Today and the Future: Mid Infrared Testing for Dairy Herd Management and Dairy Product Analysis

D. M. Barbano[1], C. Melilli[1], B. Margolies[1], H. M. Dann[2], and R. J. Grant[2]

[1] Department of Food Science Cornell University, Ithaca, NY

[2] Miner Agricultural Research Institute, Chazy, NY

NYS Cheese Manufacturers' Association

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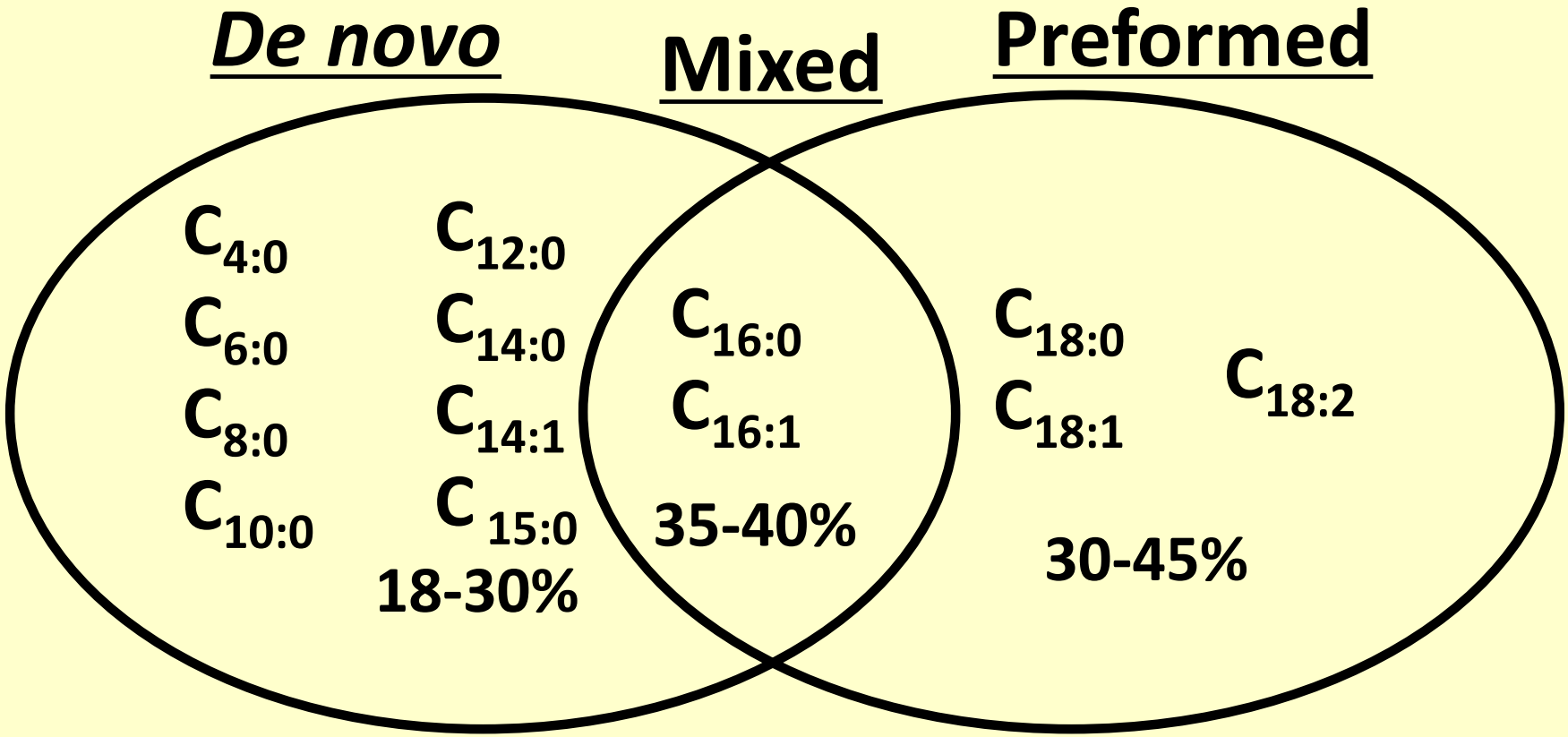
Milk Analysis for Dairy Herd Management

- **Past: Where we have been?**
- **Present: Where we are?**
- **Future: Where we are going?**

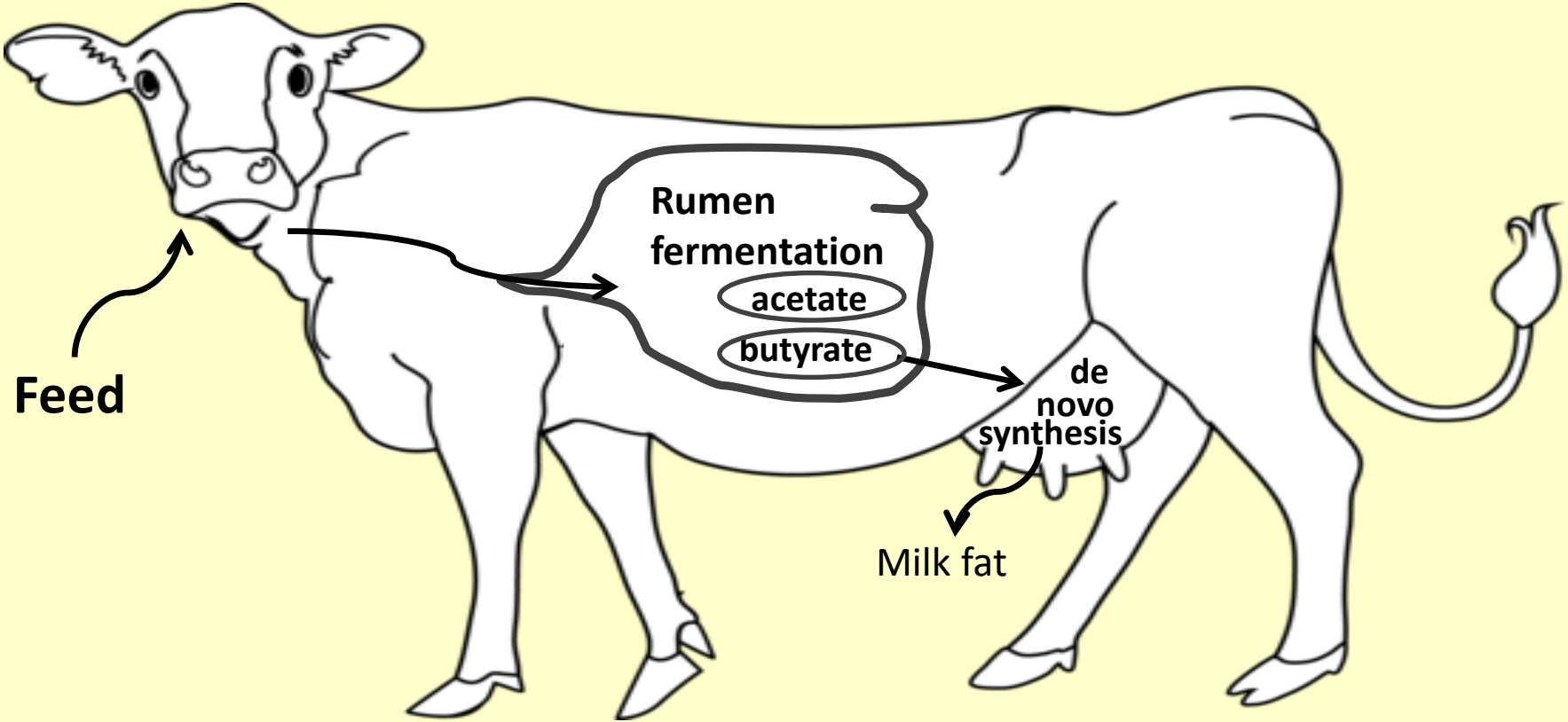
Milk Analysis for Dairy Herd Management

- **Past: Where we have been?**
  - **2014: CNC meeting first presentation on IR milk fatty acid analysis for dairy herd management. Introduction of a rapid method to measure de novo, mixed origin, and preformed fatty acids and fatty chain length and unsaturation.**
  - **First data from the St Albans Cooperative showing a strong positive correlation between bulk tank milk fat and protein test and de novo fatty acid concentration.**

