AOAC SMPR 2020.XXX; Draft AOAC Standard Method Performance Requirements (SMPRs) for Targeted Testing (TT) of formaline/formaldehyde, starch, soy protein as Adulterants for Evaluation of Liquid Raw Bovine Milk; Version 3; February 13, 2020

Intended Use

AOACI *SMPRs*® describe the minimum recommended performance characteristics to be used during the evaluation of a method. The evaluation may be an on-site verification, a single-laboratory validation, or a multi-site collaborative study.

SMPRs are written and adopted by AOACI using the consensus of stakeholders representing the industry, government, and academic and/or research institutions. AOACI SMPRs are used by AOACI expert review panels (ERPs) in their evaluation of validation study data for method being considered for *Performance Tested Methods*^{SM} or AOACI *Official Methods of Analysis*^{SM} and can be used as acceptance criteria for verification at user laboratories.

1. Applicability

This document contains assessment parameters on the performance of Targeted Testing (TT) methods to monitor liquid raw bovine milk for the detected presence of Economically Motivated Adulterants (EMAs) such as formaline/formaldehyde, starch and soy protein.

2. Analytical Technique

A targeted method to be used to detect, identify and quantify liquid raw bovine milk for the detected presence of Economically Motivated Adulterants (EMAs) such as formaline/formaldehyde, starch and soy protein.

Any method capable of detecting, identifying the presence of the defined adulterants and quantifying the amount (proportion/concentration) present in the food item will be considered.

The scope of the TT method will be defined by the authentic samples and or reference standard material (if available) used in validating the method.

3. Definitions

Applicability Statement — a general statement about the intended purpose and scope of the method entailing key aspects of expected achievements for the specific situation and circumstances. Key points to cover are the intended matrix, the purpose, and an indication of sensitivity, selectivity, enforcement and action levels where available.

Authentic Samples — Samples representative of the genuine commodity. ~~Ideally~~ these samples should represent the food's or ingredient's variability seen naturally in the commodity. The authentic samples and or reference standard materials used to validate the method will be used to properly define the TT method testing scope.

Economically Motivated Adulteration —The fraudulent addition of non-authentic substances or removal or replacement of authentic substances without the purchaser's knowledge for economic gain of the seller.

Single Laboratory Validation (SLV) — Demonstration by one laboratory of method performance on samples described in Table 1.

Multi-laboratory Validation (MLV) — Demonstration between laboratories using adulterated samples created by a third-party group and supplied blindly to the participating laboratories.

4. Method Performance Requirements

(Table 1: Method Performance Requirements for formaline/formaldehyde in raw bovine milk)

Analytical Parameter	Acceptance Criteria
Analytical Range (µg/L [ppb])	10 – 400 µg/L (ppb)
LOQ	≥ 10 ppb
Recovery	80 – 120 %

NOTES:

1. A colorimetric method of 1906 vintage (Hehner's Test) which was modified by Richmond and Boseley was claimed to be able to easily detect 1 part of formaldehyde in 200,000 parts of milk (i.e 5 x 10^{-6} [5ppm]); but the test's response is enhanced by casein, lactoalbumin, and vanillin (all milk components). Test is therefore not selective to formaldehyde alone. SF Acree; J Biol Chem. (1906), v2:145-148 (Ref A).

2. A recent HPLC method with UV detection validated as per ISO 17025 claims to be able to detect formaldehyde residues in milk over an analytical range of 0.010 to 0.40 ppm (10 – 400 ppb) with an LOQ of 0.020 ppm (200 ppb); *Flavia Borges de Freitas Rezende et al. Microchemical Journal 2017 vol 134 pp 383-389 (Ref B)*

3. Three recently published methods using mid IR, direct infusion MS, and/or FTIR were all able to readily detect and quantify formaldehyde fortified to milk at 0.074 g/L (74 ppm). Gondim, CDS et al., Food Chem 2017, v230 pp 68-75 (Ref D); Tatiane Meline Guerreiro et al., Food Research International, volume 108 pp 498-504 (2018) (Ref E); Habib Asseiss Neto et al., BioData Mining (2019) volume 12 p13-? (Ref F)
4. I recommend an analytical range of 10 - 400 ppb for formaldehyde residues in bovine milk
5. Reference to authentic/reference standards (whenever available)

(Table 2: Method Performance Requirements for starch in raw bovine milk)

Analytical Parameter	Acceptance Criteria
Analytical Range (ppm)	1 - 10
LOQ	≥ 1
Recovery	80 – 120 %
Accuracy	± 20%

NOTES:
Reference publications D and E were both able to readily measure 5 g/L (5 ppm) of starch in milk.
Recommend we peg the analytical range to 1- 10 ppm as agreed upon in our last meeting

(Table 3: Method Performance Requirements for soy protein in raw bovine milk)

Analytical Parameter	Acceptance Criteria
Analytical Range (ppm)	1 - 5
LOQ	≥ 1
Recovery	80 – 120 %
Accuracy	± 20 %

NOTES:
We don't have a recent reference publication for the analytical range, but we can leave it at 1- 5 ppm as agreed upon

5. System Suitability Tests and/or Analytical Quality Control

Suitable methods will include blanks, and appropriate check standards.

6. Reference Materials

A detailed description of the process used to obtain and evaluate authentic/reference standard materials (sources), and of the test protocol used for validating the method must be provided.

7. Validation Guidance

a. Data demonstrating method performance is required.

b. Samples: Complete documentation for the collection and use of authentic samples must be supplied by the method authors. The scope of "authentic" samples used to validate the method must be applicable to the defined scope of the TT method. Expansion of the scope is possible with the inclusion of additional authentic samples and abbreviated validation using the protocol listed in this SMPR.

c. For single lab validation studies, the method will be evaluated using prescribed adulterated materials as shown in Table 1. Methods approved at this level will proceed to a second level of evaluation (multi-laboratory) where blinded samples containing unknown adulterants will be sent to participating laboratories.

d. Statistical analysis of interlaboratory studies. Sample size needed to meet performance requirement on proportion.

8. Maximum Time-to-Results

None.