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Broad Spectra Optical Fiber Solutions

Raw Materials Identification Testing by NIR and Raman Spectroscopy

Raw Material Identification (RMID) is one of the most important aspects of quality control in the pharmaceutical, food and chemical industries. Ideally, the method for identifying raw materials should be fast, easy to implement and non-destructive. Such a method would allow testing and identification at several stages of the process, which is an important part of the Process Analytical Technology (PAT).
Fortunately, spectroscopic methods can satisfy these requirements. Spectroscopy can provide correct identification in seconds.
Recently, more and more attention is given to the study of RMID using the combined method based on Near-infrared (NIR) and Raman spectroscopy [1, 2].
Near-infrared (NIR) and Raman spectroscopy are ideally suited for even the most challenging RMID applications. These techniques are complementary to each other which increase their value and empower their more widespread use within a facility. These techniques can be used to identify a wide variety of raw materials and can even distinguish between closely related ones that have similar chemical structures.
Near-infrared spectroscopy is a technique that uses light between 800nm and 2500nm (12500 to $4000cm^{-1}$) to excite certain molecular vibrations. Also, absorption intensity is lower than in the mid-infrared region, which allows for deeper penetration of the source light into the material.
Raman spectroscopy is also a vibrational technique but differs substantially from other spectroscopic techniques. Raman spectroscopy relies on the non-elastic scattering of photons from a laser source. As the photons from the laser interact with the material, a few are absorbed. The energy from these photons is partially absorbed and the remaining energy is re-emitted as scattered light at a different frequency. The scattered light is shifted from the original laser frequency by an amount that depends on the energy absorbed by the molecular bonds.
The spectrum generated by a Raman instrument generally has sharper and better resolved peaks than NIR which can provide more chemical information of unknown samples.
The two methods are technically different in that they excite different vibrational states. Therefore, molecular bonds that are not active with one method are often active with the other.

Here, we announce the development of a new method for RMID using a combined fiber optic probe (NIRaman), which allows provide simultaneous measurements for both methods at the same point, to measure process and product characteristics in real-time, as this method allow fast and non-destructive measurements without sample preparation.
The first commercial version of the multichannel fiber optic probe (patent pending) was designed for in situ NIR diffuse reflectance and Raman measurements of solids, powders, or liquids. The unique feature of the probe is that NIR and Raman channels can work simultaneously providing hybrid modelling opportunities that were impossible in the past.

