Approaches for the Mitigation of 3-MCPD Esters and Glycidyl Esters in Baby Food

Christoph Schatzmann, Walburga Seefelder, Massimo Casella, Constantin Bertoli, Dhanavel Gokulrajan, Mathieu Dubois, Brian Craft, Kornèl Nagy, Gabriele Scholz, and Frédéric Destaillats

Symposium on MCPD Esters and Glycidyl Esters Analytics, Toxicology, Risk Assessment, Mitigation. Where we are today?

Berlin June 2017

3-MCPD-esters – Safety and Regulatory Environment

Health Based Guidance Values

	EU/EFSA	WHO/JECFA
2001 « OLD » EFSA/JECFA TDI **2** µg/kg BW/day	March 2016 NEW EFSA TDI **0.8** µg/kg BW/day **REASSESSMENT** **On-going (end 2018)**	Nov 2016 NEW JECFA TDI **4** µg/kg BW/day

Regulation

EU regulation Waiting for EFSA assessment	Code of Practice for ME/GE reduction (chaired by US, EU,..) 2020

Glycidyl-esters – Safety and Regulatory Environment

Health Based Guidance Values

No safe intake level

EU/EFSA

WHO/JECFA

Regulation

EU regulation
Most likely Jan 2018
75 ug/kg for Infant Formula (powders) / 10 ug/kg (liquids

Code of Practice for ME/GE reduction (chaired by US, EU,..) 2020

ALARA approach

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Occurrence of MCPDEs and GEs in selected vegetable oils.

Refined oils	*n*	Average 3-MCPD (mg/kg)	3-MCPD range (mg/kg)	Average Glycidol (mg/kg)	Reference
Canola	4	0.21	0.19–0.24	0.31	[45]
Canola	7	0.11	<LOQ-0.33	0.28	[25]
Coconut	4	0.4	0.3–0.4	0.4	[46]
Coconut	7	0.17	0.025–0.38	0.8	[25]
Corn	15	2.8	max 7.0[a]	nd	[47]
Corn	9	0.42	0.06–0.42	0.68	[25]
Cottonseed	2	0.43	0.14–0.72	0.5	[25]
Grape seed	4	2.6	1.38–3.19	1.93	[45]
Grape seed	3	1.74	0.24–3.91	1.14	[25]
Olive extra virgin	6	<0.1	na	nd	[45]
Olive light	4	0.84	0.58–1.56	0.52	[45]
Palm	14	3.19	1.51–7.23	3.5	[25]
Palm	5	5.5	4.7–6.0	4.8	[46]
Palm	37	4.5	max. 13[a]	nd	[47]
Palm kernel	6	1.3	1–1.4	0.4	[46]
Rapeseed	31	0.3	max 1.5[a]	nd	[47]
Rapeseed	10	1.5	0.1–4.1	0.1	[46]
Rice bran	2	4.17	0.37–8.34	2.52	[45]
Safflower	5	0.84	0.28–1.77	0.25	[25]
Sunflower	5	1.9	0.1–3.5	0.1	[46]
Sunflower	4	0.18	<0.1–0.25	0.31	[45]
Sunflower	4	0.55	0.19–0.93	nd	[25]
Sunflower	49	1	max 5.7[a]	nd	[46]
Walnut	2	7.24	2.87–11.6	0.57	[45]
Walnut	1	0.63	na	0.59	[25]

na = not applicable.
nd = not determined.
[a] Only maximum value reported.

