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## Sorbents for Trapping Organic Pollutants From Air

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A series of siliceous adsorbents with chemically bonded phases (CBPs) of different polarity were tested as sorbents for trapping air pollutants (petroleum ether) using controlled setup. Moreover, special attention was paid to the potential role of metal impurities as strong adsorption sites. Sorbents were characterized by various physico-chemical methods, such as porosimetry, inductively coupled plasma (ICP) analysis, elemental analysis, derivatography, and gas chromatography. Trapping tubes were utilized for sorption of toxic pollutants from indoor air:

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VOCs air analysis adsorbents surface characterization  
gas chromatography

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### 1. INTRODUCTION

Volatile organic compounds (VOCs) are defined as compounds with the boiling point with the highest limit between 240–260 °C, where the higher values correspond to more polar compounds. VOC sources in the air vary: chemical manufacturing plants, petroleum refineries, chemical laboratories. Industrial solvents and cleaning compounds are potential sources of VOCs (Schwarzenbach, Gschwend, & Imboden, 1993).

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A toxicological study revealed that many VOCs have various reversible and irreversible effects on the human body, ranging from acute anaesthesia to long term effects such as induction of carcinomas (*Merck Index*, 1989). Laboratory studies have shown that certain symptoms can be produced in both sensitive and healthy subjects at total volatile organic compounds levels of 5 and 25 mg/m<sup>3</sup> (Molhave, Bach, & Pedersen, 1986). Human exposure to hazardous compounds must be held within acceptable limits. Airborne contaminant vapours and particles (including aerosols) in industrial atmospheres must be monitored (Bloemen & Burn, 1993). Different solid state porous organic polymers (many kinds of Tenax) and inorganic adsorbents (zeolites, silica gel, etc.) are especially used for collecting (trapping) many hazardous substances. Activated charcoal and Tenax are very often used for this purpose as a trapping material for these applications.

- Activated charcoal is a strong adsorbent material of hydrophobic character. It is used for trapping very volatile compounds. Activated charcoal is still used in the measurement method adopted for occupational exposures (generally 1–100 ppm for a given VOC): A sample of air is pumped across a charcoal sorbent. VOCs are then recovered by a solvent such as carbon disulphide (Polish Standards: PN-Z-04016-3:1989, Polski Komitet Normalizacji, Miar i Jakości [PKNMiJ], 1989; PN-Z-04017-2:1989, PKNMiJ, 1989; PN-Z-04017-3:1990, PKNMiJ, 1990; PN-Z-04134-3:1981, PKNMiJ, 1981).
- Tenax—a hydrophobic, porous polymer (2,6-diphenyl-p-phenylene oxide), stable up to 250°C—is one of the most popular trapping materials. It is an excellent adsorbent for the trapping of nonpolar compounds. However, it has some disadvantages: Very volatile compounds are not retained well on Tenax and include artefact formation of several chemicals (e.g., benzaldehyde). In addition, polar compounds (like ketones and alcohols) are poorly retained on Tenax (*Separation Science Today*, 1995). This polymer was widely adopted during the 1970s as a more reliable sorbent than charcoal for compounds at concentration levels of ppb (Barkley et al., 1980; Krost, Pellizzari, Waburn, & Hubbard, 1982).
- Multisorbent systems—attempts were made to combine the best properties of charcoal and Tenax into a multisorbent system. A tandem system containing Tenax as the first sorbent and new types of activated charcoal (Spherocarb<sup>TM</sup>, Carbosieve<sup>TM</sup>) as second or backup were employed. Spherocarb<sup>TM</sup> and Carbosieve<sup>TM</sup> were developed to

provide more reliable recoveries. Tenax collected the bulk of the VOCs and activated charcoal collected those of VOCs that are more volatile and that "broke through" (did not adsorb) Tenax (Separation Science Today, 1995). Another system was also developed using three sorbents, such as Tenax, Ambersorb, and Spherocharb or Carbosieve (Hodgson, Girman, & Binenboym, 1986). All such systems were created for the trapping and collection of a broader range of chemical types and volatiles (Oliver, Adams, & Daughtrey, 1996).

