



SECONDA UNIVERSITÀ DEGLI STUDI DI NAPOLI

SCUOLA POLITECNICA E DELLE SCIENZE DI BASE

dfm DIPARTIMENTO DI
MATEMATICA E FISICA

CENTRE FOR ISOTOPIC RESEARCH ON

CULTURAL AND ENVIRONMENTAL HERITAGE



The role and the performance of CIRCE lab in the framework of the Isonitrate Italy Project

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The CIRCE lab

- A 3 MV accelerator mass spectrometer (AMS) for the ultrasensitive measurements of rare isotope ratios (e.g. actinides and ^{14}C).
- A mass spectrometry lab (IRMS) for the stable isotope analysis of solid, gaseous and liquids samples.
- An environmental radioactivity laboratory allowing also the ^{222}Rn and gamma emitters measurement.
- An isotope chemistry lab involved also in methodological developments (e.g. AMS measurement of ^{11}B).
- An isotope hydrology and cryogenic lab.
- A facility for the study of nuclear reactions in the framework of nuclear astrophysics.

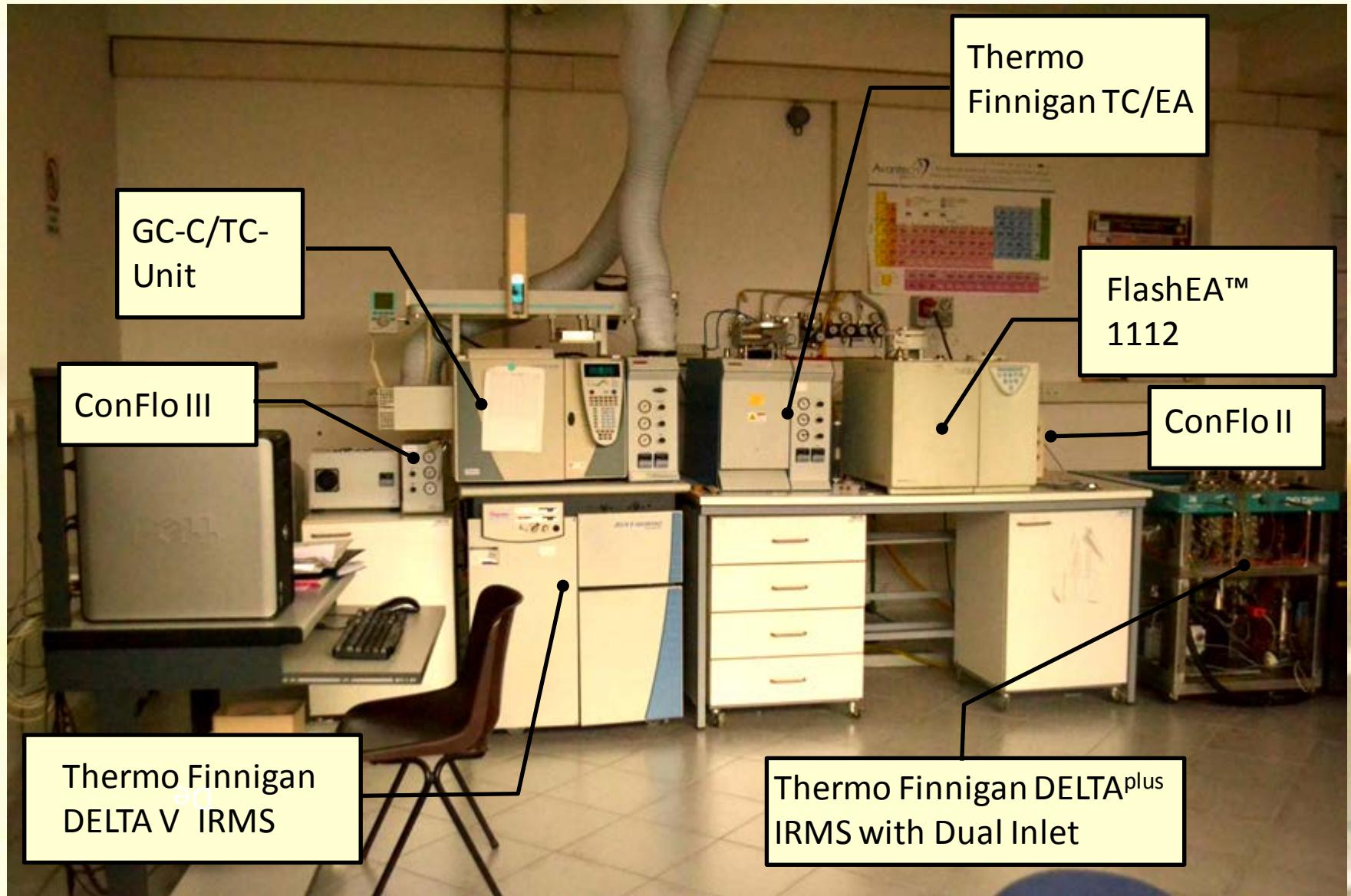


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CIRCE - FIELDS OF ACTIVITY

- a) Archaeometry by high-precision radiocarbon dating;
- b) Environmental science by ^{14}C -based global carbon cycle studies;
- c) Isotope hydrology;
- d) Nuclear astrophysics by the ERNA recoil mass separator;
- e) Nuclear safeguards and contrast to illegal nuclear fuel use by actinides AMS;
- f) Forensic applications of AMS;
