Leveraging an AI/ML based approach for the semiquantitative analysis of geological samples using ICP-OES

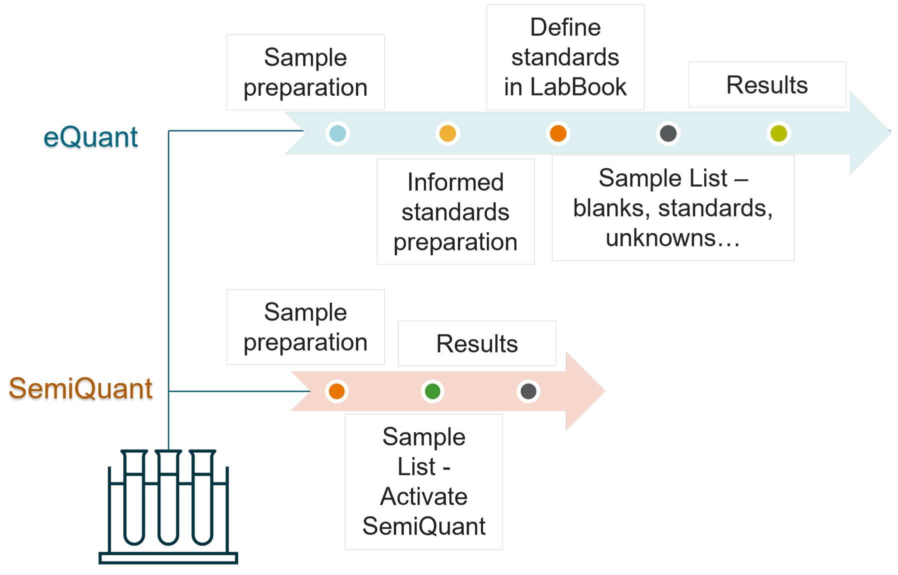
S. Sengupta[1]*, T. Stichel[2], D. Kutscher[1]
[1]Thermo Fisher Scientific, Bremen, Germany, [2]Thermo Fisher Scientific, Dreieich, Germany
*sukanya.sengupta@thermofisher.com

Abstract

Purpose: The aim of this study is to compare the effectiveness of semiquantitative analysis of geological samples using an innovative AI/ML-based approach with inductively coupled plasma optical emission spectroscopy (ICP-OES) and its comparison to fully quantitative results. The study focuses on screening geological samples for contamination and multielement data acquisition without the need for calibration standards.

Methods: The study involves analyzing geological and environmental samples, including rock digests, seawater etc. using ICP-OES and checking for accuracy.

Results: The results demonstrates that the AI/ML-based semiquantitative approach provides rapid and reliable multielement data for geological samples, showing minimal discrepancies when compared to the traditional fully quantitative method. This suggests that the semiquantitative approach can significantly speed up the process of finding suitable sample preparation methods and can be effectively used in various geological applications.



Introduction

Calibrating ICP-OES instruments for the analysis of unknown samples using suitable calibration solutions in appropriate concentration ranges can be challenging as well as time and effort intensive, specially when dealing with complex matrices such as ones typically encountered in geological samples. It can be extremely beneficial and time saving to conduct a rapid scan of unknown samples before further analysis and method development or to be able to perform a pre-screening step to ensure that only contamination-free solutions are introduced into high precision and high sensitivity instrumentation leading to best chances at accurate data. The Thermo Scientific™ iCAP™ PRO Series ICP-OES and the Thermo Scientific™ Qtegra™ ISDS Software addresses this challenge with a valuable and simple solution – SemiQuant – a novel AI/ML based approach to semiquantitative assessment and detection of a wide range of elements in unknown sample types without the need to prepare any calibration solutions[1].

Materials and methods

Samples

Three rock and one soil sample (Table 1) were acid-digested (50 mg sample size) as described elsewhere[2] and finally recovered in 2% HNO_3 (final volume 25 ml) before analysis with ICP-OES. As it is not required for SemiQuant, no calibration solutions were prepared for the semiquantitative analysis. Preliminary standards were prepared for fully quantitative (eQuant) analysis using external calibration.

For confirming accuracy via a spike recovery test, a NASS-6: Seawater reference material for trace metals from National Research Council of Canada was diluted 10X and then spiked with 0.1 $mg \cdot L^{-1}$ of a multielement standard.

