Hospital Air Emission Capture and Recovery

by

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AUTHOR'S DECLARATION

I hereby declare that I am the sole author of this thesis. This is a true copy of the thesis, including any required final revisions, as accepted by my examiners.

I understand that my thesis may be made electronically available to the public.

Abstract

Inhaled anesthetics used in hospital surgeries are typically volatile halogenated hydrocarbons. Very few of them are absorbed or metabolized by the patient during use, and, therefore, most of the exhaled gases are collected in the exhaust system and emitted directly to the environment, where they contribute to global warming and stratospheric ozone depletion. This research focuses on a capture and treatment system for the common anesthetic gases (Isoflurane, Sevoflurane and Desflurane) which includes the capture of these compounds in addition to their recovery by regeneration in a form that can possibly be re-used in hospitals or manufacture.

The first step in this research was the development of appropriate quantification methods using a gas chromatograph and an FTIR instrument for accurate measurement of the concentrations in batch and continuous flow systems, respectively. In developing a capture system, activated carbon, molecular sieves, and other adsorbents were tested in different batch experiments using several of the most common anesthetic compounds. The activated carbon Norit A48 (with 100% adsorption efficency) was 1.5 to 4 times more efficient at absorbing the halothane than either the polyethylene (25%), the molecular sieve 4A (38%), the industrial white adsorbent (57 %), and the silica gel (63 %) at room temperature. It was similar in adsorption capacity to the Activated Carbon RB 40M (99.8%), the Activated Carbon Vapure 410 (98.8%), the Molecular Sieve 13X (96 %), and the Sigma Activated Carbon (87 %). The GAC 48 (96.5 %) was 1.8 to 2 times more efficient at absorbing the Isoflurane than the industrial white adsorbent (41 %), and the silica gel (51 %) at room temperature. Isoflurane with intial volumes of 50, 100, 150 and 200 µl were adsorbed by GAC A48 with 98.8, 96.5, 74.5, and 64.1 % efficiency, respectively, after 120 minutes. In addition, sevoflurane with intial volumes of 50, 100, 150 and 200 µl were captured By GAC A48 with 99.0, 72.6, 65.3, and 50.4 % efficiency in the same time. Therefore, GAC A48 was selected as the best adsorbent for capturing both Isoflurane and Sevoflurane with the same adsorption rate at room temperature.

Adsorption isotherms were measured for activated carbon and a selection of different anesthetics. In order to account for all the factors in the adsorption process at the hospital, the effects of other parameters such as temperature, gas mixing, initial concentration, and humidity

were tested. TGA (thermal gravimetric analysis) was used to confirm that complete desorption was possible in the batch system.

Before the industrial set up was tested, a flow through system with air as the carrier gas was designed in the laboratory to closely simulate the expected hospital emissions. Satisfactory results obtained in the laboratory were followed with the installation of a tank containing the best adsorbent connected to a hospital emission system. When the tank carbon bed reached its saturation point after three weeks, desorption proceeded in two steps: "depressurization" and " heated desorption". Desorbed amounts of anesthetic in the depressurization step were negligible in comparison with the amounts collected in the heated desorption step in which a tank of activated carbon was heated while air was passed through it.

The condensation of desorbed anesthetics was successfully performed in two stages: the removal of water and the condensation of desorbed anesthetics in in a flask and a coil that were both immersed in dry ice. The temperature of the dry ice was at least -78 °C. Analysis of the condensate in the second stage confirmed the capture of relatively pure sevoflurane and desflurane. A white solid and a little oily liquid by-product were detected during the desorption process that could not be accurately identified by using available laboratory analytical methods.

The anesthetic gas absorption results using a tank of activated carbon at the hospital illustrates that the optimal adsorbent could adsorb anesthetics at about 25 % of its initial weight, although some break-through happened before the saturation point was reached. The main recommendations arising from these experiments include: Installing an online tank heating system to enhance the desorption of the anaesthetic gas collection system; and designing a heat exchanger system for effective condensation of the anesthetic gases after the desorption process.

Acknowledgements

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Dedication

This thesis is dedicated to my parents who never stop to believing in me;

And my Husband (Ali) and Beautiful Daughter (Diana) that patiently support me.

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1. Introduction

1-1 Anesthetic gases

Inhalation anesthetics are a form of narcotic which are usually made of simple halogenated hydrocarbons, paraffins or ethers that are in a liquid state at room temperature and are evaporated prior to inhalation. They are administered by an anesthesia mask, laryngeal mask airway or tracheal tubes connected to some type of vaporizer delivery system. Common anesthetics are Isoflurane (2,2,2-trifluoro-1-chloroethyl difluoro methyl ether), Sevoflurane (1,1,1,3,3,3-hexafluoro-2-(fluoromethoxy)propane), Desflurane (1,2,2,2-tetrafluoroethyl difluoromethyl ether) and Nitrous oxide (also known as “laughing gas”). The effect obtained from such halogenated hydrocarbons is normally increased by an addition of laughing gas at concentrations of 50-70 volume %.[1] Halothane (2-bromo-2-chloro-1,1,1-trifluoroethane) is another anesthetic compound that was used as anesthesia in the past but as it has some side effects it was substituted by the current gases.

The ideal volatile anesthetic agent should create smooth and trustworthy induction and continuence of general anesthesia with minimal effects on other organ systems. Furthermore it should be odorless or pleasant to inhale; safe for all ages and in pregnancy; not metabolized; rapid in onset and offset; potent and safe for exposure to operating room staff. Additionally,it should be cheap to manufacture; easy to transport and store, with a long shelf life; easy to administer and monitor with existing equipment; stable to light, plastics, metals, rubber and soda lime; non-flammable and environmentally safe. None of the current chemicals in use are ideal, while many have some of the suitable characteristics. The full mechanism of action of volatile anesthetics is not fully understood. An effect of these gases is described by physical interaction not a chemical bond actions, although the agent may bind to a receptor with a weak interaction. The three common anesthetic gases (sevoflurane, desflurane and isoflurane) undergo very little in-vivo metabolism in clinical use. Less than 5% of these volatile anesthetics is metabolized by the patient.[2] Therefore, most of the inhaled gases are exhaled and scavenged by the exhaust system to emit directly to the environment.

Isoflurane

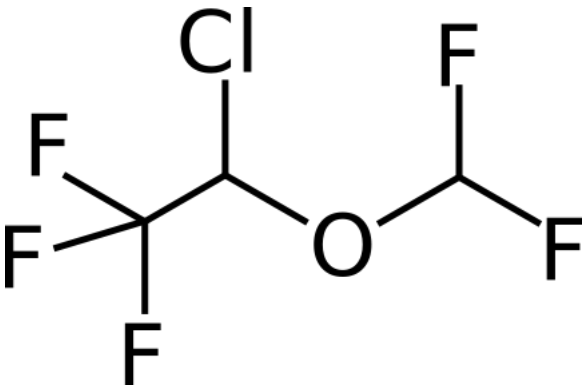

Figure 1-1: The chemical structure of Isoflurane

Isoflurane (2-chloro-2-(difluoromethoxy)-1,1,1-trifluoro-ethane) is a halogenated ether used for inhalational anesthesia as a substitute for the flammable ether and Halothane with fewer side effects . The application of this anesthetic gas recently has decreased because of a neurodegeneration risk [2] and replaced with two others Sevoflurane and Desflurane .

Isoflurane is a clear, colorless, liquid at room temperature but vaporized easily, containing no additives or chemical stabilizers. Isoflurane has a mildly pungent, musty, ethereal odor. Isoflurane does not decompose in the presence of soda lime (at normal operating temperatures), and does not attack aluminum, tin, brass, iron or copper. It is completely nonflammable. It is usually used to maintain a state of general anesthesia that has been induced with another drug, such as propofal.[3] Some of its physical properties are provided in Table (1-1).

Sevoflurane

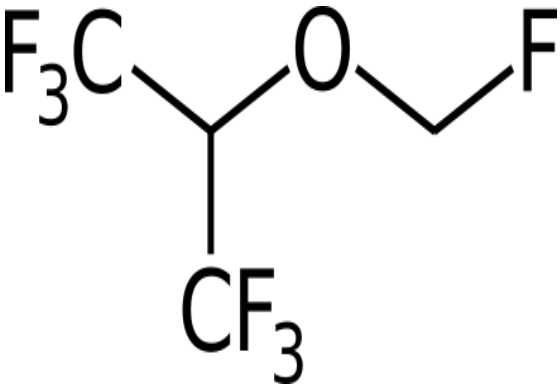

Figure 1-2: The chemical structure of Sevoflurane

Sevoflurane is a halogenated general inhalation anesthetic drug. Its chemical name is fluoromethyl 2,2,2-trifluoro-l-(trifluoromethyl) ethyl ether, with a structural formula that is provided in picture (1-2).

Sevoflurane is non-flammable and non-explosive as defined by the requirements of the International Electrotechnical Commission 601-2-13. Sevoflurane is a clear, colorless, stable liquid containing no additives or chemical stabilizers. Sevoflurane is non-pungent. It is miscible with ethanol, ether, chloroform and petroleum benzene, and it is slightly soluble in water. Sevoflurane is stable when stored under normal room lighting conditions according to instructions. In modern anesthesiology, Sevoflurane with

Desflurane has been replacing Isoflurane.[4] It is often administered in a mixture of nitrous oxide and oxygen. The fast onset and offset, low blood/gas coefficient and low irritation to mucous membranes make this gas the preferred agent especialy for a surgery using a mask induction .The major concern related to Sevoflurane is the ability to react in the presence of strong bases such as carbon dioxide absorbents. All of the currently used inhalational anesthetic agents react with some CO2 absorbents resulting in the formation of specific compounds. Sevoflurane produces a vinyl ether commonly referred to as compound A. It also creates other degradation products under special conditions that have been identified as compound B, C, D, F, and E. Desflurane, ethrane, and isoflurane can generate carbon monoxide in the presence of CO2 absorbents. Any significant clinical issue relevant to patients from any of these inhalational agent byproducts has never been proven.[5] More details about the modification of anesthetic gases is explained in (1-5).

Desflurane

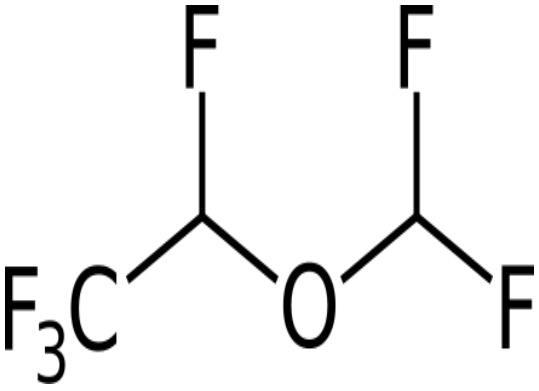

Figure 1--3: The chemical structure of Desflurane

Desflurane (1,2,2,2-tetrafluoroethyl difluoromethyl ether) is a nonflammable and highly fluorinated anesthetic gases like Sevoflurane and Isoflurane. It has a mixture of (*R*) and (*S*) optical isomers (two mirror images of a chiral molcules are called optical isomer or enantiomers). This anesthetic gas has a much higher global warming effect (discussed below) and is a colorless, volatile liquid below 22.8°C . It is also stable when stored under normal room lighting conditions according to manufacturer instructions. With its low solubility in blood, it has the fastest onset and offset in comparison with Isoflurane and Sevoflurane. Its pungency and high cost are two significant issues related to this modern inhaled anesthetic gas. [6]

Its degradation reaction ocurres through persistent direct contact with soda lime that produces low levels of fluoroform (CHF_3) and detectable levels of carbon monoxide. The amount of CHF_3 obtained is similar to that produced with MAC-equivalent doses of isoflurane. No discernible degradation occurs in the presence of strong acids. The degradation process will be

clarified in details in part (1-5).[5]

Halothane

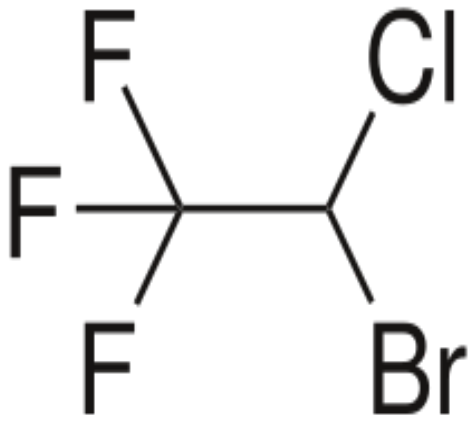

Figure 1-4: The chemical structure of Halothane

2-bromo-2-chloro-1,1,1-trifluoroethane (Halothane) is an inhalational general anesthetic gas that was replaced with the newer generation of anesthetic gases (Isoflurane, Sevoflurane and Desflurane) because of some undesirable side effects. The price of this compound is significantly cheaper than isoflurane and the other two, and it is more readily available for research purposes. However, halothane's chemical and physical properties are similar to the others. Therefore, the preliminary experiments started with this compound and were then repeated with three others. Some physical properties of the three common anesthetics and Halothane are organized in Table (1-1):

Table (1-1): Some Physical Properties of Isoflurane, Sevoflurane, Desflurane and Halothane [20]

Physical properties	Boiling point at 1atm (°C)	Density at 20°C (g/cm^3)	Molcular Weight	Vapour Pressure (Kpa) at 20°C
Isoflurane	48.5	1.496	184.5	31.7
Sevoflurane	58.6	1.522	200	22.9
Desflurane	23.5	1.465	168	88.5
Halothane	50.2	1.868	197	32.53

1-2 Environmental implication of Anesthetic gases:

Waste anesthetic gas halocarbons (similar in composition to CFCs (chloro-fluoro-carbons) and other refrigerants) have been connected to ozone depletion and to a lesser degree, global warming. The halocarbons used in anesthesia (primarily halogenated methyl ethyl ethers) are now considered significant emission sources, as other industrial and commercial halocarbon emissions have been greatly reduced by legislation and other initiatives in recent years. Although

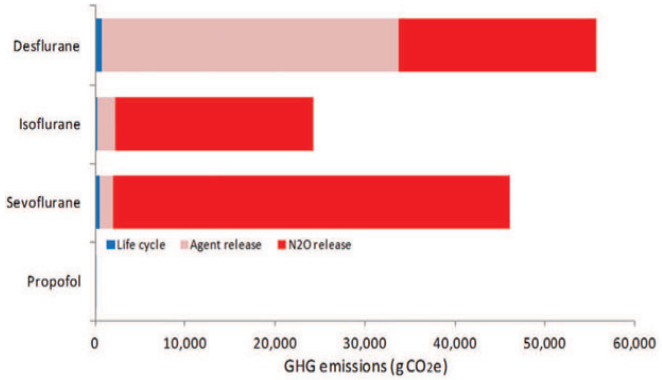

Figure 1--5: Life cycle green-house gas (GHG) emis-sions of anes-thetics, including waste anes-thetic gas emis-sions of halo-genated drugs and nitrous oxide (N2O)[Published May 21, 2012 at 620 × 322 in The environmental impact of anesthetic

no regulation has controlled waste anesthetic gas emissions so far, this will happen in the near future.

The Global Warming Potential GWP(20), which describes the total warming impact relative to CO_2 over 20 years, has values for the inhaled anesthetics of: sevoflurane 349, isoflurane 1401, desflurane 3714 and N_2O 268.[8] Although Desflurane has the best offset and onset as an anesthetic gas, it has the worst effect on global warming. N_2O in its gaseous form, usually added to these gases to improve their effect, leads to more environmental damage. Figure(1-5) demonstrates the environmental impact of the anesthetic gases as GHG (Greenhouse gas) emissions with or without N_2O.

