



CCQM and BIPM programmes supporting accurate measurements on food and feed

R. Wielgosz (BIPM)

Overview

- 1. NMI Services supporting the Food Sector**
- 2. CCQM activities for the Food Sector (CCQM Strategy 2021-2030)**
- 3. BIPM Headquarters' activities supporting the CCQM Strategy**
 - a) Comparisons of calibration CRMs**
 - b) Mycotoxins, Veterinary Drugs, Pesticides CBKT**
 - c) eLearning and qNMR Summer School**
- 4. CCQM Task Group on Food Measurement**

NMI Measurements Services supporting the Food Sector

Metrology Tools to support accurate, traceable, validated measurement results



+ Proficiency Testing Schemes and sample value assignment

CCQM: Support for the Food Analysis Sector (2021-2030)

Sector	CCQM OAWG	CCQM PAWG	CCQM NAWG	CCQM CAWG	CCQM SAWG	CCQM EAWG	CCQM IAWG	CCQM IRWG	CCQM GAWG
Climate & Environment	POPs Contaminants Microplastics Water/Soil		Species/ microbial surveillance			Seawater pH and salinity	Heavy Metal Contaminants Speciation Water/Soil	GHGs	GHGs Air Quality Emissions Particles Breath diagnostics
Health & Life Sciences	Diagnostic biomarkers Forensics Anti-doping	Diagnostic biomarkers Therapeutics	Diagnostic biomarkers Gene Therapy	Diagnostic biomarkers	Imaging diagnostics Biocompatibility In-vitro diagnostic devices	Diagnostic biomarkers	Diagnostic biomarkers Toxic Elements	Diagnostic biomarkers Forensics Anti-doping	
Food Safety	Toxins Contaminants Residues Authentication	Allergens Authentication	GMO-Foods Pathogens	Pathogens	Packaging materials		Heavy metal Contaminants Speciation	Food authentication	
Energy					Batteries Fuel/Solar cells Catalysts	Batteries Fuel Cells	Fuel Contaminants		Natural Gas LPG/LNG Hydrogen Biofuels
Advanced Manufacturing		Advanced Therapy Development	Biotechnology	Advanced Therapy Development	Nanotechnology Semiconductors Quantum devices	Nanotechnology	Nanotechnology		Trace Gases
Digitalization				Digital Pathology				Isotope Ratio Scale defining RMs Database	GHG Scales Database & Management

Inorganic Measurements in Food (CCQM-IAWG)

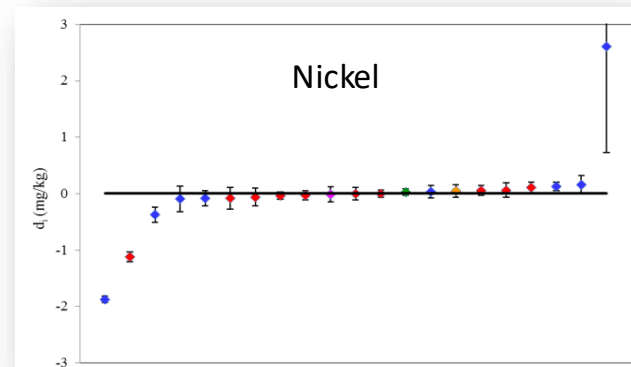
Maximum levels of metallic contaminants in food

Elements in Rice Flour (CCQM-K158)

Rice is consumed as the main foodstuff for about half of the world's population



Toxic and essential elements in bovine liver (CCQM-K145)



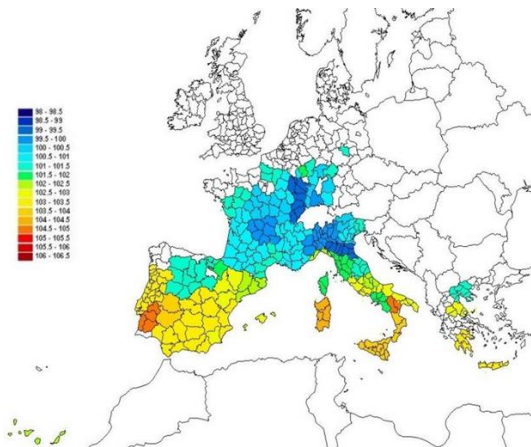
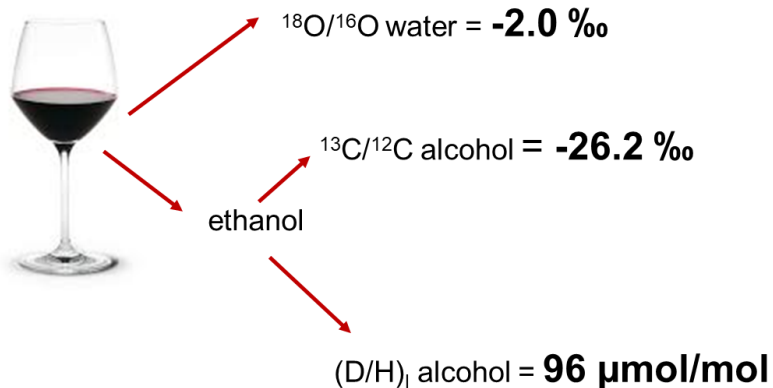
Arsenic in Seafood (CCQM-P215)