Packings with chemically bonded phases (CBPs) are now the most popular materials in chromatography. CBPs are used for the preparation of various samples of broad range matrix (water, waste water, blood, urine, plant, and fruit extracts, etc.) in solid phase extraction (SPE), membrane isolation on the phase boundary effects: liquid-solid, and gas-liquid and gas-solid phase (Unger, 1990).

Aue and Teli (1971) were the first to use silica gel (SG-C<sub>18</sub>) with chemically bonded phases (C<sub>18</sub>H<sub>36</sub>SiO<sub>3/2</sub>)<sub>n</sub>, for the sorption of organic substances such as gasoline and car exhaust, chlorinated hydrocarbons. Others authors (Suprynowicz, Buszewski, Pomorska, Gawdzik, & Lodkowski, 1986) investigated SG-C<sub>18</sub> for sorption of pesticides (chlorfenvinphos) from the air (at 6–8 μg/m<sup>3</sup> level). The CBPs were also studied for trapping and determining various chemical groups of pesticides in the air (organophosphorus, carbamates, etc.) has been researched (Pomorska, 1991).

This paper reports on an attempt to develop a simple and reproducible method for collection and preconcentration of ambient volatile organic compounds. Series of sorbents with CBPs with different alkyl chain length and polarity were prepared per analogia with solid phase extraction (SPE). All these CBPs have been characterized by the following physico-chemical methods: porosimetry, elementary, thermal analysis, and inductively coupled plasma (ICP) analysis as well as gas chromatography. The CBPs have been applied as packing material of the trapping tubes and tested for collection efficiency, and preconcentration of light hydrocarbons from air in laboratories or workshops of painting conservation and restoration. Series of CBPs were tested for the efficiency of toluene sorption. Toluene was taken as a representative of VOCs (according to Polish Standard PN-Z-04134-2:1981, PKNMiJ, 1981).

## 2. EXPERIMENT

### 2.1. Material and Reagents

Wide-porous silica gel (Polskie Odczynniki Chemiczne [POCh], Gliwice, Poland) was used as a support for chemically bonded phases. The physical characteristics of bare silica gel and charcoal applied as a reference adsorbent are listed in Table 1.

**TABLE 1. Surface Characteristics of Bare Silica Used as a Support for CBP Packings and Activated Charcoal**

Parameter	Unit	Abbreviation	Value for	
			Bare Silica Gel	Activated Charcoal
Particle size	mm	$d_p$	0.71–1.00	1.00
Specific surface area	m <sup>2</sup> /g	$S_{BET}$	250	1130
Mean pore diameter	nm	$D$	12.00	—
Pore volume	cm <sup>3</sup> /g	$V_p$	1.00	—
Specific surface area of mesopore	m <sup>2</sup> /g	$S_{ME}$	—	120
Adsorption value of micropore	mmol/g	$a_{MI}^0$	—	12.40

For surface chemical modification, the following reagents were used: phenyldichlormethylsilane, octadecyldichloromethylsilane, octylmethyltrichlorosilane, (3-aminopropyl)-triethoxysilane (Petrarch System, Levitown, PA), (3-glycidyloxypropyl)-trimethoxysilane, (3-cyanopropyl) dimethylchlorosilane, trimethylchlorosilane (Fluka, Buchs, Switzerland), morpholine—dry (Reachim, Moscow, Russia), toluene standard for gas chromatography (GC), n-hexane *ultra-resi* grade, hydrochloric acid *ultra-resi* grade (J.T. Baker, Deventer, Holland), carbon disulphide HPLC grade (Aldrich, Steinheim, Germany). Activated charcoal AG-5 for gas adsorption particle size approximately 1 mm (Coal Carbonization Plants, Hajnówka, Poland) was applied as a reference trapping material.

