

Validation of Universal Fat Analysis Method using Certified Reference Materials



Abstract

Certified reference materials (CRMs) for food analysis are created and analyzed by trusted laboratories using known, validated test methods and offer insights into the inherent variation of a given analysis. They are considered a gold standard for comparative analysis and have safeguards in place to avoid method errors commonly encountered in a production testing environment. In this application note, we highlight the results of a blind comparison study, where a wide variety of certified reference materials were gathered, then tested in the ORACLE™. This study found that the ORACLE is capable of matching certified reference material values without any method development, calibration, or prior knowledge of the samples.

Introduction

The ORACLE universal fat analyzer is a low-resolution, time-domain NMR instrument (TD-NMR). However, unlike other TD-NMR instruments that rely on free induction decay (FID) or spin echo pulse sequences, the ORACLE uses a proprietary train of pulses, which not only provides sufficient resolution to separate the fat signal from the signal of other components, but it also normalizes fat response, so that any fat molecule, regardless of source, results in a uniform signal. Because of these two properties, the ORACLE is able to accurately measure fat content in any food sample with no calibration or method development.

As part of the ORACLE validation process, nearly 40 different certified reference materials (CRMs) were gathered and analyzed on the ORACLE.

CRMs were chosen for a variety of sample types and purchased from a variety of suppliers to show broad applicability. The study was conducted in a blind fashion where the operator was not given any information on the sample being analyzed.

Method and Results

To evaluate the performance of the ORACLE system, a wide variety of CRMs were gathered and analyzed. It is important to note that for the ORACLE to provide the most accurate results possible, the sample must have sufficiently low water content (less than 10%) and must be measured at a constant temperature that is high enough for all fat molecules to be in a liquid state. For this study, a fat analysis temperature of 44.2 °C was selected. All samples were pre-dried in an air oven at 100 °C overnight to drive off any free moisture. Once the pre-dry was complete, all samples were placed in a temperature conditioning block for 45 minutes to bring the sample up to 44.2 °C, which is the same temperature at which the ORACLE magnet was set. Each sample was prepared in five separate replicates, which were each measured one time. The results, shown in **Table 1** (page 2), were compared to the CRM certified values. All ORACLE results fell within the margin of measurement error for each sample.

Table 1. Percent Moisture Analysis of CRMs

Product Type	CRM ID	Source	Target %F	=/-	Target Min	Target Max	ORACLE %F	ORACLE STDEV
Whole Wheat	703WM07011	CAIQ - China	0.92	0.52	0.40	1.44	0.59	0.02
Wheat Flour	ERM-BC382	LGC	1.39	0.17	1.22	1.56	1.33	0.08
UHT Milk		Muva Kempten	1.71	0.01	1.70	1.72	1.71	0.01
Yogurt	YO-1415	Muva Kempten	1.87	0.05	1.82	1.92	1.91	0.03
Grain Feeds Stuff	703F006011	CAIQ - China	3.34	0.79	2.55	4.13	2.85	0.03
Yogurt		Muva Kempten	3.80	0.05	3.75	3.85	3.82	0.02
Fresh Cheese		Muva Kempten	3.84	0.26	3.58	4.10	3.85	0.02
Poultry Feed	LGC7173	LGC	4.10	0.70	3.40	4.80	3.72	0.02
Processed Meat	PT-MT-733	LGC	11.57	0.44	11.13	12.01	11.62	0.06
Fresh Cheese	FK-1222	Muva Kempten	13.82	0.14	13.68	13.96	13.83	0.01
Processed Cheese		Muva Kempten	14.58	0.18	14.40	14.76	14.52	0.03
Meat Homogenate	NIST 1546a	NIST	18.96	0.40	18.56	19.36	18.83	0.75
Process Cheese	SK-0315	Muva Kempten	19.49	0.17	19.32	19.66	19.55	0.07
Boiled Sausage	BR-1001	Muva Kempten	19.75	0.36	19.39	20.11	19.68	0.07
Sweet Digestive Biscuit	LGC7103	LGC	21.17	0.45	20.72	21.62	21.38	0.02
Fresh Cheese (Lact. Red.)		Muva Kempten	24.19	0.38	23.81	24.57	24.30	0.09
Parmesan Cheese		Muva Kempten	24.98	0.12	24.86	25.10	25.03	0.04
Milk Powder		Actalia	26.12	0.20	25.92	26.32	25.92	0.01
Whole Milk Powder	MP-0210	Muva Kempten	26.14	0.11	26.03	26.25	26.21	0.04
Milk Powder	MP-0213	Muva Kempten	26.73	0.09	26.64	26.82	26.72	0.03
WMP	MP-0215	Muva Kempten	26.79	0.08	26.71	26.87	26.71	0.08
Infant Formula	703IPO6092	CAIQ - China	28.12	0.52	27.60	28.64	27.84	0.00
Process Cheese		Actalia	28.22	0.21	28.01	28.43	28.23	0.04
Powdered Infant Formula	NIST 1849a	NIST	30.43	0.95	29.48	31.38	30.31	0.02
Milk Chocolate	S-0815	Muva Kempten	39.98	0.42	39.56	40.40	40.13	0.11
Cream Powder	MP-0211	Muva Kempten	43.39	0.15	43.24	43.54	43.28	0.04
Baking Chocolate	NIST 2384	NIST	51.40	1.10	50.30	52.50	51.56	0.10
Peanut Butter	NIST 2387	NIST	51.60	1.40	50.20	53.00	50.58	0.18
Salted Butter	Muva 1308	Muva Kempten	82.00	0.78	81.22	82.78	81.91	0.52
Butter	Muva 1306	Muva Kempten	82.43	0.12	82.31	82.55	82.46	0.33
Butter	Muva 1309	Muva Kempten	82.62	0.82	81.80	83.44	82.75	0.06
Equine Feed	#201727	AAFCO	8.23	0.62	7.61	8.85	8.13	0.04
Swine Feed	#201730	AAFCO	6.64	0.64	6.00	7.28	6.41	0.28
Lamb Feed	#201731	AAFCO	2.56	1.18	1.38	3.74	1.44	0.03
Dairy-Beef Feed	#201728	AAFCO	1.54	0.80	0.74	2.34	0.97	0.12
Pheasant and Turkey	#201729	AAFCO	2.85	0.54	2.31	3.39	2.83	0.07

Conclusion

This study proves the ORACLE universal fat analyzer is capable of measuring total fat in a wide variety of samples, without the need for calibration, method development or biases. The accuracy and precision of the ORACLE matches the results of the reference chemistry, even when samples are run in a blind study. As such, the ORACLE is well-suited for not only for food production settings, but even contract labs and R&D sites where a wide variety of ingredients, in-process products and finished goods need to be accurately analyzed.

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