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333

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SR ISOTOPE ANALYSIS OF WATER SAMPLES AT THE RADIOGENIC ISOTOPE LABORATORY OF THE ISTITUTO NAZIONALE DI GEOFISICA E VULCANOLOGIA, SEZIONE DI NAPOLI - OSSERVATORIO VESUVIANO (INGV-OV)

Ilenia Arienzo[1], Alessandro Fedele[1], Barbara Capuozzo[1], Stella Tamburrino[2],
Marcello Liotta[3], Renato Somma[1]

[1]**INGV** (Istituto Nazionale di Geofisica e Vulcanologia, Sezione di Napoli - Osservatorio Vesuviano)
[2]**CNR - IAMC** (Istituto per l'Ambiente Marino Costiero)
[3]**INGV** (Istituto Nazionale di Geofisica e Vulcanologia, Sezione di Palermo)

333

Index

Introduction

Since 2000, a Thermal Ionisation Mass Spectrometer (Thermo Scientific^{TM} Triton TI$^{®}$ Mass Spectrometer) and a clean laboratory are operating at the Istituto Nazionale di Geofisica e Vulcanologia (INGV), Sezione di Napoli, Osservatorio Vesuviano (OV) to measure strontium (Sr) and neodymium (Nd) isotope compositions of volcanic products for scientific purposes.

In 2014 particular attention has been dedicated to set up the analytical procedure for extracting Sr and Nd and measuring their isotope compositions from groundwater and mineral water, due to its growing interest on environmental topics.

Strontium is considered a trace element and Sr^{2+} preferentially substitutes for Ca^{2+} in most rock types since it geochemically behaves like calcium. Groundwater is enriched in Sr during water-rock interaction processes occurring within the saturated and/or unsaturated zones. Conversely, Sr is removed from water as a result of mineral precipitation and ion exchange reactions. The most common removal process is the co-precipitation of Sr^{2+} ions with calcium carbonate. However, this latter process does not fractionate Sr^{2+} [Faure and Powell, 1972]. Therefore, the Sr isotopic composition of groundwater records an integrated signal of water-rock interaction along flow path and dissolution/precipitation events, and can be used as a dynamic tracer to constrain subsurface flow in volcanic, non volcanic and geothermal areas. Water is a natural resource, which is renewed by different processes. The aforementioned geochemical processes and reactions with dissolving/precipitating minerals have a profound effect on water quality.

Since the 1990s, Sr isotopes have been extensively used as a natural tracer of groundwater flow [Peterman and Stuckless, 1992; Bullen et al., 1996; Johnson and De Paolo, 1994; McNutt et al., 1990; McNutt, 2000; Frost et al., 2002; Gosselin et al., 2004; Klaus et al., 2007] because groundwater progressively acquires the $^{87}Sr/^{86}Sr$ isotopic ratio from the rocks with which it has interacted [Prasanna et al. 2009]. Furthermore, variable Sr isotope ratios and concentrations characterize different groundwater sources and are tracers of groundwater mixing [eg. Carucci et al., 2012].

In this work we present the first results obtained by analyzing the Sr isotope composition of two mineral waters, selected groundwater samples from Mt. Etna and a certified water sample from North America (National Research Council Canada - NRC, Certified Reference Materials - CRM TM-25.4). On the contrary, the Nd isotope ratios of such water samples have been not measured due to the low Nd content of the investigated samples. A statistically representative data set on certified international standards (NIST SRM 987, La Jolla and JNdi-1) was used to evaluate the quality of the analytical data produced at the INGV-OV Radiogenic Isotope Laboratory since 2014.

This methodological approach will allow us to isotopically characterize different water systems with the aim to trace back the water-rock interaction and mixing processes in different environments. This analytical procedure could be exported to other geological contests and applied to other types of waters (e.g. surface and thermal water).

