GLOBAL ANALYTICAL SOLUTIONS

GERSTEL

AppNote 8/2000

Volatile Organic Compounds from Adhesives and their Contribution to Indoor Air Problems

Roland F. Augustin, Heinz-Gerd Bittner, Helmut Klingenberger
Analytical Department, Deutsches Teppich-Forschungsinstitut, Charlottenburger Allee 41, D-52068 Aachen, Germany

Bernd Wiesend
Gerstel GmbH & Co. KG, Eberhard-Gerstel-Platz 1, D-45473 Mülheim an der Ruhr, Germany

Key Words
Indoor Air Pollution, Thermal Desorption, Adhesives, Floor Coverings, Emissions, Bromophenol

Abstract
Carpets for office use are nowadays in most cases applied with water-based adhesives. During the last decade the complaints about odors and emission of volatile organic compounds from these fitted carpets have increased dramatically, causing a major problem for indoor air quality. In a series of investigations it has been established that in many cases the adhesives used were the primary cause of complaints. This is initially surprising, since usually solvent-free water-based dispersion adhesives were used.

This paper describes the analytical approach of analyzing a broad variety of volatile compounds within a wide boiling point range with thermal desorption GC/MS.

INTRODUCTION

In the early 90´s, due to a German worker safety regulation (TRGS 610), solvent based adhesives for floorcoverings were changed to water-based dispersions. To realize this change in technology, instead of low boiling solvents such as methanol and toluene high boiling components such as Phenoxy ethanol, miscellaneous glycols and glycolethers were used. These components still do have the function of a solvent, but due to the solvent definition of the regulation (boiling point < 200°C), the adhesives have been declared solvent-free.

These high boiling and polar components have been identified as a major source of problems caused by glued carpets. Due to their low vapor pressure, the high boiling components diffuse only very slowly from the adhesive through the textile floorcovering, but can cause long-term indoor air pollution. Adhesives for textile floorcoverings do not only contain these high boiling components but also other components, such as terpenes or other volatile organic compounds as shown in Table I.

Table I. Volatile organic compounds from water-based dispersion adhesives.

	Compound	Source
Terpenes	Pinene Limonene Carene Longifolene Isolongifolene	Colophony resin
Glycol ethers	Butyldiglycol Butyldiglycol acetate Phenoxy ethanol Phenoxy propanol	Soft resins and solubilizer
Alcohols	2-Ethylhexanol	Emulgators and defoamer
Esters	Acryl acid esters	Acrylic resins

For the analysis of volatile organic compounds in indoor air, various sampling techniques and different adsorbing materials are in use (Figure 1). The sampling strategies depend on the boiling point of the components.

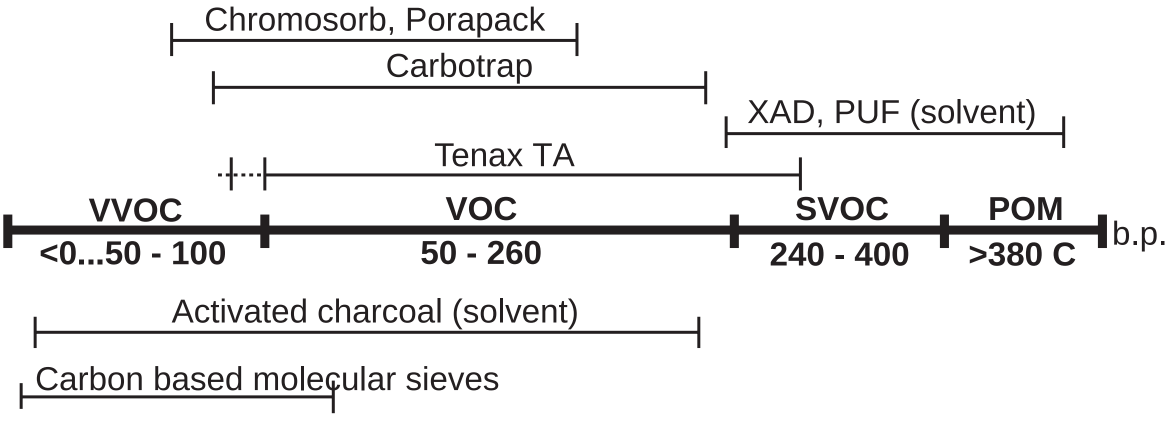

Figure 1. Boiling point range of VOC's and applicable adsorbents.