CODEX sets maximum levels of metallic contaminants in seafood to protect public health

- Cadmium 2 mg/kg in bivalve molluscs and cephalopods
- Lead 0.3 mg/kg in fish
- Mercury 1.2 mg/kg in fish
- Arsenic 0.1 mg/kg in fish

Isotope Ratio Measurements in Food (CCQM-IRWG)

Food Authenticity



Year	Method	product	Method	Isotope Ratio	Fraud
2001	OIV 17/2001	wine, must	IRMS	$^{13}\text{C}/^{12}\text{C}$	sugar addition (cane)
2003	EU Reg. 2676/90, 440/03	wine, must	IRMS	$^{13}\text{C}/^{12}\text{C}$	sugar addition (cane)
2003	OIV MA-F-AS314-03	wine	IRMS	$^{13}\text{C}/^{12}\text{C}$	technogenic CO_2

Measurements of GMOs (CCQM-NAWG)

Identification/Quantification of presence of GMO food products

- in support of regulatory environment across the world



Metrologia

Final report for CCQM-K86.b relative quantification of Bt63 in GM rice matrix sample

Lianhua Dong¹, Zhiwei Sui¹, Jing Wang¹, Vincent H M Tang², Winnie W Y Chum², Foo-wing Lee², Della W M Sin², Melina Pérez-Urquiza³, Malcolm Burns⁴, Stephen L R Ellison⁴ [+ Show full author list](#)

©2018 BIPM & IOP Publishing Ltd

[Metrologia, Volume 55, Number 1A](#)

Citation Lianhua Dong *et al* 2018 *Metrologia* 55 08017

DOI 10.1088/0026-1394/55/1A/08017

Metrologia

Final report of CCQM-K86.c. Relative quantification of genomic DNA fragments extracted from a biological tissue

Zoltan Mester¹, Philippe Corbisier², Stephen L R Ellison³, Yunhua Gao⁴, Chunyan Niu⁴, Vincent Tang⁵, Foo-wing Lee⁵, Melina Pérez-Urquiza⁶, Angel Ramirez Suárez⁶, Malcolm Burns³ [+ Show full author list](#)

©2020 BIPM & IOP Publishing Ltd

[Metrologia, Volume 57, Number 1A](#)

Citation Zoltan Mester *et al* 2020 *Metrologia* 57 08004

DOI 10.1088/0026-1394/57/1A/08004

Organic Measurements in Food (CCQM-OAWG)

Maximum levels of organic contaminants in food

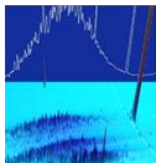
**Pesticide Residues:
Current State-of-
the-Art and
Emerging Issues**