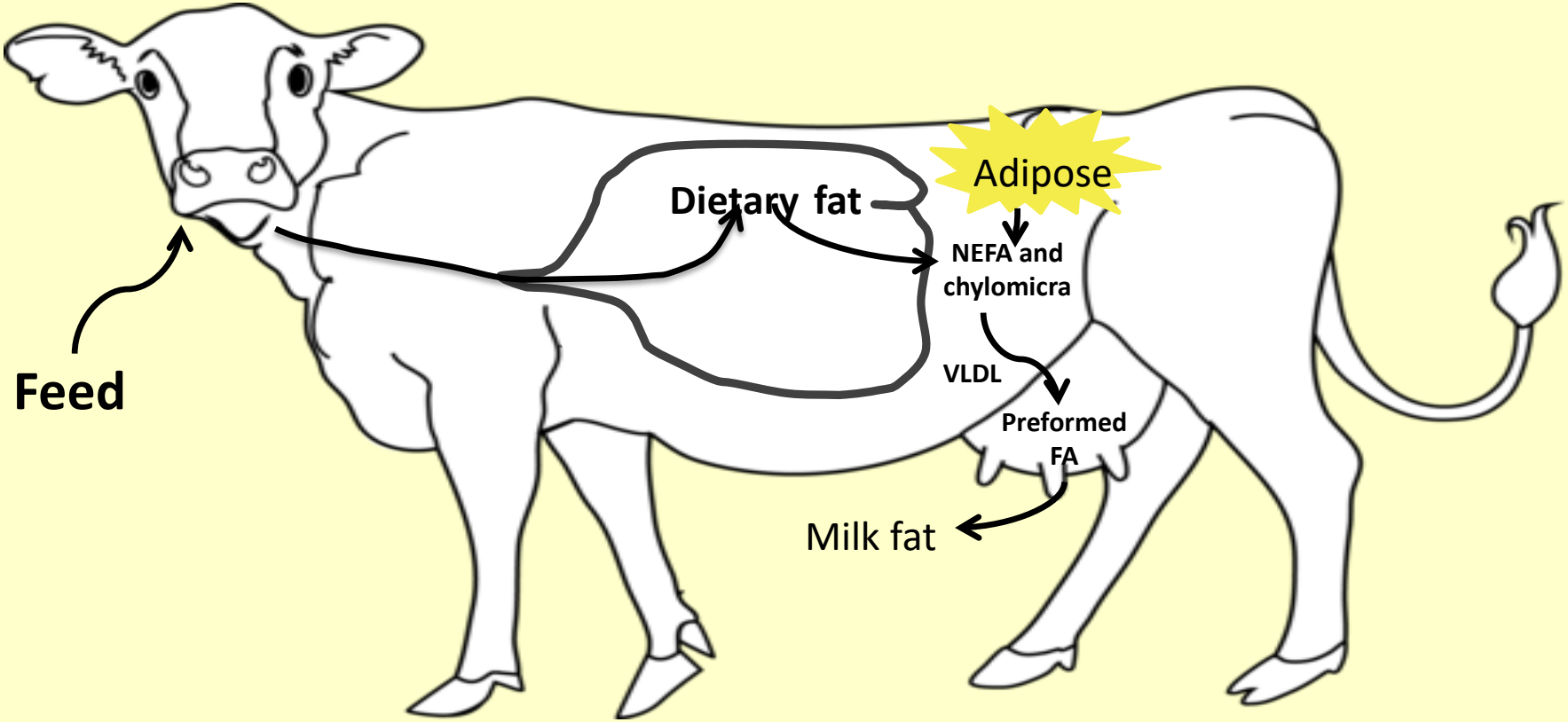
Milk Fatty Acid Origin



De novo Fatty Acid Synthesis



Preformed Fatty Acids



Milk Analysis for Dairy Herd Management

- **Past: Where we have been?**
  - **2016: CNC meeting: Introduction to the "Cow of Interest" and the quest for real-time dairy herd management detailed milk analysis began.**
    - **Results were presented for two 40 herd field studies showing herd management factors that influence de novo fatty acids and fat and protein tests.**
    - **Graphs of relationship between milk fatty acids and bulk tank fat and protein tests for Holsteins.**

Main Conclusions from Bulk Tank Milks

The strongest correlation between milk fatty acid composition and the concentration of fat and protein in milk was with *de novo* fatty acid production.

De novo fatty acid level seems to be barometer of rumen health and proper rumen function.

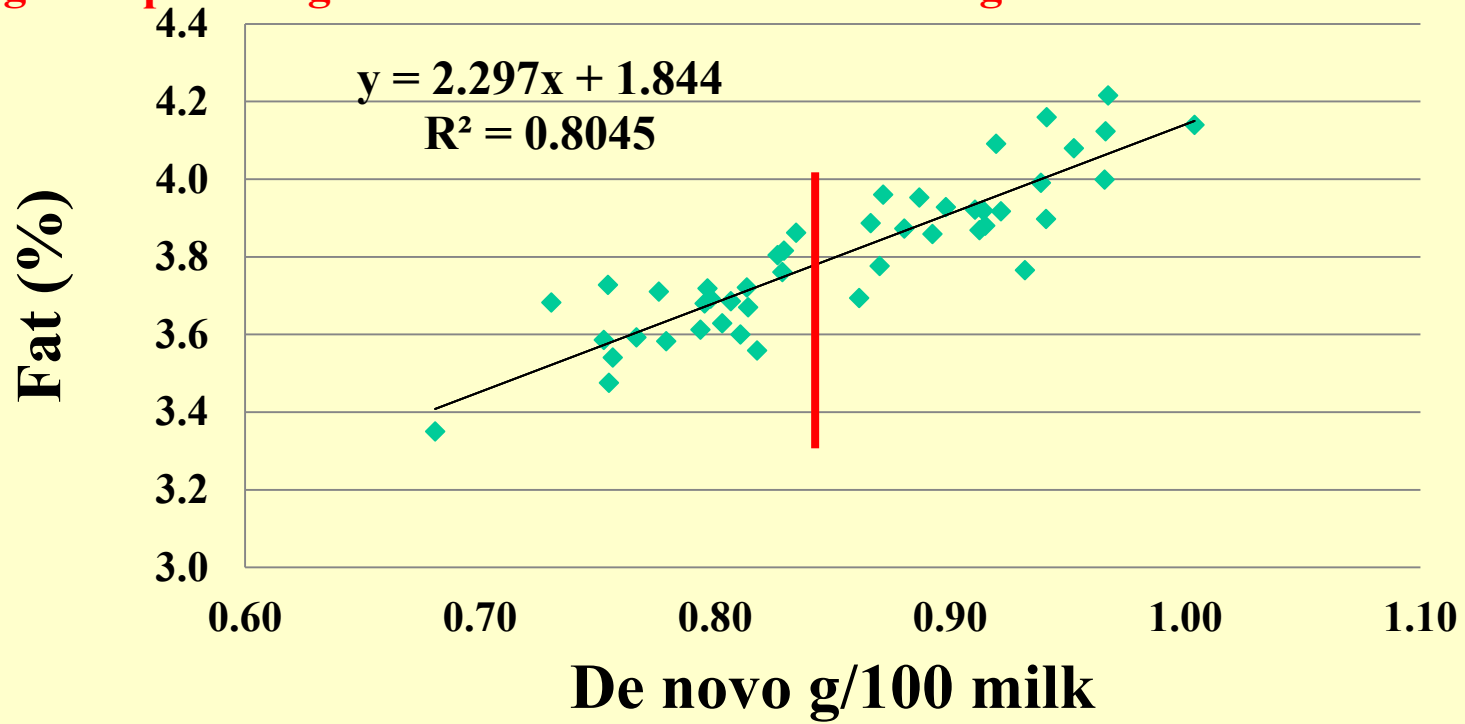
Thus, feeding and farm management strategies that produce an increase in synthesis of *de novo* fatty acids may produce an increase milk fat and milk protein percentage and possibly output of fat and protein per cow per day.

In the field studies, over crowding showed up clearly as a factor causing low de novo fatty acids, lower bulk tank fat and protein tests.

