Application Note AN-NIR-084

Quality Control of Silicone rubber

Fast determination of vinyl content without chemicals

Determination of the vinyl content of silicone rubber is a lengthy and challenging process. First, the vinyl groups must be converted to ethylene by reacting with an acid, followed by the determination of the produced ethylene with gas chromatography (GC).

This application note demonstrates that Vis-NIR (visible near-infrared) spectroscopy provides a **cost-efficient and fast** solution for the determination of **vinyl content in silicone rubber**. With the DS2500 Solid Analyzer it is possible to obtain results in **less than a minute without sample preparation or any chemical reagents**. The standard GC method requires one hour to perform, along with highly trained analysts. In contrast to the primary method, Vis-NIR spectroscopy, is a cost-efficient and fast analytical solution for the determination of vinyl content in silicone rubber.

EXPERIMENTAL EQUIPMENT

Silicone samples were measured with a DS2500 Solid Analyzer in transflection mode over the full wavelength range (400–2500 nm). A DS2500 Slurry Cup was employed, which simplifies the positioning of the sample and cleaning of the sample vessel. The 1 mm gold diffuse reflector defines the same path length for all measurements to guarantee reproducible results. As displayed in Figure 1, samples were measured without any preparation step. The Metrohm software package Vision Air Complete was used for all data acquisition and prediction model development.

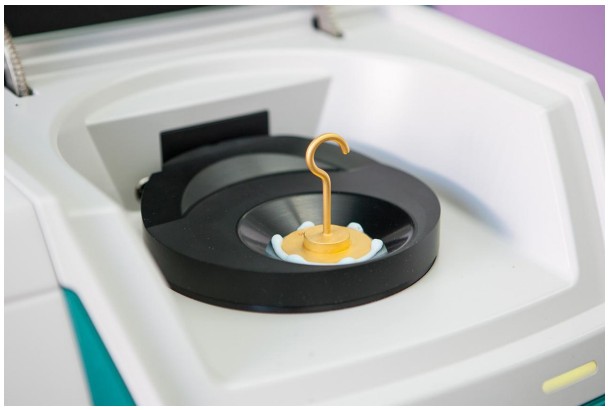

Figure 1. DS2500 Solid Analyzer with silicone rubber sample present in DS2500 Slurry Cup.

Table 1. Hardware and software equipment overview

Equipment	Metrohm number
DS2500 Analyzer	2.922.0010
DS2500 Slurry Cup	6.7490.430
Gold Diffuse Reflector 1 mm	6.7420.000
Vision Air 2.0 Complete	6.6072.208

RESULT

The obtained Vis-NIR spectra (Figure 2) were used to create prediction models for quantification of vinyl content in silicone rubber. The quality of the prediction models was evaluated using correlation diagrams, which display the correlation between Vis-NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.

