FOOD COMPOSITION AND ADDITIVES

Rapid Determination of Moisture and Fat in Meats by Microwave and Nuclear Magnetic Resonance Analysis

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Abstract

A peer-verified method is presented for the determination of percent moisture and fat in meat products by microwave drying and nuclear magnetic resonance (NMR) analysis. The method involves determining the moisture content of meat samples by microwave drying and using the dried sample to determine the fat content by NMR analysis. Both the submitting and peer laboratories analyzed 5 meat products by using the CEM SMART system (moisture) and the SMART Trac (fat). The samples, which represented a range of products that meat processors deal with daily in plant operations, included the following: (*1*) fresh ground beef, high-fat; (*2*) deboned chicken with skin; (*3*) fresh pork, low-fat; (*4*) all-beef hot dogs; and (*5*) National Institute of Standards and Technology Standard Reference Material. The results were compared with moisture and fat values derived from AOAC-approved methods, **950.46** (Forced Air Oven Drying) and **960.39** (Soxhlet Ether Extraction).

1 Summary of Results of Verification Study

1.1 Matrixes

A range of meats, including fresh meats, preblends/emulsions, and cooked meats, were analyzed for moisture and fat content.

1.2 Number of Samples

AOAC methods for moisture analysis (**950.46**) and fat analysis (**960.39**) were run 10 times for each of the 5 products by the peer laboratory. The submitting and peer laboratories independently analyzed 5 products (10 times each) for both moisture and fat, using a SMART system (microwave drying system manufactured by CEM Corp.) and SMART Trac (NMR system manufactured by CEM Corp.), respectively.

2 Safety Precautions

It is recommended that persons with heart pacemakers or other magnetically sensitive devices do not approach within 11 inches (0.3 m) of the SMART Trac magnet component. Certain heart pacemakers or other magnetically sensitive prosthetic devices may be affected by magnetic fields as low as 0.5 mT.

3 Scope

This method uses a microwave drying procedure and a rapid NMR procedure for the determination of moisture and fat, respectively, in meat products. These tests cover a variety of meat products and a wide range of moisture and fat levels.

4 References

(1) *Official Methods of Analysis* (2000) 17th Ed., AOAC INTERNATIONAL, Gaithersburg, MD
(2) Youden, W.J., & Steiner, E.H. (1975) *Statistical Manual of the AOAC*, AOAC, Arlington, VA

5 Abbreviations

(**5.1**) *NMR*.—Nuclear magnetic resonance.
(**5.2**) *RF*.—Radio frequency.
(**5.3**) *TAM*.—Texas A&M University.
(**5.4**) *NIR*.—Near infrared.
(**5.5**) *LR–NMR*.—Low-resolution time-domain NMR.

6 Principle

NMR, discovered in the middle of the last century, is based on the observation that certain nuclei will absorb and re-emit radio frequency (RF) energy over a narrow band of frequencies when placed in a static magnetic field. The frequency at which the NMR effect occurs for a given nuclear isotope is dependent on the strength of the magnetic field of the magnet, and the phenomenon is caused by the interaction between the nuclear magnetic dipole of a nucleus and the magnetic field it experiences. (The latter is the reason that the word "nuclear" is included in the description of the phenomenon. NMR does not involve the emission of ionizing radiation.)

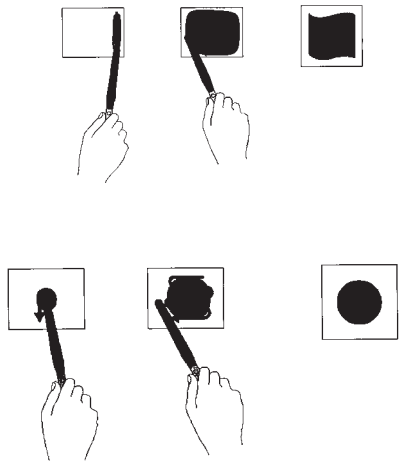

Figure 1. Two examples of how to spread the meat sample evenly. (Top) If the sample is in a paste, semisolid, or crumb form or a raw or skeletal meat product such as fresh pork, ground beef, or chicken, place the sample on the end of a spatula, and spread it across one end of the pad. Then spread the sample to a uniform thickness covering approximately 90% of the pad surface area. (Bottom) If the sample contains bound water, such as an all-meat emulsion, cooked all-meat sausage, sausage with extenders, semidry sausage, or ham, place the sample on the end of the spatula and apply the sample to the middle of the pad. Then spread the sample around the pad in a circle.

