

# Method Validation of Minerals Determination in Argan Pulp (*Argania spinosa* L.) Using Inductively Coupled Plasma Optical Emission Spectrometry and Dry Ashing

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**Abstract:** Argan is a fruit tree endemic to Morocco; a huge amount of argan by-products is generated during argan oil processing, including argan pulp and argan oil cake. These by-products are usually used as animal feed and have more recently found applications in the pharmaceutical field. This study focuses on a method for determining four mineral elements (potassium, calcium, sodium, and magnesium) in argan pulp. The method employs inductively coupled plasma optical emission spectrometry and dry ashing, which has been validated according to NF90-210 and NF V03-110 standards. The linearity, quantification limit, and accuracy profile were carefully investigated. The results demonstrate excellent linearity within the tested concentration range for all minerals. The accuracy of the presupposed limit of quantification was successfully verified. Satisfactory recoveries were obtained (between 88,70% and 101,80%), and the accuracy profiles show that all the tolerance intervals are included in the acceptability intervals. These findings demonstrate that the method can provide sufficiently accurate values for all the studied minerals.

**Keywords:** accuracy; linearity; method validation; quantification limit.

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## 1. Introduction

Minerals play an important role in the functioning of the body and are crucial for maintaining good human and animal health, as they are involved in several complicated metabolic and physiologic processes [1–5]. Potassium K, Calcium Ca, Magnesium Mg, and Sodium Na are known as macronutrients and are usually found in living beings at high levels [6]. For instance, potassium deficiency is linked with hypertension and cardiovascular diseases and is considered the most abundant cation in intracellular fluids [7]. Calcium is involved in a variety of functions, including regulating intracellular and extracellular fluid homeostasis, maintaining a healthy skeletal system, and preserving the structure of cell organelles [8]. Magnesium is the second most prevalent intracellular cation and is known to affect muscular relaxation positively and may enhance muscle function [9]. Sodium is the most prevalent cation in extracellular fluid, and both Na and K are essential for preserving cellular homeostasis [10]. Despite their important roles in the proper functioning of the human body, these minerals are only required in well-defined levels and should be provided

in well-known concentrations [1, 11]. Inadequate intake of minerals and trace elements might affect the immune system and eventually cause clinical symptoms [12]; therefore, sufficient consumption is necessary to maintain a strong immune system [12]. Furthermore, an excess of a single element has the potential to adversely impact the accessibility of other elements, leading to potentially harmful consequences for human health [6]. As a result, mineral composition should be controlled throughout the entire production process of foods and animal feed, and a powerful and reliable tool is needed for this purpose [13, 14].

Argan tree, *Argania spinosa* (L.) Skeels is an endemic tree of Morocco [15]. It is also cultivated in many countries, including South Africa, Israel, Kuwait, Mexico, Spain, and Tunisia [16,17]. In Morocco, this tree covers an area of 800,000 hectares, with a production rate of about 4000 tons of oil per year [17–19]. This makes argan oil (AO) a quintessential Moroccan product.

Argan oil by-products find multiple uses as feedstock for local producers and are also incorporated into many pharmaceutical and food products [20–25]. Argan pulp is one of the argan oil by-products; it corresponds to the outer cover of the argan fruit. Traditionally, it has been used as an animal feed supplement, but recently, several new emerging uses have appeared [26–28]. Consequently, there is a growing demand to control the mineral composition of these products, requiring a powerful analytical tool. In order to enhance the value of by-products in different fields.

This study aims to validate a method for mineralization and determination of four minerals (K, Ca, Na, and Mg) in argan pulp using an inductively coupled plasma optical emission spectrometer (ICP-OES), in accordance with the French Standard NF90-210 and NF V03-110. The validation process includes the study of the calibration function, the limit of quantification, and the accuracy profile. This standard, initially designed to validate physico-chemical analytical methods in water, has broader applications in diverse fields, such as cosmetics, agriculture, and food.

## 2. Materials and Methods

### 2.1. Sampling.

The samples of argan pulp collected in 2019 were provided by Taitmatine Cooperative in Taroudant. The collected samples were dried in the oven at 80°C for 24 h, then crushed and sieved before analysis.

### 2.2. Standards and reagents.

To calibrate each of the assessed elements (Ca, Na, K, and Mg), stock solutions containing standard concentrations of 1000 µg/mL dissolved in 2% weight HNO<sub>3</sub> (CertiPUR, Darmstadt, Germany) were utilized. Stock solutions were purchased from Merck Millipore and refrigerated at 4°C until use. GBW 07605 was used as the certified reference material. Argon alpha-gas (purity higher than 99.995) supplied by Air Liquide was used in the plasma. The acids used (Nitric and hydrochloric) were of reagent grade. Ultrapure water with a maximum resistivity of 18.2 MΩ/cm was produced using a Milli-Q Millipore system (Darmstadt, Germany) for solution preparation.

