APPLICATION NOTE - 24

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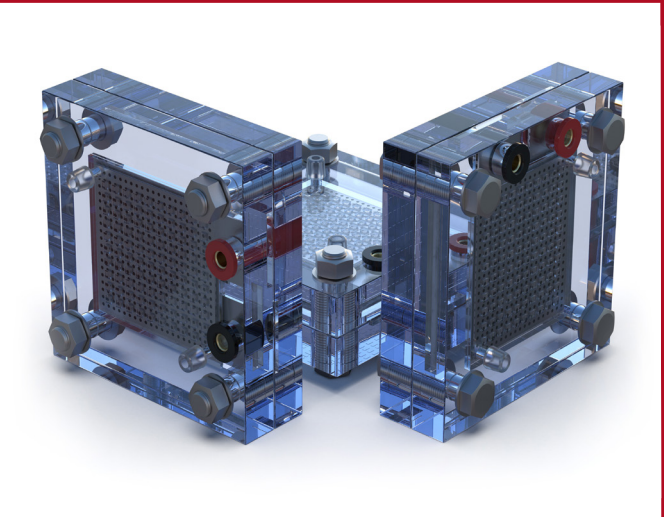
LAB-X5000



The LAB-X5000 benchtop analyzers present many advantages:

- **Compact and robust:** ideally suited in a lab and near the production line
- **Easy to use:** minimum operator training needed
- **Sample preparation to results in minutes:** fast decision making for consistent product quality
- **Sample spinner:** reliable and repeatable results, even for samples with uneven surfaces

LAB-X5000 for the rapid determination of platinum coating weight on film used in PEM fuel cells

INTRODUCTION

In the race to reduce carbon emissions, fuel cells technology is developing rapidly. All types of fuel cells include three basic components: two electrodes (anode and cathode), and an electrolyte sandwiched between them. In the case of polymer electrolyte membrane (PEM) fuel cells, also called proton exchange membrane fuel cells, the electrodes are typically coated with conductive carbon mixed with platinum (Pt) particles. Pt acts as a catalyst for the continuous conversion of fuel (hydrogen) and oxidant (oxygen) into electricity.

In the fabrication process, it is critical to control the amount or coating weight of Pt on the electrode's surface to ensure efficient chemical reactions in the cell, while minimizing production costs (Pt is very expensive).

Energy-dispersive X-ray fluorescence (EDXRF) spectrometry is one of the simplest elemental analysis techniques that can be used for the rapid control of Pt coating weight on film or fabric. The sample preparation is straightforward and the analysis is fast.

COATING ANALYSIS MADE EASY

With the Hitachi High-Tech LAB-X5000 EDXRF benchtop analyzer, the determination of Pt coating weight on film couldn't be easier. Once the LAB-X is calibrated, routine analysis is carried out by placing the sample in the instrument's analysis port and pressing a button to start the measurement. Preliminary results are displayed within seconds on the analyzer's large, industrial LCD touchscreen, showing the Pt

coating weight. Pass/Fail messages can also be setup for fast decision making and process adjustments, ensuring consistent product quality.

The LAB-X5000 is calibrated by measuring a series of well-characterized samples. Setting-up samples (drift correction monitors) are measured at the time of the calibration, and again in the unlikely event that the analyzer drifts, so there is no need to source calibration standards again or re-measure them, saving both time and money.

The combination of a high-resolution detector and optimized calibration parameters ensure that you get results you can trust. A spinner rotates the sample during the analysis to provide a result that is representative of the sample's surface.

With up to 100,000 results stored on the analyzer itself, operators can view new and old results easily, print them on the optional integrated printer for a hard-copy record, download them on a USB memory device as a CSV file, and upload them to our ExTOPE Connect cloud service or a local server via Wi-Fi for real-time access to the data anytime, anywhere.

SAMPLE PREPARATION

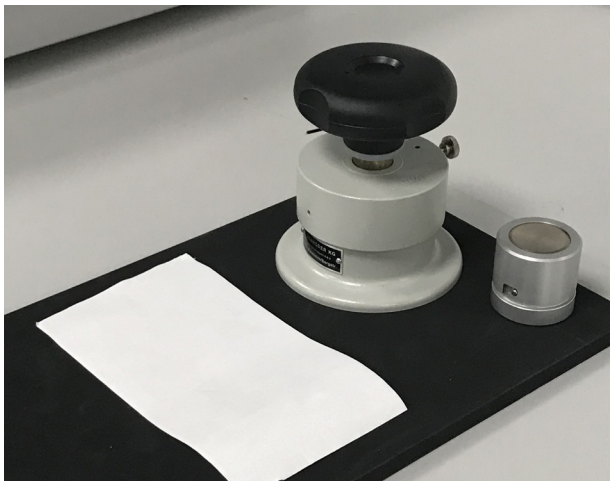
The sample preparation is very simple and only takes a few seconds: operators place the film sample on the cutting mat, cut a disk off using the cutter provided, and place the film (surface to be analyzed facing up) on the specially-design sample holder. The film sample is locked in place on the holder, and this is then placed in the LAB-X5000's analysis port. All that is left to do is to press the Start button on the analyzer to start the measurement.