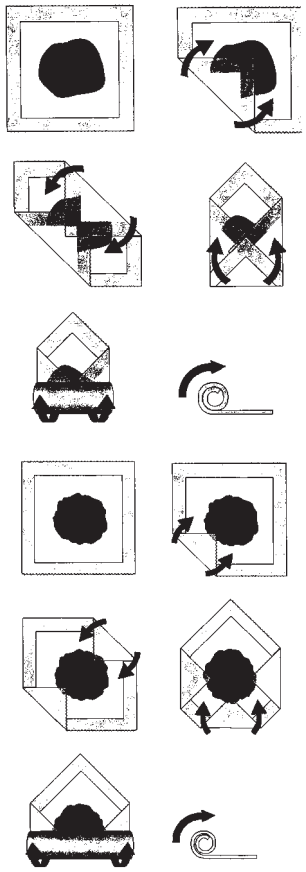

Figure 2. Two examples of how to roll pads in Trac Film. (Top) Place the 2 square pads and dried sample in the center of the Trac film. Fold the left corner of the film and pads as illustrated. Fold the right corner. Pull the lower edge of the film and sample pads toward the top, and begin to roll them into a tube. (Bottom) For samples that are rigid after being dried and more difficult to roll into a cylinder, prepare the pads as illustrated above.

Although many nuclei can be made to generate an NMR signal, the overwhelming majority of NMR experiments involve the excitation and detection of signals from the ^{1}H nucleus; this branch of the science is commonly known as "proton NMR." NMR has been widely used as the basis of an analytical spectroscopy technique (NMR spectroscopy) for several decades and is also the basis of magnetic resonance imaging (MRI), which has been used as a clinical diagnostic tool for nearly 20 years.

The NMR technique incorporated into the SMART Trac system is based on low-resolution time-domain NMR (often called LR–NMR). This is a small "offshoot" of NMR spectroscopy that has also been used for >20 years for industrial quality control. The vast majority of LR–NMR is proton NMR. The main difference between LR–NMR and NMR spectroscopy is in the effects used for discriminating between different hydrogen-containing constituents of a sample.

In NMR spectroscopy, these constituents are distinguished by small variations in the magnetic field that ^{1}H nuclei experience in different molecules and different parts of the same molecule. These variations are caused by differences in the electronic structures of molecules and lead to small differences in the NMR frequencies of ^{1}H nuclei in different molecules that can be used to discriminate between the different constituents within the sample. This phenomenon is known as the chemical shift effect.

In LR–NMR, it is not possible to detect chemical shift effects in samples containing ^{1}H nuclei because of the low field strength and homogeneity of the magnet used to generate the static magnetic field. Instead, differences in the rate of decay of the signal from different constituents (commonly known as transverse relaxation or T_2 decay) are used to distinguish between NMR signals from different constituents within the sample. Transverse relaxation can generally be approximated as an exponential decay with the time constant T_2.

In food that has undergone microwave drying, the main constituents that contain significant amounts of protons are fat, protein, and carbohydrate. There are significant differences between the proton transverse relaxation times (T_2) of these constituents. In particular, both protein and carbohydrate in dried foods exhibit "solid-like" behavior and have transverse relaxation times that are very short (typically on the order of ≤10 μs), and the signal from these substances decays very rapidly. However, the transverse relaxation times for fat

Table 1. Results from analyses of high-fat, fresh ground beef[a]

	AOAC results from TAM				SMART Trac results[b] from CEM			SMART Trac results[b] from TAM		
	Method **950.46**		Method **960.39**			Microwave	NMR		Microwave	NMR
Sample ID	Wt, g	M, %	Wt, g	F, %	Wt, g	M, %	F, %	Wt, g	M, %	F, %
1	4.9750	40.44	3.6150	46.03	3.6893	40.19	46.34	3.3149	40.69	45.68
2	3.4373	40.42	3.7455	45.86	3.5562	40.13	46.15	3.8042	40.23	46.25
3	3.7334	40.35	3.8465	45.83	4.1158	40.26	46.08	35.233	40.18	46.27
4	3.6018	40.26	3.3339	45.54	3.9324	40.20	46.25	3.5591	40.68	45.93
5	3.6140	40.49	3.3647	45.92	4.0714	40.19	46.19	3.9022	40.24	46.24
6	4.2714	40.34	3.6816	45.76	3.8083	40.35	45.99	3.7258	40.32	46.40
7	3.3022	40.41	3.8443	45.64	3.7083	40.44	45.59	3.2478	40.42	45.94
8	3.4890	40.44	2.2901	45.92	3.4759	40.33	45.65	2.9598	40.39	45.92
9	3.2051	40.30	2.2819	45.93	3.4957	40.36	45.50	3.3260	40.42	45.85
10	3.3612	40.45	2.2736	45.99	3.6916	40.32	45.87	3.4150	40.59	45.54
Mean		40.39		45.84		40.28	45.96		40.42	46.00
SD[c]		0.074		0.157		0.098	0.295		0.184	0.280