**Veterinary drug residues and
drug metabolites in food**



Mycotoxin analysis, current state-of-the art and emerging issues

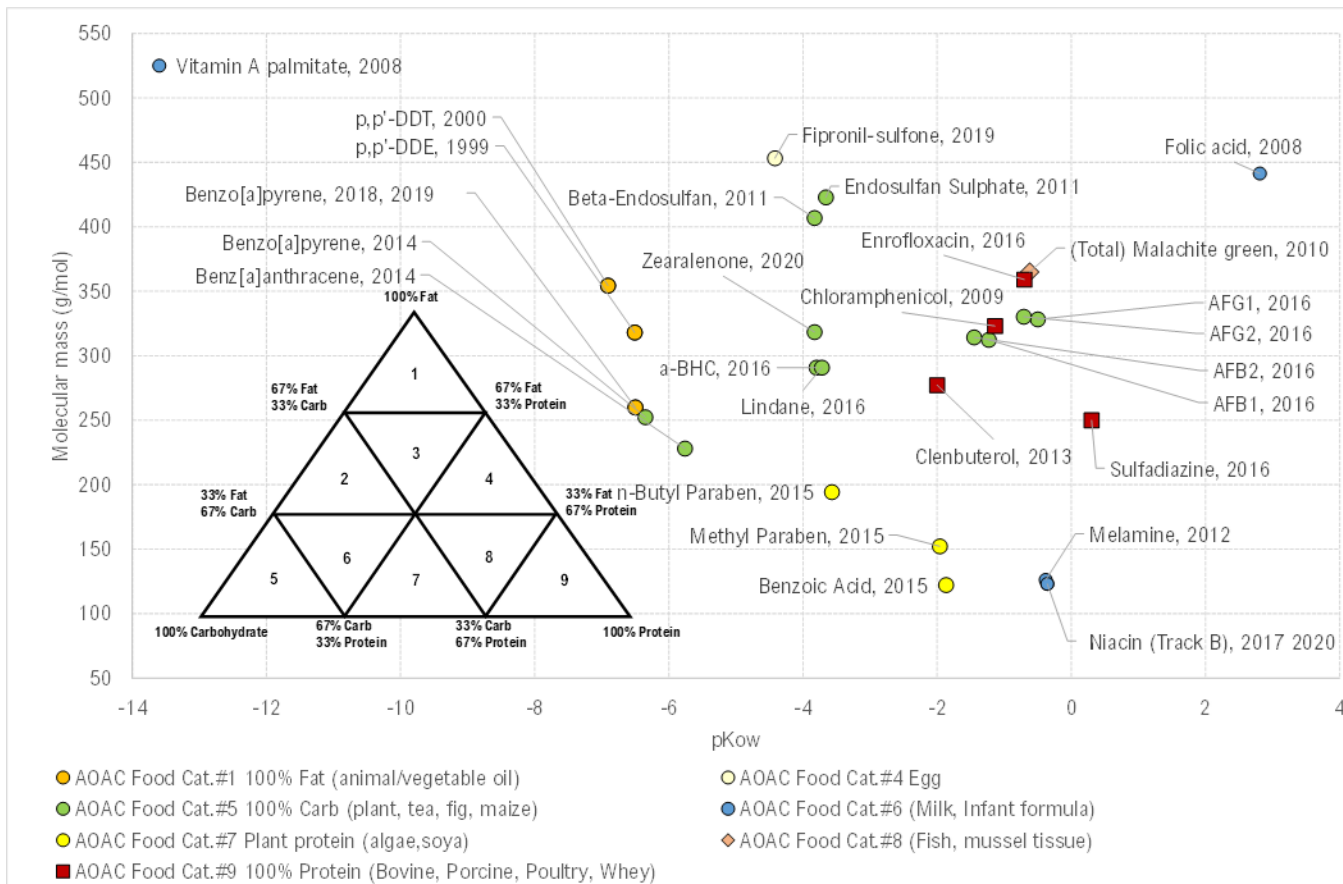


Packing contaminants in food:

The analysis of *mineral oil saturated hydrocarbons* (MOSH) and *mineral oil aromatic hydrocarbons* (MOAH) as an example

Organic Measurements in Food (CCQM-OAWG)

Food sector CCQM OAWG comparisons



BIPM Headquarters' support for CCQM Food Activities

Liaison /Coordination

The Inter-Agency Meeting (IAM)



World Metrology Day 2023:
'Measurements supporting the
global food system'

CCQM Task Group on Food Measurement (2023)

Specific activities to be undertaken by the
Task Group:

To develop a document describing the
CCQM strategy and work programme of
2021-2030 within the field of food safety

Comparisons

Contaminants in Food:

- Toxins
- Drugs
- Pesticides



Calibrants:



Knowledge Transfer

BIPM qNMR Summer School (2024)

A qNMR Summer School at the BIPM Headquarters is provisionally planned for 24-28 June 2024 for NMR practitioners in NMIs to enhance their knowledge and skills in qNMR as a robust methodology for the value assignment of organic reference materials. The onsite course will provide a hands-on training opportunity and accompanies the online knowledge transfer modules that will be available on BIPM's eLearning platform in 2024.

The course is suitable for scientists at NMIs that already have practical experience in the use of NMR and access to a spectrometer within their home institute. There is no charge for the course, but travel and accommodation costs will need to be met by participants.

Potential participants can [register their interest by completing the following form before 31 August 2023](#). Registration for the course is planned to open at the end of 2023. Space is limited to 12 participants.

qNMR Summer School

qNMR Summer School	
When and where <ul style="list-style-type: none">• Summer 2024 (1 week duration)• BIPM Headquarters (Sèvres, France)	Participant requirements <ul style="list-style-type: none">• Practical and theoretical knowledge of NMR• Access to an NMR spectrometer in their home institution
Key topics <ul style="list-style-type: none">• Sample preparation• qNMR experiment set-up• Data analysis and uncertainty evaluation	Organization <ul style="list-style-type: none">• Experienced instructors from NMIs and the BIPM• 12 places available• Networking opportunities

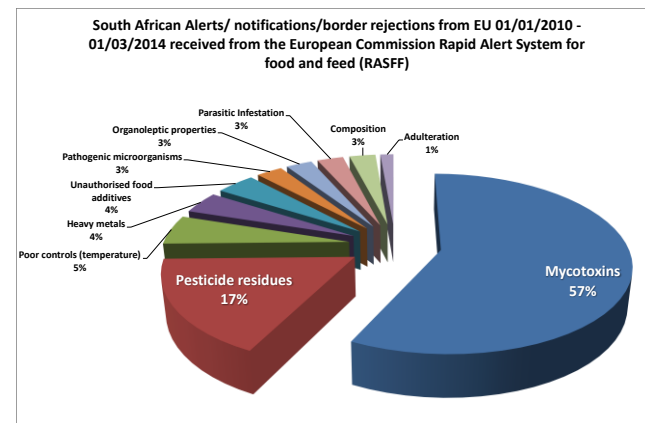
Description of course:

- Led by experienced external instructors from National Metrology Institutes and the BIPM
- qNMR sample preparation
- NMR spectrometer set-up for qNMR
- Use of MNova software for data processing and analysis
- qNMR of different types of samples and nuclei (^1H , ^{19}F and ^{13}C)
- Choice of suitable internal standards
- Measurement uncertainty
- Alternative calibration methods
- 2D NMR for impurity confirmation
- Use of orthogonal techniques to correct qNMR results

Developing Economy Needs Expressed by AFRIMETS (2014)

Food and Agriculture Organisation (FAO) estimates in Asia and Africa, 8–18 % of cereals are lost during postharvest handling and storage, the majority due to fungal growth and contamination with mycotoxins.

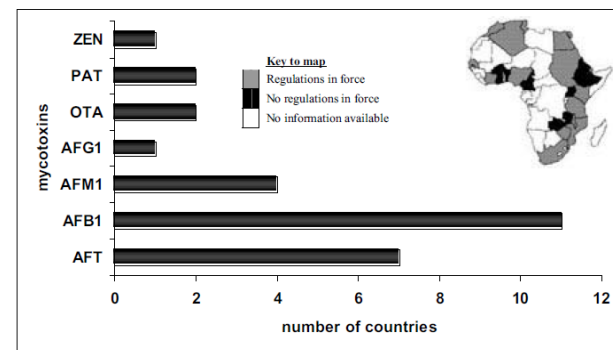
AFRIMETS has identified the regional need for certified reference materials to support its mycotoxin in food analysis requirements.



Children under 15 at huge risk of aflatoxicosis in Kenya—study

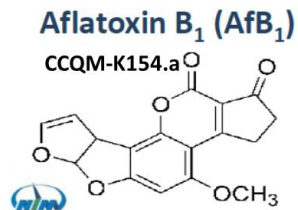
SATURDAY NOVEMBER 16 2013

Bureau
International des
Poids et
Mesures

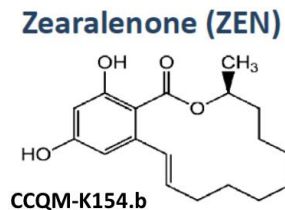
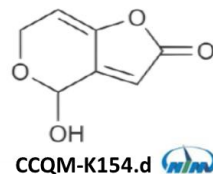


BIPM CBKT: Mycotoxin Metrology (started 2016) **CBKT**

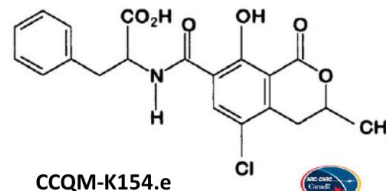
- ◆ The CBKT project is designed to allow the BIPM and National Metrology Institutes (NMIs) to work together to:
- enable NMIs to characterize selected pure mycotoxin materials, provide mycotoxin calibrants and matrix reference material and proficiency test materials to support mycotoxin testing laboratories within their countries.



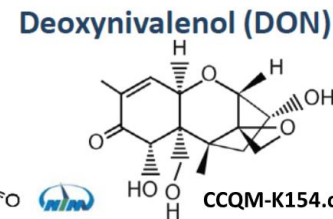
Patulin (PAT)