As a common adsorbent, activated charcoal tubes are regularly used for the determination of volatile organic compounds. However, this type of adsorbent is not suitable for the detection of high boiling and polar compounds, such as glycols and glycolethers found in water-based adhesives.

Table II. Comparison of different adsorbents and their suitability for different compound classes.

Adsorbents	Suitable Compounds	Unsuitable Compounds	Desorption
Activated carbon	Aromatics Aliphatics Terpenes	Glycol ethers Polars (Phenols)	Carbon bisulfide
Silicagel	Polars	Non-polars	Ethanol
Tenax TA	Compounds of a wide boiling point range with different polarities	Boiling points below 60°C	Thermal

As shown in Figure 2, the use of activated charcoal for the sampling of these components will lead to severely biased analytical results and incomplete information for the interpretation of the indoor air situation.

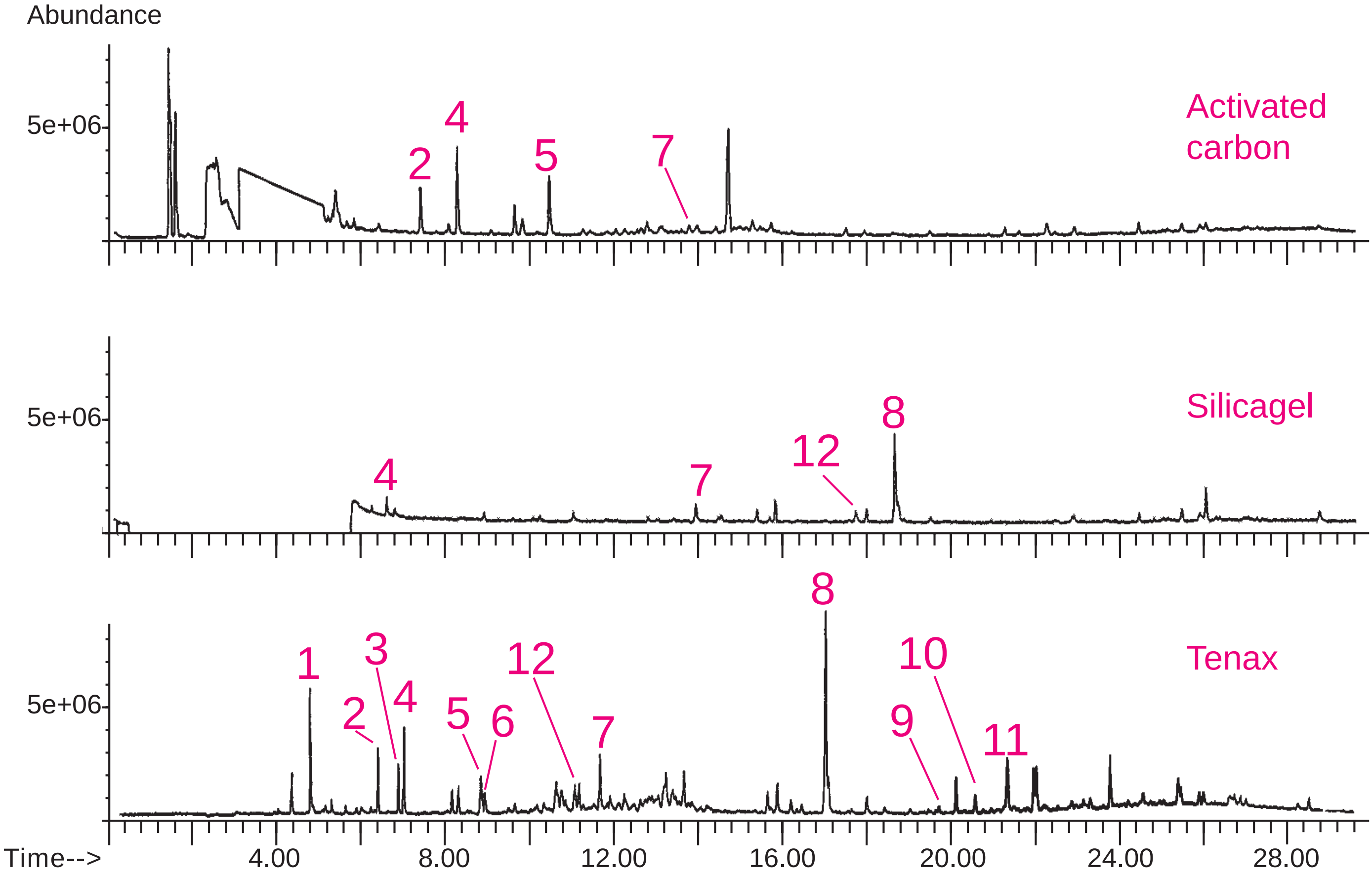

Figure 2. Comparison of different adsorbents for indoor air analysis.

Table III. List of compounds.

No.	Compound	No.	Compound
1	Butanol	7	2-Ethyl Hexanol
2	Toluene	8	Phenoxy Ethanol
3	Hexanal	9	4-Phenyl Cyclohexene
4	Butyl Acetate	10	Longifolene
