

EC DG JRC activities

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**The European Commission's
science and knowledge service**
Joint Research Centre



Content

- FCM group
- NIAS
- Mineral oil
- Revision kitchenware guideline
- Ceramics, vitreous materials
- Recycling of plastic
- PT Temperature control during migration testing
- PT Oligomers

DISCLAIMER

➤ This work does not represent the position of the European Commission

FCM topics

Migration test methods

Ceramics, vitreous materials
Revision kitchenware guideline
Migration modelling

Recycling of plastics

Standardisation

ISO, CEN

Analytical methods

Collection
Calibrants
Multi-analyte ((N)IAS)
Performance guideline
Proficiency testing
Authorisation

FCM Laboratories

FCM Ispra
FCM org. Geel
FCM inorg Geel

Quality management

ISO 17025, 17043

DG SANTE

CoE
EFSA

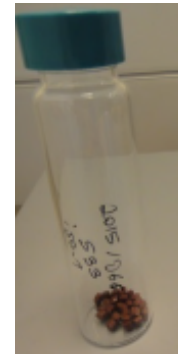
Mineral oil

NIAS – EuPC/JRC project

- **Identify IAS and NIASs migrating from common plastic FCM in the EU**
 - ✓ Use worst case migration test conditions
 - ✓ Develop in-house libraries both for GC-MS and LC-MS
 - ✓ Highlight problems during routine testing
- **Assign scores for NIAS to establish a potential hierarchy of concern**
- **Develop proposals to address any substances of potential concern**

Samples

- 70 representative FCM from EuPC
 - 63 different plastic FCM
 - ✓ food packing films
 - (non-)printed
 - ✓ trays
 - ✓ bottles and preforms
 - ✓ caps
 - ✓ cutlery
 - ✓ machine parts that contacts with food
 - 5 resins
 - PP, PS, PET, and other specific polymer)
 - 29 monolayers, 35 multi-layers (2-10)

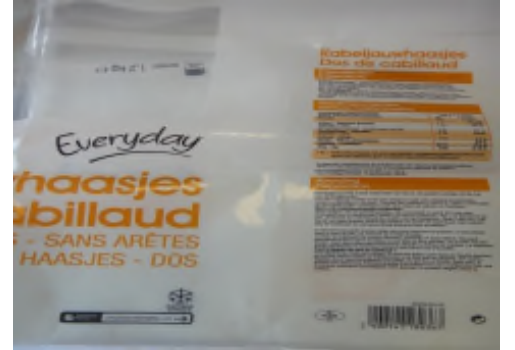


Selection migration test conditions

- Worst case foreseeable conditions specified by producer

Simplifications:

- Food simulant D2 replaced by isooctane
- In case of samples with more than two food simulants
 - ✓ maximum two food simulants for each sample
 - ✓ preference for D2
 - ✓ one aqueous food simulant with higher organic phase



food simulant A: 20 °C/10 d (5); 40 °C/10 d (7); 60 °C/10 d (22)

food simulant C: 20 °C/10 d (1); 40 °C/10 d (4); 100 °C/3 d (1)

food simulant D1: 20 °C/10 d (1); 40 °C/10 d (7); 60 °C/10 d (18)

Isooctane: 20 °C/10 d (9); 40 °C/10 d (3); 40 °C/2 d (16); 60 °C/10 d (13)

food simulant E: 20 °C/10 d (1); 60 °C/10 d (4); 100 °C/1 h (1); 121 °C/2 d (1)

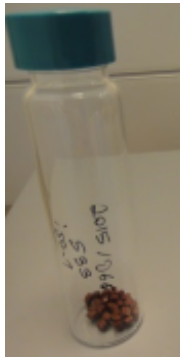
Sample preparation

Petri dishes for rigid multilayer sample ($0.14 \text{ dm}^2/1 \text{ g}$; 2 samples)

Pouch for flexible mono/multilayer
($1 \text{ dm}^2/25\text{-}50 \text{ ml}$; 25 samples)



Immersion for rigid monolayer
($1 \text{ dm}^2/100 \text{ ml}$; 21 samples),
caps, machine parts, cutlery



Immersion for resins
(25 pieces/20 ml;
7 samples)



Filling for trays, bottles
(real S/V; 11 samples)

Cell for rigid multilayer
($0.5 \text{ dm}^2/60 \text{ ml}$;
4 samples)