- g) Tribology by ^{7}Be implantation.

The IRMS lab



Number of isotopic analyses in the Isonitrate Italy project

Matrix	$\delta^{15}\text{N}_{\text{NO}_3}$	$\delta^{18}\text{O}_{\text{NO}_3}$	$\delta^{15}\text{N}_{\text{BULK}}$	$\delta^{11}\text{B}$
Surface waters	105	105		11
Groundwaters	361	361		74
Soils (2mm sieved and homogenized)			54	
Soil extracts (1:5 soil to water ratio)	32	32		
Mineral fertilizers			2	
Manure			8	in progress
Sewage effluents (at inlet of TP)			12	in progress
Sewage effluents (at outlet of TP)	9	9		in progress

Total: 668 samples

The environmental samples and isotopic analyses in the Nitrate Project



The samples reception: the Nitrobase

Screenshot of Microsoft Access 2007 showing the "ACCETTAZIONE" table.

The table contains 741 records. The columns are:

- DATA
- ID Campione COD ISPRA
- ID C
- Data campion
- Primary Key
- Tipologia A
- Sa
- MATRIC
- Regi
- Contenitore
- Data passa

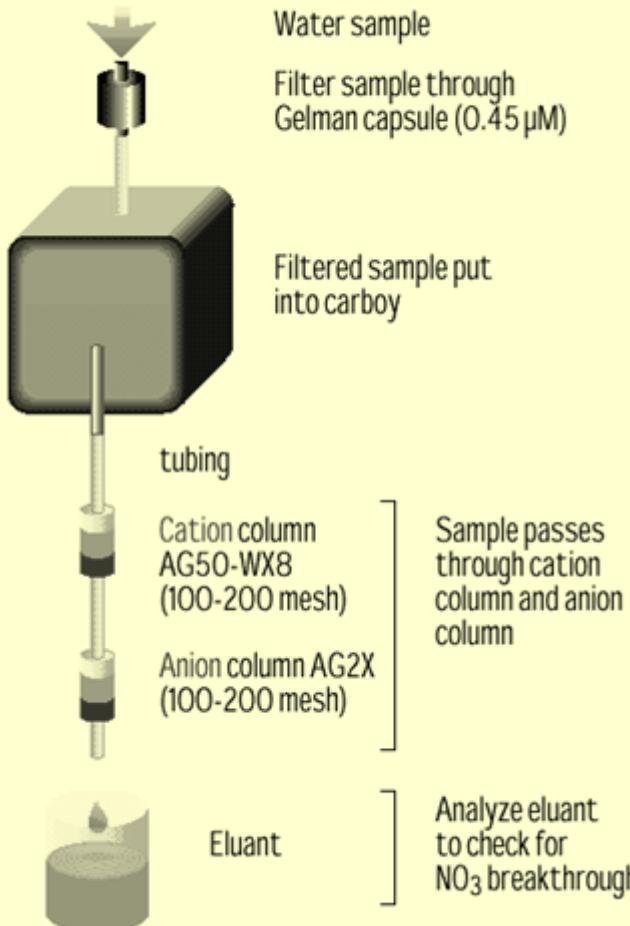
The table shows various sample details, including collection date, specimen ID, collection location (ISPRA), collection date, primary key (numbered 1 to 27), sample type (e.g., Taratura, SW, GW, CI, ZO), sample status (Sa), registration number (MATRIC), registration location (Regi), container type (e.g., Sacchetto polietilene, Colonna anionica, Bottiglia di plastica, Tubi Falcon), and processing date (Data passa).

