EDXRF APPLICATION NOTE
CHEMICAL BATH

FOR COPPER FOIL SURFACE TREATMENT

#1306

SCOPE

Measurement of aqueous chemical bath solutions containing nickel, cobalt, molybdenum, zinc and indium is demonstrated.

BACKGROUND

Surface treating is an important part in the manufacture of copper foil for the electronics industry. Surface treatments are used to clean the copper foil, to create roughing and heat resistance treatments, and to make single or multiple layer thin film coatings to prevent oxidation or enhance the electro-chemical properties of the copper foil. The chemical baths must be constantly monitored to ensure the highest quality surface treatments. Rigaku offers the NEX OL analyzer to meet these analytical needs for trend analysis of bath composition.

INSTRUMENT CONFIGURATION

Model:	Rigaku NEX OL Analyzer
Excitation:	Direct with filters
X-ray tube:	4 W Ag-anode
Detector:	Silicon Drift Detector
Film:	2 mil Kapton with 0.5 mil Teflon Coating (on wetted side)

Total Measurement Time = 160 sec

- 100 sec for Zn and In
- 60 sec for Co, Ni and Mo

SAMPLE PREPARATION and PRESENTATION

Aqueous calibration solutions were prepared by mixing powdered reagents for Zn, In, Ni, Mo and Co with the appropriate quantity of water. 5 calibrations standards were prepared for the Zn & In calibration and 5 standards were prepared for the Ni, Mo & Co calibration. All samples were measured in a static position using the auxiliary sample input loop.

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CALIBRATION and MEASUREMENT

Empirical calibrations were developed using aqueous samples. A summary of typical calibration is shown here.

Component	Concentration Range (ppm)	RMS Deviation	R^2 Confidence
Zn	750 - 1425	14.6	0.9958
In	140 - 320	4.2	0.9957
Ni	4500 - 5500	16.4	0.9973
Mo	780 - 1525	13.7	0.9976
Co	1275 - 1750	22.1	0.9805

SAMPLE RECOVERY AND PRECISION

A representative mid-concentration standard was selected and analyzed against the empirical calibrations to demonstrate effective recovery and precision. The sample was analyzed 10 consecutive times in a static position. Typical performance results are provided below.

Sample ID: Zn-In Std3 (ppm)			Units:	
Element	**Assay Value**	**NEX OL Value***	**Standard Deviation**	**RSD (%)**
Zn	1100	1118	16	1.5
In	220	222	3	1.2

** NEX OL Value reflects the average of the 10-repeat analysis.*

Sample ID: NMC Std3			Units: (ppm)	
Element	**Assay Value**	**NEX OL Value***	**Standard Deviation**	**RSD (%)**
Ni	5200	5362	36	0.7
Mo	1100	1115	4	0.4
Co	1500	1547	19	1.3

EMPIRICAL DETECTION LIMITS (LLD)

The empirical method was used to determine detection limits in a clean matrix containing no measureable elements. In the empirical method, 10 repeat analyses of a “blank” (deionized water) were taken and the standard deviation (σ) was determined. The Lower Limit of Detection (LLD) is then defined as 3σ.

Sample Type: DI Water		Units: ppm
Component	**Empirical LLD**	**Condition Count Time**
Zn	5	100
In	13	100
Ni	35	60
Mo	16	60
Co	19	60

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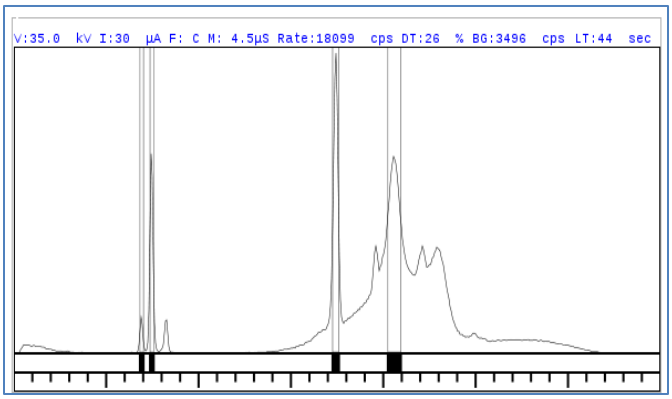
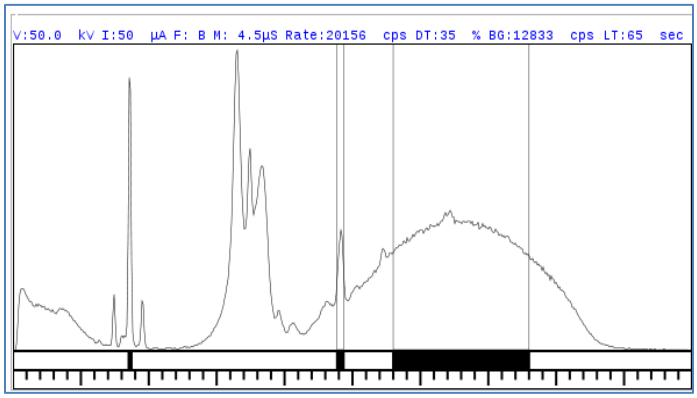
MULTI-ELEMENT ANALYSIS

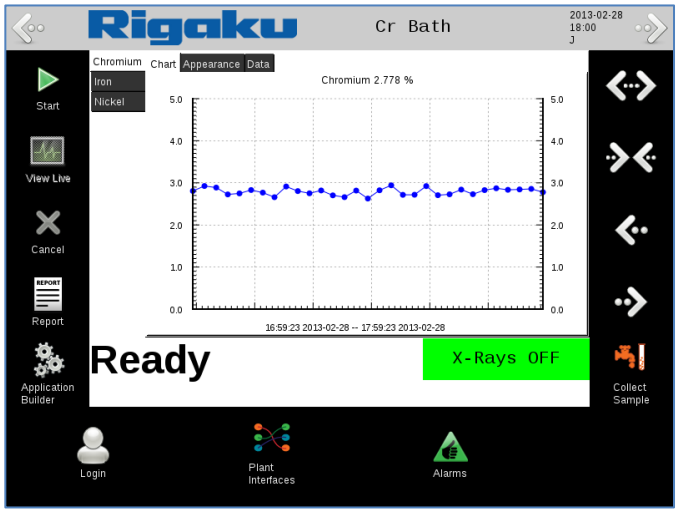
The Rigaku NEX OL system uses a high resolution semiconductor detector that achieves excellent resolution and sensitivity. Adjacent or near-adjacent elements can be measured with little or no peak overlap, as shown in these typical spectra.





NEX OL FEATURES & BENEFITS

- Real-time process control
- Trend analysis charting
- Capable of measuring elements Al to U, depending on application
- Robust Rigaku NEX QC+ optical kernel with SDD detector
- Industrial touch screen user interface
- Unique toolless flow cell design
- No dangerous radioisotopes



CONCLUSION

The NEX OL offers high-throughput manufacturers a simple yet powerful and versatile system for quantifying the elemental composition of their process stream. The results of this study indicate that given stable samples, proper sample handling and proper calibration technique, the Rigaku NEX OL EDXRF can achieve excellent results in monitoring the concentration of chemical solutions used in the manufacturing of copper foil.

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