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Removal of Toluene at Low Concentration with Activated Carbon Filter

Adsorption Analysis using Tenax Tube and TD-GCMSD

Helsinki Metropolia University of Applied Sciences

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<p>Abstract</p> <p>Various studies have demonstrated the usefulness of adsorption of activated carbon on VOCs removal. Large porosity and high adsorption allows activated carbon to remove very small particles. This project was commissioned by the company name Genano Oy to test the activated carbon filter in their air purifier and hence to provide a database for the company. The purpose of this thesis was to study the effectiveness of impregnated activated carbon filter on toluene removal.</p> <p>Experiment was conducted with a pipe model consisting activated carbon filters to simulate a ventilation system. Two batches of activated carbon were used, and one to three layers of filters containing these activated carbon were made. Toluene was prepared at low concentration. The samples before and after contacting the activated carbon filter inside the pipe model were taken by tenax tubes. At last, the results were analyzed by using a thermal desorber gas chromatography mass selective detector.</p> <p>The results of this thesis indicate that filters with one layer of impregnated activated carbon had a removal efficiency around 40 %. Based on these results, impregnated activated carbon revealed their potential on VOCs removal.</p>	
Keywords	Toluene, activated carbon, adsorption, tenax tube, TD-GCMSD, indoor air quality.

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This work was conducted in Helsinki Metropolia University of Applied Sciences in Finland along with the cooperation from the Genano OY Company where I did my internship. This work gave me a brand new perspective to evaluate my knowledge and know-how with my major in Environmental Engineering. It also tested and trained my ability to withstand pressure and difficult circumstances. This work benefited me not just in academic skills but also in moral maturity. I learnt time management, gathering and organizing information, and grabbing the chances to discuss my topic with people around me. I also realized my weakness and how superficial my knowledge was. I would say the most rewarding achievement through my study years so far is the completion of my Bachelor thesis.

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Notwithstanding all of the above support for my project, any errors and/or omissions are solely my own. I want to dedicate this to my family and my girlfriend who always supported me and encouraged me to complete my Bachelor's degree.

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Abbreviation

AC	Activated carbon
GC	Gas chromatography
IAC	Impregnated activated carbon
IAQ	Indoor air quality
IN	Inflow
OUT	Outflow
PM	Particulate matter
ppm	Parts-per-million
TD-GCMSD	Thermal desorber gas chromatography mass selective detector
VOCs	Volatile organic compounds

1 Introduction

Serious consideration toward environmental impacts and attempts to address them have developed significantly in recent decades. Among these issues, air pollution stands out as one of the principle concerns as it had drawn attention already after the industrial revolution. Air pollution resulted in anthropogenic emissions includes industrial manufacturing, domestic utility, and transportation. From these, common pollutants such as carbon monoxide (CO), sulfur oxides (SO_x), nitrogen oxide (NO_x), volatile organic compounds (VOCs), ammonia (NH₃), and particulates (PM) are discharged (Cooper and Alley, 2011). Air pollution contributes to human health problems, ecosystem threats, climate change, acidification, ozone depletion, haze and smog formation, and eutrophication. Of major believes, air pollution is highly related to lung cancers and other diseases. As reported from World Health Organization (WHO, 2014), around 7 million people died worldwide in 2012 as a result of air pollution.

Air pollution could be outdoor or indoor pollution where the term outdoor pollution often refers to the urban air quality. Both urban and indoor air quality are described as the worst pollution problems especially in developing countries in Asia. Meanwhile, regulations and treatments have been carried out to reduce emissions, for example, from factories to the atmosphere. Building ventilation and indoor air purifiers have been installed to improve indoor air quality (IAQ).

As people move into the city and spend more time indoor especially in developed countries, for instance, working or visiting in offices, factories, stores, restaurants, hotels and airports, staying at homes, and traveling inside vehicles, the effect of indoor air pollution is tremendous on human health (Cooper and Alley, 2011, pp.695). Regarding indoor air pollutants, which often contain irritation or even toxic and hazardous substances and hence decline the indoor air quality and threat occupant's health, volatile organic compounds (VOCs) are recognized as one of the major contributors along with other pollutants including CO, radon, particulates, and microbial contaminants such as mould. According to the study from U.S.EPA (EPA, 2015a), VOCs concentration tends to be much higher indoors than outdoors. As a result, proper building ventilation and air purification methods especially for treating VOCs is required to secure human health.

Toluene, a very common indoor air pollutant and model compound for VOCs, is the focus of this study. Toluene can be found as a solvent in many commercial products such as paints. In recent research, major approaches attempt to remove toluene include adsorption, thermal incineration and catalytic oxidation. For example, a study conducted by Liu, Chen and Peng (2013) has shown that adsorption method on activated carbon filters has drawn great deal of attention recently due to the characteristics of having a much larger specific surface area, faster adsorption, high fluid permeability, and a more uniform micropore structure. Hameed (2009) also described adsorption method as “the most efficient, promising and widely used fundamental approach” due to its “simplicity, economical viable, technically feasible and socially acceptable”. For these reasons, this study chose the adsorption method on activated carbon filters for toluene removal.

This thesis consists of an introduction, theoretical background, and an experimental part where the experiment using a thermal desorber gas chromatograph mass selective detector (TD-GCMSD) and tenax tubes on analysing toluene removal by activated carbon filters is described. Results of the experiment are presented and discussed and conclusions are drawn at the end of the thesis.

2 Goal and scope

This thesis was made for a company, Genano Oy (Genano) who is professional in air purification technology in buildings such as hospitals and laboratories. Genano provides services on air purification using technology including electrostatic precipitation where activated carbon filters perform as a part of the components in those devices. This project work was conducted during my internship with Genano using Helsinki Metropolia UAS facilities. The results of this thesis would be used to provide a data base and to support decision making for Genano.

The purpose of this thesis was to study the behaviour of activated carbon filter and the quality of different batches of impregnated activated carbon filters (provided by Genano) on indoor gas pollutant of toluene removal. An designed experiment was conducted to create a model of an indoor ventilation system with PVC pipes containing an activated carbon filter while fed with toluene gas. Air samples were taken by using tenax tubes and were later analysed with TD-GCMSD.

The thesis focused on homes and commercial buildings where the system is modeled to keep the toluene concentration low for analysis. The activated carbon used in the experiment was not necessary provided from Genano.

3 Theoretical background

3.1 Indoor air

Studies have shown that average person spent his/her day 86% indoor while 8% outdoor and 6% traveling in United States. In other word, people spend around 20 hours indoors per day. As a result, the quality of indoor air in residences and public buildings generates a significant concern at personal level. (Griffin, 2007, pp.291) IAQ is introduced as it refers to the air quality in a building especially relating to the occupants' health and comfort. Unpleasant air odor inside a room is also an example of IAQ concern.

Indoor air is treated in a different way from outdoor air due to two main reasons. The first reason is that indoor air is considered as private property and therefore it must be subjected to different regulations from those set for outdoor air. The second reason is technical where the air behaves differently between indoor and outdoor due to different pollutant sources and dispersion behaviour. In addition, the concentrations of certain pollutants may be higher outdoors than indoors, while those of other pollutants are vice versa. (Cooper and Alley, 2011, pp.696)

The concentrations of most indoor air pollutants tend to be higher in the winter than in the summer. This results from people tending to close the windows for heating during the winter and open the windows for cooling during the summer. Thus, the air exchange between outdoors and indoors will be greater in the summer than in the winter. (De Nevers, 2000, pp.542-543) Apart from this, using an air conditioner in the summer while closing the windows for cooling down the temperature also services like a closed system. In such cases, the IAQ relies greatly on the ventilation system and also on an air purifier if necessary. However, in most developing countries especially in China where the ambient air quality is generally poor, it does not benefit much when exchanging indoor air with outdoor air. Therefore, an air purifier is needed for the indoor space.

3.1.1 Indoor air pollutants and their sources

The six most common air pollutants identified by the Clean Air Act (CAA), also known as the criteria pollutants, are PM, ozone (ground-level), CO, NO₂, SO₂, and lead. (IDAHO, 2015) Regarding indoor air pollutants, criteria pollutants are also included along

with radon, environmental tobacco smoke, combustion emissions which contain NO₂, CO, PM and polycyclic aromatic hydrocarbons (PAHs), formaldehyde, pesticides, bio-aerosols such as molds, and VOCs. Those indoor pollutants come from five categorised sources which are combustion sources, building materials and furnishings, consumer products, people, and outdoor air. Combustion sources include stoves (cooking), kerosene heaters, and tobacco smoke. Building materials and furnishings take account of paints, particleboard, caulks, adhesives, and carpeting. Consumer products contain cleaning materials, waxes and polishes, pesticides, and cosmetics. Indoor air pollutants such as VOCs contributed by people are directly from biological or metabolic functions, and indirectly from clothing, deodorants, perfumes, etc. (Cooper and Alley, 2011, pp.696-698). In a lesser degree, outdoor air is included in these categories due to infiltration of outdoor air including CO and ozone. (Griffin, 2007, pp.291-294; Cooper and Alley, 2011, pp.696-698)

Even though VOCs is not classified as the criteria pollutant, it is considered as a major primary pollutant due to its relatively low vaporization temperature and therefore generates large emissions. Noel de Nevers from the University of Utah (2000, pp.329) claimed that “VOCs are probably the second-most widespread and diverse class of emissions after particulates”.

3.1.2 General health effects of indoor air pollution

The health effects due to the indoor air pollution can be either acute or chronic. For acute or immediate effects, which may take place after just a single exposure or frequent exposures, people may suffer from irritation of the eyes, nose and throat, headaches, dizziness, and fatigue. Other more serious symptoms from these acute effects include asthma, humidifier fever, and pneumonitis. Fortunately, most of these immediate effects usually last in a short time. Treatment for such immediate effects includes simply staying away from exposing to those pollution sources or eliminating the sources of pollution, if it can be identified. For chronic effects, usually caused by long-term exposure, people may suffer from respiratory diseases including emphysema, heart diseases, and cancers. These health effects could be fatal. Apart from the exposure frequency, the likelihood of both acute and chronic effects also depends on each person. Some people are more sensitive to certain type of pollutants while others may not. (Burke, Singh and Theodore, 2005, pp.138)

3.1.3 IAQ control

The approach for IAQ control is similar to other emission control where the first step is to reduce or even eliminate the use of energy in a household or public buildings including heating, ventilation, and air conditioning (HVAC) and other appliances. According to Energy Information Administration (EIA) (EIA 2012, p 3), this energy consumption accounted for around 40% of the total energy flow in U.S in 2011. Using a better energy efficient design of the residents and public buildings will conserve energy and thus reduce the emissions.

