Current applications and challenges along the dairy food chain

Steve Holroyd, Michel Nieuwoudt, Gavin Scott,

Cushla McGoverin

March 2023

Outline

- FTIR overview, use in value chain
- Milk adulteration – non targeted methods
- Individual fatty acids
- Milk minerals and proteins
- FTIR and data quality control - ASCA

Dairy value chain - where analysis fits in

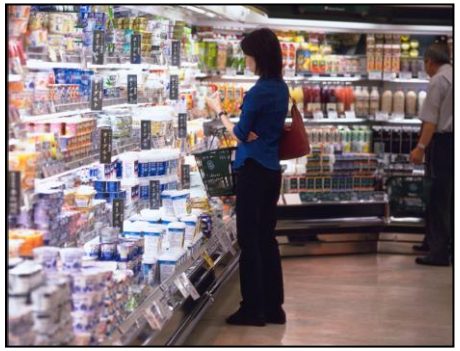
For payment

Before processing

In process

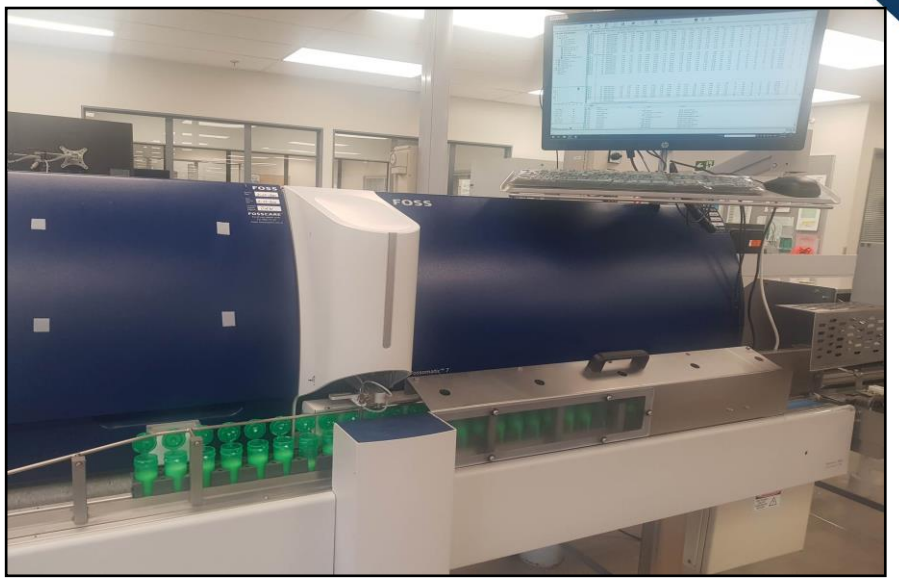
Final product grading

Customers & regulators



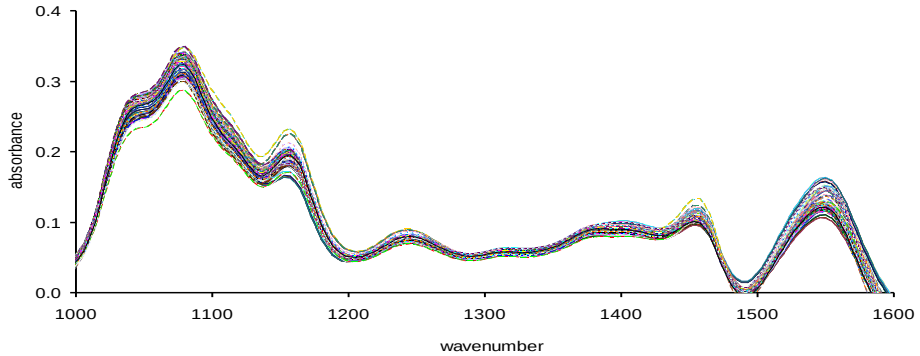
Mid infrared (MIR) spectroscopy for milk testing

- MIR testing on milk is common.
- Measures milk composition in seconds.
- Range of different Fourier transform infrared (FTIR) instrument types for use in a range of environments.
- Rapid technique for gathering information on milk using infrared absorption.



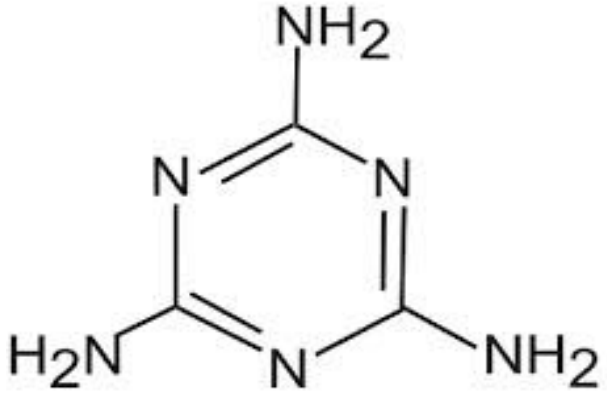
Historic measurement of milk components by MIR

- 1980-90s: Traditional quantitative calibration are for gross composition.
  - ➢ Fat, protein, lactose, total solids, solids not fat.
- 2000: Major fractions of fat and protein.
  - ➢ Casein, saturated and unsaturated fatty acids.
- 2008 onwards – adulterants.
- 2010: Individual fatty acids and proteins (“fine milk composition”) and adulterants at concentrations 100-1000+ppm.



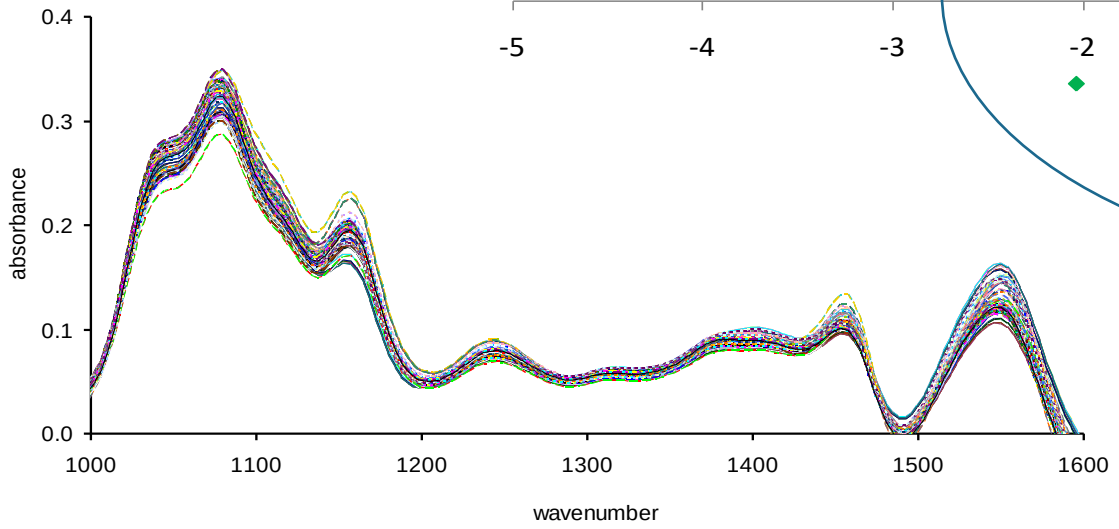
Melamine adulteration of milk powder 2008

- Melamine deliberately added to infant formula to increase protein content as tested by Kjeldahl reference test.
- Very serious food safety incident.
- Prompted considerable interest in use of non targeted applications to detect adulterants and contaminants.

