

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.

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.

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**).

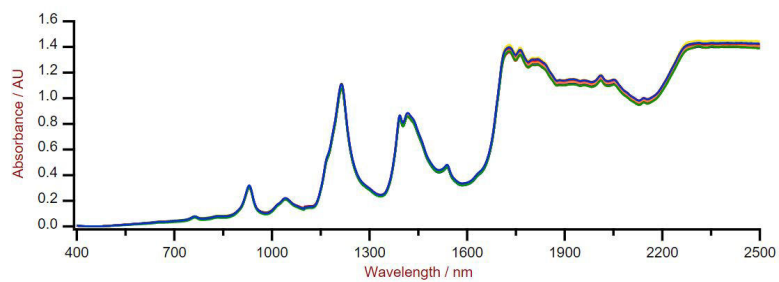


Figure 2. Selection of Vis-NIR spectra of PE samples obtained using a Metrohm DS2500 Solid Analyzer equipped with the DS2500 large sample cup.

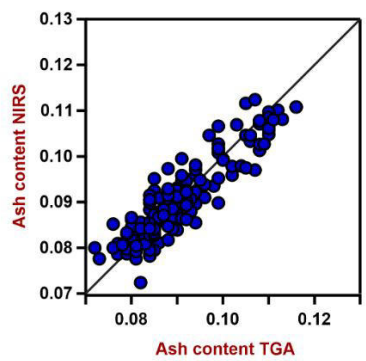


Figure 3. Correlation diagram and the respective figures of merit for the prediction of ash content in PE using a DS2500 Solid Analyzer. The lab values were determined using TGA.

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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EXPERIMENTAL EQUIPMENT



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,可以自批批量多 9 个品。



DS2500

用于在不同品位置使用 NIRS DS2500 Analyzer 采集粉末和粒反射光的大号品容器。