TXRF

TXRF spectrometry applied to food (II): Rapid screening for nutrition-relevant elements in fish

Application Note # XRF 450

Introduction

In order to check the analytical performance of TXRF spectrometry a certified reference standard (DORM-3, fish protein, National Research Council of Canada) was analyzed. An additional feasibility study was then performed with a variety of typical fish and vertebrate samples, which were purchased in a grocery store:

- Fish samples: mussels, sea bream, cuttlefish
- Vertebrate muscle samples from chicken, cattle, horse

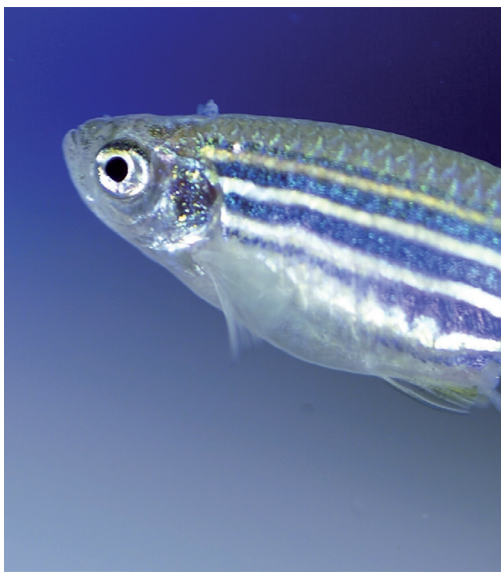

Figure 1
Fish sample

Sample preparation

The preparation of the samples for fast element screening is described in Figure 2 (left). Grinding was performed in a bench top ball mill (Retsch MM400) with Zr jar for 3 min at 50 Hz. For internal standardization a Se standard solution was added (final concentration 4 mg/kg). Microwave digestion was applied to part of the samples for comparative TXRF analysis (Figure 2, right). The weighed samples were digested in 10 ml HNO_3 / 1 ml H_2O_2 and filled up to a volume of 25 ml. After internal standardization with Se, the samples were measured with TXRF. In addition, the digested samples allowed the verification of the element concentrations through ICP-MS measurements (using Bruker's aurora M90).

Results

DORM-3 standard

Element concentrations of either suspended or digested DORM-3 standard sample were analyzed with TXRF and compared with ICP-MS data (Figure 3). The comparison confirms the accuracy of TXRF results.

Fish and vertebrate samples

Real samples contained elements over a wide concentration range (Table 1) and sometimes close or below the limit of detection (LOD) for TXRF, which influenced the quality of the results (Figure 4):

- **Na, Mg**
  Concentration values are sensitive to sample thickness and distribution due to absorption of the low energy fluorescence radiation. However, a fast screening even of suspended samples is possible.

- **K, Ca**
  Excellent concordance with ICP-MS for digested samples. Due to high standard deviation sample suspension is suitable for screening only.

- **Ti, V, Cr**
  High spectrum background and line overlaps impede accurate quantification, but element screening is still possible.

- **Mn, Fe, Ni, Cu, Zn, As, Se**
  Most TXRF results are accurate for both sample preparation procedures. Stronger deviations occur in case of concentrations close to the LOD.

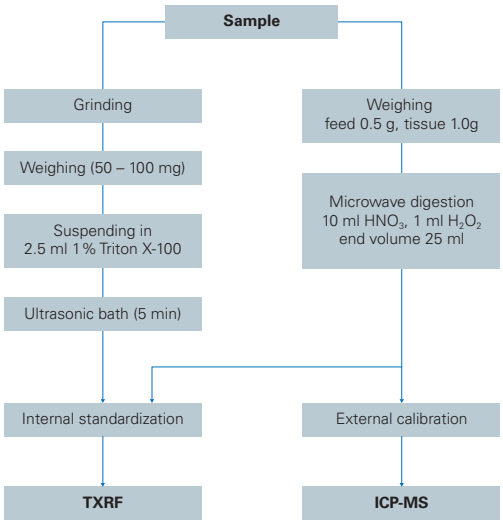

Figure 2
Methods of sample preparation of fish and vertebrate samples for TXRF and ICP-MS analysis

Element	Concentration range (mg/kg)
Na	500 – 11000
Mg	220 – 2100
K	430 – 11700
Ca	40 – 11800
Ti	0.16 – 4
V	0.02 – 700
Mn	0.08 – 3
Fe	2.7 – 95
Ni	0.01 – 2.5
Cu	0.38 – 140
Zn	4 – 105
As	0.01 – 4

Table 1
Element concentration range in fish and vertebrate samples determined by ICP-MS

Conclusion

The analysis of certified reference fish standards and typical fish and vertebrate samples have clearly demonstrated that rapid screening of macro and trace elements with TXRF is possible. Therefore, TXRF is a powerful complementary tool to more sensitive, but highly sophisticated ICP-MS spectrometry.