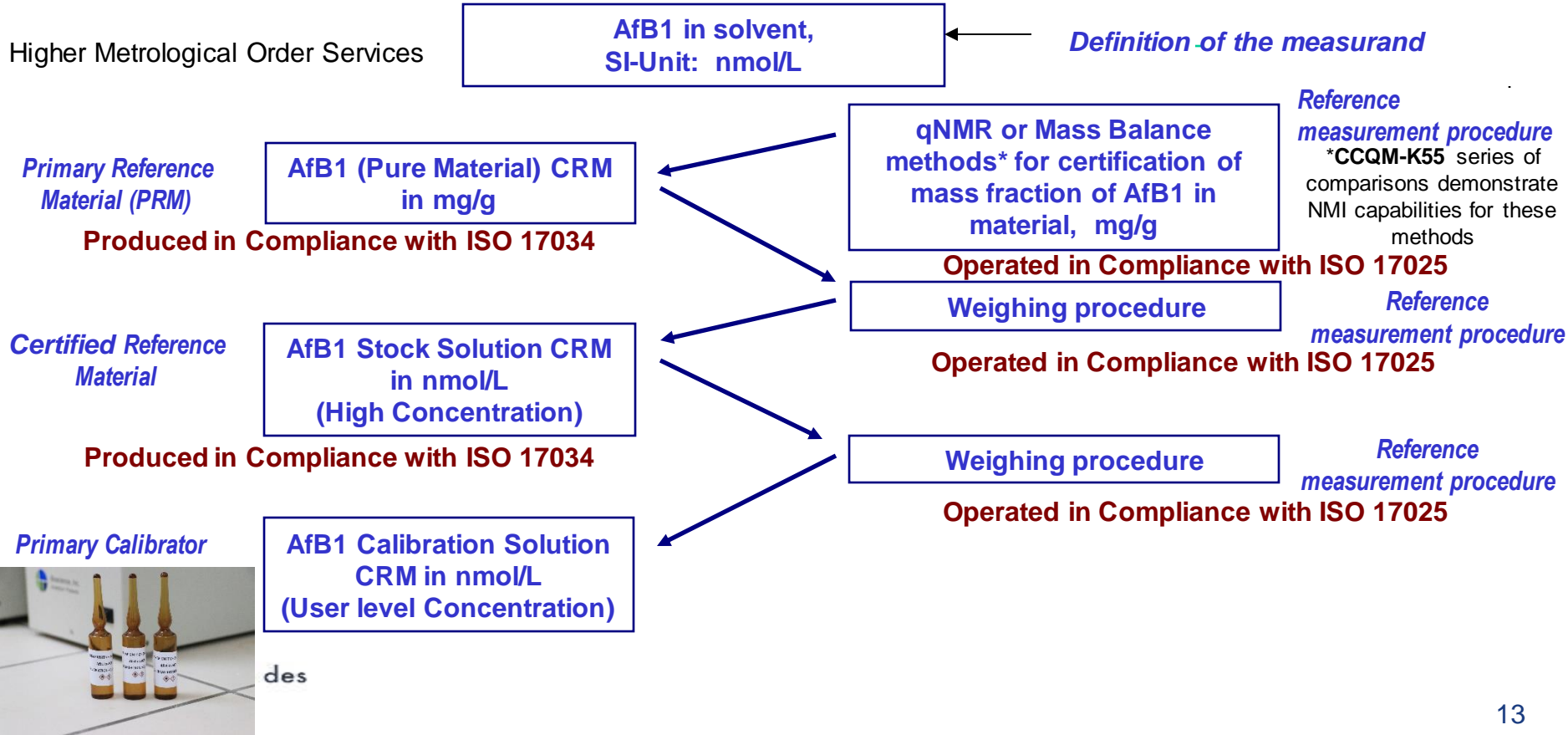
Ochratoxin A (OTA)



(2024)



Mycotoxin Primary Reference Materials and Calibrators



Purity evaluation guidelines for Mycotoxin Pure CRMs

Rapport BIPM-2021/01



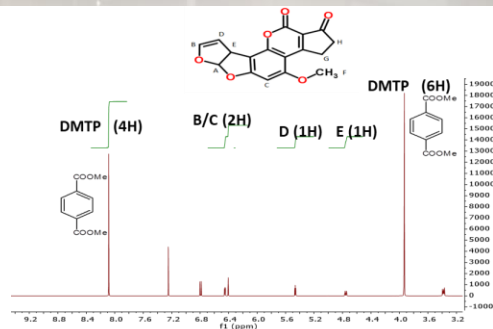
Bureau
International des
Poids et
Mesures

Purity Evaluation Guideline: Aflatoxin B₁

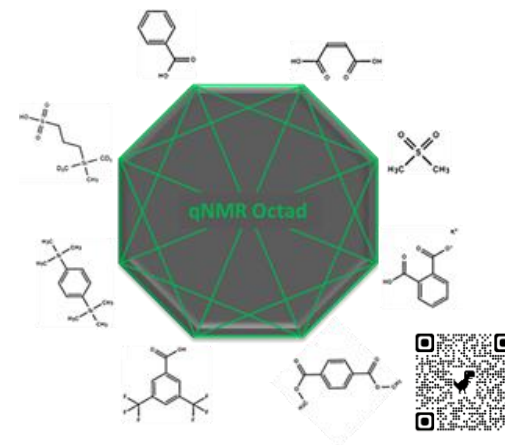
BIPM PEG-02

Authors: Steven Westwood (BIPM); Ralf Josephs (BIPM), Gustavo Martos (BIPM), Tiphaine Choteau (BIPM), Xiuqin Li (NIM China); Xiaomin Li (NIM China); Xhen Guo (NIM China); Xianjiang Li (NIM China); Bruno Garrido (INMETRO, Brazil); Ilker Un (TUBITAK UME, Turkey)

