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Comparison of Standard Liquid Extraction and Direct Thermal Desorption GC/MS Techniques for the Analysis of Charcoal Filters used for Indoor Air Purification in a PCB Contaminated Building

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Abstract

Many buildings constructed from prefabricated elements are widely contaminated with PCB originated in e.g. elastic sealants which act as permanent sources for several years. Indoor air filtration is part of a restoration strategy for these buildings using activated carbon filter media [1].

The analysis of PCBs trapped on these filters 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 PCBs in air charcoal filters without requiring any sample pretreatment.

Introduction

Indoor air pollution by polyhalogenated organic compounds often leads to severe health problems. Prolonged exposure to those substances at the ppt-level is regarded as the origin of an increased suscepitibility to allergic reactions, nervous deseases and even cancer [2].

PCBs have been used in various applications with direct access to indoor air, e.g as flame retardants or as additives to permanently elastic sealants or cable shielding. Due to their low vapor pressure PCBs outgas fairly slowly thus creating permanent low-concentration sources in indoor environments. Since these compounds show high affinity for painted walls and ceilings the contamination becomes widespread with time and, as a consequence, difficult to control.

The reduction of PCB levels to meet the targeted health standard of less than 300 ng m^{-3} [3] demands for stepwise restoration strategies, one of them being indoor air filtration. Indoor air is drawn through a recirculation filter system at a volume flow rate of 1,600 m^3 h^{-1}. Efficiency of the filtration process can be monitored by analysing filter particles from different layers of the filter system using thermal desorption GC/MS techniques to determine the PCB content of each layer.

EXPERIMENTAL

Filter construction. The first two stages consist of particle filters of F 6 (acc. to EN 779) and HEPA class respectively. The last filter stage consists of 5 layers of BLÜCHER NANOSORB™ high density filter foam (**Figure 1**) of 30 pores per inch porosity loaded with spherical activated carbon of <0.35 mm diameter. The filter foam may be described as expanded fixed bed filters which are known to have an ideal adsorbtion 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**).