According to the U.S. EPA (EPA, 1997), "controlling indoor air quality involves integrating three main strategies. First, manage the sources of pollutants either by removing them from the building or isolating them from people through physical barriers, air pressure relationships, or by controlling the timing of their use. Second, dilute pollutants and remove them from the building through ventilation. Third, use filtration to clean the air of pollutants."

Ventilation is the process of air exchange where outdoor air replaces indoor air that is being exhausted to the outdoor. Air exchange occurs in building by the means of forced ventilation, natural ventilation, and infiltration. Using fans or blowers to exchange the air forcibly is defined as forced ventilation. Natural ventilation involves allowing ambient air exchange simply through open windows or doors. Infiltration refers to the occurrence of air exchange through leakage from gaps of a building while windows and doors are closed. (Cooper and Alley, 2011, pp.704-706) To conclude, opening windows or doors frequently in permitted conditions improves IAQ significantly.

The use of filtration refers to air purifiers or air cleaners. There are many types and sizes of air purifiers commercially ranging from inexpensive portable models to expensive whole-house systems. The performance of an air purifier depends on how much air can be draw through the purifier and how well the purifier collects indoor air pollutants. However, an air purifier could be ineffective if the source of the pollutants is too strong. (Burke, Singh and Theodore, 2005, pp.151-154)

When dealing with VOCs, the emission control could be carried out by the methods of condensation, absorption (scrubbing), adsorption, and oxidation. (De Nevers, 2000, pp.329-381)

3.2 Toluene

Toluene is a common indoor air pollutant in the group of VOCs. It is the main focus of this study. The molecular formula of it is C_7H_8 . The IUPAC name for toluene is methylbenzene. Toluene is a hydrocarbon consisting of a single benzene ring (phenyl group) and a methyl group (CH_3) and is classified as an aryl (aromatic) hydrocarbon as shown in Figure 1.

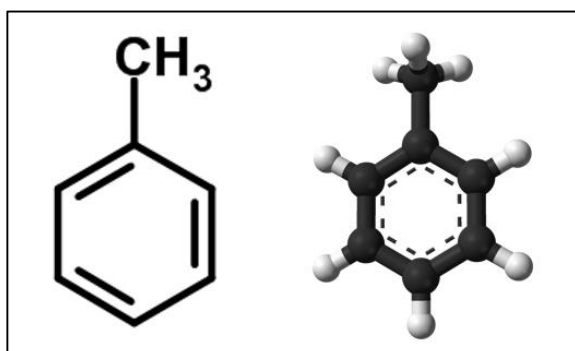


Figure 1. Structure of toluene.

Toluene behaves similarly to the other aryl hydrocarbons, which are widely used as solvents in industry and served as raw materials for manufacturing plasticizers and monomers and in polymers (Manahan, 2000, pp.360). Therefore, toluene was used as a model compound for the VOC removal in this thesis.

3.2.1 Basic properties

Some basic properties of toluene are listed in the Table 1 below.

Table 1. Physical properties of toluene (Sigma-Aldrich).

Toluene (CAS-No. 108-88-3)	
molar mass:	92.14 g/mol
density:	0.865 g/mL (at 25 °C)
form in nature:	liquid
color:	colorless, transparent
odor:	aromatic, sweet, pungent
melting point:	-93 °C

boiling point:	111 °C
vapor pressure:	29.1 hPA at 20 °C
water solubility:	0.5 g/L at 15 °C

These data were collected by using material safety data sheet (MSDS) from Sigma-Aldrich Company.

3.2.2 Source of toluene pollutant

As an example of VOCs, toluene shares similar sources with other VOCs, which include “paints, thinners, perfumes, hair sprays, furniture polish, cleaning solvents, carpet dyes, glues, dry cleaned clothing, air fresheners, candles, soaps, bath oils, molds, tobacco smoke” (Cooper and Alley, 2011, pp.698). Regarding tobacco smoke, 1,260 µg toluene is produced from smoking one cigarette (Griffin, 2007, pp.296).

3.2.3 Health effect of toluene

According to the MSDS (Sigma-Aldrich) in Appendix A, toluene is not considered as a hazardous but only dangerous chemical. However, it is necessary to take into account its properties including flammability when conducting experiment. Precaution must be taken and exposing toluene to high temperature must be avoided.

Toluene is also classified as one of the indoor air pollutants due to its occurrence from many of the normal household materials. Toluene smells unpleasant which is similar to the smell of super glue. Contacting toluene at low level in short time will only irritate nose and throat. Reports (CCOHS, 2015) have shown that inhalation of toluene at low level in longer period may cause tiredness, weakness, dizziness, drunken-type actions, and even loss in hearing, color vision, and memories. Fortunately, these symptoms are classified as chronic effects which disappears immediately when exposure is stopped. CCOHS (2015) also stated that inhaling toluene at high level in short time can cause nausea, sleepiness, and unconsciousness. In addition, inhaling toluene at high levels for a longer period can cause serious health effects. Research testing with Long-Evans rats (Kodavanti P. et al, 2015) shown brain and neuro damages and degenerative effects due to the toxicity of inhaling toluene.

3.3 Adsorption

The removal of gases and vapors typically at low concentrations at the end point of the exhaust stream can utilize the application of adsorption. Adsorption is defined as the phenomenon when molecules of a fluid or dissolved solid attach, or adhere to the surface of a collecting medium (usually solid). The material cause adsorption is called the adsorbent and the fluid being adsorbed is known as the adsorbate. The Adsorber, or the adsorption system, works on the principle of molecular or atomic interaction at its surface. It is different from absorption where the absorbed materials are dissolved into the absorbent while adsorbed materials are only attached onto the adsorbent surface. (De Nevers, 2000, pp.354; Wang, Pereira and Hung, 2004, pp.395) The term desorption is the reverse process of adsorption. The application of adsorption is usually dealing with gas phase air pollution and is usually carried out at a low vapor pressure. This thesis is mainly focused on the activated carbon and toluene as the adsorbent and adsorbate.

3.3.1 Adsorption theory

Adsorption can actually occur at the surfaces of any solids. However such adsorption is very small unless the solid is highly porous and has the structure like fine capillaries. In other word, the internal surface area, or the surface-to-volume ratio refers to the ability of adsorption for a solid material. The larger the internal surface area of an adsorbent, the higher the efficiency on adsorbing the adsorbates.

According to Wang, Pereira and Hung (2004, pp395-396), adsorption can take place in several specific means. It can be used effectively for separating gases from gases, solids from liquid, dissolved gases from liquid, and ions from liquid. He continue explained that during the process of adsorption, it involves three steps of contact, separation, and regeneration. When a fluid passes through an adsorbent, it is firstly adsorbed at the surface of the adsorbent as they get contacted. At this point, the composition of the fluid will change where one or more of its components are adsorbed. Then followed by the separation where the unadsorbed components pass through the absorbent and leave the adsorbates on the surface of the adsorbent. In the final step of regeneration, or desorption process, the adsorbates are removed from the adsorbent and collected somewhere else. In other word, regeneration is the cleaning process for the adsorbent. This can be done by applying heat to remove the adsorbates.

In the case of this work, it is interested in studying the adsorption for separating vaporized toluene from the normal indoor air using activated carbon filter. And the step of regeneration is carried out by using a thermal desorber (TD) at elevated temperature to clean the tenax tube which will be discussed in the later methodology section.

3.3.2 Physical and chemical adsorption

Theodore (2008, pp186-187) explained that adsorption mechanism is distinguished by physical and chemical adsorption due to the different manner of bonding between adsorbate and adsorbent. The processes of both adsorptions are considered to be exothermic as the gas molecules lose their kinetic energy when attached to the adsorbent in the form of heat. In the case of this work, activated carbon filter is used as a physical adsorption application.

Removing VOCs by using activated carbon adsorbent is an example of physical adsorption, also known as van der Waals adsorption. It involves a weak bonding of adsorbate (gas molecules) to the adsorbent (solid) resulted from the intermolecular cohesion. This weak bonding is akin to the attraction forces between two molecules in a liquid. The chemical nature of the adsorbed fluid remains unchanged in physical adsorption. As a result, it is a readily reversible process. Applying heat or reducing the pressure can break the forces holding the gas molecules to the solid in physical adsorption. Therefore, heating or reducing the pressure in the adsorbers can be used to regenerate or to clean the adsorbents. (Cooper and Alley, 2011, pp387; Theodore, 2008, pp186-187) The enthalpy changes in physical adsorption are small that the typical range is between 10 and 40 kJ/mol (Thomas and Thomas, 1997, pp65).

Physical adsorption can be also described by the polarity of the molecules of the fluid adsorbate and solid adsorbent. According to the chemical structure, molecules can be either polar or nonpolar. The polarity of the molecules of the solid adsorbent surface determines the type of fluid adsorbate it can adsorb most likely. Polar adsorbents adsorb polar adsorbates and nonpolar adsorbents adsorb nonpolar adsorbates. For example, activated carbon is generally a nonpolar adsorbent, thus, it is used to adsorb nonpolar adsorbates such as most of the organic compounds. Besides, polar adsorbents are used preferentially adsorb water vapor for instance. However, in certain situ-

ation, activated carbon can be also applicable when dealing with polar adsorbates. (Theodore, 2008, pp186-187)

Chemical adsorption, or chemisorption, involves chemical bonding (similar to covalent bonding) from the reaction within the adsorbate and adsorbent where the electrons are being rearranged. Examples include the oxidation of SO_2 to SO_3 on activated carbon which involves chemical reactions (Cooper and Alley, 2011, pp387). Such bonding is much stronger than physical adsorption and is not easily reversible (Theodore, 2008, pp186). The heat of chemisorption is usually larger than physical adsorption which often exceed 400 kJ/mol (Thomas and Thomas, 1997, pp65).

3.3.3 Pore size distribution

Several physical properties indicate the adsorption behavior of an adsorbent. According to Theodore (2008, pp188-190), these properties includes the surface area, internal porosity, pore volume, mean pore diameter, and bulk density. It is generally described that the larger the surface area the better the adsorption effectiveness. However, it should be noted that the effectiveness of adsorption is also determined by adsorbent pore size. If the pore size is not suitable, the adsorption works poorly no matter how large the surface area is. Theodore stated that “the surface area must be available in certain pore sizes if it is to be effective as a vapor adsorber”.

