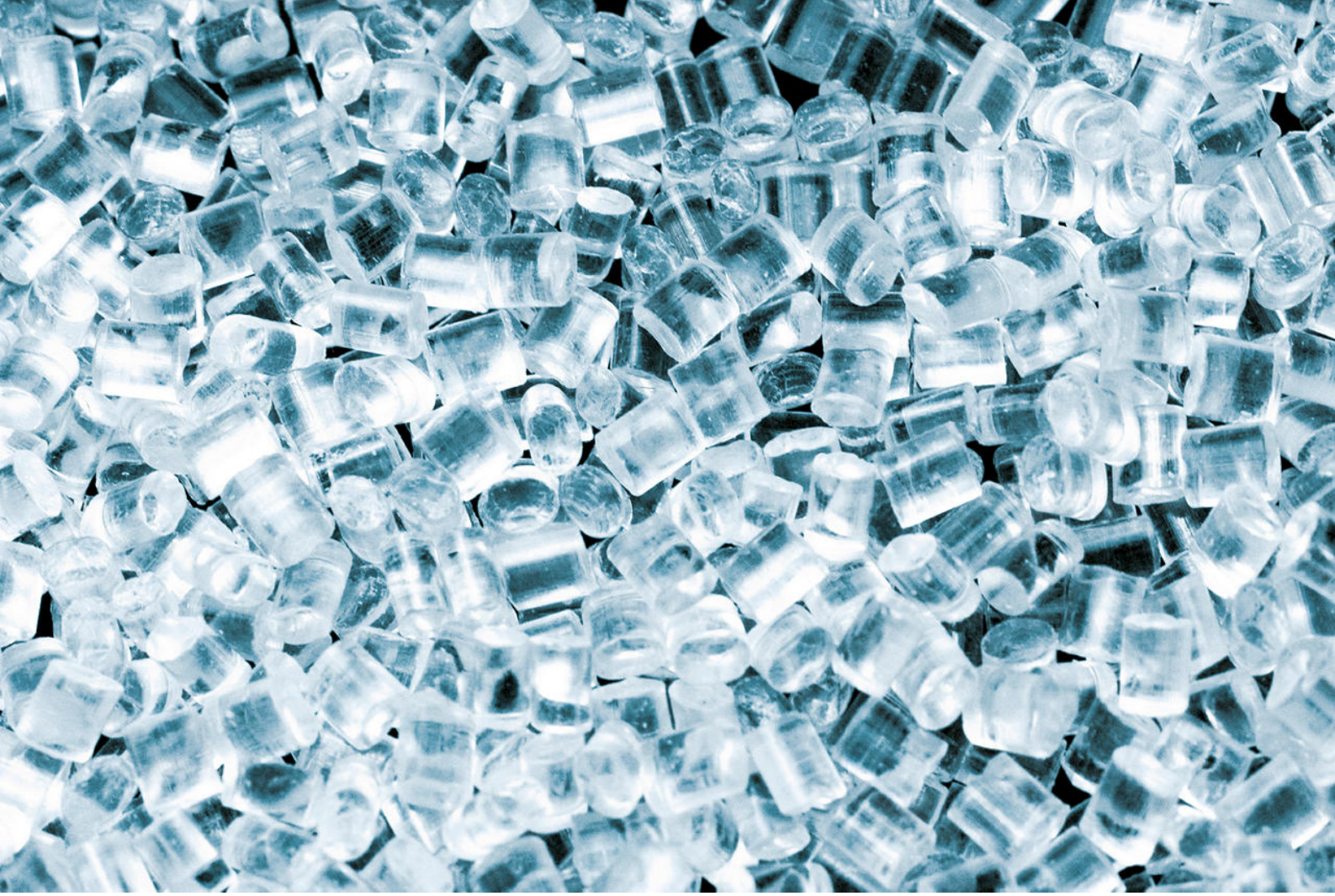

Application Note AN-NIR-100

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).

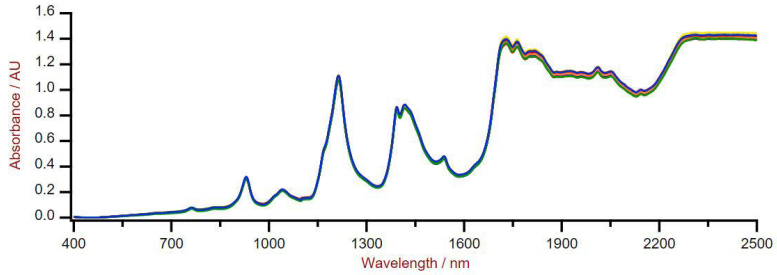

Figure 2. Selection of Vis-NIR spectra of PE samples obtained using a Metrohm DS2500 Solid Analyzer equipped with the DS2500 large sample cup.

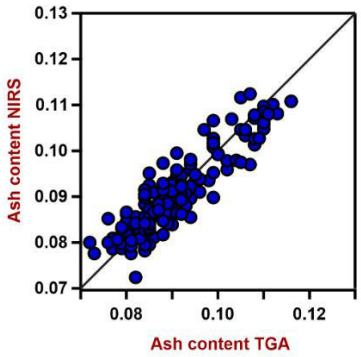

Figure 3. Correlation diagram and the respective figures of merit for the prediction of ash content in PE using a DS2500 Solid Analyzer. The lab values were determined using TGA.

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

CONTACT

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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

用于在不同样品位置处使用 NIRS DS2500 Analyzer 采集粉末和颗粒反射光谱的大号样品容器。