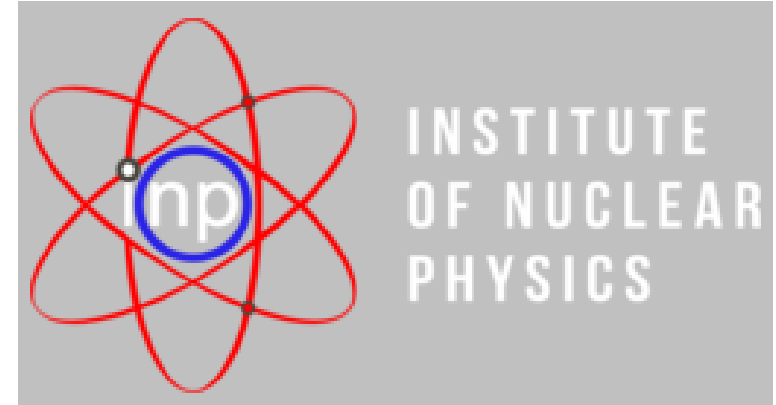




**Joint Institute for Nuclear
Research**

SCIENCE BRINGING NATIONS TOGETHER



Radiochemistry for Underground Physics

Ayagoz Baimukhanova

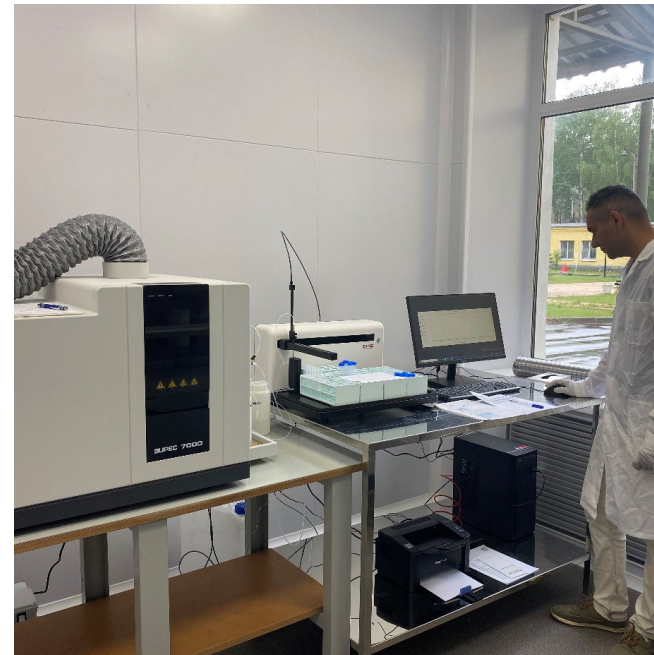
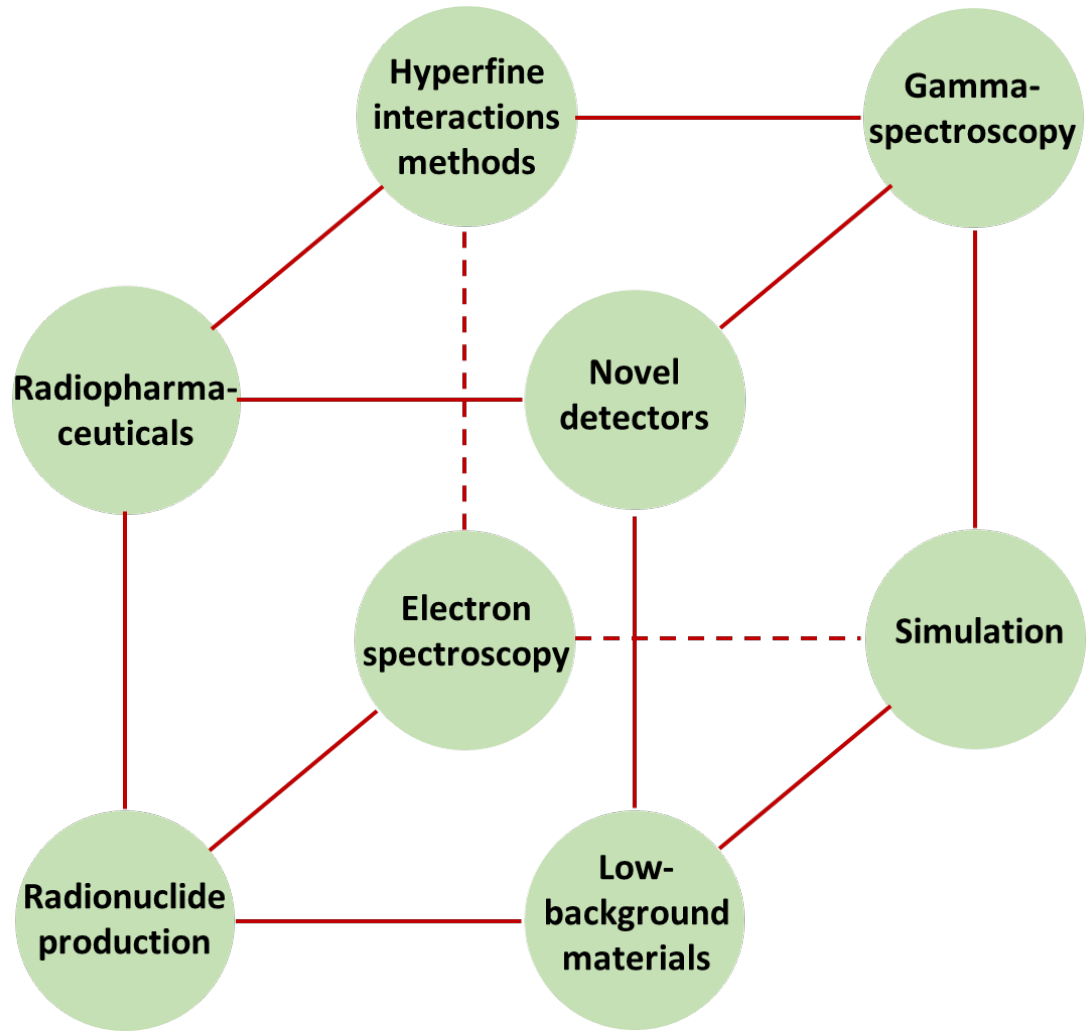
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Joint Institute for Nuclear Research
Institute of Nuclear Physics of the Republic of Kazakhstan

