Chemical Contaminants & Residues in Food Community

Subgroup – 'Environmental & Emerging Contaminants'

Tuesday, August 28th, 2018

1:30 p.m. to 3:00 p.m.

AOAC INTERNATIONAL

132nd AOAC Annual Meeting & Exposition
Toronto, Canada
August 26 – 29, 2018

Chemical Contaminants & Residues in Food Community
Subgroup – 'Environmental & Emerging Contaminants'

Agenda

- **1:30 p.m. to 1:40 p.m.**
  Introduction
- **1:40 p.m. to 2:00 p.m.**
  - **Update on MCPD and glycidyl esters – by *K. Mastovska***
  - **Bisphenol A: First Action method 2017.15 – by *K. Mastovska***
- **2:00 p.m. to 2:30 p.m.**
  - **Determination of folpet in foodstuffs – by *Th. Delatour***
  - **Artefact formation of formaldehyde in milk powders – by *Th. Delatour***
- **2:30 p.m. to 2:50 p.m.**
  New topics: furan, LC-MS multi-mycotoxins method – by *Th. Delatour*
- **2:50 p.m. to 3:00 p.m.**
  Wrap-up and closure

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- **Update on MCPD and glycidyl esters**
- **Bisphenol A: First Action method 2017.15**

– by *Katerina Mastovska* –

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- **Determination of folpet in foodstuffs**

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6.2.2016 EN Official Journal of the European Union L 31/1

II

(Non-legislative acts)

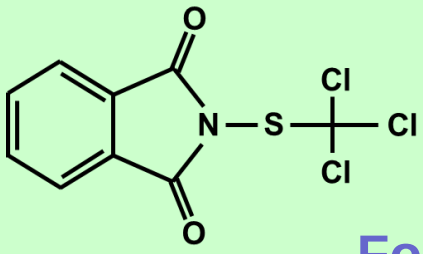
REGULATIONS

COMMISSION REGULATION (EU) 2016/156

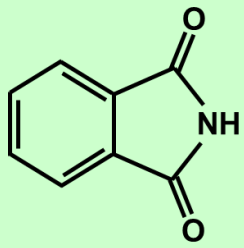
of 18 January 2016

amending Annexes II and III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for boscalid, clothianidin, thiamethoxam, folpet and tolclofos-methyl in or on certain products

(Text with EEA relevance)



Folpet



Phthalimide

Chemical Contaminants & Residues in Food Community

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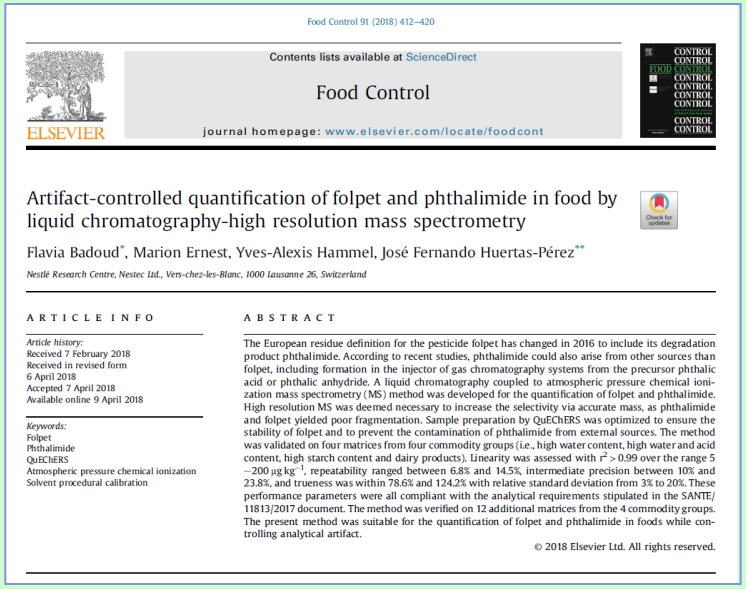

Food Control 91 (2018) 412–420

Contents lists available at ScienceDirect

Food Control

journal homepage: www.elsevier.com/locate/foodcont

Artifact-controlled quantification of folpet and phthalimide in food by liquid chromatography-high resolution mass spectrometry

Flavia Badoud*, Marion Ernest, Yves-Alexis Hammel, José Fernando Huertas-Pérez**

Nestlé Research Centre, Nestec Ltd., Vers-chez-les-Blanc, 1000 Lausanne 26, Switzerland

ARTICLE INFO

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Available online 9 April 2018

