



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).

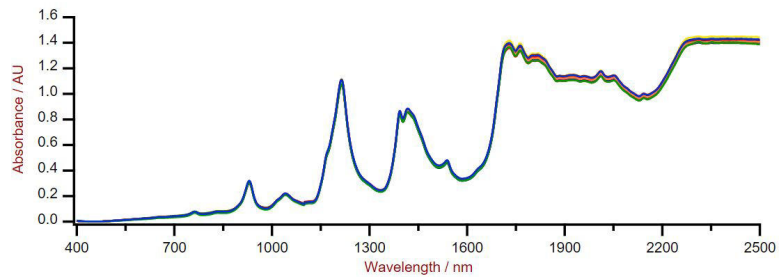


Figure 2. Selection of Vis-NIR spectra of PE samples obtained using a Metrohm DS2500 Solid Analyzer equipped with the DS2500 large sample cup.

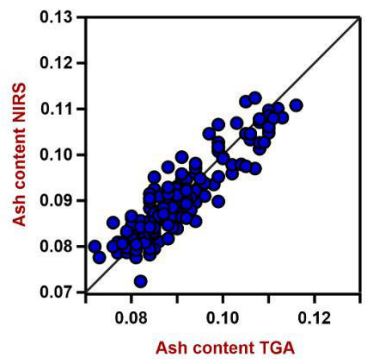


Figure 3. Correlation diagram and the respective figures of merit for the prediction of ash content in PE using a DS2500 Solid Analyzer. The lab values were determined using TGA.

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

Metrohm France
13, avenue du Québec - CS
90038
91978 VILLEBON
COURTABOEUF CEDEX

info@metrohm.fr



DS2500 Solid Analyzer

Spectroscopie proche infrarouge robuste pour le contrôle de la qualité en laboratoire et en environnement de production.

L'analyseur DS2500 Analyzer est la solution éprouvée et souple destinée aux analyses de routine de matières solides, de crèmes et, en option, de liquides, tout au long de la chaîne de fabrication. Sa conception robuste fait du DS2500 Analyzer un appareil insensible à la poussière, à l'humidité, aux vibrations ainsi qu'aux variations de température, et donc particulièrement adapté aux rudes conditions d'un environnement de production.

Le DS2500 couvre l'ensemble de la gamme spectrale de 400 à 2 500 nm et fournit des résultats exacts et reproductibles en moins d'une minute. Le DS2500 Analyzer répond aux exigences de l'industrie pharmaceutique et représente une aide précieuse pour les opérations de routine quotidiennes grâce à sa simplicité d'utilisation.

Grâce à des accessoires parfaitement adaptés à l'appareil, il atteint des performances sans précédent avec tous les types d'échantillons, quel que soit le défi qu'ils opposent (matières solides à gros grains comme les granulats ou échantillons semi-solides ou liquides telles les crèmes). La productivité lors de mesures de matières solides peut encore être augmentée par l'utilisation du MultiSample Cup, lequel permet des mesures automatisées en série jusqu'à un maximum de 9 échantillons.



DS2500 Récipient d'échantillon, grand

Grand récipient d'échantillon pour l'enregistrement de spectre par réflexion de poudres et de granulés en différents points de l'échantillon avec le NIRS DS2500 Analyzer.