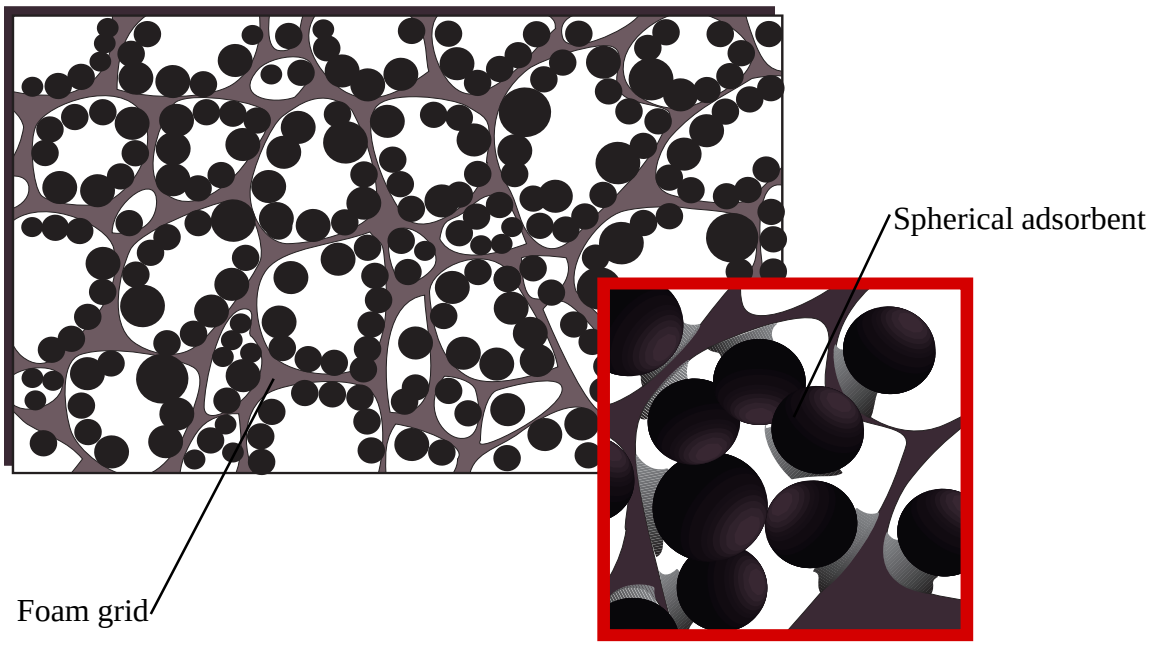

Figure 1. *Construction of the open-porous NANOSORB™ filter.*

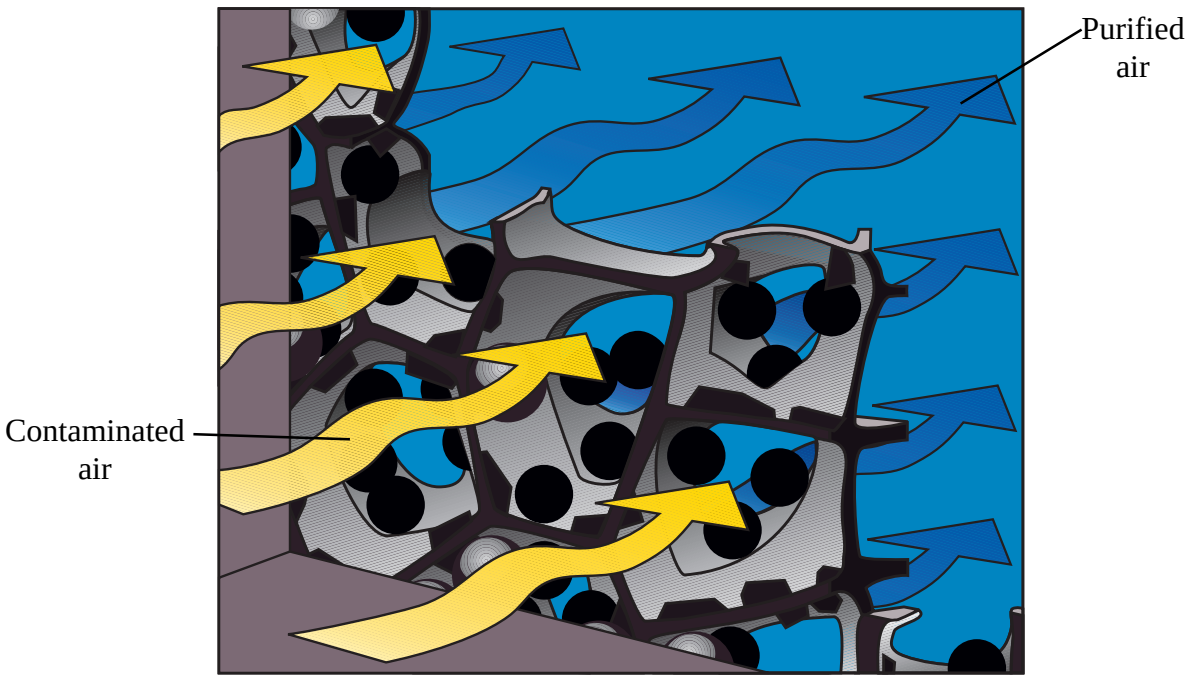

Figure 2. *Schematic of the filtration process.*

Liquid extraction: Sample preparation. A piece of every filter layer was pierced out, weighted and hot extracted with toluene, then reduced to 10-100 ml and injected (2 µl).

Instrumentation. The system consists of a gas chromatograph HP 5890 series II with split/splitless inlet and a HP 5972 mass selective detector (all Hewlett-Packard, Waldbronn, Germany).

Operation. The filtered extract is injected splitless and analysed.

Analysis conditions.

Precolumn:	1 m,	d_i = 0.25 mm
Column:	30 m DB 5 (J&W),	d_i = 0.25 mm, d_f = 0.25 µm
Pneumatics:	Carriergas He,	p_i = 70 kPa,split 1:25, 1 min splitless
Temperatures:	Split/splitless injector	260°C
	Oven	80°C (1 min); with 10°C/min to 280°C (14 min)
	MSD	280°C
MSD:	SIM	

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 6890, and a mass selective detector HP 5972 (both Hewlett-Packard, Waldbronn, Germany).

