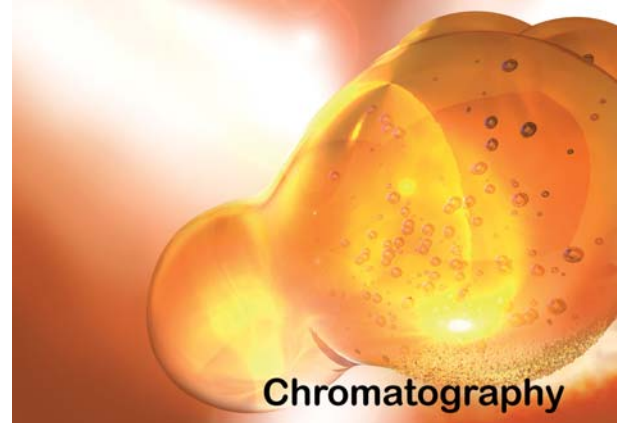


Application Note

Thermodesorption/GC-MS analysis of organic emission (VOC, FOG) in non-metallic car materials according to VDA 278



The emission of non-metallic indoor car materials is usually characterized according to the VDA 278 method [1] in which VOCs (up to C20) and less volatile substances (FOG value C16 to C32) are analyzed using thermal desorption and subsequent GC-MS analysis.

This application was carried out using the Shimadzu TD-20/GCMS-QP2010S. The TD-20 can process up to 48 sample tubes automatically and the thermally extracted analytes are refocused on a Peltier cooled cryo trap.

The sample tubes are decapped automatically and sealed in the desorber heating block. Before heating under a specific gas flow, a leak check is done and the cryo trap is cooled. In this setup only thermal extraction from the inside of the tube is transferred to the GC-MS. This excludes transfer of any contaminants from the outer surface of the tubes to the MS.

The VOC and FOG analyses were carried out with extraction temperatures of 90 °C and 120 °C respectively. The analytical column was an RTX-5 30 m, 0.25 mm, 0.25 µm with a temperature program of 40 °C, 1 min then 10 °C/min to 280 °C. The mass spectrometer was run in full scan (35-300 amu). The split ratio was set to 20:1. The recovery rates over a broad range of boiling points were checked and were 100 % even for C36.

In Fig. 1 the chromatogram of the control standard is shown. The insert shows the zoom of the overlay of two replicates indicating the high precision of the system.

The batch was started with a blank run followed by VOC standard runs. Two replicates were made for precision control. Before and after the polyurethane sample was measured, another blank was run ensuring that there was no carry-over.

