

LAB-X5000

LAB-X5000 for the rapid determination of catalysts residue and additives in PET

INTRODUCTION

Polyethylene terephthalate (PET or PETE) is used in a wide variety of applications. Its clarity, light weight and high strength make it a choice material for the packaging of foods and beverages, household products, toiletries, etc. In its fiber form, it is known as Polyester, and is used in the fabrication of fabrics, textiles, carpets... Another advantage of PET is that it is fully recyclable.

PET is produced by the polymerization of ethylene glycol and terephthalic acid. Catalysts such as antimony (Sb), cobalt (Co), or titanium (Ti) oxides are added to the mix to speed up the chemical reaction and optimize plant capacity. Other functional additives are pigments, fillers, heat and light stabilizers, plasticizers, slip agents, biocides and flame retardants.

The content of catalysts residue and additives is monitored during production to ensure quality: too little of the additive(s) can have major effects on future processing, and too much can make the PET unreliable and even unsafe. Unlike other techniques such as titration, atomic absorption, or inductively coupled plasma, energy dispersive X-ray fluorescence (EDXRF) is rapid, simple, and does not require the use of chemicals and time-consuming sample preparation. With results available in seconds, it can be used by any operator with minimal training for rapid process and quality control of PET pellets.

POLYETHYLENE TEREPHTHALATE ANALYSIS MADE EASY

With the Hitachi High-Tech LAB-X5000 EDXRF benchtop analyzer, the analysis of PET pellets couldn't be easier. Once the LAB-X is calibrated, routine analysis is carried out by placing the pellets in a sample cup, placing the cup in the instrument's analysis port, and pressing a button to start the measurement. Results are displayed within seconds on the analyzer's large, industrial LCD touchscreen, showing the elements' content. Pass/Fail messages can also be setup for fast decision making and process adjustments, reducing material waste and ensuring that what comes out of the production line meets specifications.

The LAB-X5000 is calibrated by measuring a series of assayed 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. The advantage is that there is no need to source calibration standards again and measure them, saving both time and money.

A sample spinner is used for this application to compensate for sample inhomogeneity and deliver repeatable results.



The LAB-X5000 benchtop analyzers present many advantages:

- **Compact and robust:** ideally suited in a lab and near the production line
- **Easy to use:** insert the sample cup and press Start
- **Reliable:** low cost of ownership
- **Fast:** results in seconds for fast decision making

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

The LAB-X5000 can determine a wide range of catalysts and additives in PET, and examples are shown in Performance and Results. To discuss other specific application requirements, please contact your local Hitachi High-Tech representative.

SAMPLE PREPARATION

The sample preparation is simple:

- Prepare a secondary safety window with Poly4 film
- Pour the small pellets directly in the safety window, up to the internal line. Because of its large diameter, the safety window allows the granules or pellets to sit uniformly over the analysis area and increases results reproducibility
- Gently shake the prepared sample (side to side motion) to spread the pellets inside the safety window more evenly
- Place the sample in the LAB-X's analysis port and press the green button to start the analysis.

PERFORMANCE AND RESULTS

For each application, the LAB-X5000 was calibrated by measuring a series of assayed samples to establish the relationship between the elements' content and their X-ray signal.

The following tables show the optimized parameters used and the typical calibration performance the LAB-X delivers for each calibration.

The limits of detection (LOD) were calculated from the results of 10 repeat measurements of a blank sample, and the precision from 10 repeats of samples containing the elements of interest.

Antimony (Sb), Titanium (Ti), Manganese (Mn) and Cobalt (Co) in PET

Table 1a: Optimized calibration parameters

Analyte	Region of interest (keV)	Excitation condition	Sample spinner	Regression model	Measurement time (seconds)
Sb	3.50 – 3.70	13kV 230 μ A W1 filter	ON	Linear	240
Ti	4.40 – 4.62	20kV 150 μ A A6 filter		Linear	
Mn	5.78 – 6.01			Linear	240
Co	6.80 – 7.05			Linear	

