Application Note

Food

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Fast, Accurate Analysis of Metal Contaminants in Edible Coconut Products using ICP-MS

Method compliant with FSSAI regulations using Agilent 7850 ICP-MS in helium mode

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Introduction

The coconut palm tree (*Cocos nucifera L.*) is a major plantation crop, providing an important source of nutrition and contributing to the economies of countries in the tropical regions of Asia, South and Central America, the Caribbean, and Africa. The main coconut producing countries are Indonesia, the Philippines, and India, with Sri Lanka, Brazil, Vietnam, and Papua New Guinea also being major producers.[1,2] The white flesh or "meat" of the coconut may be consumed raw or used in preparation of various food delicacies and for the extraction of coconut oil. Coconut oil, an edible oil produced from the kernel of the mature coconut, has gained immense popularity in recent years due to its numerous health benefits.[3] Other edible products derived from coconuts include coconut water, coconut milk, cream, and butter, and desiccated coconut flesh in the form of powder, flakes, chips, and flakes.

The global drive for improved food safety[4] has been evolving over decades with all stages of food production, processing, packaging, storage, transport, and preservation coming under scrutiny. Consumers and regulatory bodies are focused on minimizing the health risk due to unwanted contaminants and toxins in food products and ingredients. One aspect of ensuring food safety is controlling the levels of potentially toxic elements, which may be naturally present in foods or result from contamination. Some elemental contaminants, particularly those referred to as "heavy metals"—such as lead (Pb), arsenic (As), mercury (Hg), and cadmium (Cd)—are known to cause devastating environmental and health effects. High levels of metals can enter a food crop from contaminated soil or groundwater, by environmental exposure such as airborne deposition, and from anthropogenic sources including application of fertilizers, pesticides, and other agrochemicals. Metals may also be added, either intentionally or accidentally, as ingredients, additives, or contaminants during food production, processing, and packaging.

To address global food safety concerns, regulatory bodies around the globe publish and enforce maximum allowable concentrations for a range of elemental contaminants in various foodstuffs, ingredients, and additives. For example, the United States Food and Drug Administration (USFDA) Code of Federal Regulations Title 21 (21 CFR) Chapter 1 Subchapter B Parts 100 to 199 contain regulations that apply to the production and quality of foods and additives intended for human consumption.[5] In India, the Food Safety and Standards Authority of India (FSSAI) regulates the maximum levels of toxins, residues, and contaminants—including heavy metals—that are permitted in various food products. Table 1 shows the maximum allowable concentrations of contaminant elements regulated in coconut products listed in the FSSAI Food Safety and Standards (Contaminants, Toxins and Residues) First Amendment Regulations, 2020.[6]

Table 1. Maximum allowable concentrations of elemental contaminants in coconut products listed in FSSAI regulations.

Element	Maximum Concentration, ppm
Lead	0.1*
Copper	30
Arsenic	0.1*
Tin	250
Cadmium	1.5
Mercury	1
Nickel	1.5

** Limits quoted for Pb and As are specific for coconut oil. Limits quoted for other contaminant elements are generic limits for unspecified foods.*

ICP-MS is widely used for the determination of trace elements in samples across many industries, including food production and monitoring. The key benefits of the ICP-MS technique are its low detection limits, multielement capability, high throughput, and good accuracy due to relatively few spectral interferences.

The Agilent 7850 ICP-MS is an ideal instrument for food testing laboratories that need to analyze elemental contaminants in foods, including laboratories that are new to the ICP-MS technique or new to Agilent systems. The 7850 combines proven hardware capabilities, helpful software presets, built in method and report templates, and ease-of-use features that simplify all aspects of the analytical workflow. Varied sample types are easily handled by the exceptionally robust plasma ion source of the 7850, ensuring accurate data, good long-term stability, and lower maintenance requirements. The 7850 controls common spectral interferences using helium (He) collision cell mode and Kinetic Energy Discrimination (KED)[7,8] (known as He KED mode), while doubly-charged interferences can be addressed using half-mass correction in the Agilent ICP-MS MassHunter software.[9] These features help ensure accurate results, reducing the need for sample remeasurements. The instrument's 10 orders linear dynamic range also simplifies method setup, as major and trace analytes can be measured in a single run, meaning fewer reruns due to over-range results.

