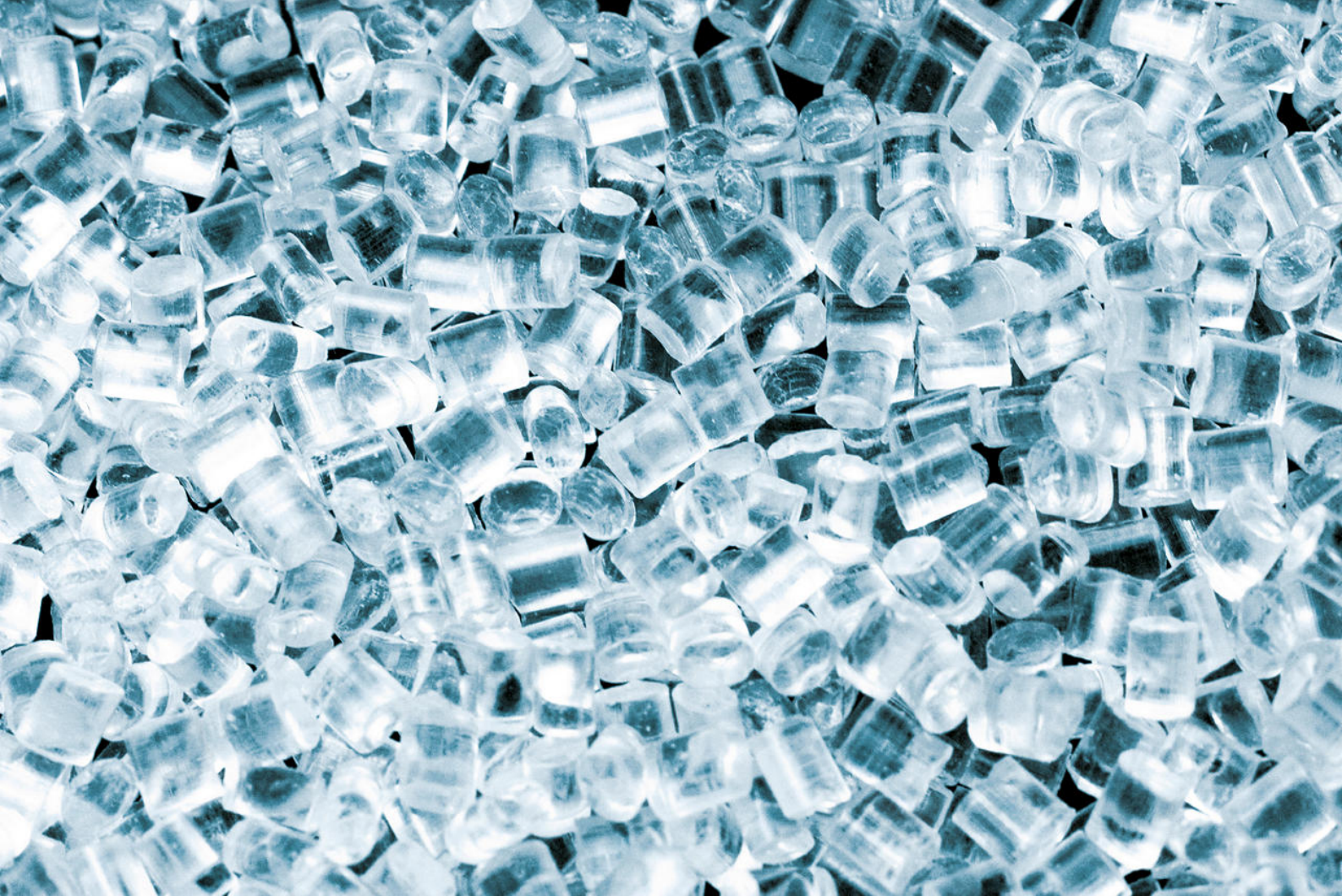




Application Note AN-NIR-100

Ash determination in polyethylene

Routine analysis of ash content in PE granulates made simple

In order to improve certain characteristics in polymers, various fillers are added to resins. One important filler in this industry is ash, which mainly consists of silicon-, magnesium-, and iron oxide. The ash content can vary in the product (depending on the particle size and the desired properties) from 0.01 wt% to up to 10 wt%. The standard test method for ash content

