

Overview of Component Testing of Milk in the USDA Federal Milk Markets

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Outline

- **Sample Handling for Reference Testing**
- **Reference vs. Secondary Methods**
- **Statistical Performance Characteristics of Methods**
- **Reference Methods**
- **Infrared Milk Analysis**

Outline

- **Sample Handling**
 - keep samples cold
 - protect from water contamination
 - warm sample (38°C) before removing test portion
 - inspect container for complete dispersion of fat (i.e., none stuck on sides)
 - mix by inversion

Outline

- **Sample Handling**
- **Reference vs. Secondary Methods**

Reference Methods

- **Methods that are sometimes considered as reference (or primary) methods differ from other methods because they do not use any material for calibration or adjustment of results. The result of the method defines the level of the compound in a sample.**

Secondary Methods

- **A secondary method is usually defined as a method that uses samples pre-analyzed by another method for calibration.**

Methods for Official Testing

- **The regulatory agency usually defines what method(s) is acceptable for use as a reference method for calibration of a secondary method.**

Examples of Reference Methods in the Federal Milk Marketing Orders

- **Fat: Mojonnier Ether Extraction**
- **Protein: Kjeldahl True Protein**
- **Total Solids: Forced Air Oven Drying**
- **Somatic Cell Count - DMSCC**
- **Lactose - Spectrophotometric Assay
using extinction coefficient to calculate
lactose content**

Examples of Secondary Methods

- **Infrared milk analysis - uses pre-analyzed milk samples.**
- **Somatic cell count by photoemission electronic cell counter - samples that have been pre-analyzed by DMSCC**
- **Microwave total solids - samples pre-analyzed by oven drying**

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Method Performance Statistics

- **Internationally Harmonized Statistics**
 - **Method Validation and Performance Documentation - Collaborative Study**
 - **Within lab (repeatability)**
 - S_r , RSD_r , and r-value
 - **Between lab (reproducibility)**
 - S_R , RSD_R , and R-value

Method Performance Statistics

example: milk - ether extraction

- Repeatability

- $S_r = 0.015$

- $RSD_r = (0.015/3.91) = 0.384\%$

- $r\text{-value} = 2.8 \times 0.015 = 0.042$

- Reproducibility

- $S_R = 0.020$

- $RSD_R = (0.020/3.91) = 0.512\%$

- $R\text{-value} = 2.8 \times 0.02 = 0.056$

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Reference Methods for Federal Milk Market Order Verification

- **Fat - ether extraction**
 - AOAC - method number 989.05
- **Kjeldahl - true protein**
 - AOAC - method number 991.22
- **Total Solids - Forced air oven drying**
 - AOAC - method number 990.20
- **Lactose – Enzymatic Method**
 - – AOAC – method number 2007.04

Other Solids

- **For payment testing in the Federal Milk Market Orders the component value of milk is the sum of the fat, true protein, and other solids value. A price per pound of each of these components is calculated monthly.**
- **There is no specific single test for other solids content of milk.**

What is Other Solids?

- **Lactose + minerals + miscellaneous minor organic compounds (e.g. urea, citric acid, creatinine, etc.).**

How is Other Solids Measured?

- **Reference Test for the Federal Milk Market Orders.**
 - **(Oven Drying Total Solids) minus (Fat by Mojonnier + True Protein by Kjeldahl)**

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Infrared Milk Analysis

- **Precalibration (published paper - JDS)**
- **Calibration – modified milk samples (i.e., UF calibration samples) – (2 published papers - JDS)**
- **Pilot Control Samples used During Routine Analysis**
- **Control milk sets of producer milk samples sent to payment testing labs**