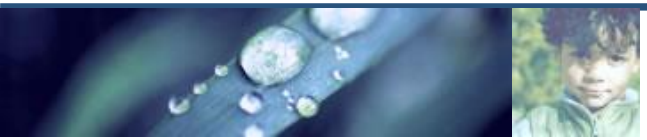


Crude Fat Methods in Corn Derived DDGS

AAFCO Meeting, August 2015

Agenda

DDG Market

DDG Working Group Method

Data

Conclusions

Source presentation EDR: 1-F-MK-PRE-0103614
Source modification date: 02/11/2011

AFIA Definition

Appendix 1. Draft – Updated AFIA Guidelines – DDG/S

****

DISTILLERS DRIED GRAINS WITH SOLUBLES (27.6)

PRODUCT DESCRIPTION

Distillers dried grains with Solubles (DDG/S) is a product obtained by condensing and drying the whole stillage remaining after the removal of ethanol by distillation from the yeast fermentation of grain or grain mixture by condensing and drying at least three-fourths of the solids of the resultant whole stillage by methods employed in the grain distilling industry. The whole stillage contains coarse material that is separated from the thin, watery portion by centrifuging or screening. The thin stillage fraction is concentrated by evaporation to a syrup (Condensed Distillers Solubles – CDS) and then added back to the coarse fraction and dried in rotary, flash, or steam-tube heated air dryers. The predominating grain must be declared as a first word in the name; i.e. Corn Distillers Dried Grains with Solubles (CDDG/S).

This product is made from a dry milling operation which makes potable, fuel, and / or other grades of ethanol.

TYPICAL ANALYSIS:

Moisture, %	8 - 12	Protein, %	25 - 28
Ether Extract, %	6 - 10	Crude Fiber, %	8 - 10
Ash, %	4 - 5		

Typical Specifications

Corn DDGs	Wheat DDGs
Minimum Crude Protein...27%	Minimum Crude Protein.....34%
Minimum Crude Fat.....8%	Minimum Crude Fat.....4%
Maximum Crude Fiber.....15%	Maximum Crude Fiber.....8.5%