### 2.2. Apparatus and Physico-Chemical Investigations

The porosity parameters of the bare silica ( $SG$ ;  $S_{BET}$ —specific surface area,  $V_p$ —pore volume,  $D$ —mean pore diameter) were determined by the low temperature adsorption-desorption of nitrogen using a Model 1800 Sorptomatic instrument (Carlo Erba, Milan, Italy). Thermal analysis

was performed by means of a Model Q-1500 D derivatograph (MOM, Budapest, Hungary). Determination of metal content in SG was performed using a Plasma Spectrometer (PU-7000 Philips Scientific, Cambridge, UK) equipped with an ultrasonic nebulizer CETAC U-5000. Samples were prepared prior to ICP analysis by microwave mineralization (microwave station MDS-2000, CEM, Matthews, NC). A CHN analyzer (Perkin Elmer Model 240, Norwalk, CA), was used to determine carbon ( $P_C$ ) and nitrogen ( $P_N$ ) percentage in the CBPs.

### 2.3. Preparation of CBPs

A batch of silica gel support was purified with diluted (20%) hydrochloric acid for 12 hrs in a Soxhlet apparatus (Buszewski, 1992a). Then, after washing in distilled water until obtaining neutral pH, it was dried at temperature 160°C under vacuum ( $10^{-2}$  Pa) for 6 hrs. In the next step, each portion of SG was treated with individual silane in a specially constructed glass reactor that makes contact of reagents with the environment impossible. Details on procedures and conditions are described elsewhere (Buszewski, 1989, 1992a, 1992b; Buszewski et al., 1989).

### 2.4. Testing of Toluene Recoveries From Silica Gel With CBPs

The setup used for sorbent testing is illustrated in Figure 1. Packing materials such as bare silica gel and chemically modified silica gel (octadecyl-, octyl-, trimethyl-, cyano-, phenyl-, amino-, diol-) were tested in this setup.

The procedure for testing trapping kits was as follows:

1. application of 10  $\mu$ l (equivalent of 0.62 mg toluene) of the standard solution of toluene in hexane to the injector,
2. passing air stream (flow rate of 1.5 L/hr) through the tube for 80 min,
3. elution of sorbed toluene using hexane (total volume 10 ml),
4. collection of the eluate in a volumetric flask of 10 ml,
5. GC analysis.

### 2.5. Procedure for Sorption Petroleum Ether From Indoor Air

Comparative studies have been undertaken with application of sorbents such as activated charcoal, washed (SG-AW-C<sub>18</sub>), and nonwashed

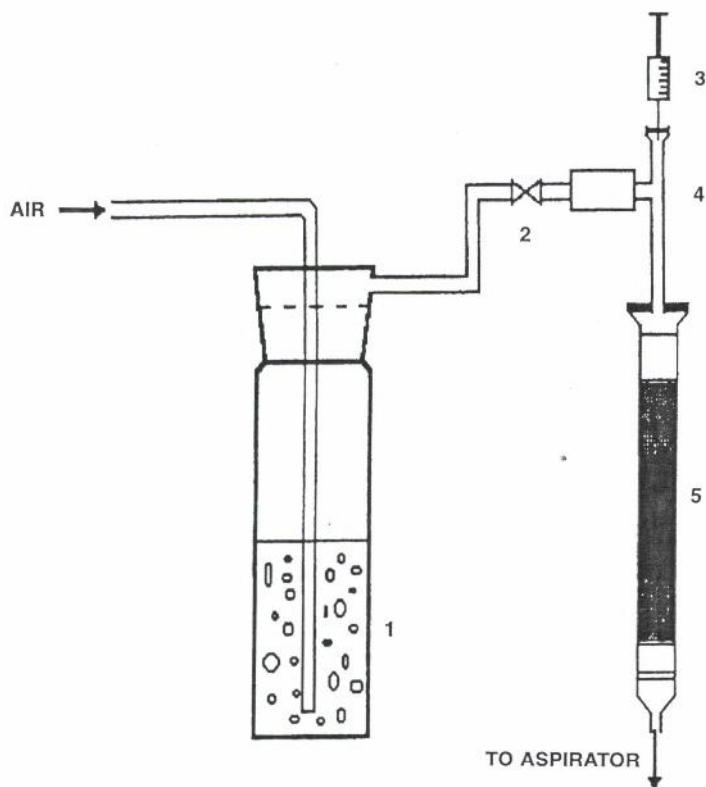


Figure 1. Controlled setup (described in the text): 1—gas dryer with molecular sieves; 2—air flow regulator and control unit; 3—syringe; 4—injector (built like a gas chromatography [GC] on-column injector); 5—trapping tube (contains 3 g of chemically bonded phases) connected to an aspirator (AM-1, Mechanics Plants, Bytom, Poland).