To explain the pores characteristic begins with understanding their size range. According to the sizes of pores, they are classified into micropores, transition pores and macropores. Pores having diameters of 400 nm or less are micropores and larger than 4000 nm are macropores. Transition pores are those in between this range. (Theodore, 2008, pp188)

Adsorption works the best if the pore size of the adsorbent is relatively similar to the size of the adsorbates or gaseous molecules. When the pore size of the adsorbent is smaller than the size of adsorbate, the adsorbate is unable to reach these surfaces. As a result, these pores cannot adsorb the adsorbates. Moreover, the larger pores (macropores) capture adsorbates with size much smaller than themselves inadequately. This is resulted from that these larger pores serve simply like a passageway to the areas with smaller pores for adsorbates. (Theodore, 2008, pp188) Therefore, the efficiency of activated carbon adsorption depends on its surface accessibility. Figure 2

below illustrates the distribution and behavior of different pore sizes in typical activated carbon adsorbent.

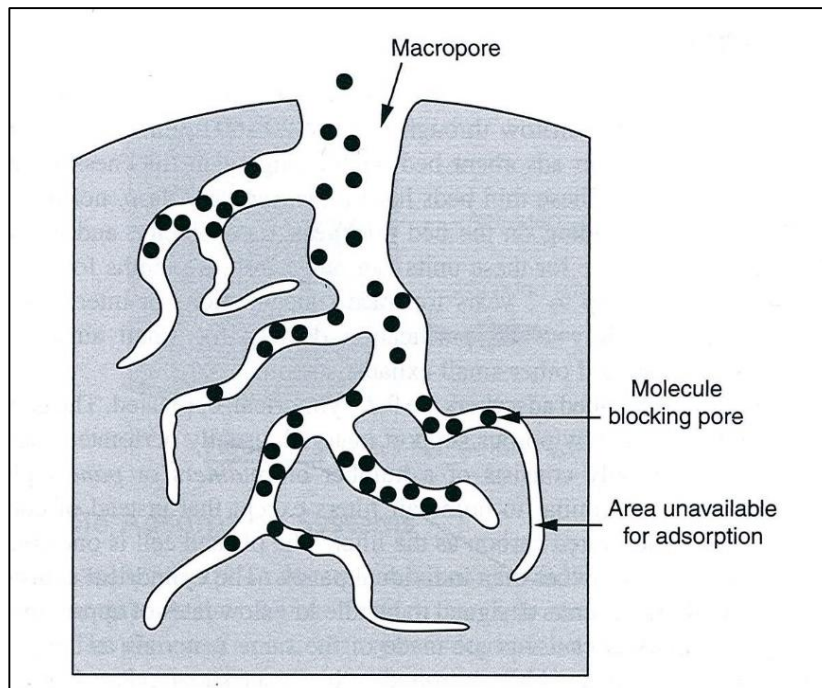


Figure 2. Pore size distribution of activated carbon (Theodore, 2008, pp189).

As described, typical air pollutant adsorbates are much smaller than macropores, therefore these molecules are unlikely to be adsorbed on the surface of macropores and will continue travel toward smaller pores. In the meantime, large molecules will be blocked at the area before entering small micropores. To be more exact, Theodore (2008, pp189) also claimed that “adsorption forces are strongest in pores whose sizes are not more than roughly twice the size of the adsorbate”

3.3.4 Regenerable and nonregenerable adsorption system

Adsorption system can be either regenerable or nonregenerable. Regenerable means that the adsorbent can be used several times after regeneration process. Nonregenerable system refers to those serves like a disposable adsorbent. In general, regenerable adsorption system is used to treat contaminates at higher concentration in cases like industrial emissions. There is usually two adsorption beds in regenerable system. When one of the bed is consumed, the adsorbates are redirected to the second bed. The consumed bed is then desorbed as described in previous section and become ready to use later. Such system allows the adsorption beds to function automatically

and sustainably. A nonregenerable system does not require the process of desorption. Adsorbents in such system are simply replaced, usually manually, by another new one once it is consumed up. Nonregenerable systems are typically used for removal of contaminants at very low concentration in unit of parts per million (ppm). The used adsorbent beds are then sent back to the manufacturer for recovery. (Theodore, 2008, pp190-194)

Regarding this work, activated carbon filter functioned as a nonregenerable adsorption system. After several experiment test, those filters were replaced manually by new ones. These activated carbon filters used during the experiment of this thesis were not necessary provided from Genano.

3.3.5 Adsorbent

Adsorbents are typically composed of either natural or synthetic materials allowing them to have a microstructure where the surfaces of the internal pore are accessible to the gas phase pollutants (Griffin, 2007, pp164). Adsorbents exist on the market are available in great variety. Most common adsorbent material used is activated carbon which can be used in various applications. Other adsorbent materials include alumina, silica gels, and bauxite. And these adsorbents are usually regenerable and can be used several times in the adsorbers. (Cooper and Alley, 2011, pp392; Griffin, 2007, pp164) Some general utilities of these adsorbents are described below in Table 2.

Table 2. General functions of some common adsorbents (Wang, Pereira and Hung, 2004, pp396).

Adsorbent	Utility
Activated carbon	solvent recovery, elimination of odors, purification of gases
Alumina	drying of gases, air, and liquids
Silica gel	drying and purification of gases
Bauxite	treatment of petroleum fractions, drying of gases and liquids

Regarding indoor air purification, alumina, silica gel, and bauxite are less likely to be used in the adsorbers. The application of this study is to use activated carbon as the adsorbent to eliminate bad odor and purify the air from toluene gas existing in indoor environment.

3.4 Activated carbon filter

Activated carbon, also known as activated charcoal, is a form of carbon used as an adsorption material. Activated carbon has very large surface area due to its high degree of microporosity inside a small volume. According to Cooper and Alley (2011, pp392), the term “activated” regarding adsorbent refers to “the increased internal and external surface area imparted by special treatment process”. Such treatment is done by applying heat in the form of steam, air, or carbon dioxide where these gases violate the carbon and cause it to increase its pore structure.

Adsorption of activated carbon is an example of physical adsorption. As described previously, activated carbon’s nonpolar surface is useful on controlling emission of nonpolar organic solvents, VOCs and gasoline vapors. In practice, it can be also used to remove unpleasant odors especially from indoor.

Activated carbon is prepared and packed in different forms according to each purposes and work basically like a filter to remove contaminates in the air. It works on the principle where activated carbon remove contaminants from liquid or vapor streams such as VOCs for the approach in this study.

3.4.1 Physical properties

As explained in the previous section, several physical properties effect the behavior of adsorbent. These properties for activated carbon are listed in the Table 3 below.

Table 3. Physical properties of activated carbon (Theodore, 2008, pp189).

Adsorbent	Surface area (m ² /g)	Internal porosity (%)	Pore volume (cm ³ /g)	Mean pore diameter (nm)	Bulk density (g/cm ³)
Activated carbon	600 - 1600	55 - 75	0.80 - 1.20	150 - 200	0.35 – 0.50

Activated carbon adsorbents have pore size between 150 and 200 nm which is suitable for application as micropores. As described earlier, Theodore (2008, pp189) claimed that adsorption forces perform most efficiently when the size of adsorbent pore is around doubled of the size of adsorbate. In addition, most gaseous molecules from air

pollutants, including VOCs, are having diameters in the range of 80-180 nm. Also, nearly 95% of activated carbon pores are micropores. Therefore, activated carbon can remove most VOCs adequately. (Theodore, 2008, pp189-190)

3.4.2 Types of activated carbon

Activated carbon can be made from carbonizing various raw materials such as coal, petroleum-based products, wood, coconut, and nutshells. Carbonization is the process of heating the material to an elevated temperature (usually 600 °C) to drive away all volatile material leaving desired carbon behind. (Theodore, 2008, pp187) Activated carbon made from different raw material and carbonizing method will behaves differently. According to different interest and purposes from manufactures, the parameters producing activated carbon vary. Based on the preparation methods, size, and applications, typical types of activated carbons include powdered, granular, extruded, and impregnated activated carbons. Impregnated activated carbon (IAC) is the type of carbon used in this study and will be described in the later section.

Powdered activated carbon (PAC), as its name, it is the carbon that being crushed or grinded into fine particles like sand dust. PAC is made from organic materials such as wood, lignite and coal giving it very high carbon content. According to EPA (2015b), PAC is typically manufactured with a diameter less than 0.1 mm. PAC has very small particle size PAC is widely used in water treatment to adsorb contaminates, taste and odor in the water (EPA, 2015b). Granular activated carbon (GAC) is the carbon in granular form that looks like little rocks and has relatively larger size compared to PAC. Raw materials used to manufacture it include bituminous coal, peat, coconut shells, wood, and lignite. It is mainly used in water treatment process. (AWWA, 2012) Extruded activated carbon (EAC), also classified as one form of GAC, is the carbon being extruded or compressed into cylindrical shape like pellets (AWWA, 2012).

3.4.3 Impregnated activated carbon (IAC)

“Impregnated activated carbons are carbonaceous adsorbents which have chemicals finely distributed on their internal surface. The impregnation optimizes the existing properties of the activated carbon giving a synergism between the chemicals and the carbon. This facilitates the cost-effective removal of certain impurities from gas streams.” (IJST, 2009).

The appearance of most of IAC is similar to a sponge or foam. As a result, it is also known as the foamed activated carbon. One obvious difference between different IACs, depending on their component and manufacturing procedure, is their softness. Some IAC filters are very soft like a sponge while some are more solid.

3.4.4 Activated carbon adsorption and saturation stage

Carbon adsorption is the process which certain pollutants are adsorbed selectively at the surface of activated carbon adsorbent. Study (Wang, Pereira and Hung, 2004, pp396) has shown that activated carbon is one of the most effective adsorbent for removing generally all the organic vapors such as VOCs, regardless of distinction in concentration and humidity. Wang, Pereira and Hung (2004, pp396-397) explained that adsorption by activated carbon happens in two stages, adsorption and saturation stages.

During the adsorption stage, fresh activated carbon filter adsorbs fluids effectively and quickly in the beginning. Activated carbon adsorbent increases its weight as adsorbates are adsorbed onto it. After adsorbing for a certain time, the activated carbon is no longer fresh, it is getting saturated with adsorbates and reaches a point where the adsorbing rate decreases rapidly. This point is recognized as the breakpoint. After exceeding such point, the saturation stage appears. (Wang, Pereira and Hung, 2004, pp397)

When saturation stage is obtained, activated carbon is said to be completely saturated with adsorbates (vapors) and stops adsorbing anymore adsorbates and ceases to increase in weight. At this stage, fluid at the inflow will simply pass through the activated carbon filter. As a result, the concentration of the selected fluid at the inlet and outlet of the stream appears to be the same. Therefore, saturation stage is also referred as the adsorption equilibrium for the adsorber system. The point of saturation stage is different for each selected fluid and activated carbon at different conditions (e.g. temperature, pressure, airflow rate, etc.). Therefore, saturation value, or the point of adsorption equilibrium for activated carbon must be determined experimentally. (Wang, Pereira and Hung, 2004, pp396-397)

The maximum amount of adsorbates the activated carbon can adsorb at the saturation stage is recognized as the adsorption capacity. Adsorption capacity is defined at a given temperature and pressure. Wang, Pereira and Hung (2004, pp397) defined that “the retention capacity of an activated carbon is expressed as the ratio of the weight of the adsorbate retained to the weight of the carbon”.

