

Eurofins Central Analytical Laboratories (ECAL)



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Testing topics - DDG products



- Why are results between laboratories important
- Overall Considerations
- Interlaboratory Studies
- Nutritional Quality: Moisture
- Nutritional Quality: Crude Protein
- Nutritional Quality: Crude Fat
- Mycotoxins: Aflatoxin, Don, Fumonisin

Why are Results between labs important?

- Commodities are bought and sold based on specifications verified by independent labs
- Seller may use one lab, Buyer uses another
- Lab representing foreign feed mills may still rely on kjeldahl methods for crude protein
- Often, multiple trades occur that may be risky to the buyers in the middle
- Domestic markets and foreign markets may have different method requirements
- Feed assay method vs ingredient assay methods may produce significantly different results

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- Mature Quality systems in place
 - GLP quality system (ISO17025 or equivalent practices)
 - Control charts updated daily and reviewed
 - ppK activities for improving repeatability
 - Active use of control samples verified by at least 3 labs

Accreditations and Programs

- ISO / IEC 17025:2017 Chemical Testing - Certificate No. 2993.01
- American Oil Chemist Society – Approved Chemist, Oilseeds, DDG, Meals, Oils
- Grain and Feed Trade Association (GAFTA) Member Analyst - Grain and Feeding stuffs
- Federation of Oils, Seeds, and Fats Associations (FOSFA) Member Analyst (D2) and Superintendent (C) - Oils and Fats
- American Association of Feed control Officials (AAFCO) – Participating Laboratory
- International Olive Council (COI) – Approved Laboratory, Chemical
- California Olive Oil Commission (COOC) – Approved Laboratory
- USDA – Approved Laboratory, Olive Oil Verification program
- USDA-AMS Laboratory Verification Program – Approved Laboratory – Export Meat
- Global Aquaculture Alliance – Approved Laboratory
- Japan Ministry of Health Agriculture, Welfare – Approved Laboratory
- FAPAS, Bipea, CFIA Proficiency Testing

AFIA - DDG Sub-Working Group 2007 recommendations

Test	Method	
Moisture/Loss on Drying	NFTA 2.2.2.5 (105C / 3 hours)	
Crude Fat	AOAC 945.16 (Pet ether)	
Crude Protein	AOAC 990.03 (Dumas)	
Crude Protein	AOAC 2001.11 (Kjeldahl/Copper)	

Interlaboratory Studies on Feed ingredients

- **AAFCO Proficiency Testing Program**

The Association of American Feed Control Officials (AAFCO), is an independent organization that provides ingredient definitions, label standards and laboratory guidance for state, federal and international feed regulators

Proficiency testing program available as well to private laboratories

All types of finished feeds and ingredients provided monthly for nutritional chemistry, and mycotoxins.

Several DDG samples have been selected in the program over the years

- Moisture common methods in feed labs
 - NFTA method, 105 °C - 3 hours
 - AOAC 935.29, 104 °C – 3 hours
 - AOAC 930.15, 135 °C – 2 hours
 - NIR

- Moisture Interlaboratory

AAFCO DDG 200833

Sample	Method	Meth short	# labs	Mean	SD
DDG 200833	AOAC 935.29	104C 3 hr	38	8.76	0.77
DDG 200833	AOAC 934.01	Vac 95C 5 hr	7	9.44	0.9
DDG 200833	AOAC 930.15	135C 2 hr	69	11.26	1.12
DDG 200833	NIR	NIR	9	8.40	0.67

- Crude protein – two primary methods
 - Dumas method (Combustion instruments manufactured by LECO and Elementar)
 - Kjeldahl method (Macro Kjeldahl and Kjeltec)
 - Methods offer very similar results but Kjeldahl method highly dependent on operator experience and proper QC controls
 - Many Labs have very good correlation between kjeldahl
 - AOCS

- Crude Protein Interlaboratory

AAFCO DDG 200833

Sample	Method	Meth short	# labs	Mean	SD
DDG 200833	AOAC 990.03	Dumas	119	26.88	0.37
DDG 200833	AOAC 976.05	Kjel-Foss	12	26.48	0.22
DDG 200833	NIR	NIR	6	25.97	0.65
DDG 200833	AOAC 988.05	Kjel - Cu	17	26.40	0.3