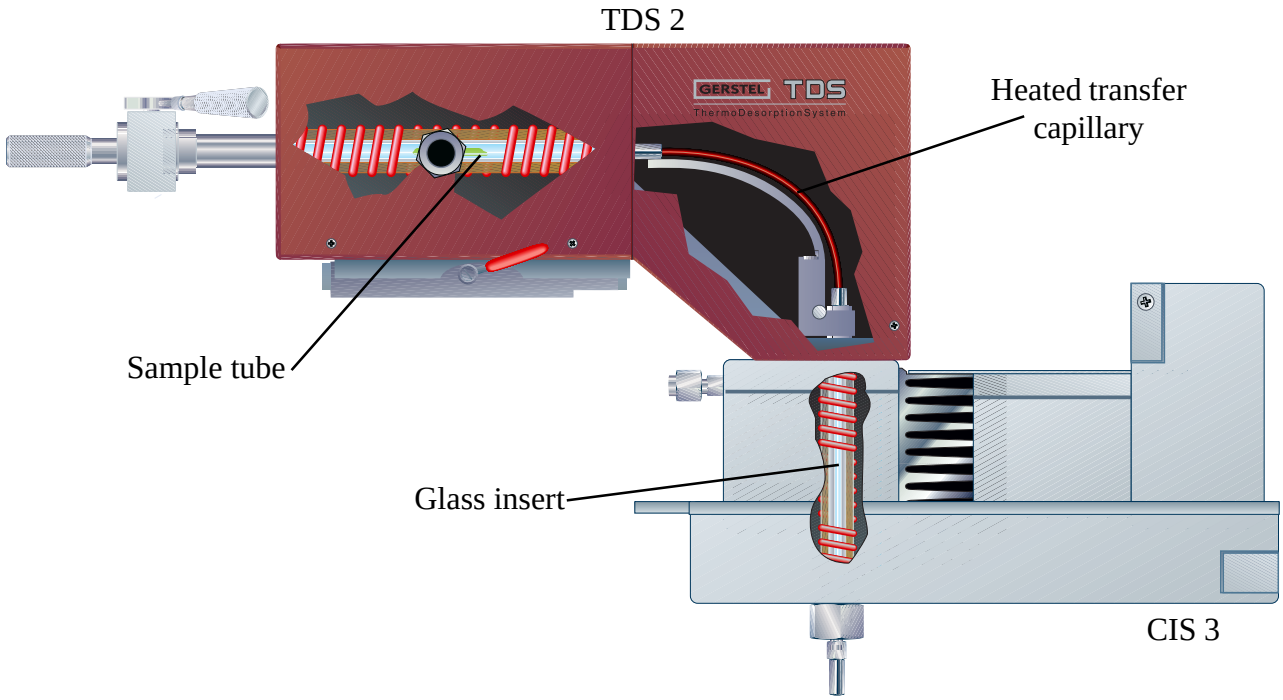

Figure 3. *Thermodesorption system TDS 2 attached to CIS 3.*

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 350°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.
After the desorption has finished the CIS is heated to 350°C to allow split- or splitless transfer of the trapped compounds to the analytical column and further mass spectrometric detection.

