# Sample Preparation of Feeds and Forage for NIR Analysis

The feed quality has a decisive influence on the productivity of livestock farming. However, high-quality forage considerably adds to the costs of stock breeding. Consequently, economic considerations focus on the quality and quantity of the components used in the production process. This includes reliable analyses of feedstuff and ingredients from receipt of goods to final inspection as the best way to ensure a balanced feeding of the animals while keeping cost and profit orientation in focus.

Near Infrared Spectroscopy is the most important analytical method for the determination of protein content, moisture, fat and ash in feeds and forage. The advantage over classical methods such as Kjeldahl is the simultaneous determination of several parameters. Moreover, NIR spectroscopy is a quick method which requires neither consumables nor reagents. Therefore it is used whenever high sample throughput and great flexibility are required. The identification and qualification of raw materials as well as the quantitative analysis of convenience products can be carried out within seconds to guarantee highest product quality and safety.

A much discussed issue related to NIR analysis is the necessity of sample preparation. Users often face the problem of having to decide whether sample preparation is required or not.

**What does sample preparation for NIR analysis involve?**

Sample preparation for NIR does not require digestion or extraction; it is mainly about size reduction of the sample material. This involves two aspects:

1. Homogenizing the sample
2. Achieving the required grind size

Whereas an inhomogeneous sample leads to systematic errors in the subsequent analysis, a sample which is too coarse causes statistical errors. This will be explained in the following.

The penetration depth of NIR radiation in grain or forage is up to 1 mm, measured in diffuse reflection. Thus, the composition of the sample's deeper layers is not detected. That is not a problem if the sample/pellet is homogeneous in itself. When examining pellets coated with fat or grains and seeds, however, the ingredients of the analyzed layers are over represented in relation to the complete sample.

If the material is homogeneous but does not have the required analytical fineness, the surface actually analyzed is reduced. This is due to the fact that the light inside the sample is scattered and not returned back to the detector. The same happens with light, fibrous samples with a low bulk density. As this effect is influenced by the filling level of the sample container and the surface actually analyzed, the statistic error is rather big in such cases.

**Test series with different samples**

The different properties of ground and unground samples when analyzed with NIR are demonstrated exemplarily with three different materials: rabbit food pellets, wheat grains and hay. The samples were analyzed 10 times, the spectrometer was refilled for every measurement.

The samples were pulverized in **RETSCH's cyclone mill TWISTER**. This mill was specially designed for sample preparation of food and feed for subsequent NIR analysis. In the TWISTER size reduction is effected by **impact and friction** between the rotor and the friction surface of the fixed grinding ring. The feed material passes through the hopper (with splash-back protection) onto the rotor, which is rotating with high speed, and is thus submitted to preliminary size reduction. The sample is then thrown outwards by centrifugal acceleration and is

pulverized between rotor and grinding ring. The **2-step grinding** ensures particularly **gentle but fast processing** so that the feed material only remains in the grinding chamber for a very short time.


The integrated cyclone provides cooling of the sample and the grinding tools which helps to preserve the moisture content. The sample properties to be determined are not altered in any way. The ground material is separated in the cyclone and collected in a sample bottle. The cyclone mill TWISTER is also suitable for processing large numbers of different materials as no cleaning between samples is required.
The quantitative measurements were carried out with the NIR spectrometer TANGO from Bruker Optik GmbH.

