MISC UPDATES

AVAILABLE RESOURCES – CURRENT AND FUTURE

AAFCO Perspective

LABORATORY CURRICULUM FRAMEWORK

Entry Level Courses

- LIMS
- Laboratory Sampling
- Method Resources
- Public Health (One Health)
- QMS
- Regulatory Programs
- Regulatory Sampling
- Safety
- Waste Management
- Accreditation / Certification
- Basic Communication Skills
- Basic Laboratory Math
- Basic Laboratory Statistics
- Basic Laboratory Techniques
- Chain of Custody
- Ethics
- ICS
- IFSS

LABORATORY CURRICULM FRAMEWORK

- Will be a great resource for AAFCO Labs
- Make great training for incoming lab personnel
- Opportunity to do reviewing. If you want to be a reviewer, contact Robyn.

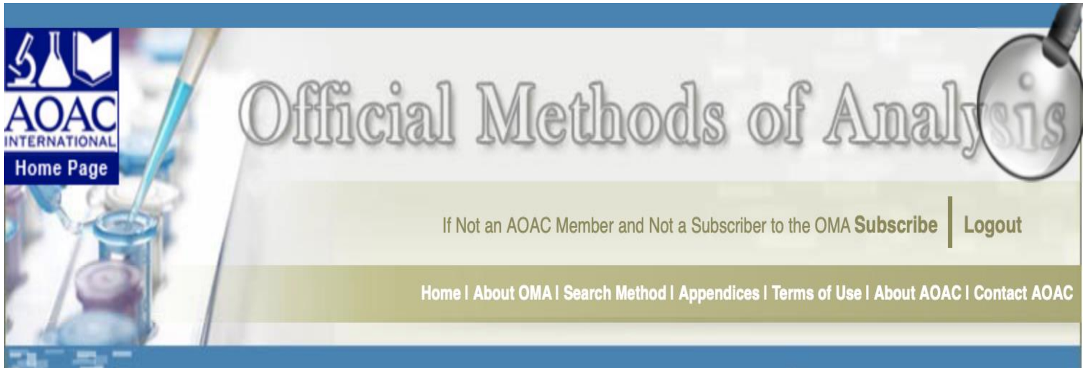
AOAC SUGARS ERP

AOAC First Action Methods – Sept. 2018

- *Fructan (Inulin, FOS, Levan, and Branched Fructan) in Animal Food (Animal Feed, Pet Food, and Ingredients*
  - This method is commercially available from Megazyme as the Fructan Assay Kit (Megazyme Cat. No. K-FRUC).

- *Sugar Profile by High Performance Anion Exchange Chromatography with Pulsed Amperometric Detection*

Fructan OMA Method 2018.07



Fructan (Inulin, FOS, Levan, and Branched Fructan) in Animal Food (Animal Feed, Pet Food, and Ingredients)

Kit Name	Fructan Assay Kit (Megazyme Cat. No. K-FRUC)
Company Info	Megazyme, Bray, Ireland
Analyte	Fructans
Analytical Technique	Fructan Assay Kit (Megazyme Cat. No. K-FRUC)
Equipment	Test Kit
Matrices	Animal Feed, Animal Feed/Swine Complete Feeds, Pet Foods, Animal Feed/Feed Ingredients
Approved By	AOAC
Method Number	2018.07

Access This Method

JOURNAL OF AOAC INTERNATIONAL
An International Journal of Analytical Science

Determination of Fructan (Inulin, FOS, Levan, and Branched Fructan) in Animal Food (Animal Feed, Pet Food, and Ingredients): Single-Laboratory Validation, First Action 2018.07

Authors: McCleary, Barry V.; Charmier, Lucie M.J.; McKie, Vincent A.; McLoughlin, Ciara; Rogowski, Artur
Source: Journal of AOAC International
DOI: https://doi.org/10.5740/jaoacint.18-0330

