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Direct Thermal Desorption GC/MS Analysis of Automotive Air Charcoal Filters

Andreas Hoffmann
*Gerstel GmbH & Co.KG, Eberhard-Gerstel-Platz 1,
D-45473 Mülheim an der Ruhr, Germany*

Jost Kames
Blücher GmbH, Parkstrasse 10, D-40675 Erkrath, Germany

Jürgen Steinhanes
*Fraunhofer-Institut für Umweltchemie und Ökotoxikologie, Auf dem
Aberg 1, D-57392 Schmallenberg, Germany*

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System CIS

ABSTRACT

The analysis of volatile organic contaminants in air trapped on an automotive air charcoal filter is usually carried out using standard liquid extraction followed by GC/MS analysis of the extract.

This standard technique will be compared to direct thermal desorption of the filter material in combination with cryo-focusing in the liner of a cooled injection system, followed by temperature programmed sample transfer to the analytical column.

It will be shown that direct thermal desorption is a reliable and fast method for the determination of volatiles in automotive air charcoal filters without requiring any sample pretreatment.

INTRODUCTION

The so called "individual traffic" has gained more and more popularity over the last years. Statistically nearly one European out of two drives a car. This habit also causes problems such as air pollution which mainly affects those who cause it: the car drivers themselves. Although the increased usage of three-way catalytic converters has reduced total emissions the interior of cars need to be protected by highly effective filter systems to lower the cars indoor air contamination, especially in driving situations like parking houses, tunnels or traffic jams.

Such a multi-layer automotive air charcoal filter was analysed layer by layer. The results are discussed in this paper. The filter examined was taken from a car with a total milage of 120,000 km. However be aware that the filter has been used only sometimes as it is activated by the driver via a switch electronic.

EXPERIMENTAL

Filter construction. The filter consists of 7 layers of BLÜCHER NANOSORB™ high density filter foam (Figure 1) of 20 pores per inch porosity loaded with spherical activated carbon of <0.6 mm diameter. The filter foam may be described as expanded fixed bed filters which are known to have an ideal adsorption characteristic because of the even distribution of small adsorber particles [1]. Polluted air has to pass this filter system, volatile contaminants are adsorbed on the spherical carbon granula (Figure 2).