### 2.3. Apparatus.

An inductively coupled plasma optical emission spectrometry (ICP-OES) was used for mineral determination (Perkin Elmer Model Optima 8000 DV, Waltham, USA), equipped with an autosampler ASX-520 supplied by Teledyne CETAC Technologies (Omaha, USA), and a charge-coupled device (CCD) detector was used for minerals determination. A Gem Tip, Cross-Flow II nebulizer coupled with the Scott chamber was used as a sample introduction system. Mineralization was carried out using a muffle furnace Nabertherm model (Cologne, Germany).

### 2.4. Preparation of the samples and quantification of minerals (K, Ca, Na, Mg).

The dry ashing strategy was adopted for sample mineralization. Indeed, one gram of sample powder was weighed into a porcelain crucible and then calcinated in a muffle furnace for 2h at 500°C. After cooling, 10 drops of water and 4 mL of HNO<sub>3</sub> (65%) were added. The nitric acid excess was evaporated on a hot plate at 120°C, and the sample was heated for 1h at 500°C. Finally, the ashes were dissolved in 10 mL 20% HCl (v/v), resulting in a clear solution. The operating conditions of the ICP-OES measurements are shown in Table 1.

**Table 1.** Operating conditions of ICP OES during minerals determination.

Condition	Description/ value	Parameter	Description/ value
Plasma gas flow rate	14 L.min <sup>-1</sup>	Sample flow rate	1.3 mL.min <sup>-1</sup>
Auxiliary gas flow rate	0.2 L.min <sup>-1</sup>	Time flush	7s
Nebulizer gas flow rate	0.8 L.min <sup>-1</sup>	Analysis mode	Axial
Plasma gas	Argon	RF power	1300 W

The wavelengths used for the quantification were as follows: K (766.490 nm), Mg (285.213 nm), Na (589.592 nm), and Ca (317.933 nm). The accuracy of the measurements was established using standard samples.

### 2.5. Validation study.

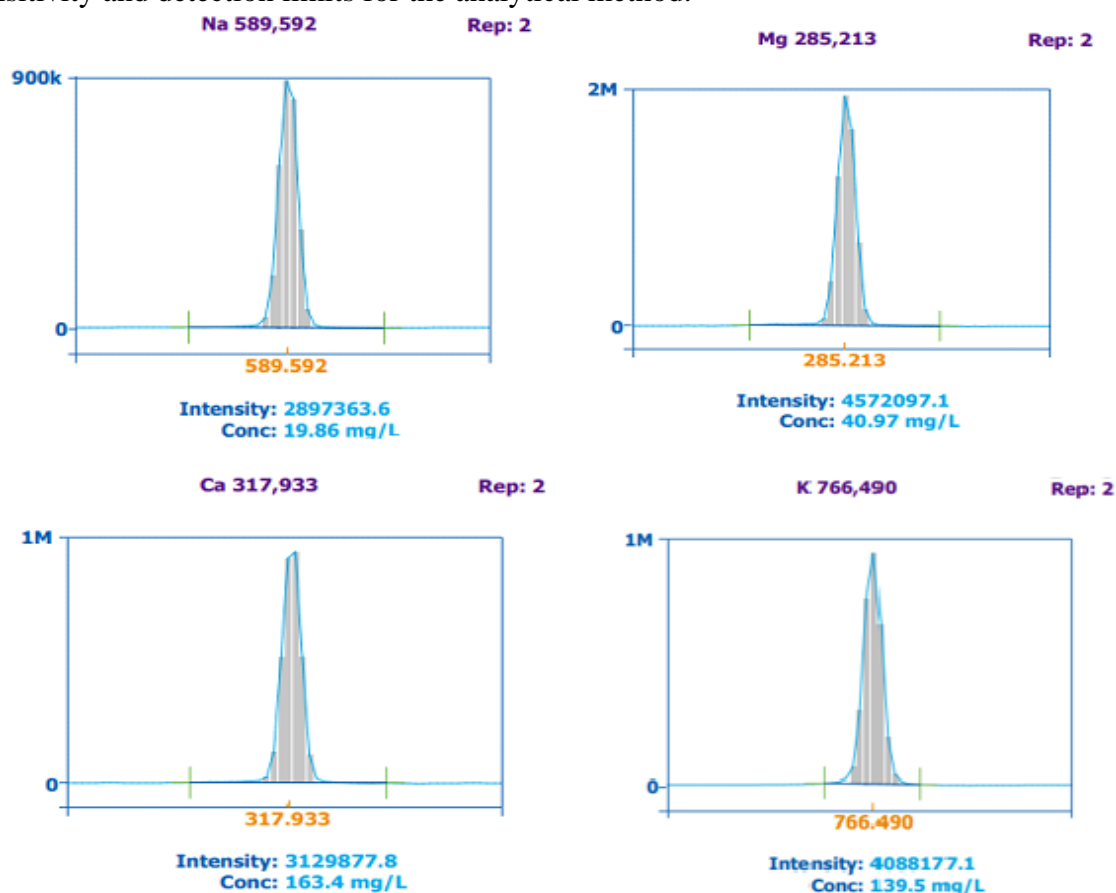
The method was validated for determining four minerals (Ca, K, Mg, and Na) following the standards NF T90-210 and NF V03-110. During the proposed method's validation process, linearity, quantification limits (LOQ), and accuracy profile were evaluated. Five independent standards are prepared for 5 days (p=5) for linearity validation under repeatability conditions. The quantification limit was validated by analyzing 5 samples; each sample was analyzed twice under repeatability conditions. An accuracy profile was performed according to NF V03-110. The validated method was used to analyze the certified reference material GBW 07605.