1. Clean Chemistry Laboratory

A Clean Chemistry Laboratory has been set up at the INGV-OV, equipped with conditioned (ca. 23°C) and over-pressured air, since year 2000. In order to separate Sr and Nd from sample solutions ion-exchange chromatographic techniques on column are used, in which the stationary bed is hosted within a quartz tube. In the INGV-OV Clean Laboratory (Fig. 1) quartz columns for Sr and Rare Earth Element (REE) separation with an internal diameter of about 0.5 cm and height of ca. 22 cm, with a 20 ml reservoir at the top (Fig. 2) were set up and calibrated in March 2015.

Figure 1. The Clean Chemistry Laboratory at the INGV-OV.

Columns were prepared by adding cation exchange resin Dowex AG 50 W X-8 (200-400 mesh). The resin has been washed several times with 6N HCl and then conditioned with 2.5N HCl. In order to know the amount of acid to be eluted and discarded before collecting Sr, a solution made of 0.5 ml of 2.5N HCl, containing ca. 70 ppm of Sr, was loaded on each column. Then, several 1 ml aliquots of 2.5N HCl were loaded into the columns and each time the eluted aliquots were collected in different, labeled beakers. The Sr content of each aliquot was measured by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) at the Geochemistry Laboratory of the Istituto per l'Ambiente Marino Costiero, Centro Nazionale Ricerche (IAMC-CNR).

From the same sample from which Sr has been separated and collected, it is possible to collect the REE fraction, in order to successively extract Nd for measuring the $^{143}Nd/^{144}Nd$ isotopic ratio. In order to calibrate the column to separate the Rare Earth Elements (REE), 0.5 ml of a solution 2.5N HCl, containing ca. 80 ppm of Nd, were loaded in each column. The amount of 2.5N HCl, corresponding to that necessary to elute and then collect Sr, was eluted and discarded. Afterwards, several aliquots of 0.5 ml of 6.3N HCl were eluted and collected in different labeled beakers. With the aim of detecting the presence of REE, in each beaker containing the eluted acid solution, *Eriochrome Black T*, a chemical indicator sensible to metal presence in basic solutions, was added [Arienzo et al., 2013].

New, smallest columns for the chromatographic separation of Nd were prepared (Fig. 2) by adding few ml of LNB25 resin, washed several times with 6N HCl and then conditioned with 0.5N HCl. Columns for Nd separation have height of 16 cm, internal diameter of 0.5 cm, and a 15 ml reservoir. The calibration has been performed by loading 0.5 ml of a 0.25N HCl solution containing about 80 ppm of Nd, on each column. Then, several 0.5 ml aliquots of 0.25N HCl were eluted, and collected in labeled beakers [Arienzo et al., 2013].