Common Commercial Requirement = 35% Profat

No Commercial Requirements for Analytical Methods

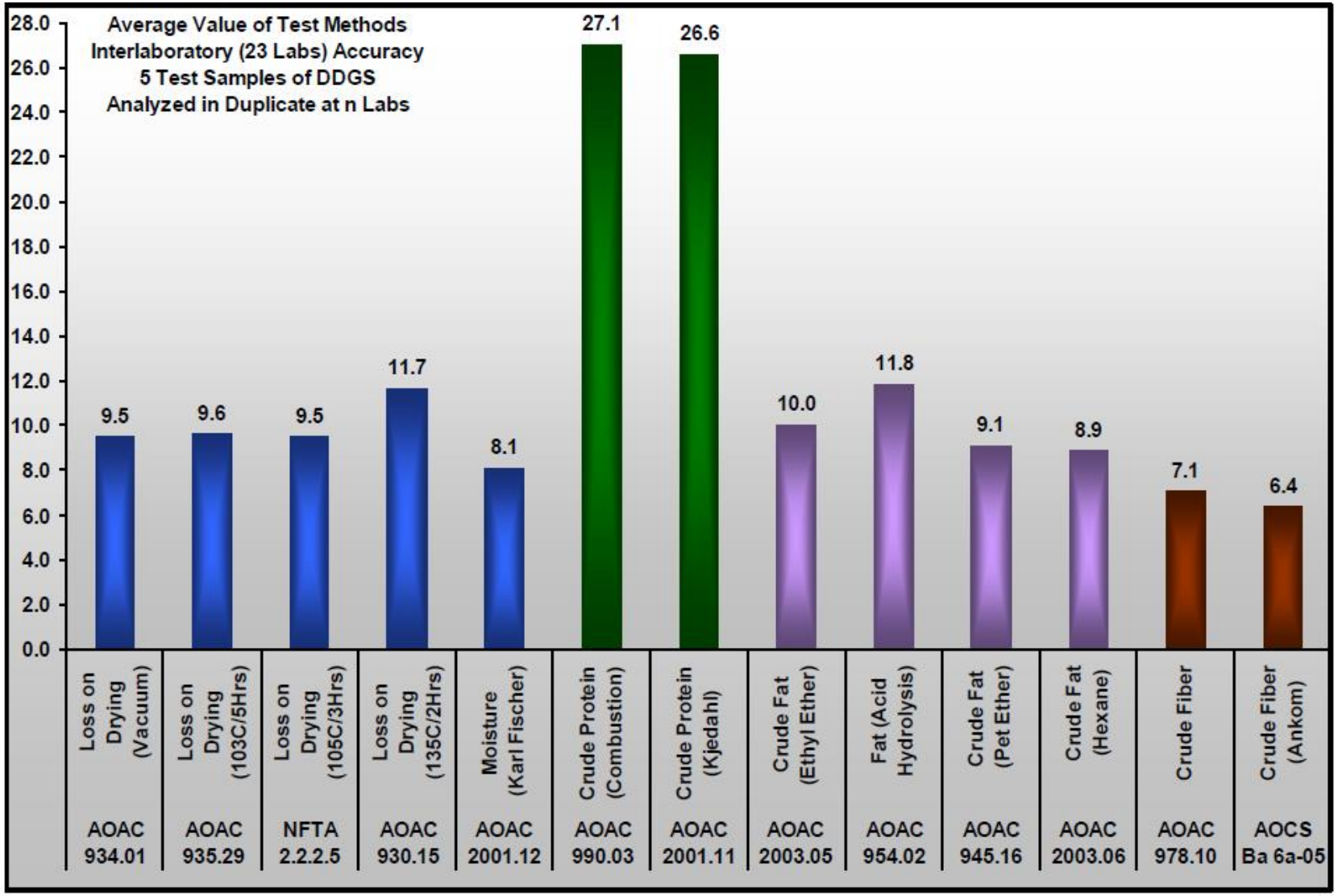
DTIWG 2006-2007

AMERICAN FEED INDUSTRY ASSOCIATION