## 3. Results and Discussion

In order to achieve the best results and a good price-quality relationship, the parameters of the method used were optimized. Linearity was assessed over a specific concentration range, and the limit of quantification was determined. Additionally, accuracy profile studies were conducted to ensure the reliability of the results.

### 3.1. Method optimization.

It is known that each atom presents one or several emission lines [29, 30]. In order to choose the best emission wavelength for the investigated elements, a spectral scan was performed, following the manufacturer's recommendations. The scan aimed to identify the least interfered lines and select those with the highest intensity values for mineral identification (Figure 1). The selection of lines with the highest intensity ensures better sensitivity and detection limits for the analytical method.



**Figure 1.** Emission lines of the investigated minerals Na, K, Ca, and Mg.

### 3.2. Method validation.

#### 3.2.1. Linearity.

Linearity was validated using five concentration levels of the investigated minerals and analyzed on five different days. The concentration levels were prepared as follows: (2.5, 5, 10, 20, 50 ppm) for Ca, K, and Mg, and (1, 2.5, 5, 10, and 20 ppm) for Na. The obtained calibration curves exhibited acceptable correlation coefficients ( $R^2$ ) ranging from 0,98108 and 0,99674, all higher than 0.98, indicating good linearity over the examined range for all analytes. At the same time, the correlation coefficient ( $R^2$ ) is commonly cited as an indicator of linearity in method validation [31]. Unfortunately, this affine function coefficient property is insufficient to demonstrate that the model fits the experimental data [32]. Consequently, statistical tests and other indicators are required [33].

In order to check the linearity, the COCHRAN Test was applied to confirm the homogeneity of variances. The test was confirmed for all the studied minerals (C test < C critical), Table 2. Additionally, the adequacy test was applied to check the regression model. The obtained values for the F test were lower than the F critical values; this confirms that the

error of the model is significantly negligible compared to the observed experimental error. Consequently, the calibration function was validated for the domain studied with a risk of 1%.

**Table 2.** Calibration curve equations, homogeneity of variances, and adequacy test of the studied minerals.

Element	Equation	R <sup>2</sup>	Homogeneity of variances		Adequation test	
			C Test	C Critical	F Test	F Critical
Ca	$y = 5731102.15054x + 123991.66667$	R <sup>2</sup> = 0.99197	0.586	0.633	0.47	4.94
K	$y = 21043037.63441x + 196558.33333$	R <sup>2</sup> = 0.98748	0.530	0.633	2.17	4.94
Mg	$y = 9214444.44444x + 175077.77778$	R <sup>2</sup> = 0.98108	0.592	0.633	2.07	4.94
Na	$y = 24530304.65950x + 314427.77778$	R <sup>2</sup> = 0.99674	0.573	0.633	1.13	4.94

### 3.2.2. Limit of quantification (LQ).

A sample with a known concentration corresponding to the presupposed limit of quantification was analyzed for five days, with two repetitions each day Table 3. According to NF T 90-210, LQ validation ensures the accuracy of the presupposed limit of quantification with respect to a maximum acceptable deviation of 60% of the LOQ by verifying the following two inequalities [34].