Problems during migration test

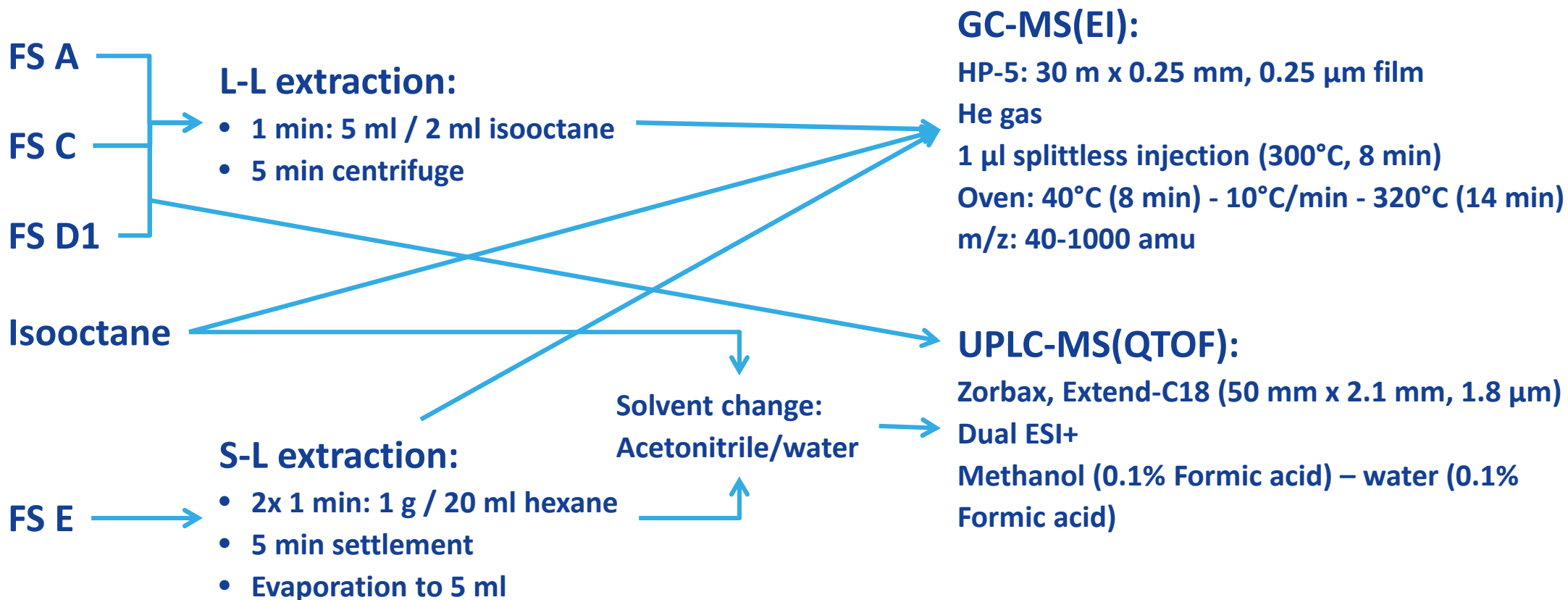
- **Delamination with isooctane (no problem with A and D1) (4x)**
 - ✓ Test with D2
- **Evaporation of ethanol and/or isooctane through multilayer (5x)**
 - ✓ Monolayers: immersion
 - ✓ Multilayers: food and D2 resp.
- **Shrinkage of multilayer in D1 and isooctane (1x)**
 - ✓ Test with food
- **Precipitation/suspension after 24 h in extract C (1x), D1 (2x) and isooctane (19x)**
 - ✓ Filtration of solution
 - ✓ Test with D2
- **Boiling with multi-layers**
 - ✓ Non-pouchable



OR reconsider time-Temperature



Migration solution preparation for analysis



GC-MS and LC-MS results

Substance	CAS No	FCM No	SML	id	N samples (GC/LC)
2,4-di-tert-butylphenol	96-76-4			y	23/13
tris(2,4-di-tert-butylphenyl) phosphate	95906-11-9			y	23/10
erucamide	112-84-5	271		y	22/22
tris(2,4-di-tert-butylphenyl)phosphite (irgafos 168)	31570-04-4	671		y	21/0
(11Z)-11-icosenamide (trans-13-docosenamide)	10436-08-5	589		High 68	10/20
2,6-di-tert-butyl-4-cresol	128-37-0	315	3	y	16/0
(15Z)-15-tetracosenamide					x/16
2,2'-(Tridecylimino)diethanol	68155-05-5				0/14
C22H38O					?/13
m/z=934, 939					?/13
icosanamide	51360-63-5				0/12
diethyl phthalate	84-66-2			y	11/0
oleamide	301-02-0	335			0/11
linoleamide	3999-01-7				0/11
docosanamide (behenamide)	3061-75-4	458			0/11
caprolactam	105-60-2	212	15	y	10/13
hexadecanoic acid, bis(2-ethylhexyl) ester (DEHA)	103-23-1	207	18	y	10/6
palmitamide	629-54-9				0/10

