ZINC DETERMINATION

by ABS, AAS and HPLC-ion

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Introduction

Zinc sulphate is an inorganic salt that is essential for human health. The salt has a wide variety of useful applications. For example, it can be consumed as a vitamin supplement and on the other hand prevent extended moss growth on roofs.(1)
According to Ph. Eur. And USP, photometric titration is the most suitable method to determine zinc sulphate. In this research project, this claim was investigated by conducting a comparative study of photometric titrations (ABS) with atomic absorption spectrophotometry (AAS) and high-performance liquid chromatography-ion (HPLC-ion).

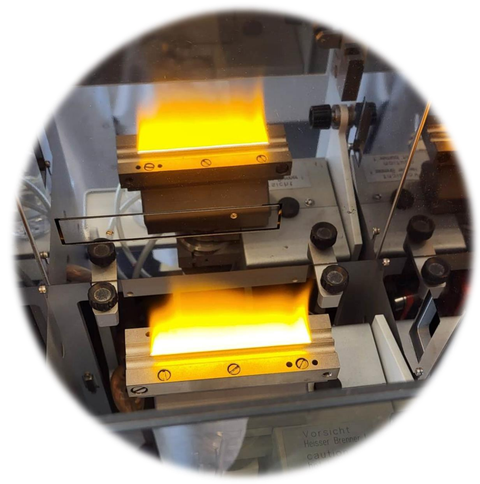
With the aim of performing this study, two unknown samples were prepared. The PRL-sample is a purchased zinc sulphate supplement with an undetermined zinc concentration, whereas the self-prepared zinc sample has a known zinc concentration. The self-prepared sample is analysed in three solvents: Milli-Q, distilled water and tap water. The PRL-sample was analysed in Milli-Q solvent. To determine the most efficient method for zinc determination, the parameters of analysis time, cost, solvent and other performance characteristics (accuracy and confidence interval) are compared.

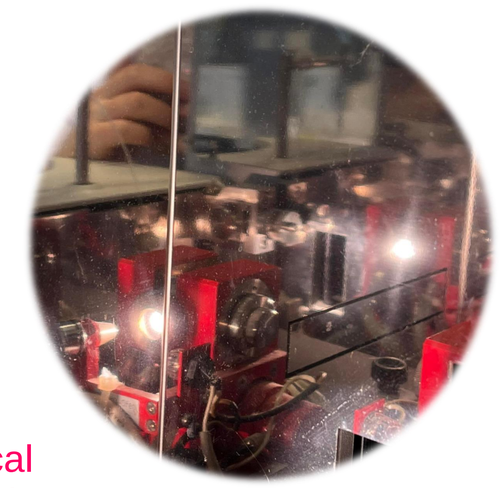
Materials and methods

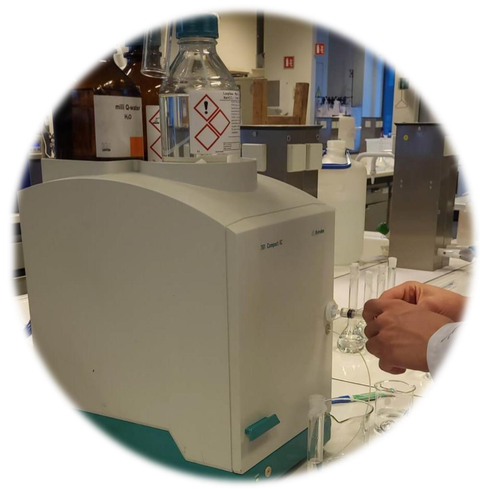
- ABS - Metrohm Eco Titrator

  The technique is performed using a standardised EDTA solution as titrant and Erio-T as indicator. Measurements were performed using a Metrohm Optrode (610 nm). The equivalence point was determined in the titration curve with the measured voltage as a function of volume of EDTA .
- AAS - novAA 400 S Analytik Jena

  In this method, zinc ions are atomised into zinc atoms by a graphite furnace or flame. The measurement principle is based on an absorbance measurement where a reduction in light intensity is observed. A calibration line is established of the measured absorbance as a function of the concentration of zinc ions in solution.
- HPLC-ion - Metrohm 761 Compact IC
  This principle is based on an equilibrium reaction between the sulphate ions in the mobile phase and the resin in the anion exchanger column. When the sulphate ions reach the suppressor column, a conductivity measurement is performed. A chromatogram is established with a peak area corresponding with the change in electric current. A calibration line is plotted with the peak area as a function of the concentration of sulphate ions in solution.







