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ICP MS measurements of ss steel for GERDA experiment

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Conclusion (*Milano meeting, Nov. 2006*)

1. Six samples of ss steel type 1.4571 were measured with ICP MS ELAN DRC II (Moscow).
2. Content of Th-232 was defined at level of

   <= 2.4 – 4.0 mBq/kg (limit)
3. Such a level of Th-232 requires application of internal Cu passive shielding with modest thickness (in the cryostat design).
4. These 6 samples will be re-measured with another MS set up, X7 ICP MS.

Samples of ss steel ICP MS measurements

The Analytic Certification Testing Center of the Institute of Microelectronics Technology & High Purity Materials of Russian Academy of Science.

- The Center has been accredited by the Federal Standart of Russia (№ РОСС RU.0001.513800 dated 25.02.2003).
- Head of the Center is Dr.Karandashev (karan@ipmt-hpm.ac.ru).
- We do certification of ^{nat}Ge metal and $^{nat}GeO_2$, and enriched Ge metal and GeO_2 (for GERDA Phase II)
- We certified two batches of ss steel:

December 2006 - 6 samples (MPIK)

Dec 2006/Jan2007 - 2 samples (LNGS)

Typical mode of the MS Spectrometer' operation

Inductively Coupled-Mass Spectrometer

X-7 ICP-MS, *Thermo Elemental*, USA was used

Plasma	13 L/min
Auxiliary Gas Flow	0,9 L/min
Sample	0.8 ml/min
Resolusion	0.8M
Range of scanning	175 - 245

Main parameters of mass-spectrums measurements:

- **Detector mode operation: double (count of pulses and analog one);**
- **Scan mode:**

	Survey Scan		**Peak Jumping**
Number of scans	5	Sweeps	400
Dwell Time	0.6 msec		10 msec
Channels Per Mass	10		1
Acquisition Duration	2 sec		19 sec

Samples of ss steel

- Samples were first etched with a mixture of HNO_3 and HCl acids, then washed by DI water and dried.
- Samples have been weighed.
- Three probes to be analyzed were placed in one-chamber autoclaves, where 0,5 mL of concentrated HNO_3 and 1,0 mL of concentrated HCl were added and heated for 40 min at 160°C. Then the autoclaves were cooled down. The solutions from the autoclaves were transferred to beaker made of polyethylene and add 10 mL of DI water.
- Just before analysis the volume of the solutions (1 mL) were adjusted by adding DI water to have concentration of samples around of ~ 1.0 g/L (to avoid matrix effect). Then analysis was done.
- Simultaneously the same procedure was performed in two autoclaves without samples and resultant solutions were used as control samples.

Calibration and internal standard

- Calibration of the set up was made with standard solution, concentration of ^{238}U is 1 μg/L and ^{232}Th is 1 μg/L.

- Processing of mass-spectrums and calculation of element content were made with X-7 ICP software. Detection limit (DL) were calculated as

$$DL = C_i + 3*\sigma$$

where: C_i - mean value of content of i-isotope if control samples are under measurements;

σ - standard deviation for isotope i (for control samples).

Relative standard deviation for elements

<= 0.3 if content of these elements <= 5*DL

and <= 0.15, if C_{Me} > 5*DL

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ICP MS measurements ss steel type 1.4571