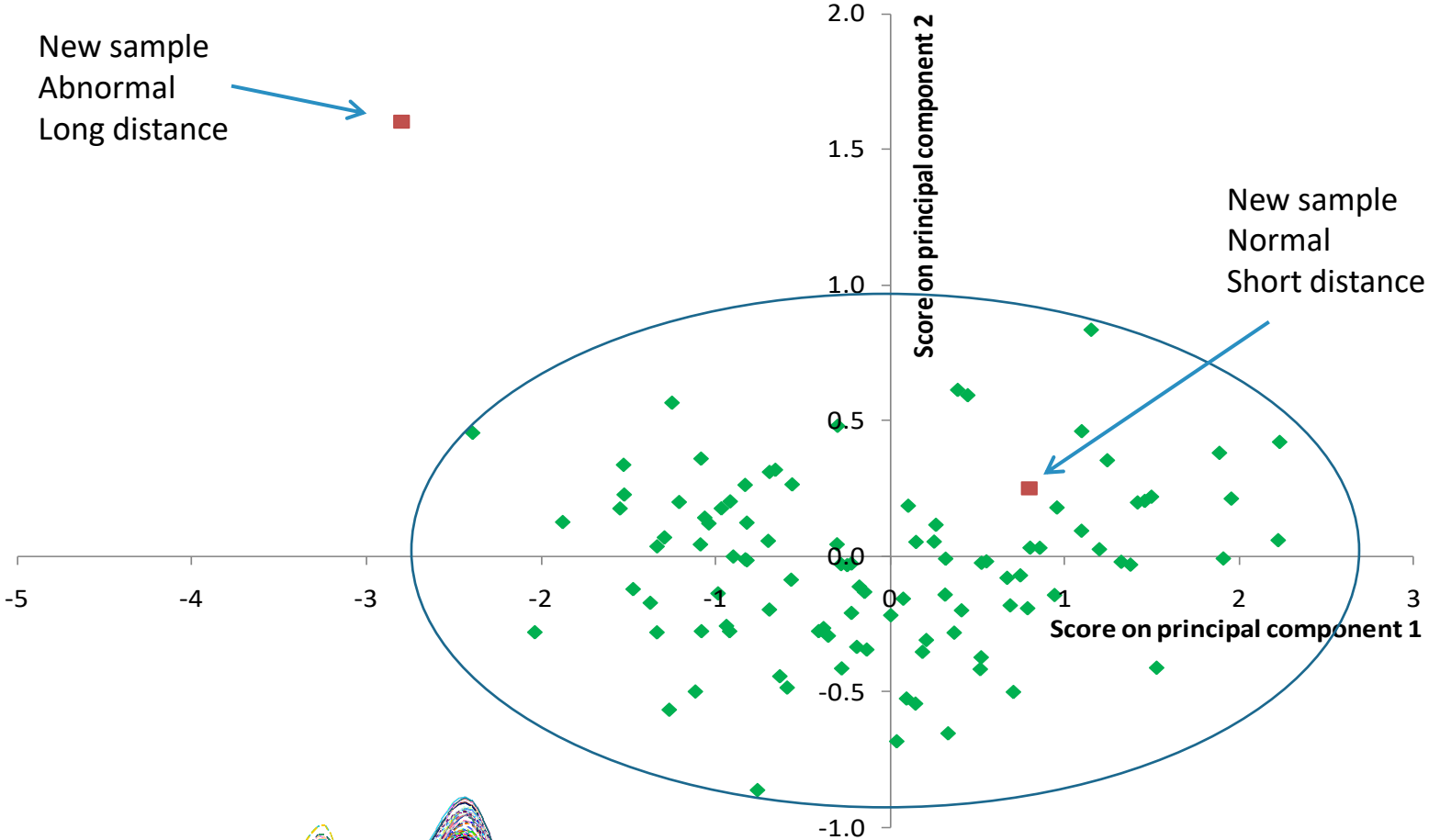


New approach to detecting unknowns

Non targeted analysis – Principle Components Analysis (PCA)

