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Application Note AN N505

Filter Dryer Endpoint Analysis using FT-NIR Spectroscopy

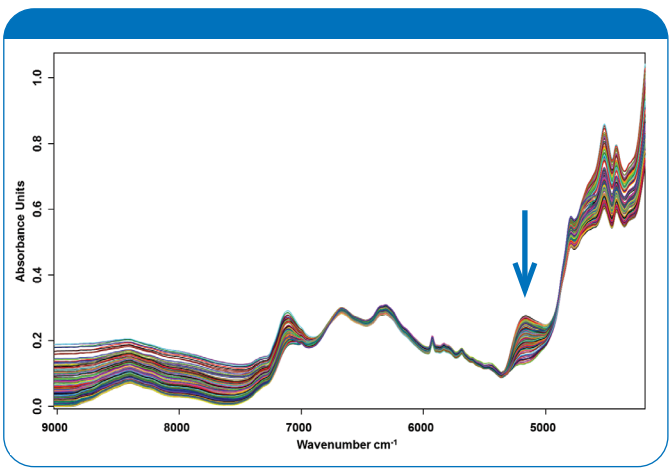
Modern process analytical technologies (PAT) in pharmaceutical production and quality control is helping to move industry to a new level of productivity. FT-NIR spectroscopy enables timely measurements of critical quality parameters and performance attributes of raw and in-process materials and processes.

The drying step is critical to ensure that the correct characteristics (moisture/solvent content) are controlled for the next step in the process. Filter/Pressure driers are commonly used in pharmaceutical manufacturing to wash and dry product prior to blending and tableting.

The standard operating method involves either stopping the process and thieving a sample for primary analysis or drying the product for a fixed period of time. Both of these do not provide a continuous monitor of the drying process and therefore do not provide the control necessary to optimize the product moisture, for example, for the next step in the process.

FT-NIR spectroscopy in combination with an in-line diffuse reflectance probe enables a continuous monitoring the drying process.

Filter driers can be modified to install measurement ports in the filter drier bowl into which the probes can be mounted. The bowls can be designed with the measurement ports located in positions optimized for the size of filter drier. For example, in R&D and pilot plant filter driers where the amounts of material may be limited, this could be close to the bottom of the bowl.



FT-NIR spectra recorded during the drying process. The decreasing OH-band at 5,200cm^{-1} allows a close monitoring of the moisture content.

Additionally larger diameter diffuse reflectance probes can be used that have the added benefit of a software controlled purging capability. This allows the probe tip to be cleaned at frequent intervals and removes the high probability of build-up on the probe tip.

Quantitative Analysis

Near-infrared spectra result from combination and overtone bands of C-H, N-H and O-H vibrations. Since most reaction mixtures and solvents contain some organic components with these bonds, they are ideal for near-infrared analysis.

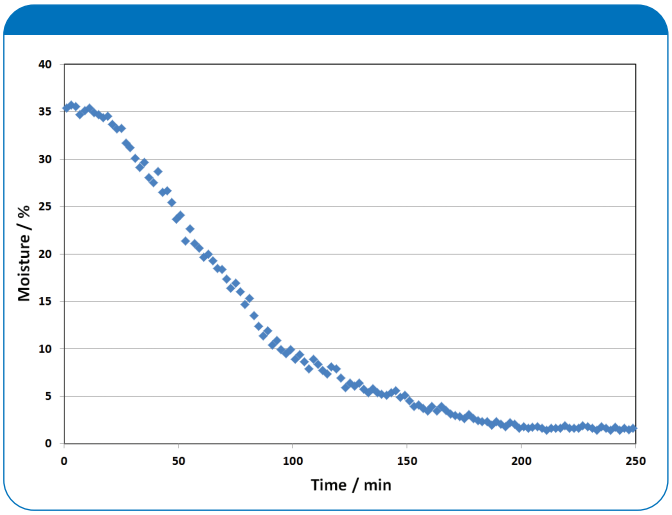
The OPUS/QUANT quantitative analysis software package uses partial lease squares (PLS algorithm) to develop quantitative models. Typically the development of a model requires measuring samples that contain a range of concentrations of the components of interest. The QUANT self-optimisation routine is then applied to develop the calibration model.

Instrumentation

Bruker Optics offers a wide variety of instrumentation to meet your specific needs. For process applications, the FT-NIR spectrometer MATRIX-F is recommended. Itss multiplexing capability, ruggedness and easy serviceability make it the perfect process system.

Various process measurement accessories are available for contact and non-contact measurements of liquids, solids and slurries. Near-infrared sample spectra can be collected from driers using diffuse reflectance probes or non-contact sensor heads.

The use of fiber optics makes it possible to locate the instrument in either an enclosure in a hazardous location close to the measurement sites or in a control room. In a process environment the MATRIX-F can be used along with our process software CMET to perform the measurement and analysis of the sample and also output the results via a variety of I/O options (4-20mA, Modbus, Profibus DP, OPC DA etc).



Moisture profile measured by FT-NIR spectroscopy

Quality Assurance in a Validated Environment

To ensure that pharma-grade products meet the strict quality parameters before their release, it is essential to carry out a completely traceable analysis according to GLP.

Bruker Optics spectrometers can be fully qualified according to US Pharmacopeia and PhEur. The operating software supports Operational Qualification (OQ) and Performance Qualification (PQ) as well as full traceability according to 21 CFR Part 11. Dedicated validation manuals containing a complete documentation of the hard- and software performance are available.

- OPUS Validation Program for OQ/PQ qualification
- Qualification according to USP<1119> and PhEur 2.2.40 with certified standards
- Internal Validation Unit for automatic PQ tests
- Validation manual with complete qualification documentation for hard- and software
- Compliance with 21 CFR Part 11
- Secure, time-stamped audit trails

Bruker Scientific LLC
Billerica, MA · USA
Phone +1 (978) 439-9899
info.bopt.us@bruker.com

Bruker Optics GmbH & Co. KG
Ettlingen · Germany
Phone +49 (7243) 504-2000
info.bopt.de@bruker.com

Bruker Shanghai Ltd.
Shanghai · China
Tel.: +86 21 51720-890
info.bopt.cn@bruker.com

www.bruker.com/optics