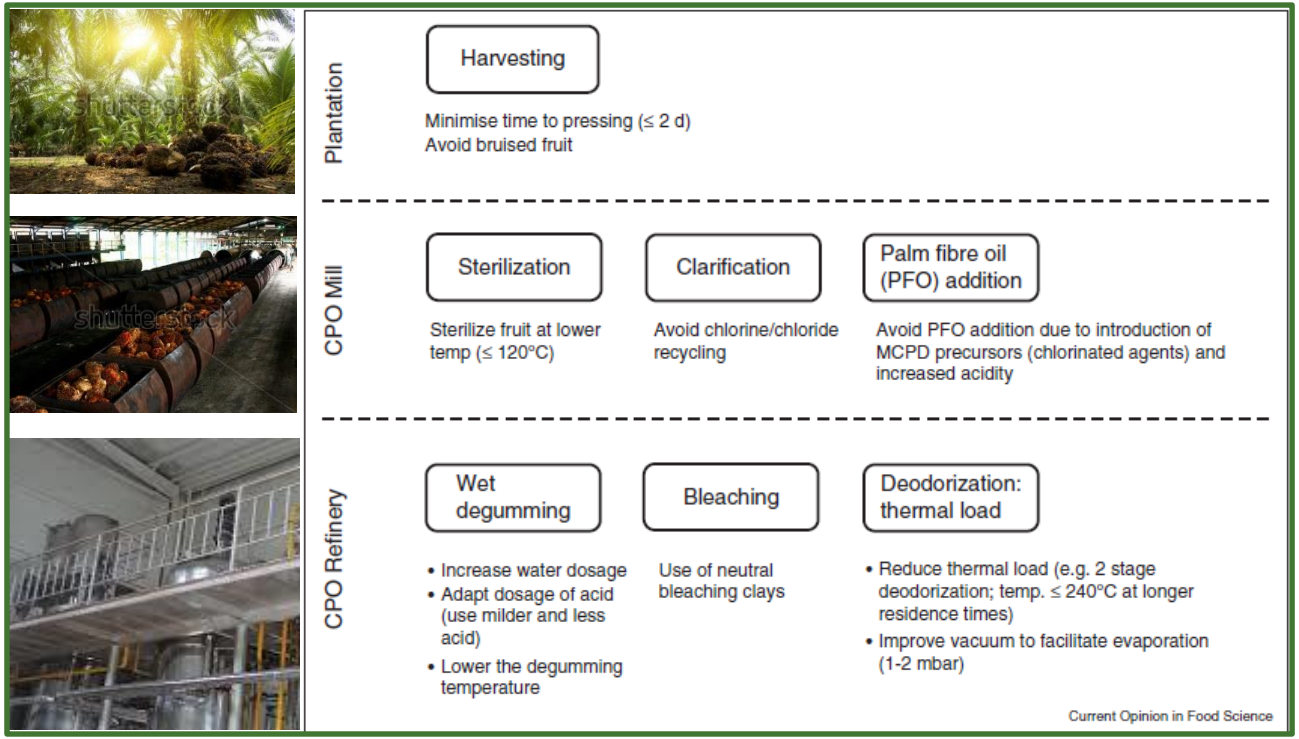
Occurrence in oils

- It is evident that palm oil and its fractions can be considered as most susceptible to the formation of both MCPDEs and GEs.
- Not only Palm oil but all refined commodity oils would require mitigation and a better science based understanding of root cause and mitigation options
- All studies published so far, for example those from the USA, Brazil and China, show comparable levels.

Monochloropropane-1,2-diol esters (MCPDEs) and glycidyl esters (GEs): an update Richard H Stadler (Nestlé, Quality Management) in *Current Opinion in Food Science* 2015, 6:12–18

Toolbox for the mitigation of MCPD- & Glycidyl Esters (BLL)

Bund für Lebensmittelrecht und Lebensmittelkunde e. V. (BLL). German Federation for Food Law and Food Science.



- **Tailor-made solutions need to be found; there is no one size fits all approach**

Nestlé Research actively contributes to better understand the fundamental mechanisms involved in the formation of MCPD- and Glycidyl-esters

Food Additives & Contaminants: Part A
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/tfac20

Formation mechanisms of Monochloropropanediol (MCPD) fatty acid diesters in refined palm (*Elaeis guineensis*) oil and related fractions

Frédéric Destaillats [a] , Brian D. Craft [a] , Laurence Sandoz [a] & Kornél Nagy [a]
[a] Nestlé Research Center, Food Science & Technology Department, Vers-chez-les-Blanc, P.O. Box 44, 1000 Lausanne 26, Switzerland

