

MATERIALS ANALYSIS

DETERMINATION OF ELEMENTS IN PRE-ALLOYED METAL POWDER SAMPLES USING THE AGILENT 4200 MP-AES



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ABSTRACT

This solution note describes a method for the quantitation of percentage level elements in pre-alloyed metal powder samples using the Agilent 4200 MP-AES. The note compares results from the 4200 MP-AES with conventional flame AA and shows the unique advantages of the 4200 MP-AES over flame AA. The spiking studies demonstrate excellent recovery results for all elements.

INTRODUCTION

Metal bonded cutting tools have been around for well over half a century, and the many and varied metal binders have been the subject of much empirical research. These tools are utilized in diamond and other precious sample cutting and processing. The required properties of these tools are slightly unusual but critical. The binder metal should be easy to sinter, hold sample particles to be processed firmly and be sufficiently wear resistant to grip each sample until this superhard component is worn away, but soft enough to expose new sample

Research into diamond impregnated tools (DIT) for stone and civil engineering applications has targeted the development of pre-alloyed powders, which has increased the productivity and reduced the cost process of these types of tools. Pre-alloyed powders for DIT, have obtained a significant position in the market. These are mainly based on Fe/Co/Cu/Sn systems, which are present at high ppm to percentage level concentrations.

The accurate and precise combination of these metals is of prime importance in producing tools with the desired properties. Conventional flame atomic absorption spectroscopy (AA) is the standard solution for the quantitation of such elements at higher ppm to percentage level analysis, but with Agilent's unique 4200 Microwave Plasma – Atomic Emission Spectrometer (MP-AES), the analysis can be performed at high metal concentration levels in alloy and metal powder samples, with minimal sample preparation steps, high sample throughput and at lower sample analysis cost.



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ANALYTICAL TECHNIQUE

Instrumentation:

All measurements were performed using an Agilent 4200 MP-AES with nitrogen plasma gas via an Agilent 4107 Nitrogen Generator equipped with the standard sample introduction system, consisting of an inert OneNeb nebulizer, double pass cyclonic spray chamber and an easy fit torch. The Agilent SPS-4 autosampler was used to deliver samples to the instrument allowing for unattended operation. Spectral and background interferences could be simultaneously and accurately corrected using the MP Expert software. Method parameters are given in Table 1.

Parameter	Setting/Value
Replicates	3
Pump rate	15 rpm
Sample uptake delay	35 seconds
Rinse time	30 seconds
Stabilization time	15 seconds
Read Time	3 seconds
Nebulizer flow	0.65-0.80 (L/min)
Fast Pump during uptake and rinse	On (80 rpm)
Background correction	Auto
Sample pump tubing	Orange/Green
Waste pump tubing	Blue/Blue

Table 1. Agilent 4200 MP-AES operating parameters.

Sample and Standard Preparation:

The calibration standards for Fe, Cu, Co and Sn were prepared using single element NIST traceable standards in 2% HNO₃ and 10% HCl. The samples were prepared in multiple replicates. 100 mg of each sample was weighed and prepared in 2% HNO₃ and 10% HCl, heated on a hot plate, transferred to a 100 ml volumetric flask and made up to the mark using Milli-Q water. The samples with known concentrations of spiked elements were prepared simultaneously. Pure grade acids were used. The samples were then diluted and analyzed. Elemental concentrations of the samples were determined against the calibration graphs.

