FOOD COMPOSITION AND ADDITIVES

Rapid Determination of Moisture/Solids and Fat in Dairy Products by Microwave and Nuclear Magnetic Resonance Analysis

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Abstract

A peer-verified method is presented for the determination of percent moisture/solids and fat in dairy products by microwave drying and nuclear magnetic resonance (NMR) analysis. The method involves determining the moisture/solids content of dairy samples by microwave drying and using the dried sample to determine the fat content by NMR analysis. Both the submitting and peer laboratories analyzed various dairy products by using a CEM SMART system (moisture) and the SMART Trac (fat). The samples included milks, creams, ice cream mix, sour cream, yogurt, cream cheese, and mozzarella, Swiss, and cheddar cheeses. These samples represented a range of products that processors deal with in daily plant operations. The results were compared with moisture/solids and fat values derived from AOAC-approved methods.

1 Summary of Results

1.1 Matrixes

This paper addresses the determination of moisture/solids and fat in a range of dairy products including milks, creams, ice cream mix, yogurt, and cheeses.

1.2 Number of Samples

The Dairy Quality Control Institute (DQCI Services, Inc.) reported the results obtained by AOAC Method **990.20** (1) for the solids analyses of the milks and creams.

CEM performed the solids analyses of the yogurt samples according to the steps outlined in AOAC Method **990.20**. Moisture analyses of the cheese samples were performed by CEM according to the steps outlined in AOAC Method **926.08**, and ice cream solids were determined by CEM according to AOAC Method **941.08**. The standard methods were run 5 times for each yogurt, cheese, and ice cream sample.

Crude fat analyses of the dairy samples were performed according to the steps outlined in AOAC Method **989.05** for the milks and creams, and the results were reported by DQCI Services, Inc.

For sour cream and yogurt samples, AOAC Method **905.02** was run 5 times by CEM. For the cheese samples, AOAC Method **933.05** was used, and for ice cream, AOAC Method **952.06** was run 5 times by CEM.

The submitting and peer laboratories independently performed analyses of 11 products (10 times each) for both moisture/solids and fat by 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 in. (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 method and a rapid NMR procedure for the determination of moisture/solids and fat, respectively, in dairy products. These tests cover a variety of dairy products and a wide range of moisture/solids and fat levels.

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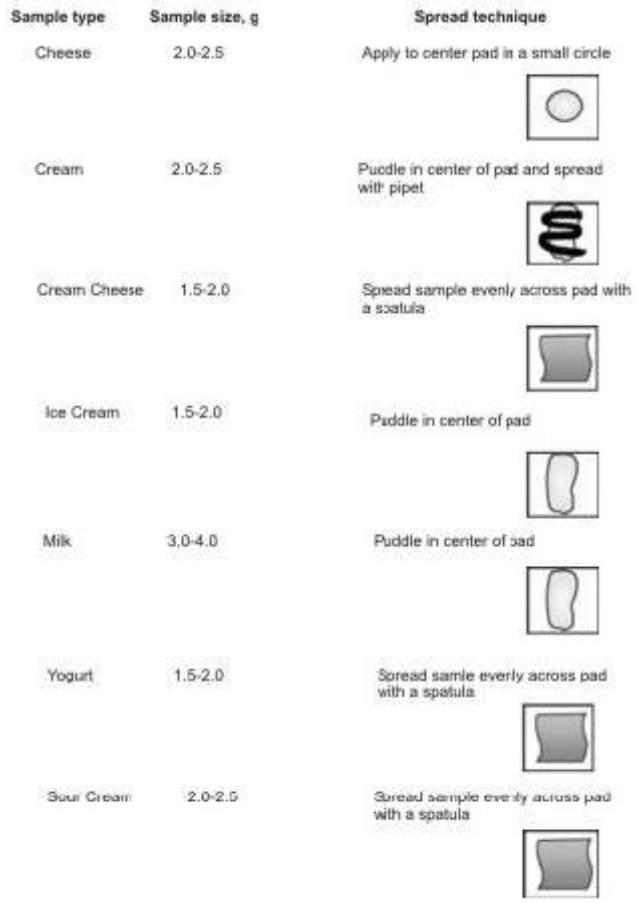

Figure 1. Approximate sample size and spread technique for various sample types.