[a] M = Moisture; F = fat.
[b] SMART system for moisture and SMART Trac system for fat.
[c] SD = Standard deviation.

are considerably longer (typically on the order of ≥10 ms), and thus the signal decays relatively slowly. In addition, any very small amounts of residual moisture that remain after microwave drying of the sample are associated with the nonlipid molecules within it (i.e., protein and carbohydrate) and also exhibit "solid-like" behavior. Thus, it is possible to discriminate between the fat and the other principal constituents of a dried food by exciting the system, waiting for the "solid-like" signals to decay, and then acquiring the remaining signal which, in the absence of moisture, is predominantly from protons contained in fat within the sample.

The intensity of an NMR signal acquired from a dried food sample by using the methodology described above will be directly proportional to the number of protons within the fat contained in the sample and, for many samples, directly proportional to the fat content of the sample.

Low-resolution NMR techniques based on methodologies similar to that described above are used widely for quality control in a number of industries, and some of these methods have been approved by international standards organizations. For example, see American Society for Testing and Materials (ASTM) D3701 and D4808 for determination of the hydrogen content of various petroleum products and International Standards Organization (ISO) 10565 for determination of oil and moisture in seeds.

7 Supplies

(**7.1**) *Glass fiber pads.*—CEM Corp. (Matthews, NC) equivalent.

(**7.2**) *Trac film.*—CEM Corp. or equivalent.

8 Apparatus and Equipment

(**8.1**) *Microwave moisture analyzer.*—Sensitivity of 0.2 mg water, moisture range of 0.01–99.99% in liquids, solids, and slurries; 0.01% resolution. Includes automatic electronic balance (0.1 mg readability), microwave drying system with temperature feedback, and microprocessor computer control (CEM Corp.); or equivalent.

(**8.2**) *NMR–RF pulse generator.*—Pulse power, 250 W nominal; pulse times, variable in 100 ns increments; transmit and receive phases, selectable 0, 90, 180, and 270°; nominal 90° pulse times, 4 μs (18 mm probe). Magnet: permanent, thermally stabilized, 0.47 T (20 MHz), and homogeneity better than 10 ppm. Signal detection: dual-channel (quadrature) detection with programmable low-pass filtering, programmable data acquisition rate up to 4 MHz per pair of points (CEM Corp.); or equivalent.

9 Sample Preparation

To prevent water loss during preparation and subsequent handling, do not use small samples. Keep ground material in glass or similar containers with air- and watertight covers. Samples were prepared for analysis as follows:

Approximately 5 lbs of freshly processed product from each predetermined category was collected from local and national meat processing plants and stored at ≤4°C for ≤3 days.

Samples of nonground meat products were diced into approximately 2 in. (5.08 cm) cubes and passed rapidly 2 times through a Hobart grinder (Model 4612; Troy, OH) equipped with a 3/16 in. (0.1875 in. or 0.4763 cm) plate.

Table 2. Results from analyses of fresh chicken with skin[a]

	AOAC results from TAM				SMART Trac results[b] from CEM			SMART Trac results[b] from TAM		
	Method **950.46**		Method **960.39**			Microwave	NMR		Microwave	NMR
Sample ID	Wt, g	M, %	Wt, g	F, %	Wt, g	M, %	F, %	Wt, g	M, %	F, %
1	5.8888	74.52	3.3288	7.36	4.0309	74.55	7.25	2.7348	74.28	7.29
2	5.9199	74.56	3.4672	7.50	3.4891	74.51	7.34	3.4454	74.23	7.14
3	6.6851	74.59	3.5339	7.02	3.7672	74.66	7.33	2.6815	74.38	7.22
4	5.9594	74.55	3.6244	7.49	3.4990	74.56	7.32	3.2946	74.18	7.21
5	6.2424	74.52	3.4877	7.08	3.8961	74.53	7.29	3.0128	74.32	7.08
6	6.5156	74.60	3.7604	7.06	3.7438	74.42	7.26	3.6523	74.42	7.16
7	6.6359	74.60	3.3748	7.16	3.7383	74.65	7.24	3.3548	74.55	7.07
8	6.2113	74.58	3.5340	7.08	3.7661	74.25	7.25	4.1013	74.40	7.10
9	6.4292	74.64	3.6059	7.41	3.8905	74.25	7.30	2.8425	74.41	7.07
10	6.4023	74.56	4.2455	7.20	4.1224	74.31	7.27	3.3257	74.54	7.11
Mean		74.57		7.24		74.48	7.29		74.37	7.15
SD[c]		0.038		0.186		0.162	0.036		0.121	0.074

[a] M = Moisture; F = fat.
[b] SMART system for moisture and SMART Trac system for fat.
[c] SD = Standard deviation.