The 7850 also includes Agilent Ultra High Matrix Introduction (UHMI) aerosol dilution technology as standard. UHMI further improves the already exceptional plasma robustness of the 7850, enabling the instrument to handle samples with percent levels of total dissolved solids (TDS).[10] The IntelliQuant function in ICP-MS MassHunter also provides a useful screening function to allow analysts to obtain semiquantitative results for up to 78 elements from the QuickScan full mass spectrum data. QuickScan only takes an additional two seconds per acquisition and does not require element-specific calibration standards. IntelliQuant's periodic table "heat map" view provides a quick and simple overview of the concentration of all elements within the sample, helping analysts to identify unexpected contaminants that were not included in the quantitative analysis.[11]

This study describes the use of a 7850 ICP-MS system for the analysis of elemental contaminants in coconut products. The analyte list included Pb, Cu, As, Sn, Cd, Hg, and Ni, which are listed in the FSSAI regulations. Samples of several popular coconut-based food products were microwave digested and analyzed using the 7850 ICP-MS, with sample delivery via an Agilent SPS 4 autosampler. The measured elemental

concentrations were evaluated for compliance with the limits defined in the FSSAI regulations.

Experimental

Coconut product samples

Three types of coconut-based food products, including coconut oil, coconut milk, and desiccated coconut powder, were selected for the study. These commercially available products were bought from a local supermarket in Bangalore, India.

Sample preparation

For each coconut product, triplicate samples were prepared by weighing 0.5 g ± 0.001 g of sample into a microwave digestion vessel. Then, 8 mL of trace metal grade nitric acid (HNO_3) and 0.5 mL trace metal grade hydrochloric acid (HCl) were added into each vessel. The samples were digested using an Anton-Paar Go microwave digestion system using the program outlined in Table 2. The digested samples yielded clear solutions that were then diluted to 25 mL with de-ionized water. The prepared samples were analyzed for trace metal content using the 7850 ICP-MS. Method blanks and spiked samples were prepared along with the coconut samples. The sample preparation and analysis processes are shown schematically in Figure 1.

Table 2. Microwave digestion program for coconut sample preparation.

Step	Ramp Time (mins)	Temp (°C)	Hold Time (mins)
1	10	120	5
2	10	160	5
3	10	180	20

Calibration standards

Calibration standards were prepared in a matrix of 2% HNO_3 and 1% HCl. HCl is routinely added to samples for analysis by ICP-MS, as the presence of the HCl ensures that elements such as Hg, which are chemically unstable in HNO_3 alone, are retained in solution. Any Cl-based polyatomic interferences formed from the HCl are easily controlled using He KED mode that is standard on all Agilent ICP-MS systems.[6] Calibration standards were prepared from Agilent mixed stock solutions including multielement calibration standard-2A (p/n 8500-6940) and its Part 2 for Hg, and multi-element standard 3 (p/n 8500-6948). Most of the elements were calibrated from 0.05 to 1000 ppb, while Hg was calibrated from 0.05 to 10 ppb.

The internal standard (ISTD) solution containing 600 ppb of Li, Sc, Ge, Rh, Tb, Lu, and Bi (Agilent p/n 5188-6525) plus Ir (the preferred ISTD for Hg) was prepared in 1% HNO_3. The internal standard solution was added online at a flow rate approximately 16 times lower than the sample flow to minimize sample dilution and maintain the best possible detection limits.

Instrumentation

An Agilent 7850 ICP-MS was used for the analysis. The 7850 includes the UHMI aerosol dilution system, which further enhances plasma robustness to enable routine analysis of percent levels of matrix, and the ORS^4 collision/reaction cell for simple, reliable control of common polyatomic interferences. The 7850 was fitted with the standard sample introduction system, consisting of a MicroMist glass concentric nebulizer, temperature-controlled quartz spray chamber, and quartz torch with 2.5 mm id injector. A nickel-plated copper sampling cone was used, together with a nickel skimmer cone.

The sample preparation applied a total dilution factor of 50 (0.5 g sample made up to a final volume of 25 mL), so the matrix level in the digests could be up to 2% TDS. However, foodstuffs such as vegetable and nut oils typically contain a very high organic content, which is destroyed during microwave digestion, so the solutions analyzed in this study contained relatively low matrix levels. Agilent ICP-MS systems are routinely optimized for good matrix tolerance (robust, high temperature plasma conditions), so UHMI aerosol dilution was not required for the samples analyzed in this study. Instrument conditions were optimized using the general purpose autotune function of the ICP-MS MassHunter software. All analytes were acquired in He KED mode to ensure effective control of any possible background and matrix-based polyatomic interferences, such as ArO, C_2, ArC, ArCl, etc. Instrument operating conditions are listed in Table 3.