Experimental methods

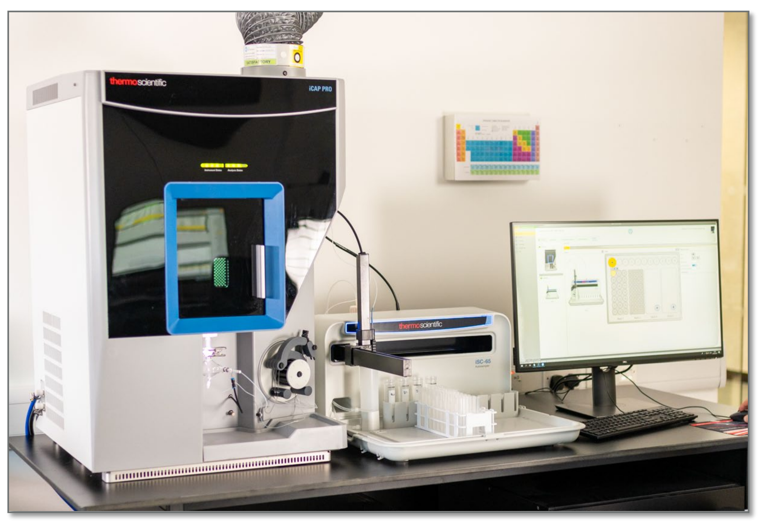
A Thermo Scientific™ iCAP™ PRO XP ICP OES Duo and a Thermo Scientific™ iSC-65 autosampler were used for analysis using SemiQuant and subsequently eQuant, i.e., the classical full quantification approach. The default tune set for aqueous samples was used for both evaluations (Table 2). The intelligent Full Range (iFR) mode enabled simultaneous data acquisition for eQuant between 167 – 852 nm (Figure 1) in both Axial and Radial views. For SemiQuant, the Qtegra ISDS software automatically selected predefined tune parameters.

Software

The SemiQuant data acquisition feature in Qtegra ISDS software utilizes artificial intelligence and machine learning to automatically choose the optimal wavelengths—free from interferences—to estimate the approximate concentrations of a wide range of elements in the sample (Figure 2).

Table 1. List of rock and soil samples

Sample name	Description
Sample 1- SDO-1	Black shale rich in organic matter[3] Certified reference material
Sample 2 - CAST	Deep sea carbonate[4] In-house reference material
Sample 3 - BE-N	Basalt rich in Fe, Ca, Mg[3] Certified reference material
Sample 4 - LOESS	Soil sample resembling the upper crust[2] In-house reference material



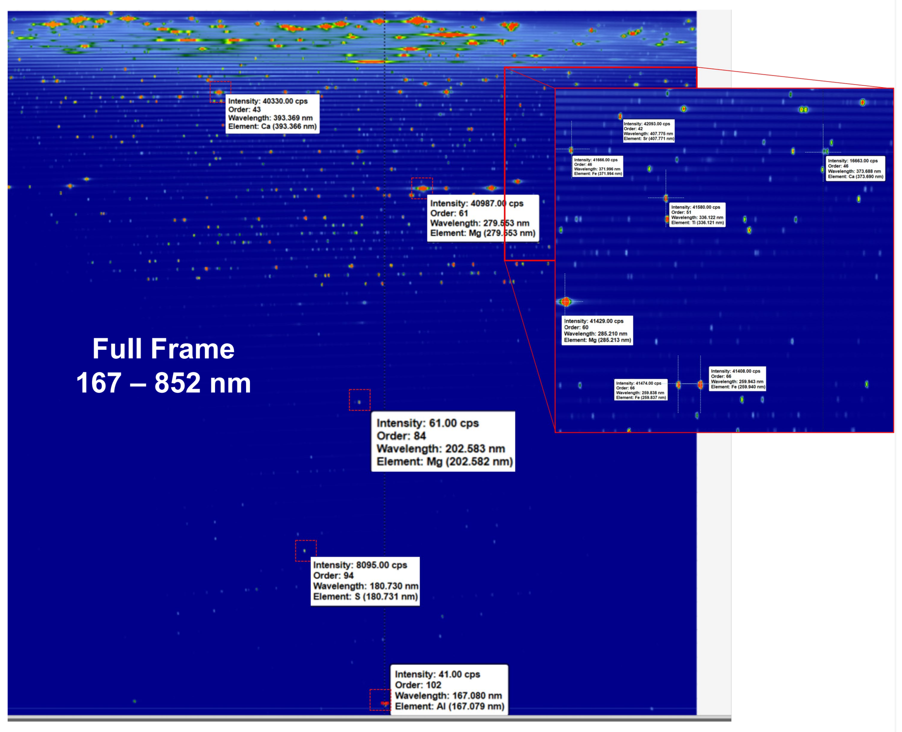


Figure 1. A Full Frame image of an unknown sample viewing the full spectrum between 167 – 852 nm simultaneously in a single exposure using an iCAP PRO XP ICP-OES

