# Investigation of the lyophilization effect on mixed milk powder products by near infrared spectroscopy

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## Introduction

Multivariate near infrared (NIR) calibration was performed on powder mixtures of whole milk, skimmed milk, whey, retentate and lactose for the determination of fat, protein, lactose, water and ash content. More than 150 samples were prepared and measured. The measured samples were dissolved in water to create artificial milk solution with different main components. These solutions were then lyophilized at 38ºC.

Multivariate NIR calibration was performed on both original and lyophilized samples and the results obtained from the same samples were compared. The qualitative evaluations were performed using the PQS method, determining the quality points of the milk powder samples.

## The aims of the study

Rapid quantitative and qualitative determination of mixtures of milk powder products using NIR. Comparison of the results achieved before and after lyophilization process.

## Materials

The composition of the raw materials (%) used is given in Table 1. Four raw materials were used and blended in different ratios for producing the calibration and prediction samples. In total, we had 104 samples.

In order to see the influences of the fat content we blended samples with high fat content milk powder as well, and we had 48 additional samples.

**Table 1. Composition of raw materials used for analysis.**

| Constituents | Skimmed milk | Whey powder | Protein conc. | Lactose |
|---|---|---|---|---|
| Fat | 0.9 | 2.8 | 0.5 | 0.0 |
| Protein | 36.3 | 15.3 | 80.3 | 0.0 |
| Lactose | 50.9 | 73.1 | 6.6 | 100.0 |
| Water | 4.1 | 1.4 | 4.8 | 0.0 |
| Ash | 7.8 | 7.4 | 7.8 | 0.0 |

## Instruments

A Leybold–Heraeus GT2 lyophilization apparatus and a PMC Spectralyzer 1025 scanning type NIR spectrophotometer were used.

## Methods

The samples were dissolved in distillated water, cooled and then lyophilized. The NIR investigations were performed before and after the lyophilization process. For statistical evaluation, multivariate analysis was performed using the software built-in to the NIR instruments.

The qualitative evaluation and diagrammatic results (Figures 1–6) the drawings were prepared using the PQS software.