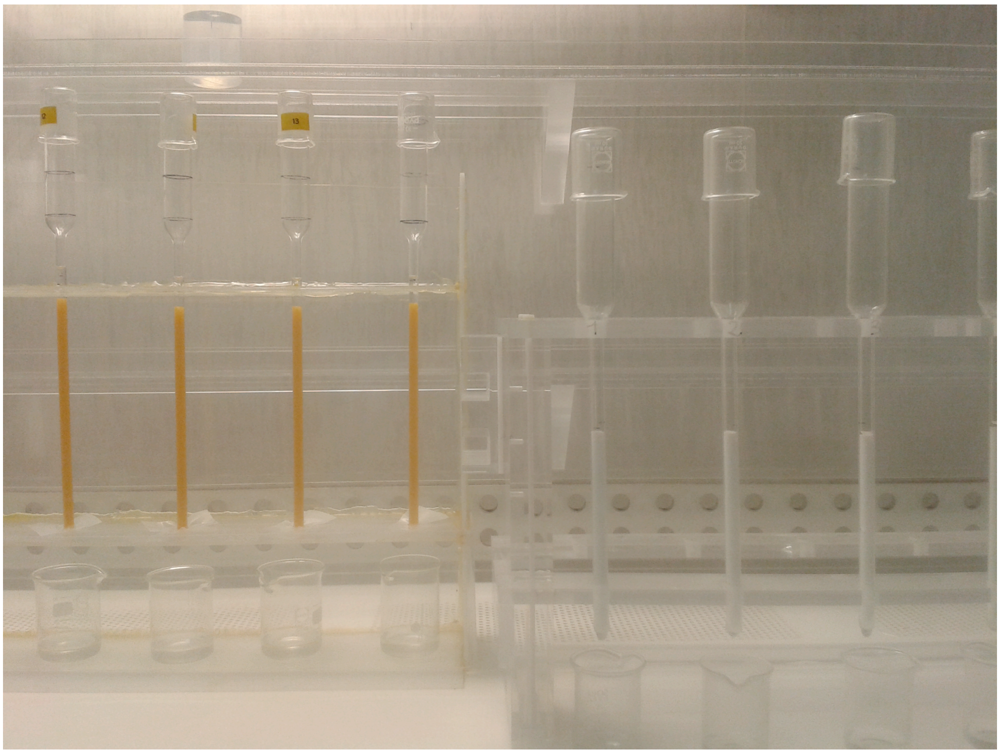

Figure 2. Detail of the quartz columns for Sr and REE (left side), and Nd separation (right side), by chromatographic techniques.

1.1 Sample selection

In order to test the procedure adopted in the Radiogenic Isotope Laboratory for measuring the Sr and Nd isotope ratios of water, a certified water sample (NRC CRM TM-25.4), two mineral waters and ~~a~~ six groundwater samples from the eastern and south flanks of Mt. Etna were selected.

Sample TM-25.4, provided by the IAMC-CNR (Naples), was a filtered water from Lake Ontario (Canada, North America) of known chemical composition. This fortified bulk CRM has concentrations in the low range and is designed by the National Research Council of Canada for method validation or as a quality control tool merely for trace element analysis. Certified values for NRC CRM TM 25.4 were obtained in an inter-laboratory program with a robust statistical analysis (ISO DIS 13528:2005(E)). The Sr elemental content of this standard is of 73.4 µg/l ± 5.4 (2σ). Currently, no isotope ratio values relative to this standard are provided by NRC nor published.

Mineral waters analyzed are Gaudianello and S. Anna di Vinadio. The former is tapped from a spring in Rionero in Vulture (Basilicata, S. Italy), the latter from a spring in locality Vinadio (Piemonte, North Italy).

Natural groundwater samples were collected by the staff of INGV, Sezione di Palermo, from the Mt. Etna basalt-hosted aquifers. These samples belong to the monthly sampling program for volcanic surveillance they carry out. At each sampling site, several aliquots are collected and treated according to the type of chemical and isotopic analysis to be performed. In particular, samples to be analyzed for Sr concentration and Sr isotopic composition were filtered with 0.45-µm-pore filters and acidified with ultrapure HNO_3 (65%) just after the sampling (labeled FA in Table 1). This treatment prevents the precipitation of carbonate minerals and the formation of hydroxides since these processes can remove significant amounts of Sr. At each site a further aliquot of not-treated sample (labeled TQ in Table.1) was collected in order to perform a comparison on the analytical results. Sr content was determined by ICP-MS (7500ce, Agilent) at INGV, Sezione di Palermo. Nd content has not been determined on such samples, although it is typically of few ppm in water [eg. Leybourne and Cousens, 2005].

1.2 Samples preparation

Fifteen to 50 ml of water samples, were evaporated to dryness in Savillex® Teflon beakers, depending on their Sr content, in order to have from ca. 40 (Sample TM-25.4) to ca. 100 ppm (groundwater samples from Mt. Etna) of Sr in the investigated sample. The residue, dissolved in a variable amount (from 1.5 to 4 cc) of 2.5N HCl, was loaded on columns and Sr extracted from the solution by conventional cation exchange methods. Once the chemical separation of Sr was completed, a few drops of concentrated ultrapure HNO_3 were added and then dried on hot plate to convert the collected fractions from chlorides to nitrates.

The Sr fractions re-dissolved in diluted HNO_3 were loaded on previously degassed zone refined Rhenium (Re) filaments (Fig. 3), to carry out mass spectrometer analysis [Arienzo et al., 2013].