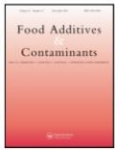
Food Additives & Contaminants: Part A: Chemistry, Analysis, Control, Exposure & Risk Assessment
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/tfac20

Factors impacting the formation of Monochloropropanediol (MCPD) fatty acid diesters during palm (Elaeis guineensis) oil production

Brian D. Craft [a] , Kornél Nagy [a] , Laurence Sandoz [a] & Frédéric Destaillats [a]
[a] Nestlé Research Center, Food Science and Technology Department, Vers-chez-les-Blanc, Lausanne, Switzerland

Glycidyl esters in refined palm (*Elaeis guineensis*) oil and related fractions. Part II: Practical recommendations for effective mitigation

Brian D. Craft, Kornél Nagy, Walburga Seefelder, Mathieu Dubois, Frédéric Destaillats *
Nestlé Research Center, Vers-chez-les-Blanc, Lausanne, Switzerland



Food Additives & Contaminants: Part A
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/tfac20

Fatty acid esters of monochloropropanediol (MCPD) and glycidol in refined edible oils

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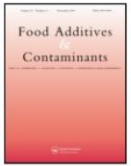
[a] Nestlé Research Center - Food Science & Technology, Vers-chez-les-Blanc PO Box 44, Lausanne 26 CH-1000, Switzerland
[b] ILSI Europe a.i.s.b.l., Av. E. Mounier 83, Brussels B-1200, Belgium

Glycidyl esters in refined palm (*Elaeis guineensis*) oil and related fractions. Part I: Formation mechanism

Frédéric Destaillats *, Brian D. Craft, Mathieu Dubois, Kornél Nagy
Nestlé Research Center, Vers-chez-les-Blanc, Lausanne, Switzerland



Food Additives & Contaminants: Part A
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/tfac20

Mass-defect filtering of isotope signatures to reveal the source of chlorinated palm oil contaminants

K. Nagy [a] , L. Sandoz [a] , B.D. Craft [a] & F. Destaillats [a]
[a] Food Science and Technology Department, Nestlé Research Center, Nestec Ltd, Vers-Chez-les-Blanc, CH-1000 Lausanne 26, Switzerland

