



Application Note AN-NIR-106

Quality control of non-nutritive sweeteners with NIR spectroscopy

NIRS gives mixture analysis results within one minute

The use of non-nutritive sweeteners as sugar substitutes in foodstuffs has risen dramatically in the last decade, e.g., in soft drinks and snacks. Two examples are sucralose, a halogenated sucrose derivative, and Stevia, derived from the leaves of the *Stevia rebaudiana* plant. Both are much sweeter than sugar and are used in much lower concentrations in foodstuffs. To ensure food safety, regulations for non-nutritive sweeteners are becoming stricter. Several

analytical methods are available to determine various sweeteners using, e.g., high-performance liquid chromatography (HPLC), ion chromatography, and thin-layer chromatography. However, these methods are time-consuming and incur high running costs. Near-infrared spectroscopy (NIRS) allows the simultaneous determination of several sweeteners in less than one minute without any chemicals or sample preparation.

EXPERIMENTAL EQUIPMENT

Mixtures of both Stevia (0.5–4.5%) and sucralose (0.5–4.5%) in sucrose (95%) were prepared and analyzed to create a prediction model for quantification.

Samples were measured with a Metrohm NIRS

DS2500 Solid Analyzer (**Figure 1**) using 15 mm disposable vials, a DS2500 holder, and a DS2500 Iris in reflection mode. The Metrohm software package Vision Air Complete was used for all data acquisition and prediction model development.

Table 1. Hardware and software equipment overview.

Equipment	Article number
DS2500 Solid Analyzer	2.922.0010
DS2500 Iris	6.7425.100
Disposable vials, 15 mm	6.7402.110
Vision Air 2.0 Complete	6.6072.208



Figure 1. Metrohm NIRS DS2500 Solid Analyzer used to determine Stevia and sucralose content in sucrose mixtures.

RESULT

All measured Vis-NIR spectra (Figure 2) were used to create a prediction model for quantification of sucralose and Stevia in sucrose. The quality of the prediction models was evaluated using correlation diagrams which display a very high correlation

between the Vis-NIR prediction and the reference values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis (Figures 3–4).

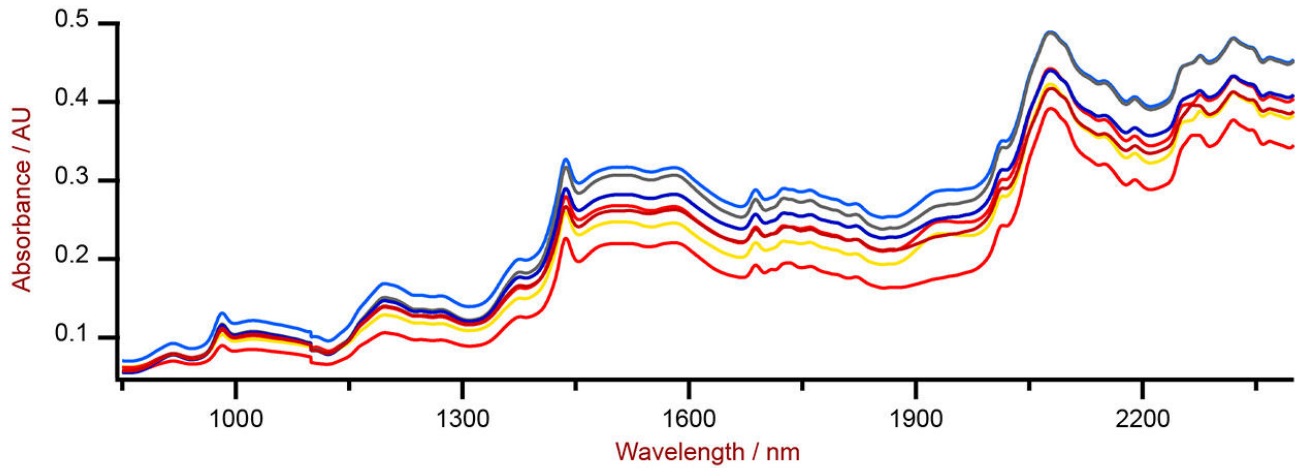


Figure 2. Selection of Vis-NIR spectra of Stevia and sucralose in sucrose samples which were analyzed on a DS2500 Solid Analyzer.

