

**FMMA / Cornell University
Modified Milk Calibration
Sample Program and IR-QC
Software**

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Outline

Calibration Sample Preparation

Comparison of Modified Milk and Producer Calibration Samples

- a. range of component concentration**
- b. uncoupled fat & protein correlation**
- c. high leverage samples**
- d. shelf life**

Producer Calibration Samples – Approach

- **Sample sets for IR calibration are prepared and distributed by several labs around the U.S.**
- **Sets are prepared by**
 - **collecting bulk samples from local farms that represent a range of component concentration**
 - **preserving (potassium dichromate or bronopol)**
 - **splitting into vials**
 - **analyzing for reference chemistry**

Producer Calibration Samples – Weaknesses

- **Short range of component concentration**
- **Correlation of fat and protein concentration**
- **High leverage samples**
- **Relatively short shelf life**

Producer Calibration Samples – Weaknesses

Short Range of Component Concentration

IR Calibration Analysis [CCORAM01.MDB]

Main Menu Edit Analysis Print Options Select

Instrument Setting **Chemistry Set**

1: 3/17/03 7:56 PM 1: 3/17/03 7:58 PM (cl 031003)

Chemistry Set No. Chemistry Set Date and Time: Batch ID No. of Samples:

1 3/17/2003 7:58:35 PM cl 031003 12

Comment: Ash = .5379 + True Protein x .0596
Measured Lactose

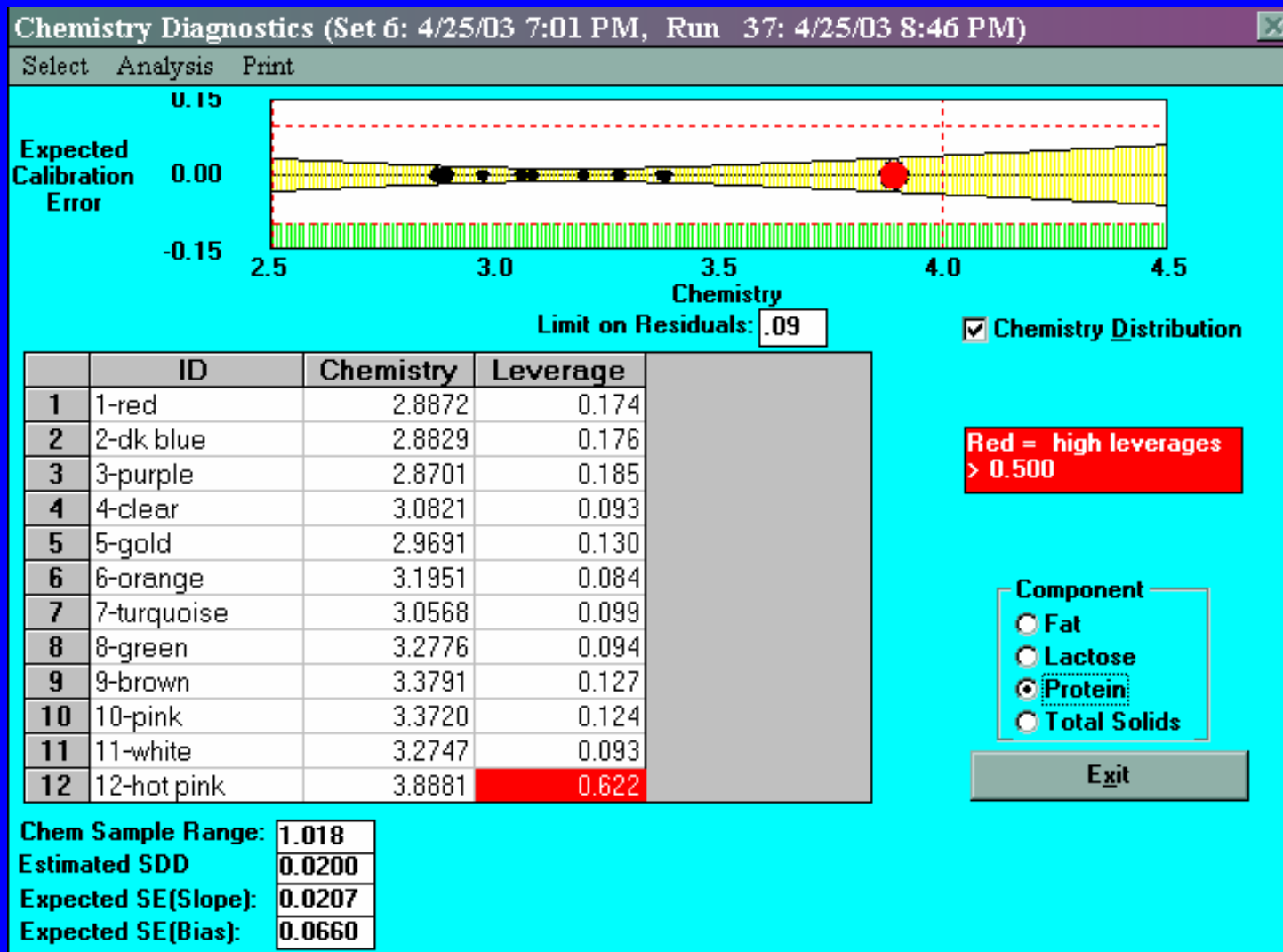
Chemical Test Results, Certified Correct By: Kaylegian, Kerry *