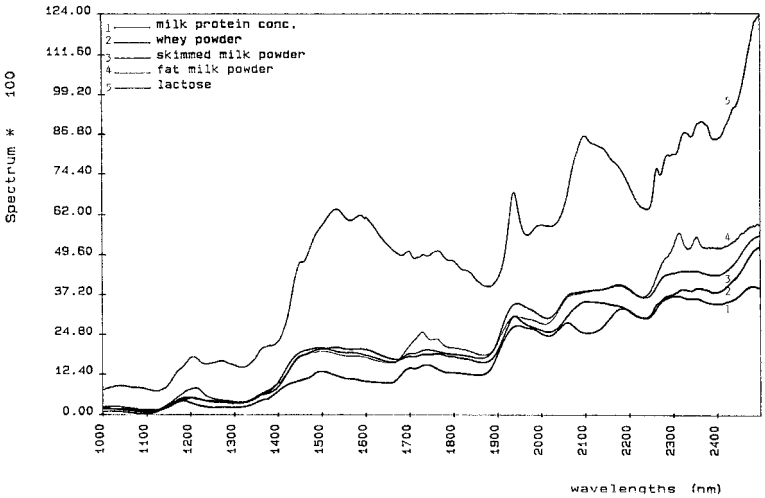


**Figure 1. The log (1/*R*) spectra of the raw materials.**

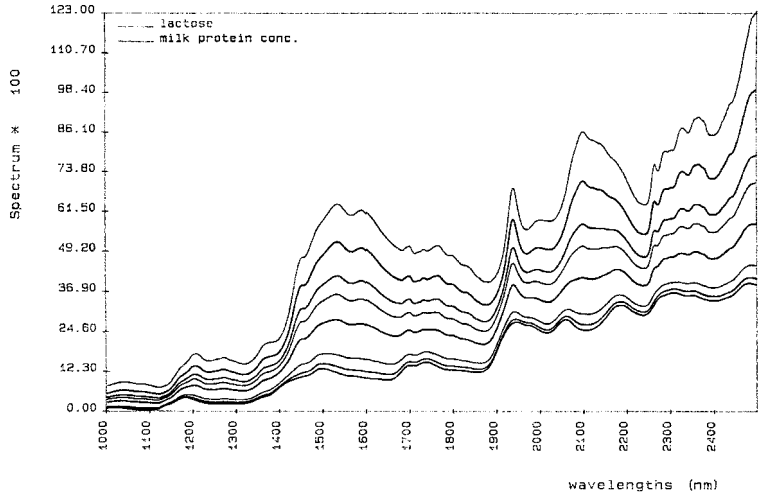


**Figure 2. The log (1/*R*) NIR spectra of the mixtures of protein concentrate and lactose.**

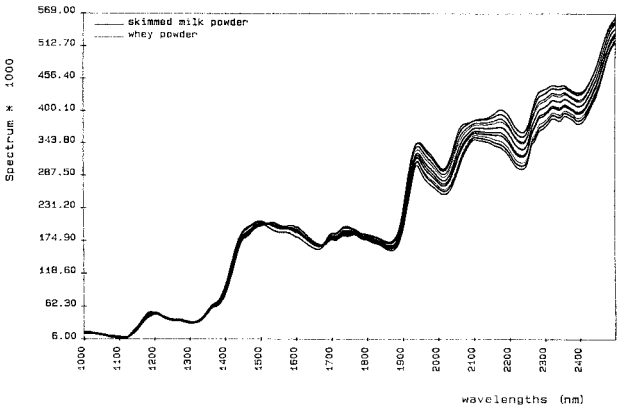


**Figure 3. The log (1/*R*) NIR spectra of the mixture of skimmed milk and whey powder.**

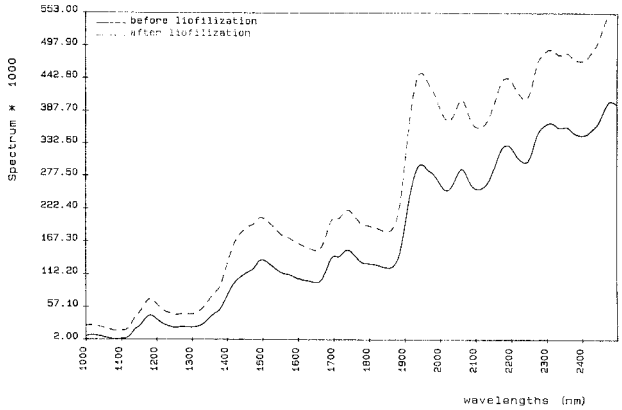


**Figure 4. The log (1/*R*) NIR spectra of the protein concentrate before and after lyophilization.**

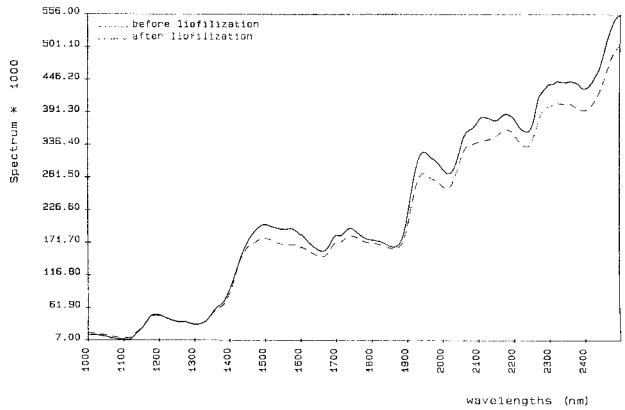


**Figure 5. The log (1/*R*) NIR spectra of the skimmed milk powder before and after lyophilization.**

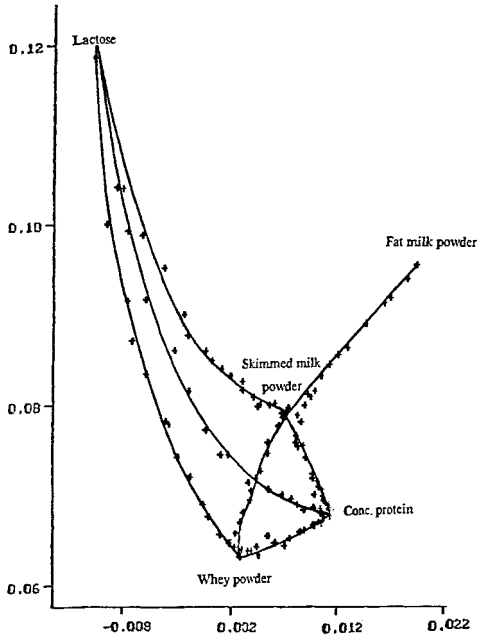


**Figure 6. Quality points of the milk powder samples, obtained as the gravity points of log (1/*R*) spectra represented in polar coordinate system from 1000 to 2500 nm.**

**Table 2. The performance data obtained by the NIR instrument before the lyophilization process.**

| Constituents | Best wavelengths (nm) | Stand. error (*SEC*) (mass%) | Corr. coeff. (*R*) | Calibr. range (mass%) |
|---|---|---|---|---|
| Fat | 1400<br>1604<br>2172<br>2188<br>2476 | 0.128 | 0.986 | 0–2.8 |
| Protein | 1534<br>1586<br>1600<br>2372<br>2498 | 0.128 | 0.986 | 0–80.3 |
| Lactose | 1084<br>1086<br>1100<br>1530<br>1600 | 2.615 | 0.995 | 6.6–100 |

**Table 2 (continued). The performance data obtained by the NIR instrument before the lyophilization process.**

| Constituents | Best wavelengths (nm) | Stand. error (*SEC*) (mass%) | Corr. coeff. (*R*) | Calibr. range (mass%) |
