Application Note

Food

Determination of Toxic Elements in Durian and Jackfruit Using an Agilent Automated ICP-MS System According to China GB 5009.268

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Abstract

This application note describes the development and validation of a method for the analysis of toxic elements in durian and jackfruit samples using an Agilent 7900 ICP-MS, in accordance with the China GB 5009.268 method.[1] The automatic ICP-MS system has been implemented to enhance operational efficiency, increase sample throughput, and minimize human error. The instrument's performance was demonstrated through long-term stability, selectivity, reproducibility, and high sensitivity, which meet the low-level standards set by the Chinese National Food Safety Standard on Maximum Levels of Contaminants in Food (GB 2762-2025). The method gave excellent linearity ($R^2 \geq 0.995$) over the range of 0.05–10 µg/L, the recovery rates were between 75–105%, and RSD < 20% was achieved across various spike concentration levels, reaching GB/T 27417-2017.[2]

Keywords: As, Hg, Cd, Pb, durian, jackfruit, ICP-MS 7900, AVS, ADS2

Introduction

Fruit is an essential part of daily human nutrition, providing vital vitamins, minerals, and dietary fiber.[3] The Chinese market, with over 1.4 billion people, has become one of the largest fruit markets in the world. It is expected that China's total fruit consumption will reach about 312 million tons by 2025.[4] Additionally, fruit imports are projected to exceed 9 million tons annually, underscoring the country's strong domestic demand and reliance on imported supplies.

Within this context, China has become a major export destination for Asian countries, including Vietnam, Thailand, Laos, Cambodia, Philippines, Indonesia, and others. Thanks to favorable climate conditions and a wide variety of tropical fruit cultivars, these countries have strategically increased their fruit exports to satisfy Chinese market demand. Specifically, products such as durian and jackfruit have gained significant popularity, playing an increasingly vital role in regional agricultural trade.

However, the biggest challenge for those countries when entering the Chinese market is strict food safety regulations, especially limits on trace elements and heavy metals such as lead (Pb), cadmium (Cd), mercury (Hg), and arsenic (As). The General Administration of Customs of the People's Republic of China (GACC) sets very low Maximum Residue Limits (MRLs, Table 1). Additionally, the large, continuous volume of fruit exports makes time a critical factor, as any delay can affect product quality and reduce its economic value. This situation places significant pressure on testing laboratories, requiring the development of stable, rapid, and accurate methods to ensure continuous and reliable operations.

To address these challenges, the Agilent ICP Workflow Automation System offers a practical solution. The ICP-MS 7900 provides high-throughput, highly sensitive, and precise measurements of trace elements and heavy metals, including Pb, Cd, Hg, and As. The combination of ADS2 and AVS helps reduce sample turnaround time and cost per analysis. The Advanced Dilution System (ADS2) automatically performs important tasks such as preparing a multipoint calibration from a single stock standard up to 400 times, diluting samples above the calibration range, or, if there is an internal standard recovery issue, eliminating the need for manual dilution. The Advanced Valve System (AVS) automatically rinses the sample introduction system while the next sample is being analyzed, virtually eliminating the delays typical of conventional analysis. Using controlled argon bubble injection between the sample and rinse solution prevents mixing, reducing uptake and rinse times. This study describes the use of the Agilent 7900 ICP-MS, Agilent SPS 4 autosampler, AVS, and ADS2 auto-dilution for the analysis of four toxic elements in durian and jackfruit samples using the China GB 5009.268 method.[1]

Table 1. Limits for heavy metals in fruits per GB 2762-2025.[5]

Element	Maximum Limit (mg/kg)
Pb	0.1
Cd	0.05
Hg	0.01*
As	0.5*

*For elements without specific fruit regulations, the lowest value among food categories is selected.