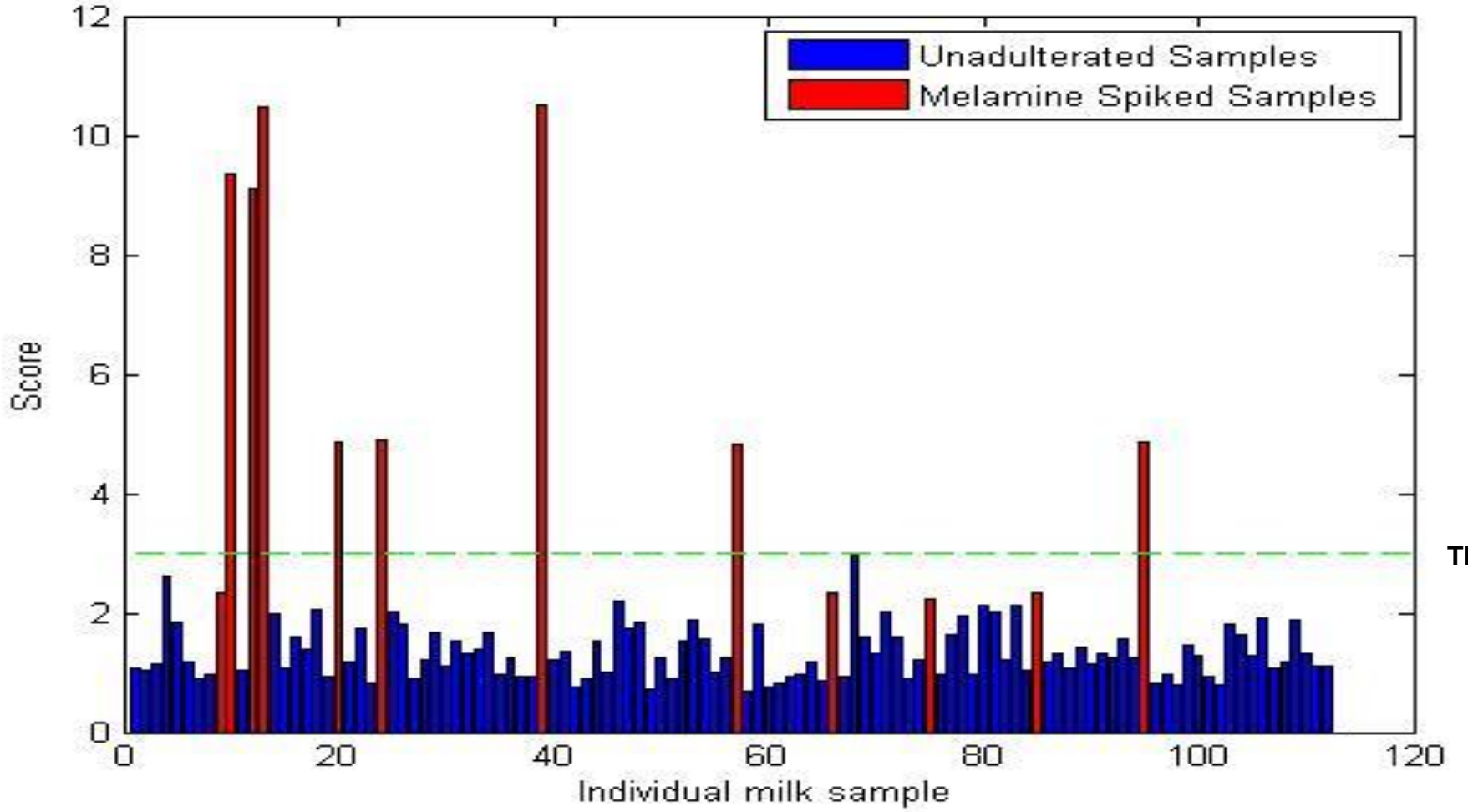


New sample
Abnormal
Long distance



New sample
Normal
Short distance

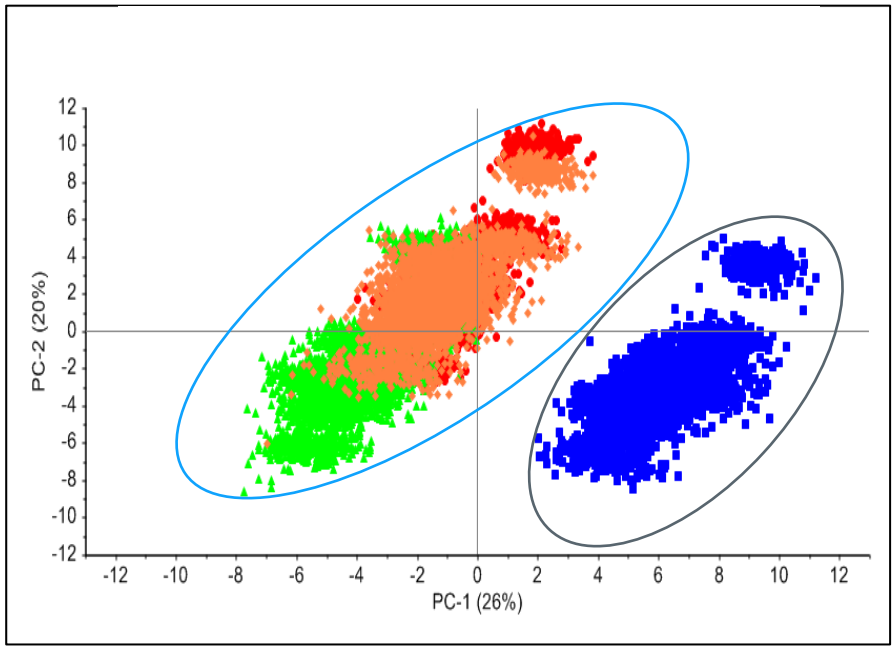
Non-targeted analysis of liquid milk by MIR

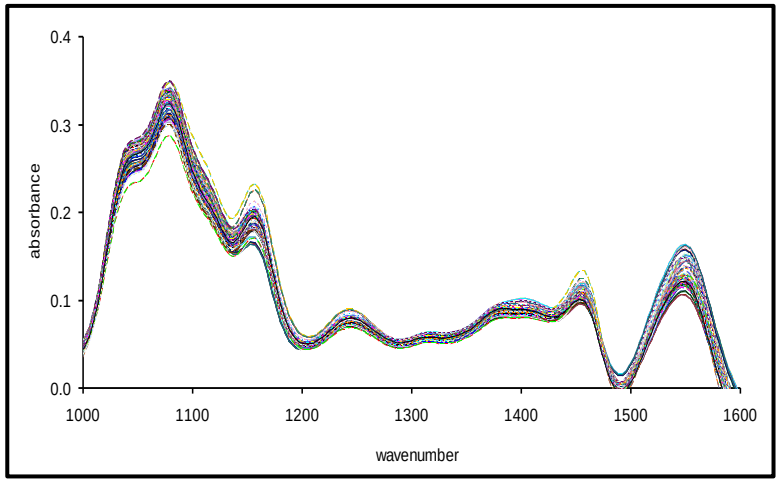


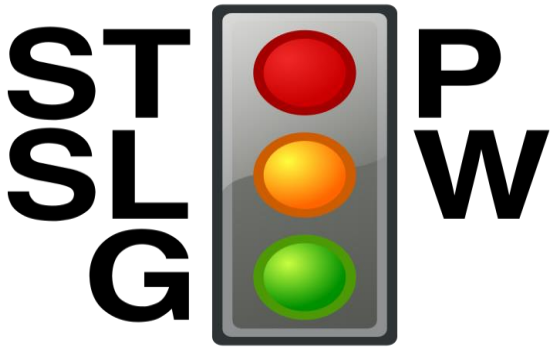
Threshold

Non-targeted methods - process

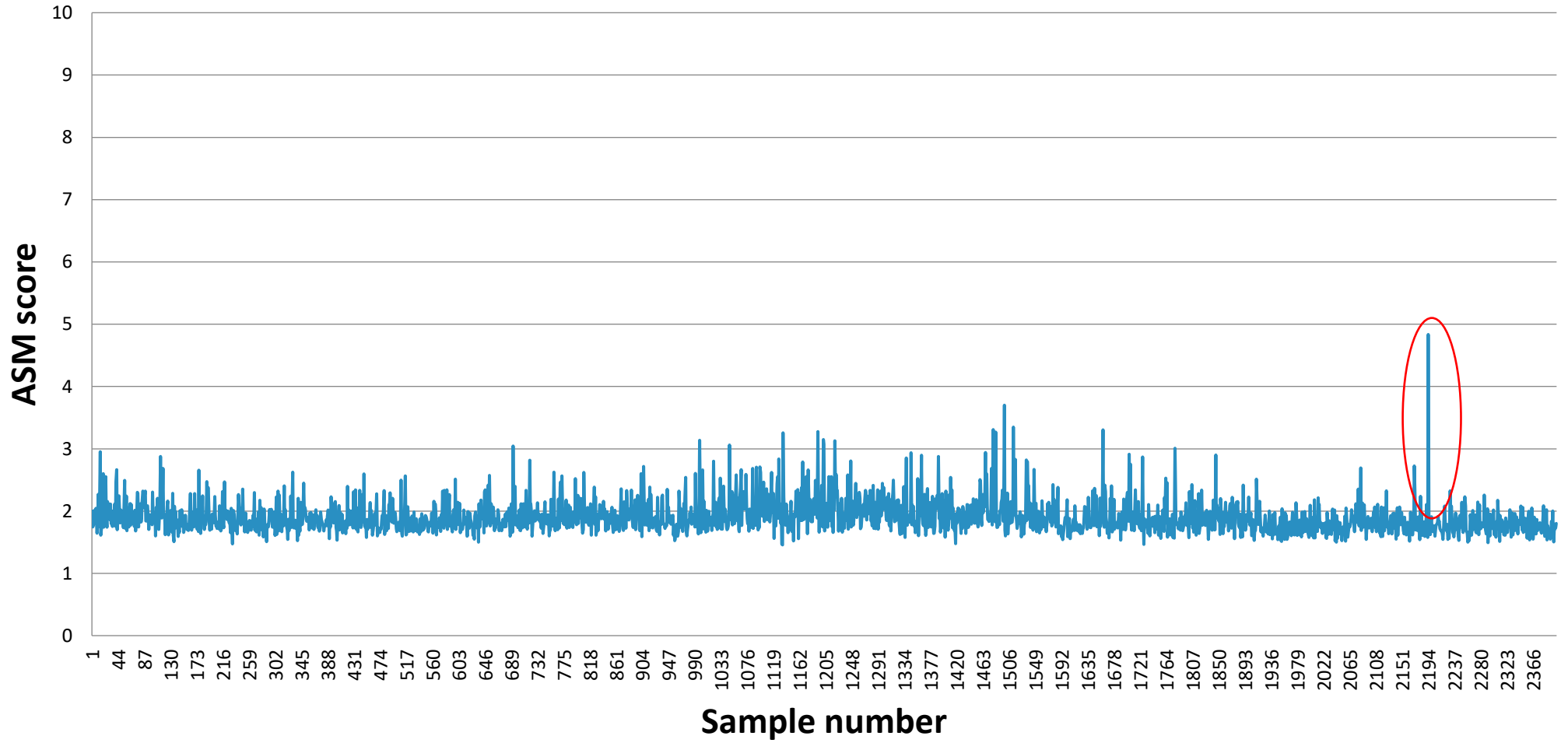
- Gather database on representative samples – “fingerprints”
- Do statistics and decide on limits.
- Measure new sample and make a decision.