40 Holstein Farms 2015

St Albans - Fat

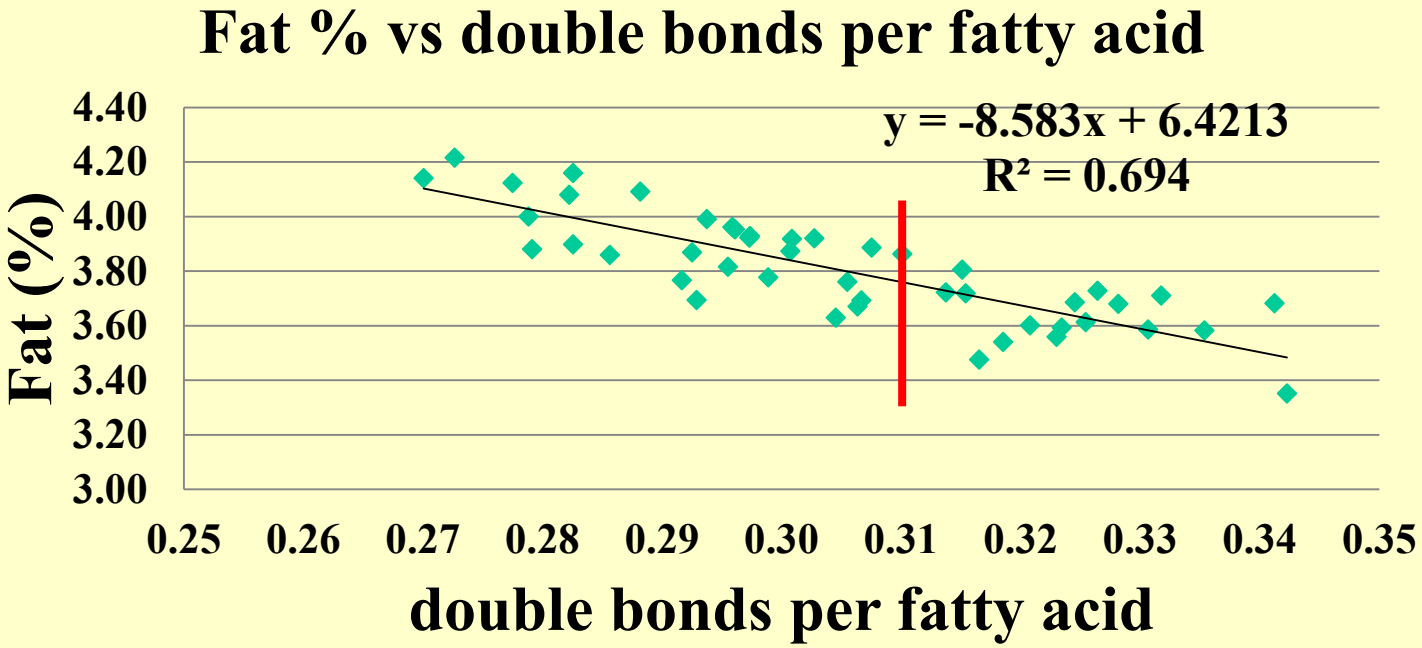
If you want a fat test > 3.75% fat in bulk tank with Holsteins, then the de novo fatty acids in grams per 100 grams of milk needs to be > 0.85 g/100 milk



40 Holstein Farms 2015

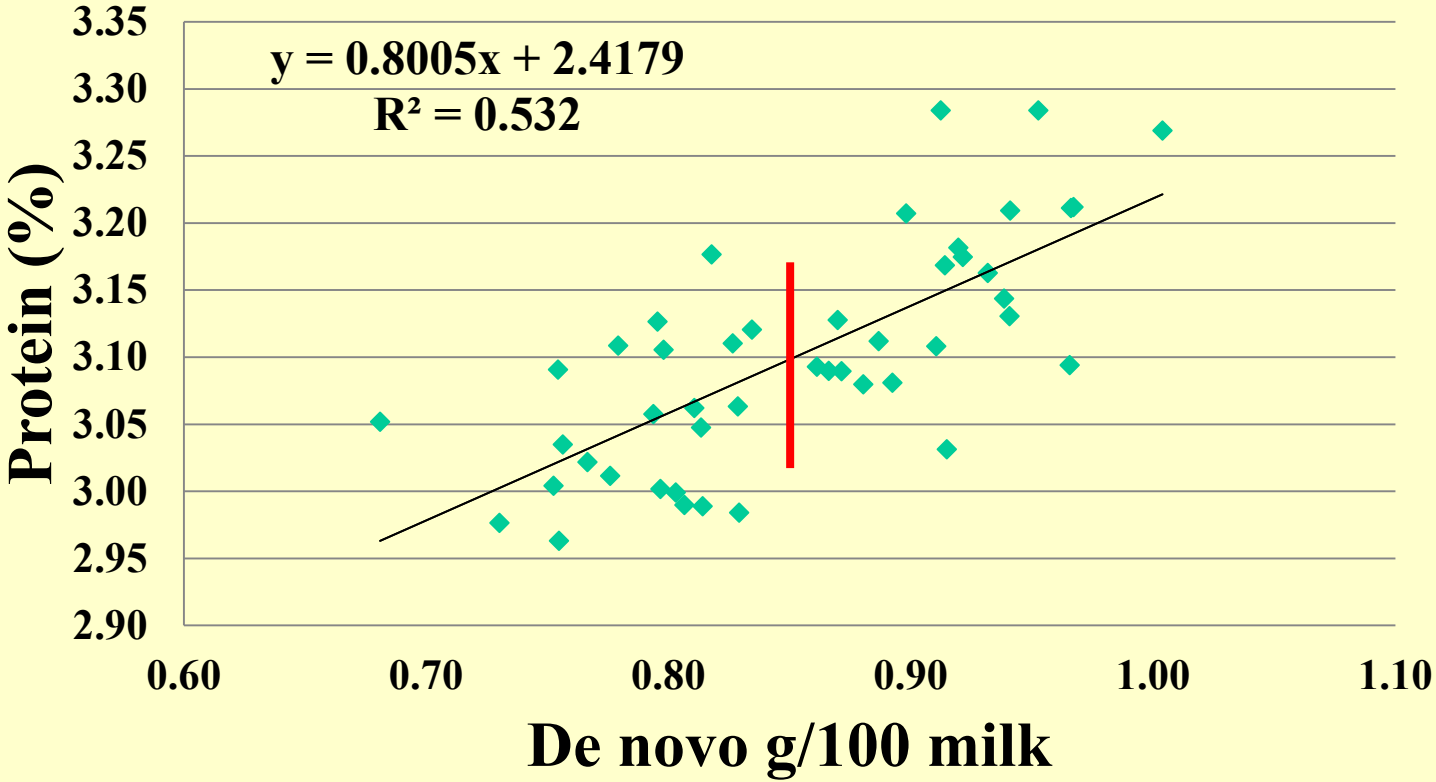
St Albans - Fat

If you want a fat test > 3.75% fat in bulk tank with Holsteins, then the double bonds per fatty acid in milk fat needs to < 0.31.