Ground meat products were then homogenized in a Robot Coupe bowl chopper (Model R6) to a paste or pâté consistency. Ground material was placed in a chilled bowl chopper (4°C) and chopped for 30 s; then the inner side wall and bottom of the bowl were wiped down with a spatula (plastic or rubber spatula with ca 2 × 4 in. straight-edge blade), and the gathered material was transferred to the body of the test sample. The process was repeated for an additional 30 s.

After homogenization, eight 4 oz (118 g) samples were collected and stored in plastic containers with screw-cap lids. Four of the 8 samples were frozen at –20°C for secondary analysis. The remaining 4 sample containers were stored overnight in a refrigerated cooler set at ≤4°C. The next day, 2 of the sample containers from the refrigerated cooler were shipped overnight to CEM Corp. for analysis. Samples remained under refrigerated storage at ≤4°C for immediate moisture and fat analysis.

10 Procedures

10.1 AOAC Moisture Determination

Meat samples were analyzed for moisture according to AOAC Method **950.46**.

10.2 AOAC Crude Fat Determination

Meat samples were analyzed for crude fat according to AOAC Method **960.39**.

10.3 CEM SMART System (Moisture)/SMART Trac (Fat)

Note: Consult manufacturer's operation manual, and perform the recommended tests to determine system functionality. A frequency optimization should be performed daily prior to system operation to correct for any drift in the SMART Trac magnetic frequency.

(**10.3.1**) On the SMART system CEM Main Menu screen, select Load Method; then select the appropriate preprogrammed item to be analyzed, i.e., GROUND BEEF. Note: Different types of sample matrixes and fat will exhibit different responses with the NMR system. To obtain accurate fat readings, ≥2 samples of the specific sample type must be analyzed by the AOAC method. The samples should cover the entire fat range to be determined. Preferably, one high-fat reference sample and one low-fat reference sample should be analyzed. The reference values are typed into the SMART Trac system, and then replicate runs of each sample are performed to determine the appropriate NMR signal values for that specific sample type. After the reference scans are completed, the SMART Trac system will establish a linear relationship for fat determination for that type of sample.

(**10.3.2**) Press the Ready Key to initiate the analysis. Place 2 glass fiber sample pads (square) in the SMART system moisture/solids analyzer microwave chamber on the balance, and press Tare on the keypad. Tare weight will be automatically recorded.

(**10.3.3**) With a Teflon-coated spatula, transfer approximately 4 g sample, from the center of the refrigerated 118 g sample vial, to the center of one of the tared sample pads.

Table 3. Results from analyses of NIST SRM 1546[a,b]

	AOAC results from TAM				SMART Trac results[c] from CEM			SMART Trac results[c] from TAM		
	Method **950.46**		Method **960.39**			Microwave	NMR		Microwave	NMR
Sample ID	Wt, g	M, %	Wt, g	F, %	Wt, g	M, %	F, %	Wt, g	M, %	F, %
1	4.6370	59.37	3.5248	21.69	3.8453	58.64	21.77	2.9107	59.24	20.97
2	4.7906	59.54	3.3892	21.69	3.9331	58.44	21.76	2.2456	58.90	21.13
3	4.9450	59.41	3.3078	21.71	3.6093	58.45	21.62	2.4838	58.76	20.91
4	5.0970	59.38	3.3027	21.37	3.7151	58.53	21.60	2.4145	59.04	20.52
5	4.7507	59.53	3.5036	21.58	3.7950	58.31	21.79	2.2786	58.92	20.94
6	4.9085	59.24	3.9291	21.32	3.6658	58.59	21.58	2.5071	58.73	20.98
7	4.9116	59.41	3.4701	21.14	4.0866	58.41	21.71	2.6993	59.02	20.62
8	4.5751	59.49	3.5912	21.93	3.7401	58.47	21.61	2.1753	58.77	21.08
9	5.0447	59.15	3.9805	21.99	3.8832	58.54	21.56	3.1707	58.70	20.76
10	4.6640	58.98	3.7927	21.94	4.1022	58.44	21.48	2.4360	58.53	21.11
Mean		59.35		21.64		58.48	21.65		58.86	20.90
SD[d]		0.178		0.286		0.095	0.104		0.205	0.207

[a] Three different cans of NIST material were used for the above results. NIST reference values: moisture = 59.5 ± 2.6%; fat = 21.0 ± 1.4%.
[b] M = Moisture; F = fat.
[c] SMART system for moisture and SMART Trac system for fat.
[d] SD = Standard deviation.

