Analysis of Metals in 100 g/l Copper Acetate

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Keywords: chemicals, inorganic

1 Introduction

A sample with 100 g/L Cu $(CH_3COO)_2.H20$ was presented to this laboratory for trace determination. As there was only one sample to be analyzed, which had a complex matrix, the standard addition method was used for analysis.

2 Principle

2.1 Technique used

The elemental analysis of a 100 g/L Cu $(CH_3COO)_2.H_20$ solution was undertaken by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). 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.

2.3 Limit of detection estimation

The limits of detection are calculated using the following formula:

$$\mathbf{LOD = k \times BEC \times RSD_0}$$

With:

LOD = limits of detection,
k= 3 for the normal 3-sigma values,
BEC = Background equivalent concentration,
RSD_0 = relative standard deviation of the blank.

To calculate the LOD, a calibration curve is constructed using two points, 0 ppm and 5 ppm, or some concentration where the calibration is linear; this gives the BEC. The RSD_0 is evaluated by running the blank ten times.

3 Sample preparation

10 g of sample was weighed and dissolved with 5 mL 69 % HNO_3 and diluted up to 100 mL with deionized water.

Two spiked samples were prepared for the standard addition method.
Addition 1: 0.1 mL of standard "A" with 25 mL of dissolved Cu $(CH_3COO)_2.H_20$.
Addition 2: 0.2 mL of standard "A" with 25 mL of dissolved Cu $(CH_3COO)_2.H_20$.

Standard "A" contained:
1 g/L of Na,
0.5 g/L of Ca,
0.2 g/L of S, Fe, Ni, K.

The concentrations of the spiked samples are listed in Table 1 below.

Table 1: Standard addition concentration

Element		Concentration of		
	Wavelength	Unknown sample	Addition1	Addition2
Ca	315.885	X	X + 2	X + 4
Fe	259.940	X	X + 0.8	X + 1.6
K	766.490	X	X + 0.8	X + 1.6
Na	589.592	X	X + 4	X + 8
Ni	231.604	X	X + 0.8	X + 1.6
S	182.568	X	X + 0.8	X + 1.6

4 Instrument specification

The work was done on a ULTIMA.The specifications of this instrument are listed below in Table 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

5 Operating conditions

The operating conditions are listed in Table 4 below.

Table 4: Operating conditions

Parameter	Condition
RF Generator power	1200 W
Plasma gas flowrate	12 L/min
Auxiliary gas flowrate	0 L/min
Sheath gas flowrate	0.2 L/min
Nebulizer gas flowrate	0.8 L/min
Nebulizer flowrate	3 bars (45 psi)
Sample uptake	1 mL/min
Type of nebulizer	Parallel
Type of spray chamber	Cyclonic
Argon humidifier	No
Injector tube diameter	3.0 mm

6 Wavelength selection and analytical conditions

For each element, the line with the highest sensitivity was used for analysis, because there were no problems with interferences. The analysis conditions were the same for all elements except the alkali elements.

Table 5: Analytical conditions

Element	Slits (µm)	Analysis mode	Integration time (sec)
All elements	20 x 15	Direct peaking	8
K	20 x 80	Gaussian	0.5

The use of the parallel nebulizer and the large internal diameter (ID) of the injector tube enabled trouble free analysis, even with the high dissolved salts. The larger the ID injector tube also ensures a minimization of the interferences. Due to the high dissolved salts, an initial conditioning of the spray chamber is advised for maximum stability. It is also imperative to use matched standards or standard addition because of the viscosity of solutions with high dissolved solids.

7 Discussion

7.1 Limits of Detection

The limits of detection were calculated using the formula in paragraph 2.3. They were calculated in µg/kg in the solid sample.

Table 6: Limits of detection

Elements	LOD (*µ*g/kg)
Ca	40
Fe	5
K	90
Na	60
Ni	15
SO_4	90

7.2 Determination of the unknown sample

Using the two spiked samples and the unknown, a calibration curve was constructed. The concentrations of the traces in the unknown are given by the intercept of their calibration curves. The results for unknown are given in Table 7 below.

Table 7: Results

Element	Concentration in mg/l	Concentration in %	Expected Value in %
Ca	0.876	0.0008	< 0.005
Fe	0.492	0.0005	< 0.002
K	0.018	< 0.00002	< 0.01
Na	3.28	0.0033	< 0.05
Ni	0.013	< 0.00002	< 0.01
SO4	0.765	0.0008	< 0.01

8 Summary

To achieve the lowest detection limits, dilution is undesirable. The results show the spectrometers are able to perform an excellent analysis, even with high dissolved solids. This enables the analysis to be performed to the best detection limits possible.

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