

SCOPE

On-line measurement of cobalt (Co), bromine (Br) and manganese (Mn) in terephthalic acid (TPA) is demonstrated.

BACKGROUND

Terephthalic acid (TPA) and purified terephthalic acid (PTA) are precursors in the making of polyester PET for polyester films, PET bottle resin, textile fabrics, and specialty chemicals. During production and use of TPA and PTA the catalysts Co, Br and Mn must be closely monitored to ensure optimum product quality. Since TPA and PTA are aggressive solutions, an on-line measurement is ideal. Continuous monitoring minimizes lab testing requirements, allows for process optimization, and is a critical step in insuring the end product meets specifications. The Rigaku NEX OL offers a simple and low maintenance on-line analytical technique for trending your process streams. Results are communicated to your plant DCS (distributed control system) via 4-20 mA current loops or MODBUS over Ethernet connection allowing for real time closed loop control.



INSTRUMENTATION

Model:	Rigaku NEX OL Analyzer
Excitation:	Direct
X-ray tube:	50 kV 4 W Ag-anode
Detector:	Silicon Drift Detector
Atmosphere:	Air
Measurement Time:	300 sec



SAMPLE PRESENTATION

No sample preparation treatment is required. To demonstrate performance all samples were measured in a static position using the auxiliary sample input loop.

CALIBRATION

An empirical calibration was built using a set of 8 standards with a measurement time of 300 seconds per sample. To demonstrate performance water-based standards were introduced through the auxiliary sample input loop and analyzed in a static position.

Component	Concentration Range (ppm)	RMS Deviation	R ² Confidence
Co	150 - 650	6.7	0.99853
Br	360 - 1800	7.4	0.99979
Mn	50 - 1000	9.2	0.99923

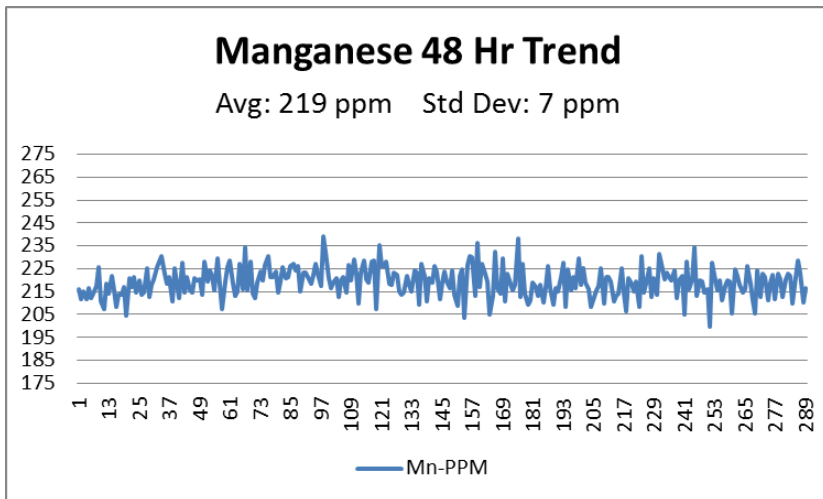
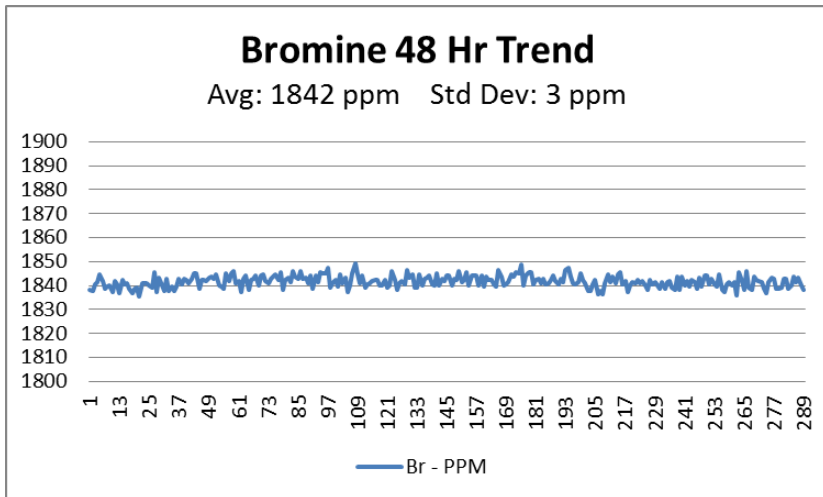
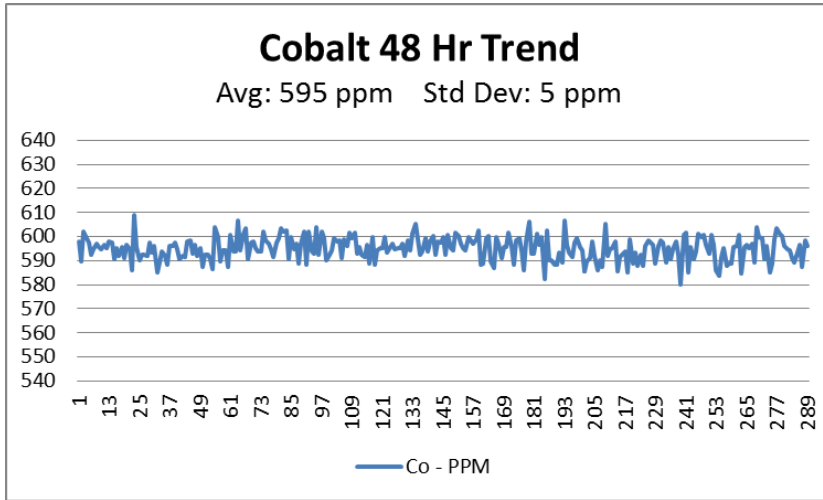
REPEATABILITY (Precision)

