



HARRPA

**Hydrocarbon Resins, Rosin Resins and Pine Chemicals
Producers Association**

Resin and Mineral Oil Analysis in Food Contact Materials

The quality of food in relation to contamination with non-food impurities has gained significant importance at consumer, governmental and industrial level. Presence of mineral oil in foodstuffs from direct and indirect food contact materials has received increased attention over the recent years (Stieger, 2016).

Tackifier resins are used extensively in diverse end uses such as diapers, packaging, medical plasters, tapes, labels, coatings and chewing gum. For decades, they have provided safe and efficient solutions, backed up by extensive regulatory clearances around the globe. Resins are derived from raw materials obtained from natural or petrochemical sources, or synthetically produced. Resins and chemically modified resins should not be mistaken for mineral oils.

Mineral oil contamination could originate from food packaging, processing aids, additives and lubricants. In case of food packaging, mineral oils could potentially migrate from the packaging into the food. The risks of mineral oil / mineral oil residues migration from packaged food should therefore be properly assessed.

A mineral oil is a generic term used to group several petroleum derived liquids, manufactured by atmospheric and vacuum distillation of crude oil followed by further refinement through extraction, dewaxing and hydrogenation or other treatment. A mineral oil is a UVCB¹ substance and not a mixture (Concawe, 2017).

Since 2009, two chromatographic terms were introduced, i.e. MOSH for the Mineral Oil saturated Hydrocarbons and MOAH for the Mineral Oil Aromatic Hydrocarbons. MOSH and MOAH are chromatographic measures of respectively the alkane and aromatic content of a mineral oil.

The MOSH in the range of C16 to C35 may accumulate in human tissue and could cause harmful health effects. The presence of MOAH is a potential concern as this is associated with polycyclic aromatic hydrocarbons (3-7 membered rings) that may have carcinogenic (and mutagenic) properties. (European Food Safety Authority, 2012).

Due to the identified health effects of MOSH and MOAH in combination with their potential migration into food, it is essential that the MOSH and MOAH content in food can be properly determined.

The Technical Committee of the European Committee for Standardization (CEN/TC 275 - Food analysis - Horizontal methods) published a standard (EN 16995:2017) that describes an analytical method for the

¹ Complex substance of Unknown or Variable composition, Complex reaction products or Biological materials



determination of saturated and aromatic hydrocarbons (from C10 to C50) in vegetable fats and oils and foodstuff on basis of vegetable oils with online-HPLC-GC-FID. The method can be used for the analysis of MOSH and/or MOAH in vegetable oils. The standard explicitly stated that it has only been proven suitable for a MOSH or MOAH content of minimal 10 mg/kg and is not intended to be applied to other matrices. It also mentioned the need for further verification of the MOSH and MOAH fraction in case of *interferences* from natural sources.

The current method for a MOSH and MOAH is described in the literature (Lommatzsch, Biedermann, Grob, & Simat, 2016). It involves an analysis based on High-Performance Liquid Chromatography-Gas Chromatography with a Flame Ionization Detector (HPLC-GC-FID). The analysis generates broad humps of unresolved peaks in a chromatogram. These humps may contain peaks that originate from hydrocarbons other than mineral oil, such as synthetic or natural hydrocarbons. A simple HPLC-GC-FID analysis does not differentiate between mineral oils and other substances; e.g. when analyzing for the presence of mineral oils, the low molecular weight fraction of Tackifier Resins could produce 'false positives' (Lommatzsch, Biedermann, Grob, & Simat, 2016). Consequently, the MOSH and MOAH content is overestimated. Other resins like derivatives of rosin resins can also result in an overestimation of the MOSH and MOAH content. More information is often required to identify the contamination in packaging materials.

According to Biedermann and Grob, the analysis of mineral oil and oligomeric mixtures is demanding. They state that two-dimensional Gas Chromatography with Mass Spectrometry and/or Flame Ionization Detection (GCxGC-MS/FID) offers the best technique for hydrocarbons analysis due to outstanding separation power and high sensitivity (Biedermann & Grob, 2015).

In the absence of a test method that can separate between mineral oil and non-mineral oil substances, it is very important to take the 'false positives' as produced by the HPLC-GC-FID tests into account before setting standards for MOSH and MOAH content in food and/or food packaging products and articles.

The Cefic sector group representing the Hydrocarbon-, Rosin Resins and Pine Chemical producers in Europe - HARRPA - is committed to supporting the safe use of our products by both the industry and the consumers. Therefore, it has taken the initiative to develop an improved test method that can better distinguish between MOSH/MOAH and tackifier resins residues, thereby contributing to establishing appropriate standards for the industry.

HARRPA collaborated with Laboratory Lommatzsch in Germany, which is specialized in the analysis of MOSH and MOAH. We completed a feasibility study that differentiates MOSH migrated from recycled cardboard and oligomers migrated from hydrogenated hydrocarbon resin (transfer via gaseous phase). The conclusion was that when the HPLC-GC-FID method is applied to mixtures of hydrocarbons from different sources, only the total amount of all hydrocarbons in the target range of C16 to C35 can be quantified, instead of just the MOSH. In addition, a qualitative evaluation of their sources is hardly feasible. In this study, it was demonstrated that oligomers from hydrocarbon resins can be misinterpreted as MOSH, thereby overestimating the resulting measured "MOSH"-concentration.

In contrast, a test method using GCxGC-MS/FID is able to distinguish between MOSH migrated from recycled cardboard and oligomers migrated from a hydrogenated hydrocarbon resin sample. It was also

shown that a quantitative determination of the different concentrations of MOSH and resin oligomers can be performed.

Based on the positive outcome of the feasibility study, HARRPA decided to continue with the development of a suitable analytical method in a comprehensive analysis of saturated and aromatic oligomeric hydrocarbons from different tackifier resins by GCxGC-MS/FID. The main resin families that are available on the market will be analysed and characterised using the two-dimensional GC method. HARRPA's study on the correct analysis of resins is a comprehensive effort, scheduled to be completed in the first quarter of 2018. Preliminary results do confirm that also other resin families can be well distinguished from mineral oils provided the correct analytical methods are being employed.

The GCxGC-MS/FID studies confirmed the false positive nature of resin species in HPLC-GC-FID based mineral oil analyses!

As a result, HARRPA reaches out to the industry stakeholders to ensure that this information is being taken into account when industry standards and test procedures are considered.

For further information, please contact your resin supplier or HARRPA secretariat.

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