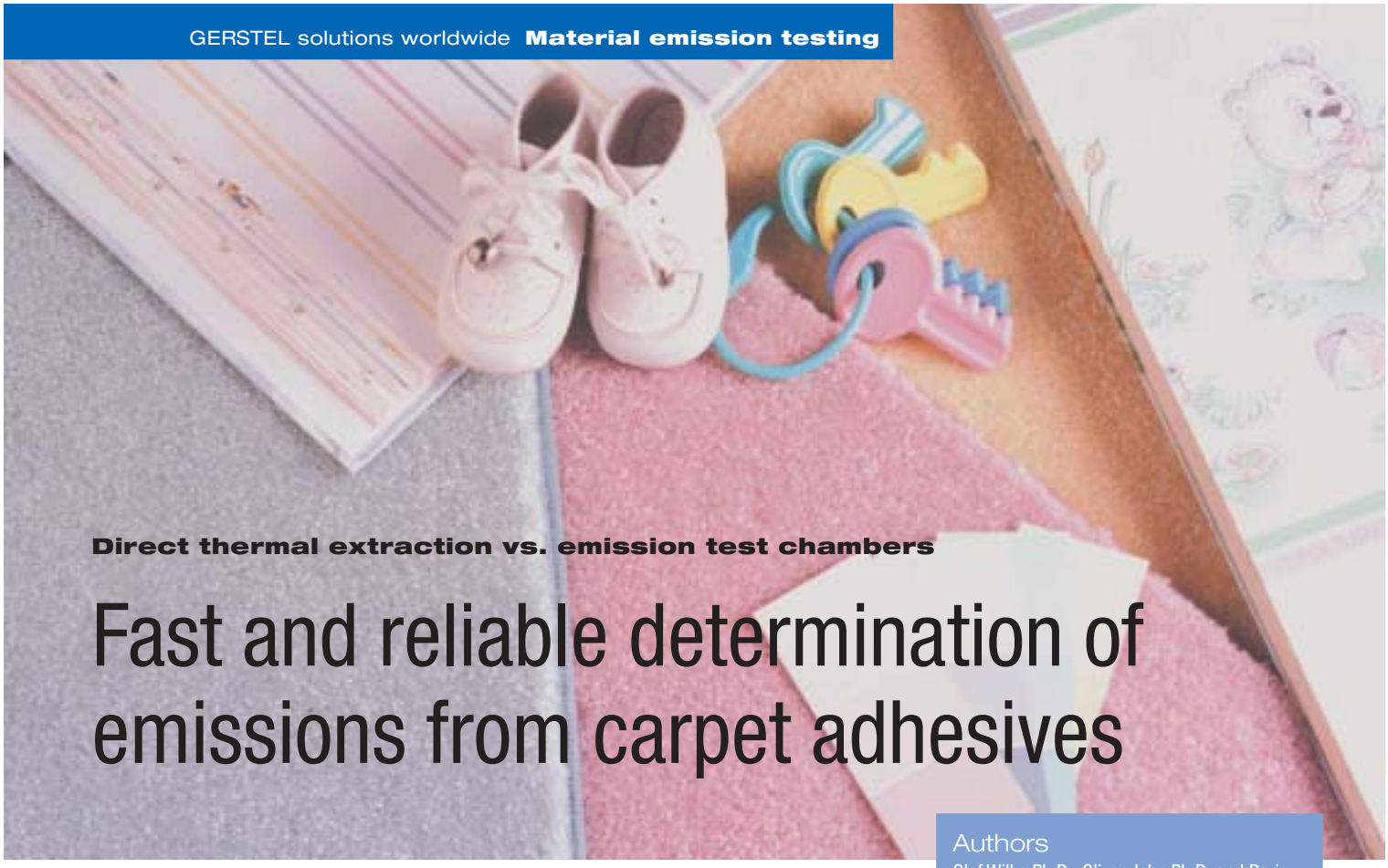

Direct thermal extraction vs. emission test chambers

Fast and reliable determination of emissions from carpet adhesives

Authors
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Upholstery, carpeting, adhesives, sealants, paints and various building materials can contain and emit volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs), which impact indoor air quality and can contribute to "sick building syndrome" (SBS).
SBS is a term used when bad indoor air quality leads to health problems or even illness among occupants. In order to test materials used inside our homes and workplaces, their VOC and SVOC emissions are determined using an emission test chamber under controlled conditions. Direct thermal extraction with the GERSTEL Thermal Desorption System (TDS) leads to comparable results, as testing of carpet adhesives shows.

