

Quality-tested pre-column (Retention Gap) for the determination of mineral oil contamination in food, cosmetics and packaging materials

Non-discrimination from C₁₀ to C₅₀

Verena Koospal, food chemist



Application note ASAN 2201

Quality Tested Retention Gap

Application note ASAN 2201

Introduction

The analysis for the determination of mineral oil contamination in food, cosmetics and packaging materials is carried out with an online LC-GC-FID coupling. In the HPLC, the substance groups mineral oil saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MOAH) are separated. The two fractions are transferred to the GC with 450 µL of solvent each, where they are first transferred to an unoccupied precolumn, a so-called Retention Gap. The majority of the solvent is evaporated via a special interface. Subsequently, the analytes are transferred to the coated GC separation column, separated according to boiling point and detected by means of FID.

GC columns

- MOSH/MOAH Retention Gap: AS-GC-4060
- MOSH/MOAH separation column: MXT-1 0.25 µm, 15 m x 0.25 mm IDAS-RE-70120

According to the current state of analytics, MOSH and MOAH are quantified between C₁₀ and C₅₀ [1]. A necessary freedom from discrimination in the determination range C₁₀ to C₅₀ is to be ensured by an alkane standard C₁₀-C₅₀ (MOSH/MOAH retention time standard, AS-RE-31076). In the past, even new Retention Gaps have repeatedly failed to ensure freedom from discrimination for C₅₀. Therefore, Axel Semrau exclusively offers a Retention Gap tested for freedom from discrimination.

Ensure freedom from discrimination

To ensure freedom from discrimination in the determination range C₁₀-C₅₀, the following peak area ratios are formed:

- C₅₀ to C₂₀ for high molecular weight boiling range
- C₁₀ to C₂₀ for the low molecular boiling range

The ratio of C₅₀ to C₂₀ should be between 0.8 and 1.2 [1]. Freedom from discrimination in the front boiling range from C₁₀ should also be ensured by a peak area ratio of C₁₀ to C₂₀ between 0.8 and 1.2. Ensuring this freedom from discrimination can be a challenge for users, as the causes can be of different origins.

Causes of discrimination from C₁₀, e.g.,

- Pressure settings on the GC not adjusted (too much evaporation)
- Shifted fraction window in HPLC
- Leakage in the system

Causes of discrimination from C₅₀, e.g.,

- (Dirty) Retention Gap
- Dirty FID nozzles
- Dirty GC separation columns
- Incorrect installation of the GC columns

In the past, the causes of discrimination of C₅₀ occurred even when a new retention gap was properly installed. The recoveries of C₅₀ were then sometimes far below 80 %.

Figure 1 shows an example of the retention time standard used to ensure freedom from discrimination. The measurement shows a strong discrimination of the alkanes from C₃₅. The recovery of C₅₀ is only 10 % relative to C₂₀. In this case, a new Retention Gap was the cause of the discrimination.

The retention gap tested by Axel Semrau shows recoveries of C₅₀ of at least 80 % and thus fulfils the requirements for non-discrimination of C₅₀ according to [1] of 80-120 %.

Figure 2 shows the standard on the front GC channel (MOSH). To ensure freedom from discrimination for both GC channels, the standard is also measured in *reverse* on the rear channel (MOAH) (Figure 3). The Retention Gap tested by Axel Semrau was used for the determination. All alkanes show comparable peak heights and areas.

Quality Tested Retention Gap

Application note ASAN 2201

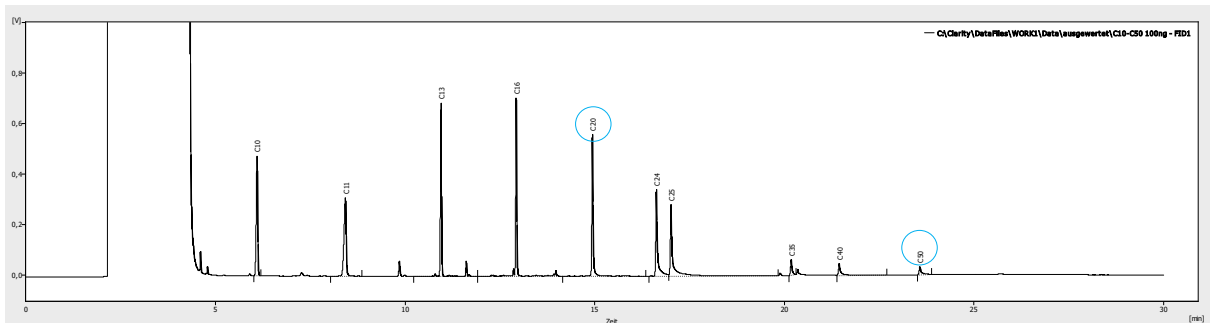


Figure 1: MOSH LC-GC-FID chromatogram of the injection of a retention time standard with the components C₁₀, C₁₁, C₁₃, C₁₆, C₂₀, C₂₄, C₂₅, C₃₅, C₄₀ and C₅₀ on the front GC channel. The alkanes C₂₄ and C₂₅ already show strong tailing. The alkanes C₃₅, C₄₀ and C₅₀ are visibly discriminated.