5	Styrene	11	Isolongifolene
6	Butyl Diglycol	12	Butyl Diglycol Acetate

According to these data, for the determination of volatile organic components from adhesives the adsorption on Tenax TA, in combination with Thermodesorption and GC/MS analysis is nowadays state of the art (Figure 3: Gerstel Thermodesorption system). Only this technique is suitable for the analysis of a broad variety of volatile compounds with a wide range of boiling points and different polarity.

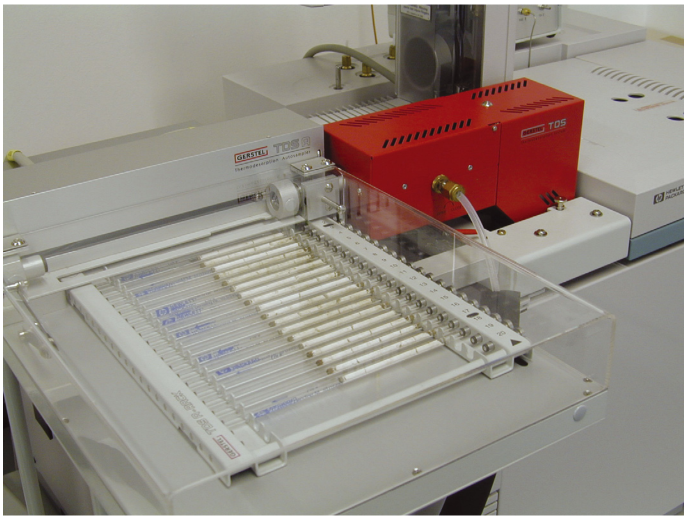

Figure 3. Gerstel TDS system with autosampler mounted on 6890 GC with 5973 MSD.

Experimental

Instrumentation. The analytical system consists of a thermodesorption system with autosampler (TDS A, TDS 2, Gerstel GmbH & Co.KG, Mülheim an der Ruhr, Germany, Figure 3), a temperature programmable vaporization inlet (CIS 4, Gerstel), a gas chromatograph (6890, Agilent Technologies, Little Falls, USA) and a mass selective detector (5973, Agilent).

Operation. The air samples are drawn on a Tenax TA tube, which is then introduced into the thermal desorption unit and thermally desorbed to release the trapped organic compounds into the cryogenically precooled PTV for subsequent GC/MS analysis.