40 Holstein Farms 2015

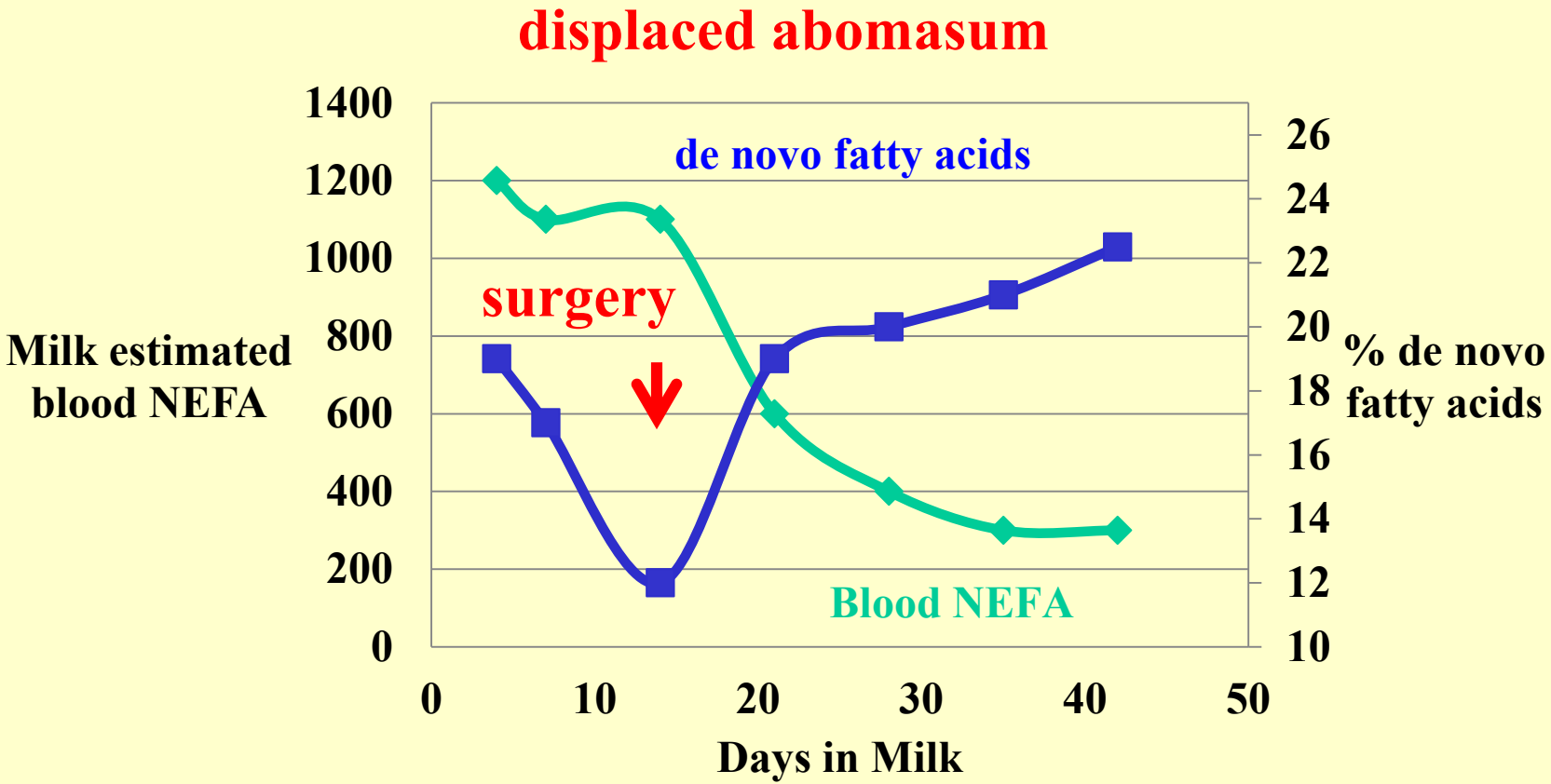
St Albans - Protein



Milk Analysis for Dairy Herd Management

- **Past: Where we have been.**
  - **2016: CNC meeting: Introduction to the "Cow of Interest" and the beginning of quest for real-time dairy herd management milk analysis.**
    - Results presented for two 40 herd field studies showing herd management factors that influence de novo fatty acids and fat and protein tests.
    - Graphs of relationship between milk fatty acids and bulk tank fat and protein tests for Holsteins.
    - **First introduction of milk estimated blood NEFA testing by mid-infrared milk analysis.**

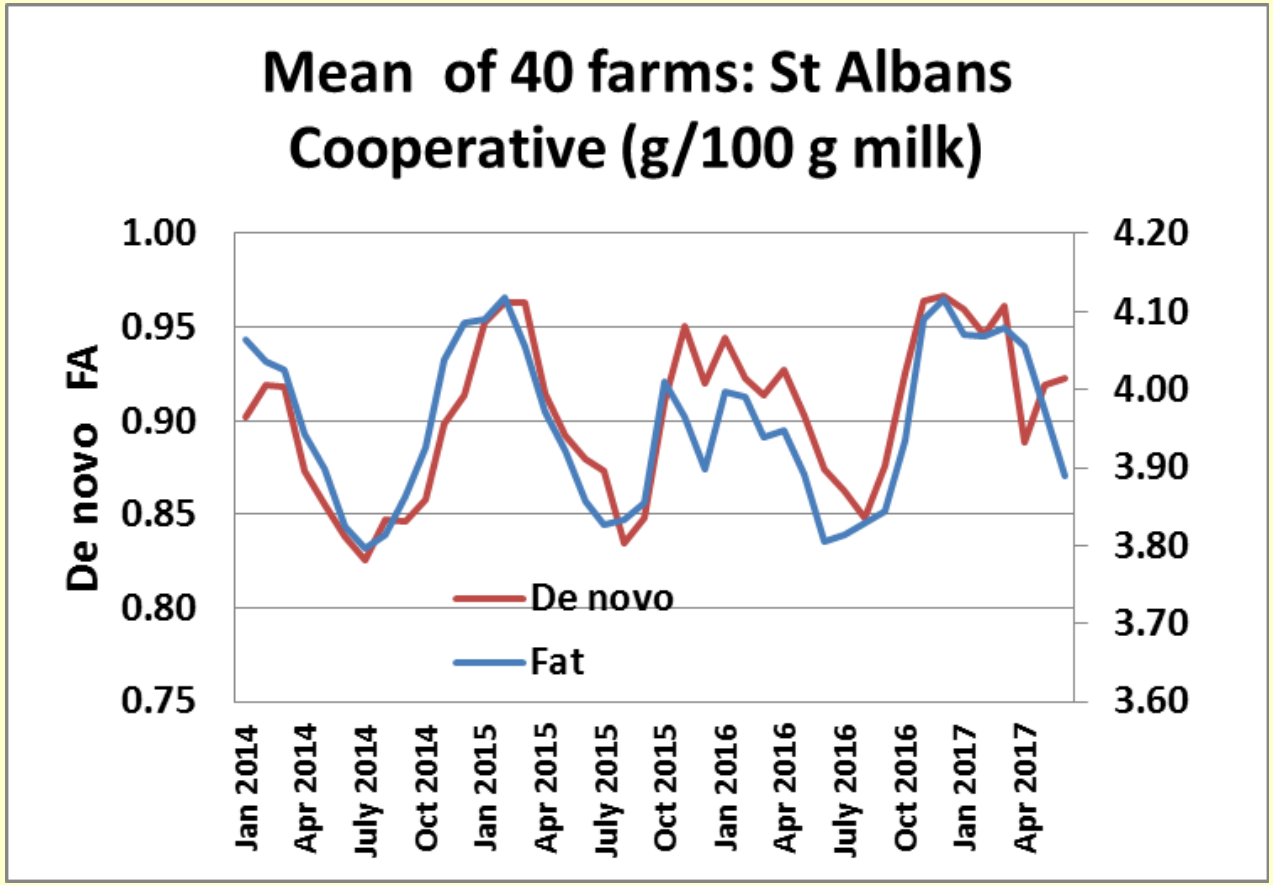
Sample Individual Cow Health Data



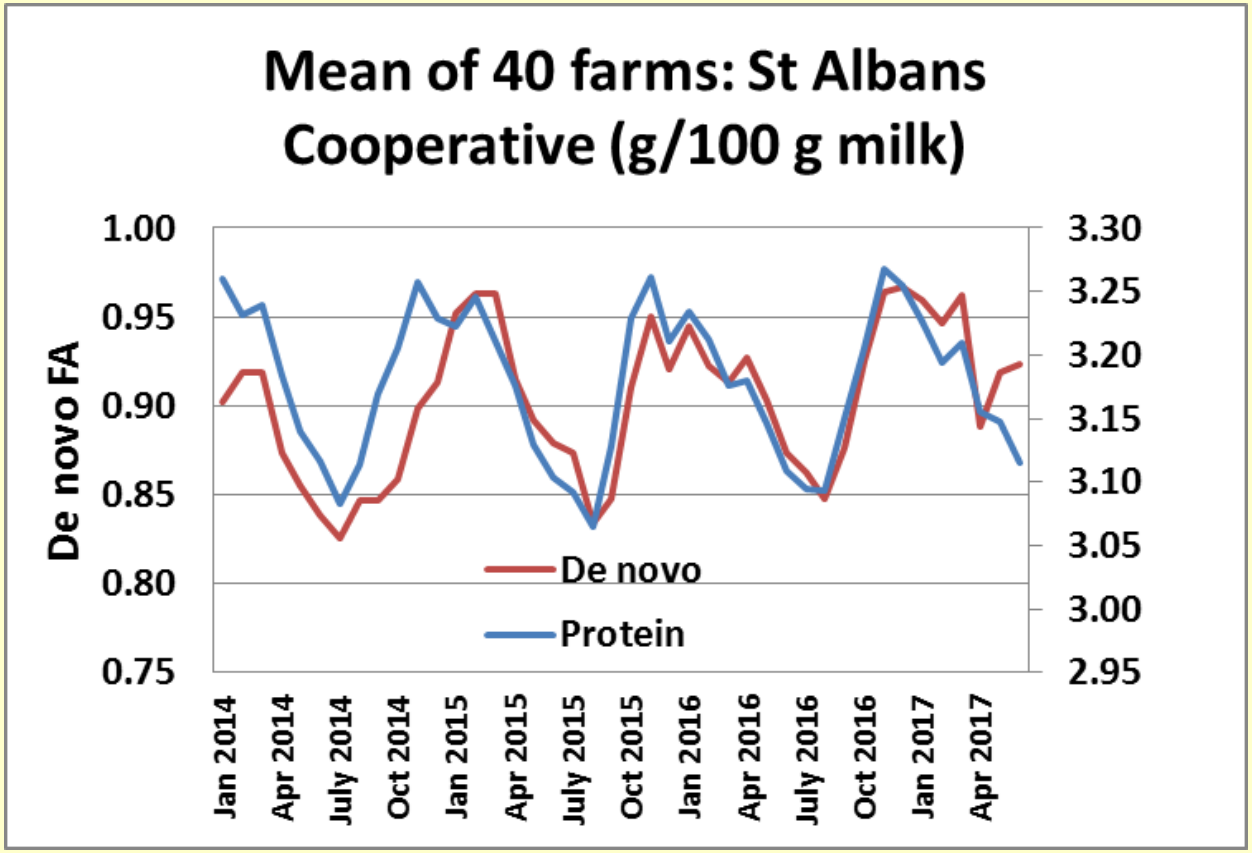
Milk Analysis for Dairy Herd Management

- **Past: Where we have been?**
  - **2017: CNC meeting: new data (herd level and individual cow level)**
  - **milk fatty acids: relation to seasonality of fat and protein**