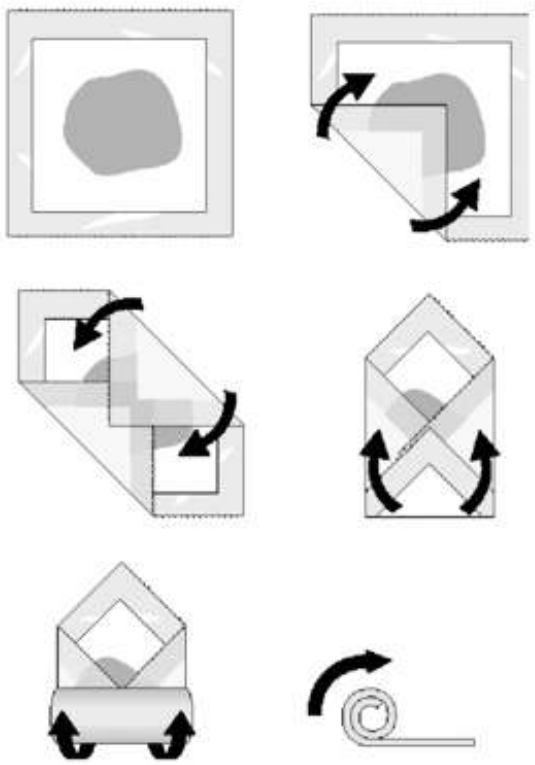

Figure 2. Place the 2 square pads and dried sample in the center of the Trac film. Fold the left corner of the film and pads as illustrated. Fold the right corner. Pull the lower edge of the film and sample pads toward the top and begin to roll them into a tube.

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 Definitions and Abbreviations

(**5.1**) *NMR*.—Nuclear magnetic resonance.
(**5.2**) *RF*.—Radio frequency.
(**5.3**) *NCSU*.—North Carolina State University.
(**5.4**) *NIR*.—Near infrared.
(**5.5**) *LR-NMR*.—Low-resolution time-domain NMR.
(**5.6**) *DQCI*.—Dairy Quality Control Institute.

6 Principle

NMR, discovered in the middle of the last century, is based on the observation that certain nuclei will absorb and re-emit 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 why the word "nuclear" is included in the description of the phenomenon. NMR does not involve the emission of ionizing radiation.)

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 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

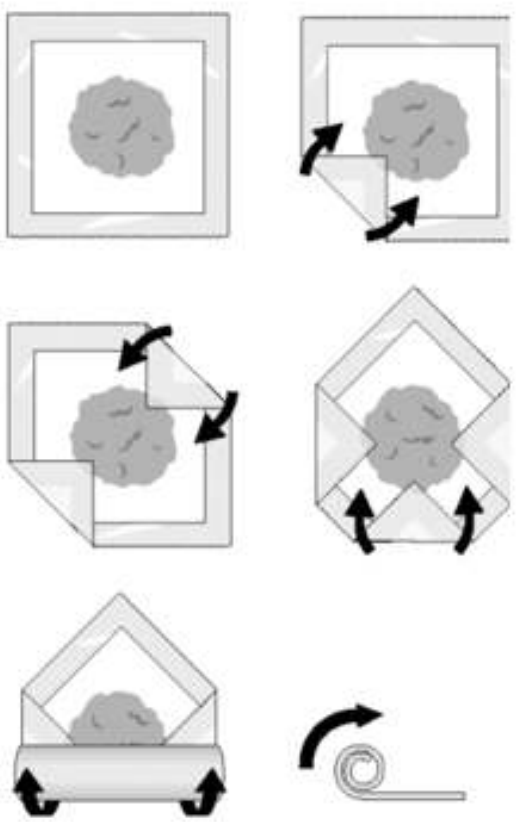

Figure 3. For samples that are rigid after being dried and more difficult to roll into a cylinder, prepare the pads as illustrated above.

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 ^1H 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 ^1H 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 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 of the order of ≤10 μs), and the signal from these substances decays very rapidly. However, the transverse relaxation times for fat are considerably longer (typically of 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 in various petroleum products and International Standards Organization (ISO) 10565 for determination of oil and moisture in seeds.

7 Standards

Not applicable.

8 Supplies

(**8.1**) *Glass fiber pads*.—CEM Corp. or equivalent.

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

9 Apparatus and Equipment

(**9.1**) *Microwave moisture/solids 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.

(**9.2**) *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.