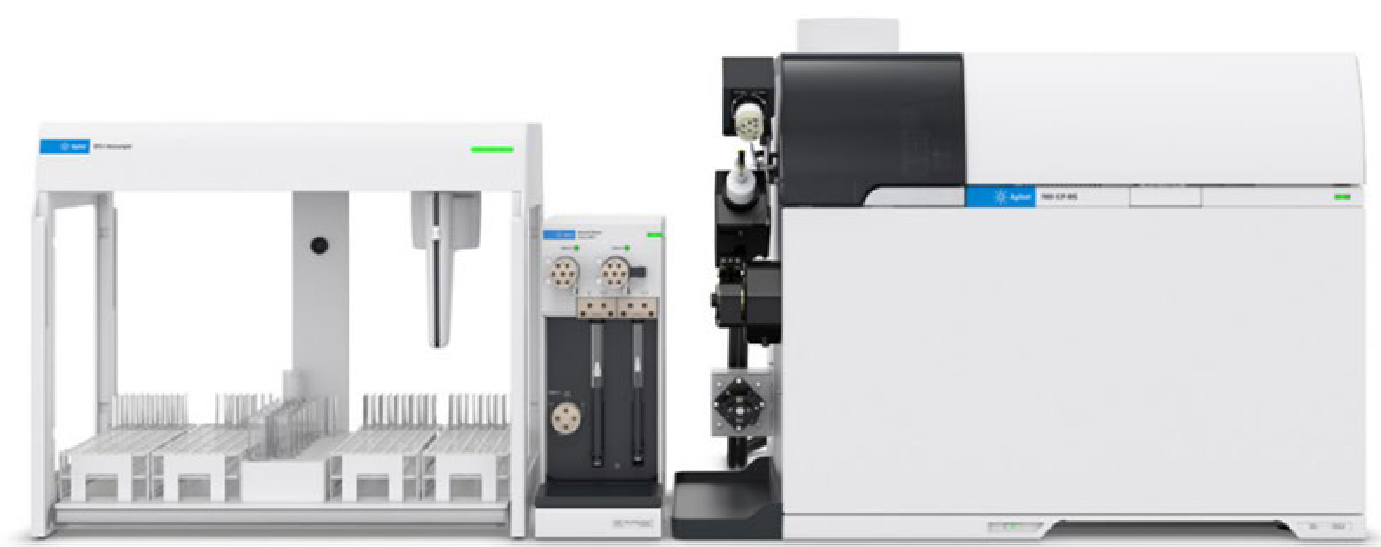

Figure 1. The Agilent 7900 ICP-MS with an integrated AVS MS switching valve connected to the Agilent ADS2 and Agilent SPS 4 autosampler.

Experimental

Sample and reagents

Durian and jackfruit samples were collected using a random sampling technique from various positions within export containers destined for China. The amount was determined to ensure the entire shipment was representative. Only edible parts were retained and ground for analysis.

Nitric acid (≥ 67%, TraceMetal Grade, p/n A509-P500) and hydrochloric acid (34–37%, TraceMetal Grade, p/n A508-P500) from Fisher Chemical and ultrapure water produced by a Milli-Q system from Millipore Sigma (Burlington, Massachusetts, United States) and were used for microwave-assisted sample digestion and the preparation of calibration standards. The Au 1,000 mg/L standard solution ($H(AuCl_4)$ in 7% HCl, 500 mL bottle, Merck, p/n 1702160500) was added to form a gold/mercury amalgam, which helps reduce mercury volatilization and minimize memory effects, preventing mercury from adsorbing onto the instrument's surfaces. This process ensures a more stable signal and highly reliable results.

Standard calibration

Intermediate standard solutions of 10 µg/L were prepared from a multi-element stock solution containing 28 components at 10 mg/L (p/n 8500-6940.L1, CPAchem) by diluting 1000 times. A 7-point calibration curve, ranging from 0.05 to 10 µg/L, was automatically generated by the ADS2, with dilution factors of 200x, 100x, 50x, 10x, 5x, 2x, and 1x using 1% nitric acid. The internal standard was a 50 µg/L solution prepared from an Agilent internal standard mix containing ^{6}Li, Sc, Ge, Rh, In, Tb, Lu, and Bi (100 mg/L, p/n 5188-6525.L1, CPAchem), diluted in 1% nitric acid. The internal standard was mixed online with the sample solution via the AVS system.

