

An alternative to handheld fibre optic probes

Christine Simard and Henry Buijs

Bomem/Hartmann & Braun, Québec, Québec, Canada G2E 5S5.

Introduction

Quantitative near infrared (NIR) spectroscopy is known in the chemical industry as a fast and convenient way to measure several parameters of importance with great precision and accuracy. These qualities often depend on the sampling technique chosen to do the analysis: it must be fast and simple and must provide precise, accurate and reliable results. Fixed-pathlength cells fulfil these conditions, since they are relatively easy to use and their controlled pathlength ensures analytical precision and accuracy. However, cleaning these cells is time-consuming and often requires hazardous chemicals. In addition to that, viscous samples cannot be analysed using this sampling technique, since they are impossible to inject into the cell.

Handheld fibre optics probes are very easy and convenient to use and their efficiency for quantitative analysis has been demonstrated in many successful applications. However, they have to be used with some care since the sample must be in close contact with the probe to get good results. This leads to the requirement to clean the probe tip, to avoid contamination of the next sample.

Disposable glass vials are a very interesting alternative for near infrared applications. This sampling technique is more flexible and allows faster analysis, without compromising analytical precision and accuracy.

Use of glass vials for NIR sampling

The glass vials that are used for NIR sampling are made of borosilicate glass, a material which is virtually transparent to NIR radiation. These vials can contain a wide range of samples, from solids to liquids, and are particularly useful to analyse viscous materials. Due to their very low cost (0.25\$ each) they can be disposed of after use, eliminating the need for potentially hazardous chemicals and the possibility of sample contamination. Samples can also be stored in vials for long periods of time.

A heated vial holder was designed to record spectra from glass vials. The holder is simply inserted in the optical path of a Bomem MB series FT-NIR spectrometer and the vial containing the sample is placed in the central hole of the holder, where a slit allows the beam to pass through the sample. The six other holes can be used to pre-heat other samples. The temperature of this holder is controlled for better spectral reproducibility. It can be heated up to 130°C, allowing the melting of solid materials.

The analysis of a sample contained in a glass vial does not require any particular attention. A vial is simply filled with the sample to analyse, put to heat in the vial holder, and its NIR spectrum is recorded. The use of glass vials reduces the sample turnaround time to 2 minutes. (Note that when a Fourier transform spectrometer is used, a reference has first to be acquired using an empty vial.)

Quantitative analysis using glass vials

Glass vials have demonstrated their suitability for quantitative analysis. However, spectra have to be normalised to compensate for differences in pathlength between vials. This is usually done by integrating a region of the spectrum, and by dividing the whole spectrum by this area. NIR bands are more intense when the vials are used, since their pathlength is usually of 3–5 mm. The effect of this longer pathlength is to make some bands intense enough to be used for calibration (for example, the OH 1st overtone and the CH 2nd overtone regions) while some others become too intense. It has to be mentioned that the use of the vials maintains features of a FT-NIR-based analyser including highly precise and accurate results and stability of calibrations.

Applications where glass vials can be used are numerous. This sampling technique has been successfully used for determination of hydroxyl value, saponification value, acid value, iodine value, humidity content, and others, in polyols, surfactants, resins etc. We have also developed calibrations for iodine value in hydrogenated vegetable oils, for alkyl sulfonate content in detergents, for humidity content of glue etc. The vials increase significantly the number of materials that can be analysed by our FT-NIR analyser and provide very precise and accurate results. It has been proven that calibrations developed using spectra acquired from vials have the same quality as those developed using a more classical sampling technique.

Conclusion

In conclusion, glass vials make NIR sampling easier, faster and safer for operators in QA/QC labs. As they are disposable, there are no chemicals to use for cleaning and to dispose of. These vials allow the analysis of thick and viscous samples and can be used to melt solid samples. They provide precise, accurate and reliable results suitable for quantitative analysis and this in a very short period of time and at low-cost.