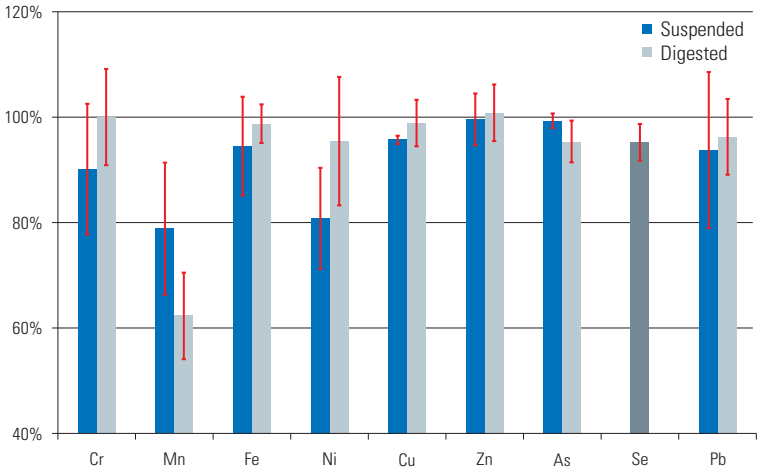

Figure 3
TXRF recovery for DORM-3 (concentration of gray elements is not certified)

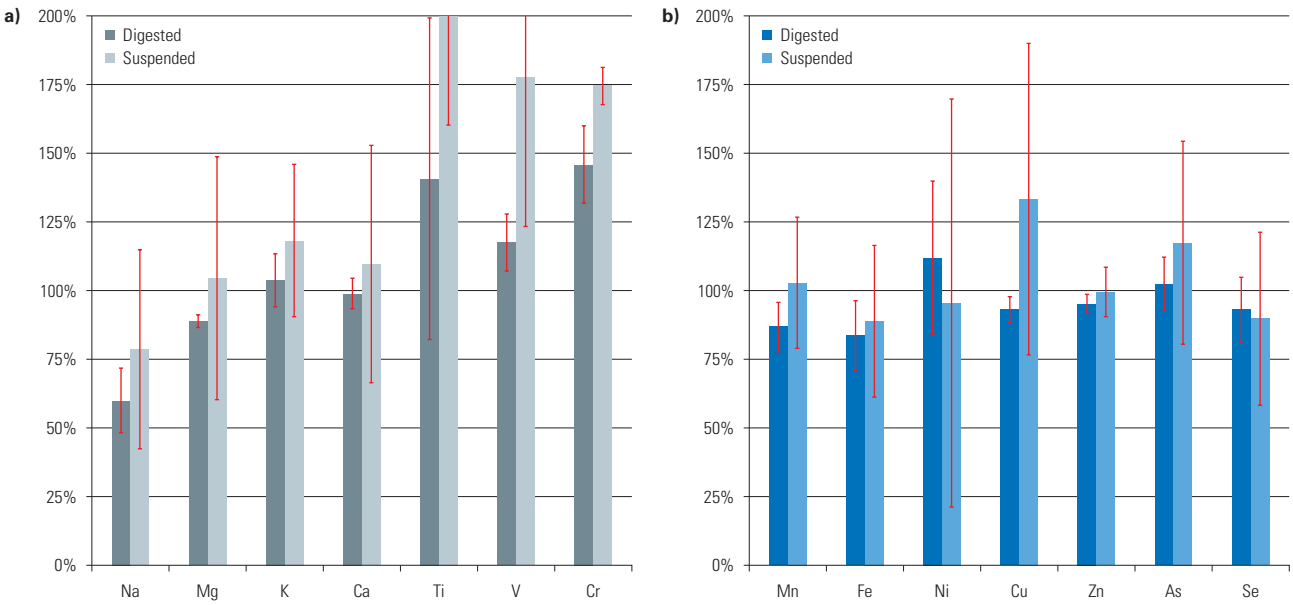

Figure 4
Recovery of light elements (a: Na to Cr) and metals (b: Mn to Se) in fish and vertebrate samples

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