Sample preparation procedure

All food samples included in this study were prepared following a digestion procedure using the microwave digestion system Multiwave 5001 (Anton Paar) (Figure 2). Approximately 0.5 g of homogenized durian and jackfruit pulp samples were accurately weighed into a 56 mL PTFE-TFM vessel. For the digestion process, the following reagents were sequentially added to each vessel: 10 µL of Au standard solution at 1,000 mg/L, 50 µL of concentrated HCl, and spike standards, where applicable. The addition of the Au standard and concentrated HCl before spiking helps create a stable environment for mercury and ensures consistent recovery rates of the target analytes. Then, 2 mL of concentrated HNO_3 was added to the mixture. The digestion vessels were capped and left to stand for about 0.5 to 1 hour to allow initial decomposition before further microwave-assisted processing. Finally, using the heating program shown in Table 2, once digestion is complete, the solution is diluted to 25 mL with DI water, filtered, and then processed for further measurement.

Table 2. Microwave digestion parameters.

Parameter	Setting
Power Limit	1,800 W
Ramp Time	20 min
Hold Time	20 min
Temperature	195 °C

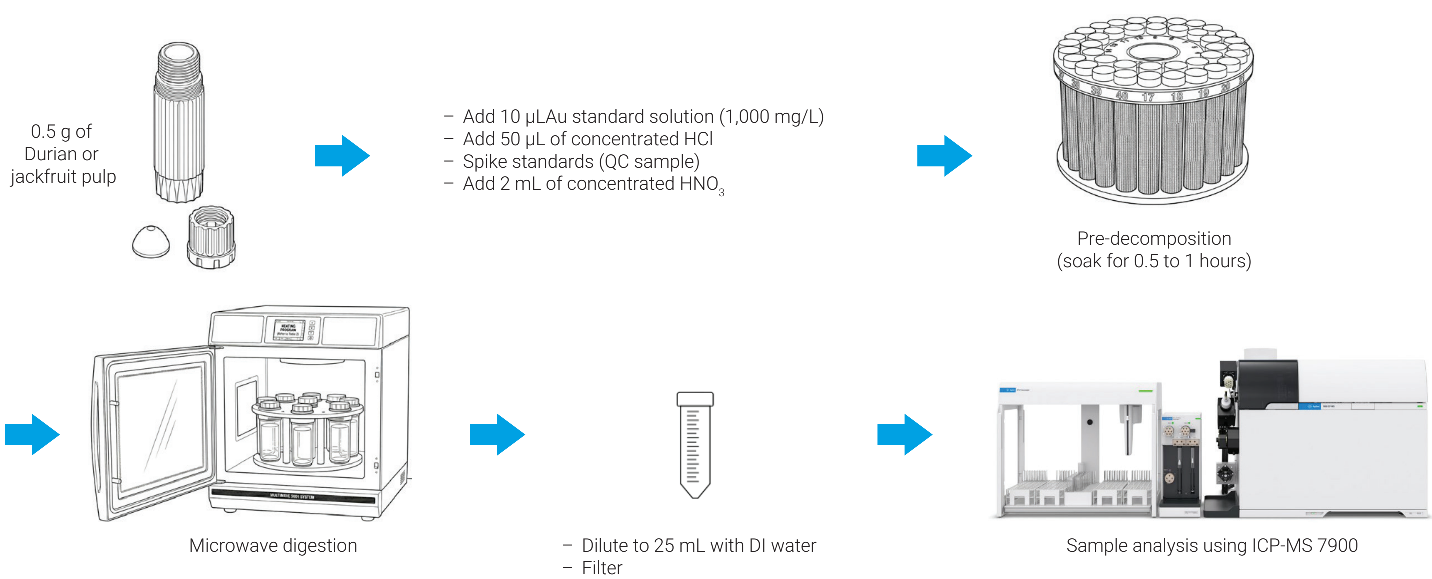

Figure 2. Sample preparation flowchart with microwave digestion.

Instrumentation

Agilent ICP-MS 7900 instruments have a wide linear dynamic range (11 orders of magnitude), allowing major and trace analytes in food samples to be measured in a single run. Also, New High Matrix Introduction (UHMI) enables the ICP-MS to handle samples with total dissolved solids (TDS) levels up to 25%.[6] The system was equipped with a standard MicroMist nebulizer, a temperature-controlled quartz spray chamber, and a one-piece quartz torch with a 2.5 mm ID injector. The interface consisted of a nickel-plated copper sampling cone and a nickel skimmer cone.[7] The ORS4 uses a small cell volume and octopole ion guide for optimal performance in He collision mode.

