EDXRF APPLICATION NOTE IRON IN ALUMINUM

#1237

SCOPE

The analysis of iron in aluminum extrussion billets is demonstrated.

BACKGROUND

Often, selection of a bauxite mine site is determined by the alumina, silica and iron content of the ore samples. In producing pure aluminum and aluminum alloys, the measurement of trace and alloy component elements is of vital importance to define



purity and alloy quality. The concentration of iron is especially important during the smelting process to control the grade of the aluminum produced. In order to ensure consistent production, the levels of iron must be closely monitored in the QA/QC process, and a simple and fast analysis is required at the production line in order to maintain the optimum product output. To meet this industry need, Rigaku offers the NEX QC benchtop EDXRF analyzer, a rugged and reliable tool for the non-destructive measurement of iron and other elements in aluminum.

INSTRUMENTATION

Model:	Rigaku NEX QC
X-ray tube:	4 W Ag-anode
Detector:	Semiconductor
Sample Type:	Solid aluminum disks
Window Ring:	Flat
Film:	Mylar
Analysis Time:	100 sec
Environment:	Air



SAMPLE PREPARATION

In the production of aluminum and aluminum alloys, the aluminum is often cast into ingots, blocks, bars or extrusion billets (cylindrical logs). Any sample that fits inside the analysis chamber can be measured as long as it has a flat, smooth surface that covers the 25mm diameter aperture. Such samples can be cut from final product, or test coupons can be made during the production process. The data collected here measures disk sample approximately 8cm diameter and 1cm thick cut from an extrusion billet cylinder.

CALIBRATION

Three assayed standards were used for calibration. Each standard was measured once using a 100 sec analysis time.

Element: Fe SEE: 0.0034 Units: % Correlation: 0.99988				
Sample	Assay Value	Calculated Value		
1	0.0001	0.0031*		
2	0.11	0.1063		
3	0.57	0.5707		



* A linear fit with offset was used. The value for sample 1 containing essentially no Fe essentially defines the zero point of the calibration and is the approximate offset of the fit.

RECOVERY

Each calibration sample was measured against the calibration shown above to demonstrate recovery.

Element: Fe Units: %				
Sample	1	2	3	
Assay Value	0.0001	0.11	0.57	
NEX QC Result	0.0032	0.1097	0.5623	

ANALYSIS OF UNKNOWN SAMPLES

Unknown samples were measured to demonstrate calibration accuracy, with assays provided for comparison to NEX QC results. Each unknown sample was measured against the calibration shown above.

Element: Fe Units: %							
Sample	Α	В	С	D	E	F	G
Assay Value	0.061	0.057	0.062	0.052	0.14	0.127	0.07
NEX QC Result	0.0630	0.0600	0.0618	0.0536	0.1356	0.1275	0.0696

REPEATABILITY

To demonstrate repeatability (precision), unknown sample F was chosen. The sample was measured in 10 repeat analyses using an analysis time of 100 sec per sample without moving the sample between measurements.

Element: Fe Units: %					
Sample	Assay Value	NEX QC Average Result	Std Dev (ஏ)	% Relative	
E	0.14	0.1432	0.0021	1.5	

DETECTION LIMIT (LLD)

The empirical method was used to determine detection limit (LLD, Lower Limit of Detection). Calibration standard 1 was measured as the blank sample. Ten repeat analyses of the blank sample are taken with the sample in static position and the standard deviation (σ) is determined. The LLD is then 3X the measured standard deviation of 10 repeat measurements of the blank sample.

Element: Fe		
LLD	Measurement Time	
26 ppm	100 sec	

QUALITATIVE ANALYSIS

The NEX QC uses a high resolution detector and a 50 kV tube, giving it the versatility to analyze other elements in the aluminum, as well as Fe. The following spectra demonstrate the qualitative power of the analyzer.



Calibration Standard 1 (0.0001% Fe)



Calibration Standard 2 (0.11% Fe)

QUALITATIVE ANALYSIS (continued)



Calibration Standard 3 (0.57% Fe)

CONCLUSION

The results show NEX QC provides excellent performance for the measurement of iron in aluminum. Spectral results also indicate the versatility of the analyzer for qualitative screening of low and high grade aluminum, in addition to capability of screening for other elements in the process. The NEX QC offers a simple and fast means of analysis during the QA/QC process in the production of aluminum and its alloys, as well as for screening at the quarry.