DATA	ID Campione COD ISPRA	ID C	Data campion	Primary Key	Tipologia A	Sa	MATRIC	Regi	Contenitore	Data passa
16/04/2014	ISPRASU5001	ISPRA	04/04/2014	1	Taratura	c	SU	VEN	Sacchetto polietilene	
16/04/2014	ISPRASU5002	ISPRA	04/04/2014	2	Taratura	c	SU	VEN	Sacchetto polietilene	
16/04/2014	ISPRASU5003	ISPRA	04/04/2014	3	Taratura	c	SU	VEN	Sacchetto polietilene	
16/04/2014	ISPRASU5004	ISPRA	04/04/2014	4	Taratura	c	SU	VEN	Sacchetto polietilene	
16/04/2014	ISPRASW5206	ISPRA	09/04/2014	5	Taratura	c	SW	VEN	Colonna anionica	
16/04/2014	ISPRASW5202	ISPRA	09/04/2014	6	Taratura	c	SW	VEN	Colonna anionica	
16/04/2014	ISPRAGW5002	ISPRA	09/04/2014	7	Taratura	c	GW	VEN	Colonna anionica	
16/04/2014	ISPRAGW5001	ISPRA	09/04/2014	8	Taratura	c	GW	VEN	Bottiglia di plastica	
16/04/2014	ISPRAGW5000	ISPRA	09/04/2014	9	Taratura	c	GW	VEN	Colonna anionica	
16/04/2014	ISPRACI5001	ISPRA	09/04/2014	10	Taratura	c	CI	VEN	Tubi Falcon	
16/04/2014	ISPRACI5000	ISPRA	09/04/2014	11	Taratura	c	CI	VEN	Tubi Falcon	
16/04/2014	ISPRASU8001	ISPRA	01/04/2014	12	Taratura	c	SU	EMR	Sacchetto polietilene	
16/04/2014	ISPRASU8002	ISPRA	01/04/2014	13	Taratura	c	SU	EMR	Sacchetto polietilene	
16/04/2014	ISPRASU8003	ISPRA	01/04/2014	14	Taratura	c	SU	EMR	Sacchetto polietilene	
16/04/2014	ISPRASU8004	ISPRA	01/04/2014	15	Taratura	c	SU	EMR	Sacchetto polietilene	
16/04/2014	ISPRAZO8001	ISPRA	01/04/2014	16	Taratura	c	ZO	EMR	Bottiglia di plastica	
16/04/2014	ISPRAZO8002	ISPRA	01/04/2014	17	Taratura	c	ZO	EMR	Bottiglia di plastica	
16/04/2014	ISPRACI8001	ISPRA	28/03/2014	18	Taratura	c	CI	EMR	Tubi Falcon	
16/04/2014	ISPRACI8002	ISPRA	28/03/2014	19	Taratura	c	CI	EMR	Tubi Falcon	
16/04/2014	ISPRAGW8002	ISPRA	25/03/2014	20	Taratura	c	GW	EMR	Colonna anionica	
16/04/2014	ISPRAGW8003	ISPRA	25/03/2014	21	Taratura	c	GW	EMR	Colonna anionica	
16/04/2014	ISPRAGW8001	ISPRA	25/03/2014	22	Taratura	c	GW	EMR	Colonna anionica	
16/04/2014	ISPRAGW8001	ISPRA	01/04/2014	23	Taratura	c	GW	EMR	Colonna anionica	
16/04/2014	ISPRAGW8002	ISPRA	01/04/2014	24	Taratura	c	GW	EMR	Colonna anionica	
16/04/2014	ISPRAGW8003	ISPRA	01/04/2014	25	Taratura	c	GW	EMR	Colonna anionica	
16/04/2014	ISPRASW8001	ISPRA	01/04/2014	26	Taratura	c	SW	EMR	Colonna anionica	

The Silver Nitrate method (Silva et al., 2000) for $\delta^{15}\text{N}_{\text{NO}_3}$ and $\delta^{18}\text{O}_{\text{NO}_3}$ analyses

Pretreatment

Concentrating Dissolved Nitrate



Protocol

Desorption of Nitrate from Anion-Column and Conversion to AgNO_3



To desorb NO_3^- from anion column apply 30ml of 3M HCl (3ml x 10) to the anion column



Neutralize with ~15g Ag_2O



Filter through Whatman #1 to remove AgCl



Add BaCl_2 to precipitate SO_4^{2-} and PO_4^{3-} . Refrigerate.



