Rapid identification of o-rings, seals and gaskets using the handheld Agilent 4100 ExoScan FTIR

Application note

Materials testing

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Introduction

In virtually every industrial and chemical plant worldwide, using the correct materials to seal valves, pipes and vessels is critical from both a productivity and safety perspective. Incorrect sealing materials can lead to leaks, loss of product, and equipment downtime, as well as affect worker health or potentially lead to catastrophic failures.

Chemical equipment manufacturers will specify the optimum sealing material for their equipment, but each type of material may have a multitude of different compositions, properties and usages. The material chosen depends on the type of equipment, the specific applications or chemicals that are being processed and the conditions to which the chemicals are being subjected. The result is that there are literally thousands of different o-ring, seals and gaskets available for the chemical, petrochemical and petroleum industries.

In manufacturing plants, handheld XRF is being used to ensure that the metals in the chemical equipment have the proper elemental composition for the manufacturing process. Similarly, there is a clear need for a handheld analyzer to ensure that the correct organic based sealing material is positively identified.

Though Near-IR and Raman have some use in this application, many of the o-rings, gaskets and seals are carbon black filled and present a special problem. The Agilent 4100 ExoScan system (Figure 1) equipped with a germanium ATR sample interface provides an excellent solution for identifying these materials, including the difficult-to-analyze carbon black filled examples.

Figure 1. Agilent 4100 ExoScan FTIR system

Experimental

A series of carbon black filled seals and o-rings were sourced and then analyzed by the 4100 ExoScan FTIR equipped with its novel spherical germanium ATR. This sample interface was chosen for two major reasons:

- The spherical shape of the ATR element results in high quality, reproducible spectral results while at the same time minimizing the pressure exerted on the surface of the sample. This is a non-destructive test (NDT) for measuring a material, that is, the gasket or o-ring is measured without cutting a sample or deforming the surface.
- Germanium is chosen as the ATR material since it has a shorter overall pathlength through the sample due to its higher refractive index and shorter depth of penetration. Carbon is a nonspecific absorber of IR radiation and if the depth of penetration of IR radiation is too great, the carbon particles will scatter the IR light. This results in a baseline shift in the resultant spectrum, obscuring the characteristic infrared absorbance bands (the IR 'fingerprint') of the sample, making identification of the material less reliable (Figure 2).

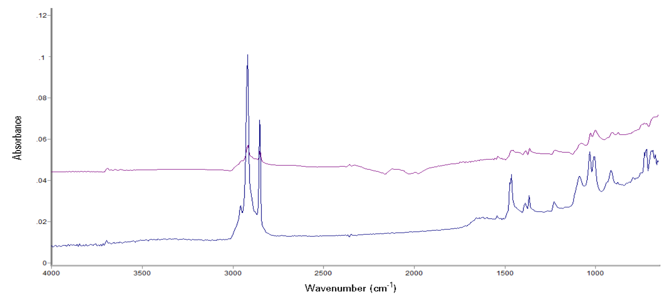

Figure 2. Comparison of carbon black filled elastomeric material recorded using diamond ATR (purple) and germanium ATR (blue). The Ge ATR has a shorter depth of penetration resulting in less scattering from the carbon black particles and less baseline shifting. The fundamental vibrational absorbances are readily visible, when compared to the spectra recorded using the diamond ATR.

Specifically, 14 samples were measured of 9 different seal materials. Samples from different manufactures were measured for 5 of the 9 materials. The following carbon filled sealing materials were measured: fluorosilicone; silicone, viton, EPR/EPDM, neoprene, butyl, kalrez, NBR, polyurethane, and natural rubber. The samples were recorded by lightly pressing against the ATR crystal to ensure contact. The Agilent MicroLab FTIR software has a real-time analysis mode that enables the user to ensure that adequate contact is made between the sample and the ATR element as evidenced by the instantaneous appearance of the desired spectra
(Figure 3).

Spectra resulted from a 20 second interferogram co-addition recorded at 8 cm^{-1} resolution. The spectra from this group of samples were used to form a reference library in the ExoScan's MicroLab software. Days later, a second set of spectra were recorded from other samples of these materials and searched against the onboard library. The top two hits were reported by the software as well as the degree of similarity (the correlation function).

Figure 3. Sample is lightly pressed against spherical Ge ATR element (left) and real time display instantly shows that adequate contact is made. Co-addition is initiated and resultant high quality spectrum is searched against onboard spectral library (right).

Results

High quality spectra (Figures 4 and 5) of the sealing materials were obtained using the previously described procedure. The spectra show clear spectral differentiation based on the class of material and the carbon particles had minimal affect on spectral quality or baseline position. These spectra formed the basis of the onboard library and when the evaluation samples were measured days later, all samples produced the correct first match, and fibe of the samples produced a close second match from within the same type of material (Table 1).

