Accelerator Mass spectrometry (AMS) for radionuclide analysis

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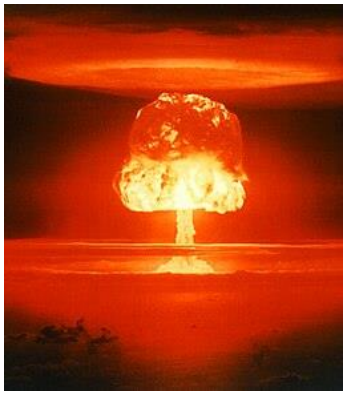
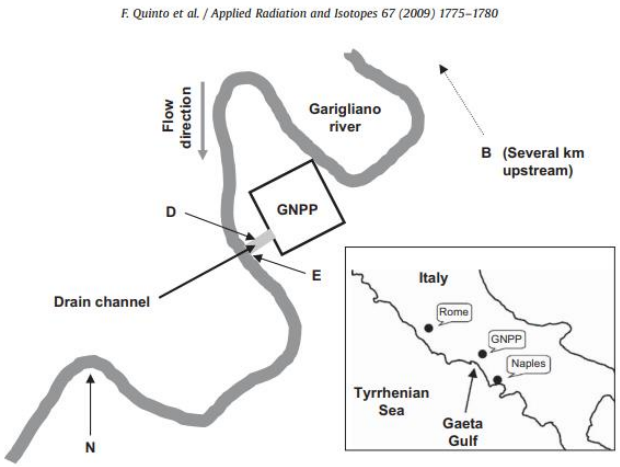
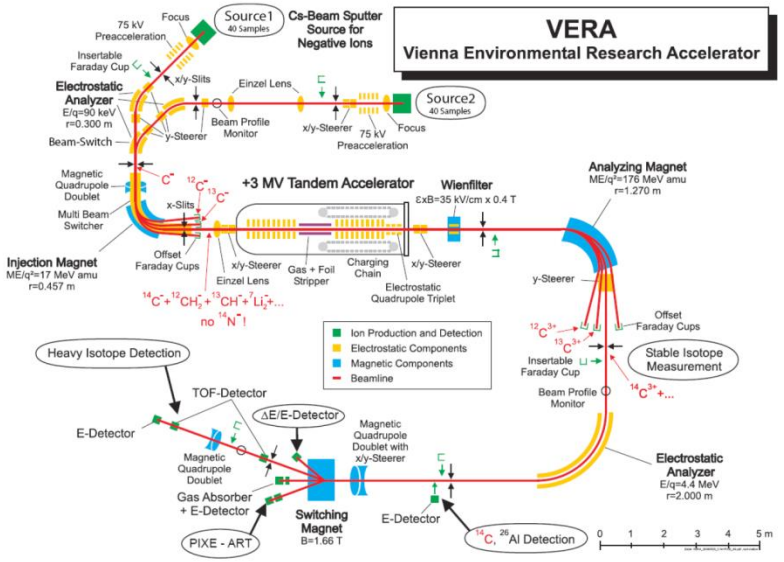






The first radionuclide determined with the highest sensitivity with AMS

^{14}C ($t_{1/2}$ = 5700 ± 30 y)

In nature: spallation on ^{14}N target atoms in the atmosphere

$$^{14}\mathrm{N}\ (\mathrm{n}, \mathrm{p})\ ^{14}\mathrm{C}$$

From nuclear reactors and nuclear bomb tests:

$$^{14}\mathrm{N}\ (\mathrm{n}, \mathrm{p})\ ^{14}\mathrm{C}$$

$$^{13}\mathrm{C}\ (\mathrm{n}, \gamma)\ ^{14}\mathrm{C}$$

$$^{17}\mathrm{O}\ (\mathrm{n}, \alpha)\ ^{14}\mathrm{C}$$

In contemporary carbon $^{14}C/^{12}C = 1.2 \times 10^{-12}$

Several orders of magnitude higher

a wide range of $^{14}C/^{12}C$ ratios in environmental samples starting from 10^{-15}

An example:

1 mg C with a natural $^{14}C/^{12}C = 1.2 \times 10^{-12}$

- **6×10^7 atoms ^{14}C**
- **$t_{1/2}$ = 5700 y**
- **Radiometric method: ca. 1 decay of ^{14}C in one hour = 0.28 mBq (< DL of Liquid Scintillation)**
- **Mass Spectrometry: atom counting**
- **AMS => Isobaric interference are removed:**

✓ **$^{14}N^-$ in the ion source**

✓ **$^{12}CH_2^-$ and $^{13}CH^-$ in the terminal of the accelerator**

- **AMS: 6 x 10^7 atoms ^{14}C counted with an efficiency of 1% in one hour with a precision ca. 0.1%**

An example:

1 µg C with a natural $^{14}C/^{12}C = 1.2 \times 10^{-12}$

- **6×10^4 atoms ^{14}C**
- **$t_{1/2}$ = 5700 y**
- **Radiometric method: ca. 0.001 decay of ^{14}C in one hour = 0.00028 mBq (<< DL of Liquid Scintillation)**
- **Mass Spectrometry: atom counting**
- **AMS => Isobaric interference are removed:**

✓ **$^{14}N^-$ in the ion source**

✓ **$^{12}CH_2^-$ and $^{13}CH^-$ in the terminal of the accelerator**

- **AMS: 6×10^4 atoms ^{14}C counted with an efficiency of 1% in one hour with a precision of ca. 4%**

Which Radionuclides?

- $t_{1/2}$ ≤ or > observation time
  +
- [radionuclide] ↑ high

→ decay counting → radiometric techniques

γ, β, α

m

- $t_{1/2}$ >> observation time
  +
- [radionuclide] ↓ low

→ atom counting

→ mass spectrometry (MS)

→ ultra-trace! → accelerator mass spectrometry (AMS)

Analytical Capability of AMS

Overall Efficiency:
$^{14}C = 10^{-2}$
$^{129}I = 10^{-3}$
actinide nuclides = 10^{-4}

→ 10^5 atoms of an actinide nuclide (corresponding to tens of attograms) is possible with a 32 % relative uncertainty

A precision of 1%:
10^6 atoms of ^{14}C
10^7 atoms of ^{129}I
10^8 atoms of an actinide nuclide

→ at least 4 and up to 7 orders of magnitude less atoms than a radiometric technique to perform equally precise measurements, with consequent reduction of sample size

abundance sensitivities:
$^{14}C/^{12}C = 10^{-15}$
$^{129}I/^{127}I = 10^{-14}$
$^{236}U/^{238}U = 10^{-13}$

→ unambiguous nuclide detection in environmental samples at concentration levels below ppq

AMS one of the most sensitive analytical techniques for the investigation of rare long-lived radionuclides in the environment

Why Accererator Mass Spectrometry?

Why ultra-trace analysis of long-lived radionuclides?

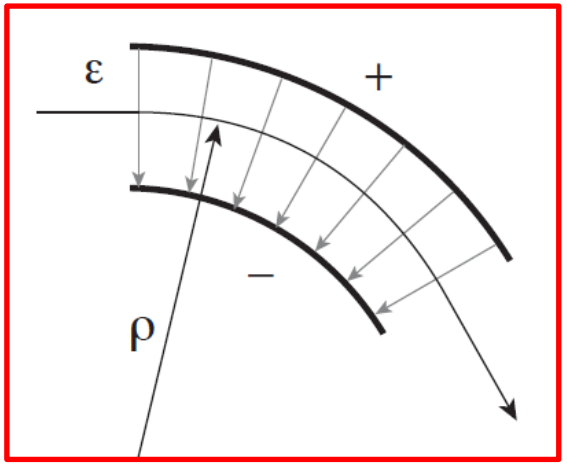
They pose no concern to radioprotection

What can we learn from such ultra-trace analysis in Environmental Research and Decommissioning & Radioactive Waste Disposal

Mass Spectrometry

A technique allowing the identification of an atom (nuclide) according to its mass

If a nuclide of mass= m and velocity= v has a certain atomic charge= q:



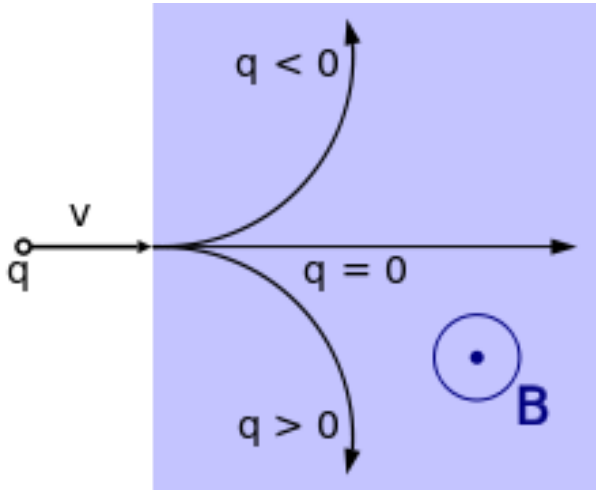
- **In an electric field, ε:**
  the ion has a trajectory with radius,

$$\rho = mv^2/q\varepsilon = 2E/q\varepsilon$$

with $E = 1/2\ mv^2$

- **In a magnetic field, B, with direction perpendicular to v: the ion has a trajectory with radius,**

$$\rho = mv/qB$$



Mass Spectrometry

A technique allowing the identification of an atom (nuclide) according to its mass

1. **Chemical preparation of the sample**
2. **Ions must be produced => ion source**
3. **Ions must be selected according to their m/q and/or E/q ratios => magnetic and electrostatic „filtering devices“**
4. **Selected ions must be detected => detector**

Accelerator Mass Spectrometry

Like in other Mass Spectrometric Techniques:

- Atoms are extracted from a sample and are ionized
- Accelerated to high energies
- Separated according to their momentum, charge and energy
- Individually counted after identification as having correct atomic number and mass

✓ **BUT in AMS the ions are accelerated at MeV by using ion accelerators**

Facilities used for AMS

Big

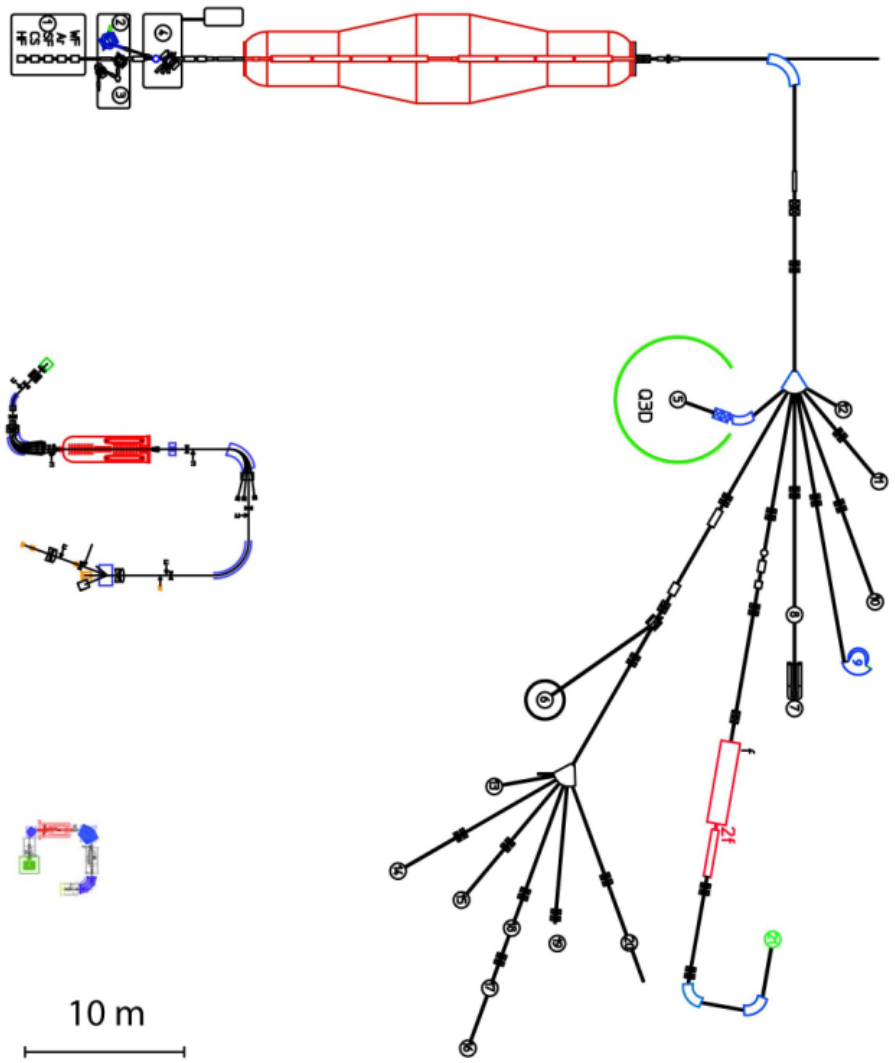
15 MV Tandem
TU and LMU München
Germany

↔

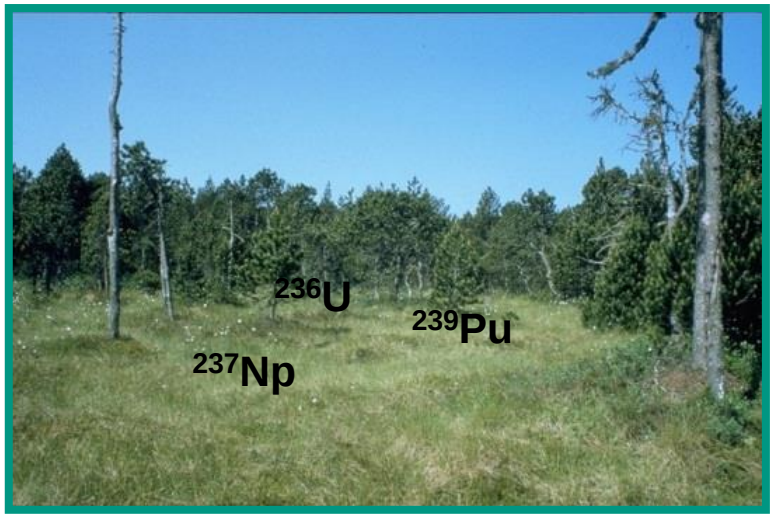
3 MV Tandem VERA
Universität Wien
Austria