$$zLQ - 2 \times sLQ > LQ - 60\% \times LQ \quad (1)$$

$$zLQ + 2 \times sLQ > LQ - 60\% \times LQ \quad (2)$$

**Table 3.** Accuracy parameters of the presupposed limit of quantification for the four elements.

Element	K	Ca	Mg	Na
Number of series: n	5	5	5	5
Number of reps per set: r	2	2	2	2
Repeatability variance: $s_{rep}^2$	0.00700	0.00004	0.00009	0.00042
Variance of means: $s(z_i)^2$	0.00325	0.00013	0.00010	0.00023
Inter-series variance: $s_b^2$	0.00000	0.00011	0.00005	0.00002
Variance of intermediate fidelity: $s_{lq}^2$	0.00700	0.00015	0.00014	0.00044
Overall average: $z_{lq}$	1.13000	0.50000	0.50300	0.25200
Intermediate fidelity Standard deviation: SQL	0.08367	0.01204	0.01183	0.02104
Intermediate fidelity CV%: $CV_{lq}$	0.07404	0.02408	0.02352	0.08347
Reference value: ref	1.00000	0.50000	0.50000	0.25000
Maximum acceptable deviation: MAD	0.60000	0.30000	0.30000	0.15000
$lq + 60\% lq$	1.60000	0.80000	0.80000	0.40000
$z_{lq} + 2 s_{lq}$	1.29733	0.52408	0.52666	0.29407
$z_{lq} - 2 s_{lq}$	0.96267	0.47592	0.47934	0.20993
$lq - 60\% lq$	0.40000	0.20000	0.20000	0.10000
The accuracy of the limit	Verified	Verified	Verified	Verified

The obtained data shows that the two inequalities  $ZLQ - 2 \times SLQ > LQ - 60\% \times LQ$  and  $ZLQ + 2 \times SLQ < LQ + 60\% \times LQ$  were verified for the four elements studied, which means that the accuracy of the presupposed limit of quantification is verified.

### 3.2.3. Accuracy profile.

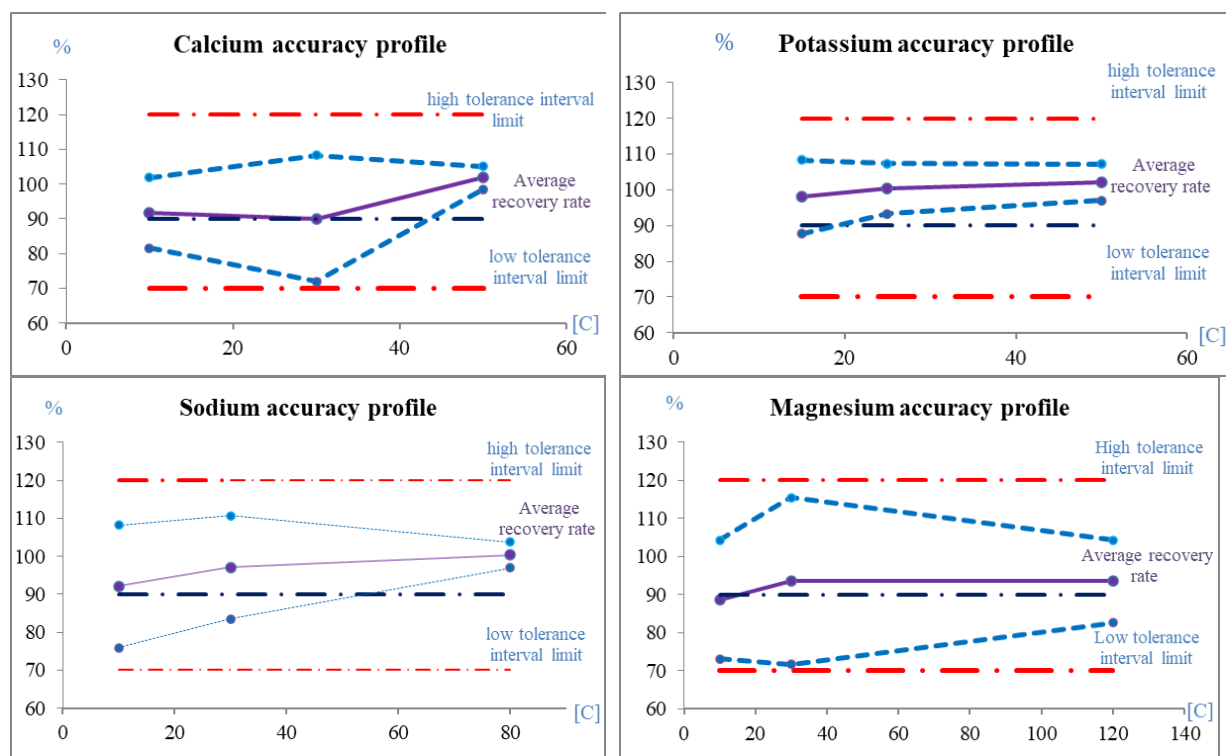
The accuracy validation was performed according to NF V03-110 standard (Table 4). Materials with reference values were used to check method accuracy. Mean concentration, collection rate, tolerance interval limit, and acceptability limit were calculated in the Table. The obtained values for recoveries ranged from 88.7% to 101.8%; these values were very satisfying.

