

MS measurements of U, Th in
Ge/GeO₂ samples

Bezrukov L.B.¹, Kornoukhov V.N.²

(1) INR RAS

(2) ITEP / INR RAS

Moscow

U&Th content in Ge diodes

^{228}Th (^{232}Th) and ^{226}Ra (^{238}U) content were extracted from HD experiment data based on analysis of α -decays

	Kiev group (GENIS, 1998)	B.Schwingenheur GSTR-05-022
U238	$4.2 \cdot 10^{-17}$ g/g $\sim 4 \cdot 10^{-8}$ ppb	0.01 $\mu\text{B}/\text{kg}$ $8.1 \cdot 10^{-7}$ ppb
Th232	$1.7 \cdot 10^{-16}$ g/g $\sim 2 \cdot 10^{-7}$ ppb	0.006 $\mu\text{B}/\text{kg}$ $1.48 \cdot 10^{-6}$ ppb

if equilibrium exists

MS techniques used in Russia

- SS MS (Spark Source MS)
- ICP MS (X7 ICP MS Thermo Elemental) + pre-concentration
- *HR ICP MS ELEMENT 2*
- *HR GD MS (Russian chemists + Element GD in Bremen, Germany)*

Spark Source MS (SS MS)

Ge metal and GeO₂, samples from:
Germany, Krasnoyarsk,
Svetlana, the ECP

U-238 < = 40 ppb

Th-232 < = 40 ppb

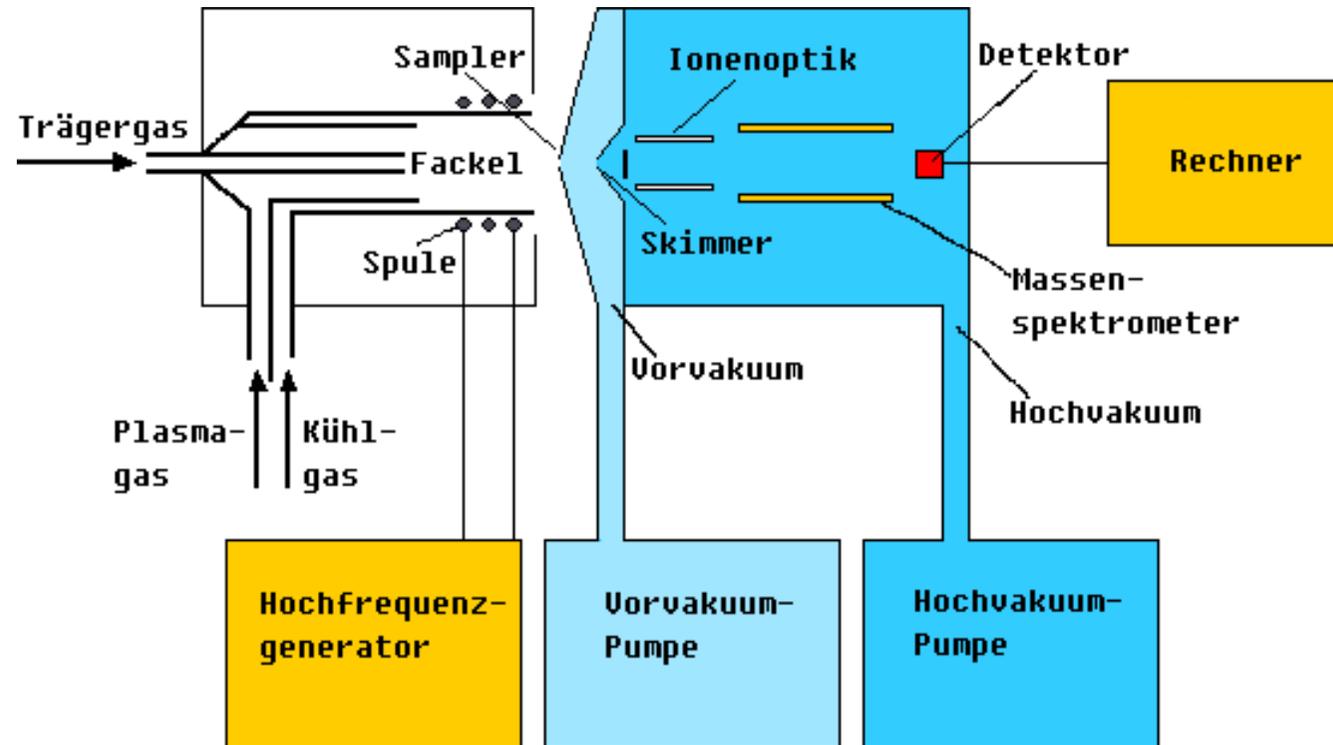
ICP MS

$^{\text{nat}}\text{GeO}_2$ and $^{76}\text{GeO}_2$ samples

Samples were produced at Svetlana, the ECP according to existing technology for enriched Ge

- Standard chemical decomposition + GeCl_4 distillation
 - U-238 ≤ 0.4 ppb
 - Th-232 ≤ 0.3 ppb
- Chemical decomposition + simultaneous GeCl_4 distillation
 - U-238 ≤ 0.002 ppb
 - Th-232 ≤ 0.006 ppb

ICP MS: how it works



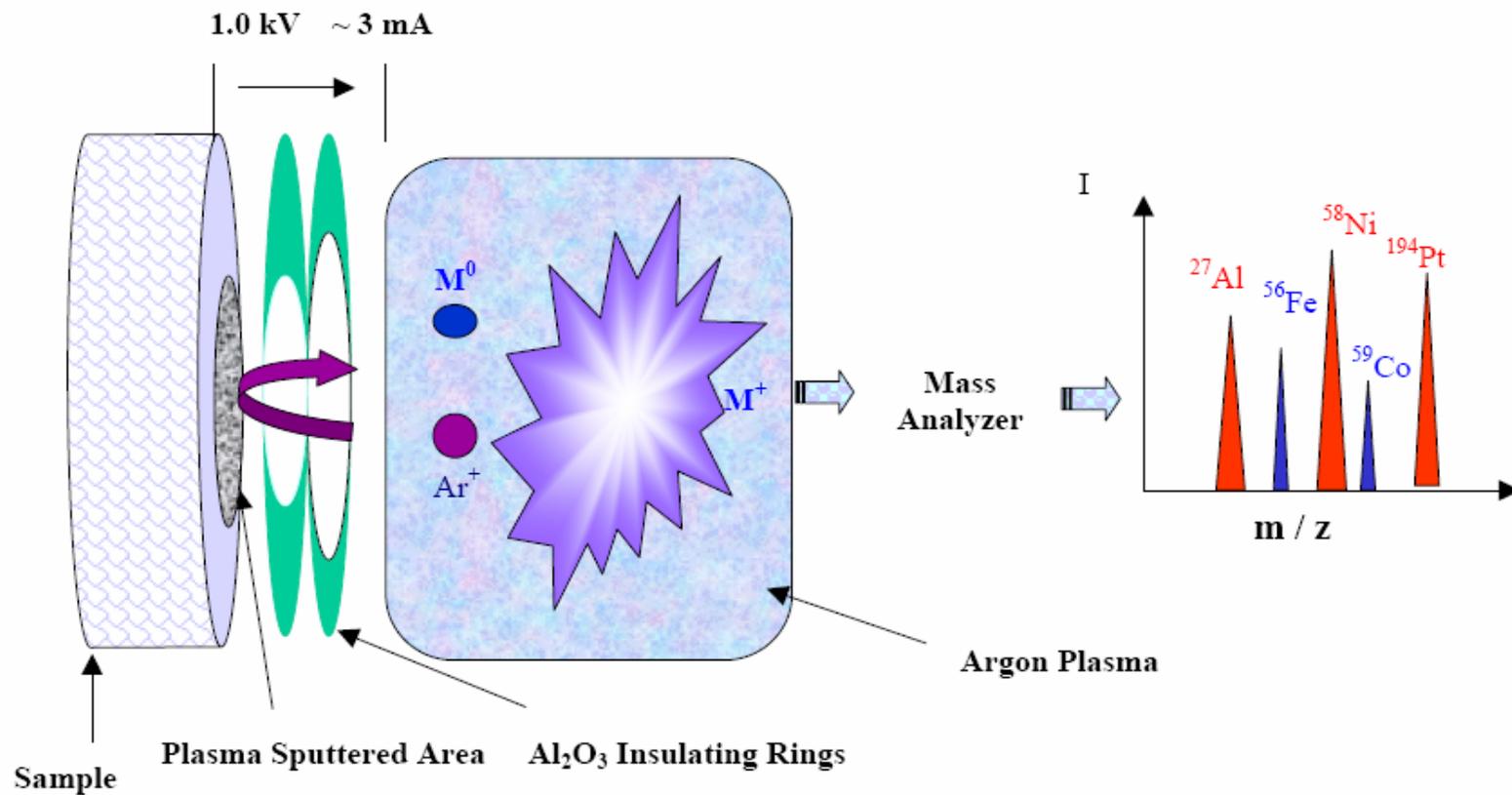
ELEMENT HR GD (glow discharge high resolution)