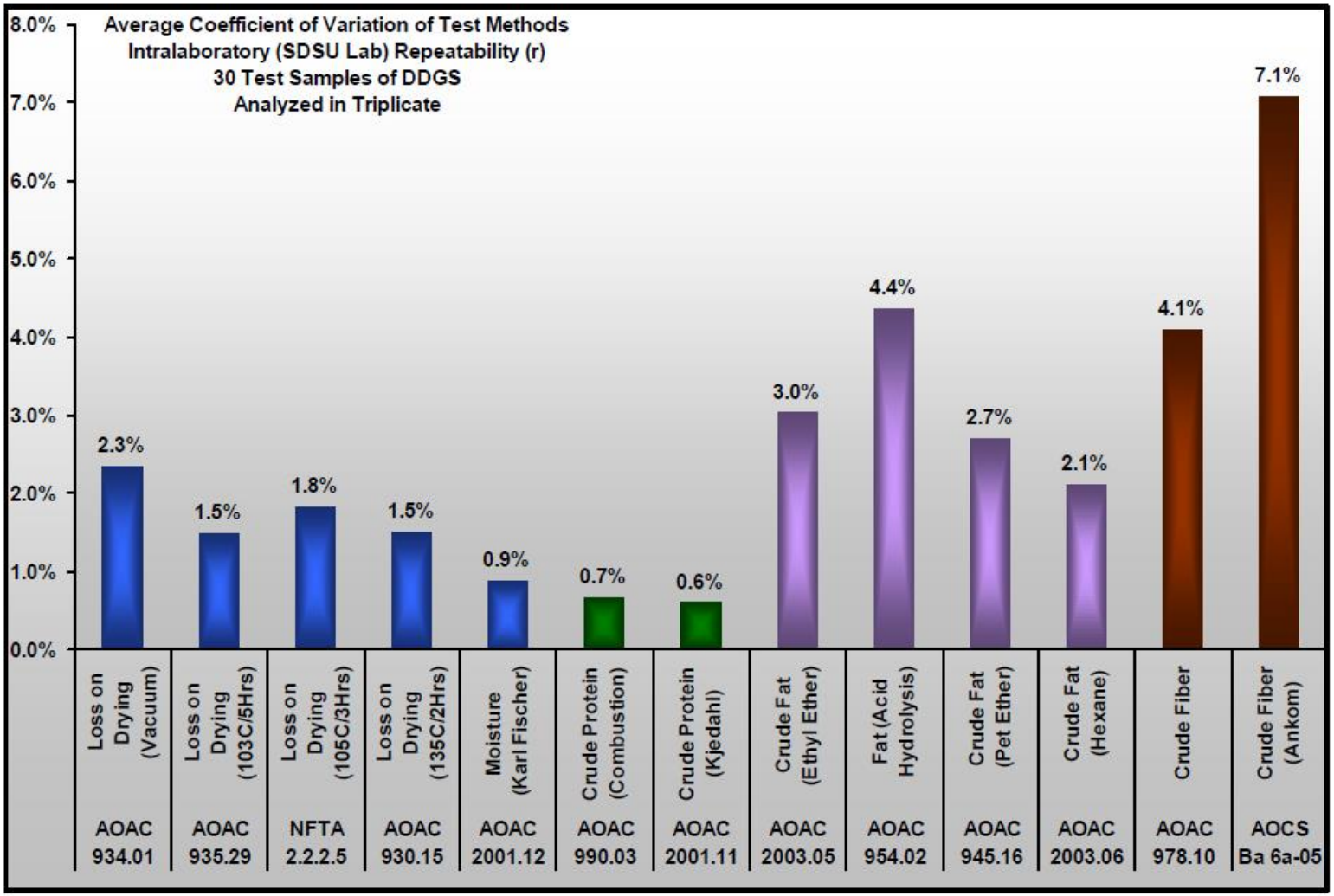
AFIA DDGS Analytical Methods Sub-Working Group (Operating under the *AFIA DDGS Technical Issues Working Group)*