Table 3. Agilent 7850 ICP-MS General Purpose preset conditions.

ICP-MS Parameter	Setting
RF Power (W)	1550
Sampling Depth (mm)	10
Nebulizer Gas Flow (L/min)	0.8
Lens Tune	Autotune
He Cell Gas Flow (mL/min)	5.0
KED Bias (V)	5

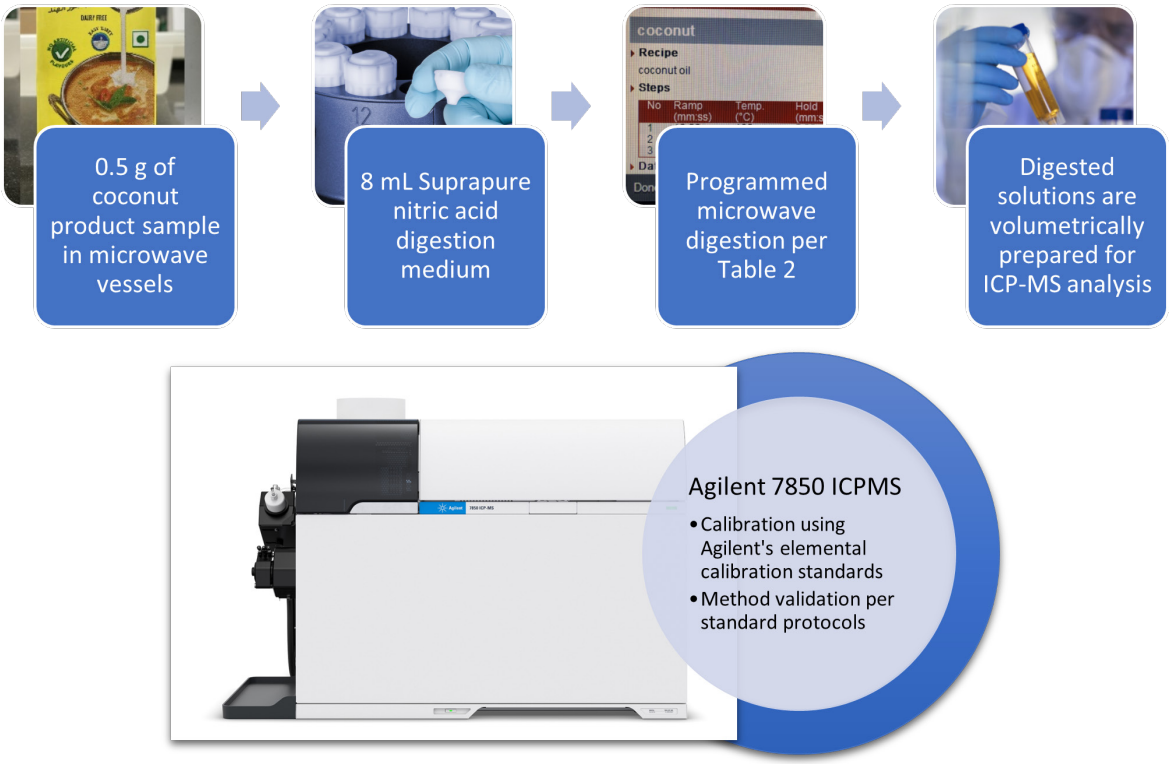

Figure 1. Workflow diagram for sample preparation and analysis of coconut products.

Results and discussion

Representative calibration curves are shown in Figure 2. Calibration parameters such as correlation coefficients, instrument detection limits (IDLs), and method detection limits (MDLs) are presented in Table 4. The MDLs were calculated based on six separate method (digest) blanks measured at the end of the sample run and corrected for the dilution factor of 50.

Table 4. Calibration linearity and DLs in solution and after correction for dilution.

