Reference system and centralised calibration for milk (payment) testing

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A modified milk calibration set has been developed for use in a network of payment testing laboratories in the US. The ser of calibration samples consist of 14 samples produced with an orthogonal matrix of composition with respect to variation in fat, protein, and lactose. The range of fat content is from 0.2 to 5.8%, true protein from 2 to 4.3%, and anhydrous lactose from 3.9 to 5.2%. The modified milk calibration samples are produced 12 times per year and serve as a proficiency test for the reference chemistry methods performed in all the laboratories and a set of calibration samples for infrared milk analyzers. These samples are used to set slope and intercept of the intercorrected mid-IR signal.

The modified milk calibration samples serve three purposes. First, each month the testing of these samples provides a proficiency test of the fat by ether extraction, the true protein by Kjeldahl, the anhydrous lactose by enzymatic, and total solids by oven drying methods. The orthogonal matrix of composition of the set of samples provides some interesting diagnostic and trouble shooting opportunities that are used to improve the performance of the laboratories that run the chemistry methods. The performance of individual laboratories and the group of laboratories for the chemistry methods has been improved. Second, the all-laboratory mean with outliers removed is used to create a fat, protein, and lactose reference value for each sample. Third, the samples are used for 1 month to set the slope and intercepts for each instrument. Because of the orthogonal matrix of composition, the data can be used to evaluate the linearity and intercorrection response of each instrument. These evaluation calculations and protocols are built into a software package we have written called IR-QC. Instrument Calibration Performance has been improved by using the modified milk calibration samples and all-lab mean reference values. The standard deviation of the difference between reference chemistry and instrument values on all components is < 0.015% and often < 0.01% using a traditional filter based calibration approach. The size of the 95% confidence interval around the slope of the regression line has been reduced greatly by the use of the modified milk calibration samples, compared to the performance that is achieved by using raw milks from individual farms for calibration. This is due to the homogeneity of the matrix of the modified milks and elimination of the influence of high leverage samples from the calibration set.

The network of laboratories does monthly pre-calibration performance evaluations of instrument performance. Homogenizer performance is monitored by a central laboratory at Cornell University using laser light scattering particle size analysis. Homogenizers that have failed the homogenization performance evaluation by particle size analysis are inspected by microscopic evaluation to determine the cause of failure.

In our research we have developed an optimized set of traditional "virtual" sample and reference filter wavelengths for use in FTIR instruments and we are in the process of publication of that information. We have also made a quantitative determination of the impact of variation in fatty chain length and unsaturation on Fat B and Fat A on absorbance at sample and reference wavelengths with a model sample system. That work is complete and in the process of publication. We continue to work toward the goal of improving the accuracy of the infrared milk testing to achieve the most accurate testing results on any instrument, on any sample, at any time.