Analysis conditions.

Table IV. Analysis conditions.

Tube	Tenax TA, 60/80 mesh, 160 mg
Column	30 m HP VOC (Agilent), di = 0.2 mm, df = 1.1 µm
Pneumatics	He, Pi = 170 kPa, constant pressure TDS-desorption flow = 50 ml/min (splitless) PTV-splitflow = 50 ml/min
TDS	10°C (2 min), 30°C/min, 250°C, 40°C/min, 300°C (6 min)
PTV	-150°C (1 min), 8°C/s, 250°C, 10°C/s, 320°C (6 min)
Oven	35°C (2 min), 25°C/min, 70°C, 6°C/min, 150°C, 10°C/min, 280°C (20 min)
Detector	MSD, 230°C / 150°C, Scan 34-450 amu

Results and Discussion

As shown above, these high boiling and polar components are not only difficult to detect, but can also influence the ambient indoor air to a great extent. Compared to low boiling solvents, these chemicals tend to migrate slowly out of the adhesive into the textile floorcovering and will furthermore lead to ongoing emissions of the material. Figure 4 shows a chromatogram of an air sample taken in a test chamber above a pure adhesive in comparison to one of a fitted carpet containing the same adhesive after 7 days (Figure 5).

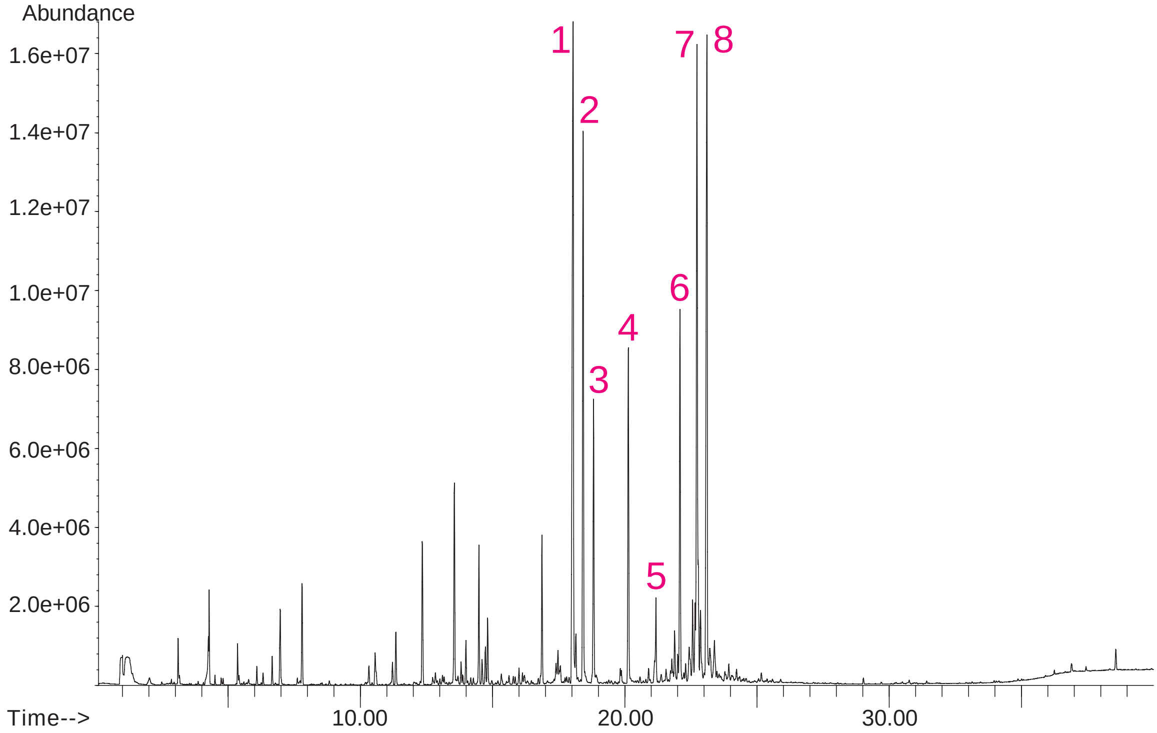

Figure 4. Adhesive after 7 days.