Results and discussion

Table 1: PRL-sample – Milli-Q analysis: AAS-flame, HPLC-ion and ABS

Average mass of zinc (g)			
	1,326	±	0,0300
	1,716	±	0,120
	1,323	±	0,100

In comparison with AAS-flame and ABS, HPLC-ion demonstrates a significant deviation on the average mass of zinc. During the analysis, multiple dilution factors were applied on the PRL-sample to fit the sample in the calibration line, resulting in dilution errors and a bigger dispersion.

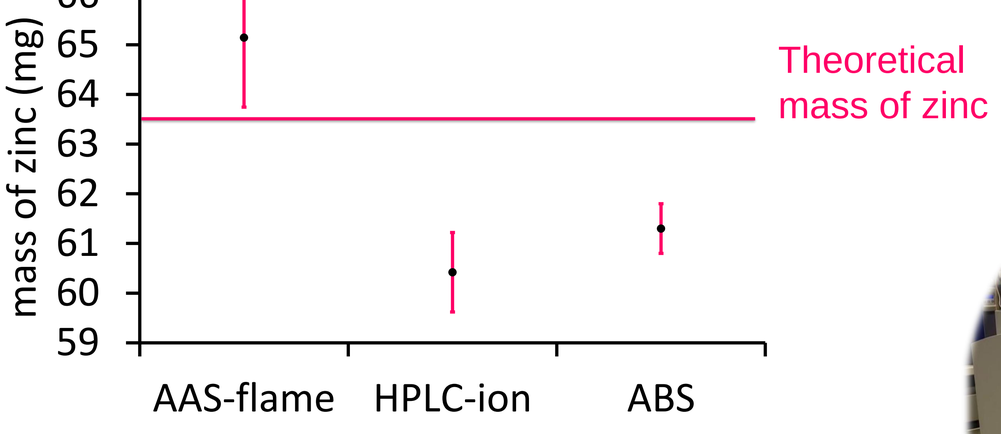


Figure 1: Self -prepared sample – Milli-Q analysis

Table 2: Self-prepared sample – Milli-Q analysis- Relative error on theoretical mass of zinc (%)

Relative error (%)		
	AAS-flame	2,445%
	HPLC-ion	4,985%
	ABS	3,601%
 (2)	AAS-flame	6,302%
	HPLC-ion	4030%
	ABS	21,23%
 (3)	AAS-flame	208,8%
	HPLC-ion	4,536%
	ABS	2,462%

The relative error indicates the extent to which the average mass of zinc ions in the self-prepared sample deviates from the theoretically calculated mass of zinc ions.

With ABS, the same titrant and primary standard solutions were used for each lab and the self-prepared sample did not have to be diluted. HPLC-ion and AAS-flame, on the other hand, both analysed a diluted sample solution. In addition, both methods used standard solutions prepared from a diluted stock solution to establish a calibration line. Therefore, the average obtained mass of zinc with HPLC-ion and AAS-flame demonstrates a higher dispersion.

Conclusion

It was concluded that with ABS the best results were obtained for the analysis of the self-prepared sample in Milli-Q solvent. The method has a minor relative error and low dispersion. The results of the PRL-sample are not included in this decision, since the average determined masses of zinc in the PRL-sample are significantly different from the theoretically calculated masses in the PRL-sample. In addition, different dilution factors were applied within each method to fit the PRL-sample in the calibration lines, which indicates unreliable results.

References

(1) Metrohm AG. Metrohm. [Updated 2022 november 9]. Available from https://www.metrohm.com/en/applications/application-notes/aa-t-001-100/an-t-090.html
(2) PNGEG. [Updated 2022 december 16]. Available from https://www.pngegg.com/en/png-zasdf
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