Seasonality of Bulk Tank Milk - Fat



Seasonality of Bulk Tank Milk – Protein



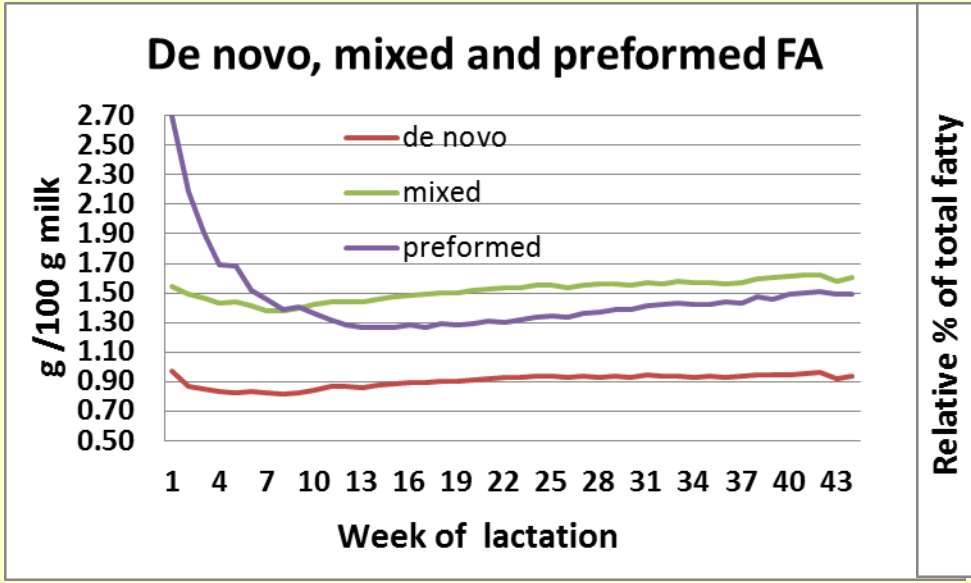
Milk Analysis for Dairy Herd Management

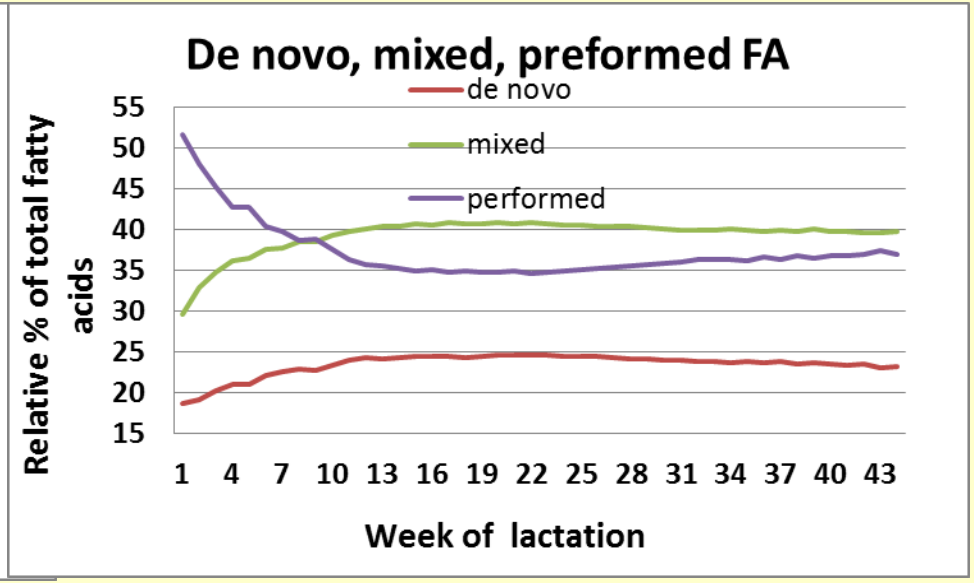
- **Past: Where we have been?**
  - **2017: CNC meeting: new data (herd level and individual cow level)**
  - **milk fatty acids: relation to seasonality of fat and protein**
  - **167 farm study of milk fatty acid from herds distributed all over the US – basically the same relationships between de novo, mixed, and preformed fatty acid with fat and protein test that we had seen in the Northeast**

Milk Analysis for Dairy Herd Management

- **Past: Where we have been?**
  - **2017: CNC meeting: new data (herd level and individual cow level)**
  - **milk fatty acids: relation to seasonality of fat and protein**
  - **167 farm study of milk fatty acid from herds**
  - **Stage of lactation effect on milk fatty composition**

Stage of Lactation – Holstein





Herd producing an average of about 92 lb (41.77 kg) per cow per day on TMR feeding system.

Milk Analysis for Dairy Herd Management

- **Current 2019: Where are we?**
  - **Instruments testing milk for de novo, mixed performed, chain length, and unsaturation (total of 14 Delta Instruments)**
    - **St Albans Cooperative, AgriMark Cooperative, and Cayuga Marketing Cooperative (1 instrument each), Merieux-Siliker, Salida CA (2 instruments)**
    - **Sterns County and Zumbrota DHIA Labs, Minnesota (2 instruments each), ADM DHIA Lab, Clovis, New Mexico**
    - **Cornell University, North Carolina State, Miner Institute, and Texas Federal Milk Market Laboratory**

Milk Analysis for Dairy Herd Management

- **Current 2019: Where are we?**
  - **Calibration Samples**
    - **A 14 sample sets of milk samples for calibration of mid-infrared milk analyzers are being produced 13 times a year at Cornell.**
    - **All Delta Instruments are calibrated with these samples**
    - **These samples are available to calibrate other brands of infrared milk analyzers when those instruments have herd management milk fatty acid models available.**

Milk Analysis for Dairy Herd Management

Current 2019: Where are we?

- **MIR Milk Testing Equipment Manufacturers (there are 3)**
  - **DELTA: Milk fatty acid models for de novo, mixed, and preformed fatty acids, fatty chain length, fatty acid unsaturation (double bonds per fatty acid), and milk estimated blood NEFA are available. Methods have been validated and results published, except for blood NEFA. All Delta Instruments in the US and 4 in the Netherlands are calibrated with samples from Cornell.**
  - **FOSS: Foss announced that they will release prediction models for de novo, mixed, and preformed fatty acids in November 2018. At the present time there are no Foss instruments calibrated with calibration samples produced at Cornell. Foss has not indicated that they are going to release chain length, double bonds per FA, or milk estimated blood NEFA models.**
  - **BENTLEY: Bentley has made no announcements on any of these models.**

Milk Analysis for Dairy Herd Management

Current 2019: Where are we?

- **How well do results agree among instruments on fatty acids?**

If the milk fatty acid parameters on each infrared milk analyzer are not calibrated with reference samples, then instruments will not agree very well. Calibration is needed if you want accurate fatty acid results.

In 2018, we did the first multi-lab comparisons of instrument results for milk fatty acid testing.

