Radiochemical Analysis- experience and challenges

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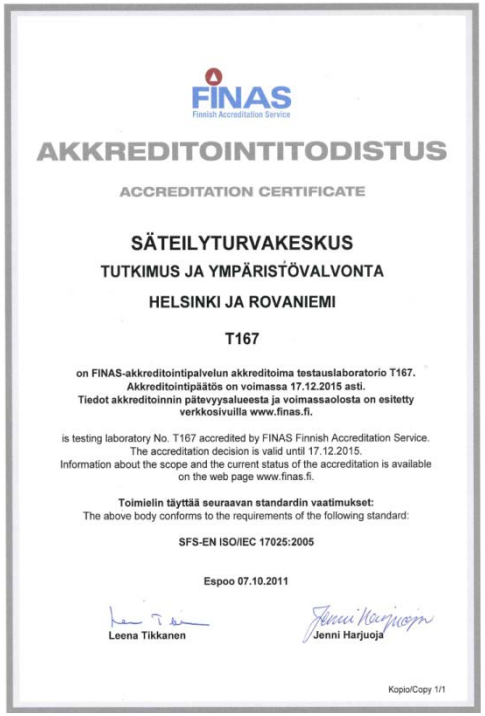
NKS-B RadWorkshop 2018

10.10.2018

Environmental Radiation Surveillance and Emergency Preparedness is accredited department

- The accreditation field is “Environmental testing”

- This accreditation is an indication that our institution meets the quality set by an ISO 17025 standard.



FINAS
Finnish Accreditation Service

AKKREDITOINTITODISTUS

ACCREDITATION CERTIFICATE

SÄTEILYTURVAKESKUS
TUTKIMUS JA YMPÄRISTÖVALVONTA
HELSINKI JA ROVANIEMI

T167

on FINAS-akkreditointipalvelun akkreditoima testauslaboratorio T167.
Akkreditointipäätös on voimassa 17.12.2015 asti.
Tiedot akkreditoinnin pätevyysalueesta ja voimassaolosta on esitetty verkkosivuilla www.finas.fi.

is testing laboratory No. T167 accredited by FINAS Finnish Accreditation Service.
The accreditation decision is valid until 17.12.2015.
Information about the scope and the current status of the accreditation is available on the web page www.finas.fi.

Toimielin täyttää seuraavan standardin vaatimukset:
The above body conforms to the requirements of the following standard:

SFS-EN ISO/IEC 17025:2005

Espoo 07.10.2011

Leena Tikkanen

Jenni Harjuoja

Kopio/Copy 1/1

Fields of testing:

- Advanced gammaspectrometric analyses
- Gammaspectrometric measurements (K-40, I-131, Cs-134, Cs-137, decay series of U-238, U-235 and Th-232)
- Direct measurements of people
- Method for local laboratories, NaI(Tl) detector
- Radiochemical analyses
  - Plutonium
  - Strontium
  - H-3
  - Uranium
  - Lead
  - Polonium
  - Radon in water
  - Gross alpha
- Uranium, strontium determinations using ICP-MS
- Airborne radon concentrations
- Sampling for environmental surveillance of radioactivity

Environmental surveillance and measurement laboratory

The laboratory provides analysis services both for internal and external customers:

- Gamma spectrometric analyses
- Radiochemical analyses measured by liquid scintillation counters, proportional counters or using alpha spectrometry
- Radiation monitoring programme in the vicinities of the Finnish nuclear power plants
- Baseline studies of mining and nuclear facility areas
- Sampling of environmental samples
- CTBT (Comprehensive Nuclear-Test-Ban Treaty) laboratory analyses

Environmental surveillance and measurement laboratory

- Environmental laboratory handling of low activities
- Most of laboratories are type C as a normal chemistry laboratory, one type B laboratory



Challenges in radiochemical analysis

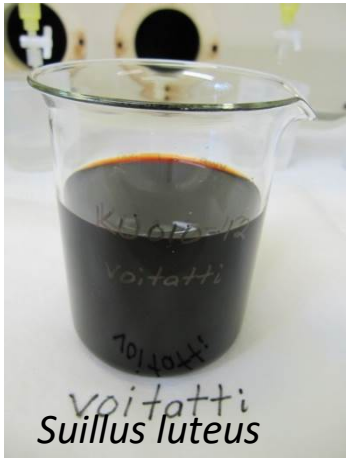
- Activity concentration in environmental samples
- Digestion of the samples
- High concentration of Fe
- Organic matter
- High amount of Si
- Natural radionuclides
- Etc.