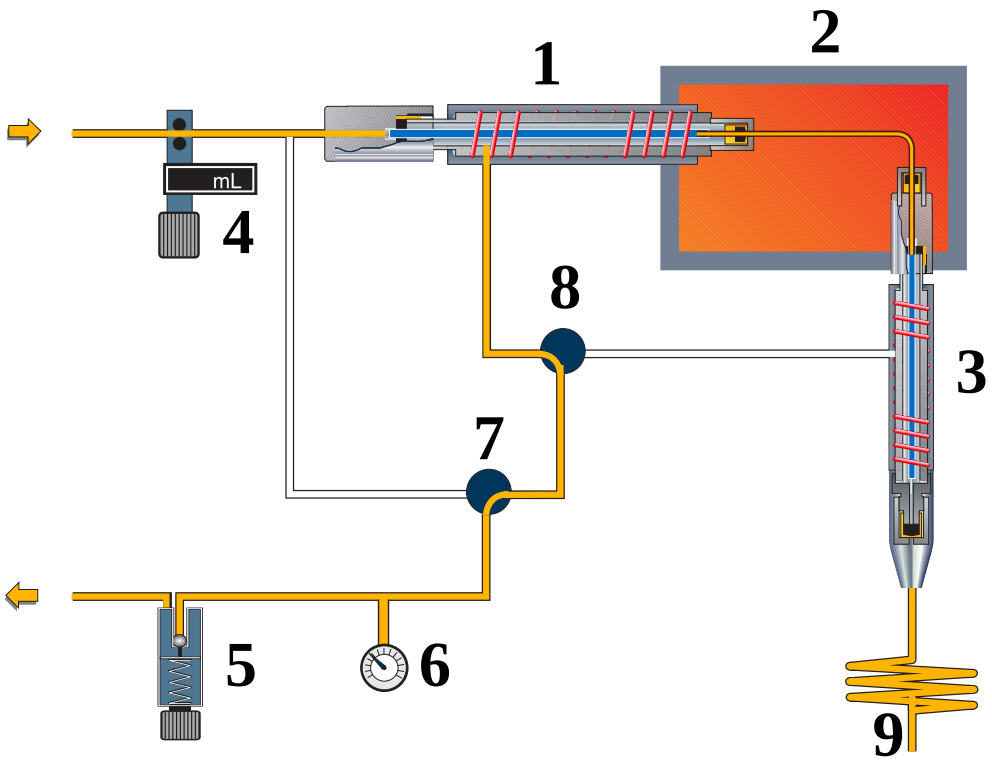

Figure 4. *Schematic of the applied system which consists of a thermodesorption system (1), a temperature controlled transfer capillary (2), a cooled injection system (3), standard backpressure pneumatics with mass-flow controller (4), backpressure regulator (5), pressure gauge (6) and split/ splitless 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.*

Analysis conditions.

Column:	60 m DB 5 (J&W),	d_i = 0,25 mm, d_f = 0,25 µm
Pneumatics:	Carriergas He,	constant flow mode (1 ml/min), split 1:50
		1 min splitless
Temperatures:	TDS	20°C; with 60°C/min to 350°C (10 min)
	CIS	-50°C; with 12°C/s to 350°C (3 min)
	Oven	60°C (1 min); with 10°C/min to 300°C (10 min)
	MSD	280°C
MSD:	SIM	

Results and Discussion

Recovery rates. The charcoal used in the filter material has a very high adsorption capability for PCBs (this is the reason why it is used as a filter media). The recovery rate for thermal desorption was obtained by comparison between liquid injection of a clophen-standard and thermal desorption of filter material spiked with the same standard. As a result a recovery rate of approx. 25% could be determined, which seems to be in the same or an even better range than the recovery rate for liquid extraction (see **Figure 9**).