Recently, an evaluation has been made that focuses on the accumulation of these anesthetic gases coming from hospitals. Worldwide yearly sales of inhaled anesthetics total in the millions of liters, given that a busy midsize US hospital might purchase approximately 1000 L of inhaled anesthetics per year. Assuming an average 4.78 metric tons of CO2 emissions/passenger car/year in the United States, this would be the same as about 100 to 1200 passenger car emissions/year/midsized hospital.[7]

Currently, gas scavenging is the most practical engineering control for removing waste anesthetic gases. The effect of gas scavenging can be improved by combining this method with other control procedures such as dilution ventilation; although, the problem is not totally solved.

1-3 Different Techniques for Capturing and Recovery of the Waste Anesthetics Gases

Anesthesia delivery systems in surgical facilities (medical, dental, and veterinary) produce significant amounts of waste anesthetic gases. Most of these gases are not metabolized by a patient's body and are collected from the patient's exhalation by a dedicated or shared vacuum system. The healthcare facilities usually apply one or more centrally-located vacuum pumps to emit waste gases from a large number of surrounding rooms. In addition, more air is added to the mixture of anesthetic gases to dilute the stream before venting to the atmosphere.

Therefore, the exhausted stream comprises (in volume percent): 25-32 percent oxygen, 60-65 percent nitrogen, 5-10 percent nitrous oxide, and 0.1-0.5 percent volatile halocarbons, including fluoro-ethers, such as isoflurane, desflurane and sevoflurane.[9]

Various methods have been suggested so far to solve the growing problem of waste anesthetic gas emissions:

In 2006, a catalyst for decomposing nitrous oxide at temperatures ranging from 200 to 600°C was suggested as a removal method to solve the non halogonated waste anesthetic gas issue. [10]

In a series of patents [9,12,13], the Berry research group had developed an efficient condensation method to collect gases in liquid or solid form.

In one patent, Berry in 2006 [3] suggested that a periodic batch process that liquifies oxygen in a cold fractionator be applied to selectively condense nitrous oxide and other volatile halocarbon gas components from waste anesthetic gases at -150 ° C.

In a second patent[12], using a low-flow scavenging system, an intelligent waste canesthetic gas collection unit was used to suction flow only in the presence of waste gas and stop all flow into the suction manifold when no waste gas was present. This method is suitable for condensation, because a smaller volume of gas is cooled using the condensation temperatures.

Further, the recovery system was changed by this group in such a way that a compressor was applied to increase the waste anesthetic gas pressure make it easier to condense the anesthetic

gas components at higher temperatures .The method was applicable with both high-flow and low-flow rate scavenging systems; however, a low –flow system with an intelligent collector was preferable.[9]

Another applicable method for capturing and recovering of anesthetic gases is the adsorption process. Limited studies have been done in this area to clarify two important key questions: "which adsorbent should be applied?" and "how should the adsorbent system be designed?"

In U.S. patents 5,515,845 [13] and 5,231,980 [14], a Canadian company developed a technology based on the adsorption process to remove the waste anesthetic gases. In this method, a canister that contains silica zeolite was applied to selectively remove an anesthetic gas from the exhaled stream in the surgery room. This process lasts until the adsorbent material is saturated and the adsorbent replaced with a fresh one, which usualy takes a few days.

This method was more extesively investigated in 2002 by Doyle to find the efficiency of this system for removing isoflurane. It was concluded that Silica zeolite was effective at completely removing 1% isoflurane from exhaled gases for periods of eight hours. The technology demonstrates promise in removing isoflurane emitted from anesthesia machine scavenging systems.[15]

In another paper, different kinds of activated carbon were investigated as adsorbents to find the one best suited for the adsorption and desorption processes. In this paper, only Isoflurane as an anesthetic gas was tested.[1]

In a third paper, Hotta specified a process that captured the waste anesthetic gases in two steps. First, an adsorbing cylinder removed all anesthetic gases except nitrous oxide. Then the stream was passed over a catalyst layer to decompose nitrous oxide .[16]

Most of these papers and patents only focus on old anesthetic gases and sometimes Isoflurane, which is currently replaced by Sevoflurane and Desflurane. In addition, there is no source that compares different adsorbents.

1-4 Adsorption Process

In a surface phenomenon,atoms, ions, or molecules from a gas, liquid, or dissolved solid adhere to a surface. This process is a surface phenomenon and is different from absorption where whole volumes participate. In an adsorption process, two substances are involved. One is the solid or the liquid on which adsorption occurs and this is called the adsorbent. The second is the adsorbate, which is the gas or liquid or the solute from a solution which gets adsorbed on the surface. Regarding the nature of forces existing between adsorbate and adsorbent molecules , two types of adsorption occur:

1. Physical adsorption (physisorption): the force of attraction existing between adsorbate and adsorbent is Vander Waal's force. This force is quite weak and can be reversed by heating or by decreasing the pressure.
2. Chemical adsorption (chemisorption): If the force of attraction existing between adsorbate and adsorbent is almost the same strength as the chemical bond, the adsorption is called chemical adsorption. It is also known as Langmuir adsorption. This process can not be reversed easily as it is a strong nature of force. Various elements affecting the adsorption consist of:

1. Nature of adsorbate and adsorbent
2. The surface area of adsorbent
3. Activation of adsorbent.
4. Experimental conditions such as :temperature, pressure, etc.

Adsorption Isotherm

The isotherm depicts the equilibrium of the sorption of a material at a surface at constant temperature. This adsorption process is usually depicated through graphs known as an adsorption isotherm. It displays the amount of adsorbate on the adsorbent as a function of pressure or concentration at constant temperature .The amount adsorbed over the mass of the adsorbent (q) is a useful parameter that allows comparison of different materials.

Freundlich Adsorption Isotherm

In 1909, Freundlich proposed an empirical expression to signify variations of the isotherm adsorption . This equation is named the Freundlich Adsorption Isotherm or the Freundlich Adsorption equation or simply the Freundlich Isotherm.

$$q = kp^{\frac{1}{n}} \qquad eq(1)$$

It is also written as:

$$q = kc^{\frac{1}{n}} \qquad eq(2)$$

q :the mass of adsorbate on mass of the adsorbent

p: Equilibrium pressure of adsorbate

C: Equilibrium concentration of adsorbate in solution

k and 1/n :constants depend upon adsorbent and adsorbate at particular temperature

Experimentally it was determined that extent of adsorption varies directly with pressure until saturation pressure Ps is reached. Beyond that point the rate of adsorption saturates even after applying higher pressure. Thus the Freundlich adsorption isotherm fails at higher pressure.

As can be observed in Figure (1-6), the value of q is rising with increasing c until it reaches a constant value.

Taking the logarithms of a second equation:

$$\log q = loqk + \frac{1}{n}\log c \qquad eq(3)$$

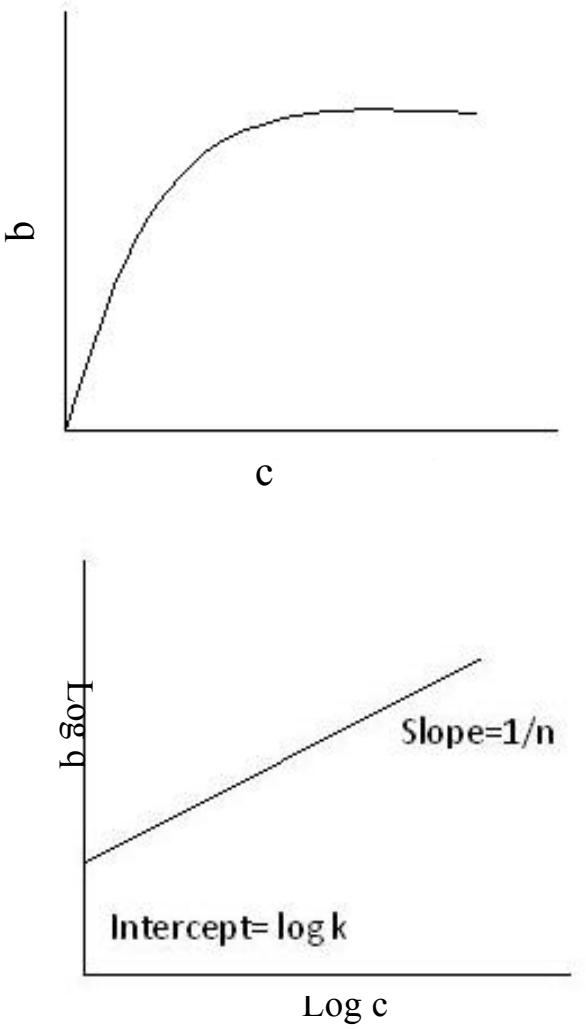

Figure 1-6:the Freundlich Adsorption Isotherm showing top (the amount adsorbed, q , as a function of equilibrium concentration in the solution) bottom(the logarithm amount

The log q is charted against log c and is a straight line in the following diagram. The slope shows 1/n and the value of the intercept presents log k. If the graph of log x/m against log p follows a straight line, it can be concluded that the freundlich adsorption isotherm fits this system.

Langmuir Adsorption Isotherm

In 1916 Langmuir suggested another adsorption isotherm known as the Langmuir adsorption isotherm. It was assumed in this theory that a dynamic equilibrium exists between adsorbed gaseous molecules and the free gaseous molecules.

Based on his theory, the Langmuir Equation describes a relationship between the number of active sites in the surface that participate in the adsorption and pressure.

$$\theta = \frac{KP}{1 + Kp} \qquad eq(4)$$

θ :the number of sites of the surface which are covered with adsorbate molecules

P :represents pressure

K : the equilibrium constant for distribution of adsorbate between the surface and the adsorbate phase .

This theory is applicable for low pressure as is the Freundlich isotherm.

At lower pressure, KP is so small that factor (1+KP) can almost be counted as zero. Therefore, the Langmuir equation is converted to

$$\theta = KP \qquad eq(5)$$

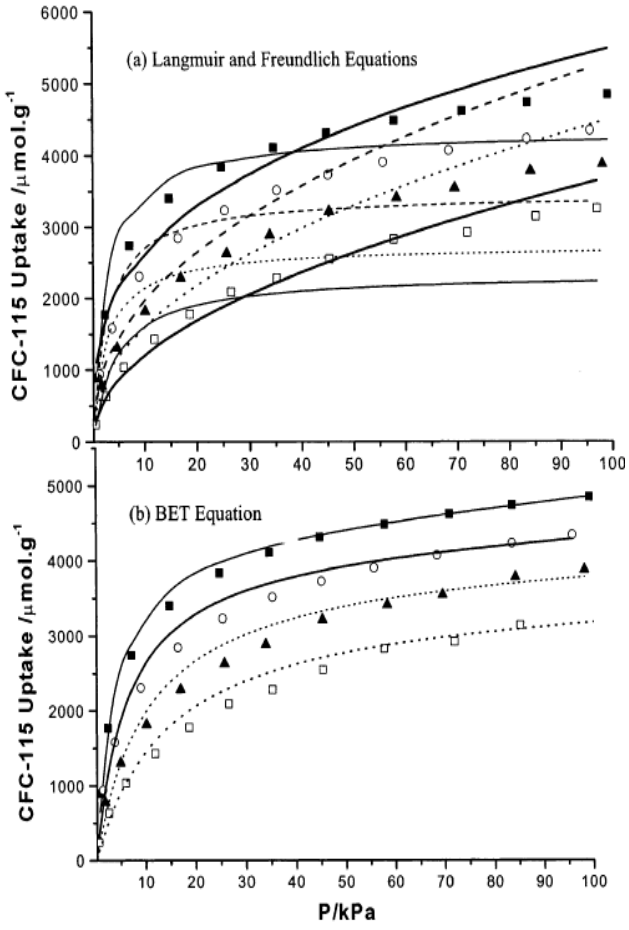

Figure 1-7: :Calculated isotherms by the Langmuir, Freundlich, and BET equations with the experimental data for CFC-115: (a) ▪, experimental value at 298 K; —, Langmuir eq (298 K); —, Freundlich eq (298 K); ○, experimental value at 318 K; · · ·, Langmuir eq (318 K); · · ·, Freundlich eq (318 K); ▲, experimental value at 338 K; · · ·, Langmuir eq (338 K); · · ·, Freundlich eq (338 K); □, experimental value at 358 K; Langmuir eq (358 K); Freundlich eq (358 K). (b) —, BET eq

At high pressure KP is so large, that factor (1+KP) in is nearly equal to KP. So the Langmuir equation is changed to:

$$\theta = \frac{KP}{KP} = 1 \qquad Eq(6)$$

BET Adsorption Isotherm

BET Theory, discovered by Brunauer, Emmett and Teller, is based on a multilayer formation that can be applied to the physical adsorption phenomena.

The Langmuir Adsorption Isotherm is based on the theory that the monolayer only happens in low pressure. In this condition, gaseous molecules would possess high thermal energy and high escape velocity. Therefore, the number of gaseous molecules near the surface of adsorbent would be limited.

In both high pressure and low temperature, multilayer adsorption happens where more adsorbent molecules would be accessible per unit surface area as the thermal energy of gaseous molecules fall. The BET equation is given as

$$V_{total} = \frac{V_{mono} C (\frac{P}{P_0})}{(1 - \frac{p}{P_0})(1 + c\left(\frac{p}{P_0}\right) - \frac{p}{P_0})} \qquad eq(7)$$

V_{mono}: adsorbed volume of adsorbate at high pressure conditions to cover the surface with a unilayer of gaseous molecules

P :The equilibrium pressure of adsorbates at the temperature of adsorption

P_0: The Saturation pressure of adsorbates at the temperature of adsorption

C: BET Constant

The BET method is extensively used in surface science for the calculation of surface area of solids by physical adsorption of gas molecules.[17] ,[18]

There is no published study about the adsorption isotherm of the three anesthetic gases of interest on activated carbon or similar adsorbents.The equiblium adsorption of CFC(CFC-115, CF_3CF_2Cl), which has a similar chemical structure to the anesthetic gases, on activated carbon was studied in 2003. Results were fitted with mathematical relevant models using Langmuir, Freundlich, and BET equations. Figure (1-7-a) shows the calculated isotherms of charcoal for CFC-115 by the Langmuir and Freundlich equations.

Figure(1-7-b) displays the BET equations and the experimental data at different temperatures. The amount of CFC-115 adsorbed increases with increasing pressure to a maximum. In very low pressure and over 26.6 kpa , there is a good fit between the experimental data and the Longmuir graph. Differences between the Freundlich equation and experimental data increase with rising pressure. The BET equation has the best fit with experimental data over the all tested temperatures. The BET constant was calculated in this paper too.[19]

1-5 The anesthetic gas degradation:

New general anaesthetics consisting of Sevoflurane, isoflurane and desflurane have successfully replaced the previous generation agents. Their suitable physicochemical characteristics , such as gas-blood partition coefficients, rapid onset of anaesthesia and quicker recovery, lower doses and better control of the patient's condition make them relevant in the medical industry . To the contrary, their chemical structure causes several issues that should be noted in the application of theses gases. Studies have been done about the degradation of anesthetics that were published both in medical and chemical papers. The knowledge of how to apply and store these compounds and how to prevent uncontrollable degradation is extremely important; especialy since these three different anesthetics are currently available.

