NEW OFFICIAL FORMULAE FOR
NIR POLARIMETRY
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ABSTRACT

NIR polarimetry becomes more and more important in the sugar industry, as the increased awareness for environmental and health topics makes it desirable to avoid the clarification with basic lead acetate. Until 1998 the ICUMSA recommended to calibrate NIR polarimeters using the well established Bünnergel formulae, although they are based on measurements at visible wavelengths only. In a co-operation between the PTB, the Sugar Institute in Braunschweig and Schmidt + Haensch new formulae for the calibration of polarimeters were evaluated, which were officially adopted at the ICUMSA meeting in Berlin 1998. The main result is, that the values by polarimeters calibrated using the old formulae are too low by 0.06 %, while the temperature correction does not change significantly.

Keywords: Polarimeter, NIR polarimetry, calibration, payment

INTRODUCTION

One of the main advantages of polarimeters over alternative methods for sugar analysis is that these instruments can be calibrated using certified standards, the quartz plates. This makes it possible for both the sugar factory and the farmers to base the payment upon a reliable polarimetric value of a sample.

A disadvantage in the past has been that standard polarimeters were not able to cope with dark samples, and dark samples are very common in the sugar analysis. So most of the samples needed to be “clarified” with basic lead acetate. With the introduction of NIR polarimeters in the 1980’s it became possible to measure even non clarified samples of the sugar industry, as they show a rather low absorbance at NIR wavelengths. This has been a significant development as the use of basic lead acetate should be avoided for safety and environmental reasons.

Until 1998 the ICUMSA1 recommended calibration of NIR polarimeters with a procedure, which was based on formulae evaluated for the standard visible wavelengths (the so called Bünnergel formulae). Works by Altenburg and Chou2, and by Paton et al.3 hinted, that the rotation of sucrose solutions were by 0.06% too low in the NIR if this procedure was used. Both groups were working with the Polartronic NIR by Schmidt + Haensch.

This was the reason for the PTB, the Sugar Institute in Braunschweig and the company Schmidt + Haensch to make precise measurements on polarimetry in the NIR. This lead to a paper by Emmerich et al.4. The results were presented and officially adopted at the 1998 Session of the ICUMSA in Berlin.
ROTATION OF QUARTZ

The rotation $\alpha$ (in angular degree) of a quartz plate of 1 mm thickness at a wavelength $\lambda$ (in $\mu$m) can be calculated as follows:

$$\alpha = -0.1963657 + 7.262667 \cdot \lambda + 0.1171867 \cdot \lambda^2 + 0.0019554 \cdot \lambda^3$$

In the visible there is no difference between the well known Bünnagel formula and this new one, while there is a slight deviation in the NIR:

$$\begin{align*}
\alpha(882.6 \text{ nm, old}) &= 9.323^\circ \\
\alpha(882.6 \text{ nm, new}) &= 9.324^\circ
\end{align*}$$

This is a deviation of 0.01% only.

The temperature effect is very similar in the visible and the NIR, with values of

$$\begin{align*}
\alpha_{20^C}/\alpha_{20^C} \text{ (VIS)} &= 1.0 + 0.000144 \cdot (t - 20) \\
\alpha_{20^C}/\alpha_{20^C} \text{ (NIR)} &= 1.0 + 0.000139 \cdot (t - 20)
\end{align*}$$

ROTATION OF SUCROSE

For sucrose the rotation $\alpha$ of a sucrose solution at a wavelength $\lambda$ (in $\mu$m) in relation to that of the same solution at 546.2271 nm ($\alpha_{546}$) is described by the following formula:

$$\begin{align*}
\alpha(\lambda) &= \alpha_{546} / (a_0 + a_1 \cdot \lambda + a_2 \cdot \lambda^2 + a_3 \cdot \lambda^3) \\
a_0 &= -0.075047659 \\
a_1 &= 3.588221904585 \\
a_2 &= 0.0519461783 \\
a_3 &= -0.006515194377
\end{align*}$$

For the Normal Sugar Solution (26 g to 100 cm$^3$) the correct value is the well known:

$$\alpha_{546} = 40.777289^\circ$$

Again there is no difference between this new formula and the old one in the visible, but in the NIR the values are:

$$\begin{align*}
\alpha(882.60 \text{ nm, old}) &= 14.844^\circ \\
\alpha(882.60 \text{ nm, new}) &= 14.836^\circ
\end{align*}$$

This is a difference of 0.06 %.