Automated sample introduction was performed using an SPS4 autosampler, ADS2, and AVS. Combining with ADS2 provides valuable benefits for routine laboratories, including:

- Automatic preparation of multilevel calibrations from single or multiple stock standards with up to 400x dilution. Calibration standards can be prepared and analyzed automatically within 20 minutes, enhancing lab efficiency.
- Smart reactive auto-dilution of samples if any targeted analytes are measured at concentrations above the calibration range. The diluted sample will be automatically added to the real-time sample list, and online analysis data of the diluted sample will be generated accordingly.
- Result-based reactive auto-dilution of samples with analytes that require different dilution factors due to matrices containing elements at various concentrations.[8]

The 7900 ICP-MS operating conditions and ADS2 sample introduction parameters are shown in Tables 3 and 4. To minimize spectral interference, arsenic was measured in KED mode to suppress the polyatomic ion $^{40}Ar^{35}Cl^+$, while the remaining elements were analyzed in both no-gas and helium modes.

Table 3. Parameters set by the Method Automation Software.

Parameter	Setting	
Plasma Mode	Low Matrix	
ORS Mode	No gas	He
RF Power	1,550 V	
Sampling Depth	8 mm	
Carrier Gas	1.09 L/min	
Ext 1 Lens	0 V	
Ext 2 Lens	–230 V	
Energy Discrimination	5 V	
Lens Tune	Autotune	
He Flow Rate	–	4.3 mL/min

Table 4. Agilent AVS and ADS2 sample introduction parameters.

Phase	Time (s)	AVS MS Uptake Pump Speed (%)	Autosampler Needle Position	Valve Position
Sample Load	14	50	Sample	Load
Stabilize	10	5	Rinse Port	Inject
Probe Rinse (Sample)	15	5	Rinse Port	Inject
Probe Rinse (Standard)	15	5	Rinse Port	Inject
Probe Rinse 1	10	50	Rinse Port	Inject
Probe Rinse 2	15	0	Rinse Port	Inject
Optional Loop Probe Wash	8	50	Rinse Port	Load
Optional Loop Probe Wash (Dilution)	8	50	Rinse Port	Load
Optional Loop Wash	1	5	Rinse Port	Inject

Results and discussion

Automatic calibration standards and Instrument stability

A 7-point calibration curve automatically prepared using the ADS 2 demonstrated excellent linearity over the concentration range of 0.05–10 µg/L. Using a 1/x weighted regression without forcing the calibration through the origin, all analytes achieved coefficients of determination (R^2) ≥ 0.995 (Figure 3), with back-calculated concentrations within 90–110% of their theoretical values, confirming the suitability of the calibration range for routine quantification. The integrated automation workflow significantly improved laboratory productivity by completing calibration preparation and analysis in approximately 1.5 minutes per sample. By minimizing manual operations and operator intervention, the ADS 2 reduced the risk of contamination and human error while enabling higher sample throughput for routine food safety testing.

To evaluate instrument stability during continuous operation, the variation in internal standard recoveries was monitored throughout an analytical sequence of more than 70 consecutive samples. The recoveries of the three internal standards across different measurement modes remained consistently within the ±20% acceptance limit throughout the analytical sequence (Figure 4). Blank samples interspersed between concentration intervals showed no evidence of carryover, demonstrating effective system cleanliness and analytical robustness. These results confirm the excellent long-term stability and robust matrix tolerance of the Agilent 7900 ICP-MS, providing reliable performance for high-throughput routine food safety testing.