Table 1. Results from analyses of DQCI milk sample 9, set 239[a]

	SMART Trac results from CEM			SMART Trac results from NCSU			AOAC results from DQCI	
Sample ID	Wt, g	Microwave, S, %	NMR, F, %	Wt, g	Microwave, S, %	NMR, F, %	Method **990.10**, S, %	Method **989.05**, F, %
1	4.1443	11.77	2.99	3.8945	11.83	3.00	11.79	3.0193
2	4.3300	11.78	2.98	4.1822	11.83	3.01	11.78	3.0094
3	4.4346	11.80	2.97	4.1213	11.81	3.00		3.0213
4	4.7113	11.81	2.98	4.0613	11.80	3.00		
5	4.4751	11.77	2.96	4.0441	11.80	3.03		
6	3.9464	11.79	3.01	4.4162	11.80	3.00		
7	3.8453	11.79	3.01	4.1109	11.77	2.98		
8	3.6758	11.78	3.01	4.0890	11.83	2.99		
9	3.6831	11.78	2.99	4.1664	11.78	3.01		
10	3.7225	11.79	3.01	4.2647	11.82	2.98		
Mean		11.79	2.99		11.81	3.00	11.79	3.02
SD[b]		0.013	0.019		0.021	0.015	0.007	0.006

[a] S = Solids; F = fat.
[b] SD = Standard deviation.

Table 2. Results from analyses of DQCI reduced milk sample 5, set 239[a]

	SMART Trac results from CEM			SMART Trac results from NCSU			AOAC results from DQCI	
Sample ID	Wt, g	Microwave, S, %	NMR, F, %	Wt, g	Microwave, S, %	NMR, F, %	Method **990.10**, S, %	Method **989.05**, F, %
1	4.3015	9.93	0.99	4.0372	9.88	0.97	9.87	0.9827
2	3.9614	9.93	0.97	3.7935	9.93	0.98	9.87	1.0133
3	4.3538	9.92	0.97	3.9850	9.94	0.99		0.9956
4	3.6101	9.87	0.98	4.1085	9.93	0.99		
5	3.5606	9.93	0.99	4.0157	9.93	0.99		
6	3.8770	9.90	0.96	4.4296	9.93	0.98		
7	4.1012	9.92	0.96	4.1261	9.91	0.98		
8	3.9439	9.93	0.96	4.1844	9.93	1.00		
9	4.1969	9.93	0.98	4.0369	9.88	1.00		
10	4.6306	9.87	0.98	4.5477	9.89	0.97		
Mean		9.91	0.97		9.92	0.99	9.87	1.00
SD[b]		0.025	0.012		0.023	0.011	0.000	0.015

[a] S = Solids; F = fat.
[b] SD = Standard deviation.

Table 3. Results from analyses of DQCI heavy cream[a]

	SMART Trac results from CEM			SMART Trac results from NCSU			AOAC results from DQCI	
Sample ID	Wt, g	Microwave, S, %	NMR, F, %	Wt, g	Microwave, S, %	NMR, F, %	Method **990.20**, S, %	Method **989.05**, F, %
1	2.5523	45.59	39.83	2.6443	45.52	39.95	45.57	39.96
2	2.3591	45.61	39.95	2.4219	45.49	39.93	45.57	39.90
3	2.4594	45.53	40.01	2.5356	45.52	39.84		
4	2.3075	45.63	39.92	2.5842	45.51	39.85		
5	2.5811	45.56	39.83	2.3451	45.35	39.96		
6	2.6227	45.61	39.86	2.0813	45.57	40.07		
7	2.4871	45.59	40.01	2.2943	45.54	40.07		
8	2.3598	45.66	40.00	2.4101	45.54	39.88		
9	2.3285	45.56	39.85	2.6731	45.52	39.95		
10	2.4629	45.65	40.00	2.2915	45.40	39.92		
Mean		45.60	39.93		45.50	39.94	45.57	39.93
SD[b]		0.041	0.078		0.068	0.079	0.000	0.042

[a] S = Solids; F = fat.
[b] SD = Standard deviation.

Table 4. Results from analyses of light cream sample 4, set 538[a]

	SMART Trac results from CEM			SMART Trac results from NCSU			AOAC results from DQCI	
Sample ID	Wt, g	Microwave, S, %	NMR, F, %	Wt, g	Microwave, S, %	NMR, F, %	Method **990.20**, S, %	Method **989.05**, F, %
1	2.3534	20.85	12.92	2.5073	20.83	12.97	20.86	12.9130
2	2.3827	20.83	12.91	2.5196	20.77	12.93	20.85	12.8951
3	2.5486	20.80	12.89	2.2672	20.85	12.92	20.85	12.8395
4	2.4339	20.82	12.92	2.2035	20.78	12.90		
5	2.4584	20.77	12.86	2.4807	20.78	12.88		
6	2.3064	20.74	12.95	2.5304	20.92	12.91		
7	2.3418	20.87	12.92	2.4260	20.75	12.88		
8	2.4656	20.83	12.83	2.3663	20.80	12.84		
9	2.3290	20.85	12.89	2.5216	20.90	12.86		
10	2.4446	20.80	12.89	2.4813	20.83	12.87		
Mean		20.82	12.90		20.82	12.90	20.85	12.88
SD[b]		0.039	0.034		0.056	0.038	0.006	0.038

[a] S = Solids; F = fat.
[b] SD = Standard deviation.