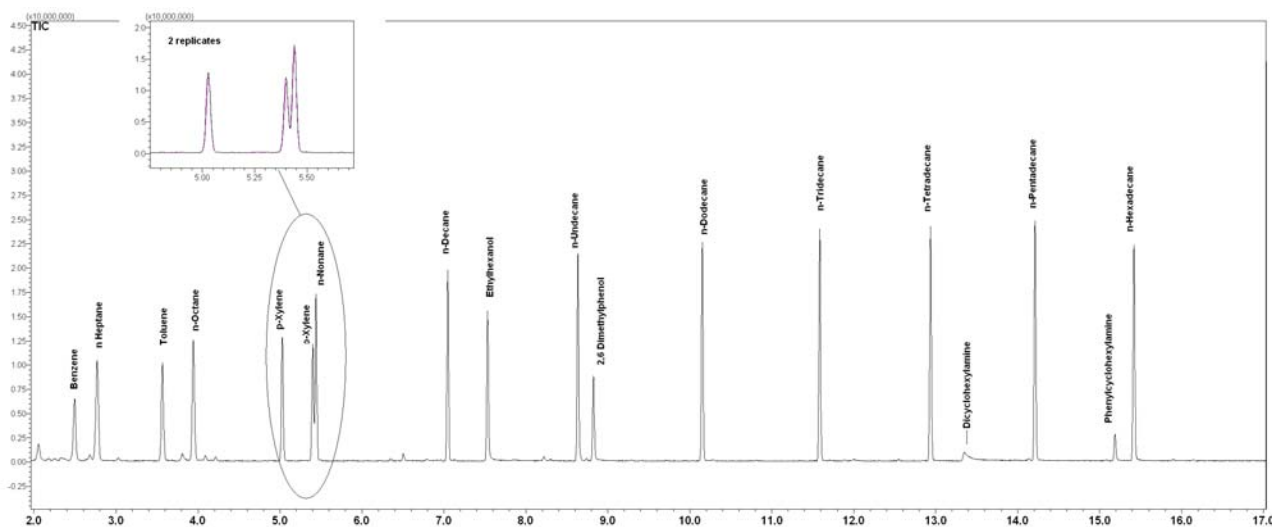


Fig. 1: TIC of the control mix injected into a thermal desorption tube and thermally extracted (VOC condition). The zoom shows the precision of two replicates (o,p-xylene, nonane)

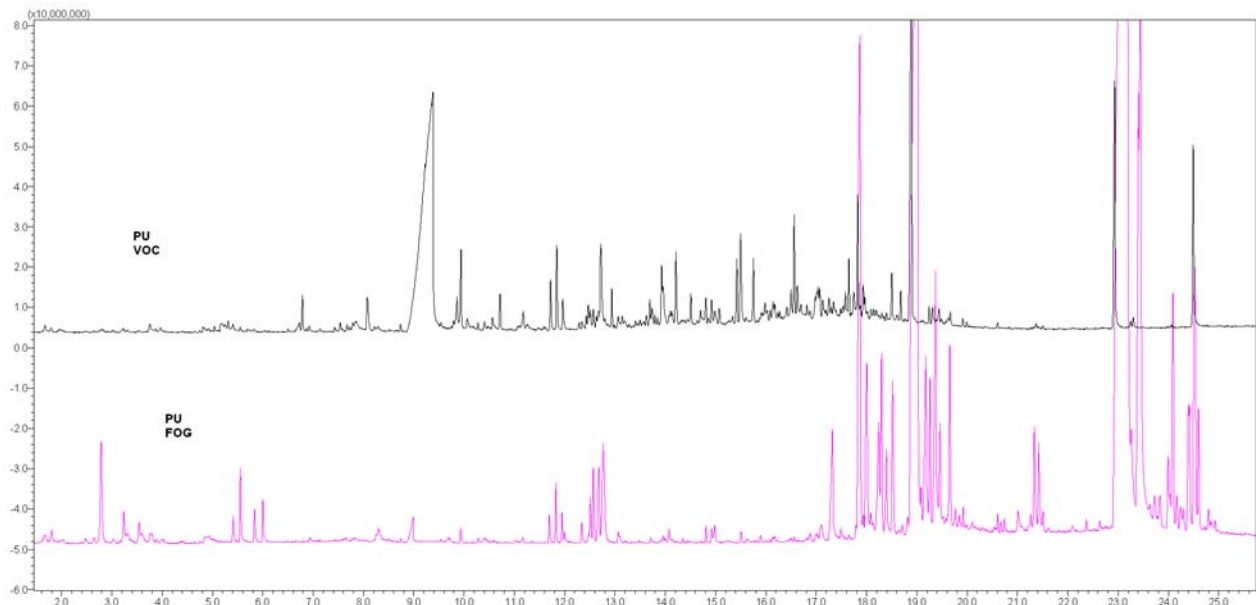


Fig. 2: TIC data of a polyurethane (PU) sample thermally extracted at 90 °C (VOC) and 120 °C (FOG).

The procedure was duplicated when performing the FOG analysis. Standards were injected with a syringe into a glass wool plug placed into the desorption tubes under a gas stream of 20 mL/min.

Fig. 2 shows the result obtained with the polyurethane sample (14.6 mg) after extraction at 90 °C (VOC) and 120 °C (FOG) respectively. As an example Fig. 3 shows the peaks relative to ethylhexanol and C16 (VOC).

The corresponding concentrations are 108 and 24 µg/g.

[1] VDA 278
Recommendation of the German Association of the Automotive Industry (Verband deutscher Automobilindustrie), September 2002

