APPLICATION NOTE - XSP-AN-001

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X-SUPREME8000

Benchtop X-Supreme8000 Analyser

Hitachi's X-Supreme8000 for the rapid determination of Co, Mn, and Br used as a catalyst in the production of Purified Terephthalic Acid (PTA)

INTRODUCTION

In the manufacturing process of polyester (PET), i.e. polyester fibre, polyester film and polyethylene terephthalic (PET) bottle resin, an intermediate product is a white crystalline powder called Puri ied Terephthalic Acid (PTA). This powder is produced by an oxidation process and a catalyst containing Co, Mn, and Br.

Analysis of these critical catalyst elements is required at various stages in the production process to ensure product quality and efficient high sample throughput.

The X-Supreme8000 is a simple to use benchtop Energy Dispersive X-ray Fluorescence (EDXRF) analyser offering rapid, accurate, simultaneous analysis of Co, Mn and Br. It can be operated by either laboratory or production staff, on a 24/7 basis leading to high quality analysis with potential cost savings.

SAMPLE PREPARATION AND PRESENTATION

To demonstrate the performance of the X-Supreme8000 for the analysis of Co, Mn and Br, a series of liquid acetic acid samples containing Co, Mn and Br at known concentrations were measured to derive a calibration. In this case the liquid was simply poured directly into the sample cell, i.e. no weighing or volumetric measurements are required, and the cell placed into one of the ten positions on the in-built sample carousel.

Note: At each carousel tray position a secondary safety window was also fitted which is used to capture any liquid in the case of sample leakage, ensuring instrument protection and long term instrument reliability.

In addition up to 10 samples can be loaded at a time allowing unattended operation leading to maximum efficiency.

For the determination of Co, Mn, and Br, the X-Supreme8000 uses the combined power of a field proven high reliability Tungsten target X-ray tube, and high resolution of the Silicon Drift Detector with optimised primary beam filters (automatically selected in the calibration) to give the highest performance with lowest limits of detection.

CALIBRATION

An elemental spectrum was first derived as shown in Figure 1, which shows the X-ray energy positions for the elements manganese and cobalt, for two samples with different concentrations. From this an X-ray “Region of Interest” (ROI) was derived and a series of samples measured, giving the calibration line shown in Figure 2 and Table 1.

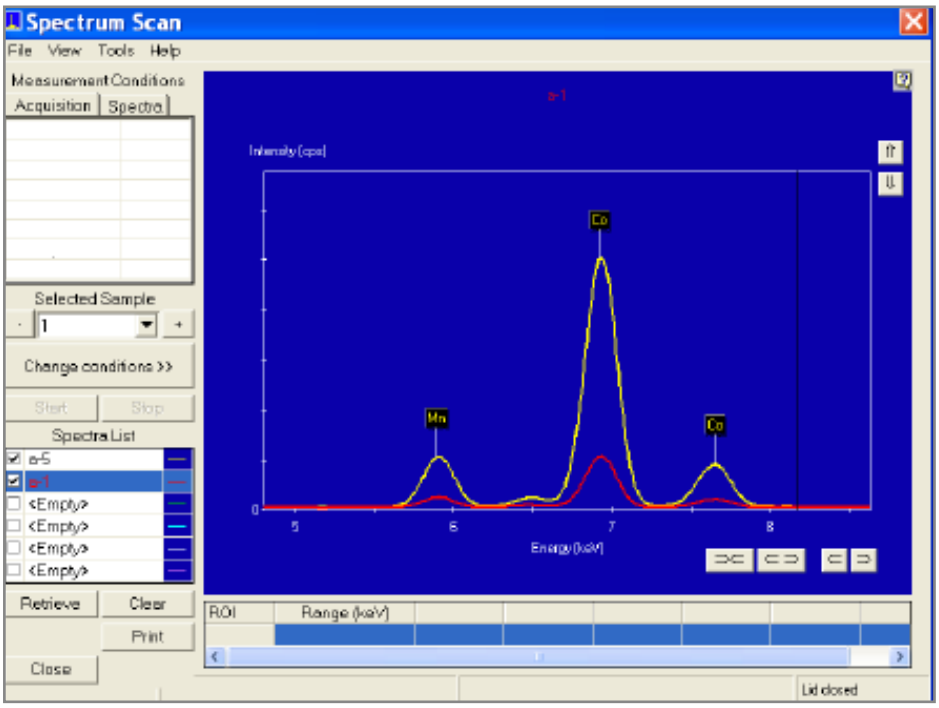

Figure 1: Spectrum display showing Mn (X-ray energy of 5.9 keV) and Co (6.9 keV)

