



GERMANY

**Deutsches Nationalkomitee
im Internationalen
Milchwirtschaftsverband - IDF
Verband der Deutschen
Milchwirtschaft e. V. - VDM**

„Open Day“ 2011

**Offenes Diskussionsforum
des Deutschen Nationalkomitees
im Internationalen Milchwirtschaftsverband – IDF**

Berlin, 16. März 2011

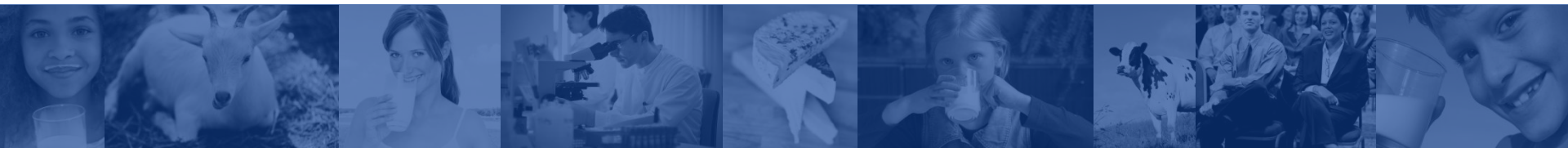


Integrierte Qualitäts- und Sicherheitsmanagementsysteme in der Milchwirtschaft

IDFs Fallstudien und Reaktionen auf Lebensmittelskandale

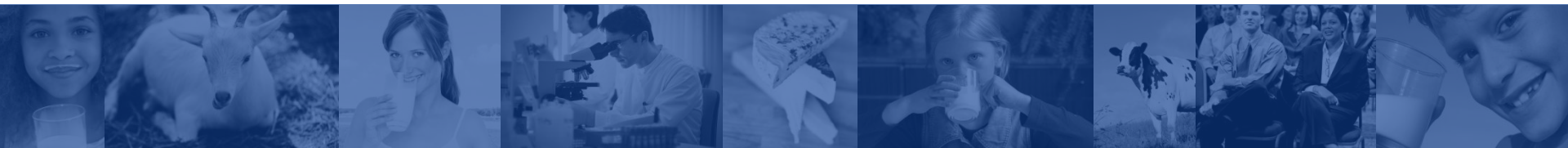
Dr. Petra Lutter

IDF Member of the Standing Committee on
Analytical Methods for Composition





Speedy Response to the Melamine Challenge



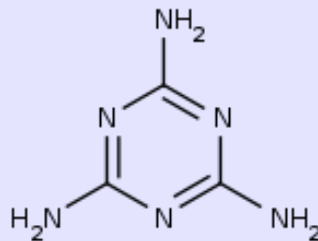
Contents



1. Past Melamine Issues
2. IDF SWIFT driven Process for a new Melamine Guideline
3. Key Events in the Evolution of the Document
4. What would have happened if IDF would not have picked it up?

Past Melamine Issues

- Renal failure in pets in the Republic of Korea (2004)
- Renal failure in pets in the United States of America (2007)
- Kidney stones in infants fed with Sanlu products (Sept. 2008)



Deliberate melamine addition to boost apparent protein content

Minimum Milk Protein Level in China

Maximum Melamine Level



- 75% - 90% of raw milk in some provinces had failed to reach the old protein level standard in 2007 and 2008.
(*South China Morning Post*, 14 July 2010)
- June 2010, Beijing lowered the mainland's minimum protein level for raw milk, from 2.955 to 2.8%.
- WHO: "Tolerable Daily Intake" (TDI) is 0.2 mg melamine per kg of body mass.
- Codex Alimentarius: max. melamine allowed in powdered infant formula is 1 mg/kg and 2.5 mg/kg in other foods and animal feed.

Methodologies for Melamine Determination

On-field



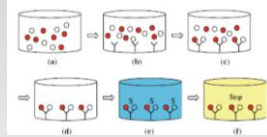
Rapid tests
(Lateral flow)

On-line



Spectroscopy
(Raman, SERS,
NIR)

Factory lab



ELISA



HPLC

Other QC lab



UPLC & HPLC



DART-TOF



GC-MS



LC-MS/MS

Simple & rapid

Complex & accurate

Product Controls & Recalls



- Milk-containing products were recalled in different countries because they were tested positive for melamine
- Food manufacturers and ingredient suppliers disagreed because they tested their products negative.
- Applied methods were not always compatible with the food matrix or not fully validated.

