NON-DESTRUCTIVE PROMPT GAMMA ACTIVATION ANALYSIS OF CHIPPED STONE RAW MATERIAL

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ARCHAEOMETRY AT THE INSTITUTE OF ISOTOPES

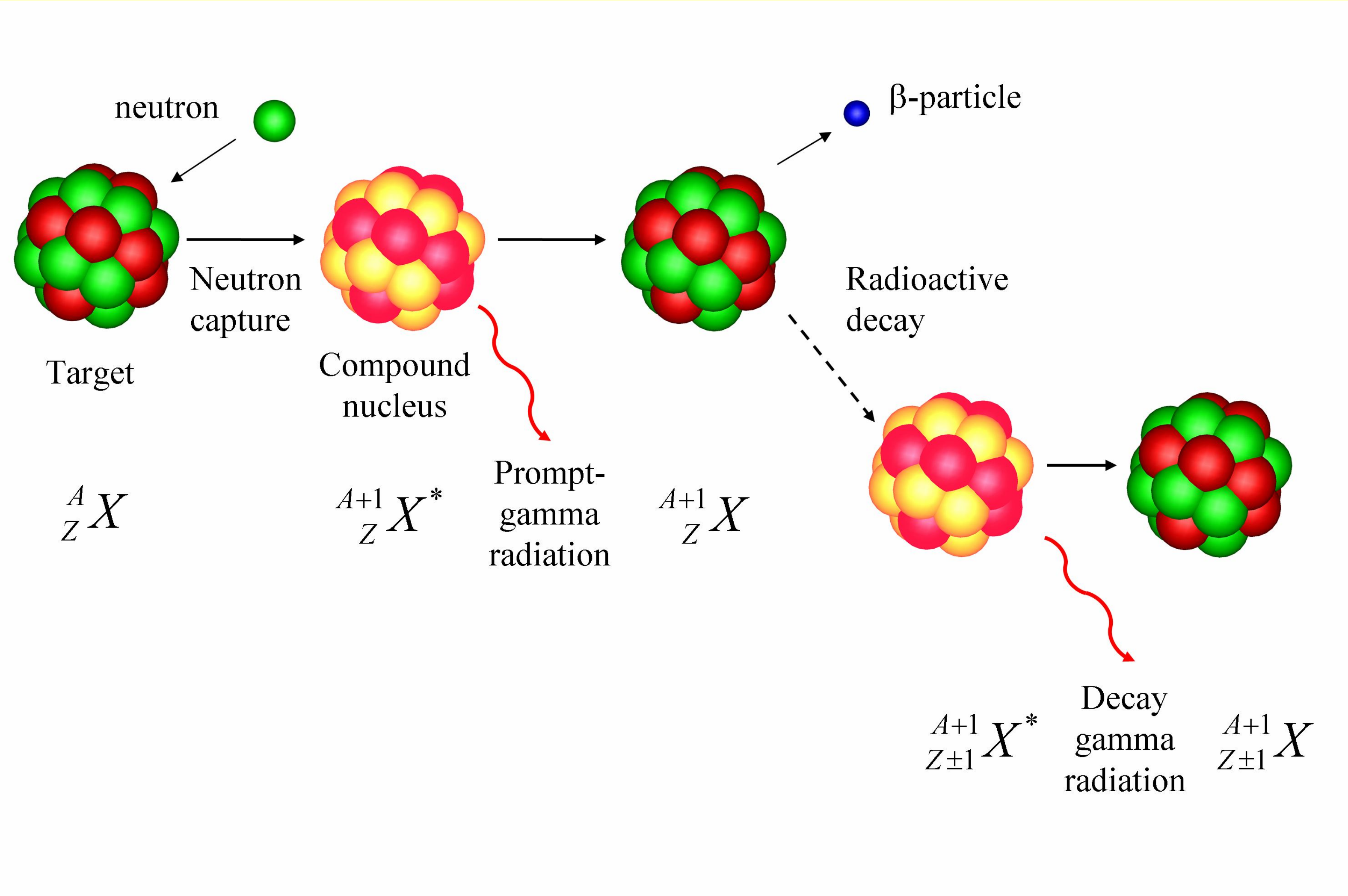
- **1996** - Prompt Gamma Activation Analysis (PGAA) at the Budapest Research Reactor
- **1998** - Applications in Archaeometry (metals, stones, pottery, glass)
- **2000** - Co-operation with the Hungarian National Museum (mostly stone tools)

- More than **150** objects from the Palaeolithic and Prehistoric collection of the HNM, mainly from the Central-European region
- Objects made of silex (flint, chert, radiolarite) or volcanite (porphyry, obsidian)
- Comparative geological material from ‘Lithotheca’ collection
- **AIM**: Classification of the objects; if possible – provenance study
- With PGAA major- and trace components measurable

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

→ **Macroscopically similar objects proved to be different !**

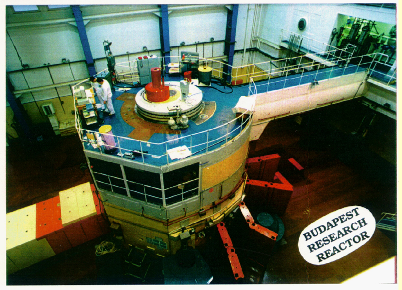
THE PHYSICAL BASIS OF PGAA

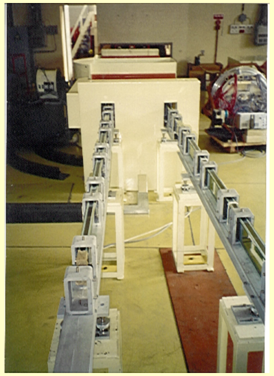


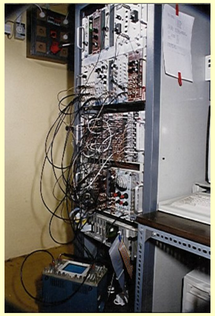
MAIN FEATURES OF PGAA

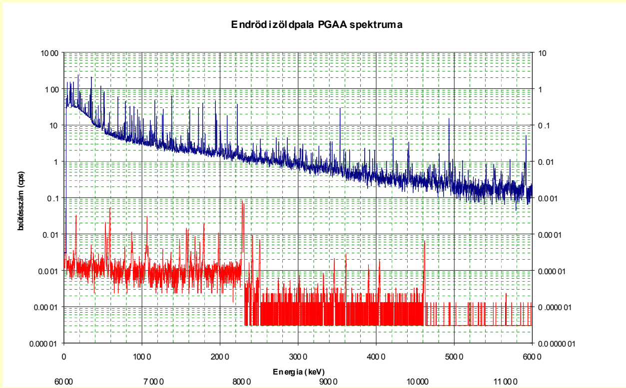
- 1996-2000 $2.5 \cdot 10^6$ $cm^{-2}s^{-1}$ thermal flux
- 2000- $5 \cdot 10^7$ $cm^{-2}s^{-1}$ cold neutron beam
- 2008- $1 \cdot 10^8$ $cm^{-2}s^{-1}$ cold neutron beam
- '**Multielement**' (main- and trace components)
- Minimal sample preparation
- **Non-destructive** (no residual radioactivity, no mechanical damage)
- Bulk composition independent of chemical and physical form of the sample