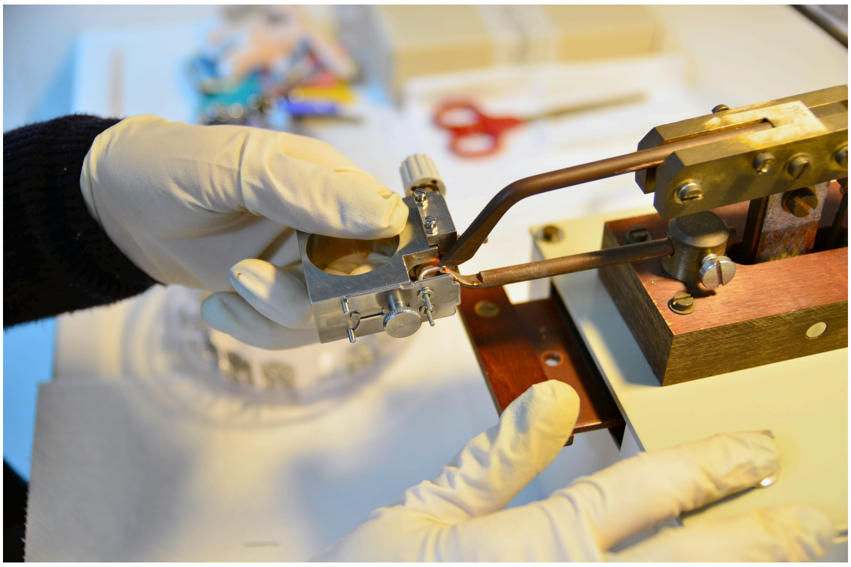

Figure 3. Preparation of the Re filaments for Sr and Nd isotope ratios measurements.

The possibility to measure the Nd isotopic composition of water samples was also tested by using two groundwater samples from Mt. Etna. For both samples we could separate Nd from the other REE. However, due to the low Nd content of water samples their $^{143}Nd/^{144}Nd$ ratios have not been measured, as the signal of the ^{144}Nd was below 0.2V.

1.3 Sr blanks

All sample preparation procedures unavoidably result in mixing between the element analyzed in the sample and the same element occurring in the reagents and laboratory environment. The level of contamination of the Radiogenic Isotope Laboratory was determined by performing the entire chemical procedure in the absence of sample. Blank determinations consist in weighting an amount of a spike containing the element to be analysed and making the same dissolution and chromatographic separation as for the natural samples, and finally measuring its isotopic composition. 0.02 g of a spike solution, containing $3.7*10^{-9}$ mol/g of Sr (available by courtesy of Prof. Riccardo Petrini, University of Pisa), was dried down, then taken up in 0.5 ml of 2.5N HCl and loaded on column [Arienzo et al., 2013]. During the period of chemistry processing the measured total procedure Sr blank was of the order of 0.2 ng and did not affect the Sr isotopic measurements.

2. Sr and Nd isotope ratios determinations

Since 2001 in the Radiogenic Isotope Laboratory of INGV, Sr and Nd isotopes on natural samples (whole rocks, minerals, groundmass and volcanic glasses) are routinely measured, in order to study the volcanic systems and to define the magmatic processes in the Earth sciences. The running conditions for determining the isotope composition of natural samples were tested by measuring the isotope ratios of reference samples. Sr and Nd standard solutions were prepared, by using as starting material $SrCO_3$ and the Nd_2O_3, in order to load ca. 200 ng of these elements on filaments. Since December 2014, we systematically measure $^{87}Sr/^{86}Sr$ of the NIST SRM 987 and $^{143}Nd/^{144}Nd$ of both the Nd La Jolla and the JNdi-1 standards, this latter kindly provided by Dr. Tanaka from the Geological Survey of Japan. The recommended values for $^{87}Sr/^{86}Sr$ and $^{143}Nd/^{144}Nd$ are 0.710248±11, 0.511856±7 and 0.512115±7, respectively [Thirlwall, 1991; Tanaka et al., 2000].