The first sample to be analyzed was rabbit food pellets. The measurement results of the ground and unground samples hardly show any difference.

| | **Ash** | **Moisture** | **Fiber content** | **Fat** | **Protein** |
|---|---|---|---|---|---|
| Ground rabbit food pellets | | | | | |
| Average | 8.58 | 10.20 | 11.68 | 3.11 | 13.70 |
| Standard deviation | 0.07 | 0.23 | 0.34 | 0.03 | 0.18 |
| Unground rabbit food pellets | | | | | |
| Average | 8.67 | 10.81 | 11.46 | 3.16 | 13.19 |
| Standard deviation | 0.10 | 0.15 | 0.59 | 0.03 | 0.13 |

*Table 1: The measurement results of various parameters in ground and unground rabbit food pellets only show slight differences*

The results for wheat are different. The table shows a considerable discrepancy between ground and unground sample, particularly with regards to ash and fiber content. This is due to the fact that only the surface of the unground wheat grains is analyzed resulting in an over representation of the kernel shell.

| | **Ash** | **Moisture** | **Fiber content** | **Fat** | **Protein** |
|---|---|---|---|---|---|
| Ground wheat | | | | | |
| Average | **2.80** | 9.68 | **1.10** | 1.17 | 9.02 |
| Standard deviation | 0.03 | 0.09 | 0.05 | 0.03 | 0.07 |
| Unground wheat | | | | | |
| Average | **0.10** | 9.80 | **6.90** | 1.38 | 8.46 |
| Standard deviation | 0.10 | 0.25 | 0.62 | 0.16 | 0.45 |

*Table 2: The analysis of wheat grains shows a considerable difference in the ash and fiber content of the ground and unground sample.*

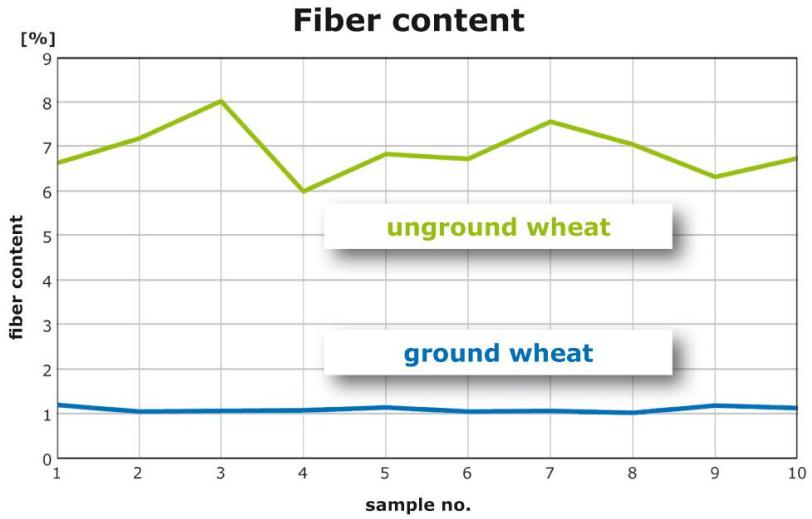


*Figure 1: Fiber content of the ground (blue) and unground (green) wheat sample resulting from 10 measurements. The analysis of the unground sample clearly shows a systematic as well as a considerable statistic error.*

The third analyzed sample is hay. This sample shows a substantial difference for the average and the standard deviation of the protein content. This is because of the varying bulk density of the unground sample.

| Parameter | Ash | Moisture | Fiber content | Fat | Protein |
|---|---|---|---|---|---|
| Ground hay | | | | | |
| Average | 9.53 | 5.70 | 28.56 | 1.36 | **6.17** |
| Standard deviation | 0.07 | 0.12 | 0.29 | 0.04 | **0.12** |
| Unground hay | | | | | |
| Average | 9.31 | 6.25 | 26.26 | 2.44 | **9.94** |
| Standard deviation | 0.07 | 0.09 | 0.68 | 0.18 | **0.43** |

*Table 3: The analysis of hay shows a strong difference in the protein content and standard deviation between ground and unground sample.*

**Conclusion**

NIR spectroscopy is a simple and suitable method to determine a series of relevant parameters in forage and grain. The general opinion is that it doesn't require any sample preparation. The results presented in this article show, however, that it does make a difference if the samples are ground in a suitable laboratory mill before being analyzed, particularly if they are inhomogeneous. Only then is it possible to guarantee meaningful and reliable analysis results.