The change with the temperature is slightly higher in the NIR than in the visible:

$$\begin{align*}
\alpha_{20^C}/\alpha_{20^C} \text{ (VIS)} &= 1.0 - 0.000474 \cdot (t - 20) \\
\alpha_{20^C}/\alpha_{20^C} \text{ (NIR)} &= 1.0 - 0.000493 \cdot (t - 20)
\end{align*}$$
IMPLICATIONS ON THE CALIBRATION

First of all: The calibration procedures for polarimeters using visible wavelengths do not have to be changed! The changes only apply to polarimeters working in the NIR.

The consequence of the new formulae is that a polarimeter calibrated to 882.60 nm following the old recommendation by the ICUMSA shows 99.94 °Z instead of 100.00 °Z for the Normal Sugar Solution. This result is close to that of Altenburg and Chou and to that of Paton et al.

If an analyst is willing to follow the new calibration procedure, he will thus have to use a new value for his quartz plate. A quartz plate showing 100.00 °Z at 589.44 nm for example should read in the NIR:

<table>
<thead>
<tr>
<th>Wavelength</th>
<th>Angle</th>
<th>°Z</th>
</tr>
</thead>
<tbody>
<tr>
<td>880.00 nm</td>
<td>14.953° ± 0.001°</td>
<td>100.18 °Z</td>
</tr>
<tr>
<td>882.60 nm</td>
<td>14.862° ± 0.001°</td>
<td>100.17 °Z</td>
</tr>
</tbody>
</table>

If the customer knows the correct reading of his quartz for 589.44 nm, he can thus calculate the reading at 880.00 nm by multiplying the value by 1.00177, for 882.60 nm he has to multiply by 1.00174.

If he knows the correct reading in the NIR following the old procedure, he has to multiply this value by 1.00062 (880.0 nm) or 1.00063 (882.6 nm), resp.

QUARTZ WEDGE SACCHARIMETER

The quartz wedge saccharimeter is well established in the sugar industry. The special advantage is that this instrument, unlike all other polarimeters, does not need a regular re-calibration.

Today the instrument is available for the well known yellow wavelength, and also for the NIR (882.60 nm).

FILTRATION

All polarimeters need clear solutions for a proper measurement. Indeed, the clarification with basic lead acetate does not only remove the colour, but also the turbidity, so that a filtration of such clarified samples is easy and fast. For NIR polarimeters the samples have to be filtered directly, with adding a filtration aid like Celite only. Procedure for making this filtration faster and more convenient are being sought. Promising tests show that the automatic filtration unit Labfilt can be used successfully.

OUTLOOK

Having a reliable way to calibrate NIR polarimeters, the door is open for collaborative tests for a wider use of this important method (for example with raw sugars).

REFERENCES

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FORMULA OFICIAL NUEVA PARA POLARIMETRÍA INFRAROJA

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RESUMEN

Polarimetría infrarroja resulta más y más importante en la industria azucarera, al aumentar el interés en tópicos ambientales y de salud, haciendo deseable evitar la clarificación con acetato de plomo. Hasta 1998 el ICUMSA recomendó calibrar polarímetros NIR usando la muy establecida fórmula de Bunnagel, aunque estos están basados solamente en medidas de longitud de onda largas visibles en una co-operación entre el PTB, el Instituto de Azúcar de Braunschweig y la compañía Schmidt y Haensch, se evaluaron nuevas fórmulas para la calibración de polarímetros, las cuales fueron oficialmente adoptadas en la reunión del ICUMSA en Berlin de 1998.

El resultado principal es que los valores de polarímetros calibrados usando la fórmula vieja son reducidos por un 0,06%, mientras la corrección de temperatura no cambia significativamente.

Palabras claves: Polarímetro, NIR polarimetría, calibración, pago

NOUVELLES FORMULES OFFICIELLE POUR LA POLARIMÉTRIE NIR

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RÉSUMÉ

La polarimètre NIR offre des possibilités intéressantes pour l’industrie sucrière et elle permettrait d’éviter l’utilisation du plomb pour la clarification. ICUMSA avait recommandé la formule Bünemel jusqu’a 1998, pour la calibration des polarimètre NIR, malgré que la formule soit basée sur des mesures aux longueurs d’onde dans le spectre visible seulement. En co-opération avec le PTB, le Sugar Institute de Braunschweig et Schmidt & Haensch, une nouvelle formule a été évaluée. Elle a été adoptée officiellement pendant la conférence ICUMSA de Berlin en 1998.

Le résultat principal est que les valeurs donnés par l’ancienne formule sont plus basse par 0,06% mais la correction pour la température ne change pas.