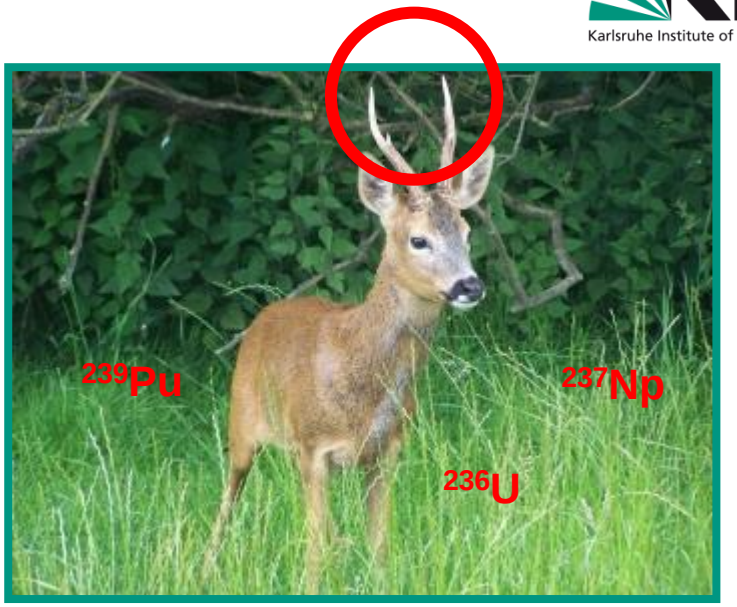
Small

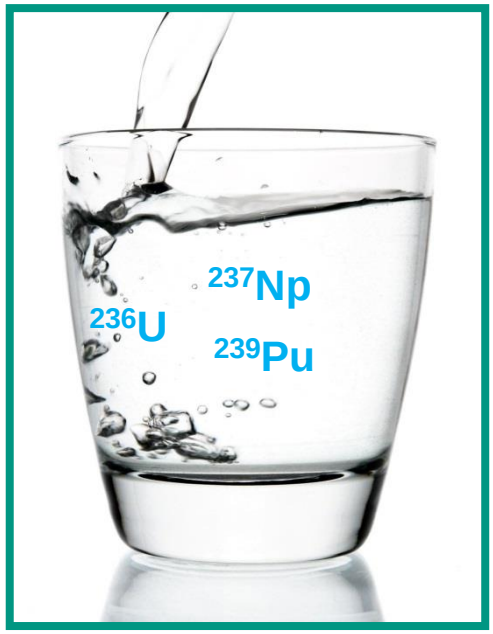
0.5 MV Tandem
ETH Zürich
Switzerland

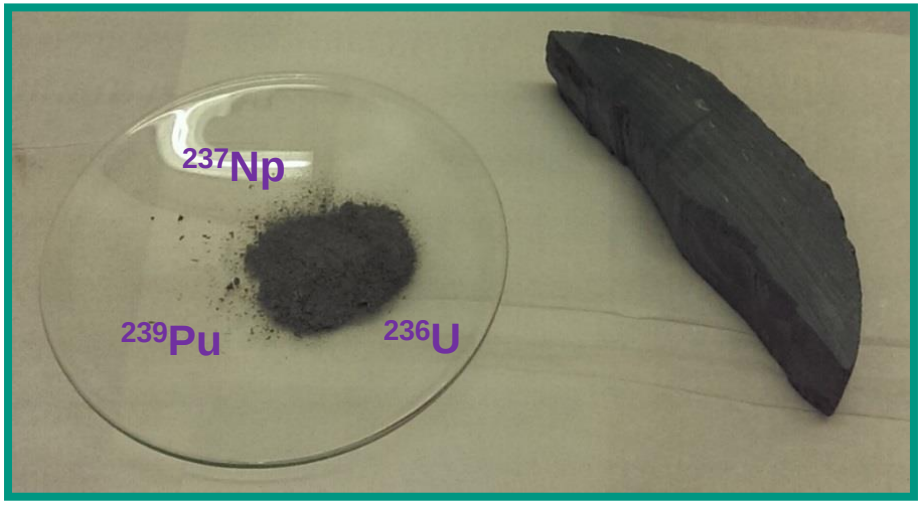


Samples

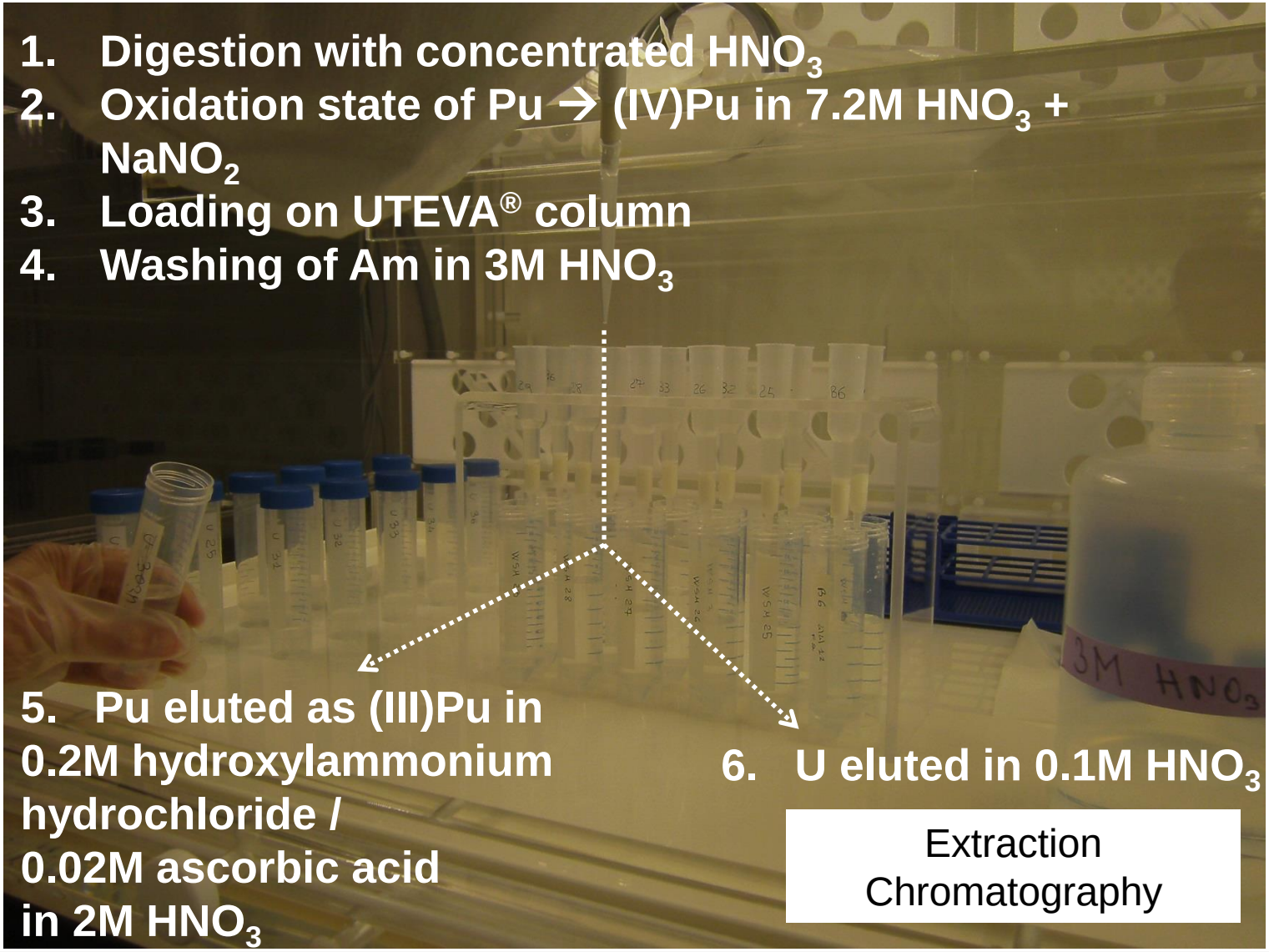

^{236}U
^{239}Pu
^{237}Np


^{239}Pu
^{237}Np
^{236}U


^{237}Np
^{236}U
^{239}Pu

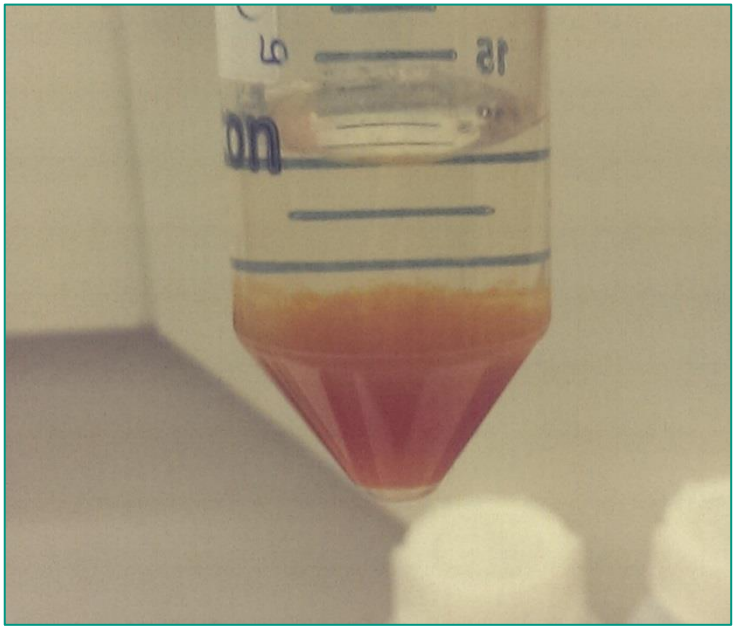

^{237}Np
^{239}Pu
^{236}U

Sample Preparation: separation of Pu and U fractions

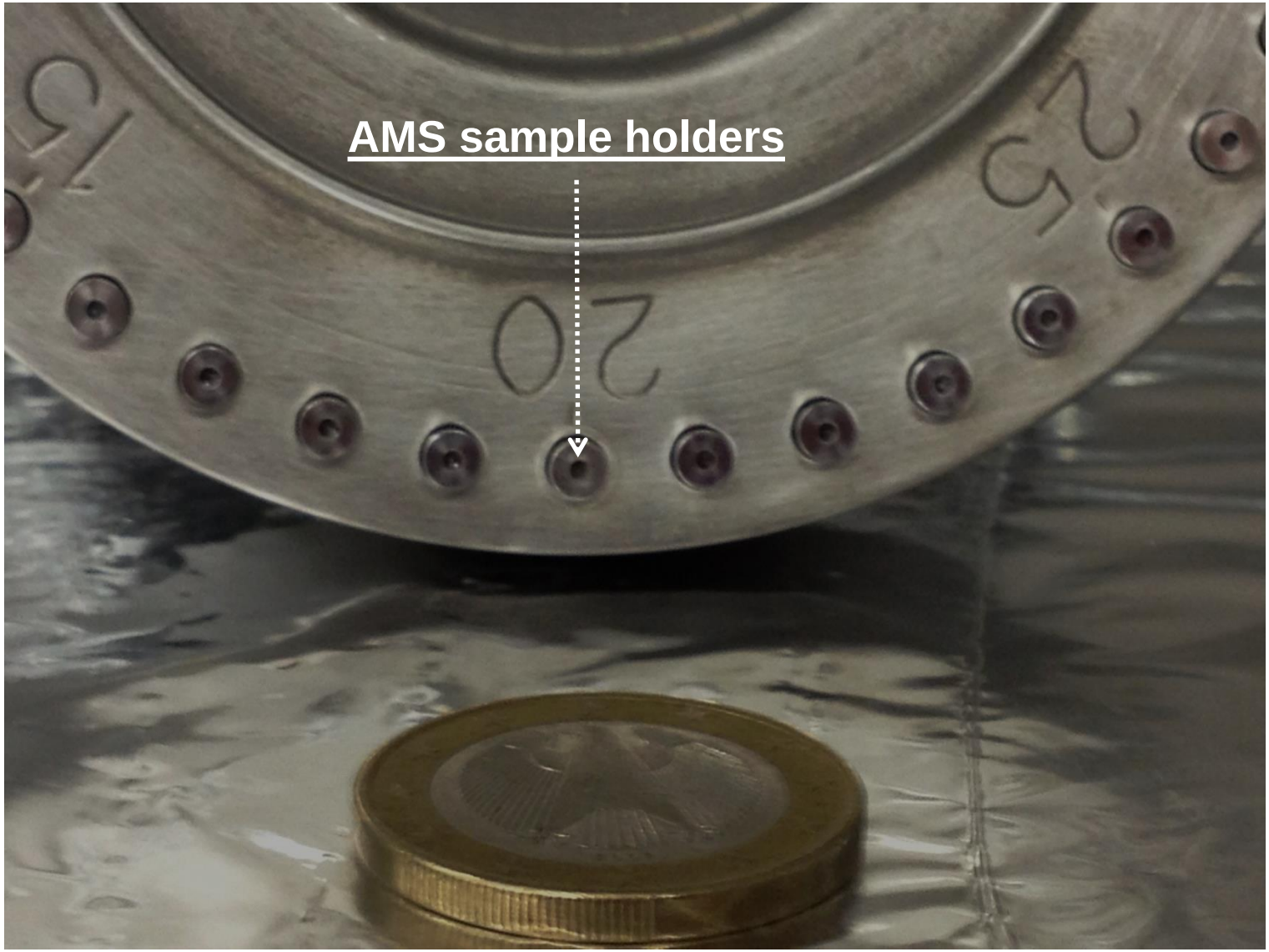


Sample Preparation: AMS sample holders

1. $Fe(OH)_3$ co-precipitation
2. Conversion to iron oxide at 900 ^{0}C
3. Pressing into the AMS sample holders

