Application Note AN-NIR-084

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.

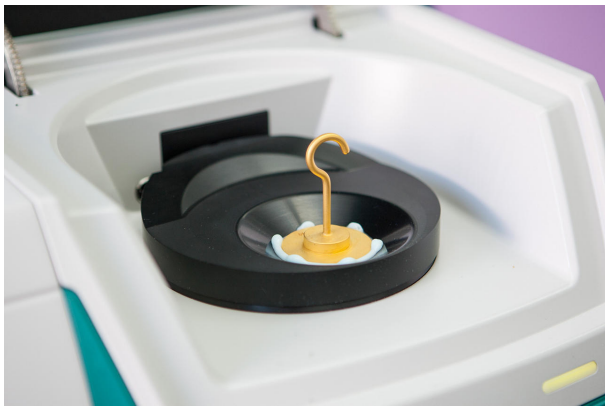

Figure 1. DS2500 Solid Analyzer with silicone rubber sample present in DS2500 Slurry Cup.

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.

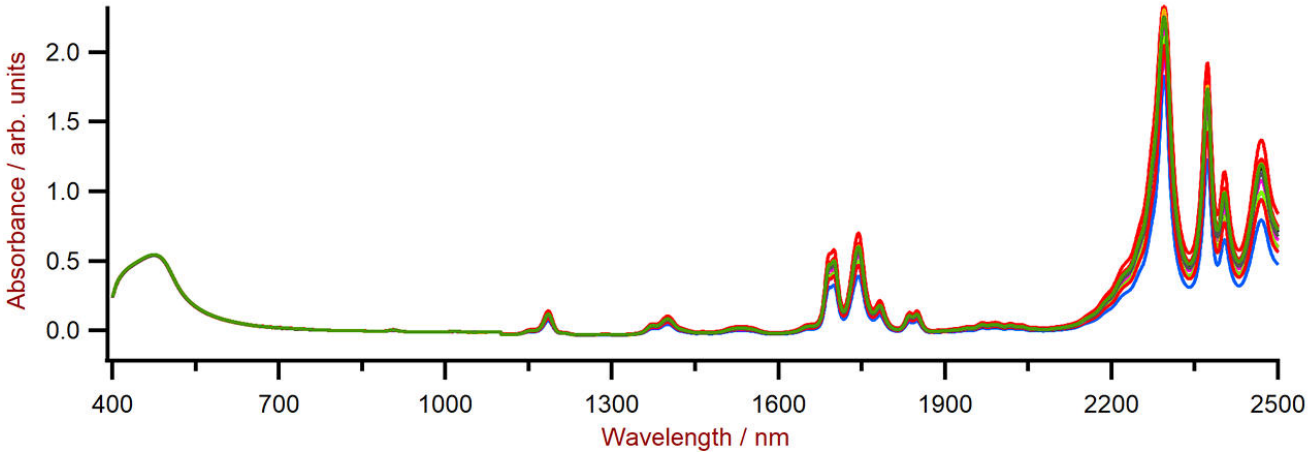

Figure 2. Selection of silicone rubber Vis-NIR spectra obtained using a DS2500 Analyzer and a rotating DS2500 Slurry Cup.

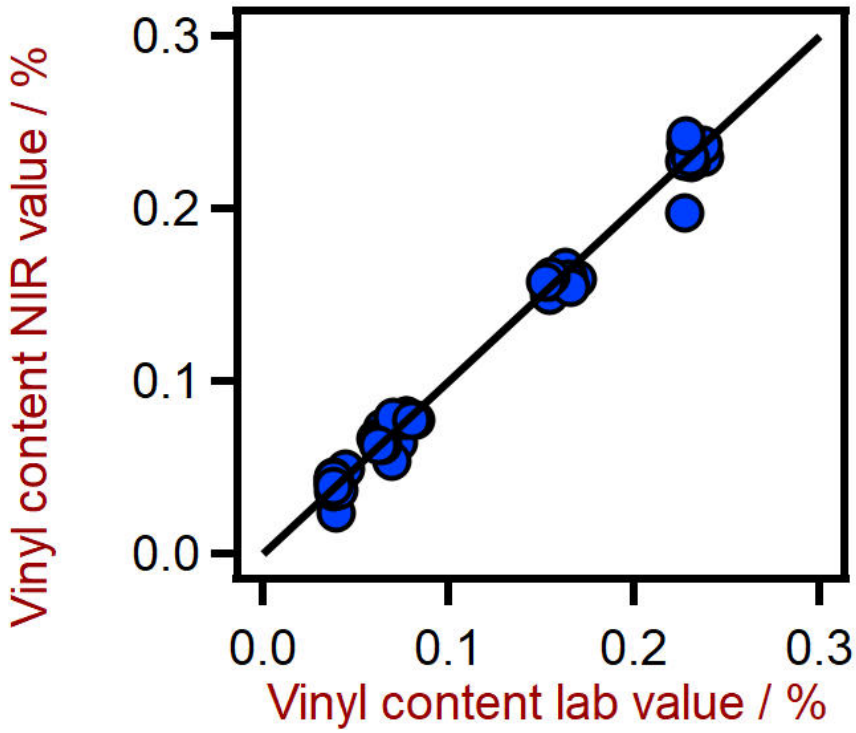

Figure 3. Correlation diagram and the respective figures of merit for the prediction of the vinyl content in silicone rubber using a DS2500 Solid Analyzer. The vinyl content lab value was evaluated using gas chromatography.

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%

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

固的近外光,用于生境和室中的量。

DS2500 分析是的活解决方案,用于整个生程中的固体、乳膏和液体行常分析。其固的使 DS2500 Analyzer 分析不受灰、湿度、振和温度波的影,因此非常用于在劣的生境中使用。

DS2500 涵盖了从 400 到 2500 nm 的整个光范,并能在不到一分内提供准和可再的果。DS2500 Analyzer 足制行的要求,并由于操作便而能助用完成其日常工作任。

由于与匹配,附件可以承受任何具有挑性的品型,例如:粒料之的粗粒固体或乳膏之的半固体品,可得果。量固体的候,使用 MultiSample Cup 可以提高生率,可以自批量量多 9 个品。

DS2500 Slurry Cup

Slurry Cup 是使用 DS2500 分析高粘度物的理想品容器。由于采用放式,在 Slurry Cup 中定位膏和油十分便,同可以行快速有效的清。

与 Liquid Kit (6.7400.010) 相合,也可以透明的粘性品。

NIRS 1 mm

液体行透射反射量的金反射器。可与下列合使用:

- NIRS DS2500 Analyzer(**号:2.922.0010**)
- NIRS XDS MasterLab Analyzer(**号:2.921.1310**)
- NIRS XDS MultiVial Analyzer(**号:2.921.1120**)
- NIRS XDS RapidContent Analyzer(**号:2.921.1110**)
- NIRS XDS RapidContent Analyzer - Solids(**号:2.921.1210**)

Vision Air 2.0 Complete

Vision Air – 通用的光分析件。

Vision Air Complete 是用于管范境的先易用的件解决方案。

Vision Air 点一:

- 便捷的件用和配的用界面保了直的操作方式
- 操作程的建与方式
- SQL 数据,可安全且地管理数据

Vision Air Complete (66072208) 版本包含所有用于可近外光分析量保程的用:

- 器和数据管理用
- 方法用
- 常分析用

其它 Vision Air Complete 解决方案:

- 66072207 (Vision Air Network Complete)
- 66072209 (Vision Air Pharma Complete)
- 66072210 (Vision Air Pharma Network Complete)