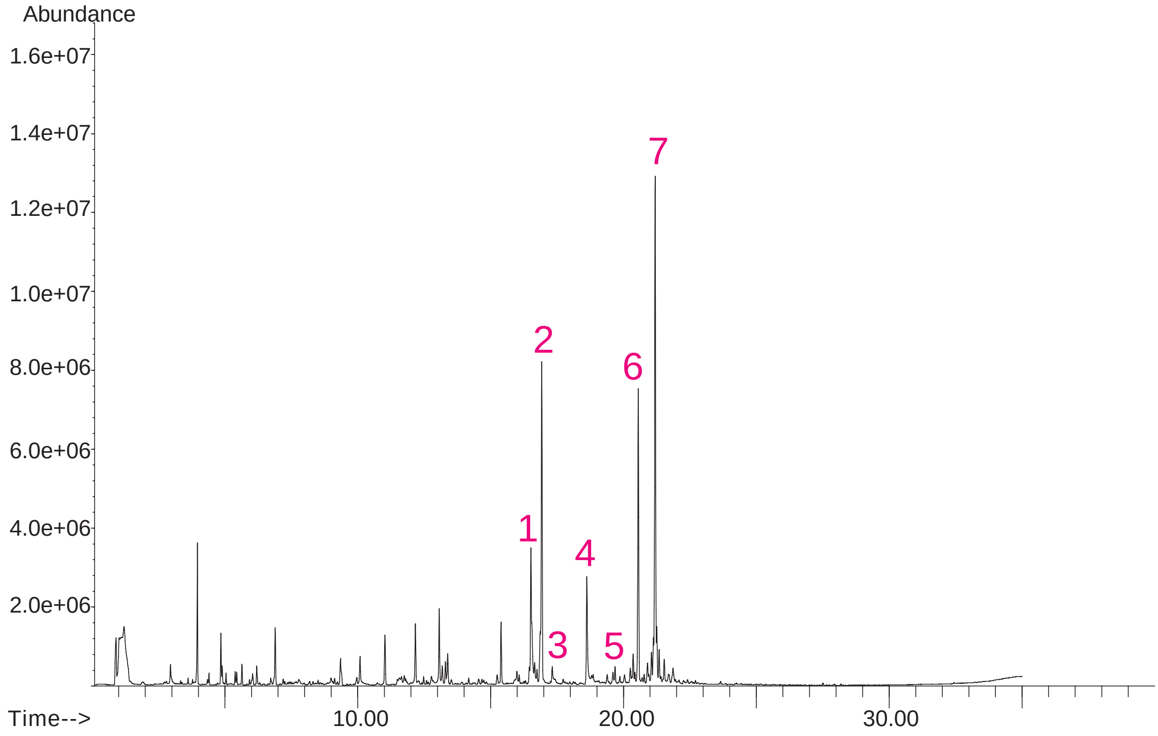

Figure 5. Adhesively fitted carpet after 7 days.

Table V. List of compounds.

No.	Compound	No.	Compound
1	Butyl Diglycol	5	Butyl Diglycol Acetate
2	Methoxypropenyl Benzene	6	Terpene
3	Phenoxy Ethanol	7	Longifolene
4	Dimethylbicycloheptene Ethanol	8	Butyl Tryglycol

Another experiment is shown in Figure 6. For the determination of long-term emissions from adhesives (prediction for real rooms), a chamber measurement has been performed over 100 days. A carpet, glued on a glass plate was measured for this long period of time in a test chamber (Figure 7) to obtain more information concerning the long-term emission behavior of glycols and glycol ethers.