From the chemical point-of-view, the carbon-fluorine bond, C-F, is unique. With respect to differences in the electronegativity of elements, the electron cloud of the bond creates a dipole with negative charge on the fluorine atom and positive charge on the carbon atom. A similar polarization is observed in carbon-oxygen (C-O) bonds. The formation of partial charges is the reason for the reactivity. Each site may be the center of reactions leading to molecular breakdown and formation of new, unfavorable or even toxic compounds. Sevoflurane is more sensitive, as the monofluormethyl group is susceptible to the attack of Lewis acids . Sevoflurane produces a variety of degradation products such as *compound A* [fluoromethyl-2,2-difluoro-1-(trifluoromethyl)vinyl ether] also called PIFE (pentafluoroisopropenyl fluoromethyl ether) and *Compound B* [1,1,1,3,3-pentafluoro-2-(fluoromethoxy)-3-methoxypropane] also called PMFE (pentafluoromethoxy isopropyl fluoromethyl ether) on contact with a carbon dioxide adsorbent such as the soda lime. Different factors effect the degradation products quality and quantity such as temperature, flow rate etc.[21]

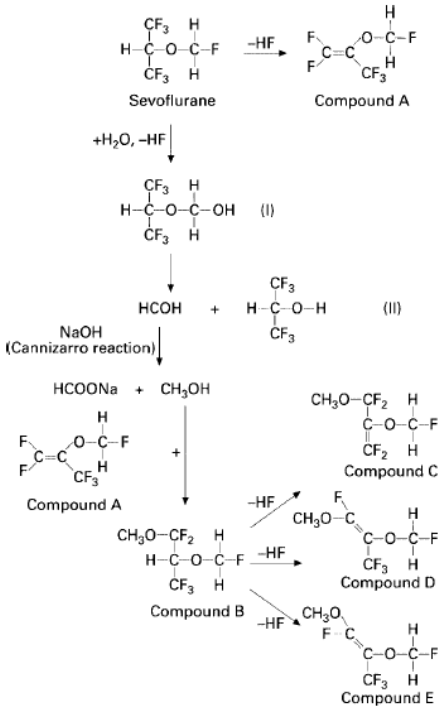

Figure 1- 8: Sevoflurane degradation products[21]

In 1996, it was reported that Sevoflurane was not consistently stable and could be degraded by Lewis acids. A Lewis acid, such as $FeCl_3$, $Fe_2O_3$and BF_3, is a metal-containing molecule that can produce a covalent bond by accepting an unshared pair of electrons. Lewis acids react with sevoflurane and produce hydrogen fluoride and several other products. These compounds might be found in different quantities adhered to the components of the anesthesia machine, vaporizer, circuit, shipping and/or storage containers. There is a limited amount of information about degradation products and their hazardous effects. For example fifty parts per million (ppm) of hydrogen fluoride exposure for 1 hour will cause serious adverse effects such as eye and skin irritation, a burning sensation, respiratory tract irritation (coughing, laryngitis, bronchospasm), respiratory tract injury (pulmonaryedema, respiratory failure, pulmonary fibrosis), achycardia, and hypertension. Hydrogen fluoride etches glass containers releasing aluminum oxides, which

are Lewis acids and a natural component of glass. This can start a self-perpetuating cycle of reactions. Fortunately, any hydrogen fluoride and silicon tetrafluoride produced (if the reaction happens in the glass container) are highly pungent gases with characteristic smells, and can be easily recognized. Hydrogen fluoride is detectable by smell at concentrations 100-fold lower than toxic levels. The contaminated sevoflurane was first identified in such a way in 1986. The Abbott Lab. company, the only manufacturer of sevoflurane that year, added water (up to 300-1000 ppm) to block the formation of a Lewis acid and replaced glass containers with plastic ones (polyethylene naphthalate). At present, sevoflurane is produced with different water content and packaging. The plastic or aluminium-lined containers are produced by two companies:

Table (1-2): Comparison of preparations containing Sevoflurane [5]

Producer	Abbot Lab.	Baxter
Name	Sevorane/Ultane	Sevoflurane
Purity	More than 99.99%	More than 99.99%
Water Content	330 ppm	130 ppm
Container	PEN(Polyethylene Naphthalate)	Aluminium Lined with Phenolic resin

The chemical structure of sevoflurane is similar to diethyl ether and haloform (chloroform, fluoroform), therefore it acts as an organic solvent. It may cause local weakness or damage to organic polymers, such as polyethylene naphthalate or phenolic resin. In a inappropriate packing and storage conditions, some part of the content may evaporate. This was the issue with use of a series of Ultane containers .In addition, in a plastic container some additives may act as potential Lewis acids and cause degradation. Therefore, the containers of this agent were selected specifically by the two manufactures to prevent any degradation issues (Table (1-2).

Another problem regarding the new generation anaesthetics is their instability in the precense of CO_2 absorbents used in anaesthetic machines . This effect was discovered when halothane was used in the closed circuit with a CO_2 absorbent and extensive amounts of toxic products of degradation formed. Isoflurane and desflurane break down only to CO while sevoflurane degrades to various products when in contact with soda lime or other adsorbents that have an OH group .

In a closed anesthesia circuit, different kinds of degradation products can be formed where the quality and quantity of them depend mainly on temperature and moisture of the absorbent . Moisture less than 15% apparently increases the capacity of the adsorbent to degerade the anesthetic . As the majority of reactions happening in the absorbent are exothermal, heating the absorber increases the degree of degradation of the anaesthetic , which, in turn, raises the temperature and decreases moisture (flow of warm, dry gas dries the absorber). This problem is more serious in a low flow system as the adsorbent temperature is increased more rapidly.[22,23,24,25]

Researches have demonstrated that active sodium and potassium hydroxides enhance the degradation of anaesthetics .For other absorbents, which do not contain hydroxides, degeradation issues have not been reported.[5]

The other point that should be noted with respect to safely applying modern inhaled anaesthetics is proper equipment. Suitable safety and control may be provided only in a unit with trained personnel and appropriate devices. Incorrect equipment may cause serious problems; there have been cases when the use of improper vaporizers damaged the anaesthetic machines .[5]

1-6 Objective of the Current Research

Regarding the importance of the waste anesthetic gases problem and shortage of studies in this area, the objects of this project,therefore, was planned as such

1- Determining accurate methods for measuring anesthetic gas quantity in both batch and continuous experiments
2- Designing batch experiments to compare the adsorption capacity of different adsorbents with the new anesthetic gases (Isoflurane, Sevoflurane and Desflurane)
3- Obtaining the isotherm of three agents on the best adsorbent and investigating experimental condition effects on the adsorption process
4- Preparing in the laboratory a column of adsorbent to do a pre-test before industrial set up of the adsorbent container
5- Setting and monitoring the industrial system to determine how well the system performs and to identify any opportunities for improvements
6- Precisely analyzing the desorption process in two steps of the depressurizing and the heating desorption to find the best condition for performance of this process through testing different parameters such as temperature, flow rate etc.
7- Designing and developing an effective cryogenic condensation system for recovery of the adsorbed gases and testing the purity of the captured gases

2.Experiments and Results

2-1 The Calibration of Gas Chromatography (GC)

The composition of anaesthetic gases (Halothane, Isoflurane, Sevoflurane and Desflurane) during experiments were analyzed throgh GC (HP5890 Series II) equipped with a split/splitless injector, a flame ionization detector (FID) and an electron capture detector (ECD) with a 30 m by 0.53 mm RTX502.2 column (Restek, Pennsylvania). Data acquisition involved a starting oven temperature of 50^{o}C followed by a ramp temperature rate of 2 ^{o}C/min to a final temperature of 60 ^{o}C . The first step was to set an appropriate method and calibration curve for this instrument to measure the composition parameters.

Halothane Calibration Data

To obtain a calibration curve which demonstrates the relationship between the concentration of sample and the GC peak area, 1 µl of Halothane was injected into a standard glass bulb with a volume of 2000 ml, then after half an hour of mixing at room temperature three different volumes from the bulb were collected and injected into the GC. This procedure was repeated for 60 µl of Halothane . The results are summarized in Table(2-1) and Figure(2-1):

Table (2-1): The Gas Chromatography Calibration Data for Halothane

Injected 1 µl Halothane to Standard bulb				Injected 60 µl Halothane to Standard bulb			
Volume(ml)	Amount(ng)	ECD Area	FID Area	Volume(ml)	Amount(ng)	ECD Area	FID Area
100	93.6	3008	98393	100	5616	119285	2969980
300	280.8	8439	224715	300	16848	285626	6816550
500	468	16600	400583	500	28080	6.33E+05	1.31E+07

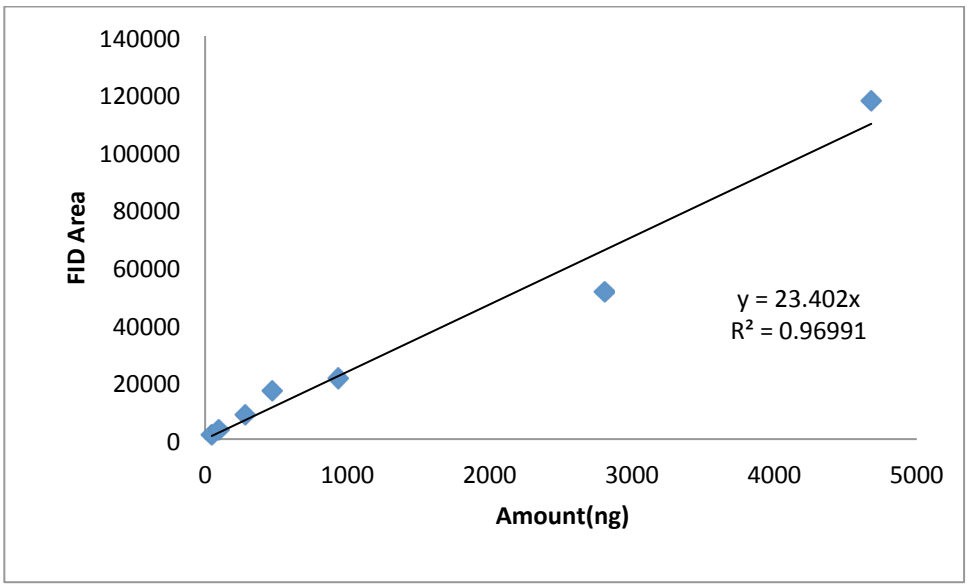

Figure 2-1: Halothane Gas Chromatography calibration Curve with FID

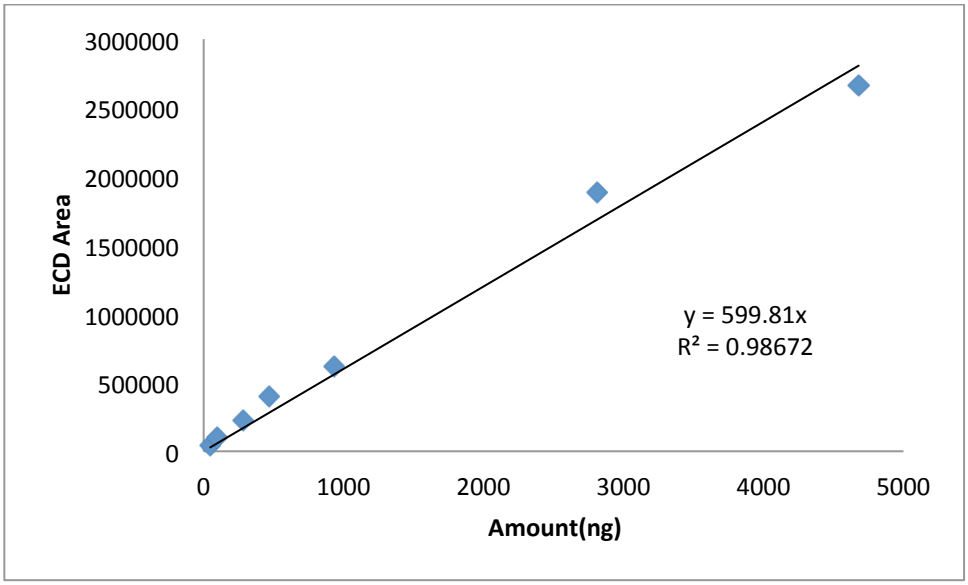

Figure 2-2: Halothane Gas Chromatography calibration Curve with ECD

The electron capture detector (ECD) is generally used for detecting electron-dense compounds (high electronegativity) such as halogenated compounds in gas chromatography and can be 10 to 1000 times more sensitive to these types of compounds than FID. The ECD is more sensitive to compounds containing chlorine than those having only fluorine. Isoflurane, with one chlorine, was the only compound of the three anesthetic gases studied that was detected by the ECD.

However, the ECD was only used briefly for isoflurane in the laboratory when studying this compound. This method was applicable only with a very dilute gas sample where the ECD response was within the range of 27,0571 to 250,380 area counts. For most of the experiments, the FID detector was used for the quantification of the anesthetic gases.

Isoflurane and Sevoflurane Calibration Curves

For the calibration of Isoflurane and Sevoflurane, 10 µl of each anaesthetic in liquid form was injected into the standards bulb containing nitrogen and the mixture was stirrred for half an hour at room temperature. For calibration of Desflurane, 100 µl of a 1% (v/v in air) was injected to the standard bulb contaning nitrogen. Various volumes from the mixtures were injected into the GC to prepare calibration curves. As explained before, only the FID method was selected for detection of these gases. Standard curves were prepared with ranges of 380 to 22800 ng, 374 to 22440 ng, and 90 to 18940 ng, respectively. The individual peaks were sufficiently separated for identification and quantification with retention of approximately 0.722, 0.677 and 0.651 min, respectively.

