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Application News

No. SCA_280_081

GCMS

Analysis of VOC and FOG emissions from moulded components for automobiles according to VDA 278

Introduction

In Germany millions of new cars are produced and licensed every year. In the supply chain of a vehicle, control standards have to be followed to ensure a high quality final product. The VDA regulations e.g. address the organic emissions from automotive components. Based on thermodesorption techniques, VDA 278 regulates the test procedure for non-metallic materials used for moulded components in automobiles. With this, two classes of compounds are distinguished: highly and medium volatile substances (VOC) up to C25 and those of low volatility (FOG) in the range of C14 up to C32.

In this application the upper layer material of fairing parts used in automobiles has been analyzed according to VDA 278 regarding its organic emission. The influence of open storage time on the VOC and FOG content has been additionally investigated.

Workflow and analytical conditions

According to VDA 278 the workflow shown in figure 1 has to be followed. A control standard solution of 18 compounds has to be checked to ensure instrument performance followed by the measurement of 2 standards as calibration substances. In case of VOC toluene, for the FOG calibration hexadecane is used to determine the respective response factor. Each sample has to be measured twice: One sample is used for a VOC analysis only, the second one is measured under VOC conditions subsequently followed by FOG analysis.

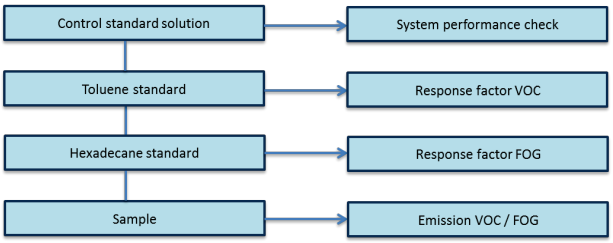

Figure 1: Workflow according to VDA 278

Table 1: Analytical conditions for VOC

TD-20	
Desorption temperature:	90 / 120 °C
Desorption flow:	80 mL/min
Desorption time:	30 min
Trap cool temperature:	-20 °C
Line temperature:	280 °C
Interface temperature:	280 °C

GCMS	
Column:	Optima5MS 50 m, 0.32 mm ID, 0.5 µm film thickness
Oven Temp.:	40 °C, 2 min → 3 °C/min to 92°C → 5 °C/min to 160 °C → 10 °C/min to 280 °C, 10 min
Column flow:	1.3 ml/min
Split:	100:1
Ion Source Temp.:	280 °C
Interface Temp.:	200 °C
Acquisition Mode:	Scan (29 - 450 u)

The measurements were carried out using GCMS-QP2010 SE combined with the TD-20 thermodesorption unit. Following VDA 278, desorption temperatures for the tubes were set to 90 °C (VOC) and 120 °C (FOG) respectively, desorption time was 30 min each. The chromatographic separation was performed by means of an Optima5MS column with 50 m length, 0.32 mm ID, 0.5 µm film thickness. The oven temperature for the FOG run was programmed beginning at 50 °C, held for 2 min, ramped with 25 °C/min to 160 °C followed by a second ramp of 10 °C/min up to 280 °C final temperature, held for 30 min. The settings for the VOC run are summed up in table 1. Compound detection was done by a MS full scan over the expected mass range. Due to the high concentrations both in the standards as well as the samples, a split of 100:1 was used to prevent detector saturation. Additionally, the amount of standards injected into the TD tubes could be decreased by a factor of 4 to 0.5 µg absolute. The sample was cut into small pieces of around 10 mg placed into empty TD tubes as shown in figure 2. For the standard solution, tubes filled with Tenax were used, the solvent was evaporated after injection under a continuous flow of nitrogen gas (5 min at 100 ml/min).

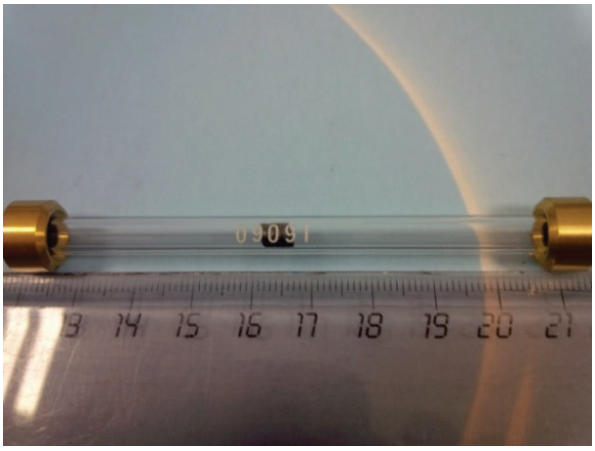

Figure 2: Sample placed into the TD tube

Results and discussion

The chromatogram of the control standard solution is shown in figure 3. As required by VDA 278, o-xylene and n-nonane are baseline separated. Undecane and 2,5-dimethylphenol coelute (see insert in figure 3) but can both be identified using the library search. Recovery rates for the compounds checked were well within the limits of 60 - 140%. For toluene, the recovery was 98%. Response factors calculated for VOC and FOG were 0.08 and 0.06, respectively. With these, the emissions from the upper layer material of fairing parts were measured directly after opening the package. The chromatograms for the VOC and FOG run are shown in figure 4. For emission calculations all peaks have been summed.