Determination of uranium from environmental samples

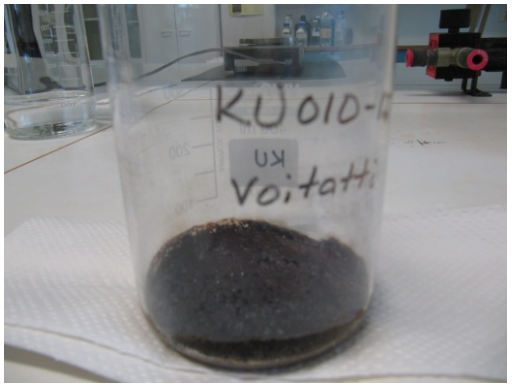
- Challenges :
  - High concentration of Fe → yield of uranium poor
  - Indissoluble organic matter follows to the final source preparation → impurities in alpha spectrum
  - Po-210 (5,304 MeV) impurity, U-232 (5,320 MeV)
  - High amount of Si → problems with the ion exchange

Determination of uranium from environmental samples

- Solutions :
  - Oxidation of organic matter with HNO_3 and hydrogen peroxide (H_2O_2), repeated several times. After oxidation process, samples are burned in 450°C if needed

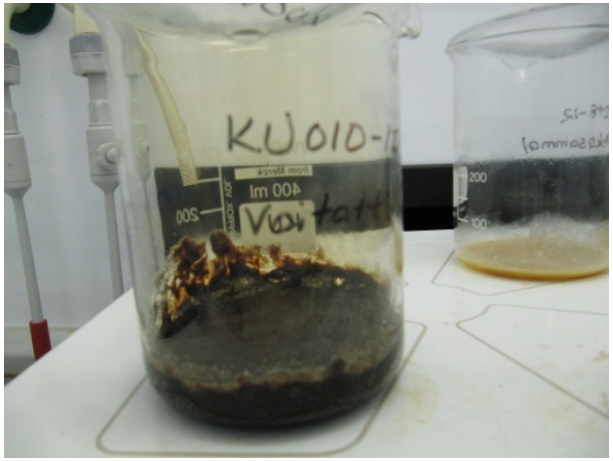


Sample after MARS5- digestion

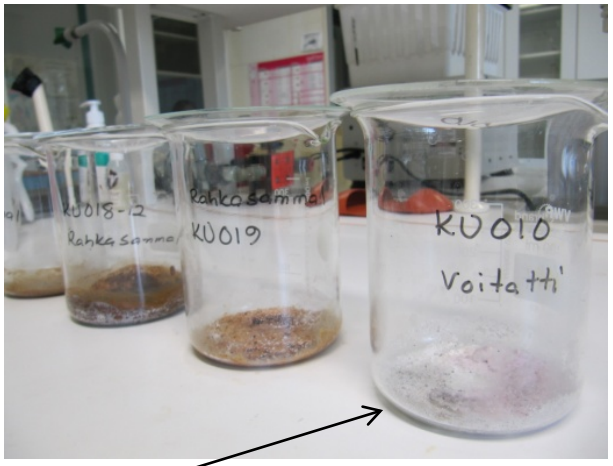


Sample after evaporation

Determination of uranium from environmental samples



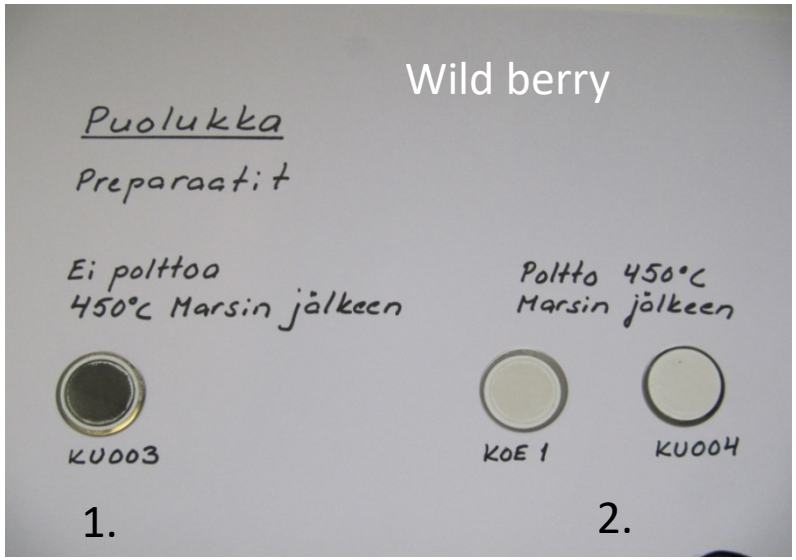