PERFORMANCE AND RESULTS

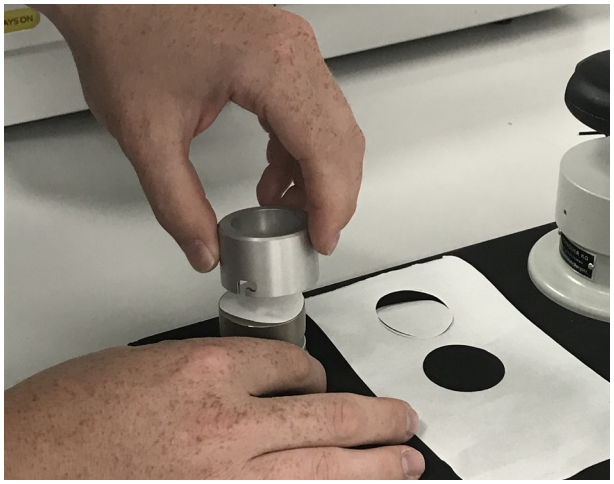
For this application, the LAB-X5000 was calibrated by measuring a series of well-characterized film samples to establish the relationship between the platinum content and its X-ray signal.

Table 1 shows the typical calibration performance the LAB-X delivers for the determination of platinum coating weight on film.

The limit of detection (LOD) was calculated from the results of 10 repeat measurements of a uncoated film, the precision from 10 repeats of samples coated with carbon/platinum. The analysis time was set to 60 seconds.



Sample cutter, cutting mat, and sample holder



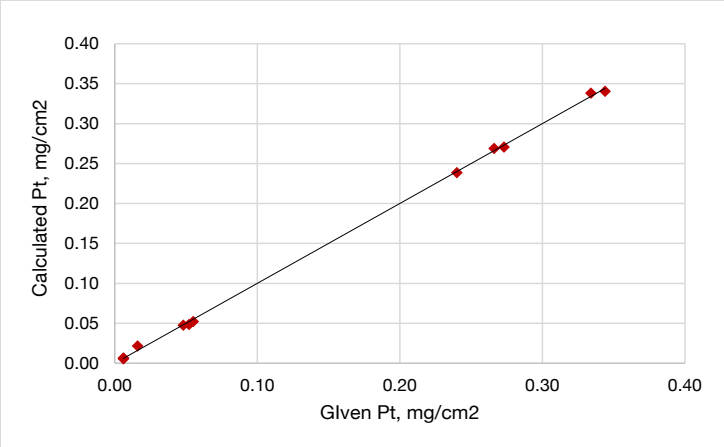
Preparing the sample for analysis

Table 1: Typical calibration performance for the determintion of Pt coating weight on film

Analyte	Calibration range (mg/cm^2)	Standard error of calibration (mg/cm^2)	Guaranteed limit of detection (3σ) (mg/cm^2)	Limit of quantification (10σ) (mg/cm^2)	Precision (95% confidence) (mg/cm^2)
Pt	0 – 0.35	0.004	< 0.001	0.002	0.001 at 0.050 0.003 at 0.265 0.003 at 0.330

To obtain lower limits of detection (LOD) and quantification (LOQ), longer measurement times can be used (to halve the LOD and LOQ, you need to increase the measurement time by a factor of 4).

Graph 1: Calibration graph for Platinum



SUMMARY

Once calibrated, Hitachi High-Tech's LAB-X5000 provides cost-effective, accurate and repeatable determination of platinum coating weight on film, enabling operators to make process decisions fast and ensure products meet specifications. The analyzer's ease of use and ruggedness make it an ideal quality control tool close to the production line, in the plant's laboratory, or in a research facility, delivering results fast for maximum productivity.

ORDERING INFORMATION

The instrument configuration and accessories used to produce the data in this application note were as follows:

- LAB-X5000 with Pd-target X-ray tube
- Sample spinner
- LAB-X5000 standard accessories pack
- Paper/film accessories pack
- Setting-up samples: customer to use samples with the highest Pt coating weight, and an uncoated film.

Alternatively, you can order the LAB-X5000 "Si coating weight on paper" package.

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

ADDENDUM: CALIBRATION PARAMETERS

The performance shown in this application note was achieved on the LAB-X5000 calibrated using the parameters in the tables below.

Table 2: Analytes and conditions

Analyte	Condition	Measurement time (seconds)	Region of interest (keV)	Used in calibration
Pt	30kV 100µA Z1 filter Sample spinner ON	60	9.29 - 9.58	Yes
Si*			1.66 - 1.83	No

* Si is included in the calibration to correct for matrix variation effects.

Table 3: Display settings

Analyte	Display name	Display unit	Number of decimals
Pt	Pt	mg/cm^2	3
Si	Si	n/a	n/a

Table 4: Regression

Analyte	Correction model
Pt	Intensity correction: Si

Table 5: Restandardization

Analyte	Low SUS	High SUS
Pt	Uncoated film	Sample with highest Pt
Si	Uncoated film	Sample with highest Si