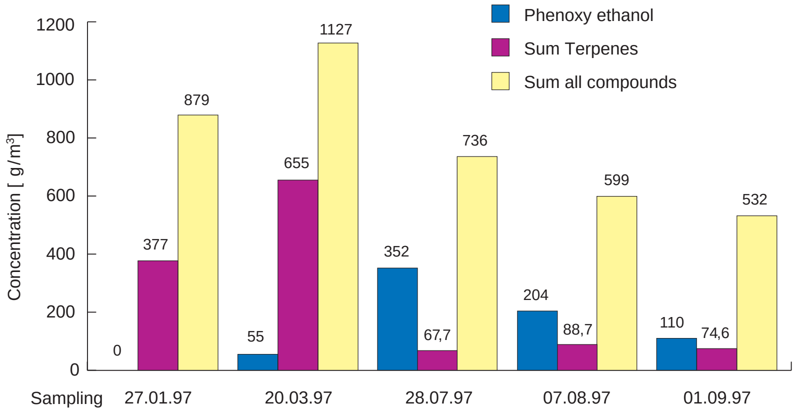

Figure 8. Real room situation in a newly constructed office building.

An actual situation similar to the test chamber model, was observed after the new construction of the TFI-building. Instead of vaporizing within the first days after installation of a new carpet, one of the adhesives used, containing these high boiling compounds, has lead to a long lasting emission of volatile components into the indoor air. As shown in Figure 8 in a real room situation these high-boiling components, such as phenoxy ethanol, do not appear immediately, but instead after a period of time after installation. In this particular case, the office had to be renovated after nine months due to the complaints of the users and according to the emission data.

Another example shows that air analysis alone does not necessarily solve indoor air odor problems. In this case an extremely annoying bad smell was reported in an office room. Indoor air analysis resulted in the detection of bromophenol (Figure 9), but neither the floor covering nor the adhesive contained even traces of this compound. The combination of carpet and adhesive led to the formation of bromophenol and placing a piece of carpet (with the adhesive applied) in the thermal desorption unit and performing thermal extraction could reproduce the bad smell.

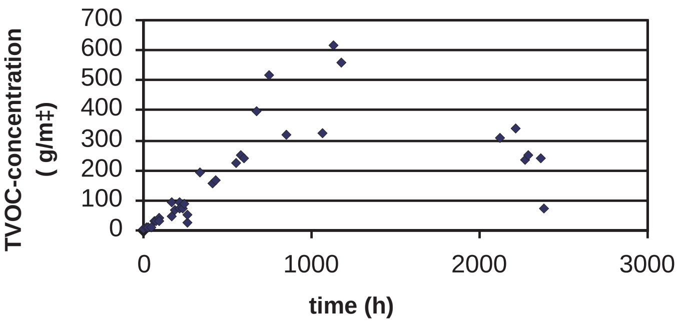

Figure 6. Long-term emission (TVOC) of a glued carpet in a test chamber.

