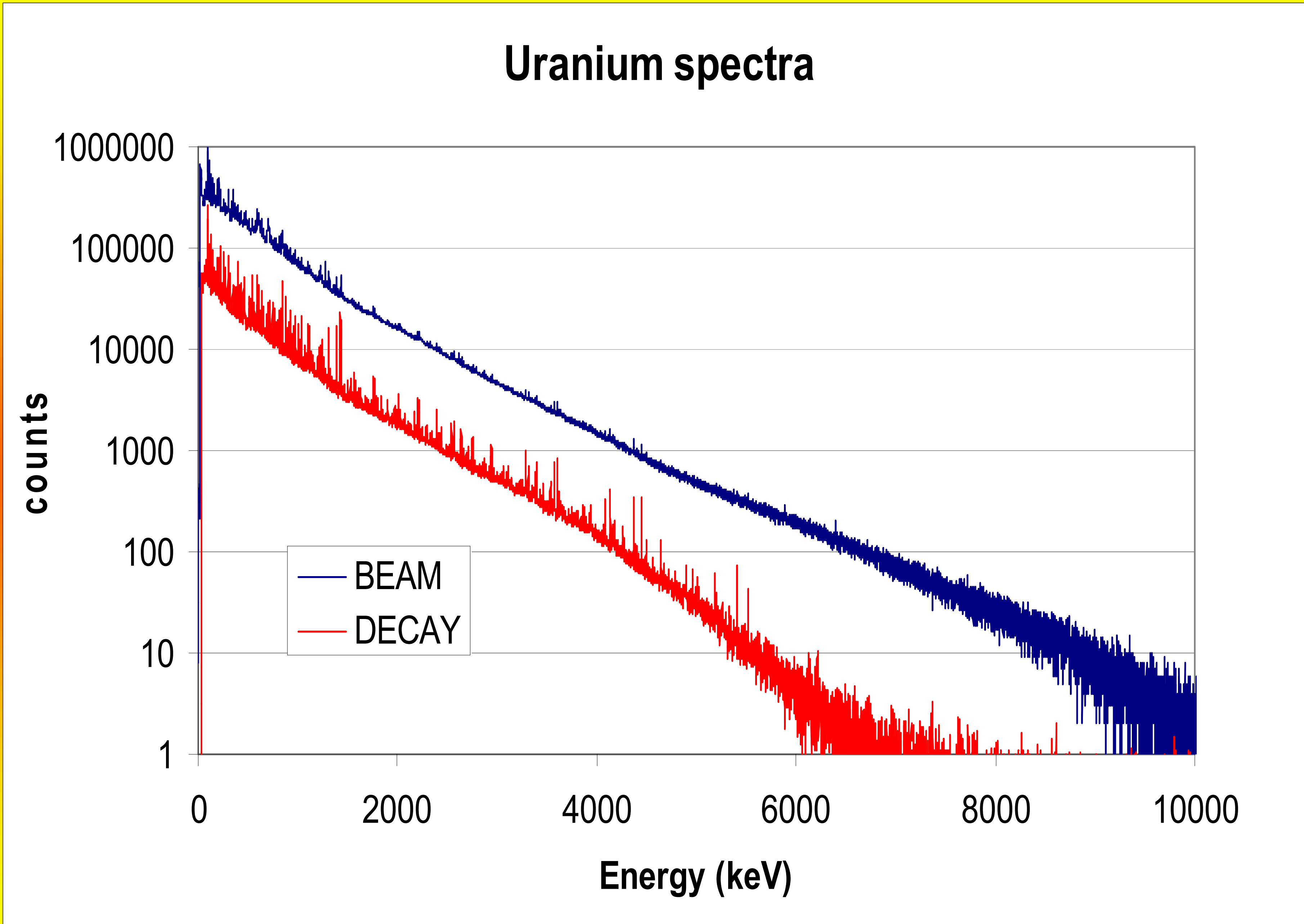


New uranium measurements

- Nat. uranil-acetate
  - PGAA
  - chopped beam PGAA
  - decay measurements regularly after irrad.
- enriched (95%) U_3O_8
  - PGAA
  - chopped beam PGAA
  - decay measurements regularly after irrad.

