JOINT INSTITUTE FOR NUCLEAR RESEARCH.

FRANK LABORATORY OF NEUTRON PHYSICS.

FINAL REPORT ON THE START PROGRAMME.

"*ELEMENTAL COMPOSITION OF THE SAMPLES FROM FUMAROLIC ZONE DETERMINED BY ICP-OES*"

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Contents.

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Abstract.

The elemental composition of water, soil and algae samples collected in the municipality known as “Los Azufres” was determined by Inductively Coupled Plasma Optical Emission. The content of the following elements was determined in the samples: Ba, Cd, Fe, Mn, Ni, Pb, Zn, Cu, Co, Cr, Al, Sr, V, P, S, Ca, Mg, K, Na.

Additionally, he became familiar with neutron activation analysis and Genie-2000 software.

Dubna, 2024.

Introduction.

"Los azufres" is a municipality in the state of Michoacán in Mexico surrounded by enormous amounts of volcanic fumaroles where lakes and hot springs are also found, a result of the extensive geographic map of fumaroles found on the surface, these are located in that area. of Michoacán extending to different places on the American continent and part of the European continent, this area has been characterized by the frequency of tourists. This site is used for the supply of geothermal energy. The Los Azufres Power Plant annually supplies around 600 gigawatts/hour, this represents approximately 1.7 times the total electrical energy consumption of the city of Morelia.

Something very interesting about the fumaroles in "Los azufres" where plants, ferns and algae live near the fumaroles in very acidic conditions. Samples from these samples contain a high level of sulfur, but it is interesting to know what other elements they may contain. This task can be solved using multi-element techniques such as NAA or ICP-OES.

Project goals.

The main purpose of the project is to assess the level of chemical elements in the samples collected in Los Azufres as well as to get acquainted with new analytical techniques for elemental analysis.

Scope of work.

1. Water, soil and algae samples analysis using ICP-OES.

2. Preparation of the literature review for the manuscript.

3. Description of the results.

4. Introduction to neutron activation analysis.

5. Getting acquainted with the Genie-2000 software used for gamma spectra processing.

Samples analysis.

ICP-OES is an analytical technique used to determine the presence and concentration of chemical elements in a sample, where an ionized argon plasma is used to excite the atoms of the sample and measure the light emissions characteristic of each element, this allows us to identify and quantify the presence of chemical elements in the samples.

The main tool for the analysis of the samples was the Plasma Quant PQ 9000Elite equipment.

Figures 1 and 2 show the equipment and its calibration method for optimal operation, performed before the analysis.

The steps followed were the following:

1. Introduction of water samples into the equipment.

2. Nebulization: the water sample is converted into a fine mist that is introduced into the plasma.

3. Inductively coupled plasma (ICP): The plasma is generated by an inductively coupled coil that produces a magnetic field that ionizes the argon gas.

4. Atomization and excitation: the atoms of the sample are atomized and excited in the plasma, causing light to be emitted at different wavelengths.

5. Optical Emission Spectroscopy (OES): The spectrometer measures the light emitted by the atoms and breaks it down into an emission spectrum.

6. Data analysis: The equipment software analyzes the emission spectrum and determines the concentration of the elements present in the samples.

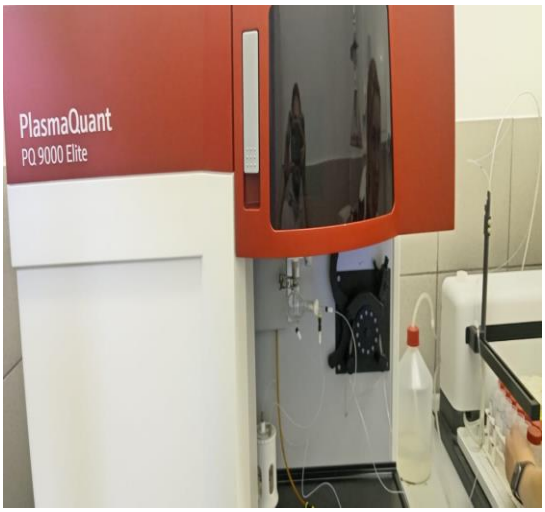

Figure 1 "Plasma Quant PQ 9000 Elite".

Figure 2 "equipment calibration".

Water Samples.