Need of an international reference method was identified !

« SWIFT »



- « SWIFT » is the new driver for the further development of IDF
- « SWIFT » = **S**peedy, **W**orldwide visible, **I**mpactful, **F**ocused and **T**ransparent
- The five key concepts ensure that the organization continues to deliver timely outcomes that meet expectations and the changing needs of IDF membership.



- *ISO Technical Specifications/IDF Review Method document*
 - This new ISO/IDF TS gives guidance on sampling, test procedures, performance and examples for the determination of melamine and cyanuric acid using LC-MS/MS.
 - Two example methods are given in the annex chapters
 - Co-managed by S. Holroyd (NZ) and T. Delatour (CH)

Guideline with two Example Methods



Annex A (informative)

Example A — Cow milk and milk-based infant formula — Simultaneous quantitative determination of melamine and cyanuric acid by liquid chromatography electrospray ionization tandem mass spectrometry

A.1 Scope

This annex specifies an in-house validated method for the quantitative determination of melamine and cyanuric acid contents in cow milk (CM) and milk-based powdered infant formula (PIF) by electrospray ionization liquid chromatography tandem mass spectrometry (LC-MS/MS) using the selected reaction monitoring (SRM) mode.

The positive identification of melamine and cyanuric acid in the confirmation criteria defined in EU Commission Decision 2002/657/E

The quantification is performed by an isotopic dilution approach using labelled ($^{13}\text{C}_3$, $^{15}\text{N}_3$)-cyanuric acid as internal standards (ISs). For proficiency-test results, see Reference [10].

The method allows an accurate quantification at the following reported

- melamine and cyanuric acid contents in CM at levels of 0,05 mg
- melamine and cyanuric acid contents in PIF at levels of 0,05 mg
- melamine and cyanuric acid contents in PIF at a level of 1,00 mg

TECHNICAL
SPECIFICATION

ISO/TS
15495

IDF/RM
230

First edition
2010-##-##

Milk, milk products and infant formulae —
Guidelines for the quantitative
determination of melamine and cyanuric
acid by LC-MS/MS

*Lait, produits laitiers et formules infantiles — Lignes directrices pour la
détermination quantitative de la mélamine et de l'acide cyanurique par
CL-SM/SM*

ISO/TS 15495:2010(E)
IDF/RM 230:2010(E)

Annex B (informative)

Example B — Quantitative analysis of melamine and cyanuric acid in milk-based infant formula and cow milk by liquid chromatography using linear ion-trap mass spectrometry

B.1 Scope

This annex of this Technical Specification specifies a method involving the determination of melamine and cyanuric acid in milk-based infant formula and cow milk by the use of a linear ion-trap tandem mass spectrometry (LC-MS/MS) method with an electrospray ion source and in selected reaction monitoring (SRM) mode. Quantification is performed by the isotopic dilution approach using labelled ($^{13}\text{C}_3$, $^{15}\text{N}_3$)-melamine and labelled ($^{13}\text{C}_3$, $^{15}\text{N}_3$)-cyanuric acid as internal standards (ISs).

Key Events in the Evolution of the Document



IDF World Dairy Summit, 20-24 September 2009, Berlin, Germany



IDF World Dairy Summit, 4-11 November 2010, Auckland, NZ



H2 2008

H1 2009

H2 2009

H1 2010

H2 2010

« **Melamine incident** »
2008

IDF/ISO Analytical Week,
18-22 May 2009, Sochi, Russia



IDF/ISO Analytical Week,
17-21 May 2010, Montreal,
Canada

Key Dates in the Evolution of the Document



April 2009

- New IDF Work Item was proposed and distributed to National Committees

October 2009

- Action Team sends 1. DRAFT to the National Committees for approval

May 2010

- IDF/ISO Analytical Week, Montreal
- Draft presented to the National Committees

H2 2008

H1 2009

H2 2009

H1 2010

H2 2010

« Melamie outbreak 2008

Q4 2008

- IDF proposed several methods to WHO

May 2009

IDF/ISO Analytical Week, Sochi, Russia

- IDF “Action Team” was created to write a guideline
- Co-managed by T. Delatour (CH) and S. Holroyd (NZ)
- Agreement:
 - IDF Review Method/ISO Technical Specifications
 - Guideline with methods in the annex

