Analysis of Animal Feeds - Reference Chemistry Limitations and Challenges with Mineral Additives

Introduction

The testing of animal feeds has great importance in part due to the raising of various animals for consumption. In order to obtain the highest quality meats, the diets of the animals are strictly controlled to maximize their profitability. Obtaining accurate and repeatable reference results on these animal feeds is of paramount importance. In the US, animal feed is tested under AOAC 920.29, an ether extraction method, while in Europe, it is tested under ISO 11085, an acid hydrolysis method. This difference in technique often leads to differences in fat values. The AOAC extraction method utilizes ether as the solvent and cannot extract any bound fat. Conversely, the ISO acid hydrolysis method can potentially extract non-lipid components, thereby inflating the fat values. In addition to the risk of inaccurate results from under- or over-extraction, both methods suffer from poor repeatability, caused by the wide and often changing variety of grains, protein sources, roughage, and other added minerals that comprise animal feeds.

Since the ORACLE™ was introduced in 2016, it has proven to be incredibly successful in accurately determining fat values in countless applications, without the need for calibration or method development. In several independent studies, the ORACLE has proven to be just as accurate and more precise than the chemical analysis methods for a broad range of applications. However, animal feed samples showed significantly lower results on the ORACLE, compared to reference chemistry methods. To determine the cause of this discrepancy between the two results, several different animal feed certified reference materials (CRM) were sourced and analyzed with the ORACLE, as well as both extraction methods listed above.

Current Issue

When analyzing animal feeds on the ORACLE, some samples had lower than expected fat values. Several hypotheses were considered, most of which centered on a comparison of the reference methods and rapid techniques. The initial hypothesis was that the extraction techniques were over extracting non-lipid components, similar to what has been observed with Whey Protein Concentrates (WPC).[1] Upon testing the CRM animal feed samples, it was observed that most of the samples produced results comparable to the reference method with excellent precision. Certain samples, however, had low results that were outside of the acceptable statistical range. Upon analysis of the NMR data, it was discovered that there was a rapidly relaxing fat signal that corresponded to the difference between the ORACLE result and the reference technique. To understand why this portion of the fat signal behaved differently, a detailed study of the composition of these feeds was performed.

One feature that stood out in samples with low ORACLE results was that they contained higher concentrations of paramagnetic metals in comparison to the other feed samples. These samples contained approximately 200 ppm or more of manganese, added as a nutritional supplement. Paramagnetic materials behave differently in a magnetic field, compared to diamagnetic materials. Diamagnetic atoms affect the magnetic field by bending the field slightly away from them, while paramagnetic atoms bend the magnetic field towards themselves. The presence of paramagnetic metals will affect the surrounding molecules by aligning the surrounding protons toward the paramagnetic atom. This results in a higher base

energy state, which in turn, shortens the relaxation time. Studies have shown that the increased presence of elements such as Mn(II), Gd(III) and Cr(III) can significantly reduce the T1 relaxation time, while other metals such as Fe(III), Ni(II) Cu(II) and Co(II) have minimal effects.[2]

Method and Results

Once the cause of the reduced relaxation time was discovered, a new "Animal Feed" tag was implemented in the ORACLE software. This tag increases the analysis window of what the ORACLE system will consider as fat. The increase in the analysis window allows the ORACLE to capture the rapidly relaxing portion of the fat signal, but this area also has some overlap with the relaxation signal of water. Due to the interference of water, all samples that are run with the "Animal Feed" tag must be completely moisture free. With the new feed tag, the ORACLE results of samples containing paramagnetic metals aligned well with the extraction technique results, as shown in **Table 1**.

Table 1. Animal Feed Tag Results

	Reference	ORACLE	Animal Feed Tag
Swine Feed	6.66-8.15	6.21	8.11
		6.29	8.12
		6.20	8.13
		6.23	8.15
	Average	6.23	8.13

Software Implementation

The remainder of this document describes the software implementation for this tag in both possible ORACLE analysis configurations: Fat only (stand-alone ORACLE), and Moisture and Fat (SMART 6™ & ORACLE™).