Table (2-2): The Gas Chromatography Calibration Data for Isoflurane, Sevoflurane and Desflurane

Inject 10 µl Isoflurane and 10 µl Sevoflurane to standards bulb				
Volume(µl)	Sevoflurane Amount(ng)	Isoflurane Amount(ng)	Sevoflurane FID Area	Isoflurane FID Area
50	374	380	14490	8935
100	748	760	28091	18436
200	1496	1520	49817	31854
300	2244	2280	78560	50401
500	3740	3800	120055	78070
Inject 60 µl Isoflurane and 60 Sevoflurane to standards bulb				
50	2244	2280	61356	38682
100	4488	4560	278536	177400
200	8976	9120	395699	251261
300	13464	13680	723410	461620
500	22440	22800	61356	38682

Desflurane amount (ng)	Desflurane FID area
90	633
180	1171
11361	65869
18937	133010

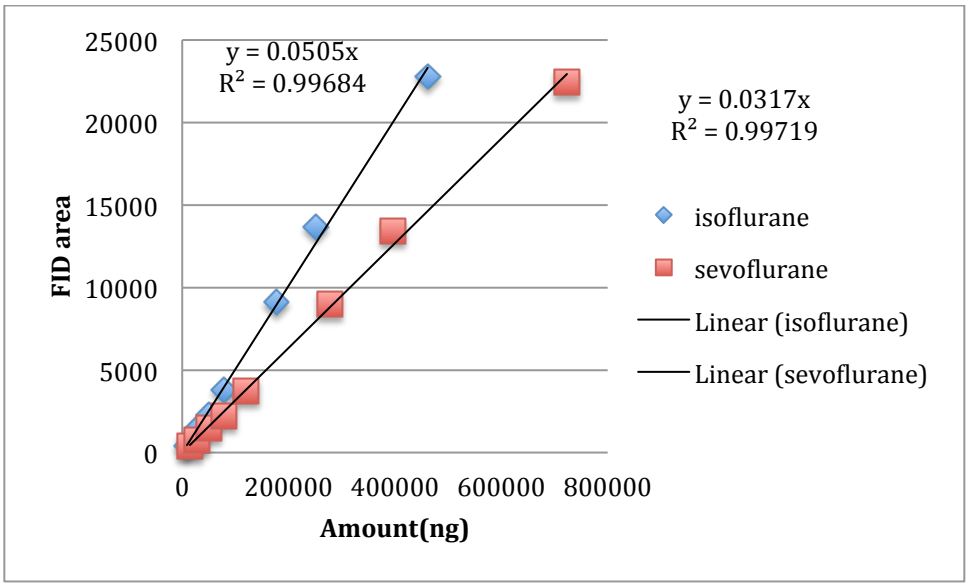

Figure 2-3: Isoflurane and Sevoflurane Gas Chromatography calibration Curve with FID

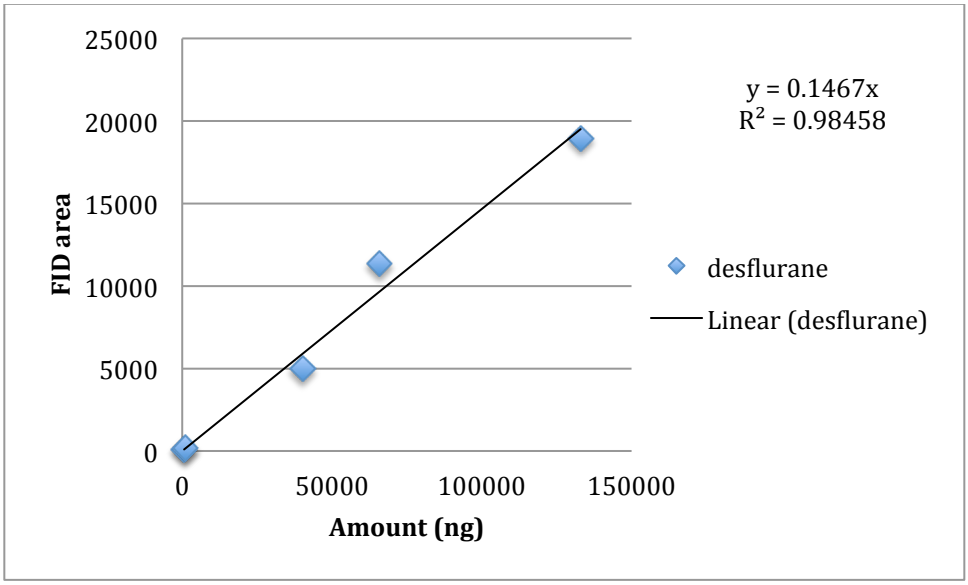

Figure 2-4: Desflurane Gas Chromatography calibration Curve with FID

Therefore, with regards to different retention times for Isoflurane, Sevoflurane and Halothane, their concentration can be easily monitored by the GC in both mixed or separate samples.

2-2 Adsorbent Selection

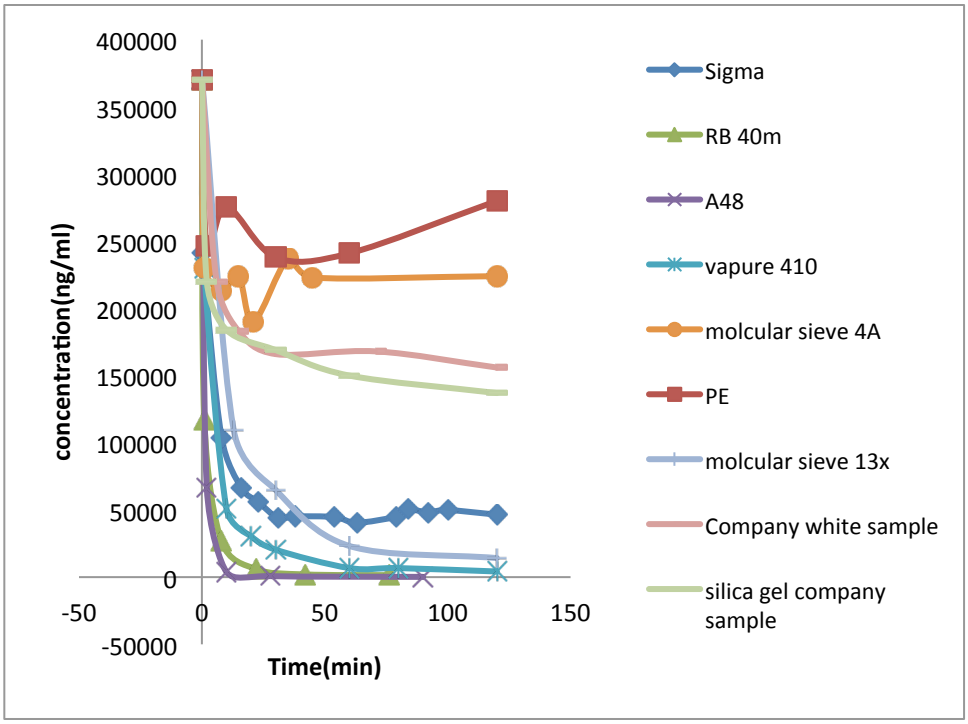

Figure 2-5: The Concentration of the Halothane in the Bulb with different adsorbents over time

The experiments were started with the old anesthetic "Halothane" that is significantly cheaper than the three more modern agents (Isoflurane, Sevoflurane and Desflurane). The price was not the only reason for selection of this compound (around $1.3 per ml of Halothane in comparison to $313 per ml of Isoflurane with respect to Sigma Aldrich supplier price), the similar physical and chemical characteristics of this anesthetic compared to the other three, especially Isoflurane, created a very good source for initiating different tests (more details were explained in Section (1-1)).

To find the best adsorbent, the adsorption of Halothane on different relevant adsorbents was tested (Figure 2-5). An initial volume of Halothane (50 µl) was injected into a glass bulb (volume 250 ml) which contained a known weight of adsorbent. Then, the concentration of Halothane in the bulb was measured by removing a 500 µl gas sample for GC analysis over time. The same experiments were repeated with Isoflurane. The results are shown in Table (2-3). As can be observed in Figure 2-5 and Table 2-3, the GAC 48 (100%) was 1.5 to 4 times more efficient at absorbing the halothane than either the polyethylene (25%), the molecular sieve 4A (38%), the Industrial white sample (57%), and the silica gel (63%) at room temperature. It was similar in adsorption capacity to the Activated Carbon(RB 40M, 99.8%), the Activated Carbon Vapure 410 (98.8%), the Molecular Sieve 13X (96%), and the Sigma Activated Carbon (87%).

Table (2-3): The adsorption properties of relevant adsorbent on Halothane and Isoflurane

Adsorbent Name	Injected Gas volume	Anaesthetic Gas Name	Adsorbent weight (mg)	Gas Concentration at First (ng/ml)	Gas Concentration at Equilibrium (ng/ml)	Adsorbed Anaesthetic gas%
Industrial white sample*	100	Isoflurane	259	598000	352000	41
Silica Gel	100	Isoflurane	262	598000	290000	51
Industrial white sample*	50	Halothane	257	374000	156000	57
Silica Gel	50	Halothane	294	374000	137000	63
Activated Carbon(GAC 48)	100	Isoflurane	294	374000	137000	96.5
Activated Carbon(GAC 48)	50	Halothane	257	374000	Too low to calculate	100
Activated Carbon(Sigma)	50	Halothane	259	598000	20900	87
Activated Carbon(RB 40M)	50	Halothane	254	3.74e5	700	99.8
Activated Carbon(vapure 410)	50	Halothane	257	370000	Too low to calculate	98.8
Molecular Sieve 13x	50	Halothane	255	374000	46500	96
Molecular Sieve 4A	50	Halothane	254	374000	700	38
Poly Ethylene	50	Halothane	252	374000	4200	25

*provided by Class 1 as a possible zeolite type adsorbent

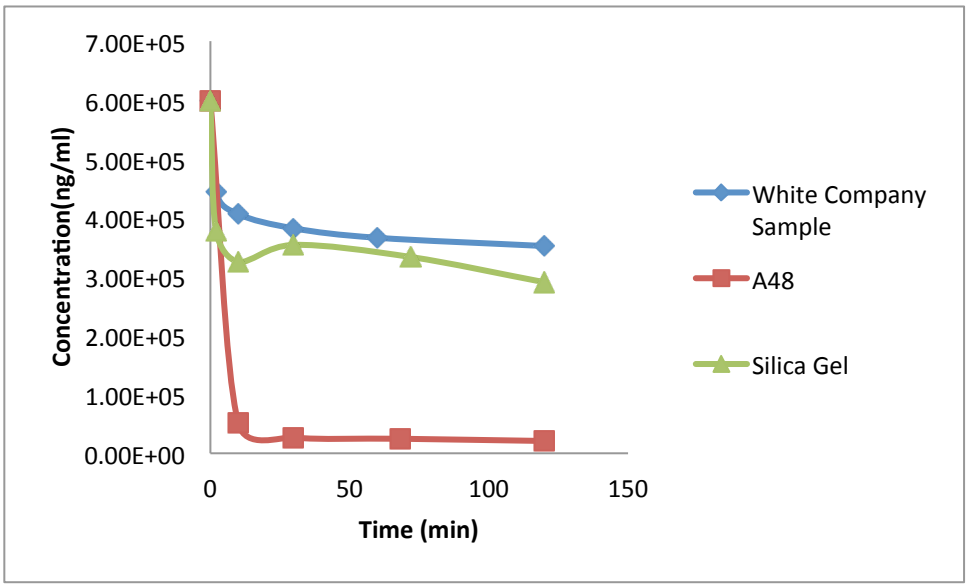

Figure 2-6: The Concentration of Isoflurane in the bulb with adsorbent over time

Activated Carbon Norit GAC 48 M-1869 (GAC A48), as the best adsorbant, demonstrated adsorption efficiencies of 100% with an initial volume of 50 µl of halothane and 96.5% with an initial volume of 100 µl of Isoflurane. As can be observed in Figure (2-6) and Table (2-3), the GAC 48 (96.5%) was 1.8 to 2 times more efficient at absorbing the Isoflurane than the Industrial white sample (41%), and the silica gel (51%) at room temperature.In addition, this particular activated carbon (A48) adsorbed most of the Isoflurane gas molcules in less than 10 minutes of the experiment. Therefore, this adsorbant was significantly more efficient in capturing the anaesthetic gases compared to the other adsorbents tested. In addition, between the different kinds of activated carbon, GAC A48 exhibited the better adsorption at different initial concentrations. The white adsorbent provided by Class 1 Inc., initially used as the adsorbent in a small canister in surgery rooms, was demonstrated to have only half the adsorption capacity of the GAC A48 (Table 2-3). More experiments were completed to investigate the effect of different factors on the adsorption of activated carbon using GAC A48.

2-3 The effect of initial composition of anesthetic gas on the adsorption process

Various initial volumes of halothane were injected into a bulb (250 ml) which contained GAC A48 (with a weight around 255 mg). The concentrations, over a 2-hour period, were measured using the GC. The results are organized in the Table (2-4) and Figure (2-7):

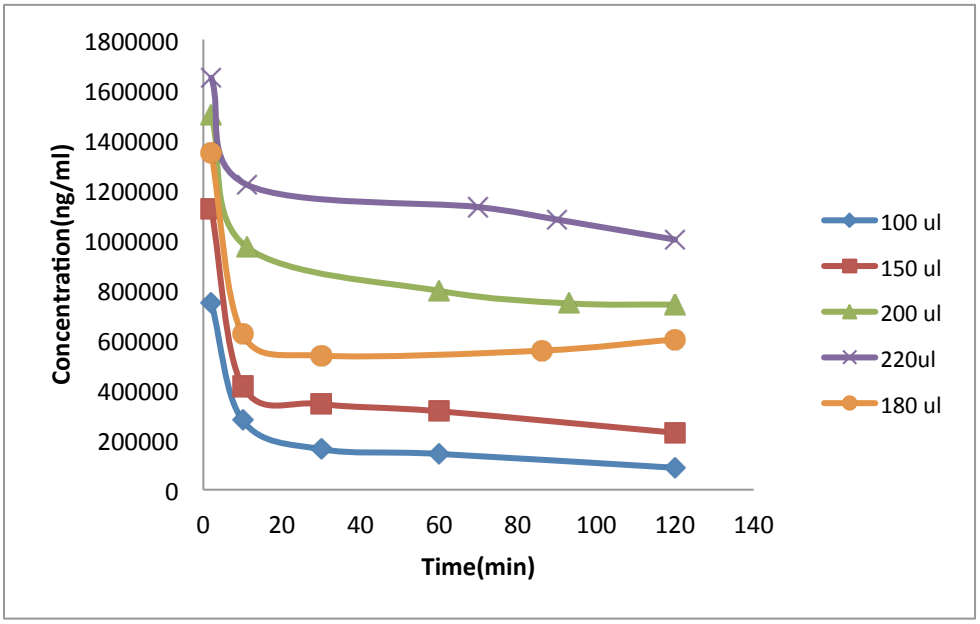

Figure 2-7: The Concentration of Halothane with different initial injected volumes over time

Table (2-4) The Concentration of Halothane in the bulb with different initial injected volumes

Time (min)	Initial halothane Volume(50ul)		Initial halothane Volume(100ul)		Initial halothane Volume(150ul)		Initial halothane Volume(180ul)		Initial halothane Volume(200ul)		Initial halothane Volume(220ul)	
	Concentration (ng/ml)	Adsorption effiency%	Concentration (ng/ml)	Adsorption effiency%	Concentration (ng/ml)	Adsorption effiency%	Concentration (ng/ml)	Adsorption effiency%	Concentration (ng/ml)	Adsorption effiency%	Concentration (ng/ml)	Adsorption effiency%
0	374400	0	0	0	0	0	0	0	0	0	0	0
2	6.68E4	82.16	5.59E5	25.37	7.17E5	35.98	1.10E6	18.52	---	---	----	----
10	3.85E3	98.97	2.77E5	63.02	4.14E5	63.04	9.41E5	30.30	9.70E5	34.90	1.22E6	26.06
30	739	99.80	1.62E5	78.37	3.42E5	69.46	6.49E5	51.93	7.95E5	46.64	1.13E6	31.52
60	-	-	1.43E5	80.91	3.14E5	71.96	3.64E5	73.04	7.45E5	50.00	1.08E6	34.55
120	-	-	8.76E4	88.30	2.27E5	79.73	3.35E5	75.19	7.40E5	50.34	1.00E6	39.39

The same experiments were repeated for isoflurane and sevoflurane. The results are provided in Table(2-5), Table(2-6), Figure(2-8) and Figure(2-9):