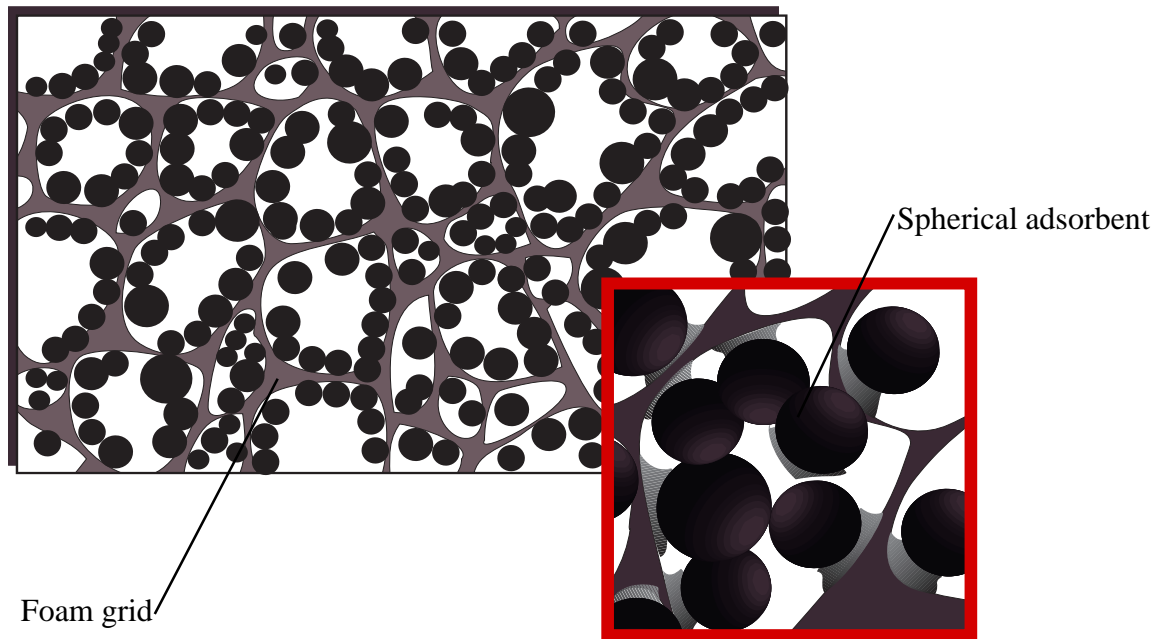


Figure 1. Construction of the open-porous NANOSORB™ filter.

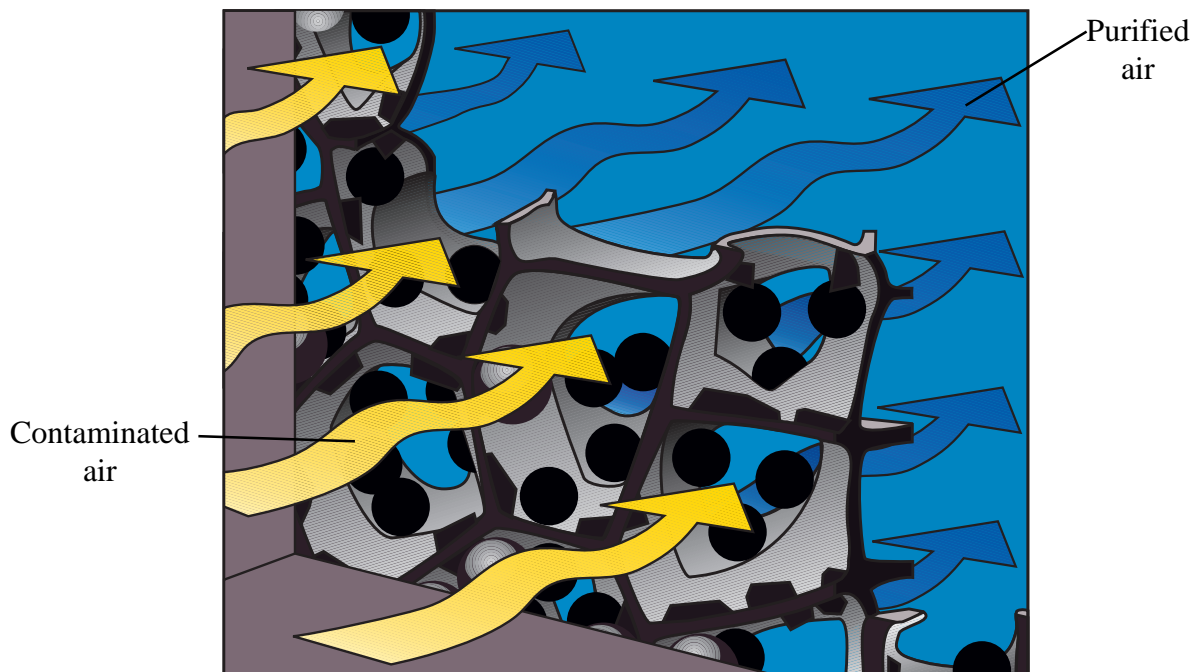


Figure 2. Schematic of the filtration process.

Liquid extraction: Sample preparation. A piece of every filter layer was pierced out, weighted and extracted with 10 ml of CS₂ for 20 hours, then filtered and injected (1 µl).

Instrumentation. The system used for qualitative analysis consists of an autosampler HP 7673, a gas chromatograph HP 5890 series II with split/splitless inlet and a HP 5970 B mass selective detector (all Hewlett-Packard, Waldbronn, Germany).

