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APPLICATION BRIEF 30489

Routine analysis of 6N high purity copper

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Keywords

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Goal

To demonstrate the capability of the Element GD Plus MS for the routine analysis of ultra-trace impurities in high purity copper with minimum sample preparation.

Analytical Challenge

Trace elemental analysis of high purity 5N or 6N metals is challenging due to both the complete elemental coverage required in the assay and the ultra low target levels.

For example, the sum of all impurities in a 6N Cu (99.9999% purity) is only 1 ppm. For such high purity materials, a routine, full mass scan quantification of all elements at single digit $ng \cdot g^{-1}$ levels is required.

In this application brief the Thermo Scientific™ Element™ GD Plus high resolution glow discharge sector-field mass spectrometer is assessed for the impurity analysis of high purity copper.

Method

In industrial manufacturing environments, the Element GD Plus MS is used for routine production quality control in a 2 or 3 shift rotation. Sample throughput is therefore critical to ensure a timely feedback based on analytical results. Sample surfaces were prepared by wet grinding with SiC paper (grit 120). Milling would be an appropriate alternative for sample preparation.

The instrumental parameters listed in Table 1 were defined to meet the needs of a routine production lab.

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Results

- The results in Table 2 demonstrate excellent detection limits for all trace elements analyzed.
- Sample throughput at such low quantification levels is relatively high with 5 samples per hour.
- A well characterized in-house control sample with elemental concentrations in the $\mu g \cdot g^{-1}$ level can be used to monitor daily performance.
- Typically a one-point calibration using a reference material (e.g. ERM-EB385) is sufficient for accurate trace element quantification due to the intrinsic linearity of the complete system.
- When suitable reference materials are not available, semi-quantitative analyses provide accuracies to within ± 30 % using the Standard RSF calibration strategy.
- The Element GD Plus GD-MS analytical process is simple enough that routine analyses are performed by shift operators.
- Residual precious metals are of additional interest in Cu refining processes; the most challenging of which is rhodium, as it suffers from polyatomic interferences. As can be seen in Figure 1, these can be easily resolved by the Element GD Plus MS. Since the resolution switching is fast (< 1 s) and automatic, elements that require the highest resolution, such as Rh, can be routinely analyzed at the $ng.g^{-1}$ level. and further optimization, depending on individual lab requirements.

Conclusion

The Element GD Plus MS provides a routine, reliable and straightforward solution for ultra-trace analysis of high purity copper. Under routine operation, detection limits of <0.01 $\mu g \cdot g^{-1}$ in 5N or 6N copper samples are easily achieved. A fast sample turn-around of 5 samples per hour guarantees rapid feedback to production with the capacity of analyzing ~120 samples per day. Automatic resolution switching (within 1 s) enables the routine use of optimum mass resolution for the elimination of matrix induced interferences.

Table 1. Instrumental parameters.

Parameter	Value
Matrix sensitivity	2×10^{10} cps total Cu (MR)
Analysis time	5 min pre-sputter 5 min acquisition
Discharge current	45 mA
Discharge voltage	~750 V
Anode parts	High purity graphite

Table 2. Results obtained from repeat analysis of a 6N Cu sample at multiple spots.

Element	Typical LoD [$\mu g \cdot g^{-1}$]	Obtained LoD [$\mu g \cdot g^{-1}$]
Ag	<0.01	0.005
As	<0.01	0.001
Bi	<0.01	0.0003
Fe	<0.01	0.001
Pb	<0.01	0.0004
S	<0.1	0.03
Sb	<0.01	0.001
Se	<0.01	0.002
Te	<0.01	0.001

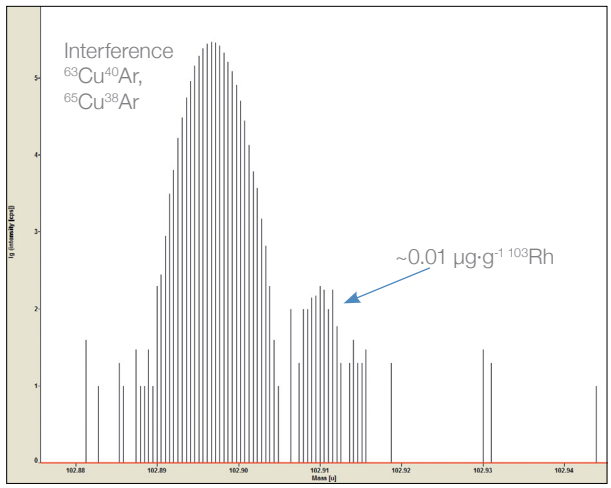

Figure 1. High mass resolution (R>10000) spectrum showing baseline separation between the CuAr interferences and the monoisotopic rhodium. Note the logarithmic intensity scale.

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