

## Application News

NO. SP-06-ADI-056

## ICPMS-2030

### Estimation of heavy metals in Tea and Coffee powder using Shimadzu inductively-coupled-plasma-mass spectrometry

#### Introduction

**Tea**, traditionally known as a beverage made by brewing the leaves of *Camellia sinensis*, tea is one of the most consumed non-alcoholic drink around the world due to its nutritional importance as a source of several minerals (e.g., Fe, Mn, K, and Zn) and antioxidants such as catechin and flavonoids. The health benefits related to tea consumption also include the prevention of diseases, such as skin cancer, myocardial infarction, and Parkinson's disease, as well as the reduction of cholesterol levels in the blood and the risk for type 2 diabetes.

The inorganic components of tea include essential minerals such as Cu, Fe, Mn, and Zn and non-essential minerals or contaminants such as As, Cd, and Pb. The accumulation of trace elements in tea leaves is based on the acidophilic nature of their plants: metal dissolution increases in acidic soils compared with neutral or alkaline soils.

**Coffee** is consumed by around 40 % of the world's population. For many people, especially in Western countries, the coffee drinking is a part of their lifestyle and an everyday habit. The coffee beverage can be consumed for many reasons, including its stimulatory effects resulted from the presence of caffeine, rich phytochemistry, health benefits, and primarily excellent taste and aroma. Due to a habitual consumption of coffee, its chemical composition, namely the presence of essential, non-essential and toxic elements, has to be known and kept under control in terms of its safety, and to assist its quality, nutritional value, and certain sensorial properties.

The main purpose of this study is to verify the applicability of the ICPMS 2030 for the determination of inorganic constituents in tea and coffee.

**Table 1: FSSAI permissible elemental Limits for tea and coffee.**

Elements	FSSAI Limits (ppm)	
	For Tea	For Coffee
Arsenic (As)	1.1	1.1
Cadmium (Cd)	1.5	1.5
Copper (Cu)	150	70
Mercury (Hg)	1.0	1.0
Lead (Pb)	10	5
Tin (Sn)	250	250
Zinc (Zn)	50	50

#### Experimental

A sample of Tea and coffee powder from branded source was purchased locally for the extraction of elements for this study.

Table 1 shows maximum residual limit (MRL) as per FSSAI [1][2] for elements. Table 2 shows limit of quantification (LOQ) which are achieved based on 20% of maximum residual limit (MRL).

Elements	For Tea		For Coffee	
	LOQ (ppm)	10x LOQ (ppm)	LOQ (ppm)	10x LOQ (ppm)
Arsenic (As)	0.22	2.2	0.22	2.2
Cadmium (Cd)	0.3	3.0	0.3	3.0
Copper (Cu)	30	300	14	140
Mercury (Hg)	0.2	2.0	0.2	2.0
Lead (Pb)	2.0	20	1	10
Tin (Sn)	50	500	50	500
Zinc (Zn)	10	100	10	100

## Sample Preparation:

0.05g of tea/coffee sample powder was accurately weighed into a microwave vessels. Samples were kept for pre digestion after carefully adding ultra pure water, nitric acid (HNO<sub>3</sub>) and hydrochloric acid (HCl).The vessels were then heated in microwave digester system (MDS) under controlled temperature program (Table 3)

After digestion, samples were left to cool to ambient temperature and filled up using ultra-pure water in a standard volumetric flask (50 ml).

Pre spiked recovery studies were carried out at LOQ, & 10x LOQ levels (considering weight & dilution factor) by spiking samples with standard solution of metals and digested as like sample.

**Table 3: Microwave digester programme**

Steps	Ramp (min)	Temp (°C)	Hold time (min)
1	10	120	05
2	10	180	20

The digested solutions obtained are analysed on Shimadzu ICPMS-2030. Yttrium (Y) and bismuth (Bi) were added as internal standards (IS). Gold solution and IS solution are added at a final concentration of 100 ppb and 20 ppb respectively to blank, samples and linearity standards.

Note: Gold (Au) is added to stabilize Hg

## Calibration standard preparation

Sigma Aldrich 1000 ppm individual certified reference standards were used for preparation of intermediate stock solution. Calibration standard solutions were prepared by diluting intermediate stock solution to prepare concentrations as shown in Table 5 for tea and Table 6 for coffee.

## Analytical Conditions

A Shimadzu ICPMS-2030 coupled with auto sampler AS-10 (Figure 1) was used for analysis of measuring elements in tea and coffee matrix. The detailed instrument configurations and operating parameters are summarized in Table 4.



