



Lab Report XRF 466

S2 PICOFOX

Rapid and cost-effective analysis of lead in tea drinks by TXRF spectroscopy

Introduction

The analysis of lead in beverages like tea drinks is an important task due to the toxicity of this element. Short-term exposure to high levels of lead can cause brain damage, paralysis (lead palsy), anemia and gastrointestinal symptoms. Long-term exposure can cause damage to the kidneys, and the reproductive and immune systems, in addition to effects on the nervous system.

In most countries, the legal limits for Pb in food and beverages are typically between 0.1 and 0.2 mg/l. Failing to comply with these limits can lead to significant financial damages for manufacturers caused by fines and import rejections.

Due to the strong matrix of the tea drinks containing several wt% of sugar, analytical techniques like atomic adsorption (AAS) or inductively coupled optical emission spectroscopy (ICP-OES) require an expensive and laborious acid digestion of the samples.

Total reflection X-ray fluorescence (TXRF) spectroscopy, however, is an easy analytical technique for trace element analysis. In this lab report its suitability for the analysis of lead in tea drinks is shown.

It's tea time



Fig. 1: Iced tea is a popular beverage, especially in summer

Sample preparation

Representing a usual tea sample, a lemon flavored tea drink was obtained at a supermarket. The tea was spiked with a Pb mono-element standard solution in order to prepare samples with 0 (blank), 10, 50, and 200 µg/l Pb. For internal standardization 500 µg/l gallium were added to all samples.

After thorough homogenization 10 µl sample were pipetted onto quartz glass sample carriers and dried at 40 °C on a hot plate. All samples were prepared as triplicates.

For determination of the method detection limit (MDL) and the limit of quantification (LOQ) according to EPA (EPA 40 CFR Part 136, APPENDIX B Revision 1.11), the sample containing 50 µg/l of Pb was prepared sevenfold. This sample was also prepared on disposable acrylic glass carriers which are often used for routine laboratory applications.

All samples were measured using a S2 PICOFOX 800 with the parameters shown in table 1.

Results

The measurement results are summarized in table 2. Lead concentrations of 50 µg/l or higher can easily be analyzed in the samples on the reusable quartz glass carriers as well as on the disposable acrylic glass carriers.

Measured lead concentrations of 10 µg/l or below are close to the 3σ detection limit (LLD) and the reproducibility of the measurements is not sufficiently reliable. However, considering the calculated values of 6.4 µg/l for the MDL and of 20.5 µg/l for the LOQ, the safe determination of lead in tea drinks even at low concentrations is possible.

Conclusion

The measurement results for the determination of the lead concentration in tea drinks proves the low cost benchtop TXRF spectrometer an ideal solution for this analytical task.

Table 1 Measurement parameters

S2 PICOFOX 800	
Parameter	Specification
X-ray tube	Micro focus, Mo target
High voltage	50 kV
Current	1000 µA
Detector	XFlash® 6 60 SDD
Active area	60 mm ²
Measurement time	1200 s

Table 2 Measurement results for tea drinks spiked with Pb

Sample/ spiked Pb conc.	measured Pb conc.	s	LLD
Blank	7.4	2.3	3.9
10 µg/l	6.2	2.3	3.6
50 µg/l, quartz disc	48	2.1	2.4
50 µg/l, acrylic glass disc	51	1.6	2.6
200 µg/l	193	3.9	2.4

The analysis does not require laborious digestion procedures or external calibrations as samples can be prepared directly after addition of an internal standard. TXRF is a simultaneous method and other toxic elements like Cr, Ni, As, and Hg are also automatically measured and quantified.

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