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

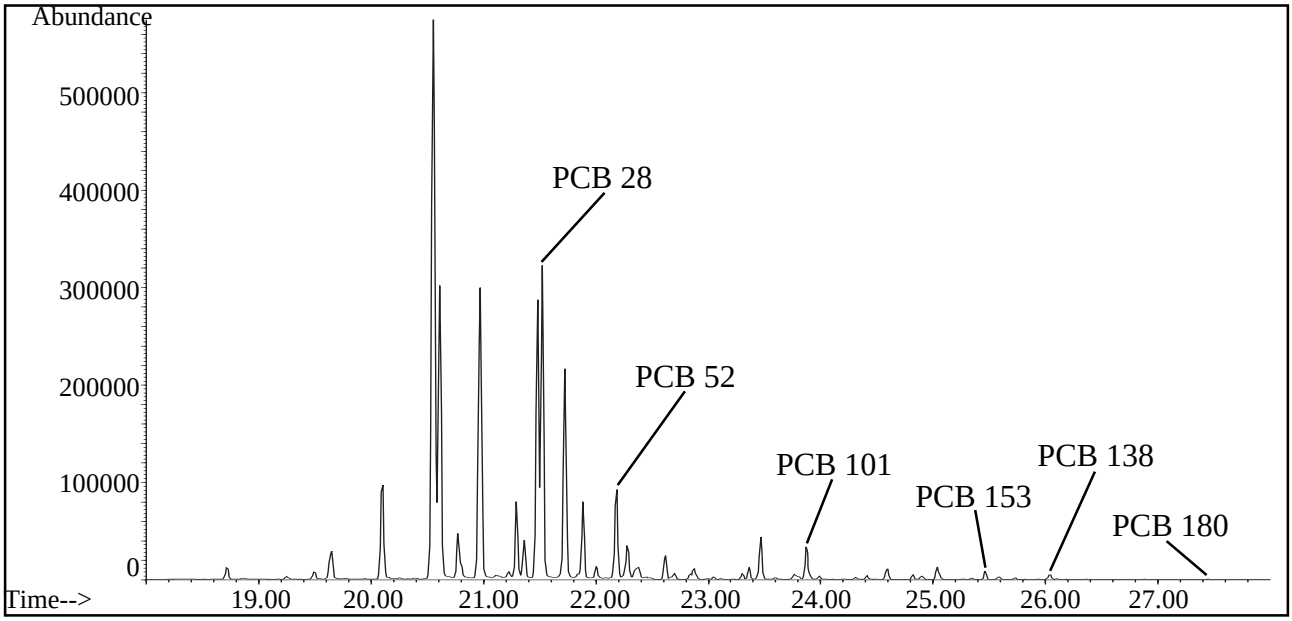

Figure 5. *Selected ion chromatogram of the first filter layer, PCB traces.*

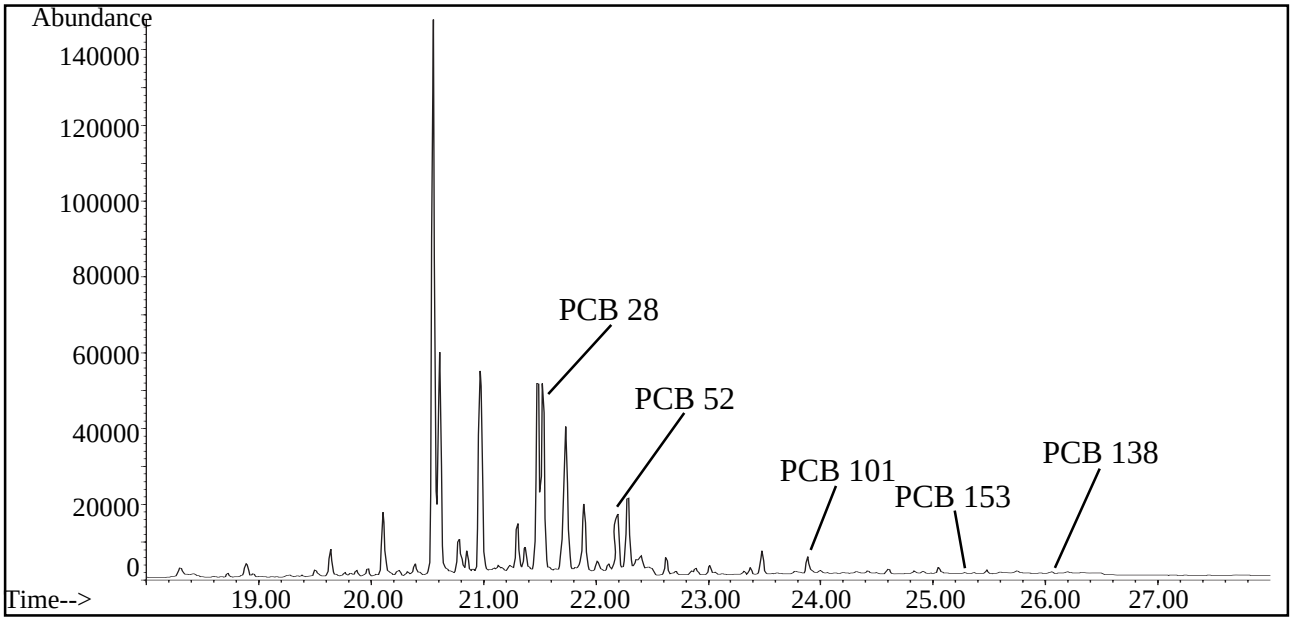

Figure 6. *Selected ion chromatogram of the last filter layer, PCB traces.*

Comparison of standard liquid extraction and thermal desorption. **Figures 7** and **8** (or **Tables I** and **II**, respectively) show the percental distribution of each PCB in the different filter layers. Both techniques here show similar results.

