



## APFAN PT-2 Workshop

Food Analysis Workshop: Proficiency Testing and Reference Materials Development

# Role of Reference Materials in Optimizing Reliability of Food Analysis

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APFAN Food Analysis Workshop:  
Proficiency Testing and Reference  
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## 1985 Florentino Survey of RM use in Asian food laboratories

- \* Survey included government, private, food industry and university labs involved food composition analysis in 12 countries in Asian region
- \* Only a third of respondents were aware of and used reference materials (presumably) to improve quality assurance
- \* Other respondents cited logistical reasons and/or unavailability of suitable RMs.
- \* So what progress has been made?

Ref: Frensenius Zeitschrift fur analytische Chemie, 1987, 326, Issue 7, 673-678

19<sup>th</sup> - 21<sup>st</sup> June 2019, Bangkok, THAILAND

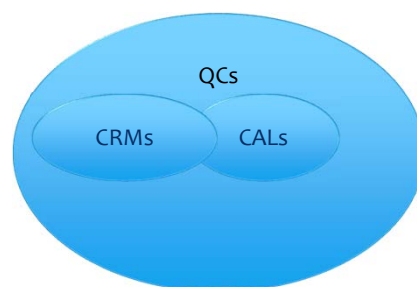
## What are reference materials?

ISO REMCO defines a reference material as :

- \* *a material which is sufficiently homogeneous and stable with respect to one or more properties, which has been established to be fit for its intended use in a measurement process.*
- \* The term RM is a generic description and includes CRMs, calibrants, primary standards and QCs (and RMs)

## What are RMs?

- \* The EC Institute for Reference Materials and Measurements (IRMM) subscribes to the use of an "RM Family" incorporating Calibrants, CRMs, RMs and QC materials

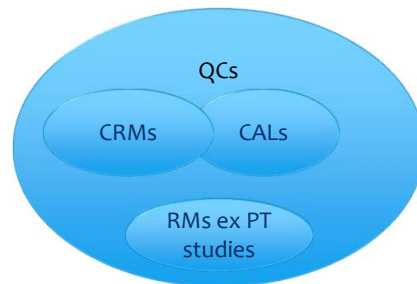


Ref:Accred Qual Assur (2006)  
10:576-578,690-691

The RM Family

## What are RMs?

- \* The EC Institute for Reference Materials and Measurements (IRMM) subscribes to the use of an "RM Family"
- \* Should the 'family' make room for another sibling named 'RMs ex PT studies'?



"The RM Family"

## CRMs and non-CRMs

A CRM is defined as

- \* "a reference material, characterised by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability". (ref ISO/IEC 34)



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## CRMs

- \* Available from accredited standard agencies e.g., NIST, IRMM, NMI
- \* Accompanied with certificates of certified values, measurement uncertainty and traceability
- \* Certified values maybe based on collaborative studies involving highly reputable laboratories using standard methods and/or a variety of measurement techniques
- \* Generally expensive and with limited certified analytes
- \* CRMs provide a valuable comparative basis for laboratories in establishing method accuracy

## Role of RMs

RMs are fundamental tools to laboratory quality assurance programs and may include:

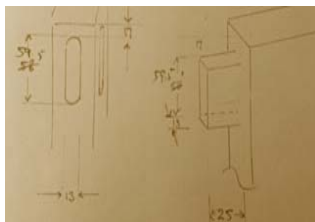
- \* In-house method development and validation/verification
- \* Calibration
- \* Staff training
- \* Internal quality control
- \* MU estimation
- \* Inter-laboratory collaborative and proficiency programs
- \* Validation and standardization of new methods and procedures

## Need for accuracy and precision in woodworking!

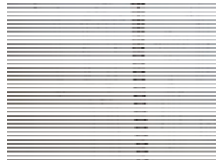
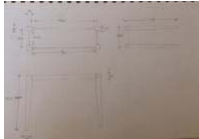
- \* Since retirement, I have taken an interest in wood working through membership of a Men Shed.
- \* A 'Men Shed' is an Australian concept where retired men meet at 'shed' or building specifically fitted-out to undertake activities such as wood and metal work, bicycle repair, art classes, music and just getting together over coffee to discuss politics, books and the latest 'tweets' of President Trump.
- \* concepts of accuracy and precision are not just the province of analytical chemists!



## Construction of a table - making use of a reference sample



Reference sample to 'calibrate'  
cutting saws to ensure accuracy and  
precision of table legs



## RM Characteristics

Ideally RM samples for food analysis have the following properties/characteristics:

- \* Stability for the duration of the intended period of use
- \* High level of homogeneity
- \* Compositional properties similar to the test samples
- \* Where available, assigned values based on reputable collaborative or PT studies
- \* Analytical values at similar levels to the test samples
- \* Readily available at reasonable/affordable cost

## RM Characteristics

Other considerations include:

- \* Ease of preparation
- \* Ease of sub-sampling into test portions
- \* Continuation of supply/availability (e.g., PT provider, food processors, retail super-market )
- \* Covers a number of analytes
- \* Ease of storage (solid, liquid, gas)
- \* Non-hazardous

## Selection of RMs

- \* Selection and preparation of RMs with specific analytes are based around perceived AQ/QC needs of laboratories.
- \* PT providers will prioritise RM development based on market needs for proficiency studies for current and new methods or for emerging analytes/contaminants.