In the Radiogenic Isotope Laboratory isotopic measurements were performed with a Thermal Ionisation Mass Spectrometer (TIMS) Thermo Scientific^{TM} MAT Multicollector Triton TI$^{®}$ [e.g., Wieser and Schwieters, 2005] (Fig. 4). The room where the instrument is located was maintained at constant temperature of 24 °C.

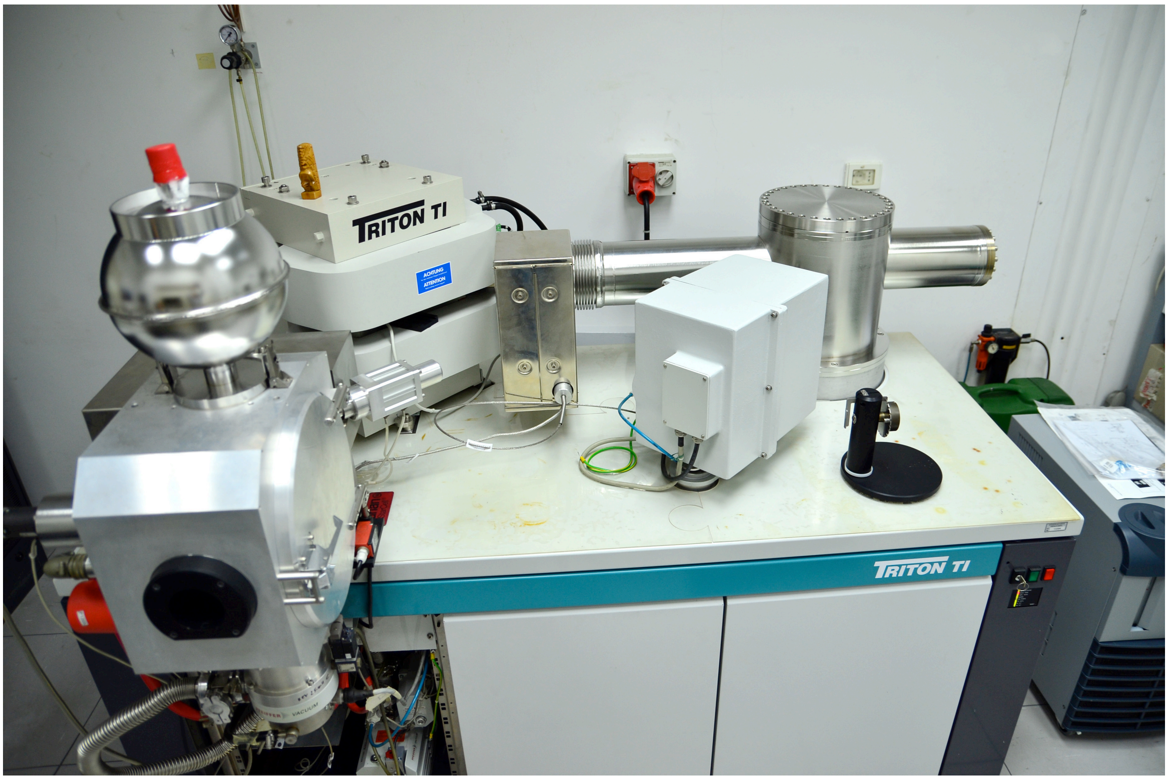

Figure 4. The Thermal Ionisation Mass Spectrometer (TIMS) installed at the INGV-OV.

Measurements were performed with ^{88}Sr and ^{144}Nd signals of ca. 6 Volt and 2 Volt and integrating time of 16 and 8 seconds, respectively. The measured isotopic ratios are mean values of 175 ratios, divided in 5 blocks of 35 measures for Sr and 7 blocks of 25 measures for Nd. We generally report the analytical error as ± 2 times the standard error (2se). [Arienzo et al. 2013].