Filter through Whatman #1 to remove barium precipitate.



Pass sample through cation resin to remove soluble barium.



Neutralize sample with ~ Ag_2O



Filter through Whatman #1 to remove AgCl



Separate liquid samples for ^{15}N and ^{18}O analysis.

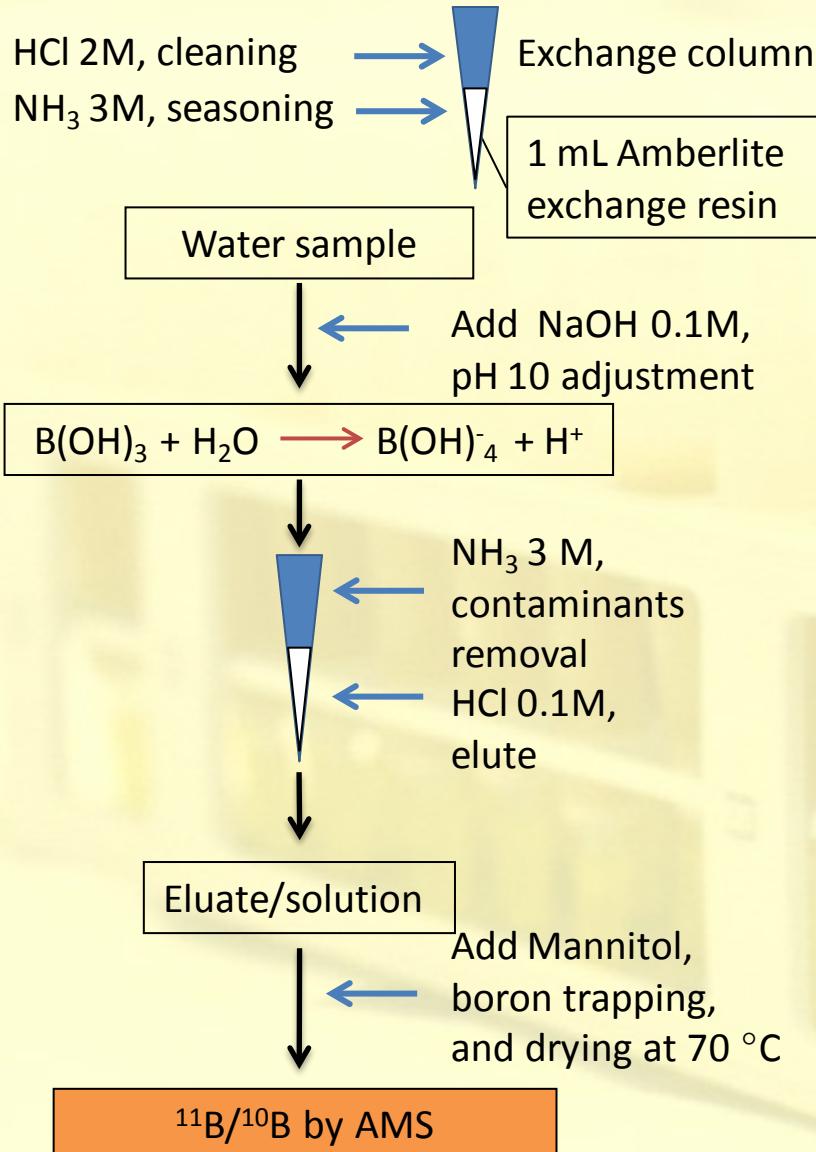
Minimum quantity of NO_3^- :

6 mg (100 μmol)

3 Internal Reference Materials (RM)
in two replicates processed along
with Unknown samples:

- 1) **USGS 34** ($\delta^{15}\text{N}=-1.8\pm0.2\text{\textperthousand}$;
 $\delta^{18}\text{O}=-27.9\pm0.6\text{\textperthousand}$);
- 2) **SiAl KNO₃** ($\delta^{15}\text{N}=26.9\pm0.8\text{\textperthousand}$;
 $\delta^{18}\text{O}=23.8\pm0.1\text{\textperthousand}$);
- 3) **CIRCE KNO₃ 3** ($\delta^{15}\text{N}=2.5\pm0.5\text{\textperthousand}$;
 $\delta^{18}\text{O}=24.8\pm0.5\text{\textperthousand}$).