analysis is thermogravimetric analysis (TGA). Although TGA is easy to perform, it is time-intensive and requires the use of nitrogen gas. In contrast to the primary method, near-infrared spectroscopy (NIRS) is a fast analytical technique which can measure multiple parameters including ash content in polymers within one minute.

EXPERIMENTAL EQUIPMENT

154 polyethylene samples with varying ash content from 0.07% to 0.12% were analyzed by NIR spectroscopy on a Metrohm DS2500 Solid Analyzer (**Figure 1**) equipped with a DS2500 large sample cup. All measurements were performed in rotation to average the subsample

spectra. This setup reduces the influence of the particle size distribution of the polymer pellets. Data acquisition and prediction model development were performed with the software package Vision Air Complete.

Table 1. Hardware and software equipment overview.

Equipment	Metrohm number
DS2500 Solid Analyzer	2.922.0010
DS2500 large sample cup	6.7402.050
Vision Air 2.0 Complete	6.6072.208



Figure 1. DS2500 Solid Analyzer.

RESULTS

The obtained Vis-NIR spectra (Figure 2) were used to create a prediction model for ash content in PE samples. To verify the quality of the prediction model, correlation diagrams were created which display a correlation value (R^2) of

0.77 between the Vis-NIR prediction and primary method (TGA) values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis (Figure 3).

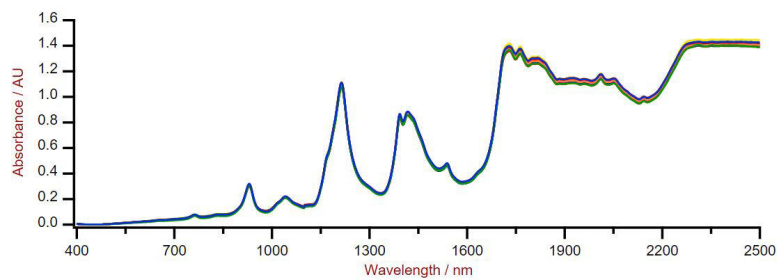


Figure 2. Selection of Vis-NIR spectra of PE samples obtained using a Metrohm DS2500 Solid Analyzer equipped with the DS2500 large sample cup.

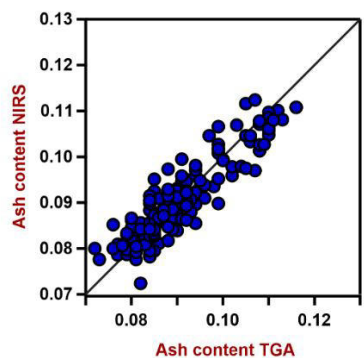


Figure 3. Correlation diagram and the respective figures of merit for the prediction of ash content in PE using a DS2500 Solid Analyzer. The lab values were determined using TGA.

Table 2. Figures of merit for the prediction of ash content in PE using a DS2500 Solid Analyzer.

Figures of Merit	Value
R^2	0.77
Standard Error of Calibration	0.0055%
Standard Error of Cross-Validation	0.066%

CONCLUSION

This Application Note shows the feasibility of NIR spectroscopy for the analysis of low levels of ash content in polyethylene granulates within one

minute. In comparison to the standard method (Table 3), the time savings for ash content analysis in PE by using a NIRS analyzer is clear.

Table 3. Time to result with conventional TGA method.

Parameter	Method	Time to result
Ash content	TGA	~2 hours

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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ズ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

NIRS DS2500 Analyzerを用いた、様々なサンプル位置における反射中の粉末および顆粒のスペクトル記録のための、大きなサンプル容器です。