Available online: 31 Aug 2011

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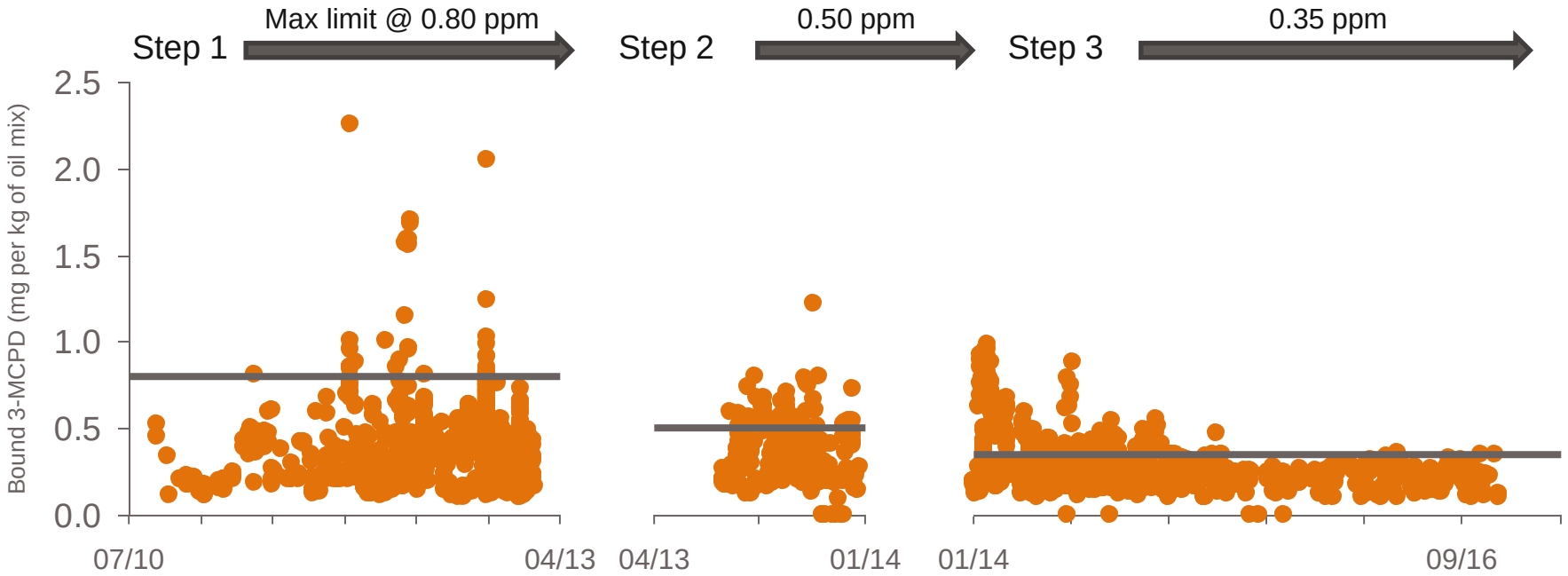
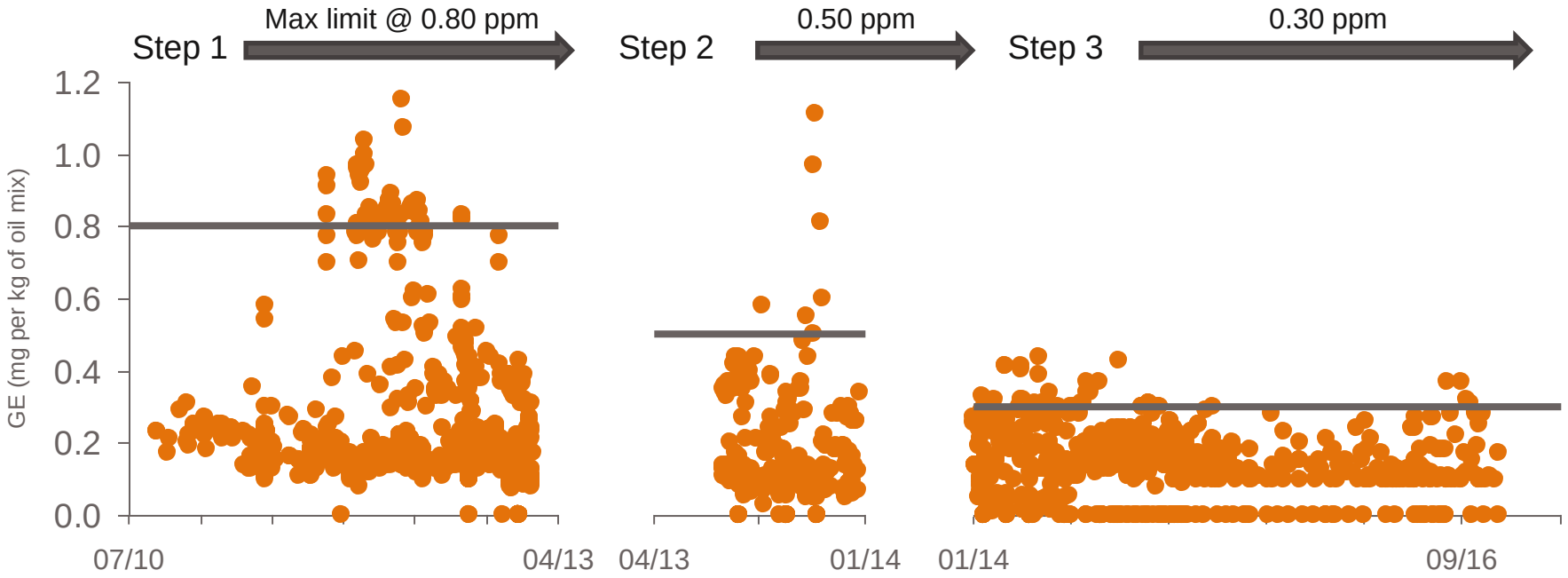
Impact on continuous improvement done with suppliers on the bound 3-MCPD Ester in Oil mix used in Infant Formula



