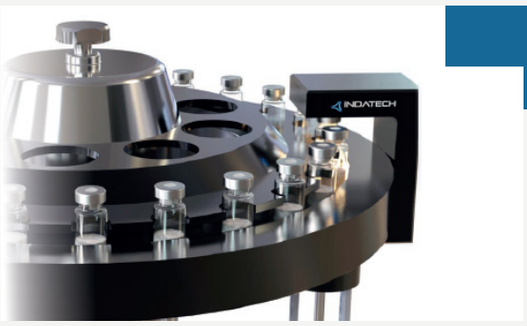
SAM-Spec®

CHAUVIN ARNOUX INDATECH

Pharma Biotech



Analysis of lyophilized vaccines using our SAM-Spec® multipoint NIR technology

Because of its direct influence on public health, the pharmaceutical industry attaches great importance to quality control of its products. This is particularly significant in the case of freeze-dried or lyophilized vaccines, for which features such as the moisture level and physical appearance of the lyocakes are strategically important. Strict quality control thus becomes essential to ensure patients' safety and comply with the rigorous standards governing this crucial industry.

Goal: inspection of the moisture level and structural defects in lyophilized samples

The goal of this study is to quantify the level of residual moisture in lyophilized vaccine samples and analyze appearance defects in the lyocakes by means of spatially-resolved near-infrared spectroscopy NIR-SRS. This experimental approach was implemented in collaboration with a customer whose identity and that of the product examined have been withheld for reasons of confidentiality. The sample analyses were performed in static mode at the scale of the laboratory to demonstrate the capability of the SAM-Spec system, with the aim of using it in-line in the future for real-time inspection of the products manufactured.



SAM-Spec®: the solution for in-line analysis of the critical parameters

Indatech offers a unique inspection solution capable of checking both the physical and chemical characteristics of solids and liquids. SAM-Spec lets you perform quick, non-destructive, multipoint analyses in-line. It is capable of:

- Measuring up to 27 channels simultaneously
- Precisely analyzing the physical and chemical composition of solids and liquids
- Performing high-speed acquisition with integration times < 1 ms

How to measure the lyophilized products

The samples were analyzed with our SAM-Spec NIR spectrometer connected to our 13-point SPC probe by optical fibers. The probe measures the samples contained in vials from below.

The spectral range is from 900 to 1650 nm. The acquisition parameters for this initial static analysis on the laboratory were defined with an 8 ms integration time and 50 scans. These 50 scans yielded 50 automatically-averaged measurements, thus improving the signal/noise ratio.

Results and discussion

All the data analyses were performed using MATLAB R2022b and PLS_Toolbox 9.1 from Eigenvector.

1-Quantification of moisture content:

A total of 60 spectra of samples at known concentrations (measured with the Karl Fischer reference method) were obtained. The different 13-point spectra measured were averaged and then preprocessed (SG1 + SNV). 49 samples were used to develop a PLS (Partial Least Squares) predictive model, and the 21 remaining samples were used to validate the model built.

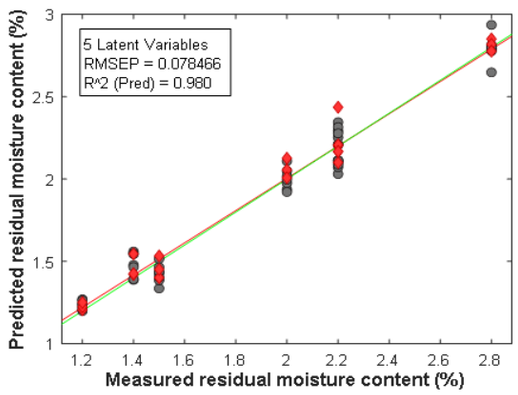


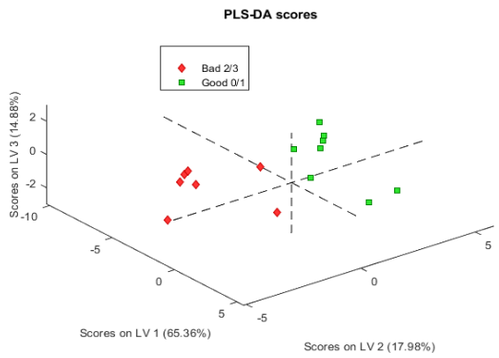
Figure 1: Results of prediction of the validation set using the PLS model

The PLS model predicted the validation set with an RMSEP (Root Mean Square Error of Prediction) of approximately 0.1%. The moisture content was quantified correctly, so high-speed inspection of this critical parameter is now possible by measuring the samples through their container.

2-Inspection of lyocake defects:

A total of 19 samples was measured with known categories (Good lyocake=9; Bad lyocake=10). Several positions of the same sample were measured and 57 spectra were obtained in this way. 41 spectra were used to develop a PLS-DA (Discriminant Analysis) classification model, and the 16 remaining spectra (Good lyocake=8; Bad lyocake=8) were used to validate the model built.

The teams at INDATECH will be delighted to answer your questions by telephone: +33 4 80 78 01 40



Confusion Table:

	Actual Class	
	Bad 2/3	Good 0/1
Predicted as Bad 2/3	8	0
Predicted as Good 0/1	0	8
Predicted as Unassigned	0	0

Figure 2: Results of classification of the validation set using the PLS-DA model

The whole validation set was classified correctly. Our SAM-Spec technology gives access to physical data by analyzing a large area of the lyocake with different measuring points. This made it possible to obtain these relevant results.

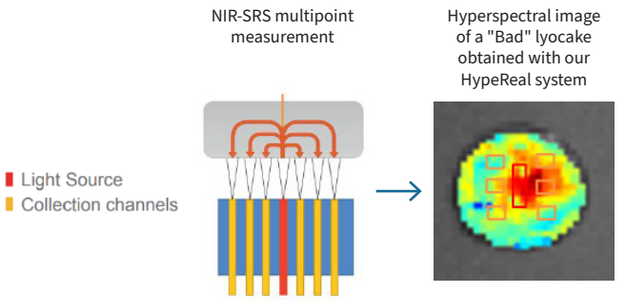


Figure 3 : Illustration of the multipoint NIR measurement of lyocake defects

Advantages of the SAM-Spec® spectrometer

- Very fast measurement making it possible to check various parameters such as moisture content, API or contaminant concentrations and the homogeneity of the powders and tablets
- A set of probes, flow-cells and cuvette-holders enabling reliable, reproducible measurement
- Physical and chemical characterization of liquid or solid products by means of multipoint SRS measurement.

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