Laboratory request for PET NIAS evaluation

Reference : Labarotry_NIAS_analysis_request	Version : 1	Date : Nov.12th 2019

This document to be completed and sent to the laboratory together with the sample.

General information

Company Name	
Sample reference	
Date	

Analysis to be carried out

Tick the box corresponding to determination to be carried out.

	Volatile	***Quantification of Targeted NIAS***: - Acetaldehyde - 2- methyl 1,3-dioxolane, - Benzene - Limonene
	Volatile	***Qualification and Quantification of Non-Targeted NIAS***
	Semi – Volatile/ non Volatile	***Quantification of Targeted NIAS***: - Bisphenol A - Ortho-phtalates
	Semi- Volatile	***Qualification and Quantification of Non-Targeted NIAS***
	Non - Volatile	***Qualification and Quantification of Non-Targeted NIAS***
	Oligomers	***Quantification of Oligomers***

The analytical methods implemented to deliver the results will comply with those described in ***Appendix 1***.

Migration assessment

Tick the box corresponding to the migration assessment conditions to be carried out.

	10 days at 60°C
	10 days at 40°C
	365 days at 25°C
	Other – to be specified

The analytical results report will comply with the requirements listed in ***Appendix*** 2.

REMAINING TEST MATERIAL

The laboratory will keep the remaining sample for 6 months, in suitable conditions to avoid any contamination.

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Appendix 1

As a result of previous experience, the choice for the analytical techniques to be applied for PET resins and derivates (bottle wall – preforms …) are described below.

Volatile Analysis

Sample preparation

The sample must be cryogenically ground to a particle size below 750µm in order to be homogenised and to facilitate thermal desorption from small particles; it is very important to avoid both product degradation, losses of volatile substances (placing, weighing and sealing the sample in the HS vial as quick as possible is recommended) and cross contamination between samples when grinding them. The non-grinding method can be applied for specific cases but only when data show that there is no significant difference between grinding and non-grinding.

Thermal desorption

The thermal desorption conditions must be adapted to PET which means temperatures high enough to desorb volatile substances like acetaldehyde and less volatile substances such as limonene; the thermal desorption conditions should be such that they do not generate degradation of the PET sample. The desorption temperature that is recommended is 200°C, other temperatures could be used if data shows the relevance, (ie. acetaldehyde regeneration). The time at 200°C is typically set at 1h.

Quantification of Targeted NIAS:

The main targeted substances listed in the following table:

Name	EC-Number	CAS-Number
Acetaldehyde	200-836-8	75-07-0
1,3-Dioxolane, 2-methyl-	207-841-4	497-26-7
Benzene	200-753-7	71-43-2
Limonene	205-341-0	138-86-3

Volatile substances will be analysed using Headspace HR-GC–MS. Quantification will be done by adding an internal standard. To have a more accurate quantification GC-FID is preferred, with a method sensitivity limit of 10 to 30µg/kg for targeted NIAS. A triplicate analysis is required.

Qualification and Quantification of Non-Targeted NIAS

Regarding HR-GC-MS, the use of The National Institute of Standards and Technology (NIST) library as a well-known and validated data base is preferred. However, other libraries can be used as long as their robustness can be demonstrated. All substances found will be listed and a semi-quantification will be carried out with a Limit of Quantification of 30 µg/kg.

Semi-Volatile Analysis

Sample preparation

The sample must be cryogenically ground to a particle size below 750µm in order to be homogenised and to facilitate thermal desorption from small particles; it is very important to avoid both product degradation, losses of volatile substances and cross contamination between samples when grinding

them. The non-grinding method can be applied for specific cases but only when data show that there is no significant difference between grinding and non-grinding.

Solvent extraction
The solvent and the extraction conditions must be adapted to PET; the choice of solvent, justification and the extraction method will be reported.

Quantification of Targeted NIAS
Main targeted substance are Bis-Phenol A and some ortho-phthalates GC can be used with appropriate detector. The analytical method including operating settings and calibration method will be reported. It is aimed at having a method sensitivity limit at 0.1mg/kg PET for Bis-Phenol A and at 0.1 to 0.3 mg/kg PET for o-phtalates A triplicate analysis is preferred

Qualification and Quantification of Non-Targeted NIAS
Semi – volatiles substances can be analysed using GC-High Resolution MS The choice of MS library will be reported together with a robustness validation description. All substances found will be listed and a semi-quantification will be carried out with a Limit of Quantification of 0.1 mg/kg.

