Short Note No 222/2016
Lead determination in instant noodles

Wet Digester B-440:
Acid digestion and ashing using the Wet Digester B-440 followed by spectroscopic lead determination

Lead occurs in the earth's crust and is present in air, soil and dust. It can be deposited on or absorbed by plants, which are used for food processing. Lead that gets in or on the plant cannot always be completely removed by washing or other food processing steps. Therefore, food products may contain minute amounts of the element - in the range of parts per billion (ppb).

Exposure to large amounts of lead, from whatever source, can affect the human body, e.g. the central nervous system, the kidneys and the immune system. In children, chronic exposure to lead, even at low levels, is associated with impaired cognitive function.

Here an effective method to prepare instant noodle samples for lead analysis by acid digestion and ashing is presented.

1. Introduction

The Food Safety and Standards Authority of India (FSSAI) is an agency of the Ministry of Health & Family Welfare, Government of India. According to FSSAI, for instant noodles included in the "foods not specified category", the permitted level of lead is 2.5 ppm. [1]

In 2015, Indian state governments found that some instant noodles contained far higher levels of lead than legally allowed. The aim of this application note was to determine lead from instant noodles as per the FSSAI method using the Wet Digester B-440.

2. Experimental

Equipment:
Wet Digester B-440 with 90 mL crucibles
Scrubber K-415 QuadScub [2]

Chemicals:

Sulphuric Acid concentrated 98 % AR Grade
Nitric Acid concentrated 69 % AR Grade
Hydrochloric Acid concentrated 37 % AR Grade
Distilled Water

Samples:
Instant noodles from Indian supermarket

Two experiments were carried out with instant noodles. The first experiment was to check the lead content in noodles. In the second experiment the samples were spiked with a standard solution to determine the recovery.

For digestion, 10 g of the homogenized sample was put into a clean crucible, 10 mL of 20 % sulphuric acid were added and then mixed and dried thoroughly. The 90 mL ceramic crucibles were placed in the Wet Digester at 250 ^{0}C. The temperature was increased at a rate of 50 ^{0}C / h to 500 ^{0}C where it was kept for about 6 hours (Figure 1). The ash should be white or brownish-red and essentially be carbon free.

If the ash contains carbon particles, the crucibles were removed from Wet Digester cooled for 5 min and the sides of the crucible were washed down with distilled water then 2 mL of HNO_3 were added carefully, the sample mixed well and dried thoroughly on a Wet Digester at 150 ^{0}C. The crucibles were kept on Wet Digester and the ashing was continued at 500 ^{0}C for another 30 minutes. Nitric acid treatment using 1 mL increment of HNO_3 was repeated until a white or brownish-red carbon free ash was obtained.

When a clean ash was obtained the crucibles were removed from the Wet Digester, cooled down to room temperature and 1 mL of HNO_3 and 10 mL of distilled water were added. The crucibles were heated at 150 ^{0}C on the Wet Digester until the sample ash dissolved. The content of the crucible was quantitatively transferred to a 50 mL volumetric flask. Then the crucibles were heated at 150 ^{0}C with 10 mL of 50 % HCl for 5 min and the solution was transferred again to the same volumetric flask. The volume was made up with distilled water to the 50 mL mark.

The blanks were prepared by adding 10 mL of 20 % sulphuric acid to the crucible. Both samples and blanks were identically treated.

These samples were stored and analyzed by inductively coupled mass spectroscopy (ICP-MS).

Figure 1. Digestion and ashing using the Wet Digester B-440.

3. Results

To achieve a good recovery rate, care has to be taken when H_2SO_4 is used for digestion. Because of $PbSO_4$ precipitation low recoveries might be obtained.

The first experiment revealed that the lead content in the noodle samples are below the permissible limit. Detected values were close to the blank value. The results are shown in Table 1.

Table 1. Results of lead determination in instant noodles Experiment 1.

	Sample weight [g]	Lead content* [ppm]
Blank	0	0.048
Sample 1.1	10.5462	0.068
Sample 1.2	10.1447	0.077
Sample 1.3	10.0694	0.099

*Lead content calculated considering dilution factor.

The second experiment shows the lead recovery of the spiked sample with a standard solution (Table 2). Lead content in Sample 2, a different noodle sample, was found higher than Sample 1, but also far below the permitted level for instant noodles.

Table 2. Recovery of lead spiked in sample Experiment 2.

	Sample weight [g]	Actual Conc.* [ppm]	Lead content [ppm]	Recovery [%]**
Blank	0	0	0.048	
Sample 2	10.0279	0	0.245	
Sample + 5 ppm Spike	10.5276	4.749	4.15	86.37
Sample + 10 ppm Spike	10.0428	9.999	9	89.60

*Actual Conc. is concentration of lead in sample after spiking standard solution

**Recovery calculated after subtracting blank value and considering dilution factor.

4. Conclusion

Lead determination by ICP-MS using the Wet Digester B-440 for ashing gives reliable results with a recovery rate above 85 % and is in accordance to the FSSAI regulation. For the measured instant noodle samples the lead content was far below the permissible limit.

Lead determination using the Wet Digester B-440 is reproducible, well convenient due to programmability, safe and economic.

5. Acknowledgement

Geo-Chem Laboratories (P) Ltd is kindly acknowledged for the ICP-MS experiments and the fruitful discussions.

6. References

[1] FSSAI Manual of Methods of Analysis of Foods Metal. *Lab Manual 9.*

[2] AN 95/2012 Determination of Trace Metals by Aqua Regia Digestion according to ISO 11466