**Figure 1. ICPMS-2030 Inductively coupled plasma mass spectrometer with AS-10.**

**Table 4: Instrumental parameters**

Plasma torch	Mini torch
Radiofrequency	1.2 kW
Sampling depth	5 mm
Plasma gas flow rate	10 L/min
Auxiliary gas flow rate	1.1 L/min
Carrier gas flow rate	0.7 L/min
Collision gas	Helium
Collision gas flow rate	6.0 mL/min
Chamber	Cyclone chamber (electronically cooled)
Chamber temperature	5 °C

## Results

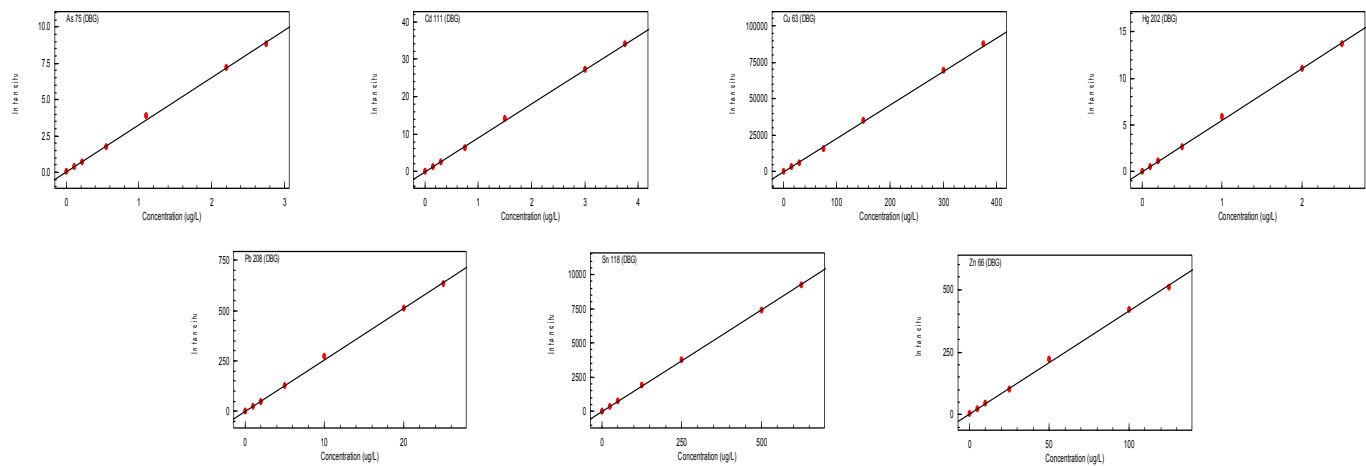
1. Quantitative results are given in Table 7 for tea and Table 8 for coffee.
2. To check the accuracy of the method for actual sample analysis, a spike recovery test was carried out. The four samples were spiked with respective elements. Using the ICPMS-2030, excellent spike recoveries were achieved for most elements in the spiked samples. All recoveries were within  $\pm 20\%$  for all elements.
3. The Calibration standard solutions showed good linear response (Figure 2 & 3) with correlation coefficient  $\geq 0.999$  for all elements.
4. %RSD of result obtained for 4 preparation (n=4) are less than 6%.