Table 2. Instrument parameters

Instrument parameter	Setting
Spray chamber	Glass cyclonic
Nebulizer	Glass concentric nebulizer
Center tube	2.0 mm, quartz
Torch	EMT glass torch
Pump tubing	Sample: Tygon™ orange/white; Internal Standard: Tygon™ orange/blue; Drain: Tygon™ white/white
Pump speed	45 rpm for data acquisition 125 rpm for Fast Uptake
Nebulizer gas flow	0.5 $L \cdot min^{-1}$
Auxiliary gas flow	0.5 $L \cdot min^{-1}$
Coolant gas flow	12.5 $L \cdot min^{-1}$
RF Power	1150 W
Autosampler	iSC-65 autosampler

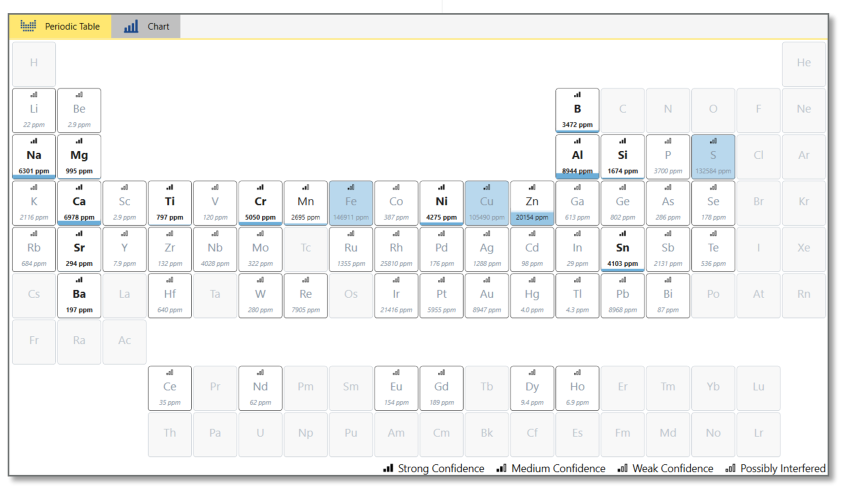


Figure 2. One of the several different data visualization options for SemiQuant available within the Qtegra ISDS software. In the example above, a pooled sample with different combination of elements was chosen for demonstration purposes.

Results

Water samples

Comparing the SemiQuant results to expected concentrations and those from the eQuant results, as shown in Figure 3, 23 elements exhibited recovery within the ±20% accuracy range. Typically, SemiQuant accuracy is expected to be within ±30%, which was observed for 9 elements. The AI/ML model clearly pointed out which elements were confidently identified, with indications for different confidence levels, and which were very likely interfered upon (Figure 3). These findings indicate a high likelihood of obtaining nearly accurate data even without using calibration solutions.

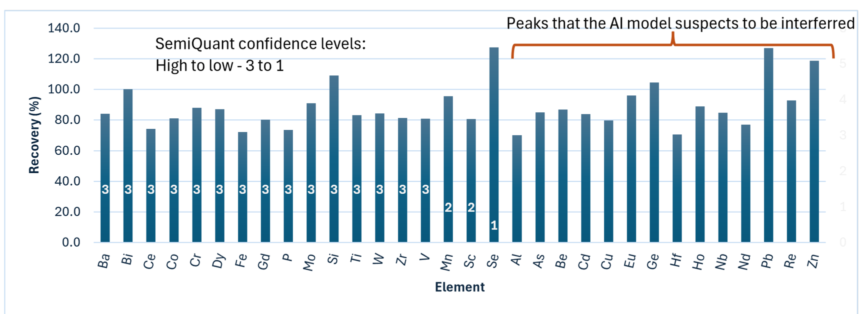


Figure 3. Results of spike recovery estimates from SemiQuant compared to expected values. Even for elements with interfered peaks several estimates were in an excellent accuracy range, thus demonstrating the advantages of an AI/ML approach of parallelly assessing multiple wavelengths and peaks per analyte.

Rock/soil samples

The accuracy of the SemiQuant data was further assessed by measuring four different rock and soil sample digests, each distinctly different from the other (sample descriptions in Table 1), and amongst all four forming a good representation of different geological sample types and comparing the SemiQuant data to concentrations obtained by the X-ray fluorescence (XRF) technique from previous studies[2]. This data is presented in Table 3 and illustrated in Figure 4.

The results from the fully quantified XRF and the semiquantitative approach in this study show excellent comparability. Several elements, such as Ca in all four samples, and Sr and V in SDO-1 and Loess, demonstrated exceptional accuracy within the ±10-15% range. Overall, most elements fell within the typically expected ±30% range, with poorer recoveries observed for elements with interferences or very high concentrations. Additionally, SemiQuant data compared to eQuant data from this study showed good agreement with similar accuracy levels. These results suggest that the SemiQuant software tool provides a reliable overview of element presence and concentration in complex matrices like rock and soil digests, without the need for calibration solutions.