- Crude fat – several solvent extraction based methods
 - GAFTA method (pet ether)
 - AOCS Ba 3-38 (pet ether)
 - AOAC 920.39 (ethyl ether)
 - AOAC 954.02 (Acid hydrolysis)
 - NIR

- Crude fat Interlaboratory

AAFCO DDG 200833

Sample	Method	Meth short	# labs	Mean	SD
DDG 200833	Soxtec	Pet Ether	25	9.33	0.13
DDG 200833	Soxtec	Ethyl Ether	25	10.33	0.83
DDG 200833	AOAC 920.39	Ethyl Ether	24	9.84	0.77
DDG 200833	NIR	NIR	11	9.36	0.59
DDG 200833	Ankom	Ethyl Ether	11	9.82	0.36
DDG 200833	AOAC 954.02	AH-EthEther	36	11.11	0.76

Total Fat by AOAC Method 996.01

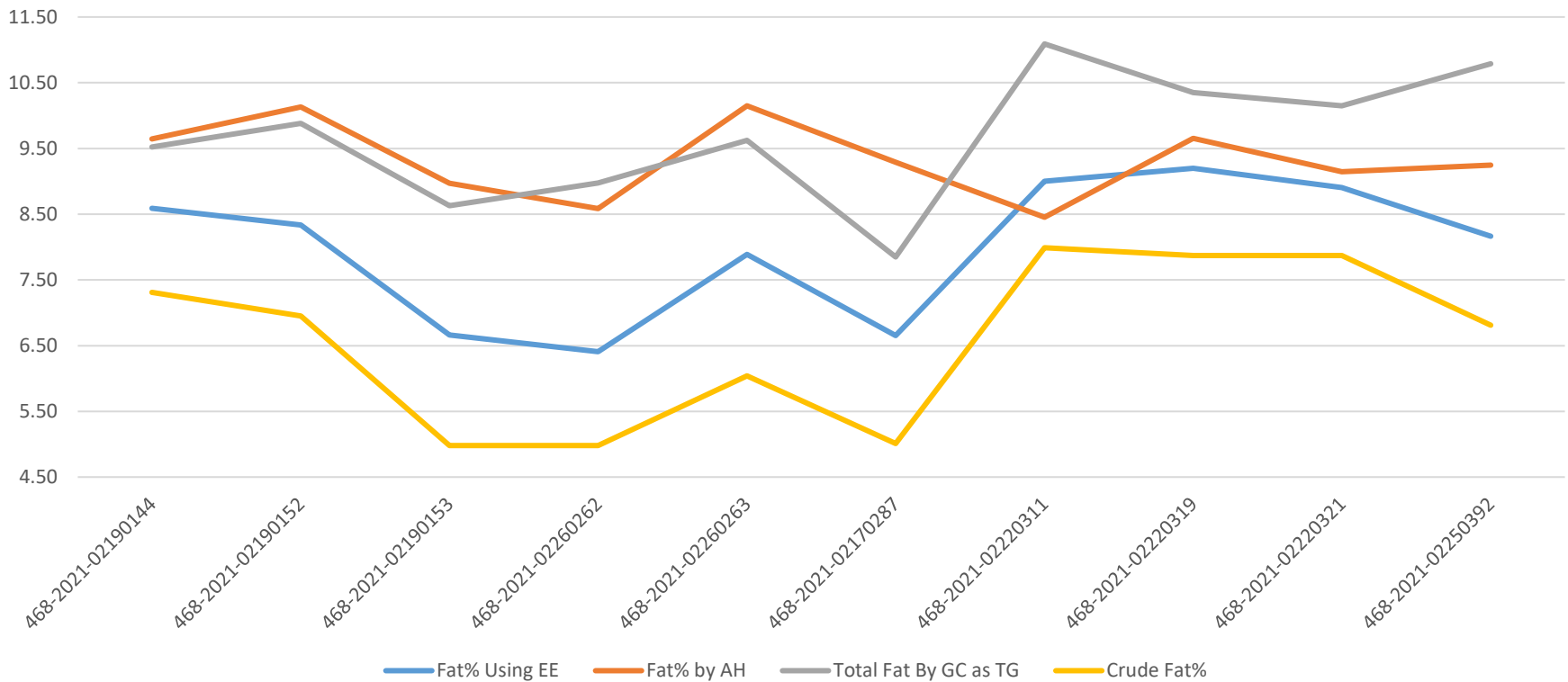
- Method based on GC analysis of fatty acids
- Aligned with FDA definition of fat for food NLEA labeling
- Mandated for label testing labs for assay of total fat, unsaturated, saturated fat profiles