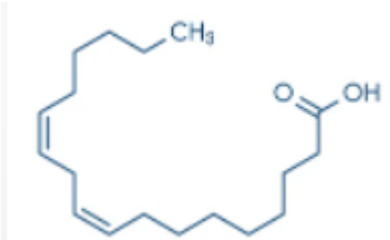
Non-targeted MIR screening of NZ milk
Abnormal screening module (ASM)



Outline

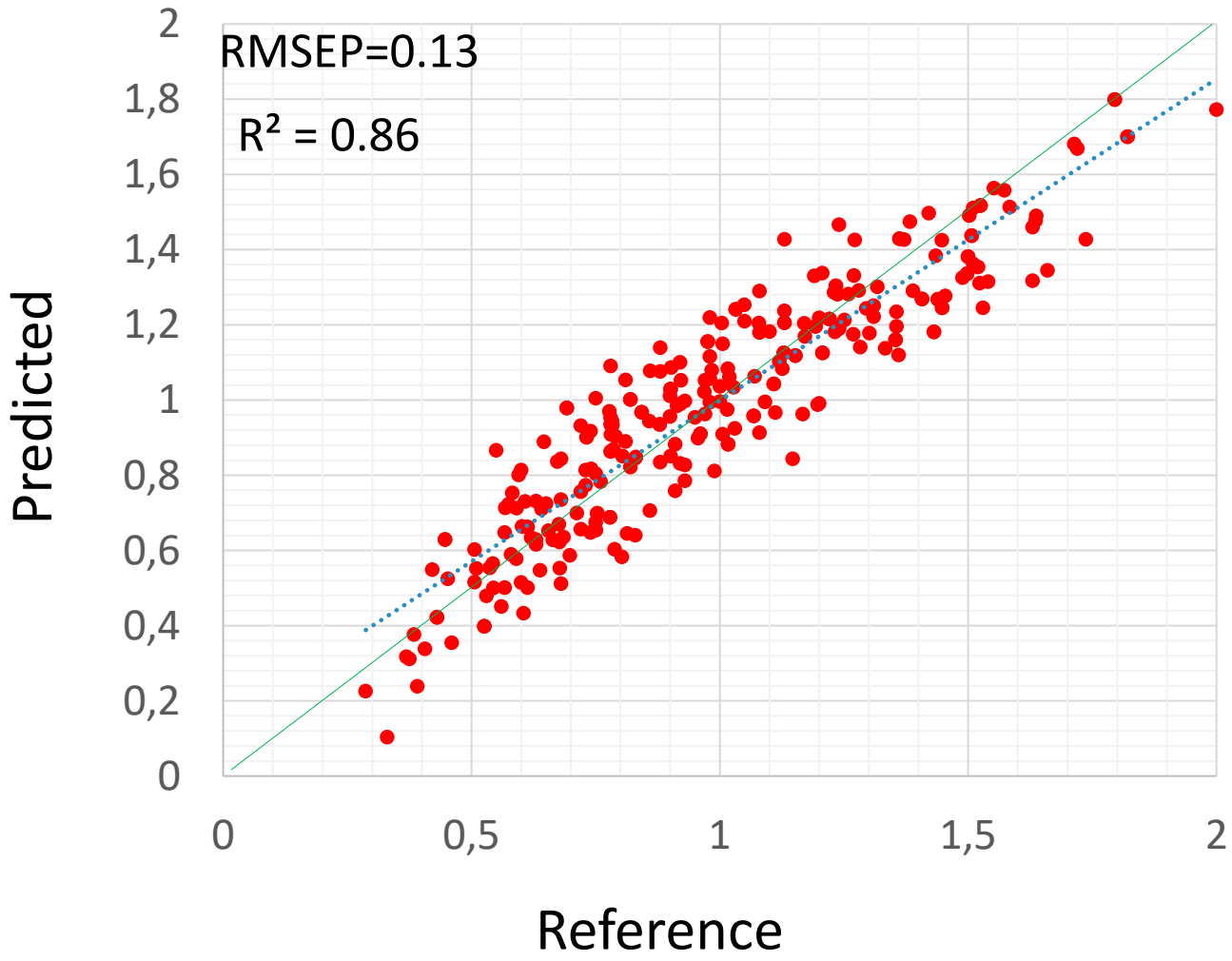
- FTIR overview
- Milk adulteration – non targeted methods
- Individual fatty acids
- Milk minerals and proteins
- FTIR and data quality control - ASCA

Conjugated linoleic acid (CLA)