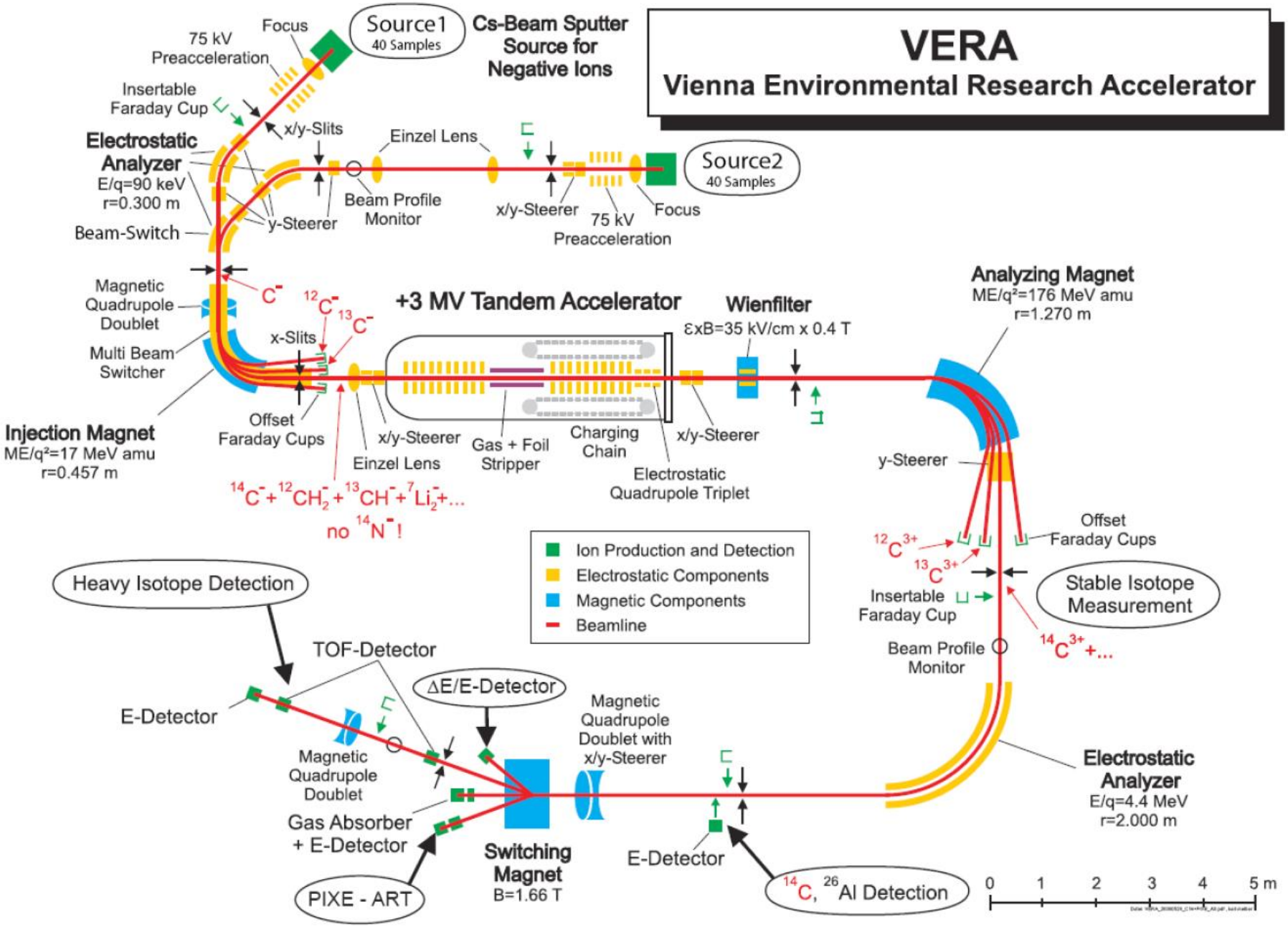


Sample Preparation: AMS sample holders

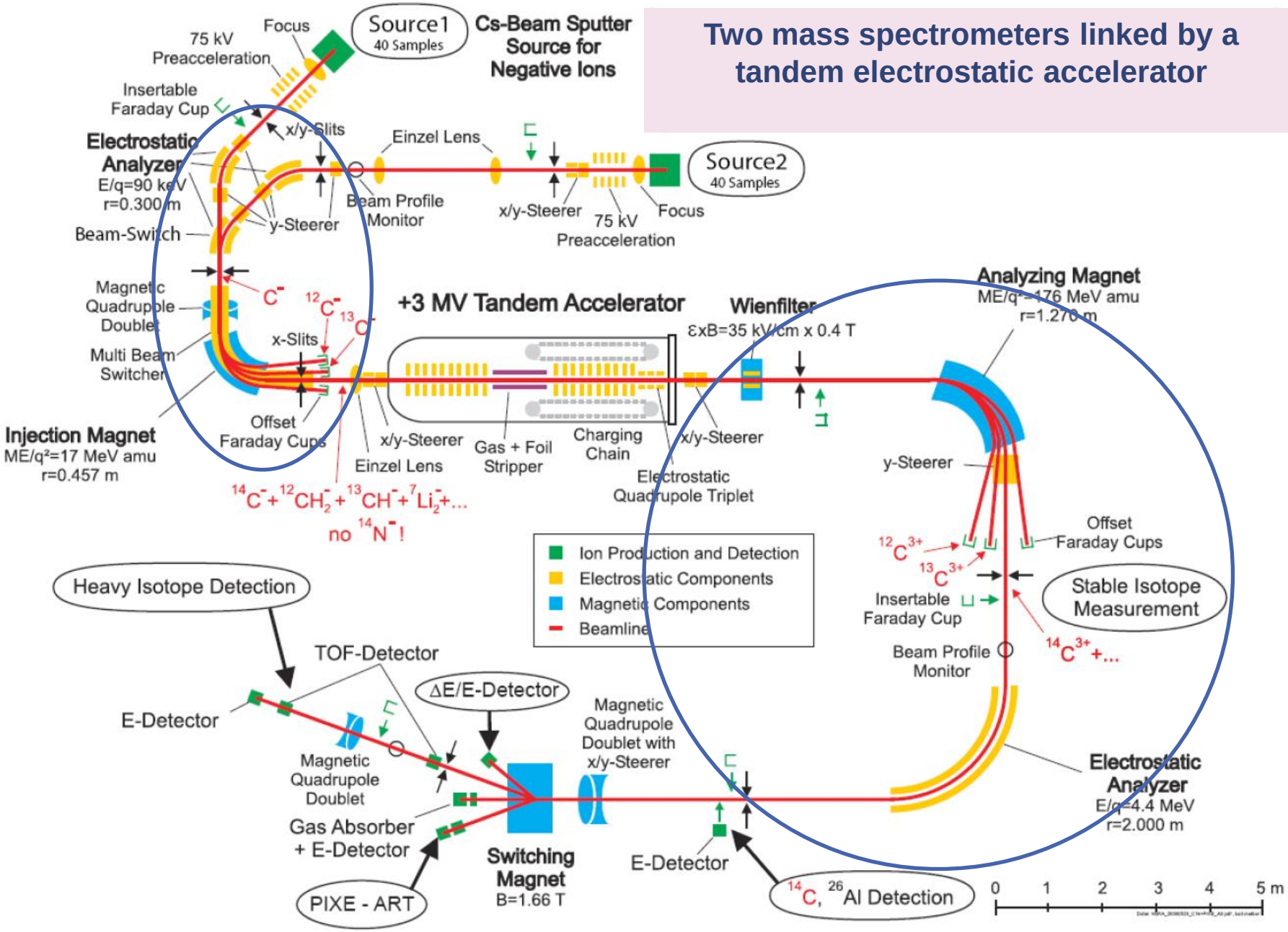


Principal Components of AMS

- ❑ Ion source
- ❑ Filtering devices,e.g.:
  - Dipole magnets
  - Electrostatic analyzers
- ❑ Devices for the destruction of molecular isobars:
  - Tandem Electrostatic Accelerator


VERA
Vienna Environmental Research Accelerator
Source1
40 Samples
Cs-Beam Sputter Source for Negative Ions
Focus
75 kV Preacceleration
Insertable Faraday Cup
Electrostatic Analyzer
E/q=90 keV
r=0.300 m
Beam-Switch
x/y-Slits
Einzel Lens
Beam Profile Monitor
y-Steerer
x/y-Steerer
75 kV Preacceleration
Focus
Source2
40 Samples
Magnetic Quadrupole Doublet
Multi Beam Switcher
Injection Magnet
ME/q²=17 MeV amu
r=0.457 m
C⁻
12C⁻
13C⁻
x-Slits
Offset Faraday Cups
+3 MV Tandem Accelerator
x/y-Steerer
Einzel Lens
Gas + Foil Stripper
Charging Chain
Electrostatic Quadrupole Triplet
Wienfilter
εxB=35 kV/cm x 0.4 T
Analyzing Magnet
ME/q²=176 MeV amu
r=1.270 m
y-Steerer
14C⁻+12CH2⁻+13CH⁻+7Li2⁻+...
no 14N⁻!
Ion Production and Detection
Electrostatic Components
Magnetic Components
Beamline
12C3+
13C3+
Offset Faraday Cups
Insertable Faraday Cup
Beam Profile Monitor
14C3+...
Stable Isotope Measurement
Heavy Isotope Detection
TOF-Detector
E-Detector
ΔE/E-Detector
Magnetic Quadrupole Doublet
Gas Absorber + E-Detector
PIXE - ART
Switching Magnet
B=1.66 T
Magnetic Quadrupole Doublet with x/y-Steerer
E-Detector
14C, 26Al Detection
Electrostatic Analyzer
E/q=4.4 MeV
r=2.000 m
0 1 2 3 4 5 m

Two mass spectrometers linked by a tandem electrostatic accelerator



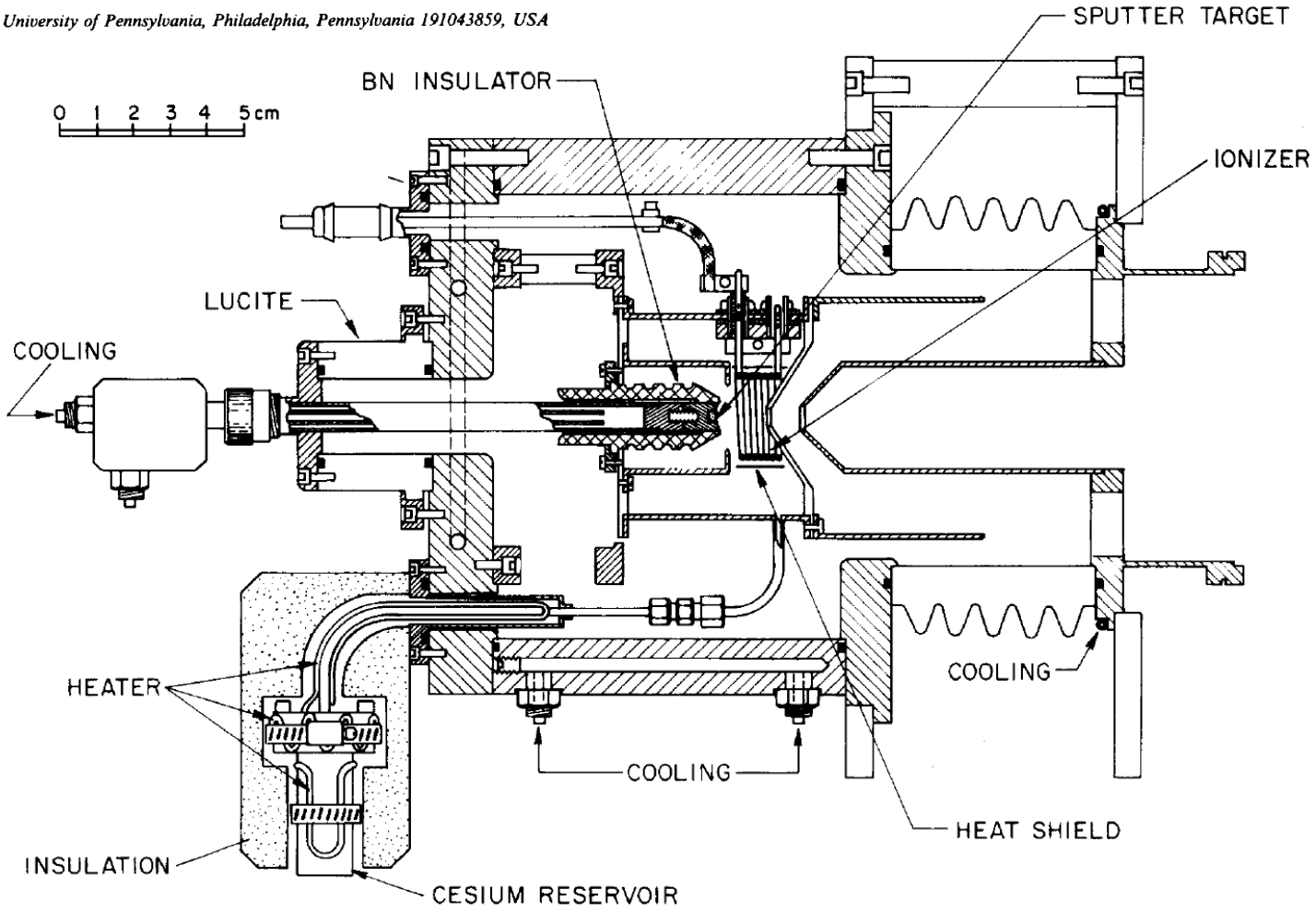
Nuclear Instruments and Methods in Physics Research 220 (1984) 105–106
North-Holland, Amsterdam

Cs sputtering negative ion source

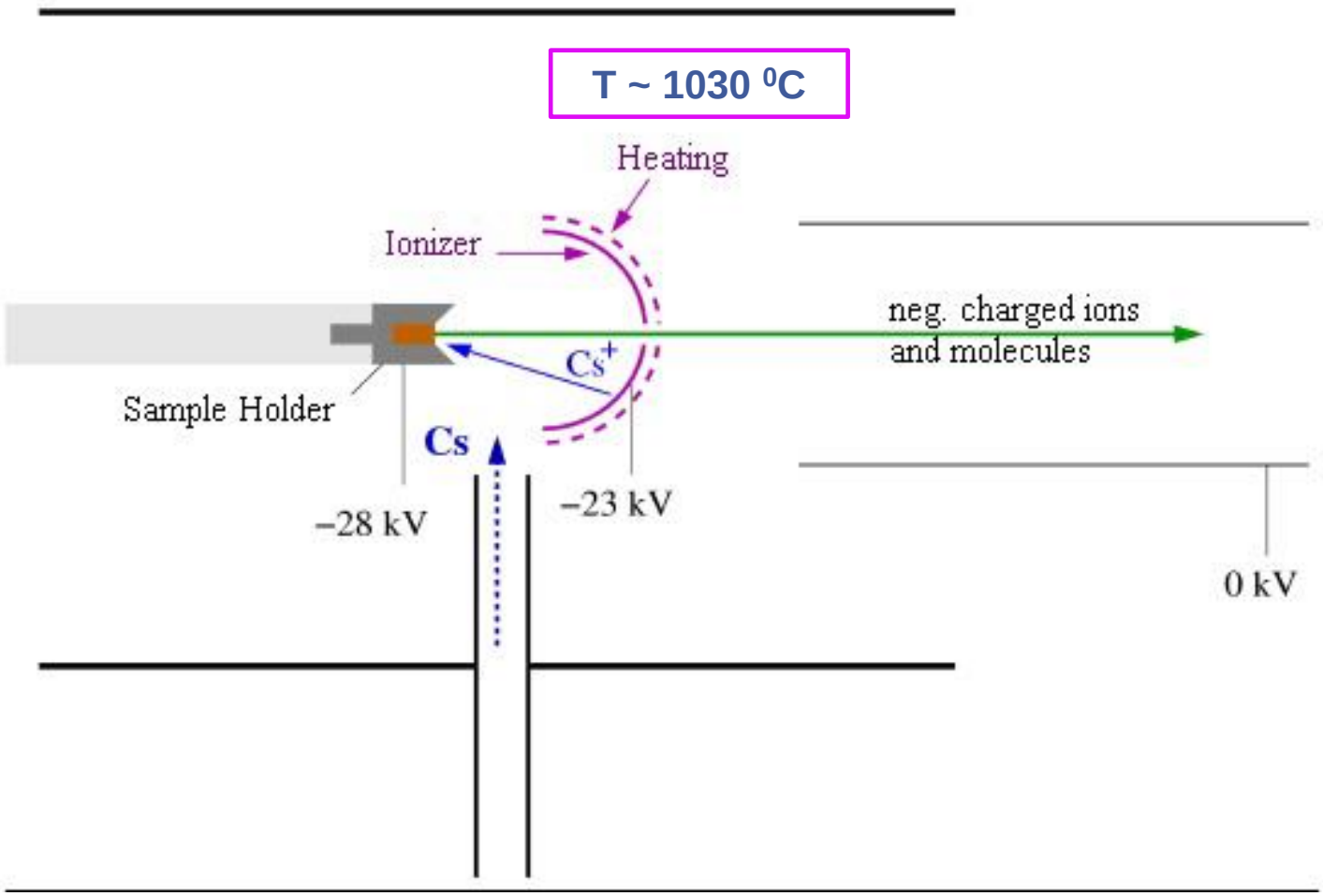
Section III. Ion sources

A VERSATILE HIGH INTENSITY NEGATIVE ION SOURCE *

R. MIDDLETON
Physics Department, University of Pennsylvania, Philadelphia, Pennsylvania 191043859, USA

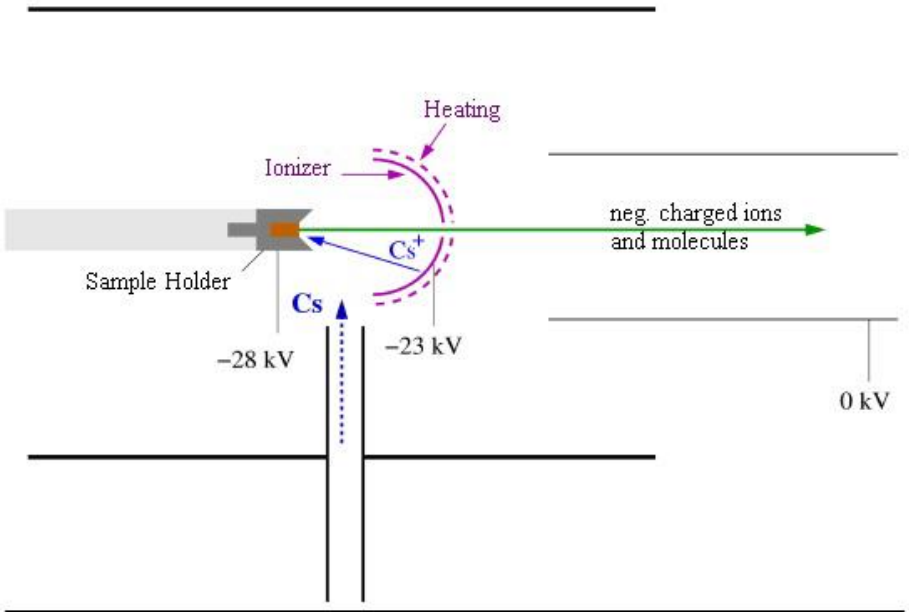


Cs sputtering negative ion source



Diplomathesis P. Ludwig (2010) Schematic display of a high-current cesium sputter source of Middleton type with a spherical ionizer.

Cs sputtering negative ion source



~~$^{14}N^-$~~

~~$^{129}Xe^-$~~

$^{14}C^-$

$^{129}I^-$

$^{236}UO^-$

$^{244}PuO^-$

Separation of atomic isobars !

Diplomathesis P. Ludwig (2010) Schematic display of a high-current cesium sputter source of Middleton type with a spherical ionizer.