The system used for quantitative analysis consists of an autosampler HP 7673, a gas chromatograph HP 5890 series II with split/splitless inlet and an HP 5921 atomic emission detector (all Hewlett-Packard, Waldbronn, Germany).

Operation. The filtered extract is injected splitless and analysed.

Analysis conditions.

Column:	60 m HP-624 (Hewlett-Packard),	d _i = 0.32 mm,	d _r = 1.8 µm
Pneumatics:	Carriergas He,	constant flow mode (1 ml/min),	split 1:30, 1 min splitless
Temperatures:	Split/splitless injector	250°C	
	Oven	40°C (1 min); with 3°C/min to 150°C; with 10°C/min to 250°C (20 min)	
MSD:	Scan, 33-350 amu		
AED:	wave length C 193 and C 496 nm		

Thermal desorption: Sample preparation. A piece of every filter layer was pierced out, weighted and inserted into a blank TDS tube.

Instrumentation. The system consists of a thermodesorption system (TDS, Gerstel GmbH, Mülheim an der Ruhr, Germany, **Figure 3**), a temperature programmable cooled injection system (CIS 3, Gerstel GmbH, Mülheim an der Ruhr, Germany, **Figure 3**), a gas chromatograph HP 5890 series II, and a mass selective detector HP 5972 (both Hewlett-Packard, Waldbronn, Germany).

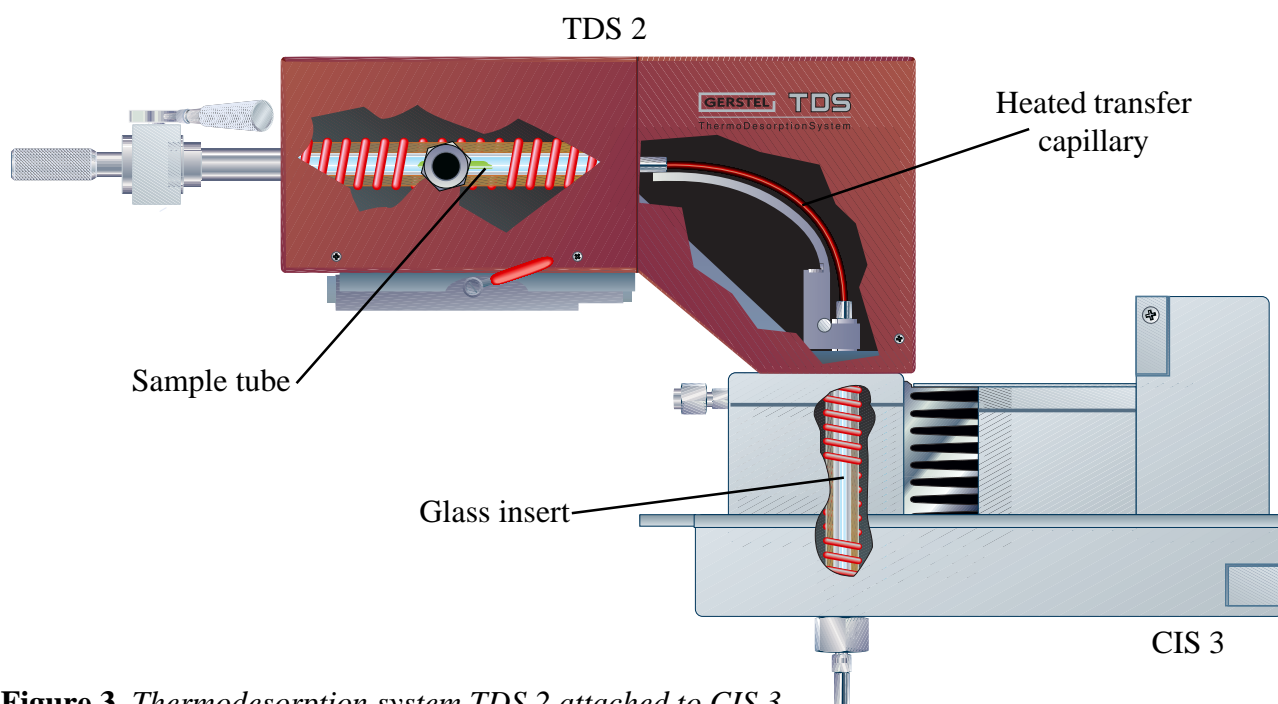


Figure 3. Thermodesorption system TDS 2 attached to CIS 3.