3.4.5 Adsorption equilibrium and isotherms

To determine the behavior of adsorption system, the adsorption system must be conducted under equilibrium conditions. As described earlier, adsorption equilibrium is defined as the condition when sufficient time is applied for adsorbates contacting with adsorbent, the amount of adsorbate molecules coming from the inflow attached to the surface of adsorbent is equal to the amount of adsorbate molecules leaving the adsorbent. Adsorption equilibrium can be also referred as the saturation stage where the adsorbent is saturated with adsorbates and can remove no more contaminants. Thus, adsorption equilibrium is used to determine the capacity, or the maximum amount of adsorbate molecules, or vapors that an adsorbent can hold at a given condition including temperature and pressure. Stated by Cooper and Alley (2011, pp387), “generally, the capacity of an adsorbent is directly proportional to the molecular weight and inversely proportional to the vapor pressure of the adsorbate.”

According to Theodore (2008, pp194-195), three types of equilibrium relationship, isotherm, isobar, and isostere, are used to express the adsorption equilibrium. Isotherm means at constant temperature; isobar means at constant pressure; and Isostere means at constant amount of vapors adsorbed. Among these three methods, Theodore claimed that isotherm is the most commonly used for describing adsorption equilibrium data. Theodore described that “The isotherm is a plot of the adsorbent capacity vs. the partial pressure of the adsorbate at a constant temperature.” Isotherm represents the capacity of an adsorbent for a specific gas or vapor which is usually expressed in weight percent as grams of adsorbate per certain grams of adsorbent.

Generalized equations are difficult to develop for predicting the behavior of adsorption isotherm. This is due to the fact that adsorption is very complex and sensitive and the shape of the isotherm graph varies from a curve to S-curve and other shapes depending on each adsorption system. Therefore, adsorption equilibrium must be determined

experimentally. (Theodore, 2008, pp194-195) Figure 3 shows an example of adsorption isotherm for activated carbon.

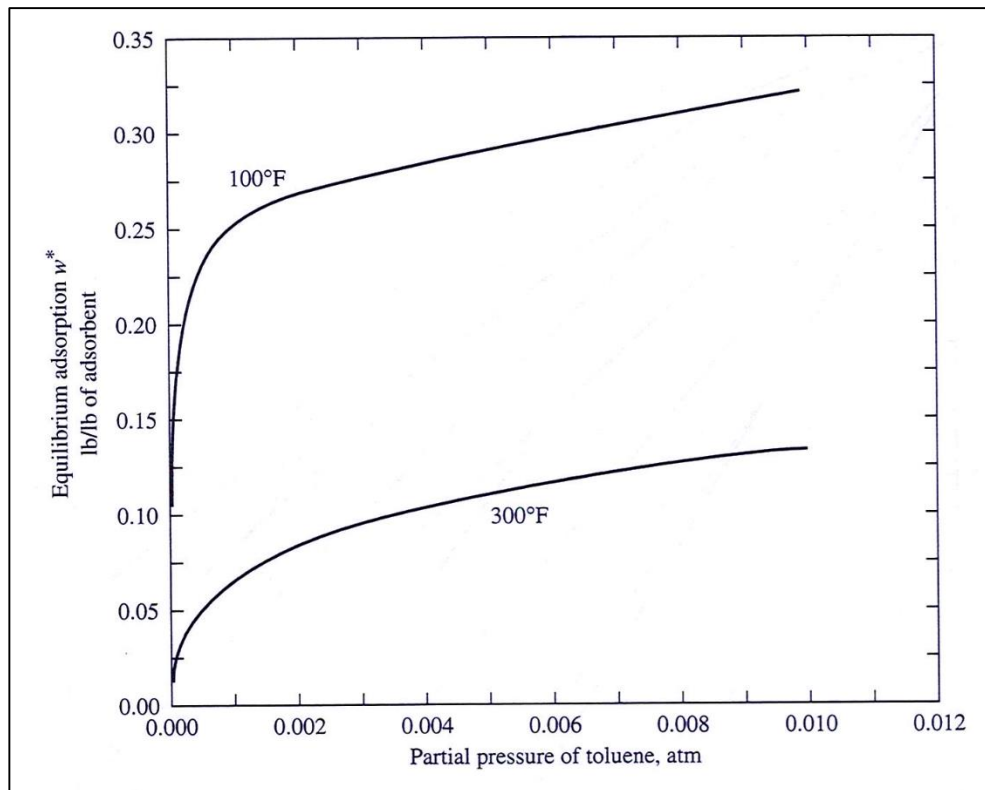


Figure 3. Adsorption isotherm for toluene on activated carbon (De Nevers, 2000, pp358).

In addition, there are several existing mathematical equations or models designed to express the adsorption equilibrium and each of them are used specifically in different cases. According to Cooper and Alley (2011, pp387-389), Langmuir model would be one of the best model for describing isotherm. Foo and Hameed (2009) described Langmuir isotherm model as a model developed for gas to solid phase adsorption onto activated carbon. They explained that Langmuir model deals with “homogeneous adsorption which each molecule possess constant enthalpies and sorption activation energy with no transmigration of the adsorbate in the plane of the surface”. Langmuir isotherm equation is written as below.

$$W = \frac{k_1 P_p}{k_2 P_p + 1} \quad (\text{Cooper and Alley, 2011, pp389}) \quad (1)$$

where P_p is the adsorbate’s partial pressure. W is equilibrium adsorptivity, mass of adsorbate adsorbed per unit mass of adsorbent. And k_1 and k_2 are coefficient constants.

When the adsorbate partial pressure is very low, the value of K_2P_p is approximately equal to zero, and then Eq. (1) could be rewrite as

$$W_{low} = k_1P_p \text{ (Cooper and Alley, 2011, pp389)} \quad (2)$$

Equilibrium adsorptivity or capacity W represents the maximum amount of adsorbate the adsorbent can hold at a given temperature and partial pressure. However, in practice, the activated carbon filter lost its function as it reaches the saturation stage. Activated carbon filters are then replaced before they reach equilibrium. Thus, the actual capacity, or working capacity of activated carbon system is smaller than the theoretical equilibrium capacity. Working capacities can range from 10 - 70% of the equilibrium capacity (Wang, Pereira and Hung, 2004, pp396-397; Theodore, 2008, pp196). In other word, the equilibrium capacity also indicates the life time of activated carbon filters.

However, calculating the adsorption equilibrium and life time of activated carbon filter is not the focus of this study since every set of test on the filters were replaced by a fresh one. This report will focus more on the experimental work of testing the removal efficiency of toluene with designed conditions.

3.5 Gas chromatograph mass selective detector

Gas chromatography (GC) is one type of chromatographic analysing method dealing with gases or vaporized substances. GC can be used for identifying different components of a mixture by separation. As explained by Agilent Technologies (2002, pp10), "GC creates a time separation rather than normal physical separation such as filtration and distillation." GC allows the analysing vaporized mixture of gaseous sample to be separated in time when pass through the tube which contains a material that retards some components more than others. The detected result shown on a computer is a plot of chromatogram. Each peaks with their corresponding retention time on the chromatogram represent different components of the original mixture. The retention time of each component is distinctive and thus can be used to identify each component. The peak size (area) measures the amount of the component and can be later calculated into concentration for example. (AT, 2002, pp10)

The basic system of a GC system consists the source of carrier gases, an injection port, a column, a detector, and a corresponding data interpretation program on a computer. The flow chart of a GC is shown as in Figure 4. In this thesis, the carrier gas is helium, the injection port is thermal desorber, the GC is capillary column, and the detector is mass selective detector.

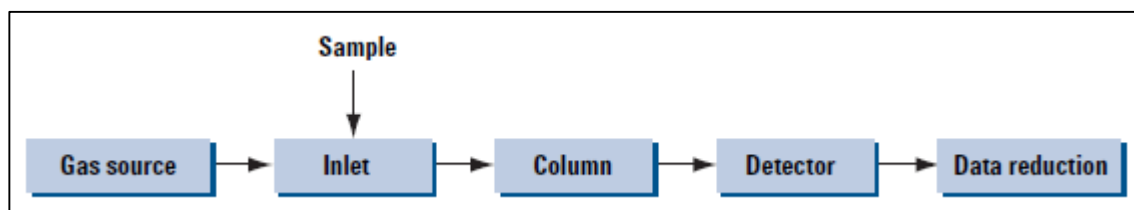


Figure 4. Flow chart of a gas chromatography system (AT, 2002, pp11).

GC works on the procedure of that the analysing sample is first injected and vaporized at the injection port and then being transported by carrier gas to the column. The sample is heated up at a very high temperature inside the column. Part of the sample may already evaporates to the surrounding at this point. As it continues its path by the carrier gas, it reaches the detector. The detector will then detect the left over sample where the data is transmitted and displayed on a computer. After being detected, the rest of the sample will evaporate through the escaping hole to the surrounding.

3.5.1 Carrier gas and tubing

In GC, gaseous sample is transported through the tubing in the column by a gaseous mobile phase which is known as the carrier gas. Carrier gas must be supplied along the gas lines into the GC in order to transport analysing samples throughout the analysing process. The most common carrier gas is helium which is compatible with most GC system. According to Harris (2010, pp575), “Gas flow through a narrow column may be too low for best detector performance, so extra *makeup gas* is sometimes added between the column and the detector. Makeup gas that is optimum for detection can be a different gas from that used in the column.”

In this thesis, helium gas and air gas tanks were used as the carrier gas and extra *makeup gas* respectively for the GC system.

3.5.2 Injection port

Inject port introduces and vaporizes the sample into the carrier gas stream along the pipe line. The choice of injection port depends on the type of column (capillary or packed, which are described in the next section) being used. Packed column uses packed port while capillary column uses split/splitless port.