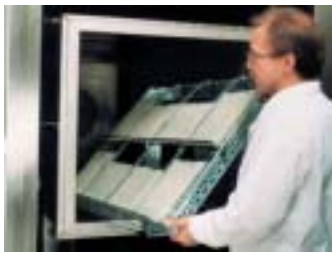


Figure 1
Loading a 1 m^3 test chamber with adhesive on glass plates

Figure 2
GERSTEL TDS 3 and TDS A with samples for direct thermal extraction

Emission testing of materials is generally performed in test chambers. An emission profile is generated over a period of 28 days under well-defined conditions. This process is time-consuming and extremely costly. The process can be simulated by direct thermal extraction: A large number of extractions are performed in a much smaller volume and over a much shorter period of time.

Thermal extraction is an extremely efficient method for determining the emission potential of a material or product, for example, in the development phase. To perform a thermal extraction determination using the GERSTEL TDS, only 5 to 20 mg sample is required. The sample is introduced into a TDS glass tube and then heated to the specified temperature in a controlled gas flow (helium) to simulate test chamber conditions. Determination of the analytes is performed by GC/MS.

Emission Test Chamber

The Emission test chambers used for analysis of the adhesives comply with the European standard EN 13419-1.

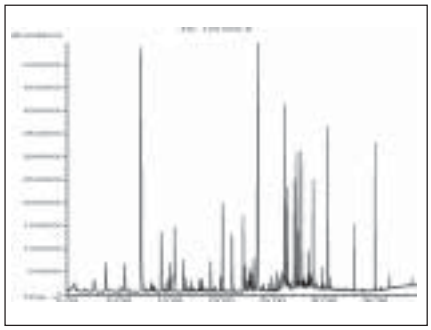
The operating temperature was kept at 23 ± 0.5°C, the relative humidity

50 ± 3 % and the area-specific air flow rate set to q = 1.25 m^3/m^2 h (loading 0.4 m^2/m^3, air exchange 0.5^{h-1}).

Chamber air sampling

Air sampling from the test chamber was performed using GERSTEL TDS glass tubes (length 178 mm, outside diameter 6 mm, inside diameter 4 mm), filled with Tenax TA (200 mg, 60/80 MESH) mounted with plugs of deactivated glass wool. Between 1 and 2 L of chamber air was sampled at a flow of 100 mL/min.

Just before the sample was drawn, the glass wool was spiked with 1 µL internal standard containing cyclodecane in methanol. For calibration, a blank tube was spiked with 1 µL of a standard mixture in methanol. After adding the internal



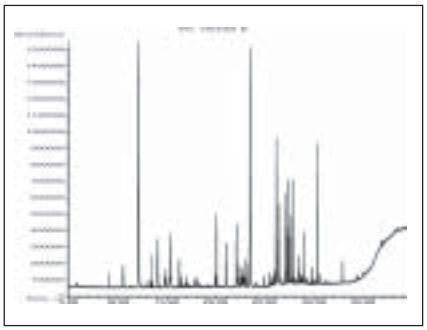


Figure 3 Chromatograms from test chamber air analysis after 24 hours (left) and from direct thermal extraction analysis after 1 h (right)

standard, 1 L of clean air from an empty emission test chamber was drawn through the tube to purge out the methanol.