Water samples brought from "Los Azufres" were analyzed to detect the concentrations of elements present where this is expressed in units of mass per volume (for example mg/L or µg/L), said samples were treated with nitric acid. The following parameters pH, temperature, µs/ms, Ppm, %, SG, mV, as well as the chemical elements were determined in the two samples 1Al-1Aα and the sample 1AL-1Aβ (fig3).

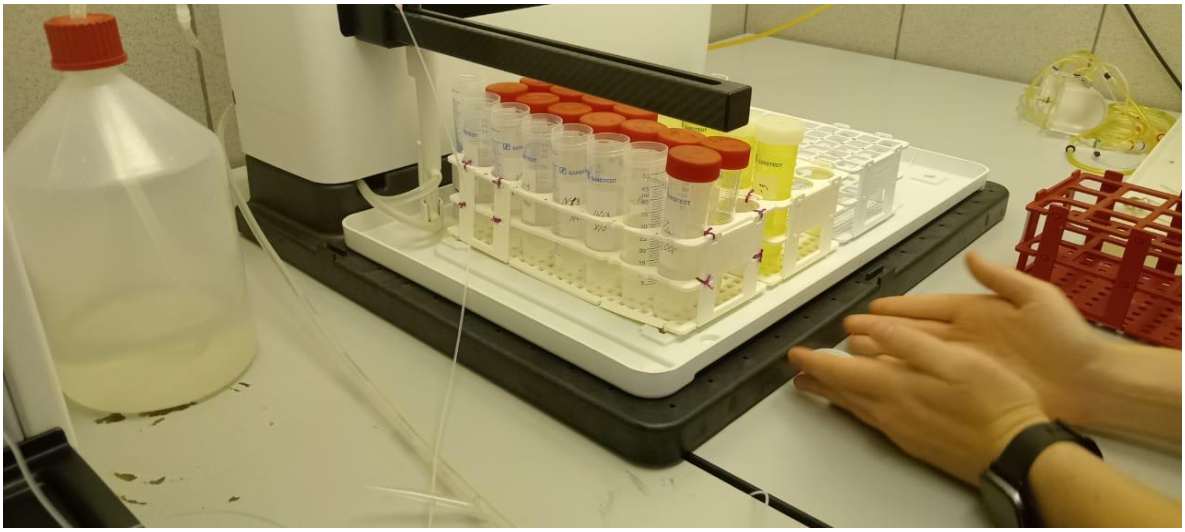

figure 3 "water samples prepared for the ICP-OES analysis process".

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The concentrations of the elements such as: Ba, Cd, Fe, Mn, Ni, Pd, Zn, Cu, Co, Cr, Al, Sr, V, P, S, Ca, Mg, K, Na, were determined in water samples (Table 1).

Plant samples.

The analysis of plant samples was a different process before implementing the use of the “ICP-OES” method; For this, the samples had to be prepared for work by separating them into smaller tubes in the chemistry laboratory, where the weights of each of the samples were recorded, where fig. 4 shows the bottles containing the samples.

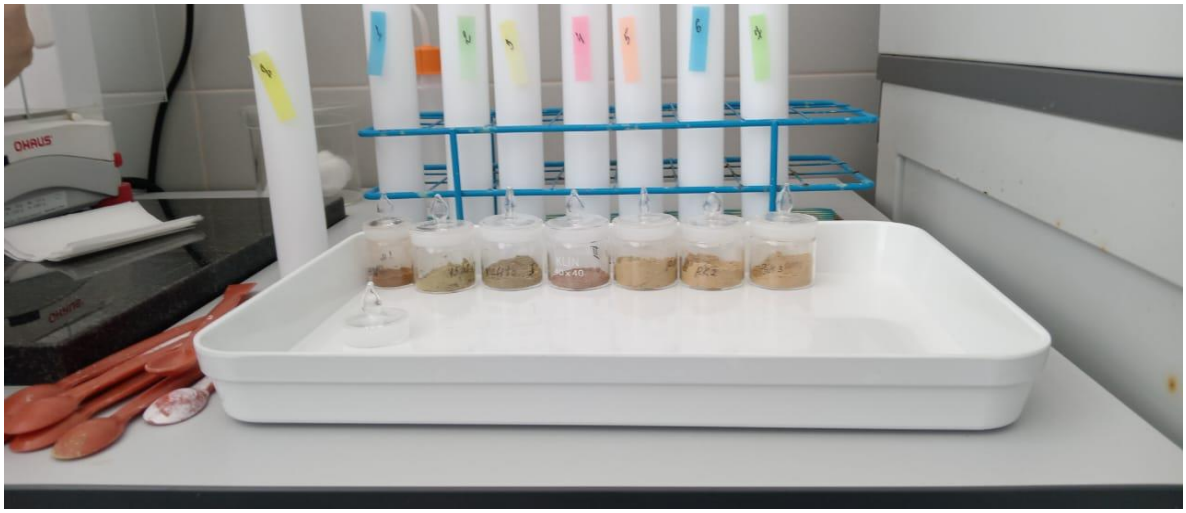


Figure 4. “content samples”.