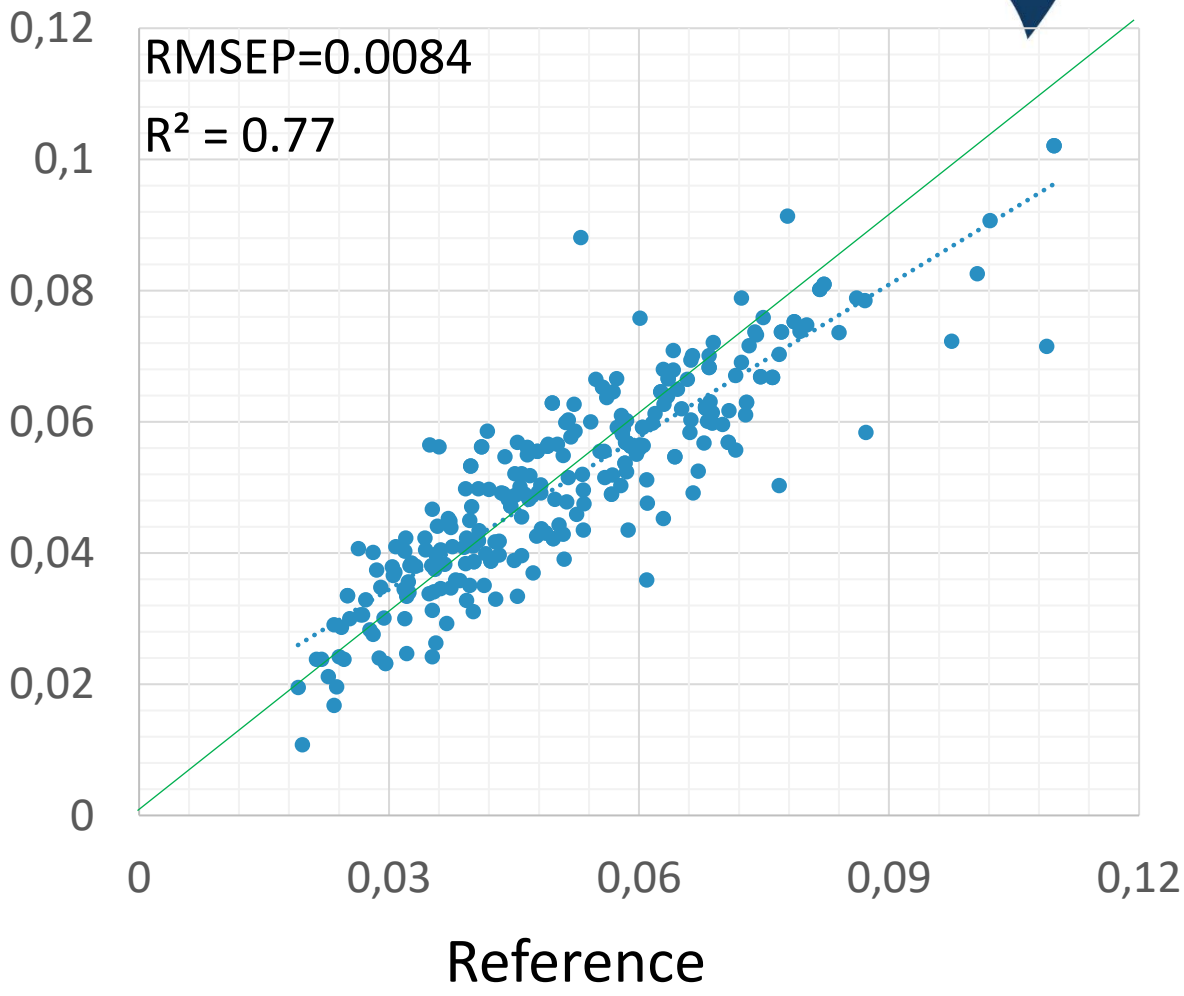


Fonterra
Dairy for life

CLA pred (g/100g fat)

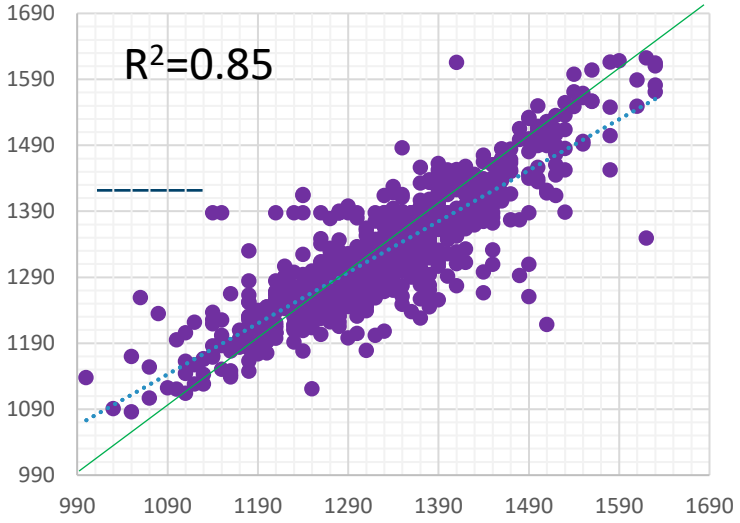


CLA ref (g/100L milk)

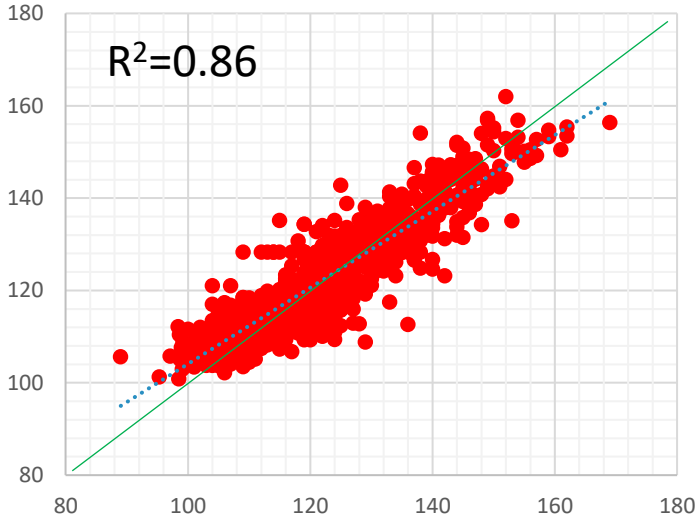


Minerals

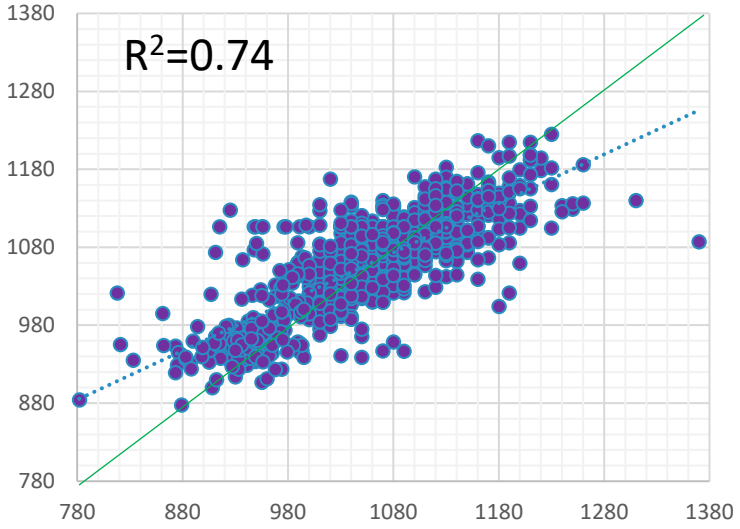
Calcium (mg/g)



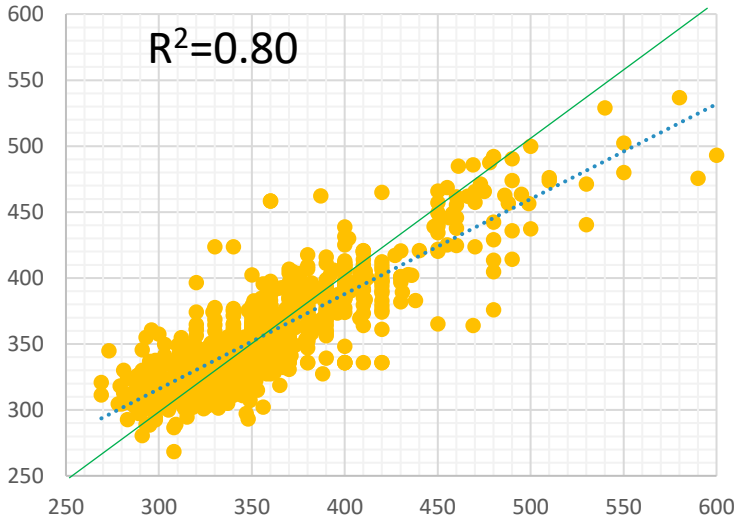
Magnesium (mg/g)



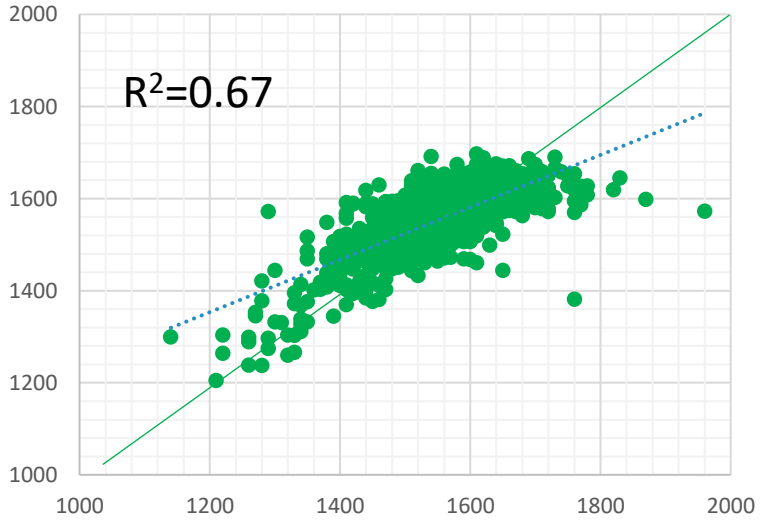
Phosphorous (mg/g)