Traditional enzyme-based methods for measurement of fructan were designed to measure just inulin and branched-type (agave) fructans. The enzymes employed, namely exo-inulinase and endo-inulinase, give incompletely hydrolysis of levan. Levan hydrolysis requires a third enzyme, endo-levanase. This paper describes a method and commercial test kit (Megazyme Fructan Assay Kit) for the determination of all types of fructan (inulin, levan, and branched) in a variety of animal feeds and pet foods. The method has been validated in a single laboratory for analysis of pure inulin, agave fructan, levan, and a range of fructan containing samples. Quantification is based on complete hydrolysis of fructan to fructose and glucose by a mixture of exo-inulinase, endo-inulinase, and endo-levanase, followed by measurement of these sugars using the PAHBAH reducing sugar method which gives the same color response with fructose and glucose. Before hydrolysis of fructan, interfering sucrose and starch in the sample are specifically hydrolyzed and removed by borohydride reduction. The single-laboratory validation (SLV) outlined in this document was performed on commercially available inulin (Raftiline) and agave fructan (Frutafit®), levan purified from Timothy grass, two grass samples, a sample of legume hay, two animal feeds and two barley flours, one of which (Barley MAX®) was genetically enriched in fructan through plant breeding. Parameters examined during the validation included working range, target selectivity, recovery, LOD, LOQ, trueness (bias), precision (repeatability and intermediate precision), robustness, and stability. The method is robust, quick, and simple.

Affiliations: Megazyme, Bray Business Park, Southern Cross Rd, Bray, Ireland A98 YV29

Appeared or available online: January 15, 2019

Fructan Method Repeatability

Table 2018.07. Results of SLV for Fructan Assay Kit

	AOAC SMPR® 2018.002	Megazyme (K-FRUC)
Operating range (% w/w)	0.2 to 100	0.21 to 98.4[a]
Limit of quantitation (LOQ) (% w/w)	0.20	0.119[b]
RSD_r, % (0.2 to 1% w/w Fructan)	7	4.74
RSD_r, % (>1 to 10% w/w Fructan)	5	3.59
RSD_r, % (>10 to 100% w/w Fructan)	3	2.96
RSD_l, % (0.2 to 1% w/w Fructan)	14	8.47
RSD_l, % (>1 to 10% w/w Fructan)	10	6.36
RSD_l, % (>10 to 100% w/w Fructan)	6	5.77

[a] Precise range dictated by fructan content in samples tested.

[b] Based on replicate measurements for a sample with ~ 1% (w/w) fructan.

First Action AOAC Method 2018.16

SUGAR PROFILE BY HIGH PERFORMANCE ANION EXCHANGE CHROMATOGRAPHY WITH PULSED AMPEROMETRIC DETECTION

AOAC Method 2018.16

- Method manuscript is with copy editors
- Does not yet appear in OMA

Table 1. Method performance requirements in the AOAC SMPR 2018.001[1]

Analytes		Fructose, Glucose, Sucrose, Maltose, Lactose, and Galactose	
Analytical range, %*	0.1-5	>5-50	>50-100
Recovery, %	90% –110%	95% – 105%	97% – 103%
% RSD_r	≤ 7%	≤ 5%	≤ 3%
% RSD_R	≤ 10%	≤ 8%	≤ 4%
* Reported as the individual sugars (fructose, glucose, sucrose, maltose, lactose, and galactose)			

Summary of matrix categories, matrices, and samples used in the multi-lab validation study

Matrix Category	Matrix Material ID	AOAC food pyramid sector	Sample Type	Validation site
Food Products	Fat/Oil Palm oil	1	Innate, spikes	MSN
	Baking chocolate	2	Innate, spikes	MSN
	High fat Peanut butter	3	Innate, spikes	SGP, HAR
	Lunch meat Salami	4	Innate, spikes	BC
	Cereal NIST 3233	5	CRM	BC, HAR, MSN, SGP
	Lemon Juice	5	Innate, spikes	BC, HAR, MSN, SGP
	Artificial Foodstuff BCR-644	5	CRM	BC, HAR, SGP
	Vegetable Spinach	7	Innate, spikes	MSN
	Lunch meat Turkey	7	Innate, spikes	BC
	Lunch meat Ham	8	Innate, spikes	BC
	Meat Mince	9	Innate, spikes	HAR
	Seafood Tuna	9	Innate, spikes	BC
Infant Formula and Adult Nutritional	Infant formula NIST 1849a	6	CRM	HAR, MSN, SGP
Dietary Supplements	Gummy	5	Innate, spikes	MSN
	Tablet	5	Innate, spikes	MSN, HAR
	Premix	5	Innate, spikes	MSN
	Protein powder drink	6	Innate, spikes	MSN
Pet Food and Animal Feed	Horse feed	5	Innate, spikes	MSN
	Swine feed	5	Innate, spikes	MSN
	Milk replacement supplement	6	Innate, spikes	MSN
	Dry dog food	6	Innate, spikes	MSN
	Dry cat food	7	Innate, spikes	MSN, HAR
	Wet cat food	7	Innate, spikes	BC