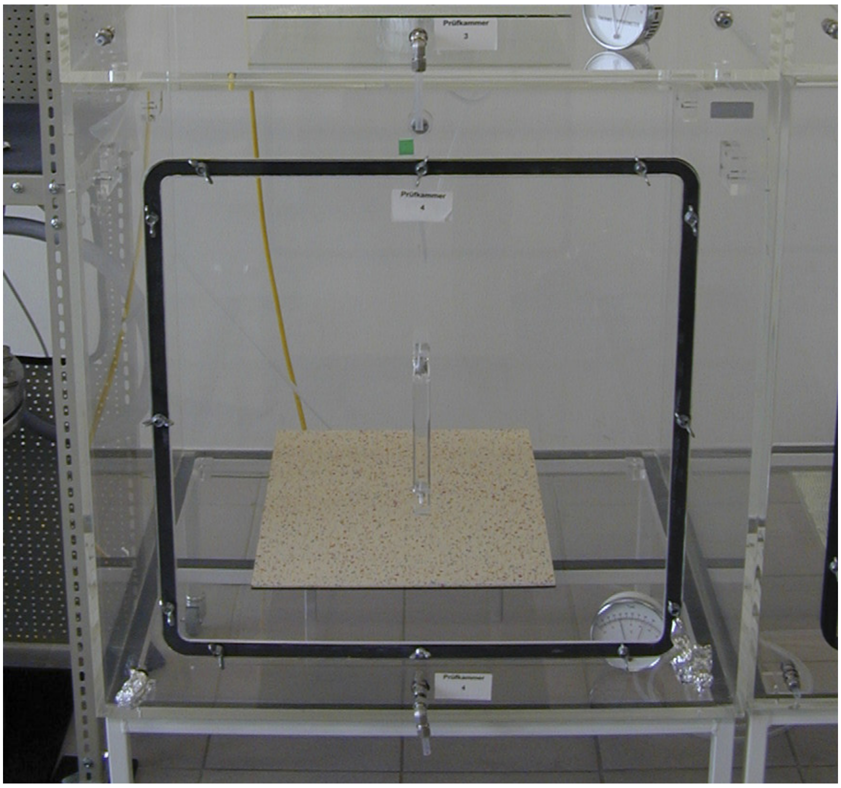

Figure 7. Test chamber.

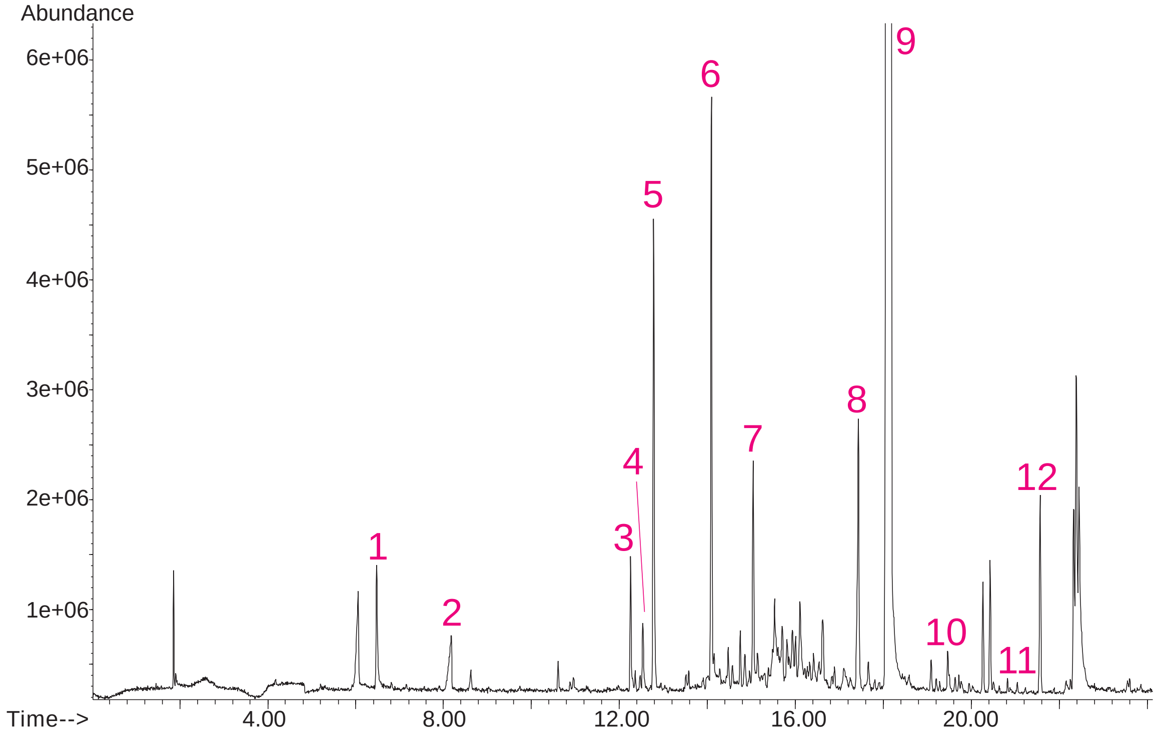

Figure 9. Direct thermal extraction of carpet material and an adhesive.