Sodium (mg/g)



Potassium (mg/g)



New Zealand results						Literature comparisons		
Mineral	RMSECV mg/g (LV's)	R^2 (CV)	RPE [1] %	Mean mg/g	SD mg/g	RMSEP[1] g/L or SECV [2] (LV's)	R^2 (pred)[1] Or R^2(CV)[2]	RPE [1] %
Ca	63.21 (13)	0.85	4.77	1325	105.4	80 (10)[1], 95[2]	0.80[1], 0.77[2]	6.3[1]
Mg	7.54 (10)	0.81	6.10	123.5	13.14	10 (5)[1], 11[2]	0.46[1], 0.50[2]	8.1[1]
P	60.66 (11)	0.74	5.73	1058	79.60	40 (12)[1], 50[2]	0.87[1], 0.83[2]	5.4[1]
K	85.64 (16)	0.67	5.50	1556	98.28	110 (17)[1], 136[2]	0.33[1], 0.23[2]	7.5[1]
Na	32.58 (10)	0.80	9.19	354.5	47.4	70 (9)[1], 64 [2]	0.63[1], 0.77[2]	18.8[1]

[1] *RPE* = the percentage of RMSEP relative to the ICP-MS reference test mean values for the prediction model [1,5]

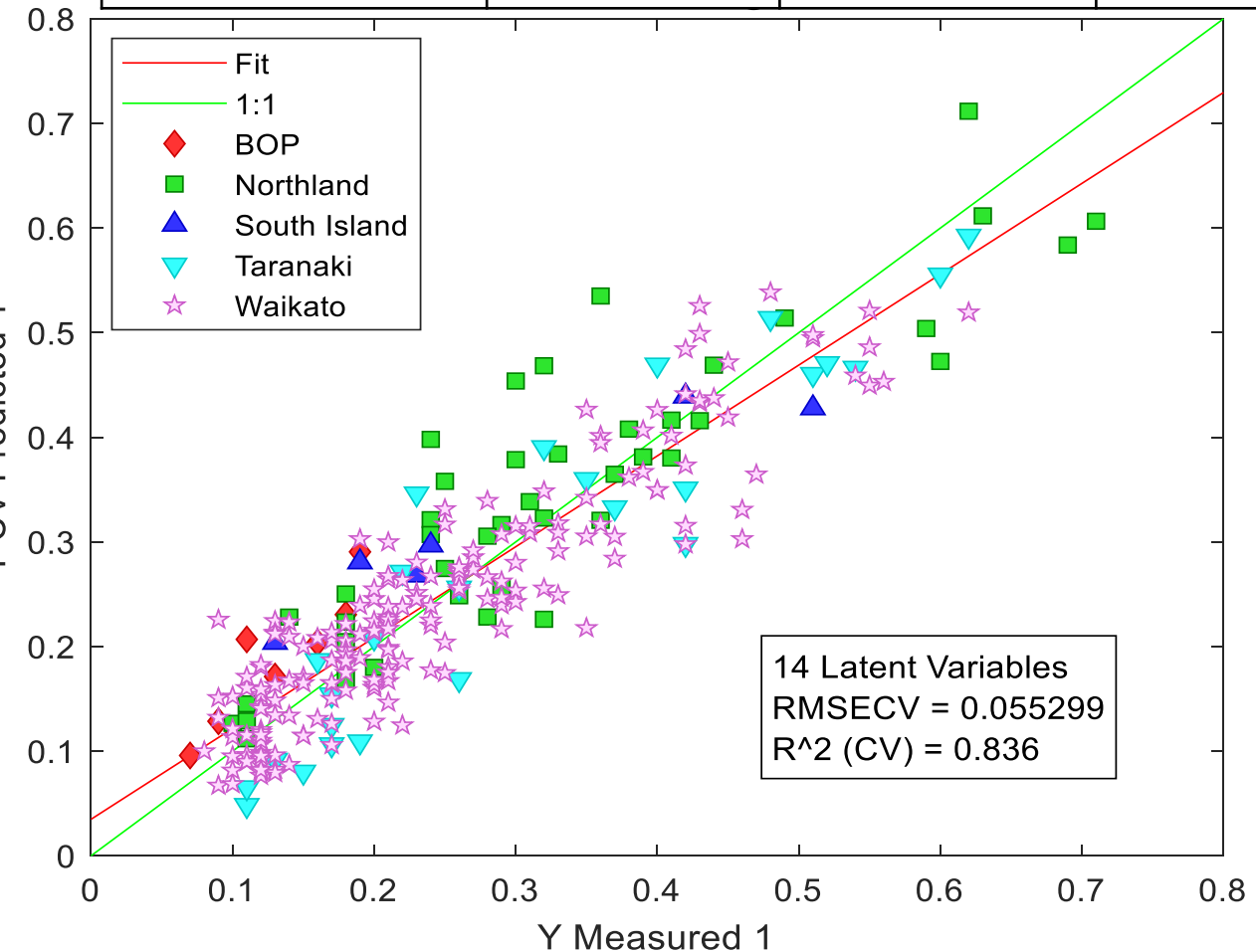
[2] Total SECV of analysed samples, given in [2].

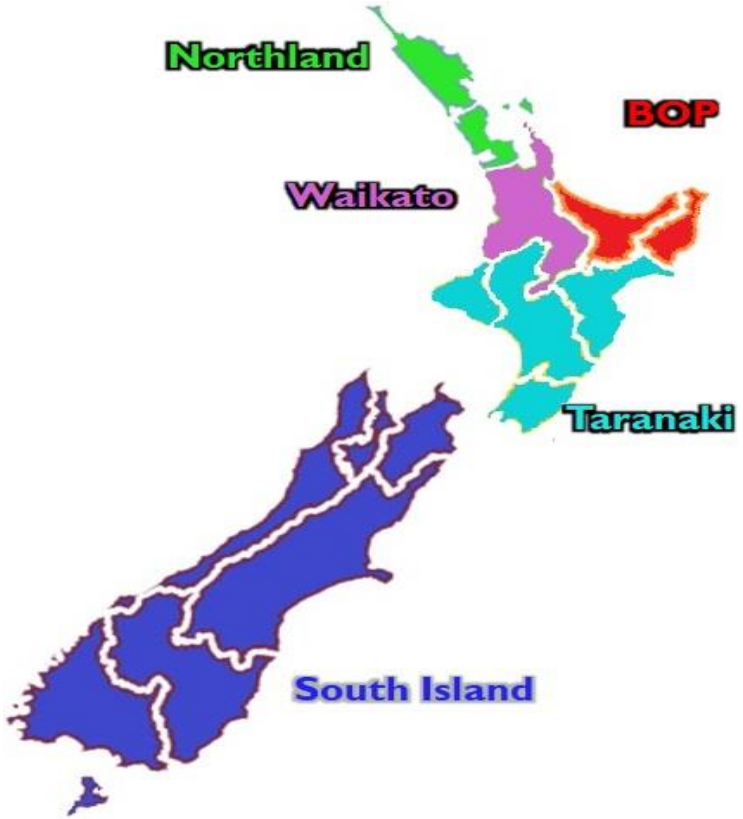
[1] Zaalberg, R. M., Poulsen, N. A. Bovenhuis H., Sehested J.,Larsen L. B.,2 and Buitenhuis A. J., J. Dairy Sci. 2021, 104 (8), https://doi.org/10.3168/jds.2020-19638.