Table 1b: Typical calibration performance for Sb, Ti, Mn and Co in PET

Analyte	Calibration range (ppm)	Standard error of calibration (ppm)	Guaranteed limit of detection (3σ) (ppm)	Limit of quantification (10σ) (ppm)	Precision (95% confidence) (ppm)
Sb	0 - 151	2.5	2.0	4.4	1.9 at 90 ppm
Ti	0 - 10	1.9	2.7	6.0	1.3 at 7 ppm
Mn	0 - 73	2.2	1.1	2.5	0.7 at 64 ppm
Co	0 - 14	1.0	1.0	2.1	0.5 at 9 ppm



Sample introduction



Starting the analysis

Titanium Dioxide (TiO₂) in PET

Table 2a: Optimized calibration parameters

Analyte	Region of interest (keV)	Excitation condition	Sample spinner	Regression model	Measurement time (seconds)
TiO ₂	4.40 – 4.62	20kV 150μA A6 Filter	ON	Self-absorption	40

Table 2b: Typical calibration performance for TiO₂ in PET

Analyte	Calibration range (%)	Standard error of calibration (%)	Guaranteed limit of detection (3σ) (%)	Limit of quantification (10σ) (%)	Precision (95% confidence) (%)
TiO ₂	0.04 – 2.3	0.01	n/a	n/a	0.005 at 0.3 %

Sulfur (S) in PET

Table 3a: Optimized calibration parameters

Analyte	Region of interest (keV)	Excitation condition	Sample spinner	Regression model	Measurement time (seconds)
S	2.22 – 2.40	13kV 230μA W1 Filter	ON	Self-absorption	60

Table 3b: Typical calibration performance for S in PET

Analyte	Calibration range (%)	Standard error of calibration (%)	Guaranteed limit of detection (3σ) (%)	Limit of quantification (10σ) (%)	Precision (95% confidence) (%)
S	0.2 – 1.1	0.01	n/a	n/a	0.003 at 0.2 %

Barium (Ba) in PET

Table 4a: Optimized calibration parameters

Analyte	Region of interest (keV)	Excitation condition	Sample spinner	Regression model	Measurement time (seconds)
Ba	4.36 – 4.57	20kV 150μA A6 Filter	ON	Linear	90

Table 4b: Typical calibration performance for Ba in PET

Analyte	Calibration range (%)	Standard error of calibration (%)	Guaranteed limit of detection (3σ) (%)	Limit of quantification (10σ) (%)	Precision (95% confidence) (%)
Ba	0.10 – 0.26	0.004	n/a	n/a	0.002 at 0.14 %

SUMMARY

Once calibrated, Hitachi High-Tech's LAB-X5000 provides accurate and repeatable determination of a wide range of elements in PET. The analyzer's ease of use and ruggedness make it an ideal tool alongside production and in the laboratory. With results available in seconds, operators can accept/reject products before they are introduced into production (incoming inspection), and make immediate adjustments to the manufacturing process to ensure consistent quality day after day while minimizing material waste and its associated costs.



ORDERING INFORMATION

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

- | LAB-X5000 with Ti-target X-ray tube
- | Sample spinner
- | Secondary safety windows
(3 included in LAB-X Basic accessories pack P/No. 10009936)
- | Poly4 sample film (P/No. 10000780; 100m roll).

Setting-up samples (SUSs):

- | Please consult our Applications specialists to identify suitable SUSs for your application
- | Alternatively, you can mold or press a blank polymer and the highest standard in your calibration into 35mm- or 40mm-diameter discs (4 to 5mm thick), and use these as low and high SUSs
- | 35 to 40mm-diameter pellet holder (P/No. 10001316) if using pressed PET discs as SUSs.

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



Hitachi High-Tech Analytical Science

This publication is the copyright of Hitachi High-Tech Analytical Science and provides outline information only, which (unless agreed by the company in writing) may not be used, applied or reproduced for any purpose or form part of any order or contract or regarded as the representation relating to the products or services concerned. Hitachi High-Tech Analytical Science's policy is one of continued improvement. The company reserves the right to alter, without notice the specification, design or conditions of supply of any product or service.

Hitachi High-Tech Analytical Science acknowledges all trademarks and registrations.

© Hitachi High-Tech Analytical Science, 2020. All rights reserved.