Sample	ID	Fat	True Prot.	TS	Lactose	SNF	OS	Drop
1	1-red	3.9750	2.8220	12.2220	4.5253	8.2470	5.4250	
2	2-dk blue	3.6470	2.9520	12.3700	4.8670	8.7230	5.7710	
3	3-purple	3.2440	2.9700	11.7390	4.6203	8.4950	5.5250	
4	4-clear	3.8430	3.1860	12.7260	4.7732	8.8830	5.6970	
5	5-gold	4.2420	3.2420	13.1860	4.7776	8.9440	5.7020	
6	6-orange	3.0180	2.9920	11.7840	4.8632	8.7660	5.7740	
7	7-turquoise	4.2000	3.3930	13.2370	4.7110	9.0370	5.6440	
8	8-green	3.6160	3.1410	12.3750	4.7033	8.7590	5.6180	
9	9-brown	5.3770	3.8980	15.0470	4.8058	9.6700	5.7720	
10	10-pink	4.2710	3.3100	13.0720	4.5665	8.8010	5.4910	1
Average		4.0927	3.2668	13.0183	4.7339	8.9256	5.6588	
Range		2.3590	1.0760	3.3080	0.3417	1.4230	0.3490	

Settings Chem Data IR Data Residuals Calc Comp. Evaluation Charts

Producer Calibration Samples – Weaknesses

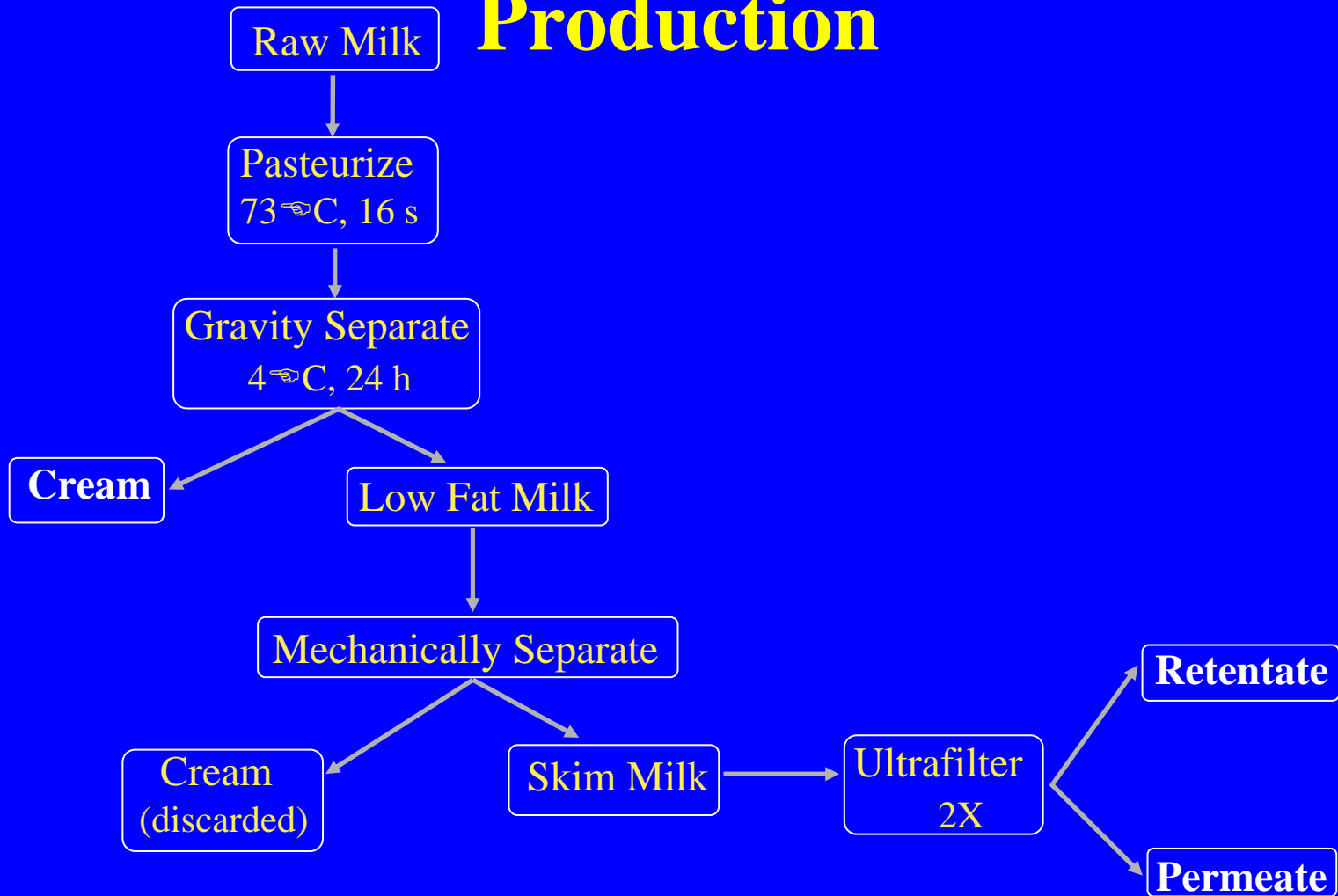
High Leverage Samples



Modified Milk Calibration Samples – Approach

- **Sample are designed to meet specific composition targets**
- **Samples are prepared by:**
 - **recombining gravity separated cream, permeate and retentate from ultrafiltered (UF) skim milk, and lactose and water, if needed**
 - **preserving (potassium dichromate, microtab, unpreserved)**