[2] Soyeurt, H, Bruwier, 1 D., Romnee, J.-M., Gengler, N. Bertozzi, C., Veselko, D. and Dardenne, P., Potential estimation of major mineral contents in cow milk using mid-infrared spectrometry, J. Dairy Sci., 2009, 92:2444–2454.

Lactoferrin

Component	# samples	# outliers	# PCs	Mean	StdDev	RMSEP	R^2	RPD
Lactoferrin	299	12(9%)	14	0.282	0.166	0.055	0.84	3.1

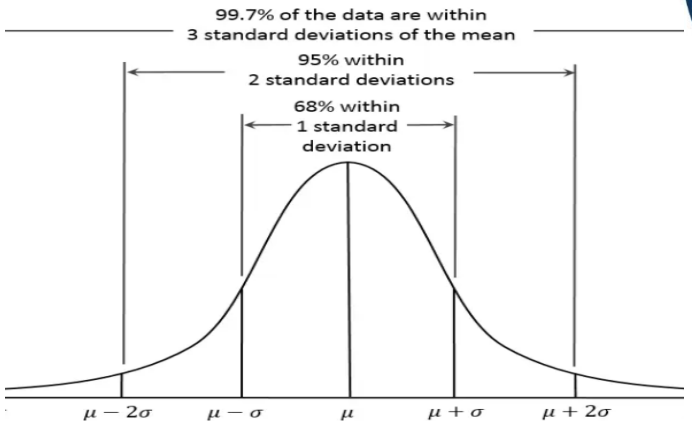




Outline

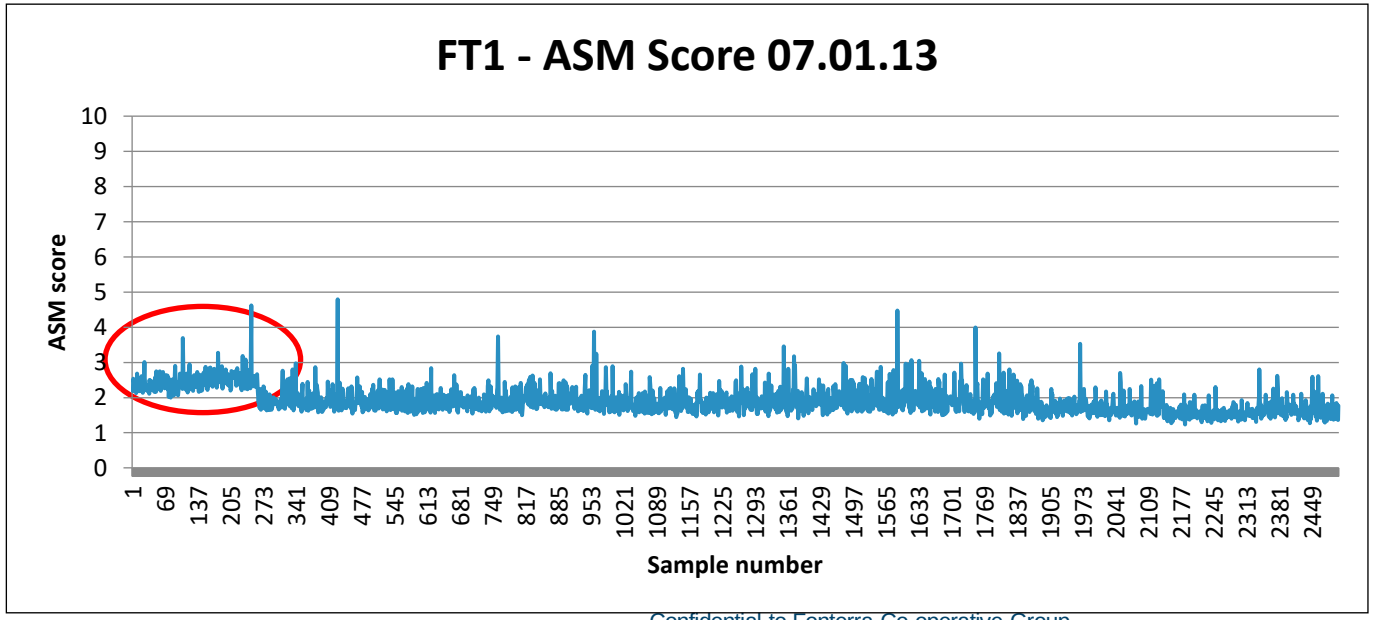
- FTIR overview
- Milk adulteration – non targeted methods
- Individual fatty acids
- Milk minerals and proteins
- FTIR and data quality control - ASCA

Challenges specific to MIR for fine milk composition



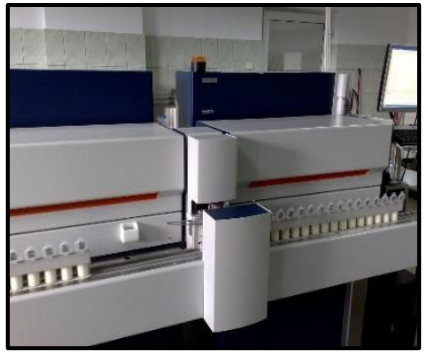
- Reference data
  - Quantitative or qualitative?
- Standardization of the MIR spectra between FTIR instruments.
- Stability of FTIR instruments over time.

Grelet, C., *et al*. "Standardization of milk mid-infrared spectrometers for the transfer and use of multiple models." Journal of Dairy Science 100(10) 7910-7921. 2017.