Spread the meat sample evenly across the square pad (*see* Figure 1). *Note:* Sample size should be 3–5 g.

(**10.3.4**) Cover the sample with the other tared square pad as if making a sandwich, and return the pads to the SMART system moisture/solids analyzer microwave chamber on the balance.

(**10.3.5**) Dry the sample by pressing Start on the keypad. A temperature feedback system allows rapid measurement of the temperature of the sample during drying to adjust the microwave power delivery. Percent Moisture will be displayed on the screen (±0.01%) after the sample has dried to a constant

Table 4. Results from analyses of all-beef hot dogs[a]

	AOAC results from TAM				SMART Trac results[b] from CEM			SMART Trac results[b] from TAM		
	Method **950.46**		Method **960.39**			Microwave	NMR		Microwave	NMR
Sample ID	Wt, g	M, %	Wt, g	F, %	Wt, g	M, %	F, %	Wt, g	M, %	F, %
1	2.4646	51.51	3.2652	30.59	4.0588	51.55	30.63	2.5094	51.79	30.30
2	2.2422	51.45	3.6297	30.61	4.3006	51.42	30.64	0.9061	51.96	30.77
3	2.0909	51.64	4.1140	30.44	3.5390	51.64	30.28	2.9303	51.58	30.60
4	2.0910	51.54	3.1469	30.53	4.0012	51.53	30.46	3.2794	51.59	30.29
5	2.1961	51.63	3.5239	30.63	3.6598	51.46	30.65	3.4650	51.97	30.36
6	2.4098	51.75	3.6645	30.36	3.9586	51.47	30.36	3.3049	51.47	30.63
7	2.2961	51.87	3.4951	30.72	4.1737	51.38	30.72	2.8597	51.66	30.45
8	2.2859	51.89	3.1308	30.20	3.6191	51.26	30.57	3.0330	51.97	30.40
9	2.4673	51.80	3.2411	30.54	3.5878	51.44	30.42	2.7080	51.69	30.63
10	2.0816	51.69	3.1027	30.18	3.7391	51.17	30.71	2.7289	51.49	30.71
Mean		51.68		30.48		51.43	30.54		51.72	30.51
SD[c]		0.152		0.183		0.138	0.154		0.195	0.175

[a] M = Moisture; F = fat.
[b] SMART system for moisture and SMART Trac system for fat.
[c] SD = Standard deviation.

Table 5. Results from analyses of low-fat fresh pork[a]

	AOAC results from TAM				SMART Trac results[b] from CEM			SMART Trac results[b] from TAM		
	Method **950.46**		Method **960.39**			Microwave	NMR		Microwave	NMR
Sample ID	Wt, g	M, %	Wt, g	F, %	Wt, g	M, %	F, %	Wt, g	M, %	F, %
1	7.3366	73.90	3.7666	3.75	3.8205	73.90	3.75	2.5704	73.65	3.77
2	6.7329	73.91	4.1604	3.86	4.2475	73.79	3.74	2.3533	73.76	3.85
3	7.1051	73.83	3.3838	3.44	3.8189	73.74	3.77	3.0565	73.72	3.94
4	8.0669	73.93	3.6228	3.69	4.0823	73.60	3.78	1.9321	73.88	3.87
5	7.4594	74.06	3.6303	3.84	3.8975	73.77	3.78	2.0746	73.73	3.92
6	7.0622	74.03	3.7274	3.95	3.5714	73.74	3.77	1.8361	73.69	3.93
7	8.1275	74.00	3.4831	3.76	3.8836	73.66	3.79	1.9611	73.81	3.80
8	7.7963	74.02	3.7028	3.78	4.3785	73.70	3.74	2.3695	73.46	4.05
9	7.2248	73.92	3.3244	3.58	4.1558	73.73	3.74	2.6486	74.00	3.79
10	8.0085	73.80	3.8148	3.78	4.1257	73.58	3.77	1.9676	73.75	3.85
Mean		73.94		3.74		73.72	3.76		73.75	3.88
SD[c]		0.087		0.146		0.094	0.019		0.142	0.085

[a] M = Moisture; F= fat
[b] SMART system for moisture and SMART Trac system for fat.
[c] SD = Standard deviation.

weight. *Note*: Five short beeps will be heard when drying is complete.