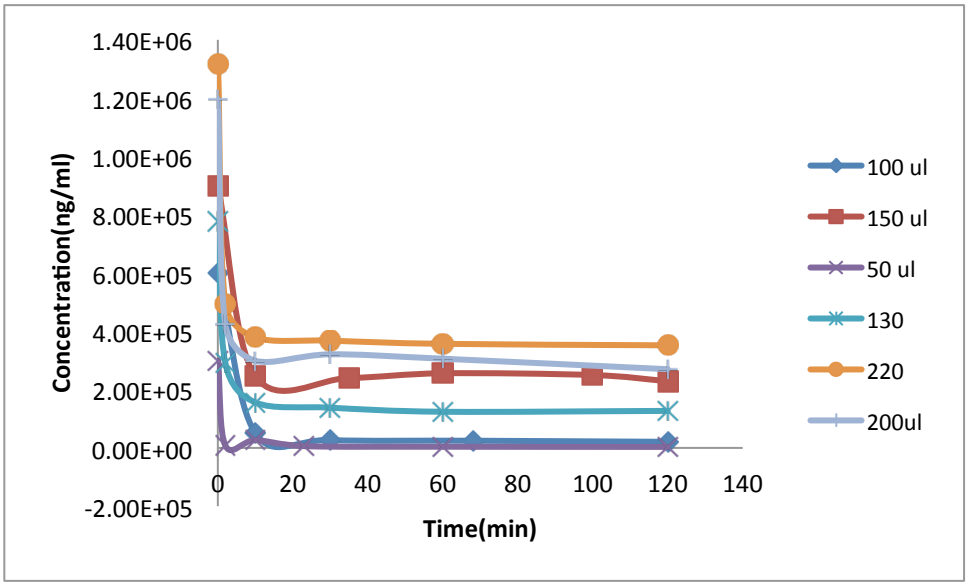

Figure 2-8 The Concentration of Isoflurane in the bulb with different initial injected volumes over time

Table (2-5) The Concentration of Isoflurane in the bulb with different initial injected volumes

Time (min)	Initial Sevoflurane Volume(50ul)		Initial Sevoflurane Volume(100ul)		Initial Sevoflurane Volume(150ul)		Initial Sevoflurane Volume(200ul)	
	Concentration (ng/ml)	Adsorption effiency%	Concentration (ng/ml)	Adsorption effiency%	Concentration (ng/ml)	Adsorption effiency%	Concentration (ng/ml)	Adsorption effiency%
0	2.99E+05	0.00	5.98E+05	0.00	8.98E+05	0.00	11.97E+05	0.00
2	----	----	5.11E+04	91.45	2.46E+05	72.61	6.30E+05	47.37
10	2.70E+04	90.97	2.68E+04	95.52	2.40E+05	73.27	5.43E+05	54.64
30	6162	97.94	2.47E+04	95.87	2.56E+05	71.49	4.99E+05	58.31
60	4059	98.64	2.09E+04	96.51	2.50E+05	72.16	4.72E+05	60.57
120	3462	98.84	2.06E+04	96.51	1.27E+05	74.50	2.29E+05	64.08

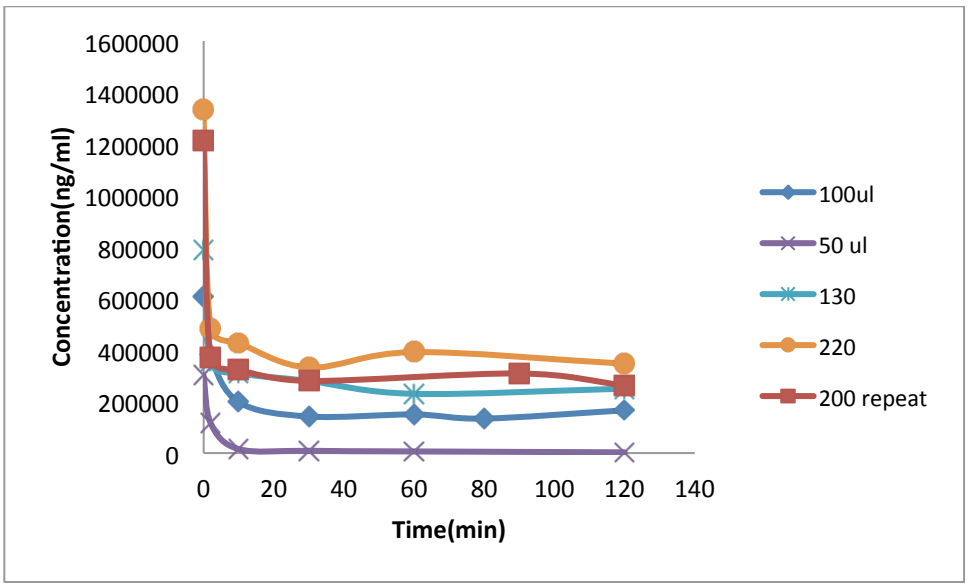

Figure 2-9: The Concentration of Sevoflurane in the bulb with different initial injected volumes over time

Table (2-6): Concentration of Sevoflurane in the bulb with different initial injected volumes

Time (min)	Initial Sevoflurane Volume(50ul)		Initial Sevoflurane Volume(100ul)		Initial Sevoflurane Volume(150ul)		Initial Sevoflurane Volume(200ul)	
	Concentration (ng/ml)	Adsorption effiency%	Concentration (ng/ml)	Adsorption effiency%	Concentration (ng/ml)	Adsorption effiency%	Concentration (ng/ml)	Adsorption effiency%
0	303400	0.00	606800	0.00	910200	0.00	1213600	0.00
2	1.16E+05	61.77	3.82E+05	37.05	4.73E+05	48.03	9.90E+05	18.42
10	1.54E+04	94.92	1.98E+05	67.37	3.90E+05	57.15	5.39E+05	55.59
30	8889	97.07	1.42E+05	76.60	3.70E+05	59.35	5.38E+05	55.67
60	6461	97.87	1.50E+05	75.28	3.30E+05	63.74	6.07E+05	49.98
120	3000	99.01	1.66E+05	72.64	3.16E+05	65.28	6.02E+05	50.40

It can be observed in Table(2-5), Table(2-6), Figure(2-8) and Figure(2-9), Isoflurane with intial volumes of 50, 100, 150 and 200 µl were adsorbed with 98.8, 96.5, 74.5, and 64.1% efficiency, respectively, after 120 minutes. In addition, sevoflurane with intial volumes of 50, 100, 150 and

200 µl were captured with 99.0, 72.6, 65.3, and 50.4 % efficiency in the same time. Therefore, both gases had approximately the same adsorption rate within 30 minutes.

2-4 Mixtures of Isoflurane and Sevoflurane

The other factor that was thought to possibly affect the adsorption process was competition of the Isoflurane and Sevoflurane molecules on the active sites of adsorbent in a mixture of both gases. The adsorption of diverse mixtures of these two anaesthetic gases with more, less, and equal percentages of Isoflurane was tested. The results are shown Figure(2-10), (2-11) and (2-12):

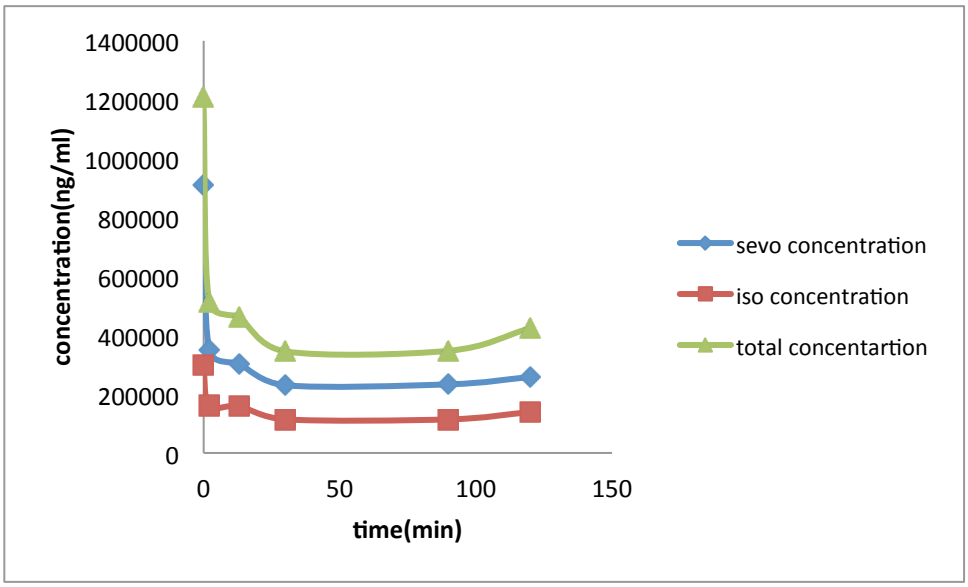

Figure 2-10: The Concentration of Isoflurane and Sevoflurane in the mixture of 50 µl of Isoflurane plus 150 µl of Sevoflurane in the bulb with the activated carbon A48 over time

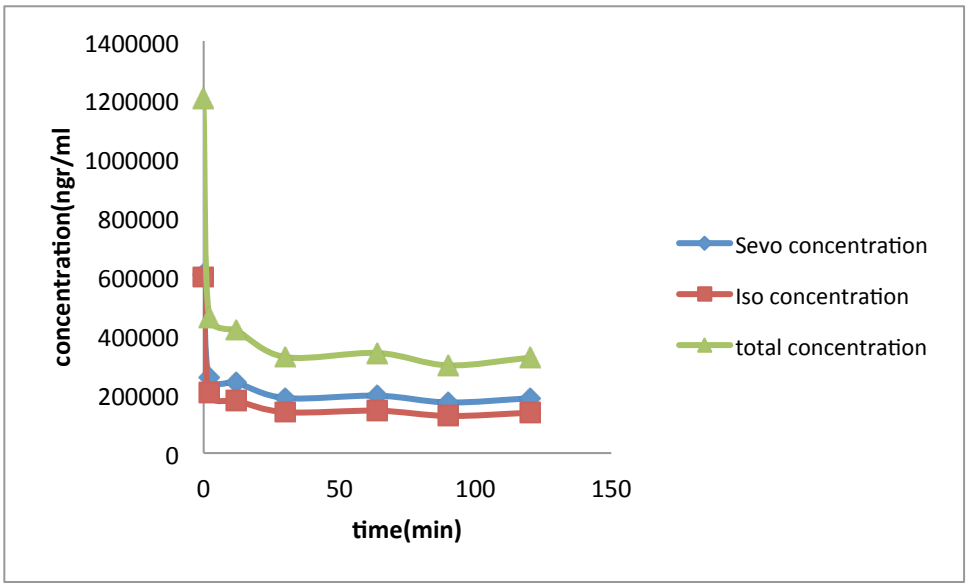

Figure 2-11: The Concentration of Isoflurane and Sevoflurane in the mixture of 100 µl of Isoflurane plus 100 µl of Sevoflurane in the bulb with the activated carbon A48 over time

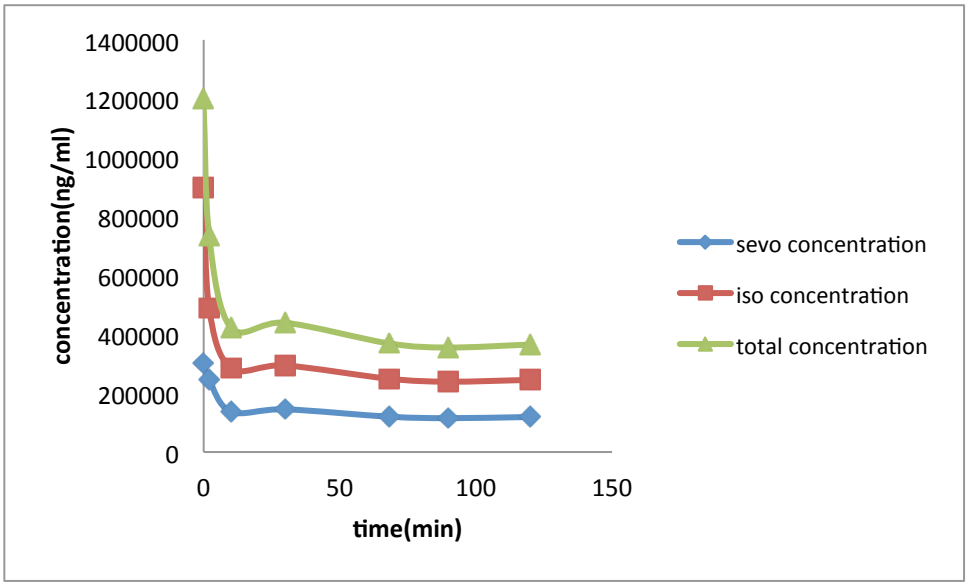

Figure 2-12: The Concentration of Isoflurane and Sevoflurane in the mixture of 150 µl of Isoflurane plus 50 µl of Sevoflurane in the bulb with the activated carbon A48 over time

It was obvious that in a mixture of gases there was no significant competition effects for the adsorption on the activated carbon. Therefore, based on an initial composition, gas molecules occupied the activated carbon sites until an equilibrium was reached.

Table (2-7): The isoflurane and sevoflurane concentrations in the bulb with activated carbon A48 in different mixtures of them

Inject 150 µl Sevoflurane and 50 µl Isoflurane to bulb with 257 mg GAC			
Time(min)	Sevoflurane Concentration(ng/ml)	Isoflurane Concentration(ng/ml)	Total Concentration(ng/ml)
0	910200	299200	1209400
2	3.51E+05	1.61E+05	5.13E+05
13	3.03E+05	1.59E+05	4.62E+05
30	2.30E+05	1.14E+05	3.45E+05
90	2.33E+05	1.14E+05	3.47E+05
120	2.58E+05	1.39E+05	4.24E+05
Inject 50 µl Sevoflurane and 150 µl Isoflurane to bulb with 258.5 mg GAC			
Time(min)	Sevoflurane Concentration(ng/ml)	Isoflurane Concentration(ng/ml)	Total Concentration(ng/ml)
0	303400	897600	1201000
2	2.46E+05	4.90E+05	7.36E+05
10	1.37E+05	2.86E+05	4.23E+05
30	1.45E+05	2.94E+05	4.39E+05
68	1.20E+05	2.49E+05	3.69E+05
90	1.15E+05	2.39E+05	3.55E+05
120	1.19E+05	2.45E+05	3.65E+05
Inject 100 µl Sevoflurane and 100 µl Isoflurane to bulb with 255 mg GAC			
Time(min)	Sevoflurane Concentration(ng/ml)	Isoflurane Concentration(ng/ml)	Total Concentration(ng/ml)
0	606800	598400	1205200
2	2.57E+05	2.04E+05	4.61E+05
12	2.39E+05	1.78E+05	4.17E+05
30	1.87E+05	1.39E+05	3.26E+05
64	1.95E+05	1.44E+05	3.40E+05
90	1.72E+05	1.26E+05	2.98E+05
120	1.86E+05	1.37E+05	3.24E+05

2-5 The effect of temperature on the adsorption process

The initial volumes of 100, 150 and 200 µl of halothane were selected to investigate the effect of temperature on the Gac A 48 adsorption. Each adsorption process was performed at three temperatures 23, 35 and 50°C that expected to operate at the planned hospital emission system.(Grand River Hospital)