Analyte	R Value	IDL (ppb)	MDL in Sample (ppb)
^{60}Ni	0.9999	0.013	0.625
^{63}Cu	1.0000	0.007	0.357
^{75}As	0.9999	0.015	0.747
^{111}Cd	1.0000	0.007	0.347
^{118}Sn	1.0000	0.010	0.524
^{208}Pb*	1.0000	0.001	0.049
^{201}Hg	0.9999	0.013	0.673

**Pb was measured as the sum of the three most abundant isotopes, 206, 207, and 208*

The 7850 ICP-MS demonstrated good linearity for all elements, shown by the correlation coefficients (R values) of better than 0.9999 for all the calibrated elements listed in Table 4. The MDLs were all at low- or sub-ppb level in the original coconut product samples. These limits easily meet the requirements specified in the 2020 amendment to the FSSAI regulations for coconut oils intended for human consumption (Table 1).

Quantitative results for trace elements in coconut products

Table 5 summarizes the mean quantitative results and Relative Standard Deviation (RSD) for the seven target elements measured in the coconut products.

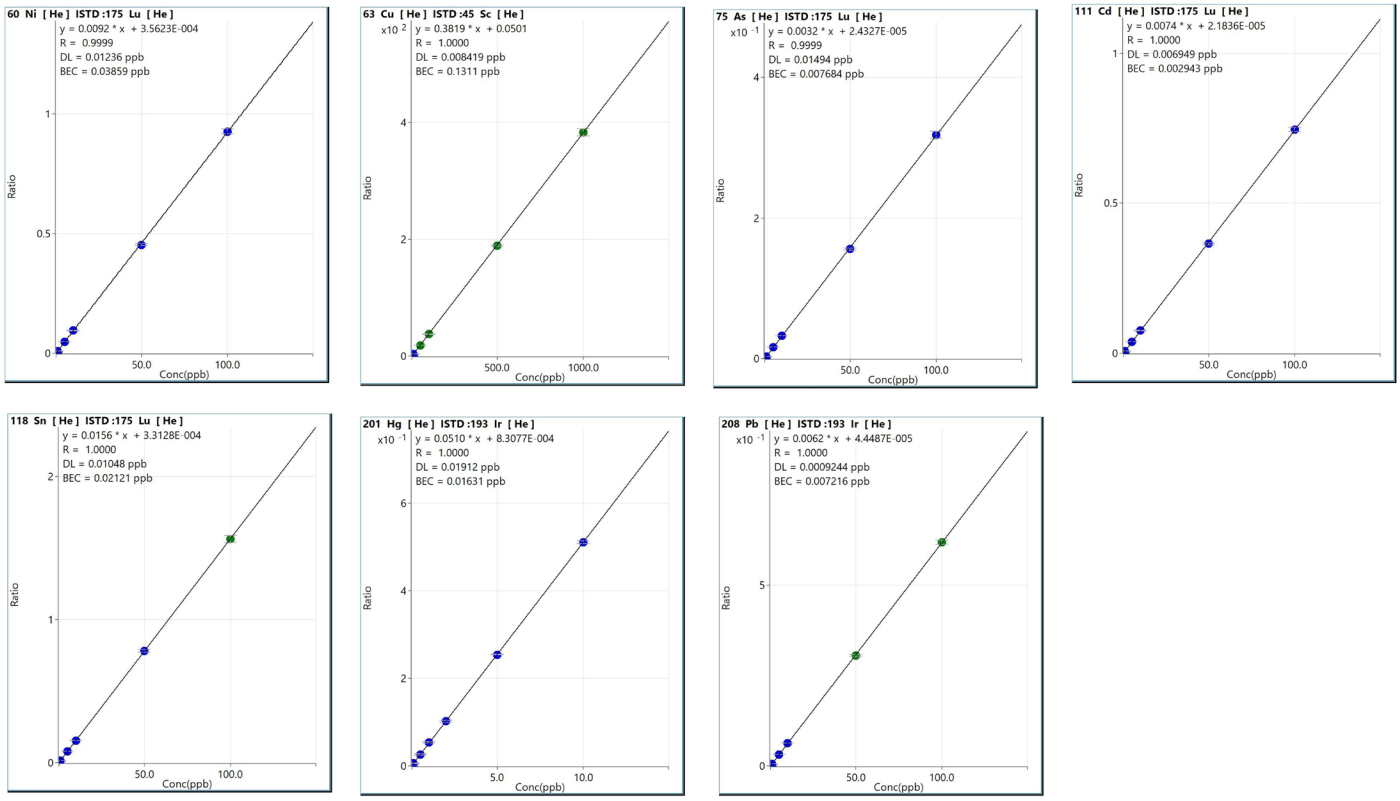

Figure 2. Calibration curves for elements required by the FFSAI regulations.