Some semi-volatile substances can also be detected with a non-volatile analysis method.

Non-Volatile and oligomers Analysis:

Sample preparation:
The sample must be cryogenically ground to a particle size below 750µm in order to be homogenised and to facilitate thermal desorption from small particles; it is very important to avoid both product degradation, losses of volatile substances and cross contamination between samples when grinding them. The non-grinding method can be applied for specific cases but only when data show that there is no significant difference between grinding and non-grinding.

Solvent extraction:
The solvent and the extraction conditions must be adapted to PET; the choice of solvent, justification and the extraction method will be reported.

Quantification of Targeted NIAS:
The main targeted substances are Bis-Phenol A, oligomers and some ortho- phthalates (if not already considered as a semi-volatile substance). LC will be used with appropriate detector. The analytical method including operating settings and calibration method will be reported. It is aimed at having a method sensitivity limit at 0.1mg/kg PET for Bis-Phenol A and at 0.1 to 0.3 mg/kg PET for ortho-phthalates.

Quantification of Oligomers
Oligomers should be identified and excluded from the non-targeted NIAS evaluation. An overall oligomer amount in mg/kg will be provided.

Qualification and Quantification of Non-Targeted NIAS:

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Non- volatile substances can be analysed using LC-HR-MS. The choice of MS library will be reported together with a robustness validation description. All substances found will be listed and a semi-quantification will be carried out with a Limit of Quantification of 0.1 to 0.3 mg/kg.

Some non-volatile substances can also be detected with a semi-volatile analysis method

Substance Identity Confirmation

Due to the fact that methods of analysis somehow overlap and many substances can fall into the common region of ‘’heavier’’ volatiles and ‘’lighter’’ semi-volatiles, and the same applies with the ‘’heavier’’ semi-volatiles and the ‘’lighter’’ non-volatiles, it is highly recommended to use semi-volatiles analysis method as identification confirmation tool for ‘’heavier’’ volatiles and ‘’lighter’’ non-volatiles and vice-versa.

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Appendix 2

The analytical report to be delivered to the client will include the following :

General information

The report will describe:

- Coordinates of laboratory including name of accountable person.
- Date and conditions of sample at reception.
- Reference number of the sample as mentioned on the label
- Date of analysis
-

Methods

All methods used to deliver the results will be reported and include all of the elements which are listed in Appendix 1.
If no accredited method is used the level of repeatability of LOD must demonstrated. The level of interpretation by any laboratory employee should be limited by clear SOP which guide the tolerance allowed for a triplicate measurement of a sample

For each method used, describe:

- Sample preparation conditions
- Thermal desorption conditions or solvent extraction conditions
- Gas or Liquid chromatography equipment and analysis conditions including detectors that were used
- Calibration method
- Mass Spectroscopy conditions with library reference.

Results

All substances, targeted and non-targeted will be reported in a table, and classified into 3 categories , volatile, semi-volatile and non-volatile.

Targeted substances

The result will be expressed in µg/kg or mg/kg
For each substance, both LOD and LOQ will be reported.
If triplicate, the 3 results will be included.

Non – Targeted substances

For each substance identified, the following information will be reported:

- Identification, molecular weight, elemental composition (level of confidence)
- CAS number
- Semi-quantified level for all identified substances, even if below LOQ.
- If the substance is a listed CMR, it must be reported.

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Migration assessment

The estimation of migration of substances will be carried out using a mathematical model. For high volume polymers such as PET, a pragmatic migration model has been established and is a scientifically recognised, widely used tool for food law compliance evaluation purposes.
Migration models which can be used are available from several companies/institutes such as , but not limited to :

- French National Institute for Agricultural Research (INRA) Safe Food Packaging Portal version 315,

- MIGRATEST software16 and AKTS-SML Software.

The laboratory will provide a migration assessment for each identified NIAS, the concentration will be calculated for a PET container with a surface-volume ratio of 6 dm2 per 1 kg food and storage conditions according to customer request.