Table VI. List of compounds.

No.	Compound	No.	Compound
1	Butanol	7	Bromophenol
2	Methyl Propionic Acid	8	Phenoxy Propanone
3	Butylisopropylene Glycol	9	Phenoxy Propanol
4	Benzaldehyde	10	4-Phenyl Cyclohexene
5	Phenol	11	Isolongifolene
6	2-Ethyl Hexanol	12	Longifolene

Figure 10 shows the mechanism of formation: phenoxy propanol (from the adhesive) is hydrolyzed to phenol, which itself reacts with inorganic bromide (from the latex back of the textile covering) forming bromophenol.

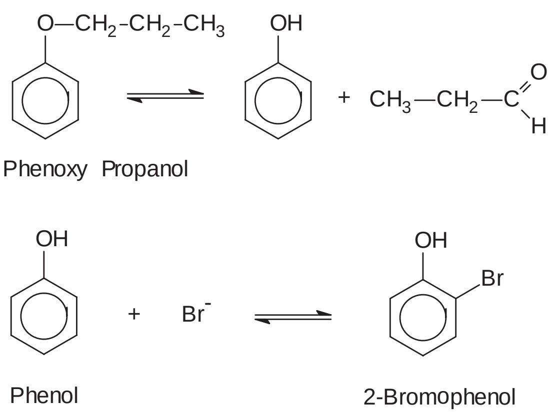

Figure 10. Mechanism of formation of bromophenol.

Conclusions

Volatile organic components from water-based adhesives have a major influence on the indoor air quality. Due to the use of high-boiling and polar compounds, the impact of the problem has been shifted from the installation process to the consumer or inhabitant of the office. The emissions of these compounds are a major problem of indoor air pollution. As shown in this paper, the influence of adhesive components on the long-term emission is substantial and by using the wrong analytical technique the true magnitude of the problem for the indoor air situation can be severly underestimated.

After having learned about the situation, a new testing scheme for the long-term emission of adhesives was developed by the association of adhesive manufacturers and the association of environmentally friendly carpets.

GERSTEL GmbH & Co. KG
Eberhard-Gerstel-Platz 1
45473 Mülheim an der Ruhr
Germany
+49 (0) 208 - 7 65 03-0
+49 (0) 208 - 7 65 03 33
gerstel@gerstel.com
www.gerstel.com

GERSTEL Worldwide

GERSTEL, Inc.
701 Digital Drive, Suite J
Linthicum, MD 21090
USA
+1 (410) 247 5885
+1 (410) 247 5887
sales@gerstelus.com
www.gerstelus.com

GERSTEL AG
Wassergrabe 27
CH-6210 Sursee
Switzerland
+41 (41) 9 21 97 23
+41 (41) 9 21 97 25
swiss@ch.gerstel.com
www.gerstel.ch

GERSTEL K.K.
1-3-1 Nakane, Meguro-ku
Tokyo 152-0031
SMBC Toritsudai Ekimae Bldg 4F
Japan
+81 3 5731 5321
+81 3 5731 5322
info@gerstel.co.jp
www.gerstel.co.jp

GERSTEL LLP
10 Science Park Road
#02-18 The Alpha
Singapore 117684
+65 6779 0933
+65 6779 0938
SEA@gerstel.com
www.gerstel.com

GERSTEL (Shanghai) Co. Ltd
Room 206, 2F, Bldg.56
No.1000, Jinhai Road,
Pudong District
Shanghai 201206
+86 21 50 93 30 57
china@gerstel.com
www.gerstel.cn

GERSTEL Brasil
Av. Pascoal da Rocha Falcão, 367
04785-000 São Paulo - SP Brasil
+55 (11)5665-8931
+55 (11)5666-9084
gerstel-brasil@gerstel.com
www.gerstel.com.br