Sample	Element	Content in sample g/g	Th-232	Activity Ra228/Th228 mBq/kg (HPGe)
1 494257	U	≤ $4{,}0 \cdot 10^{-10}$		
	Th	**≤ $5{,}0 \cdot 10^{-10}$**	**≤ 2,0**	**< 0, 86/0,11**
2 493553	U	≤ $1{,}2 \cdot 10^{-9}$		
	Th	**≤ $5{,}0 \cdot 10^{-10}$**	**≤ 2,0**	***<3,3/=1,1***
3 254533	U	≤ $1{,}5 \cdot 10^{-9}$		
	Th	**≤ $5{,}0 \cdot 10^{-10}$**	**≤ 2,0**	**= 1,0/1,5**
4 255455	U	≤ $5{,}0 \cdot 10^{-10}$		
	Th	**≤ $7{,}0 \cdot 10^{-10}$**	**≤ 2,8**	**<3,0/=5,1**
5 50609522	U	≤ $4{,}0 \cdot 10^{-10}$		
	Th	**≤ $5{,}0 \cdot 10^{-10}$**	**≤ 2,0**	**<1,0/0,41**
6 charge	U	≤ $1{,}9 \cdot 10^{-9}$		
#5991	**Th**	**≤ $1{,}2 \cdot 10^{-9}$**	**≤ 4,8**	***<2,9/=5,1***
#1 (LNGS)	U	≤ $4{,}0 \cdot 10^{-10}$		
	Th	**≤ $3{,}0 \cdot 10^{-10}$**	**≤ 1,2**	**?**
#2 (LNGS)	U	≤ $4{,}0 \cdot 10^{-10}$		
	Th	**≤ $3{,}0 \cdot 10^{-10}$**	**≤ 1,2**	**?**

Conclusion

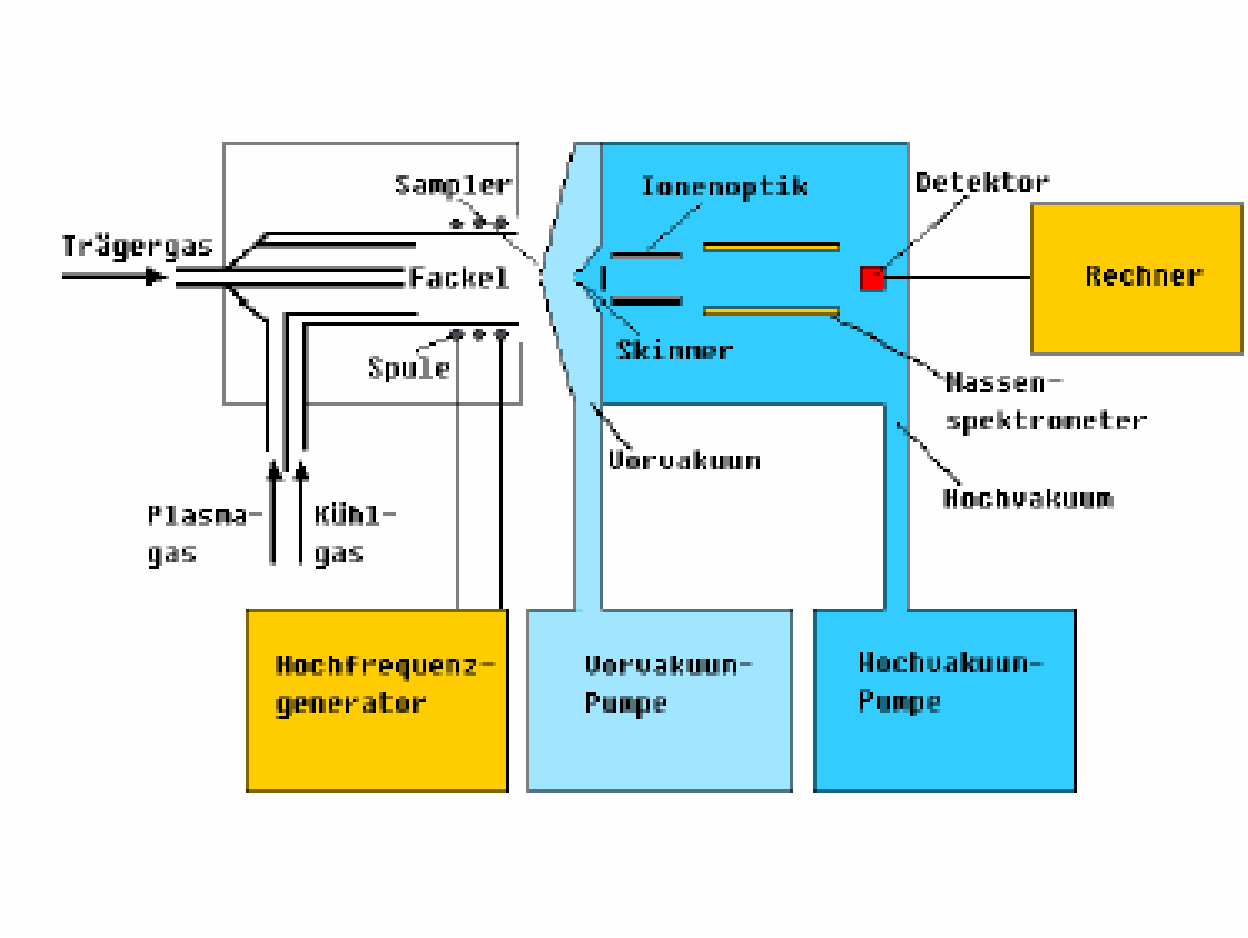
1. Eight samples of ss steel type 1.4571 were measured with X7 ICP MS (six of them were re-measured).