Operation. A blank glass tube is filled with the sample and then inserted into the TDS desorption chamber which is cooled down to ambient temperatures in order to prevent premature desorption. After purging the air out of the system, the tube is then heated to the 300°C, while the carrier gas flowing through the tube transfers the volatiles in splitless-mode (**Figure 4**) into the pre-cooled CIS, where they are cryofocused and concentrated.

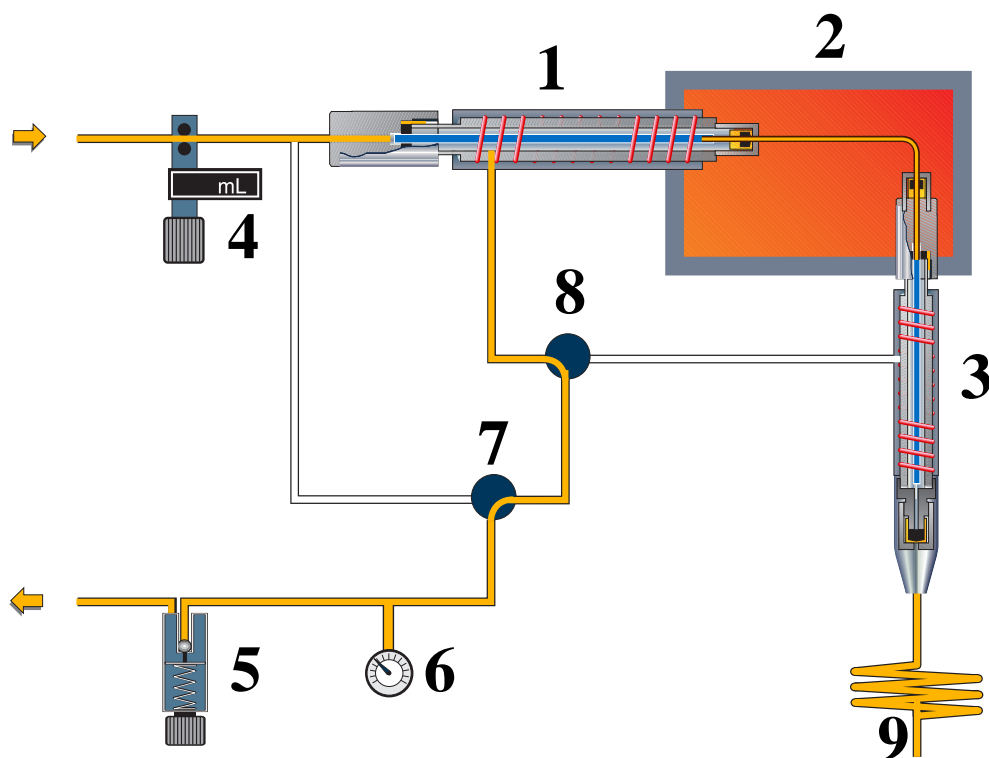


Figure 4. Schematic of the applied system which consists of a thermodesorption system (1), a temperature controlled transfer capillary (2), a cooled injection system (3), standard backpressure pneumatics with mass-flow controller (4), backpressure regulator (5), pressure gauge (6) and split/splitless valve (7), including an additional 3/2-way solenoid (8) to switch the splitflow between TDS and CIS. The analytical column (9) is directly connected to a mass selective detector.

After the desorption has finished the CIS is heated to 300°C to allow split- or splitless transfer of the trapped compounds to the analytical column and further mass spectrometric detection.