Table 5. Results from analyses of mozzarella cheese[a]

	SMART Trac results from CEM			SMART Trac results from NCSU			AOAC results from CEM	
Sample ID	Wt, g	Microwave, M, %	NMR, F, %	Wt, g	Microwave, M, %	NMR, F, %	Method **926.08**, M, %	Method **933.05**, F, %
1	2.2308	46.15	24.25	2.2527	46.28	24.40	46.17	24.32
2	2.0764	46.02	24.23	2.1932	46.10	24.44	46.18	24.47
3	2.1903	46.22	24.25	2.2509	46.19	24.40	46.10	24.30
4	1.9632	46.12	24.32	2.2067	46.01	24.40	46.05	24.13
5	1.9422	45.83	24.30	2.0898	46.11	24.41	46.24	24.36
6	1.7798	45.95	24.37	2.2554	46.29	24.43		
7	1.9395	45.81	24.43	2.0403	45.99	24.28		
8	2.1878	45.92	24.56	2.4189	46.08	24.32		
9	1.8352	46.08	24.53	2.1369	46.07	24.43		
10	2.3110	46.24	24.34	2.0268	46.06	24.31		
Mean		46.03	24.36		46.12	24.38	46.15	24.32
SD[b]		0.146	0.110		0.0098	0.054	0.066	0.110

[a] M = Moisture; F = fat.
[b] SD = Standard deviation.

Table 6. Results from analyses of Swiss cheese[a]

	SMART Trac results from CEM			SMART Trac results from NCSU			AOAC results from CEM	
Sample ID	Wt, g	Microwave, M, %	NMR, F, %	Wt, g	Microwave, M, %	NMR, F, %	Method **926.08**, M, %	Method **933.05**, F, %
1	2.7200	40.08	27.88	2.2181	39.84	28.07	39.95	27.84
2	2.5223	39.97	28.01	2.3263	39.70	27.91	39.78	27.85
3	2.6170	39.98	27.70	2.4166	39.75	27.81	39.82	28.20
4	2.7229	39.90	27.74	2.0604	39.71	27.80	40.00	27.87
5	2.5451	39.91	27.88	2.0643	39.83	27.89	40.27	28.15
6	2.5906	40.00	28.15	2.2466	39.78	28.01		
7	2.6066	40.11	27.96	2.3289	40.02	28.07		
8	2.7935	39.95	28.01	2.6000	39.88	28.09		
9	2.5475	39.90	27.81	2.1072	39.71	28.17		
10	2.8223	39.98	28.18	2.3289	39.93	28.07		
Mean		39.98	27.93		39.82	27.99	39.96	27.98
SD[b]		0.068	0.153		0.101	0.121	0.173	0.159

[a] M = Moisture; F = fat.
[b] SD = Standard deviation.

Table 7. Results from analyses of cheddar cheese[a]

	SMART Trac results from CEM			SMART Trac results from NCSU			AOAC results from CEM	
Sample ID	Wt, g	Microwave, M, %	NMR, F, %	Wt, g	Microwave, M, %	NMR, F, %	Method **926.08**, M, %	Method **933.05**, F, %
1	2.0178	36.59	31.43	2.0936	36.79	31.35	36.79	31.41
2	2.1036	36.99	31.16	2.2693	36.67	31.35	36.68	31.43
3	2.0657	36.59	31.15	2.2849	36.89	31.37	36.83	31.07
4	2.1941	36.80	31.28	2.2639	36.83	31.49	36.77	31.32
5	2.0218	36.66	31.35	2.0009	36.85	31.16	36.75	31.24
6	2.1164	36.62	31.51	2.1508	36.69	31.05		
7	2.3500	36.68	31.30	2.0369	36.88	31.37		
8	2.4276	36.70	31.32	2.3932	36.81	31.10		
9	2.1461	36.63	31.43	2.0830	36.57	31.28		
10	1.9509	36.55	31.29	2.3385	36.65	31.41		
Mean		36.68	31.32		36.76	31.29	36.76	31.29
SD[b]		0.123	0.109		0.104	0.136	0.050	0.131

[a] M = Moisture; F = fat.
[b] SD = Standard deviation.

