

Hitachi's X-Supreme8000 for the determination of Silicone coating weight on paper and films

Instrument Package 10011625

INTRODUCTION

Silicone coated papers are widely used as release liners, with the major application being stick-on labels of all types. Other uses include double-sided tapes and decorative trims. In all cases the silicone-coated paper protects a pressure-sensitive adhesive layer until it is needed, at which time the paper is peeled away. It is important to closely control the amount of silicone applied because too little will prevent the protective paper from being removed cleanly and too much is a waste of expensive material. Benchtop X-ray fluorescence (XRF) analyzers have been used for nearly twenty years to provide a cost-effective answer to this problem, offering rapid, precise analysis with minimal sample preparation and operation by non-laboratory staff. The success of the method is that for the thin layers of silicone there is a simple linear relationship between the XRF silicon signal and the coating weight.

For this analysis, Hitachi offers the X-Supreme, a compact, rugged, and user-friendly instrument that can easily fit into a testing facility.

INSTRUMENTAL

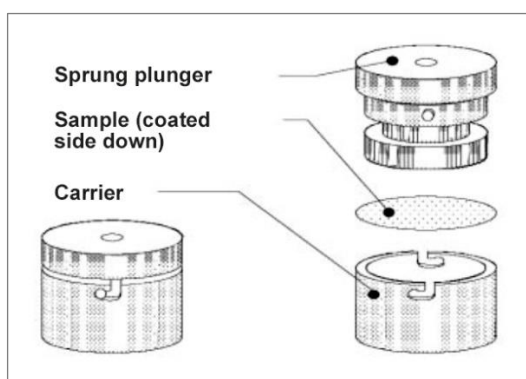
To obtain the best performance for low atomic number elements, the Hitachi X-Supreme includes the Focus SD analysis head. Focus SD combines a field-proven X-ray tube and a high-performance, high-resolution silicon drift detector (SDD), to deliver optimal sensitivity for the determination of silicone coating weight on paper.

All control of the instrument is through the X-Supreme's integrated PC and software. Although the X-Supreme's integrated PC and software offer the expert analyst powerful methodology, to the routine user it provides simplicity of use and flexibility with no requirement for specialist training. The software features easy data manipulation and storage, a report writing facility and data export. Its compactness and robustness make the X-Supreme ideal for location either in a laboratory or in a production site for twenty-four-hour operation.

The X-Supreme includes a ten-position autosampler to enable simple and unattended multiple analysis.

SAMPLE PREPARATION AND PRESENTATION

Sample preparation is simply a matter of punching out a 35 mm diameter disc from the paper sheet using Hitachi's sample cutter and placing it in Hitachi's special paper holder. The holder ensures each piece of paper is flat and provides a consistent x-ray signal for best accuracy and precision. The prepared holder is then placed on the sample tray in the instrument, a sample label is entered at the integrated keypad, data entry confirmed, and the measurement starts "at the press of a button". Results are presented on the integrated flat panel monitor. Additionally, the results can be printed in a standard or user-defined format and transmitted through an ethernet interface in the same format.



CALIBRATION

Each X-Supreme comes pre-programmed with operating parameters that are optimized for this application. It is then simply a matter of following the Hitachi method sheet and running at least six standards with known silicone coating weights.

QUALITY CONTROL AND INSTRUMENT CORRECTION

Setting-up-samples (SUSs) are measured at the time of calibration. They act as long term reference for the sensitivity of each element's X-rays. From time to time, the instrument needs restandardizing by measuring the setting-up-samples supplied in this package.

Capitalizing on the excellent stability of the X-Supreme, the best strategy is regular measurement of a quality control (QC) sample and only to restandardize when a result exceeds control limits. This is an easy process on the X-Supreme when using the QC check sample routine. This routine displays the QC check sample's results over time, in both graphical and numerical format, allowing a rapid assessment. If the results are inside customer specified tolerances then routine analysis can proceed but if outside then restandardisation is required.

PRE-DEFINED METHOD FOR PACKAGE 10011625

The X-Supreme comes pre-loaded with one method. This method's parameters can be used to derive calibrations for "normal" paper as well as clay-coated, clay-filled paper and/or silicone extractables, providing you have one calibration per sample type.

TYPICAL PERFORMANCE AND RESULTS

Table 1 shows typical calibration performance that illustrates how the X-Supreme readily covers the range of specifications for silicone-coated paper.

TABLE 1: TYPICAL PERFORMANCE FOR X-SUPREME FOR SILICONE ON PAPER

Analyte	Calibration range (g.m ⁻²)	Total analysis time (seconds)	Calibration standard error (g.m ⁻²)	Guaranteed limit of detection (g.m ⁻²)	Precision at mid-range (95% confidence) (g.m ⁻²)
Si	0.2 - 2.3	50	0.01	0.004	0.003

Note:

1. For a full description of applying an XRF benchtop analyzer to the control of silicone coating weight see the paper "Calibration, Maintenance and Operation of XRF Spectrometers for Silicone Coatweight Measurement," L Price and L Morrison, Spectroscopy, Vol. 7, No. 6, July/August 1992, pp 32-38. Although this refers to a LAB-X1000 the techniques it describes are relevant to the X-Supreme.
2. For a description of the "Determination of silicone extractables by coat weight analysis" see the article in the magazine "Labels and Labelling" dated September/October 1996 by Dow Corning SA.