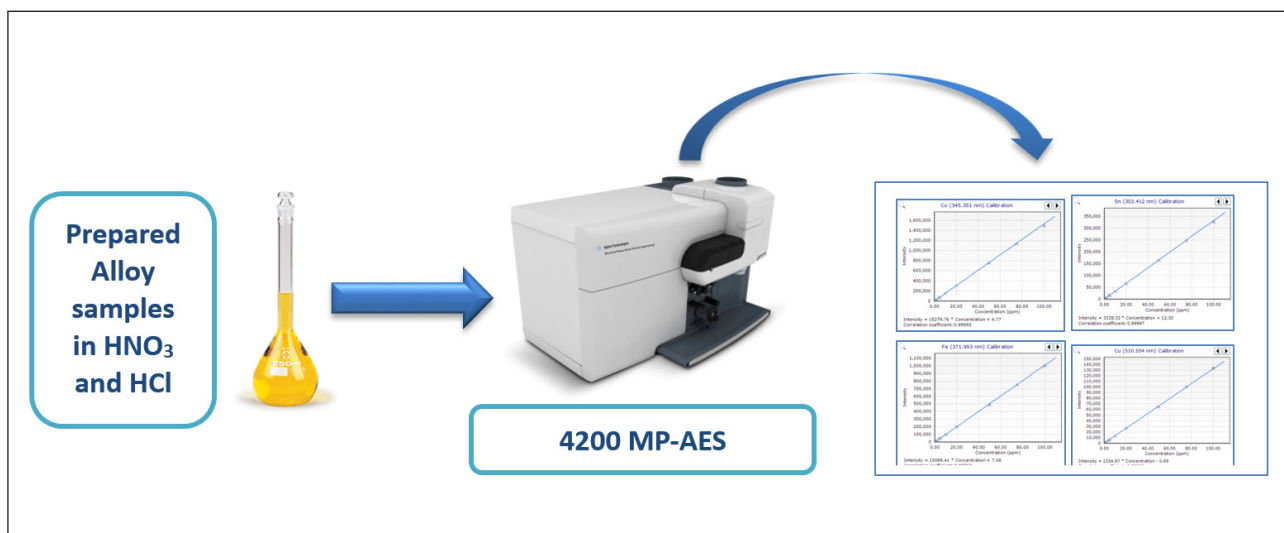


Figure 1. Schematic diagram of alloy sample analysis workflow using the Agilent 4200 MP-AES.

RESULTS AND DISCUSSION

Linear range

The linear calibration range for Fe, Cu, Co and Sn was established by preparing a series of standards at concentrations from 1ppm to 100ppm, and analyzing these using the 4200 MP-AES. The calibration graphs obtained are shown in Figure 2. These show that the linearity for all four analytes was acceptable up to a concentration of 100 ppm, with excellent correlation coefficient (R^2) values.

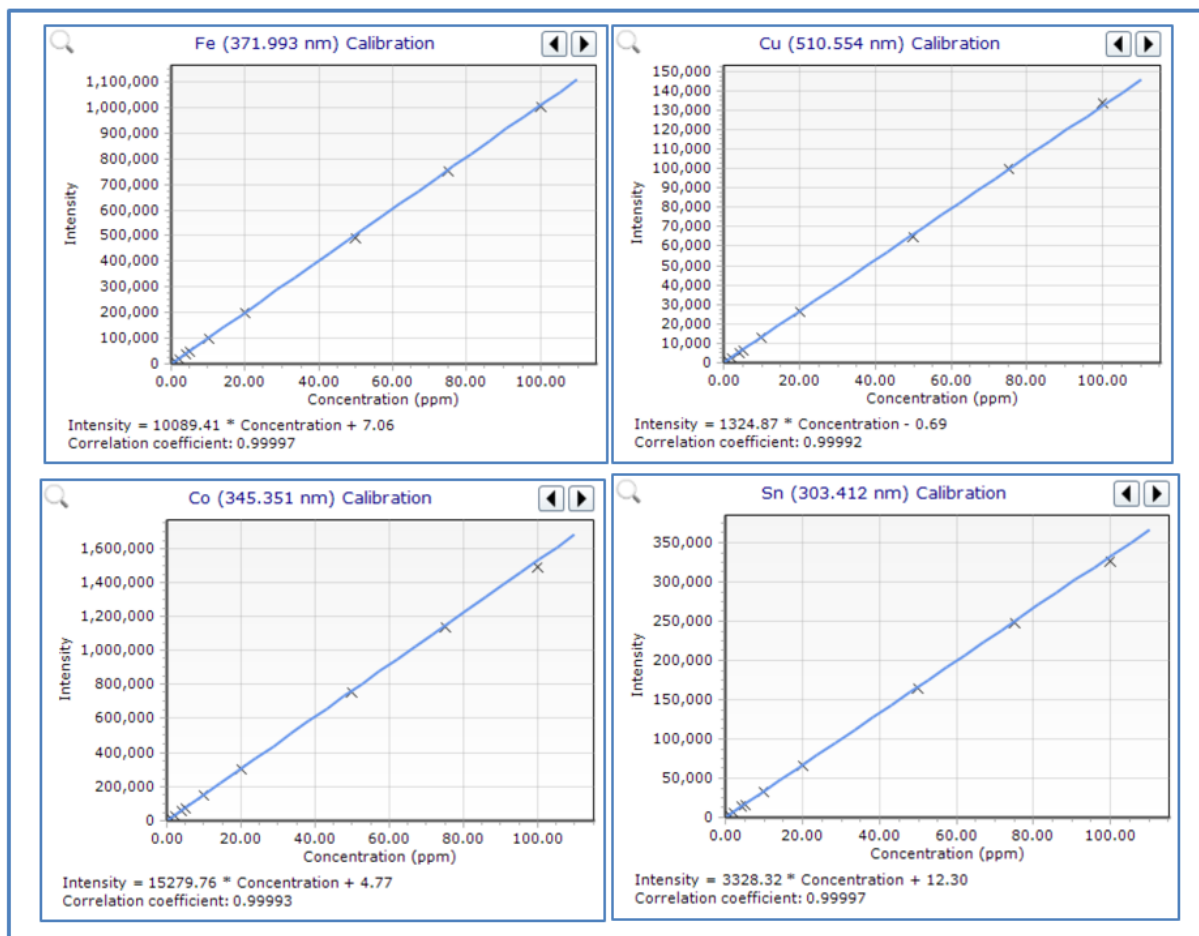


Figure 2. Calibration Curves for Fe, Cu, Co and Sn.