NIAS: Criteria for scoring

Cramer score

- Identification of the Cramer Class using in-silico tools (Toxtree, OECD Toolbox)
 - Class I: score -5; Class II: score 0; Class III: score +5

Sample score

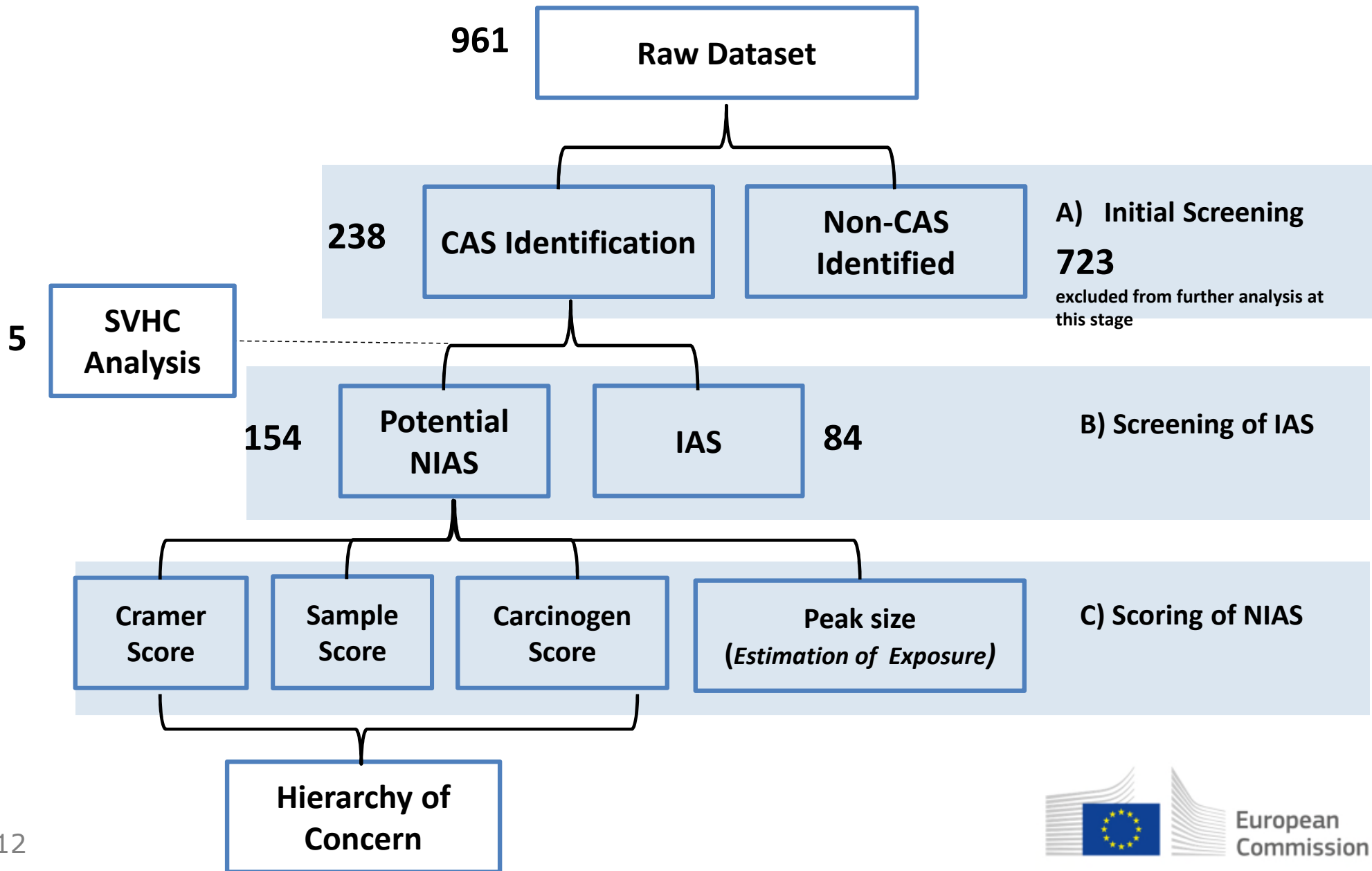
- Occurrence of a NIAS in analysed samples
 - ✓ Score: N of samples in which the NIAS has been identified

Carcinogen score

- Segregate potential CMR and assign maximum concern score
 - CMR: score 10; Non-CMR: score 0

Peak size

- No quantitative analysis
- Only as Small, Medium, High (not used as scoring system)