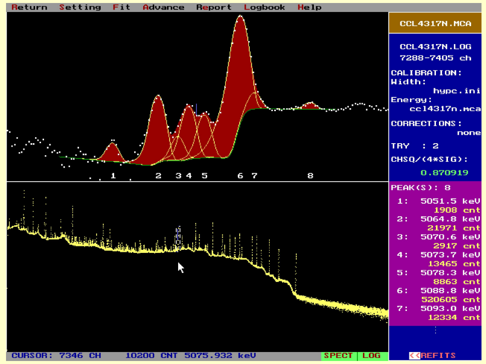
MAIN STEPS OF THE ANALYSIS





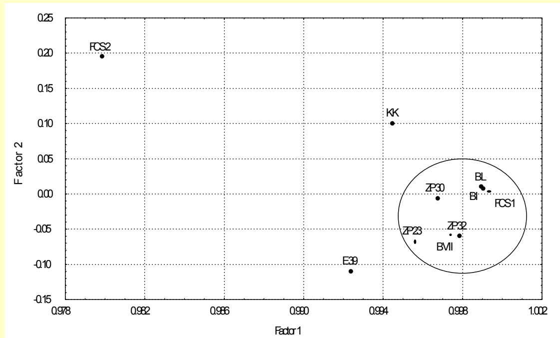






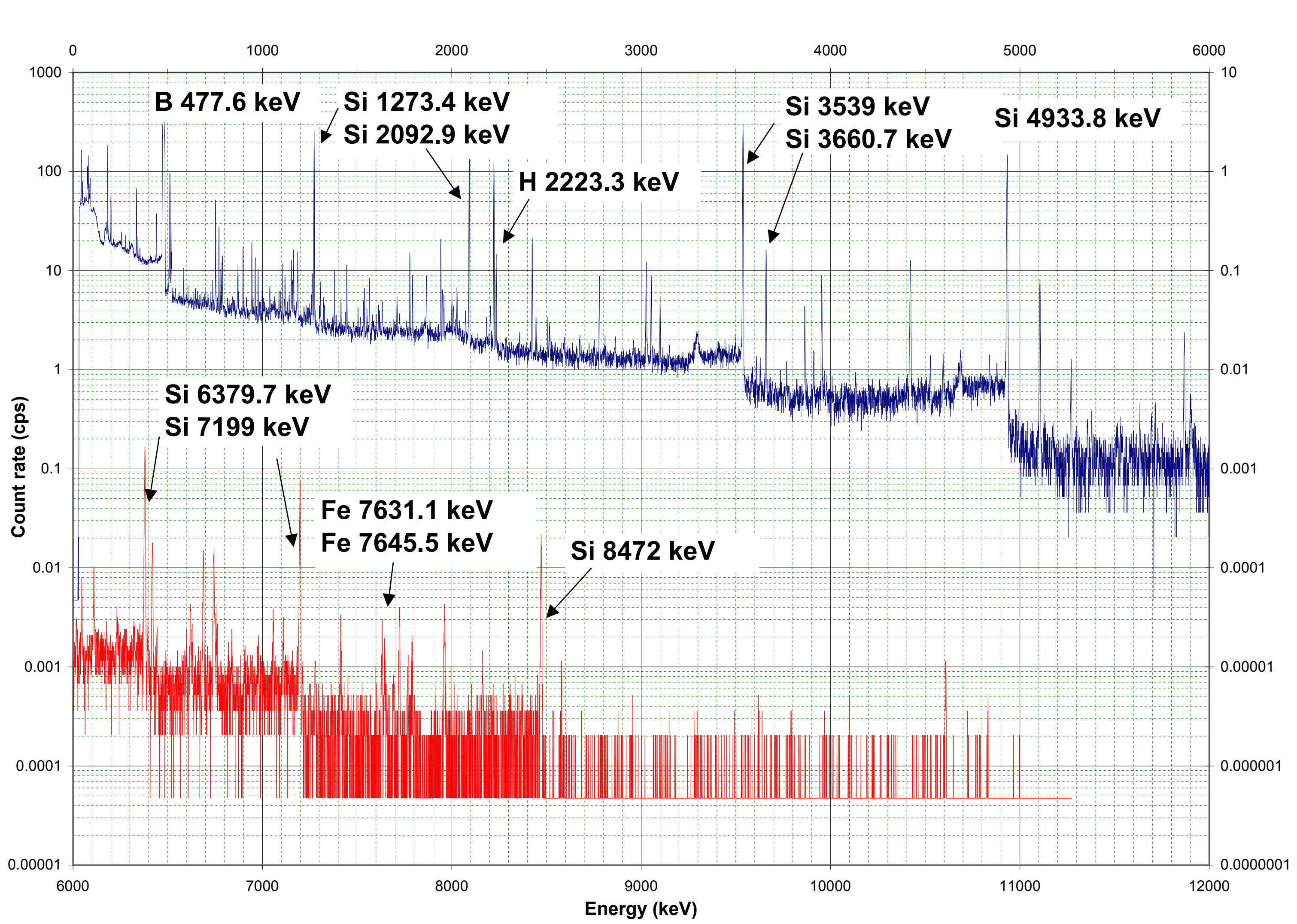
Spectrur C:\HYPC\SPECTRA\ARCHEO\ZOLDPALA\FV41I03C.MCA
Live Tim 3290.48