This process was carried out in order to prepare samples where the samples were divided into 5 vials and mixed with 5 ml of nitric acid, 2 ml of hydrogen peroxide and 1 ml of hydrofluoric acid and allowed to rest for 25-30 minutes and placed in the mineralizer.

Figure 5. “Sample Mineralizer”.

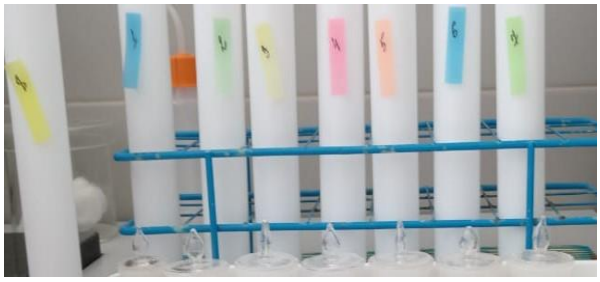


Figure 6. “vials with separate samples”.

Once the sample treatment process is finished, we carry out the analysis process using ICP-OES to determine Cd, Cu and Pd. The other elements will be determined using the “NAA” neutron activation analysis technique.

This entire process was also carried out to also analyze the soil and algae samples, the process of which will be described below.

Soil and Alga samples.

As mentioned in the previous point, the process for preparing the soil and algae samples was the same as for the plants, the samples were divided into 5 vials with a concentration of 5 ml of nitric acid, 2 ml of hydrogen peroxide and 1 ml of hydrofluoric acid. Acid and let it sit for 25-30 minutes and place it in the mineralizer, figure 5 shows the mineralizer.

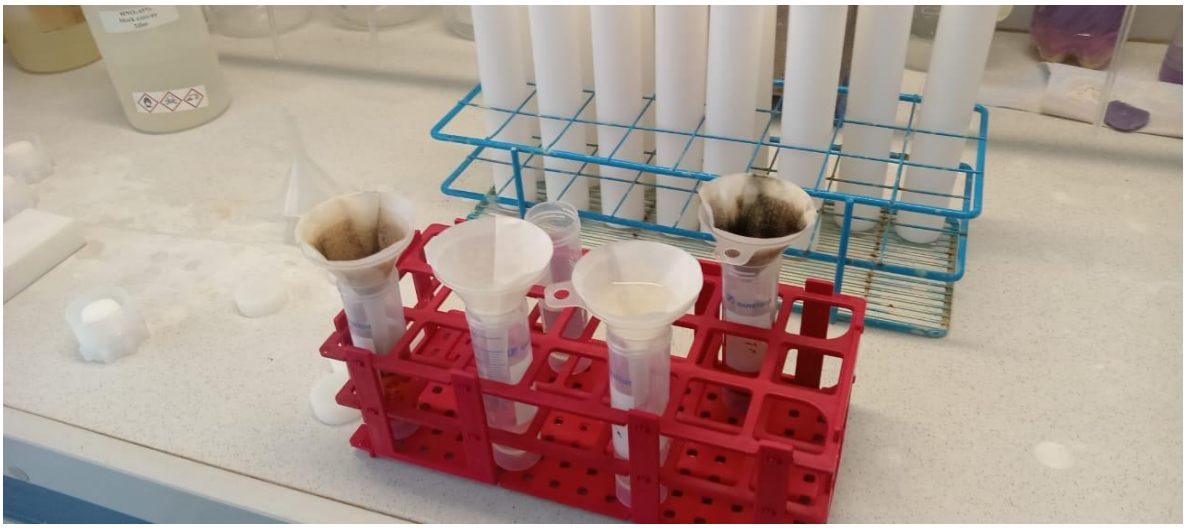


Figure 7 “soil and algae samples”.

In these samples, as in the plant and root samples, the elements Cd, Cu and Pd were determined. The other elements will be determined by using the “NAA” neutron activation analysis technique.