To demonstrate repeatability (precision), samples containing low and high amounts of Co, Br and Mn were chosen from the set of calibration standards. Each sample was measured in a static position for ten repeat analyses using a measurement time of 300 seconds per measurement.

Co Units: ppm				
Sample ID	Standard Value	Average Value	Std Dev	% Relative
8	650	653	5	0.8
1	150	147	4	2.7
3	223	220	4	1.8

Br Units: ppm				
Sample ID	Standard Value	Average Value	Std Dev	% Relative
8	1185	1181	5	0.4
1	966	962	4	0.4
3	362	365	2	0.6

Mn Units: ppm				
Sample ID	Standard Value	Average Value	Std Dev	% Relative
8	999	994	9	0.9
1	50	56	5	10
3	864	869	8	0.9



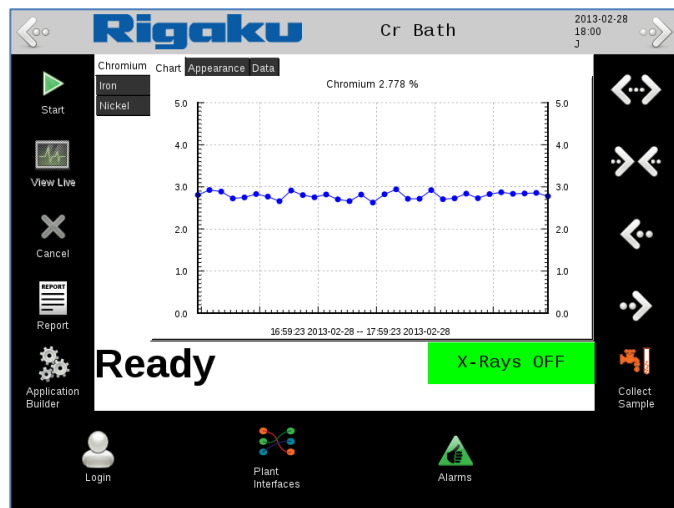
TYPICAL DETECTION LIMITS

To determine the Lower Limit of Detection (LLD) using the empirical method, ten repeat analyses of a blank sample (DI water) is measured and the standard deviation calculated. The LLD is then defined as three times the standard deviation. The following detection limits are shown using a measurement time of 300 seconds per measurement.

Element	LLD
Co	8 ppm
Br	14 ppm
Mn	12 ppm

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



General example of trend charting

CONCLUSION

The NEX OL offers real time on-line trend analysis in a simple yet powerful and versatile system for quantifying the elemental composition of a 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 Co, Br and Mn in TPA and PTA solutions.