Table 5. Dilution-corrected quantitative results in µg/g (ppm) for elements in coconut products (n=6).

Element	Coconut Oil		Coconut Milk		Coconut Powder	
	Mean	%RSD	Mean	%RSD	Mean	%RSD
^{60}Ni	< MDL		0.35	3.28	2.79	3.79
^{63}Cu	0.32	3.17	1.28	3.40	10.47	3.59
^{75}As	< MDL		< MDL		< MDL	
^{111}Cd	< MDL		< MDL		< MDL	
^{118}Sn	< MDL		< MDL		< MDL	
^{201}Hg	< MDL		< MDL		< MDL	
^{208}Pb*	< MDL		< MDL		< MDL	

**Pb was measured as the sum of the three most abundant isotopes, 206, 207, and 208*

Spike recoveries

The method for determining elemental contaminants in coconut products was validated using a multi-level spike recovery test. After sample digestion, the sample solutions were spiked at three levels: Level 1, 0.005 mg/kg; Level 2, 0.01 mg/kg; Level 3, 0.02 mg/kg using multi-element standards. Figure 3 shows the spike recovery data for all three spike levels in each of the three coconut sample digests. All the spike recoveries met the acceptance criteria of 80 to 120% defined in AOAC Standard Method Performance Requirements Guidelines.[12]

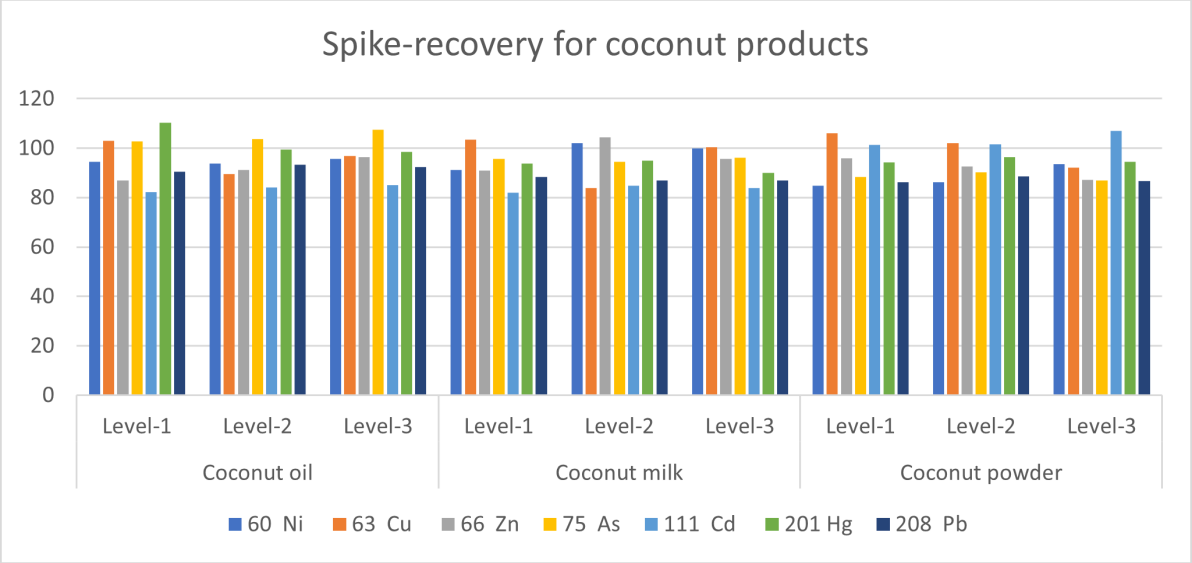

Figure 3. Spike recoveries (%) for three spike levels in coconut product sample digests.

Reproducibility study

To check the reproducibility of the entire sample preparation and analysis method described in this study, the coconut sample analysis was performed independently on two different days (n=6) after spiking a specific concentration to sample solutions. The between-day reproducibility was compared to the within-day repeatability. The results shown in Table 6 demonstrate the excellent reproducibility of the sample preparation approach and the quantitative analysis using the 7850 ICP-MS.