Split/splitless port has two operating mode, split and splitless. Due to the property of capillary column which has very small capacity for samples, sample injected to column must avoid overloading it. Handling such small sample size is usually difficult. As a result, the split mode which transfers only part of the sample to the column, is used control and provide small enough sample. The rest of the sample is vented to waste. Split mode is used also when one wants to keep the sample, due to, for example the difficulty of preparing the same sample, and the residual part is not being vented to waste. This can be done when using a tenax tube, which will be discussed in the later section. For splitless mode, it is well suited with low concentration samples, so that the sample amount does not need to be reduced. (AT, 2002, pp17-22) Figure 5 below illustrates the port in splitless mode.

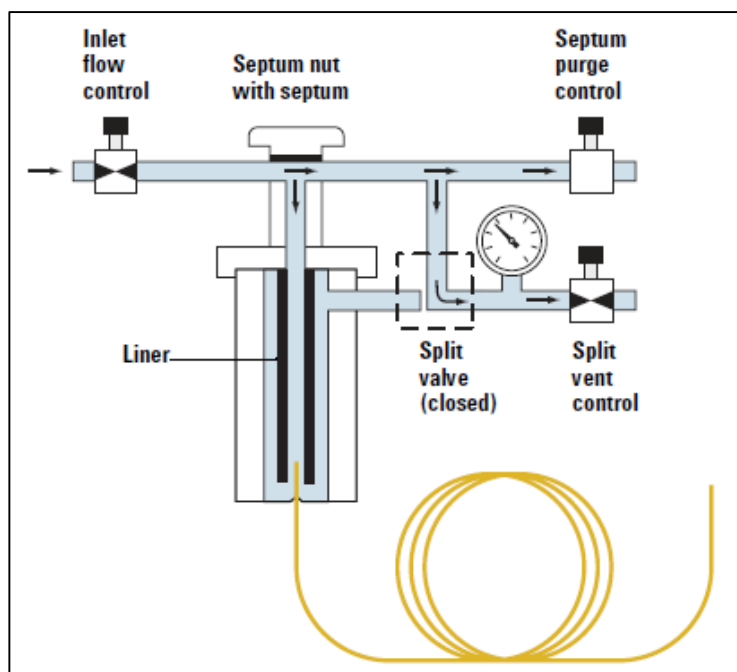


Figure 5. Split/splitless port in splitless mode (AT, 2002, pp21).

The split valve in Figure 5 can be managed by computer program in which closing the valve for splitless mode and opening the valve for split mode.

3.5.3 Column

There are several types of columns, such as capillary and packed columns, which are selected for different performance. However, they all work on the similar principle. Typical column is shown in Figure 6.

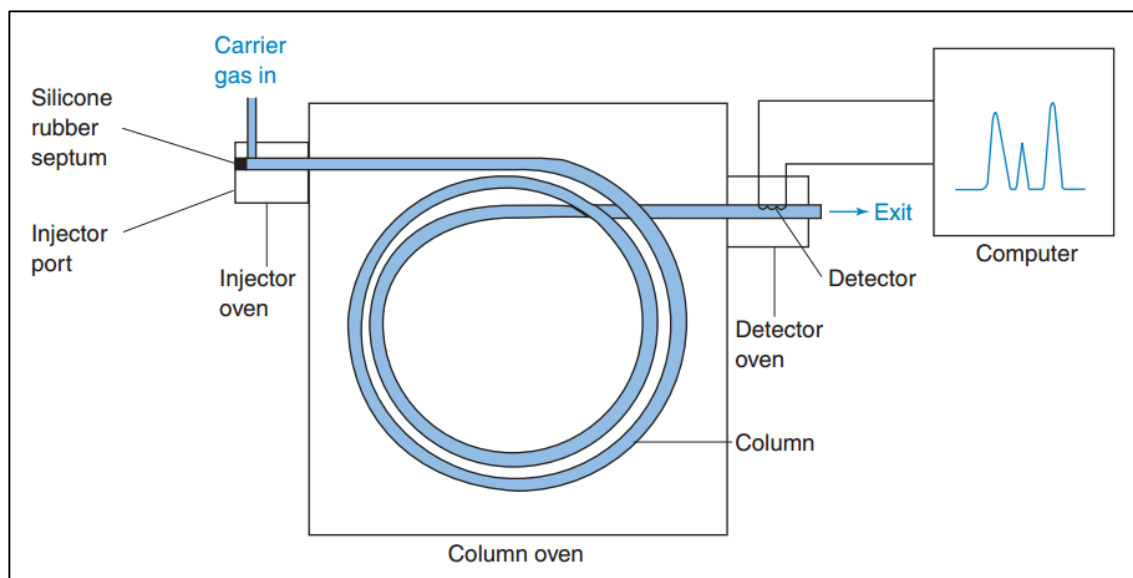


Figure 6. Schematic diagram of a gas chromatograph column (Harris, 2010, pp566).

The sample separation is carried out in the column as the column oven is heated to around 300 °C to obtain the gaseous sample in mobile phase. The coating on the inner surface of column diffuses the components of the gaseous sample as they were carried by the carrier gas. Different types of coating have different characterises. These diffusing forces can be “solubility, volatility, polarity, specific chemical interaction, or any other property that differs from one component to another” (AT, 2010, pp29). The component easily attracted by the coating will move slower than those invulnerable ones. As a result, different components are separated by their speed which is then detected as in different retention time. (AT, 2002, pp28-29)

The length of column can be up to 100 m where the common length is around 30 m. (Harris, 2010, pp567) Such long pathway allows the sample to be heated evenly with sufficient time. In order to reduce volume and ease the heating, the column piping is looped in a circle around a frame as shown in Figure 7 below. The inner diameters of columns are typically between 0.10 and 0.53 mm. According to Harris (2010, pp567),

“Narrow columns provide higher resolution than wider columns but require higher operating pressure and have less sample capacity.” The difference in thickness and length will alter the retention time. The retention time increases as the thickness is increased or the length is reduces. (Harris, 2010, pp566-567)

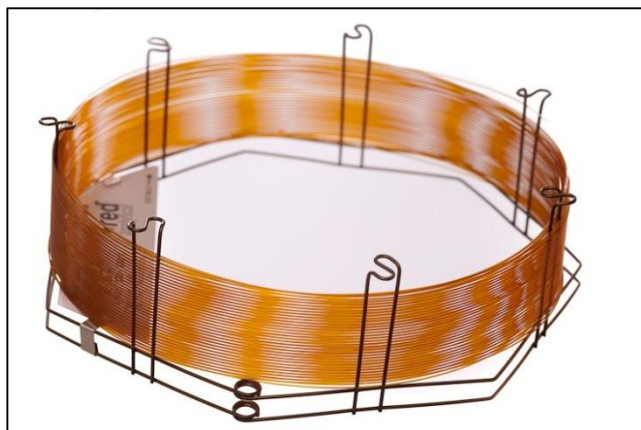


Figure 7. Fused silica capillary gas chromatography column (Dot red, n.d.).

A capillary column, also known as open tubular columns can finely separate complex samples by providing very narrow peaks. In general, capillary column performs better than packed columns as they can deal with various conditions and separate samples in more detailed and need only a small amount of samples. The more narrow the peaks, the better the efficiency. (AT, 2002, pp30) However, when dealing with large amount of gas sample, a packed column could be more suitable (AT, 2002, pp35).

3.5.4 Detector

There are several types of detectors including thermal conductivity, flame ionization, electron capture, and mass selected detector. Detector analyses the individual components which has been diffused from column. Detected components are compared with the detector's spectra library (Harris, 2010, pp579). Thus, each component can be identified and the data are collected in the computer program. Mass selected detector (MSD), also called mass spectrometry, is a sensitive detector that provides both quantitative and qualitative information of the detecting samples. MSD can detect even very small amount of samples in ppb and ppt level with detailed narrow peaks. (Harris, 2010, pp582-584)

3.6 Tenax tube

Tenax tube is also known as thermal desorption sorbent tube as it is a matching part to the thermal desorber. It works as a media to store and transport the sample which can be later inserted into the injection port of thermal desorber. Tenax tube is shown in Figure 8 below. The left side of the tenax tube that has a ring of groove as shown in Figure 8 is the head while the right side is the tail. Tenax tubes look the same which can be confusing when recording the data, however their identical numerical coding help to distinguish them from each other.



Figure 8. Tenax TA tubes manufactured from Marke International. Each tubes are labeled with numerical code. The side with a ring of groove is the head of the tenax tube.

Tenax tube has adsorbent inside which can trap volatiles and semi-volatiles from air or water. Tenax TA is the type of tenax tube used in this study. According to SIS (n.d.), “tenax TA is a porous polymer resin based on 2.6-duphenylene oxide”. Tenax TA is a good trapping agent with very low level of impurity. More information can be found in Appendix C.

4 Methodology

In order to analyze the behavior of activated carbon filters on removing toluene pollutant, a system was created to demonstrate this indoor air purifying process. A model was designed and built to carry out this approach. Activated carbon filters with one, two, and three layers were made and installed and tested in the model individually. Tenax TA tubes were used to take samples from the model. The samples were then analyzed by TD-GCMSD. To decide the parameters for operating the system, pre-work was carried out with several trails.

The activated carbon type used is impregnated activated carbon (IAC) with PUR substrate (See Appendix B). Thermal desorber gas chromatography mass selective detector (TD-GCMSD) was used for analyzing the toluene samples collected by the tenax tubes. Capillary column was used for the GC. The injection port was thermal desorber and the detector is the mass selective detector. The model of GC system used in this study is HP 6890 series combined with a MSD HP 5973. The model of thermal desorber used is UNITY (serial No. GB00U10282) manufactured from Markes International. The TD-GCMSD device used is shown in Figure 9 below.



Figure 9. TD-GCMSD.

The analyzing program installed in the computer is MSD ChemStation (version G1701DA D.01.02.16 15-JUNE-2004) from Agilent Technologies and Thermal Desorption System Control program (TDSC program) (version 2.0.0) from Markes International. TDSC program is used to control the thermal desorber while MSD ChemStation is used to operate the GCMSD. The instructions for using TD-GCMSD and these two programs to analyse and clean the tenax tube are explained in detailed in Appendix F and G.

5 Experimental work

5.1 System and model

As described, the purpose of this experimental work was to test the activated carbon filter behavior on removing toluene pollutant in the air stream. The system was designed to demonstrate such approach. The model is shown in Figure 10. More pictures can be found in Appendix D.