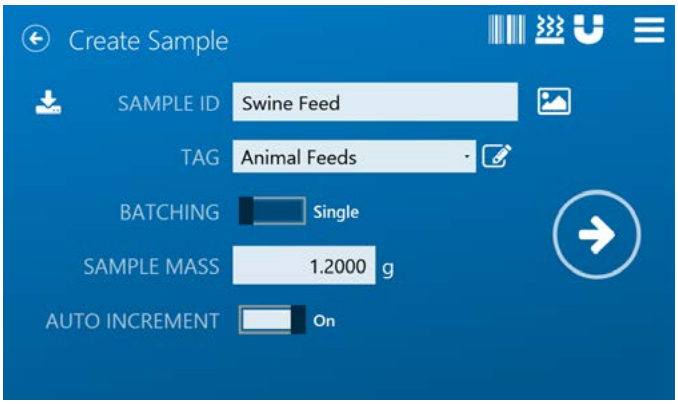
ORACLE: Fat Only Analysis

Implementation in stand-alone ORACLE systems is done through the use of a specific Tag, named "Animal Feed". Upon selection of this Tag in the Create Sample screen, a change in the time constant is applied to the ORACLE after calculation. The final displayed ORACLE result is the true fat result, taking into account any paramagnetic metals in the sample.

Step 1: Activate Tags

Select "Menu" → "Settings" → "Tags" →ON

Step 2: Select "Animal Feed" Tag on Create Sample Screen



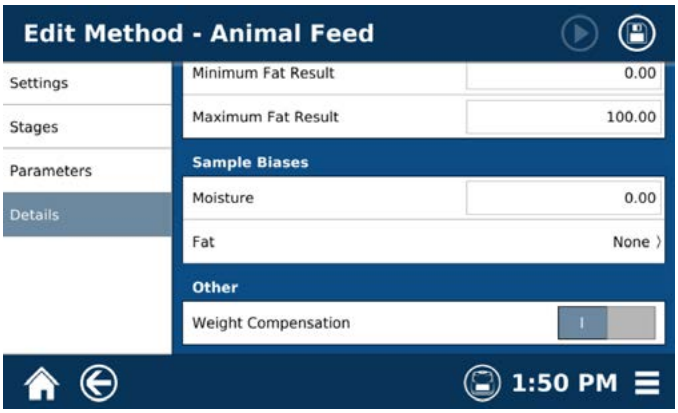
*__Note:__ *the "Animal Feed" Tag comes standard on every ORACLE with software version 1.9.0 and later. The time constant is embedded in the Tag and is not editable.*

SMART 6 & ORACLE: Moisture and Fat Analysis

Many customers analyzing Animal Feeds also require rapid moisture/solids for their starting materials and other products. The "Animal Feed" Tag is applicable on both the ORACLE and SMART 6. On the SMART 6 the Tag is called "Time Constant"

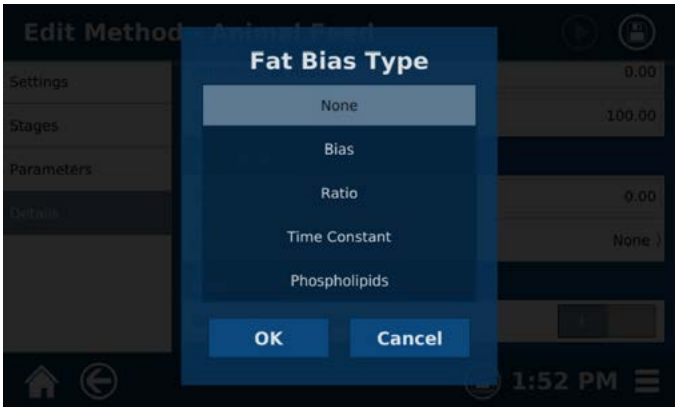
*All method parameters for the combination system (i.e. drying, cooling, QuikPrep™, biases are accessed through the SMART 6.

Step 1: Create/Edit Method



Enter method creation and press “Details” on the left side of the screen.

Step 2: Activate “Time Constant” Bias



Scroll to the “Sample Biases” and press “Fat”. Select “Time Constant” and Press “OK” and save method.

Conclusion

Animal feed samples present a unique challenge in universal fat analysis. As unique application-specific challenges present themselves, new and innovative solutions are required. With slight changes to the window of analysis, the ORACLE is able to capture the rapidly relaxing feature associated with high mineral content while still maintaining a universal, calibration-free approach to total fat analysis.

References

[1] Aued-Pimentel et al. *Quim. Nova*, **2010**, 33, 76–84.

[2] Barnhart et al. *Investigative Radiology*, **1986**, 21, 132-136.

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