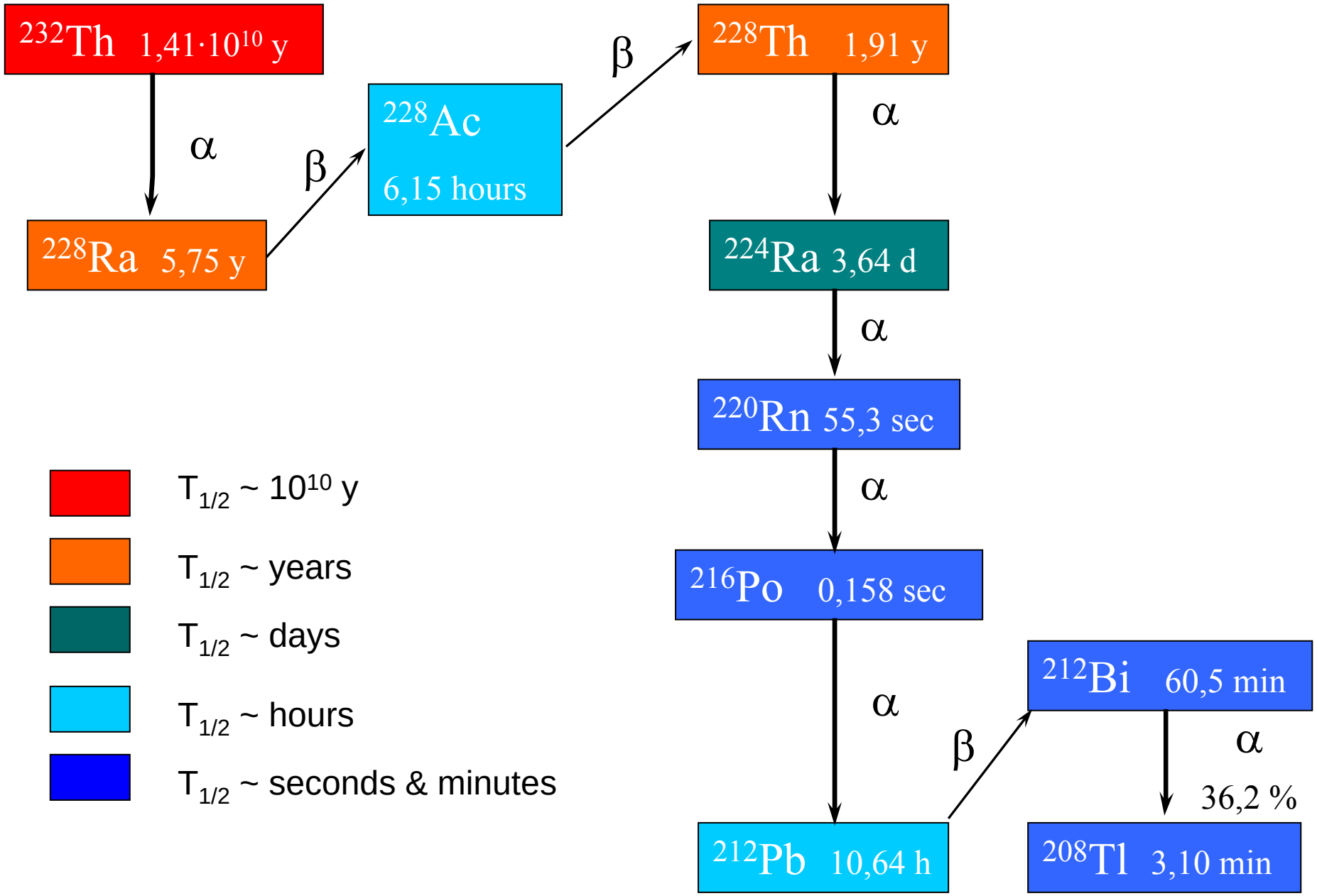
2. DL (Th232) for last 2 samples is 1,2 mBq/kg → good