*Symposium on Science @ PAUL
17 January 2024*

Radiochemistry and spectroscopy for astrophysics and nuclear medicine

Scientific directions



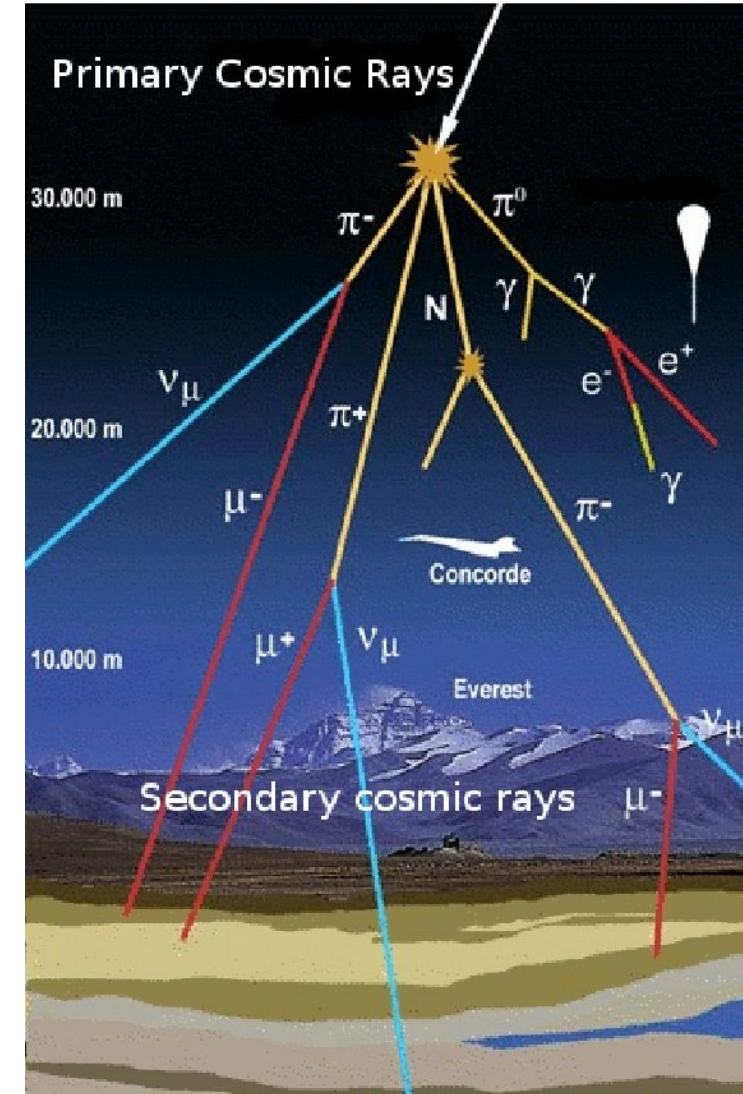
Low-Background Experiments

Background sources:

1. Cosmic rays.
2. The presence of radioactive impurities in detector materials, in measured samples and in structural materials of experimental facilities and laboratories.
3. Radioactive gases in the atmosphere (radon and its decay products: $^{210,214}\text{Bi}$, $^{210,214}\text{Pb}$, $^{210,214}\text{Po}$).

Methods of analysis:

ICP-MS, ICP-AES with chemical preparation, neutron activation analysis, gamma-ray screening and etc.



Giving new life to old equipment. F. Barradas-Solas .
Physics Education 2007, V. 42, N. 1, P. 9-11. DOI 10.1088/0031-9120/42/1/F03

Materials and Methods Developed in JINR

Samples for measurement

^{48}Ca

^{82}Se

^{96}Zr

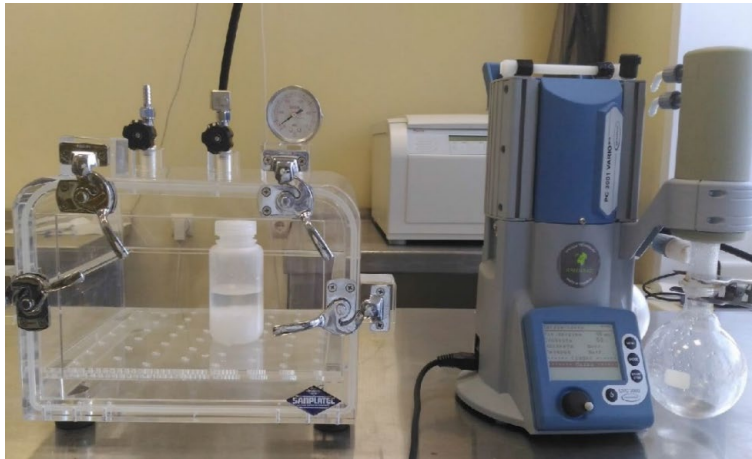
^{106}Cd



Solders and fluxes

archaeological Pb+Sn

NH_4Cl , $\text{CH}_3\text{COONH}_4$

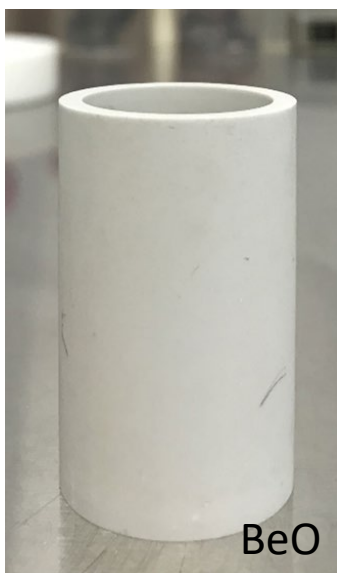
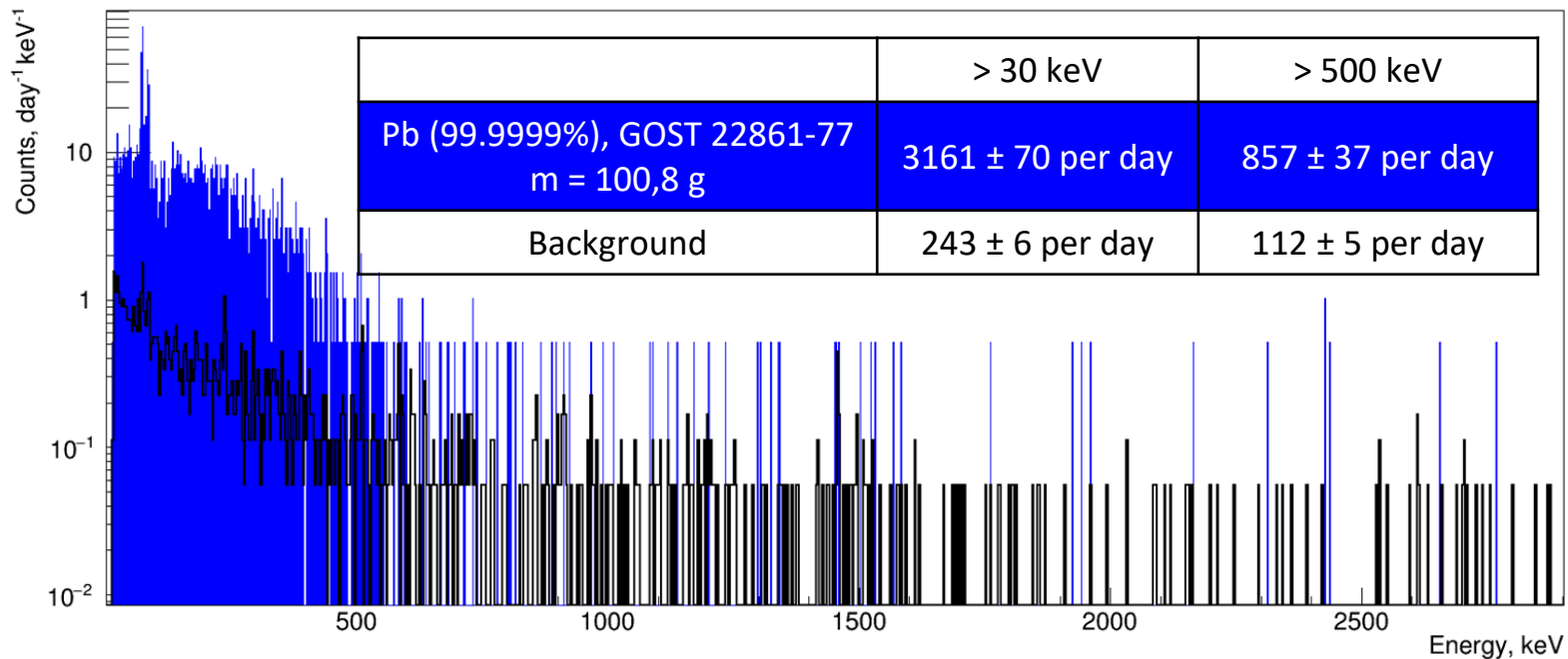