(SG-NAW-C<sub>18</sub>) silica gel with bonded octadecyl hydrocarbon chains. The first trapping tube was charged with 100 mg of CBPs, the second tube was charged with 100 mg of activated charcoal. These trapping tubes were connected by a Y-shaped connector with an aspirator. Through both tubes an air stream was passing, 1.5 L/hr of flow rate, for 3 hrs. After the air passed, sorbents were removed to a vial with a cap. Next, 1.00 ml of CS<sub>2</sub> was added into the vial and slowly mixed for 15 min. The airflow (25 ml/min), weight of sorbents (100 mg), and time of sampling (3 hrs) were selected according to Polish Standard PN-Z-04134-3:1981(PKNMiJ, 1981).

## 2.6. Chromatographic Measurements

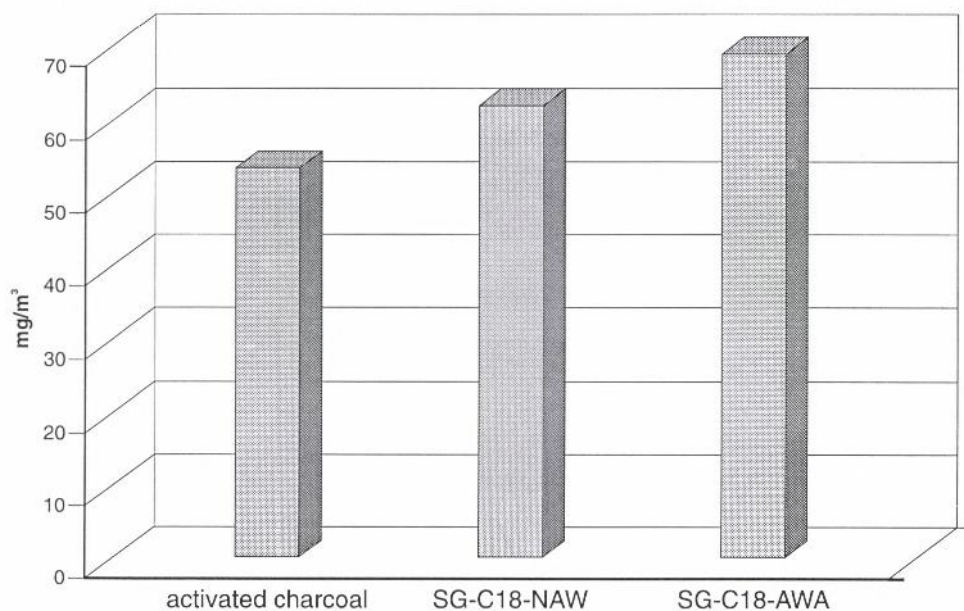
Extracts from sorbent testing were immediately analyzed by injecting 1  $\mu\text{l}$  of the sample into a gas chromatograph (Fisons 8160, Milan, Italy) with a flame ionization detector (FID). The Supelcowax-10 (30 m  $\times$  0.32 mm  $\times$  0.25  $\mu\text{m}$ , Supelco, Bellefonte, PA) capillary column was used to separate and quantify the sample compounds. The carrier gas (helium—99.999%) flow rate was 2.0 ml/min. The temperatures were as follows: injector—220°C, detector—250°C, column—75°C (for analysis of toluene), and for analysis of carbon disulphide extracts a temperature gradient was used: 60°C—hold 4 min, rate—15°C/min to 200°C. Data were collected and processed by a computer equipped with Chromcard software (Fisons, Milan, Italy).