Results.

The results obtained for the water, plant, roots and soil samples using the ICP-OES method are presented below.

Table 1. water samples 1AL-1Aa-1 and 1AL-1Aa-2

Samples	Elements	mg/L	SD	Samples	Elements	mg/L	SD
1AL_1Aa_1	Ba	0.2375	0.0004	1AL_1Aa_2	Ba	0.2263	0.0011
1AL_1Aa_1	Cd	0.0004	0.0001	1AL_1Aa_2	Cd	0.0004	0.0001
1AL_1Aa_1	Fe	40.62	0.3557	1AL_1Aa_2	Fe	40.57	0.0515
1AL_1Aa_1	Mn	2.194	0.0111	1AL_1Aa_2	Mn	2.194	0.0143
1AL_1Aa_1	Ni	0.0311	0.0001	1AL_1Aa_2	Ni	0.0306	0.0012
1AL_1Aa_1	Pb	0.016	0.0007	1AL_1Aa_2	Pb	0.0161	0.0006
1AL_1Aa_1	Zn	0.3555	0.003	1AL_1Aa_2	Zn	0.3555	0.001
1AL_1Aa_1	Cu	0.0309	0.0004	1AL_1Aa_2	Cu	0.0311	0.0001
1AL_1Aa_1	Co	0.0168	0.0002	1AL_1Aa_2	Co	0.0168	0.0001
1AL_1Aa_1	Cr	0.0051	0.0002	1AL_1Aa_2	Cr	0.0048	0.0001
1AL_1Aa_1	Al	15.92	0.0019	1AL_1Aa_2	Al	15.68	0.176
1AL_1Aa_1	Sr	0.4163	0.0009	1AL_1Aa_2	Sr	0.4193	0.0016
1AL_1Aa_1	V	0.0283	0.0003	1AL_1Aa_2	V	0.0281	0.0001
1AL_1Aa_1	P	0.472	0.0016	1AL_1Aa_2	P	0.4585	0.0021
1AL_1Aa_1	S	400.1	5.191	1AL_1Aa_2	S	396.9	3.893
1AL_1Aa_1	Ca	55.65	0.0101	1AL_1Aa_2	Ca	55.93	0.1391
1AL_1Aa_1	Mg	21.34	0.1178	1AL_1Aa_2	Mg	21.36	0.2322
1AL_1Aa_1	K	69.76	0.8759	1AL_1Aa_2	K	69.28	0.062
1AL_1Aa_1	Na	96.45	0.2501	1AL_1Aa_2	Na	94.36	0.2517

Table 2 . water samples 1AL-1AB-1 and 1AL-1AB-2.

Samples	Elements	mg/L	SD	Samples	Elements	mg/L	SD
1AL_1AB_1	Ba	0.2366	0.0001	1AL_1AB_2	Ba	0.2829	0.0017
1AL_1AB_1	Cd	0.0005	0.0001	1AL_1AB_2	Cd	0.0005	0.0001
1AL_1AB_1	Fe	87.18	0.5669	1AL_1AB_2	Fe	87.85	0.0036
1AL_1AB_1	Mn	2.936	0.0031	1AL_1AB_2	Mn	2.94	0.0011
1AL_1AB_1	Ni	0.0552	0.0005	1AL_1AB_2	Ni	0.0548	0.0001
1AL_1AB_1	Pb	0.0075	0.0004	1AL_1AB_2	Pb	0.0082	0.0005
1AL_1AB_1	Zn	0.303	0.0023	1AL_1AB_2	Zn	0.3457	0.0012
1AL_1AB_1	Cu	0.0242	0.0003	1AL_1AB_2	Cu	0.0248	0.0001
1AL_1AB_1	Co	0.0311	0.0006	1AL_1AB_2	Co	0.0307	0.0001
1AL_1AB_1	Cr	0.0046	0.0001	1AL_1AB_2	Cr	0.005	0.0001
1AL_1AB_1	Al	18.53	0.1425	1AL_1AB_2	Al	19.02	0.0991
1AL_1AB_1	Sr	0.5022	0.0012	1AL_1AB_2	Sr	0.495	0.0027
1AL_1AB_1	V	0.0252	0.0001	1AL_1AB_2	V	0.0248	0.0002
1AL_1AB_1	P	0.623	0.0053	1AL_1AB_2	P	0.6099	0.0025
1AL_1AB_1	S	516	0.9716	1AL_1AB_2	S	516.4	4.202
1AL_1AB_1	Ca	64.86	0.9286	1AL_1AB_2	Ca	65.86	0.4618
1AL_1AB_1	Mg	28.49	0.0176	1AL_1AB_2	Mg	28.74	0.0187
1AL_1AB_1	K	89.93	0.0482	1AL_1AB_2	K	92.05	1.054
1AL_1AB_1	Na	107.1	0.0499	1AL_1AB_2	Na	107.2	0.05

