Lab Report XRF 172

S2 PUMA Series 2

- Detecting low levels of Pt in wastewater products

Introduction

Platinum (Pt) is a precious metal which is used in various applications in medicine, electronic devices, jewelries, and chemical catalysts. However, a substantial amount of Pt is lost in waste catalysts, electronic scraps and industrial wastewater. The limited geological reserve and high cost of extraction make it an imminent task to recover lost Pt from various Pt-containing waste materials. Pt is often recovered through leaching automotive catalytic converters (or electronic scraps) followed by extraction using ion exchange or electrolysis.

At the final stage however, often after some additional treatment, Pt concentrations are still as high as 20 mg/L.

The S2 PUMA Series 2 energie-dispersive X-ray fluorescence (EDXRF) spectrometer can quantify traces of metals such as Pt quickly and reliably. The liquid samples can be analyzed without any preparation (like dilution or filtration) besides filling them into sample cups. This decreases the time-to-result substantially, enhancing the sample throughput.

Instrumentation

The S2 PUMA is perfectly equipped for industrial environments. The combination of a 50 Watt X-ray tube with closely-coupled beam optics and the new HighSense™ detector technology enables optimal sample excitation, resulting in outstanding analytical performance (Figure 1).

The S2 PUMA is equipped with Bruker's unique SampleCare™ technology, a multi-layer system that protects the instrument's vital components from accidental spills or sample breakages. This guarantees a high system uptime and easy maintenance. The intuitive TouchControl™ interface allows for independent routine operation in island mode, without external PC.

Sample Preparation

A set of six secondary standard samples (including a blank sample) with varying concentrations of Pt was used for the calibration. First, the samples were mixed for 30 s using a vortex mixer. Then 10 g per sample were filled into liquid cups with a thin film window (Figure 2). For heavy analytes like Pt the foil does not affect the measured intensities.

Figure 2: Preparation of liquid samples.

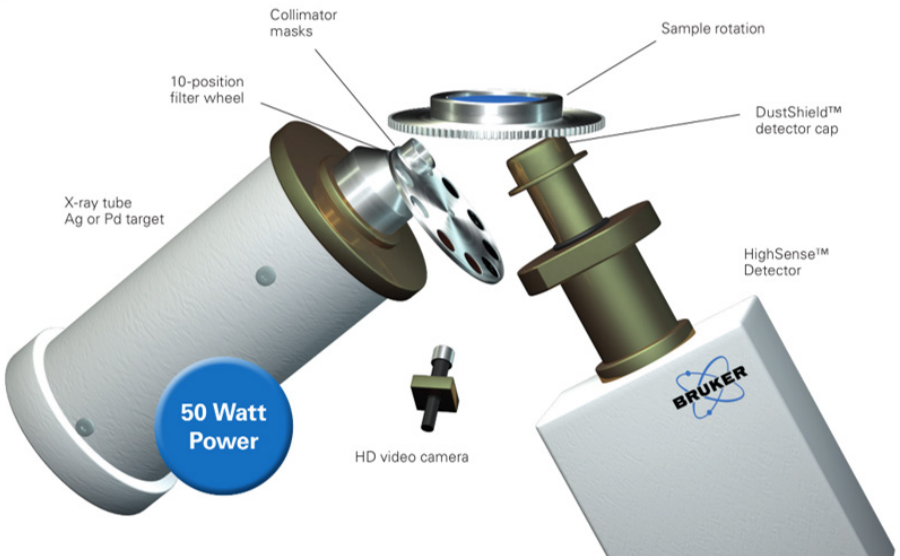

Figure 1: HighSense™ beam path of the S2 PUMA Series 2.

Voltage [kV]	20
Current [mA]	Automatic
Filter	500 µm Al
Rotation	Yes
Time [s]	180

Table 1: Measurement Parameters.