Sputtered AMS targets

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journal homepage: www.elsevier.com/locate/nimb

Ionisation efficiency improvements for AMS measurement of actinides

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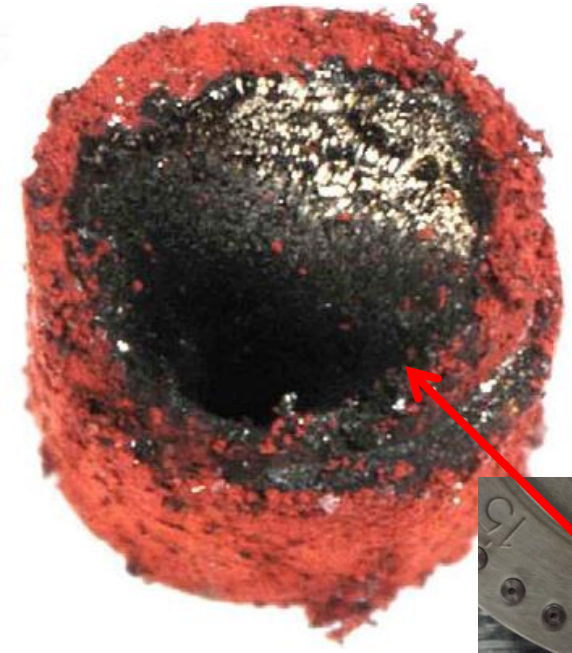
[b] Jožef Stefan Institute, Jamova 39, SI-1000 Ljubljana, Slovenia



Nuclear Instruments and Methods 214 (1983) 139–150
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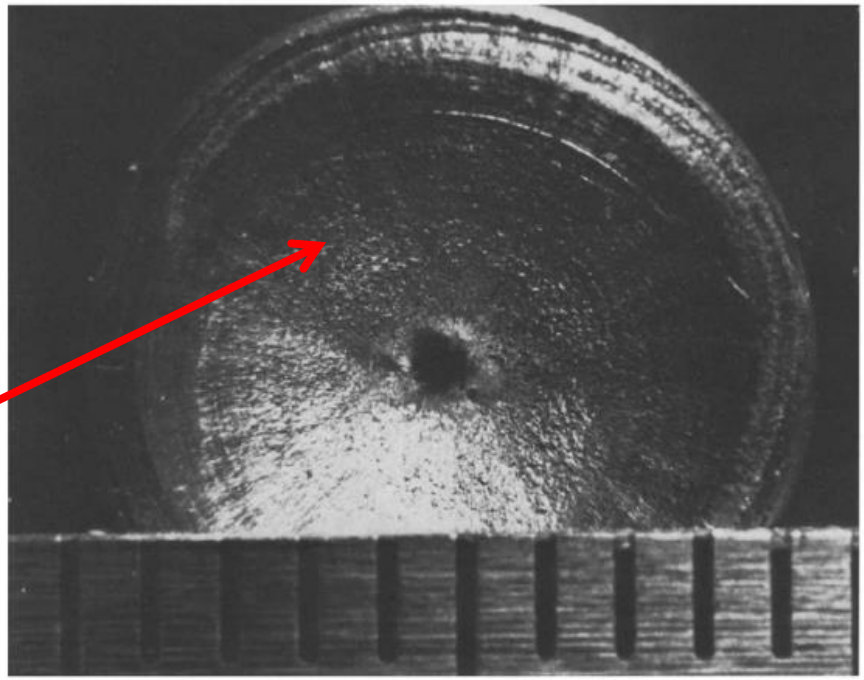
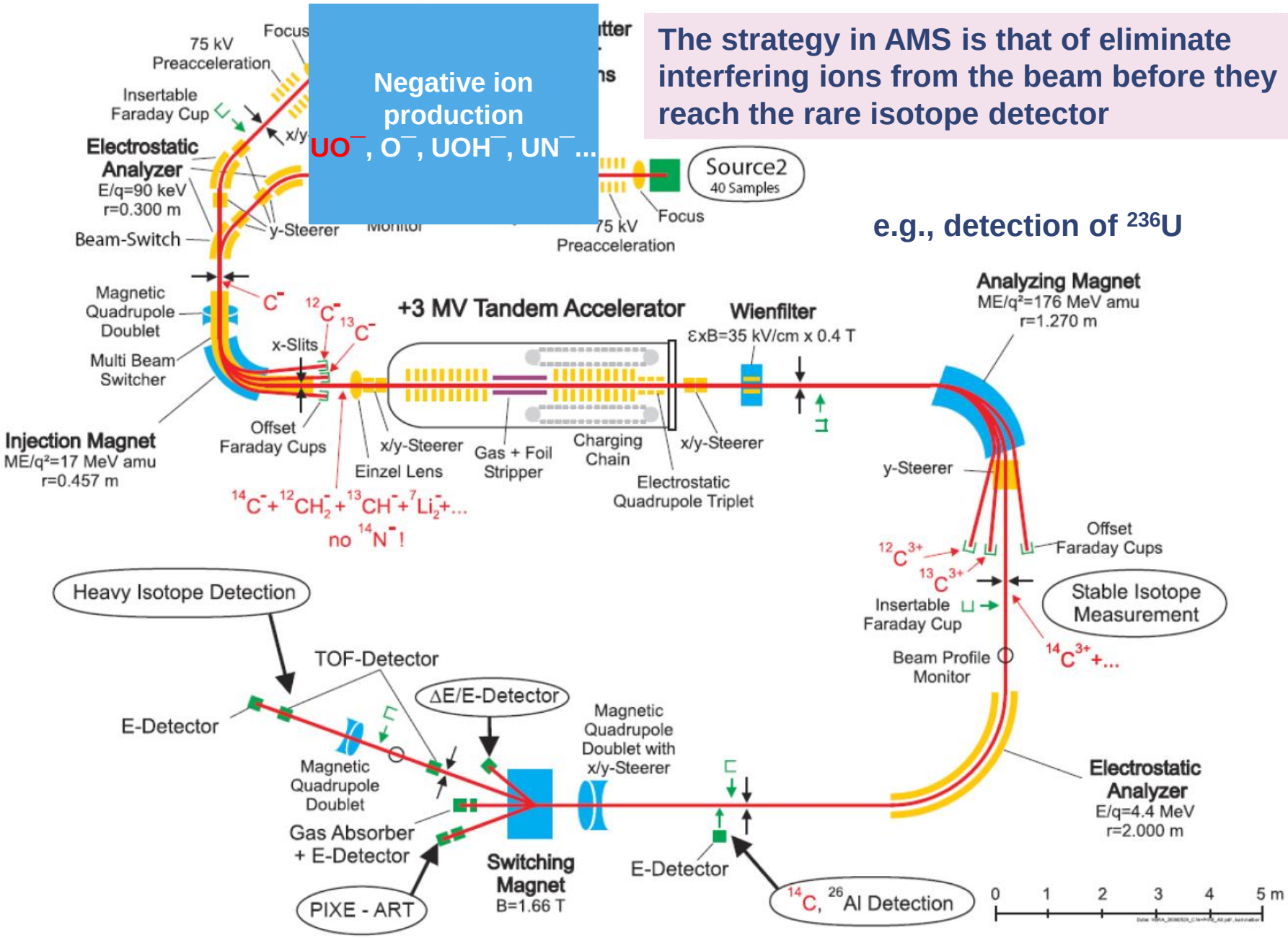
A VERSATILE HIGH INTENSITY NEGATIVE ION SOURCE *

R. MIDDLETON
Physics Department, University of Pennsylvania, Philadelphia, Pennsylvania 191043859, USA

Received 23 December 1982




Negative ion production
UO⁻, O⁻, UOH⁻, UN⁻...
The strategy in AMS is that of eliminate interfering ions from the beam before they reach the rare isotope detector
e.g., detection of 236U
Source2
40 Samples
75 kV Preacceleration
Focus
Insertable Faraday Cup
Electrostatic Analyzer
E/q=90 keV
r=0.300 m
Beam-Switch
y-Steerer
Magnetic Quadrupole Doublet
Multi Beam Switcher
Injection Magnet
ME/q²=17 MeV amu
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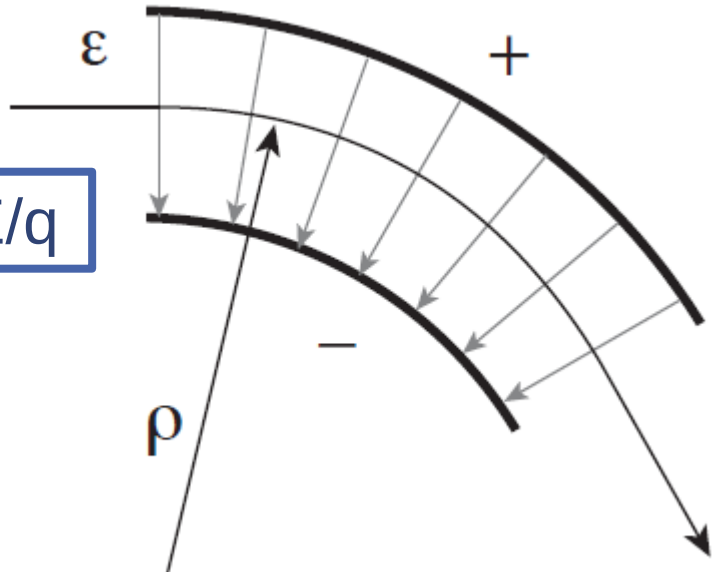
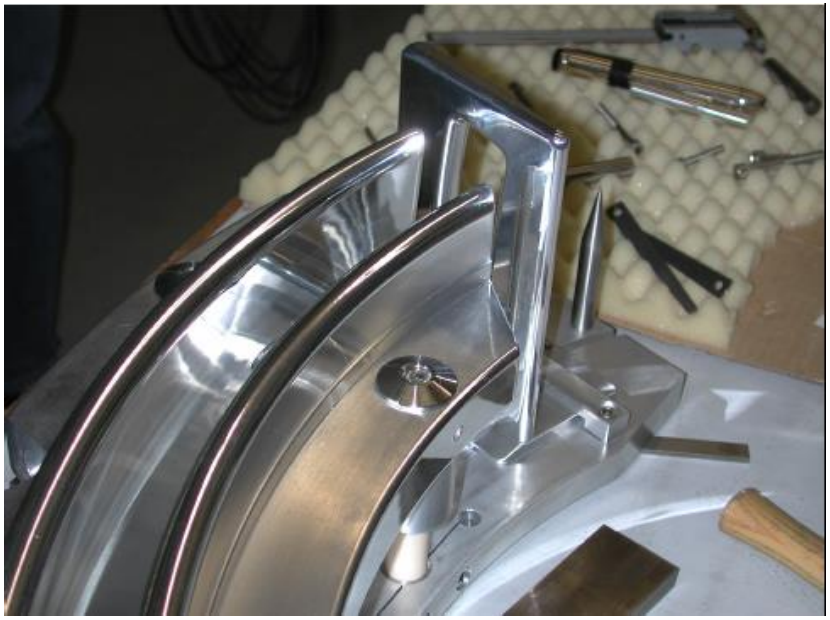
E/q=4.4 MeV
r=2.000 m
0 1 2 3 4 5 m

Electrostatic Analyzer - ESA



Selection of ions with a certain E/q

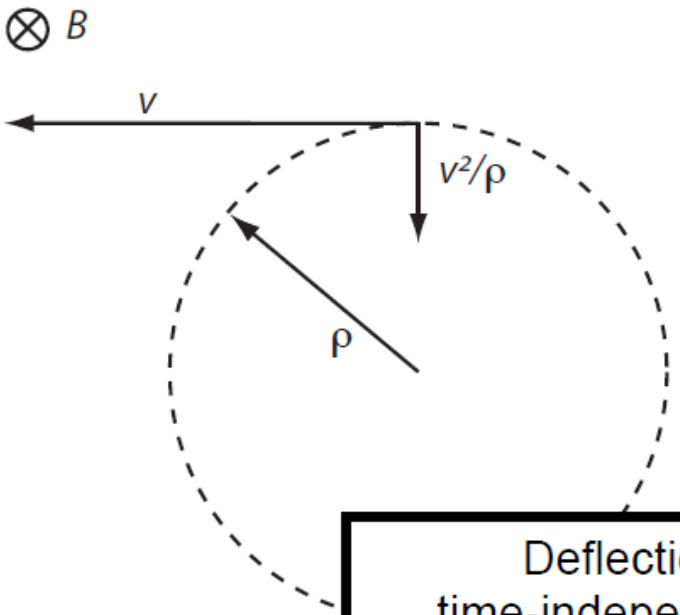


Deflection in a electric field between a pair of curved electrodes:

$$\varepsilon = \frac{\Delta U}{d}, \quad \varepsilon q = \frac{mv^2}{\rho}$$

$$\Rightarrow \varepsilon\rho = \frac{mv^2}{q} = \frac{2E}{q}$$

Dipole Magnet – 90^0



Selection of ions with a certain m/q

Deflection in a uniform, time-independet magnetic field:

$$Bqv = \frac{mv^2}{\rho}$$

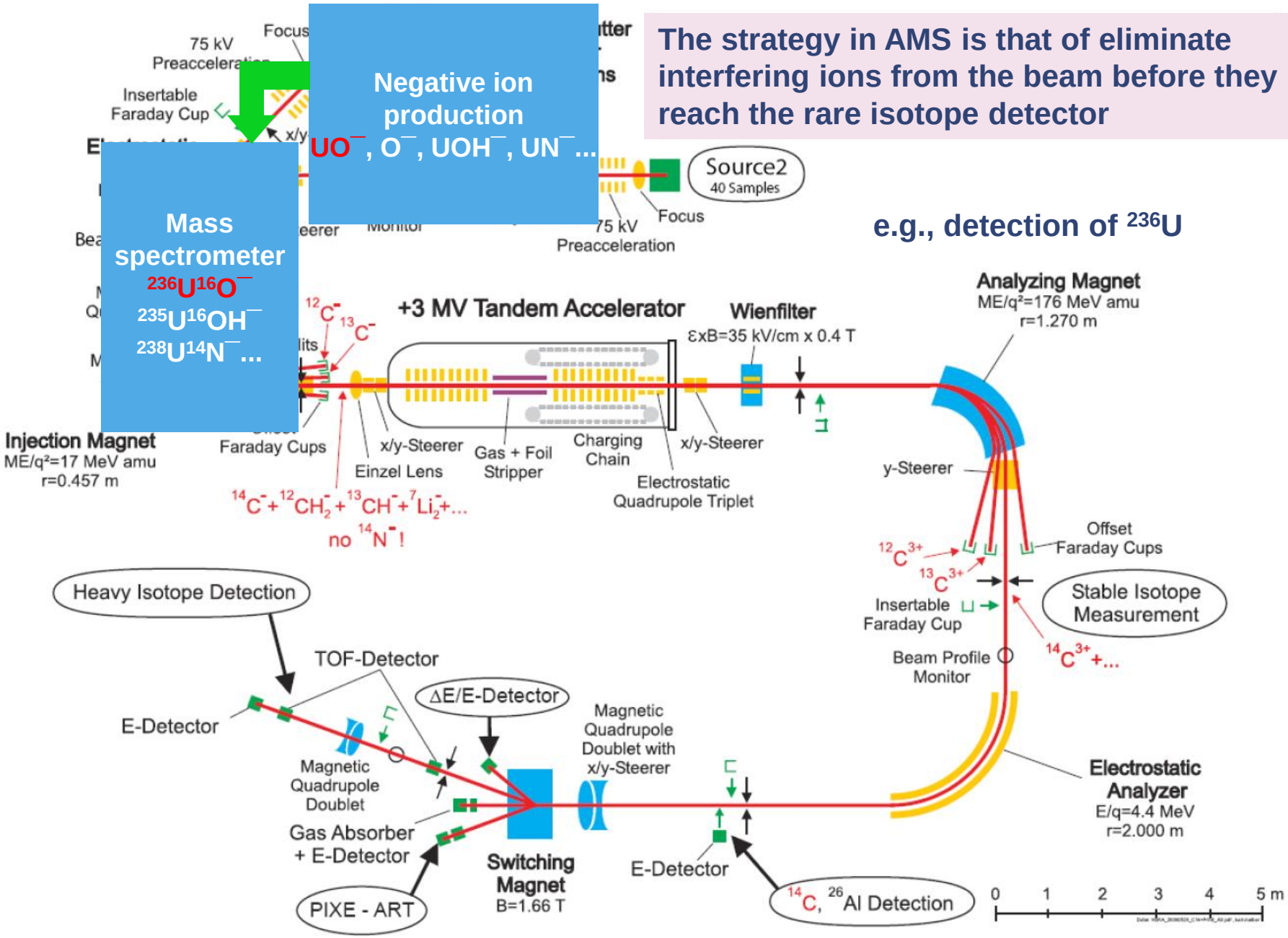
$$\Rightarrow B\rho = \frac{mv}{q} = \frac{p}{q} = \frac{\sqrt{2Em}}{q}$$

The strategy in AMS is that of eliminate interfering ions from the beam before they reach the rare isotope detector

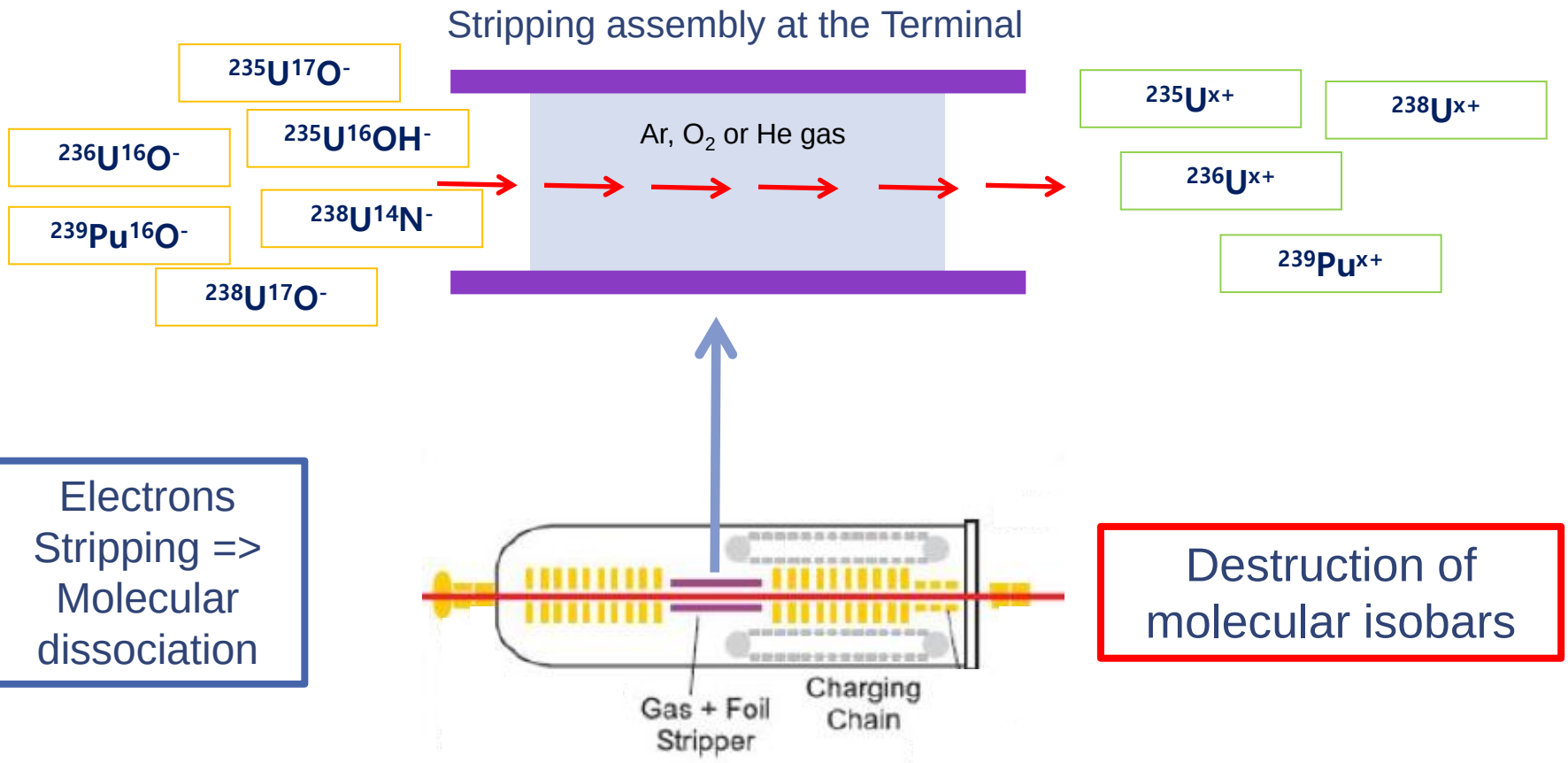
Negative ion production
UO^-, O^-, UOH^-, UN^-...