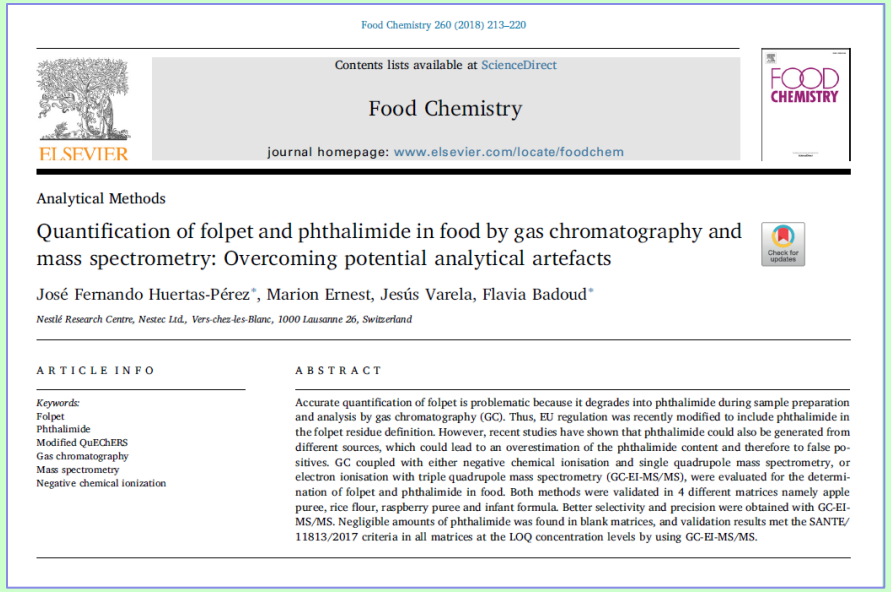
Keywords:
Folpet
Phthalimide
QuEChERS
Atmospheric pressure chemical ionization
Solvent procedural calibration

ABSTRACT

The European residue definition for the pesticide folpet has changed in 2016 to include its degradation product phthalimide. According to recent studies, phthalimide could also arise from other sources than folpet, including formation in the injector of gas chromatography systems from the precursor phthalic acid or phthalic anhydride. A liquid chromatography coupled to atmospheric pressure chemical ionization mass spectrometry (MS) method was developed for the quantification of folpet and phthalimide. High resolution MS was deemed necessary to increase the selectivity via accurate mass, as phthalimide and folpet yielded poor fragmentation. Sample preparation by QuEChERS was optimized to ensure the stability of folpet and to prevent the contamination of phthalimide from external sources. The method was validated on four matrices from four commodity groups (i.e., high water content, high water and acid content, high starch content and dairy products). Linearity was assessed with $r^2 > 0.99$ over the range 5 −200 μg kg^{-1}, repeatability ranged between 6.8% and 14.5%, intermediate precision between 10% and 23.8%, and trueness was within 78.6% and 124.2% with relative standard deviation from 3% to 20%. These performance parameters were all compliant with the analytical requirements stipulated in the SANTE/11813/2017 document. The method was verified on 12 additional matrices from the 4 commodity groups. The present method was suitable for the quantification of folpet and phthalimide in foods while controlling analytical artifact.

Badoud *et al.* **2018**

Food Control, *91*, 412-420



Food Chemistry 260 (2018) 213–220

Contents lists available at ScienceDirect

Food Chemistry

journal homepage: www.elsevier.com/locate/foodchem

Analytical Methods

Quantification of folpet and phthalimide in food by gas chromatography and mass spectrometry: Overcoming potential analytical artefacts

José Fernando Huertas-Pérez*, Marion Ernest, Jesús Varela, Flavia Badoud*

Nestlé Research Centre, Nestec Ltd., Vers-chez-les-Blanc, 1000 Lausanne 26, Switzerland

ARTICLE INFO

Keywords:
Folpet
Phthalimide
Modified QuEChERS
Gas chromatography
Mass spectrometry
Negative chemical ionization

ABSTRACT

Accurate quantification of folpet is problematic because it degrades into phthalimide during sample preparation and analysis by gas chromatography (GC). Thus, EU regulation was recently modified to include phthalimide in the folpet residue definition. However, recent studies have shown that phthalimide could also be generated from different sources, which could lead to an overestimation of the phthalimide content and therefore to false positives. GC coupled with either negative chemical ionisation and single quadrupole mass spectrometry, or electron ionisation with triple quadrupole mass spectrometry (GC-EI-MS/MS), were evaluated for the determination of folpet and phthalimide in food. Both methods were validated in 4 different matrices namely apple puree, rice flour, raspberry puree and infant formula. Better selectivity and precision were obtained with GC-EI-MS/MS. Negligible amounts of phthalimide was found in blank matrices, and validation results met the SANTE/11813/2017 criteria in all matrices at the LOQ concentration levels by using GC-EI-MS/MS.