**Table 5: Calibration standard solution concentration (ppb) for Tea**

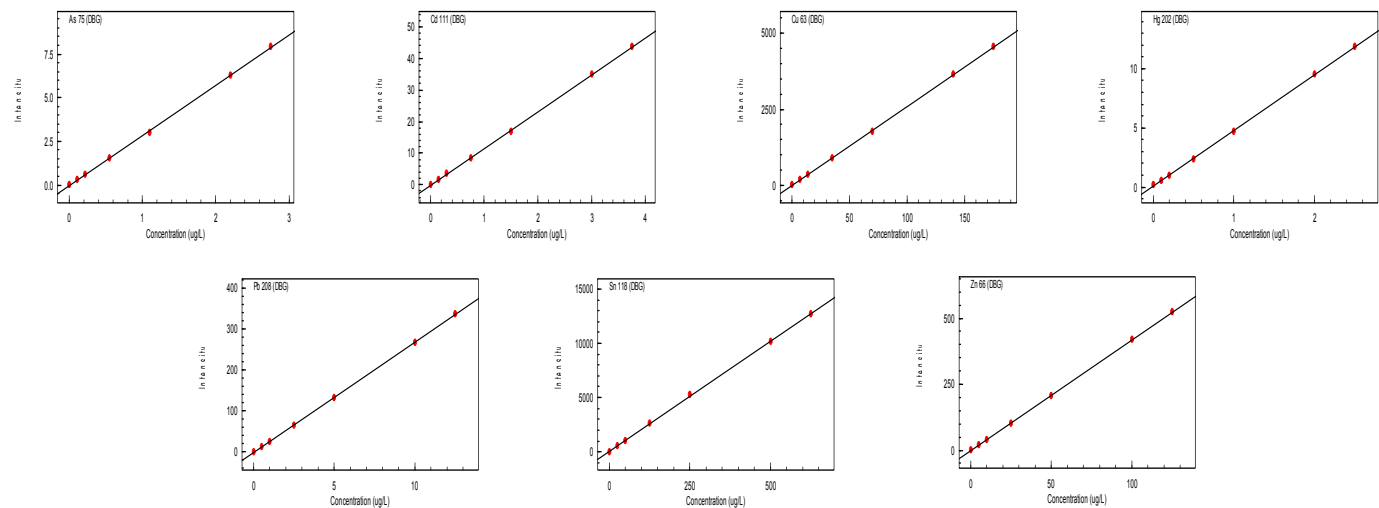
Element	As	Cd	Hg	Pb	Cu	Sn	Zn
Calibration Std Level 1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Calibration Std Level 2	0.11	0.15	0.10	1.00	15.0	25.0	5.00
Calibration Std Level 3	0.22	0.30	0.20	2.00	30.0	50.0	10.0
Calibration Std Level 4	0.55	0.75	0.50	5.00	75.0	125	25.0
Calibration Std Level 5	1.10	1.50	1.00	10.0	150	250	50.0
Calibration Std Level 6	2.20	3.00	2.00	20.0	300	500	100
Calibration Std Level 7	2.75	3.75	2.50	25.0	375	625	125

**Table 6: Calibration standard solution concentration (ppb) for Coffee**

Element	As	Cd	Hg	Pb	Cu	Sn	Zn
Calibration Std Level 1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Calibration Std Level 2	0.11	0.15	0.10	0.50	7.00	25.0	5.00
Calibration Std Level 3	0.22	0.30	0.20	1.00	14.0	50.0	10.0
Calibration Std Level 4	0.55	0.75	0.50	2.50	35.0	125	25.0
Calibration Std Level 5	1.10	1.50	1.00	5.00	70.0	250	50.0
Calibration Std Level 6	2.20	3.00	2.00	10.0	140	500	100
Calibration Std Level 7	2.75	3.75	2.50	12.5	175	625	125



**Figure 2. Linearity curves for targeted elements for Tea analysis**



**Figure 3. Linearity curves for targeted elements for Coffee analysis**

**Table 7: Average accuracy at LOQ, 10x LOQ (n=4 replicates) in Tea matrix**

Elements	LOQ spike %recovery	LOQ %RSD	10x LOQ spike %recovery	10x LOQ %RSD	Content in Sample (ppm)
As	90.0	4.8	92.4	1.8	BLOQ
Cd	92.1	2.1	90.7	4.4	BLOQ
Cu	88.5	4.0	95.3	2.6	BLOQ
Hg	103.8	1.8	102.0	2.7	BLOQ
Pb	97.6	2.6	101.3	3.6	BLOQ
Sn	99.4	2.1	90.2	4.4	BLOQ
Zn	91.6	9.5	93.3	3.0	20.5

**Table 8: Average accuracy at LOQ, 10x LOQ (n=4 replicates) in Coffee matrix**

Elements	LOQ spike %recovery	LOQ %RSD	10x LOQ spike %recovery	10x LOQ %RSD	Content in Sample (ppm)
As	95.1	1.4	97.2	1.7	BLOQ
Cd	92.2	2.3	93.2	0.2	BLOQ
Cu	92.5	1.6	93.7	0.6	BLOQ
Hg	94.3	2.8	98.6	0.5	BLOQ
Pb	97.8	2.1	98.0	0.6	BLOQ
Sn	102.3	2.9	94.0	0.6	BLOQ
Zn	88.7	1.6	95.5	0.5	BLOQ

**Note: BLOQ = Below Limit Of Quantitation**

## Conclusion

The analysis of a wide range of trace and minor elements in tea and coffee is easily performed using the Shimadzu ICPMS-2030. The octaplate technology in collision cell not only removes polyatomic interference but also provides high throughput and hence increasing sensitivity.

Good recovery results successfully proved validation of microwave-assisted sample preparation method.

The ICPMS-2030 is suitable for trace metal quantitation of tea and coffee, as well as related products. The analysis can be carried out at all stages of production to ensure product quality control and products that are free of toxic metals.

## References

- [1] Food Safety and Standards (Contaminants, Toxins and Residues) Regulations, 2011
- [2] AOAC Official Method 2015.01