Table 8. Results from analyses of ice cream[a]

	SMART Trac results from CEM			SMART Trac results from NCSU			AOAC results from CEM	
Sample ID	Wt, g	Microwave, S, %	NMR, F, %	Wt, g	Microwave, S, %	NMR, F, %	Method **941.08**, S, %	Method **952.06**, F, %
1	1.6988	37.87	9.73	1.8188	37.91	9.81	38.02	9.76
2	1.9009	37.91	9.77	1.8875	37.90	9.75	38.07	9.74
3	2.0709	38.00	9.78	1.8883	38.00	9.80	37.85	9.78
4	1.1972	38.07	9.73	2.0424	37.97	9.77	37.93	9.80
5	2.1537	38.08	9.71	1.8710	38.03	9.73	37.86	9.75
6	1.9221	37.98	9.78	2.1444	37.87	9.73		
7	2.1258	38.01	9.72	2.0934	37.95	9.76		
8	1.7855	37.97	9.76	1.8591	37.97	9.77		
9	1.6788	37.93	9.78	1.9112	37.95	9.78		
10	2.1355	38.07	9.72	1.9741	37.98	9.83		
Mean		37.99	9.75		37.95	9.77	37.95	9.77
SD[b]		0.072	0.029		0.048	0.033	0.087	0.022

[a] S = Solids; F = fat.
[b] SD = Standard deviation.

Table 9. Results from analyses of sour cream[a]

	SMART Trac results from CEM			SMART Trac results from NCSU			AOAC results from CEM	
Sample ID	Wt, g	Microwave, S, %	NMR, F, %	Wt, g	Microwave, S, %	NMR, F, %	Method **990.20**, S, %	Method **905.02**, F, %
1	2.2018	25.93	18.40	2.2358	25.94	18.39	26.03	18.26
2	2.6194	26.00	18.49	2.2176	25.91	18.41	26.12	18.37
3	2.1480	26.01	18.48	2.3220	26.06	18.44	26.03	18.58
4	2.3803	26.07	18.47	2.4422	25.91	18.39	26.00	18.51
5	2.3554	26.00	18.51	2.3928	26.05	18.46	25.93	18.37
6	2.2559	26.02	18.52	2.1009	26.15	18.53		
7	2.4510	26.02	18.49	2.0944	26.10	18.56		
8	2.4436	25.93	18.50	2.5110	26.16	18.48		
9	2.4227	25.99	18.46	2.1413	26.04	18.41		
10	2.3467	26.01	18.50	2.2793	25.99	18.49		
Mean		26.00	18.48		26.03	18.46	26.02	18.42
SD[b]		0.042	0.034		0.092	0.059	0.061	0.113

[a] S = Solids; F = fat.
[b] SD = Standard deviation.

Table 10. Results from analyses of cream cheese[a]

	SMART Trac results from CEM			SMART Trac results from NCSU			AOAC results from CEM	
Sample ID	Wt, g	Microwave, S, %	NMR, F, %	Wt, g	Microwave, S, %	NMR, F, %	Method **926.08**, S, %	Method **933.05**, F, %
1	2.1385	41.05	28.23	1.8721	41.22	28.11	41.17	28.26
2	2.1463	41.15	28.20	1.7500	41.15	28.12	41.18	28.10
3	1.8897	40.97	28.16	1.7736	41.11	28.13	41.16	28.04
4	2.0791	41.16	28.17	1.7596	40.97	28.12	41.13	28.28
5	1.7843	41.11	28.26	1.7385	41.22	28.17	41.09	28.19
6	1.6406	40.92	28.20	2.0077	41.06	28.24		
7	1.8609	41.10	28.10	1.9284	41.15	28.16		
8	2.0020	40.95	28.23	1.6891	41.09	28.26		
9	1.7305	41.07	28.24	1.8422	41.19	28.22		
10	1.7953	41.20	28.21	1.7073	41.10	28.25		
Mean		41.07	28.20		41.13	28.18	41.15	28.17
SD[b]		0.095	0.047		0.077	0.059	0.033	0.092

[a] S = Solids; F = fat.
[b] SD = Standard deviation.

Table 11. Results from analyses of yogurt[a]

	SMART Trac results from CEM			SMART Trac results from NCSU			AOAC results from CEM	
Sample ID	Wt, g	Microwave, S, %	NMR, F, %	Wt, g	Microwave, S, %	NMR, F, %	Method **990.20**, S, %	Method **905.02**, F, %
1	1.8034	12.89	3.23	1.6898	12.92	3.19	13.00	3.21
2	1.9221	12.99	3.25	1.7745	13.05	3.26	13.01	3.28
3	1.7733	12.99	3.29	2.0980	13.04	3.24	12.90	3.19
4	1.9049	13.01	3.27	1.8623	12.95	3.19	12.95	3.28
5	1.7933	12.93	3.25	1.7562	12.99	3.25	12.96	3.28
6	1.8733	12.99	3.26	2.0185	12.99	3.22		
7	1.6752	12.98	3.26	2.0428	12.96	3.20		
8	1.7605	12.99	3.27	1.8348	13.04	3.26		
9	1.9152	13.01	3.24	1.9762	13.01	3.23		
10	2.1500	13.07	3.24	1.8810	13.06	3.24		
Mean		12.99	3.26		13.00	3.23	12.96	3.25
SD[b]		0.048	0.018		0.047	0.027	0.039	0.040

[a] S = Solids; F = fat.
[b] SD = Standard deviation.