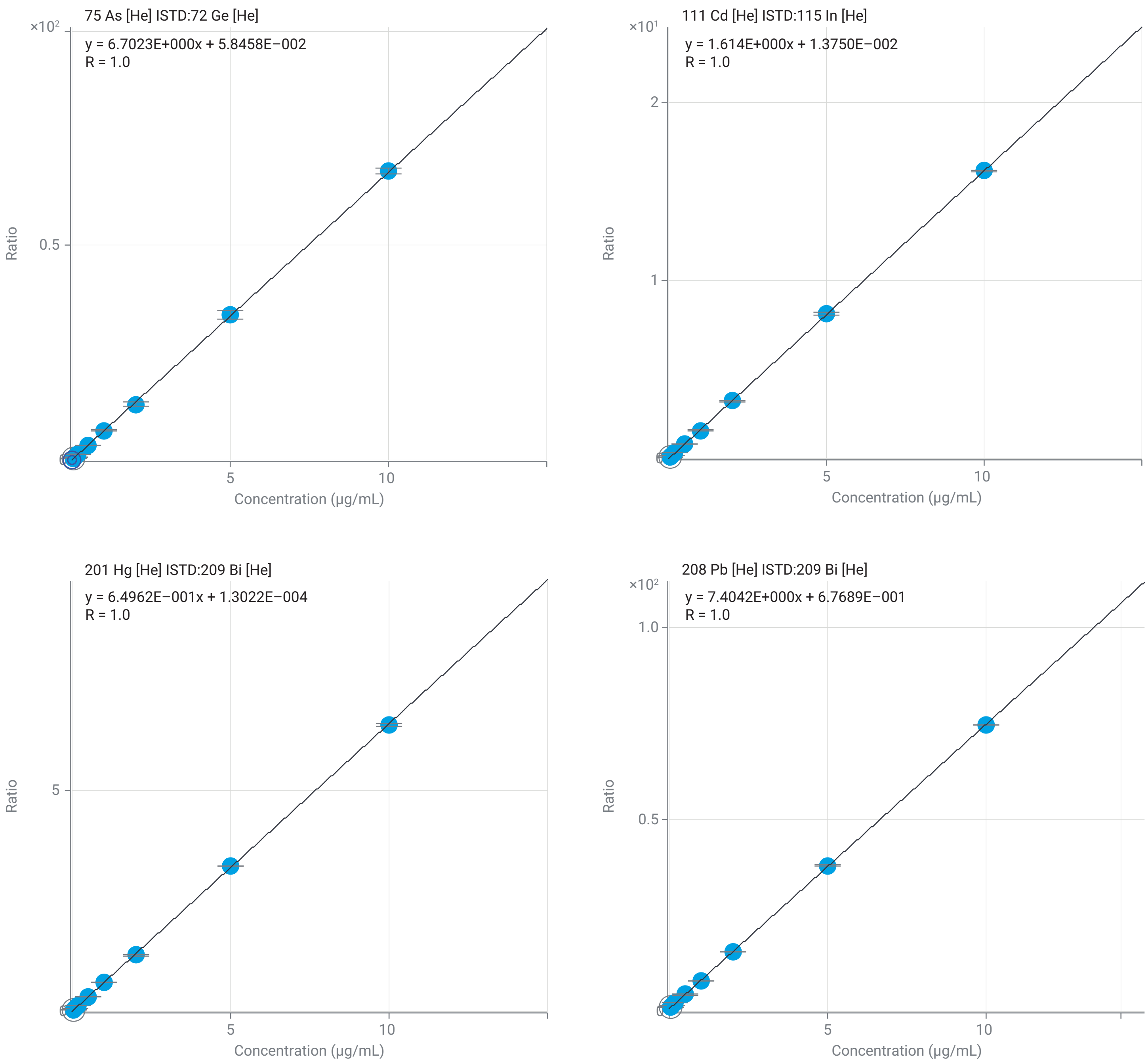

Figure 3. Representative calibration curves for four elements.