(**10.3.6**) Remove pads and roll both pads in Trac Film (*see* Figure 2).

(**10.3.7**) Compress the rolled sample in the plastic sleeve by using the compression tool, and insert the sample into the NMR chamber for analysis. (The sample is placed in the core of an 89 kg magnet and pulsed with RF energy while in the static magnetic field. The resulting signal is recorded and analyzed for the total proton activity of fat present in the sample. Proprietary software analyzes the NMR data and provides the moisture and fat results.)

(**10.3.8**) Press Ready to continue the fat analysis; then press Start to analyze for fat. Percent Fat will be displayed on the screen (±0.01%).

11 Test Results Report

The results from the study are given in Tables 1–5. TAM used the AOAC moisture and fat methods to analyze the 5 products. TAM and CEM, the participating laboratories, both

Table 6. Statistical summary for determination of moisture

Parameter[a]	Pork, fresh, low fat	Hot dogs, all beef	NIST SRM 1546	Chicken, fresh, with skin	Beef, fresh, high fat
AOAC Method **950.46**					
$\bar{x}$, %	73.94	51.68	59.35	74.57	40.39
S_o	0.087	0.152	0.178	0.186	0.074
CV_o, %	0.118	0.294	0.300	0.249	0.183
Rapid microwave drying method					
$\bar{x}$, %	73.73	51.57	58.67	74.42	40.35
S_o	0.120	0.169	0.160	0.143	0.148
S_x	0.115	0.258	0.308	0.156	0.171
CV_o, %	0.163	0.328	0.272	0.192	0.366
CV_x, %	0.156	0.499	0.525	0.209	0.424

[a] $\bar{x}$ = Mean; S_o and S_x = within-laboratory repeatability standard deviation and between-laboratories reproducibility standard deviation, respectively; and CV_o and CV_x = the corresponding coefficients of variation.

Table 7. Statistical summary for determination of fat

Parameter[a]	Pork, fresh, low-fat	Hot dogs, all-beef	NIST SRM 1546	Chicken, fresh, with skin	Beef, fresh, high-fat
			AOAC Method **960.39**		
$\bar{x}$, %	3.74	30.48	21.64	7.24	45.84
S_o	0.146	0.183	0.286	0.186	0.157
CV_o, %	3.904	0.600	1.322	2.569	0.342
			NMR fat method		
$\bar{x}$, %	3.82	30.53	21.28	7.22	45.98
S_o	0.062	0.165	0.164	0.059	0.288
S_x	0.100	0.158	0.550	0.114	0.274
CV_o, %	1.612	0.539	0.769	0.811	0.626
CV_x, %	2.606	0.516	2.584	1.573	0.597

[a] $\bar{x}$ = Mean; S_o and S_x = within-laboratory repeatability standard deviation and between-laboratories reproducibility standard deviation, respectively; and CV_o and Cv_x = the corresponding coefficients of variation.

used the SMART system and SMART Trac system to determine moisture and fat, respectively, in the 5 products.

Data were analyzed, and the statistical summaries are given in Tables 6 and 7. The values shown for each product category include means, standard deviations for within-laboratory repeatability, and standard deviations for between-laboratories reproducibility. Corresponding coefficients of variation are also included. The statistical data indicate the SMART system and SMART Trac system compare favorably with the AOAC methods for determination of both moisture and fat.

The standard deviation of the between-laboratories reproducibility was larger for the NIST SRM 1546 sample than for the other 4 products. However, 3 separate cans of the product were used to collect AOAC, TAM, and CEM results. The specifications for the product indicate a moisture value of 59.5 ± 2.6% and a fat value of 21.0 ± 1.4%. Given the specified product variability, the standard deviation of the between-laboratories reproducibility is not only acceptable, but also expected.

12 Conclusions from Ruggedness Testing (Attachment A)

Initial sample weight can have an adverse effect on moisture and fat determinations. Sample weight should be in the range of 3–5 g.

Initial sample weights of >5 g do not have a significant effect on moisture and fat determinations; however, the ideal weight range of 3–5 g should be maintained to eliminate the chance of burning during the drying stage.