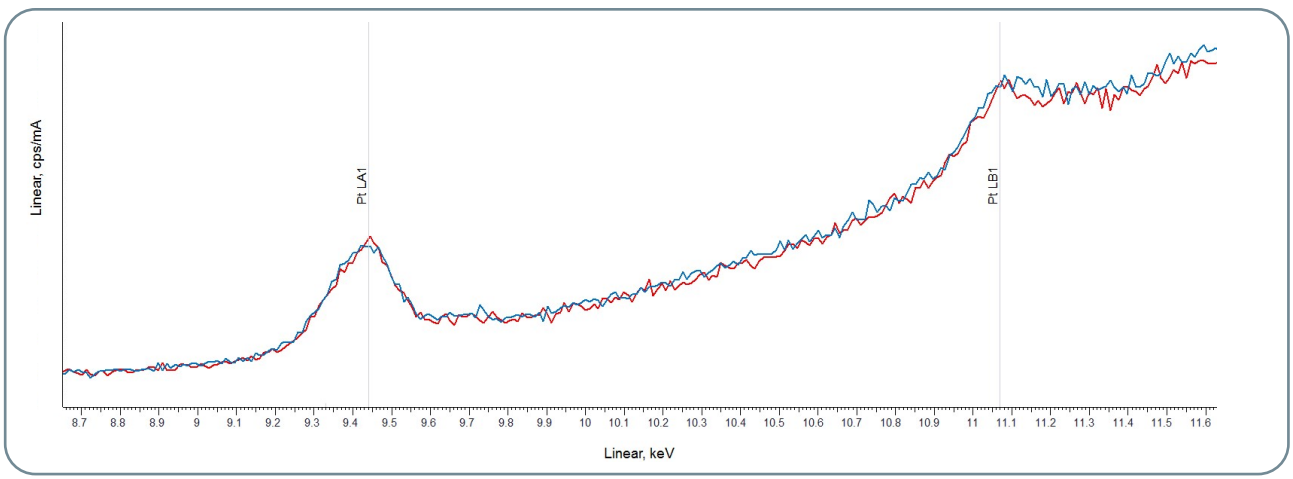

Figure 3: Comparison of measurement in He and air mode.

Measurement Method

Measuring liquid samples requires Helium or air mode. Heavy analytes (heavier than S) can be conveniently measured in air mode (Figure 3). The parameters for this method are listed in Table 1. The counting time was 3 min. The sample rotation allows to obtain representative bulk compositions, even for slightly heterogeneous samples. The automatic current setting adjusts the current to reach the highest count rates and thus optimal performance.

Calibration

The calibration curve and exemplary Pt peaks are shown in Figure 4. The curve shows excellent correlation without applying any matrix corrections. The achievable detection limit is 0.3 ppm (mg/kg) Pt. Integrating over the peak increases the detected intensities and improves the measurement statistics.

Precision Testing

Ten preparations of a secondary standard that was not part of the calibration set, were measured. Each time fresh material and a new liquid cup were being used. The low standard deviation of the results (Table 2) demonstrates the suitability of the sample preparation and the outstanding stability of the S2 PUMA Series 2.

Repetition	Pt [mg/kg]
01	14.7
02	14.8
03	14.9
04	14.4
05	15.0
06	14.9
07	15.0
08	14.4
09	14.9
10	14.8
Average **Std. Dev.** **Rel. Std. Dev.**	**14.8** 0.2 1.6 %

Table 2: Analytical precision test using a secondary standard containing 15 mg/kg Pt.