Jan 2010

- ISO/IDF members approved the DRAFT

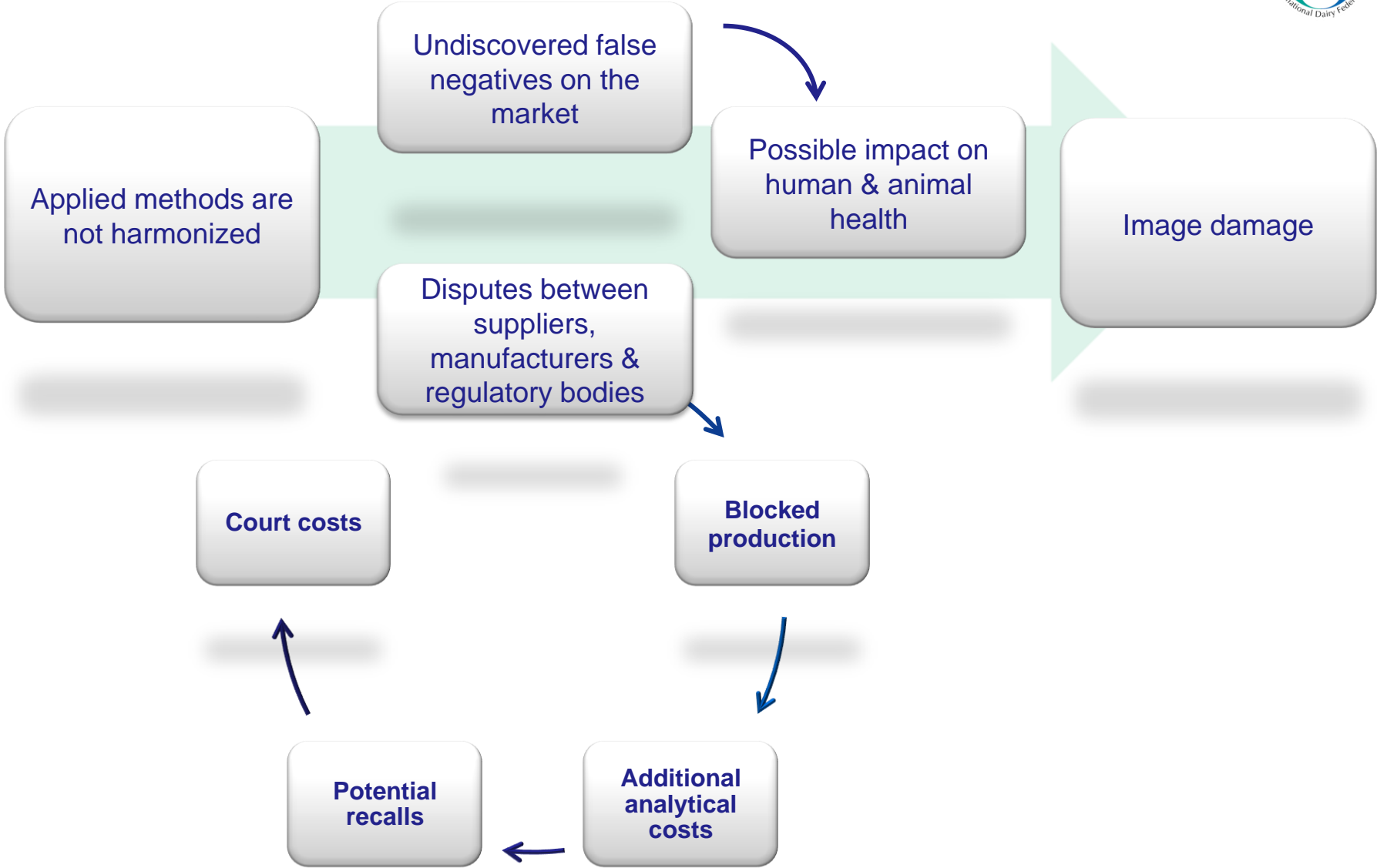
Oct 2010

- Proof joint ISO/IDF document ready for publication

Nov 2010

- Publication of ISO/IDF document

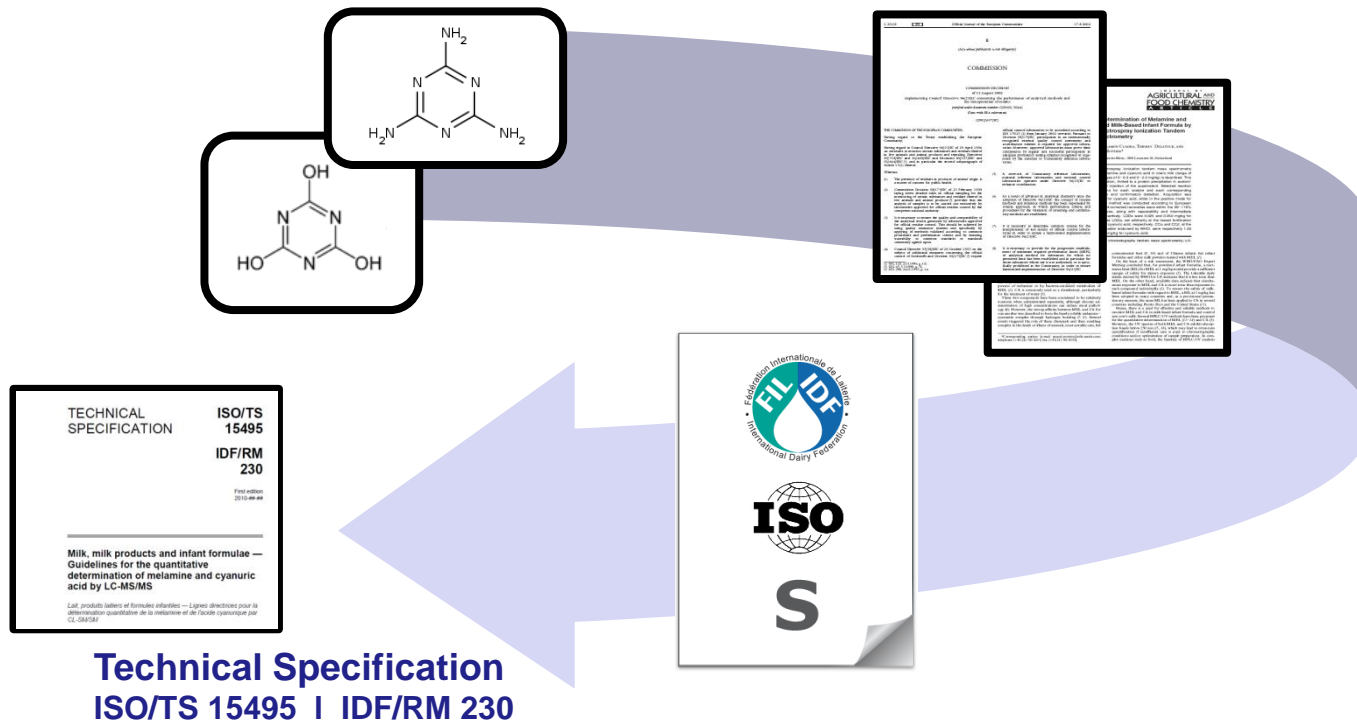
What would have happened if IDF would not have taken the initiative?



SWIFT Success



- IDF took the leadership in a very collaborative way
- Overall outcome is better and faster than if an individual organisation would have done it.



« **IDF Guide on Maintaining the Integrity of Suppliers' Milk - Assessment, prevention and monitoring** » to be published within few months

Contact



Dr. Petra Lutter
Nestec Ltd.
Nestlé Research Center
Lausanne, Switzerland

petrasilke.lutter@rdls.nestle.com





Thank you