Total score

Score	<0	0-5	6-10	11-15	16-20	>20	TOTAL
N	28	9	88	12	15	1	154

Frequency of occurrence

Frequency	1	2	3-4	5-6	7	>7	TOTAL
N	88	20	25	13	2	6	154

Next - RA

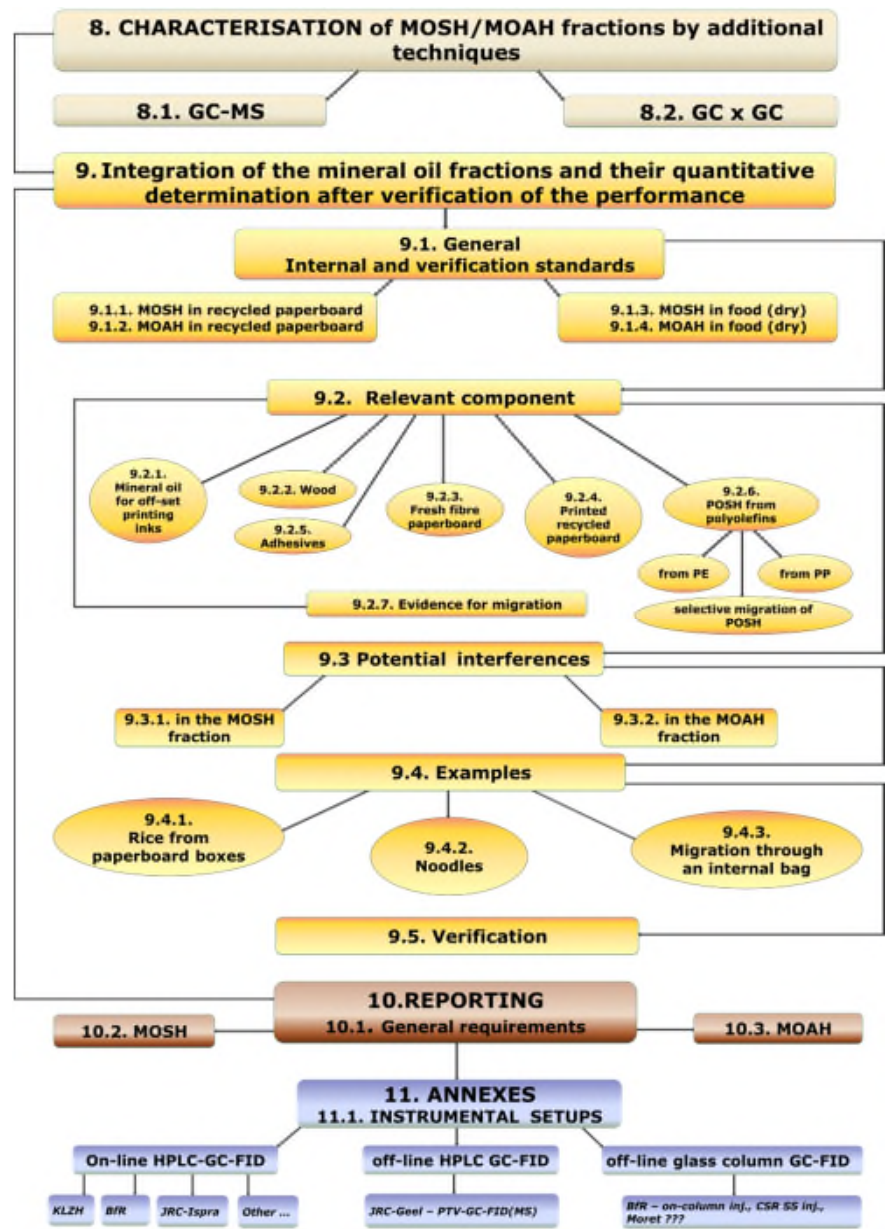
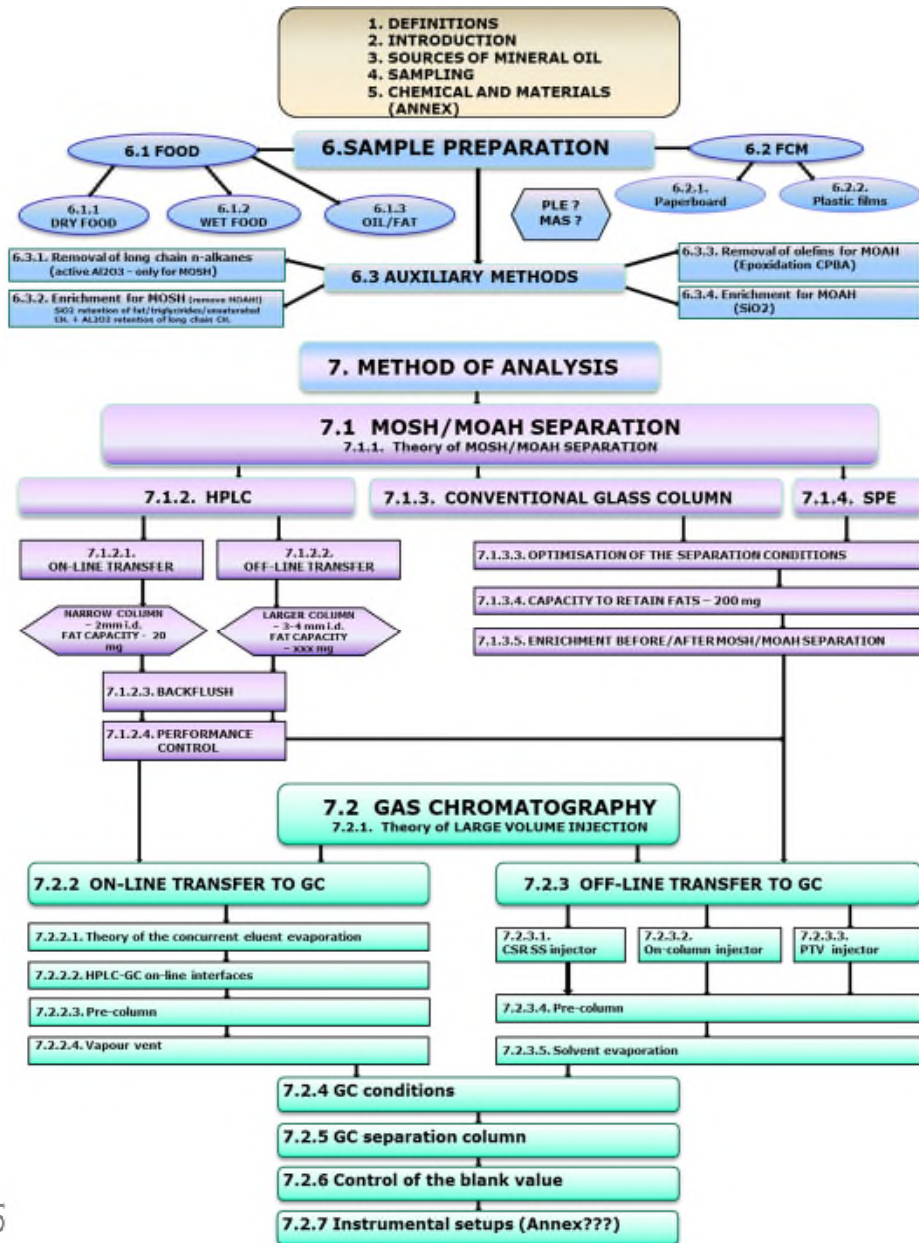
- Risk Assessment based on Matrix Exposure tool
- priority to substances exceeding a score threshold
- Pilot Risk Assessment Scheme

