

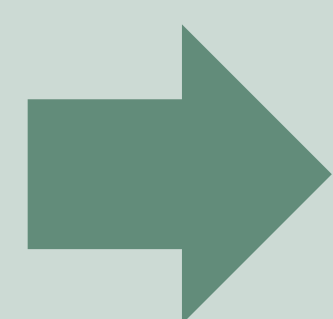
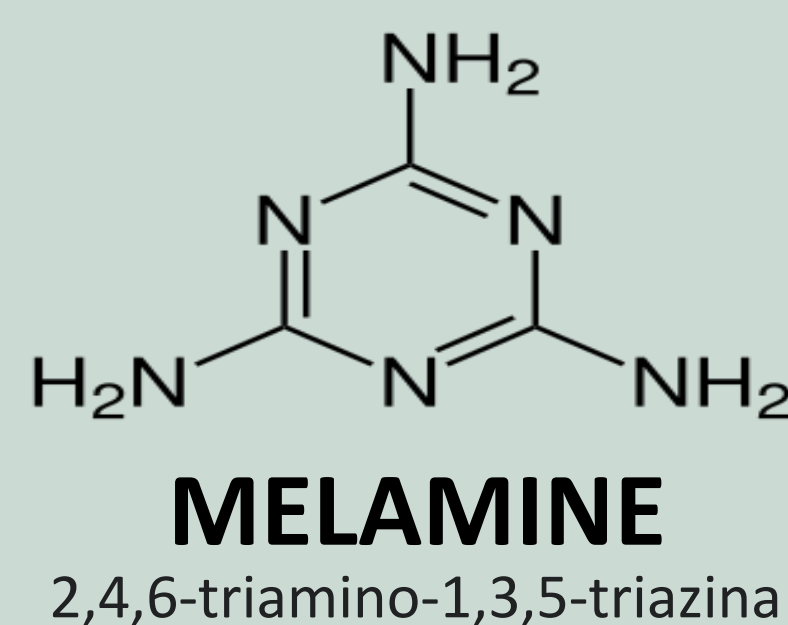
DETECTION AND QUANTIFICATION OF MELAMINE IN MILK POWDER BY NIR SPECTROSCOPY

CARTIF

M. Isabel Campos^{1,2}, Raquel Sánchez², Luis Debán²

¹ CARTIF Technology Center, Agrifood and Processes Division, Parque Tecnológico de Boecillo, 205. Valladolid, Spain. marcam@cartif.es

² Analytical Chemistry Department, Faculty of Sciences, University of Valladolid. Spain.



**MILK
POWDER**



**September 2008 China
300,000 children poisoned**

ABSTRACT

Melamine is a nitrogenous compound that has been fraudulently used to increase the apparent protein content in dairy products, leading to severe health consequences due to its toxicity. In this work, near infrared spectroscopy (NIRS), combined with chemometric methods, has been used to detect and quantify melamine in milk powder samples.

METHODOLOGY

Analytical technique: **NEAR INFRARED SPECTROSCOPY (NIRS)**

Accurate, fast and low cost per measurement. Non-destructive and non-invasive, without sample pre-treatment or reagents.

Equipment: Fourier Transform (FT) NIR spectrometer model MPA (Bruker Optik), He-Ne laser beam of 633 nm. PbS detector.

Reference method: HPLC with UV-Visible detection (λ , 204 nm). Temperature, 20°C ; Flow rate, 1 mL/min; Injection volume, 5 μ L

Mobile phase: Water and acetic acid (1%) in a 90:10 ratio

Stationary phase: Column C18

Standard solution 100 ppm melamine in deionised water, from which standard solutions of different concentrations are prepared