ISTD recovery (%)

The complete analytical sequence performed for this study—including calibration standards, samples, spikes, and method blanks—had a run time of approximately two hours. Figure 4 shows the ISTD stability control chart for one complete sample batch. The ISTD recoveries are normalized to the signals in the calibration blank. All the ISTD recovery plots were within ±20%, with no internal standard failures throughout the run. The results demonstrate the excellent stability and matrix tolerance of 7850 ICP-MS. No significant signal drift or divergence in the signals for low- and high-mass ISTD elements was observed during the sequence. The recovery test shows that the plasma was able to decompose the variable sample matrices effectively.

Table 6. Within-day repeatability and between-day reproducibility for elemental contaminants in coconut products. Concentration units: µg/g.

Analyte	Coconut Oil				Coconut Milk				Coconut Powder			
	Repeatability (n=6)		Reproducibility (n=12)		Repeatability (n=6)		Reproducibility (n=12)		Repeatability (n=6)		Reproducibility (n=12)	
	Mean	%RSD	Mean	%RSD	Mean	%RSD	Mean	%RSD	Mean	%RSD	Mean	%RSD
^{60}Ni	0.52	2.41	0.51	3.18	0.60	3.57	0.60	3.49	3.05	1.40	3.07	1.43
^{63}Cu	0.87	2.26	0.86	3.33	1.42	3.26	1.42	3.83	9.78	1.59	9.98	2.76
^{75}As	0.63	2.64	0.62	3.70	0.32	4.84	0.31	4.90	0.53	2.77	0.53	2.07
^{111}Cd	0.50	2.22	0.48	4.75	0.30	3.63	0.30	4.52	0.46	1.99	0.45	3.08
^{118}Sn	0.54	2.04	0.55	2.49	0.35	3.40	0.35	3.29	0.49	1.38	0.51	3.69
^{201}Hg	0.51	3.01	0.50	4.24	0.29	3.37	0.29	3.40	0.43	1.71	0.41	4.57
^{208}Pb*	0.52	2.71	0.54	3.71	0.30	2.96	0.32	4.91	0.44	2.16	0.44	2.35

**Pb was measured as the sum of the three most abundant isotopes, 206, 207, and 208*

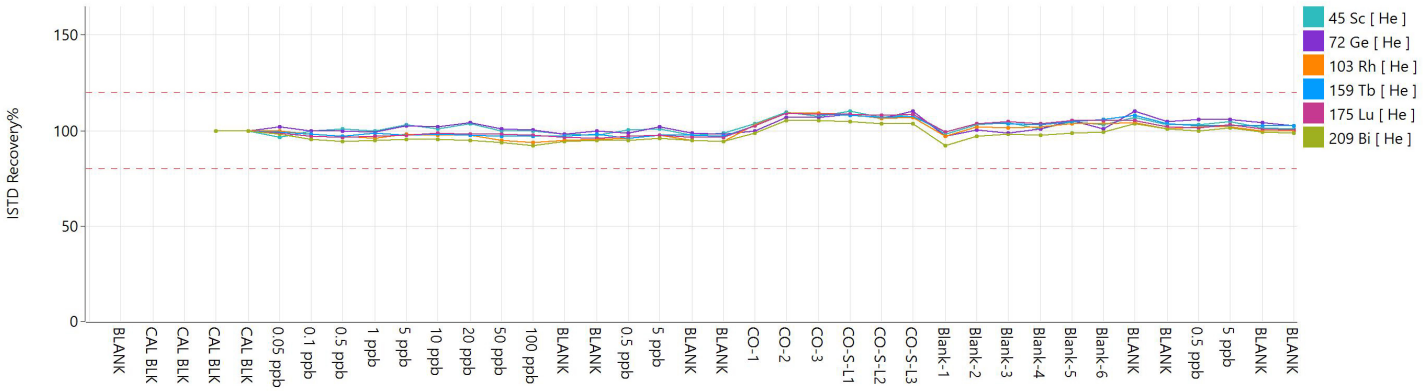

Figure 4. Stability of ISTD measurements over two hours.