- Crude fat – Inhouse study on 10 corn DDG

Sample ID	Crude Fat% GAFTA/AOCS	Fat% Using EE	Fat% by AH	Total Fat By GC as TG
468-2021-02190144	7.31	8.59	9.65	9.53
468-2021-02190152	6.95	8.34	10.13	9.88
468-2021-02190153	4.98	6.67	8.97	8.63
468-2021-02260262	4.98	6.41	8.59	8.98
468-2021-02260263	6.04	7.89	10.15	9.63
468-2021-02170287	5.01	6.66	9.29	7.85
468-2021-02220311	7.99	9.00	8.46	11.09
468-2021-02220319	7.87	9.20	9.66	10.35
468-2021-02220321	7.87	8.91	9.15	10.15
468-2021-02250392	6.81	8.17	9.25	10.79
average of all samples	6.58	7.98	9.33	9.69

- Crude fat – Inhouse study on corn DDG

Fat% By Various Methods of DDG



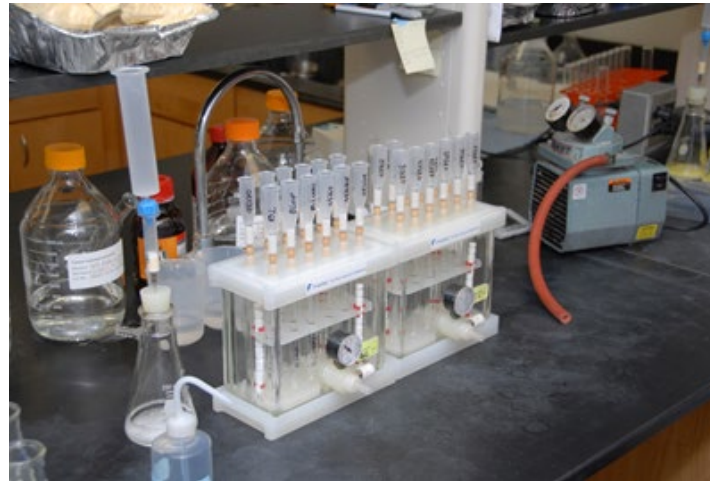
- Aflatoxin Common Methods
 - Neogen Veratox ELISA
 - R-Biopharm Ridasceen ELISA
 - HPLC with Fluorescence (LC-FLD)
 - LC-MSMS

- Aflatoxin Interlaboratory

- AAFCO DDG201963

Sample	Method	Meth short	# labs	Mean	SD
DDG 201963	Neogen Veratox	ELISA	11	25.3	9.6
DDG 201963	r-Biopharm Ridascreen	ELISA	7	13.5	4.6
DDG 201963	HPLC	LC-FLD	6	17.75	1.68
DDG 201963	LC-MSMS	LC-MSMS	3	14.5	5.4

- DON Common Methods
 - Neogen Veratox ELISA
 - R-Biopharm Ridascreen ELISA
 - HPLC
 - LC-MSMS



- DON Interlaboratory
 - AAFCO DDG201963

DDG 201963	Neogen Veratox	ELISA	8	9532	1951
DDG 201963	r-Biopharm Ridascreen	ELISA	9	11025	2128
DDG 201963	HPLC	LC-FLD	4	10343	1067
DDG 201963	LC-MSMS	LC-MSMS	12	10206	2120

- Fumonisin Common Methods
 - Neogen Veratox ELISA
 - R-Biopharm Ridascreen ELISA
 - HPLC
 - LC-MSMS



- Fumonisin Interlaboratory
 - AAFCO DDG201963

Sample	Method	Meth short	# labs	Mean	SD
DDG 201963	Neogen Veratox	ELISA	8	2091	688
DDG 201963	r-Biopharm Ridascreen	ELISA	9	2123	1036
DDG 201963	HPLC	LC-FLD	1	2326	
DDG 201963	LC-MSMS	LC-MSMS	6	1886	193

Final comments

- Results for methods for Crude fat may be significantly different depending on method chosen
- Acid Hydrolysis may be considered as a viable method for DDG as a feed ingredient
- Export trade contracts written on GAFTA rules may result in lower fat values than methods used domestically

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