Table 3 quality control

Name	Line	mg/L	SD	Pasp (mg/L)	%
Merck_X	Ba	0.0483	0.0002	0.05	97
Merck_X	Cd	0.0191	0.0001	0.02	96
Merck_X	Fe	0.102	0.0006	0.1	102
Merck_X	Mn	0.0316	0.0002	0.03	105
Merck_X	Ni	0.0478	0.0005	0.05	96
Merck_X	Pb	0.0252	0.0012	0.025	101
Merck_X	Zn	0.0536	0.0008	0.05	107
Merck_X	Cu	0.0214	0.0004	0.02	107
Merck_X	Co	0.023	0.0001	0.025	92
Merck_X	Cr	0.0201	0.0005	0.02	101
Merck_X	Sr	0.1128	0.0008	0.1	113
Merck_X	V	0.0496	0.0007	0.05	99
Merck_X	Ca	35.27	0.0172	35	101
Merck_X	Mg	15.55	0.063	15	104
Merck_X	K	2.614	0.0012	3	87
Merck_X	Na	6.951	0.0432	8	87

This following table shows the results obtained from the plant, root, algae and soil samples along with table 5 where the srm is shown.

Table 4. soil, plant and root samples

Samples	Weight in (g)	mg/kg	SD	mg/kg	SD	mg/kg	SD
		Cu		Cd		Pb	
MS2 2AL	1.8270	24.14	0.2087	0.273	0.009	17.799	0.136
MS3 2AL	1.8198	24.68	0.1275	0.175	0.005	13.756	0.137
V3 1AL-1Aα	0.8015	22.44	0.1476	0.083	0.008	4.707	0.235
V2 1AL-1Aβ	0.8004	26.53	0.1332	0.249	0.003	6.969	0.091
V P P.A.	8.8401	5.582	0.006	0.029	0.001	0.602	0.078
V P R	8.4142	8.101	0.056	0.060	0.002	1.938	0.038

Table 5 soil, plant and root samples "SRM".

Name	Element	mg/kg	sd	pasp	%
458	Cu	42.93	0.5252	48.1	89.3
458	Cd	0.45342067	0.00559767	0.49	92.5
458	Pb	32.7285917	0.26799069	35.5	92.2
m2	Cu	69.25	0.6079	68.1	101.7
m2	Cd	0.4274	0.0033	0.454	94.1
1547	Cu	3.457	0.0237	3.7	93.4
1547	Pb	0.8262	0.0282	0.87	95.0

With these results obtained from the ICP-OES process we have very relevant and important data.

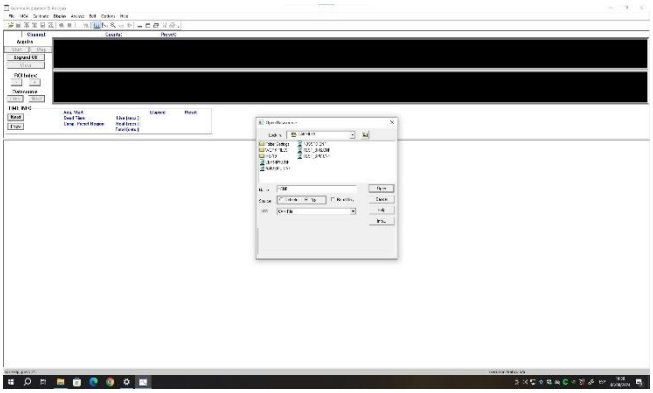
Introduction to neutron activation analysis.

The neutron activation analysis technique is based on the measurement of the radiation released by the decay of radioactive nuclei formed by neutron irradiation of the material. The most suitable neutron source for this application is usually a research reactor. Samples that can be analyzed with this method come from various fields, such as medicine, nutrition, biology, chemistry, forensic science, environment and mining.