Beginning of the oxidation with H_2O_2 and HNO_3



After the oxidation and 450°C

Determination of uranium from environmental samples

- Precipitated sample for the alpha spectrometry



1. No extra oxidation and burning after MARS-digestion

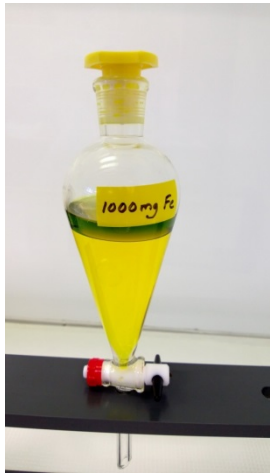
2. Extra burning after MARS-digestion and oxidation

Determination of uranium from environmental samples

- Solutions :
  - High amount of silica (for example in plant samples)
    problem: precipitated silica, ion exchange column is blocked

Extra oxidation **and** Extra ashing

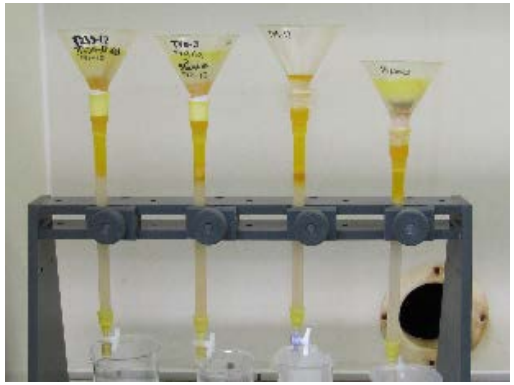
  - High amount of Fe (for example in mushrooms, in some moss samples)
    → reduction of Fe(III) first by ascorbic acid, after that with $TiCl_3$
    → extraction of Fe into diethyl ether (Fe 99%, U 0%)