10 Sample Preparation Procedures

(**10.1**) *Dairy products/milk*.—AOAC Official Method **925.21**.

(**10.2**) *Dairy products/cheese*.—AOAC Official Method **955.30**.

11 Procedures

11.1 AOAC Moisture/Solids Determination

Dairy samples were analyzed for moisture/solids according to AOAC Method **990.20** for the milks, creams, sour cream, and yogurt. Cheese samples were analyzed for moisture according to AOAC Method **926.08**, and ice cream samples were analyzed for solids according to AOAC Method **941.08**.

11.2 AOAC Crude Fat Determination

Dairy samples were analyzed for crude fat according to AOAC Method **989.05** for the milks and creams. Samples of sour cream and yogurt were analyzed by AOAC Method **905.02**. Cheese samples were analyzed by AOAC Method **933.05**, and ice cream samples, by AOAC Method **952.06**.

11.3 CEM SMART System (Moisture/Solids)/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.

(**11.3.1**) On the SMART system **CEM Main Menu** screen, select **Load Method**; then select the appropriate preprogrammed item to be analyzed, e.g., **Milk**.

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 analyzed. Preferably, 1 high-fat reference sample and 1 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.

(**11.3.2**) 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.

(**11.3.3**) Transfer the approximate amount of sample onto the center of 1 of the tared sample pads. (*See* Figure 1 for approximate sample size and spread technique.) Spread the sample according to the illustrations in Figure 1.

(**11.3.4**) 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.

(**11.3.5**) 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/Solids** will be displayed on the screen (±0.01%) after the sample has dried to a constant weight. *Note*: Five short beeps will be heard when drying is complete.

Table 12. Statistical summary for determination of solids in dairy products

Parameter[a]	DQCI milk sample	DQCI reduced milk sample	DQCI heavy cream	DQCI light cream
AOAC Method **990.20**				
$\bar{x}$, %	11.79	9.87	45.57	20.85
S_o	0.007	0.000	0.000	0.006
Rapid microwave drying method				
$\bar{x}$, %	11.80	9.91	45.55	20.82
S_o	0.017	0.024	0.056	0.049
S_x	0.022	0.023	0.090	0.046
CV_o, %	0.148	0.241	0.124	0.233
CV_x, %	0.188	0.229	0.198	0.222

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

(**11.3.6**) Remove pads and roll both pads in Trac film (Figures 2 and 3).

(**11.3.7**) 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. Moisture and fat results are calculated and displayed.)

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

12 Test Results Report

The results from the study are given in Tables 1–11. CEM and DQCI used the AOAC solids/moisture and fat methods to analyze the 11 products. NCSU and CEM, the participating laboratories, both used the SMART system and SMART Trac system to determine moisture/solids and fat, respectively, in the 11 products.

Data were analyzed, and the statistical summaries are given in Tables 12–19 (2). The statistical data for each product category include the mean, the standard deviations for within-laboratory repeatability and between-laboratories reproducibility, and the corresponding coefficients of variation. The data indicate the SMART system and SMART Trac system compare favorably with the AOAC methods for determination of both moisture/solids and fat. Of utmost importance are the statistical data for the milks and the creams because these products serve as the raw materials for dairy products. Therefore, the favorable results for the finished products, including the cultured products, in this study were expected.

13 Conclusions from Ruggedness Testing (Attachment A)

Initial sample weight can have an adverse effect on moisture/solids and fat determinations. Sample weight should

Table 13. Statistical summary for determination of fat in dairy products

Parameter[a]	DQCI milk sample	DQCI reduced milk sample	DQCI heavy cream	DQCI light cream
AOAC Method **989.05**				
$\bar{x}$, %	3.02	1.00	39.93	12.88
S_o	0.006	0.015	0.042	0.038
NMR fat method				
$\bar{x}$, %	3.00	0.98	39.93	12.90
S_o	0.017	0.011	0.078	0.036
S_x	0.017	0.013	0.075	0.034
CV_o, %	0.561	1.152	0.196	0.281
CV_x, %	0.573	1.351	0.189	0.267

[a] *See* footnote a in Table 12.