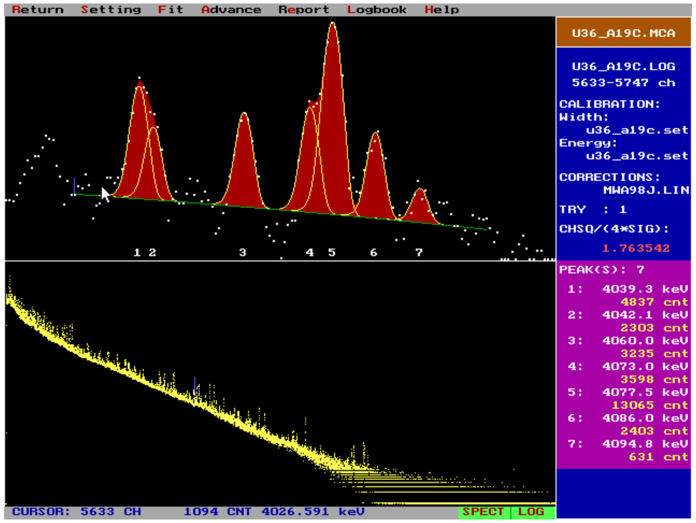
Irradiation of 50mg U_3O_8

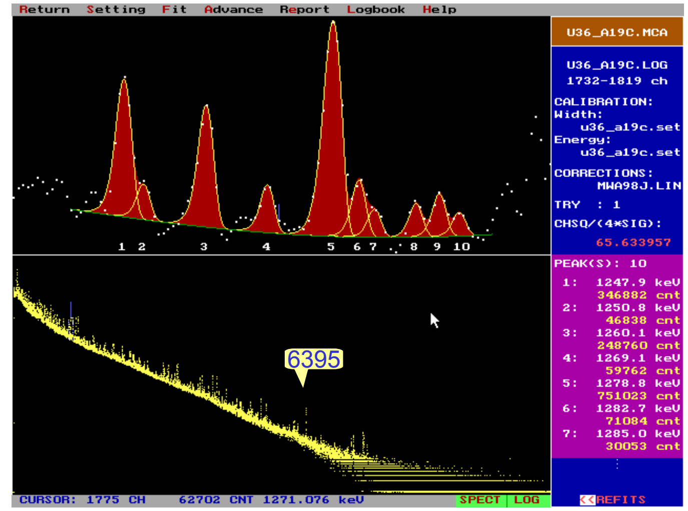
Background	1 cps
natural activity	~5 cps
decay	~90 cps
total in-beam	~700 cps


Uranium spectra
counts
1000000
100000
10000
1000
100
10
1
0
2000
4000
6000
8000
10000
Energy (keV)
BEAM
DECAY

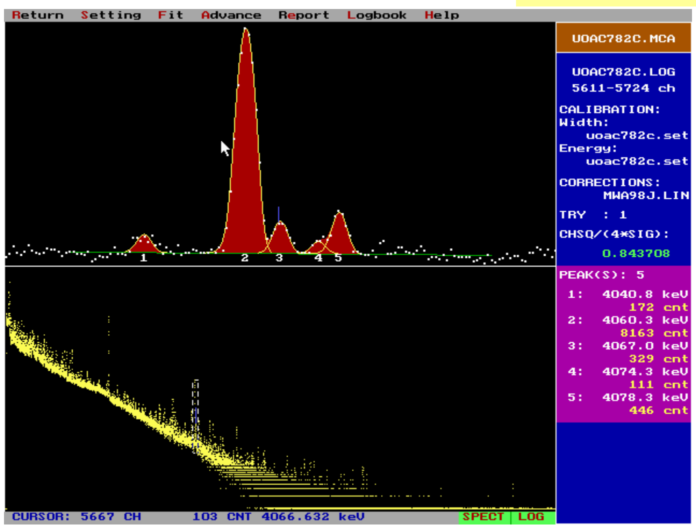
U-239 capture line - *4060 keV*   Te-134 fission line - *1279 keV*

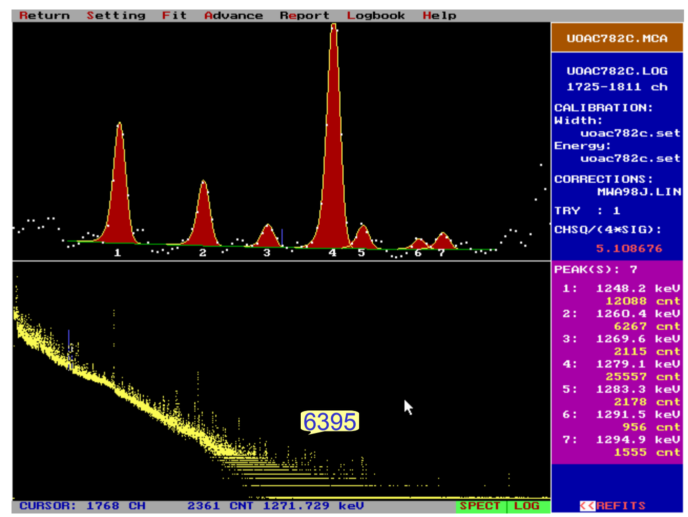
Enriched U (36% U-235)





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)
^{238}U	^{239}U	PGAA	4060	0	0.98	**0.192(2)**	–
	^{239}Np	Decay	278	2.36 d	0.074	**0.382(6)**	0.383(6)
	^{234}Th	Nat	92	$4.468 \cdot 10^{9}$ y	0.045(11)		
^{235}U	^{236}U	PGAA	6395	0	0.011	**0.0038(2)**	
	^{134}Te	Chopped PGAA	297	0	1.60	**0.22(2)**	0.22(2)
	^{134}Te	Chopped PGAA	1279	0	0.49	**0.20(1)**	0.22(2)
	^{140}Ba	Decay	537	12.75 d	0.0122	**0.066(3)**	0.064(1)
	^{235}U	Nat	186	$7.037 \cdot 10^{8}$ y	0.073		
^{99}Tc	^{99}Tc	PGAA	172		3.0	**16.61(15)**	-
	^{99}Tc	PGAA	223		0.24	**1.472(13)**	-
	^{99}Tc	PGAA	263		0.21	**1.425(12)**	-
	^{100}Tc	Decay	539	16 s	0.14	**1.604(14)**	-
	^{100}Tc	Decay	591	16 s	0.10	**1.296(11)**	
	^{99}Tc	Decay	89	211 000 y	4.3×10^{-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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