

Analysis of Impurities in Silver Matrix by Atomic Absorption Spectrophotometry

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Summary: A procedure for the analysis of aluminium, chromium, copper, lead, mercury, nickel and zinc mainly using flame less atomic absorption spectrophotometry has been described. The results depict that the presence of silver does not introduce any significant interference, when standards are prepared in matching silver matrix solutions. The calibration curves obey the straight-line equations passing through the origin. Thus the separation of silver matrix from the analyte solutions is not necessary. The method has been successfully applied for the analysis of silver foils, wires, battery grade silver oxides and silver nitrate samples containing analyte elements in the concentration range 2 to 40 ppm.

Introduction

Atomic absorption spectrophotometry has been widely used for the analysis of metals and their alloys [1-3]. Most of the papers appearing in various journals on the analysis of impurities in silver by atomic absorption spectrophotometry have used flame [4-6] or graphite furnace [7,8]. The analyses have been carried out after separating the impurities from the bulk either by precipitation [9] or solvent extraction [10] of silver.

In the present work, an attempt has been made to estimate impurities in silver leaves and other silver articles without prior separation of impurities from the matrix. The results show that elements under study can be determined quite satisfactorily by using matching (in silver concentration) standards.

Results and Discussion

The analytical program followed for the analyte elements is presented in Table-1. Remaining instrumental conditions were adjusted following the instrument manual. All the elements with the exception of zinc and mercury were analyzed on graphite furnace. Mercury was analyzed by cold vapor generation using tin chloride as reducing agent, which is equivalent to sodium tetraborate [11]. Zinc was analyzed by flame AAS. For standardization of the instrument one blank i.e., 1% silver solution in 1% nitric acid and two standards prepared in the same matrix were used for each element.

Solutions of five different concentrations were prepared for each element by the addition of analyte element in the matrix solution. These solutions were

Table-1

A:-

Element	Wave-length (nm)	Lamp Curr. (mA)	Dry time/ Dry temp. (sec/°C)	Char time/ Char temp. (sec/°C)	Atom. Time/Atom. Time(sec °C)
Al	309.3	20	30/105	20/1400	10/2700
Be	467.9	20	30/125	20/1200	10/2700
Cd	457.6	10	20/100	20/300	20/2100
Cr	357.9	20	30/100	20/1100	10/2700
Cu	324.8	15	20/100	20/900	15/2650
Pb	566.6	10	20/100	20/1000	10/2300
Ni	464.0	20	20/100	20/1000	10/2700

B:-

Element	Wave-length (nm)	Lamp Curr. (mA)	Slit	Flame
Zn	427.7	15	0.1	Air/Acetylene
Ag	328.1	15	0.2	Air/Acetylene
Hg	253.7	15		Cold vapor generation

then analyzed. The results are presented in Table-2 as recovered concentrations against the added concentrations. It is evident from the results that the elements under study follow linear response (straight line equation) in the range studied. The linear regression curves for each element are enlisted below:-

Al;	$Y = 0.9978X + 0.0025$
Be;	$Y = 0.987X + 0.0002$
Cd;	$Y = 1.022X + (-0.0017)$
Cr;	$Y = 0.99X + 0.0021$
Cu;	$Y = 0.993X + 0.0015$
Pb;	$Y = 1.022X + (-0.0037)$
Hg;	$Y = 1.024X + (-0.0007)$
Ni;	$Y = 0.992X + (-0.002)$
Zn;	$Y = 0.997X + 0.0008$

Where: Y = concentration recovered and X = concentration added

By using the blank and standards of matching matrix the intercepts are almost zero with the straight line of the calibration passing through the origin. The concentration recovered matches concentration of the element added. All these regression curves show that silver presented only additive error factor or interference which can be nullified by blank and standards prepared in silver matrix. Further more, the presence of silver did not introduce significant effect or interference.