Neutron activation analysis can be performed in various ways, depending on the element and the corresponding radiation levels to be measured, as well as the nature and degree of interference from other elements present in the sample. Most of the methods used are non-destructive and are based on the detection of gamma radiation emitted by the irradiated material after or during irradiation.

Getting acquainted with the Genie-2000 software used for gamma spectra processing.

During the analysis process it was necessary to use the Genie-2000 computer program to analyze the data resulting from the NAA. Below are screenshots of the program and a description of what was being done for the analysis of the data. samples.



This is the first step to start working with the program, where we must locate the file called GENIE2K and go to the CAMFILES folder and select the spectra.

Figure 8. "folders and file selection".

One next figure are presented all steps of spectra processing.

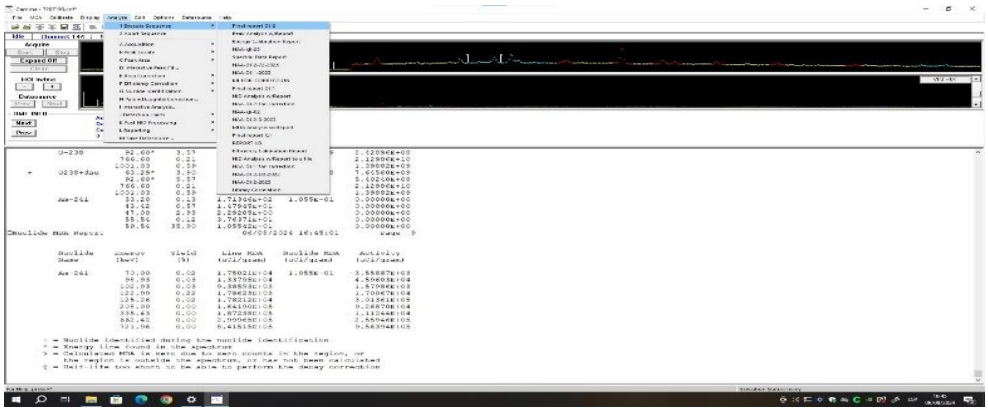


FIgure 9. "process selection".

We will use the alphabetical procedure where we will start with B "unidentified 2nd Drift which is figure 10 and C peak area 1 sum/ non-linear which is figure 11.

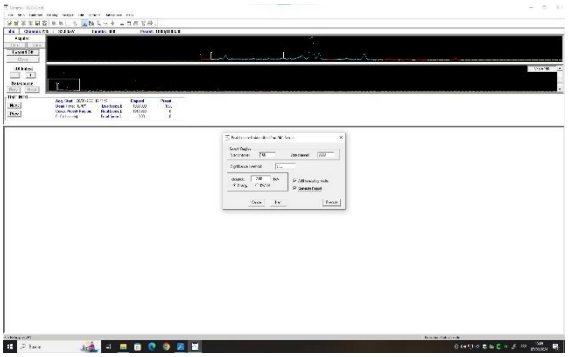


Figure 10 “B unidentified 2nd Drift”.

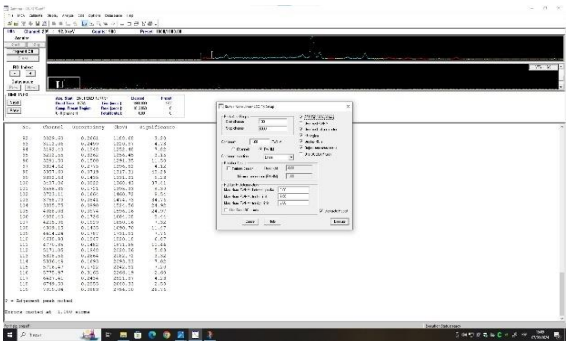


Figure 11”C peak area 1 sum/ non-linear”.

In the following figure you can see the interference process and energy graphs.

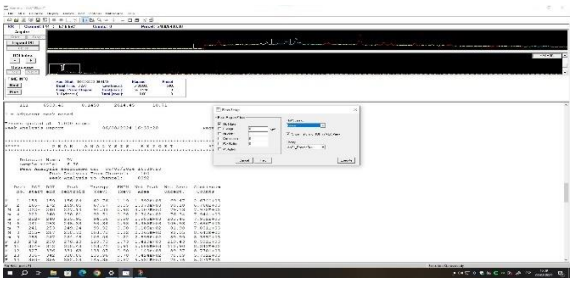


Figure 12 “ D interactive Peak”.