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						

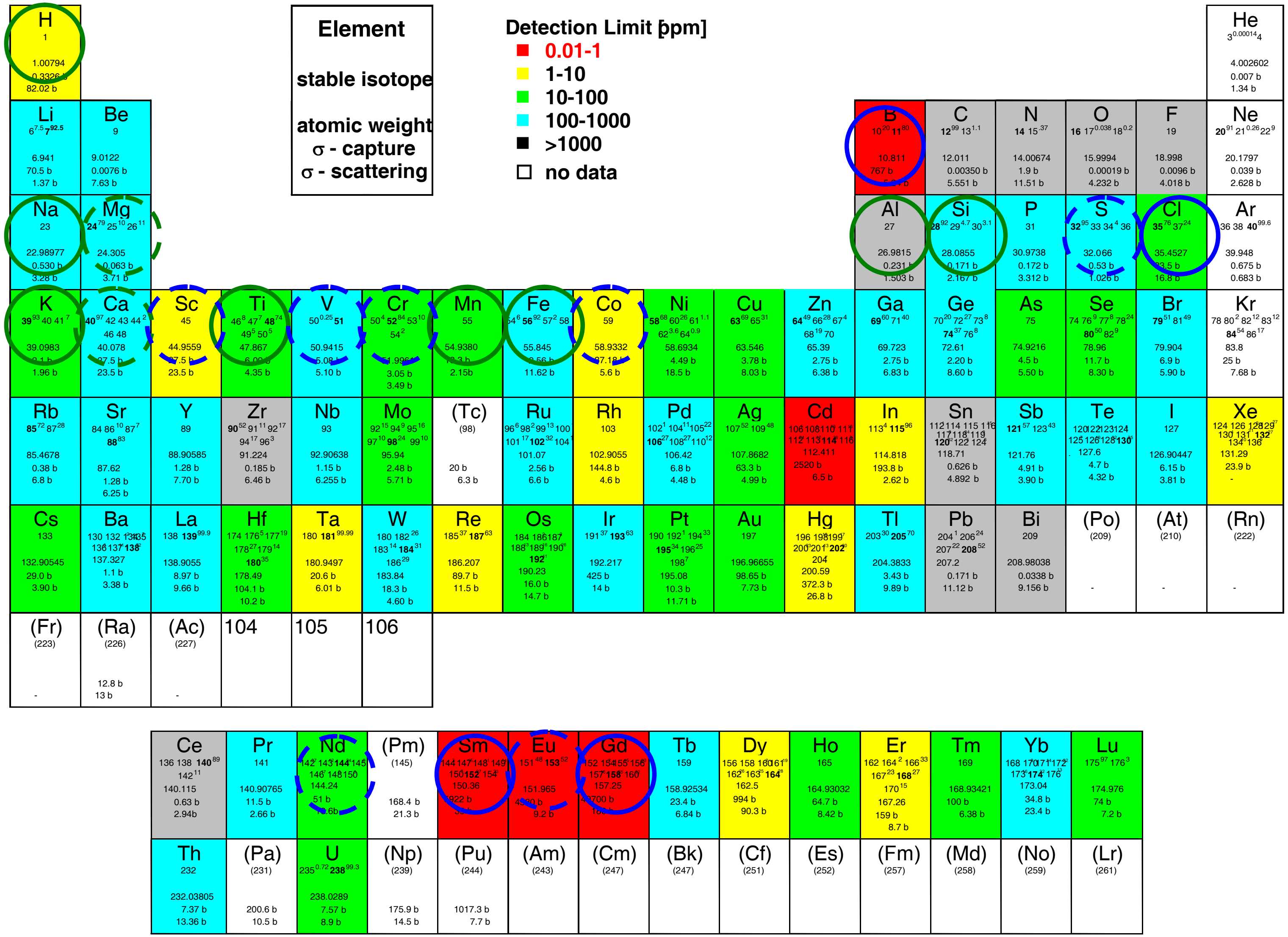


THE PGAA SYSTEM

PGAA SPECTRUM OF A GREY FLINT SAMPLE

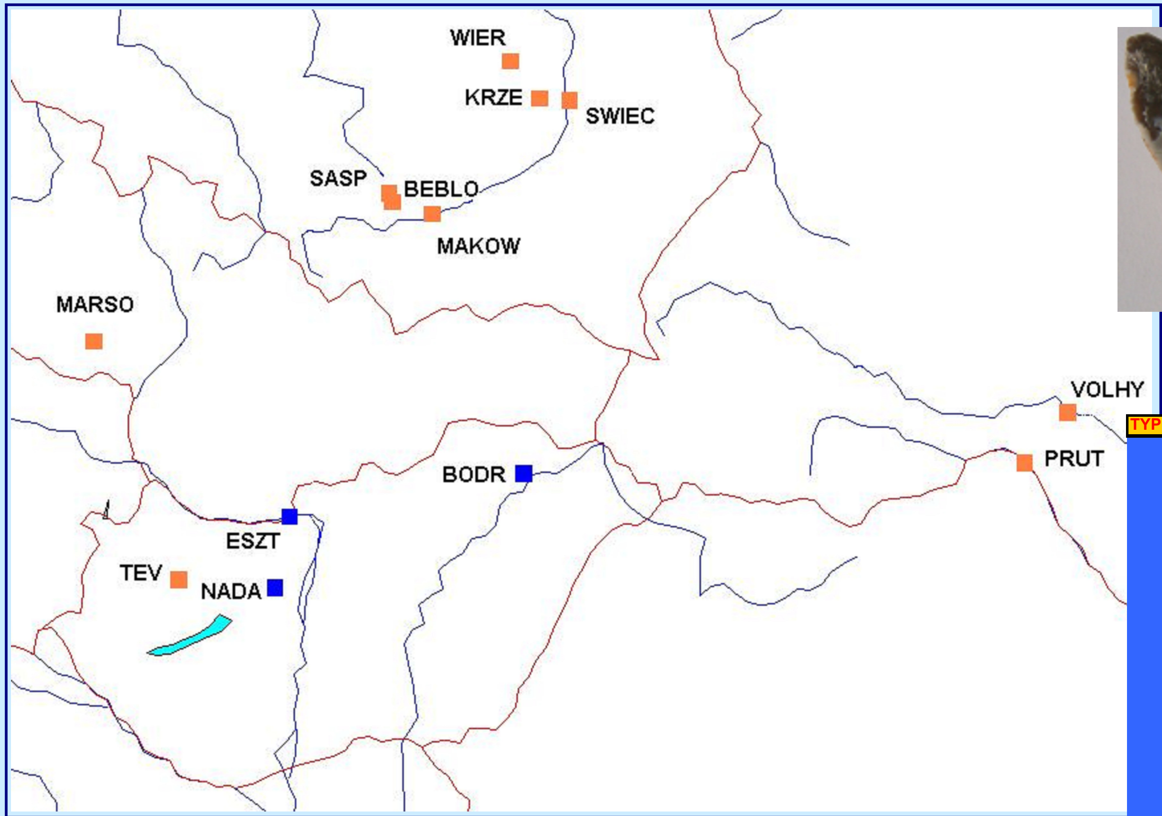


APPROXIMATE DETECTION LIMITS FOR BUDAPEST PGAA SYSTEM WITH COLD NEUTRON SOURCE



FLINT
(*Shallow water sedimentary siliceous rock*)

- **AIM**: Provenance study of **24** chipped stone objects from Upper Palaeolithic sites of Esztergom, Nadap and Bodrogkeresztúr
- **COMPARATIVE MATERIAL**: HNM Lithotheca (outcrops of Hungary, Bohemia, Poland, Romania and Ukraine)
- **COMPOSITION: >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

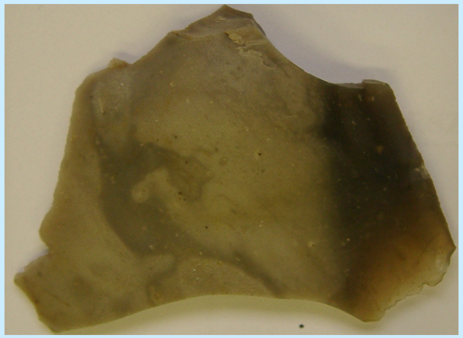


F1

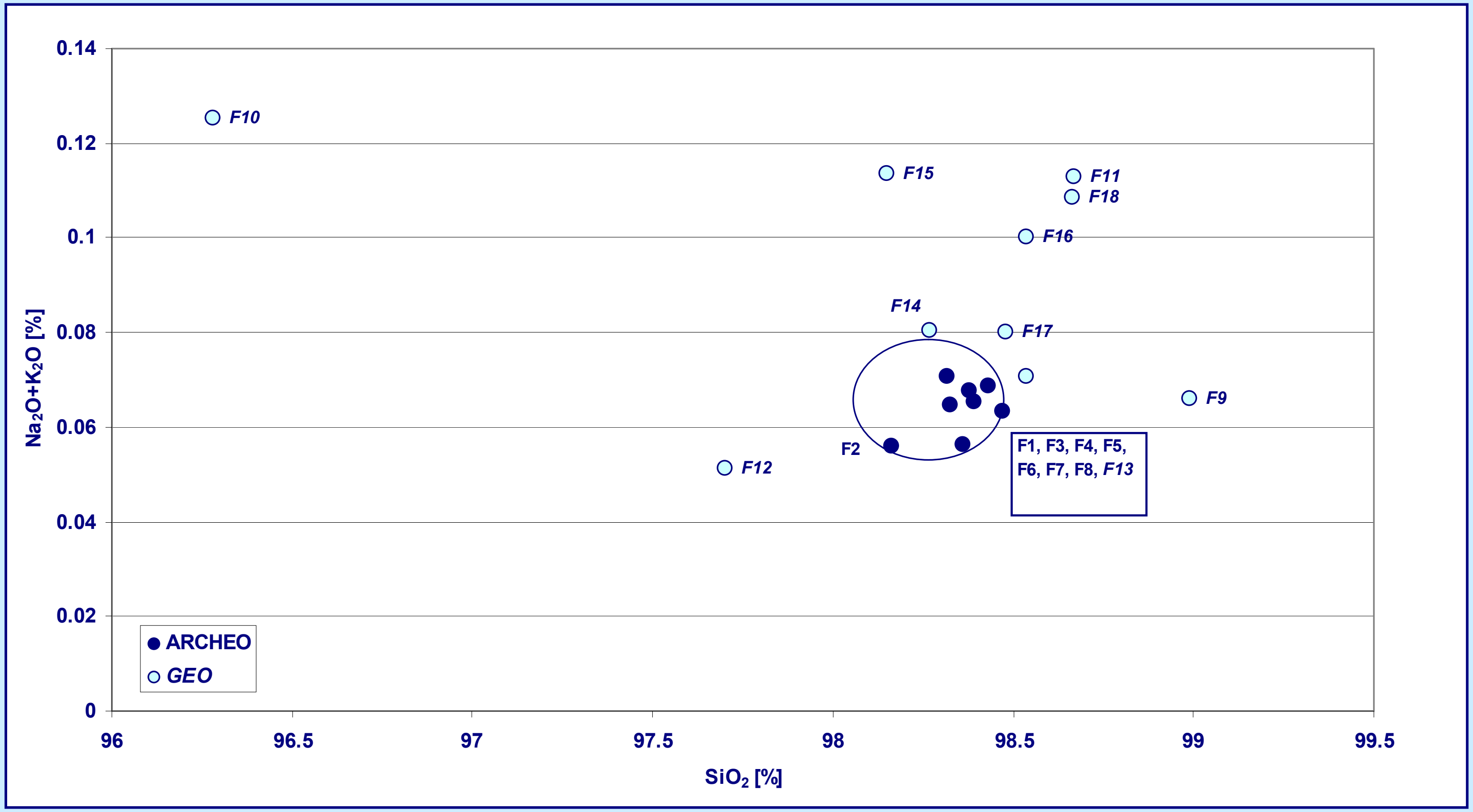
F8

TYPE	SAMPLE	Inv. Nr.		CODE NE
FLINT ARC	BODROGKERESZTÚR 1	PB 83/643 (1) 17	Volhynian/Prut Flint	FA01
	BODROGKERESZTÚR 2	PB 83/643 (2) 18	Volhynian/Prut Flint	FA02
	ESZTERGOM 1	PB 88/234 (1) 14	Prut Flint	FA03
	ESZTERGOM 2	PB 88/234 (2) 15	Prut Flint	FA04
	ESZTERGOM 3	PB 88/234 (3) 16	Prut Flint	FA05
	NADAP 1	PB 86/47 (1) 11	Erratic Flint	FA06
	NADAP 2	PB 86/47 (2) 12	Erratic Flint	FA07
	NADAP 3	PB 86/47 (3) 13	Erratic Flint	FA08
	JÁSZLADÁNY 17.	Ö 35/1939.46	grey flint	FA09
	KUNSZENTMÁRTON	Ö 25/1928.29	grey flint	FA10
	TISZAPOLGÁR	Ö 53.35.170	grey flint	FA11
	KÁLLÓ 1	Ö 59.10.8	grey flint	FA12
	KÁLLÓ 2	Ö 59.10.12	grey flint	FA13
	PESKŐ BARLANG 1	PB 914	grey flint	FA14
	PESKŐ BARLANG 2	PB 915	grey flint	FA15
	PESKŐ BARLANG 3	PB 55/12	grey flint	FA16
	KUP-EGYES	–	grey flint	FA17
	MIKOLA 2	–	flint	FA18
	ZEIT 1	–	Silex	FA19
	BOINESTI 1	BM 4829	silex	FRA01
	BUSAG 2	BM 33632	silex	FRA02
	CALINESTI II/2		flint	FRA03
	CALINESTI DSM 5	SM 42664	silex	FRA04
	COSAUTI 1	-	"obsidian"	FRA05
FLINT GEO	SASPÓW		Jurassic Kraków Flint	FG01
	SWIECIECHÓW		Swieciechów Flint	FG02
	NAGYTEVEL		Tevel Flint	FG03
	MAKÓW	L89/107	Erratic (Baltic) Flint	FG04
	PRUT	L86/248	Prut Flint	FG05
	VOLHYNIA		Volhynian Flint	FG06
	WIERZBYCZA		Chocolate Flint	FG07
	BEBLO		Jurassic Kraków Flint	FG08
	KRZEMIONKI		Krzemionki Flint	FG09
	MARSOVICE		Les Type Chert	FG10
	KANIV-AMAZONKA 1		grey flint	FG11
	KANIV-AMAZONKA 2		grey flint	FG12
	BUGOYOVO-BUCHAK I/1		grey flint	FG13
	BUGOYOVO-BUCHAK I/2		grey flint	FG14
	BUGOYOVO-BUCHAK I/3		grey flint	FG15
	BUGOYOVO-BUCHAK I/4		grey flint	FG16
	BUGOYOVO-BUCHAK II/1		grey flint	FG17
	BUGOYOVO-BUCHAK II/2		grey flint	FG18
	BUGOYOVO-BUCHAK II/3		grey flint	FG19
	BUGOYOVO-BUCHAK TALBERG		grey flint	FG20
	BELAEVKA-ZELEZNY HUTOR 1		grey flint	FG21
	BELAEVKA-ZELEZNY HUTOR 2		grey flint	FG22
	BELAEVKA-ZELEZNY HUTOR 3		grey flint	FG23
	TORUN 1	L 86/218	grey flint	FG24
	TORUN 2	L 86/218B	grey flint	FG25
	OVIDIOPOL		grey flint	FG26