Analysis of the Chamber Air

The analysis was performed by thermal desorption GC/MS using a TDS 2 coupled to a GC 5890 II Plus / MSD 5972 GC/MS system. The thermal desorption conditions were: Heating from 40°C to 280°C at 40°C/min, hold time 5 minutes at 280°C. Cryofocussing at -150°C in the GERSTEL Cooled Injection System (CIS 3). Helium desorption flow 20 mL/min. Following tube desorption, the CIS 3 was heated to 280°C at a rate of 12°C/s in splitless mode to transfer the analytes to the GC column. A Restek Rtx-200 column was used, length 30 m, film thickness 1 µm, i.d. 0.25 mm.

Direct Thermal Extraction

Using a spatula, the adhesives were applied to aluminum foil between two adhesive strips, which were used to get a defined film thickness.

After 1 hour of drying, a small strip (3 mm x 20 mm) with 5-10 mg of adhesive was cut out and transferred to an empty glass TDS tube. Thermal extraction was performed at 23°C for 12.5 minutes under a helium flow of 20 mL/min. Multiple thermal extractions were performed by repeating the extraction at the end of each GC run, enabling a rate of one extraction per hour.

Between extractions, the glass tube containing the adhesive was sealed airtight by the GERSTEL TDS A autosampler (Fig. 2). The only difference in analysis parameters from the chamber air analysis is that the TDS 2 temperature is kept constant at 23°C for thermal extraction.

Results

The chromatograms in Figure 4 show that direct thermal extraction and test chamber measurements for flooring adhesives yield comparable results. Chromatograms obtained with the two methods show the same compounds and peak pattern. Our results also show that multiple thermal extraction can simulate the long term emission behavior of a material measured in a test chamber.

Discussion

The method of direct thermal extraction was first used at higher temperatures (up to 200°C) as a screening tool for VOC and SVOC emissions from building products. The results for flooring adhesives show that keeping the temperature at 23°C, as in the chamber experiments, gives a good correlation to the chamber measurements.

The differences between test chamber measurement and direct thermal extraction using a TDS are the gas used, the different specific flow rate (q) and the mass of the applied adhesive. In the chambers, humidified air is used with q = 1.25 m^3/m^2 h and 300 g/m^2 adhesive applied. For direct thermal extraction, helium is used with q = 20 m^3/m^2 h with 100 g/m^2 adhesive applied. These parameters are compensated by using different test times: 1 hour after application for thermal extraction, 24 hours after application for chamber measurements. Using the TDS for direct thermal extraction analysis of inhomogeneous or thick materials such as floor coverings showed no such correlation to test chamber measurements. When using 4 mm i.d. tubes for thermal extraction, test conditions such as emitting surfaces and edges are too different. For inhomogeneous or thicker materials, we will test the GERSTEL Thermal Extractor TE 2 with its larger 14 mm i.d. extraction tube.

Toyota uses GERSTEL TE 2 for materials emission testing

When leading automobile manufacturers and their suppliers world-wide perform materials emission testing, they turn to a method from the German association of car manufacturers (VDA). Before a material is accepted for use in a car interior, it is tested for emission of volatile organic compounds (VOCs) as well as for emission of organic compounds in the boiling point range corresponding to the range from n-C_{16} to n-C_{32}.

The amount of higher-boiling compounds in a material is expressed in a so-called fog-value, a name derived from condensation of these compounds forming a film on the windshield (fogging).

The VDA 278 method describes the "thermal desorption analysis of organic emissions for the characterization of non-metallic materials used in automobiles".

The method is based on the use of the GERSTEL Thermal Desorption System (TDS 2) that incorporates the GERSTEL Cooled Injection System (CIS 4) as well as a twenty position autosampler.

TDS tubes, which are used for the VDA 278 method, have an internal diameter of 4 mm. Sample size is thus limited, making it difficult to analyze representative samples of foams, molded plastics and inhomogenous materials.

Toyota has now optimized the VDA 278 method for larger samples by using the GERSTEL Thermal Extractor (TE 2) with its larger inner tube for the thermal extraction step. Thanks to the larger internal diameter of its extraction tube, the GERSTEL TE 2 facilitates direct thermal extraction of a representative sample amount.

Thermal extraction is performed at temperatures that could be encountered in your car interior when parked in direct sunlight on a warm summer's day.