Application Brief

Chemicals & Electronics

# Determination of Elemental Impurities in Copper Sulfate using ICP-OES

## Fast, accurate results using the Agilent 5800 Vertical Dual View (VDV) ICP-OES with smart tools

**Author**

Ruby Bradford

Agilent Technologies, Inc.

**Figure 1.** Crystals of copper sulfate pentahydrate.

## Monitor impurities in copper sulfate

Copper (Cu) is one of the most important metals in the manufacturing of electronic devices, due to its high electrical conductivity. The Cu found in many electrical components, such as printed circuit boards, are typically manufactured by electroplating electrolytes such as copper sulfate ($CuSO_4$). Any metal impurities present in the $CuSO_4$ will also be electroplated onto the surface, potentially reducing the quality and conductivity of the electrical device. Therefore, the elemental purity of the $CuSO_4$ needs to be carefully monitored using a suitable analytical technique such as Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES).

The Agilent 5800 VDV ICP-OES with Agilent ICP Expert Pro pack instrument control software was used for the multi-elemental analysis of high purity $CuSO_4$. The array of smart tools, sensors, and diagnostics provide useful information on the samples and the operational status of the instrument, greatly assisting with method development, speed and quality of the quantitative analysis, and maintenance tasks. Tools including IntelliQuant Screening, Intelligent Rinse, and Early Maintenance Feedback (EMF) improved data accuracy and reduced unscheduled downtime for maintenance, providing analysts with greater confidence in the method and results. By maximizing uptime and minimizing remeasurements, the intelligent 5800 ICP-OES boosts productivity and reliability making it ideal for routine applications.

### Simple sample preparation procedure

To prepare the samples, 1 g of anhydrous $CuSO_4$ (Figure 1) was dissolved in 50 mL of 18.2 MΩ de-ionized water (Merck Millipore) to generate a 2% $CuSO_4$ solution. Standards were matrix-matched to the samples to create a final matrix of 2% $CuSO_4$ at analyte concentrations between 5 and 1000 µg/L. The standards and samples were analyzed by the 5800 VDV ICP-OES using default parameters and Intelligent Rinse to optimize rinse times between samples.

## Simplify your method development

Once the samples had been prepared, the IntelliQuant Screening software function was used to estimate the concentrations of major impurity elements in the $CuSO_4$ samples. Using the periodic table 'heat map' view of the results (Figure 2), IntelliQuant quickly and clearly informed the analyst of an appropriate calibration range for each analyte, saving development time. IntelliQuant Screening also helped with the selection of the best wavelengths to use for analytes in the quantitative method. It provides a star-ranking system to indicate which wavelengths are free from spectral overlaps. The Screening worksheet can then be easily converted into a quantitative method.

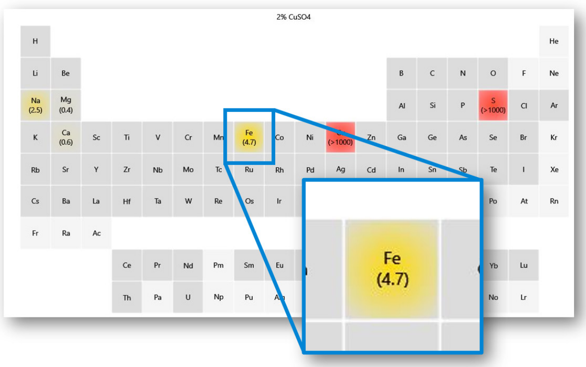


**Figure 2.** IntelliQuant Screening elemental 'heat map', showing elements present in a sample and their semiquantitative concentration (ppm).

## Maximize sample throughput

The Intelligent Rinse software feature was used throughout the application. When low-level analyte analysis is required, long rinse times are often used to minimize carry-over and cross-contamination between samples. However, the same lengthy rinse is applied to all samples, even if a short, five-second rinse time is sufficient. Intelligent Rinse adjusts the rinse time between samples based on the actual time required to rinse out each element. The software monitors the intensities of analyte wavelengths until a user-specified threshold is met. So, Intelligent Rinse minimizes unnecessary rinse-time, increasing overall throughput while maintaining accurate results.

The accuracy of the method was evaluated by conducting a sample spike recovery test of a $CuSO_4$ sample. Recoveries were within ±10% for all elements, as shown in Table 1.

**Table 1.** Method Detection Limits (MDLs), quantitative results, measured spike concentration, and recoveries for elements in a $CuSO_4$ sample, before dilution factor applied.

| Element and Wavelength (nm) | MDL (µg/L) | Sample Results (µg/L) | Measured Spike (µg/L) | Spike Recovery (%) |
|---|---|---|---|---|
| Ag 328.068 | 0.366 | <MDL | 49.7 | 101 |
| Al 396.152 | 0.787 | <MDL | 52.2 | 99 |
| As 193.696 | 2.86 | <MDL | 48.9 | 99 |
| Ca 396.847 | 8.43 | <MDL | 48.1 | 95 |
| Cd 226.502 | 0.0822 | <MDL | 49.6 | 99 |
| Co 238.892 | 0.279 | 91.2 | 50.4 | 101 |
| Cr 267.716 | 0.357 | <MDL | 50.8 | 101 |
| Fe 238.204 | 1.54 | 2512 | 213 | 107 |
| In 410.176 | 2.39 | <MDL | 52.9 | 105 |
| K 766.491 | 5.01 | <MDL | 54.3 | 106 |
| Mg 279.553 | 1.91 | 1476 | 51.0 | 102 |
| Mn 257.610 | 0.0813 | <MDL | 50.7 | 101 |
| Na 589.592 | 1.687 | 2131 | 209 | 106 |
| Ni 231.604 | 0.693 | 207 | 50.5 | 101 |
| Pb 220.353 | 1.00 | 12459 | 206 | 103 |
| Sn 189.925 | 3.35 | 2754 | 206 | 98 |
| Ti 337.280 | 0.675 | <MDL | 49.5 | 100 |
| Tl 190.794 | 2.77 | <MDL | 49.0 | 97 |
| Zn 206.200 | 1.04 | 25894 | 199 | 100 |

## Prevent unnecessary downtime

In this study, the high levels of Cu present in the samples caused a buildup on the torch after prolonged use. An EMF maintenance counter was created to remind the user to swap the torch after 10 plasma-on hours. Keeping the instrument in optimal condition preempts unplanned downtime, reduces the need for QC remeasurements, and improves instrument performance.

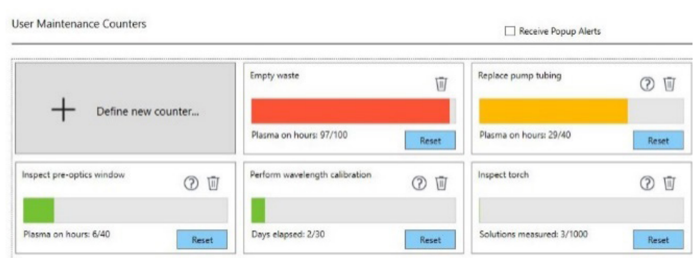


**Figure 3.** Example of EMF counters within the Agilent ICP Expert software.

## More information

Agilent application note: 5994-6337EN

**www.agilent.com/chem/5800icp-oes**


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