

Determination of Trace Metals in High-Purity Copper Using the GTA-95 Graphite Tube Atomizer

Application Note

Atomic Absorption

Author

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Introduction

In the manufacture of high-purity copper for electrical purposes the concentration of trace metals has to be carefully controlled. It has been found, for example, that the presence of selenium and tellurium affects the electrical conductivity of the copper. Arsenic has a similar effect, but also causes brittleness which makes coiling of wire difficult.

The elements of interest are lead, bismuth, selenium, tellurium, arsenic, antimony and tin. The levels to which they are monitored are:

Arsenic, tin and lead	5 µg/g in solid
Antimony, bismuth, selenium and tellurium	2 µg/g in solid

Although the elements are those which normally lend themselves to hydride generation, problems can be encountered with this technique in the presence of large concentrations of metal. Furnace atomization is the alternative method of analysis and is ideally suited to these low level estimations.

The study has resulted in a procedure for the analysis of these metals using the GTA-95 graphite tube atomizer. Because of the relatively volatile nature of the metals of interest it was thought useful to investigate the performance of the pyrolytic platform.



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Sample Preparation

Sample preparation requires the dissolution of the copper in concentrated nitric acid which is then made up with water to give a 2% solution of copper. This is suitable for the analysis of all seven elements.

Instrumental Conditions

The GTA-95 graphite tube atomizer with programmable sample dispenser was used with an M-975 spectrophotometer in this study. Agilent hollow cathode lamps were selected for all elements (Table 1). The pyrolytic platform was used in the furnace with the selected conditions given in Table 2.

Table 1. Instrumental Conditions

Element	Lamp current (mA)	Wavelength (nm)	SBW (nm)
Arsenic	7	193.7	1.0
Tin	7	235.5	0.5
Lead	5	217.0	1.0
Antimony	10	217.6	0.2
Bismuth	10	223.1	0.2
Selenium	10	196.0	1.0
Tellurium	7	214.3	0.5

Table 2. Furnace Parameters for Platform Atomization

Element volume (µL)	As 10		Tin 10		Pb 5		Sb 10		Bi 10		Se 20		Te 10		Ar gas flow L/min	Read signal
	°C	Sec	°C	Sec	°C	Sec	°C	Sec	°C	Sec	°C	Sec	°C	Sec		
Step	75	5	75	5	75	5	75	5	75	5	75	5	75	5	3	
1	75	5	75	5	75	5	75	5	75	5	75	5	75	5	3	
2	115	50	115	50	115	50	115	50	115	50	115	50	115	50	3	
3	500	1	600	1	400	1	600	1	400	1	650	1	500	1	3	
4	500	10	600	10	400	10	600	10	400	10	650	10	500	10	3	
5	500	10	600	10	400	10	600	10	400	10	650	10	500	10	0	
6	2500	1.1	2600	1.1	2400	2	2600	1	2200	2	2600	1.1	2600	1.1	0	R
7	2500	1.1	2600	1.1	2400	2	2600	1	2200	2	2600	1.1	2600	1.1	0	R
8	2500	1.1	2600	1.1	2400	2	2600	1	2200	2	2600	1.1	2600	1.1	3	

Pyrolytic Platform

The pyrolytic platform is a piece of solid pyrolytic graphite which can be installed in the standard graphite tube. Physical contact between the platform and the tube is only at the corners. The sample deposited on the platform is heated principally by radiation from the tube wall rather than by conduction. Figure 1 shows the position of the platform inside the graphite tube.

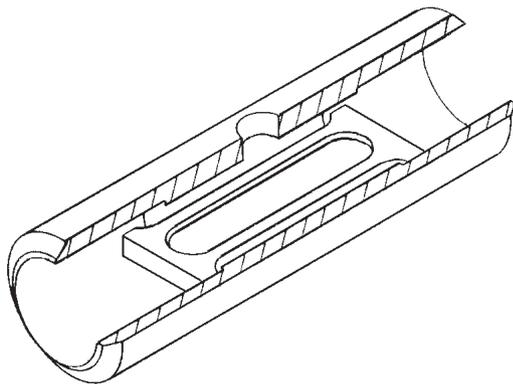


Figure 1. Platform located inside graphite tube.

One of the benefits of the platform is the greater freedom from interferences and background which can be achieved for volatile elements. This is a result of the delay in atomization of the element as the temperature of the platform lags behind the temperature of the tube wall and the atomization environment.

Figure 2 shows the benefit of using the platform in the determination of arsenic in copper compared to atomization directly from the tube wall. The background is considerably reduced during atomization and the atomic peak is delayed by about 2 seconds. These findings were confirmed for the other elements of interest.