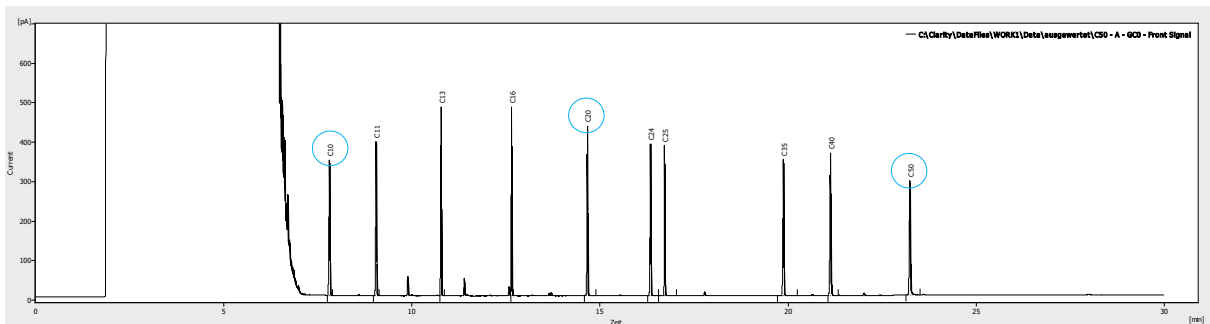


Figure 2: MOSH LC-GC-FID chromatogram of the injection of a retention time standard with the components C₁₀, C₁₁, C₁₃, C₁₆, C₂₀, C₂₄, C₂₅, C₃₅, C₄₀ and C₅₀ on the front GC channel. All alkanes show comparable peak heights and areas.

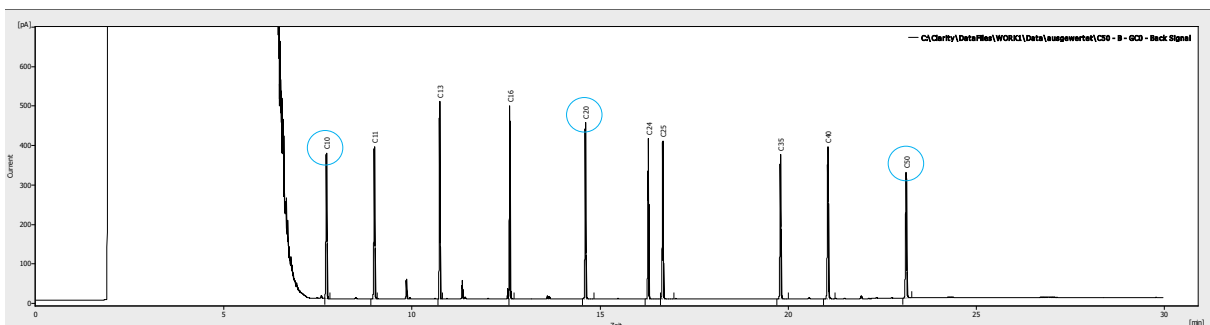


Figure 3: MOSH LC-GC-FID chromatogram of the injection of a retention time standard with the components C₁₀, C₁₁, C₁₃, C₁₆, C₂₀, C₂₄, C₂₅, C₃₅, C₄₀ and C₅₀ on the rear GC channel. All alkanes show comparable peak heights and areas.

Table 1 shows the peak areas and peak area ratios to the measurements of the standard from Figure 2 (MOSH channel) and Figure 3 (MOAH channel). The peak area ratios are in the range

of 0.95-1.01 and thus fulfil the requirements for freedom from discrimination according to [1] very well .

Quality Tested Retention Gap

Application note ASAN 2201

Table 1: Determination of the peak area ratios C_{10} to C_{20} and C_{50} to C_{20} in the retention time standard to the measurements in Figure 2 and 3. The requirements for freedom from discrimination according to [1] are fulfilled.

GC channel	Peak areas			Peak Area ratio	
	C_{10}	C_{20}	C_{50}	C_{10} to C_{20}	C_{50} to C_{20}
MOSH	655,685	653,94	623,674	1,00	0,95
MOAH	672,541	667,133	645,298	1,01	0,97

Summary

The internal quality control of the Retention Gap, which is exclusively available from Axel Semrau, ensures that the requirements for non-discrimination of C_{50} and C_{10} are met. Discrimination caused by a new Retention Gap can be

excluded by the internal comprehensive quality check. The quality-tested Retention Gap has already proven itself in routine use by a large number of users. However, if problems with discrimination should occur again when a new Retention Gap is installed, it can be exchanged without incurring any costs.

Literature

- [1] Bratinova, S. and Hoekstra, E., Guidance on sampling, analysis and data reporting for the monitoring of mineral oil hydrocarbons in food and food contact materials, EUR 29666 EN, Publications Office of the European Union, Luxembourg, 2019, ISBN 978-92-76-00172-0, doi:10.2760/208879, JRC115694.

The quality check of the Retention Gap is a development by Axel Semrau.

Subject to technical changes

Axel Semrau GmbH & Co. KG
 Part of the Trajan Family
 Stefansbecke 42
 45549 Sprockhövel
 Tel.: +49 2339 / 12090
www.axelsemrau.de
info@axelsemrau.de