Multi-lab Validation of Results (Delta Instruments)

Instruments: **A mixture of 9 Delta FTA's and Delta Combi's**

Calibration:

1. **De novo, mixed, and preformed calibrated every 4 weeks with Cornell calibration samples.**

2. **Chain length and double bonds/fatty acid calibrated once per year. That frequency will increase in the future.**

Validation: **Individual farm milks (8) from different regions of the US. None of these milks are part of the PLS model development samples or calibration adjustment.**

Multi-lab Comparison of Results (Delta Instruments)

	de novo Reference	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6	Lab 7	Lab 8	lab 9
1	0.8991	0.860	0.862	0.874	0.860	0.870	0.894	0.920	0.890	0.890
2	0.8484	0.820	0.810	0.838	0.820	0.822	0.828	0.840	0.820	0.830
3	0.7209	0.720	0.732	0.743	0.730	0.715	0.748	0.750	0.720	0.720
4	0.8179	0.810	0.811	0.819	0.800	0.789	0.804	0.840	0.800	0.830
5	0.7540	0.720	0.729	0.754	0.750	0.731	0.740	0.740	0.730	0.740
6	0.9635	0.930	0.937	0.964	0.940	0.933	0.953	0.950	0.930	0.950
7	0.7910	0.810	0.798	0.803	0.820	0.796	0.804	0.840	0.810	0.810
8	1.3033	1.220	1.224	1.252	1.240	1.234	1.220	1.240	1.230	1.250
	0.887 Mean	0.861	0.863	0.881	0.870	0.861	0.874	0.890	0.866	0.878
	MD	-0.026	-0.024	-0.006	-0.017	-0.026	-0.013	0.003	-0.021	-0.010
	SDD	0.031	0.029	0.023	0.029	0.022	0.032	0.035	0.027	0.022

Multi-lab Comparison of Results (Delta Instruments)

	Mixed Reference		Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6	Lab 7	Lab 8	lab 9
1	1.3295		1.480	1.445	1.438	1.420	1.419	1.471	1.480	1.490	1.460
2	1.1070		1.220	1.170	1.162	1.180	1.163	1.168	1.170	1.220	1.200
3	0.9481		1.050	1.042	1.041	1.010	0.996	1.035	1.030	1.060	1.040
4	1.1063		1.240	1.232	1.208	1.210	1.158	1.186	1.260	1.260	1.230
5	1.0260		1.100	1.098	1.103	1.100	1.049	1.078	1.070	1.100	1.080
6	1.3599		1.490	1.455	1.472	1.440	1.414	1.482	1.440	1.450	1.460
7	1.3105		1.330	1.261	1.267	1.300	1.227	1.225	1.290	1.300	1.280
8	1.5220		1.660	1.625	1.648	1.640	1.580	1.630	1.650	1.680	1.620
Mean	1.2136		1.321	1.291	1.292	1.288	1.251	1.285	1.299	1.320	1.296
		MD	0.108	0.077	0.079	0.074	0.037	0.071	0.085	0.106	0.083
		SDD	0.043	0.055	0.054	0.039	0.052	0.070	0.059	0.057	0.051

Multi-lab Comparison of Results (Delta Instruments)

	Preformed Reference		Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6	Lab 7	Lab 8	lab 9
1	1.4988		1.370	1.419	1.426	1.480	1.451	1.405	1.410	1.380	1.390
2	1.4982		1.390	1.479	1.492	1.450	1.468	1.484	1.470	1.400	1.440
3	1.5371		1.410	1.438	1.427	1.460	1.480	1.458	1.470	1.390	1.490
4	1.5798		1.440	1.471	1.544	1.510	1.561	1.563	1.430	1.400	1.490
5	1.4224		1.370	1.371	1.370	1.380	1.438	1.429	1.440	1.350	1.460
6	1.7128		1.560	1.635	1.606	1.690	1.677	1.622	1.660	1.620	1.660
7	1.3716		1.310	1.414	1.434	1.370	1.442	1.477	1.410	1.340	1.400
8	1.7819		1.690	1.739	1.695	1.750	1.784	1.774	1.730	1.650	1.760
Mean	1.5503		1.443	1.496	1.499	1.511	1.538	1.526	1.503	1.441	1.511
		MD	-0.108	-0.055	-0.051	-0.039	-0.013	-0.024	-0.048	-0.109	-0.039
		SDD	0.036	0.049	0.058	0.026	0.041	0.066	0.059	0.046	0.052

Multi-lab Comparison of Results (Delta Instruments)

	CL Reference		Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6	Lab 7	Lab 8	Lab 9
1	14.7434		14.63	14.76	14.80	14.72	14.65	14.65	14.67	14.76	14.76
2	14.7429		14.64	14.78	14.79	14.69	14.61	14.69	14.71	14.77	14.78
3	14.8803		14.75	14.85	14.91	14.83	14.76	14.73	14.82	14.88	14.88
4	14.7634		14.64	14.72	14.76	14.68	14.64	14.65	14.64	14.77	14.73
5	14.7897		14.67	14.75	14.78	14.71	14.66	14.67	14.73	14.76	14.78
6	14.8062		14.61	14.74	14.77	14.69	14.63	14.61	14.70	14.77	14.77
7	14.7861		14.67	14.79	14.83	14.73	14.68	14.69	14.73	14.76	14.82
8	14.4498		14.32	14.38	14.46	14.37	14.25	14.32	14.32	14.43	14.47
Mean	14.7452		14.616	14.721	14.763	14.678	14.610	14.626	14.665	14.738	14.749
		MD	-0.129	-0.024	0.017	-0.068	-0.135	-0.119	-0.080	-0.008	0.004
		SDD	0.029	0.039	0.032	0.028	0.037	0.043	0.035	0.023	0.028

Multi-lab Comparison of Results (Delta Instruments)

	DB/FA Reference		Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6	Lab 7	Lab 8	Lab 9
1	0.2651		0.260	0.275	0.289	0.270	0.281	0.277	0.260	0.290	0.270
2	0.2974		0.290	0.308	0.318	0.288	0.301	0.310	0.300	0.310	0.300
3	0.3405		0.320	0.329	0.344	0.326	0.334	0.328	0.330	0.340	0.340
4	0.2987		0.290	0.299	0.311	0.291	0.307	0.309	0.290	0.310	0.300
5	0.3237		0.310	0.316	0.325	0.305	0.319	0.321	0.310	0.320	0.320
6	0.3065		0.290	0.299	0.310	0.286	0.301	0.293	0.300	0.310	0.300
7	0.2841		0.280	0.302	0.311	0.282	0.302	0.306	0.290	0.300	0.300
8	0.2649		0.250	0.255	0.273	0.245	0.259	0.268	0.250	0.260	0.250
Mean	0.2976		0.286	0.298	0.310	0.287	0.301	0.302	0.291	0.305	0.298
		MD	-0.011	0.000	0.013	-0.011	0.003	0.004	-0.006	0.007	0.000
		SDD	0.006	0.011	0.010	0.009	0.010	0.013	0.007	0.010	0.009

Milk Analysis for Dairy Herd Management

- **Future: Where we are going?**
  - **De novo, mixed, preformed, chain length and double bonds per fatty acid graphs for Jersey cattle.**
  - **Improved milk fatty acid analysis PLS models (2nd generation).**
  - **More milk estimated blood metrics (i.e., haptiglobin).**
  - **Milk fat and protein quality metrics.**
  - **In-line on-farm FT-MIR optics (high power laser FT MIR).**

Mid-infrared (MIR) analysis of Cheddar cheese and other dairy products

1. Background on cheese yield estimation and the importance of accurate analytical results on milk and cheese.
2. Development of a rapid MIR method for cheese analysis.

Metrics of Cheese Yield Performance

J. Dairy Sci in press
https://doi.org/10.3168/jds.2016-12295

Effect of uncertainty in composition and weight measures in control of cheese yield and fat loss in large cheese factories

Brenda Margolies, Michael C. Adams, Joice Pranata, Kathleen Gondoutomo, and David M. Barbano[1]
Northeast Dairy Foods Research Center, Department of Food Science, Cornell University, Ithaca, NY 14853

Uncertainty in the accuracy of milk and cheese composition were identified as important factors that influence the ability to manage business profitability and consistency of cheese quality.

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The uncertainty in cheese composition has a large impact on controlling and managing for profitability. In analytical performance evaluations of commercial cheese quality assurance laboratories, we found that analytical uncertainty was typically a bias as large as 0.5% on fat and moisture.

Metrics of Cheese Yield Performance

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The impact of having a high bias of 0.5% moisture or fat will produce a missed cheese yield opportunity of 484 kg of cheese per day in a plant that processes 940,000 kg (i.e., 2.07 million pounds) of milk per day.