Mass spectrometer
$^{236}U^{16}O^-$
$^{235}U^{16}OH^-$
$^{238}U^{14}N^-$...

e.g., detection of ^{236}U



Background Suppression Device



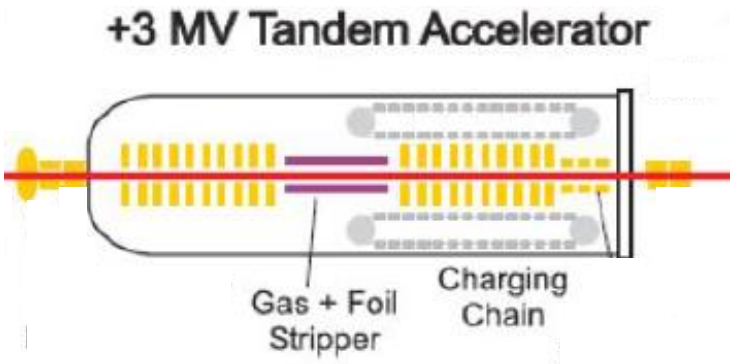
Electrons Stripping => Molecular dissociation

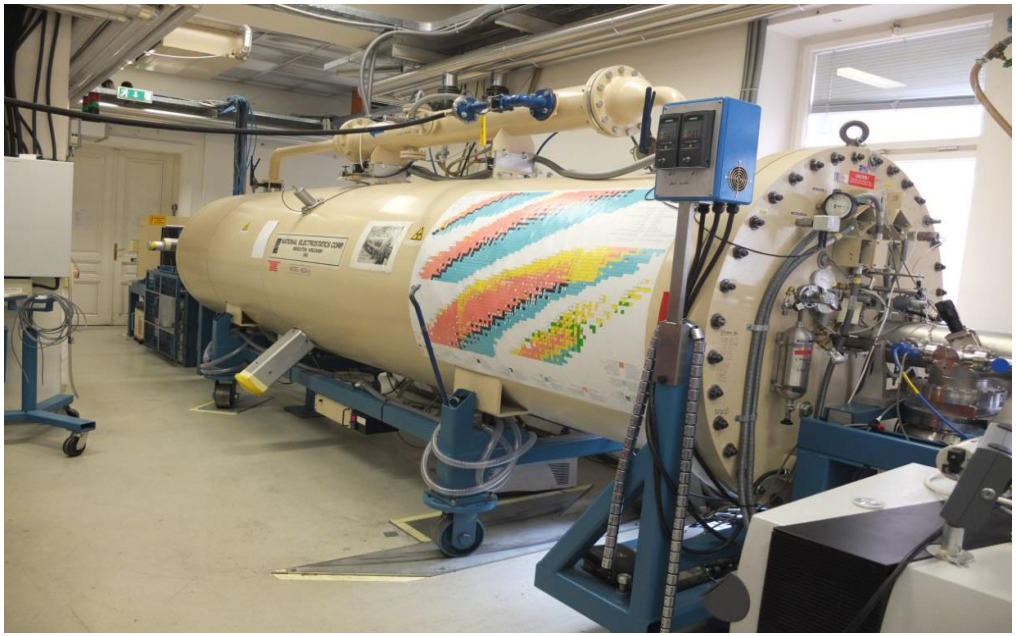
Destruction of molecular isobars

Tandem Electrostatic Accelerator

Kinetic energy of the monoatomic cations at the high energy end of the tandem - MeV

$$E = (E_{\mathrm{inj}} + \mathrm{e\,TV})\frac{M}{M_{\mathrm{inj}}} + q\,\mathrm{e\,TV}$$





The strategy in AMS is that of eliminate interfering ions from the beam before they reach the rare isotope detector

Negative ion production

UO^-, O^-, UOH^-, UN^-...

Mass spectrometer

$^{236}U^{16}O^-$

$^{235}U^{16}OH^-$

$^{238}U^{14}N^-$...