F3

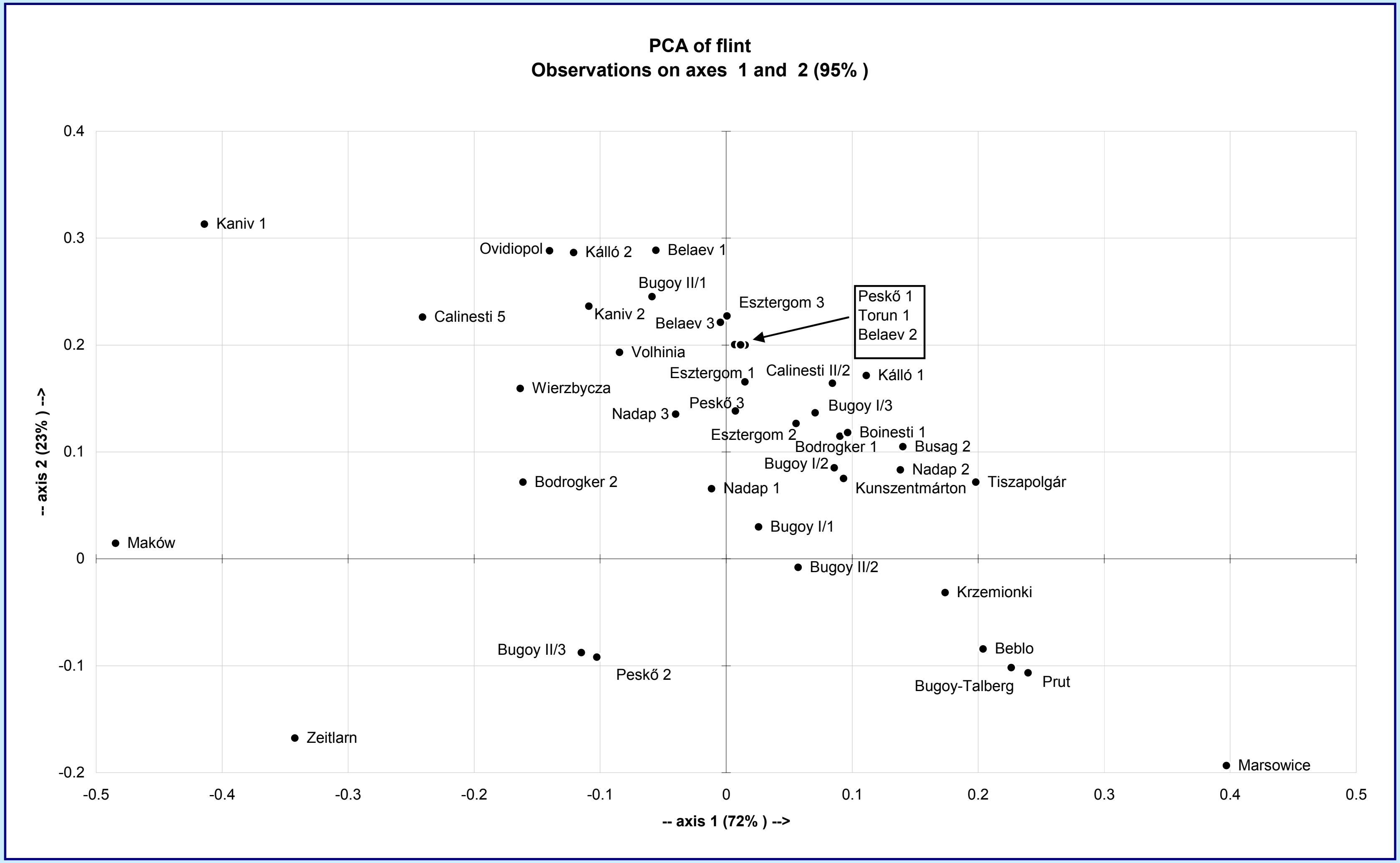


F13



RESULTS

- The archaeological samples have very similar composition
- They are different from most of the geological references
- Most similar to the raw material from **Prut** and **Volhynia**
- More samples must be measured



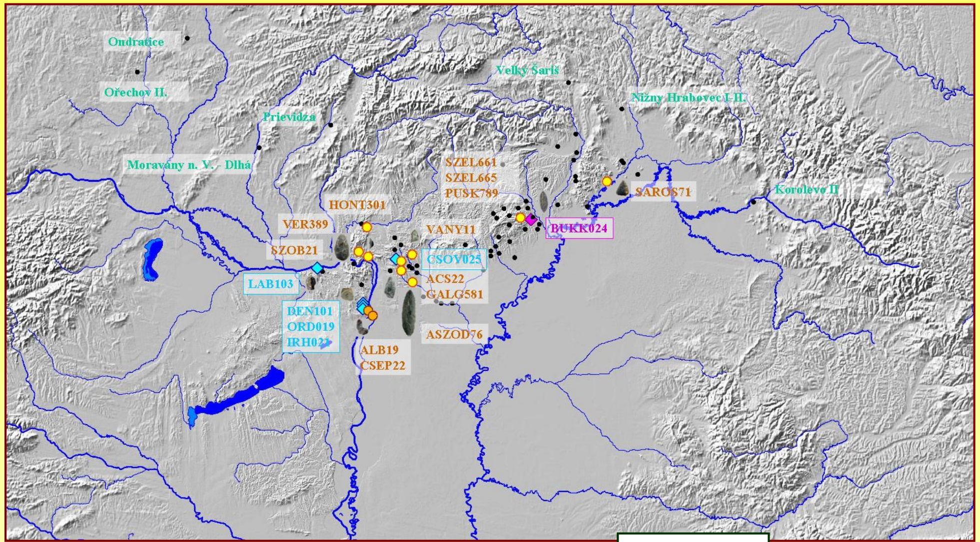
PCA of flint
Observations on axes 1 and 2 (95%)
-- axis 2 (23%) -->
-- axis 1 (72%) -->
0.4
0.3
0.2
0.1
0
-0.1
-0.2
-0.5
-0.4
-0.3
-0.2
-0.1
0
0.1
0.2
0.3
0.4
0.5
Kaniv 1
Ovidiopol
Kálló 2
Belaev 1
Bugoy II/1
Calinesti 5
Kaniv 2
Esztergom 3
Belaev 3
Peskő 1
Torun 1
Belaev 2
Volhinia
Esztergom 1
Calinesti II/2
Kálló 1
Wierzbycza
Peskő 3
Nadap 3
Bugoy I/3
Esztergom 2
Boinesti 1
Bodrogker 1
Busag 2
Bugoy I/2
Nadap 2
Bodrogker 2
Nadap 1
Kunszentmárton
Tiszapolgár
Bugoy I/1
Maków
Bugoy II/2
Krzemionki
Bugoy II/3
Beblo
Peskő 2
Bugoy-Talberg
Prut
Zeitlarn
Marsowice

PCA of Chipped Stones total, selection of siliceous raw materials
Observations on axes 1 and 3 (88%)
1
0.5
0
-0.5
-1
-1.5
11
12
13
14
15
16
17
18
3rd Principal Component
1st Principal Component
Calinesti 7, menilite
Flint
Porphyry
Hornstone
Radiolarite
LQ
Obsidian
Other

'SZELETIAN' FELSITIC PORPHYRY, HORNSTONE, RADIOLARITE

- **SFP** classical Palaeolithic raw material in Hungary (Szeleta-cave, Bükk mts.) Previously identified as flint...
- Also in Danube-Bend, Börzsöny, Cserhát, Zemplén mts.
- **COMPARATIVE: Hornstone** and **Radiolarite** (Buda hills, Trans-Danubian mts. - **SiO_2 > 90%)**

