

# Study of Matrix Effects on Determination of Trace Elements in Aluminium Using Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) (II) Criterion for Selection of Spectral Lines

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**Summary:** Selection of spectral lines for determination of twelve impurities in aluminium has been considered in light of the true detection limits as the basic criterion. The lines with lowest true detection limit values are recommended for determination of these elements in the aluminium matrix.

## Introduction

Aluminium and its alloys are, at present considered important structural materials due to their wide spread use for fabrication of items of varied nature [1]. All aluminium parts and components, whether they are for domestic use for fabrication of window and door frames or employed as reactor materials, must conform to specified chemical composition and qualified for the envisaged use.

A number of instrumental techniques have been applied for determination of alloying and the impurity elements present in aluminium and its alloys [2-3]. Considering the speed of analysis and the detection limits ICP-AES technique is one of the most favoured choice as it could be easily adapted for multi-elemental analysis of simple and complex matrices [4]. However accurate analysis of impurities in a material would require well-documented information on matrix effects. This is considered to be valid even when using ICP-AES [5] as it is now positively confirmed that this technique is equally subjected to matrix effects contrary to earlier reports [6,7]. There are many factors which could lead to matrix effects in ICP-AES and spectral interferences are considered to be

one of the basic and important element for delineation of such effects. These may become dominant especially when traces of impurity elements are to be analyzed in the main matrix.

Aluminium is considered, in general, a noble matrix and does not present a complex spectral interference picture as one may be confronted with line rich spectra of uranium [8-10]. Yet due consideration had to be given to spectral interferences due to the Al matrix while selecting spectral lines of the impurity analyte elements present. Such exercise is considered imperative for an accurate analysis methodology.

In a recent study matrix effects of Al specially related to spectral interferences and selection of spectral lines for trace impurities have been considered on the basis of line sensitivity, line width and matrix contribution to the enhancement of background [11]. In the present study twelve analyte elements B, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Ni, Si and Zn were selected for investigation with a view to establishing criterion on the basis of true detection limits for selection of the analyte spectral lines. Suitable lines with lowest true

detection limit values are recommended for determination of these impurities in Al matrix.

### Results and Discussion

The selection of spectral lines of the analyte elements, being determined in the presence of a matrix is generally considered on the basis of low background, no direct or wing overlap and low matrix effect to the change of line sensitivity. To accomplish this task knowledge of the blank solution background and line spectra of the analyte element of interest with and without the presence of the particular matrix, within a selected band width or spectral window would be required. In line with this approach a recent study on selection of analyte element spectral lines in Al matrix has been described [11]. The spectral lines were selected after a judicious assessment of matrix interferences. Parallel to this approach line selection could also be followed using true detection limit value as a criterion for such selection as suggested by Bouman *et al.* [12]. Overall 47 spectral lines of the twelve analyte elements were studied. True detection limits ( $C_L$ ) were calculated following the relation.

$$C_L(\text{True}) = 2/5 \frac{S_i}{S_A} C_i + C_{L(\text{conv})}$$

where  $S_i$ ,  $S_A$ , and  $C_i$  represent interferent matrix sensitivity, analyte sensitivity and matrix concentration respectively. The  $C_{L(\text{conv})}$  to be defined and calculated as under:

$$C_{L(\text{conv})} = 0.01 \times 2\sqrt{2} \times \text{RSDB} \left[ \text{BEC} + \sum \frac{S_{ij}}{S_A} C_{ij} + \sum \frac{S_{wj}}{S_A} C_{ij+..} \right]$$

Where RSDB, BEC,  $S_{ij}$ ,  $S_{wj}$  and  $C_{ij}$  represent relative standard deviation of blank, background equivalent concentration, interferent sensitivity (line overlap), interferent sensitivity (wing overlap) and the interferent concentration respectively. RSDB was considered to be 1% in all these calculations.