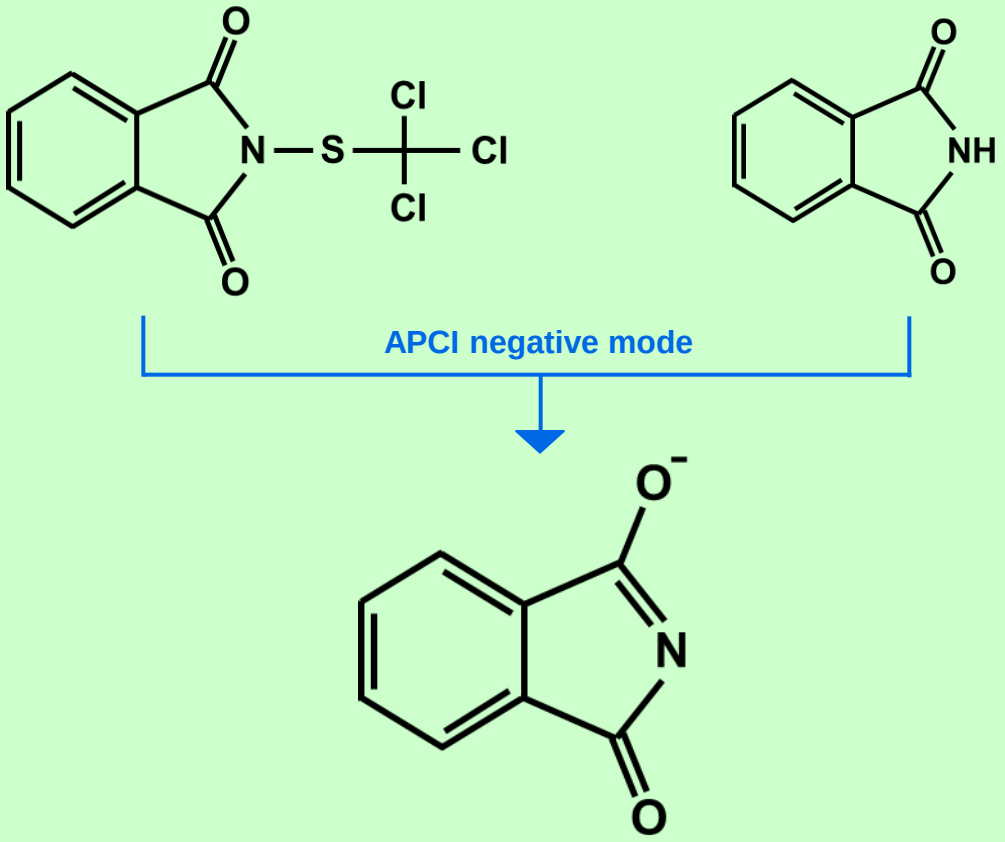
Huertas-Pérez *et al.* **2018**

Food Chemistry, *260*, 213-220

Investigated matrices

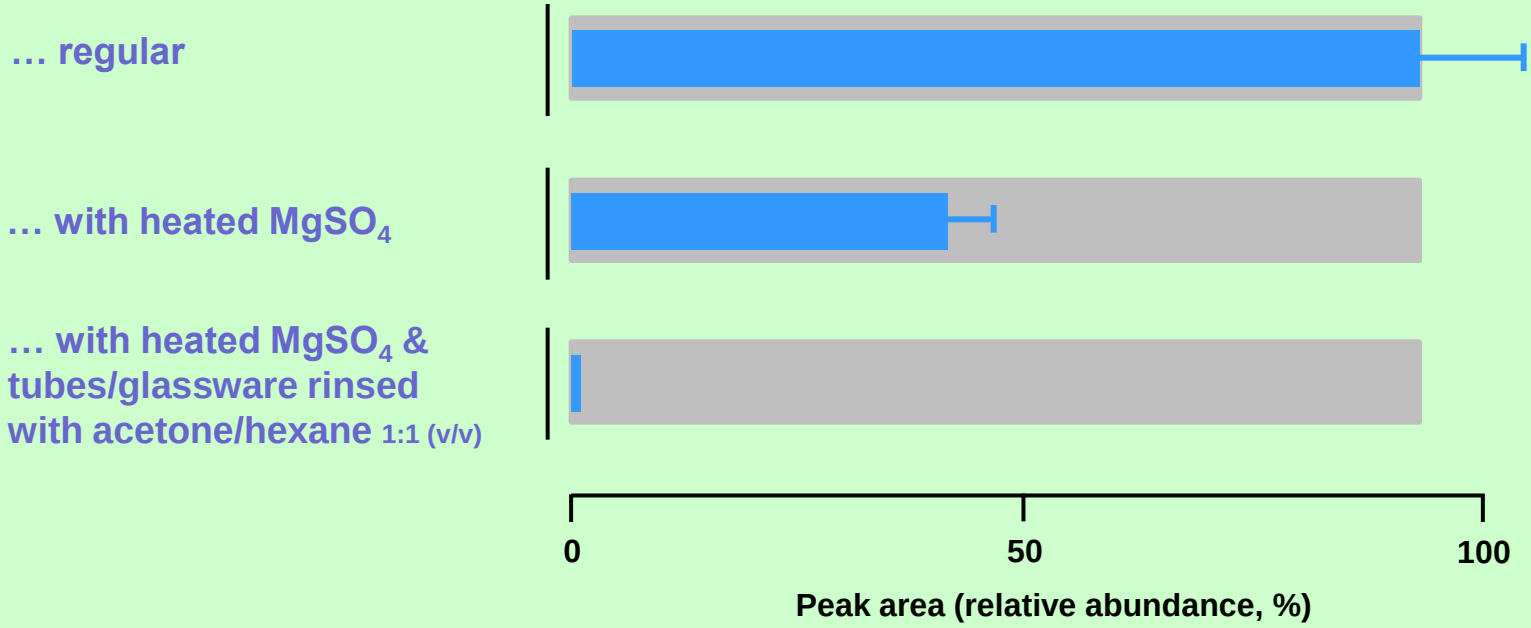
- **High water content**
  Tomato paste, Red bell peppper, Carrot, Pineapple puree, Banana puree, Onion & Sweet potato
- **High acid and water content**
  Blueberry & Strawberry
- **High starch and/or protein content**
  Wheat semolina
- **Dairy products**
  Hydrolyzed infant formula