TYPE	SAMPLE	Inv. Nr.		CODE NE
PORPH ARCH	BECSKE	–	Seletian Felsitic porhyry	PA01
	ERDŐKÜRT	–	Seletian Felsitic porhyry	PA02
	PILISMARÓT-DIÓS	PB 81/60	Seletian Felsitic porhyry	PA03
	JÁSZFELSŐSZENTGYÖRGY	PB 93/152	Seletian Felsitic porhyry	PA04
	DEBERCSÉNY	PB 87/167	Seletian Felsitic porhyry	PA05
	CSOBÁNKA	PB 409	Seletian Felsitic porhyry	PA06
	BAJÓT	PB 557	Seletian Felsitic porhyry	PA07
	LEGÉND-ROVNYA	–	Seletian Felsitic porhyry	PA08
	VANYARC-TOVI	–	Seletian Felsitic porhyry	PA09
	VANYARC-DOLINA	–	Seletian Felsitic porhyry	PA10
	ZSÁMBOK	–	Seletian Felsitic porhyry	PA11
	ACSA	PB 2001/22	Seletian Felsitic porhyry	PA12
	SZOB	PB 21/1935	Seletian Felsitic porhyry	PA13
	HONT	PB 99/301	Seletian Felsitic porhyry	PA14
	VERŐCE	PB 86/383	Seletian Felsitic porhyry	PA15
	GALGAGYÖRK	PB 2001/581	Seletian Felsitic porhyry	PA16
	SZELETA 1	PB 661	Seletian Felsitic porhyry	PA17
	SZELETA 2	PB 665	Seletian Felsitic porhyry	PA18
	SÁROSPATAK	PB 71/2	Seletian Felsitic porhyry	PA19
	ASZÓD	PB 76/1	Seletian Felsitic porhyry	PA20
	PUSKAPOROS	PB 789	Seletian Felsitic porhyry	PA21
	VANYARC 11	–	Seletian Felsitic porhyry	PA22
	KEHNEC 1	464 (124/55)	Seletian Felsitic porhyry	PA23
	CECEJOVCE	CE 24	Seletian Felsitic porhyry	PA24
	NIZNY HRABOVEC	NH I+II	Seletian Felsitic porhyry	PA25
	KOROL 2A	–	Glassy Porhyry ?	PA26
	KOROL 3	KOR 75.6.3	Glassy Porhyry ?	PA27
	BÜKKSZENTLÁSZLÓ	L 86/024	Seletian Felsitic porhyry	PG01
HORN ARCH	ALBERTFALVA	–	hornstone	HA01
	CSEPEL	–	hornstone	HA02
HORN GEO	ÖRDÖG-OROM	L 86/019	hornstone	HG01
	IRHÁS-ÁROK	L 86/021	hornstone	HG02
	CSŐVÁR	L 86/025	hornstone	HG03
	DENEVÉR U	L 87/101	hornstone	HG04
RADIO ARCH	TATA	PB 2001/140	radiolarite	RA01
	SÁGVÁR 1	PB 61/1950.29	radiolarite	RA02
	REMETEA SOMOS I/2	BM 1885	radiolarite	RA03
RADIO GEO	LÁBATLAN	L 86/103	radiolarite	RG01
LQ ARCH	GALGAGYÖRK-CSONKÁS	–	limnic quartzite	LQA01
	MIKOLA 3		limnic quartzite	LQA02
	BOINESTI 2	BM 5147	limnic quartzite	LQRA01
	BOINESTI 3	BM 4531	limnic quartzite	LQRA02
	CALINESTI II/2	SM 44906	limnic quartzite	LQRA03
	CALINESTI HURCA 1	SM 40939	limnic quartzite	LQRA04
	CALINESTI DSM 10	SM 41617	limnic quartzite	LQRA05
JASPER ARC	BUSAG 3	BM 33634	jasper	JRA01
	CALINESTI DSM 4	SM 42773	jasper	JRA02
	CALINESTI DSM 8	SM 41603	jasper	JRA03
	REMETEA SOMOS I/1	BM 5173	jasper	JRA04
	REMETEA SOMOS I/3	SM 1191	jasper	JRA05

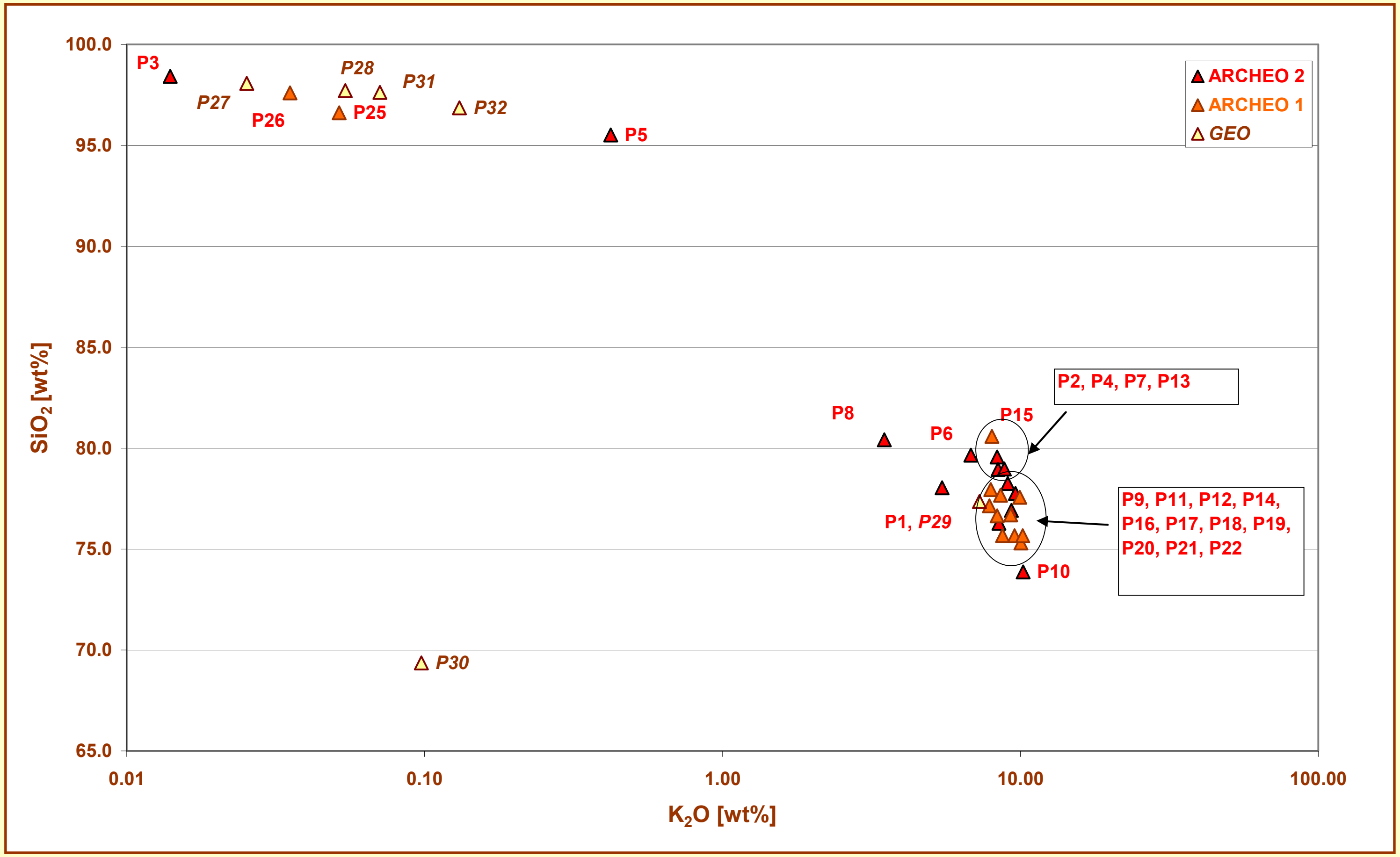


P31

P22

P18

P21



RESULTS

• Siliceous Radiolarite and Hornstone **SiO_2 > 90%**) can be distinguished from Szeletian Porphyry (**70-80% SiO_2**)

• Archaeological pieces from Cserhát mts. lies **>100 km** from the geological source