Ge metal, samples #1 – 5
from Germany, Krasnoyarsk,
after experiment # 2 (August 2005)

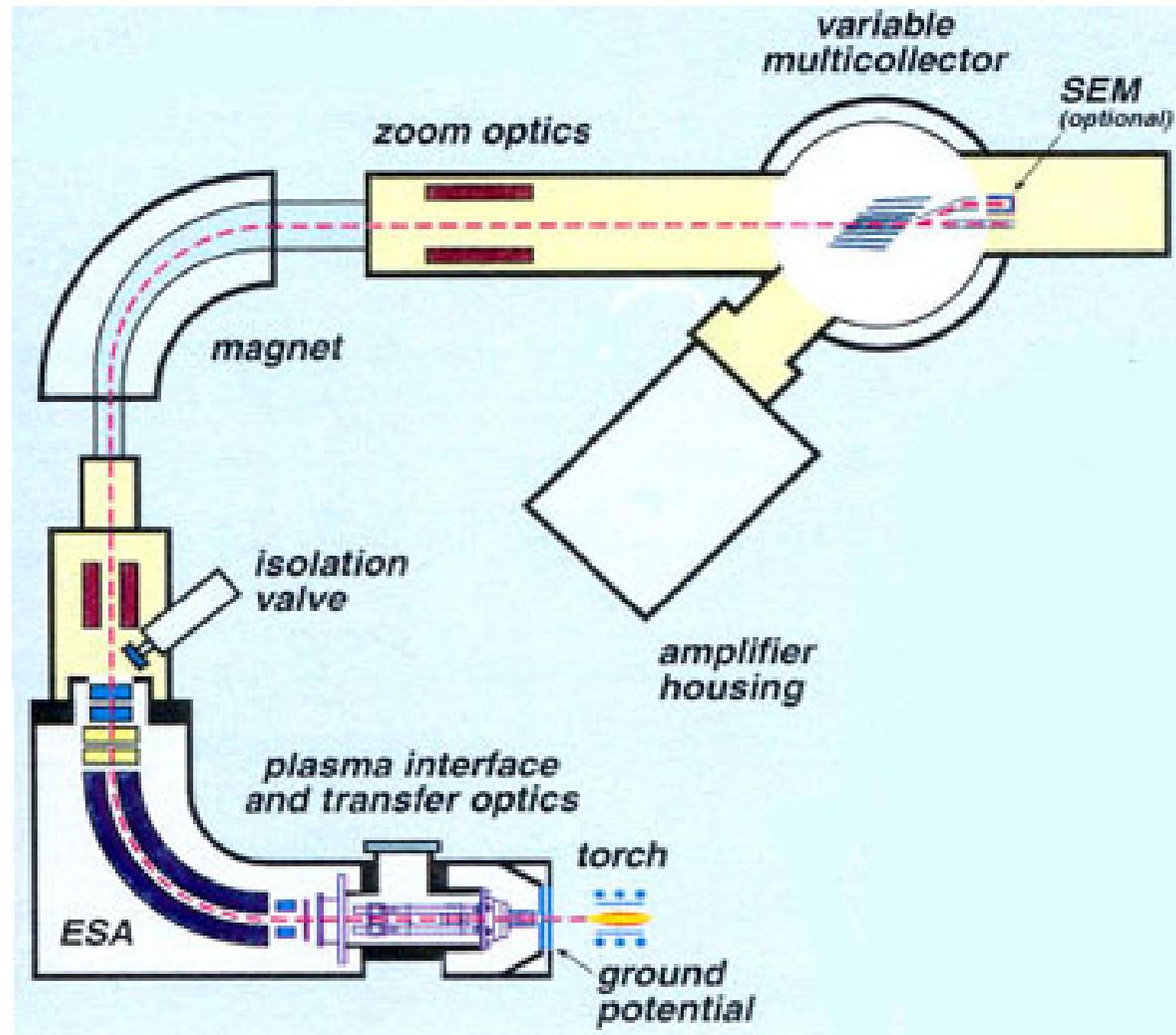
U-238	< = 0.003 - 0.018 ppb
Th-232	< = 0.006 - 0.014 ppb
K-39	< = 1.5 ppb