IntelliQuant data

When an analyst creates a method in ICP-MS MassHunter based on a preset method, an IntelliQuant QuickScan acquisition in He mode is automatically included in the method. QuickScan collects data for all masses with only two seconds of extra measurement time. QuickScan enables the analyst to quickly see which elements are present in each sample, making it perfect for screening unknown samples. QuickScan can be used to identify unexpected elements that were not included in the quantitative analysis and to confirm their presence by automatically comparing the measured mass spectrum to the theoretical isotopic abundances.

IntelliQuant uses the QuickScan data to calculate the semiquantitative concentrations for up to 78 elements based on a stored mass/response curve. No special setup or separate calibration is needed, simplifying the analysis. IntelliQuant data is acquired in He KED mode, so analytes are largely free from errors caused by polyatomic ion overlaps, ensuring the quality of data.

In this study, QuickScan data was collected for each of the three types of coconut products and IntelliQuant was used to calculate the concentration of "all elements" measured in each sample. The QuickScan data can be displayed in a periodic table heat map, as shown in Figures 5, 6, and 7 for coconut oil, coconut milk, and coconut powder, respectively. The color intensity of each element indicates the concentration of that element in the selected sample, with darker colors indicating higher concentrations. The data can also be reported in a conventional table of concentration values.

Taking sodium as an example, the heatmap for coconut oil indicates a relatively low content of 188 ppb (Figure 5). Coconut oil is typically produced by cold-pressing kernels, a method that avoids the use of additional ingredients. In the case of coconut milk, which is predominantly used in foods, sodium chloride is added as a preservative and for taste. This salt addition accounts for the higher sodium content of 78 ppm (Figure 6). Figure 7 shows sodium at 5 ppm in the desiccated coconut powder, likely a result of the combined sodium content in the oil and the kernel being concentrated during manufacturing.

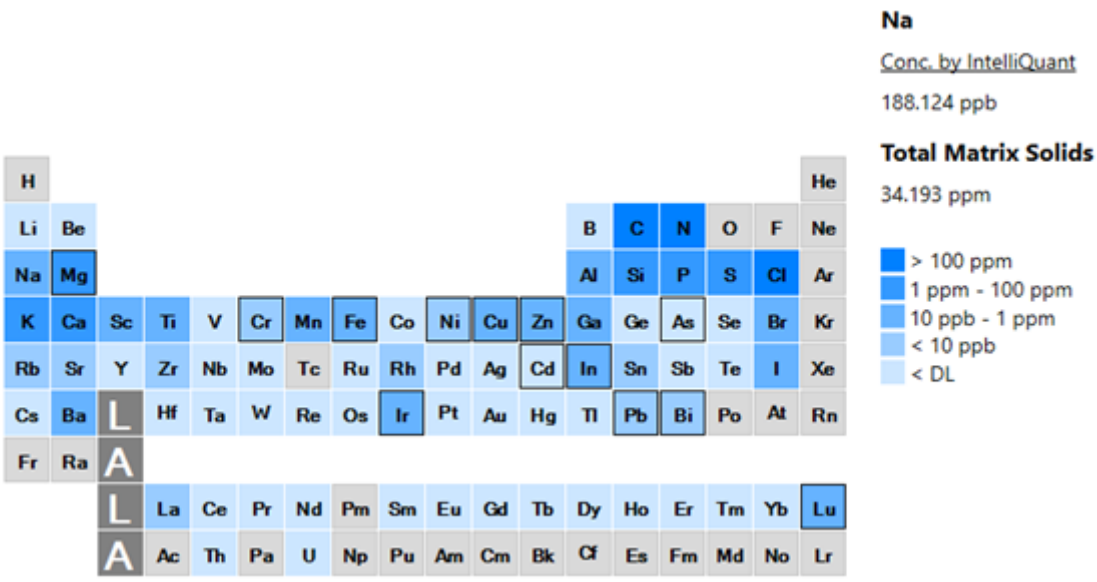

Figure 5. IntelliQuant periodic table heat map view of coconut oil sample.