Analysis conditions.

Column:	60 m DB 5 (J&W)	$d_i = 0,25 \text{ mm}$	$d_f = 0,25 \text{ }\mu\text{m}$
Pneumatics:	Carriergas He	$p_i = 120 \text{ kPa}$	split x:100
Temperatures:	TDS	20°C; with 60°C/min to 300°C (5 min)	
	CIS	-150°C; with 12°C/s to 300°C (3 min)	
	Oven	50°C (1 min); with 5°C/min to 100°C; with 10°C/min to 300°C (10 min)	
	MSD	300°C	
MSD:	Scan, 35-350 amu		

RESULTS AND DISCUSSION

Figures 5 and 6 show total ion chromatograms of the first (Figure 5) and the last (Figure 6) filter layer.

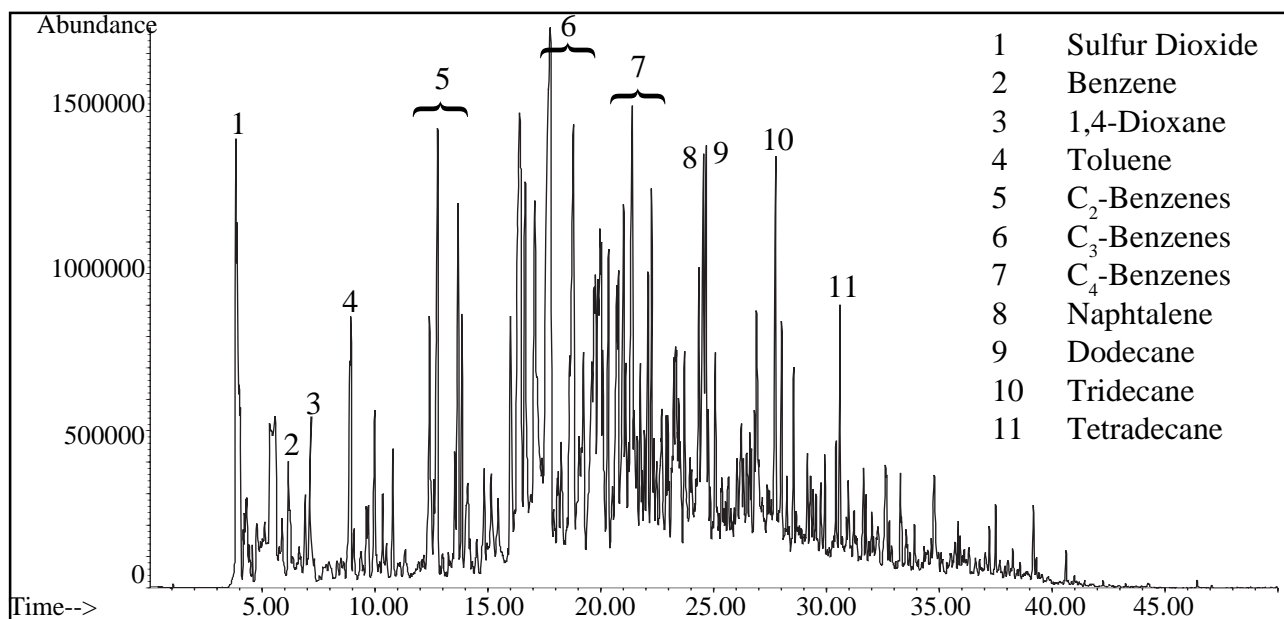


Figure 5. Total ion chromatogram of the first filter layer.