Cheese Composition: testing options

Classical chemistry (slow but accurate if done correctly)

- **Fat - Babcock, Gerber, ether extraction**
- **Protein – Kjeldahl**
- **Moisture – forced air oven or vacuum oven**
- **Salt – Volhard silver nitrate titration or chloridometer type salt meter**

Cheese Composition: QC routine testing method options

NIR Cheese Analysis (rapid and simple sample preparation)

- **Parameters measured: fat, moisture, and salt (sometimes protein)**
- **Local NIR calibration (*very difficult and extensive calibration required for each cheese type - requires several hundred samples for each cheese type in each factory*)**
- **Standard Error of Prediction (SEP) for NIR of cheese components are in the range of 0.2 to 0.4%.**

Cheese Composition: testing options

MIR Cheese Analysis

Goal to improve accuracy (i.e., develop a more simple global calibration for Cheddar cheese and achieve a lower SEP than with NIR)

- **Parameters measured:** fat, protein, moisture, and salt.
- **Instrument**: Delta FTA (Delta Instruments, Netherlands)
- **Sample Preparation**: Blend the cheese with a ***cheese dissolver solution*** and analyze the cheese on the same MIR analyzer used for milk and whey analysis.
- **Calibration:** a set of liquid cheese standards (similar to calibration done for MIR milk testing)

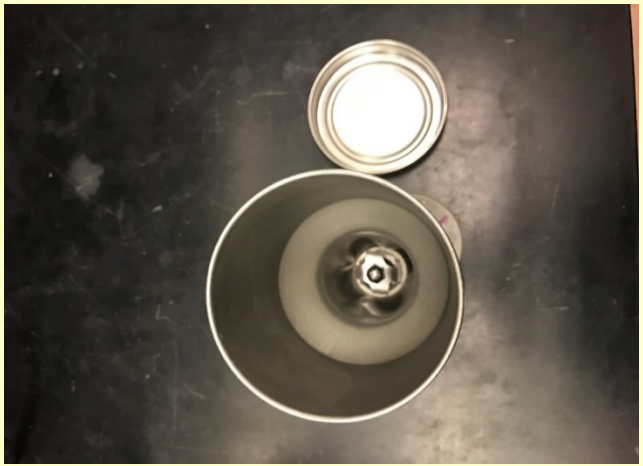
Materials and Methods: Experimental Design

- **34 full-fat annatto colored cheddar cheeses were collected from one factory and were used for calibration of the MIR.**
- **36 full-fat annatto colored cheddar cheeses were collected from 4 factories and used for validation of the MIR.**
- **All cheeses were analyzed by NIR and the coulometric method in each factory were produced.**
- **All cheeses were analyzed by reference chemistry (fat: ether extraction, moisture: oven drying, protein: Kjeldahl, and salt: silver nitrate titration) and MIR at Cornell.**

Cheese Composition: Sample Preparation

MIR Cheese Analysis Method:

- **Dissolver was composed of pentasodium triphosphate ($Na_5P_3O_{10}$) and disodium metasilicate ($Na_2Si_2O_3$).**
- **Dissolver was warmed and 81 g of 65°C solver was weighed and added to the stainless steel Eberbach blender jar**



Cheese Composition: Sample Preparation

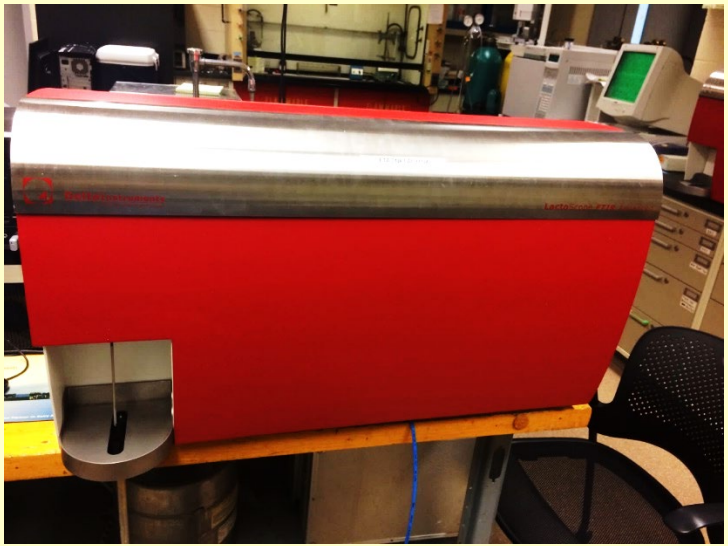
MIR Cheese Analysis Method:

1. **Warm cheese dissolver solution to 65°C**
2. **Weigh 81 g of 65°C dissolver and add to stainless steel Eberbach blender jar**
3. **Weigh 9 g of blended cheese (3 to 4 mm pieces) and add to the blender jar containing the dissolver**
4. **Blend at low speed for 15 seconds and then increase to high speed for 45 seconds.**
5. **Pour the liquid into a 4 oz plastic snap lid vial and place into 40°C water bath.**
6. **After 20 to 30 minutes test on the Delta FTA like a milk sample.**

Cheese Composition: Mid-infrared

MIR Cheese Analysis

Instrument: Delta FTA



Cheese Composition: NIR vs MIR results

Moisture

Factory	MIR	Reference	Difference	NIR	Reference	Difference
1	36.91	36.99	-0.08	36.99	36.99	0.00
2	37.50	37.37	0.13	37.22	37.37	-0.15
3	37.11	37.12	-0.01	37.32	37.12	0.20
4	36.15	36.15	0.00	36.15	36.15	0.00
Mean	36.92	36.91	0.01	36.92	36.91	0.01
SDD			0.09			0.14

Cheese Composition: NIR vs MIR results

	Fat					
Factory	MIR (Fat A)	Reference	Difference	NIR	Reference	Difference
1	33.81	34.06	-0.25	33.06	34.06	-0.99
2	33.73	33.75	-0.02	33.50	33.75	-0.25
3	33.43	33.47	-0.04	33.26	33.47	-0.21
4	34.61	34.65	-0.04	34.57	34.65	-0.04
Mean	33.89	33.98	-0.09	33.60	33.98	-0.37
SDD			0.11			0.42

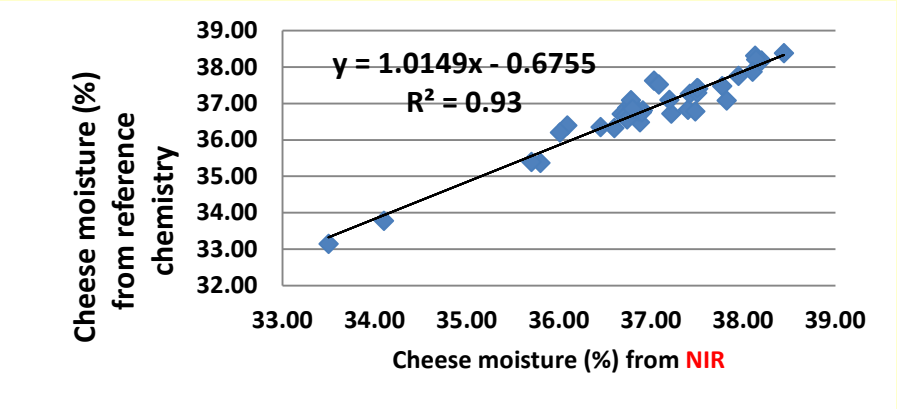
Cheese Composition: NIR vs MIR results

Salt

Factory	MIR	Reference	Difference	Coulometric	Reference	Difference
1	1.61	1.66	-0.04	1.72	1.66	0.06
2	1.59	1.69	-0.10	1.82	1.69	0.13
3	1.75	1.85	-0.11	1.76	1.85	-0.10
4	1.76	1.89	-0.12	1.73	1.89	-0.16
Mean	1.68	1.77	-0.09	1.75	1.77	-0.02
SDD			0.04			0.14

