

## Certifying Nothingness: Blank Matrix Reference Materials

Tuesday, September 15, 2009 3:00 p.m. EDT

Various well-characterized materials are required for the development, validation and verification of methods for the analysis of food and feed. This includes frequently matrix-matched materials which should "not contain" the analyte of interest. This symposium is devoted to the presentation and discussion of approaches by which such blank matrices can be qualified as certified reference materials (CRMs), which information has to be provided to the users and how such CRMs should be properly used in the analytical laboratory.

### Chairs:

CO-CHAIR: Hendrik Emons

Head of Unit

European Commission - Institute for Reference Materials and Measurements  
Geel, Belgium

CO-CHAIR: Donna L. Zink, Director

AIM Research Enterprises

Carthage, MO USA

### Speakers:

Hendrik Emons

European Commission - Institute for Reference Materials and Measurements  
Geel, Belgium

### Topic: Why to Certify and Use Blank Matrix Materials

Blank materials are required for the development, validation and performance control of methods for food and feed analysis. Such materials should sufficiently match the analytical behavior of the real test samples, but should not contain the analyte of interest to a detectable extent. Laboratories are often lacking reliable reference materials with such characteristics. Therefore, various reference material producers in the world are devoting efforts to characterise and certify appropriate blank matrix materials. This is a challenging tasks, as it does not only push the capabilities of current measurement methods to their limits, but requires also different concepts and tools for calculating the certified values and estimating their uncertainties. The presentation will explain the approaches of IRMM to resolve such problems and will illustrate experiences and challenges by various examples from mycotoxin to GMO analysis.

Steve Wood

LGC Limited

Middlesex, UK

### Topic: Nothing in it - how can we be sure? Critical steps in the production of blank RMs

New regulations and exposure guidelines are specifying ever lower limits for the presence of specific and generic analytes. Such limits test analytical measurement capability and challenge RM producers to provide well characterised CRMs for calibration and validation. To have confidence in the results of measurements of low analyte levels, blank RMs are necessary to help establish the limit of detection of

the method and to ensure that application of the method does not introduce any contamination of the analyte(s) under investigation.

The production of any reference material involves the following stages: specification, procurement and preparation of the units, homogeneity assessment, characterisation of the required analyte values, assessment of stability and the assignment of analyte value(s) and associated uncertainty. Following certification, the material must be stored appropriately and monitored regularly to confirm no significant changes have occurred.

The key challenges of producing blank RMs will be discussed with reference to the critical stages in the production some of some specific examples e.g. malachite green (MG) in fish; flame retardants in electrical and electronic components; chromium species in waters, perfluorooctane sulfonate (PFOS).

David Lee Duewer and Katherine E. Sharpless

Analytical Chemistry Division, Chemical Science and Technology Laboratory  
National Institute of Standards and Technology (NIST)  
Gaithersburg, MD USA

### **Topic: Blank? Who Needs a Blankety-Blank Blank?**

Blanks are samples that do not contain the analyte(s) of interest. Reagent blanks are typically just the materials used in the preparative steps of the measurement process and are analyzed to characterize the instrumental baseline signal. Method blanks contain all the sample matrix components except the analyte(s) and are used to confirm the absence of contamination throughout all stages of the measurement process. Method blanks are most critical when measurements must be made close to the detection limit and when “zero tolerance limits” are set by regulation. Certified method blanks, matrix reference materials that have been authoritatively characterized and documented to not contain the analyte at a higher-than-specified level, can facilitate the development and validation of many analytical systems; for some systems, they may be essential for documenting regulatory compliance.

The Analytical Chemistry Division of NIST supplies a number of method blank materials, both as stand-alone Standard Reference Materials (SRMs) such as SRM 2570 Lead Paint Film For Portable X-Ray Fluorescence Analyzers – Blank (Color Code: White) and as one component of a multi-level package as in SRM 1507b 11-Nor-Delta-9-Tetrahydrocannabinol-9-Carboxylic Acid in Freeze-Dried Urine. No matter how supplied, the identification, production, and certification of such blank materials can be challenging (and sometimes... quite aggravating.) We will survey some of the strategies used at NIST to meet past challenges and some of the novel issues we are currently facing.