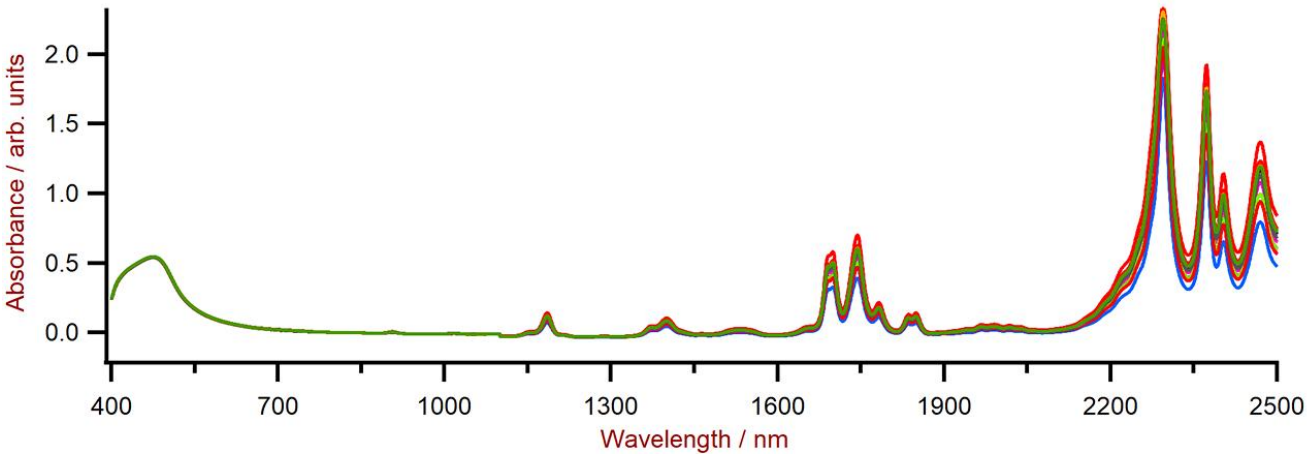

Figure 2. Selection of silicone rubber Vis-NIR spectra obtained using a DS2500 Analyzer and a rotating DS2500 Slurry Cup.

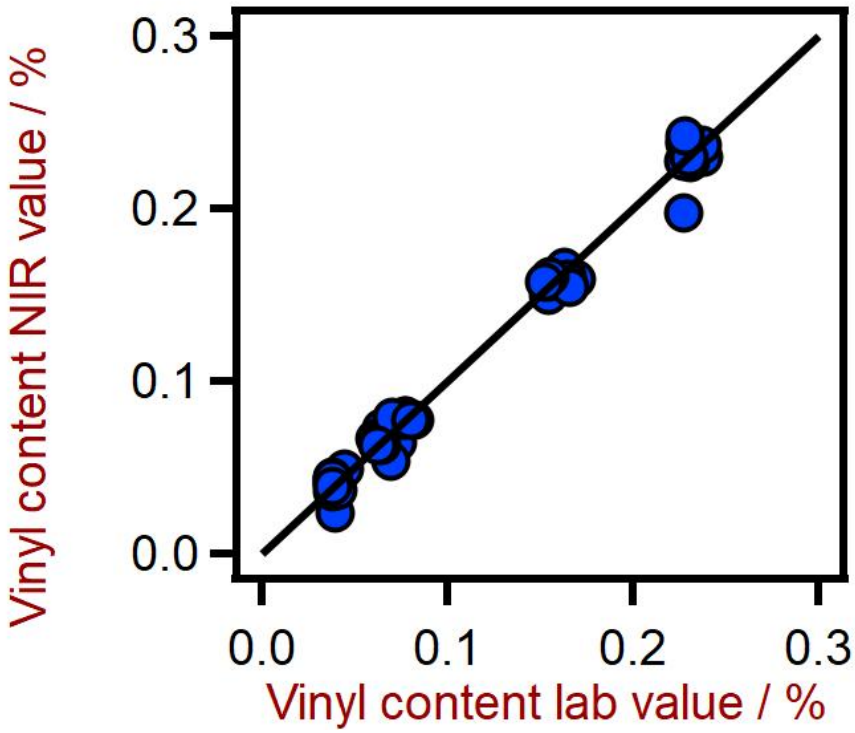

Figure 3. Correlation diagram and the respective figures of merit for the prediction of the vinyl content in silicone rubber using a DS2500 Solid Analyzer. The vinyl content lab value was evaluated using gas chromatography.

Table 2. Figures of merit for the prediction of the vinyl content in silicone rubber using a DS2500 Solid Analyzer.

Figures of merit	Value
R^2	0.989
Standard error of calibration	0.0076%
Standard error of cross-validation	0.0089%

CONCLUSION

This application note demonstrates the feasibility of NIR spectroscopy for the analysis of vinyl content in silicone rubber. In comparison to gas chromatography methods (Table 3), the time to result is a major advantage of NIR spectroscopy, since a single measurement is performed in less than a minute.

Table 3. Time to result overview for the parameter vinyl content.

Parameter	Method	Time to result
Vinyl content	Gas chromatography	10 min (preparation) + 50 min (GC)

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