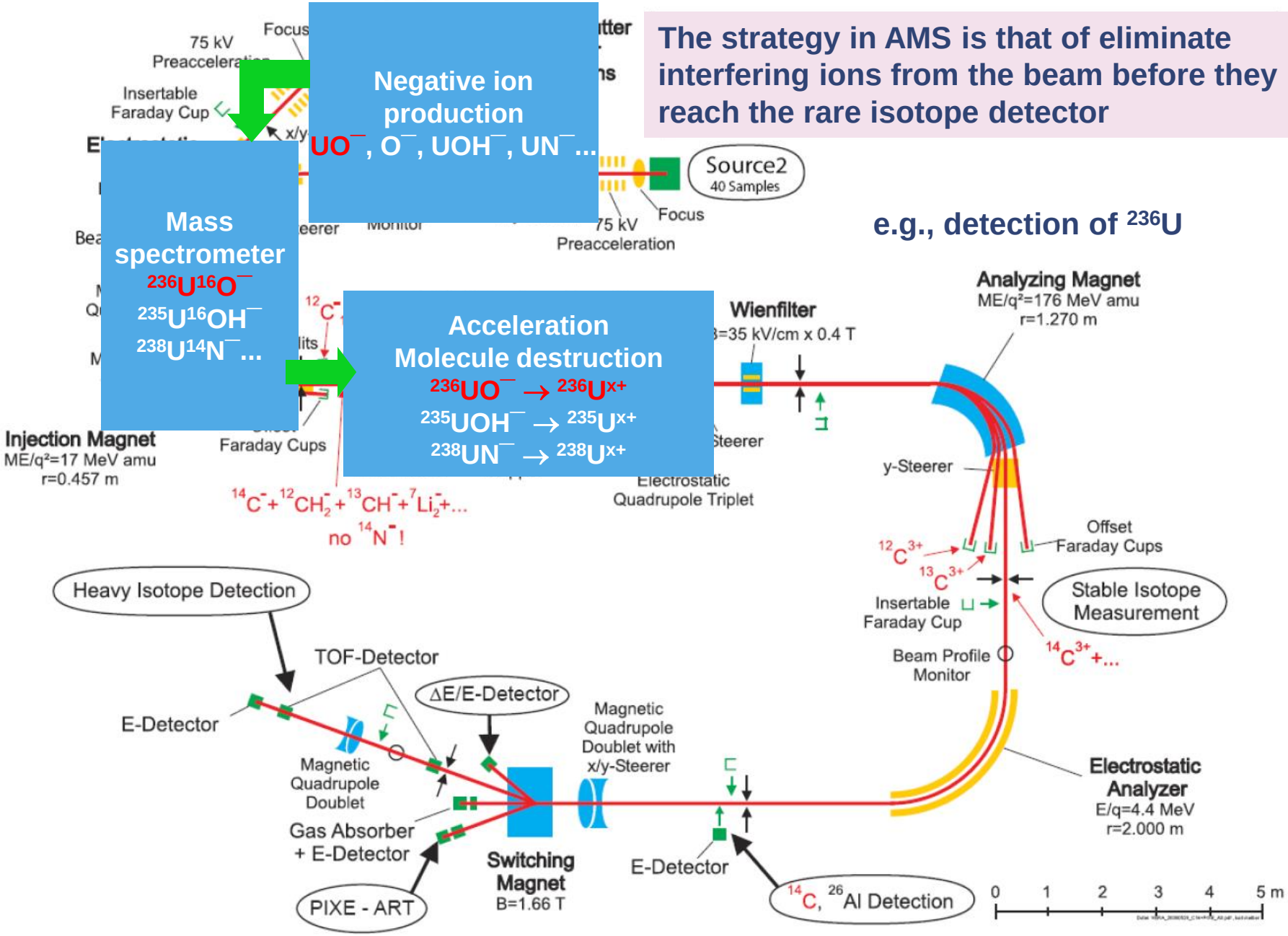
Acceleration Molecule destruction

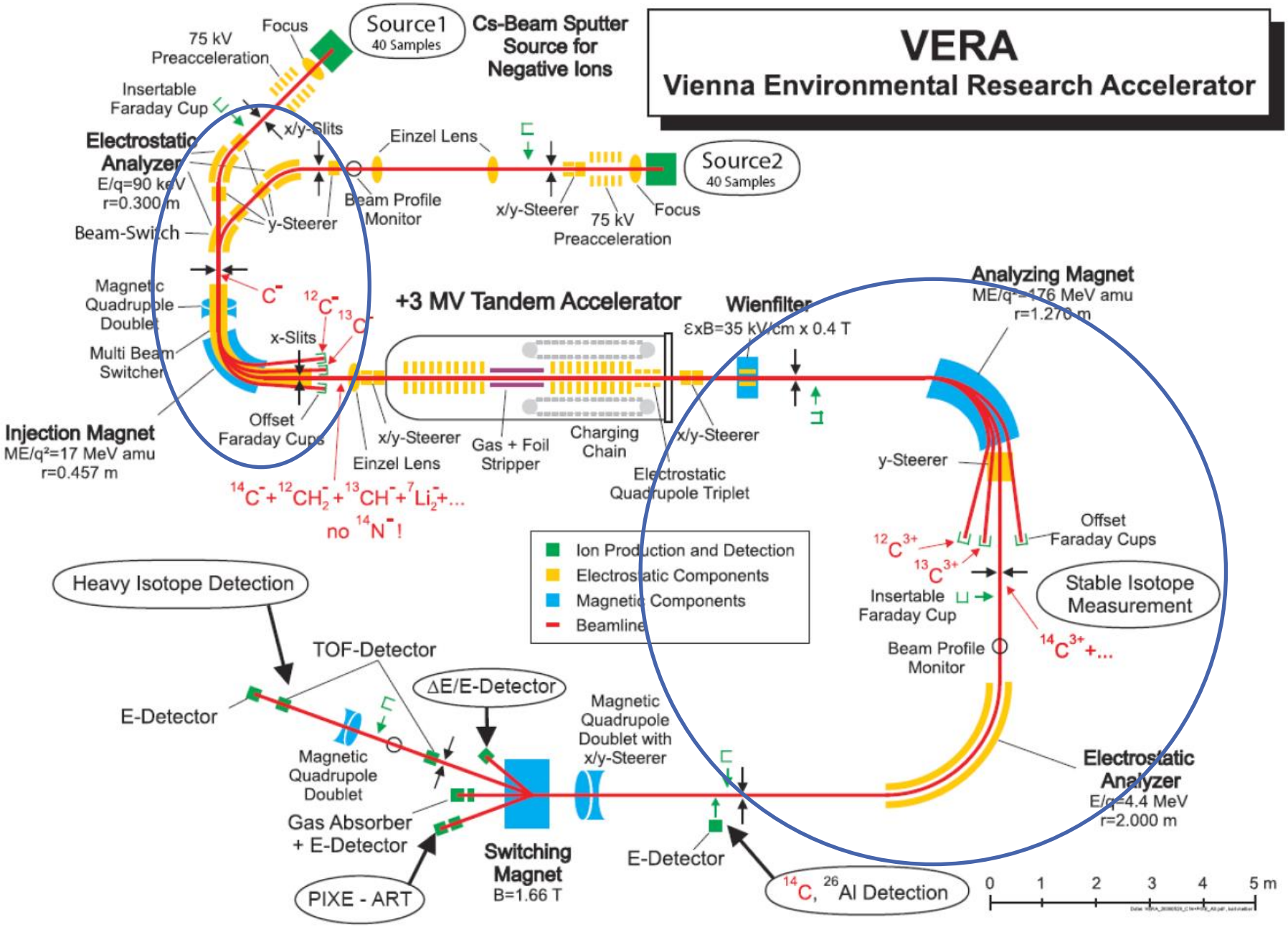
$^{236}UO^- \rightarrow {}^{236}U^{x+}$

$^{235}UOH^- \rightarrow {}^{235}U^{x+}$

$^{238}UN^- \rightarrow {}^{238}U^{x+}$

e.g., detection of ^{236}U

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Source2
40 Samples
Magnetic Quadrupole Doublet
Multi Beam Switcher
C-
12C-
13C-
x-Slits
+3 MV Tandem Accelerator
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ME/q²=176 MeV amu
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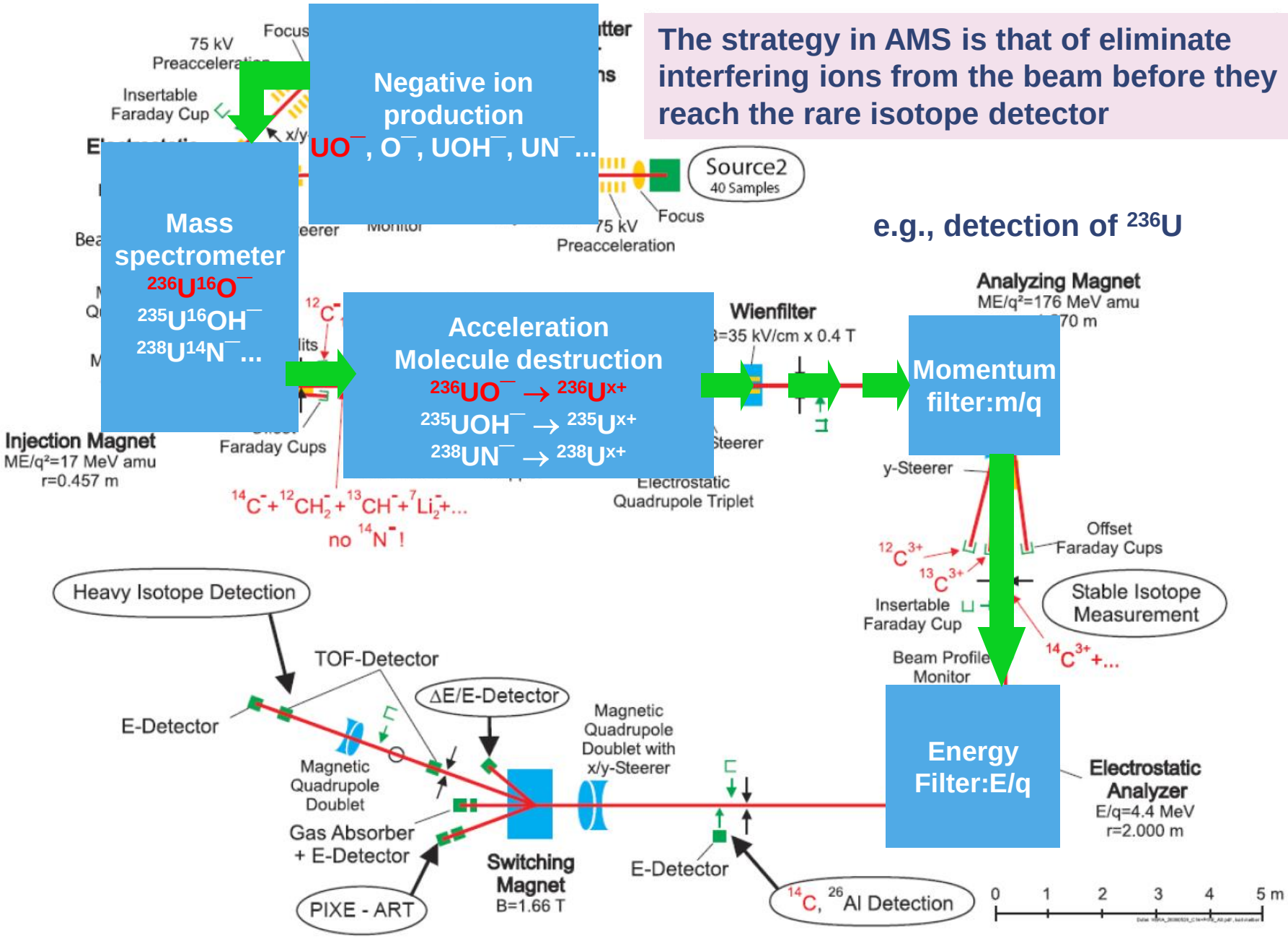
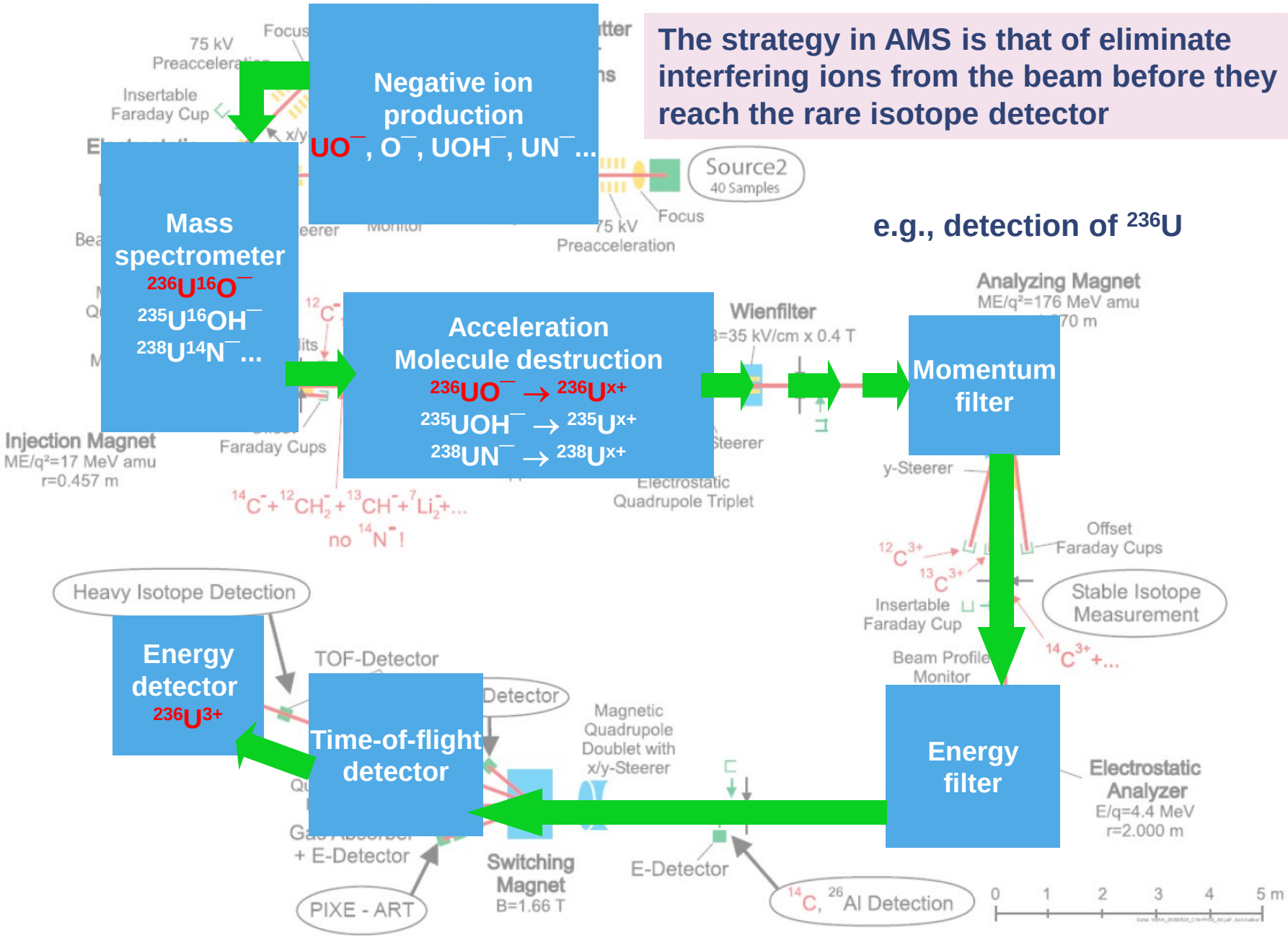
Offset Faraday Cups
x/y-Steerer
Einzel Lens
Gas + Foil Stripper
Charging Chain
x/y-Steerer
Electrostatic Quadrupole Triplet
y-Steerer
14C-+12CH2-+13CH-+7Li2-+...
no 14N-!
Ion Production and Detection
Electrostatic Components
Magnetic Components
Beamline
12C3+
13C3+
Offset Faraday Cups
Insertable Faraday Cup
Stable Isotope Measurement
Beam Profile Monitor
14C3+ +...
Heavy Isotope Detection
TOF-Detector
E-Detector
ΔE/E-Detector
Magnetic Quadrupole Doublet
Gas Absorber + E-Detector
PIXE - ART
Switching Magnet
B=1.66 T
Magnetic Quadrupole Doublet with x/y-Steerer
E-Detector
Electrostatic Analyzer
E/q=4.4 MeV
r=2.000 m
14C, 26Al Detection
0 1 2 3 4 5 m

The strategy in AMS is that of eliminate interfering ions from the beam before they reach the rare isotope detector

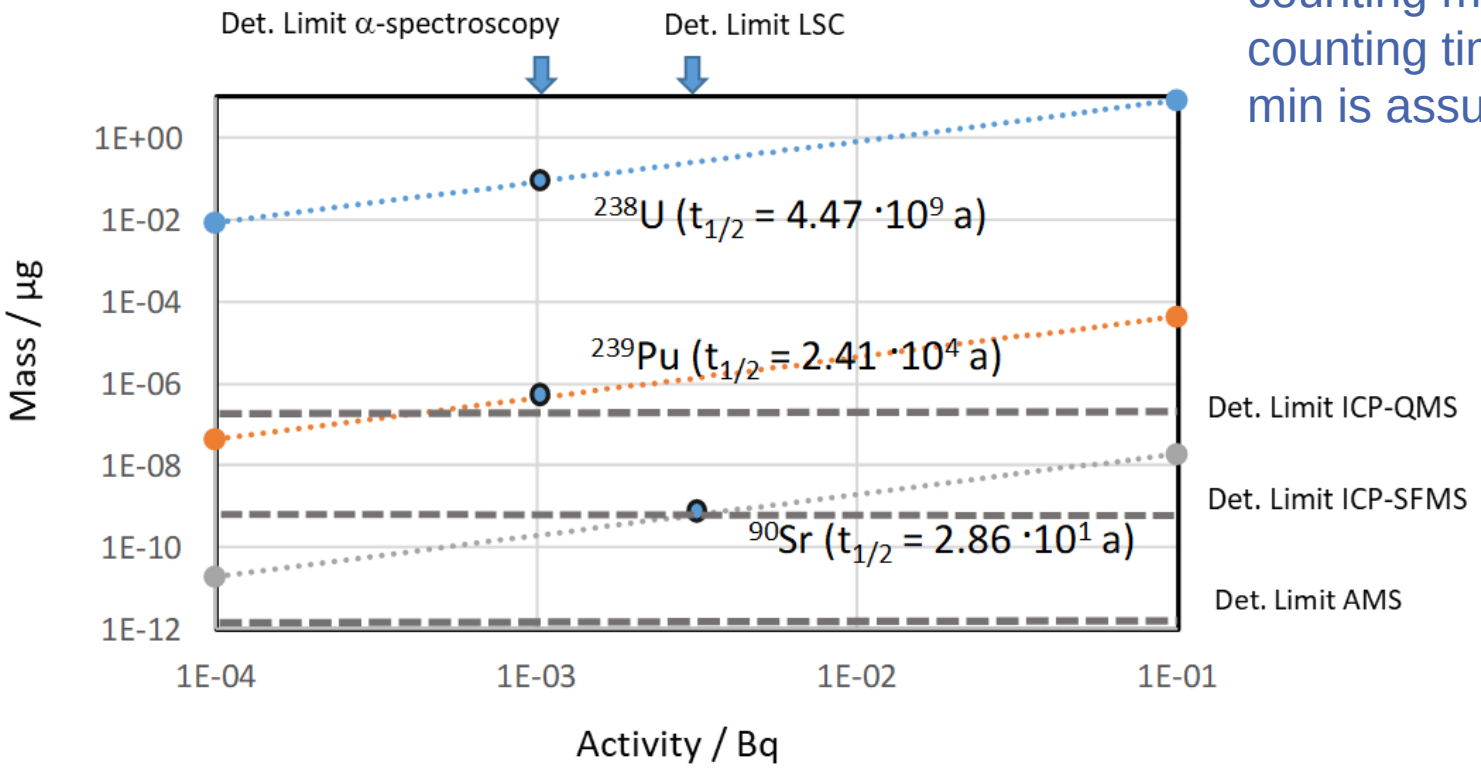


The strategy in AMS is that of eliminate interfering ions from the beam before they reach the rare isotope detector



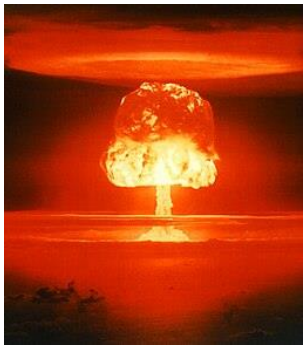
Comparison of detection sensitivities of mass spectrometric methods with those of a-spectrometry and liquid scintillation counting

for the radiation counting method a counting time of 1000 min is assumed



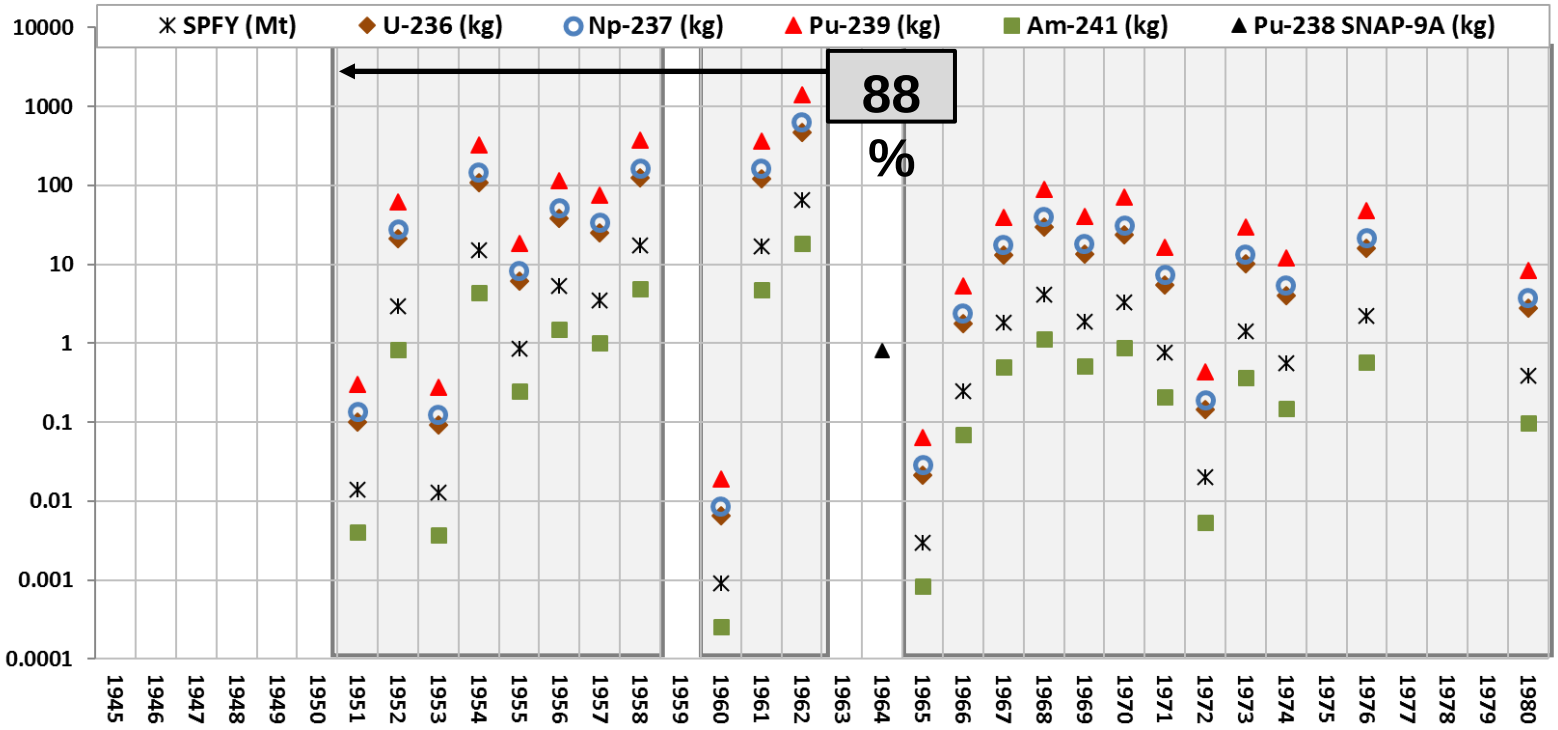
Estimated detection limits are related to the mass or activity per sample

Trace Analysis of Radionuclides by Mass Spectrometry Techniques. *Strahlenschutz-Praxis* 2023; 29 (3), 44–47.



Global fallout actinide

Stratospheric-partitioned fission yield in Mt, SPFY (UNSCEAR, 2000) per year from atmospheric testing of thermonuclear devices, Corresponding estimates for produced yearly inventories of ^{236}U, ^{237}Np, ^{239}Pu, and ^{241}Am (1 January 2016)*



*Chapter 8. "Sources and behaviour of actinide elements in the environment" of "Experimental and Theoretical Approaches to Actinides Chemistry: From Fundamental Systems to Practical Applications". Pag. 378 – 444. Edited by John Gibson and Wibe Albert de Jong. Publisher: John Wiley & Sons, 1. Edition March 2018. Ltd. ISBN: 978-1-119-11552-6.

Isotopic analysis: Inventories of ^{236}U, ^{237}Np, ^{239}Pu and ^{241}Am from global fallout and the nuclear accidents at Chernobyl and Fukushima, together with the related $^{236}U/^{238}U$, $^{236}U/^{239}Pu$, $^{237}Np/^{239}Pu$ and $^{240}Pu/^{239}Pu$ atomic ratios

	Global fallout	Chernobyl nuclear accident	Fukushima nuclear accident
^{236}U (kg)	900-1698	48.5	$5x10^{-4}$
^{237}Np (kg)	1500	0.136	/
^{239}Pu (kg)	2800-3108	5.6	$2x10^{-4}$; $5x10^{-4}$
^{241}Am (kg)	41-43	0.52	/
$^{236}U/^{238}U$	not measured in stratospheric debris	0.019	/
$^{236}U/^{239}Pu$	0.22 – 0.30	8.7	1.1
$^{237}Np/^{239}Pu$	0.44 – 0.59	0.024	/
$^{240}Pu/^{239}Pu$	0.18 integrated value	0.38 - 0.42	0.303 - 0.330

Chapter 8. “Sources and behaviour of actinide elements in the environment” of “Experimental and Theoretical Approaches to Actinides Chemistry: From Fundamental Systems to Practical Applications”. Pag. 378 – 444. Edited by John Gibson and Wibe Albert de Jong. Publisher: John Wiley & Sons, 1. Edition March 2018. Ltd. ISBN: 978-1-119-11552-6.