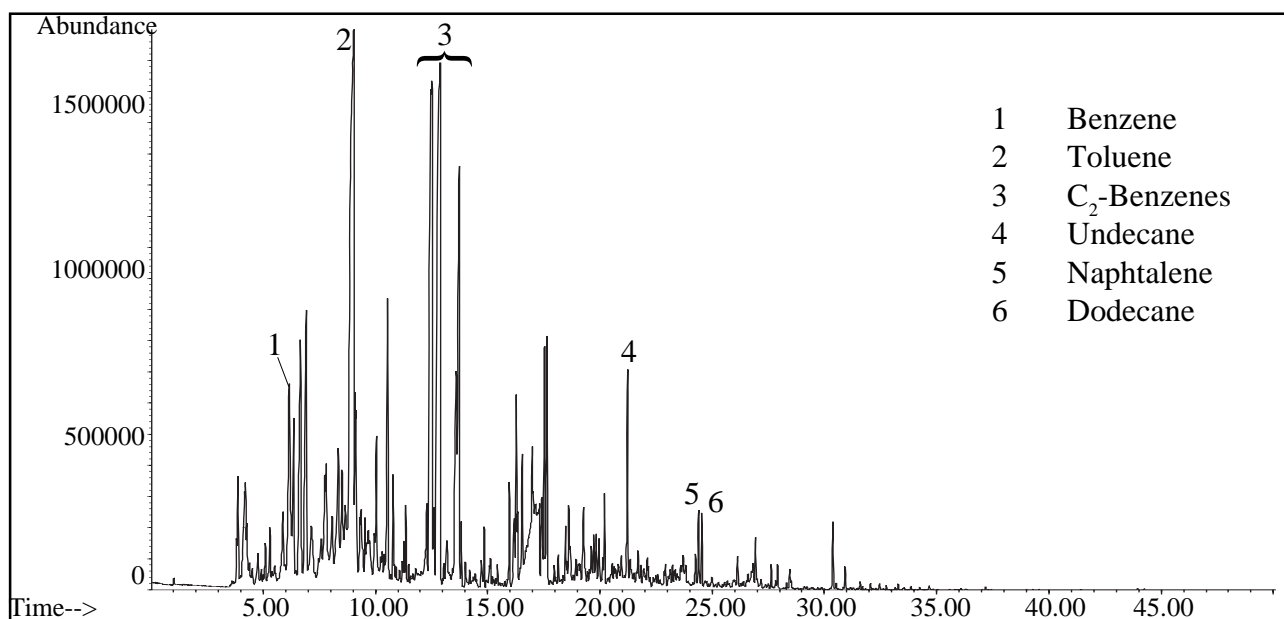


Figure 6. Total ion chromatogram of the last filter layer.

The compounds found can be directly related to car exhaust, especially aromatics are detected in high concentrations. In addition they show a very high affinity to the charcaol used in the filter material. Obiously (and expected) the highest concentration of organic volatiles is found in the first layer of the filter. Figure 7 shows the percental distribution of organic volatiles in the different filter layers.

