Method Development for the Determination of Heavy Metals such as Copper, Iron, Manganese and Zinc in Aluminum Alloys by ICP-OES, based on wastewater analysis method

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Abstract
The determination of Heavy Metals such as Copper, Iron, Manganese and Zinc in Aluminum alloys is usually achieved following ASTM E3061-17 Method. Alternatively, for time and cost saving, the laboratory's EN ISO 11885:2009 method for wastewater analysis was appropriately modified producing satisfactory results. Parameters such as matrix effect, wavelength, plasma conditions, calibration standards and dilution conditions of the alloy were examined for the accurate measurement of these elements in Aluminum alloys (e.g. CRMs, according to their Certified Values).
Keywords: Aluminum alloys, Heavy Metals, ICP-OES, Modified Wastewater Method, Matrix Effect.

1. Introduction

Aluminum is one of the most widely used metals in the world presented in an incredible range of industrial and household products. Massive types of Aluminun alloys are produced by adding elements and impurities during the production process in order to produce materials with specified properties.

Chemical analysis of such alloys is described in ASTM E3061-17 (Standard Test Method for Analysis of Aluminum and Aluminum alloys by Inductively Coupled Plasma Atomic Emission Spectrometry ICP-AES) and can be applied for the determination of 32 elements including Copper, Iron, Manganese and Zinc with a sufficient application range. Depending on the element of interest, the standard suggests five different methods of dissolution. In addition, it provides guidelines for the preparation of Aluminum Stock Solution for Matrix Matching of standards and samples in order to avoid matrix effects.

In our laboratory, ICP-OES is used for the simultaneous determination of heavy metals in wastewater based on EN ISO 11885:2009 (Water quality – Determination of selected elements by inductively coupled optical emission spectrometry ICP-OES). The method for extracting trace elements from water samples is based on EN ISO 15587-1:2002 (Water quality – Digestion for the determination of selected elements in water – Part1: Aqua regia digestion). Necessary adjustments from the guidelines of ASTM Standard to interlaboratory method analysis were made in order to save time and reduce the cost of the procedure.

2. Experimental

2.1. Instruments and reagents

- ICP-OES PerkinElmer Optima 7300DV instrument equipped with WinLab32 4.0 software
- Vertical drill
- Al Certified Reference Material (CRM for stock solution)
- CRM for Calibration
- CRM to ensure the results' validity
- Hydrochloric acid fuming 37 % w/w
- Nitric acid 65 % w/w
- Ultrapure Water 18.2 MΩ*cm
- Pure Argon 99.999 %
- Pure Nitrogen 99.999 %

2.2. Drilling

Representative samples were obtained by vertical drilling machine using titanium drill bits.

2.3. Instrument Set-up

The parameters' set-up of gas flows, generation power and sample introduction flow was based on interlaboratory ISO method. The suggested wavelengths by the ASTM method are in complete agreement with the corresponding wavelengths of ISO method.

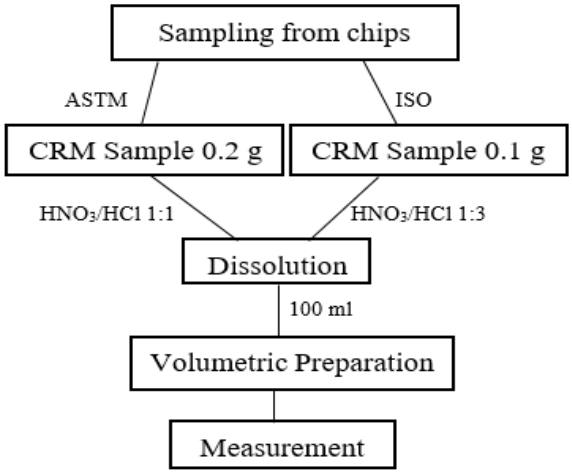
2.4. Calibration

An eight-point calibration curve with the same concentration but different matrix was designed for both methods. Regarding the ASTM method, matrix-matched calibration solutions were prepared containing the approximate amount of aluminum. The calibration standards of the ASTM method contain 5% HNO_3 and

1% HCl while the ISO method standards contain 5% HNO_3.

2.5. Sample Dissolution

The aluminum samples were divided into two groups (Figure 1). The ASTM method was applied in the first



group and the ISO method in the second group.
Figure 1. Analytical procedure for Sample Dissolution

3. Results and Discussion

The results and recoveries based on both ISO and ASTM methods are presented in Table 1 and Figure 2.

Table 1. Results and recovery percentage of each element for both methods

Element	BS EN ISO 11885:2009		ASTM E3061-17		CRM Certified Value
	Results	**Recovery**	**Results**	**Recovery**	
	% w/w	**%**	**% w/w**	**%**	**% w/w**
Cu	0.094	101.0	0.093	99.5	0.0931
Fe	0.405	103.6	0.397	101.6	0.391
Mn	0.490	98.99	0.490	99.0	0.495
Zn	0.170	107.4	0.157	99.4	0.158

As it is presented in Figure 2, satisfactory recovery percentage for all the elements of interest using both ISO and ASTM methods are achieved.

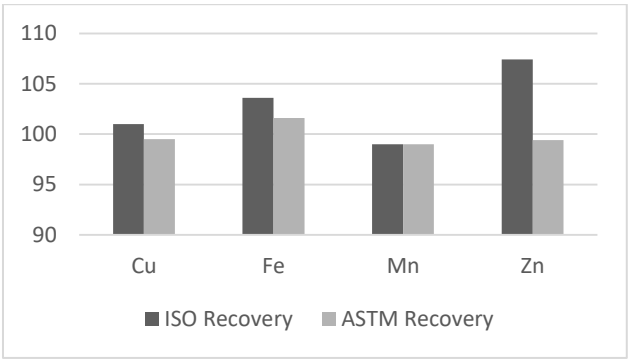


Figure 2. Graph of recovery percentage of each element for both methods (%)

4. Conclusion

Both methods (ISO-ASTM) provide similar results and their recoveries are in the range 90-110% thus being satisfactory. Based on the above, both methods are equivalent, hence ISO method can be successfully applied for the determination of Copper, Iron, Manganese and Zinc in Aluminum alloys. Due to the fact that no changes in consumables and instrument setup were made, the goal of saving time and reducing the cost of the procedure is achieved.

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