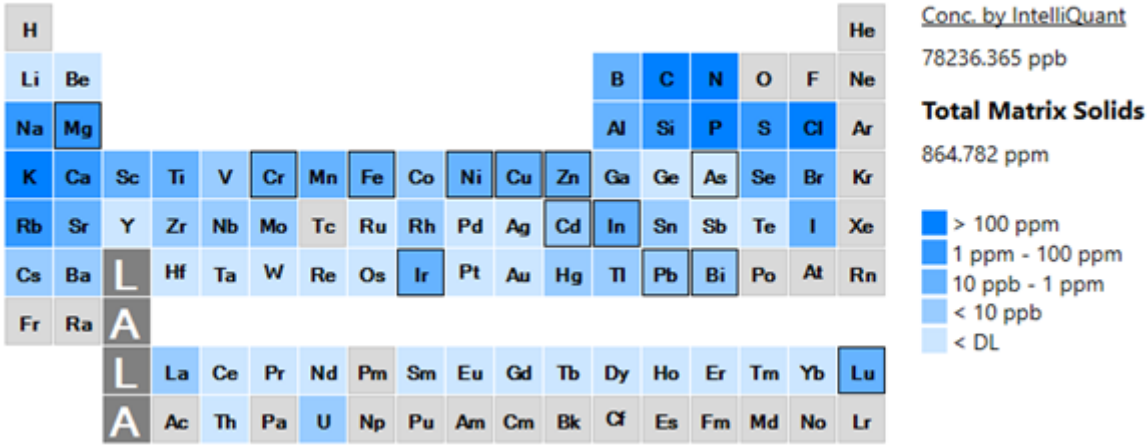

Figure 6. IntelliQuant periodic table heat-map view of coconut milk sample.

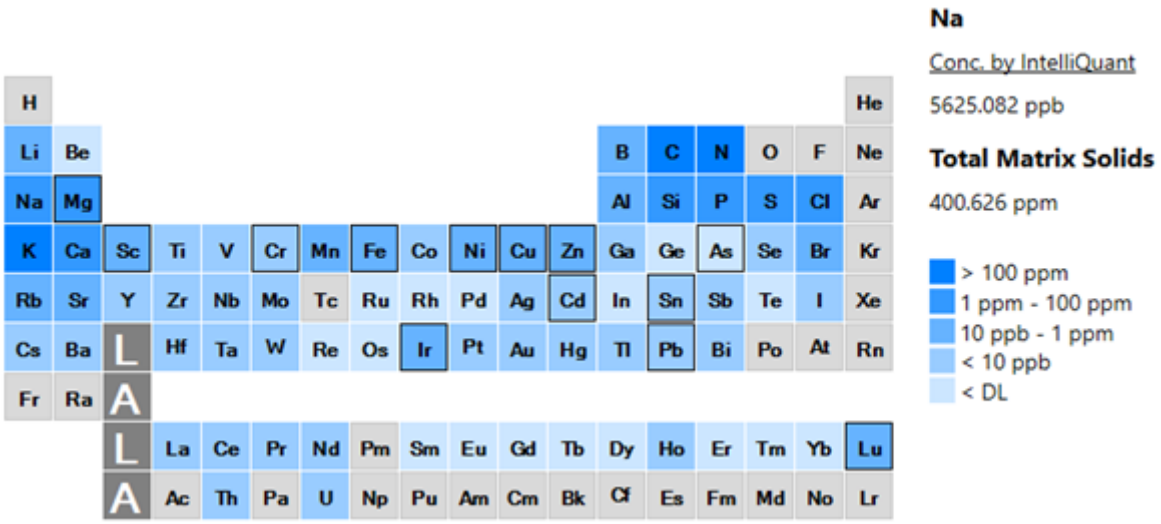

Figure 7. IntelliQuant periodic table heat map view of coconut powder sample.

Conclusion

The Agilent 7850 ICP-MS was used to quantify heavy metals and trace elements in edible coconut products, including coconut oil, coconut milk, and coconut powder. Microwave assisted digestion was used for the preparation of the samples for analysis. Good calibration linearity was obtained for all analytes with linear regressions over a calibration range from 0.05 to 1000 ppb (0.05 to 10 ppb for Hg). Data validation was done using a spike-recovery study performed at three concentration levels. An inter-day reproducibility study was conducted and the data comparison indicated that the between-day reproducibility was comparable to the excellent within-day repeatability. These results confirm that this analytical method can be used for the routine detection and accurate quantification of various elements in edible coconut products in accordance with FSSAI regulations.

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Products used in this application

Agilent products

7850 ICP-MS

SPS 4 autosampler

Multi-element calibration standard-2A

Multi-element Calibration Standard 3

Internal standard mix, for ICP-MS systems

www.agilent.com/chem/7850icpms

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