sum all	40189,3	37478,5
sum without CNO	862,5	846,5
sum of metals only	196,9	180,1

GD (glow discharge): how it works



HR MC-ICP MS NEPTUNE



Isotopic composition measurements

Product	Laboratory	Method, device	Ge76	ΔC
Natural Ge	ЦЗЛ ПО "ЕСР"	EIMS MI 1201 №15, TIMS MI 1201-AT №5	7.75	
Natural Ge	РАН ИГЕМ	ICP MS NEPTUNE	7.74	
Batch 42	ЦЗЛ ПО "ЕСР"	EIMS MI 1201 №15, TIMS MI 1201-AT №5	0.52	± 0.03
Batch 42	ВНИИНМ	TIMS SEKTOR 54	0.53	
Batch 42	РАН ИГЕМ	ICP MS NEPTUNE	0.57	
Batch 415	ЦЗЛ ПО "ЕСР"	EIMS MI 1201 №15, TIMS MI 1201-AT №5	0.69	± 0.03
Batch 415-2	РАН ИГЕМ	ICP MS NEPTUNE	0.68	
Batch 414-1	ВНИИНМ	TIMS SEKTOR 54	0.56	
Batch 414-2	РАН ИГЕМ	ICP MS NEPTUNE	0.60	

Conclusion - I

U and Th have been measured in Ge/GeO₂ samples from the ECP and Germany

- SS MS: ≤ 40 ppb (U&Th)
- ICP MS + pre-concentration (^{nat}GeO₂ from Svetlana):

U-238 ≤ 0.002 ppb

Th-232 ≤ 0.006 ppb

- Pilot measurements of Ge metal (!) with HR GD MS (Thermal Electron, Bremen):

U-238 $\leq 0.003 - 0.018$ ppb

Th-232 $\leq 0.006 - 0.014$ ppb

K-39 ≤ 1.5 ppb

Conclusion - II

No U & Th have been found at level of sensitivity:

Samples of ^{76}Ge from Svetlana, the ECP:

U-238 ≤ 0.04 ppb

Th-232 ≤ 0.3 ppb

Samples of $^{\text{nat}}\text{Ge}$ from Germany:

U-238 $\leq 0.003 - 0.018$ ppb

Th-232 $\leq 0.006 - 0.014$ ppb

Procedure of sample preconcentration

General procedure is to decompose the samples and distill GeCl_4 out.

M (Ge metal/ GeO_2) \approx 0.8 g of each of the samples.

M (probe) \approx 300 mg (Ge metal) and \approx 200 mg (GeO_2).

3 controls samples were also decomposed.

- a) The probes of Ge metal were twice etched with a mixture of concentrated $\text{HNO}_3 + \text{HCl} + \text{HF}$ to remove surface contaminants.
- b) The probes were placed in autoclaves, where 1 mL of concentrated HNO_3 + 2 mL of concentrated HF and heated for 1 h at 160 and 1 h at 180°C.
- c) Then the autoclaves were cooled to room temperature.
- d) The solutions from the autoclaves \rightarrow Teflon beaker (50 mL of volume) + { 2 mL + 2 mL of concentrated HNO_3 }.
- e) Simultaneously the same procedure was performed in three autoclaves without samples and resultant solutions were used as control samples.