Chapter 5

Recommended Laboratory Analytical Procedures for DDGS

Introduction

Laboratory analysis of feed ingredients is a common practice in the feed industry in order to verify that the ingredient meets guaranteed specifications, determine nutrient composition for use in animal feed formulation, and determine the presence and concentration of potential contaminants. Therefore, the accuracy of measurement of various chemical compounds in feed ingredients including DDGS is essential.

Analytical procedures can be categorized based on the level of validation of a specific laboratory method (Thiex, 2012). A single laboratory validation applies to a specific laboratory, technician, and equipment, whereas, a multi-laboratory validation involves validating a procedure in 2 to 7 laboratories to provide information on how well the results of a method are reproduced outside of the original laboratory. A full harmonized protocol collaborative study validation occurs when at least 8 laboratories provide acceptable data using the same procedure. An excellent summary of recommended analytical procedures for DDGS has been published by Thiex (2012) and key points are summarized in this chapter.

Recommended Procedures for Meeting DDGS Trading Standards (AFIA, 2007)

Moisture	**NFTA 2.2.2.5** Lab Dry Matter (105°C/3hr)
Crude protein	**AOAC 990.03** Protein (Crude) in Animal Feed **AOAC 2001.11** Protein (Crude) in Animal Feed and Pet Food Copper Catalyst
Crude fat	**AOAC 945.16** Oil in Cereal Adjuncts (Petroleum Ether)
Crude fiber	**AOAC 978.10** Fiber (Crude) in Animal Feed and Pet Food (F.G. Crucible)

Recommended Procedures for Nutrient Analysis of DDGS for Diet Formulation

Acid detergent fiber – **AOAC 973.18** Fiber, Acid Detergent, and Lignin, H_2SO_4 in Animal Feed and **ISO, 2008** are equivalent

Acid detergent lignin – **AOAC 973.18** Fiber, Acid Detergent, and Lignin, H_2SO_4 in Animal Feed and **ISO 13906:2008** are equivalent

Amylase-treated neutral detergent fiber – **AOAC 2002.04** Amylase Treated Neutral Detergent Fiber in Feeds and **ISO 16472:2006** are equivalent

Ash – **AOAC 942.05** and **ISO 5984:2002** are equivalent

Note: if the ash contains unoxidized carbon, the sample should be re-ashed

Trace minerals - Solubilization involves either dry ash followed by dissolving in acid, or wet ash using various acids depending on the elements being measured. Detection includes gravimetric techniques, visible spectrophotometry, flame and graphite furnace atomic absorption spectrophotometry (**AOAC 968.08; ISO 6869:2000**), or atomic mass spectroscopic detection (ICP-MS; **ISO 27085:2009**).

Sulfur – **AOAC 923.01** Sulfur in Plants and **ISO 27085:2009** are comparable

Phosphorus – **AOAC 965.17** Phosphorus in Animal Feed, Photometric Method, **ISO 6491:1998**

Determination of Total Phosphorus Content – Spectrophotometric Method, and **ISO 27085:2009** can be used

Selenium – **AOAC 996.16** Selenium in Feeds and Premixes, Fluorometric Method and **AOAC 996.17** Selenium in Feeds and Premixes, Continuous Hydride Generation Atomic Absorption Method are acceptable

Chlorine – **AOAC 969.10** Potentiometric Method, **AOAC 943.01** Volhard Method, and **ISO 6495:1999**

Chromium – No official methods. No methods have been validated

Fluorine – Microdiffusion technique (Mineral Tolerances of Animals, 2005). No methods have been validated.

Iodine – ICP-MS technique (Mineral Tolerances of Animals, 2005). No methods have been validated.

Amino acids – **AOAC 994.12** for all amino acids except tyrosine and tryptophan, **ISO 13903:2005**

Tryptophan – **AOAC 988.15**

Starch – No official method. AOAC 920.40 is no longer valid because of discontinued production of the enzyme needed for the assay, **AOAC 996.11** is most commonly used but has defects.

Recommended Procedures for Measuring Possible Contaminants in DDGS (Caupert et al., 2012)

Mycotoxins

See **Chapter 10** for recommended and GIPSA approved rapid mycotoxin testing kits.

Recommended instrumental methods for mycotoxin testing

Aflatoxins – AOAC 994.08

Deoxynivalenol – MacDonald et al. (2005a)
Fumonisins – AOAC 2001.04 and Rottinghaus et al. (1992)
T-2 – Romer Labs (2010)
Zearalenone – AOAC 994.01 and McDonald et al. (2005b)
Aflatoxins, Deoxnivalenol, Fumonisins, T-2, and Zearalenone - (Sulyok et al., 2007)

Antibiotic residues

FDA CVM has used a liquid chromatography and ion trap tandem mass spectrometry procedure (Heller, 2009) to determine 13 antibiotics in distillers grains including:

Ampicillin
Bacitracin A
Chloramphenicol
Chlortetracycline
Clarithromycin
Erythromycin
Monensin
Oxytetracycline
Penicillin G
Streptomycin
Tylosin
Virginiamycin M1

Extraction efficiency of this procedure ranged from 65% to 97% with quantitation limits from 0.1 to 1.0 µg/g. Accuracy ranged from 88 to 111% with coefficients of variation from 4 to 30%. The only FDA approved method for detecting virginamycin residues is a bioassay procedure Phibro (QA@Phibro.com), which is recommended over the LC-MS method of Heller (2009) which only measures one of the two subunits of virginamycin.

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