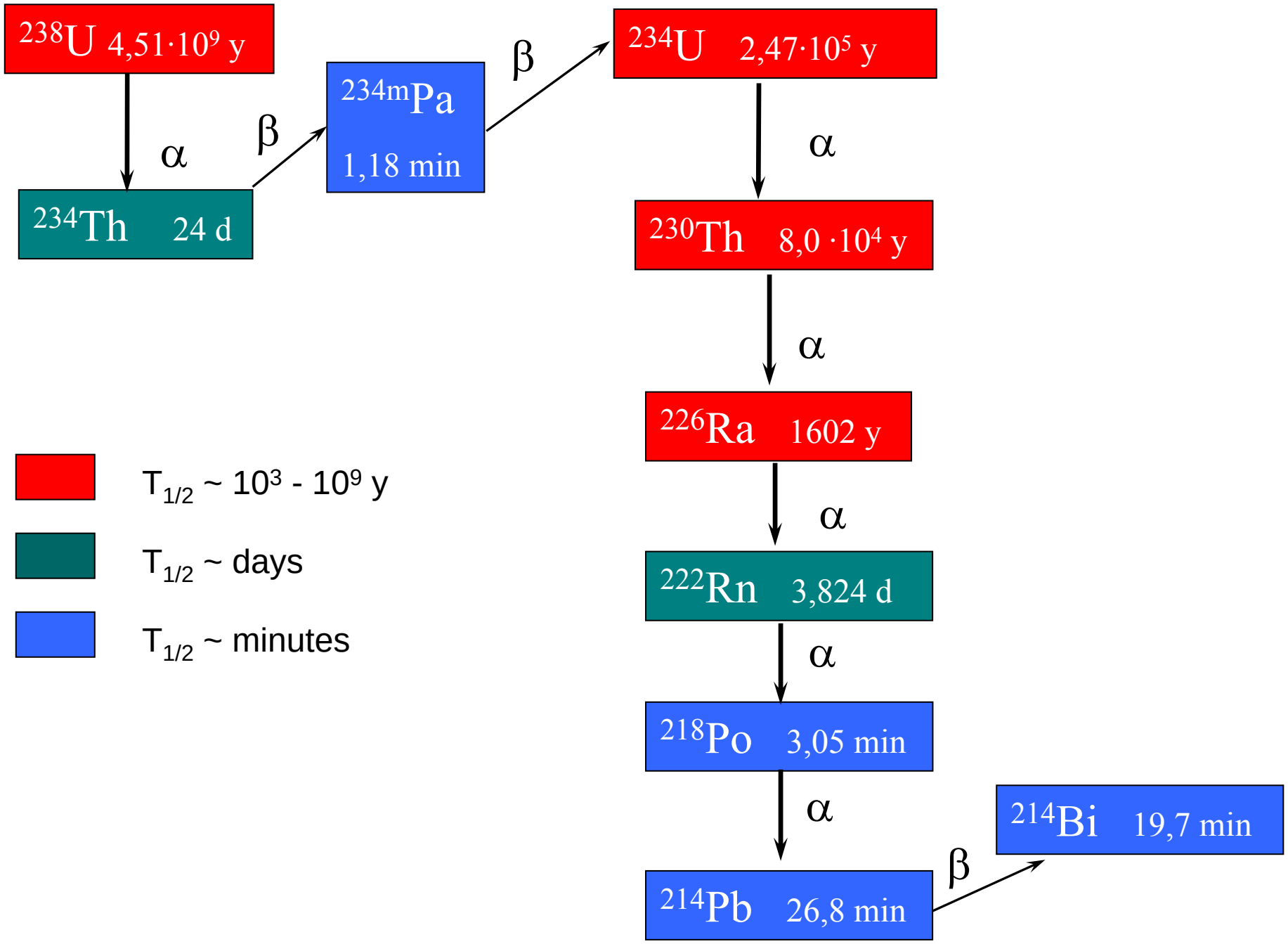
3. Content of Th-232 was defined at level of
   <= 1.2 – 4.8 mBq/kg (limit).