Members: Shon Van Hulzen	Broin Management
Dr. Lance Forster	ADM
Charlie Staff	Distillers Grain Technology Council
Dr. Thomas Robb	Abengoa Bioenergy
Dr. Phil Smith	Tyson Foods, Inc.
Thomas Sliffe	Perten Instruments
Trace Yates	Tyson Foods
Mark Host	FOSS North America
Lars Reimann	Eurofins Scientific

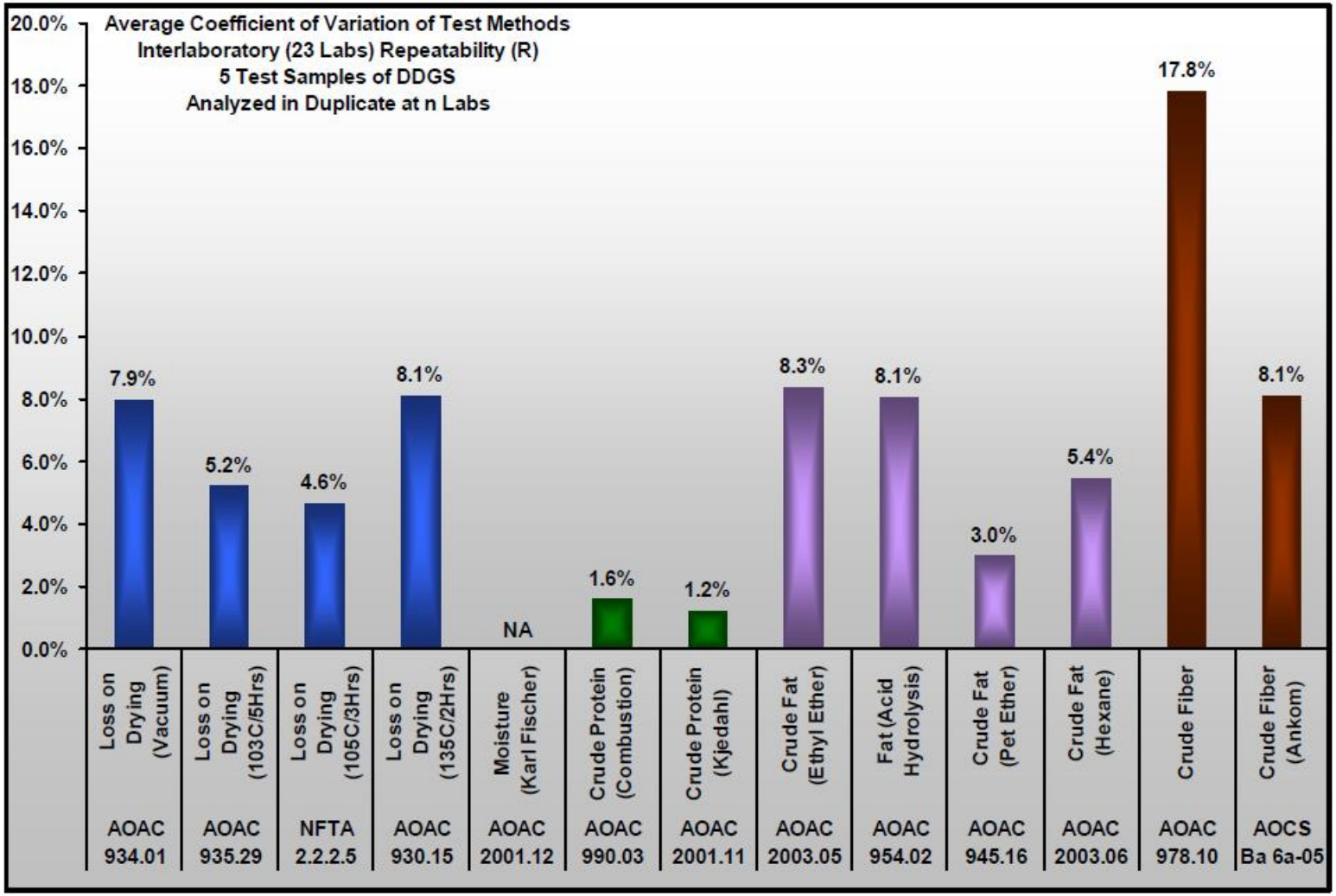
SDSU Study - Interlab Mean Comparisons



Intralaboratory Precision



Interlaboratory Precision



DITWG conclusion – Crude Fat

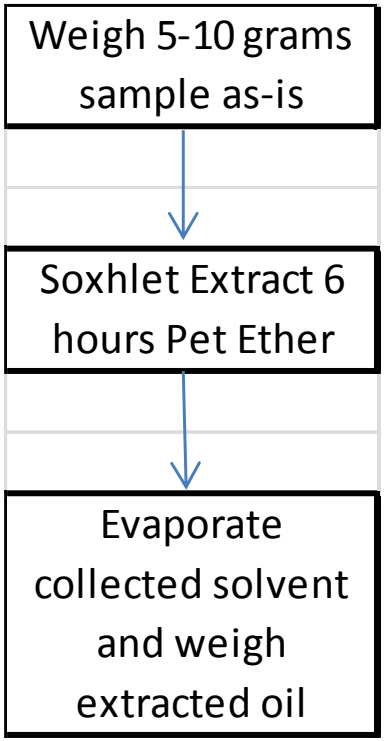
Results Summary Table			Intralaboratory (SDSU Lab) Results Summary				Interlaboratory (23 labs) Results Summary				
Method	Description	Units	StdDev	CV	Avg Value	Range	StdDev	CV	Avg Value	Range	n
AOAC 2003.05	Crude Fat (Ethyl Ether)	%	0.28	3.04%	9.22		0.84	8.34%	10.02		7
AOAC 954.02	Fat (Acid Hydrolysis)	%	0.57	4.37%	13.03		0.96	8.07%	11.84		9[b]
AOAC 945.16	Crude Fat (Pet Ether)	%	0.24	2.71%	8.85		0.27	2.95%	9.13		8[a]
AOAC 2003.06	Crude Fat (Hexane)	%	0.19	2.11%	9.00	4.18	0.48	5.45%	8.85	2.99	5

AFIA Working Group - Crude Fat

"The three non-hydrolysis fat methods (AOAC 2003.05, AOAC 945.16, and AOAC 2003.06) were determined to be statistically equivalent methods for the analysis of DDGS, however, in the interlaboratory portion of the study, **AOAC 945.16, *Oil in Cereal Adjuncts (Petroleum Ether)*, had a significantly lower coefficient of variation than the other non- hydrolysis methods and has thereby proven to be a more robust method in the analytical community, and is therefore chosen as the recommended test method for the analysis of fat in DDGS"**