## 3. RESULTS AND DISCUSSION

Test traps filled with activated charcoal are used for studying indoor air (recommended by Polish National Standards in general [see references]). Investigations of VOC sorption from urban air, indicated exceptional property of silicous sorbent with chemically bonded phases (CBPs; Buszewski, Ligor, Górna-Binkul, & Gadzała-Kopciuch, 1997; Suprynowicz, Buszewski, & Pomorska, 1981). CBPs are widely used as stationary phases in solid phase extraction technique (SPE) for sorption and preconcentrations of hazardous compounds from various liquid matrices (*BakerBond SPE*<sup>TM</sup>, 1992; Buszewski, 1990). The use of SG-C<sub>18</sub> for sorption of indoor pollutants of air in the conservation and restoration of a painting laboratory was investigated. Petroleum ether was frequently used for cleaning old paintings in this laboratory. The values of concentrations for light fractions of hydrocarbons on CBPs were compared to concentrations on activated charcoal (Polish Standard PN-Z-04134-2:1981, PKNMiJ, 1981). According to this standard, analysis with the use of charcoal indicated the concentration of petroleum ether at the level of 5.1 mg/m<sup>3</sup> (*SD* = 0.765). In contrast, concentrations on the nonwashed silica gel SG-NAW-C<sub>18</sub> were about 5.7 mg/m<sup>3</sup> (*SD* = 0.456). However, the use of washed silica gel (SG-AW-C<sub>18</sub>) has given a higher concentration. It was 6.1 mg/m<sup>3</sup> (*SD* = 0.600). Concentrations of a compound after using the various sorbents are presented in Figure 2. These differences can be explained by higher coverage density for



washed silica gel (Buszewski, 1990). Probably, exerted heteroatoms of metals and the heterogeneity of the surface influenced these results. The presence of heteroatoms (metal atoms) localized on the support surface resulted in energetic heterogeneity and influenced negatively the sorption processes. From the literature it is known that heterogeneity of surface has a negative influence on the chemical modification of siliceous support. Especially, the formation of a chemically bonded film strongly depends on the purity of SG support (Buszewski, 1992b). Properties of the surface were examined on the basis of studies before and after acid washing.



**Figure 2.** Indoor air concentration of light hydrocarbons fraction obtained on activated charcoal and SG-C<sub>18</sub> (nonwashed silica gel SG-C<sub>18</sub>-NAW and washed silica gel SG-C<sub>18</sub>-AWA).

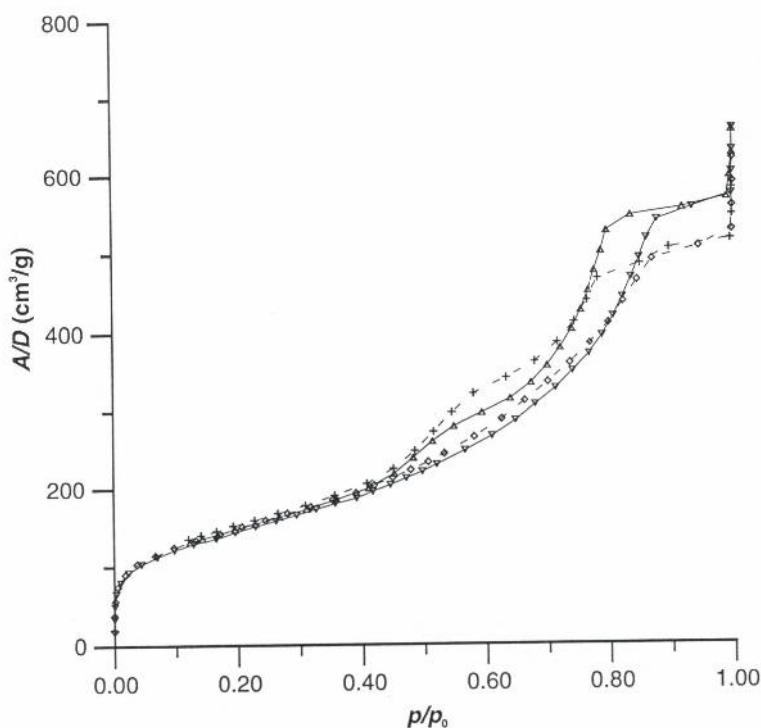
Elemental analysis was performed for determining carbon in CBPs to explain the aforementioned facts. From Table 2, it can be seen that washing the surface of silica gel increases the efficiency of the covering process by C<sub>18</sub> stationary phase (about 59%). This effect is caused by higher population of accessible silanols. Various recoveries for SG-NAW-C<sub>18</sub> and SG-AW-C<sub>18</sub> CBPs are caused by different porosity, metal impurities, and various solubility of VOCs in organic bonded phases. For a precise explanation of differences in sorption properties of washed and nonwashed silica support, various physico-chemical methods were used.