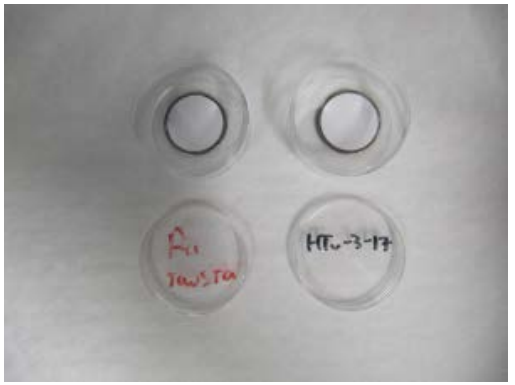


Determination of uranium from environmental samples

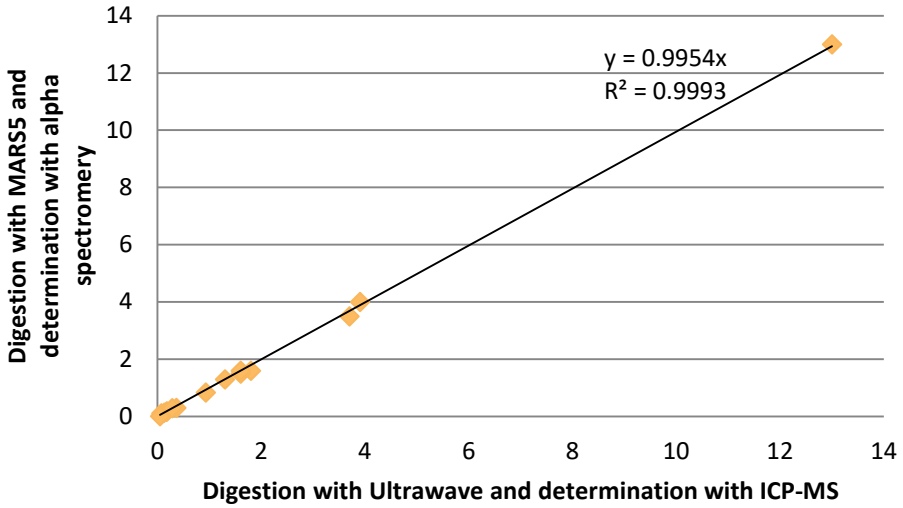
- Po-210 (for example in plant samples)

  → repeat the ion exchange with new anion exchange column





Digestion the samples with high pressure and temperature

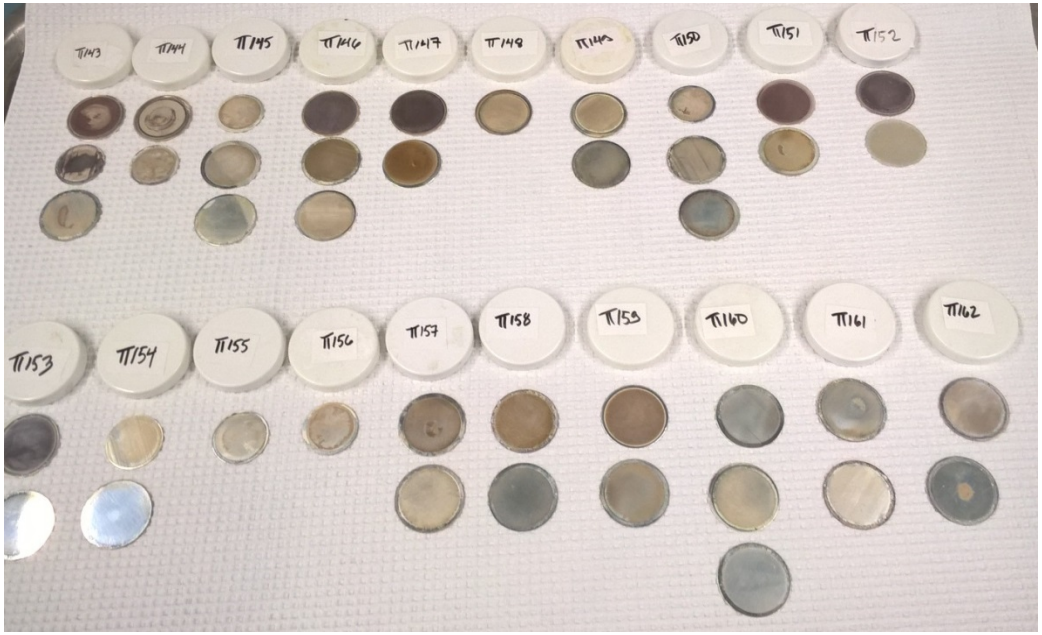


220°C, 90 bar

Industrial samples

Metal-concentrate samples:

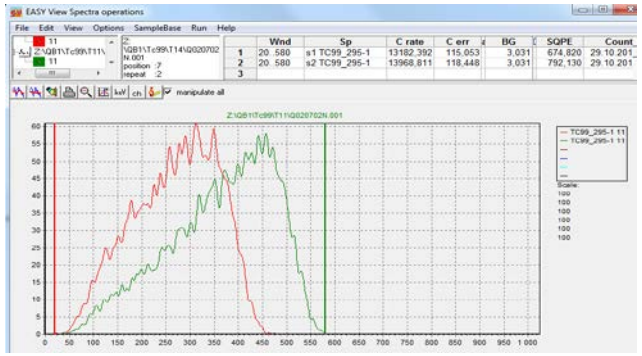
- Amount of sample in analysis: suitable amount below 1 g in Po deposition for alpha spectrometry