The true detection limit values calculated by extracting data from the spectral scans are given in Table-1. Some of the detection limits so obtained may represent values relatively higher than one

Table-1: True Detection Limit

S.No.	Element	Spectral Line	True Detection Limit (ng ml <sup>-1</sup> )
1)	B	249.678(I)	24
2)		249.773(I)*	12
3)	Ca	317.993 (II)	4.0
4)		393.367 (II)*	0.8
5)		396.846 (II)	0.9
6)		422.673 (I)	36
7)	Cd	214.438 (II)	143
8)		226.502(II)	64
9)		228.802 (I)*	8
10)	Co	237.862 (II)	600
11)		238.892 (II)*	15
12)		345.350 (I)	103
13)	Cr	205.552 (II)	273
14)		206.149 (II)	370
15)		267.716 (II)*	8
16)		283.562 (II)	14
17)		284.325 (II)	15
18)	Cu	213.598 (II)	801
19)		224.700 (II)	25
20)		324.754 (II)*	12
21)		327.396(I)	22
22)	Fe	238.203(II)	10
23)		239.562 (II)	20
24)		259.939 (II)*	11
25)		263.105 (II)	47
26)	Mg	279.553 (II)*	4
27)		280.270 (II)	5
28)		285.213 (I)	6
29)	Mn	257.610(II)	9
30)		259.373 (II)*	1
31)		260.569 (II)	2
32)		280.106(I)	74
33)		293.306 (II)	12.2
34)	Ni	221.647 (II)	92
35)		231.603(II)*	21
36)		341.476 (I)	59
37)		352.454 (I)	55
38)	Si	250.690 (I)	66
39)		251.432 (I)	24
40)		251.611 (I)*	27
41)		251.920 (I)	381
42)		288.158(I)	42
43)		252.412 (I)	39
44)		252.851 (I)	31
45)	Zn	202.551 (II)	106
46)		206.191 (II)	214
47)		213.856 (I)*	63

\*Line recommended for determination of the analyte element.

may be expecting for some of the analyte elements in this matrix. These values however provided an empirical criterion for selection of analyte element spectral lines and may be understood as applicable to this extent only. A line with the lowest value of the true detection limit, invariably represent the best choice for determination of that element in Al matrix. Following this rigorous approach the spectral lines finally recommended have also been marked in Table-1 and these happen to be the same

as selected through the relatively simpler approach [11].

### Experimental

An ICP-AES system consisting of JY-38 monochromator with 3600 groves  $\text{mm}^{-1}$  holographic grating and JY-2300, 40.68 MHz plasma generator of Jobin Yvon, France was employed in this study. For sample introduction a Meinhard type concentric nebulizer with 1.5  $\text{ml min}^{-1}$  flow rate was used. The power was adjusted to 1.0 KW and all observations were carried out at a viewing height of 15 mm above the load coil. The entrance and exit slits were fixed at 25 and 30 microns respectively. Argon gas flow rates for the plasma, intermediate and nebulizer were adjusted to 12.0, 0.35 and 0.8  $\text{L min}^{-1}$  respectively.

The following solutions were prepared using Analar grade chemicals and used throughout this study. However, the analyte element solutions were prepared using the Spex 1  $\text{g L}^{-1}$  standard elemental solutions.

- \* Blank solution, 0.1M  $\text{HNO}_3$  in double distilled deionized water.
- \* Analyte solutions in 0.1M  $\text{HNO}_3$  containing 1  $\text{mg L}^{-1}$  of the analyte element in each case.
- \* Aluminium matrix solution containing 10  $\text{g L}^{-1}$  of Al in 0.1 M  $\text{HNO}_3$ .

The instrument was operated in scanning mode at the optimum operating conditions. For each

selected spectral line three scans, one each for the blank, Al matrix and analyte solution were recorded. The data obtained was used to calculate the background equivalent concentration (BEC), analyte sensitivity ( $S_A$ ) and matrix sensitivity ( $S_M$ ) factors.

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