Analyses made on the Sr and Nd standards in the time interval from March to July 2015 are plotted in Figures 5a and b, respectively. Mean values of the measured isotope ratios are 0.710215 ($2\sigma = 1.4x\ 10^{-5}$, N=77), 0.511843 ($2\sigma= 1.5x\ 10^{-5}$, N= 22) and 0.512101 ($2\sigma= 10x\ 10^{-5}$, N=16) for the NBS 987, La Jolla and JNdi-1, respectively. The error is expressed as 2σ, where σ is the standard deviation among all measurements performed in that time span, and represents the external reproducibility [Goldstein et al., 2003].

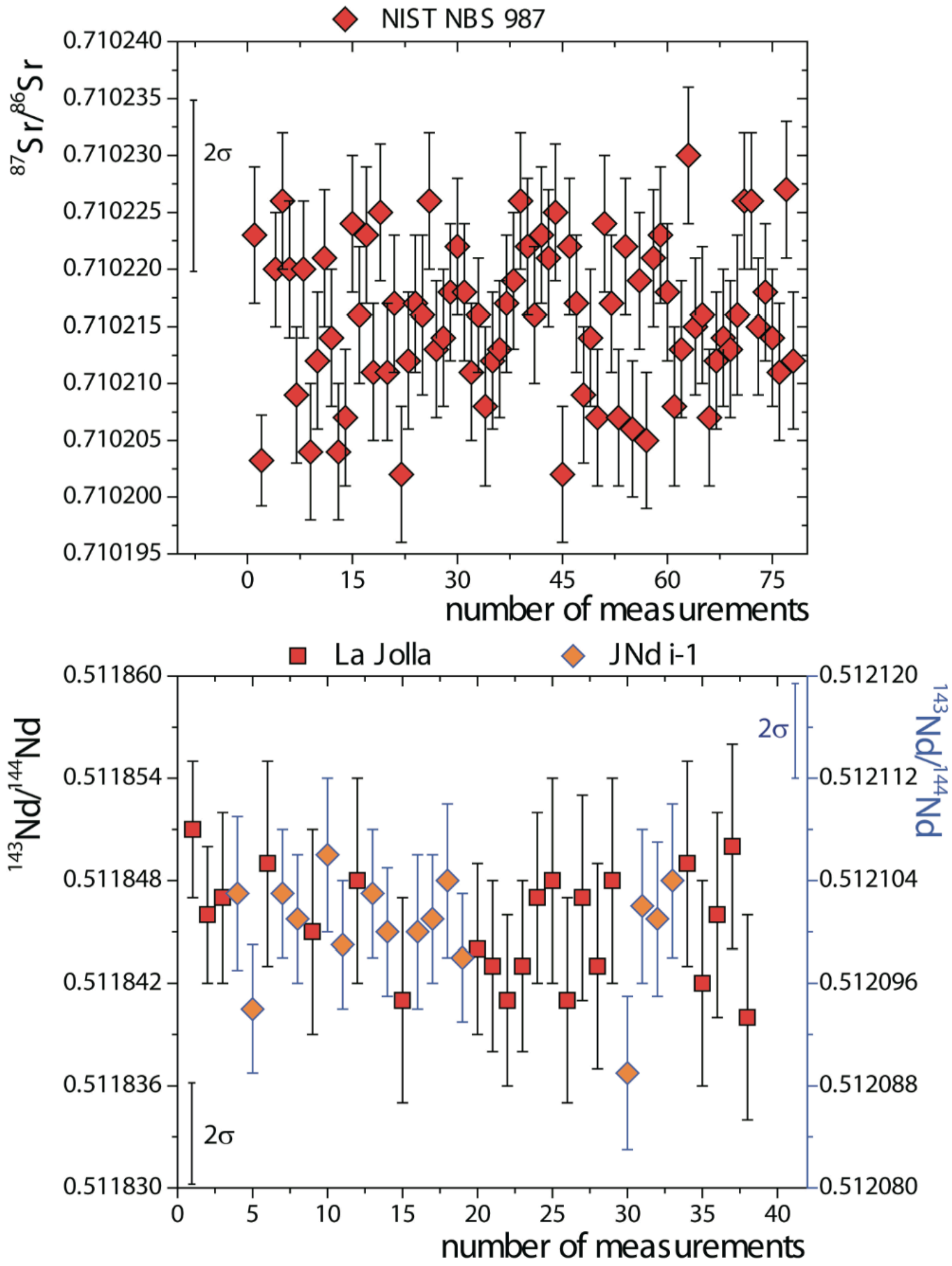

Figure 5. a) $^{87}Sr/^{86}Sr$ (NIST SRM 987) and b) $^{143}Nd/^{144}Nd$ (La Jolla and JNdi-1) ratios measured from March to July 2015.