The drying temperature of the sample does have an effect on moisture and fat determinations. Temperatures of <125°C will adversely affect the analyses; however, temperatures of >125° to 150°C had very little adverse effect on the sample that was tested. Temperatures of >125°C are not recommended because of the chance of burning during the drying stage.

Sample temperature does have a significant adverse effect on fat determination for high-fat samples. Samples should be cooled to 40°C before NMR analysis.

13 Quality Assurance

To prevent water loss during sample preparation and subsequent handling, do not use small samples. Keep all prepared samples in air- and watertight containers. The size of the test sample should be 3–5 g. Samples should be dried at 125°C and conditioned to 40°C before NMR analysis.

14 Comments

Overall the 2 participating laboratories were pleased with the method because it provides safe and rapid analyses of meat products for moisture and fat with results that compare favorably with those obtained by the AOAC methods.

14.1 Peer Laboratory Comments

The SMART Trac system offers a new, safe, and rapid method for determining moisture and fat in meat samples. The SMART Trac system provided results comparable with those obtained by the AOAC methods except for the results for the high-fat (30–50%) beef sample and the NIST standard (Tables 1 and 3). The variability (standard deviation) was higher for the analysis of the beef sample by the SMART Trac system than for the analysis by AOAC Method **960.39**. This could have been the result of variation in the sample containers or inconsistency in the homogenate or temperature of the sample at the time of analysis (our samples were 1°–2°C). Another variation was observed with the NIST sample. The NIST standard was a mixed-species sample of pork and mechanically deboned poultry, which have different lipid profiles and fat melting points. The fat results from our laboratory were approximately 1% lower than the results from the collaborating laboratory (Table 3). The recorded values from both laborato-

ries were within the acceptable range for the NIST standard of ±1.4% fat. Because the NIST sample comes in sealed containers and cannot be identified by batch or sequential container numbers, the inconsistency of the analytical results could be due to differences between containers.

The SMART Trac system does appear to offer reliable results that are comparable with those obtained by the AOAC methods for determination of moisture and fat. Compared with other analytical techniques for the determination of fat and moisture (Modified Babcock, Hobart, Ohaus, etc.), the SMART Trac system provides a safe and rapid analytical method. Earlier automated solvent extraction and AOAC ether extraction methods potentially expose workers to hazardous chemicals. The new CEM SMART Trac system eliminates the need for costly chemical disposal, large amounts of glassware, and special extraction equipment used in the AOAC Soxhlet and Goldfisch ether extraction methods. Earlier microwave drying systems had problems with overheating, charring, and oxidation of samples containing spices; especially high percentages of salt but no difficulties were noted with the SMART Trac system microwave unit that uses an infrared detector to monitor temperature. Overheating of samples can result in high estimates of percent moisture because of volatilization of fat components. The TAM laboratory is also equipped with a near infrared (NIR) analyzer. NIR analyzers offer rapid measurement of fat and moisture; however, calibration of these types of instruments can be difficult and time consuming, and the analytical range for a particular product can be limited. With the supplied programs for selected products, the SMART Trac system can provide quick, safe, and reliable results. Rapid testing is becoming a critical factor for the incorporation of raw materials into finished products, and few methods are available that offer speed, reliability, accuracy, and safety.

Attachment A: Ruggedness Testing

Example 1: Pork, Fresh, Low-Fat

Test: Effect of sample weight on the determination of moisture and fat by using microwave drying and NMR, respectively.

In pork Example 1, the weight of the sample was decreased to below the recommended parameter of 3–5 g. Results showed that sample size can have an effect on moisture and fat determinations. A drying temperature of 125°C was inadequately maintained during the drying cycle, because of the sample size. Therefore, results for moisture in the above samples ranged from 0.25–0.39% below the AOAC average of 73.94%. All the water was not eliminated from the sample during the drying process; therefore, additional hydrogen protons were present and caused an increase in the reported fat. When NMR technology is used for fat determination, all water must be eliminated from the sample before the sample is placed in the nuclear magnetic field for fat determination. If the hydrogen protons from water are not completely eliminated, these protons will be calculated as fat. This resulted in a range of 0.27–0.41% above the AOAC average of 3.74% fat.

Example 2: Pork, Fresh, Low-Fat

Test: Effect of sample weight on the determination of moisture and fat by using microwave drying and NMR, respectively.

In pork Example 2, the size of sample was increased beyond the recommended parameter of 3–5 g. Results showed that larger sample sizes have very little effect on moisture or fat determinations.

Example 3: Pork, Fresh, Low-Fat

Test: Effect of drying temperature on the determination of moisture and fat by using microwave drying and NMR, respectively.

