



# Analysis of geological samples using the Thermo Scientific iCAP 7000 Plus Series ICP-OES

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## Goal

To demonstrate accurate and precise analysis of geological samples via the Thermo Scientific iCAP 7400 ICP-OES Radial, presenting fusion as a suitable sample preparation technique.

## Introduction

The analysis of geological samples falls into two distinct groups each with different requirements<sup>1</sup>. Complete or “whole rock” analysis is used in both pure and applied research geology in the characterization of rock types. Traditionally, complete silicate analysis has been the concern of academically orientated studies. Sample numbers are usually limited while the emphasis is on the number of elements to be determined and the precision of results. The most important area of applied geochemistry is in the field of mineral exploration. In this application, samples of soil and rocks are systematically collected from large areas of land with a view to identifying small pockets or anomalies where the concentration of some elements is higher than the average. These high concentrations are usually indicative of mineral deposits, which sometimes have no surface evidence. The most important elements are the common ore elements such as Cu, Fe, Ni, Zn and Sn. In addition, other elements known as pathfinders are determined. The pathfinders are elements associated with the deposit which become more widely dispersed than the target elements making them easier to detect and less likely to be missed by the sampling strategy.

## Instrumentation

A Thermo Scientific™ iCAP™ 7400 ICP-OES Radial was selected for this analysis specifically to reduce matrix interferences. The instrument was configured with a concentric nebulizer and a cyclonic spray chamber (further instrument configuration details are shown in Table 1). The iCAP 7400 ICP-OES employs the powerful core technologies of the Thermo Scientific iCAP 7000 Plus Series ICP-OES to enable high-performance and high sample throughput capability together with application flexibility and low cost of ownership.

**Table 1. Instrument parameters.**

Parameter	Setting
Pump Tubing (Standard Pump)	Sample Tygon® orange/white Drain Tygon® white/white
Pump Speed	50 rpm
Nebulizer	Glass concentric
Nebulizer Gas Flow	0.55 L·min <sup>-1</sup>
Spray Chamber	Glass cyclonic
Auxiliary Gas Flow	0.5 L·min <sup>-1</sup>
Coolant Gas Flow	12 L·min <sup>-1</sup>
Center Tube	1.5 mm
RF Power	1150 W
Repeats	3
Exposure Time	UV 15 s, Vis 5 s

## Sample preparation<sup>2</sup>

A 0.25 g aliquot of finely ground sample was transferred to a clean platinum crucible. Using a polythene rod, the sample was mixed with 1.25 g of high purity lithium metaborate. The mixture was fused in a muffle furnace for 30 minutes at a temperature of 1000 °C (45 minutes for resistant samples). The crucible was swirled with the crucible tongs several times during the fusion and then allowed to slowly cool to room temperature after the allotted fusion time. It is recommended that the melt is allowed to gradually cool for a few minutes, just inside the furnace door, before completing the cooling process by placing crucible and melt outside the furnace door. This procedure helps to keep the silica in a soluble state in preparation for later dissolution.

100 mL of 5% nitric acid were added to the melt and this was placed on a magnetic stirrer for 1 – 2 hours or until dissolution was complete. It was ensured that the sample was not heated; this could cause the silica in the sample to be polymerized, rendering it insoluble. The dissolved sample was transferred to a 250 mL volumetric flask with deionized water and diluted to volume.

For the calibration, a reagent blank was prepared by dissolving 1.25 g of lithium metaborate in 250 mL 2% nitric acid – one reagent blank per standard. The standards were prepared from 1000 mg·kg<sup>-1</sup> single element stock solutions and matrix matched to the samples by diluting the stock standards with the reagent blank. Concentrations used in the analysis are presented in Table 2.

**Table 2. Standard concentrations in mg·kg<sup>-1</sup>.**

Element	Blank	Standard 1	Standard 2
Al	0	5	20
Ba	0	0.5	2
Ca	0	3	20
Cu	0	0.5	2
Fe	0	5	40
K	0	5	40
Mg	0	2	20
Mn	0	0.5	2
Ni	0	0.5	2
Si	0	20	100
Ti	0	2	5
Zn	0	0.5	2
Zr	0	0.5	2

## Method development and analysis

The plasma was ignited and a LabBook created in the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software, using default parameters (as shown in Table 1). A method detection limit (MDL) study was carried out by analyzing the calibration blank with ten replicates and multiplying the standard deviation of this analysis by three. This was repeated three times and the average values for detection limits were calculated. All element subarrays were checked for any necessary background corrections or spectral interferences.

Calibration was carried out using the working standards. Utilizing the advanced statistical capabilities of the Qtegra ISDS Software, the calibration curve was checked to ensure an accurate fit. Dilutions were used as needed and were made up to volume with a sample blank solution. Following samples were used for analysis:

Sample 1: SARM Geostandard AC-E

Sample 2: SARM Geostandard GS-N

Sample 3: US Geological Survey Marine Sediment MAG-1

The Standard Reference Materials (SRM) or their alternatives are available from the named research institution and from MBH Analytical Ltd., Holland House, Queens Road, Barnet, EN5 4DJ, UK.

## Results

The final results of the sample analysis are shown in Table 3. For most of the analytes and reference materials, the recoveries of the measured concentrations were within  $\pm 10\%$  of the expected value. In SRM AC-E the recovery of Mg and Ti was  $<90\%$ , however concentrations were inside the expected range. Zn had a recovery of 83% in SRM GSN, meaning this analyte may be investigated further to determine the reason for low recoveries.