**TABLE 2. Contributions of Carbon and Nitrogen (Data From Elemental Analysis) in CBP Packing With Different Functional Groups**

Number	Type of Packing	P <sub>C</sub> (%)	P <sub>N</sub> (%)	a <sub>N</sub> (nm <sup>-1</sup> )
1	*SG-C <sub>18</sub>	8.07	—	1.91
2	SG-C <sub>18</sub>	15.28	—	2.16
3	SG-(CH <sub>3</sub> ) <sub>3</sub>	3.60	—	2.60
4	SG-C <sub>8</sub>	9.67	—	2.89
5	SG-Ph	4.02	—	1.45
6	SG-DIOL	4.06	—	1.09
7	SG-CN	4.92	1.76	3.12
8	SG-NH <sub>2</sub>	3.32	1.02	2.44

Notes. CBP—chemically bonded phases, \*—packing with controlled low carbon coverage (prepared on SG-NAW, without an activator), P<sub>C</sub>—measured carbon (%), P<sub>N</sub>—measured nitrogen (%), a<sub>N</sub>—number of bonded ligands per nm<sup>2</sup> of surface area.

In order to investigate the influence of acid washing on the surface topography of silica gel, the porosimetry analysis of SG samples was performed. Analyzing low-temperature adsorption isotherms of nitrogen (at 77 K) for acid washed (SG-AW) and nonacid washed (SG-NAW) silica gel, superposition of the left branches of these curves in the range of relative pressure  $p/p_0 = 0-0.4$  (Figure 3) can be seen. Changes of the isotherm shape (in the case of SG-AW) from  $p/p_0$  values of 0.5 to 0.9 should be attributed to the increase of the amount of macropore (Ościk, 1982). As a result of cleaning, the secondary siliceous structure occurring in mesopores was removed leading to the exposure of cylindrical pores with a higher contribution of macropores. Simultaneously, an increase of the population of micropores was not observed. Presumably, from the thermodynamic point of view of the separation processes, the mass transfer of cleaned material will be better (easy "coming" and "going" of adsorbant molecules into and from the pores) than for sorbent not treated with acid. The course of the hysteresis loop for SG-NAW can indicate the siliceous structure inside pores (Vansant, Van Der Voort, & Vrancken, 1995). According to the shape of the branch ( $p/p_0 = 0.8-1$ ) of adsorption-desorption isotherm for SG-AW (Figure 3) it can be stated that during the cleaning process, the siliceous matrix was not changed, only pores were purified from the secondary siliceous structure (silicates). Further course of the curve indicates saturation of pores and formation of the polylayer of nitrogen molecules on the surface of micro-, meso-, and macropores. This testifies to the presence of pores of 200 Å diameter (confirmed by porosimetry analysis) in the material. The



**Figure 3.** Low temperature adsorption-desorption curves of nitrogen obtained on SG-NAW (----) and SG-AW (—). Notes.  $A/D$ —adsorption/desorption,  $p/p_0$ —relative pressure.

result of analysis were calculated for the presence of the populations of three different kinds of pores, that is, micropores ( $D < 20 \text{ \AA}$ )—20%, mesopores ( $D = 20\text{--}200 \text{ \AA}$ )—65% and macropores ( $D > 200 \text{ \AA}$ )—15%.

Metal impurities were suspected of influence on the creation of a film of chemical phases on the support surface. The results from ICP analysis of acid washed and nonacid washed silica gel are presented in Figure 4. Significant differences in metal content for SG-AW and SG-NAW can be seen. The biggest reduction took place in the case of Ca (99.8%) and Mg (98.9%), and the smallest for Ti (44.7%).

