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.

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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.
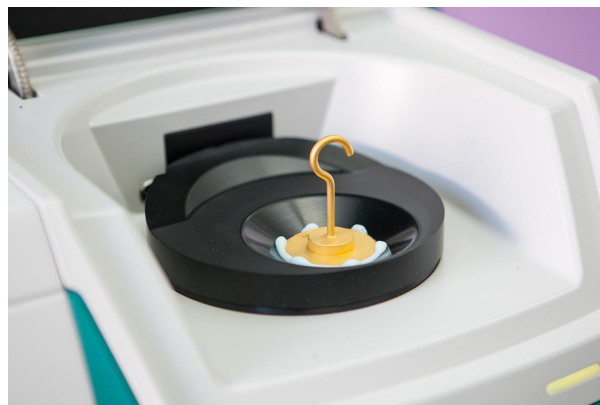

Figure 1. DS2500 Solid Analyzer with silicone rubber sample present in DS2500 Slurry Cup.

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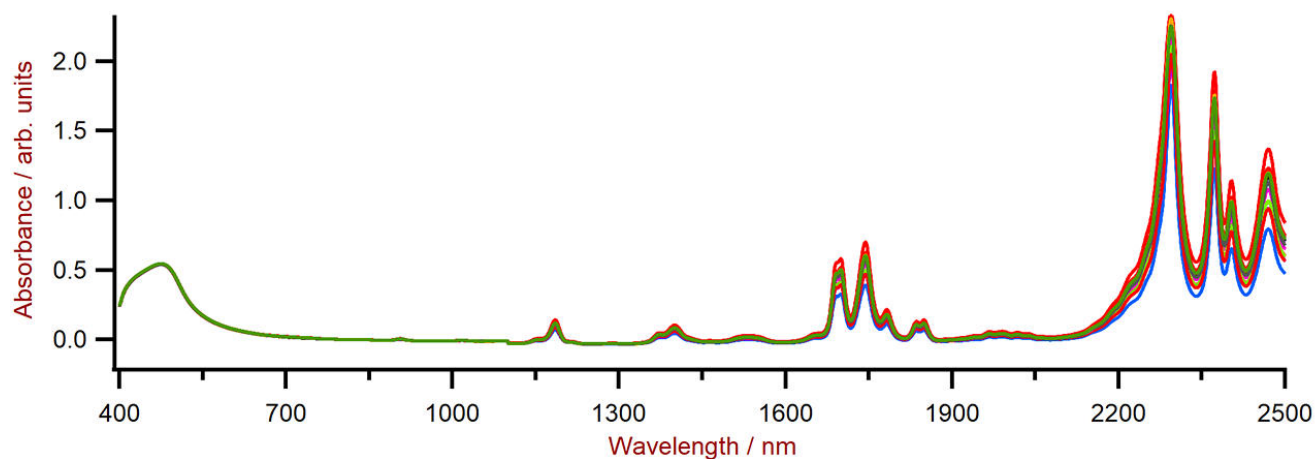
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Figure 2. Selection of silicone rubber Vis-NIR spectra obtained using a DS2500 Analyzer and a rotating DS2500 Slurry Cup.
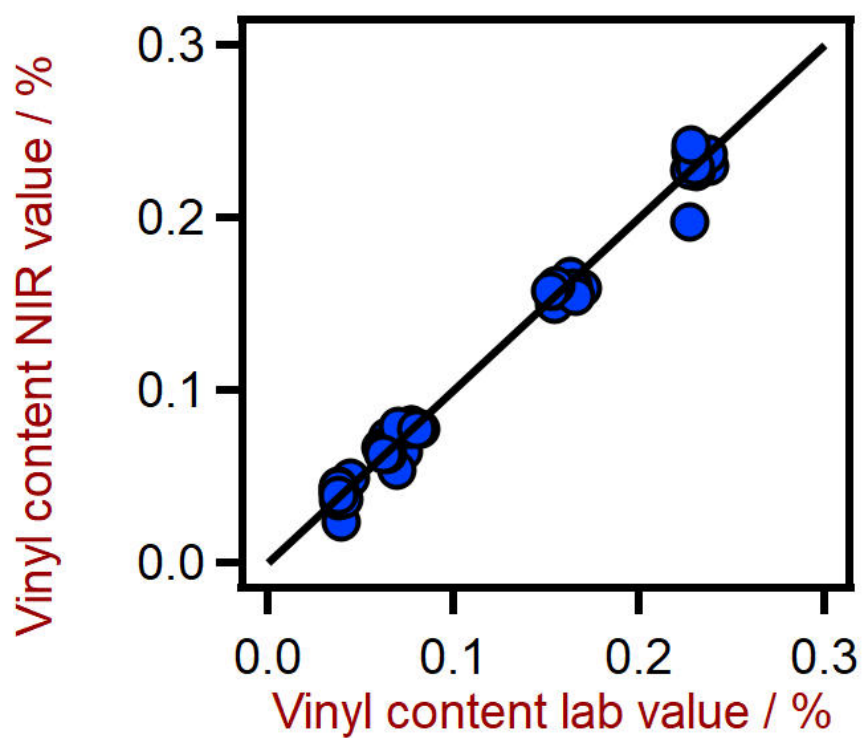