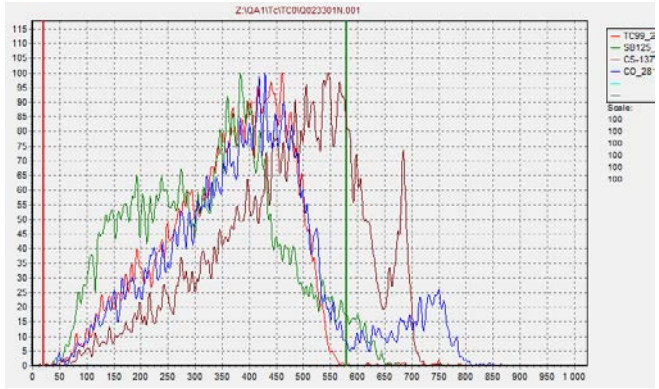
Determination of Tc-99 from waste samples

- Challenges :
  - High radioactivity concentration (laboratory facilities)
  - High concentrations of other radionuclides (Co, Sb, Cs)
  - Half life of the tracer

Liquid scintillation countings

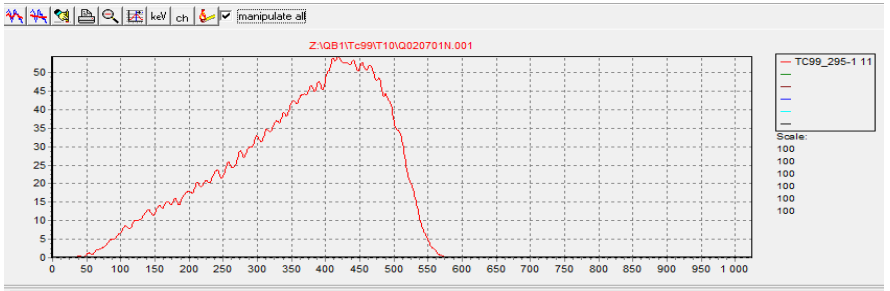


