

The Determination of Iodine in Food with the ELAN DRC-e ICP-MS

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Acknowledgements

The method development has been done by Fabien Bolle and Khalid Boutakhrit of the Belgian Scientific Institute of Public Health.

Introduction

Iodine is essential for the production of thyroid hormones. These hormones stimulate metabolism in the body, as well as mental growth and development. The recommended daily iodine intake is 0.1-0.2 mg, with the most common sources of iodine being fish, seafood, milk and food supplements. The determination of iodine in food has always been difficult due to the low concentrations (mg/kg), difficult sample preparation and the volatility of iodine.

This work demonstrates the ability of the ELAN[®] ICP-MS to measure iodine in food samples.

Experimental

Sample Preparation

Samples consisted of three certified reference materials: skimmed milk powder, cod, and mussel. Prior to weighing, the samples were mixed or slightly crushed; 0.25-0.5 g of sample was then added to PFA tubes, followed by 4.5 mL H₂O (Milli-Q) and 1 mL TMAH (25%).

After capping, the tubes were placed in a drying oven at 90 °C for 3 hours. After cooling, Milli-Q[®] water was added to a final volume of 10 mL. These solutions were then centrifuged at 3000 rpm for 15 minutes. If any visible particulates remained after centrifuging, the samples were then filtered. The resulting solutions can then be analyzed directly or with an extra dilution if high matrix concentrations are present.

Instrumental Conditions

The instrument used for this analysis was an ELAN DRC[™]-e ICP-MS. Instrumental operating parameters are shown in Table 1. All measurements were done in standard mode.

Calibration standards ranging from 5 to 20 µg/L were used, prepared in 0.5% (v/v) TMAH. After each standard and sample were analyzed, a 45 second rinse with of 0.5% TMAH was performed.

Table 1. Instrumental Conditions.

Spray chamber	Cyclonic
Nebulizer	Meinhard [®]
Sample Uptake Rate	1 mL/min
RF Power	1100 W
Plasma Gas Flow (L/min)	15
Nebulizer Gas Flow (L/min)	0.93
Auxiliary Gas Flow (L/min)	1.2
Dwell Time (ms)	50
Sweeps per reading	25
Replicates	3
Delay Time (s)	50
Wash Time (s)	45

Results

Table 2 shows the results of the analysis, along with the certified values for the samples. These results demonstrate that iodine can be accurately recovered in standard mode, indicating that there are no common interferences present. The limit of detection (3 times the standard deviation of the blank) in this method is 0.335 µg/L in the solution and 6.7 µg/kg in the original sample.

Iodine is known to have long washout times due to its volatility. This issue is overcome by rinsing with 0.5% TMAH between standards and samples. Iodine forms a non-volatile complex in basic environments, so using the TMAH rinse greatly reduces the washout time, compared to aqueous or acidic wash solutions.

Conclusion

This work demonstrates the ability of the ELAN to measure iodine in food samples using a simple digestion procedure. The problem of iodine washout was overcome by using a basic digestion medium and a basic wash between samples. The ELAN DRC operating in standard mode provided accurate results; the DRC mode was not needed due to the lack of common spectral interferences on iodine.

Table 2. Results for Iodine Analysis in Food SRMs.

	Sample	Measured Value (µg/kg)	Certified Value (µg/kg)
BCR 150	Skimmed milk (powder)	1237 ±73	1290 ±90
BCR 422	Cod	4889 ±456	4950 ±490
SRM 2977	Mussel	23540 ±750	26000 (n.c.)