Detector, protection and construction materials

Etching methods for copper surface

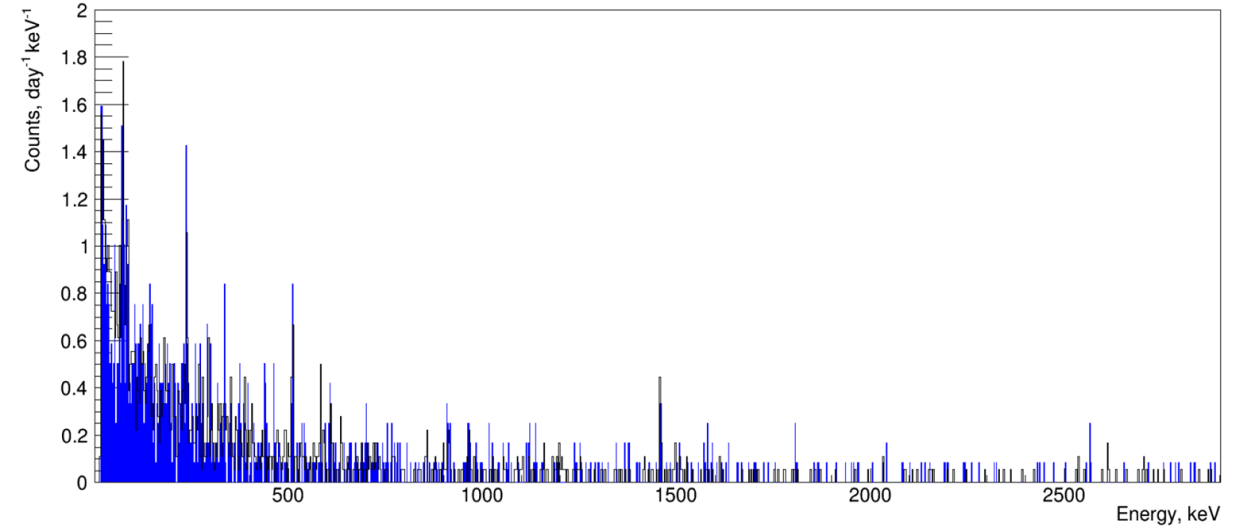
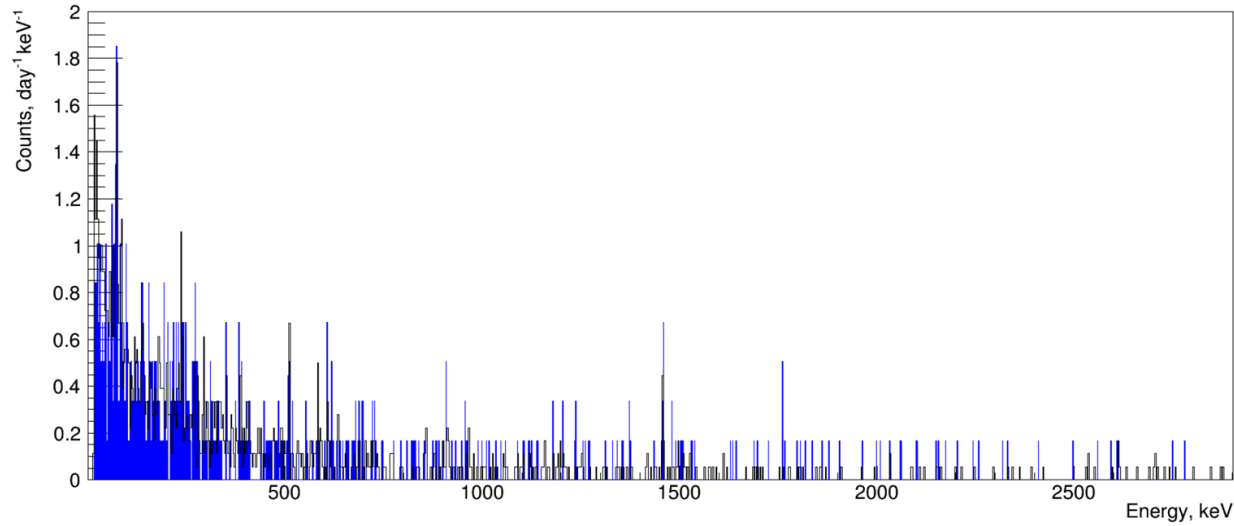
Protocols of surface cleaning

Preparation of the Solder Made of Archaeological Pb and Sn. Part 1



²³² Th decay chain		²³⁸ U decay chain	
Radionuclide	Activity, mBq/kg	Radionuclide	Activity, mBq/kg
²⁰⁸ Tl	< 18	²¹⁴ Pb	< 17
²⁰⁸ Tl	< 27	²¹⁴ Bi	< 13
²¹² Bi	< 144	²¹⁰ Pb	< 78
²¹² Pb	< 34		
²²⁸ Ac	< 79		
		Radionuclide	Activity, mBq/kg
		⁴⁰ K	< 94