Summary of CRM/SRM accuracy validation data

Matrix	Sample ID	# of labs	Reps	Analyte	RM Range (%)	RM Range (%)	Lab Overall Mean (%)	Individual Lab Average Range (%)
Cereal	NIST 3233	4	45	Fructose	0.81	0.42 - 1.20	0.727	0.694 -0.764
				Glucose	1.04	0.68 - 1.40	0.923	0.906 -0.938
				Sucrose	13.42	12.67 -14.17	13.6	13.2 - 13.9
				Maltose	0.46	0.37 - 0.55	0.443	0.415 -0.485
				Total Sugar	15.8	14.3 - 17.3	15.7	15.2 - 16.0
Artificial Foodstuff	BCR-644	3	27	Fructose	16.2	15.1 - 17.3	15.8	15.6 - 16.0
				Sucrose	10.81	10.56 -11.06	10.7	**10.5** – 10.8
				Lactose Monohydrate	15.85	15.56 -16.14	15.6	15.4 – 15.8
Infant formula/Drink	NIST 1849a	3	27	Lactose Monohydrate	47.6	42.1 - 53.1	49.5	48.0 – 50.7

Bold indicates individual result outside established range.

Summary of intermediate precision (%RSDi) and reproducibility (%RSDR) validation data

Matrix Material ID	# of labs	Total # or replicates	Analyte	%RSDi range (individual lab precision)	%RSDR
Cereal NIST 3233	4	45	Fructose	0.9 – 7.3	6.6
			Glucose	1.3 – 3.2	3.3
			Sucrose	2.0 – 4.1	3.5
			Maltose	1.5 - 7.1	7.6
			Total Sugar	1.7 – 4.0	3.4
Artificial Foodstuff BCR-644	3	30	Fructose	1.1 – 2.9	2.5
			Sucrose	1.9 – 2.9	2.6
			Lactose Monohydrate	1.9 – 3.3	2.8
Infant formula NIST 1849a	3	27	Lactose Monohydrate	1.8 – 6.0	4.5
Lemon juice	4	36	Glucose	1.2 – 3.2	2.7
			Fructose	0.9 – 4.0	3.3
			Total Sugar	1.8 – 2.4	2.2
Peanut butter	2	18	Sucrose	2.5 – 5.1	3.9
Tablet	2	18	Sucrose	2.5 – 2.6	2.6
			Maltose	3.0 – 6.7	5
			Total Sugar	2.5 (both labs)	2.6

Summary of intermediate precision (%RSDi) validation data

Matrix	# of labs	Total # or replicates	Analyte	%RSDi
Spinach	1	9	Glucose	2.9
			Sucrose	4.4
			Total Sugar	2.8
Baking chocolate	1	9	Sucrose	2.5
Ham	1	9	Glucose	2.3
			Sucrose	1.7
			Total Sugar	0.9
Salami	1	9	Glucose	4.5
			Sucrose	2.4
			Total Sugar	1.9
Turkey	1	9	Fructose	1.9
			Glucose	3.5
			Sucrose	1.1
			Maltose	2.5
			Total Sugar	0.8
Mince	1	9	Glucose	2.9
Gummy	1	9	Fructose	2.6
			Glucose	1.3
			Sucrose	1.3
			Maltose	1.2
			Total Sugar	1.3
Premix	1	8	Glucose	2.5
			Sucrose	2.2
			Maltose	1.7
			Total Sugar	2.4

Protein powder drink	1	9	Glucose	4
		9	Lactose	1.5
		8	Maltose	**7.2**
		9	Total Sugar	1.7
Dry Dog Food	1	9	Glucose	1.0
			Sucrose	1.5
			Fructose	1.2
			Total Sugar	1.7
Dry cat food	1	9	Sucrose	1.6
Wet Cat Food	1	9	Glucose	3.0
			Maltose	2.9
			Total Sugar	3.0
Horse Feed	1	9	Fructose	1.7
			Glucose	1.1
			Sucrose	7.0
			Maltose	4.8
			Total Sugar	1.6
Swine Feed	1	9	Fructose	3.0
			Glucose	2.0
			Sucrose	3.4
			Lactose	2.3
			Maltose	3.1
			Total Sugar	2.2
Milk Replacement Supplement	1	9	Galactose	0.7
			Glucose	1.9
			Lactose	5.8
			Maltose	5.6
			Total	

NEW! Two additional sugars methods under review

- Six Common Sugars by HPLC-MS
  - Interest in availability of a second technology
- Determination of Sugars in Animal Feed, Pet Food and Human Food Applying Ion Chromatograph with Pulsed Amperometric Detection (IC-PAD)
  - Interest in combining this with current First Action method for collaborative study
- A vote for both of these methods was delayed during the July 25 ERP meeting pending more data.