3. Results

The Sr isotope ratio determined on the TM 25.4 was 0.709374±0.000006, this value being in the range of those obtained for the sediments close to the Lake Ontario [Skuce et al., 2015; Ardakani et al., 2013 and Hollings et al., 2011]. The analyzed mineral waters have Sr isotopic compositions strictly influenced by water/rock interaction processes. In particular, that from Mt. Vulture (Gaudianello) is featured by $^{87}Sr/^{86}Sr$ similar to those measured for whole rocks [eg. Peccerillo 2005] and in the range of those reported by Barbieri and Morotti [2003] for spring waters, from Mt. Vulture. Water from the Alp spring (S. Anna di Vinadio) is characterized by the highest Sr isotope ratios among all those analyzed, in the range of values reported for the metamorphic basement of the westernmost part of the Southern Alps [e.g. H. Voshage et al., 1987; Pinarelli and Boriani, 2007]

Groundwater samples from the eastern flank of Mt. Etna volcano were analyzed for $^{87}Sr/^{86}Sr$ using both treated (filtered and acidified in the field - FA) and untreated aliquots (TQ). Considering the $^{87}Sr/^{86}Sr$ isotopic ratios of all the analyzed groundwater samples, they fall within the range of the Mt. Etna volcanics analyzed by Corsaro et al. [2009] and Correale et al. [2014] and in the range of ground water analysed by Pennisi et al. [2001], Analytical results indicated that treated and untreated samples provided similar $^{87}Sr/^{86}Sr$ isotopic ratios, i.e. within the analytical error (Table 1). However, since acidification prevents the precipitation of carbonate minerals and then, the Sr co-precipitation and the formation of hydroxides, before performing the analysis of the chemical composition is extremely important that groundwater samples are acidified, if Sr quantification and $^{87}Sr/^{86}Sr$ ratio determination are to be performed.

Table 1. Sr concentrations (μg/L) and isotopic ratios ($^{87}Sr/^{86}Sr$) of groundwater samples from Mt. Etna.

Groundwater sample	**Number of replicates -standard deviation**	**TDS mg/L**	**Sr (μg/L)**	**$^{87}Sr/^{86}Sr$**	**2 sigma**
PR FA	5 – 4.3%	1887	739	0.703380	0.000006
PR TQ		n.a.	n.a.	0.703389	0.000006
S 26 FA	5 – 2.8%	1741	719	0.703375	0.000006
S 26 TQ		n.a.	n.a.	0.703385	0.000006
SG FA	5 – 2.4%	574	559	0.703281	0.000006
SG TQ		n.a.	n.a.	0.703283	0.000006
Gaudianello		n.a.	n.a.	0.706062	0.000006
S. Anna di Vinadio		n.a.	n.a.	0.739914	0.000006

4. Conclusions

Although Sr isotopes do not fractionate we suggest that, to determine the chemical and isotopic compositions of water samples, these latter must be acidified, in order to prevent major and trace elements (among them Sr) precipitation, and hence to correctly interpret the observed chemical and the isotopic variations. The obtained preliminary results show that Sr isotope ratios of the analyzed waters reflect those of the host rocks. In light of this, together with chemical composition, Sr isotope ratios represents a powerful tool to track the groundwater subsurface flow in complex geological settings such us volcanic areas and geothermal systems. This analytical procedure could be exported to other geological contests and applied to other types of waters (e.g. surface and thermal water), and will allow discriminating between the contribution of different sources (i.e. sea water, meteoric water, deep uprising fluids and so forth).

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