

Strategy for introducing NIR spectroscopy and multivariate calibration techniques in industry

M.S. Larrechi, M.P. Callao

Analytical methods based on near infrared spectroscopy (NIR) and multivariate calibration techniques have important characteristics for industrial applications because they are quick and can be used to make direct determinations and extract information from several parameters from one single measurement. They can also be used in on-line applications. However, they have not been used in industry as much as one might expect because the validity of the calibration model must be assured when the signal is transformed. There are chemometric techniques for tackling every problem, but there are no specific guidelines about which procedure to use. In this article, we describe a practical strategy for introducing these methods. We discuss the relative merits of the various univariate and multivariate control techniques and describe how they might be used. We also discuss the standardisation of the calibration models and describe how to obtain robust models at low experimental cost. Finally, we illustrate our strategy with a practical case study from the petrochemical industry.

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1. Introduction

NIR combined with multivariate processing techniques has been widely studied in recent years. There have been important applications, for example in the agricultural industry [1], the pharmaceutical industry [2] and the petrochemical industry [3].

In the petrochemical industry, the method made a strong impact when it was used to determine the number of octanes in different types of petrol [4]. The method is interesting because a wide variety of samples can be measured without the need for pre-treatment and because the determinations can be made quickly. Both of these are highly desirable features for on-line applications.

However, NIR has not been fully introduced in the above areas as an alternative to conventional analytical methods. We believe this may partly be because it is sensitive to factors such as temperature, the ageing of the source, and small instrumental disturbances that can affect the validity of the models over time. This suggests that the system needs to be controlled. It may also be because the goodness of the results depends on whether the samples analysed are of the same type as those used as calibration samples. Outliers in prediction should therefore be easily detected.

These problems (control, suitability of the system if a change is detected, and outlier detection) have been widely studied [5–7]. Such studies have illustrated the chemometric approaches to solving the problems, but, in our opinion, they have always dealt with just one of the problems and have been too theoretical. We think that these techniques need general practical guidelines that can be applied to each particular field.

Another problem is that these methods are normally implemented by specialised personnel but are used on a day-to-day basis by operators who are unfamiliar with the science on which they are based. It would be useful, therefore, if they contained means by which the operators could feel that they were involved in analysing the results they helped to produce.

We are developing NIR methods for use in the petrochemical industry [8–10]. In this article, we use this experience to discuss a practical strategy for using these methods effectively. As an example, we will discuss the determination of the ethylene

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Table of Contents Application Of Multivariate Calibration And Nir

1. Understanding the eBook Application Of Multivariate Calibration And Nir
 - The Rise of Digital Reading Application Of Multivariate Calibration And Nir
 - Advantages of eBooks Over Traditional Books
2. Identifying Application Of Multivariate Calibration And Nir
 - Exploring Different Genres
 - Considering Fiction vs. Non-Fiction
 - Determining Your Reading Goals
3. Choosing the Right eBook Platform
 - Popular eBook Platforms
 - Features to Look for in an Application Of Multivariate Calibration And Nir
 - User-Friendly Interface
4. Exploring eBook Recommendations from Application Of Multivariate Calibration And Nir
 - Personalized Recommendations
 - Application Of Multivariate Calibration And Nir User Reviews and Ratings
 - Application Of Multivariate Calibration And Nir and Bestseller Lists

5. Accessing Application Of Multivariate Calibration And Nir Free and Paid eBooks
 - Application Of Multivariate Calibration And Nir Public Domain eBooks
 - Application Of Multivariate Calibration And Nir eBook Subscription Services
 - Application Of Multivariate Calibration And Nir Budget-Friendly Options
6. Navigating Application Of Multivariate Calibration And Nir eBook Formats
 - ePub, PDF, MOBI, and More
 - Application Of Multivariate Calibration And Nir Compatibility with Devices
 - Application Of Multivariate Calibration And Nir Enhanced eBook Features
7. Enhancing Your Reading Experience
 - Adjustable Fonts and Text Sizes of Application Of Multivariate Calibration And Nir
 - Highlighting and Note-Taking Application Of Multivariate Calibration And Nir
 - Interactive Elements Application Of Multivariate Calibration And Nir
8. Staying Engaged with Application Of Multivariate Calibration And Nir
 - Joining Online Reading Communities
 - Participating in Virtual Book Clubs
 - Following Authors and Publishers Application Of Multivariate Calibration And Nir
9. Balancing eBooks and Physical Books Application Of Multivariate Calibration And Nir
 - Benefits of a Digital Library
 - Creating a Diverse Reading Collection Application Of Multivariate Calibration And Nir
10. Overcoming Reading Challenges
 - Dealing with Digital Eye Strain
 - Minimizing Distractions
 - Managing Screen Time
11. Cultivating a Reading Routine Application Of Multivariate Calibration And Nir
 - Setting Reading Goals Application Of Multivariate Calibration And Nir
 - Carving Out Dedicated Reading Time
12. Sourcing Reliable Information of Application Of Multivariate Calibration And Nir
 - Fact-Checking eBook Content of Application Of Multivariate Calibration And Nir
 - Distinguishing Credible Sources
13. Promoting Lifelong Learning

- Utilizing eBooks for Skill Development
 - Exploring Educational eBooks
14. Embracing eBook Trends
- Integration of Multimedia Elements
 - Interactive and Gamified eBooks

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