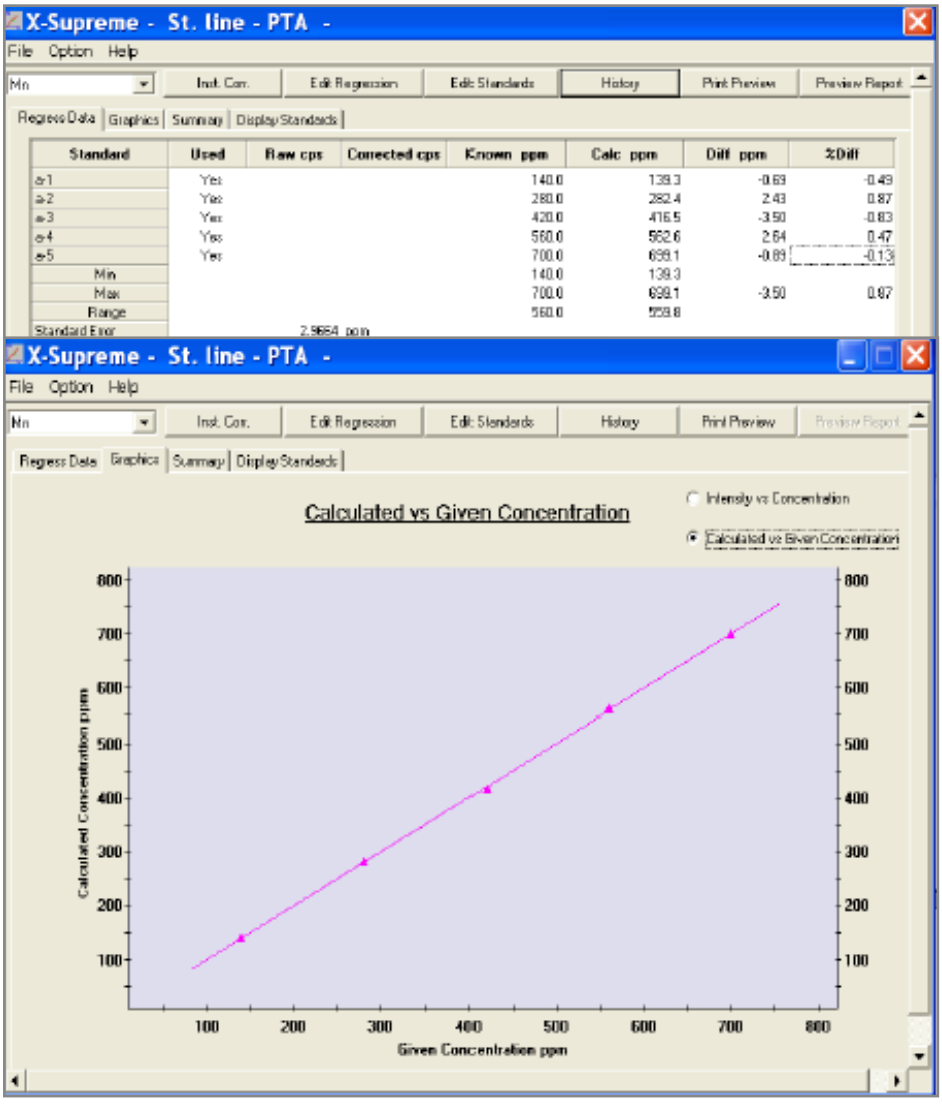

Figure 2: Calibration and regression line for Mn)

This procedure was repeated for Co and Br and Table 1 is a summary of the overall calibration performance.

PERFORMANCE AND RESULTS

Using the calibrations shown above, the following is a summary of the analytical performance obtained on the X-Supreme8000 instrument:

Analyte	Concentration range (ppm)	Standard error (ppm)	Theoretical limit of detection- 3 sigma (ppm)	Limit of quantification -10 sigma (ppm)	Precision at mid range **95% confidence (ppm)**	Measurement time (simultaneous seconds)
Mn	140-700	4	5	15	10	
Co	300-1500	4	4	13	12	90
Br	400-2000	6	3	10	6	

Table 1: Summary of results measuring Mn, Co and Br.

Using the above calibration curve a sample was measured four times, i.e. leaving the sample in the instrument and taking four repeat readings, to demonstrate instrument repeatability (precision). Results are shown below:

Reading	Mn (ppm)	Co (ppm)	Br (ppm)
#1	421	902	1205
#2	425	910	1210
#3	424	908	1211
#4	427	915	1210
Average	424	909	1209
STD Dev (1 sigma)	2.5	5.4	2.7

X-SUPREME8000 INSTRUMENT CONFIGURATION

- X-Supreme8000 fitted with W tube and high performance SDD.
- Disposable sample cells P/No 54-LX6922.
- Instrument parameters: 26 kV, 105uA, W5, 90s, Air path.
- Helium purge option (if acidic environment).

Note: For those users in the petroleum sector who wish to measure sulphur in oil as the principal application and Mn, Co, Br as a secondary application a Ti target X-ray tube X-Supreme8000 can also be used giving similar results.

CONCLUSION

The above data shows the X-Supreme8000 offers simple, rapid, cost-effective quality control analysis of Mn, Co and Br in catalyst materials used in the production of Purified Terephthalic Acid (PTA).

The field proven X-Supreme8000 can be located close to the production process and operated by production personnel, ensuring continuous production thereby minimising any cost associated with delays in the production process.

Visit **www.hitachi-hightech.com/hha** for more information.

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