Methods currently used in the commercial trade

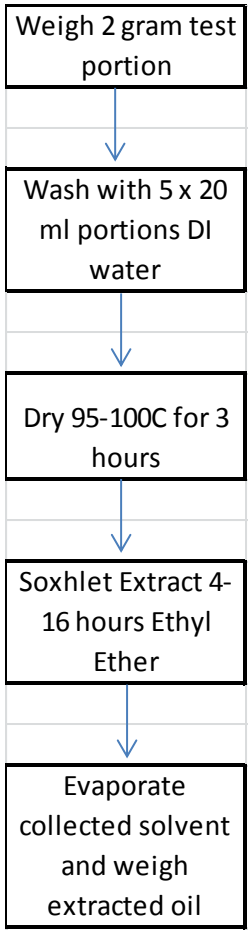
AOAC 945.16 – Pet Ether Soxhlet Extraction



Total processing time 8 – 9 hours

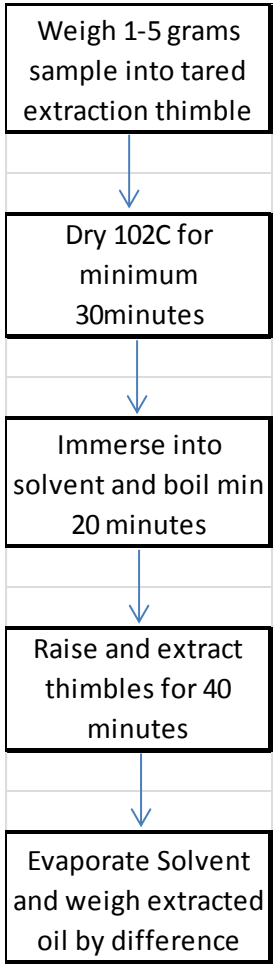
Methods currently used in the commercial trade

AOAC 920.39 – Ethyl Ether Soxhlet Extraction



Methods currently used in the commercial trade

AOAC 2003.06 – Hexane-Randall/SoxTec Submersion Extraction



AAFCO Interlab Results 2009

AAFCO Sample 200833	DDG	Corn Distillers Grain								
	Method Ref	AAFCO Method	# Labs	Pass 1 Mean	Pass 1 Stdev	Avg Range of Dups	# Labs	Pass 2 Mean	Pass 2 Stdev	Avg Range of Dups
Fat, Ethyl Ether, Direct	920.39	3.00	24	9.85	0.77	0.21	24	9.93	0.86	0.19
Fat, Ethyl Ether Indirect	920.39	3.01	1	9.84	0.19	0.27	1	9.84	0.19	0.27
Fat, Pet ether		3.06	23	9.52	0.28	0.08	22	9.53	0.28	0.07
Fat, Soxtec, Ethyl ether		3.09	25	10.33	0.81	0.12	24	10.29	0.79	0.10
Fat, Soxtec, Pet Ether		3.10	25	9.33	0.13	0.08	24	9.33	0.12	0.07
Fat, NIR		3.11	11	9.36	0.59	0.04	10	9.38	0.62	0.03
Fat, Hexane		3.12	3	9.65	0.23	0.18	3	9.65	0.23	0.18
Fat, Soxtec, Hexane		3.13	5	9.49	0.24	0.17	5	9.49	0.24	0.17
Fat, Ankom		3.14	11	9.82	0.36	0.12	10	9.75	0.29	0.09
Fat, Misc		3.99	10	9.80	0.57	0.37	9	9.85	0.50	0.22
Method			138	9.72	0.65	0.14	131	9.72	0.63	0.11

Inter-lab Precision on both ethyl ether methods significantly higher than Intra-lab precision

Ethyl ether extraction results remain higher in general but Soxtec / EE is much higher