 - **splitting into vials**
 - **analyzing for reference chemistry**
- **Approved IDF procedure, Standard 141C:2000**
 - **Now used by USDA Federal Milk Market Administrator labs**

Modified Milk Calibration Samples – Production



Modified Milk Calibration Samples – Formulation

Formulation Ingredient Composition

(%) Ingredient	Fat	True Protein	Lactose
Cream	26.60	2.21	3.58
Retentate	0.15	6.32	4.65
Permeate	0.00	0.07	5.00
Lactose Monohydrate	0.00	0.00	95.00

Modified Milk Calibration Samples – Formulation

	Composition (g/100g)		
	Fat	Protein	Lactose
1	0.20	4.30	4.00
2	0.62	2.27	4.55
3	1.05	3.96	5.10
4	1.47	2.61	4.96
5	1.89	3.62	4.28
6	2.32	2.95	4.55
7	2.74	3.28	4.55
8	3.16	3.12	4.42
9	3.58	3.45	4.68
10	4.01	2.78	4.14
11	4.43	3.79	4.82
12	4.85	2.44	4.00
13	5.28	4.13	4.55
14	5.70	2.10	5.10

Outline

Calibration Sample Preparation

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- d. shelf life**

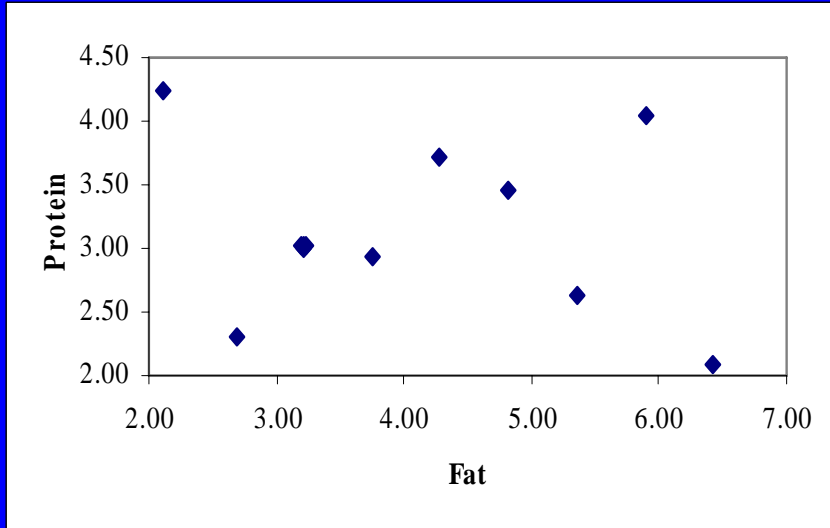
UF Calibration Samples – Advantages

Range of Component Concentration

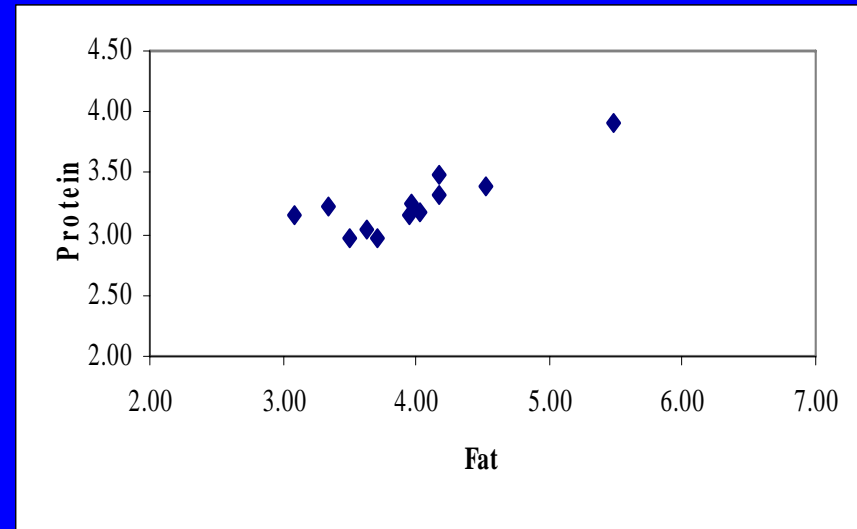
Fat	0.2 to 5.8%
True Protein	2 to 4.3%
Lactose	4 to 5.1%

Modified Milk Calibration Samples – Advantages Uncoupled Fat & Protein Correlation