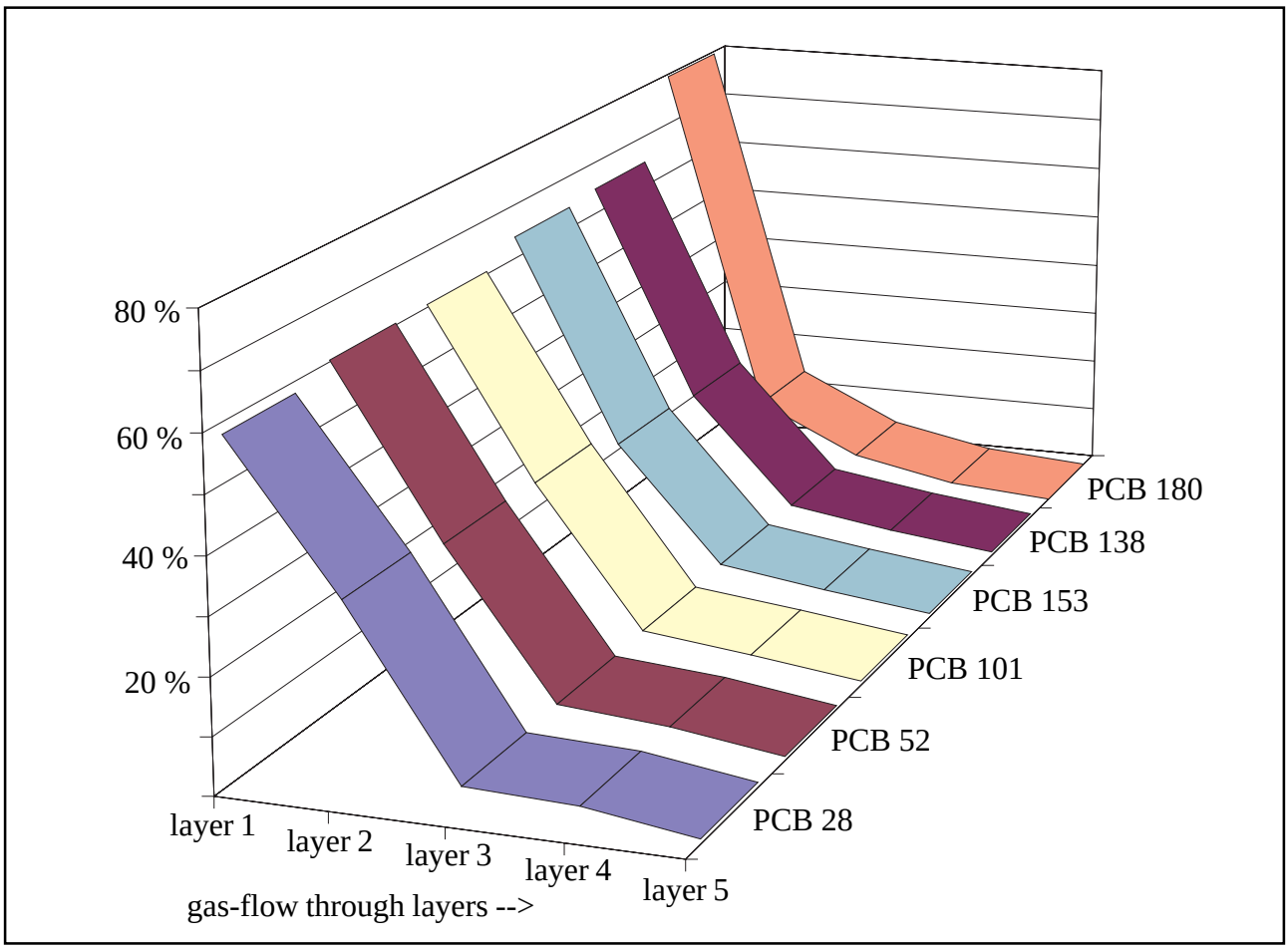

Figure 7. *Percental distribution of PCBs in different filter layers, liquid extraction (see also Table I).*

	layer 1 (%)	layer 2 (%)	layer 3 (%)	layer 4 (%)	layer 5 (%)
PCB 180	79.5	14.1	5.1	1.3	0.0
PCB 138	64.8	25.7	5.8	2.9	0.7
PCB 153	64.0	26.6	5.7	3.0	0.7
PCB 101	60.5	30.2	5.3	3.2	0.8
PCB 52	60.3	30.6	4.9	3.4	0.7
PCB 28	58.0	33.0	4.4	3.7	0.9

Table I. *Percental distribution of PCBs in different filter layers, liquid extraction.*