Figure 3. Correlation diagram and the respective figures of merit for the prediction of the vinyl content in silicone rubber using a DS2500 Solid Analyzer. The vinyl content lab value was evaluated using gas chromatography.

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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)

Internal reference: AW NIR CN-0016-082019

CONTACT

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DS2500 Solid Analyzer

Robust near-infrared spectroscopy for quality control, not only in laboratories but also in production environments.

The DS2500 Analyzer is the tried and tested, flexible solution for routine analysis of solids, creams, and optionally also liquids along the entire production chain. Its robust design makes the DS2500 Analyzer resistant to dust, moisture, vibrations, and temperature fluctuations, which means that it is eminently suited for use in harsh production environments.

The DS2500 covers the full spectral range from 400 to 2500 nm and delivers accurate, reproducible results in less than one minute. The DS2500 Analyzer meets the demands of the pharmaceutical industry and supports users in their day-to-day routine tasks thanks to its simple operation.

Thanks to accessories tailored perfectly to the instrument, optimum results are achieved with every sample type, no matter how challenging it is, e.g. coarse-grained solids such as granulates or semi-solid samples such as creams. The MultiSample Cup can help improve productivity when measuring solids, as it enables automated measurements of series containing up to 9 samples.
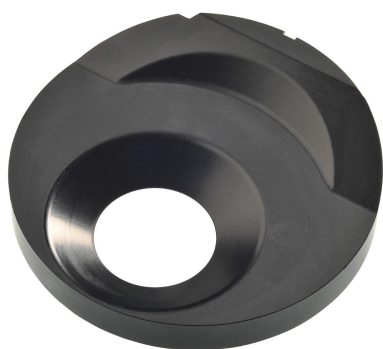
DS2500 Slurry Cup

The Slurry Cup is the ideal sample cup for analyzing high-viscosity substances with the DS2500. The positioning of pastes and creams in the Slurry Cup is made simple by its open design, which also permits rapid and effective cleaning.

In combination with the Liquid Kit **(6.7400.010)**, clear viscous samples can also be investigated.

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NIRS gold diffuse reflector, 1 mm total pathlength

Gold diffuse reflector for the transflection measurement of liquids. Can be used in combination with the following instruments:
- NIRS DS2500 Analyzer (**order number: 2.922.0010**)
- NIRS XDS MasterLab Analyzer (**order number: 2.921.1310**)
- NIRS XDS MultiVial Analyzer (**order number: 2.921.1120**)
- NIRS XDS RapidContent Analyzer (**order number: 2.921.1110**)
- NIRS XDS RapidContent Analyzer - Solids (**order number: 2.921.1210**)

Vision Air 2.0 Complete
Vision Air - Universal spectroscopy software.

Vision Air Complete is a modern and simple-to-operate software solution for use in a regulated environment.
Overview of the advantages of Vision Air:
- Individual software applications with adapted user interfaces ensure intuitive and simple operation
- Simple creation and maintenance of operating procedures
- SQL database for secure and simple data management

The Vision Air Complete version (66072208) includes all applications for quality assurance using Vis-NIR spectroscopy:
- Application for instrument and data management
- Application for method development
- Application for routine analysis

Additional Vision Air Complete solutions:
- 66072207 (Vision Air Network Complete)
- 66072209 (Vision Air Pharma Complete)
- 66072210 (Vision Air Pharma Network Complete)

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