Some of the RNs measured with the highest sensitivity with AMS

^{236}U ($t_{1/2}$ = 23.42 My)

In nature:
spontaneous neutron capture on ^{235}U

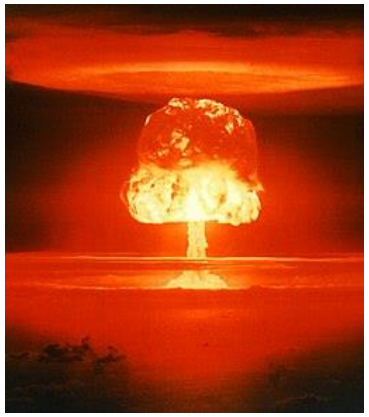
$$^{235}U\ (n,\gamma)\ ^{236}U$$

In nuclear reactors and nuclear bomb tests:

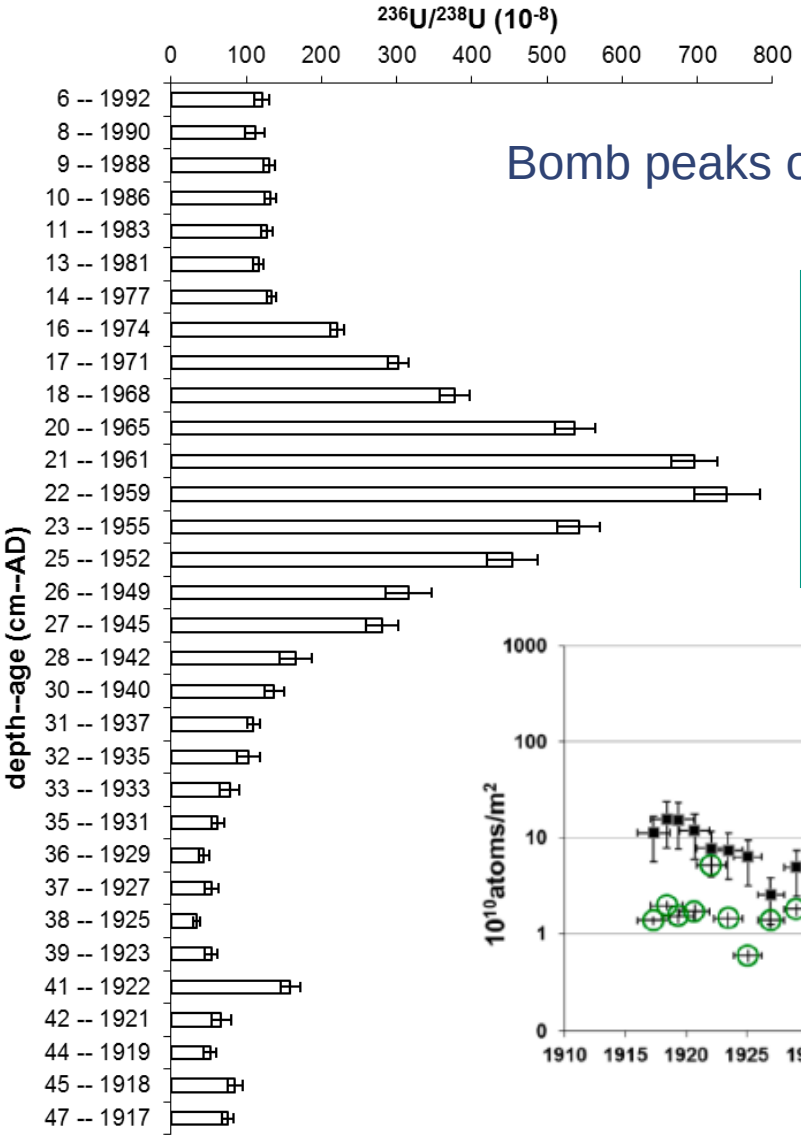
$$^{235}U\ (n,\gamma)\ ^{236}U$$

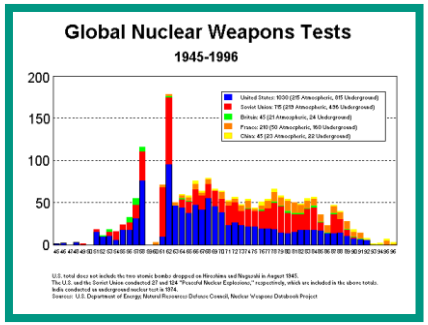
$$^{238}U\ (n,3n)\ ^{236}U$$

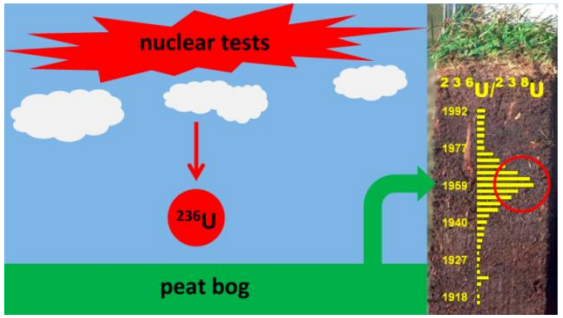
a wide range of $^{236}U/^{238}U$ ratios in environmental samples spanning from below 10^{-14} to up to 10^{-2}

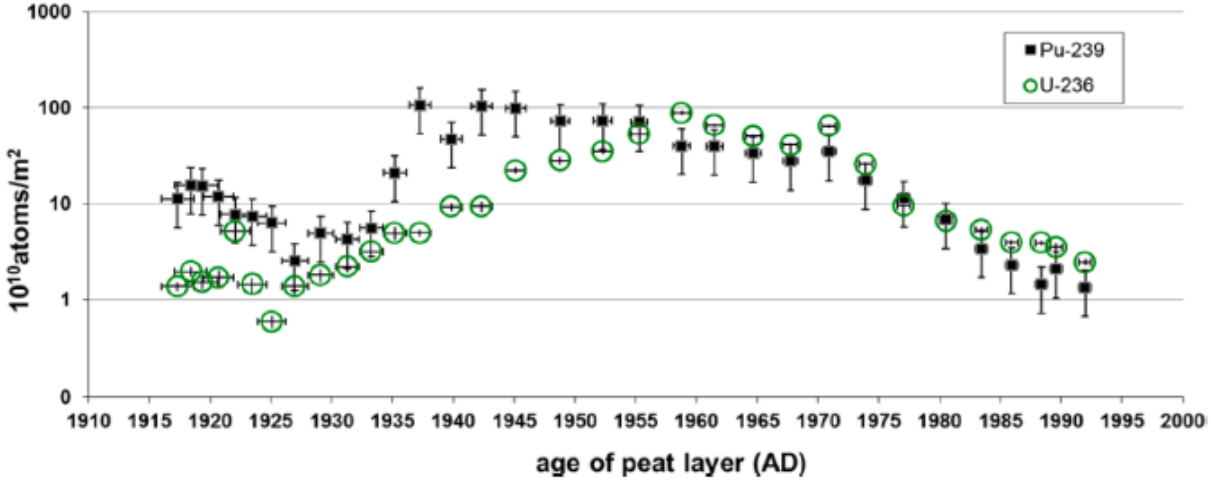


Bomb peaks of ^{236}U and ^{239}Pu along the ^{210}Pb dated profile of an ombrothrophic peat bog core

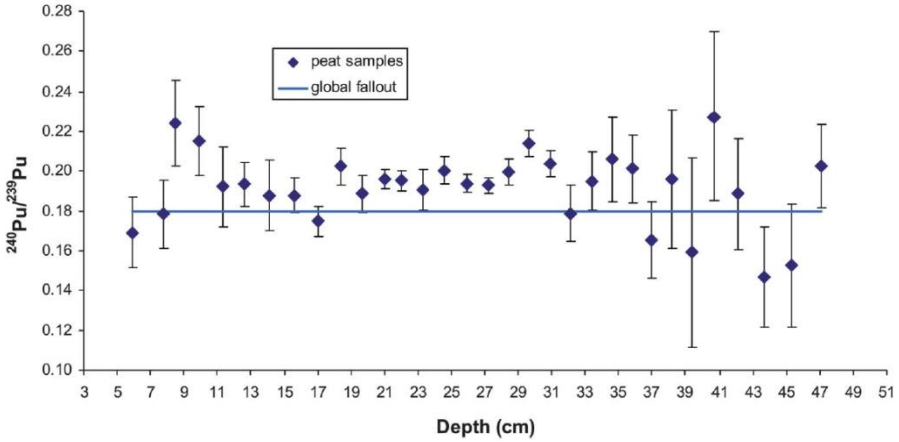


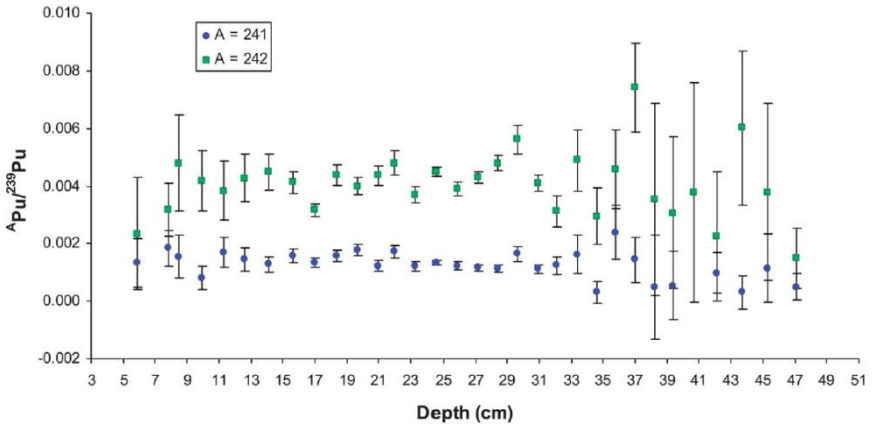




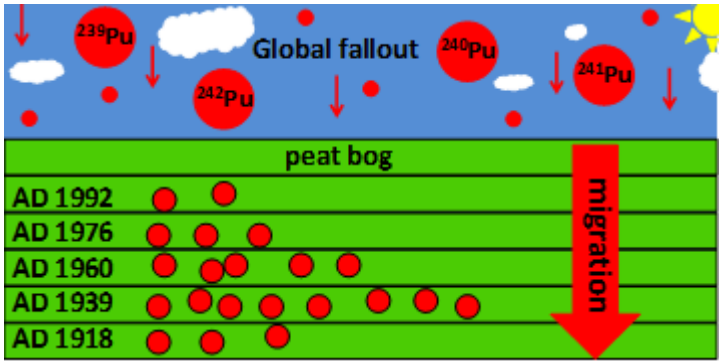


dx.doi.org/10.1021/es400026m | *Environ. Sci. Technol.* 2013, 47, 5243–5250





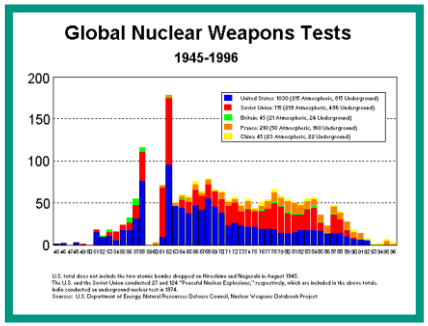
Isotopic ratios of $^{2340,241,242}Pu/^{239}Pu$ along the profile of the ombrotrophic peat bog core revealing the oring of plutonium from stratospheric nuclear weapon tests



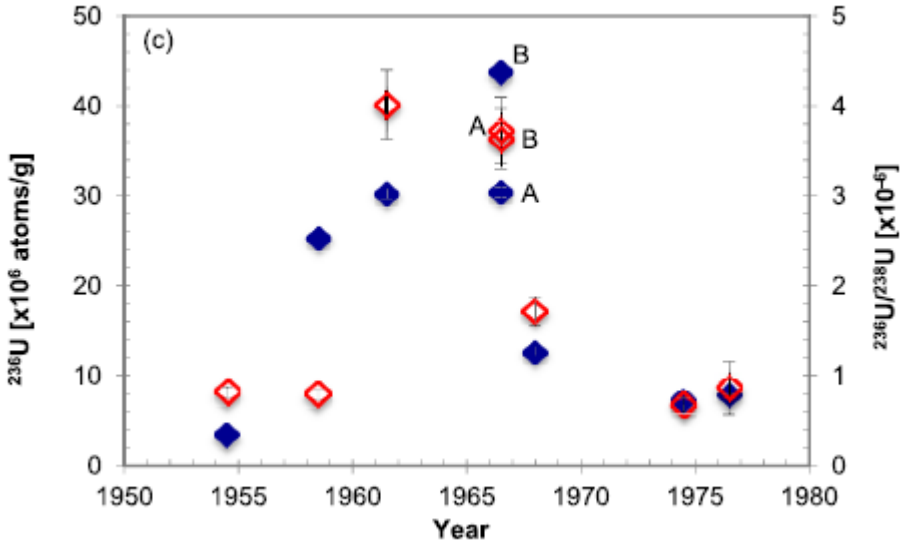
Environ. Sci.: Processes Impacts, 2013, **15**, 839–847

Bomb peak of ^{236}U in roe deer antlers

roe deer shed their antlers annually, and hence antlers may provide a time-resolved environmental archive for fallout radionuclides

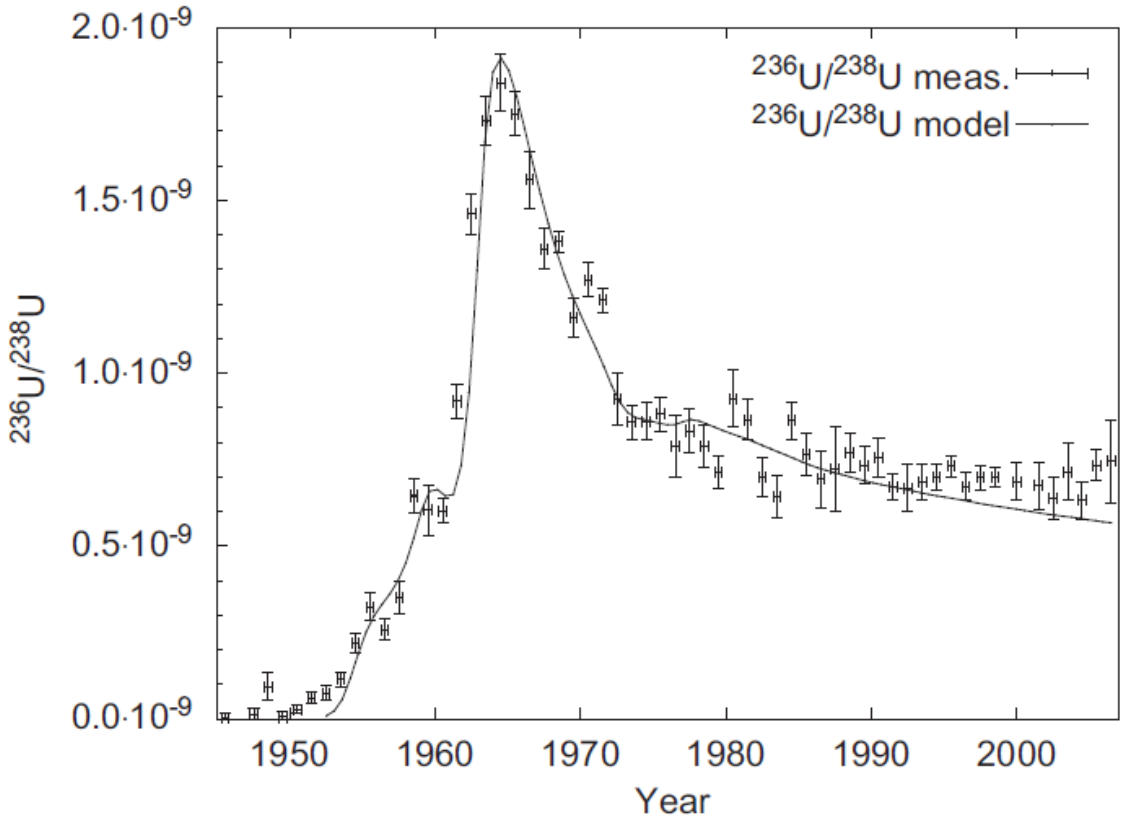


The antlers were hunting trophies, and hence the hunting area, the year of shooting and the approximate age of each animal was known



Journal of Environmental Radioactivity 151 (2016) 587–592

^{236}U as oceanic tracer: The investigation of reliable geological archives in the form of coral cores



Corals build uranium into their aragonite skeleton at a level of 2-4 ppm by substitution of uranium for calcium in the lattice

An ideal archive to trace the input of ^{236}U by nuclear testing and further evolution in the ocean

Bomb pulse of ^{236}U in an annually resolved coral core from the Caribbean Sea

S.R. Winkler et al. / Earth and Planetary Science Letters 359-360 (2012) 124–130

^{236}U in river sediments near a shutdown nuclear power plant

The Garigliano NPP, presently in the decommissioning phase, started operation in 1964, and was shut down in 1978.