**Table 4.** Method accuracy studied parameters.

Element	Ca			K			Mg			Na		
R.V	10.00	30.00	50.00	15.00	25.00	50.00	10.00	30.00	120.00	10.00	30.00	80.00
M.C.F	9.17	27.02	50.90	14.7	25.06	51.06	8.87	28.07	112.20	9.21	29.12	80.30
R	91.70	90.06	101.80	98.0	100,2	102.1	88.70	93.56	93.50	92.10	97.06	100.37
Bias	-8.30	-9.90	1.80	-2.00	0.20	2.10	-11.30	-6.40	-6.50	-7.90	-2.93	0.375
LTIL	81.60	71.86	98.48	87.60	93.20	97.00	73.13	71.70	82.64	75.94	83.50	96.90
HTIL	101.79	108.20	105.11	108.34	107.26	107.20	104.26	115.43	104.35	108.25	110.63	103.77
LAL	70.00	70.00	70.00	70.00	70.00	70.00	70.00	70.00	70.00	70.00	70.00	70.00
HAL	120.00	120.00	120.00	120.00	120.00	120.00	120.00	120.00	120.00	120.00	120.00	120.00

R.V: Reference value; M.C.F: Mean concentration found; R: Recovery (%); LTIL: Low tolerance interval limit; Bias (%) HTIL: High tolerance interval limit; LAL: Low acceptability limit; HAL: High acceptability limit.

An accuracy profile is a graphical tool that aims to assist analysts in making decisions regarding the validity of analytical procedures. It is based on combining the tolerance interval and the acceptability interval within the same graph [35,36]. This strategy was introduced by Hubert et al. [35]. The calculated limits are presented as an accuracy profile in Figure 2.



**Figure 2.** Accuracy profiles of the studied elements (Ca, K, Na, and Mg) in argan pulp.

The previous figures show that all the tolerance intervals are included within the acceptable intervals. This indicates that the method can provide sufficiently accurate results for all the studied minerals. Additionally, these graphs provide other indications, particularly for calcium, and it appears that the accuracy varies according to the concentration. Indeed, the recovery rate, which reflects the accuracy, is approximately 90% for 30 ppm, increasing to close to 100% for 50 ppm. This bias can be explained by the variation in handling conditions from day to day.

### 3.3. Application to the real CRM.

The validated method was employed to analyze the certified reference material GBW 07605, and the resulting results were summarized in Table 5. The obtained data demonstrate

that all recoveries were very satisfactory, ranging between 90% and 110%. This indicates the precision and robustness of the method employed for extracting and analyzing minerals from plant material.

**Table 5.** GBW 07605 analysis results.

Element	Certified value (mg/kg)	Found value(mg/kg)	Recovery %
K	16600	16217	97.62
Ca	4300	4589	106.72
Mg	1700	1552	91.29
Na	44	40	90.90

#### 4. Conclusions

In this study, a simple method for the detection of minerals in argan pulp using an inductively coupled plasma optical emission spectrometer and dry ashing method was optimized and validated following the guidelines outlined in NF T90-210 and NF V03-110. Three validation criteria were checked: linearity, quantification limit, and accuracy. The acquired results indicate that the method exhibits linearity across the concentration range for the four minerals. The accuracy of the assumed limit of quantification has been confirmed. Satisfactory recoveries were achieved, and the accuracy profiles show that all the tolerance intervals are within the acceptable ranges, establishing the method's capability to deliver sufficiently accurate results for all investigated minerals. This validation can potentially enhance the value of argan tree by-products, specifically the pulp, across diverse applications.

#### Author Contributions

All authors have read and agreed to the published version of the manuscript.

#### Institutional Review Board Statement

Not applicable.

#### Informed Consent Statement

Not applicable.

#### Data Availability Statement

Data supporting the findings of this study are available upon reasonable request from the corresponding author.

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#### Conflicts of Interest

The author has no conflict of interest to declare.

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