Next - analysis

- Quantification
- Development of multi-analyte methods
- In-house library-building

MOH Guidelines – Recommendation (EU) 2017/84

- **Performance criteria for analytical method**
 - ✓ Unit, significant figures, rounding, measurement uncertainty
 - ✓ LOQ
 - ✓ Recovery
 - ✓ Within laboratory precision
 - ✓ Ability to determine up to C₅₀ (recovery of ≥ 80 %)
 - **Sample preparation methods and deviations to prescribed.**
 - **Auxiliary methods used and deviations to prescribed**
 - **Identified sources and description of strategy identifying**
 - ✓ analysis of the food at different stages of the supply chain + FCM
 - ✓ presence of mineral oil identifiers fraction, e.g. pristane, phytane, hopanes in MOSH, or use of recycled paper (DIPN) by GC-MS or GCxGC-FID/MS
 - ✓ Identification of PAO or POH shall be reported
 - **File containing pictures of chromatograms – voluntary**
- obligatory**
- Total MOSH
 - MOSH ≥ C10 to ≤ C16
 - MOSH > C16 to ≤ C20
 - MOSH > C20 to ≤ C25
 - MOSH > C25 to ≤ C35
 - MOSH > C35 to ≤ C40
 - MOSH > C40 to ≤ C50
 - Total MOAH
 - MOAH ≥ C10 to ≤ C16
 - MOAH ≥ C16 to ≤ C25
 - MOAH > C25 to ≤ C35
 - MOAH > C35 to ≤ C50
- Voluntary**
- MOSH Hump n (n=...)
 - MOAH Hump n (n=...)