The bigger mass ( $\Delta m$ ) for SG-AW (about 1%) can be calculated from the shape of the thermal gravimetry (TG) curve (Figure 5). It can be explained through the increase of the population of active silanols in water sorption. Obviously, a higher concentration of accessible groups exerts a positive influence on the chemical modification process (higher overage density of modifier molecules on the SG support surface; Buszewski, 1992b).

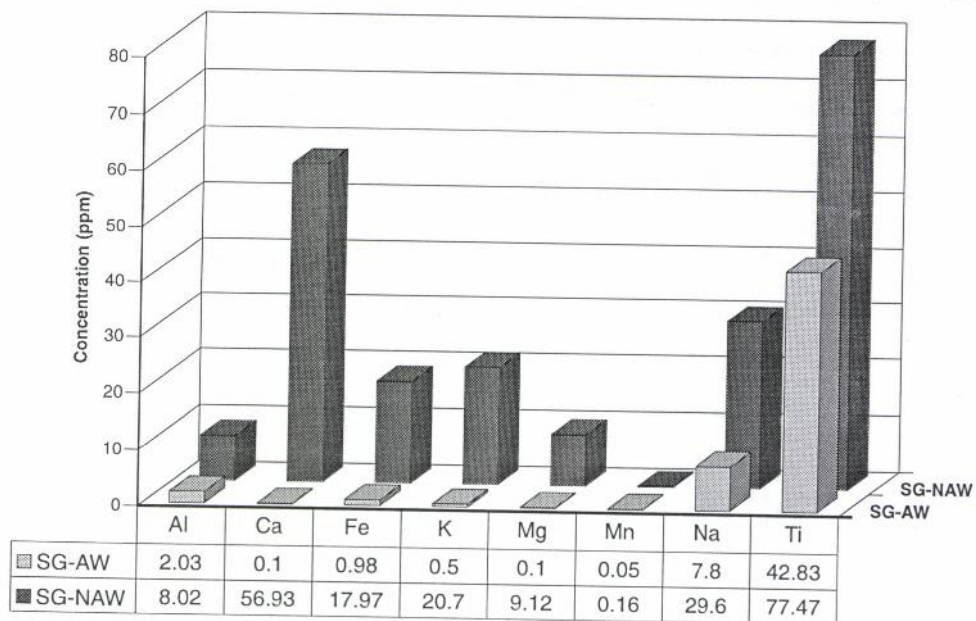


Figure 4. Content of metals (ppm; data from inductively coupled plasma analysis) in silica gel before and after acid washing (20% HCl).

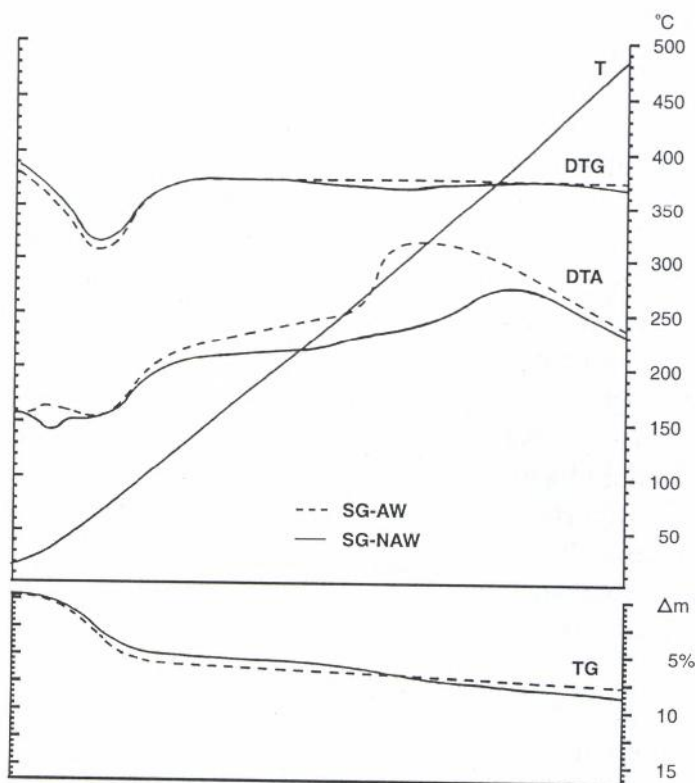


Figure 5. Thermogravimetry analysis of bare nonwashed and acid washed silica gel.

