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Determination of Aluminium in Food by ICP-MS: Influence of Microwave Digestion Parameters on the Recovery

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Aluminium (Al) is a metal that can be found in the crust of the earth and occurs naturally in drinking water and agricultural products. Humans are exposed to aluminium through the consumption of food and drinking water, and the use of consumer products and pharmaceuticals. Official food control requires validated methods for the analysis of aluminium in food and feed. The European Committee for Standardization has published a European Standard (EN) for the determination of Al by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) (EN 17264:2019).

In a proficiency test (PT) organised by the European Union Reference Laboratory for Metals and Nitrogenous Compounds in Feed and Food, high variations were observed between the participants' results for the Al concentration in a cocoa powder PT material. This observation was linked to the microwave digestion parameters used by the different participating laboratories. Four parameters were identified and their potential impact on the Al extraction was assessed: (1) Test portion size, (2) reagent(s) used for the digestion, (3) maximum temperature used and (4) digestion duration. This assessment was performed using the same PT material and three certified reference materials: NIST SRM 1566b Oyster tissue, NIST SRM 8439 Durum wheat flour and NBS 1572 Citrus leaves.

This work showed that varying test portion size between 0.2 and 0.5 g did not change recoveries. In contrast, the digestion temperature had a significant impact on the recovery of Al for the NIST SRM 1566b and NIST SRM 8439. The highest recoveries were obtained with maximum digestion temperatures of 240 and 280 °C. There was no significant difference between the Al recoveries for digestion times between 20 and 50 min. The reagents used had an effect on the recovery with the use of nitric acid with ultrapure water giving the highest recoveries.

These results confirmed those obtained from the analysis of the PT material by our laboratory and the PT participants. This suggests that a temperature of at least 240 °C for at least 25 min is required for a satisfactory microwave-assisted digestion, which is different from the recommendation in the EN (200 °C and 20 min). These findings provide important additional information to the EN method and will help the laboratories to improve their protocol for determining aluminium concentration in food samples by ICP-MS.

aluminium, food, feed, ICP-MS, proficiency test, EURL