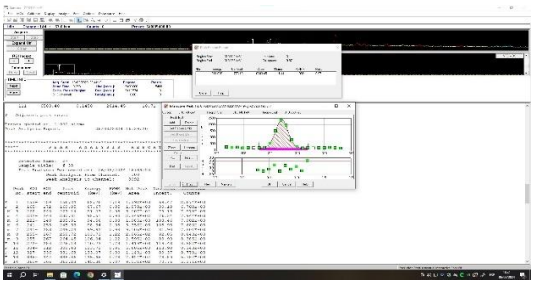


Figure 13.” D interactive Peak”.

Figures 12 and 13 show the error corrections of the peaks, below the generation of nuclide identification will be shown.

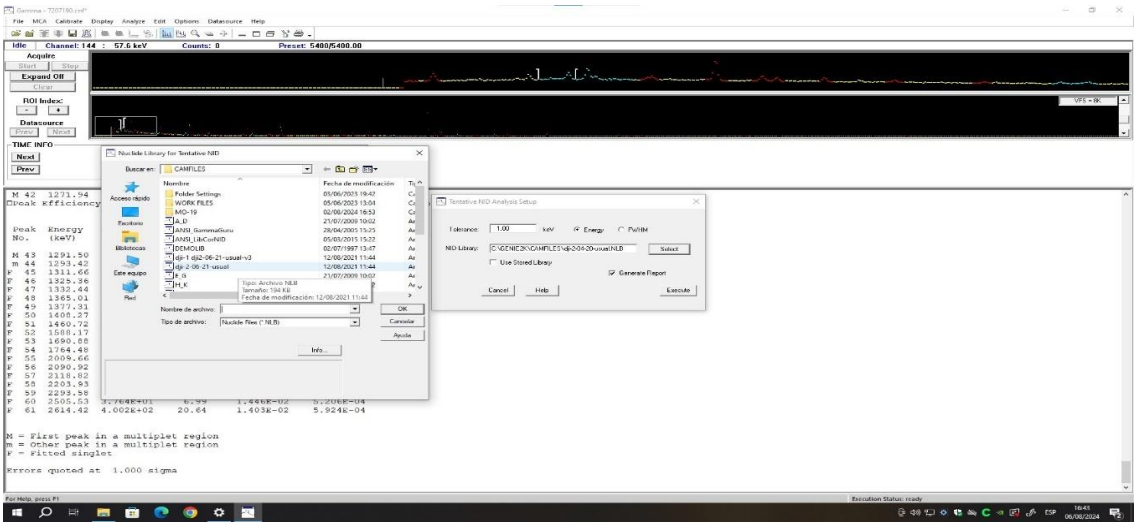


Figure 14 ” E Area correction”.

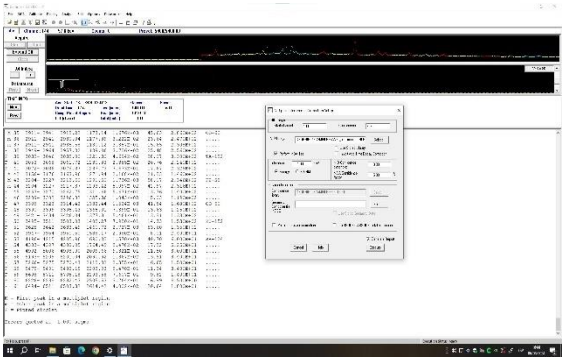


FIgure 15 “G Nucleide identification”.

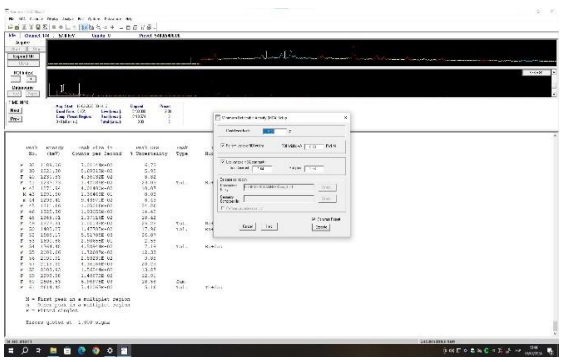


FIgure 16 “G Nucleide identification NID!”.

Finally, the MDA currie limit generation is used, shown in figure 17.