4. Such a level of Th-232 requires application of internal Cu passive shielding with modest thickness (in the cryostat design).
5. In 2007, AC IPTM-HPM RAS will start measurements with new set up: ELEMENT2 ICP MS (from Bremen).

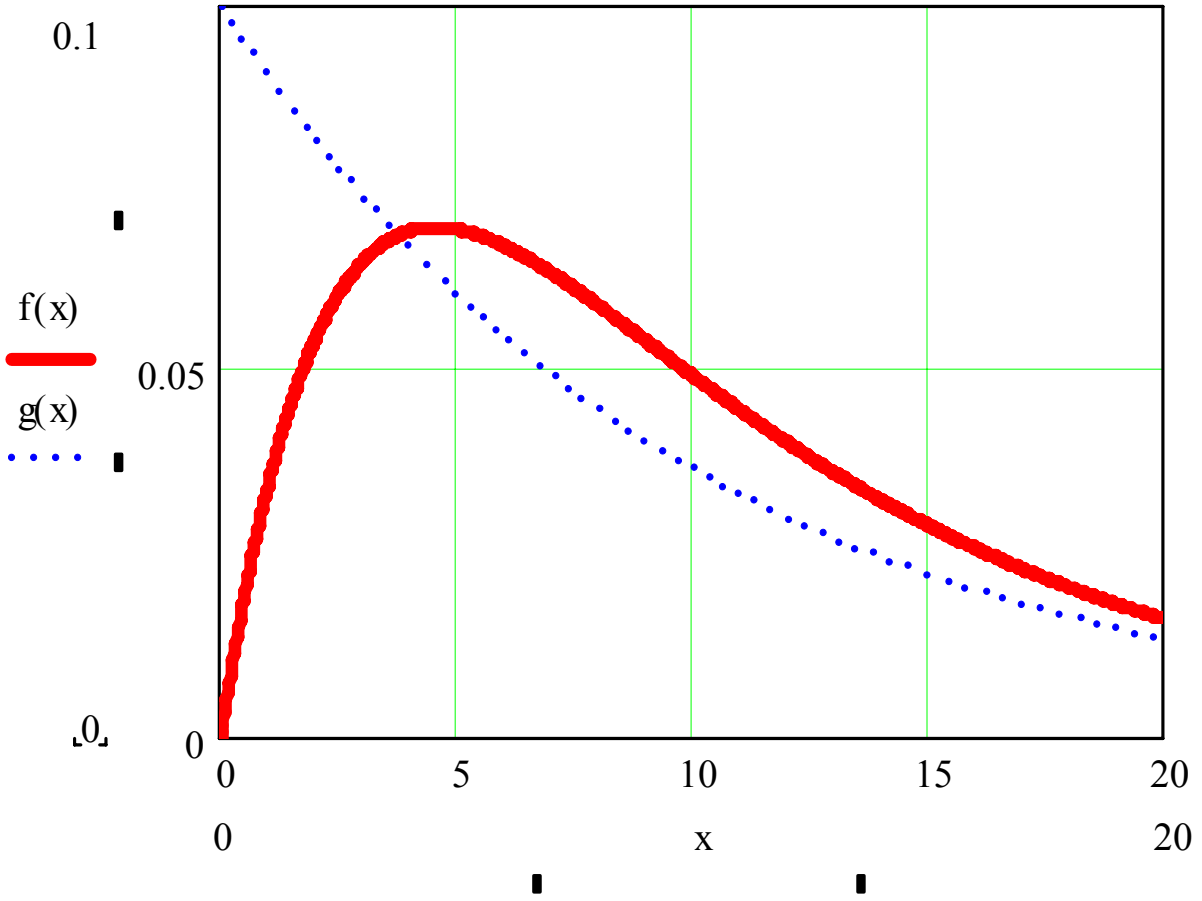
ICP MS: how it works




232Th 1,41·10^10 y
α
228Ra 5,75 y
β
228 Ac
6,15 hours
β
228Th 1,91 y
α
224Ra 3,64 d
α
220Rn 55,3 sec
α
216Po 0,158 sec
α
212Pb 10,64 h
β
212Bi 60,5 min
α
36,2 %
208Tl 3,10 min
T1/2 ~ 10^10 y
T1/2 ~ years
T1/2 ~ days
T1/2 ~ hours
T1/2 ~ seconds & minutes


238U 4,51·10⁹ y
α
234Th 24 d
β
234mPa 1,18 min
β
234U 2,47·10⁵ y
α
230Th 8,0 ·10⁴ y
α
226Ra 1602 y
α
222Rn 3,824 d
α
218Po 3,05 min
α
214Pb 26,8 min
β
214Bi 19,7 min
T1/2 ~ 10³ - 10⁹ y
T1/2 ~ days
T1/2 ~ minutes

Ra-228 and Th-228 activity