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### Selection of RMs for Collaborative Studies

- \* Collaborative studies for MDV on new methods often require both blank and incurred (or spiked) matrix-based samples to identify possible 'false positives', particularly for low-level analytes, such as trace contaminants and allergens in foods.
- \* Such samples require a high degree of preparation and pre-testing to ensure integrity prior to distribution for testing

### In-house RMs

- \* RMs prepared for routine use as in-house QCs may not need the rigorous preparation of PT samples
- \* Homogeneity and shelf-life remain important
- \* Incurred client samples or 'off-the-shelf' food samples are often useful candidate RM samples due to low cost, on-going availability and, for liquids and many powdered solids, may require minimal preparation.
- \* Flour or cereal-based matrices are also useful materials for spiking with key analytes where commercial PT samples are unavailable

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## Commercial RMs

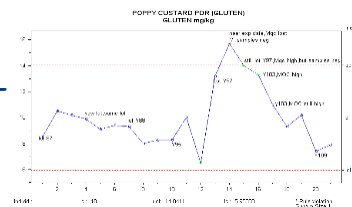


- \* PT providers are often an excellent source of RMs.
- \* PT samples are well prepared, have confirmed stability and levels of analytes of interest to many food laboratories.
- \* Surplus PT samples generally provide some statement on the levels of analytes including a CV and MU based on statistical analysis of results of participating laboratories.
- \* Some test-kit providers (ELISA) may also include suitable RMs as part of the kit for use as QC sample

## Use of RMs as QC Samples

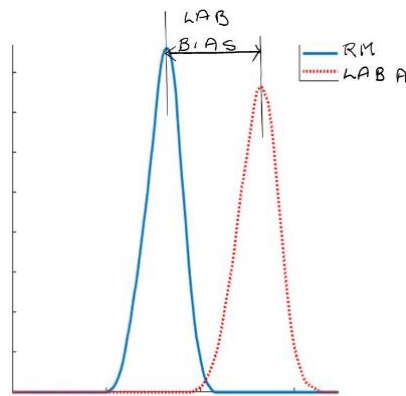


- \* The most practical and more common use of RMs is as QC samples
- \* IEC/ISO 17025 requires laboratories to employ QC samples and use real-time QC charts wherever possible to verify that the analytical process is 'in statistical control'



## Bias estimation from RM samples

- \* Use of RMs derived from PT studies which have assigned values may provide a reasonable measure of method bias as compared to the consensus mean value derived from the study participants
- \* In the absence of PT -derived samples, some indication of method accuracy can be derived from recoveries of spiked samples.



## Comparison of Precision from RM samples

- \* The precision (or CV) associated with RMs from PT studies provide a useful guide to the precision expected from replicate/reproducible analysis undertaken in-house.
- \* Unless the laboratory has limited experience in the analysis, the precision derived from in-house verification studies should be higher given fewer variables i.e., common method, instrumentation etc.
- \* the Horwitz relationship (HorRAT) provides a useful guide to the expected CV for the level of analyte in the sample although modern methods of analyses are expected to provide tighter CVs

## Estimation of MU from RMs

- \* PT derived RMs are generally assigned an MU based on the CV of the study.
- \* Where laboratories have a validated estimate of bias, this must be included in the MU estimate.
- \* Where labs have no reliable estimate of bias, the practical approach is to estimate the MU from CV and to incorporate a bias based on recoveries but this can be problematic particularly where recoveries are variable from sample to sample

## What to do when CRMs are unavailable ?

- \* For many analytes particularly for food allergenic proteins, CRMs are as yet unavailable (other than for total milk protein)
- \* Laboratories need to rely on spiking studies and PT samples for verification of key method characteristics such as LODs, LOQ/LORs, method precision and MU
- \* In regard to food allergens determined using indirect analysis based around ELISA, where recoveries between 50 to 150% are considered acceptable, CVs of around 25% are normal and the use of a default MU of 50% could be given consideration.



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### Summary comments

- \* RMs are essential tools for AQA
- \* Limited CRMs are available for food composition analysis
- \* PT programs are useful sources for food-matrix RMs with assigned values
- \* Matrix-based food RMs from robust PT studies are suitable candidates for establishing 'method' MU
- \* Practical workshops such as PT1 and PT2 are valuable teaching formats for technology transfer of PT organisation and statistical techniques.

### So what progress on the Florentino Survey Recommendations?

- \* Three well-established regional centres (Thailand, Malaysia and Philippines) are currently accredited to undertake PT studies relating to food analysis
- \* Regional PT studies have produced an increasing number of RMs many for redistribution
- \* Over the last decade or so, ASEAN and APFAN PT seminars and workshops have significantly increased the level of understanding of RM development and use within the Region
- \* Is it appropriate and timely to set up a central register and repository of regional food RMs for sale/distribution within the region?

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