OBSIDIAN

Volcanic glass, **~75% SiO_2**, classical subject of archaeometry studies

- **We have investigated**

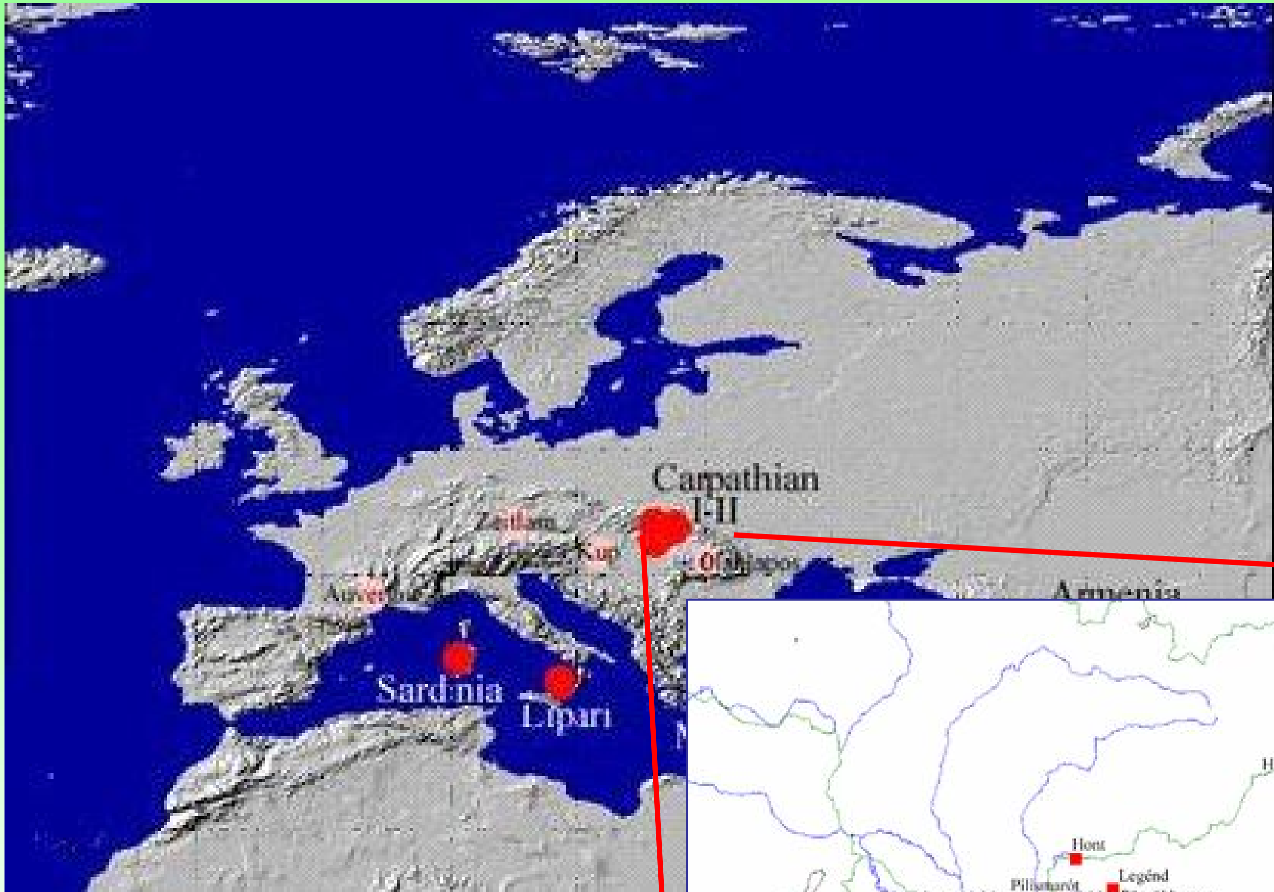
43 Archaeological pieces

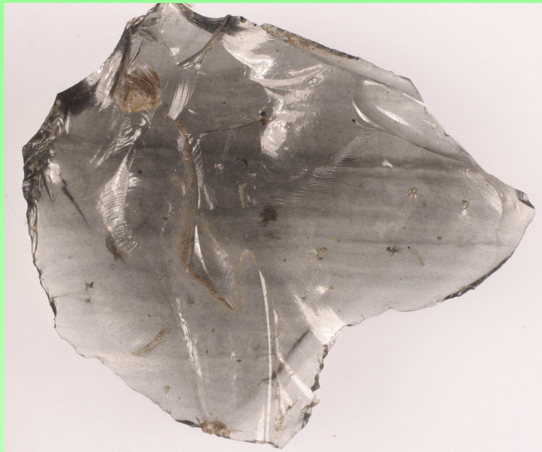
18 Comparative geological material

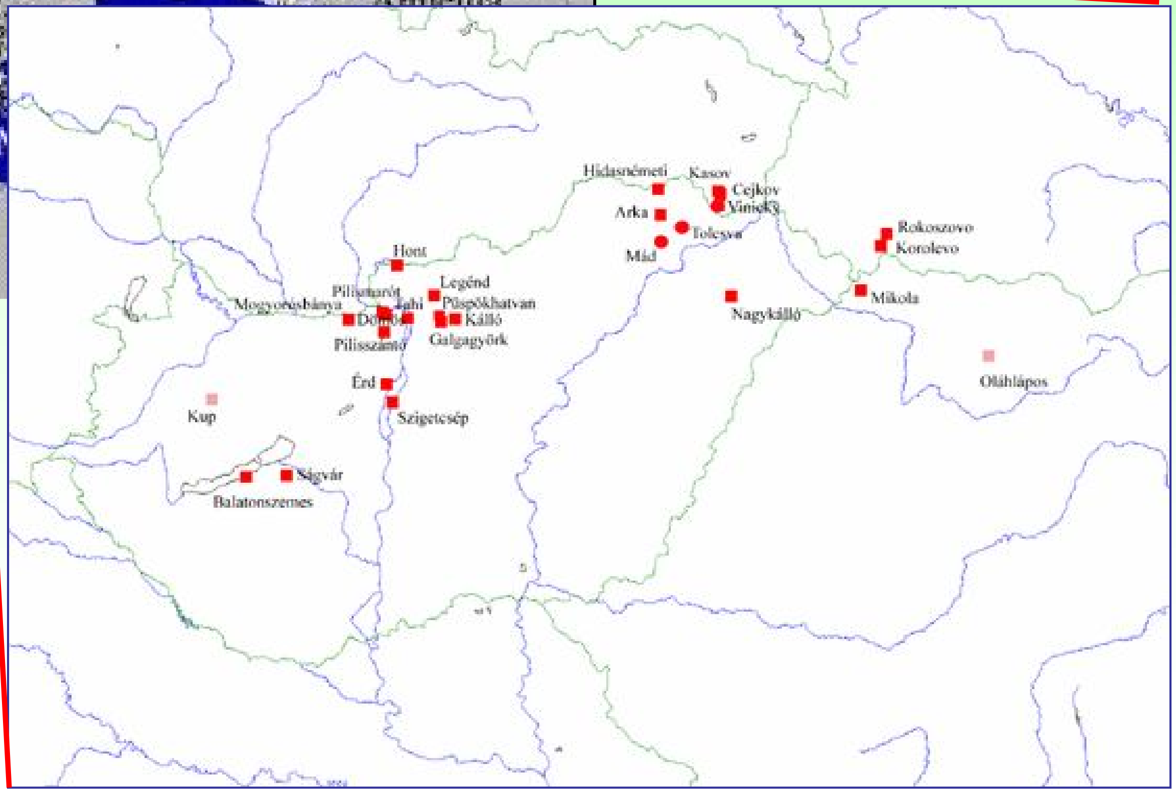
(Central Europe / Carpathian I,II; Mediterranian)

3 Modern slag

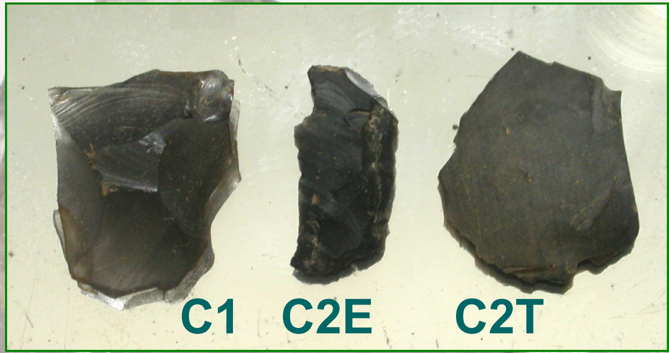
12 Other obsidian-like samples


Carpathian
I-II
Sardinia
Lipari

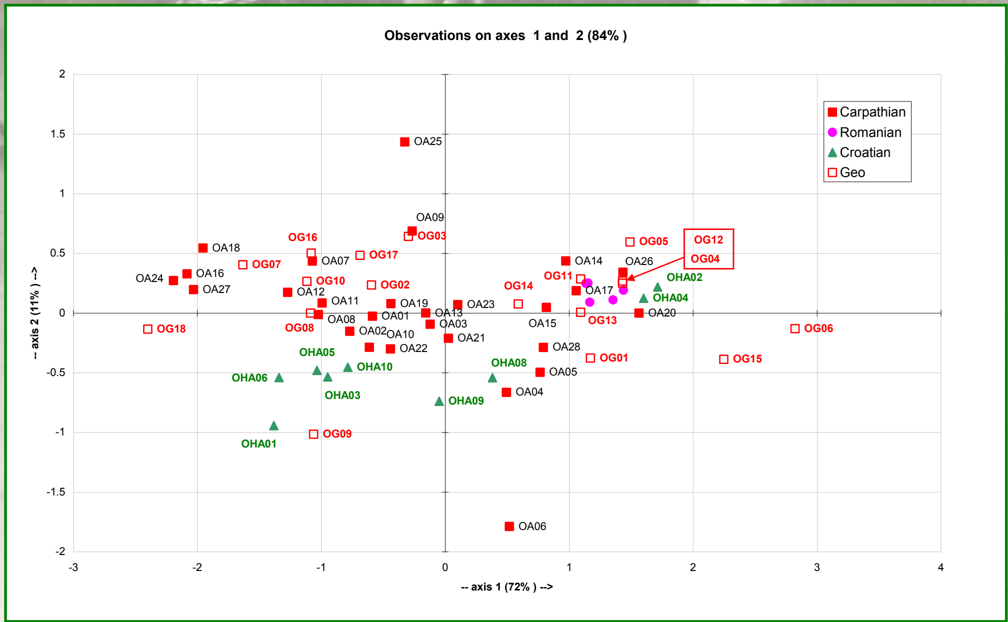



Hidasnémeti
Kasov
Cejkov
Arka
Tolcsva
Mád
Rokoszovo
Korolevo
Hont
Legénd
Mogyorósbánya
Tahi
Püspökhatvan
Kálló
Pilisszántó
Galgagyörk
Nagykálló
Mikola
Oláhlápos
Érd
Kup
Szigetcsép
Ságvár
Balatonszemes