Revision kitchenware guideline

- **All materials covered**
- **Harmonisation of test conditions**
 - ✓ Plastic vs ceramic concept
- **First finalise plastic**
 - ✓ Surface-to-volume = 6 dm²/kg food vs. envelop volume method
 - ✓ New proposed test conditions vs. guideline China-Hong Kong imports
 - ✓ Time factor 4 for replacement T>100°C to T=100°C or reflux
 - ✓ Other t-T conditions?
 - ✓ Product testing vs testing individual parts
- **Second phase discussion per material**

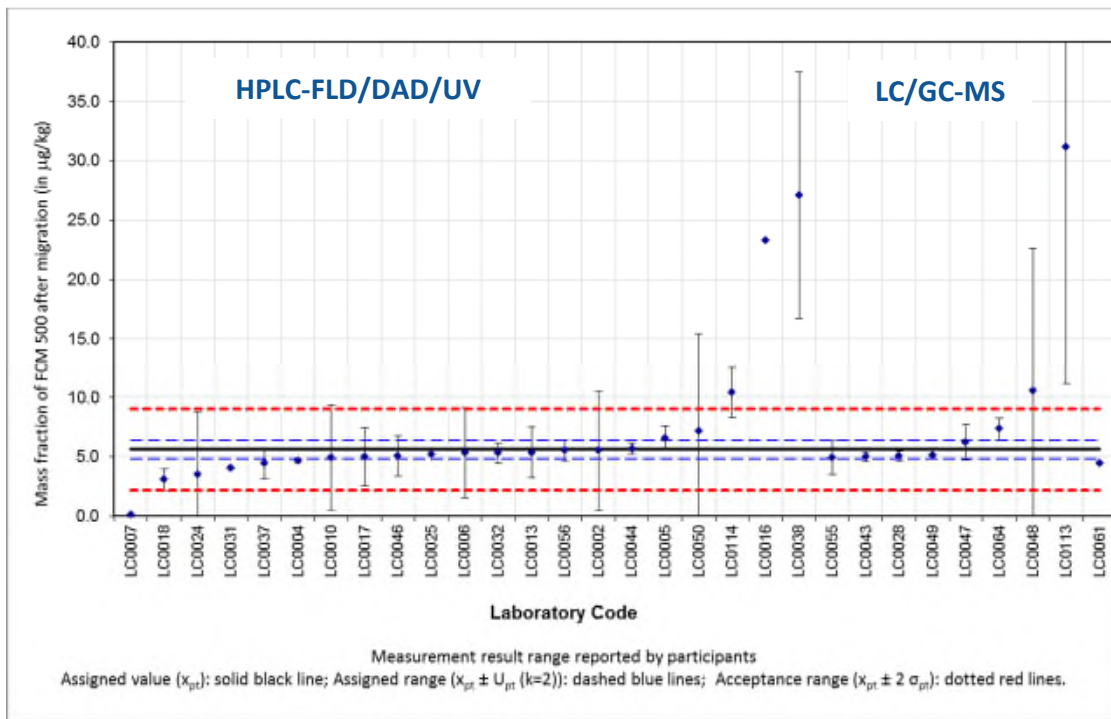
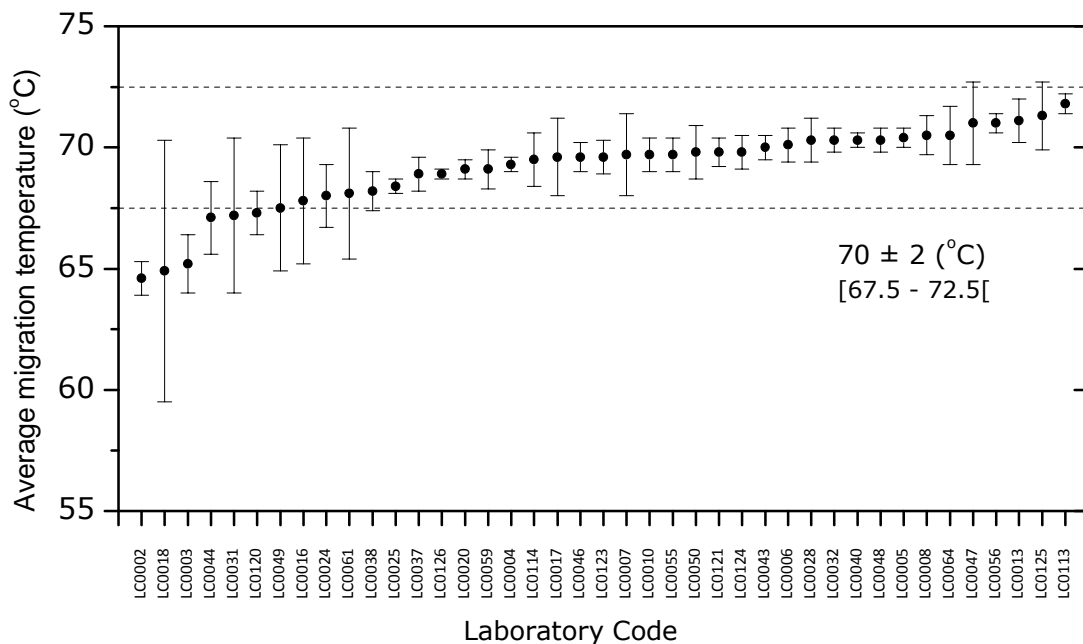
Ceramics, vitreous materials