The spectrum measured from an individual o-ring contains chemical information from both the elastomer material and any fillers used. For this reason, the first match always returns the same brand o-ring. Other o-rings made of the same elastomer may come up as a close second match if the fillers used are similar. Two examples of this in the tested sample set are the silicone and fluorosilicone o-rings. These samples contained very little filler. Their infrared spectra contained information primarily from the elastomer material, and hence both o-rings in these two categories had similar match qualities. By comparison, the NBR sample was highly filled. The two samples of NBR were not close second matches because the amount of filler differed between the two samples.

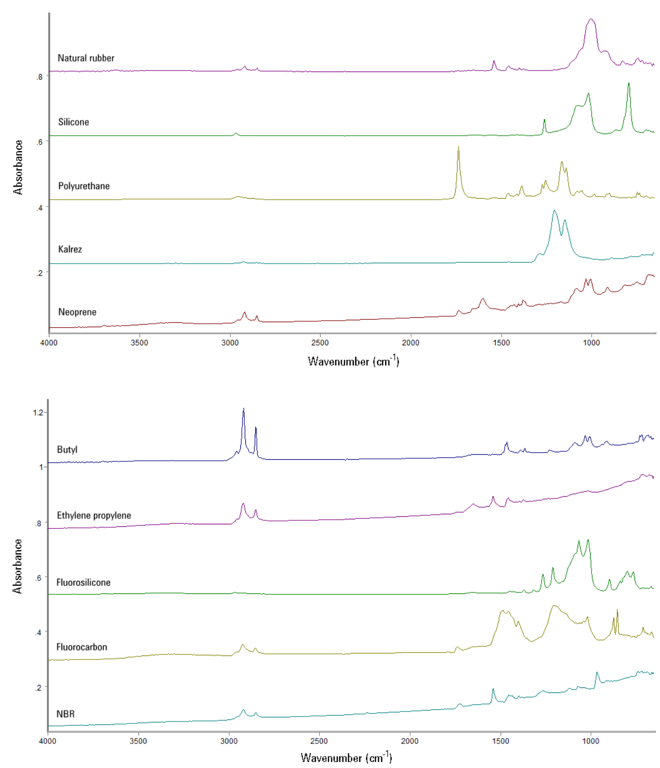

Figure 4 and 5. Spectra of seals were recorded using a 20 second co-addition of interferograms at 8 cm^{-1} resolution. The spectra are offset for clarity.

Table 1. Library search results for a test set of 14 o-rings matched against an o-ring spectral library. Samples and matches are listed by material (item number). The similarity values are automatically calculated in the MicroLab software and are based on a first derivative normalized dot product correlation.

Sample	1st match	Similarity	2nd match	Similarity
NBR (618316)	NBR (618316)	0.84	Ethylene propylene (618337)	0.21
NBR (618333)	NBR (618333)	0.819	Natural rubber (618332)	0.71
Silicone (618341)	Silicone (618341)	0.977	Silicone (618318)	0.963
Kalrez (618327)	Kalrez (618327)	0.953	Kalrez (618328)	0.951
Viton (618313)	Viton (618313)	0.604	Kalrez (618328)	0.15
Viton (618344)	Viton (618344)	0.71	Viton (618313)	0.1
Neoprene (618325)	Neoprene (618325)	0.55	EPR (618337)	0.14
Fluorosilicone (618311)	Fluorosilicone (618331)	0.95	Fluorosilicone (618319)	0.89
Fluorosilicone (618319)	Fluorosilicone (618319)	0.97	Fluorosilicone (618331)	0.963
Polyurethane (618340)	Polyurethane (618340)	0.955	Kalrez (618328)	0.017
Silicone (618318)	Silicone (618318)	0.976	Silicone (618341)	0.975
EPR (618337)	EPR (618337)	0.5	EPR (618314)	0.37
EPR (618314)	EPR (618314)	0.46	EPR (618337)	0.288
Butyl (618339)	Butyl (618339)	0.47	Neoprene (618315)	0.33

Conclusion

The Agilent 4100 ExoScan FTIR equipped with spherical Ge ATR is proven to be an effective analyzer for determining and verifying the identify of carbon black filled polymer and elastomeric based seals, gaskets and o-rings used in the chemical industry. These types of materials have traditionally been difficult to analyze by spectroscopic methods such as near-IR and Raman and thus the 4100 ExoScan FTIR system offers a more universal solution for determining the identify of both carbon filled and non carbon filled materials. Other manufacturing industries such as aerospace, food and pharmaceuticals face similar issues.

Because of its performance, size and mobility , the 4100 ExoScan FTIR can be used in both the lab and/or where the sample is located. This means that sealing materials can be examined at the site of use, without having to send them to a lab for measurement and waiting for results. The dedicated MicroLab FTIR software makes the system easy to use, so factory personnel can accurately identify materials. Moreover, this is truly a non-destructive methodology so samples do not need to be cut or otherwise modified for measurement thereby maintaining the integrity of the seal.

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