FDA, NADSA, AAFCO FOOD SAFETY IMPLEMENTATION FRAMEWORK

NASDA Model Animal Food Safety Implementation Framework

- Collaboration of FDA, NASDA, AAFCO
- Published in October 2018.
- https://www.nasda.org/foundation/food-safety-cooperative-agreements/animal-food-resources
- The Laboratory Services chapter starts on page 77.
- While written for the implementation of PCAF, the laboratory section is generic and the concepts could be used for implementing any laboratory initiative.

Checklists to prepare for PCAF Implementation

1) defining program needs
2) a gap analysis of current resources against the needs defined in the first phase.

The Elements for Initial Assessment

- Identify the regulations
- Identify products that will be collected
- Identify analyte(s) of concern, and the concentration of concern for each analyte
- Establish the required confidence level (maximum tolerable measurement error) to make a regulatory decision
- Identify tests/methods that are fit-for-purpose at the concentration of concern (achieves performance criteria within error tolerance), listing options for facility, equipment, and training requirements for each test/method
- Determine set up costs for each method, including ongoing costs for each method
- Determine the projected capacity requirements (e.g., projected numbers of samples per time period, monitoring capacity, and surge capacity).

Element's contd.

- Establish how inference will be made; determine the statistical requirements (replicates, etc.)
- Identify the quality requirements to meet program objectives
- Determine the physical and data storage requirements (legal mandates or QA needs)
- Define data capture and reporting requirements for generating reports. Consider data fields (e.g., quality data, final results, test methods, limit of detection, limit of quantitation, error or uncertainty, chain of custody). Evaluate the mechanism necessary for communicating and archiving data and results.
- Determine whether data will be shared with another agency. If so, determine if data meets the checklist published *Best Practices for Submission of Actionable Food and Feed Testing Data Generated in State and Local Laboratories*.

GAP Analysis

Evaluation and assessment of projected needs against current infrastructure and personnel resources.

- Laboratory facility assessment
  - Determine whether facility has adequate laboratory space and utilities to meet the needs.
  - Determine whether facility has adequate biological, radiological and/or chemical safety infrastructure to meet the needs.
  - Determine whether facility has adequate building security to meet the needs.
- Personnel and training
  - Determine whether organization has sufficient staff without hiring additional positions. If additional position(s) are needed, determine the required competencies.
  - Assess training needs for staff and format and accessibility of training. A model competency framework is under development.
- Equipment requirements
  - Assess if current equipment inventory meets requirements and if there is need for acquisition of new equipment.
  - Assess equipment maintenance needs (e.g., service contracts) and replacement cycle.

GAP Analysis Cont'd

Evaluation and assessment of projected needs against current infrastructure and personnel Quality requirements

- Assess whether the laboratory's Quality Management System meets the quality requirements identified by the program objectives.

- Data capture, reporting, and archiving requirements (Laboratory Information Management System)
  - Assess current reporting capability against projected program needs under consideration.
  - Evaluate the compatibility of database with program and other agency databases.
  - Assess security requirements, electronic communications, and storage
- Assess whether data, quality system, and reporting requirements meet the checklist published in the Data Acceptance White Paper.

OTHER STUFF

PFP Published

- Partnership for Food Protection Laboratory Science Workgroup's "Human and Animal Food Test Laboratories Best Practices Manual"
  - Published around the first of the year
  - Many of you worked on the document
  - FDA still has old version posted
  - PFP IFSS web site – link is broken
  - APHL has new Best Practices posted
    - https://www.aphl.org/programs/food_safety/APHL%20Documents/LBPM_Dec2018.pdf#search=pfp%20laboratory%20science%20best%20practices

Data Acceptance Document

- "Best Practices for Submission of Actionable Human and Animal Food Testing Data Generated in State and Local Laboratories"
  - Can be downloaded at: https://www.aphl.org/aboutAPHL/publications/Documents/FS-2019Jan-Best-Practices-Human-Animal-Food-Data.pdf.
- Drafted to facilitate interagency data sharing.