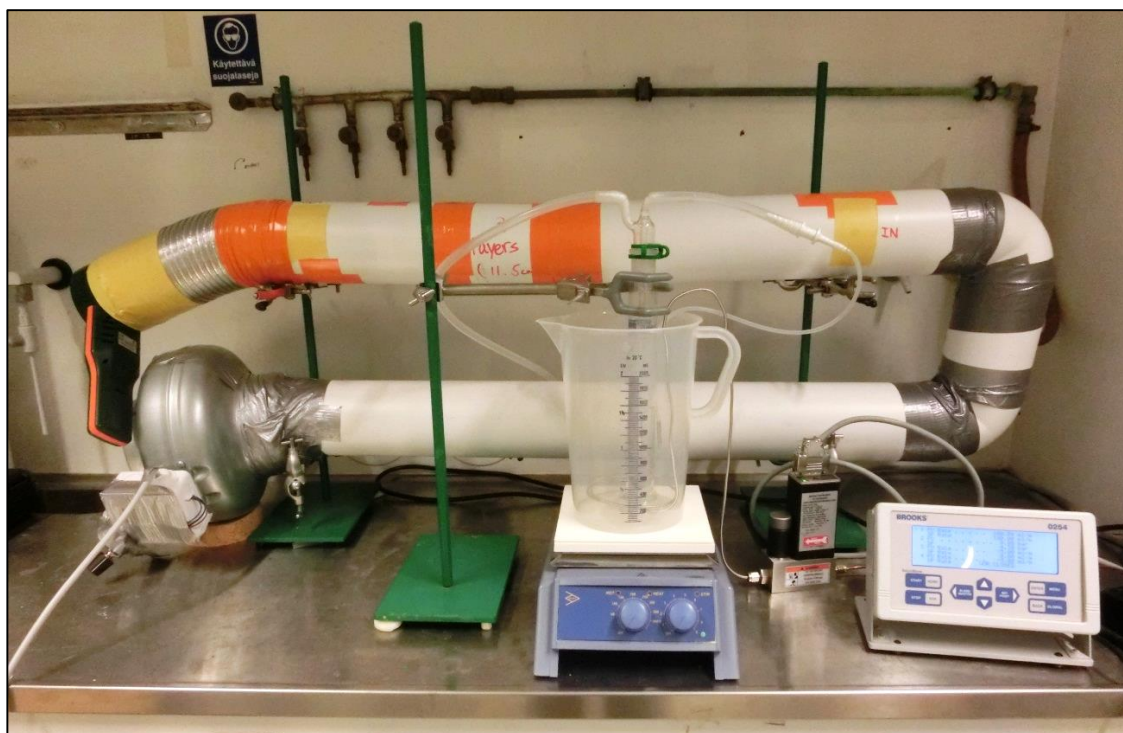


Figure 10. Experimental system and model.

The model was constructed with PVC pipes having an inner diameter of 10 cm. The model was built in a U-shape due to the size of the available fume cupboard. The pipe line needed to be long enough to enable the airflow being mixed and stabilized. Two mixers were fixed inside the pipe line before and after the U-turn to help stabilizing the airstream. An airflow meter was attached to the other end of the pipe. Airflow meter was used to measure the air speed in order to adjust the fan. Every joints and openings of the pipe system were fixed and sealed by duct tape. To eliminate possible secondary toluene pollutant from duct tape, parafilm was used to wrap the joints and cover the openings before taping.

Toluene gas was prepared and fed into the pipe model through silicon tube. Toluene (Riedel-de Haen, min. 99.7%) was placed in a measuring cylinder which was coupled with a Dreschel bottle head as shown in Figure 11. By using the silicon tubes, one end of the gas bubbler was connected to nitrogen gas tank and the other end to the pipe model. Nitrogen gas generated toluene being bubbled and vaporized. The flow of the nitrogen was controlled by a flow meter (see Appendix D). A small magnet was placed in the measuring cylinder to help evaporate toluene evenly with the use of a plate stirrer.



Figure 11. Measuring cylinder coupled with Dreschel bottle head (sinter porosity grade 0) (Dreschel bottle heads, camlab, n.d.).

IAC filters were assembled at the top layer of the pipe system. IAC provided from Genano has a thickness of 2 cm (see Appendix B). These IAC were first cut into circles with diameter of 10 cm which can fit inside PVC pipes. Additional PVC pipes were cut into three cylinders with desired length of 7.5 cm, 11.5 cm, and 13.5 cm. IAC filters were made by fitting IAC into these PVC cylinders. Three IAC filters were made having one layer, two layers, and three layers of IAC of each. IAC were fixed inside the PVC pipes by PVC joint pipes as shown in Figure 12. These filters could be easily removed and displaced during the experiment. New parafilm and duct tape were always used when replacing a new filter.



Figure 12. IAC filters with one layer and two layers. IAC (black part) is placed in between two PVC pipe joints when fit into the corresponding PVC cylinders. One-layer and two-layer IAC filter have length of 7.5 cm and 9.5 cm. Three-layer IAC filter has a length of 11.5 cm.

Two openings were drilled before and after the filter section on the pipe for tenax tubes to take air samples. Samples taken from the opening before the filter on the right side is the inlet flow (IN). Samples taken from the opening after the filter on the left side is the outlet flow (OUT). These two openings were drilled to have the size that fits the tenax tube which is covered with silicon tube. These two openings were sealed with parafilm and duct tape.

The tenax tubes used are the TA type. They collect samples with the help of a pump for extracting air volume inside the pipe model as shown in Figure 13.

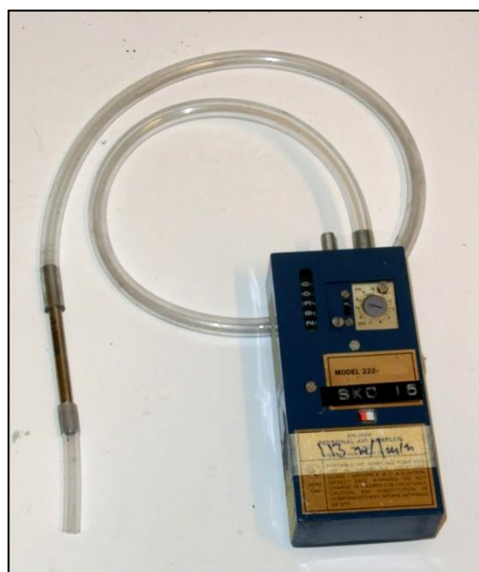


Figure 13. Tenax tube and pump. Silicon tube at the head is 6 cm long which enables it to reach the center point of the airstream in the pipe when inserting it into the opening for taking samples. (The length of the head part of tenax tube is 1 cm, and the radius of the pipe is 5 cm.).

The efficiency of the pump is 113 mL/min. The pump is connected by a silicon tube attaching to the tail of tenax tube. The head part of tenax tube is also connected to another silicon tube. This silicon tube provides an air tight function and is the part being inserted into the openings when taking samples. Moreover, samples must be taken at the center point of the airstream inside the tube. Therefore, silicon tube connected at the head must have a desirable length. After the sample was taken, the tenax tube was immediately sealed with the silicon caps on both ends and stored in ziploc bags before inserting to the TD for analysis.

5.2 Pre-work

In order to decide the parameters on preparing toluene fed into the pipe model (at inflow) which was desired to be small enough in the unit of ppm, several pre-works were conducted based on the result from TD-GCMSD analysis. These parameters included the nitrogen gas flow rate and the pumping time when taking sample by tenax tube.

Pre-work was carried out with changing in nitrogen gas flow rate and the pumping time. The filter used in the pre-work was one layer while the speed of fan which was kept constant with 1 m/s at all time. Samples were only taken at the inflow opening by the tenax tubes. Pre-work started with the smallest possible nitrogen gas flow which was 10 mL/min and increased up to 100 mL/min. The pumping time was tested from 1 min up to 10 min. After several rounds of trials, nitrogen gas flow rate and pumping time were concluded to be reasonable with 100 mL/min and 5 min respectively (see Appendix E). With this setting, the result shown roughly few ppm of toluene at the inflow before contacting the filter.

5.3 Experiment

Based on the result from pre-work, toluene was prepared with nitrogen flow of 100 mL/min and feed into the pipe model. Figure 14 below illustrates the conceptual schema of the model. The vaporized toluene was prepared by using nitrogen gas mixed with toluene liquid in a Dreschel bottle as explain in previous section.

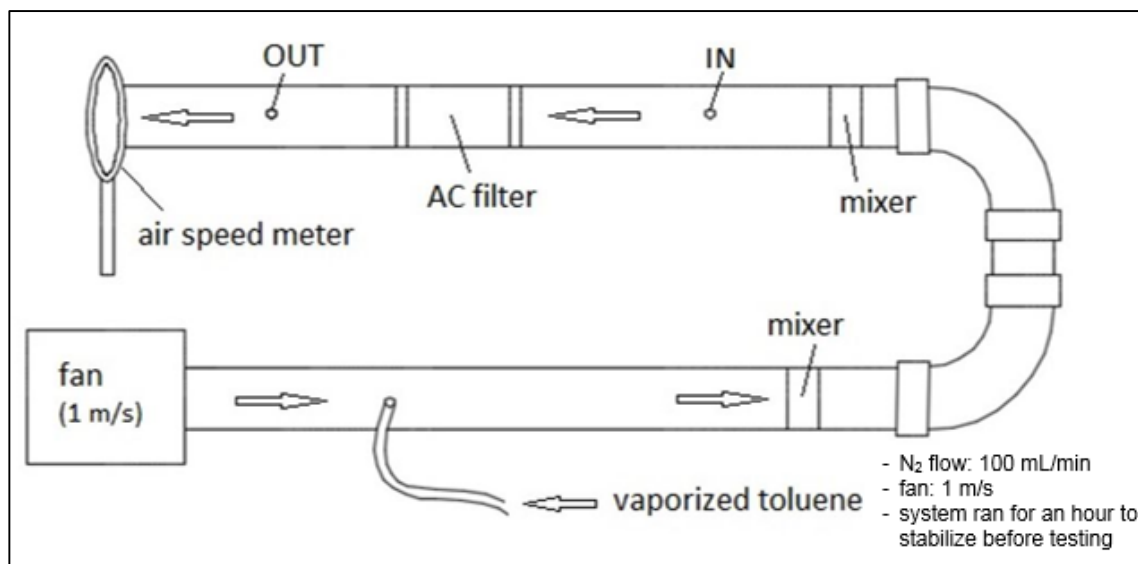


Figure 14. Illustration of the pipe model.

There were two different batch of IACs, September and October. These IACs were made into different layers of AC filters, as in one, two, and three layers. Each AC filter was tested separately. After installing an AC filter, nitrogen gas tank was opened, toluene vapor was feed in, fan was controlled to 1 m/s, the sampling openings were sealed, and the system was kept running for about an hour to stabilize. When one AC filter finished the test, another AC filter was replaced and carried out the same procedure. Due to the pressure drop caused by having more layers of AC filters, the power of the fan must be adjusted or increased to keep the reading on the air speed meter to be 1 m/s at all time. As a result, the system could be carried out in the same condition.