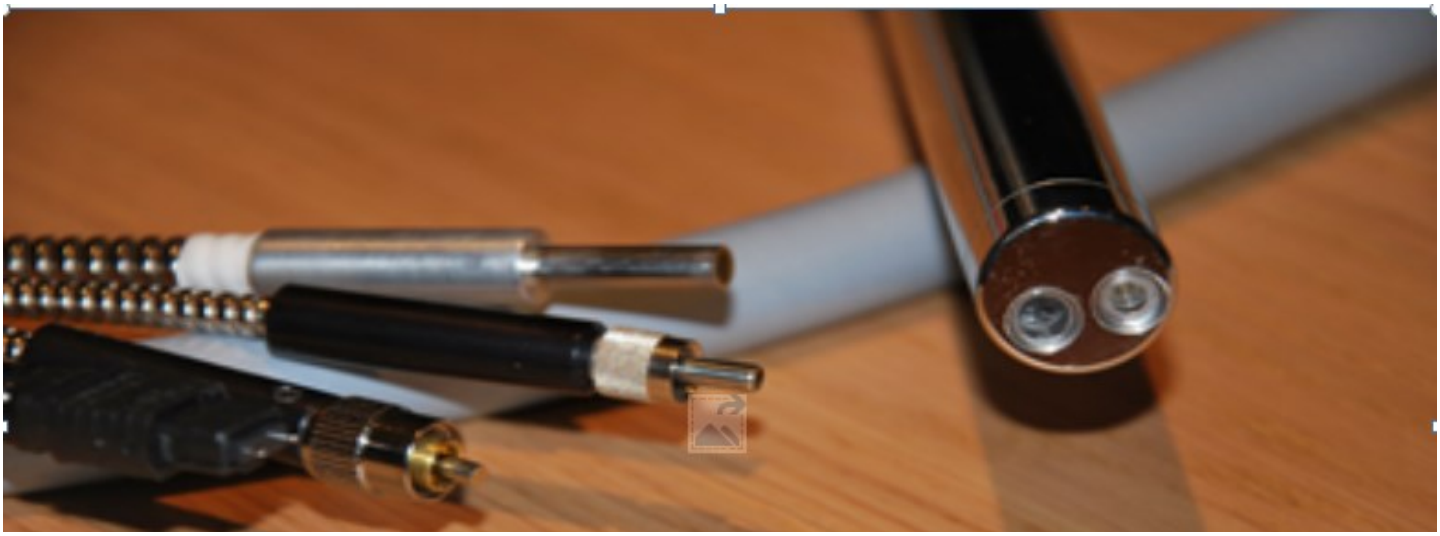




Fig.1 NIR-DR + Raman Combi Probe is a multichannel fiber optic probe designed for in situ NIR diffuse reflectance (NIR-DR) and Raman measurements of powders or liquids. NIR-DR and Raman channels of the probe can work simultaneously – the wavelength ranges of both channels are separated: Raman 785 – 1000nm; NIR-DR 1000-2500nm. The probe shaft is electrical heated to prevent moisture conden-

Specification of NIRaman Probe

Probe Shaft

Material: Stainless Steel 1.4435 (316L)

Outer Diameter: 19,0+0/-0,1mm

Length: 170mm

Window: Sapphire or Cubic Zirconia, heated

Glue: Epotek 353ND or similar

Separate NIR and Raman channels

Raman – Channel

Fibers: low OH silica fibers with Al-coating to suppress laser induced fluorescence
Laser Wavelength: 785nm
Connector 1: FC (launch fiber - 105µm core)
Connector 2: MTP-Male with 48 x 105µm fibers
Other connectors on request.
Capable to pass USP requirements (tested on Kaiser RXAndor DVA420A-OE and RXn4 Raman-Spectrometer).
Fiber Length: 3+/-0,1m

NIR – Channel

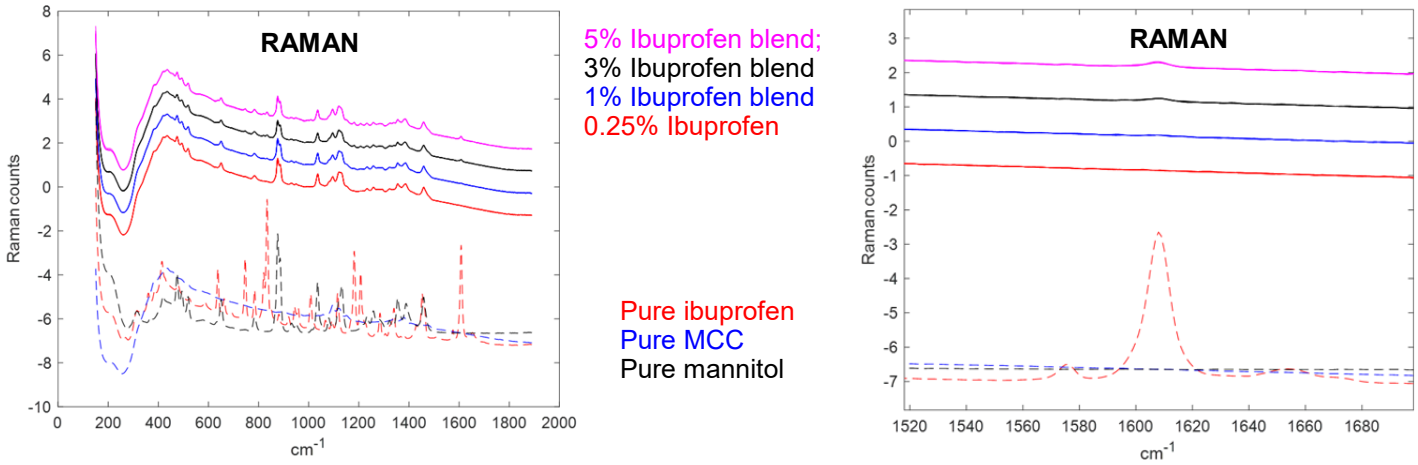
Fibers: low OH silica fibers
Stray Light: less than 1%
Illumination bundle: 32x NIR400/440 NA=0.22
Detection fiber: 1x NIR400/440 NA=0.22
Connector 1: SMA905
Other connectors on request
Connector 2: 5mm ferrule
Capable to pass USP requirements (tested on Sentronic Sentropat system)