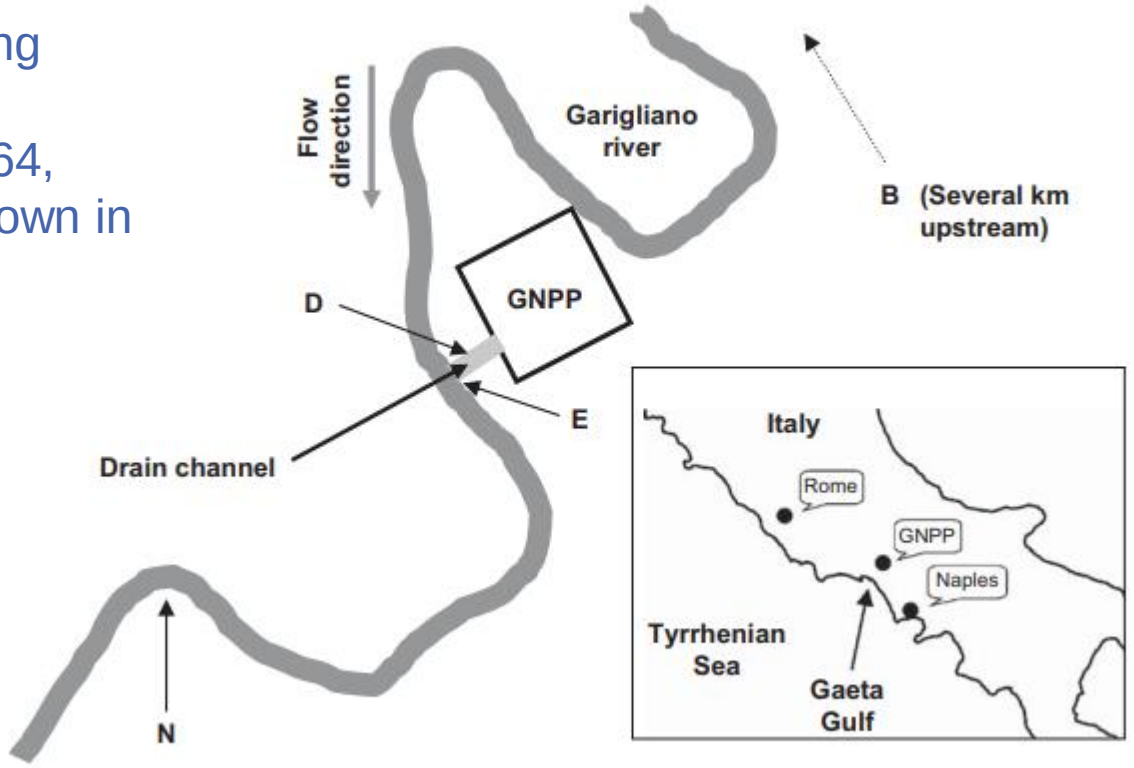


Fig. 1. Schematic representation of the sampling points; the insert shows the location of the Garigliano Nuclear Power Plant in Southern Italy.

F. Quinto et al. / Applied Radiation and Isotopes 67 (2009) 1775–1780

^{236}U in river sediments near a shutdown nuclear power plant

$^{239+240}$Pu activities, detected by alpha-spectrometry, between 0.5 and 6 mBq/g (the total activity of plutonium in soil from global fallout is usually less than 4 mBq/g, (Hardy et al., 1973)

^{240}Pu/^{239}Pu isotopic ratios determined by AMS indicates global fallout origin (about 0.2), for all four cores

^{236}U has global fallout origin

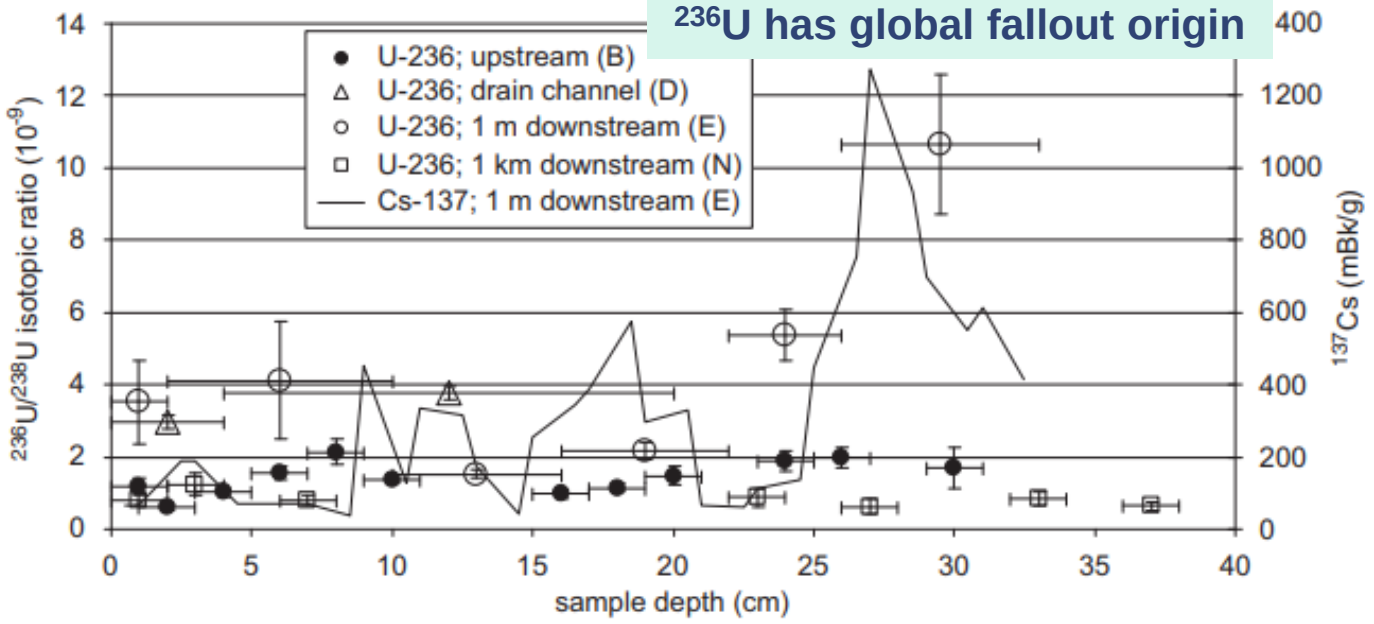


Fig. 2. ^{236}U/^{238}U isotopic ratios vs. depth for all four cores. The horizontal error bars indicate the span of the core section, the vertical error bars are the measurement uncertainty. The ^{137}Cs activity is shown for the 1 m downstream core E (solid line).

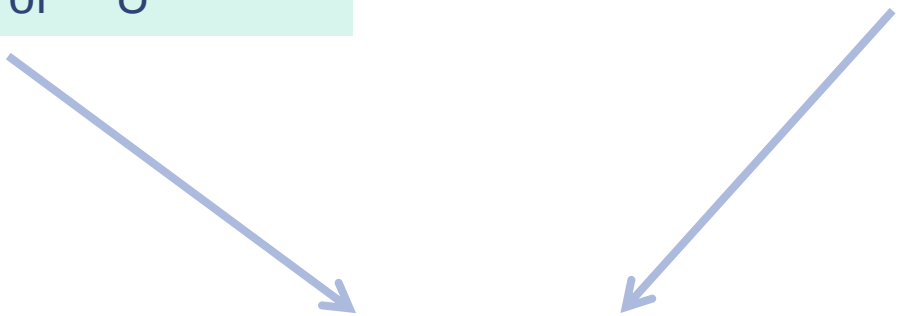
F. Quinto et al. / Applied Radiation and Isotopes 67 (2009) 1775–1780

Some of the RNs measured with the highest sensitivity with AMS

^{129}I ($t_{1/2}$ = 15.7 ± 0.4 My)

In nature:
spallation on Xe target atoms in the atmosphere and
spontaneous fission of ^{238}U

In nuclear reactors and nuclear bomb tests:
fission of ^{235}U and ^{239}Pu

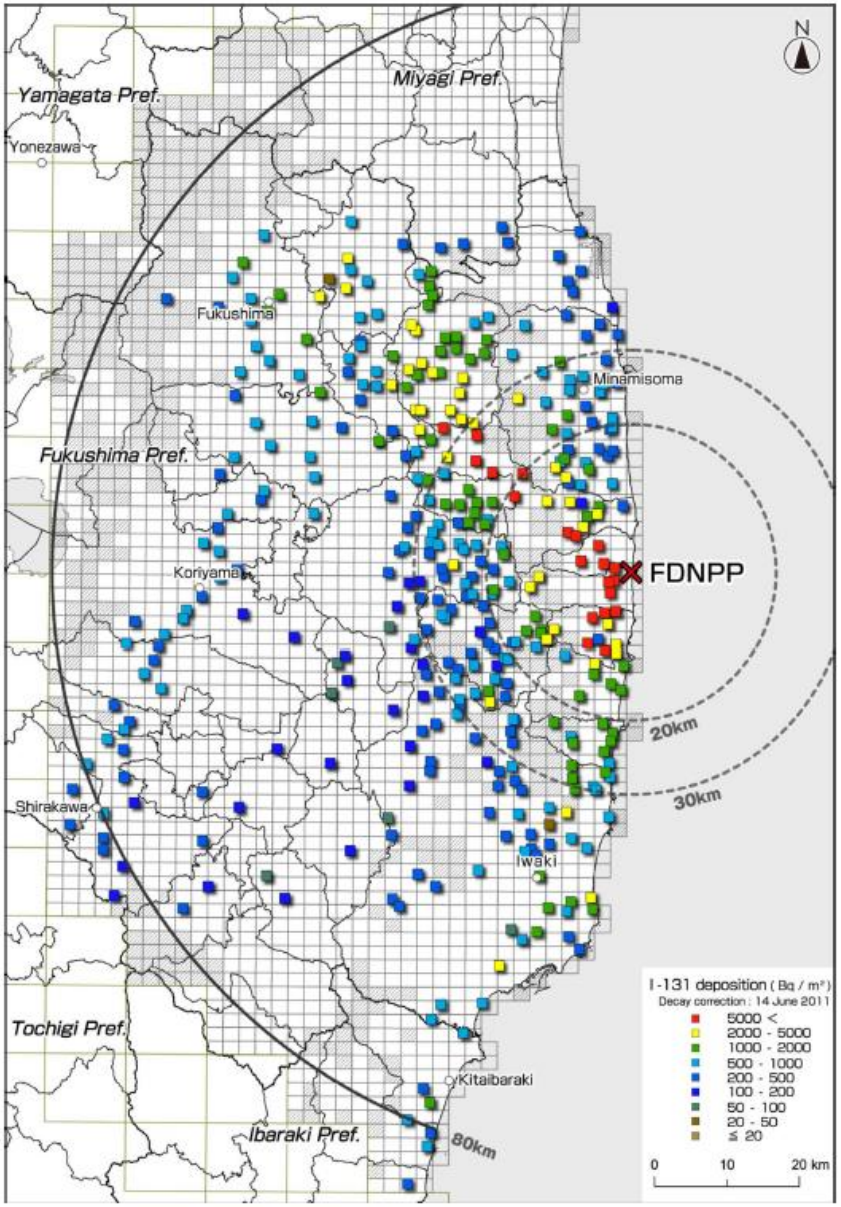


a wide range of $^{129}I/^{127}I$ ratios in environmental samples spanning from below 10^{-14} to up to 10^{-6}

Retrospective dosimetry of ^{131}I after nuclear accidents by measuring ^{129}I with AMS

Following the accident, the short half life of ^{131}I (8 d) made it impossible to obtain adequate sample coverage for a direct determination of the regional deposition patterns

Small amounts of ^{129}I produced in the reactor were also released during the accident with a ratio $^{129}I/^{131}I$ almost constant in the samples were both RNs could have been measured



Deposition map (Bq/m^2) of ^{131}I reconstructed from ^{129}I analysis in Fukushima Prefecture.

Y. Muramatsu et al. / Journal of Environmental Radioactivity 139 (2015) 344–350

Some of the RNs measured with the highest sensitivity with AMS

^{14}C ($t_{1/2}$ = 5700 ± 30 y)

In nature: spallation on ^{14}N target atoms in the atmosphere

$$^{14}\mathbf{N}\ (\mathbf{n}, \mathbf{p})\ ^{14}\mathbf{C}$$

In nuclear reactors and nuclear bomb tests:

$$^{14}\mathbf{N}\ (\mathbf{n}, \mathbf{p})\ ^{14}\mathbf{C}$$

$$^{13}\mathbf{C}\ (\mathbf{n}, \boldsymbol{\gamma})\ ^{14}\mathbf{C}$$

$$^{17}\mathbf{O}\ (\mathbf{n}, \boldsymbol{\alpha})\ ^{14}\mathbf{C}$$

Procedure Blanks $^{14}C/^{12}C$ ca. 10^{-15}
Range age for radiocarbon dating up to 50,000 y

In contemporary carbon $^{14}C/^{12}C = 1.2 \times 10^{-12}$

Several orders of magnitude higher

a wide range of $^{14}C/^{12}C$ ratios in environmental samples starting from 10^{-15}

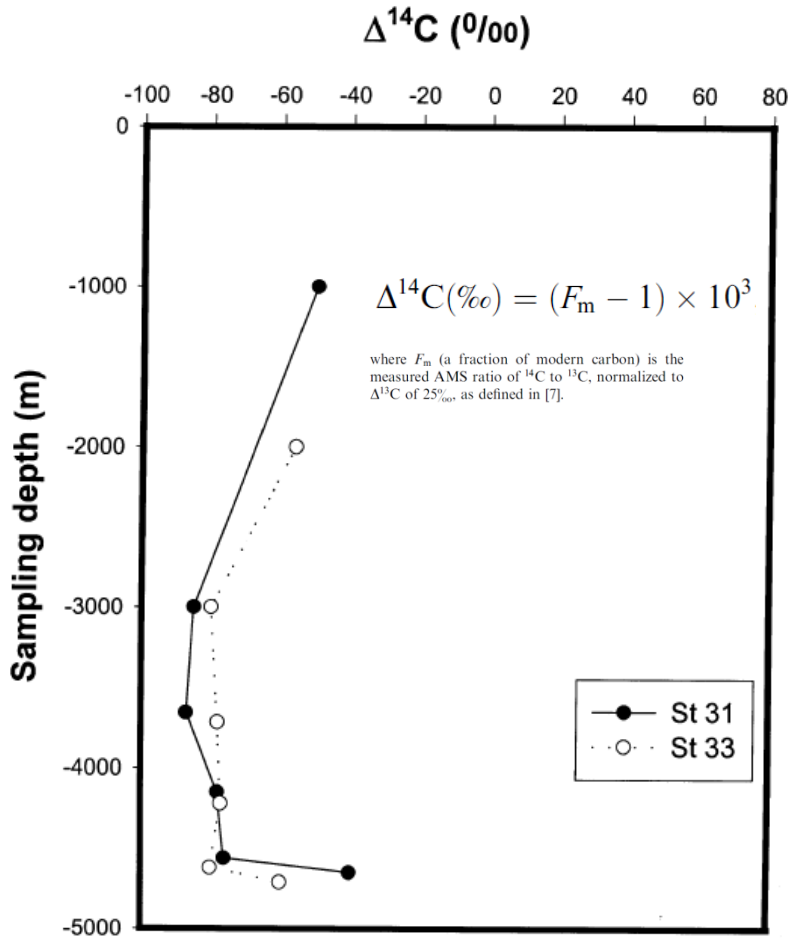


Fig. 2. ^{14}C water profiles in the NE Atlantic Ocean (1992).

AMS measurements of ^{14}C in seawater around radioactive waste dump sites

Slightly elevated ^{14}C values observed close to the bottom interpreted as a continuous leakage of ^{14}C from dumped containers

Hypothesis supported by the observation of an elevated concentration of ^{238}Pu, $^{239,240}Pu$ and ^{241}Am with ratios higher than global fallout (not reported)

P.P. Povinec et al. / Nucl. Instr. and Meth. in Phys. Res. B 172 (2000) 672–678

Summing up

The strategy in AMS is that of eliminate interfering ions from the beam before they reach the rare isotope detector

AMS one of the most sensitive analytical technique for the investigation of rare long-lived radionuclides in the environment

at least 4 and up to 7 orders of magnitude less atoms than a radiometric technique to perform equally precise measurements, with consequent reduction of sample size

unambiguous nuclide detection in environmental samples at concentration levels below ppq

Fields of Application:

Archeology
Biomedical research
Environmental science
Geology
Monitoring of nuclear contamination
Nuclear Physics and Astrophysics
Safety of nuclear waste disposal

AMS analysis is ultra trace analysis

ag/g level concentrations (10^{-15}g/L)
➔ a grain of sugar in lake Constance