**Table 3. Measured and expected concentrations as well as calculated recoveries. All results are presented as percentages unless otherwise stated.**

Element and wavelength (nm)	AC-E Measured	AC-E Expected	Recovery (%)	GSN Measured	GSN Expected	Recovery (%)	MAG-1 Measured	MAG-1 Expected	Recovery (%)
Al 237.312	7.42	7.78 $\pm$ 0.03	95.4	7.41	7.76 $\pm$ 0.16	95.5	8.43	8.68 $\pm$ 0.16	97.1
Ba 493.409	52 mg $\cdot$ kg $^{-1}$	55 $\pm$ 2.6 mg $\cdot$ kg $^{-1}$	94.5	1436 mg $\cdot$ kg $^{-1}$	1400 $\pm$ 44 mg $\cdot$ kg $^{-1}$	102.6	461 mg $\cdot$ kg $^{-1}$	480 $\pm$ 41 mg $\cdot$ kg $^{-1}$	96.0
Ca 373.690	0.22	0.24 $\pm$ 0.014	91.7	1.72	1.79 $\pm$ 0.04	96.1	0.96	0.98 $\pm$ 0.07	98.0
Cu 327.396	4 mg $\cdot$ kg $^{-1}$	4 $\pm$ 0.9 mg $\cdot$ kg $^{-1}$	100.0	20 mg $\cdot$ kg $^{-1}$	20 $\pm$ 0.7% mg $\cdot$ kg $^{-1}$	100.0	31 mg $\cdot$ kg $^{-1}$	30 $\pm$ 3 mg $\cdot$ kg $^{-1}$	103.3
Fe 239.562	1.65	1.77 $\pm$ 0.014	93.2	2.49	2.62 $\pm$ 0.03	95.0	4.77	4.76 $\pm$ 0.42	100.2
K 766.490	3.40	3.73 $\pm$ 0.02	91.2	3.53	3.82 $\pm$ 0.05	92.4	2.78	2.95 $\pm$ 0.14	94.2
Mg 280.270	0.013	0.02 $\pm$ 0.006	65.0	1.33	1.39 $\pm$ 0.03	95.7	1.80	1.81 $\pm$ 0.06	99.4
Mn 257.610	436 mg $\cdot$ kg $^{-1}$	449 $\pm$ 15.5 mg $\cdot$ kg $^{-1}$	97.1	410 mg $\cdot$ kg $^{-1}$	434 $\pm$ 31 mg $\cdot$ kg $^{-1}$	94.5	734 mg $\cdot$ kg $^{-1}$	759 $\pm$ 70 mg $\cdot$ kg $^{-1}$	96.7
Ni 231.604	N/A	N/A	N/A	33 mg $\cdot$ kg $^{-1}$	34 $\pm$ 4 mg $\cdot$ kg $^{-1}$	97.1	49 mg $\cdot$ kg $^{-1}$	53 $\pm$ 8 mg $\cdot$ kg $^{-1}$	92.5
Si 198.898	31.83	32.88 $\pm$ 0.03	96.8	29.67	30.76 $\pm$ 0.09	96.5	23.2	23.56 $\pm$ 0.45	98.5
Ti 190.820	0.06	0.07 $\pm$ 0.02	85.7	0.40	0.41 $\pm$ 0.02	97.6	0.44	0.45 $\pm$ 0.04	97.8
Zn 206.200	207 mg $\cdot$ kg $^{-1}$	224 $\pm$ 6 mg $\cdot$ kg $^{-1}$	92.4	40 mg $\cdot$ kg $^{-1}$	48 $\pm$ 3.3 mg $\cdot$ kg $^{-1}$	83.3	118 mg $\cdot$ kg $^{-1}$	130 $\pm$ 6 mg $\cdot$ kg $^{-1}$	90.8
Zr 343.823	781 mg $\cdot$ kg $^{-1}$	780 $\pm$ 20 mg $\cdot$ kg $^{-1}$	100.1	234 mg $\cdot$ kg $^{-1}$	235 $\pm$ 15 mg $\cdot$ kg $^{-1}$	99.6	127 mg $\cdot$ kg $^{-1}$	130 $\pm$ 13 mg $\cdot$ kg $^{-1}$	97.7

N/A: No analytic results available.

Method detection limits calculated from the lithium metabolite blank were sufficiently low to determine the expected concentrations (see Table 4).

**Table 4. Method detection limits (MDL) of the analyzed wavelengths.**

Element and wavelength (nm)	MDL ( $\mu$ g $\cdot$ kg $^{-1}$ )
Al 237.312	6.7
Ba 493.409	0.1
Ca 373.690	3.7
Cu 327.396	0.9
Fe 239.562	3.4
K 766.490	13.3
Mg 280.270	0.3
Mn 257.610	0.2
Ni 231.604	1.6
Si 198.898	19.6
Ti 190.820	1
Zn 206.200	0.4
Zr 343.823	0.8

## Conclusion

The described method shows that the Thermo Scientific iCAP 7000 Plus Series ICP-OES enables a precise and accurate determination of major, minor and trace elements. Sample preparation is straightforward involving fusion, acid dissolution of the melt and low detection limits. The high-resolution spectrometer ensures freedom from spectral interferences caused by the sample matrix. The use of the high performance concentric nebulizer and cyclonic spray chamber combined with the high-resolution optics ensures excellent accuracy and low detection limits.

The high-performance Charge Injection Device (CID) detector has inherent non-blooming characteristics, which enable analysis of high concentration major elements alongside trace elements without any interferences.

## References

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2. A Handbook of Decomposition Methods in Analytical Chemistry, Bock R, translated by Iain Marr, Blackie Group 1979, ISBN 0 7002 0269 2.

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