- Continuation work on development of test conditions for cookware and bakeware

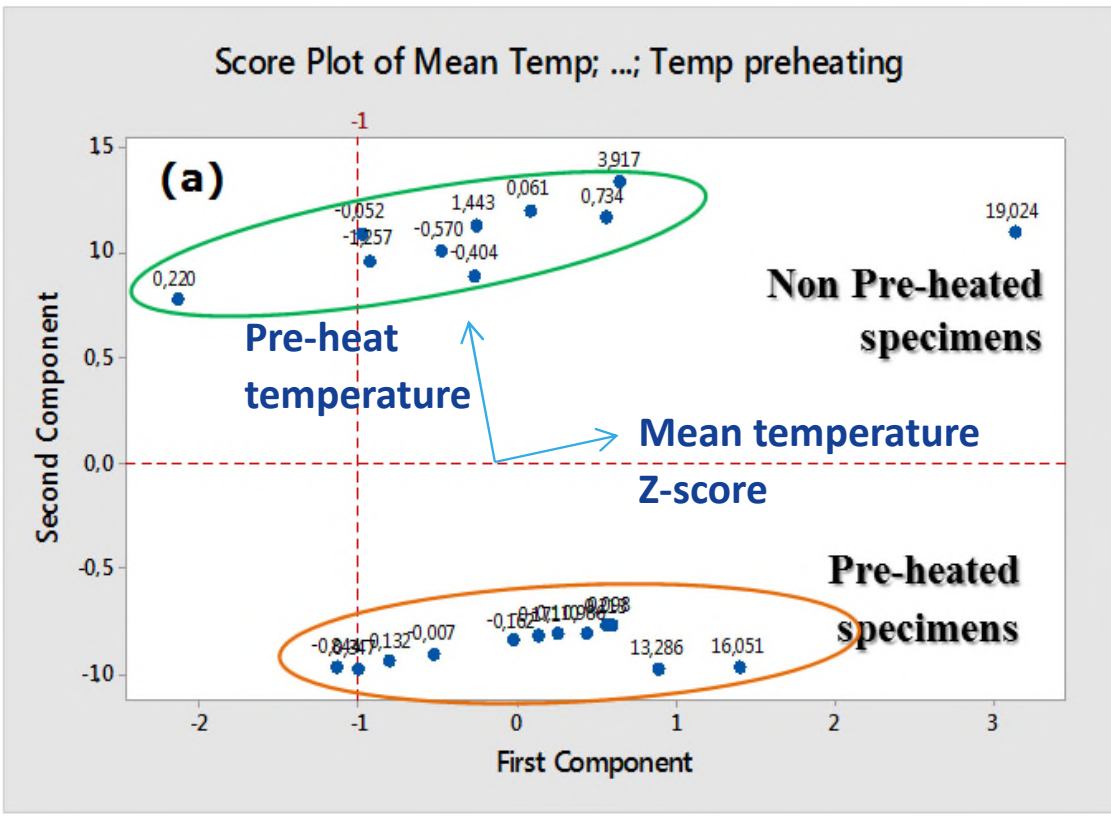
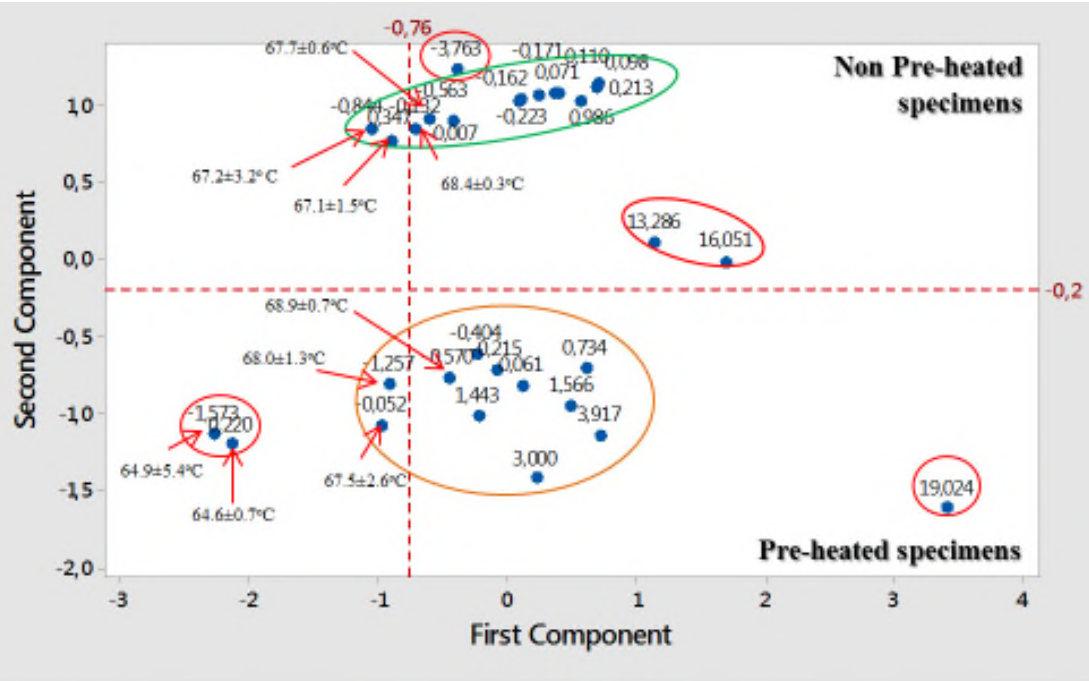
Recycling of plastic