LC-APCI-HRMS



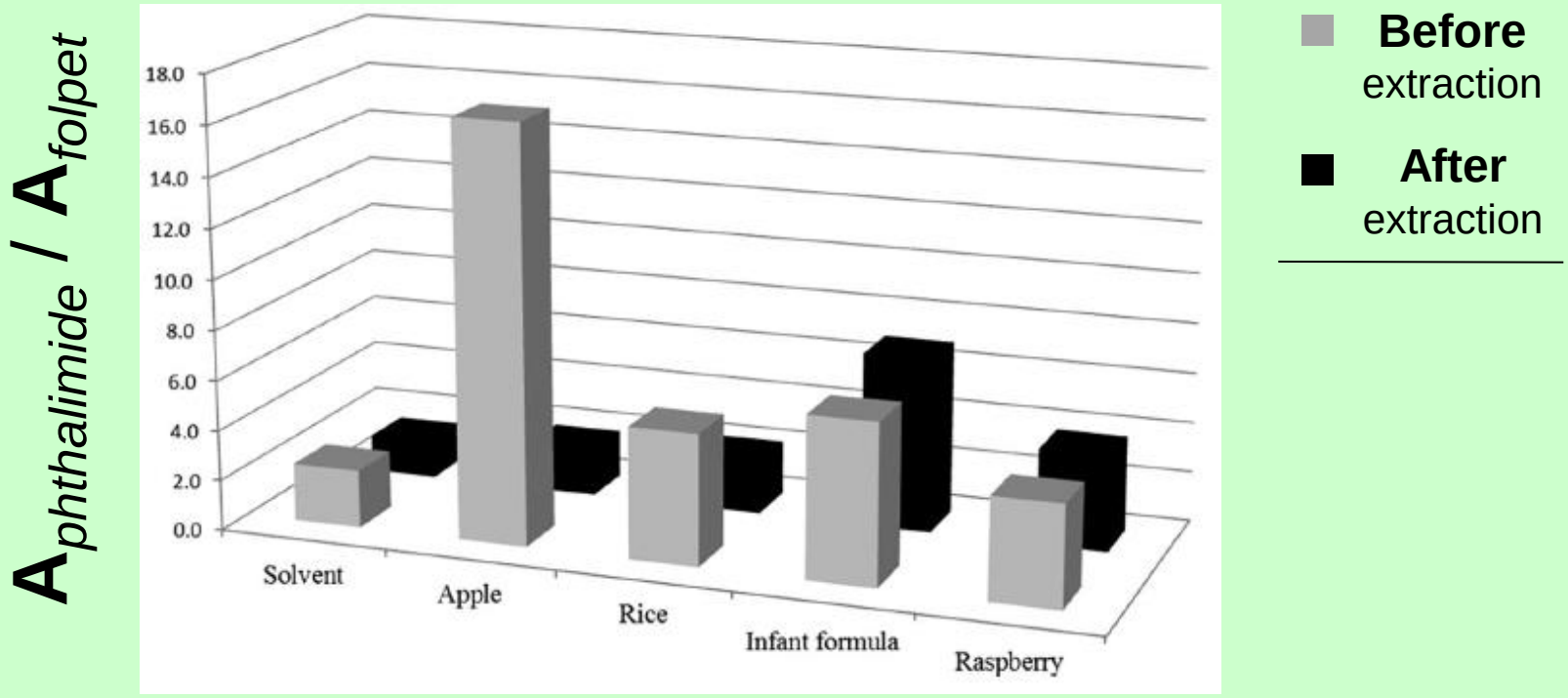
Monoisotopic mass at *m/z* 146.0247

Acidified QuEChERS …



Phthalimide formation in the blank reagent (n = 3)

GC-EI-MS/MS



Formation of phthalimide in various matrices

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- **Artefact formation of formaldehyde in milk powders**

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Food Control 93 (2018) 23–31

Contents lists available at ScienceDirect

Food Control

journal homepage: www.elsevier.com/locate/foodcont

Artefact formation of formaldehyde in milk powders: Impact of analytical conditions