Modified Milk Samples

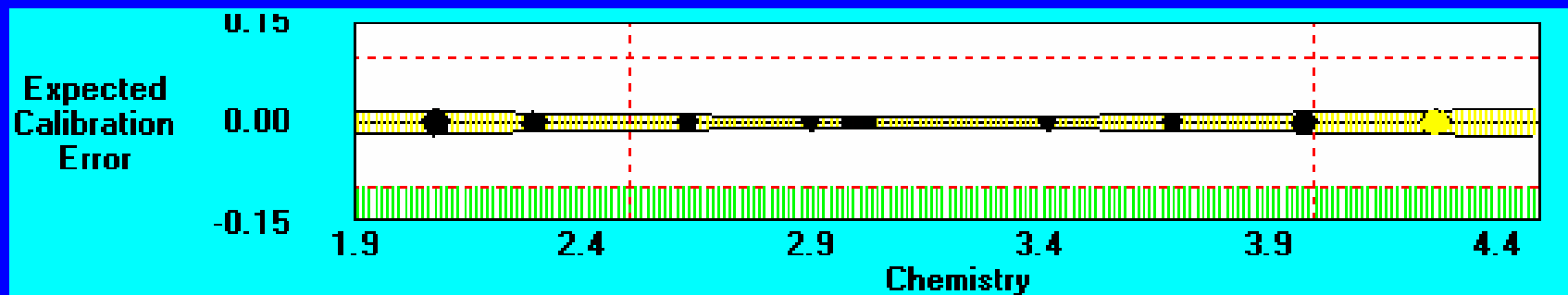


Producer Cal Samples

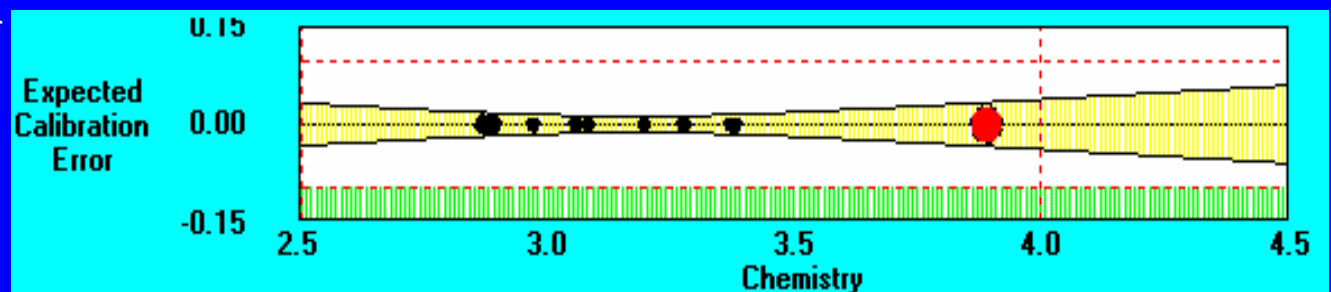


Modified Milk Calibration Samples – Advantages No High Leverage Samples

Modified Milk Samples – Protein



Producer Cal Samples – Protein



UF Calibration Samples – Advantages

Sample Shelf Life

- Shelf Life

	Modified	Producer
lag time from production		
to use (chemistry)	1.5 wk	1 wk
active time	4 wk	2 wk

- Indicators

- visible deterioration
- change in slope & bias
 - changes in FFA (Barbano et al., 1999, JDS 82(Supp 1):26)
- IR-QC[©] determination of sample out of specification

Performance of Calibration Samples Agreement between Chemistry & IR Prediction

**Standard Deviation of the Difference (SDD) of
the Residuals of Calibration Samples**

Sample	Chemistry	IR	IR-Chem
1	2.107	2.107	0.000
2	2.684	2.687	0.002
3	4.270	4.293	0.023
4	3.756	3.727	-0.030
5	4.825	4.831	0.006
6	5.363	5.359	-0.005
7	5.890	5.898	0.008
8	6.419	6.410	-0.009
Mean	4.415	4.415	0.000
SDD			0.014

Performance of Calibration Samples Agreement between Chemistry & IR Prediction

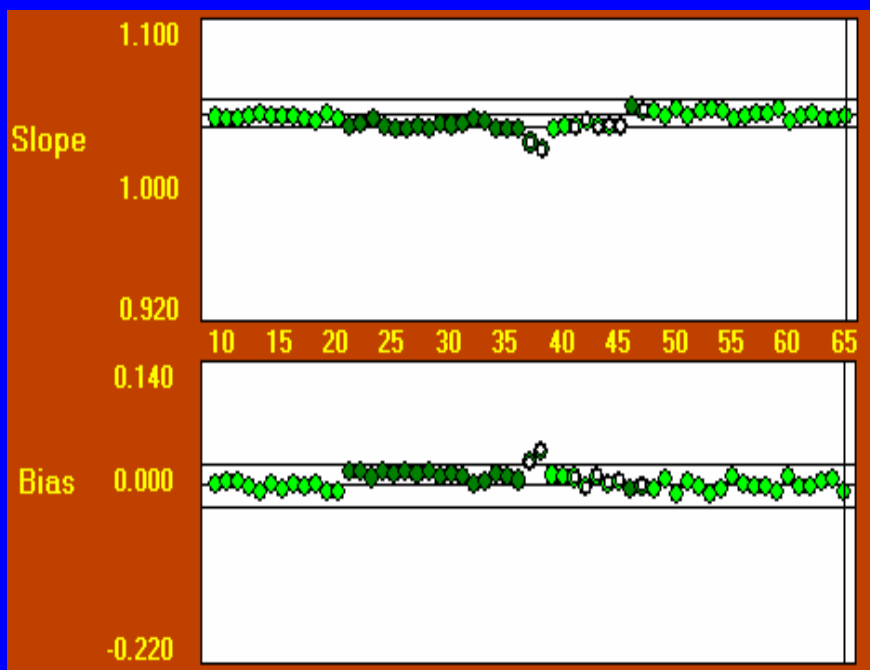
Typical SDD of the Residuals of
Calibration Samples