Version 1.0 : February 16th 2021



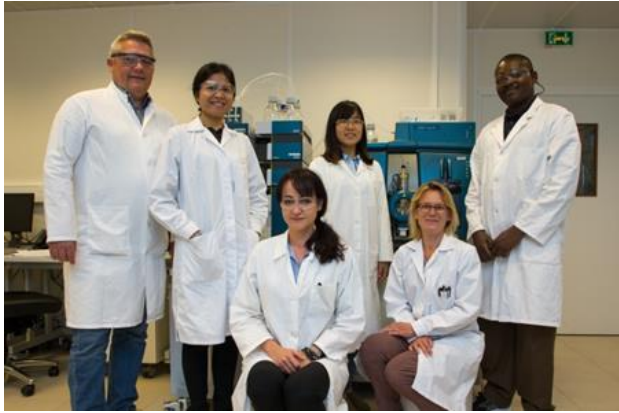
qNMR or Mass Balance methods* for certification of mass fraction of AfB₁ in material, mg/g



Internal standards for qNMR (NMIJ-BIPM)



Onsite/online Knowledge Transfer by BIPM



**Training Secondments at the BIPM
TS4: Calibration Solution
Preparation and Value Assignment
12 week Training Secondment**

**PTB support for 4
visiting scientists**



Week	Description
1	Safety and Quality documentation introduction
2-3	Accurate weighing, uncertainty calculation, ZEN ampoule solution preparation
4-5	LC-UV instrument operation and implementation of ZEN analysis method
6-7	Homogeneity testing and calculation of between ampoule variability
8-9	Stability testing and evaluation of results
10-11	Value assignment of unknown calibration solution
12	Finalisation of secondment report



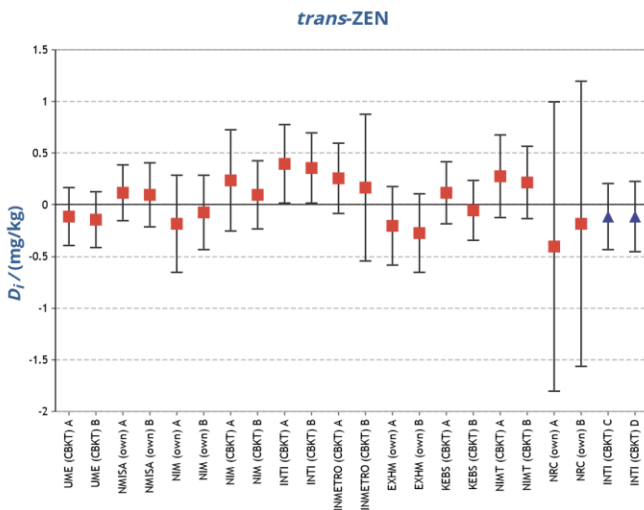
**NON STRUCTURE-RELATED
IMPURITY CONTENT IN ORGANIC
PURE MATERIALS**

The course is intended for NMI scientists working in organic analysis and wishing to further their theoretic...

**Online course offered to
over 100 participants**



Demonstration of Measurement Capabilities



Metrologia

KEY COMPARISON

Key comparison study - organic solvent calibration solution - gravimetric preparation and value assignment of deoxynivalenol (DON) in acetonitrile (ACN)

R D Josephs¹ , M Bedu¹, A Daireaoux¹, Z Guo^{1,2}, Xianjiang Li^{1,2}, Y Gao^{1,2}, Xiuqin Li^{1,2}, T Choteau¹, G Martos¹ , S Westwood¹  + [Show full author list](#)

Published 16 December 2022 • © 2022 BIPM & IOP Publishing Ltd

[Metrologia, Volume 60, Number 1A](#)

Citation R D Josephs *et al* 2023 *Metrologia* 60 08002

DOI 10.1088/0026-1394/60/1A/08002

Metrologia

Key comparison study—organic solvent calibration solution—gravimetric preparation and value assignment of trans-zearalenone (*trans*-ZEN) in acetonitrile (ACN)

R D Josephs¹, A Daireaoux¹, M Bedu¹, Xiuqin Li^{1,2}, Xiaomin Li^{1,2}, Z Guo^{1,2}, T Choteau¹, G Martos¹, S Westwood¹, R I Wielgosz¹ + [Show full author list](#)

©2020 BIPM & IOP Publishing Ltd

[Metrologia, Volume 57, Number 1A](#)




Citation R D Josephs *et al* 2020 *Metrologia* 57 08019

DOI 10.1088/0026-1394/57/1A/08019

Metrologia

KEY COMPARISON

Key Comparison Study - Organic Solvent Calibration Solution - Gravimetric preparation and value assignment of aflatoxin B1 (Afb1) in acetonitrile (ACN)

R D Josephs¹ , M Bedu¹, A Daireaoux¹, Xiuqin Li^{1,2}, Xiaomin Li^{1,2}, Z Guo^{1,2}, Xianjiang Li^{1,2}, T Choteau¹, G Martos¹ , S Westwood¹  + [Show full author list](#)

Published 1 January 2022 • © 2022 BIPM & IOP Publishing Ltd

[Metrologia, Volume 59, Number 1A](#)