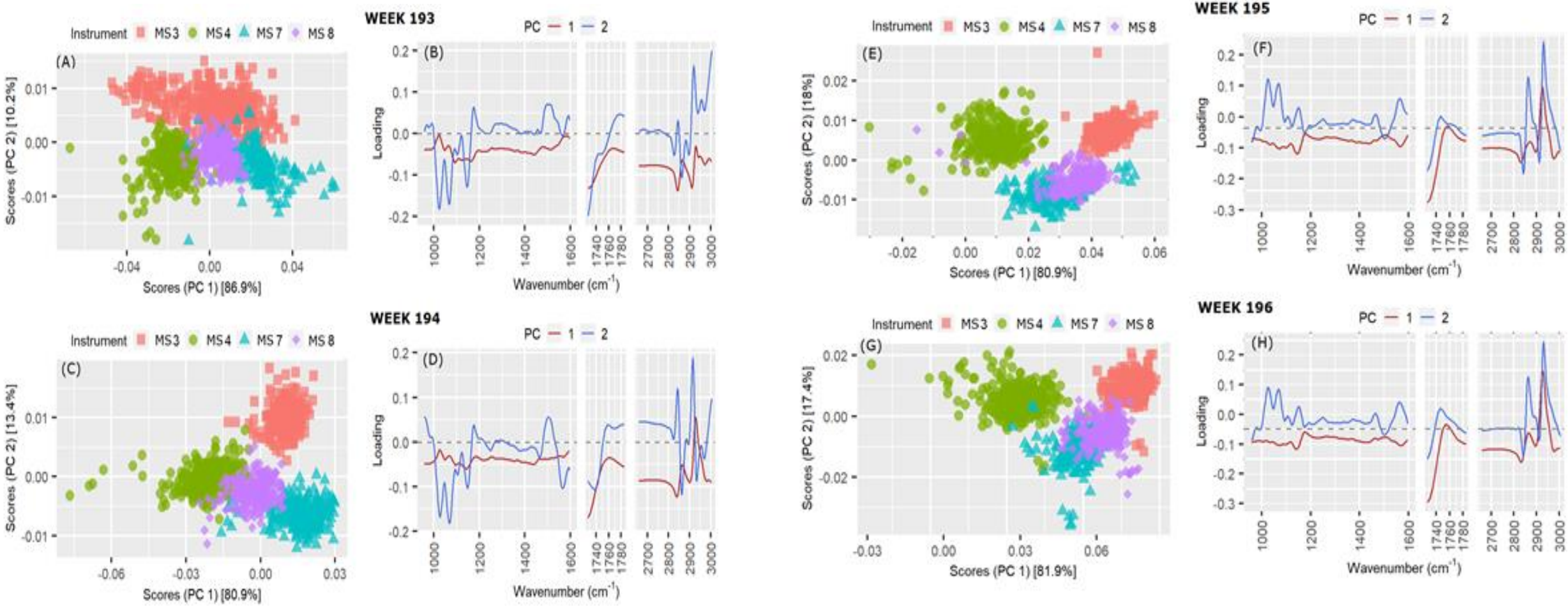
Pilot milk samples study

- NZ central milk testing laboratory.
- Seek to determine contribution of variation from instruments over time.
- Foss FT6000/FT+/Milkoscan 7 instruments
- Over 207 weeks (207 pilot samples) 2016 – 2021.
- Pilot samples recorded every hour of the same sample over a week.
- ANOVA with simultaneous Principle Component Analysis (ASCA).



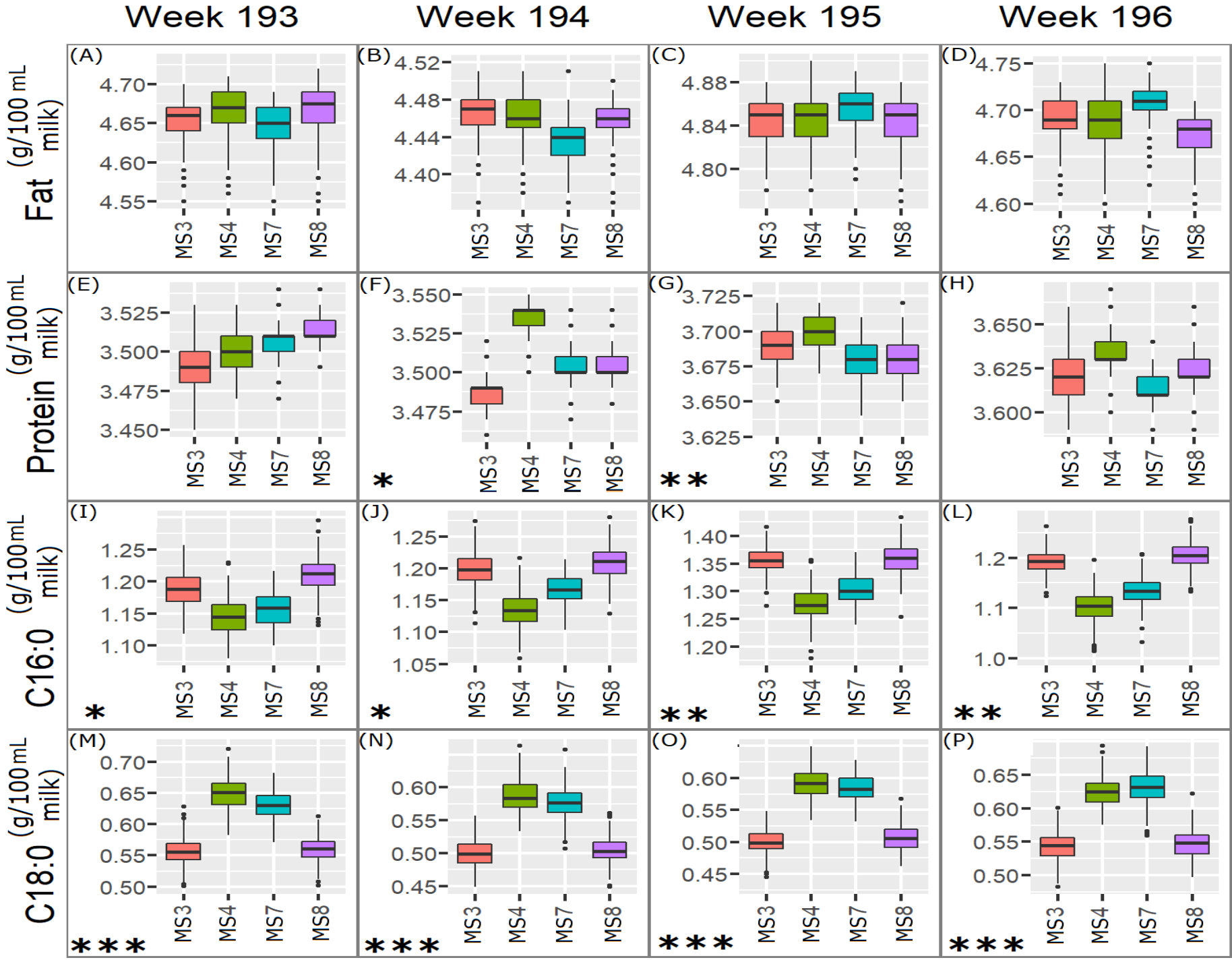
Reference: Nieuwoudt M.K., Giglio C., Marini F., Scott G., Holroyd S.E., Routine Monitoring of Instrument Stability in a Milk Testing Laboratory With ASCA: A Pilot Study, Frontiers in Chemistry (2021) 9, article 73331.

PCA of a Series of Four Successive Weeks: 193 to 196



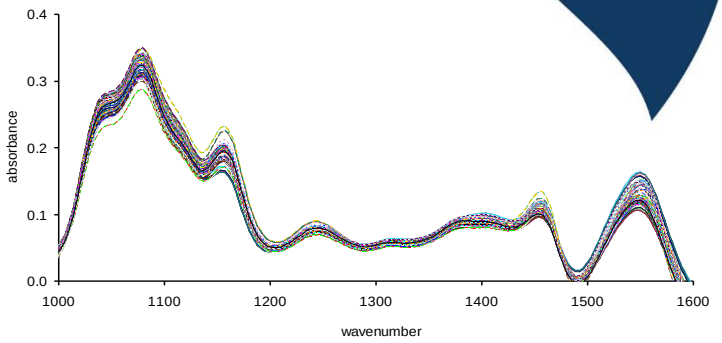
Impact on predictions

- MS3/4 and MS7/8 are different FTIRs.
- Results for fat/protein may differ a small amount.
- Results for individual fatty acids can differ more.



Conclusions

- MIR systems widely used for fine milk compositional analysis.
- Current standards and systems for MIR for milk apply to more historic applications.
- FTIR instruments are complex and vary over time which can impact predictive performance.
- Many stakeholders: Central milk analysis, breeding, researchers, milk processors, instrument vendors.



Acknowledgements

- Milktest NZ: Scott Middleton, Callie Smith,
- University of Auckland: Michel Nieuwoudt, Cushla McGoverin.
- Fonterra: Gavin Scott, Paul Jamieson.
- Foss: Per Waaben Hansen