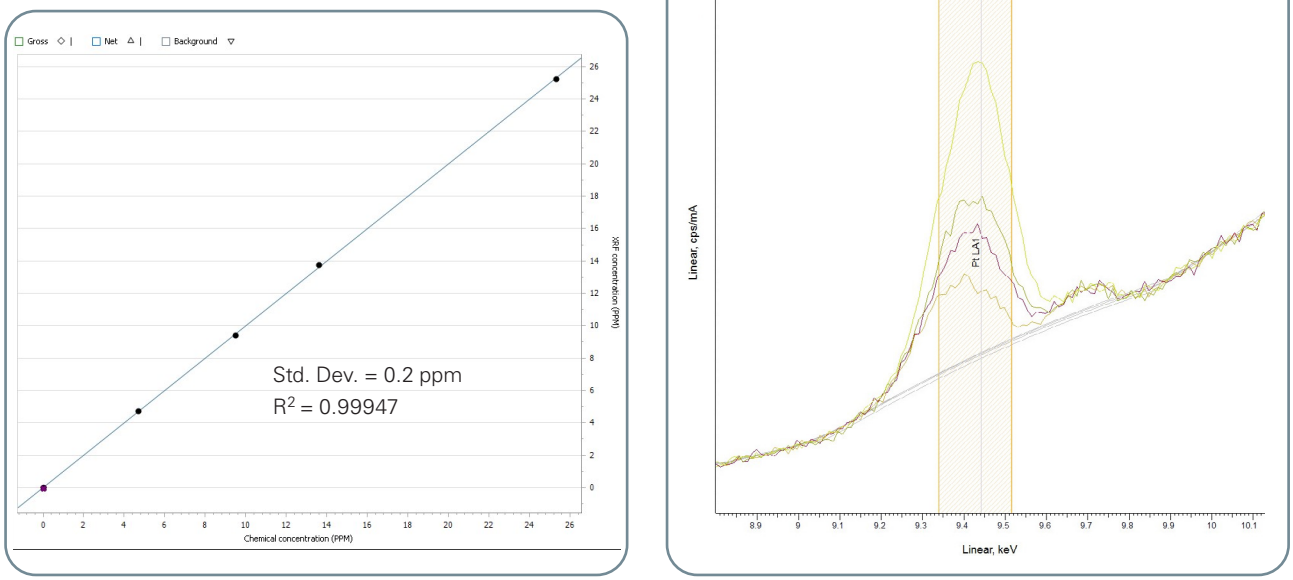

Figure 4: Calibration details. Left: Calibration curve. Right: Overlaid spectra of the standards. The integrated range used for the calibration is indicated in yellow.

SampleCare™

Bruker's unique SampleCare technology (Figure 5) protects the vital system components in the event of sample breakage or liquid cup leakage. The sample detection system prevents the measurement of liquid or powder samples under vacuum. SampleCare is key for the highest system uptime and short & easy maintenance.

Conclusion

This lab report summarizes the outstanding performance of the S2 PUMA analyzing trace levels of Pt in wastewater samples, combining easy & fast preparation, short-measurement time and minimal costs of operation.

The S2 PUMA EDXRF spectrometer is ready for challenging industrial environments and applications with its TouchControl interface and its SampleCare technology. The 22-position XY Autochanger (Figure 6) enables high sample throughput while keeping all fexibility. Mixed batches can be loaded (liquid, powder, solid, ...) and samples can be added at any time. You need to change priorities and quickly analyze a production sample? No problem with the S2 PUMA XY Autochanger!

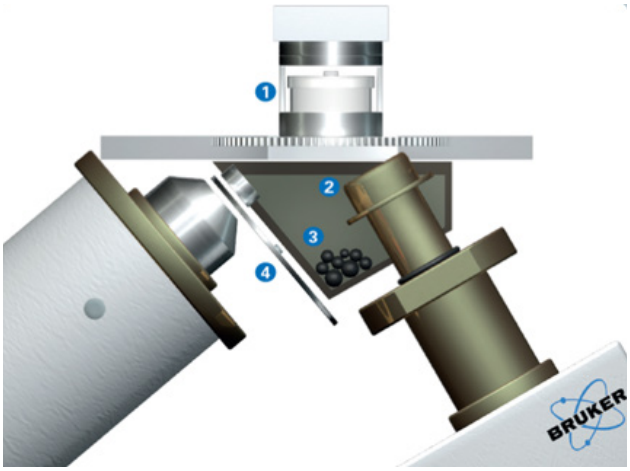

Figure 5: SampleCare™:
1. Grabber with automatic sample detection.
2. DustShield™ detector cap.
3. Dust reservoir.
4. Filter wheel.

Figure 6: XY Autochanger with 20-position EasyLoad tray and 2 extra fixed positions.

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