Well-defined peak at 12 minutes. 1.07 ppm LOD and 3.26 ppm LOQ

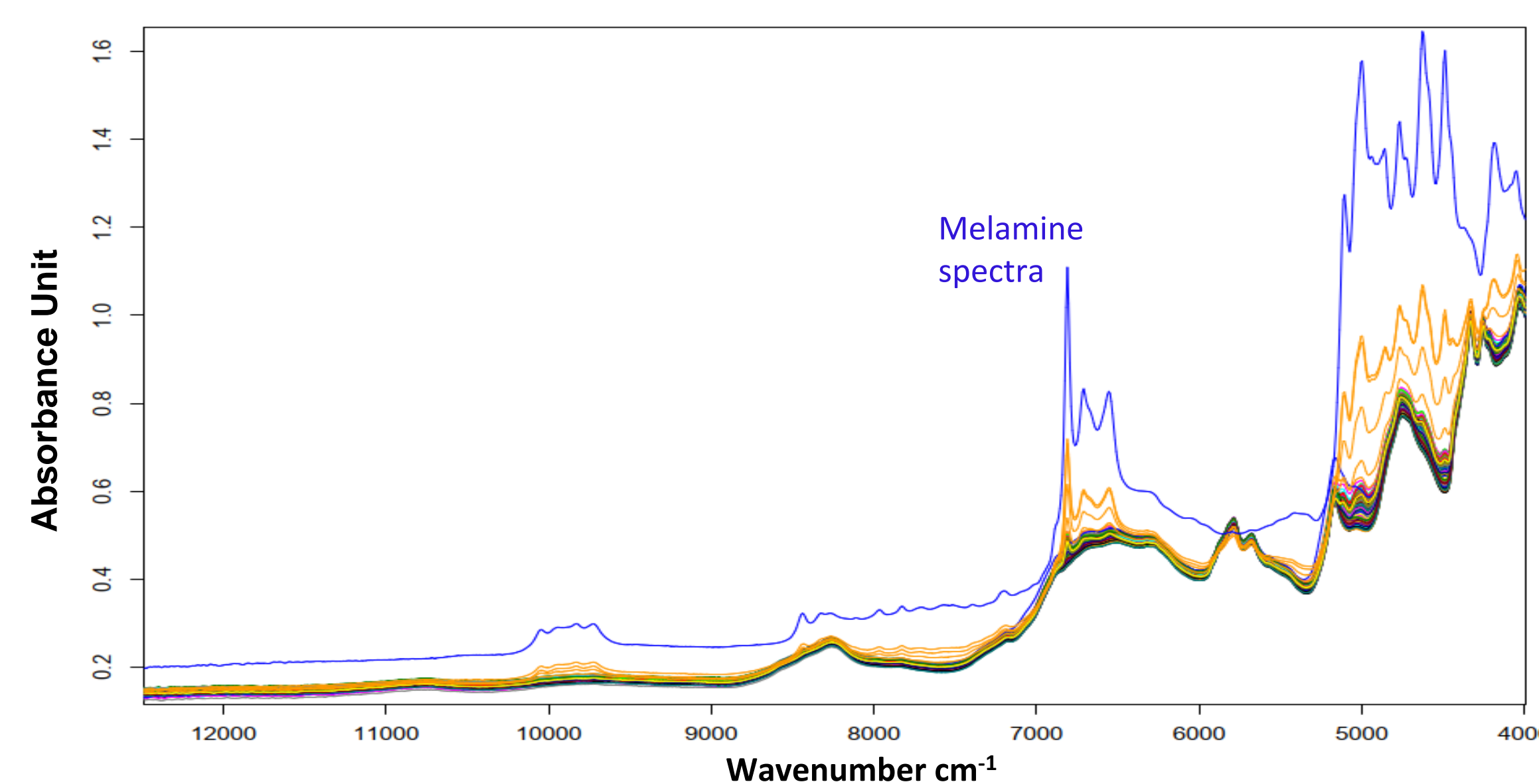
NIRS Methodology

A total of 130 solid samples with different melamine concentrations were prepared from commercial milk powders. Reflectance spectra were collected using a NIR fibre optic probe in a spectral range between 4000 and 12500 cm^{-1} . Scans were performed at a wavenumber resolution of 16 cm^{-1} .