Preparation of the Solder Made of Archaeological Pb and Sn. Part 2

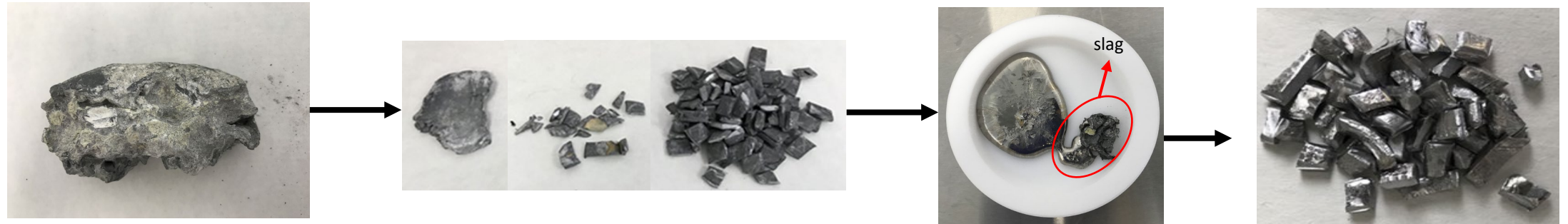


	> 30 keV	> 500 keV
Tin (99.9999%), GOST 860-75 m = 75,1 g	269 ± 12 per day	139 ± 9 per day
Background	243 ± 6 per day	112 ± 5 per day

	> 30 keV	> 500 keV
Arch. Lead m = 114 g	243 ± 7 per day	112 ± 5 per day
Background	243 ± 6 per day	112 ± 5 per day

Preparation of the Solder Made of Archaeological Pb and Sn. Part 3

I.



The original piece of archaeological lead

The molten lead

III.

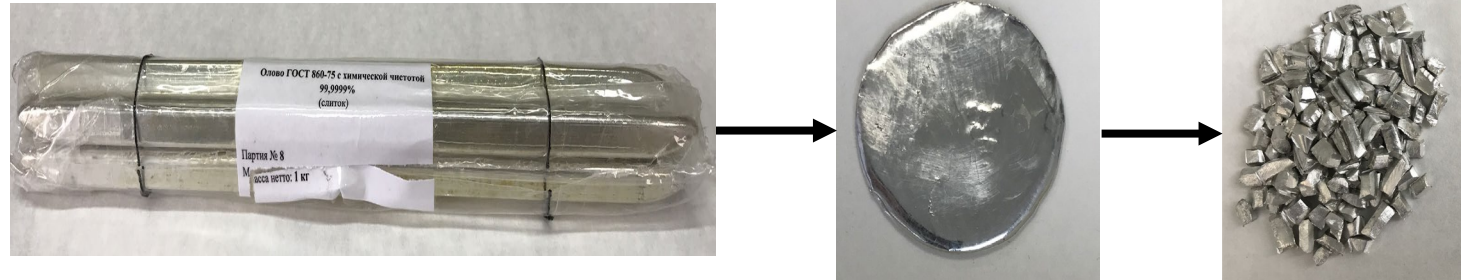


metal cutting
and weighing

melting at
 $T=400\text{ }^{\circ}\text{C}$

pouring into a
Teflon cup

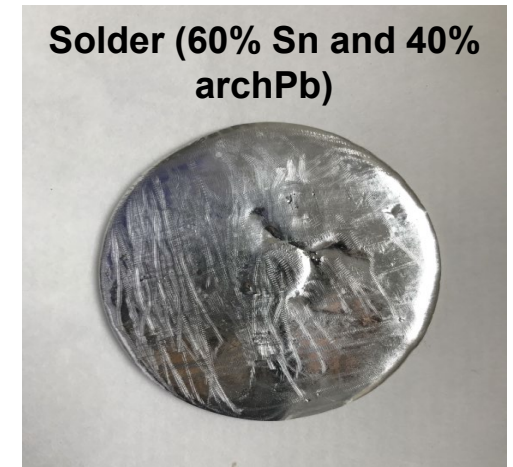
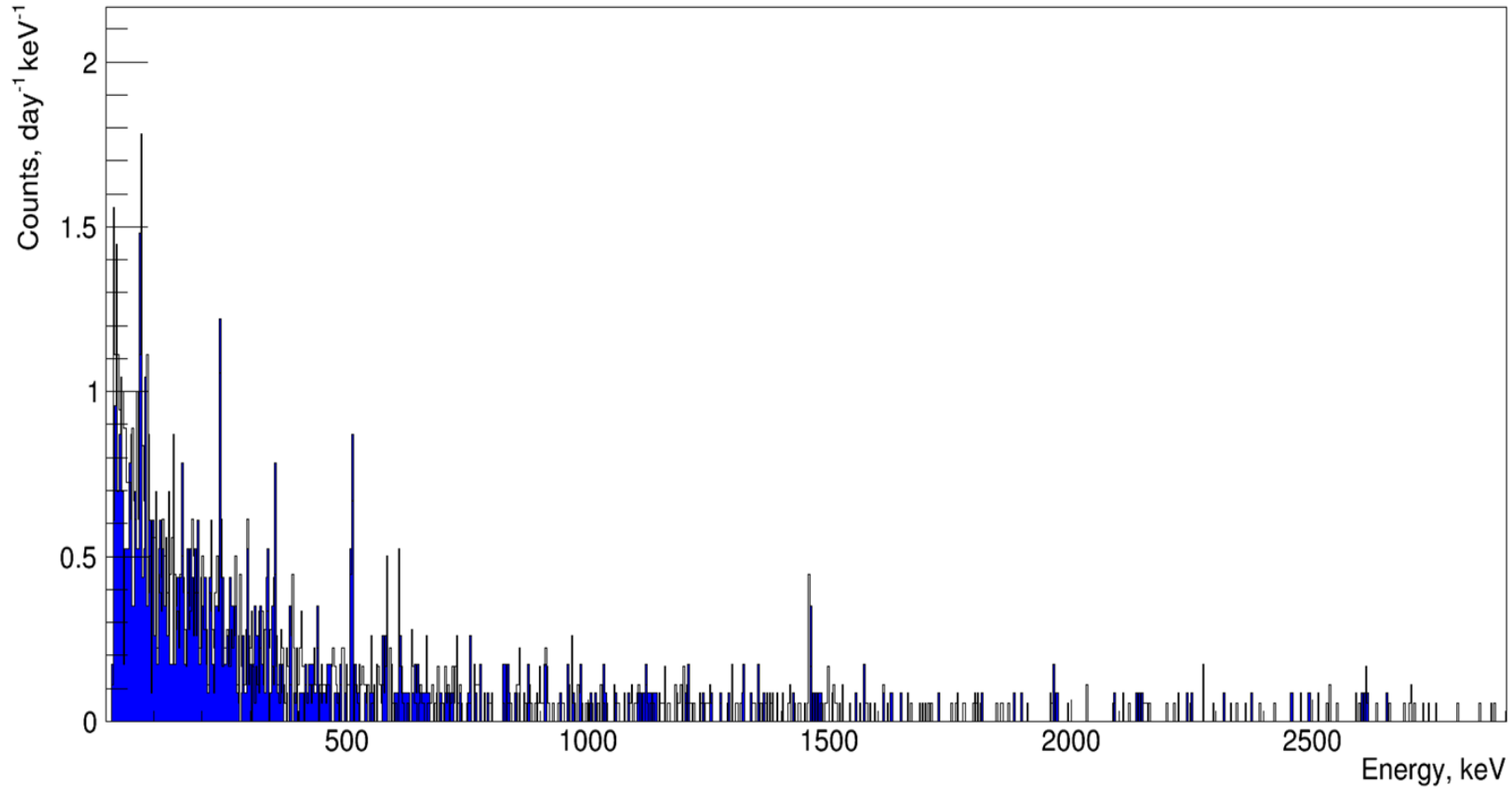
II.