Citation R D Josephs *et al* 2022 *Metrologia* 59 08002

DOI 10.1088/0026-1394/59/1A/08002

Outputs: Accurate Calibrants for Food Safety testing

The image is a collage illustrating the global availability of food safety calibrants. At the center is a world map with blue circular callouts in North America, Europe, Asia, and South America. The text "Locally accessible standards" is overlaid on the map. Surrounding the map are several photographs of calibrant products:

- Top Left:** A large white bag of MYCO-1 and a white plastic bottle with a blue cap, also labeled MYCO-1. Several small glass ampoules are shown in front.
- Top Center:** Three brown glass ampoules with white labels, each containing a yellowish liquid. The labels include "MPT-CO-08-05-21", "MPT-CO-08-05-21", and "MPT-CO-08-05-21".
- Top Right:** A row of seven clear glass ampoules with white labels, each containing a clear liquid.
- Bottom Left:** Four clear glass ampoules with white labels, each containing a clear liquid. The labels include "MPT-CO-08-05-21" and "MPT-CO-08-05-21".
- Bottom Center:** Five small glass ampoules with white labels, each containing a yellowish liquid. The labels include "nmis" and "MPT-CO-08-05-21".
- Bottom Right:** Four small brown glass ampoules with white labels, each containing a clear liquid. The labels include "Reference Mix M-S-5036", "Reference Mix M-S-5037", "Reference Mix M-S-5038", and "Reference Mix M-S-5039".

Institutes

Summary of response

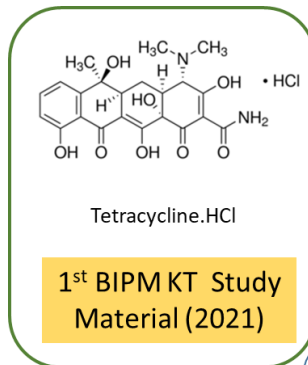
NMISA

Antibiotics in poultry: TETRACYCLINES

Africa Food Safety Network (AFoSaN)

- Tetracyclines in chicken matrix RM**
 - ethical clearance delays for dosing study by ARC-OVR
 - Chlortetracycline, doxycycline, epitetraacycline, doxytetracycline and tetracycline
 - Morocco to assist with incurred materials by Q3
- Training on in-house RM production at NMISA**
 - Incurred materials
 - Nigeria (aflatoxins in peanut butter)
 - Morocco (tetracyclines in chicken)
 - Botswana (penicillins in beef, *spiked*)
 - Benin (pesticides in cassava)
 - July-Sept 2019
 - Similar training planned for phase III of RAF 5078 AFoSaN project (2020-2023)

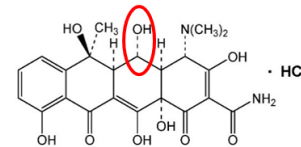
© NMISA 2019 / TQM-0161-A



500 mg
Food Safety CBKT Programme
Tetracycline.HCl

Unit No 001
BIPM OGP.039

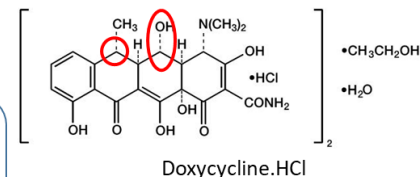
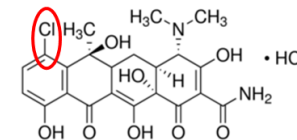
Health Hazard
SDS available
Laboratory use only



CCQM-K148.b
Material (2023)

UME

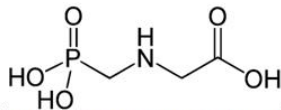
Bureau International des
Poids et
Mesures



CBKT Pesticide project includes:

- **Glyphosate** – highly regulated use in EU, concern for human and animal health, particularly impact on bee population
- **Endosulfan** – global ban since 2012 for acute toxicity, persistent in the environment and role as an endocrine system disruptor
- **Dimethoate** – neurotoxic, banned in EU 2019, recently (Sep 23) banned in Australia

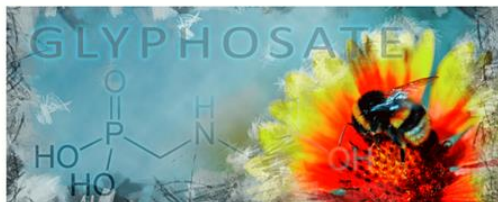
In collaboration with NIM (China)



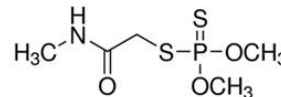
France court bans two glyphosate-based pesticides

Share

Submitted by pane on May 22, 2023 - 12:56

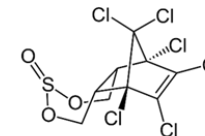


The Administrative Court of Montpellier banned two glyphosate products marketed by Syngenta. The company did not submit the mandatory risk assessment on the impacts on bees, other insects, soil and water life. For this reason, re-authorisation should not have



Dimethoate use suspended after insecticide residue found to exceed safety levels

By national regional reporter Nathan Morris
Posted Fri 22 Sep 2023 at 11:36pm, updated Tue 26 Sep 2023 at 2:03am



Endosulfan Banned Worldwide

Persistent Pollutants: Certain uses of pesticide can continue until 2017

by Cheryl Hogue
May 5, 2011 | A version of this story appeared in Volume 89, Issue 19

Countries have agreed to a global phaseout of endosulfan, an environmentally persistent pesticide that can cause neurological and reproductive problems in people and wildlife.