Component	Modified Milk	Producer Cal
Fat B	.0130	.0201
Protein	.0115	.0346
Lactose	.0146	.0184
Fat A	.0112	.0523
Number of Samples	12	12

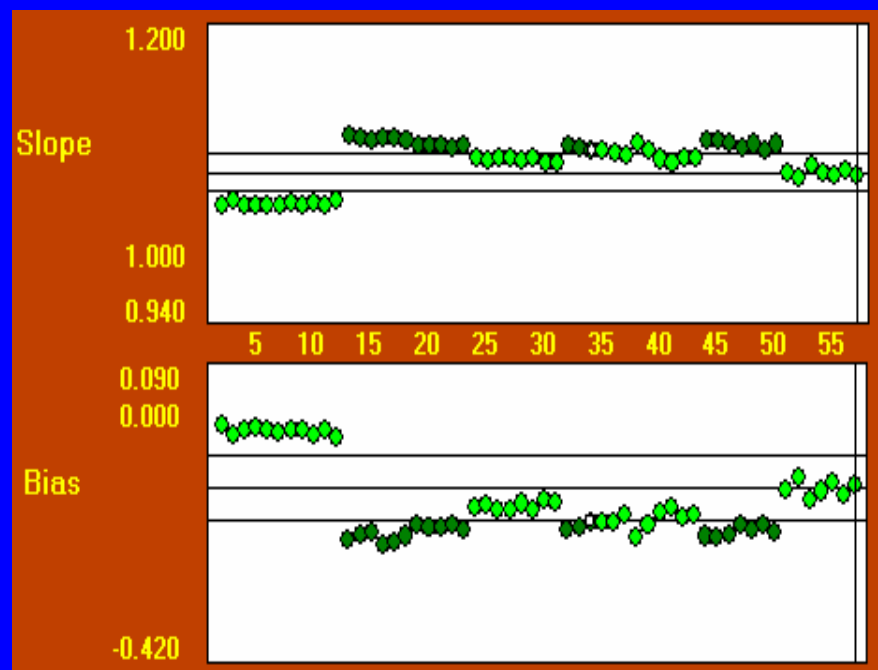
Performance of Calibration Samples

Calibration Stability of Slope & Bias

Modified Milk – Protein



Producer Cal – Protein



Summary

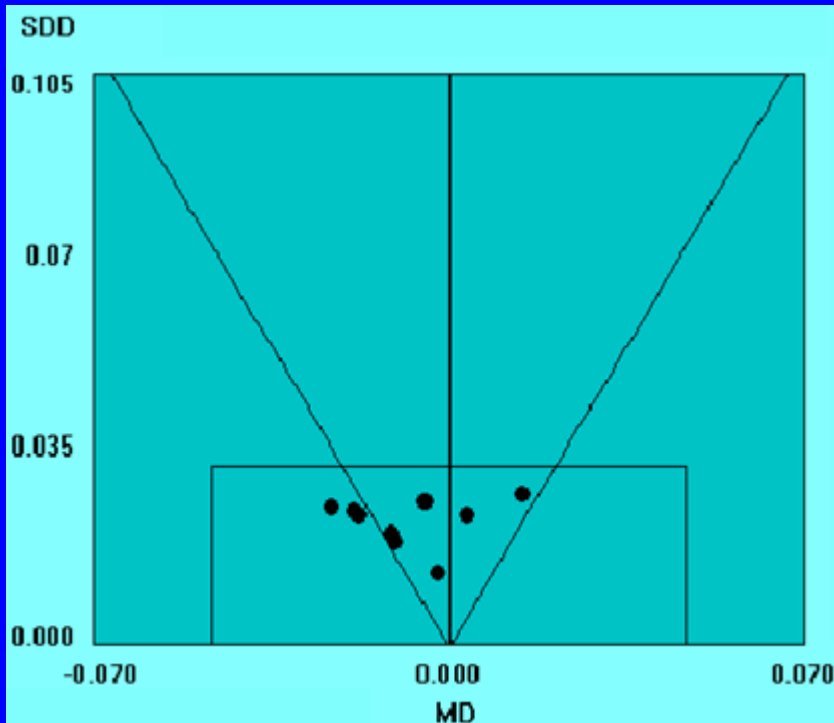
An alternative approach to produce calibration samples for IR analysis of milk is to recombine gravity separated cream, permeate and retentate from ultrafiltered skim milk, lactose and water to create a sample set that improves calibration compared with producer calibration samples