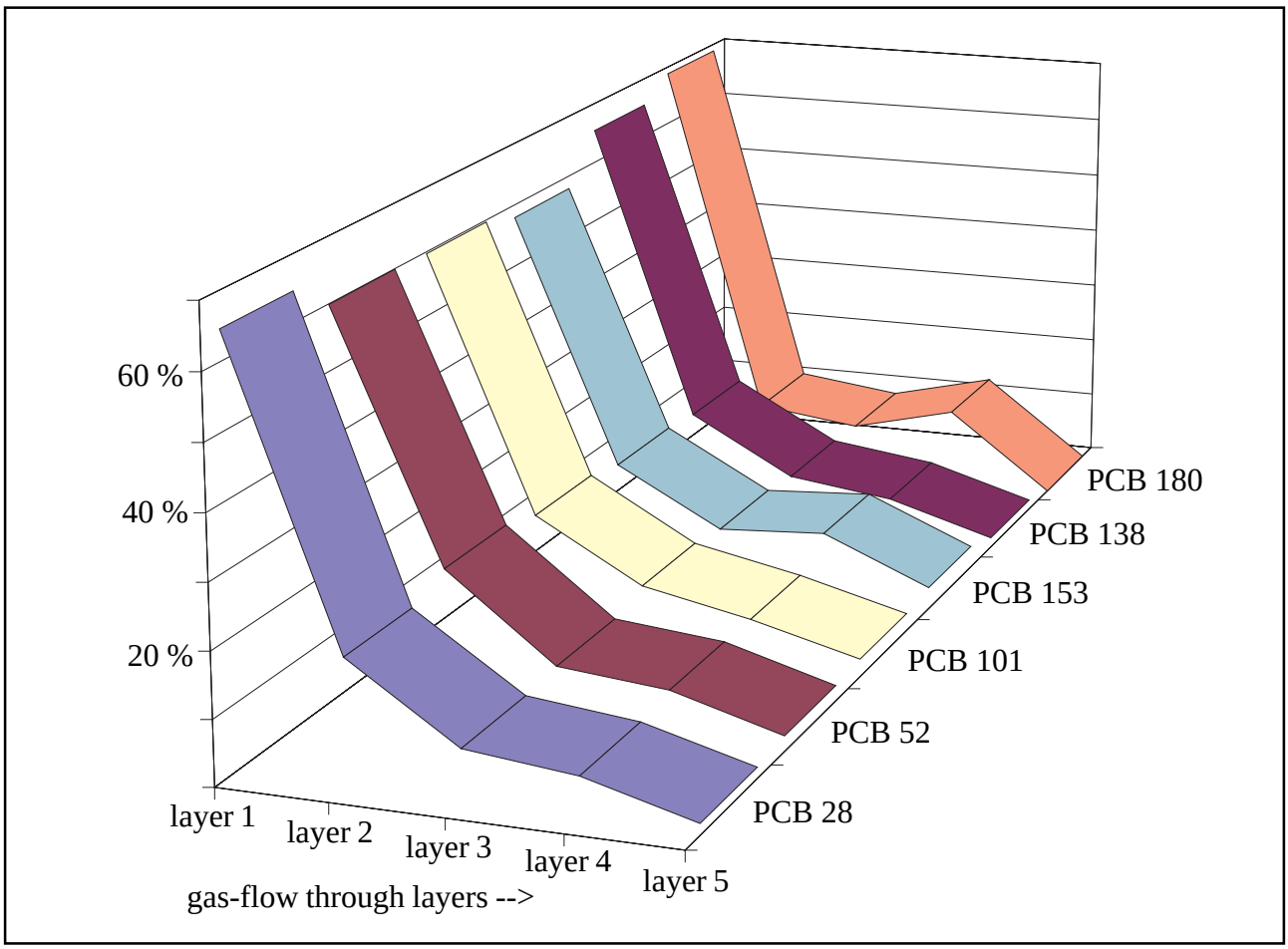

Figure 8. *Percental distribution of PCBs in different filter layers, thermal desorption (see also Table II).*

	layer 1 (%)	layer 2 (%)	layer 3 (%)	layer 4 (%)	layer 5 (%)
PCB 180	68.8	10.4	8.3	12.5	0.0
PCB 138	65.4	17.8	8.7	6.5	1.6
PCB 153	57.9	18.5	9.5	10.6	3.5
PCB 101	59.7	19.9	10.5	7.1	2.8
PCB 52	59.9	21.7	8.8	7.2	2.4
PCB 28	64.6	19.3	8.1	6.3	1.7

Table II. *Percental distribution of PCBs in different filter layers, thermal desorption.*

A difference could be observed when looking at the total amount of PCBs found, where thermal desorption seems to offer higher recovery rates (**Figure 9, Table III**).