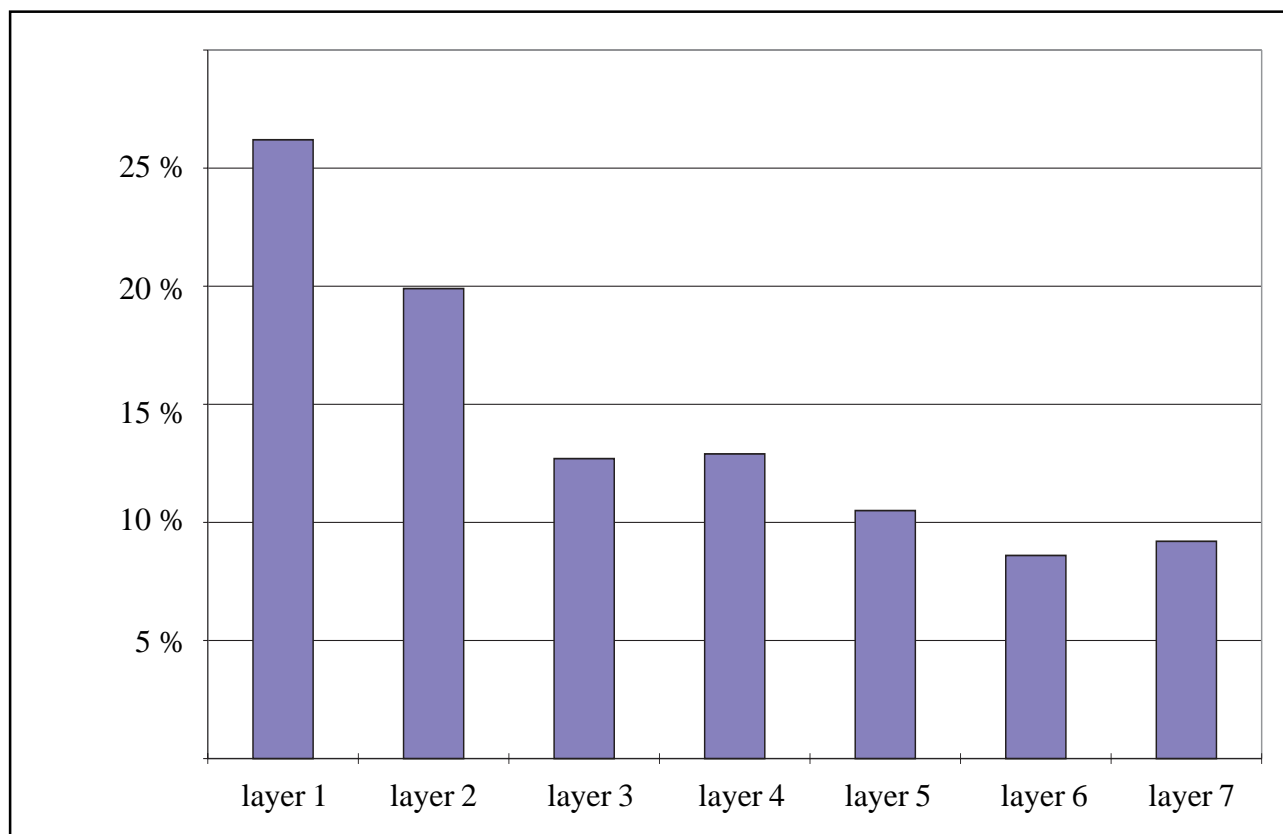


Figure 7. *Percental distribution of organic volatiles in the different filter layers.*

To monitor the filter efficiency in regard of different volatilities of compounds seven target compounds were chosen (Benzene, heptane, toluene, ethyl benzene, propyl benzene, dodecane and tetradecane). Their percental distribution per single layer is shown in **Table I** and **Figure 8**.

	layer 1 (%)	layer 2 (%)	layer 3 (%)	layer 4 (%)	layer 5 (%)	layer 6 (%)	layer 7 (%)
Benzene	4.9	3.9	3.3	2.9	2.1	2.3	3.1
Heptane	5.3	4.6	5.4	5.0	8.2	10.4	11.9
Toluene	20.4	24.1	32.7	33.1	39.2	47.8	49.4
Ethyl Benzene	13.6	25.8	27.7	43.1	45.7	37.5	32.1
Propyl Benzene	20.3	29.3	24.9	15.2	4.8	2.0	2.4
Dodecane	24.0	10.0	5.0	0.1	0.0	0.0	0.1
Tetradecane	11.5	2.3	1.0	0.0	0.0	0.0	0.0

Table I. *Percental distribution of target compounds in the different filter layers.*

