SHIMADZU APPLICATION NEWS

LAAN-A-UV-E008

● SPECTROPHOTOMETRIC ANALYSIS

No.A380

Measurement of Hexavalent Chromium, Aluminum, and Nickel in Plating Solution by Absorption Spectrophotometry —Analysis of Plating Solution —

■ History

The RoHS (Restriction of the Use of Certain Hazardous Substances in Electrical and Electronic Equipment) directive was officially announced in Europe in February, 2003.

In principle, the objectives of the RoHS directive are to restrict the use of heavy metals (lead, mercury, cadmium, hexavalent chromium) and brominated flame retardant PBB (polybrominated biphenyl) and PBDE (polybrominated diphenyl ether) in new electrical equipment products. The directive is going to be put into effect on July 1, 2006. The directive will require the electrical and electronic equipment manufacturers to take into consideration the quantities of these toxic substances contained in their products introduced into the European market. Although the specific criteria values are not to be announced, there is great interest in the RoHS directive among the companies, who plan to export their products to Europe.

Examples of absorption spectrophotometric measurements on these substances will be introduced here. Using this method, hexavalent chromium, aluminum, and nickel contained in widely used plating solutions can be analyzed by diluting the solutions and then adding packed reagents.

For details on analyzing plating products, please refer to Shimadzu Application News No. A361, and for details on analysis using generally used UV spectrophotometers, please refer to Shimadzu Application News No. A374.

■ Diluting Plating Solution and Adding Packed Reagent

Plating solutions can be analyzed just by diluting them. Since the UVmini-1240 and water quality measurement program pack have built-in calibration curves, process of creating calibration curves using standard samples can be omitted. The concentration is automatically displayed by adding the packed reagent manufactured by Kyoritsu Chemical-Check Lab. to the sample and setting the sample onto the UVmini-1240 (with built-in calibration curves).

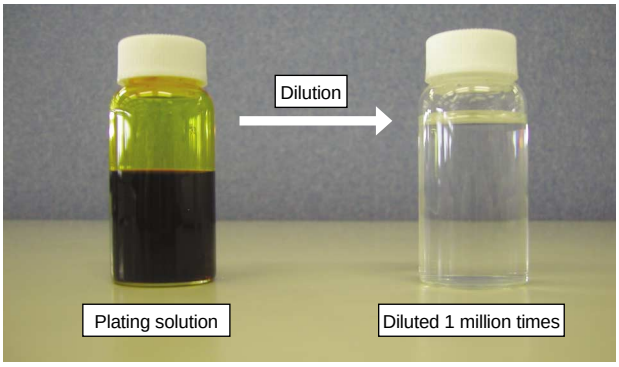

Fig.1 Diluting Plating Solution

Fig.2 Packed Reagent by Kyoritsu Chemical-Check Lab.

Fig.3 Shimadzu Spectrophotometer UVmini-1240

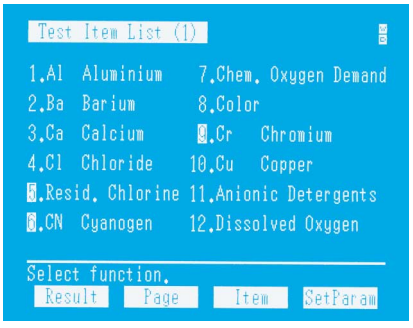

Fig.4 List of Measurement Items

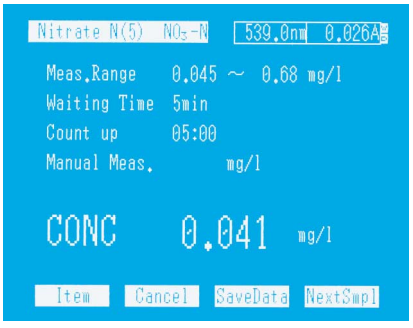

Fig.5 Measurement Result Printout

■ Results and Comparison with Data Obtained by ICPS

Hexavalent chromium plating solution, sulfuric acid plating solution, and electroless nickel plating solution were diluted, and hexavalent chromium, aluminum, and nickel were colored using the packed reagent from Kyoritsu Chemical-Check Lab. The concentrations calculated from the absorbance are shown in the tables below. For aluminum and nickel, the concentration values obtained by analyzing the same samples using Shimadzu Sequential Plasma Spectrometer ICPS-8100 are also shown.

The standard samples in the tables were prepared by diluting reagents for atomic absorption spectrophotometry by Wako Pure Chemical Industries, Ltd. and the tables show the reproducibility when using the packed reagent.

Table 1 Results of Hexavalent Chromium

Standard sample	
[mg/L]	0.5ppm
UV 1	0.507
UV 2	0.510
UV 3	0.509
UV 4	0.502
UV 5	0.509
UV Ave.	0.507
SD	0.003
CV(%)	0.633

Actual sample (hexavalent chromium plating solution)	
[mg/L]	Diluted 1 million times
UV 1	0.132
UV 2	0.130
UV 3	0.132
UV 4	0.129
UV 5	0.125
UV Ave.	0.130
SD	0.003
CV(%)	2.223

Cr^{6+}(Measurement range 0.02 - 1.0mg/L)

Table 2 Results of Aluminum

Standard sample			
[mg/L]	0.1ppm	0.2ppm	0.33ppm
UV 1	0.086	0.216	0.347
UV 2	0.095	0.211	0.351
UV 3	0.093	0.206	0.345
UV Ave.	0.091	0.211	0.348

Actual sample (Al in sulfuric acid plating solution)			
[mg/L]	Diluted 30,000 times	Diluted 15,000 times	Diluted 10,000 times
UV 1	0.079	0.247	0.352
UV 2	0.083	0.237	0.357
UV 3	0.084	0.241	0.373
UV Ave.	0.082	0.242	0.361
ICPS 1	0.117	0.236	0.356
ICPS 2	0.117	0.234	0.354
ICPS 3	0.118	0.237	0.361
ICPS Ave.	0.117	0.236	0.357

Al(Measurement range 0.05 - 0.4mg/L)

Table 3 Results of Nickel

Standard sample			
[mg/L]	1ppm	2ppm	5ppm
UV 1	1.007	2.016	4.641
UV 2	0.940	2.022	4.545
UV 3	0.960	2.021	4.199
UV Ave.	0.969	2.020	4.462

Actual sample (Ni in electroless nickel plating solution)			
[mg/L]	Diluted 5,000 times	Diluted 2,500 times	Diluted 1,000 times
UV 1	0.811	1.710	3.969
UV 2	0.859	1.754	4.051
UV 3	0.826	1.686	3.696
UV Ave.	0.832	1.717	3.905
ICPS 1	0.869	1.715	4.305
ICPS 2	0.866	1.697	4.291
ICPS 3	0.864	1.714	4.301
ICPS Ave.	0.866	1.708	4.299

Ni(Measurement range 0.5 - 8mg/L)

■ Summary

Responding to the RoHS directive, manufacturers are increasingly aware of the quality of plating solutions, as well as the plated finished products. Using the UVmini-1240 and the water quality measurement program pack together with the packed reagent, easy measurement without troublesome pretreatment is realized.

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