Tin with chemical purity 99.9999%

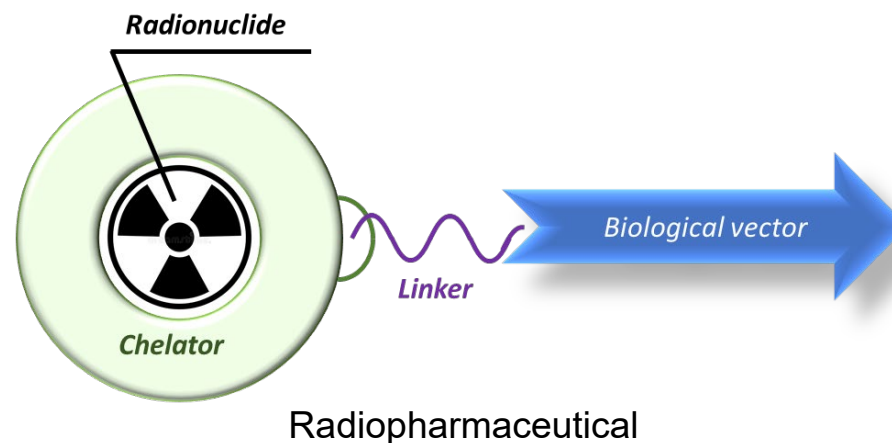
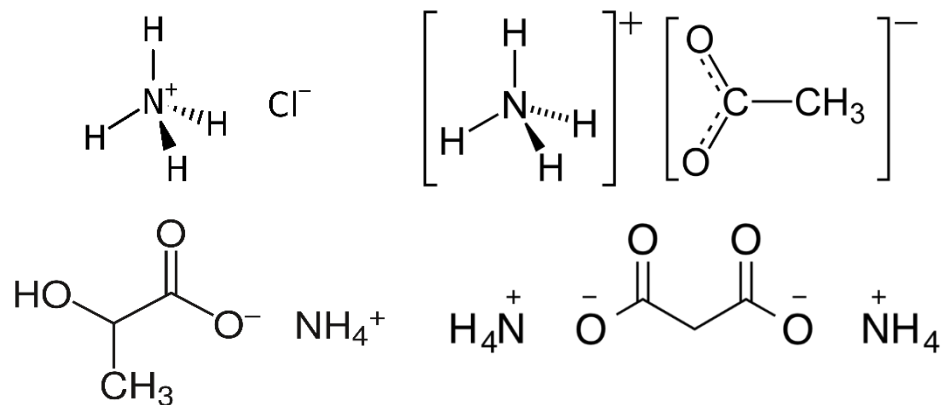


Preparation of the Solder Made of Archaeological Pb and Sn. Part 4



	> 30 keV	> 500 keV
Solder Sn(60%)Pb(40%) m = 154.5 g	222 ± 8 per day	105 ± 6 per day
Background	243 ± 6 per day	112 ± 5 per day

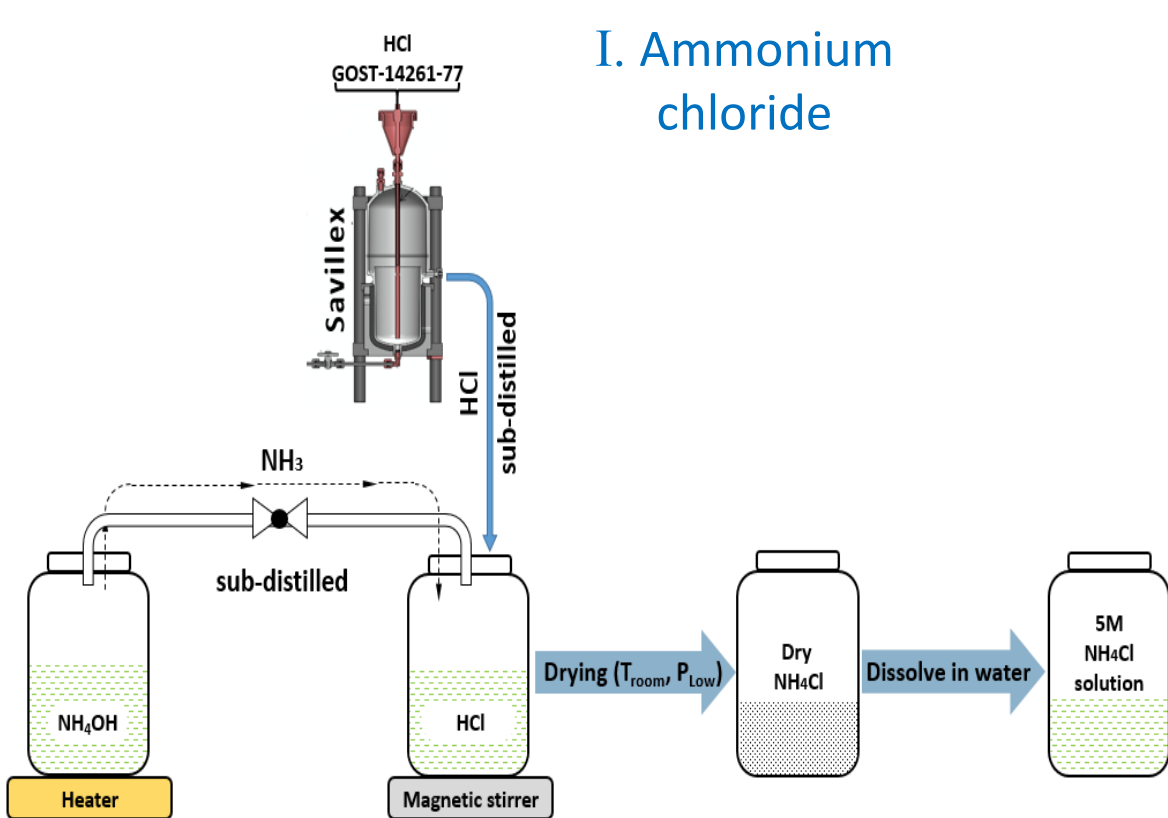
High-Purity Ammonium Salts for Fluxes and Radiolabeling. Part 1