Summary

- **Better agreement between chemistry and IR prediction (lower SDD of the residuals of the calibration samples)**
 - wide range of component concentration
 - uncoupled fat & protein correlation
- **Improved slope and bias stability between calibration sample sets**
 - consistent component range from set to set
 - fewer high leverage samples
- **Improved slope and bias stability within calibration sample sets**
 - increased shelf life

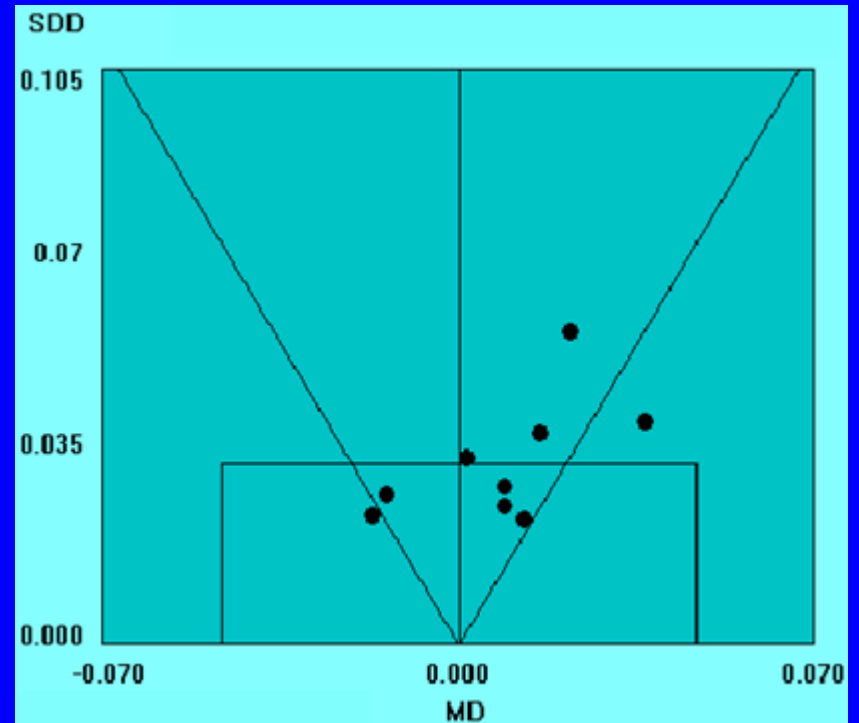
Results: Fat Validation

Modified Milk Calibration



9 instruments
each dot = 1 instrument