C2Tr

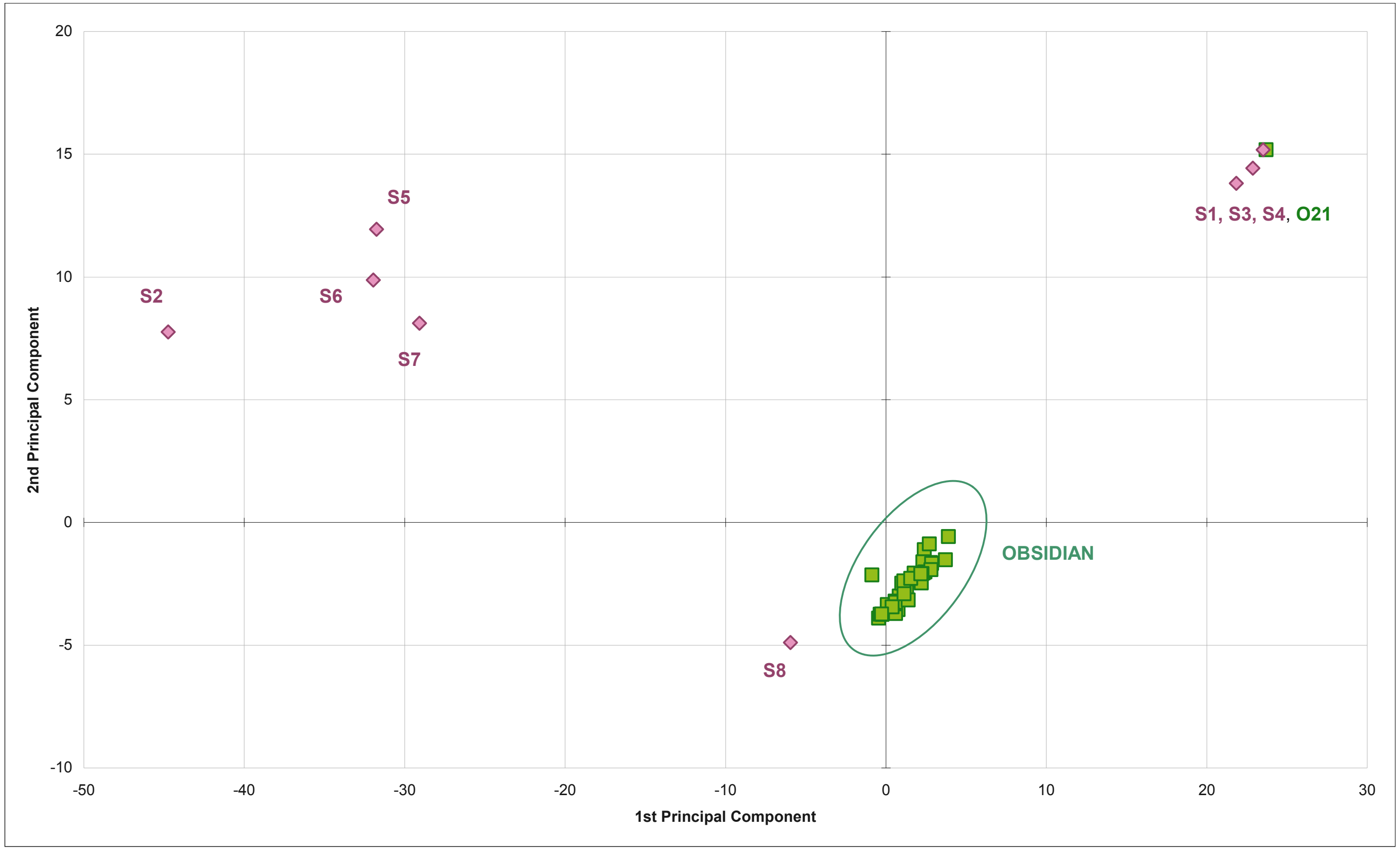


C1



TYPE	SAMPLE	Inv. Nr.		CODE NEW
OTHER	ZEIT 2	–	Slag ?	SA01
	KUP		Slag	SA02
	OLAH1		Slag ?	SA03
	OLAH 2		Slag ?	SA04
	KOROL 1	KOR 75.4286	Andesite	SA05
	BOINESTI 4	BM 4527	Andesite	SRA01
	HALMEU-VAMA 1	SM 44908	Andesite	SRA02
	CALINESTI HURCA 2	SM 40942	Menilite	SRA03
	CALINESTI DSM 6	SM 42387	Menilite ?	SRA04
	CALINESTI DSM 7	SM 41781	Menilite ?	SRA05