TABLE 3. Recoveries of Toluene Form Various Sorbents ( $n = 3$ )

Kinds of Sorbents		$M$ (mg)	$SD$	% $RSD$	Recovery (%) - $R^*$
SiO <sub>2</sub>	AWA	0.442	0.037	8.5	71.39 ± 1.31
SiO <sub>2</sub>	NAW	0.548	0.043	8.0	88.38 ± 1.39
C <sub>18</sub> -	NAW	0.445	0.026	6.0	71.77 ± 1.11
C <sub>18</sub> -	AWA	0.590	0.039	6.7	95.18 ± 1.14
C <sub>8</sub> -	AWA	0.600	0.052	8.7	96.72 ± 3.05
(CH <sub>3</sub> ) <sub>3</sub> -	AWA	0.146	0.007	4.5	23.50 ± 0.32
CN-	AWA	0.675	0.064	9.5	108.81 ± 2.32
Ph-	AWA	0.657	0.054	8.2	105.98 ± 2.53
NH <sub>2</sub> -	AWA	0.669	0.058	8.6	107.87 ± 1.18
DIOL-	AWA	0.446	0.027	6.1	71.95 ± 1.41

Notes. \*—recovery was calculated as a quotient of the mean value of toluene obtained after sorption to applied toluene mass (see section 2.4.).

The carbon chain and different ligand polarity effects, various kind of CBPs (-C<sub>8</sub>, -Ph, DIOL, etc.) were synthesized for the study (Table 3).

Evaluation of the sorption properties of tested packing materials was accomplished on the basis of toluene recoveries obtained for these materials. It can be seen from Table 3 that the recoveries vary from 23% (in the case of SG-(CH<sub>3</sub>)<sub>3</sub>) to 108% (for SG-CN packing). Recovery values exceeding 100% can be explained by very strong bonding of toluene molecules to adsorbent surface (irreversible sorption). High recovery for SG-Ph packing (105 ± 2.5%) is a result of interactions among aromatic sextets (π-electrons) of the rings of toluene and rings that are present on the surface of the phenyl phase. Similarly, high recoveries for phases with polar groups (SG-CN, SG-NH<sub>2</sub>) can be a result of interactions of toluene aromatic sextets with free electron pairs of nitrogen atoms. Recovery obtained for octyl (SG-C<sub>8</sub>) and octadecyl (SG-C<sub>18</sub>) phase is also very high (about 95%). Taking into account hydrophobicity of toluene molecules, they can be easily sorbed by nonpolar hydrocarbon chains (-C<sub>8</sub>H<sub>17</sub>, -C<sub>18</sub>H<sub>37</sub>) present on the sorbent surface. The lowest recovery of toluene observed for SG-(CH<sub>3</sub>)<sub>3</sub> phase were caused in the sorption process by locking active silanols by trimethylchlorosilan (TMCS) used as a modifier for the formation of this phase.

It should be noticed that recovery for SG-AW-C<sub>18</sub> packing is higher than 23% compared to the SG-C<sub>18</sub> phase prepared on nonacid washed silica gel. An increase of the coverage density of chemically bonded film influences significantly toluene sorption (higher number of chains that

can interact with sorbate molecules) and, as a consequence, gives higher recoveries.

#### 4. CONCLUSIONS

CBPs are useful for sampling and enriching naphta vapours and toluene from indoor air. Using these phases results in higher recoveries (higher concentration values) and lower standard deviation than still used activated charcoal. The etched silica gel is an excellent support for chemically bonded phases. The washing process gives better sorption properties and assures "easy" mass transfer of adsorbant molecules into and from pores of silica gel. On the other hand, analysis costs are lower because CBPs can be used several times (minimum 15 times) but activated charcoal can only be used once. The determination limit for investigated compounds was lower than  $0.2 \mu\text{g}/\text{m}^3$ .

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