Application Note AN-NIR-084

硅橡胶质量控制

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.

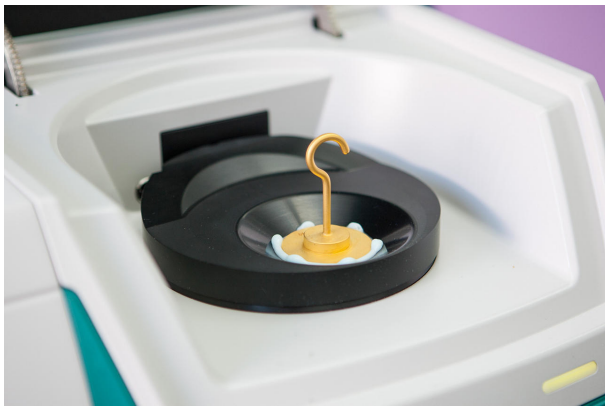

Figure 1. DS2500 Solid Analyzer with silicone rubber sample present in DS2500 Slurry Cup.

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.

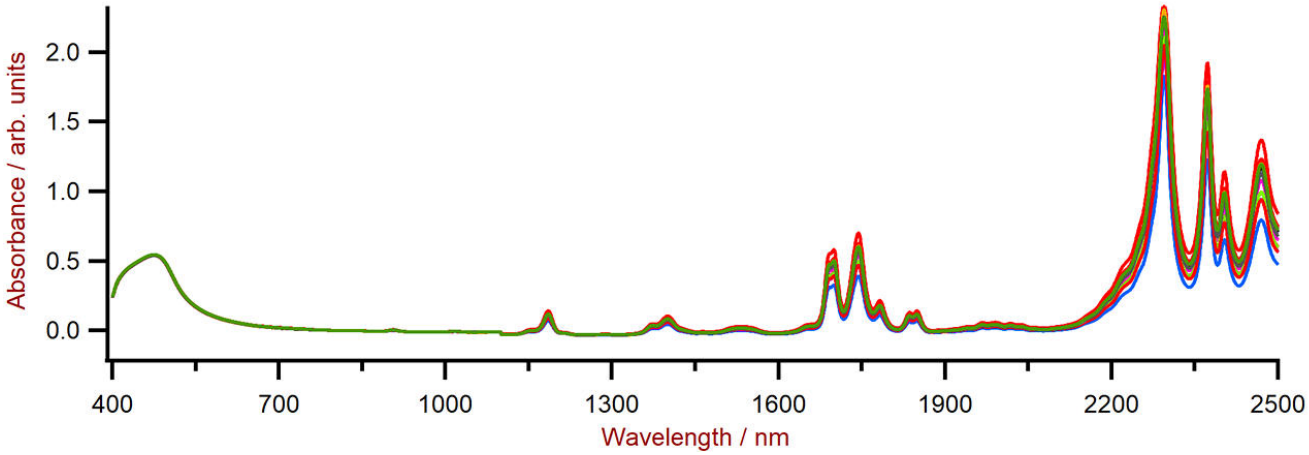

Figure 2. Selection of silicone rubber Vis-NIR spectra obtained using a DS2500 Analyzer and a rotating DS2500 Slurry Cup.

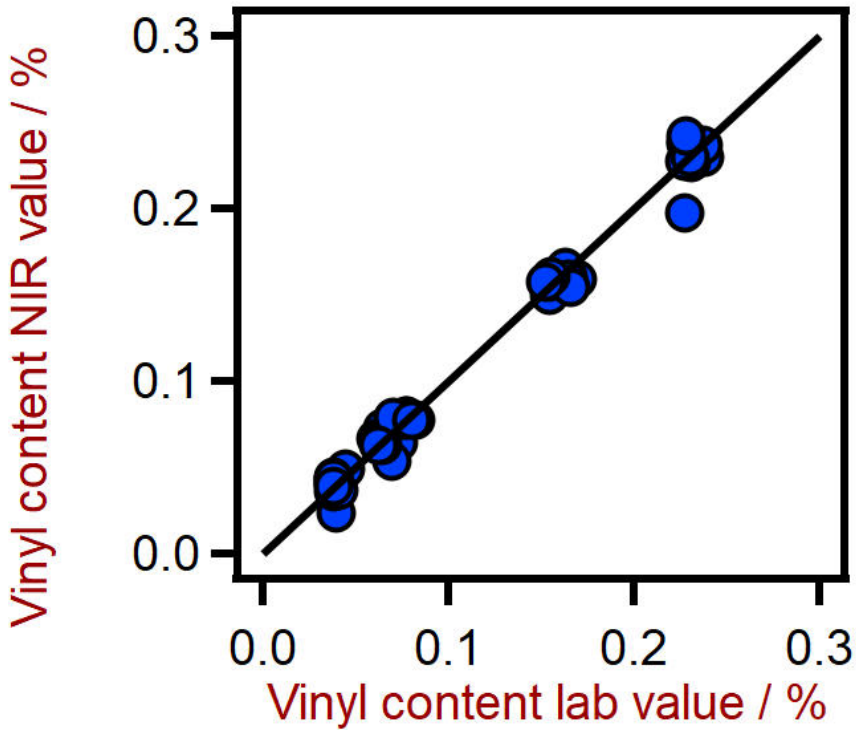

Figure 3. Correlation diagram and the respective figures of merit for the prediction of the vinyl content in silicone rubber using a DS2500 Solid Analyzer. The vinyl content lab value was evaluated using gas chromatography.

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)

Internal reference: AW NIR CN-0016-082019

CONTACT

Metrohm Taiwan Ltd.
46 5
108
info@metrohm.com.tw

DS2500 Solid Analyzer

坚固结实的近红外光谱仪,用于生产环境和试验室中的质量检查。

DS2500 分析仪是经过验证的灵活解决方案,用于对整个生产链过程中的固体、乳膏和液体进行常规分析。其坚固结实的设计使 DS2500 Analyzer 分析仪不受灰尘、湿度、振动和温度波动的影响,因此非常适用于在恶劣的生产环境中使用。

DS2500 涵盖了从 400 到 2500 nm 的整个光谱范围,并能在不到一分钟时间内提供准确和可再现的结果。DS2500 Analyzer 满足制药行业的要求,并由于操作简便而能帮助用户完成其日常工作任务。

由于与设备匹配,附件可以承受任何具有挑战性的样品类型,例如:粒料之类的粗颗粒固体或乳膏之类的半固体样品,可获得结果。测量固体的时候,使用 MultiSample Cup 可以提高生产率,可以自动批量测量多 9 个样品。

DS2500 Slurry Cup

Slurry Cup DS2500 , Slurry Cup ,

Liquid Kit (6.7400.010) ,

NIRS 1 mm

:

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

Vision Air 2.0 Complete

Vision Air –

Vision Air Complete

Vision Air :

-
-
- SQL ,

Vision Air Complete (66072208) :

-
-
-

Vision Air Complete :

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