The preparation protocol for $\delta^{11}\text{B}$ analyses



Minimum quantity of B required:

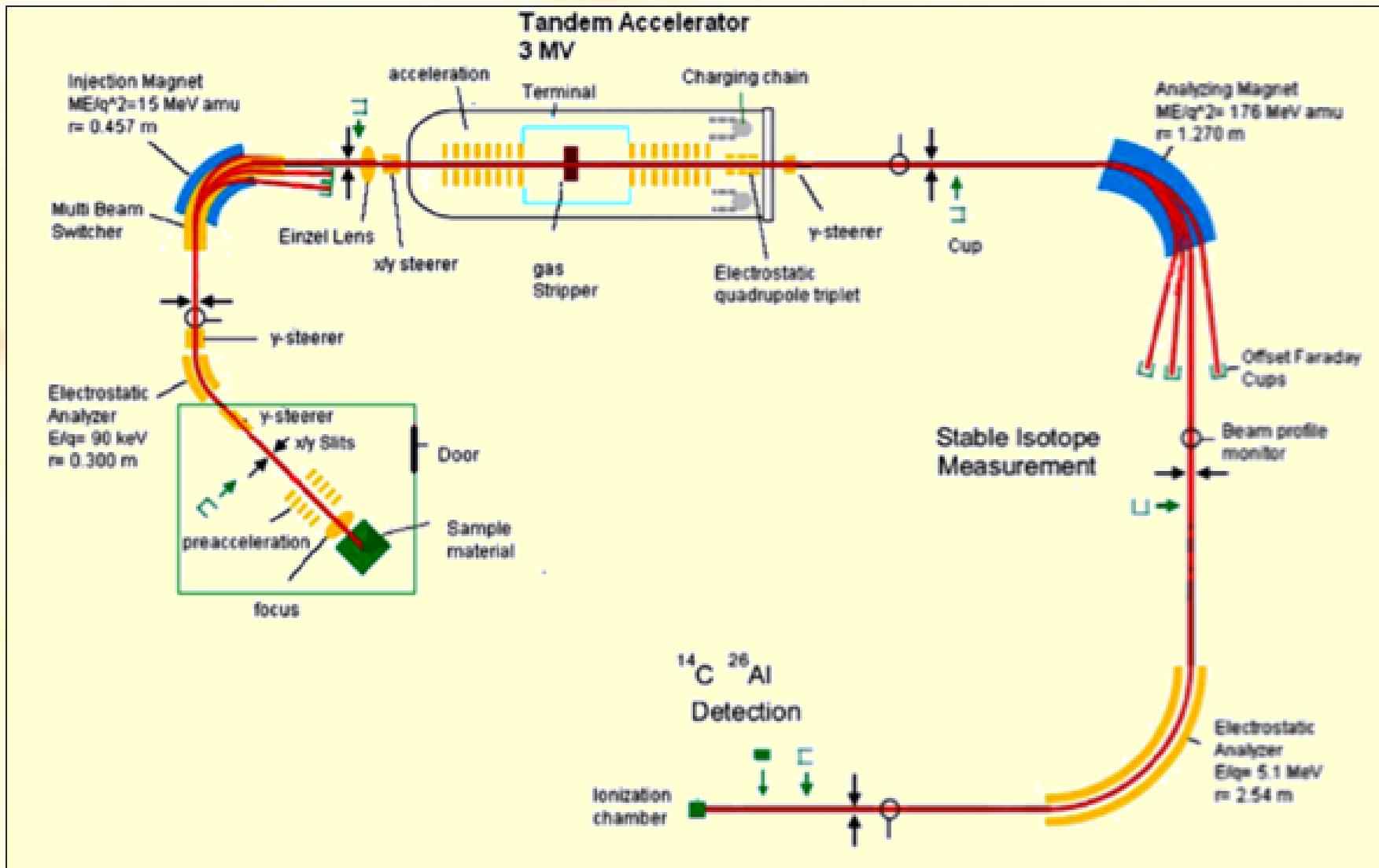
500 μg

International Reference Materials
(RM) in 5 replicates processed along
with Unknown samples:

NIST951 ($\delta^{11}\text{B}=0\text{ ‰}$)

$$\delta^{11}\text{B}[\text{‰}] = \left[\frac{R_{\text{sample}}}{R_{\text{SRM } 951}} - 1 \right] \times 1000$$

The AMS analysis of $\delta^{11}\text{B}$



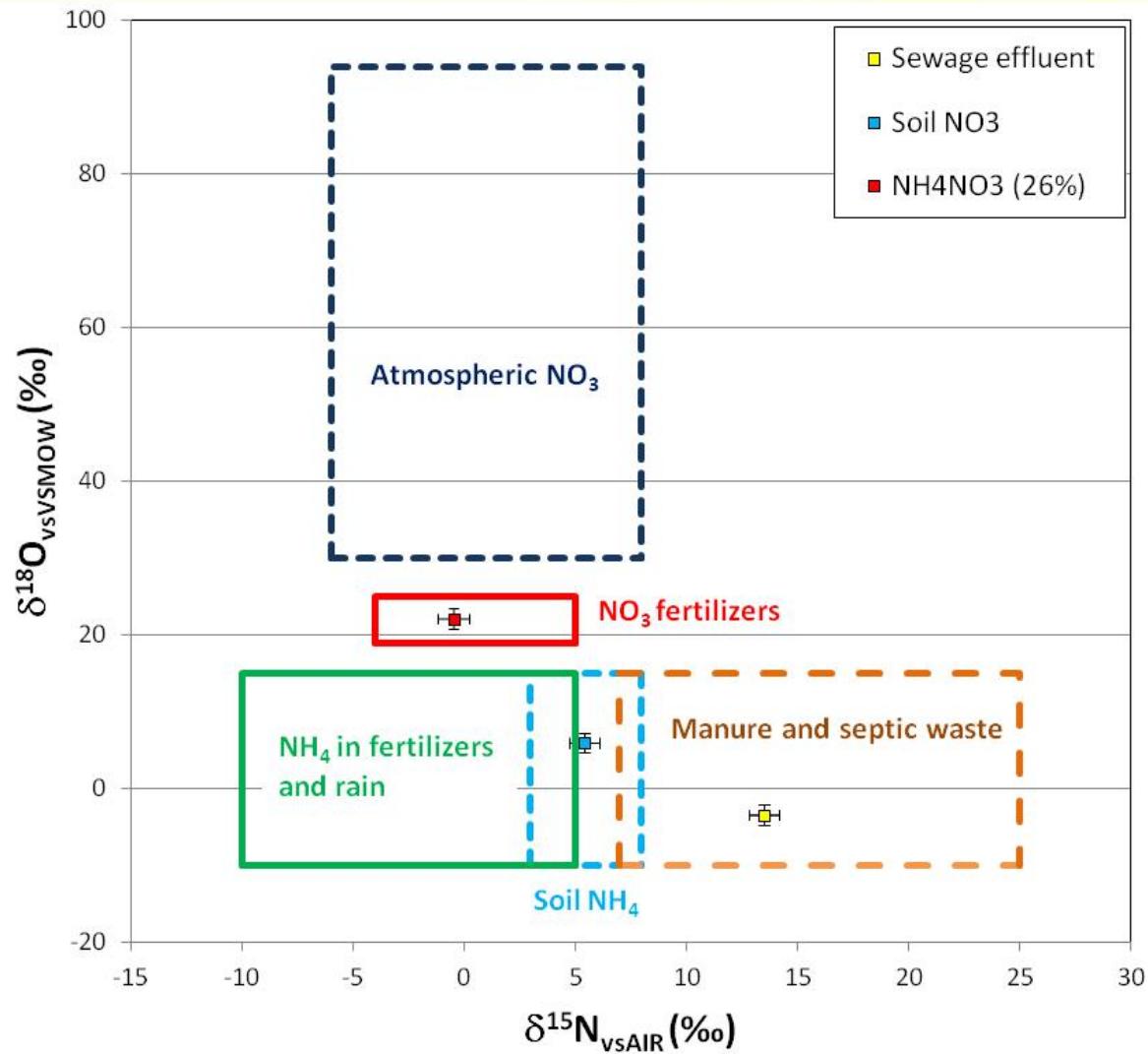
The isotopic analyses: Measurement performances

