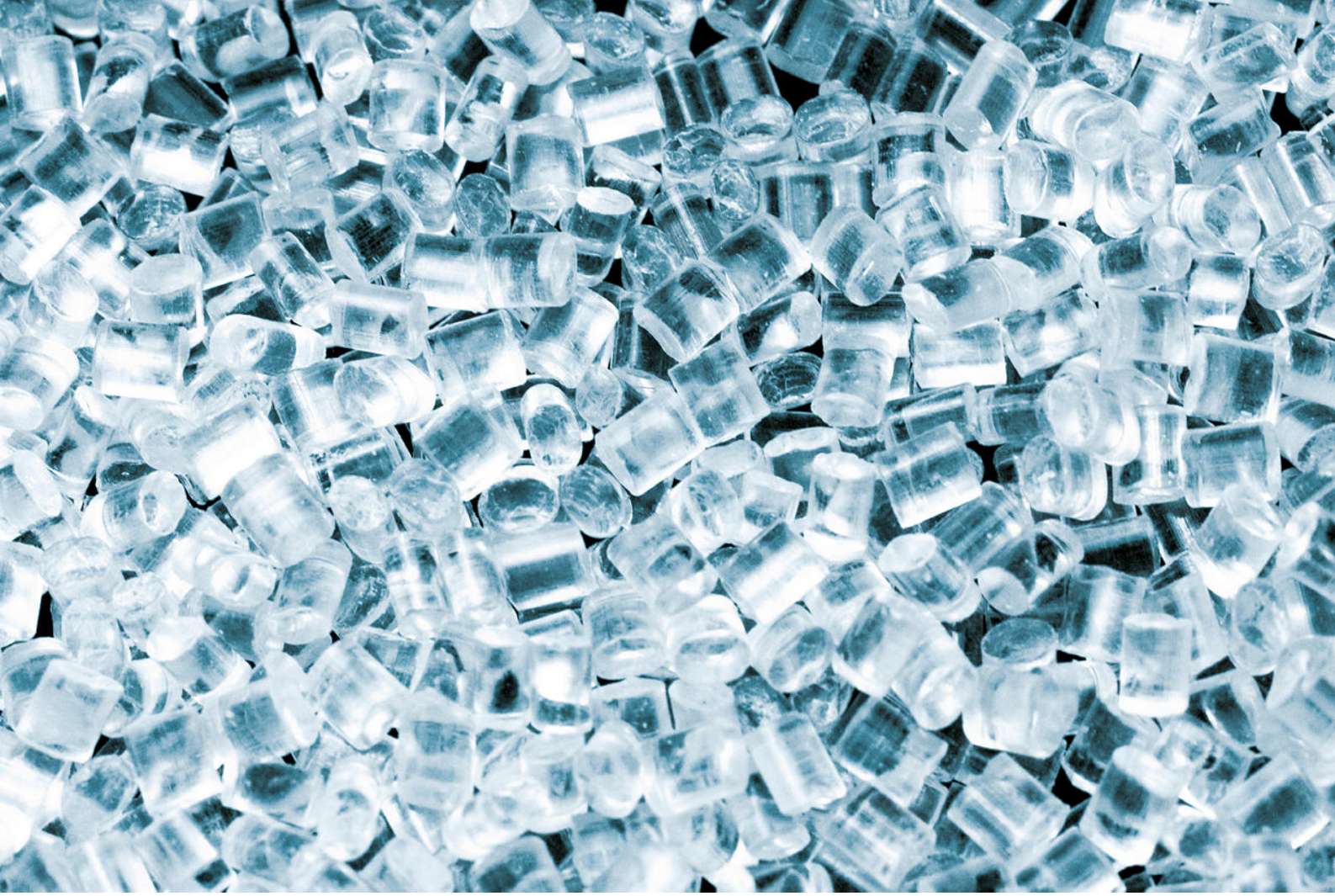


Application Note AN-NIR-100

# 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).

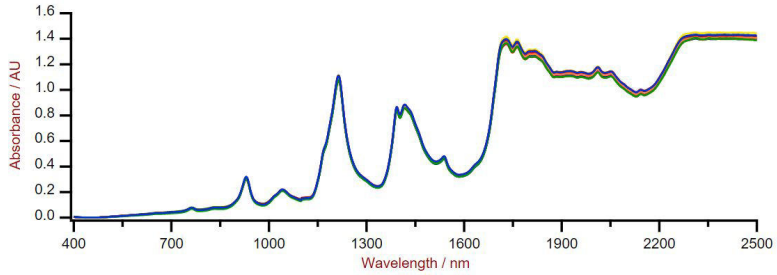



**Figure 2.** Selection of Vis-NIR spectra of PE samples obtained using a Metrohm DS2500 Solid Analyzer equipped with the DS2500 large sample cup.

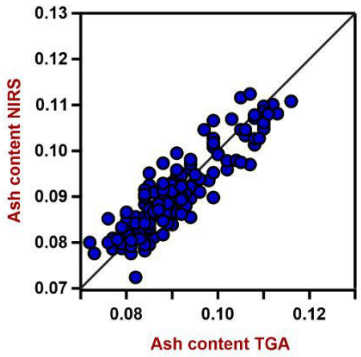



**Figure 3.** Correlation diagram and the respective figures of merit for the prediction of ash content in PE using a DS2500 Solid Analyzer. The lab values were determined using TGA.

**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

**Robuste Nahinfrarotspektroskopie für die Qualitätskontrolle im Labor sowie im Produktionsumfeld.**

Der DS2500 Analyzer ist die bewährte, flexible Lösung für die Routineanalytik von Feststoffen, Cremes und optional auch Flüssigkeiten entlang der gesamten Produktionskette. Das robuste Design macht den DS2500 Analyzer unempfindlich gegen Staub, Feuchtigkeit, Vibrationen sowie Temperaturschwankungen und damit hervorragend geeignet für den Einsatz im rauen Produktionsumfeld.

Der DS2500 deckt den gesamten Spektralbereich von 400 bis 2500 nm ab und liefert in weniger als einer Minute genaue und reproduzierbare Ergebnisse. Der DS2500 Analyzer erfüllt die Anforderungen der pharmazeutischen Industrie und unterstützt durch die einfache Bedienung die Anwender in ihren täglichen Routineaufgaben.

Durch perfekt auf das Gerät abgestimmtes Zubehör werden bei jedem noch so herausfordernder Probentyp, wie z.B. grobkörnige Feststoffe wie Granulate oder halb fest-flüssige Proben wie Cremes, bestmögliche Ergebnisse erzielt. Bei Messungen von Feststoffen kann die Produktivität gesteigert werden durch Einsatz des MultiSample Cups, welches automatisierte Messungen in Serie von bis zu 9 Proben ermöglicht.



## DS2500 Probengefäss, gross

Grosses Probengefäss für die Spektrenaufnahme von Pulvern und Granulaten in Reflektion an unterschiedlichen Probenstellen mittels NIRS DS2500 Analyzer.