Monitoring done on same material over 6 years (1800 samples analyzed)

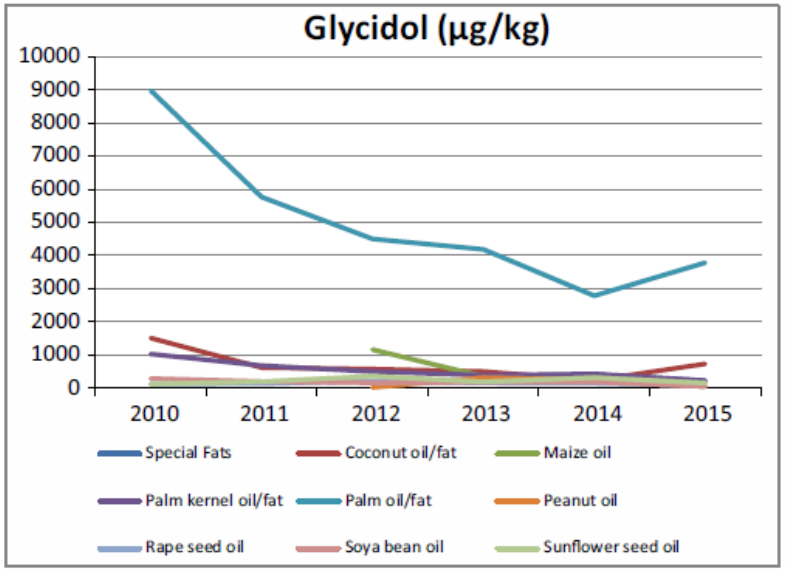
Nestlé Research

Impact on continuous improvements done with suppliers on the bound Glycidyl Ester in Oil mix used in Infant Formula



Monitoring done on same material over 6 years (1800 samples analyzed)

Mitigation efforts lead to a reduction of > 50% for GEs in Fats and Oils (EFSA report March 2016):

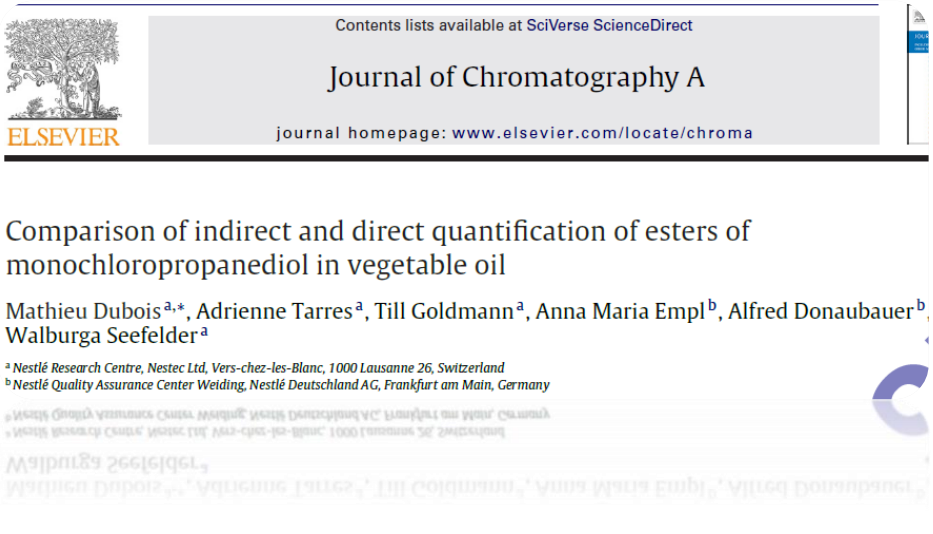


Graph showing the evolution across the years 2010-2015 of the average level of Glycidol from esters in different types of oils and fats (EFSA report March 2016).