RESULT SUCRALOSE CONTENT IN SUCROSE

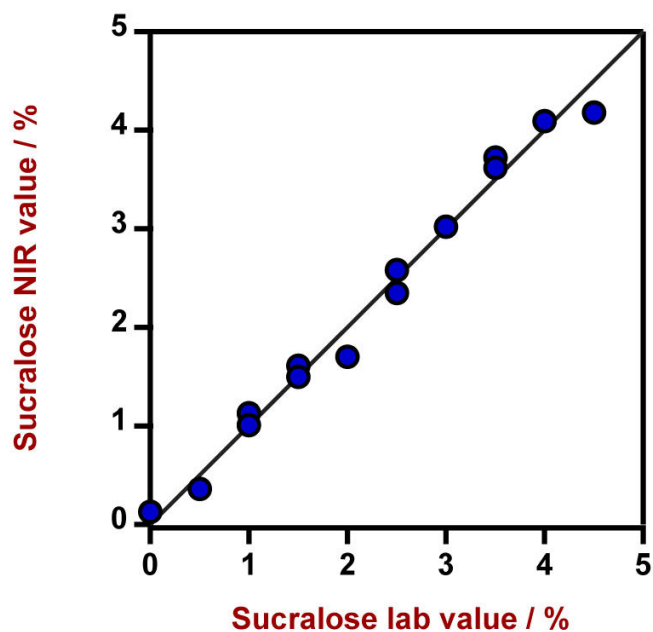


Figure 3. Correlation diagram and the respective figures of merit for the prediction of sucralose content in sucrose using a DS2500 Solid Analyzer. The lab values were determined using HPLC.

Figures of Merit	Value
R^2	0.9854
Standard Error of Calibration	0.1898%
Standard Error of Cross-Validation	0.1997%

RESULT STEVIA CONTENT IN SUCROSE

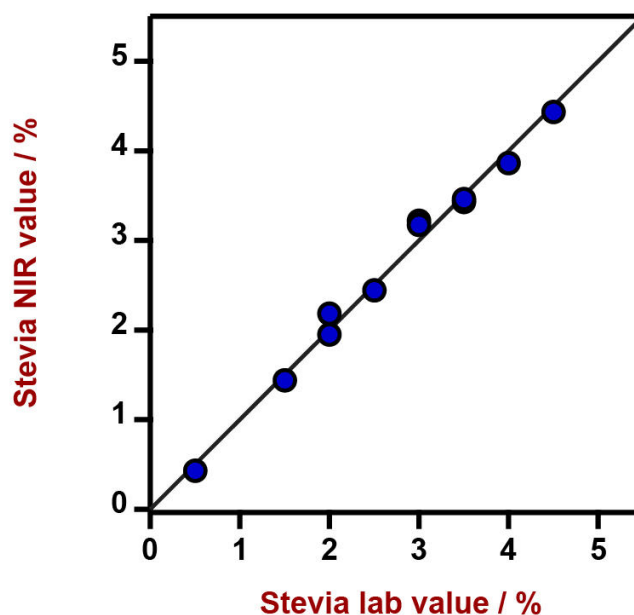


Figure 4. Correlation diagram and the respective figures of merit for the prediction of Stevia content in sucrose using a DS2500 Solid Analyzer. The lab values were determined using HPLC.

Figures of Merit	Value
R^2	0.9885
Standard Error of Calibration	0.1500%
Standard Error of Cross-Validation	0.1997%

CONCLUSION

This Application Note demonstrates the feasibility to determine the concentration of the non-nutritive sweeteners sucralose and Stevia in sucrose blends with NIR spectroscopy. Vis-NIR spectroscopy enables

fast and cost-effective measurements with high accuracy, thereby offering a suitable alternative to other standard analytical methods (Table 2).

Table 2. Time to result overview for the different non-nutritive sweeteners examined in this study.

Parameter	Method	Time to result
Stevia	HPLC	5 min (preparation) + 40 min (HPLC)
Sucralose	HPLC	5 min (preparation) + 40 min (HPLC)

Internal reference: AW NIR AE10-0002-072021

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CONFIGURATION



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.