Samples	Purity, %	^{238}U		^{232}Th		^{40}K	
		10^{-9}g/g	mBq/kg	10^{-9}g/g	mBq/kg	10^{-9}g/g	mBq/kg
ZnCl_2	99.999	55 ± 15	679 ± 185	< 200	< 812	< 64	< 2.0
NH_4Cl	99.999	60 ± 20	741 ± 247	330 ± 140	1340 ± 568	< 72	< 2.2
H_3PO_4	99.998	27 ± 12	333 ± 148	< 74	< 300	< 66	< 2.1
$\text{C}_9\text{H}_8\text{O}_4$	99.9	160 ± 30	1970 ± 370	170 ± 90	690 ± 365	< 44	< 1.4

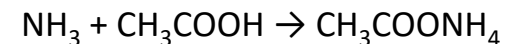
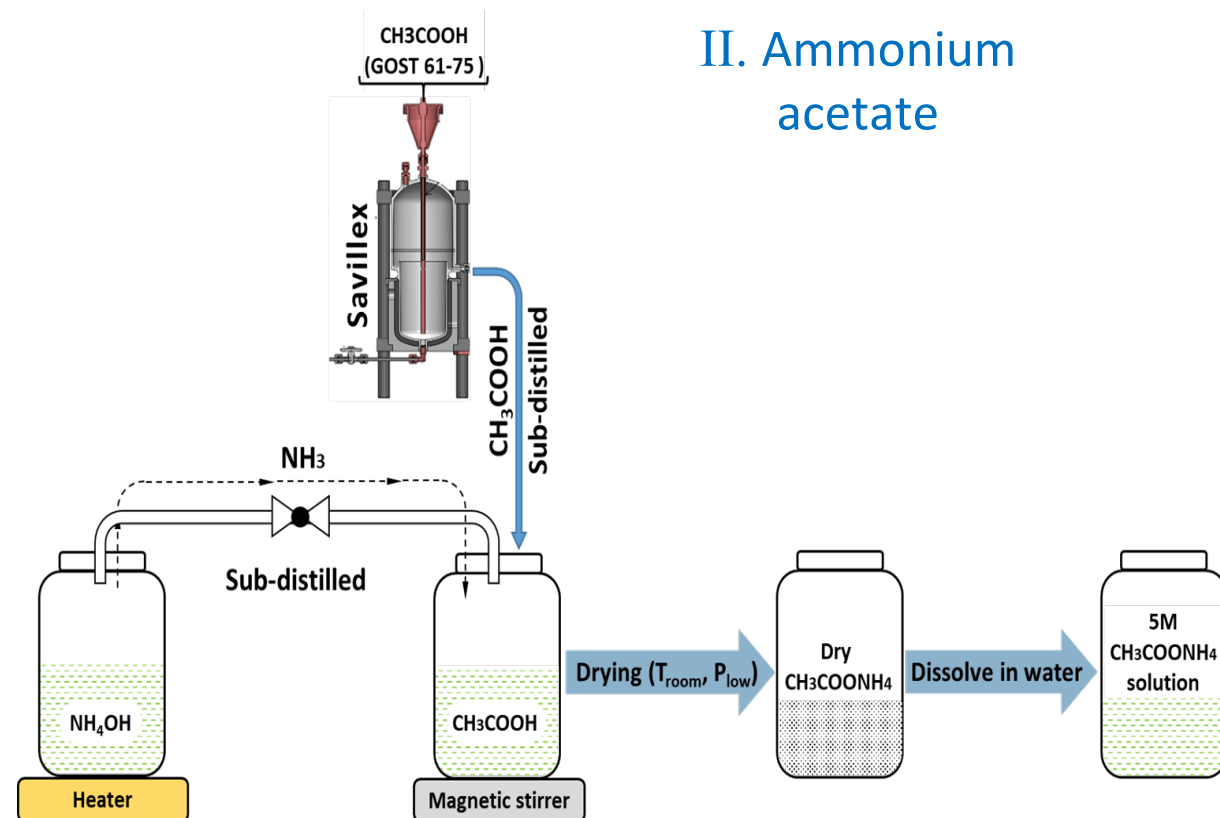
Synthesis of High-Purity Ammonium Salts. Part 2

I. Ammonium chloride



- 1) purification of HCl;
- 2) synthesis of ammonium chloride solution (transfer of gas phase ammonia to the acid);
- 3) obtaining NH_4Cl in the solid state by drying it in a vacuum at room temperature.

II. Ammonium acetate

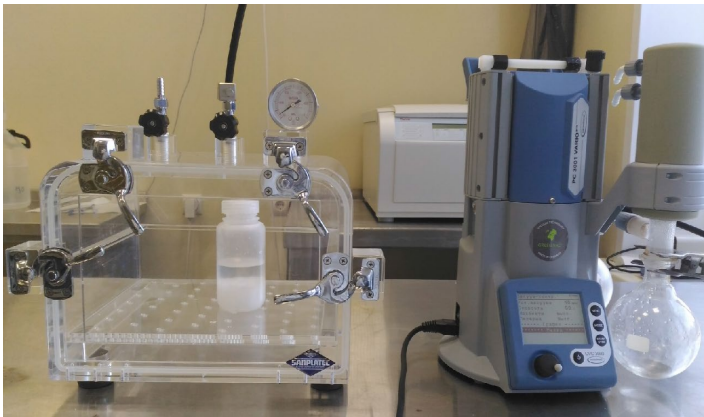


- 1) purification of the 16 M CH_3COOH acid;
- 2) synthesis of ammonium acetate solution (transfer of gas-phase ammonia to the acid);
- 3) obtaining $\text{CH}_3\text{COONH}_4$ in the solid state by drying it under vacuum at room temperature.

Analysis of High-Purity Ammonium Salts. Part 3

Impurities of ^{232}Th , ^{238}U and ^{40}K in the synthesized ammonium salts (ICP-MS, ICP-AES)