Thomas Bessaire, Marie-Claude Savoy, Adrienne Tarres, Claudia Mujahid, Till Goldmann, Irène Perrin, Pascal Mottier*

Nestlé Research Center, Route du Jorat 57, Vers-chez-les-Blanc, 1000 Lausanne 26, Switzerland

ARTICLE INFO

Keywords:
Formaldehyde
Milk powder
Derivatization
2,4-Dinitrophenylhydrazine (DNPH)
LC-MS/MS

ABSTRACT

Formaldehyde (FA) was analyzed in milk powders by isotope dilution LC-MS/MS using 2,4-dinitrophenylhydrazine (DNPH) as the derivatization agent. Analytical conditions (temperature and time) applied for derivatization were shown to strongly impact the levels of FA detected in micronutrient-fortified milk powders. In particular, vitamin C and ferrous iron catalyzed FA formation when derivatization was conducted at 60 °C, as described by several authors. Artefact formation of FA was demonstrated in a model study using $^{2}H_{2}$-glycine and a freeze-dried raw cow's milk with added micronutrients. A limited survey of commercial fortified milk powders showed that FA levels were about 4-fold lower when analyses were conducted at room temperature as compared to 60 °C (0.65 ± 0.25 mg/kg and 2.57 ± 0.51 mg/kg, respectively). Based on data obtained at room temperature, FA exposure from fortified milk powders does not represent a safety concern even under worst-case assumptions. This study highlights the need and importance of adopting an international standard for FA analysis in foods.