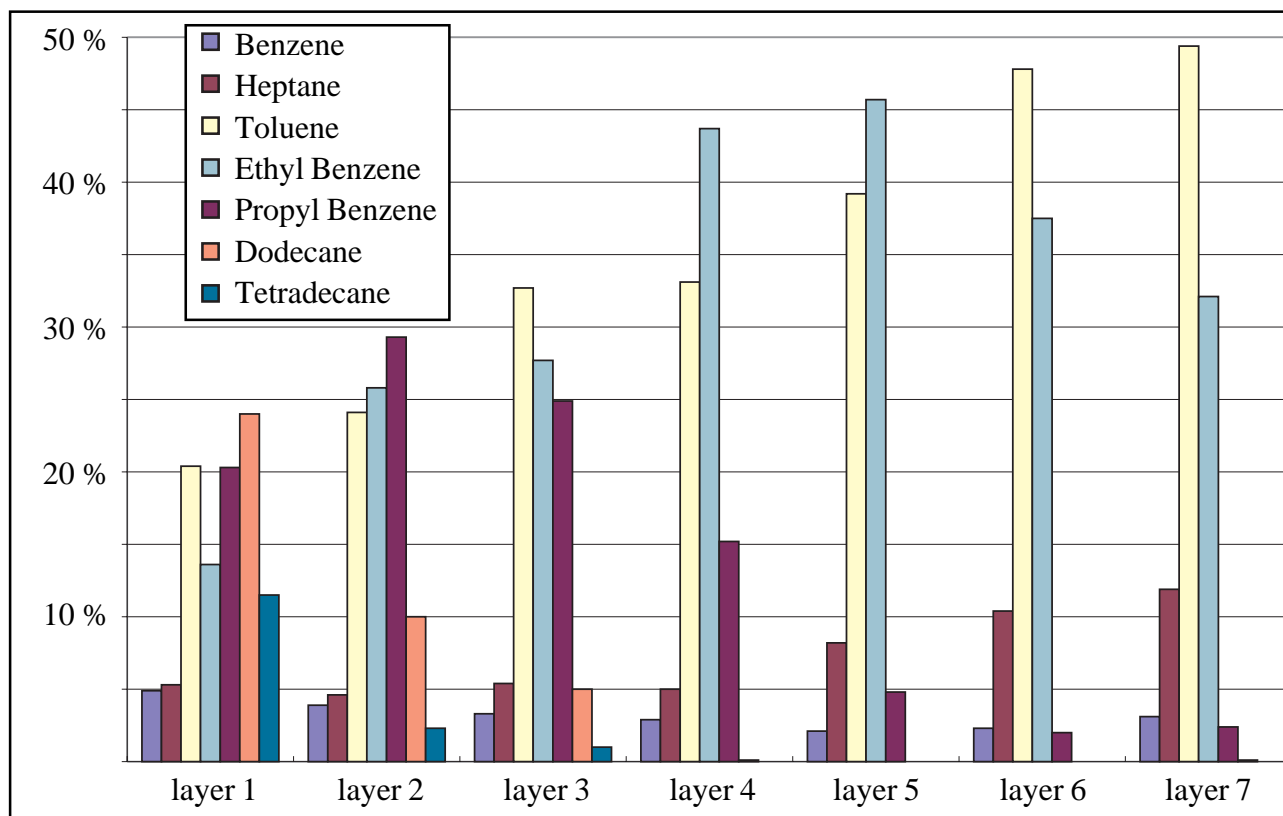


Figure 8. *Percental distribution of target compounds in the different filter layers.*

The results show that the low volatile compounds (benzene, heptane, toluene) have broken through, whereas the maximum concentrations of higher boiling compounds found in some of the mediate layers (propyl benzene, dodecane, tetradecane) indicate that the filter was able to retain them.

CONCLUSION

Thermal desorption offers a technique for the analysis of organic volatiles in automotive air charcoal filter materials with neither requiring any sample preparation or pretreatment nor sacrificing sensitivity or precision.

The results obtained show that some compounds, especially low volatiles, have already broken through the different filter layers although the charcoal filter material usually has excellent adsorption capabilities. The filter was used in a car with a total mileage of 120,000 km and this seems to be the reason for these results. Regarding healthcare and based on these results car manufacturers should think about changing the filters in regular intervals.



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
- gerstelag@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

Level 25, North Tower
One Raffles Quay
Singapore 048583

- +65 6622 5486
- +65 6622 5999
- SEA@gerstel.com
- www.gerstel.com

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



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