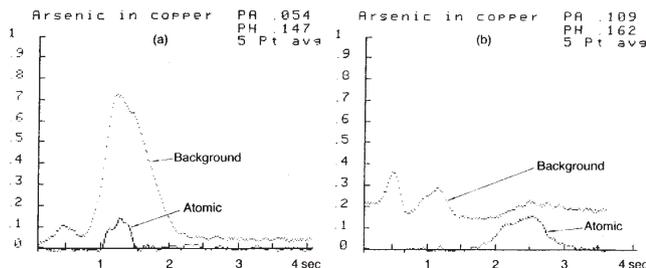


Figure 2. Arsenic in copper atomized from wall of graphite tube; Arsenic in copper atomized from pyrolytic platform.

Because of the lag in temperature of the platform, and because at low temperatures radiation is negligible, it was necessary to use somewhat higher drying temperatures than

would normally be the case with tube wall atomization. The pyrolytic platforms also require atomization temperatures higher than normally recommended.

Argon is preferred over nitrogen as a protective gas since it yields increased graphite lifetime and higher sensitivity.

Figure 3 shows the use of the platform for lead. The difference between the blank and the standard is clearly shown, as is the separation between the atomic peak and the background peaks.

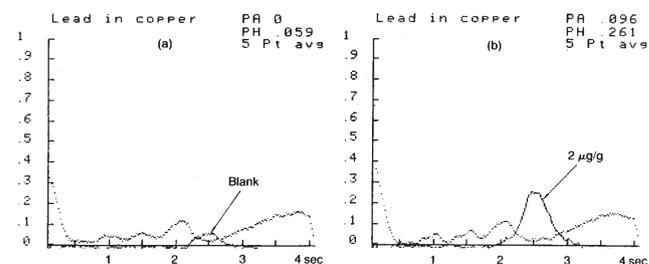


Figure 3. Lead in copper atomized from pyrolytic platform: blank; 2 µg/g in the solid.

Results

Matrix matched standards were prepared using pure copper to cover the following ranges of concentration in the solid:

Lead, arsenic, tin	2 to 8 µg/g
Selenium, Tellurium, Bismuth	1 to 3 µg/g
Antimony	2 to 6 µg/g

Figures 4 and 5 show recorder traces obtained from the standards for lead and arsenic, respectively.

Figures 6 and 7 show the calibration graphs obtained for each of the seven elements.

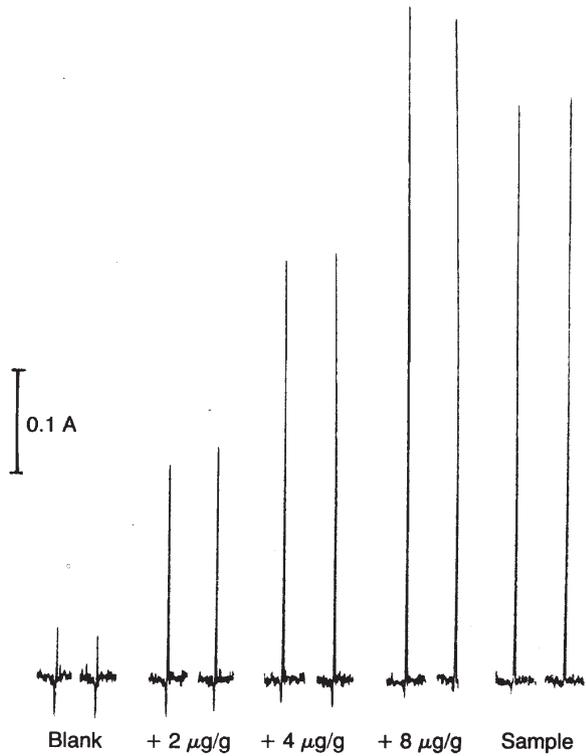


Figure 4. Lead in copper atomized from pyrolytic platform: standards and sample.

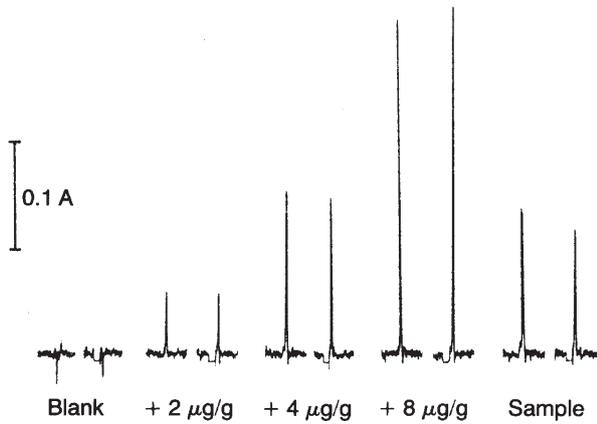


Figure 5. Tin in copper atomized from pyrolytic platform: standards and sample.