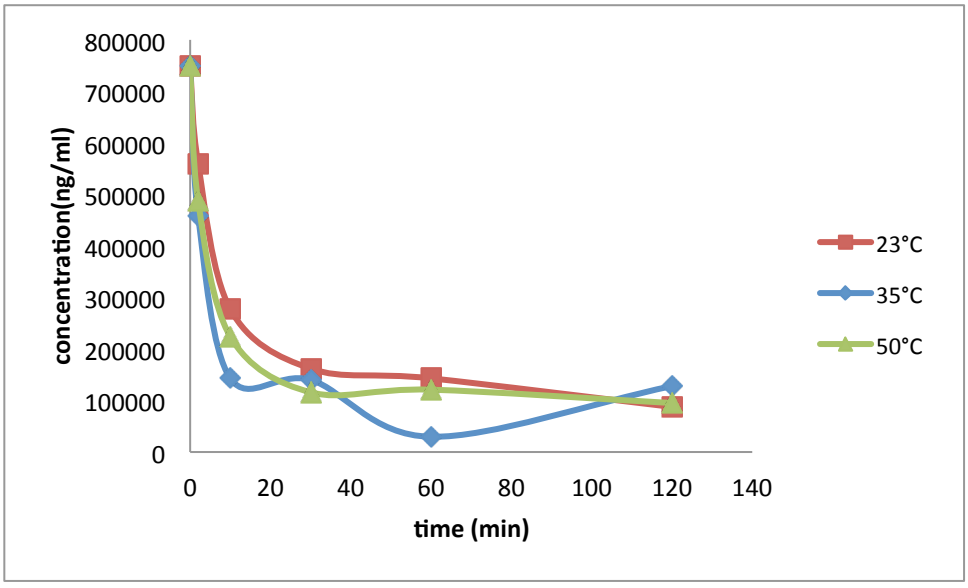

Figure 2-13: The Concentration of Halothane in the bulb with the activated carbon A48 at different temperature over time with the 100 µl initial injected volume

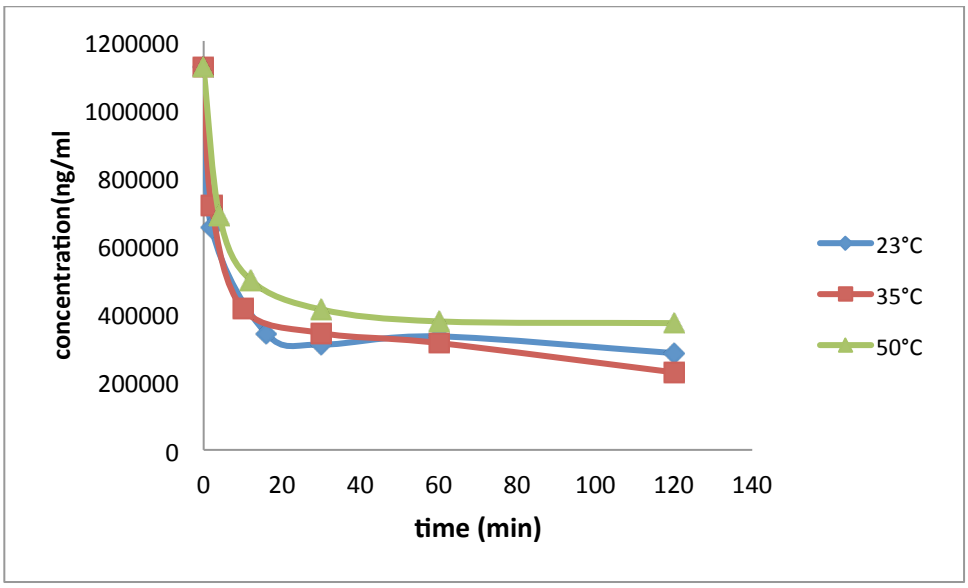

Figure 2-14: The Concentration of Halothane in the bulb with the activated carbon A48 at different temperature over time with the 150 µl initial injected volume

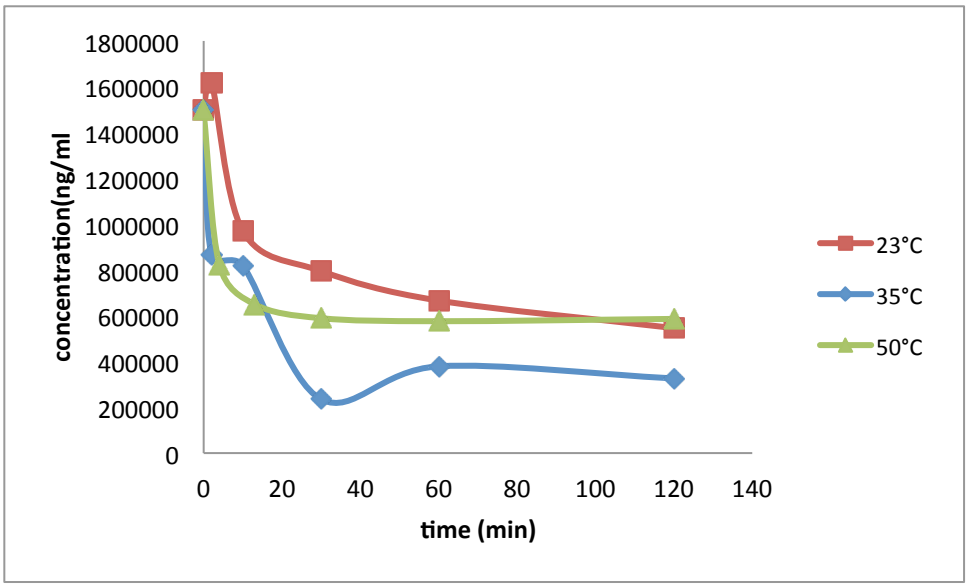

Figure 2-15: The Concentration of Halothane in the bulb with the activated carbon A48 at different temperature over time with the 200 µl initial injected volume

Table (2-8): Concentration of Halothane in the bulb with the activated carbon A48 at different temperature

Inject 100 µl halothane to bulb with GAC A48			
Time(min)	Concentration in 23C(ng/ml)	Concentration in 35C(ng/ml)	Concentration in 50C(ng/ml)
0	748800	748800	748800
2	559355	4.58E+05	4.87E+05
10	277361	1.43E+05	2.24E+05
30	161890	1.40E+05	1.16E+05
60	143013	2.96E+04	1.21E+05
120	87600	1.28E+05	9.51E+04
Inject 150 µl halothane to bulb with GAC A48			
Time(min)	Concentration in 23C(ng/ml)	Concentration in 35C(ng/ml)	Concentration in 50C(ng/ml)
0	1123200	1123200	1123200
2	717357	6.52E+05	6.88E+05
16	414549	3.41E+05	4.98E+05
30	342765	3.09E+05	4.12E+05
60	313614	3.33E+05	3.77E+05
120	2.28E+05	2.84E+05	3.72E+05
Inject 200 µl halothane to bulb with GAC A48			
Time(min)	Concentration in 23C(ng/ml)	Concentration in 35C(ng/ml)	Concentration in 50C(ng/ml)
0	1497600	1497600	1497600
2	1.62E+06	8.67E+05	8.23E+05
10	9.70E+05	8.18E+05	6.50E+05
30	7.95E+05	2.35E+05	5.89E+05
60	6.66E+05	3.79E+05	5.76E+05
120	5.46E+05	3.26E+05	5.86E+05

The results demonstrated that, within experimental error, there was no detectable effect of temperature on the adsorption over the range of 23 to 50°C, which is same temperature used for the adsorption process in a tank of adsorbent in a hospital emission system.

2-6 The Isotherm of adsorption

The process of adsorption is usually studied through the adsorption isotherm. It is the graph or equation comparing the amounts of adsorbent at equilibrium (C∞(ng/ml)) with the mass of species adsorbed over the mass of adsorbent (q) at constant temperature.

Container Isotherm

Different volumes of Halothane were injected into the 250 ml bulb without any adsorbent to observe the amount of halogenated compound that was adsorbed by a container walls. The results for the isotherm of Halothane on the glass container are shown as follows:

Table (2-9): Different parameters for calculating the adsorption isotherm of Halothane on the glass container

Halothane volume(µl)	C0(ng/ml)	C∞(ng/ml)	W(container weight) (gr)	q(ng/ng)
50	374400	344020	131	5.79771E-05
100	748800	503347	131	0.000468422
150	1123200	734490	131	0.000741813
200	1497600	1102170	131	0.000754637

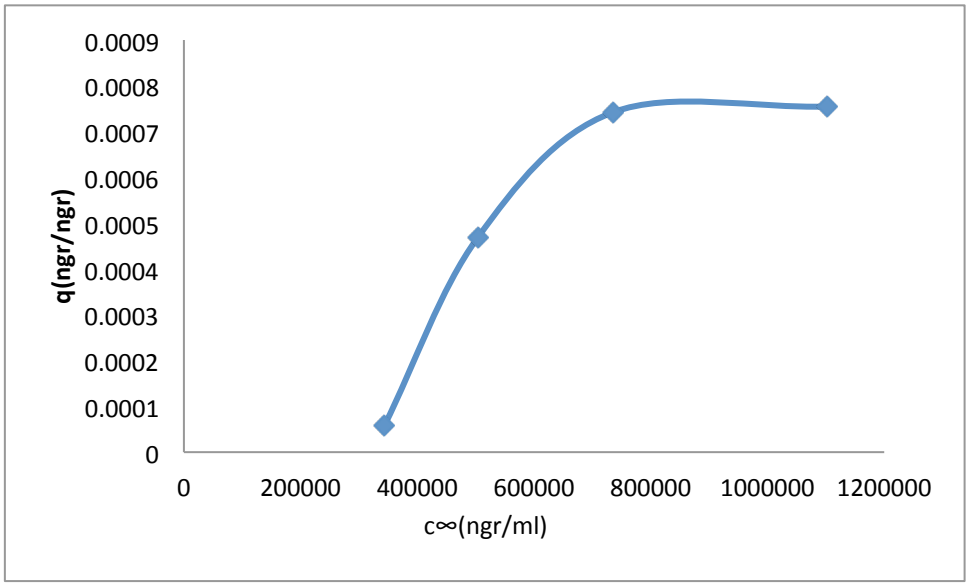

Figure 2-16: Masses of Halothane adsorbed over the glass container (q) based on amount of Halothane at equilibrium (C∞(ng/ml))at room temperature

The results demonstrate that the amount of adsorbed halothane by the container walls was very low in comparison to the amount of gas adsorbed to the activated carbon, i.e. the container did not have any special effect on the adsorption process. In fact, since the container q was 1000 times lower than the adsorbent q, it was neglected in the isotherm calculation .

The Anesthetics Isotherm:

The isotherms of Halothane, Isoflurane and Sevoflurane on the activated carbon A48 are pictured below:

Table (2-10): Different parameters for calculating the adsorption isotherm of Halothane on the activated carbon A48

Halothane volume(µl)	C0(ng/ml)	C∞(ng/ml)	W(activated carbon weight(ng)	q(ng/ng)
100	748800	87600	2.53E+08	6.53E-01
150	1123200	227750	2.57E+08	8.71E-01
180	1347840	3.35E+05	2.56E+08	9.89E-01
200	1497600	5.46E+05	2.57E+08	9.26E-01
220	1647360	6.45E+05	2.54E+08	9.87E-01

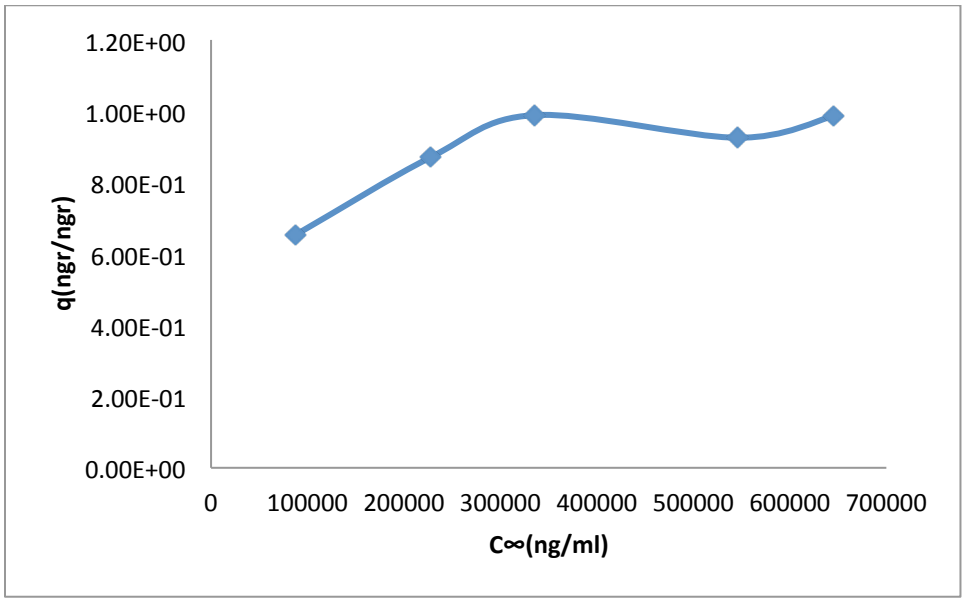

Figure 2-17: Masses of Halothane adsorbed over the activated carbon A48 (q) based on amount of Halothane at equilibrium (C∞(ng/ml))at room temperature

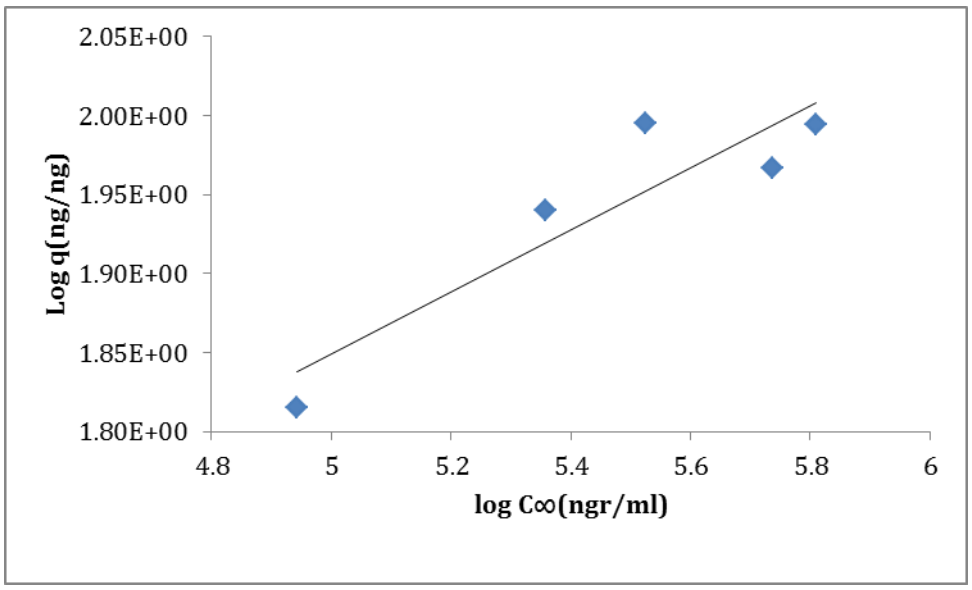

Figure 2-18: Log q (ng/ng)over log (C∞(ng/ml))at room temperature for Halothane on the activated carbon A48

Freundlich: q = 0.868 C^0.196 where q: ng/ng C: ng/ml

Table (2-11): Different parameters for calculating the adsorption isotherm of Isoflurane on the activated carbon A48

Isoflurane volume(µl)	C0(ng/ml)	C∞(ng/ml)	W(activated carbon weight(ng)	q(ng/ng)
90	538560	4.58E+04	2.58E+08	4.77E-01
100	598400	6.73E+04	2.57E+08	5.17E-01
120	718080	1.82E+05	2.55E+08	5.25E-01
150	897600	3.20E+05	2.57E+08	5.63E-01
170	1017280	3.38E+05	2.55E+08	6.66E-01
190	1136960	4.47E+05	2.57E+08	6.70E-01