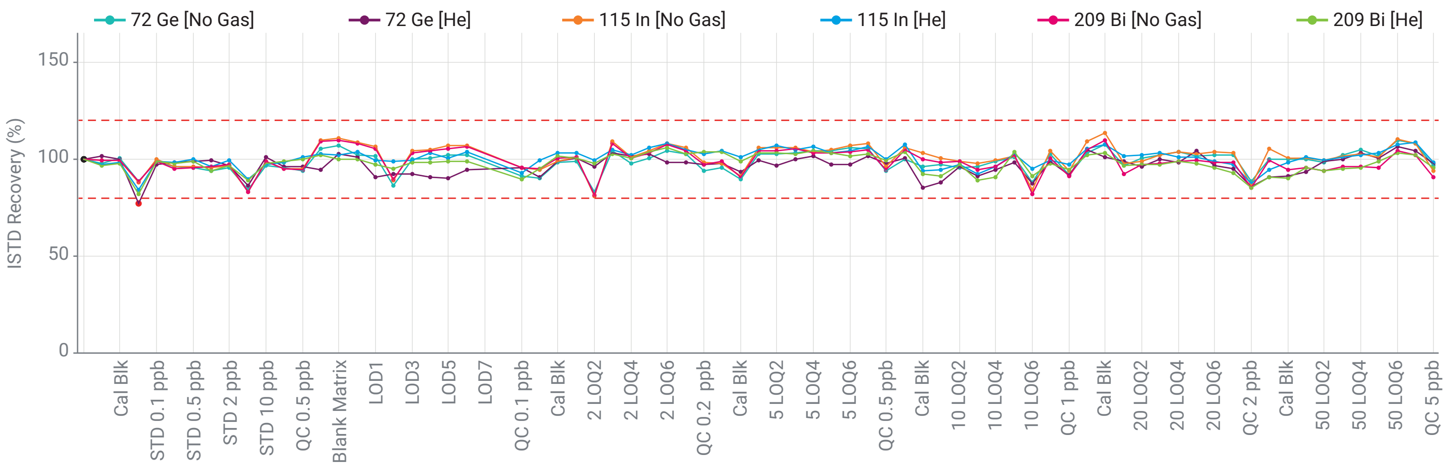

Figure 4. ISTD stability throughout the analysis of a total of over 70 consecutive samples. ISTD recoveries for all samples have been normalized to the calibration blank.

Method accuracy and precision

In determining trace toxic elements in food matrices, method sensitivity and recovery are critical indicators of analytical performance. According to GB 2762-2025, maximum allowable levels for toxic elements in food are strictly regulated, requiring analytical methods with sufficiently low limits of quantification (LOQs) (Table1).[8]

In this application note, a unified LOQ of 5 µg/kg was applied to all four target elements, providing a simplified and efficient workflow for routine analysis. Method accuracy was evaluated using pre-spiked durian and jackfruit matrices at three concentration levels: 5 µg/kg (LOQ), 10 µg/kg (2×LOQ), and 25 µg/kg (5×LOQ), with a total of 14 replicate analyses performed across the study.

The distribution of recovery performance is presented in Figure 5. Overall, recoveries for all four target elements at the 5 µg/kg spike level ranged from 71 to 115% across both durian and jackfruit matrices, meeting the acceptance criteria specified in AOAC Appendix F and GB/T 27417-2017.[2,9] The average recoveries in durian were 85.37, 83.94, 75.87, and 103.31% for As, Cd, Hg, and Pb, respectively, while the corresponding values in jackfruit were 92.69, 107.01, 93.89, and 94.74%. Similar trends were observed at the higher spike levels (10 and 25 µg/kg), where the recovery distributions became noticeably tighter and more consistently clustered around the expected values, reflecting improved quantitative precision and the robustness of the analytical workflow. Slight matrix-dependent differences were observed between the two fruit matrices. The jackfruit matrix generally exhibited higher mean recoveries, whereas the durian matrix showed a more consistent recovery distribution across replicate analyses. Despite these differences, all recoveries met the