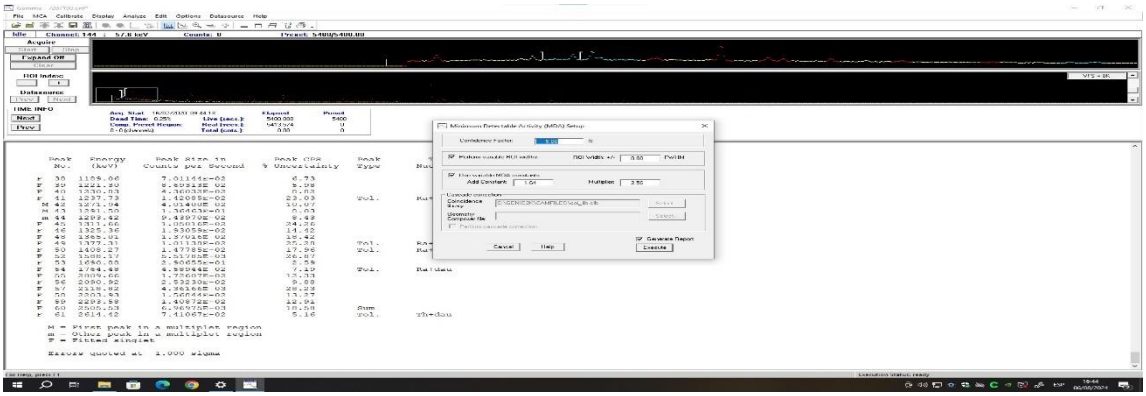


Figure 17 “J Detection Limits”.

At the end, a report of elements found is generated, like the one shown below.

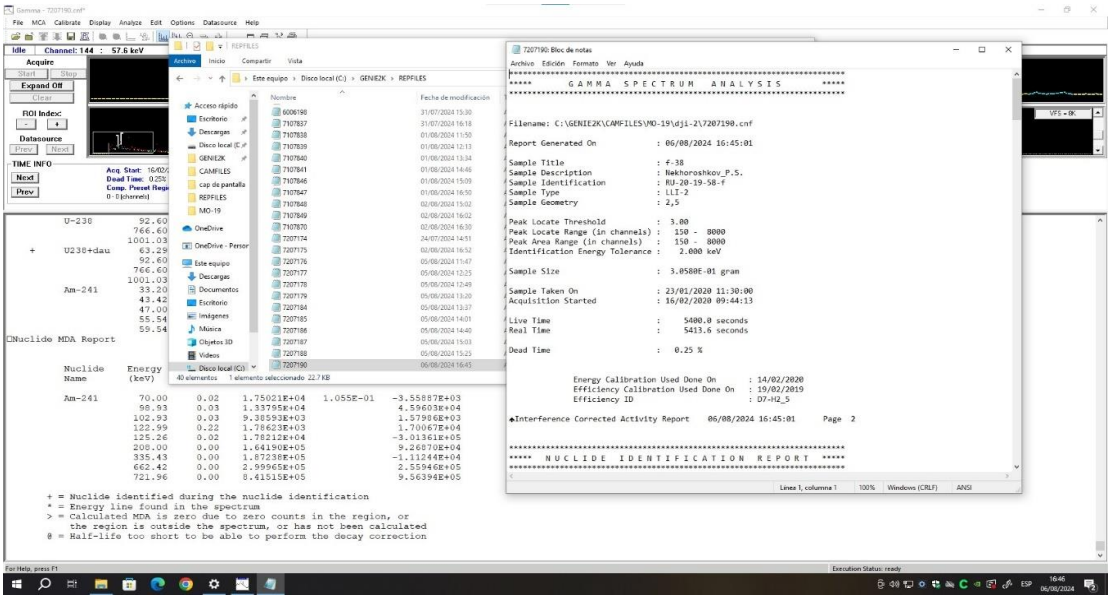


Figure 18 “Final report NAA”.

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Conclusion.

In this work it was shown which elements can be determined in samples from coastal waters to Volcanic fumaroles using ICP-OES, as well as that the NAA method can be used to determine elemental composition with much greater precision.

Acknowledgement.

for the JINR team and the START program as well as for my supervisor Dr. Inga Zinicovscaia and her colleagues for the great opportunity to learn new research methods and to be working alongside great scientists for high-level research, it has been a great honor to accompany you and to be able to live with you on this stay has been wonderful, just as you allowed me work and visit wonderful places within the JINR institute.

Dubna, 2024.