More than 120 nations struck the deal on April 29 under the Stockholm Convention, an international treaty for controlling persistent organic pollutants. They endorsed the endosulfan ban after five days of negotiations in Geneva.

Under the deal, most uses of endosulfan will cease in 2012. However, this organochlorine insecticide may be used on certain combinations of crops and pests until



Protesters in the Philippines expressed their support for a global endosulfan ban while negotiations took place in Geneva.

qNMR eLearning and Summer School (2024)



- qNMR online eLearning course in development
- Pure Organic Standard Value Assignment
- Onsite practical summer school

BIPM qNMR Summer School (2024)

A qNMR Summer School at the BIPM Headquarters is provisionally planned for 24-28 June 2024 for NMR practitioners in NMIs to enhance their knowledge and skills in qNMR as a robust methodology for the value assignment of organic reference materials. The onsite course will provide a hands-on training opportunity and accompanies the online knowledge transfer modules that will be available on BIPM's eLearning platform in 2024.

The course is suitable for scientists at NMIs that already have practical experience in the use of NMR and access to a spectrometer within their home institute. There is no charge for the course, but travel and accommodation costs will need to be met by participants.

Potential participants can [express their interest by completing the following form before 31 August 2023](#). Registration for the course is planned to open at the end of 2023. Space is limited to 12 participants.

qNMR Summer School

When and where

- Summer 2024 (1 week duration)
- BIPM Headquarters (Sèvres, France)

Participant requirements

- Practical and theoretical knowledge of NMR
- Access to an NMR spectrometer in their home institution

Key topics

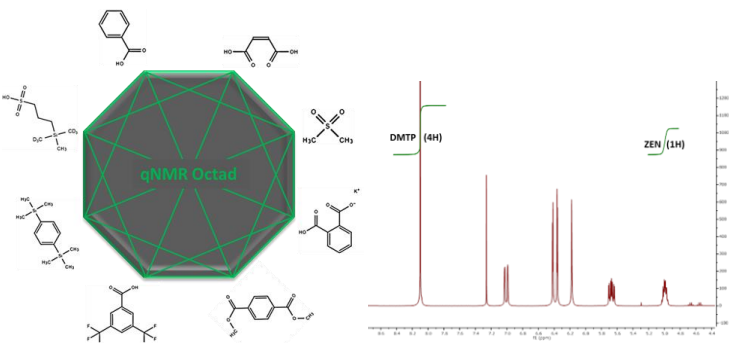
- Sample preparation
- qNMR experiment set-up
- Data analysis and uncertainty evaluation

Organization

- Experienced instructors from NMIs and the BIPM
- 12 places available
- Networking opportunities

Description of course:

- Led by experienced external instructors from National Metrology Institutes and the BIPM
- qNMR sample preparation
- NMR spectrometer set-up for qNMR
- Use of MNova software for data processing and analysis
- qNMR of different types of samples and nuclei (^1H , ^{13}C and ^{31}P)
- Choice of suitable internal standards
- Measurement uncertainty
- Alternative calibration methods
- 2D NMR for impurity confirmation
- Use of orthogonal techniques to correct qNMR results



CCQM Task Group on Food Measurement

Being established in 2023, with draft tasks:

- To develop a document describing the CCQM strategy and work programme of 2021-2030 within the field of food safety and measurement, as well as activities in the RMOs and in capacity building and knowledge transfer, based on the published CCQM 2021-2030 strategy, that can be used as publicly accessible reference to describe how the international metrology community is planning to meet measurement needs related to food and food safety;
- To identify gaps in measurements needs related to food and food safety not yet identified in the CCQM 2021-2030 strategy, and propose how the strategy could be updated to address these;
- To identify unmet stakeholder engagement opportunities and how these could be exploited to promote the benefits of metrology and NMI services or used to identify new measurement needs;
- To liaise with other CCs and identify measurements related to food and food safety covered by activities in these communities that could be incorporated into broader document covering food measurements issues and needs including those outside chemical and biological measurements.

Acknowledgements:

- All participating institutes
- Visiting Scientists and provision of materials: NIM (China), UME (Turkey), NMIJ (Japan), NRC (Canada), PTB (Germany)

Thank you for your attention

Robert Wielgosz,
International Bureau of Weights and Measures (BIPM)