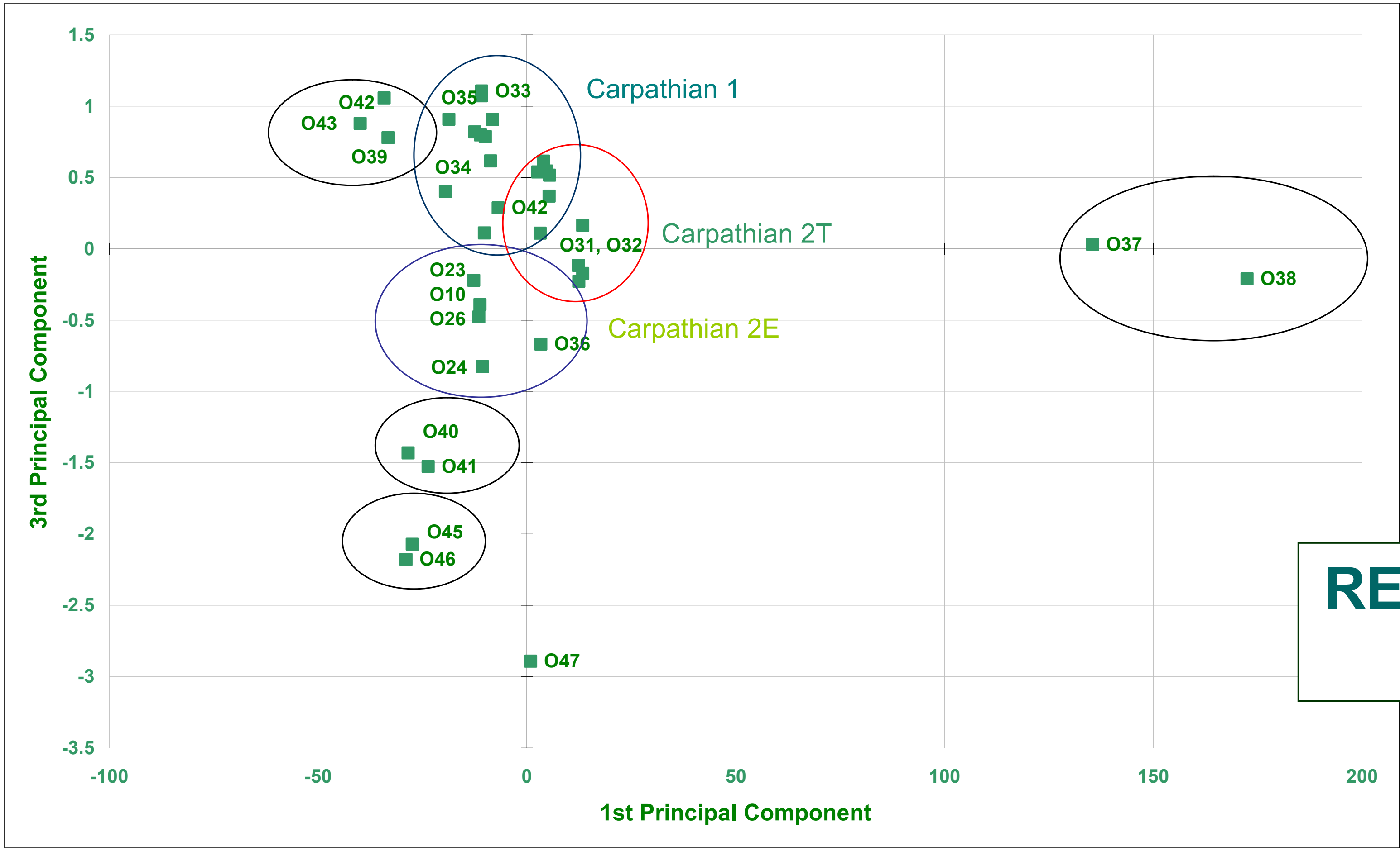
TYPE	SAMPLE	Inv. Nr.		CODE
OBS ARCH	MEGYASZÓ 1	94/737	mahagonian	OA01
	MEGYASZÓ 2	94/437	mahagonian	OA02
	MEGYASZÓ 3	95/373	mahagonian	OA03
	MEGYASZÓ 4	95/264	mahagonian	OA04
	MEGYASZÓ 5	95/263	mahagonian	OA05
	ABOVIAN	87/064	mahagonian	OA06
	BOGAZKÖY	88/040	mahagonian	OA07
	ARKA	PB 63/949	Carpathian 2Tr	OA08
	KÁLLÓ - PUSZTA	–	Carpathian 2Tr	OA09
	LEGÉND 1	–	Carpathian 1	OA10
	LEGÉND 2	–	Carpathian 2T	OA11
	LEGÉND 3	–	Carpathian 2E	OA12
	LEGÉND 4	–	mahagonian	OA13
	DÖMÖS	PB 73/111	Carpathian 1	OA14
	PILISMARÓT	PB 91/129	Carpathian 1	OA15
	HONT	PB 99/234	Carpathian 2E ?	OA16
	BALATONSZEMES	PB 17/21	Carpathian 1	OA17
	GALGAGYÖRK	–	Carpathian 2T	OA18
	PÜSPÖKHATVAN	–	Carpathian 2T ?	OA19
	MOGYORÓSBÁNYA	PB 2000/735	Carpathian 1	OA20
	SÁGVÁR 2	PB 51/159.12	Carpathian 1	OA22
	PILISSZÁNTÓ	PB 51/110	Carpathian 1	OA23
	HIDASNÉMETI	–	Carpathian 1	OA24
	ÉRD	1960/40.4	Carpathian 2E ?	OA25
	MIKOLA 1		Carpathian 1	OA26
	NAGYKÁLLÓ	1961.3.204	Carpathian 1	OA27
	SZIGETCSÉP	PB 1977/7.408	Carpathian 2T	OA28
	TAHITÓTFALU	1965/15.8	Carpathian 1	OA29
	TASNAD-SERE 1	SM 44911		ORA01
	URZICENI 1	SM 44913		ORA02
	BUSAG 1	BM 33633		ORA04
	CALINESTI I/1	SM 32347		ORA05
	CALINESTI DSM 1	SM 43001		ORA06
	VELA SPILA			OHA01
	CEPIN			OHA02
	DANILO	4116		OHA03
	KOLOJVAR	7634		OHA04
	DEVRSKE			OHA05
	SMILCIC	2856		OHA06
	CREVNI OTOK			OHA07
	OSTROVICA	957		OHA08
	SUSAC BLACK			OHA09
	SUSAC RED			OHA10
OBS GEO	BODROGOLASZI	–		OG01
	TOLCSVA 1	PB 88/027		OG02
	TOLCSVA 2	L86/170		OG03
	CEJKOV	L 86/186		OG04
	VINICKY 1	L 86/191		OG05
	VINICKY 2	L 86/152		OG06
	MÁD	L 86/124		OG07
	LIPARI1	L 86/240		OG08
	LIPARI2	L 89/152		OG09
	SARDINIA 1	L 86/244		OG10
	MELOS 1	L 87/51		OG11
	MELOS 2	L 89/151		OG12
	KASOV	L 86/188		OG13
	M ARCI	L 86/244		OG14
	AUVREGNE	L 89/150		OG15
	SEVAN 1	L 86/268		OG16
	SEVAN 2	L 89/153		OG17
	ROKOSZOVI	L 92/129		OG18



SAMPLES DIFFERENT FROM OBSIDIAN:

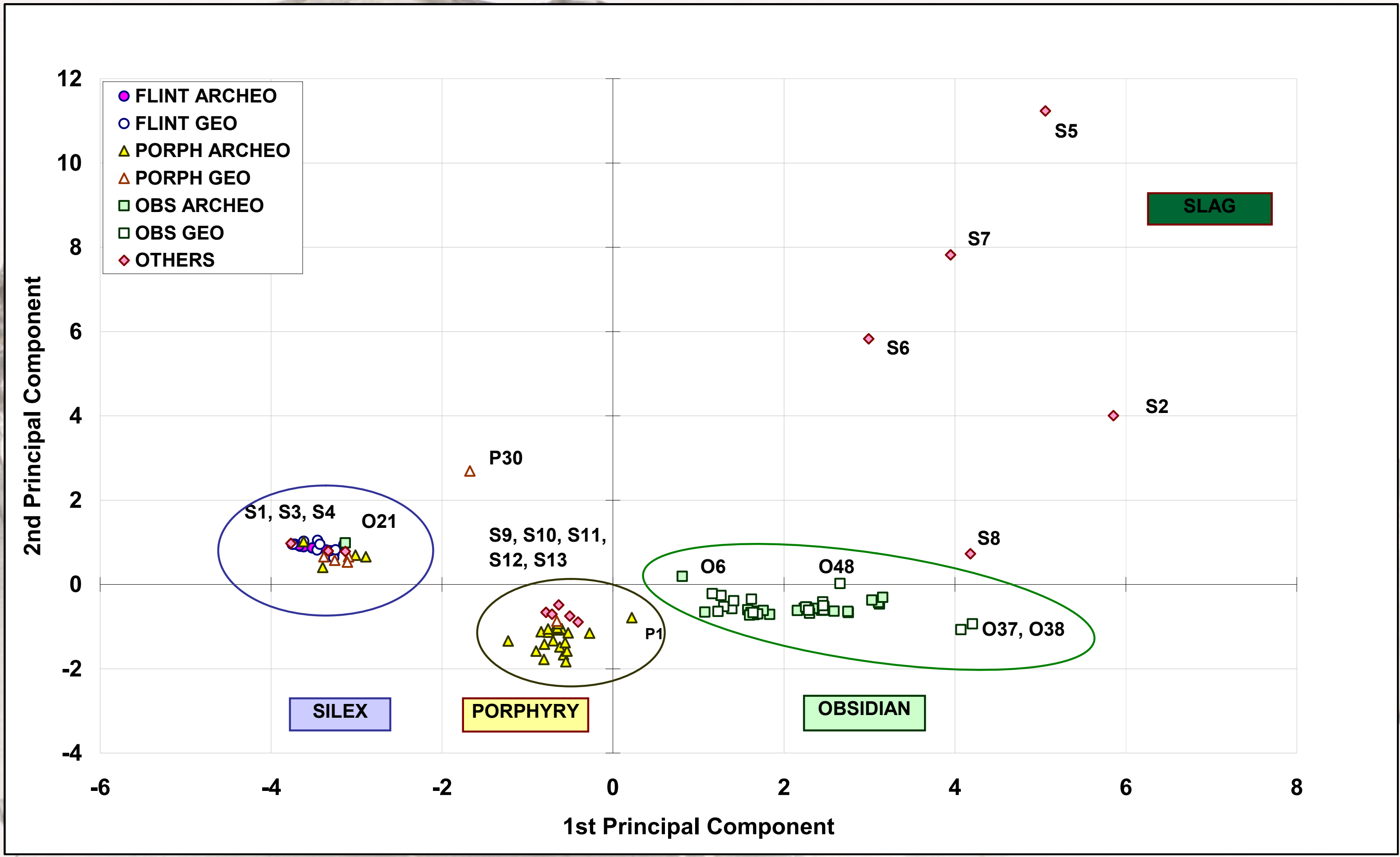
- **Oláhlápos, Zeitlarn, Kup** (artificial? *Slag)*
- **Mikola** (flint, limnic quarzite)
- **Ságvár** (radiolarite – thought to be obsidian)
- **Korolevo** (andesite)

RESULTS I.



RESULTS II.

- Carpathian obsidians are different from others (**Melos, Lipari, Sevan, Rokosovi, Auvergne**)
- C I. (SL) and C II. (HU) form two subgroups
- Further subgroups (C2T, C2E, C2Tr) with **PGAA**?



CONCLUSION

With **PGAA** we can differentiate between various chipped stone raw materials (Silex, Porphyry, Obsidian, etc.) with **non-destructive** way! Further subgroups might be determined

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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

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

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

will give the mass fraction of

arbitrary elements