Measurement precision:

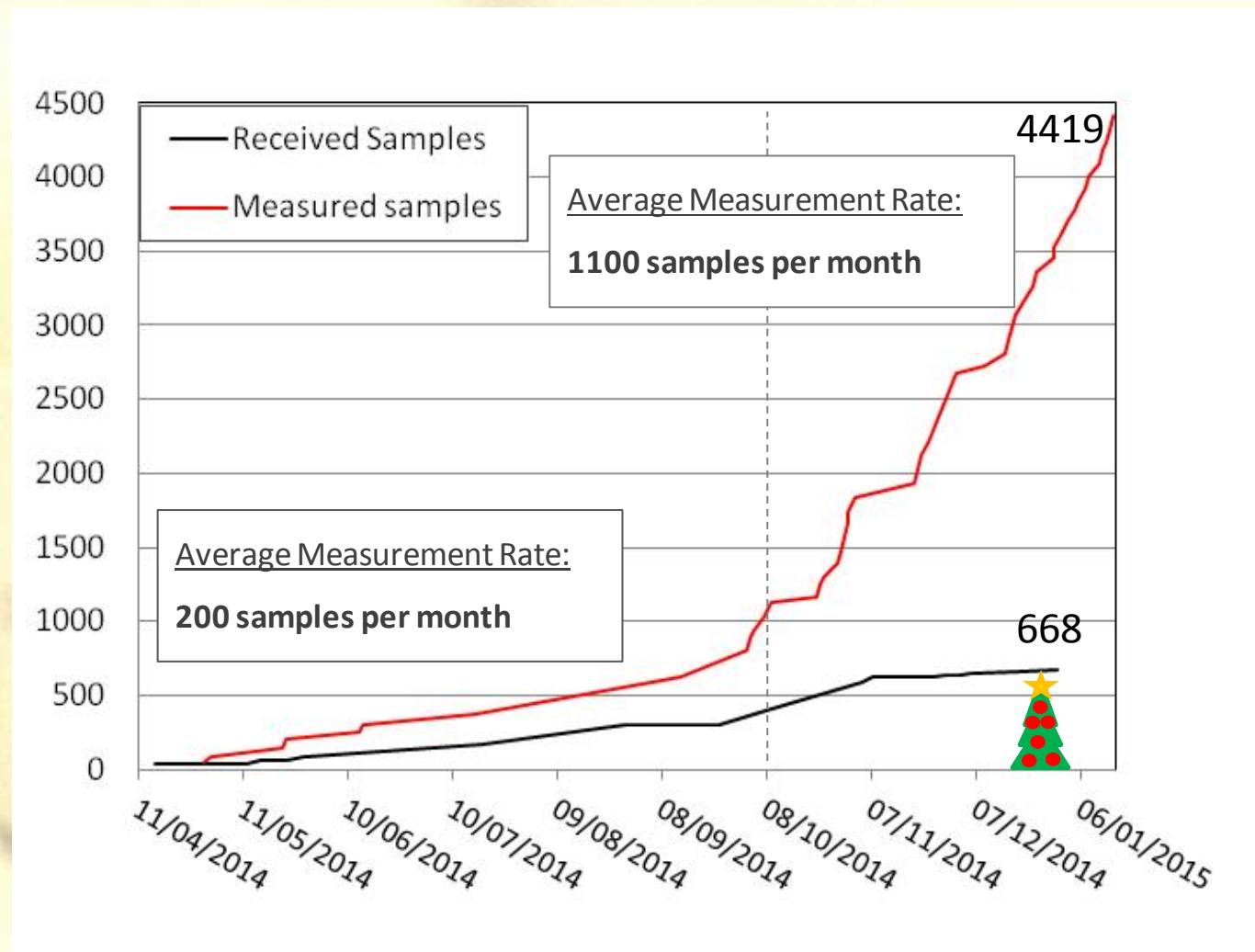
$$\delta^{15}\text{N}_{\text{NO}_3} = \pm 0.7 \text{ ‰}$$