Table 2. Elemental concentrations in rock and soil sample digests were determined using XRF (from a different study) and SemiQuant, with the accuracy of SemiQuant expressed as recovery against XRF concentrations. Elements for which SemiQuant data is missing were highly interfered and can be resolved using methods such as Inter Element Correction (IEC), which is also available in the Qtegra ISDS Software[5].

Elements	Concentrations ($mg \cdot kg^{-1}$)		Recovery SemiQuant vs XRF (%)	Concentrations ($mg \cdot kg^{-1}$)		Recovery SemiQuant vs XRF (%)
	XRF	SemiQuant		XRF	SemiQuant	
	Sample 1 - SDO-1			Sample 2 - CAST		
Ti	4256	3400	80	1197	1147	96
Al	61921	55313	89	18393	12608	69
Fe	61342	37652	61*	12364	7888	64*
Mn	325	259	80	462	-	-
Mg	9287	-		10066	7102	71*
Ca	7504	6601	88	292956	256703	88
Na	2671	3356	126	11719	12510	107
K	27810	23075	83	5649	-	-
P	567	424	75	423	491	116
S	53500	53744	100	-	-	-
Cr	66.4	57.1	86	45.0	38.9	86
Cu	60.2	45.7	76	53.0	45.7	86
Mo	134	129.3	96	-	5.7	-
Ni	100	73.2	73	40.0	33.3	83
Sr	75.1	74.1	99	1291	984.0	76
V	160	146.4	92	37	32.0	86
Y	40.6	28.3	70	14.0	9.5	68
	Sample 3 - BE-N			Sample 4 - Loess		
Ti	15647	13397	86	3982	3458	87
Al	53295	37674	71	41810	31773	76
Fe	89529	49860	56*	18326	11673	64*
Mn	116	-	-	410	313	76
Mg	79604	53854	68*	8553	6751	79
Ca	99342	91475	92	44506	43657	98
Na	23591	22073	94	8160	9267	114
K	11539	14754	128	16112	14495	90
P	4582	4281	93	516	422	82
S	300	311	104	-	-	-
Cr	360	305.3	85	73.8	67.2	91
Cu	72.0	68.4	95	4.0	-	-
Ni	267	175.3	66	21.3	16.0	75
Sr	1370	1065.5	78	164	153.5	94
V	235	206.4	88	50.2	47.7	95
Y	24.7	19.7	80	29.3	22.0	75

*Low recoveries associated with very high concentrations of respective matrix elements in the samples. As next steps, diluted aliquots will be measured where better recoveries are expected.

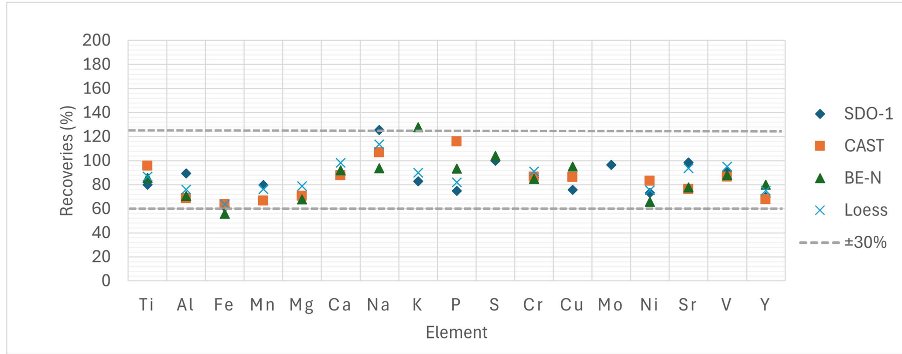


Figure 4. SemiQuant measurements of geological samples in present study compared to XRF analysis from previous studies to demonstrate accuracy of the semiquantitative analysis. Low recoveries <70% are linked to high concentrations of the respective matrix elements e.g., up to 9% Fe

Conclusions

The study demonstrates that the semiquantitative analysis or SemiQuant feature of the Qtegra ISDS Software is an effective tool for screening samples for a wide range of elements, even in challenging matrices commonly encountered in geological samples, with excellent data comparability to full quantification methods both within Qtegra (i.e., eQuant) as well as with data from completely independent techniques such as XRF. Accurate concentration estimates for several elements within unknown samples are achieved automatically using a novel AI/ML approach that requires no calibration.

Some key highlights of the study are as follows:

- The accuracy of the SemiQuant analyses was validated through a spike recovery test on a seawater reference material and by measuring rock and soil digests with known concentrations from previous XRF measurements.
- The SemiQuant, XRF, and eQuant data showed very good agreement, demonstrating the accuracy of the semiquantitative approach compared to traditional quantification techniques.
- SemiQuant results are achieved solely using the iCAP PRO Series ICP-OES instrumentation and the advanced features of the Qtegra ISDS software, eliminating the need for calibration solutions. This approach accelerates workflows, saving analysts and laboratories valuable time, effort, and costs.

Acknowledgements

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