Nestlé has a history of a dedicated Analytical Research Program for MCPD- and Glycidyl-esters

ANALYTICS

Comparison of direct & Indirect methods



Contents lists available at SciVerse ScienceDirect

Journal of Chromatography A

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

Comparison of indirect and direct quantification of esters of monochloropropanediol in vegetable oil

Mathieu Dubois[a,*], Adrienne Tarres[a], Till Goldmann[a], Anna Maria Empl[b], Alfred Donaubauer[b], Walburga Seefelder[a]

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Article

Determination of Seven Glycidyl Esters in Edible Oils by Gel Permeation Chromatography Extraction and Liquid Chromatography Coupled to Mass Spectrometry Detection

Mathieu Dubois*†, Adrienne Tarres†, Till Goldmann†, Guenter Loeffelmann‡, Alfred Donaubauer‡, and Walburga Seefelder†

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Nestlé Quality Assurance Center Weiding, Nestlé Deutschland AG, Frankfurt am Main, Germany

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Analytical landscape is complex, no standardized approach exists yet

Indirect determination
(ester cleavage releases the 3 core analytes, GC-MS)

alkaline

Early **DGF C-III 18 (09)**
Σ 3-MCPD + glycidol
DGF C-VI 17 (10); fast

Late **DGF C-III 18 (09) A,B**
A: Σ 3-M + g, B: 3-MCPD
Withdrawn by DGF

BfR method 09
3-MCPD
fast

DGF C-VI 18 (10) A, B
A: Σ 3-M + g, B: 3-MCPD
AOCS Cd 29c-13; fast

Küsters et al. 2010
3-MCPD, Glycidol
fast

SGS **"3-in-1"** Kuhlmann 2011
3-MCPD, 2-MCPD, Glycidol
AOCS Cd 29b-13; slow

acidic

Divinova et al. 2004
Zelinkova et al. 2006
3-MCPD; slow

BfR method 08
3-MCPD
slow

"Unilever"
Ermacora et al. 2013
3-MCPD, 2-MCPD, Glycidol
AOCS Cd 29a-13; slow

enzymatic

Myasaki et al. 2012
3-MCPD, 2-MCPD, Glycidol fast

Koyama et al. 2015
3-MCPD, Glycidol
fast

Direct determination
(determination of a selected number of contaminant esters)

Dilute & shoot

Blumhorst et al. 2011
GE
LC-MS²

Haines et al. 2011
3-MCPD-E, GE
LC-MS²

SPE or SPE²

Masukawa et al. 2010/11
GE
SPE²; LC-MS:
AOCS Cd 28-10

Granvogl et al. 2011
GE
SPE; LC-MS²

Dubois et al. 2011
3-MCPD-E, 2-MCPD-E, GE
SPE²; LC-MS²

Steenbergen et al. 2013
GE
l/l; LC; GC/MS

MacMahon et al. 2013
3-MCPD-E, 2-MCPD-E, GE
2 x SPE²; 2 x LC-MS²

Presentation from J. Kuhlmann at AOAC Midyear Meeting March 2017

Preliminary study is indicating inconsistences between methods applied to Infant Formula (internal trials)

Product	Content In Finished Goods					
	Bound 3MCPD µg/kg			Bound Glycidol µg/kg		
	AOCS Cd 29b-13	AOCS Cd 29c-13	AOCS Cd 29a-13	AOCS Cd 29b-13	AOCS Cd 29c-13	AOCS Cd 29a-13
A	30	32	32	27	37	28
B	29	32	45	33	39	32
C	59	59	67	42	59	38
D	104	104	120	35	62	38

AOCS Cd 29b-13
AOCS Cd 29c-13
AOCS Cd 29a-13

Different methods used in differents labs

Results not fully consistent and requiering further investigation and collaborative studies

MCPD/GE Infant Formula Method Validation (AOAC/SPIFAN)

RECOMMENDATIONS

COMMISSION RECOMMENDATION
of 10 September 2014
on the monitoring of the presence of 2 and 3-monochloropropane-1,2-diol (2 and 3-MCPD), 2- and 3-MCPD fatty acid esters and glycidyl fatty acid esters in food

(Text with EEA relevance)

(2014/661/EU)

HAS ADOPTED THIS RECOMMENDATION:

1. Member States should, with the active involvement of feed and food business operators, perform monitoring for the presence of 2 and 3-MCPD, 2 and 3-MCPD fatty acid esters and glycidyl fatty acid esters in food, and particularly in:

(a) vegetable oils and fats and derived products such as margarine and similar products,

(b) foods for particular nutritional uses as defined in Directive 2009/39/EC of the European Parliament and of the Council (1) and intended for infants and young children, including infant- and follow on formulae as defined in Commission Directive 2006/141/EC (2) and dietary foods for special medical purposes as defined in Commission Directive 1999/21/EC (3) intended for use by infants,

(c) fine bakery wares, bread and rolls,

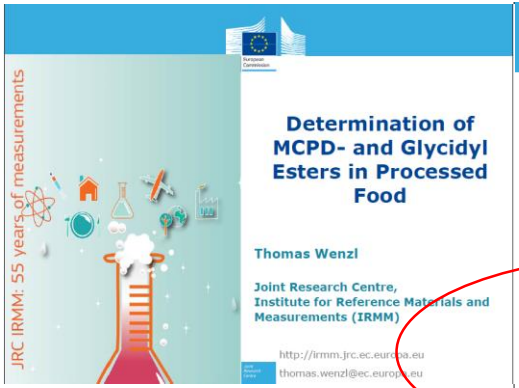
(d) canned meat (smoked) and canned fish (smoked),

(e) potato- or cereal-based snacks, other fried potato-based products,

(f) vegetable oil containing foods and foods prepared/produced with vegetable oils.

It is recognised that the analysis of 2 and 3-MCPD, 2 and 3-MCPD fatty acid esters and glycidyl fatty acid esters in foods mentioned in points (b) to (f) is very challenging and no methods of analysis, which have been validated by a collaborative study, are yet available. Therefore particular attention has to be paid when analysing foods mentioned in points (b) to (f) in order to ensure that the generated data are reliable.

Therefore, Member States which intend to analyse the presence of 2 and 3-MCPD, 2 and 3-MCPD fatty acid esters and glycidyl fatty acid esters in foods mentioned in points (b) to (f) may request, if appropriate and needed, the technical assistance of the Commission's Joint Research Centre, Institute for Reference Materials and Measurements (IRMM), Unit Standards for Food Bioscience.



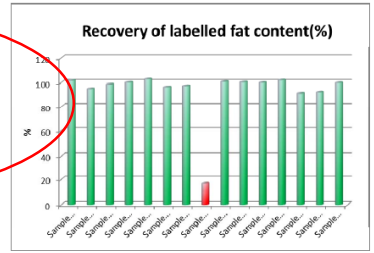
Summary fat extraction

General

- Proposed procedure applies mild conditions
- Works well for most food matrices
  - *Fat extraction yield: 97 ± 10 per cent of labelled fat content*

Limitations: Infant formula

- Proposed analysis method is not suitable for infant formula!
- Alternative extraction method (proposed by BfR)



30 October 2015

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AOAC
MIDYEAR MEETING
AOAC INTERNATIONAL 7th Annual Midyear Meeting, March 13-17, 2017

AOAC Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN)

Current state of MCPD and glycidol analysis in infant formulae and related foods

Estimated timing

March 2017 – Proposal due to AOAC-SPIFAN: Funding commitment from ISDI members

September 2017 – AOAC-SPIFAN will potentially designate AOAC Official Method First action

Nestlé Research

Key Messages

- Industry mitigation efforts have been acknowledged in the EFSA report to have so far achieved the 50% reduction of GE over the period 2010-2015 (mainly in Palm oil).
- Not only palm oil but all refined commodity vegetable oils would require mitigation and require a better science based understanding of root causes and mitigation options
- The Baby Food Association is actively engaged with its members to discuss the mitigation strategies to prevent the formation of 3-MCPDE and GE during the production and processing of fats and oils. This is done in collaboration with our suppliers, manufacturing fats and oils.
- Nestlé is actively engaged in the international discussions aiming at developing a harmonized method to analyze these compounds in infant and follow-on formulae. These developments will ensure accessibility to accurate test methods for the sector, satisfying the criteria established by the relevant Codex standard (CODEX STAN 193-1995).