Microwave Digestion for Determining Mercury

Fig. 1: speedwave MWS-3+ microwave digestion system

Fig. 2: Flexible, modular vessels

Introduction

Mercury has been in use since the Classical Antiquity and is very widespread today owing to its mobility. Still today, enormous quantities of the toxic element are being emitted into the environment by human beings, especially in combustion reactions. Mercury bioaccumulates in the food chain and primarily organic compounds (for example methyl mercury) accumulate in fatty tissue and hence reach the food chain.

Proof of the presence of mercury in food usually arises after wet chemical digestion by means of cold vapor AAS (CV AAS). In this process the art lies more in loss-free digestion of the sample material rather than in proving its presence. Loss frequently occurs owing to vaporization at high digestion temperatures. For this purpose complete digestion is essential to ensure that methyl or ethyl compounds are not converted into the more volatile organochlorine compounds during digestion.

Furthermore in quotes from older literature and individual standards [1] quartz glass is recommended as a material for vessels instead of PTFE because the surface roughness and porosity of the material can bring about lower results. If TFM-PTFE, especially isostatically compressed TFM-PTFE is used, these effects do not occur any more today and current standards also approve this material [2].

It is intended to demonstrate here that mercury from food samples can be analyzed reproducibly and with sufficient accuracy for years by means of isostatically compressed TFM-PTFE pressure vessels. The service life of the vessels significantly reduces the operating costs for the system.

Description of Apparatus and Experiments

All digestion processes were carried out in a speedwave MWS-3+ from Berghof Products + Instruments GmbH using DAP-60+ vessels (60 mL, 40 bars, 260°C). These are massive pressure vessels made from isostatically compressed TFM-PTFE. A pressure jacket and/or lid made from a different plastic material which is partly microwave-transparent and is not resistant to acid is not required. All of the vessels consist of only a few individual parts and are correspondingly fast and easy to lock or open. This is done manually without any special tools. Losses are successfully avoided by means of the pressure-resistant, permanent seal on the vessels by a TFM-PTFE lid with integrated sealing lip. In this case a burst disk serves as a safeguard against overpressure.

The system has been in use, including the vessels, for approximately six years now for routine analyses of food samples. 250–500 mg of dry sample material is weighed up for digestion and then digested in 15–20 min in an acid mixture consisting of 5 mL HNO_3 and 2 mL H_2O_2 at 180°C. The samples are flushed over with bidistilled water in a volumetric flask and diluted to 25 mL. The vessels are placed freestanding in the turntable so that they are able to give off heat di-

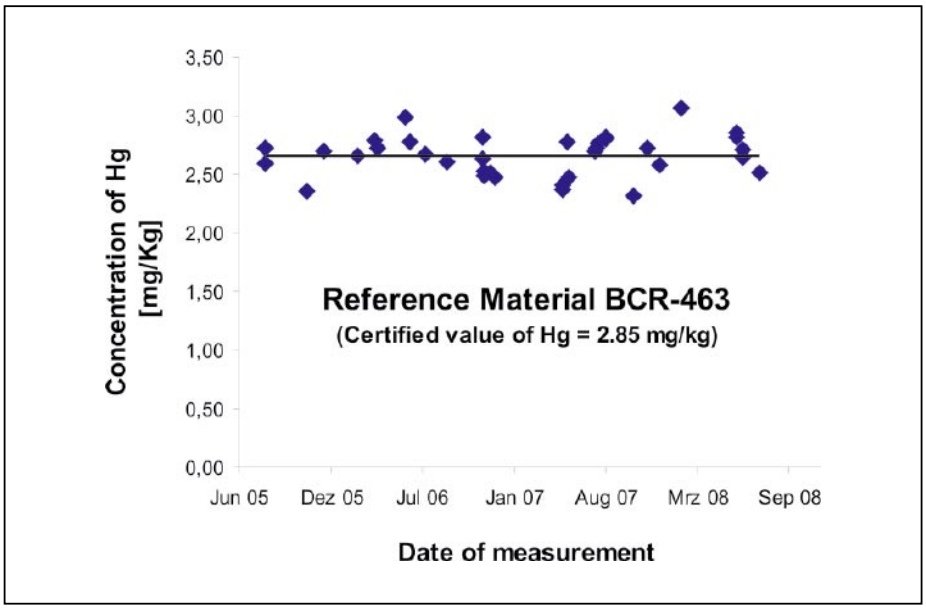

Fig. 3: Analysis of mercury from tuna following digestion in a speedwave MWS-3+ with DAP-60+ vessels (Reference Material BCR-463, certified value 2.85 mg/kg)

rectly into the oven air flow. In this way they are already cooled down sufficiently in 15 min that they can be opened without any danger.

The vessels are cleaned by simply flushing them with bidistilled water and diluted acid or in case of visible contamination with the help of a blind/test digestion.

Results

In principle it is possible to determine mercury and other heavy materials in vegetable matter with sufficient accuracy and reproducibility using the process described. This was proved by 6-fold determination of certified tomato leaves (reference material NIST 1573a) (table 1). The recovery rates for elements examined range between 94 and 106 % and the determination limits between 0.01 and 0.05 mg/kg in the dry matter.

Figure 3 displays the analysis results for mercury in certified tuna (reference material BCR-463) during the period from August 2004 to July 2008. The average recovery rate over this period is 93.3 % with a 6.4 % variation coefficient. The mercury present in this material is mainly methyl mercury (certified value 3.04 mg/kg). This makes it clear that the methyl mercury is completely decomposed by this digestion process and is determined by the subsequent determination process.

Furthermore the data prove that the digestion technique presented delivers reliable analysis results. It is not necessary to use expensive quartz glass vessels that might also break easily.

Conclusion

All analysts today endeavor to reduce the running costs for their processes. When using solid TFM-PTFE pressure vessels it is possible to achieve the considerable service life lasting three or even more years without having to accept any significant loss of quality. It means that the ongoing operating costs are reduced to the replacement of minor spare parts such as TFM-PTFE interior lids and burst disks.

Table 1: Analysis of heavy metal from tomato leaves using speedwave MWS-3$^+$ and DAP-60+ vessels (Reference Material NIST 1573a)

Element	Limit of determination [µg/g]	Result [µg/g]	Certified value [µg/g]	Recovery [%]
Cd	0.01	1.52 ± 0.01	1.52 ± 0.04	100
Cr	0.05	1.94 ± 0.23	1.99 ± 0.06	97.5
Ni	0.05	1.49 ± 0.10	1.59 ± 0.07	93.7
Hg	0.01	0.036 ± 0.004	0.034 ± 0.004	106

References

[1] DIN EN 13805, June 2002 and section 35 LMBG (Foodstuffs and Commodities Act, L00.00-19/1 December 2003

[2] e.g. DIN EN 1483, July 2007, EPA 3051a and EPA 3052

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