Three replicates were taken from both IN and OUT individually by the tenax tube for each AC filter. They were taken in the order of first one IN, second one OUT, third one IN, fourth on OUT, and so on. The opening was covered immediately after taking the sample by tenax tube. The time intervals between each sampling taking were less than 2 min. After the sample was taken, the tenax tube was immediately sealed with the silicon caps on both ends and stored in ziploc bags before inserting to the TD for analysis. Tenax tubes' numerical codes were recorded down to distinguish from each other and eased the data collecting.

There were two batches having three AC filters and three replicates were taken for both IN and OUT. Therefore, 18 samples (tenax tubes) were taken in one set of experiment. And testing both batches would need a total of 36 samples.

5.4 Analysis

Tenax tubes were analyzed based on the instruction in Appendix F. After tenax tube was been analyzed, it is regenerated or cleaned by the TD (see Appendix G).

The data were collected and recorded by using Microsoft Excel sheet. The areas from the GC graph were interpreted into unit of ppm. The unit conversion was calculated by using an online converter from Markes International (n.d.).

6 Result

The result for batch September (AC 1-3) was shown in Table 5 below (Experiment were carried out in the condition of N₂: 100 mL/min; fan: 1 m/s; pump time: 5 min; sample volume: 0.565 L). Three replicates were taken for each position of each AC filters and the average value were calculated and listed in the Table 5. More detailed data were recorded in Appendix I.

Table 5. Batch Sep. AC1-3 results (N₂: 100 mL/min; fan: 1 m/s; pump time: 5 min; sample volume: 0.565 L).

AC layer	position	average GC area	average mass (µg)	average concentration (mg/m ³)	average ppm
AC 1	IN	1017129356	12.48	22.10	5.84
	OUT	610021062	7.49	13.25	3.45
AC 2	IN	872315110	10.71	18.95	4.78
	OUT	674139293	8.27	14.65	3.71
AC3	IN	825547438	10.13	17.93	4.51
	OUT	901935102	11.07	19.59	5.04

Based on this result, the removal efficiency of IAC filter (batch Sep.) could be calculated.

$$efficiency = 1 - \frac{concentration(OUT)}{concentration(IN)} \quad (3)$$

Therefore,

Efficiency (AC1, batch Sep.) = 40%

Efficiency (AC2, batch Sep.) = 22.7%

Efficiency (AC3, batch Sep.) = -9.3%

The initial expectation was that the more AC filters, the better the removal efficiency. Surprisingly, this results showed that the more AC filters, the less the efficiency. However, the 40% removal efficiency shown a reasonable result which was similar to the Genano Oy Company's test.

Batch November was carried out following the same parameters and procedures. However, the results were incomprehensible where the concentrations of toluene at

OUT and IN were similar. Therefore, it was not appropriate to carry out the data analysis. More details could be found in Appendix J.

Due to the possible technical problem with the TD-GCMSD, personnel (Jussi Erkkilä) from Genano brought and used their own tenax tube to take few samples from the batch October. The result is shown in the table 6 below (also see Appendix K).

Table 6. Batch October AC2 result from Genanoy Oy Company.

AC layer	position	concentration ($\mu\text{g}/\text{m}^3$)	ppm
AC 2	IN	1550	4.78
		1760	5.43
		1963	6.06
	OUT	1160	3.58
		1580	4.88
		1790	5.52

Regarding the result from Genano Oy Company, batch October AC2 had an efficiency of 14.1 % on removal of toluene.

7 Discussion

The results from the TD-GCMSD throughout the experiment were inconclusive. After several test, the reliability of the devices were being doubted. Several attempts were made to solve the problem. However, they were not very effective.

All the device used were the best available from Leiritie campus. However, they were more than ten years old, and some were even twenty years old. Therefore, there could be possible experimental errors. TD-GCMSD was broken down during the experiment and technicians came to fix it. And this happened several times throughout the experiment. The trap and the ring in TD were too old.

Regarding tenax tubes, they have a life time of 100 times usage which means they could be used around 5 to 10 years. In addition, tenax TA tubes might not be the most suitable type of tenax tube for this study. However, these tenax TA tubes were the only available ones which were purchased more than ten years ago. Therefore, the test on the quality of tenax tubes was carried out by analyzing the empty tenax tubes after being cleaned by TD. The tenax tube should be empty inside and show no plot in GC graph after being cleaned. However, several tubes showed very poor results with high noises and contamination. These tenax tubes were then separated according to the GC graph in to broken tubes and usable tubes. The broken tubes were ready to be thrown away. Appendix H describes the criteria I made and also shows a list of usable tubes and bad tubes. The experiment was then carried out by using these usable tubes.

To improve this study, new TD and tenax tubes would be needed. Also, some modification on the pipe model could be set to eliminate any possible leaking.

8 Conclusion

It was unfortunate that the experiment could not be carried out according to the initial expectation. The results were inconclusive. However, if the devices were reliable, and the results were actually correct, it might be suggested that the more layers of AC filter or the thicker the AC filter, the less effective of the toluene removal. But I believe this could not be true.

Despite the irrelevant results, activated carbon filter showed its ability on removing VOCs like toluene vapor. And this pipe model was successful in preparing toluene vapor in low concentration.

Further research could be conducted on improving the system by using non-PVC pipes and eliminating all possible leaking at the joints and openings. Instead of tenax TA tubes, other types of tenax tubes could be examined.

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Appendix A. MSDS for toluene (Sigma-Aldrich)**Full text of H-Statements referred to under sections 2 and 3.**

Asp. Tox.	Aspiration hazard
Flam. Liq.	Flammable liquids
H225	Highly flammable liquid and vapour.
H304	May be fatal if swallowed and enters airways.
H315	Causes skin irritation.
H336	May cause drowsiness or dizziness.
H361d	Suspected of damaging the unborn child.
H373	May cause damage to organs through prolonged or repeated exposure.
Repr.	Reproductive toxicity
Skin Irrit.	Skin irritation
STOT RE	Specific target organ toxicity - repeated exposure

Full text of R-phrases referred to under sections 2 and 3

F	Highly flammable
Xn	Harmful
R11	Highly flammable.
R38	Irritating to skin.
R48/20	Harmful: danger of serious damage to health by prolonged exposure through inhalation.
R63	Possible risk of harm to the unborn child.
R65	Harmful: may cause lung damage if swallowed.
R67	Vapours may cause drowsiness and dizziness.
Repr.Cat.3	Toxic to Reproduction Category 3

Precautionary statement(s)

P210	Keep away from heat, hot surfaces, sparks, open flames and other ignition sources. No smoking.
P261	Avoid breathing vapours.
P281	Use personal protective equipment as required.
P301 + P310	IF SWALLOWED: Immediately call a POISON CENTER or doctor/physician.
P331	Do NOT induce vomiting.

Information on toxicological effects**Acute toxicity**

LD50 Oral - Rat - > 5.580 mg/kg

LC50 Inhalation - Rat - 4 h - 12.500 - 28.800 mg/m³

LD50 Dermal - Rabbit - 12.196 mg/kg

Skin corrosion/irritation

Skin - Rabbit

Result: Skin irritation - 24 h

Serious eye damage/eye irritation

Eyes - Rabbit

Result: No eye irritation

(OECD Test Guideline 405)

Appendix B. Impregnated activated carbon filter data

The information of IAC filter from Genano Oy Company. IAC filters used in this thesis were not necessary the filters from Genano Oy Company.



Technical Data Sheet for Carbon Filter Media

Description:	<i>Activated carbon impregnated sorption media on PUR substrate</i>	
Carrier material:	PUR Foam open celled:	PPI 20
	Density:	120 kg/m ³
	Foam Density:	30 kg/m ³
	Nominal thickness:	20 mm ± 1 mm
	Binder:	copolymer of vinyl acetate and ethylene
	Binder content:	10 %
	Carbon add-on (%):	300%
Carbon:	Powder Carbon type:	Carbopal SC 44-1
	Based on:	coconut shell
	H ₂ O content:	< 10 %
	Ashes content:	< 7 %
	pH-value:	9 – 11
	Molasses-factor:	2,2 ± 0,2
	Net carbon quantity:	1800 g/m ²
	Internal surface:	1 000 m ² /g (BET method)
	Iodine adsorption:	950 mg/g
	Methylbluetiter:	12 ml
	Grounfineness:	75 (weight-%) < 40 micro-m
Pressure drop:	at 1,0 m/s	25 Pa
Temperature resistance:		100 (120) °C
Fire retardation:		F 1 (DIN 53438)
Special remarks:	Operating Temperature:	below 50 °C

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Appendix C. Tenax TA tube details

Sourced from SIS and Markes International.

Properties

- Chemical Structure or Name: 2,6-diphenylene-oxide polymer resin
- Temperature Limit: 350 °C
- Affinity for Water: low
- Specific Surface Area: 35 sq. m/g
- Pore Volume: 2.4 cc/g
- Average Pore Size: 200 nm
- Density: 0.25 g/cc
- Mesh size: 60/80 mesh
- Other mesh sizes available on special request

Major Areas of Application

- Purge and Trap of Volatiles from Water
- Trapping Volatiles from Environmental Air
- Trapping of Volatiles from Human Breath
- Trapping Flavors and Fragrances from Plants and Commercial Products
- Trapping of Volatiles from Soil (PNA's and PCB's)
- Personal Exposure Monitoring

8.1.2 Tenax TA or GR (range of mesh sizes available)

Sorbent Strength:	Weak
Specific Surface Area (m ² /g):	»35
Approximate analyte volatility range:	n-C7 to n-C30 Bpt. 100°C to 450°C
Example Analytes:	Aromatic compounds (except benzene), apolar components boiling point >100°C, polar components boiling point >150°C, PAHs and PCBs.
Sorbent Maximum Temperature:	350°C
Recommended Conditioning Temperature:	325°C
Recommended Desorption Temperature:	Up to 300°C
Notes:	Hydrophobic, low inherent artifacts (< 1ng), inert, graphitised form best for PAHs and PCBs, efficient desorption, use 35-60 mesh to minimise fines and eliminate "leakage" through conventional sorbent retaining gauzes

Appendix D. Experimental work model for toluene removal by IAC filter

Some more photo for the system model.



Air speed meter used to control the fan.



Air flow meter used to control the nitrogen gas flow. (Model: Brooks Instrument Delta Smart II Mass Flow (left) and Brooks Instrument 0254 (right).)

Appendix E. Pre-work result

- Condition: AC 1 layer, fan 1 m/s, stabilized for 1 hr.
- Sampling position IN for the first table.