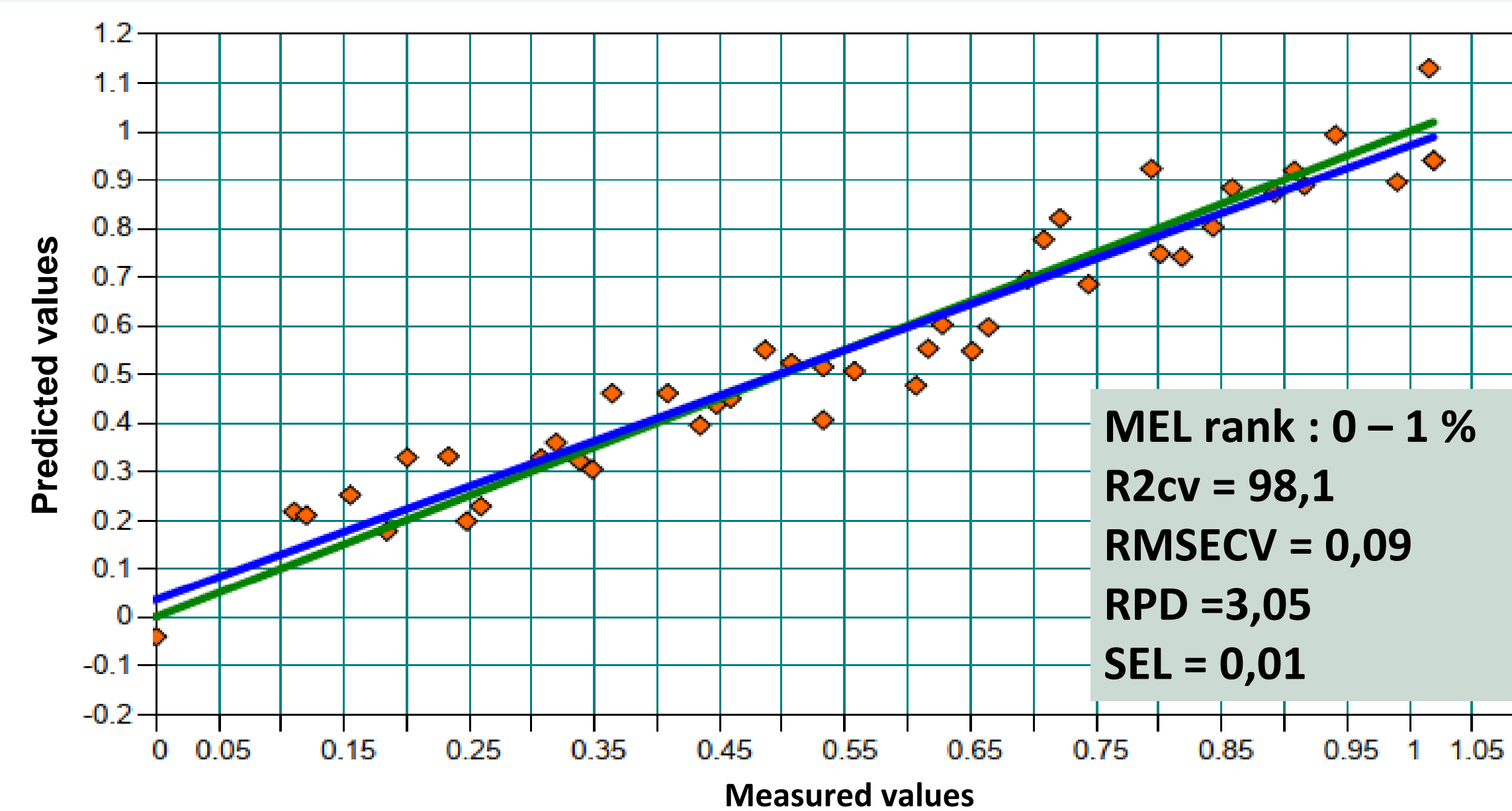
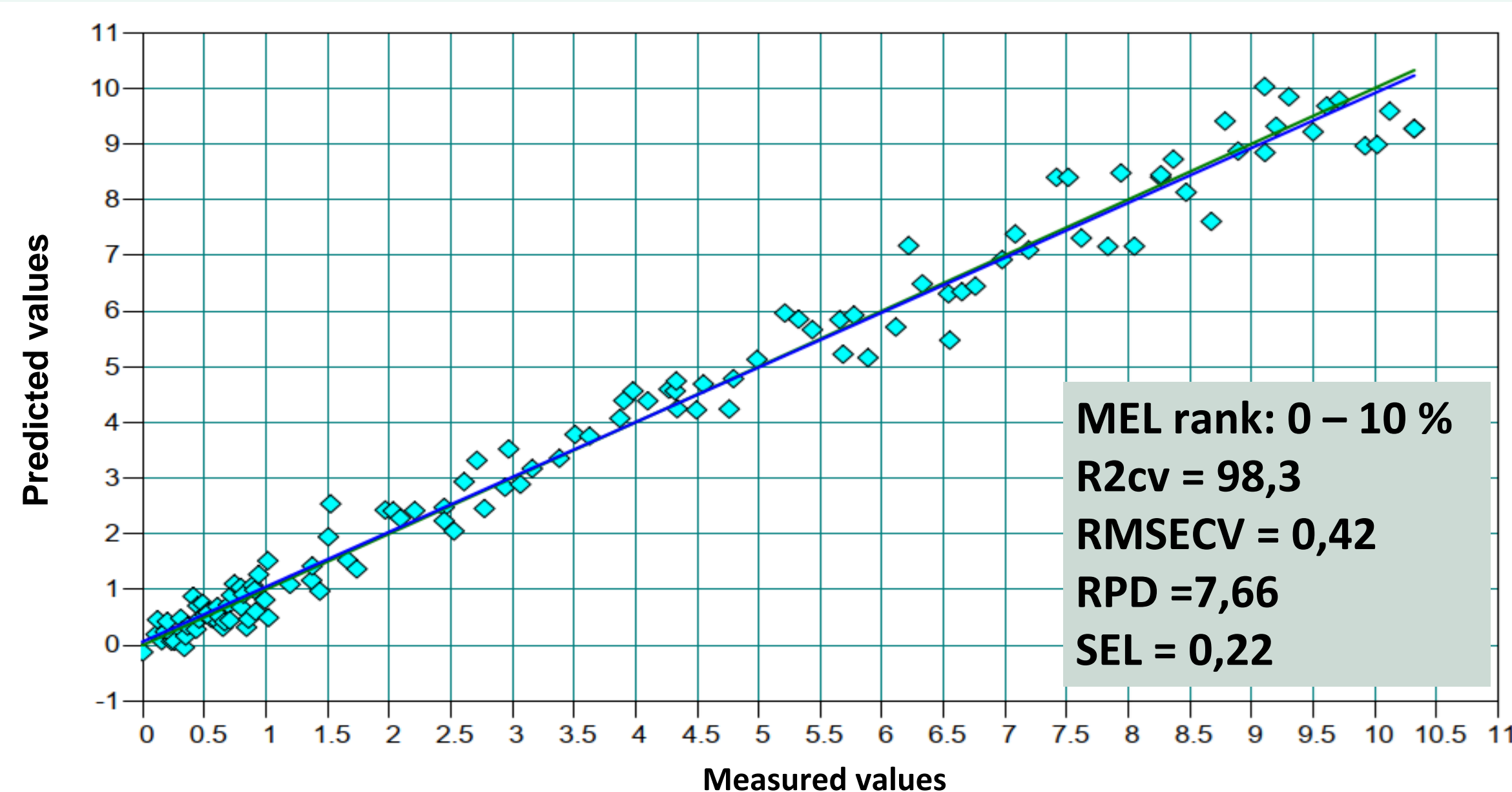
Prediction models: Spectral data were analysed by principal component analysis (PCA) and cross-validated calibration equations were constructed using partial least squares regression (PLSR).