Bessaire *et al.* **2018,** Food Control, *93*, 23-31

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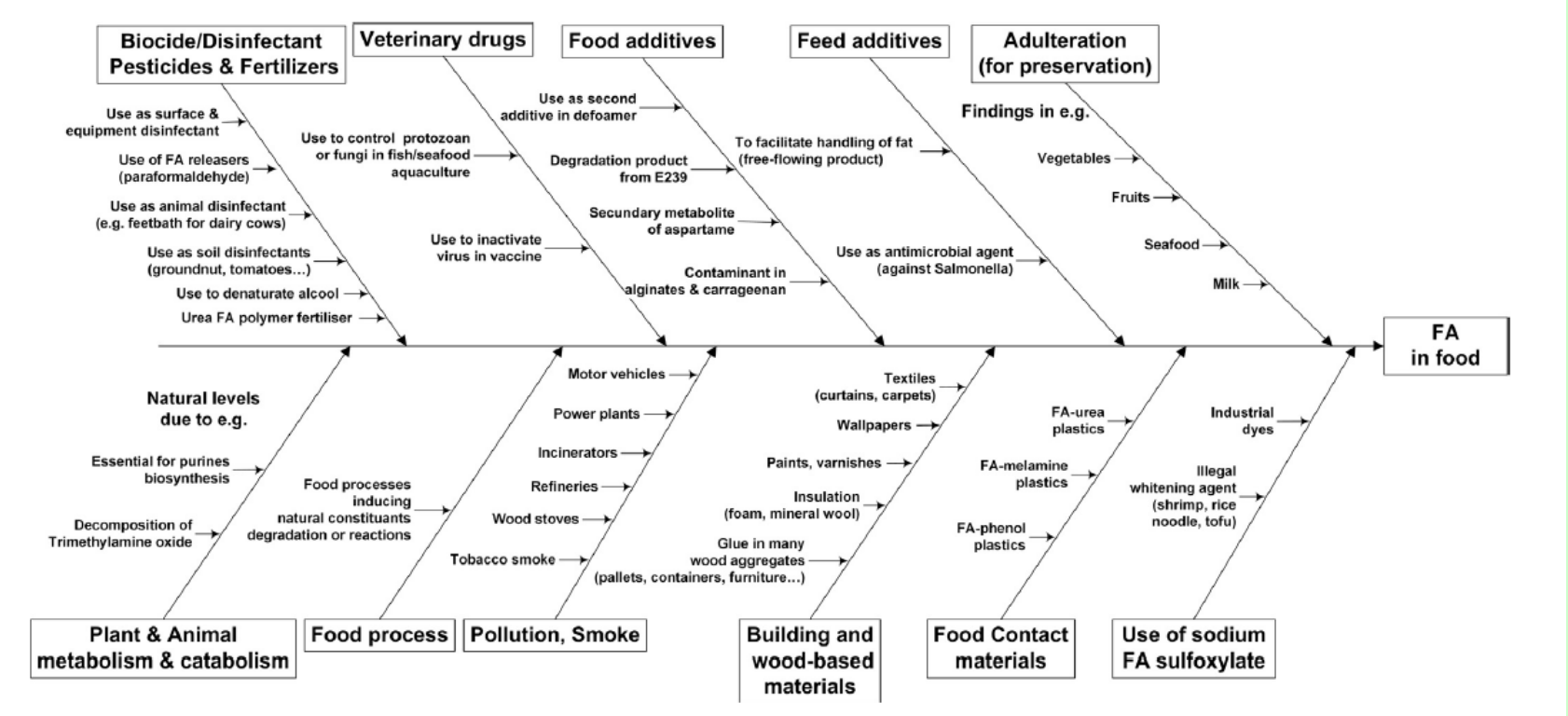
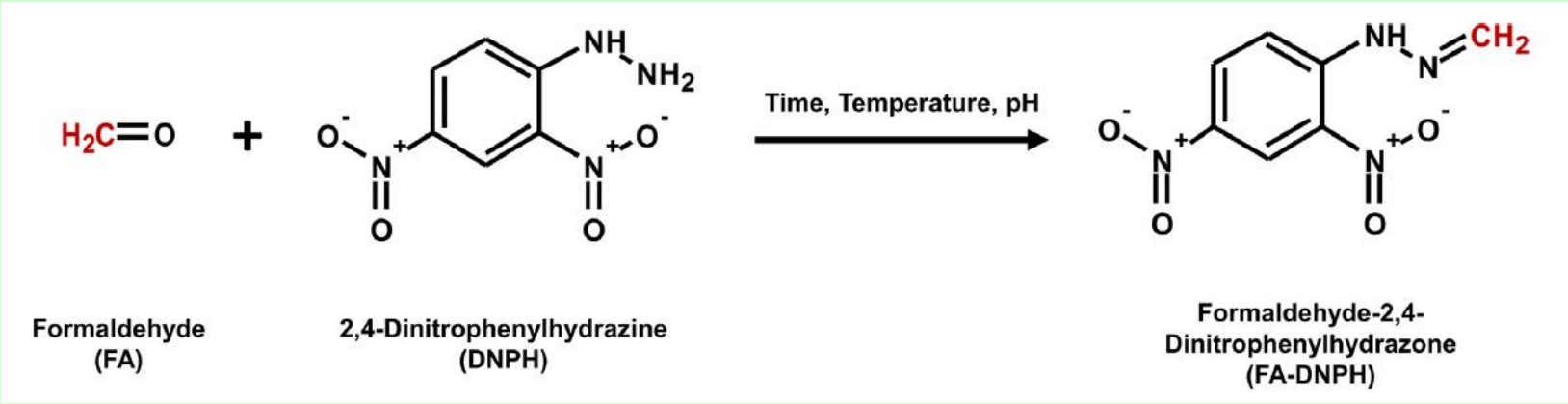
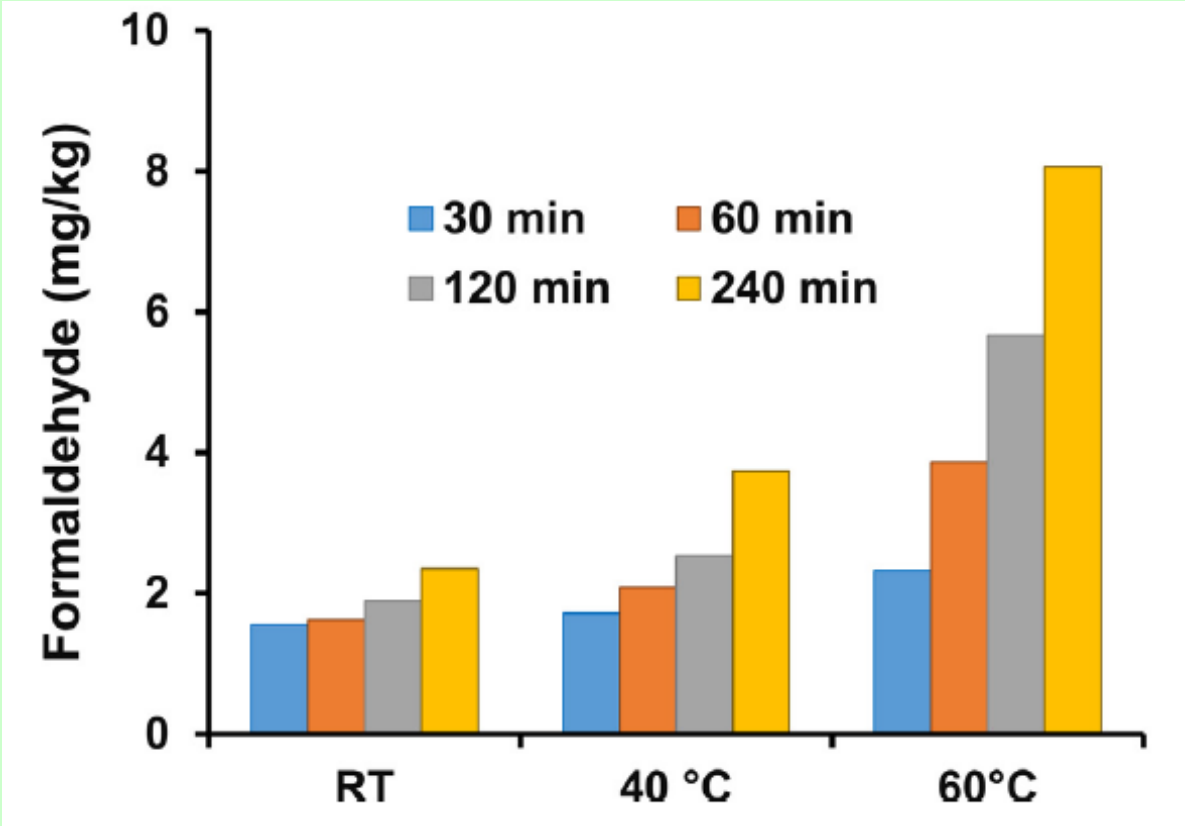