Table-2:

CONCENTRATION (PPM)					
Aluminium					
Added	0.1	0.2	0.5	0.7	1.0
Recovered	0.1	0.2	0.51	0.699	0.998
Beryllium					
Added	0.01	0.02	0.03	0.04	0.05
Recovered	0.01	0.02	0.03	0.0397	0.0495
Cadmium					
Added	0.0025	0.005	0.0075	0.0125	0.015
Recovered	0.0025	0.005	0.0072	0.0127	0.0152
Chromium					
Added	0.05	0.1	0.15	0.2	0.25
Recovered	0.055	0.1	0.148	0.201	0.251
Copper					
Added	0.1	0.2	0.25	0.3	0.4
Recovered	0.1	0.2	0.25	0.299	0.398
Lead					
Added	0.05	0.1	0.15	0.2	0.25
Recovered	0.049	0.096	0.15	0.201	0.25
Mercury					
Added	0.02	0.04	0.06	0.08	0.1
Recovered	0.021	0.0395	0.06	0.0802	0.103
Nickel					
Added	0.1	0.2	0.3	0.4	0.5
Recovered	0.101	0.202	0.286	0.4	0.498
Zinc					
Added	0.1	0.2	0.5	0.7	1.0
Recovered	0.1	0.2	0.5	0.698	0.997

Table-3:

Conc.(ppm)	Goodfellow foil Cat. No. AG000090	Goodfellow wire Cat. No. AG005220	Goodfellow powder Cat. No. AG006020	Battery grade AgO (99.99%)
Al (Std. Value)	4.0	5.0	-	-
Al (M.S.)	3.90	5.93	-	-
Al (D.M.)	3.98	5.98	-	-
Cd (Std. Value)	2.0	3.0	-	-
Cd (M.S.)	1.92	2.98	-	-
Cd (D.M.)	1.99	2.98	-	-
Cr (Std. Value)	20.0	-	-	-
Cr (M.S.)	19.96	-	-	-
Cr (D.M.)	19.98	-	-	-
Cu (Std. Value)	20.0	35.0	< 25.0	30.0
Cu (M.S.)	19.90	34.89	24.4	29.8
Cu (D.M.)	20.01	35.0	24.45	29.97
Pb (Std. Value)	40.0	< 3.0	< 10.0	35.0
Pb (M.S.)	39.7	2.95	9.87	34.8
Pb (D.M.)	39.98	2.96	9.91	34.99
Ni (Std. Value)	5.0	< 3.0	< 5.0	-
Ni (M.S.)	4.98	2.98	4.88	-
Ni (D.M.)	5.0	2.98	4.88	-
Zn (Std. Value)	-	10.0	-	-
Zn (M.S.)	-	9.97	-	-
Zn (D.M.)	-	10.02	-	-

Where; M.S. = Analysis after matrix separation

D.M. = Analysis by the direct method (described in this paper)

It can therefore be concluded that impurities in silver can be estimated reliably without their separation from matrix solution by the standards of

matching matrices that effectively remove the additive error introduced due to background impurities. Another advantage of this method is that negative error usually introduced during matrix separations is also avoided. This method has been successfully employed in our laboratory for the analysis of impurities in silver alloys, foils, wires and battery grade materials. Some of the analysis results are presented in Table-3. In addition of these, detailed analysis of silver foils (Waraq chandee) employing the present method has been reported in a previous publication [12].

Experimental

(a) Instrumentation

FMD-4 atomic absorption spectrophotometer of M/Z Zeiss, West Germany and graphite furnace model 76B manufactured by M/S Perkin Elmer GmbH, West Germany were used for the determination of impurities in silver.

(b) Reagents and standards

Standards of analytes

Standard solutions of all the elements to be analyzed were prepared from 1000 ppm atomic absorption standard solutions (BDH) in 0.1N HNO₃ containing 1% silver.

Silver solution

99.5% silver nitrate (Reidel - de Haen) was dissolved in 0.1N HNO₃ to prepare silver matrix solution.

(c) Determination of mercury by cold vapor generation

25 ml of sample solution was taken in vapor generation flask. 5 ml of 1:1 nitric acid was added to it followed by 2 ml of 10% tin chloride solution. The flask was immediately connected to the vapor generation apparatus/set up. The absorbance reading was noted for total of 20 seconds integration time.

All the solutions and their dilutions were made using double distilled/deionized water.

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