



Analysis of Silver: Analysis of Ag, Dopants and Impurities

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1 Introduction

In electronics, silver alloys are used because of their conductive, mechanic and contact properties. These properties are improved by treatment of silver with different metallic oxides. This Application Note examines the analysis of Ag and metal oxides, with impurities such as Cd, Cu, Fe, Mg Ni, Sn and Zn.

2 Principle

2.1 Technique used

The elemental analysis of solutions was undertaken by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). The sample is nebulized then transferred to an argon plasma. It is decomposed, atomized and ionized whereby the atoms and ions are excited. We measure the intensity of the light emitted when the atoms or ions return to lower levels of energy. Each element emits light at characteristic wavelengths and these lines can be used for quantitative analysis after a calibration.

2.2 Wavelength choice

The choice of the wavelength in a given matrix can be made using the "profile" function, or by using Win-IMAGE, which is rapid semi-quantitative analysis mode using multiple wavelengths. The principle is the same in either case: record the scans of analytes at low concentration, and of the matrix. By superimposing the spectra, we see possible interferences.

3 Sample Preparation

The samples were prepared at 1 g/L or 2 g/L of Ag by dissolving with 68% HNO₃ and diluting to give a final concentration of HNO₃ is about 1.5%. The standards were prepared using Spex^(*) standard single element solutions and deionized water. Matrix matching was not required, and we were able to analyze both major elements and traces using a simple calibration.

4 Calibration and analysis mode

The solutions used for calibration are listed in Table 1 (mg/L). A weighted regression was applied for the Cu and Zn calibration curves.

The diluted samples were analyzed directly. The "100 % analytical mode" was used for the analysis, meaning that the sum of all the analyzed elements is assumed to be 100%. If the results do not add up to 100%, an adjustment is made over all the measured elements. This type of method improves the accuracy of such analyses, by compensating for any small drift that may occur over a period of time. The results are given in Section 7 and are reported as % in the solid after correcting for the weight, volume and dilution factors.

(*): SpexCertiprep www.certiprep.com

**Table 1: Concentration of standards**

Element	Std0	Std1	Std2	Std3	Std4	Std5	Std6	Std7
Ag	0				55			100
Cd	0	0.6						
Cu	0	0.05						1
Zn	0	0.01						
Fe	0		0.18					
Mg	0	0.01	0.17					
Ni	0			2				
Sn	0			22	1	2	25	
Zn	0	0.01		22	1	2	25	

5 Instrument specification

The work was done on a JY ULTIMA. The specifications of this instrument are listed below in Tables 2 and 3.

Table 2: Specification of spectrometer

Parameters	Specifications
Mounting	Czerny Turner
Focal length	1m
Nitrogen purge	Yes
Variable resolution	Yes
Grating number of grooves	2400 gr/mm
Order	2nd order

Table 3: Specification of RF Generator

Parameters	Specifications
Type of generator	Solid state
Observation	Radial
Frequency	40.68 MHz
Control of gas flowrate	by computer
Control of pump flow	by computer
Cooling	air

Table 5: Wavelength selection and analytical conditions

	Wavelength (nm)	Measured point	Calculating point	Integration time (sec)	Entrance slit (μm)	Exit slit (μm)	Increment (nm)	Calculation mode
Ag	328.068	1	1	4.0	22	80	0.002	Max
Cd	214.438	5	1	0.5	22	15	0.002	Max
Cu	324.754	5	1	1.0	22	15	0.003	Max
Fe	259.940	5	1	0.5	22	15	0.002	Max
Mg	285.213	5	1	0.5	22	15	0.002	Max
Ni	231.604	5	1	1.0	22	15	0.002	Max
Sn	189.989	5	1	0.5	22	15	0.002	Max
Zn	206.200	5	1	1.0	22	15	0.002	Max

The operating conditions are listed in Table 4 below.

Table 4: Operating conditions

Parameter	Condition
RF Generator power	1100 W
Plasma gas flowrate	12 L/min
Auxiliary gas flowrate	0 L/min
Sheath gas flowrate	0.2 L/min
Nebulizer flowrate	2.84ars, 0.57 ml/min
Sample uptake	1 mL/min
Type of nebulizer	Meinhard K3 type
Type of spray chamber	Cyclonic
Argon humidifier	No
Injector tube diameter	3.0 mm

6 Wavelength selection and analytical conditions

The acquisition parameters used for analysis are listed in Table 5.



7 Results

Six samples were analyzed and the results were compared to those obtained in the quality control laboratory.

8 Conclusion

The results show the feasibility of the analysis of Silver alloys by using a high resolution ICP-OES instrument. Results were close to the ones expected, even with the high dilution factors, due to the high sensitivity and stability of the JY ULTIMA 2 spectrometer. Precision would be further improved by the use of a simultaneous internal standard using the optional internal standard monochromator available from JY. This enables simultaneous measurement of both analyte and internal standard.

Table 6: Results

Element	Sample 1 0.105 g; 50 mL 20 fold dilution			Sample 2 0.1132 g; 100 mL 10 fold dilution			Sample 3 0.11078 g; 50 mL; 20 fold dilution		
	Conc (%)	RSD (%)	Expected conc (%)	Conc (%)	RSD (%)	Expected conc (%)	Conc (%)	RSD (%)	Expected conc (%)
Ag	99.18	0.58	99.33	91.18	0.37	90.6	99.63	0.29	99.86
Cd	0.665	0.44	0.61	8.46	1.9	9.1	0.0401	0.18	
Cu	0.071	1.3	0.055	0.0674	1.15	0.05	0.0285	1.55	
Fe	0.236	2.45		0.014	4.48		0.125	0.86	
Mg	0.00207	5.08	0.00446	6.48			0.00199		7.93
Ni	0.0112	2.9	0.0024	0.224	0.52	0.195	0.148	0.35	0.13
Sn	< LD			< LD			0.00829		13.3
Zn	0.0117	1.62		0.0361	0.75		0.0162	4.45	

Table 7: Results

Element	Sample 1 0.105 g; 50 mL 20 fold dilution			Sample 2 0.1132 g; 100 mL 10 fold dilution			Sample 3 0.11078 g; 50 mL; 20 fold dilution		
	Conc (%)	RSD (%)	Expected conc (%)	Conc (%)	RSD (%)	Expected conc (%)	Conc (%)	RSD (%)	Expected conc (%)
Ag	54.4	0.16	54.1	99.57	0.28	99.69	99.37	0.6	99.3
Cd	0.104	0.62	0.04	0.0392	0.85		0.0362	0.16	
Cu	21.77	0.22	22	0.00807	3.7		0.51	1.41	0.498
Fe	0.0182	1.68		0.0103	2.06		0.0173	2.31	
Mg	0.00242	5.83		0.198	0.78	0.174	0.0037	5.7	
Ni	0.00939	0.44		0.138	2.72	0.13	0.0135	2.32	
Sn	1.67	3.22	1.97	< LD			< LD		
Zn	22.02	1.15	21.89	0.034	1.78		0.0356	0.71	



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