Analysis of real samples and spiked samples

The prepared samples were further diluted 10 times and analyzed using the 4200 MP-AES. The results are summarized in Table 2.

Sample Label	Replicate	Element (% by wt.)			
		Fe	Cu	Co	Sn
		371.993 nm	510.554 nm	345.351 nm	303.412 nm
Sample C1	Replicate 1	64.20	28.85	-	2.89
	Replicate 2	63.56	29.01	-	2.88
	Average	63.88	28.93	-	2.89
Sample C2	Replicate 1	63.03	28.64	-	2.83
	Replicate 2	64.13	28.93	-	2.87
	Average	63.58	28.79	-	2.85

Table 2. Real sample analysis results using the 4200 MP-AES.

Sample C3	Replicate 1	62.98	28.69	-	2.82
	Replicate 2	64.28	28.71	-	2.84
	Average	63.63	28.70	-	2.83
Sample N1	Replicate 1	28.01	41.83	27.28	-
	Replicate 2	28.54	42.94	27.88	-
	Average	28.28	42.39	27.58	-
Sample N2	Replicate 1	28.24	41.24	26.91	-
	Replicate 2	28.84	42.37	27.74	-
	Average	28.54	41.81	27.33	-
Sample N3	Replicate 1	28.19	41.33	27.00	-
	Replicate 2	28.56	41.40	27.55	-
	Average	28.38	41.37	27.28	-

Table 2. Real sample analysis results using the 4200 MP-AES.

Along with the pre-alloyed metal powder samples, matrix spiked samples were also analyzed at 5ppm levels for all metals (Fe, Cu & Co) in samples N1 and N2. The recoveries obtained during these studies were between 90-105 %. The results are summarized in Table 3

Sample Label	Fe	Cu	Co
	371.993 nm	510.554 nm	345.351 nm
	% Average spike recovery		
Sample N1	94.76	92.03	96.59
Sample N2	103.75	91.95	96.13

Table 3. Spike and recovery results using the 4200 MP-AES.

4200 MP-AES results comparison & verification with flame AA

The method was verified by analyzing the samples using flame AA (Agilent 240 AA) and comparing the results using both techniques. The results are shown in Table 4.

Sample Label	Comparison of 4200 MP-AES & flame AA results (% by wt.)							
	Fe		Cu		Co		Sn	
	AA	MP-AES	AA	MP-AES	AA	MP-AES	AA	MP-AES
Sample C1	63.44	63.88	28.89	28.93	-	-	2.66	2.89
Sample C2	63.16	63.58	29.22	28.79	-	-	2.84	2.85
Sample C3	63.39	63.63	29.18	28.70	-	-	2.71	2.83
Sample N1	28.30	28.28	42.75	42.39	27.78	27.58	-	-
Sample N2	28.51	28.54	42.49	41.81	27.98	27.33	-	-
Sample N3	28.33	28.38	41.64	41.37	28.03	27.28	-	-

Table 4. Comparison of 4200 MP-AES & flame AA results.

CONCLUSIONS

The Agilent 4200 MP-AES is a powerful tool for the analysis of all kind of matrices, including samples such as alloy and metal powder samples with elements ranging from ppb to % level. The advantage of Agilent's 4200 MP-AES over conventional flame AA is the wide linear dynamic range and choice of wavelengths selection, which helps in analyzing samples with minimal or no dilution steps, reducing manual dilution errors and the possibility of adding additional impurities via acids, water etc.

The 4200 MP-AES also reduces running costs, as it runs purely on nitrogen as a plasma gas which can easily be extracted from atmospheric air by means of a compressor and the 4107 nitrogen generator. There is also no need for element specific hollow cathode lamps which are required in flame AA. All these features drastically reduce the sample analysis cost. Also, as nitrogen is an inert gas, the instrument can be run unattended.

This solution note shows the ability of the Agilent 4200 MP-AES in analyzing the elements in alloy and metal powder samples accurately and precisely, with excellent recovery results for matrix spiked samples. The results are also verified using flame AA and are in good agreement.

REFERENCES

Craig Taylor, New methodology for determination of gold and precious metals using the Agilent 4100 MP-AES, Agilent Technologies Publication 5990-8597EN, Sept 2011.



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