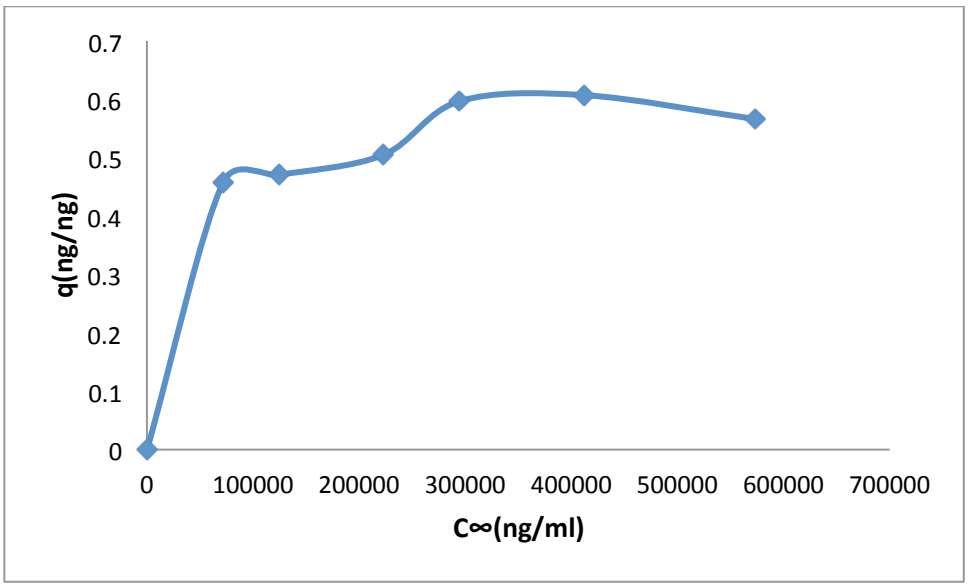

Figure 2-19: Masses of Isoflurane adsorbed over the activated carbon A48 (q) based on the amounts of Isoflurane at equilibrium (C∞(ng/ml))at room temperature

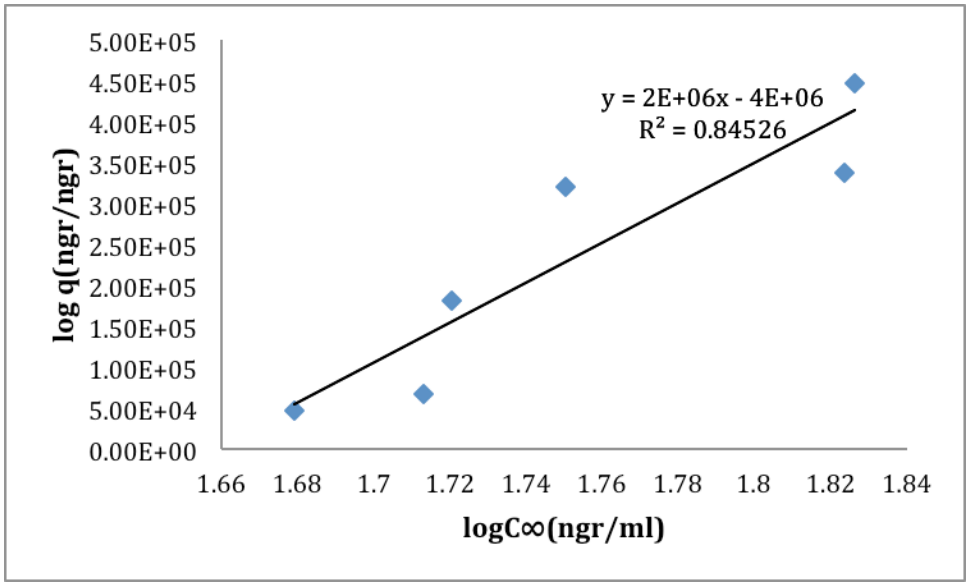

Figure 2-20: Log q (ng/ng) over log (C∞(ng/ml))at room temperature for Isoflurane on The activated carbon A48

Freundlich: q = -4E+6 C^2E+6 where q: ng/ng C: ng/ml

Table (2-12): Different parameters for calculating the adsorption isotherm of Sevoflurane on the activated carbon A48

Isoflurane volume(µl)	C0(ng/ml)	C∞(ng/ml)	W(activated carbon weight(ng)	q(ng/ng)
90	546120	7.20E+04	2.59E+08	4.58E-01
100	606800	1.25E+05	2.56E+08	4.71E-01
120	728160	2.23E+05	2.51E+08	5.05E-01
150	910200	2.94E+05	2.58E+08	5.97E-01
170	1031560	4.12E+05	2.55E+08	6.07E-01
190	1152920	5.73E+05	2.56E+08	5.66E-01

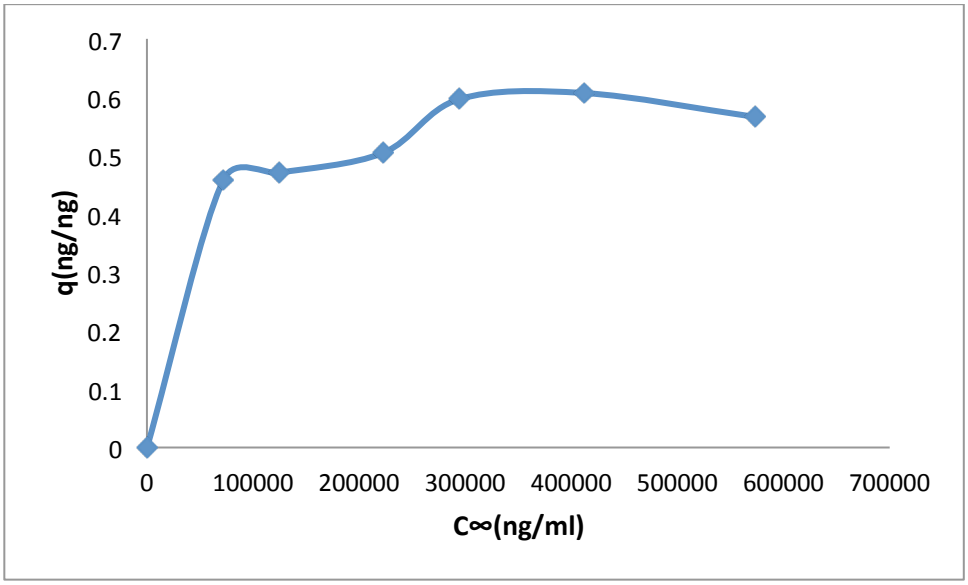

Figure 2-21: Masses of Sevoflurane adsorbed over the activated carbon A48 (q) based on the amounts of Sevoflurane at equilibrium (C∞(ng/ml))at room temperature

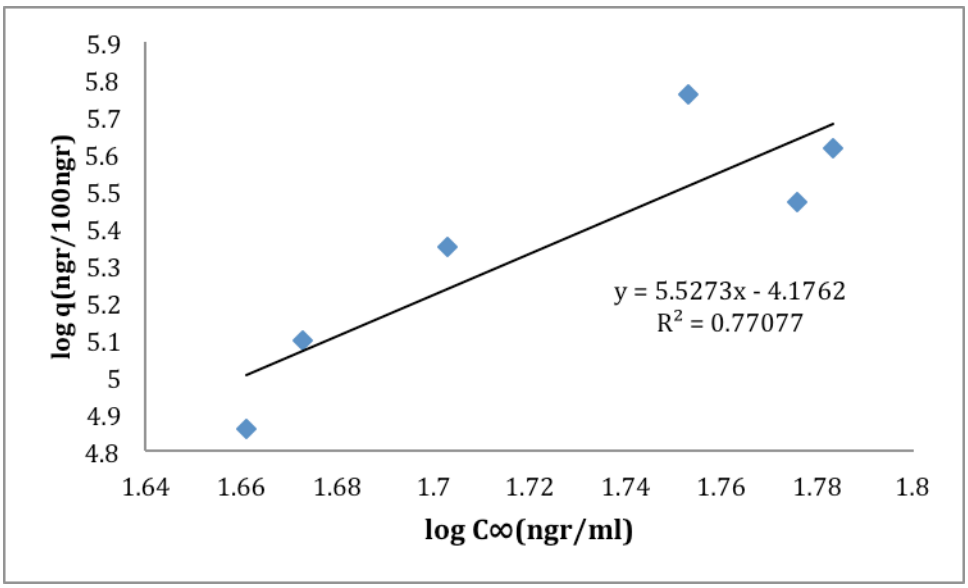

Figure 2-22: Log q (ng/ng)over log (C∞(ng/ml))at room temperature for Sevoflurane on the activated carbon A48

Freundlich: q = -4.1762 C^5.5273 where q: ng/100ng C: ng/ml

In all three graphs the quantity of adsorbed gas on the activated carbon increased until it reached a constant value. As these concentrations are relatively low and do not cover a very wide range, the saturation behavior that is reperesented by the Langmuir isotherm was not expected to be present. Therefore, the Freundlich isotherm was assumed to be the best theory for explaining the adsorption behavior of the anesthetics on the activated carbon A48. The curves in Figures (2-18), (2-20) and (2-22) were used to find the Freundlich equation constants (k and n in equation $q = kc^n$). With regards to the R^2 value, the K and n were measured with satisfactory accuracy for the purposes of comparison and preliminary design. Although Isoflurane and sevoflurane have some similarity in their chemical and physical properties, their K and n constants were found to be significantly different.

2-7 Desorption Process In Batch System

TGA (Thermogravimetric analysis) test

The desorption amount of the gases from these samples was monitored by TGA (Thermogravimetric analysis). To quantify the degree of desorption, all samples of GAC A48

with the adsorbed gases at the equilibrium concentration were first removed from the bulb and allowed to sit in the air for 10 min to ensure any room-temperature off-gassing was completed before the TGA test commenced. This was required to ensure that desorption commenced at the same initial desorption temperature for all samples. As a control, the raw activated carbon without any adsorbed gases was tested first as a reference sample. As can be observed in Figure 2-23, the weight (%) of the sample reduced from 100 % to approximately 91 % after 400 ^{o}C and there was an initial peak observed at 50 ^{o}C related to the changing weight (deriv Weght (%/^{o}C)) in the sample. This was attributed to the loss of water only from the activated carbon sample.

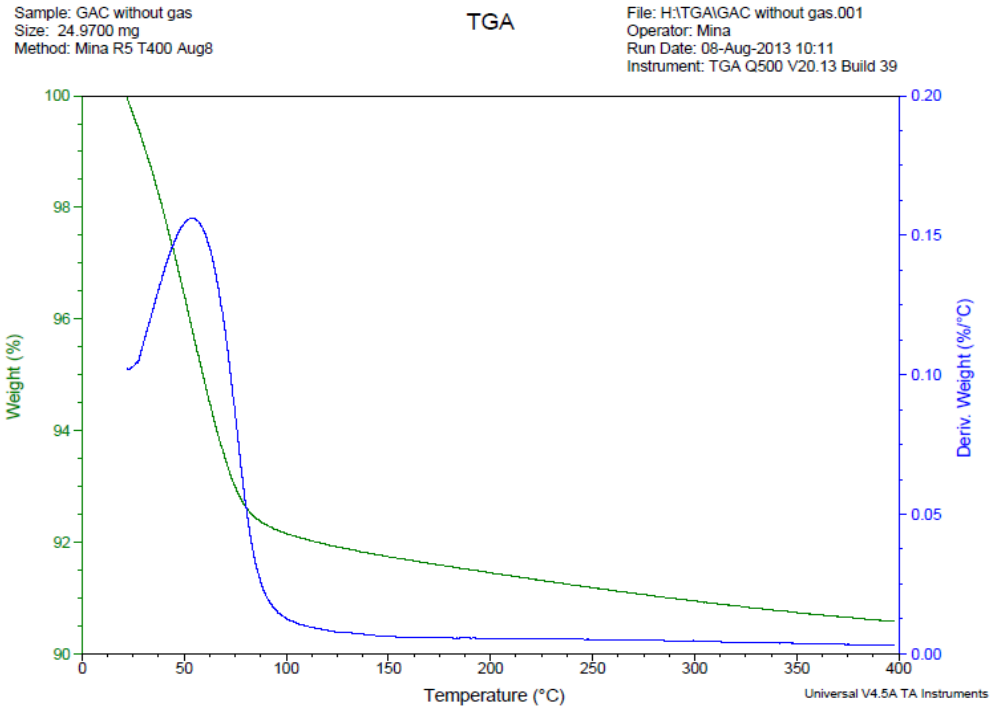

Figure 2-23: The raw activated A48 carbon TGA test

TGA tests were performed with different GAC A48 samples that had been exposed to injected volumes of 50, 100, 150 and 200 µl Halothane, and 200 µl of isoflurane and sevoflurane.

As can be observed in Table 2-13 and Figures 2-24 to 2-27, the desorption percentages were the same (26 %) for the 100, 150, and 200 µl of halothane injected. For the 50 µl initial injection, however, the desorption percentage was only 20 %. This was attributed to the presence of

unoccupied adsorption sites in the carbon, which were all occupied at the higher injected volumes.

For Isoflurane and Sevoflurane, only one TGA sample using an injected volume of 200 µl was analyzed. As can be observed in Figures 2-28 and 2-29, the behaviour is similar to that observed for the 200 µl halothane. Therefore, it was concluded that the maximum mass of anaesthetic gas that could be adsorbed was equal to approximately 26 % of the initial activated carbon mass.