In pork Example 3, the drying temperature was decreased to 100°C. The recommended drying temperature is 125°C. Results showed that drying temperature has a significant effect on both moisture and fat determinations. The same effect as in pork Example 1 was evident.

Example 4: Pork, Fresh, Low-Fat

Test: Effect of drying temperature on the determination of moisture and fat by using microwave drying and NMR, respectively.

In pork Example 4, the drying temperature was increased to 150°C. The recommended drying temperature is 125°C. Results showed that a higher drying temperature has very little effect on

Table A.1. Test results for example 1[a]

					AOAC	
Sample ID	Wt, g	Microwave, M, %	NMR, F, %	Microwave temperature, °C	M, %	F, %
1	1.3403	73.62	4.01	125	73.94	3.74
2	1.0345	73.67	4.06	125		
3	1.0017	73.55	4.15	125		
4	1.1002	73.69	4.08	125		

[a] M = Moisture; F = fat.

Table A.2. Test results for example 2[a]

					AOAC	
Sample ID	Wt, g	Microwave, M, %	NMR, F, %	Microwave temperature, °C	M, %	F, %
1	7.2790	73.75	3.74	125	73.94	3.74
2	7.4323	73.69	3.79	125		
3	9.7027	73.57	3.78	125		
4	7.3326	73.61	3.81	125		
5	8.1285	73.52	3.81	125		

[a] M = Moisture; F = fat.

A.3. Test results for example 3[a]

					AOAC	
Sample ID	Wt, g	Microwave, M, %	NMR, F, %	Microwave temperature, °C	M, %	F, %
1	4.0520	73.43	4.12	100	73.94	3.74
2	4.1170	73.71	4.02	100		
3	3.9093	73.26	4.12	100		
4	4.3017	72.96	4.41	100		
5	4.2387	73.04	4.24	100		

[a] M = Moisture; F = fat.

Table A.4. Test results for example 4[a]

					AOAC	
Sample ID	Wt, g	Microwave, M, %	NMR, F, %	Microwave temperature, °C	M, %	F, %
1	3.8377	73.77	3.76	150	73.94	3.74
2	4.1631	73.74	3.75	150		
3	3.7595	73.82	3.81	150		
4	4.0015	73.93	3.72	150		
5	4.2828	73.84	3.74	150		

[a] M = Moisture; F= fat.

Table A.5. Test results for example 5[a]

					AOAC	
Sample ID	Wt, g	Microwave, M, %	NMR, F, %	Microwave temperature, °C	M, %	F, %
1	3.5729	51.58	27.52	125	51.68	30.48
2	3.7322	51.79	27.29	125		
3	3.8488	51.77	27.31	125		
4	4.3164	51.75	27.59	125		
5	4.1137	52.07	27.05	125		

[a] M = Moisture; F = fat.

Table A.6. Test results for example 6[a]

					AOAC	
Sample ID	Wt, g	Microwave, M, %	NMR, F, %	Microwave temperature, °C	M, %	F, %
1	4.0332	73.93	3.73	125	73.94	3.74
2	4.3699	73.43	3.69	125		
3	4.3127	73.36	3.76	125		
4	4.0411	73.65	3.61	125		
5	4.1143	73.50	3.71	125		

[a] M = Moisture; F = fat.

either moisture or fat. Any temperatures resulting in burning or degradation of the sample would decrease the fat value.

Example 5: Hot Dogs, All-Beef

Test: Effect of sample temperature on the determination of fat by using NMR.

In all-beef hot dogs Example 5, the temperature at which the sample is introduced to the NMR instrumentation was tested. The magnet is maintained at a constant temperature of 40°C; therefore, all samples measured should be conditioned to 40°C. The samples in Example 5 were taken directly from the microwave at a temperature of 125°C and put immediately into the NMR instrument. Results showed that sample temperature does have an adverse effect on fat determination. The AOAC average for this sample was 30.48% fat.

Example 6: Pork, Fresh, Low-Fat

Test: Effect of sample temperature on the determination of fat by using NMR.

In pork Example 6, the temperature at which the sample is introduced to the NMR instrumentation was tested. The magnet is maintained at a constant temperature of 40°C; therefore, all samples measured should be conditioned to 40°C. These samples were taken directly from the microwave at a temperature of 125°C and put immediately into the NMR instrument. Unlike Example 5, which was a sample with high-fat content, this sample was low in fat and therefore cooled to 40°C before it was introduced to the NMR instrumentation. There was no effect on samples with low-fat content.