Results: Cheese Moisture

A- NIR



NIR Moisture vs. Reference Chemistry

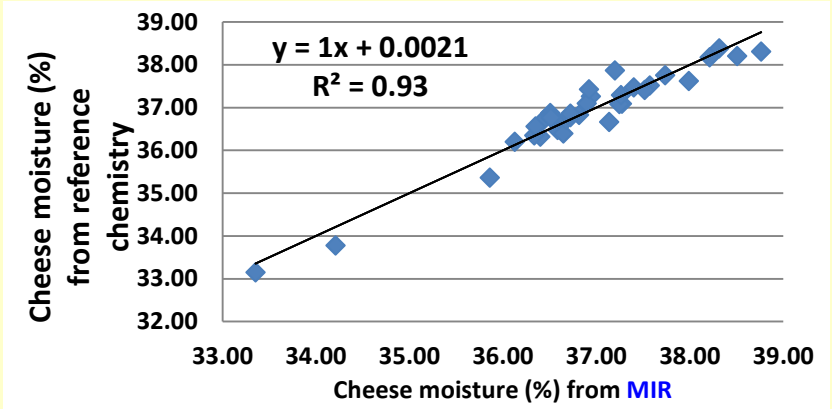
MD = 0.13

SDD = 0.29

CV = 0.79

SEP = 0.30

B - MIR



MIR Moisture vs. Reference Chemistry

MD = 0.00

SDD = 0.28

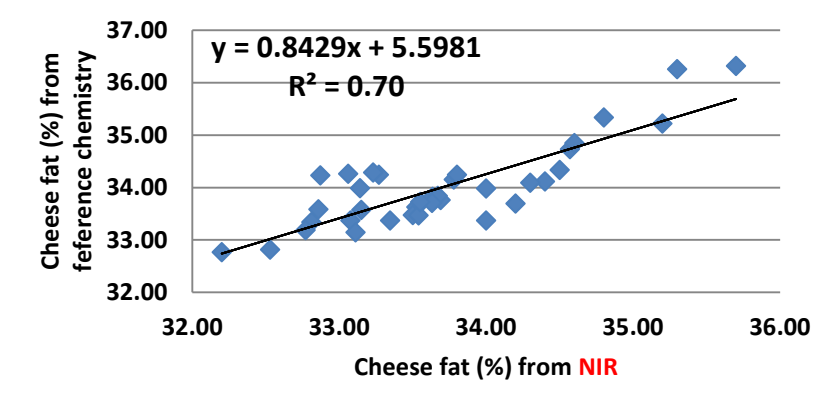
CV = 0.76

SEP = 0.28

NIR and MIR similar for accuracy for Moisture

Results: Cheese Fat

A- NIR



NIR Fat vs. Reference Chemistry

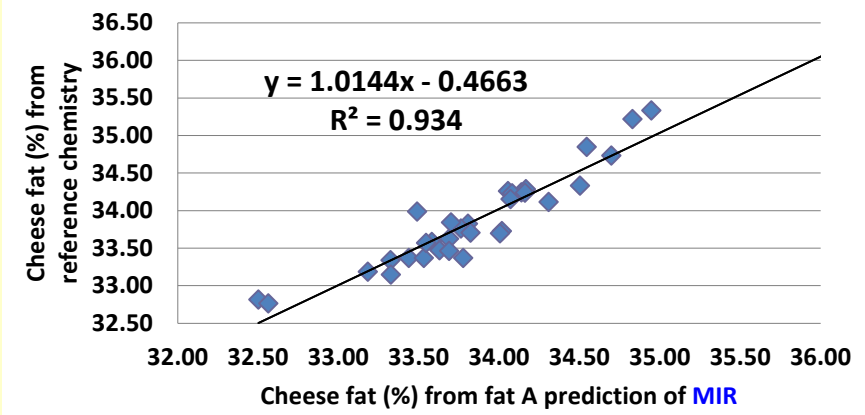
MD = -0.30

SDD = 0.44

CV = 1.29

SEP = 0.44

B - MIR



MIR Fat A vs. Reference Chemistry

MD = -0.07

SDD = 0.23

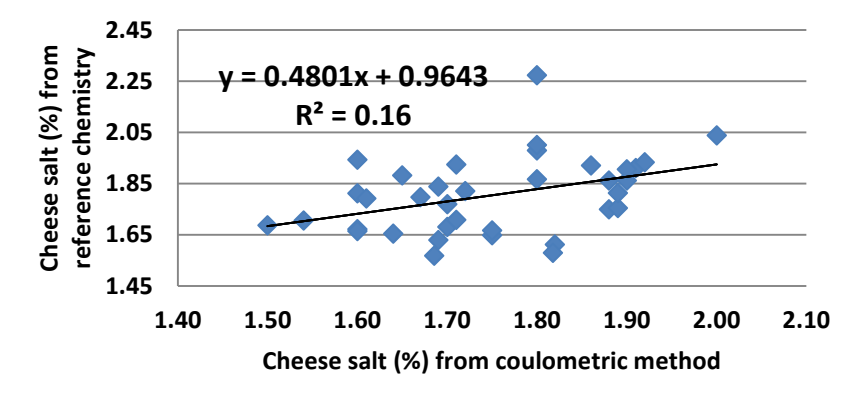
CV = 0.67

SEP = 0.23

MIR better accuracy for Fat than NIR

Results: Cheese Salt

A- NIR/Coulometric



Coulometric Salt vs. Reference Chemistry

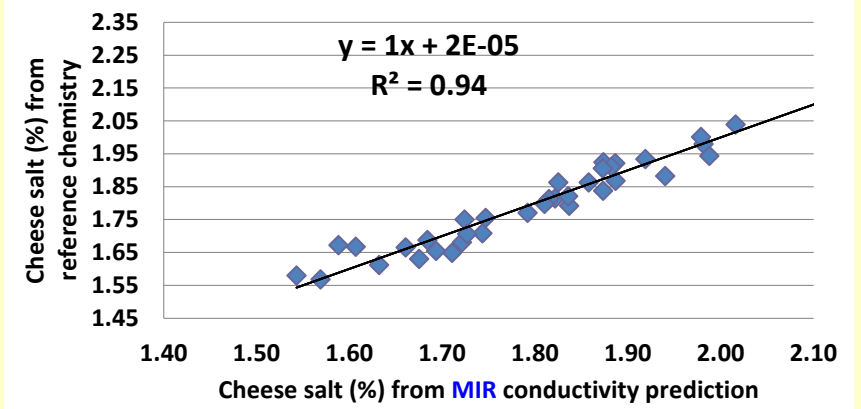
MD = -0.055

SDD = -0.137

CV = 7.590

SEP = 0.139

B – MIR - conductivity



MIR Salt vs. Reference Chemistry

MD = -0.100

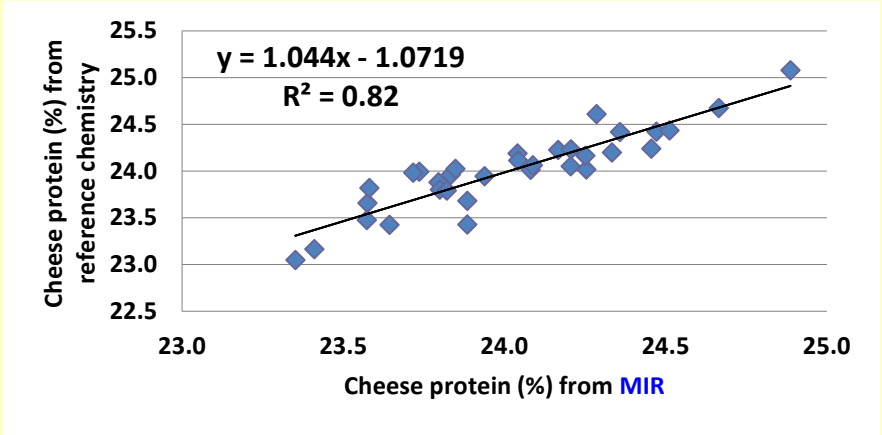
SDD = 0.035

CV = 1.950

SEP = 0.036

MIR/conductivity had better accuracy for Salt than NIR/coulometric method

Results: Cheese Protein



MIR Protein vs. Reference Chemistry

MD = -0.09

SDD = 0.23

CV = 0.94

SEP = 0.19

Conclusions

1. **Standard Error of Prediction (SEP) on moisture was as good or better than NIR analysis of cheese.**
2. **SEP for fat and salt by MIR were better than those reported for NIR analysis of cheese.**
3. **SEP for protein indicates that MIR analysis could be used for cheese protein determination.**
4. **The MIR calibration developed based one factory's colored Cheddar cheese was valid across several manufacturing plants.**
5. **A liquid cheese calibration sample set (8 samples) needs to be developed.**

Future Work

- **Once we have a routine method for testing Cheddar cheese composition using MIR, then we will focus on methodology to predict the aging characteristics of Cheddar cheese based on 2 or 3 testing dates during the first month of cheese aging.**
- **The approach for Cheddar cheese analysis will be tested on Mozzarella cheese, cottage cheese, cream cheese, and sour cream.**
- **An approach to test nonfat dry milk powder composition using reconstituted liquid samples is being developed with the goal of measuring moisture, fat, protein, lactose, and heat denaturation of whey protein in the milk powder (i.e., undenatured WPN).**

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Questions?