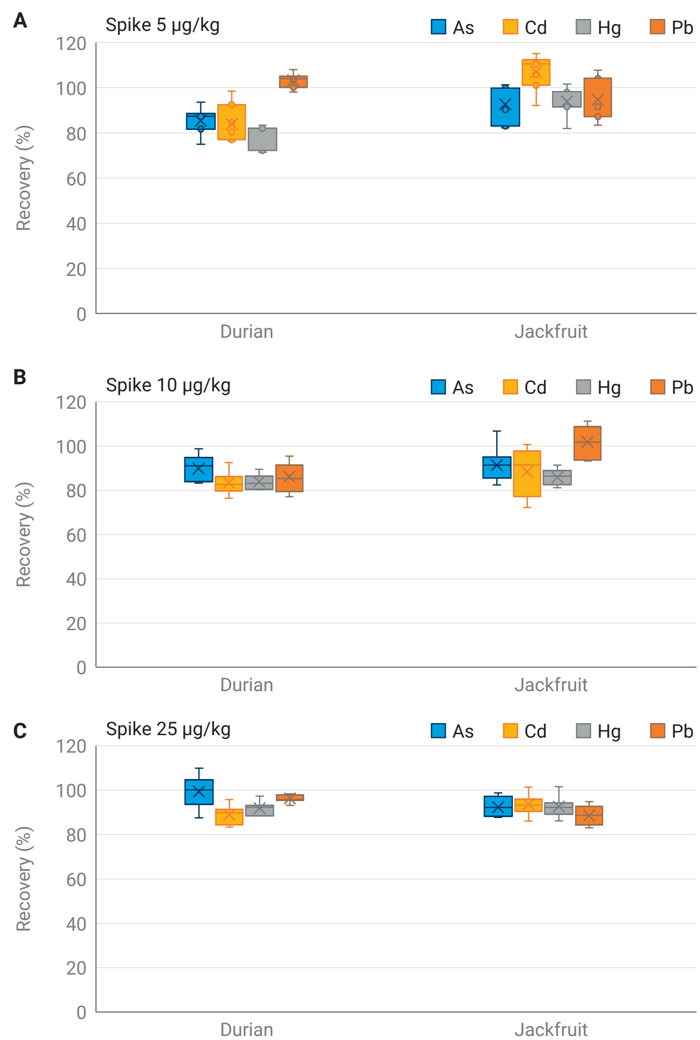

Figure 5. Recovery distribution of As, Cd, Hg, and Pb in two matrices prespiked at (A) 5 µg/kg, (B) 10 µg/kg, and (C) 25 µg/kg.

acceptance criteria of GB/T 27417-2017, demonstrating the robustness of the method and the ability of the Agilent 7900 ICP-MS to deliver reliable quantitative results across diverse and challenging food matrices.

Precision was evaluated based on repeatability (RSD_r) and reproducibility (RSD_R). Repeatability was determined by a single analyst performing seven replicate analyses (n = 7) under identical conditions. Subsequently, a second analyst repeated the procedure on a different day to assess reproducibility, yielding a total of 14 replicates (n = 14) across both analysts.

Figure 6 illustrates the repeatability (RSD_r) and reproducibility (RSD_R) of the method at three spike levels across both durian and jackfruit matrices. Overall, excellent precision was achieved for all four target elements, with both RSD_r and RSD_R values consistently below 10%. Specifically, RSD_r values ranged from 3 to 8% across both matrices, with an average of approximately 5.5%, while RSD_R values ranged from 5 to 9%, with an average of approximately 7.5%. The consistently low RSD_r and RSD_R values across all spike levels demonstrate the method's excellent repeatability and reproducibility over a wide concentration range. These results confirm the robustness, consistency, and reliability of the analytical workflow for determining trace toxic elements in complex fruit matrices and fully satisfy the performance criteria specified in AOAC Appendix F and GB/T 27417-2017.[2,9]

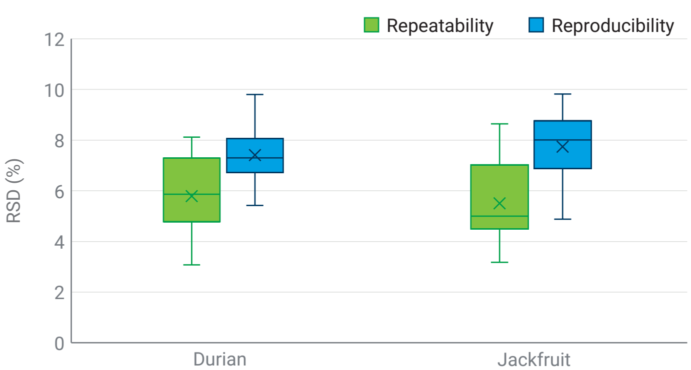

Figure 6. Method performance evaluation in terms of repeatability (n = 7) and reproducibility (n = 14) at spike 5 µg/kg, 10 µg/kg, and 25 µg/kg.

Conclusion

The Agilent 7900 ICP-MS, integrated with the SPS 4 autosampler, ADS 2, and AVS, delivers a fully automated, high-throughput workflow for routine trace-level multi-element analysis. By eliminating manual standard preparation and minimizing operator intervention, the solution reduces labor costs, improves data consistency, and empowers laboratories to maximize productivity and analytical capacity to meet increasing testing demands.

Excellent sensitivity, long-term stability, and robust interference removal, together with satisfactory recoveries, repeatability, and reproducibility for As, Cd, Pb, and Hg at an LOQ of 5 µg/kg, demonstrate reliable analytical performance. Compliance with Chinese regulatory requirements further position the Agilent automation solution as a cost-effective platform for high-volume food safety testing and export quality assurance, enabling laboratories to confidently support the growing demand for exports to the Chinese market.

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