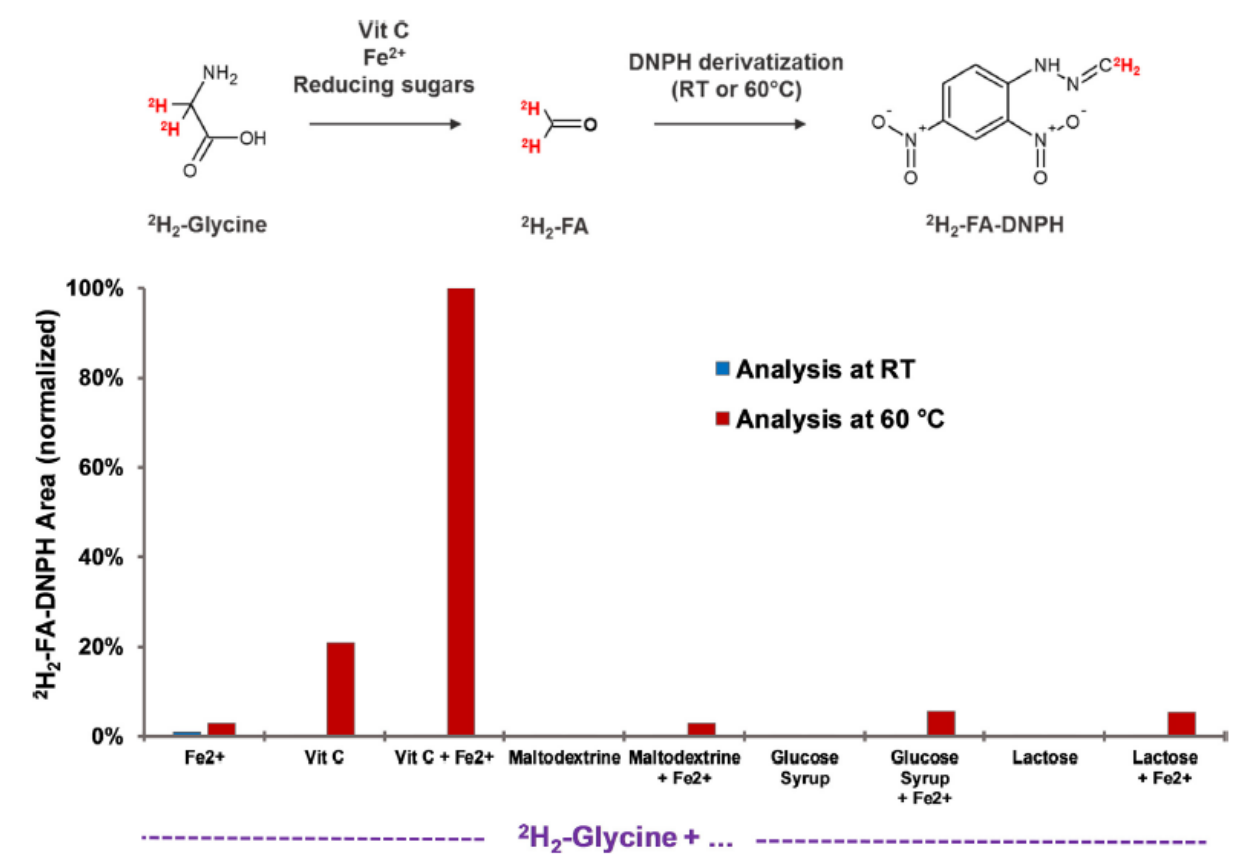
Sources of formaldehyde in food



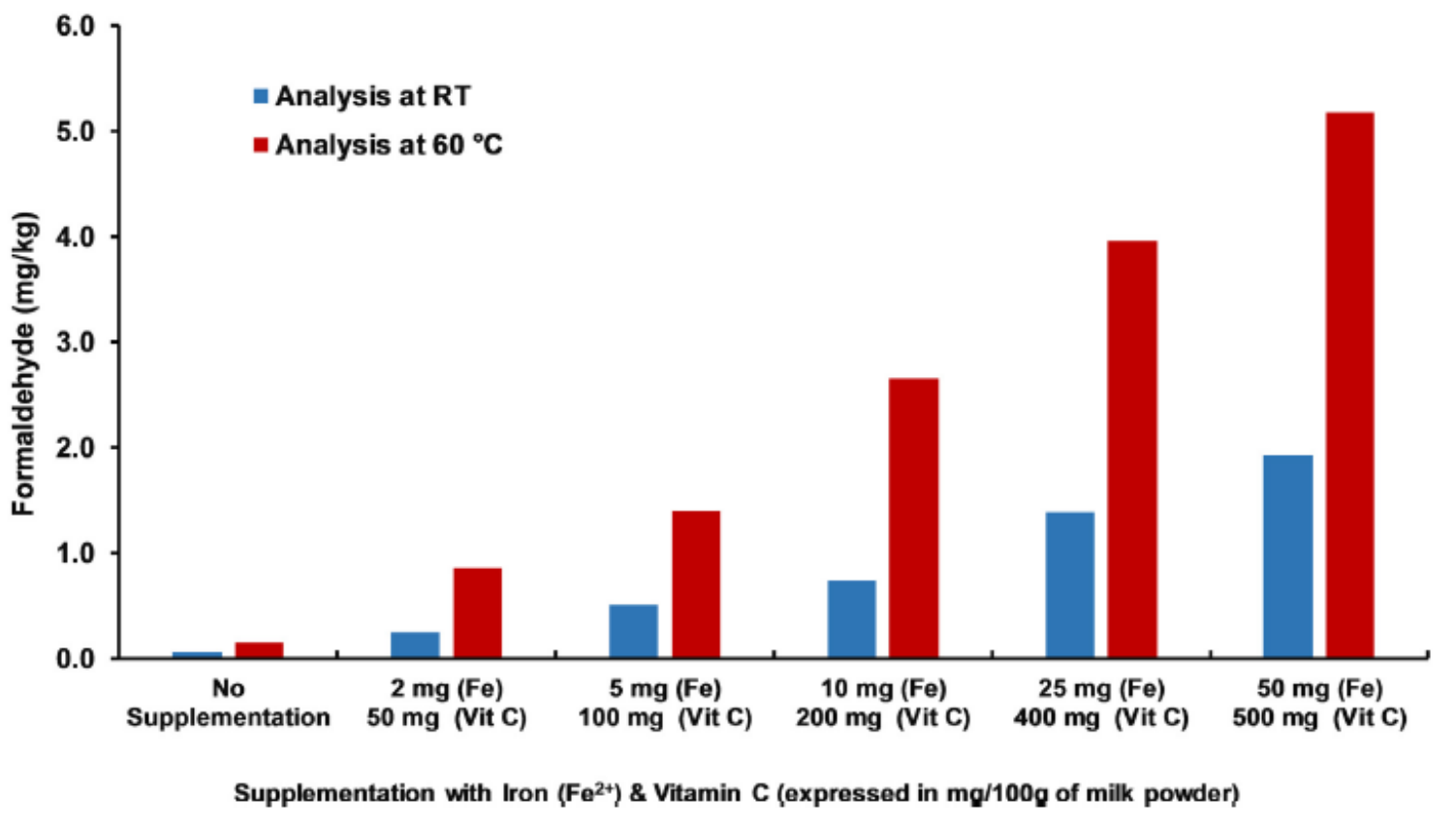
2,4-Dinitrophenylhydrazine derivatization prior to LC-MS/MS analysis



Impact of derivatization time and temperature in micronutrient-fortified milk powders



Glycine-derived formation of formaldehyde in presence of iron and vitamin C



Levels of formaldehyde in iron- (Fe^{2+}) and vitamin C-supplemented milk powder

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- **Status initiative at AOAC to establish a standard on furan and alkylfuran**

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July 31, 2018

AOAC met with CHCC (Chemistries of Heated Carbohydrates Consortium), comprised of trade association members including:

- Grocery Manufacturers Association
- American Beverage Association
- American Bakers Association
- American Institute of Bakers International
- National Confectioners Association
- Juice Products Association

Investigation of heat-formed carbohydrates with particular emphasis on:

- Furan
- 5-Hydroxymethylfurfural
- Furfural
- Furfuryl alcohol
- Acrylamide

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- **LC-MS multi-mycotoxins method**

CEN Working Group 5 – Biotoxins

Analyte scope: 12 Regulated mycotoxins.

Material to be provided: Analytical standards, Immunoaffinity columns, Samples including reference materials.

Matrix scope: Raw cereals (wheat, corn, rye …), Nuts (peanuts, almonds, hazelnuts …), Spices (chili, paprika, pepper …), Infant cereals (finished products), Milk-based products, and Dried fruits/Coffee/Cocoa if possible.

Number of participants: Up to 20 laboratories.

Number of samples: Up to 12 samples dispatched as blind duplicates.

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New Topics to be Adressed?

Thierry DELATOUR

Group *Untargeted Screening*
Department *Analytical Science*
Nestlé Research Centre
Vers-chez-les-Blanc
1000 Lausanne 26, Switzerland

Phone: +41.21.785.9220
E-mail: thierry.delatour@rdls.nestle.com