- **Development of a monitoring approach for input and output quality of recycling process**
 - ✓ Suitable for use by business operators,
- **Set up a reporting system that can do statistical analysis on the data.**
 - ✓ Identification of recurrent substances
 - ✓ Identification of possible sources of groups of contaminants of concern
 - ✓ Identification of substances that can be used as a signal that recycled materials originate from a non-food source, such as substances that are representative of detergents, or mineral oils
- **Inventory of present monitoring activities**

Proficiency test temperature control and migration



Principle component analysis

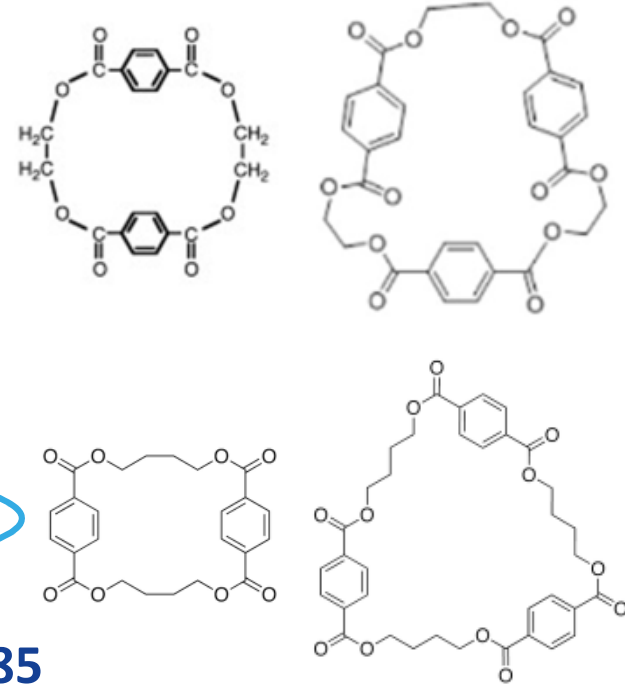


Recommendations:

1. Preheating the FCM before migration shall be avoided
2. Use of calibrated thermometers or data loggers is compulsory
3. Food simulant losses minimised by using proper covering/seal of the cup
4. Filling time should be done within half a minute

Proficiency test oligomers

- **Cyclic potential more toxic**
- **Substances**
 - ethylene terephthalate cyclic dimer (PET cyclic dimer)
 - ethylene terephthalate cyclic trimer (PET cyclic trimer)
 - butylene terephthalate cyclic dimer (PBT cyclic dimer)
 - butylene terephthalate cyclic trimer (PBT cyclic trimer)
- **Matrix**
 - Spiked food simulant D1
 - Spiked food simulant D1 migration solution of PET
- **Method given but voluntary: HPLC-UV**



FCM no 885

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