An analogous measurement was repeated after 7 days of open sample storage in a neutral environment as is compulsory for VDA278 analyses. The emission values for all measurements before and after storage are summarized in Table 2. As would be expected, the emission was significantly decreased after longer storage time. The VOC value decreased drastically compared to the FOG content as these compounds have higher volatilities.

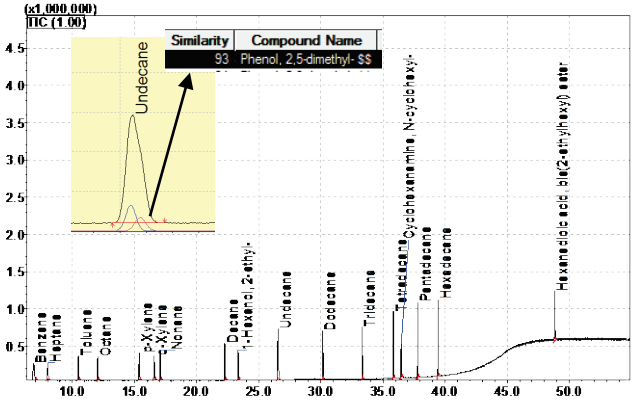

Figure 3: Chromatogram of the control standard

Table 2: VOC and FOG emission from the upper layer material of fairing parts

After unpacking:		After 7 days of open storage:	
Emission VOC 1:	299 µg/g	Emission VOC 1:	160 µg/g
Emission VOC 2:	290 µg/g	Emission VOC 2:	156 µg/g
Emission FOG:	234 µg/g	Emission FOG:	164 µg/g

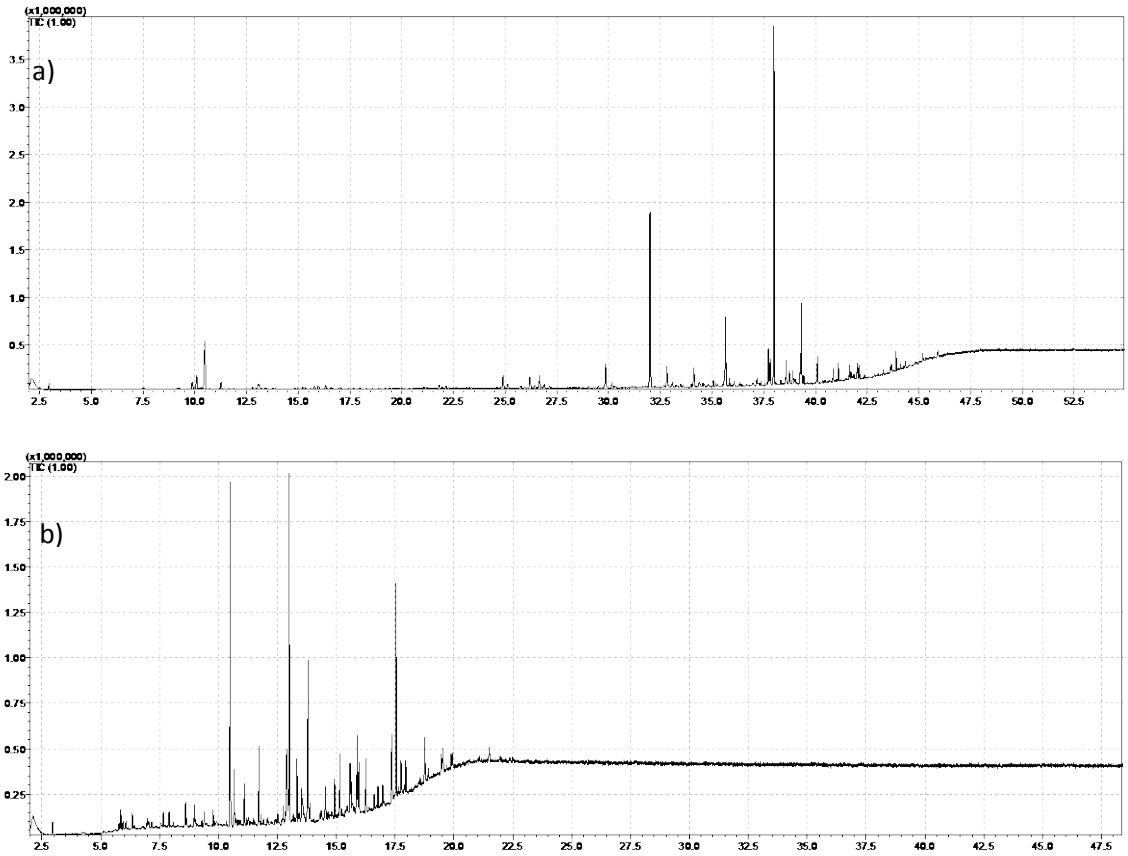

Figure 4: Chromatograms of the upper layer material of fairing parts measured under a) VOC and b) FOG conditions

Conclusions

In this application the upper layer material of fairing parts used in automobiles was analyzed for VOC and FOG compounds according to VDA 278. The GCMS-QP2010 SE in combination with TD-20 proved to be fully sufficient for such kind of analysis.

Furthermore, a high influence of the storage time on the emission values was determined indicating that following the defined storage time in VDA278 is extremely important for reliable and reproducible detection of the emission values.

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