$$\delta^{18}\text{O}_{\text{NO}_3} = \pm 1.3 \text{ ‰}$$

$$\delta^{11}\text{B} = \pm 6 \text{ ‰}$$

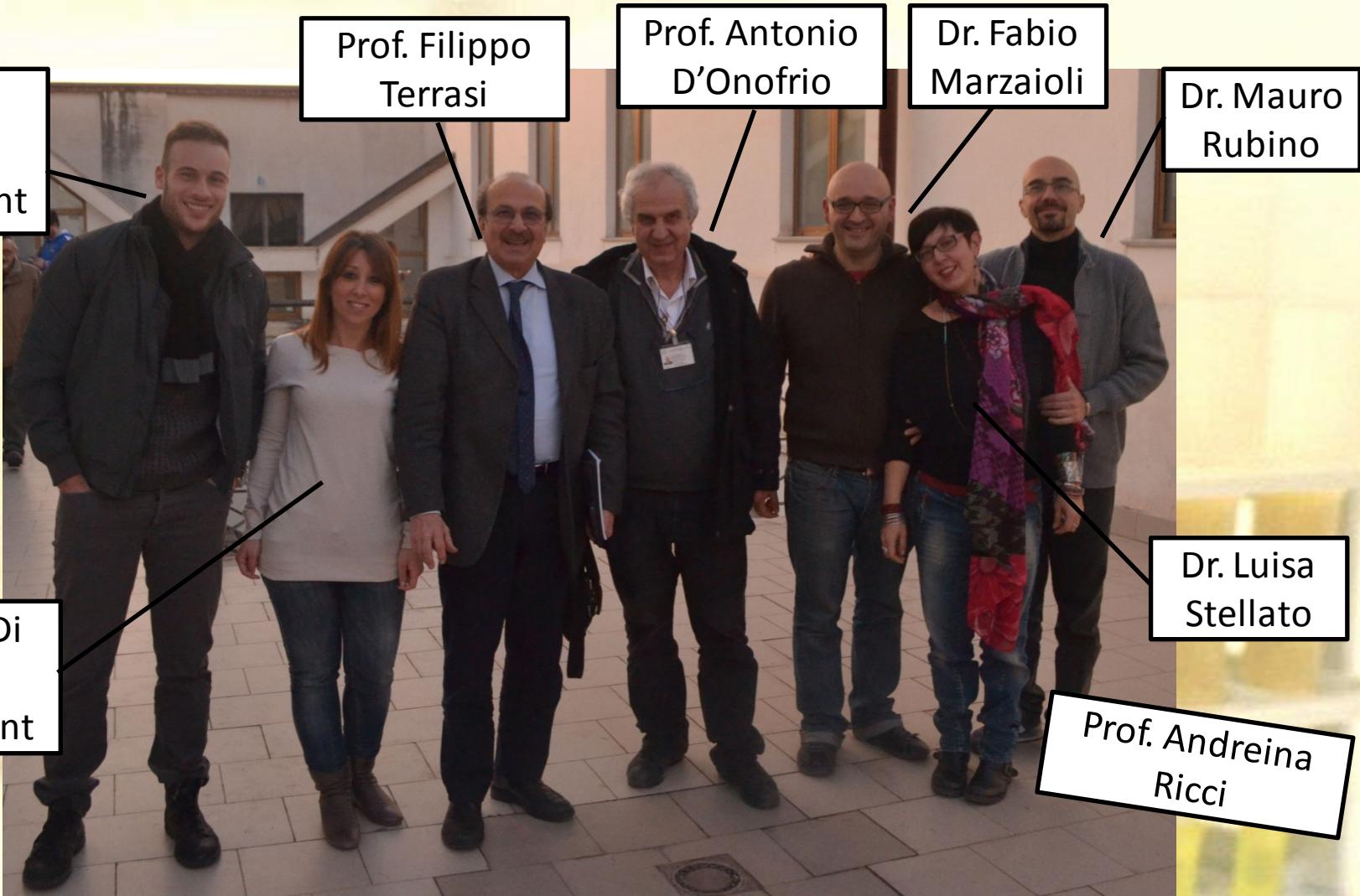


Throughput of the CIRCE lab for the Isonitrate Italy project



Average Measurement Rate: 510 samples per month

The CIRCE working group





(Artwork made of TC/EA consumables)