Single Lab Comparison Ethyl Ether vs Pet ether 2015

Cal ID	AOAC 920.39 Ethyl Ether Extraction (%)	AOAC 945.16 Pet Ether Extraction (%)	Difference %	Relative Diff %
0713B029	**5.33**	**4.51**	**0.82**	**18%**
0713B030	**6.05**	**5.20**	**0.85**	**16%**
0713B031	**5.47**	**4.70**	**0.77**	**16%**
0713B032	**5.37**	**4.47**	**0.9**	**20%**
0713B033	**6.59**	**5.94**	**0.65**	**11%**
0713B034	**6.32**	**5.38**	**0.94**	**17%**
0713B035	**5.03**	**4.20**	**0.83**	**20%**
0713B036	**6.79**	**5.84**	**0.95**	**16%**
0609B019	**8.21**	**7.73**	**0.48**	**6%**
0609B017	**9.49**	**8.42**	**1.07**	**13%**

Single Lab Precision AOAC 945.16 Long Term

Analyte:	Fat		
CAL SOP:	M-4.1		
QC Sample ID:	DDG-7.2		
QC Sample desc	Dried Distillers Grain		
Expected value:	6.57	%	
Running stats: (used only for statistics & bias)			
Average:	6.60	StDev:	0.10
+1Stdev:	6.71	-1StDev:	6.50
+2StDev:	6.81	-2StDev:	6.39
+3StDev:	6.92	-3StDev:	6.29
# Data points	190	% RSD:	1.59%
Reporting units	%		
NOPA stats (used for determining in/out of spec)			
		Avg StDev:	0.10
+1Stdev:	6.68	-1StDev:	6.47
+2StDev:	6.78	-2StDev:	6.36
+3StDev:	6.89	-3StDev:	6.26
		Avg Bias	0.03

Single Lab Precision Short Term 920.39

AOAC 920.39

Variance of Pairs over range of DDG Fat values

Rep1	Rep2	Avg	Std	%RSD	Average RSD
5.06	5.01	5.035	0.035355	0.70%	
7.20	7.25	7.225	0.035355	0.49%	
8.29	8.13	8.21	0.113137	1.38%	
9.61	9.37	9.49	0.169706	1.79%	1.09%

Intra-lab comparison with other Fat Methods

Sample ID	Pet Ether	Ethyl Ether	AH -EE	by AH- GC
468-2015-0713B035	4.22	5.06	7.72	7.06
468-2015-0724B004	6.16	7.69	8.91	8.33
468-2015-0727B031	7.17	8.54	8.99	8.83
468-2015-0728B002	7.15	7.51	9.04	8.76

Fat in Crude extracts

So far inconsistent data

Observations on AOAC 920.39/945.16

- Ethyl ether is much more volatile, and tap water cooling of condensers must be 18-20 degrees C. In summer, requires chiller unit in some parts of the USA
- AOAC 920.39 requires 2-day completion as written
- 920.39 Higher bias appears to be generally higher than original SDSU study comparing Ethyl ether vs. Pet ether methods.
- 920.39 Intra-lab Precision appears to be improved over original SDSU study comparing Ethyl ether vs. Pet ether methods
- A proficiency study or ring trial is needed to produce updated Inter-lab data for these current methods

AOCS DDGS Proficiency series

Dear LPP Participant,

AOCS is excited to introduce a proficiency testing series for Distillers Dried Grains with Solubles (DDGS) from corn meal. The first year includes 6 samples (2 in September; 2 in December; 2 in March). The analytes of interest are: moisture, crude protein, crude fat, crude fiber, and ash (optional). Results are requested in duplicate for method validation purposes. Laboratories are encouraged to use the listed methods (see below) though alternate methods are acceptable provided a method reference is remitted.

Methods
Moisture: NFTA 2.2.2.5 (105 °C/3 hr)
Protein: AOAC 990.03 (Combustion) and AOAC 2001.11 (Kjeldahl, Cu Catalyst)
Crude Fat: AOAC 945.16, AOAC 2003.06, and AOAC 920.39
Crude Fiber: AOAC 978.10 and AOCS Ba 6a-05

Enrollment will open August 1, 2015, with a deadline of August 20, 2015. Sign up today!

AOCS DDGS PT Series

Benefits of a trade based Proficiency Series

1) Comparison between labs on recent methods
2) Restudy Precision in commercially practicing labs
3) Provide a vehicle for improved lab harmonization

Thank you!!

25 Years of Laboratory Excellence