* GC graph for toluene was calibrated by Patrik Paul where
1 unit area = (430.31/35057368443) µg

N ₂ flow (mL/min)	pump time (min)	tenax #	pumping volume (L)	GCMSD area	mass (µg)	ppm
10	1	059783	0,113	35823552	0,439714484	0,8
	1,1	059764	0,1243	-	-	-
	1	058988	0,113	27945460	0,343015218	0,8
	2	058986	0,226	12088827	0,148383732	0
	2	059093	0,226	33194935	0,407449649	0,27
	2	058984	0,226	189769096	2,329311735	0,65
20	1	059761	0,113	2637004	0,03236778	0
	1	059763	0,113	-	-	-
	1	058983	0,113	17115690	0,210085722	0,27
	2	034850 (?)	0,226	-	-	-
	2	034842	0,226	4691815	0,05758946	0
	2	034843	0,226	5936557	0,072867986	0
	3	059770	0,339	-	-	-
	3	059785	0,339	7838430	0,096212436	0
	3	059786	0,339	9333573	0,114564497	0
	3					
50	1	059764	0,113	-	-	-
	1	059093	0,113	29157789	0,357895893	0,8
	1	058986	0,113	7681740	0,094289152	0
	2	058984	0,226	122901648	1,508550427	1,59
	2	034842	0,226	15363745	0,188581557	0
	2	059763	0,226	15655576	0,192163622	0
	3	034849	0,339	27931513	0,342844027	0,27
	3	059761	0,339	23003212	0,282351831	0
	3	034850	0,339	31828403	0,390676217	0,27
	5	059782	0,565	30868862	0,378898377	0
	5	030481	0,565	29578511	0,363060025	0
	5	034843	0,565	50474426	0,619545939	0,27
	10	058983	1,13	132154160	1,622119946	0,27
	10	058982	1,13	353299701	4,336560361	0,8
10	059768	1,13	173519318	2,129854608	0,27	

N ₂ flow: 100 mL/min	position	tenax #	pumping volume (L)	GCMSD area	mass (µg)	ppm
pump time: 5 min	IN	059783	0,565	281319479	3,453042552	1,59
	IN	059785	0,565	461292244	5,662109689	2,65
	IN	059786	0,565	550234538	6,753827642	2,92
	OUT	059761	0,565	364542329	4,474557463	1,86
	OUT	059762	0,565	457222191	5,612152017	2,39
	OUT	059765	0,565	539135842	6,617597226	2,92

* AC 1 layer filter had been placed there for more than half year, so as expected that the result between IN and OUT were similar. However, 5 min pumping time shown the most stable result

Appendix F. Instruction for tenax tube analysis

MSD ChemStation (version G1701DA D.01.02.16 15-JUNE-2004) from Agilent Technologies and Thermal Desorption System Control program (TDSC program) (version 2.0.0) from Markes International were used. "Unit" program refers to the TDSC program.

1. switch on the thermal desorber device
2. open program "Unity"
3. click [File] -> [Open] -> [Splitless Tenax tube desorption.mth] -> [open]
4. click [File] -> [Load] (Standard 2(3))
5. Check air tank, open it.
6. Check Inlets and change to back and GasSaver off
7. go to program "Instrument #1" and click the green arrow
8. change the "Data File Name" to the tenax tube number for testing and write down Misc.Info if needed, and press [start run] and [ok]
9. put the tenax tube into the thermal desorber device
10. go to program "Unity" and press the run bottom and ok and wait about 45 minutes
11. when the test is ready, go to program "Instrument #1" and click [View] -> [Data Analysis (offline)]
12. when it change to a new window (Enhanced Data Analysis), click [File] -> [Load Data File] and then select the tenax tube and click ok
13. then click [Spectrum] -> [Select Library] -> [Browse] -> [NBS75K.L] and click ok
14. click [Chromatogram] -> AutoIntegrate
15. click [Spectrum] -> [Library Search Report] and choose only [on screen] and press ok to check the retention time for toluene
16. then click [Chromatogram] -> [Percent Report] to find the area of toluene

Appendix G. Instruction for tenax tube cleaning

MSD ChemStation (version G1701DA D.01.02.16 15-JUNE-2004) from Agilent Technologies and Thermal Desorption System Control program (TDSC program) (version 2.0.0) from Markes International were used. "Unit" program refers to the TDSC program.

1. switch on the thermal desorber device
2. open program "Unity"
3. click [File] -> [Open] -> [Tenax condition.mth] -> [open]
4. set time to 10 min and temperature to 300 and flow path temp to 180.
5. click [File] -> [Load]
6. put the tenax tube into the thermal desorber device
7. go to program "Unity" and press the run bottom and ok and wait about 20 minutes

Appendix H. List of good and bad tenax tube from empty tube testing

Clean (empty) tenax TA tubes were tested and the result were shown in the table below. The usable (Good) tubes were the tubes which had a noises or contaminates lower than 1000000 unit of the GC graph. And the bad ones were those above that amount. There were all together thirty-one tenax TA tubes available in Leiritie campus. Based on this parameter, 18 were usable and the rest 13 were said to be ready to be thrown away.

Good (noises or contaminate < 1000000)	Bad (noises or contaminate > 1000000)
030481	058982
030482	058983
030486	058984
030590	058985
034842	058986
034843	058987
034845	058988
034849	058989
034850	059093
059761	059764
059762	059767
059763	059782
059765	059783
059768	
059770	
059785	
059786	
059790	

Appendix I. Batch September AC1-3 result

Experiment were carried out in the condition of N₂: 100 mL/min; fan: 1 m/s; pump time: 5 min; sample volume: 0.565 L; stabilization time (between each AC filters): 1 hour.




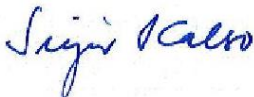
AC layer	position	tenax #	GC area	mass (µg)	concentration (mg/m ³)	ppm
AC 1	IN	058989	1074451901	13.18830871	23.34213931	6.10
		059764	1081571160	13.27569371	23.49680303	6.10
		059765	895365006	10.99011514	19.45153122	5.04
	OUT	059786	555816174	6.822339167	12.07493658	3.18
		059783	683138011	8.385144994	14.84096459	3.71
		059767	591109000	7.25553928	12.84166244	3.18
AC 2	IN	030590	905938280	11.11989629	19.68123237	5.04
		059782	834695461	10.2454297	18.13350389	4.78
		059768	876311588	10.75624487	19.03760154	5.04
	OUT	059763	542436736	6.658113893	11.78427238	2.92
		059762	678374971	8.326681287	14.737489	3.71
		059785	801606172	9.839276797	17.4146492	4.51
AC 3	IN	059761	793712314	9.742384012	17.24315754	4.51
		058984	861785018	10.57793918	18.72201624	4.78
		059790	821144981	10.07910498	17.83912385	4.51
	OUT	034843	620580062	7.617280427	13.48191226	3.45
		034849	918448408	11.27345126	19.95301108	5.04
		034850	1166776835	14.32154672	25.34787029	6.63

Appendix J. Batch October AC1-3 result

Experiment were carried out in the condition of N₂: 100 mL/min; fan: 1 m/s; pump time: 5 min; sample volume: 0.565 L; stabilization time (between each AC filters): 1 hour.

AC layer	position	tenax #	GC area	mass (µg)	concentration (mg/m ³)	ppm
AC 1	IN	059761	731203212	8.975119016	15.8851664	3.98
		034845	922051219	11.31767379	20.03128105	5.31
		030482	1051520472	12.90683797	22.84396102	5.84
	OUT	034843	655836149	8.050029589	14.24783998	3.71
		059785	971377330	11.92312479	21.10287574	5.57
		030482	1070380885	13.13833922	23.25369773	6.1
AC2	IN	059763	1037494256	12.73467385	22.53924576	5.84
		059765	1169730606	14.35780264	25.41204007	6.63
		030486	1267535946	15.55830963	27.5368312	7.16
	OUT	059790	1160820623	14.24843747	25.21847341	6.63
		059786	1066322532	13.08852516	23.16553125	6.1
		059768	1101371560	13.51873278	23.92696067	6.1
AC3	IN	034850	1006658262	12.35617891	21.86934321	5.57
		030481	924176874	11.34376504	20.07746025	5.31
		059762	1306934177	16.04190134	28.39274573	7.43
	OUT	034842	810339988	9.946479605	17.60438868	4.51
		030590	870102256	10.68002872	18.9027057	4.78
		059770	1183796943	14.53045922	25.71762694	6.63

Appendix K. Batch October AC 2 layer result from Genano Oy Company

		TESTAUSSELOSTE 2015-26637	1(1) 04.12.2015
Tilaaaja 2175218-0 Genano Oy Erkkilä Jussi	Maksaja Genano Oy Ab Laskutus	 	
Kimmeltie 3 02110 ESPOO	Niinistönkatu 8 05800 HYVINKÄÄ		
Näytetiedot	Näyte Sisäilma VOC	Kellonaika	
	Näyte otettu 25.11.2015	Kellonaika 09.45	
	Vastaanotettu 26.11.2015	Näytteenoton syy Tilaustutkimus	
	Tutkimus alkoi 26.11.2015		
	Näytteen ottaja Erkkilä Jussi		
	Viite Testi/Genano		
Tolueenin pitoisuudet:			
1) 1550 ug/m ³			
2) 1160 ug/m ³			
3) 1760 ug/m ³			
4) 1580 ug/m ³			
5) 1963 ug/m ³			
6) 1790 ug/m ³			
	Analyyssi Yksikkö Menetelmä Epävarmuus-%	TVOC tolueenina (TD-GC-MSD/FID) ug/m ³ ISO 16000-6:2011 30	
Näyte		*	
26637-1, Sisäilma VOC, in 1, genano		x	
26637-2, Sisäilma VOC, out 1, genano		x	
26637-3, Sisäilma VOC, in 2, genano		x	
26637-4, Sisäilma VOC, out 2, genano		x	
26637-5, Sisäilma VOC, in 3, genano		x	
26637-6, Sisäilma VOC, out 3, genano		x	
*=-näyte tutkittu akkreditoidulla menetelmällä			
Yhteyshenkilö	Lukkarinen Timo, 010 3913 431, Kemisti		
			
	Kalso Seija toimitusjohtaja		
Tiedoksi	Erkkilä Jussi, jussi.erkkila@genano.fi		
Akkreditointi ei koske lausuntoa. Analyysitulokset pätevät ainoastaan analysoiduille näytteille. Analyysitodistuksen saa kopioida vain kokonaan. Muussa tapauksessa kopioinnista on saatava lupa.			
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