Counting efficiency, Quenching

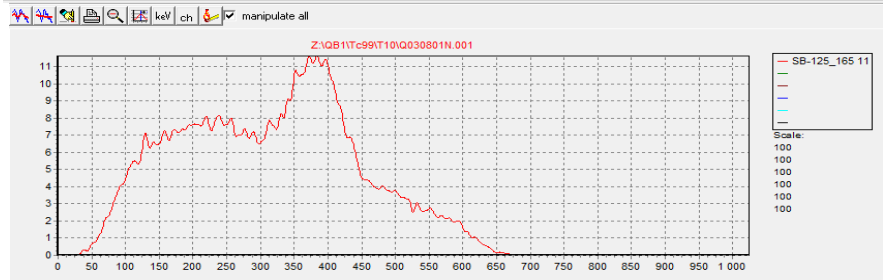


Tc-99, Sb-125, Cs-137, Co-60

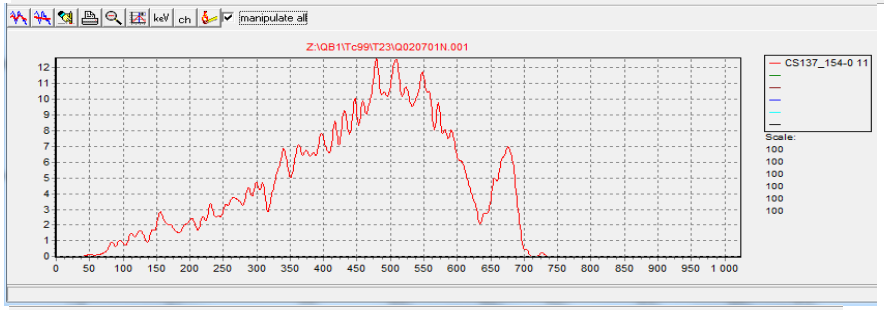
Tc-99



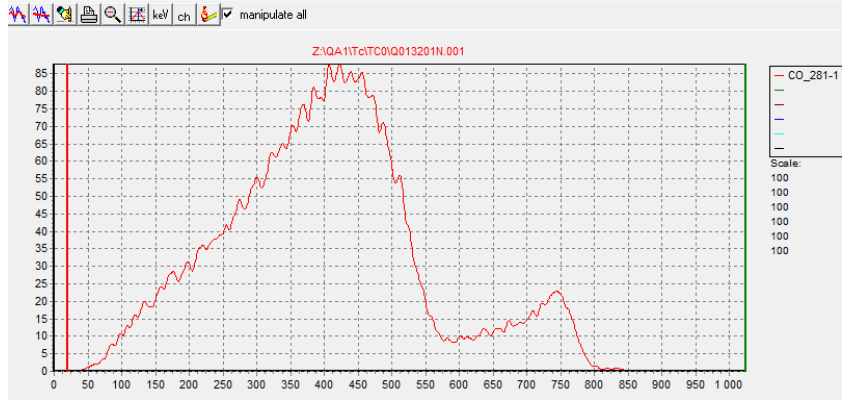
Sb-125



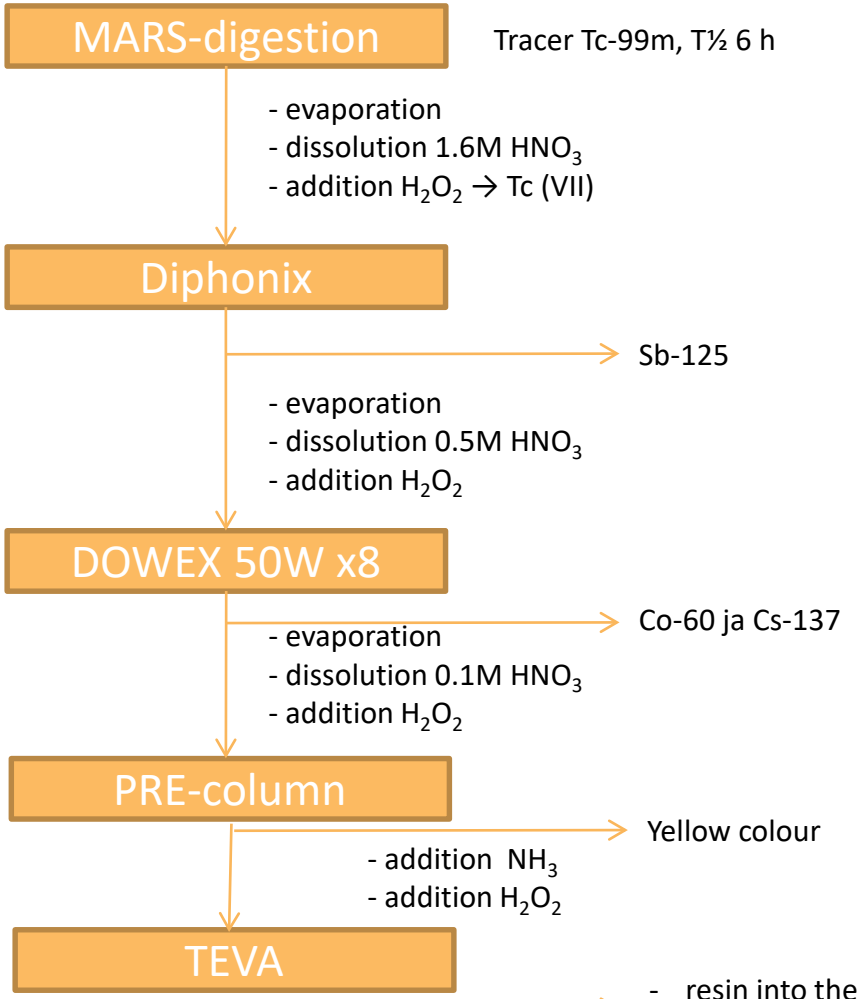
Cs-137



Co-60



Determination of Tc-99 from waste samples



Tc-99 < 10 Bq/g

Thank you!