(OPUS/QUANT chemometric software)

NIR spectra of milk powders

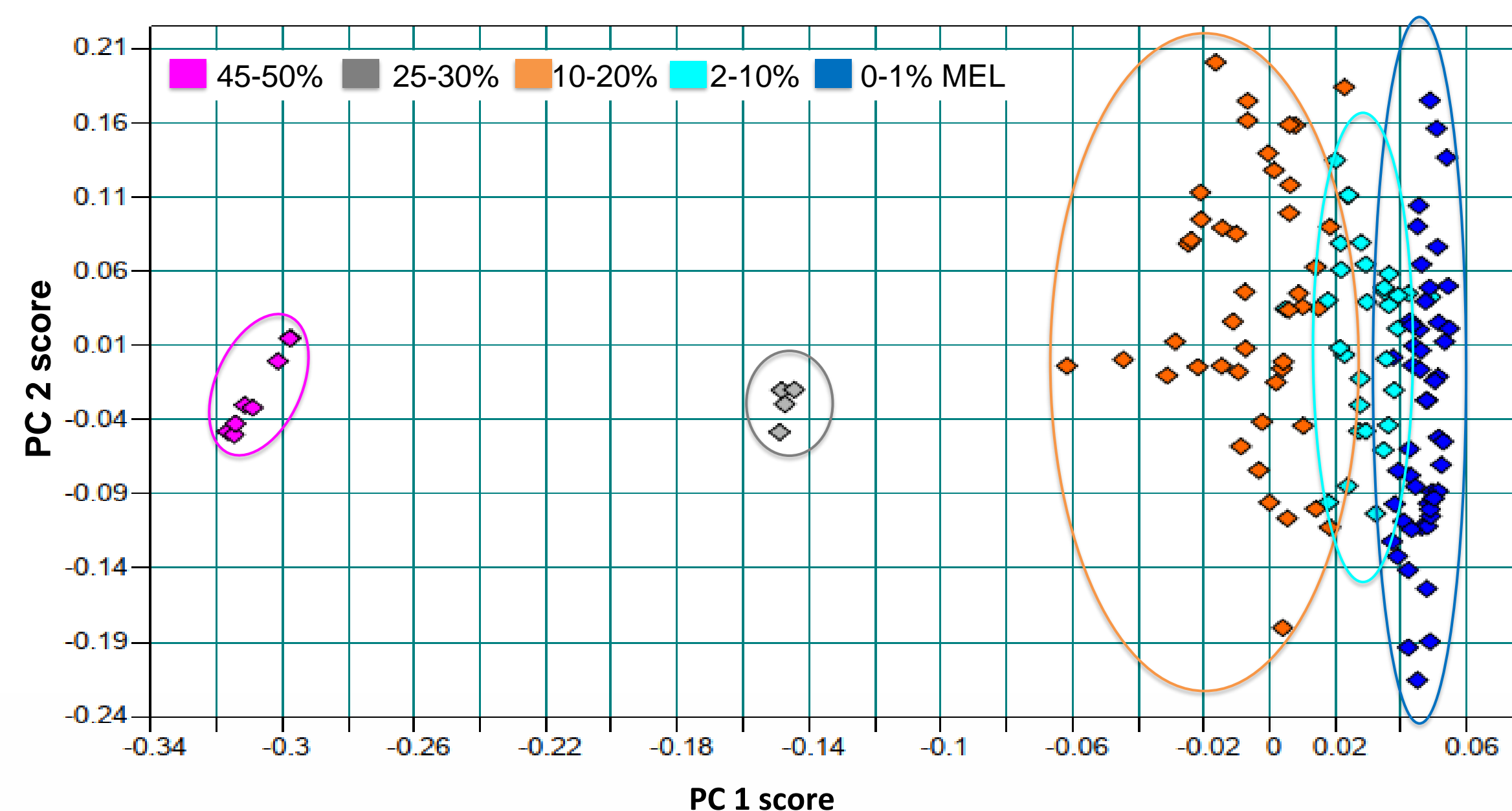


QUANTITATIVE PREDICTIVE MODELS: NIR prediction plot: True vs predicted (% melamine)



QUALITATIVE MODEL:

Principal component score plot of the NIR spectra of milk powders grouped according to melamine content



CONCLUSIONS

Prediction models in different melamine concentration ranges between 0-10%, obtaining cross-validation correlation coefficients (R^2_{CV}) higher than 98%. The predictive capacity in the external validation reached values of 7.66 and 3.05, with standard errors of prediction of 0.42% and 0.09%. PCA enabled the classification of samples according to their melamine concentration, detecting the degree of contamination solely by spectral information. **These results confirm that NIRS is a viable alternative for the detection and quantification of melamine in milk powder samples.**

Venkatasami et al. (2010). A rapid, acetonitrile-free, HPLC method for determination of melamine in infant formula. *Anal. Chim. Acta*.
Ting et al. (2020). Feasibility of fraud detection in milk powder using a handheld near-infrared spectroscopy. *AIP Conf. Proc.*
Liang et al. (2021). Detecting melamine-adulterated raw milk by using near-infrared transmission spectroscopy. *J. Food Process Eng.*
Shutevska et al. (2024). Rapid quantification models for assessing melamine adulteration in sport nutrition supplements via NIRS. *Spectrochim Acta A*