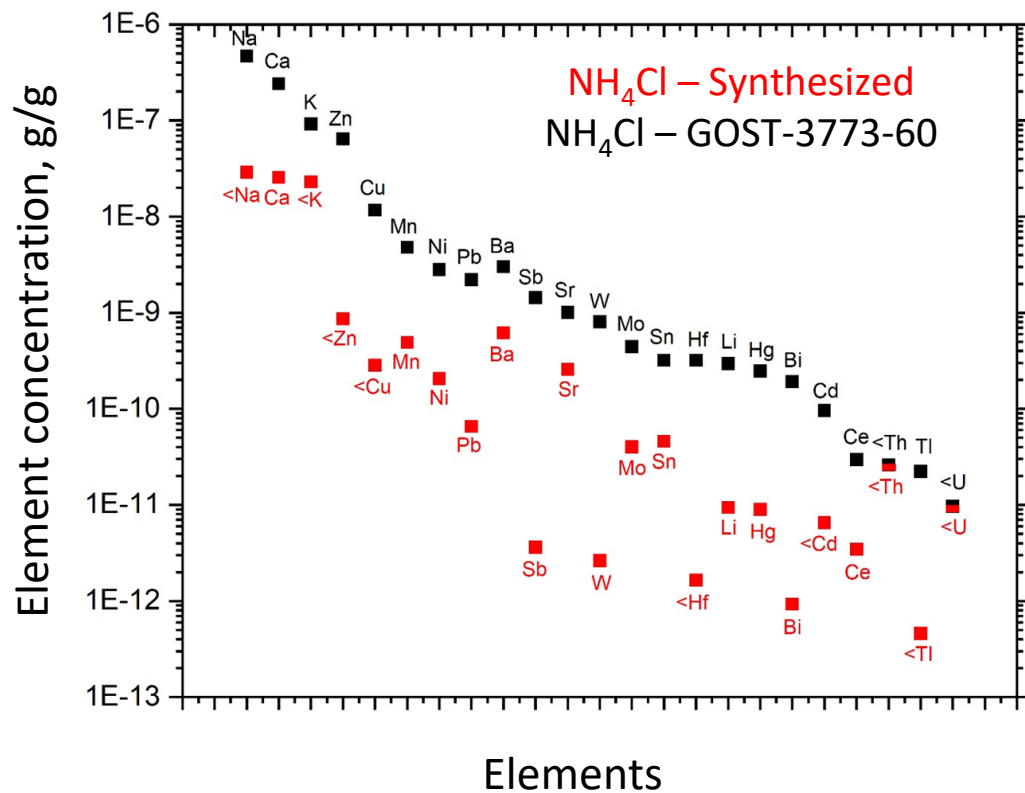
Samples	^{232}Th , mBq/kg	^{238}U , mBq/kg	^{40}K , mBq/kg
NH_4Cl	< 0.1	< 0.1	< 0.7
$\text{CH}_3\text{COONH}_4$	< 0.1	< 0.1	< 0.7



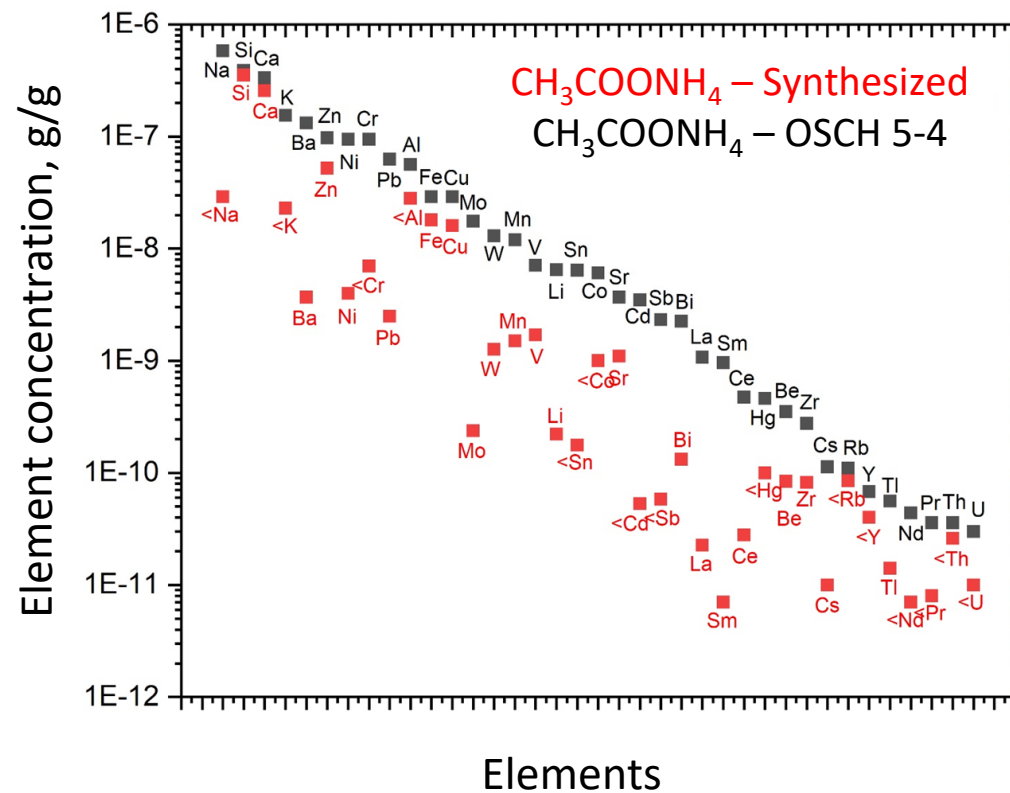
Measurement results of the synthesized NH_4Cl (low-background HPGe)

Radionuclide	E_γ , keV	Activity, mBq/kg
^{40}K	1461	< 77
^{208}Tl	2614	< 5
^{210}Pb	46	< 91
^{212}Pb	239	< 2
^{214}Pb	242	< 10
^{214}Pb	352	< 5
^{214}Bi	609	< 10
^{214}Bi	1765	< 5
^{228}Ac	911	< 5

Analysis of High-Purity Ammonium Salts. Part 4

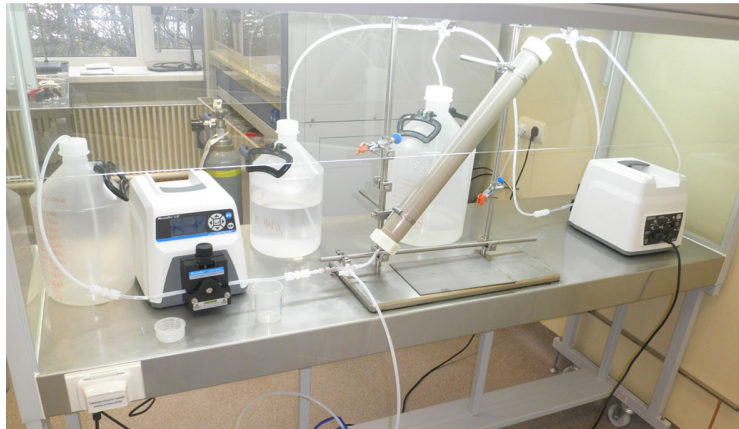


ICP-MS and ICP-AES results of the commercial (GOST 3773-60) and synthesized NH₄Cl



ICP-MS and ICP-AES results of the commercial (OSCH 5-4) and synthesized CH₃COONH₄

Method for Purification and Conditioning of Selenium. Part 1



II.



III.



IV.

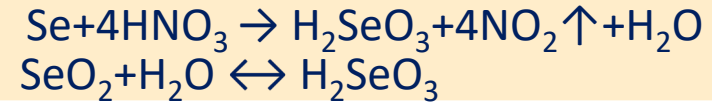


V.



VI.