Table 14. Statistical summary for determination of solids in cultured dairy products

Parameter[a]	Sour cream	Yogurt
AOAC Method **990.20**		
$\bar{x}$, %	26.02	12.96
S_o	0.061	0.039
Rapid microwave drying method		
$\bar{x}$, %	26.01	12.99
S_o	0.071	0.048
S_x	0.072	0.047
CV_o, %	0.275	0.366
CV_x, %	0.276	0.358

[a] *See* footnote a in Table 12.

Table 15. Statistical summary for determination of fat in cultured dairy products

Parameter[a]	Sour cream	Yogurt
AOAC Method **905.02**		
$\bar{x}$, %	18.42	3.25
S_o	0.113	0.040
NMR fat method		
$\bar{x}$, %	18.47	3.24
S_o	0.048	0.023
S_x	0.049	0.029
CV_o, %	0.260	0.705
CV_x, %	0.266	0.906

[a] *See* footnote a in Table 12.

be in the range appropriate for the corresponding sample type, as shown in Figure 1.

The drying temperature of the sample can have an effect on the moisture/solids determinations with very little effect on the fat determinations. Samples may not be completely dried if the appropriate drying temperature is not used.

Sample temperature does have a significant adverse effect on fat determinations. Samples should be cooled to 40°C before NMR analysis.

14 Quality Assurance

Keep all prepared samples in air- and watertight containers. Samples should be dried at the temperature and weight appropriate for the sample type and conditioned to 40°C before NMR analysis.

15 Comments

Overall, the 2 participating laboratories were pleased with the method because it provides a safe and rapid method of analyzing dairy products for moisture/solids and fat that compares favorably with the AOAC methods.

15.1 Peer Laboratory Comments

The CEM SMART Trac system offers improvements in the determinations of solids and fats in dairy products through increased speed of testing and improved accuracy of results and safety.

The NCSU dairy processing operation uses the Babcock Method (AOAC Method **989.04**) for the determination of fat in raw milk and heavy cream and in processed milks with reduced and high fat content. Ice cream and ice milk mixes are also produced and tested by using the Babcock Method. The SMART Trac system, when compared with the Babcock Method, has increased the efficiency of laboratory analysis during production, dramatically reducing the time for generation of in-process fat analysis from 25 to 4 min. Operator variability, which produced significant error with the Babcock Method, has been reduced to an insignificant level with the SMART Trac system.

Table 16. Statistical summary for determination of moisture in cheese products

Parameter[a]	Mozzarella	Swiss	Cheddar	Cream cheese
AOAC Method **926.08**				
$\bar{x}$, %	46.15	39.96	39.76	41.15
S_o	0.066	0.173	0.050	0.033
Rapid microwave drying method				
$\bar{x}$, %	46.08	39.90	36.72	41.10
S_o	0.131	0.090	0.120	0.087
S_x	0.138	0.144	0.128	0.092
CV_o, %	0.284	0.227	0.327	0.211
CV_x, %	0.299	0.360	0.348	0.223

[a] *See* footnote a in Table 12.

Table 17. Statistical summary for determination of fat in cheese products

Parameter[a]	Mozzarella	Swiss	Cheddar	Cream cheese
AOAC Method **933.05**				
$\bar{x}$, %	24.32	27.98	31.29	28.17
S_o	0.110	0.159	0.131	0.092
NMR fat method				
$\bar{x}$, %	24.37	27.96	31.31	28.19
S_o	0.091	0.145	0.130	0.053
S_x	0.088	0.144	0.125	0.053
CV_o, %	0.374	0.520	0.415	0.189
CV_x, %	0.362	0.514	0.399	0.188

[a] *See* footnote a in Table 12.

The elimination of spent reagents produced by the Babcock Method has substantially reduced hazardous waste generation. Safety-related issues have been minimized through the reduction of hazardous chemicals, glassware, and centrifuge operation.

For a university multiuse laboratory, the versatility and accuracy provided by the SMART Trac system for the determination of solids, fat, and moisture in milks, mixes, cheeses, butter, and meat products have expanded the scope of available analyses while minimizing variation among multiple users.

Attachment A: Ruggedness Testing

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

In milk Example 1 (Table A.1.), the weight of the sample was decreased to below the recommended parameter of 3–4 g. Results showed that sample size can have an effect on solids and fat determinations. A drying temperature of 105°C was inadequately maintained during the drying cycle, because of the sample size. Therefore, results for solids in the above samples ranged from 0.09 to 0.14% below the AOAC average of 11.79%. 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 in 2 of the samples. 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 0.10 to 0.14% above the AOAC average of 3.02% fat for 2 of the samples.