Sample identification by NIR and Raman Spectroscopy

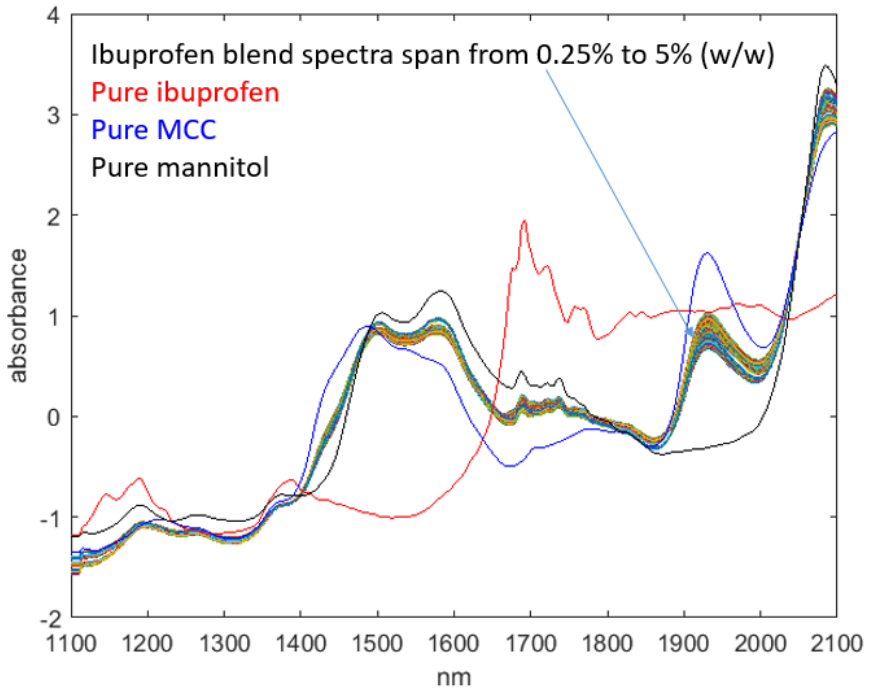
NIRAMAN system was used to produce spectra from the test raw materials. Samples of each material were scanned by placing the fiber probe in the reactor.

Apart from raw material identification, NIRaman probes are also very efficient in the analysis of blend potency. This can be used for in process blend verification as the probe can be easily mounted onto production machinery.

The addition of the probe to a production process can avoid the interruption of the process for sampling and thus considerably increase production efficiency and also the safety for the operators as they are no longer exposed to the product while taking samples.



NIR



Conclusions

By using a new method with combined fiber optic probe (NIRaman System), which allows provide simultaneous non-destructive measurements for both methods: NIR & Raman at the same point, without sample preparation, even the most challenging raw materials can be identified.

The two spectroscopic techniques are complementary with some materials being better suited for NIR, while others are better suited for Raman. NIR has better sample penetration and samples a larger area, whereas Raman is generally limited to the width of the laser. Therefore, large grained heterogeneous samples are more conducive to analysis with NIR. In aqueous samples where the water bands might be interfering, Raman is possibly a better choice than NIR.

[1]. NIR and Raman: Complementary Techniques for Raw Material Identification, *Thermo Fisher Scientific, Technical Note: 51768*

[2]. Raw Materials Identification Testing by NIR Spectroscopy and Raman Spectroscopy, Shimadzu, FTIR TALK LETTER