I. Dissolving



II. Chromatography purification



Conditioning

III. Reduction to elemental Se with SO_2



IV. Centrifugation
(washing with alcohol, water)



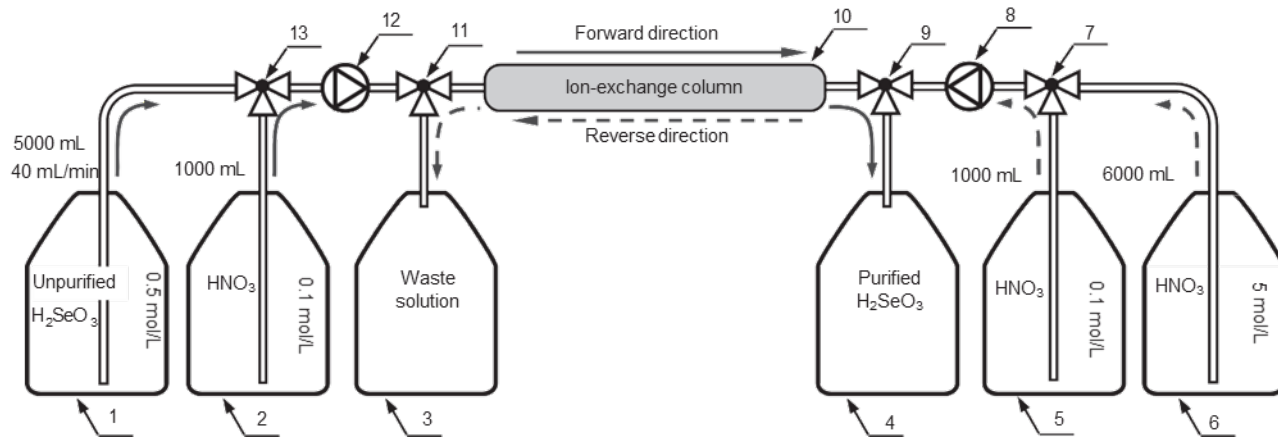
V. Homogenization of selenium (in alcohol)



VI. Vacuum drying

At room temperature

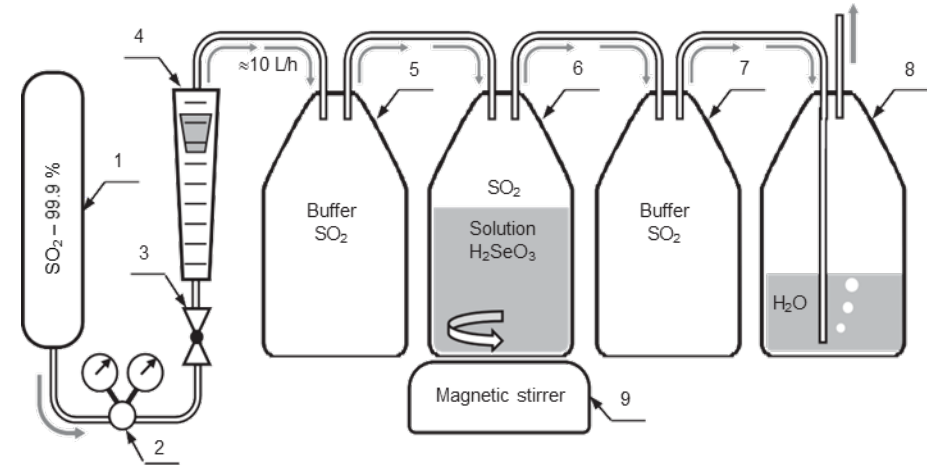
Method for Purification and Conditioning of Selenium. Part 2



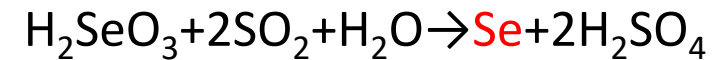
1 ÷ 6 – solutions; 7, 9, 11, 13 – triple valves; 8, 12 – pumps;
10 – ion exchange column

Dowex 50W×8, h=500 mm
For m=100 g of selenium → d=10 mm
For m>100 g → d=28 mm.

Reverse chromatography scheme of H₂SeO₃ purification

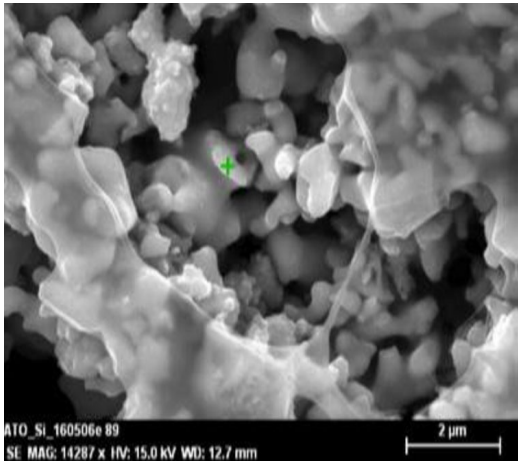
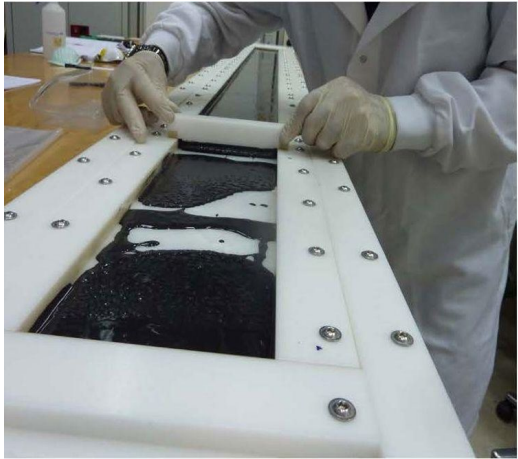


1 - SO₂; 2 - reducer; 3 – valve; 4 – rotameter;
5 ÷ 8 vessels (10 L); 9 - magnetic stirrer



Reduction scheme of selenous acid to elemental state

Method for Purification and Conditioning of Selenium. Part 3



Samples	Specific activities of radionuclide impurities, $\mu\text{Bq/kg}$			
	^{40}K	$^{234\text{m}}\text{Pa}$	^{208}Tl	^{214}Bi
^{82}Se	58700 ± 100	17300 ± 100	390 ± 10	1500 ± 40

Samples	Specific activities of radionuclide impurities, $\mu\text{Bq/kg}$			
	^{40}K	^{238}U ($^{234\text{m}}\text{Pa}$)	^{232}Th (^{208}Tl)	^{226}Ra (^{214}Bi)
Purified ^{82}Se	$< 9300^*$	$< 2500^*$	22 - 150 (8 - 54)	< 600

Improvement factor of purified ^{82}Se	> 6	> 7	7 - 50	> 2.5
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* ICP-MS results. Element content converts from g/g to $\mu\text{Bq/kg}$

< - detection limit

Summary

1. A wide experience in production of low-background materials with the level of impurities of mBq/kg (and lower) has been gained.
2. We have an expertise in separation of radionuclides and their purification with a high level of separation/purification factors.
3. Analytical methods for determination of ultralow concentration of elements/radionuclides (ICP-MS, ICP-AES, NAA, γ -spectrometry) are being developed.