|---|---|---|---|---|
| Water | 1020<br>1496<br>1610<br>1966<br>2350 | 0.101 | 0.998 | 0–5 |
| Ash | 1124<br>1380<br>2144<br>2440<br>2460 | 0.285 | 0.993 | 0–8 |

**Table 3. The performance data obtained by the NIR instrument after the lyophilization process.**

| Constituents | Best wavelengths (nm) | Stand. error (*SEC*) (mass%) | Corr. coeff. (*R*) | Calibr. range (mass% ) |
|---|---|---|---|---|
| Fat | 1412<br>2192<br>2268<br>2314<br>2408 | 0.166 | 0.976 | 0–2.8 |
| Protein | 1392<br>2080<br>2110<br>2450<br>2452 | 2.772 | 0.933 | 0–80.3 |
| Lactose | 1198<br>1212<br>1274<br>1934<br>1942 | 3.476 | 0.991 | 6.6–100 |
| Water | 1104<br>1392<br>1722<br>1952<br>2066 | 0.343 | 0.986 | 0–10.42 |

**Table 3 (continued). The performance data obtained by the NIR instrument after the lyophilization process.**

| Constituents | Best wavelengths (nm) | Stand. error (*SEC*) (mass%) | Corr. coeff. (*R*) | Calibr. range (mass% ) |
|---|---|---|---|---|
| Ash | 1132<br>2060<br>2180<br>2256<br>2268 | 0.519 | 0.975 | 0–8.0 |

## Results

The results of the NIR analysis before and after the lyophilization process are shown in Tables 2 and 3.

## Conclusions

Scanning type NIR instruments can be used as multicomponent analysers for determining the percentages of the constituents in the milk powder mixtures. The achievable accuracy data for the five constituents are given. These are acceptable for industrial practice. The PQS qualification system proved to be a very useful and simple method without time consuming calibration.