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

In milk Example 2 (Table A.2.), the drying temperature was decreased from the recommended 105° to 80°C. Results showed that drying temperature has an effect on solids determination (0.09% higher) and very little effect on fat determinations.

Table 18. Statistical summary for determination of solids in ice cream

Parameter[a]	Ice cream
AOAC Method **941.08**	
$\bar{x}$, %	37.95
S_o	0.087
Rapid microwave drying method	
$\bar{x}$, %	37.97
S_o	0.061
S_x	0.063
CV_o, %	0.161
CV_x, %	0.167

[a] *See* footnote a in Table 12.

Table 19. Statistical summary for determination of fat in ice cream

Parameter[a]	Ice cream
AOAC Method **952.06**	
$\bar{x}$, %	9.77
S_o	0.022
NMR fat method	
$\bar{x}$, %	9.76
S_o	0.031
S_x	0.034
CV_x, %	0.316
CV_x, %	0.351

[a] *See* footnote a in Table 12.

Table A.1. Test results for Example 1 milk sample 9[a]

Sample ID	Wt, g	Microwave, S, %	NMR, F, %	Microwave, °C	AOAC, S, %	AOAC, F, %
1	2.0049	11.65	3.00	105	11.79	3.02
2	1.2185	11.68	3.12	105		
3	1.3778	11.70	3.14	105		

[a] S = Solids; F = fat.

Table A.2. Test results for Example 2 milk sample 5[a]

Sample ID	Wt, g	Microwave, S, %	NMR, F, %	Microwave, °C	AOAC, S, %	AOAC, F, %
1	3.7666	10.05	1.00	80	9.91	0.97
2	4.0067	9.97	1.02	80		
3	3.7409	9.99	1.00	80		

[a] S = Solids; F = fat.

Table A.3. Test results for Example 3 milk sample 5[a]

Sample ID	Wt, g	Microwave, S, %	NMR, F, %	Microwave, °C	AOAC, M, %	AOAC, F, %
1	3.8394	9.87	0.97	120	9.91	0.97
2	3.7428	9.88	0.98	120		
3	3.8394	9.88	0.99	120		

[a] M = Moisture; F = fat.

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

In milk Example 3 (Table A.3.), the drying temperature was increased from the recommended 105°C to 120°C. Results showed that a higher drying temperature has very little effect on solids or fat determinations. However, samples with higher fat content may tend to brown or burn if run at temperatures higher than those recommended.

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

In heavy cream Example 4 (Table A.4.), the weight of the sample was decreased to below the recommended 2–2.5 g. Results showed that sample size can have an effect on solids and fat determinations. A drying temperature of 100°C was inadequately maintained during the drying cycle, because of the sample size. Therefore, results for solids in the samples averaged 0.08% higher than the AOAC average result of 45.57%. 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 in 2 of the samples. 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 hydrogen protons from water are not completely eliminated, these protons will be calculated as fat. This resulted in an average fat increase of 0.29% above the AOAC average of 39.93%.

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

Table A.4. Test results for Example 4 heavy cream[a]

Sample ID	Wt, g	Microwave, S, %	NMR, F, %	Microwave, °C	AOAC, S, %	AOAC, F, %
1	1.4612	45.65	40.10	100	45.57	39.93
2	1.4386	45.58	40.26	100		
3	1.0988	45.71	40.31	100		

[a] S = Solids; F = fat.

Table A.5. Test results for Example 5 heavy cream[a]

Sample ID	Wt, g	Microwave, S, %	NMR, F, %	Microwave, °C	AOAC, S, %	AOAC, F, %
1	3.7924	45.57	39.90	100	45.57	39.93
2	4.0953	45.61	39.63	100		
3	4.1566	45.43	39.57	100		

[a] S = Solids; F = fat.

Table A.6. Test results for Example 6 heavy cream[a]

Sample ID	Wt, g	Microwave, S, %	NMR, F, %	Microwave, °C	AOAC, S, %	AOAC, F, %
1	2.5615	45.61	39.65	100	45.57	39.93
2	2.7932	45.59	39.25	100		
3	2.2800	45.59	39.14	100		

[a] S = Solids; F = fat.

In heavy cream Example 5 (Table A.5.), the weight of the sample was increased to above the recommended 2–2.5 g. Results showed that sample size can have an effect on solids and fat determinations. Because there was some browning of the samples, this browning effect caused more variability in the overall results, and the fat content was somewhat lower in samples 2 and 3.

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

In heavy cream Example 6 (Table A.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 100°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 39.93% fat.