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.

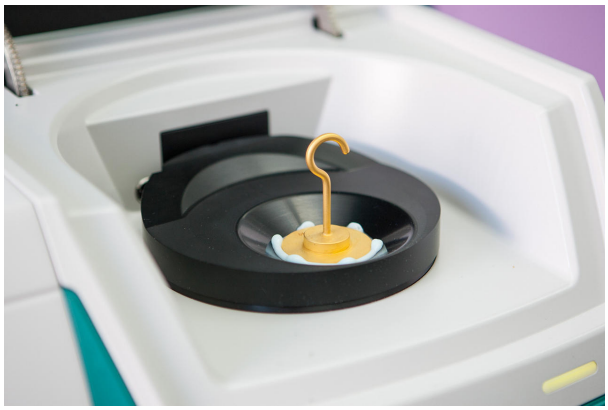

Figure 1. DS2500 Solid Analyzer with silicone rubber sample present in DS2500 Slurry Cup.

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

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.

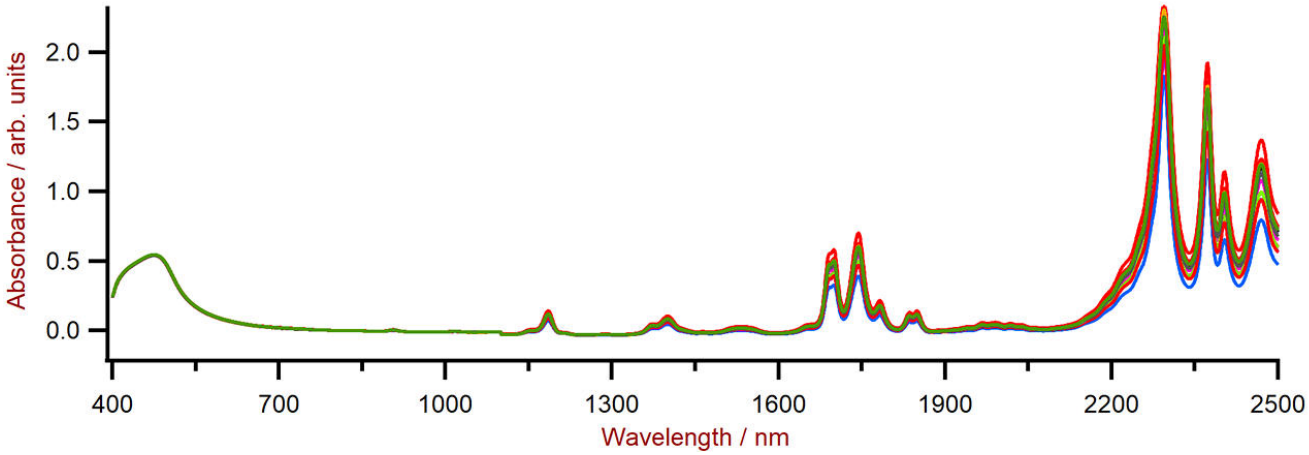

Figure 2. Selection of silicone rubber Vis-NIR spectra obtained using a DS2500 Analyzer and a rotating DS2500 Slurry Cup.

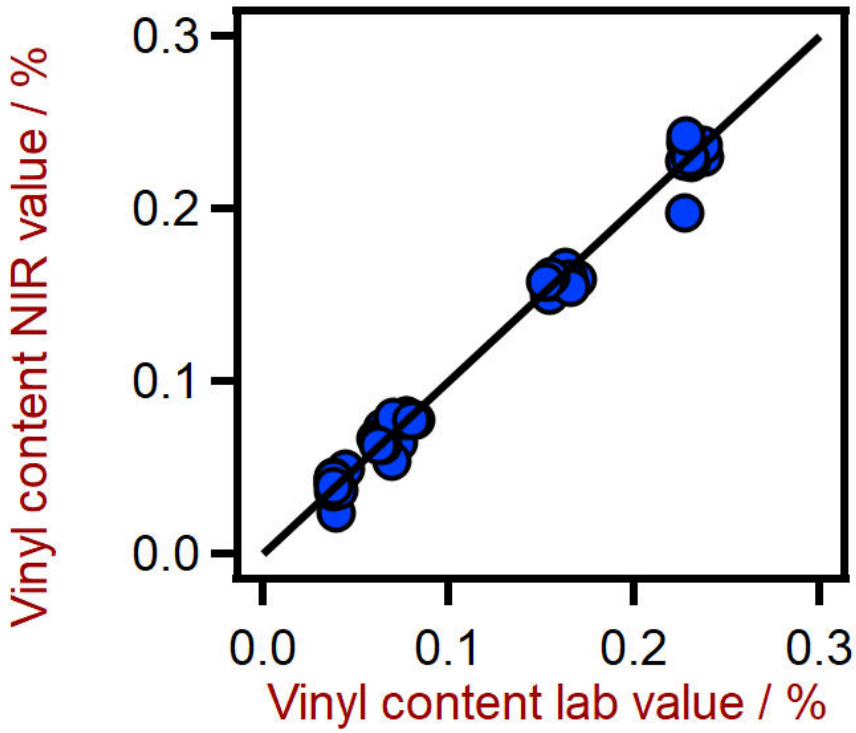

Figure 3. Correlation diagram and the respective figures of merit for the prediction of the vinyl content in silicone rubber using a DS2500 Solid Analyzer. The vinyl content lab value was evaluated using gas chromatography.

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

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DS2500 Solid Analyzer

ラボおよび生産環境における品質管理用の堅牢な近赤外分光法。

DS2500 Analyzerは、生産チェーン全体に沿った固形物、クリーム、およびオプションとしての液体のルーチン分析に実績のあるフレキシブルなソリューションです。頑丈な仕様により、DS2500 Analyzerは粉塵、湿気、振動や温度変動に強い為、過酷な生産環境での使用に理想的です。

DS2500は400 ~ 2500 nmのスペクトル範囲全体をカバーし、1分以内に正確で再現性の高い結果を提供します。DS2500 Analyzerは製薬業界の要件を満たしており、簡単な操作により日常的な作業においてユーザーをサポートします。

装置に完全に適応した付属品により、 顆粒のような粒の荒い固形物、またはクリームのような半固形液体サンプルなどのあらゆる困難なタイプのサンプルにおいても、最良の結果を得ることができます。固形物の測定においては、9つまでのサンプルのシリースの自動測定を可能にするMultiSample Cupを使用することで、生産性を高めることができます。

DS2500 Slurry Cup

Slurry Cupは、DS2500を用いた高粘度の物質の分析に最適なサンプル容器です。開放型のデザインにより、Slurry Cupにペーストおよびクリームを簡単に配置することができ、洗浄も迅速かつ効率的に行うことができます。

Liquid Kit (6.7400.010) と組み合わせることで、透明で粘性のあるサンプルも検査することができます。

NIRS 1 mm

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- 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 :

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Vision Air Complete (66072208) :

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Vision Air Complete :

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