Milano 13 – 15 of November 2006

ICP MS measurements ss steel type 1.4571

Sample	Element	Concentration in solution µg/L	Content Activity in sample g/g	Th-232 mBq/kg	Ra228/Th228 (HPGe)
1 494257	U	0,000826	$5{,}1 \cdot 10^{-10}$		
	Th	0,001651	$1{,}0 \cdot 10^{-9}$	<4,0	< 0, 86/0,11
2 493553	U	0,001788	$1{,}2 \cdot 10^{-9}$		
	Th	0,001319	$8{,}5 \cdot 10^{-10}$	< 3,4	<3,3/=1,1
3 254533	U	0,001354	$1{,}0 \cdot 10^{-9}$		
	Th	0,001485	$9{,}5 \cdot 10^{-10}$	< 3,8	= 1,0/1,5
4 255455	U	0,000423	$2{,}9 \cdot 10^{-10}$		
	Th	0,001230	$8{,}3 \cdot 10^{-10}$	< 3,3	<3,0/=5,1
5 50609522	U	0,000590	$3{,}9 \cdot 10^{-10}$		
	Th	0,001192	$8{,}0 \cdot 10^{-10}$	< 3,2	<1,0/0,41
6 charge	U	0,002035	$1{,}5 \cdot 10^{-9}$		
#5991	Th	0,000836	$6{,}1 \cdot 10^{-10}$	< 2,4	<2,9/=5,1