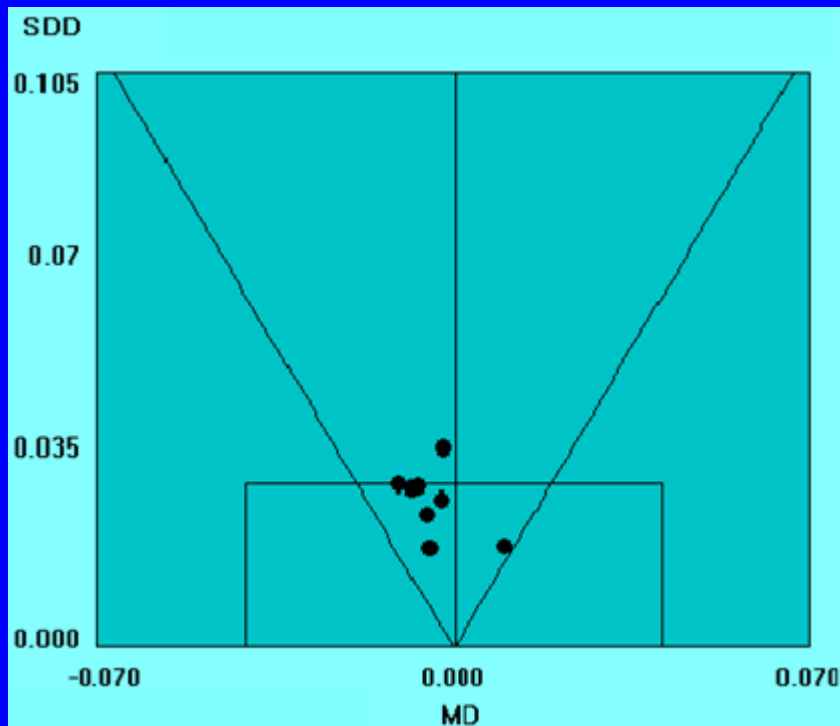
Producer Milk Calibration



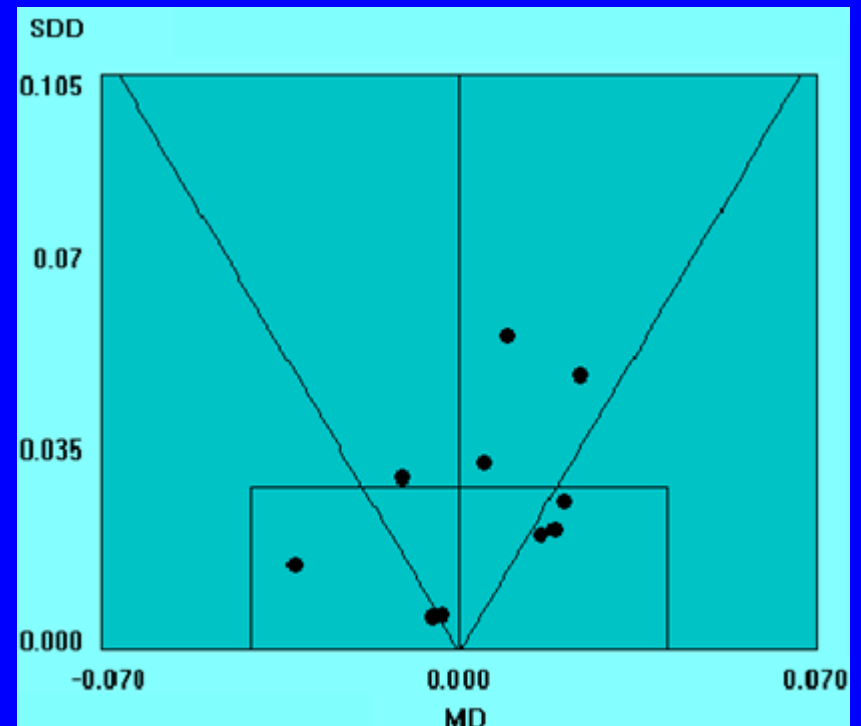
10 instruments
each dot = 1 instrument

Results: Protein Validation

Modified Milk Calibration

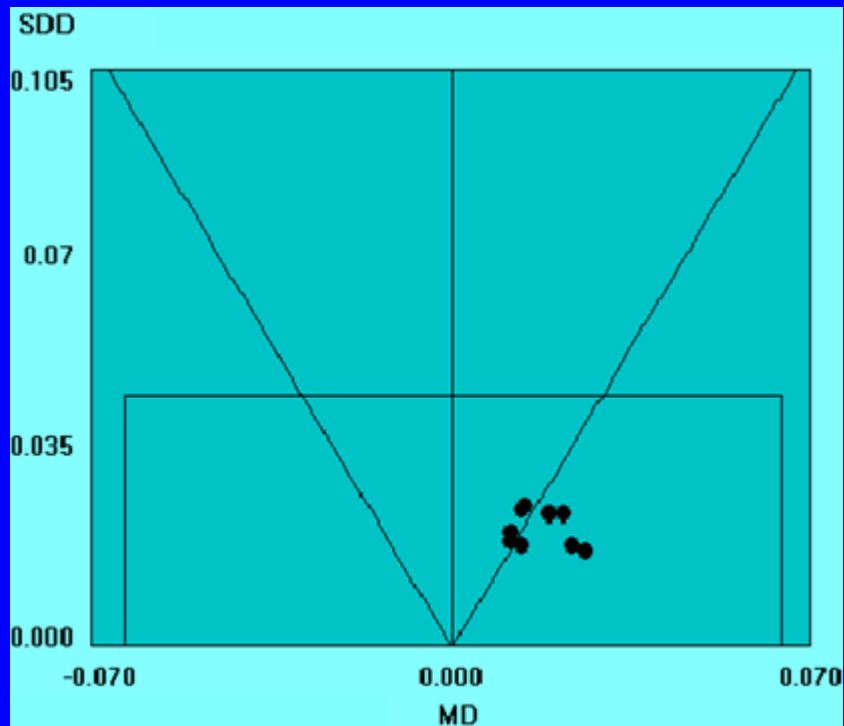


Producer Milk Calibration

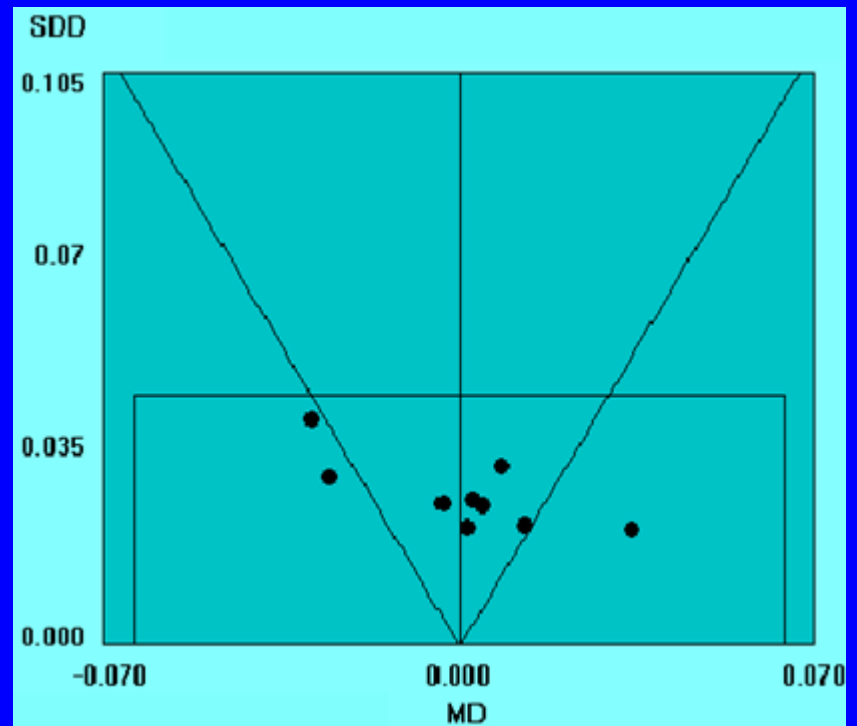


Results: Lactose Validation

Modified Milk Calibration

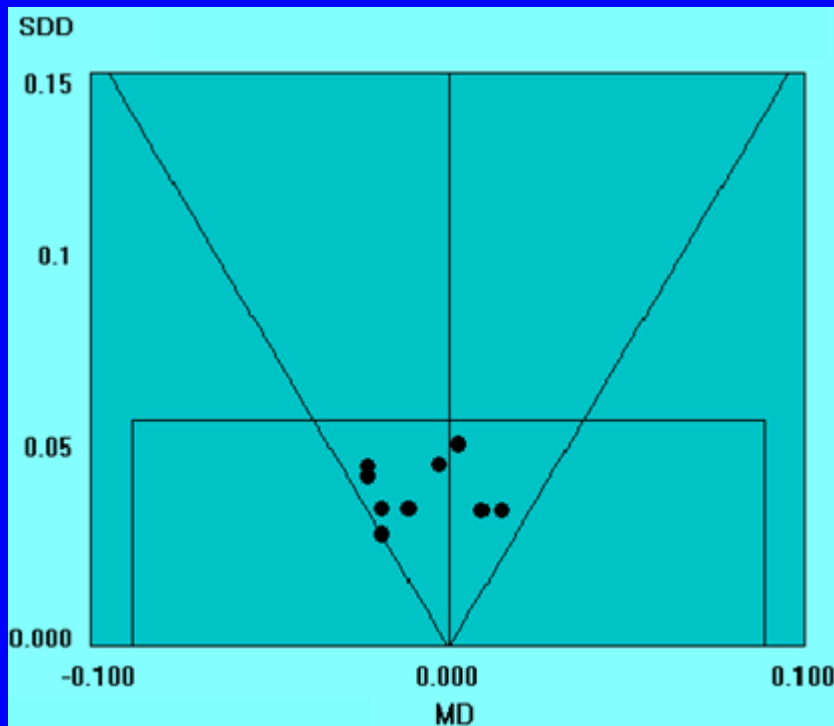


Producer Milk Calibration

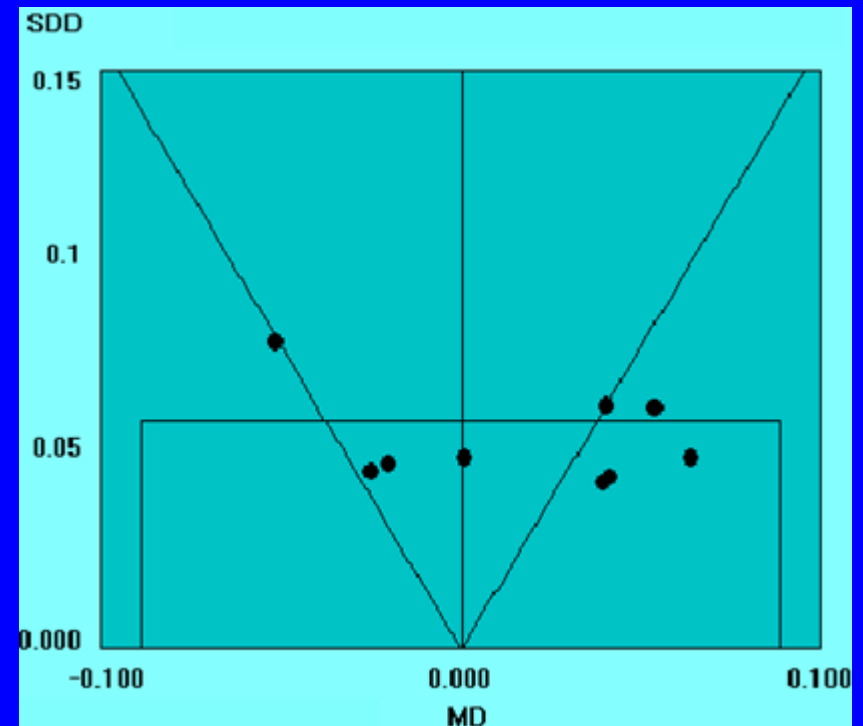


Results: Total Solids Validation

Modified Milk Calibration



Producer Milk Calibration



Conclusions on Validation

- Calibration with **modified** milk samples resulted in:
 - a reduction in the range of difference from chemistry (MD) by at least 30% and as much as 85%
 - a lower and more consistent SDD between instrument and all lab mean chemistry
- The improvement in calibration was observed in labs across the US, using different types and models of IR analyzers

Conclusions on Validation

The use of **modified** milk samples to calibrate IR analyzers improved the accuracy of prediction of fat, protein, lactose, and solids content compared with IR analyzers calibrated with producer milk samples