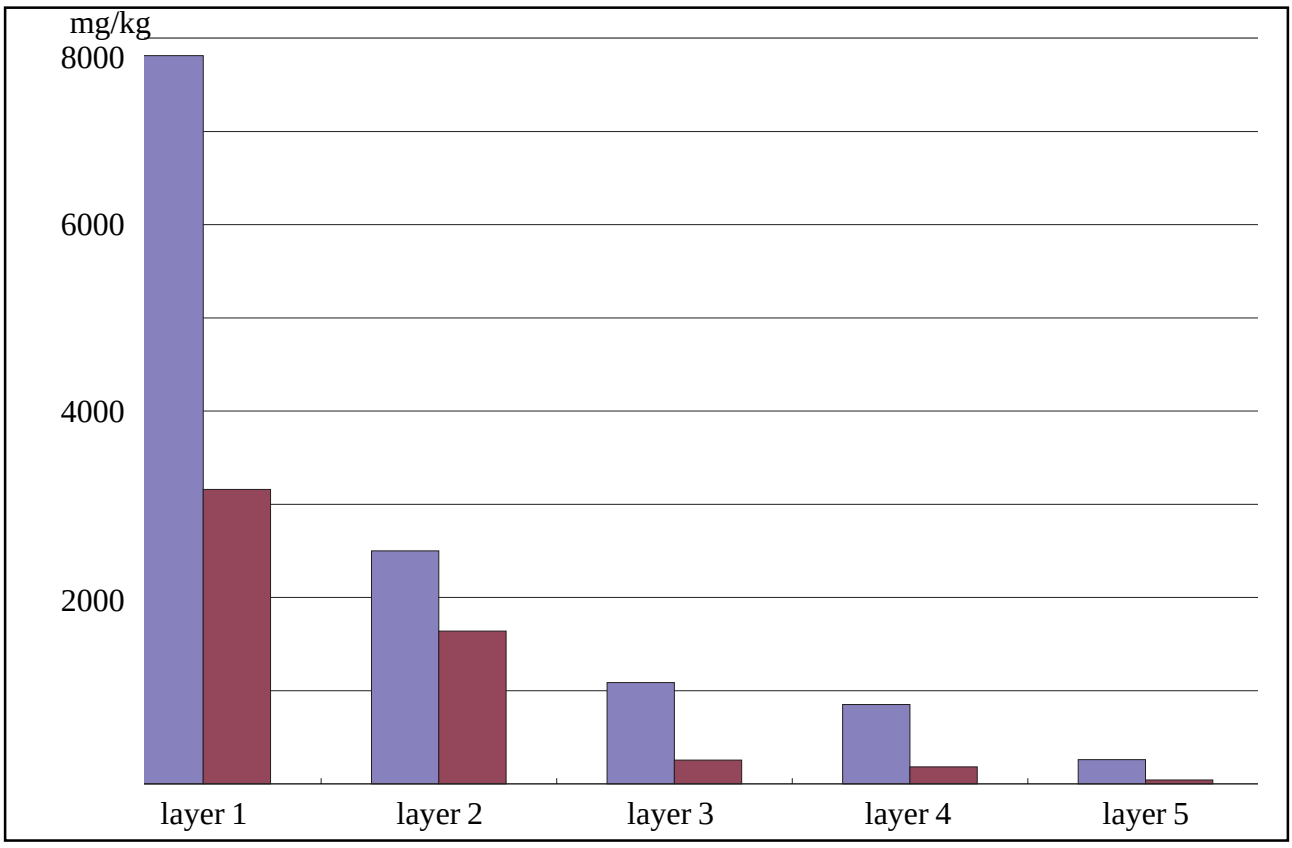

Figure 9. *Comparison of total amounts of PCBs found in different layers, thermal desorption (light grey) and liquid extraction (dark grey, see also Table III).*

	layer 1 (%)	layer 2 (%)	layer 3 (%)	layer 4 (%)	layer 5 (%)
thermal desorption	7813	2500	1085	851	260
liquid extraction	3160	1640	255	182	41.5

Table III. *Percental distribution of PCBs in different filter layers.*

Conclusion

Thermal desorption offers a technique for quantitative trace analysis of PCBs in charcoal filter materials without sacrificing sensitivity or precision compared to standard liquid extraction techniques. The results obtained with standard methods could be reproduced in much less time and without sample preparation.

References

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