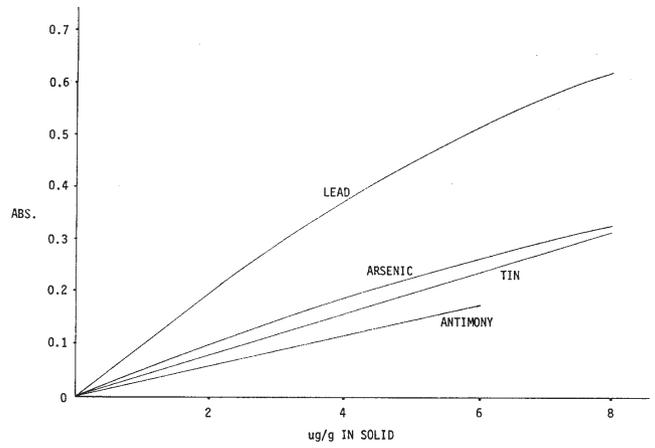


Figure 6. Calibration graphs for lead, arsenic, tin and antimony, using sample volumes listed in Table 2.

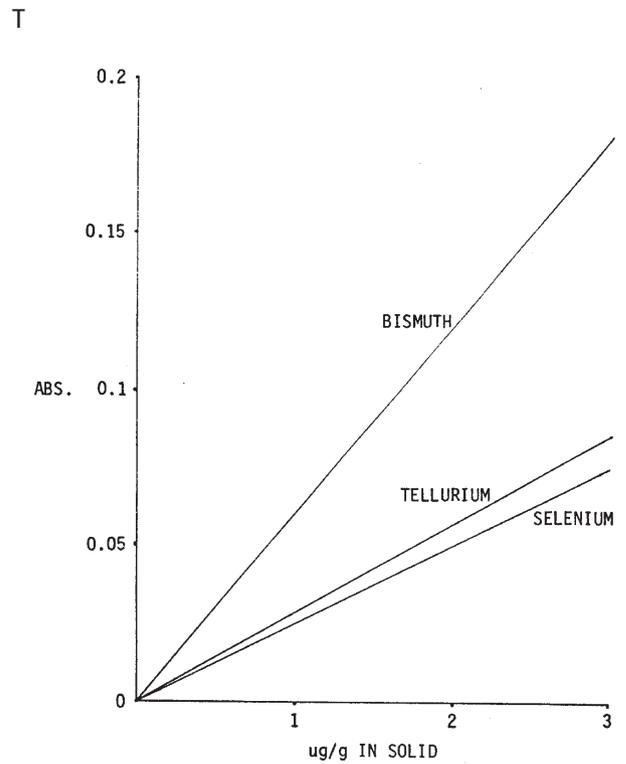


Figure 7. Calibration graphs for bismuth, tellurium and selenium, using sample volumes listed in Table 2.

Typical precision measured (for 10 replicate measurements of lead) ranged from 1.4% to 4.2% RSD depending on the concentrations (Figure 8).

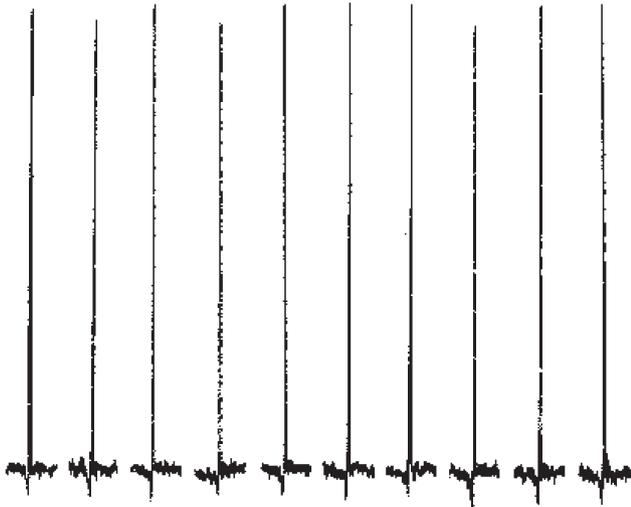


Figure 8. Ten replicate determinations of lead at 4 µg/g in the solid (RSD = 1.4%).

Conclusion

Trace elements in high-purity copper have been readily determined on the GTA-95. The pyrolytic platform is beneficial in minimizing background during atomization. This has advantages when measuring very low levels of these elements, particularly at short wavelengths. In sensitivity-limited situations or at particularly low concentrations, the analytical signal can be further improved by increasing the injection volume or by using the multiple injection facility of the GTA-95 autosampler.

Reference

Doidge, "Pyrolytic Graphite Platforms", Varian Instruments at Work, AA-25, September 1982

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