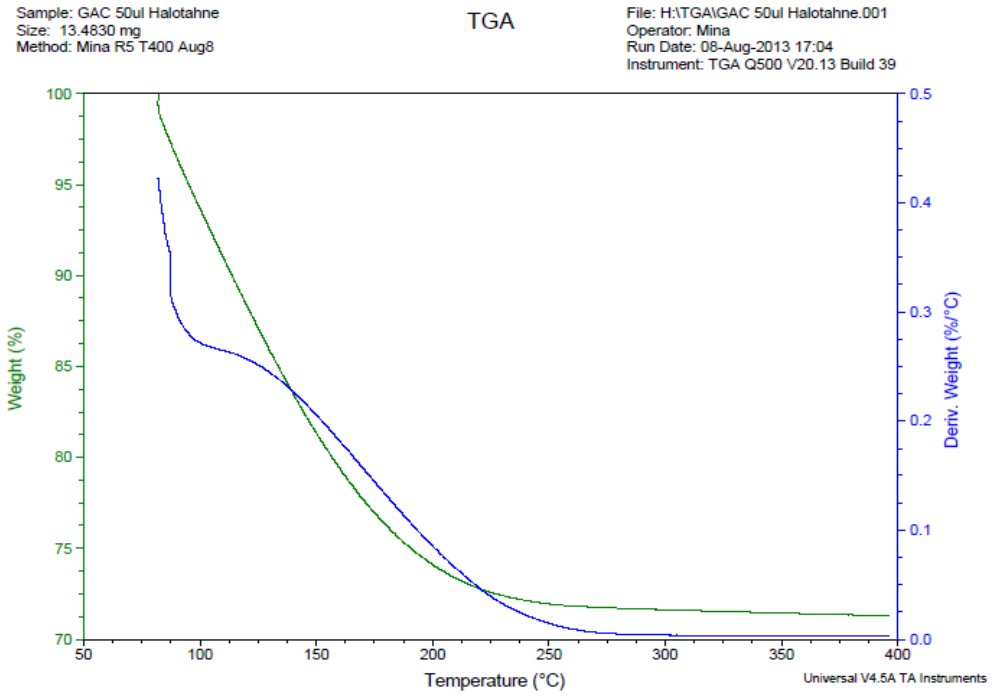

Figure 2-24: The Activated carbon A48 TGA test after adsorption in the bulb with 50 µl initial injected Halothane

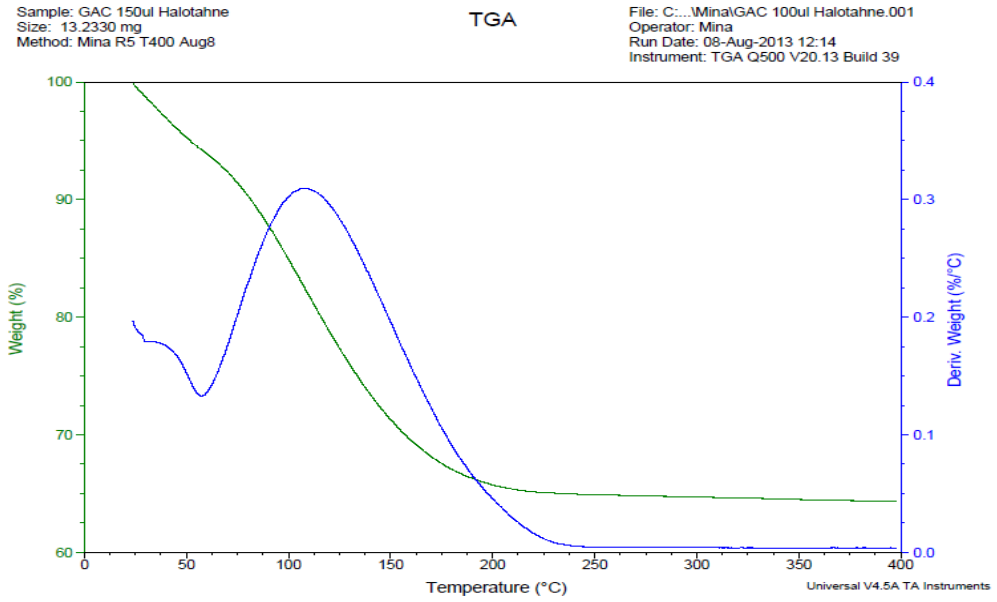

Figure 2-25: The Activated carbon A48 TGA test after adsorption in the bulb with 50 µl initial injected Halothane

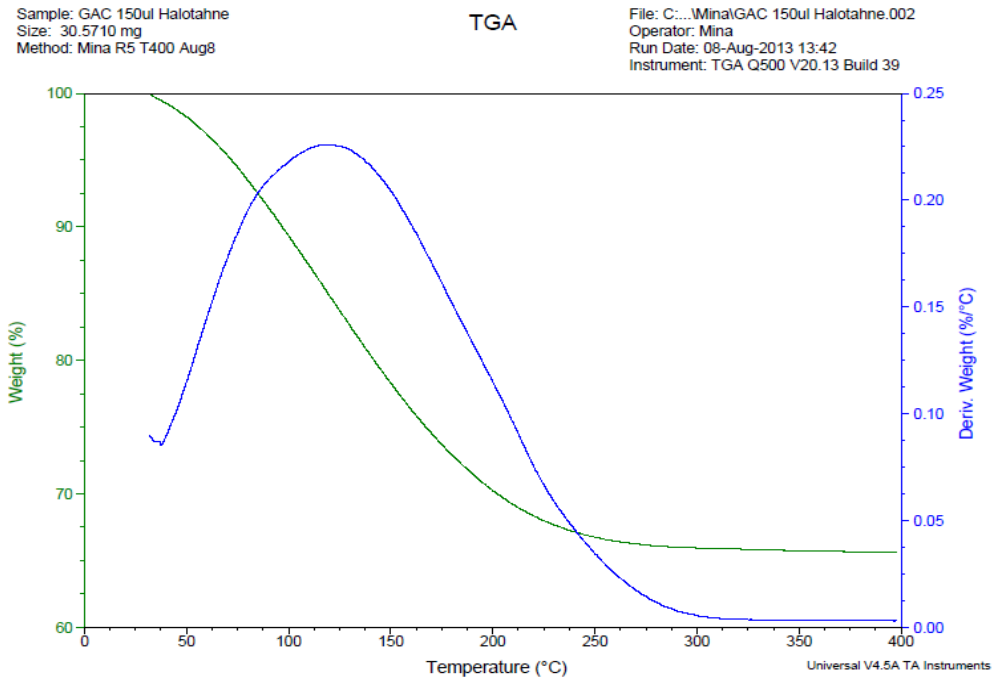

Figure 2-26: The Activated carbon A48 TGA test after adsorption in the bulb with 150 µl initial injected Halothane

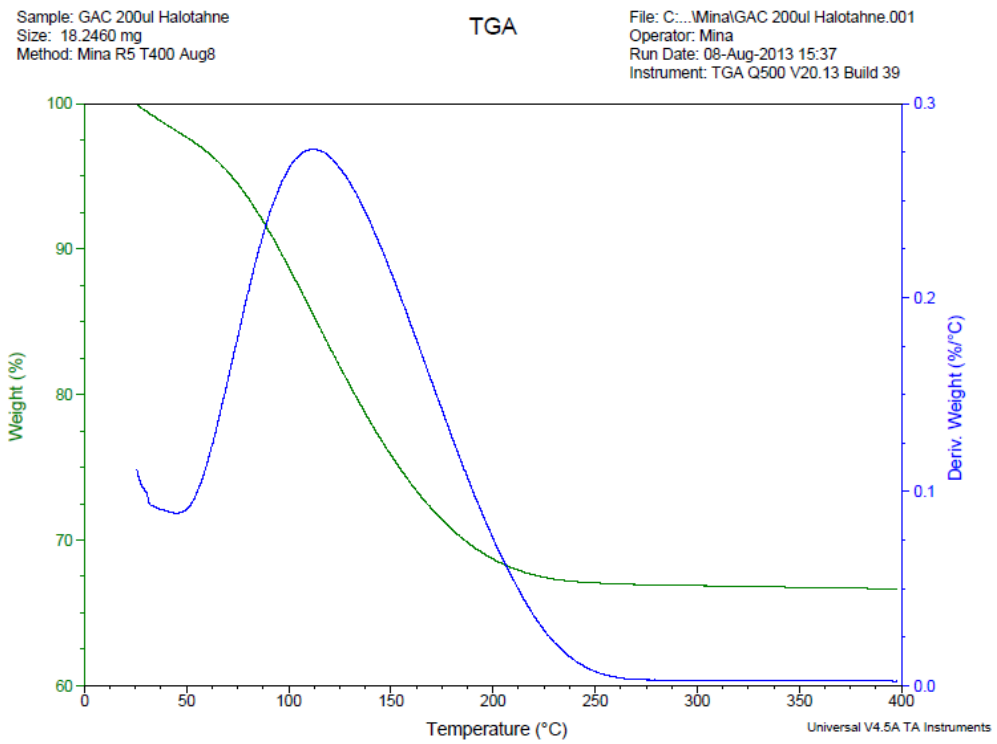

Figure 2-27: The Activated carbon A48 TGA test after adsorption in the bulb with 200 µl initial injected Halothane

Table (2-13): Isotherm parameters based on Desorption

Halothane Volume(µl)	Initial Desorbent weight(mg)	Total Desorption-humidity percentage	Total desorption Weight(mg)	q(mg/mg) Based on Desorption
50	256	20	51.2	0.2
100	258	26	67.08	0.26
150	256.5	26	66.69	0.26
200	258	26	67.08	0.26

Table (2-14): Isotherm parameters based on Adsorption

Halothane Volume(µl)	C0(ng/ml)	C∞(ng/ml)	W(activated carbon weight(ng)	q(mg/mg) Based on adsorption
50	374400			
100	748800	87600	253000000	0.87106
150	1123200	227750	257000000	0.925681
200	1497600	546000	257000000	0.65336

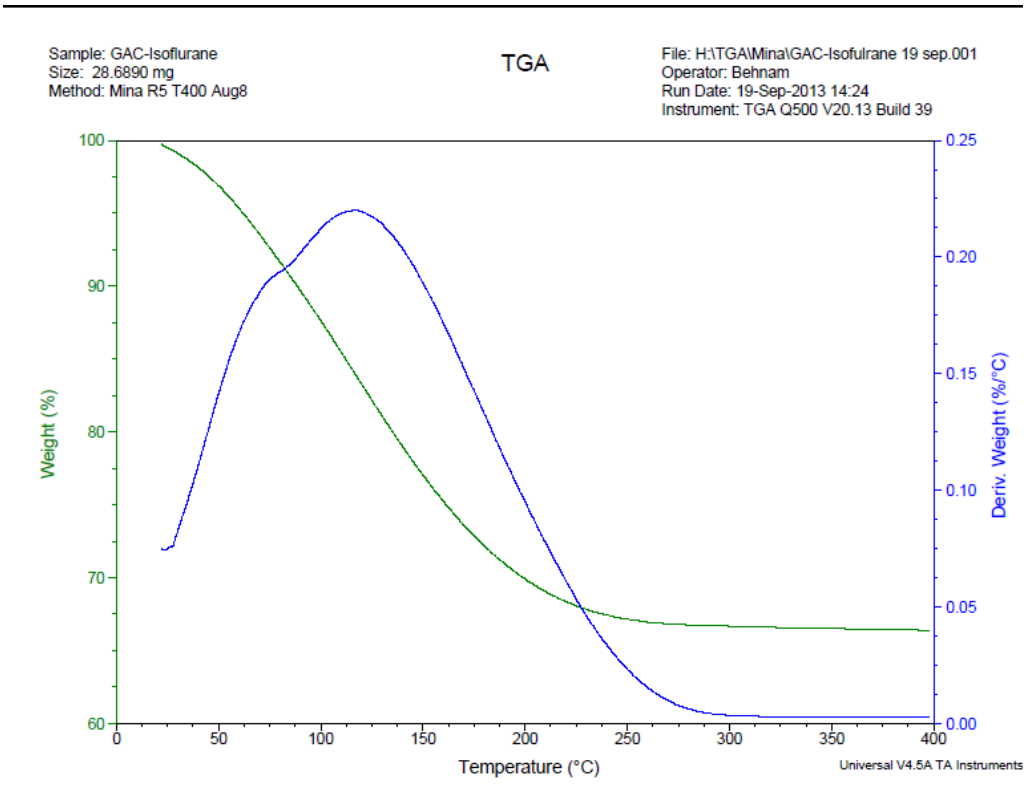

Figure 2-28: The Activated carbon A48 TGA test after adsorption in the bulb with 200 µl initial injected Isoflurane

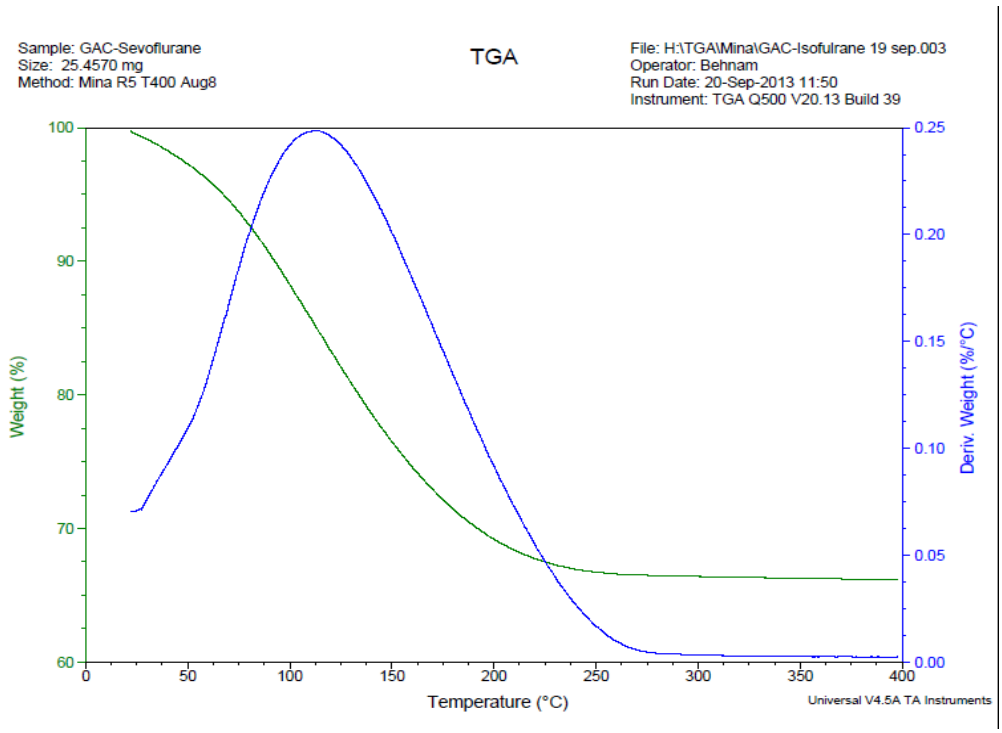

Figure 2-29: The Activated carbon A48 TGA test after adsorption in the bulb with 200 µl initial injected Sevoflurane

2-8 Continuous system

Designing a continuous system

To observe the adsorption by activated carbon in a continuous system, a specific concentration of anaesthetic gas, using nitrogen as the carrier gas, was passed through a column of activated carbon. The concentration of anaesthetic gas was measured before and after the column using the GC. Since only limited sources of anaesthetic gases were available, experiments were limited to the necessary tests that were similar to a hospital emission setting.

The maximum adsorption capacity in a column of Activated Carbon A48

Different volumes of anaesthetic gas were injected into a flask that was connected to the column containing adsorbent (Gac A48). A known amount of anaesthetic gas was passed from the flask to the column by the carrier gas at a constant flow rate. Then, the concentration of anaesthetic gas, exiting the column of activated carbon, was measured during the time required to obtain the maximum adsorption capacity, which was a maximum amount of anaesthetic gas that could be injected into the flask without detecting any GC peak after the column.

Figure 2-30: The laboratory column of activated carbon for testing continuous experiments

Table (2-15): Maximum adsorption in continuous tests with different flow rates

Flow Rate(ml/min)	Maximum adsorption(µl)	Adsorbent/ Weight(g)	Anaesthetic Gas
200	900	A48/5	Halothane
500	700	A48/5	Halothane
700	600	A48/5	Halothane

In a batch experiment, the Gac A48 adsorbed an amount of anaesthetics gas equal to 26% of its initial weight. In the continuous experiment, using a flow rate of 200 ml/ min, the adsorption weight was around 33% of the GAC weight in the column before breakthrough was detected. This amount decreased to 21% with an increasing flow rate up to 700 ml/min. Although the batch and continuous experimental conditions were not the same, these results predicted a good adsorbent pattern for the GAC A48 in the continuous system, which was more representative of the hospital emission system.

2-9 The industrial set up

A stainless steel tank with a volume of 33.98 liters containing 14.514 kg of activated carbon was set up at the Grand River Hospital for an industrial experiment .Emissions from the surgery rooms were collected and diluted with the air from the main hospital compressor .After dilution, the air flowed through a dryer for water removal before reaching the absorbent tank . There are two tanks of adsorbent but only one of them is online at any one time. The input and output of the absorbent tank was connected to an FTIR for monitoring of the composition of the combined anesthesia gases . More details are shown in Figure (2-31).

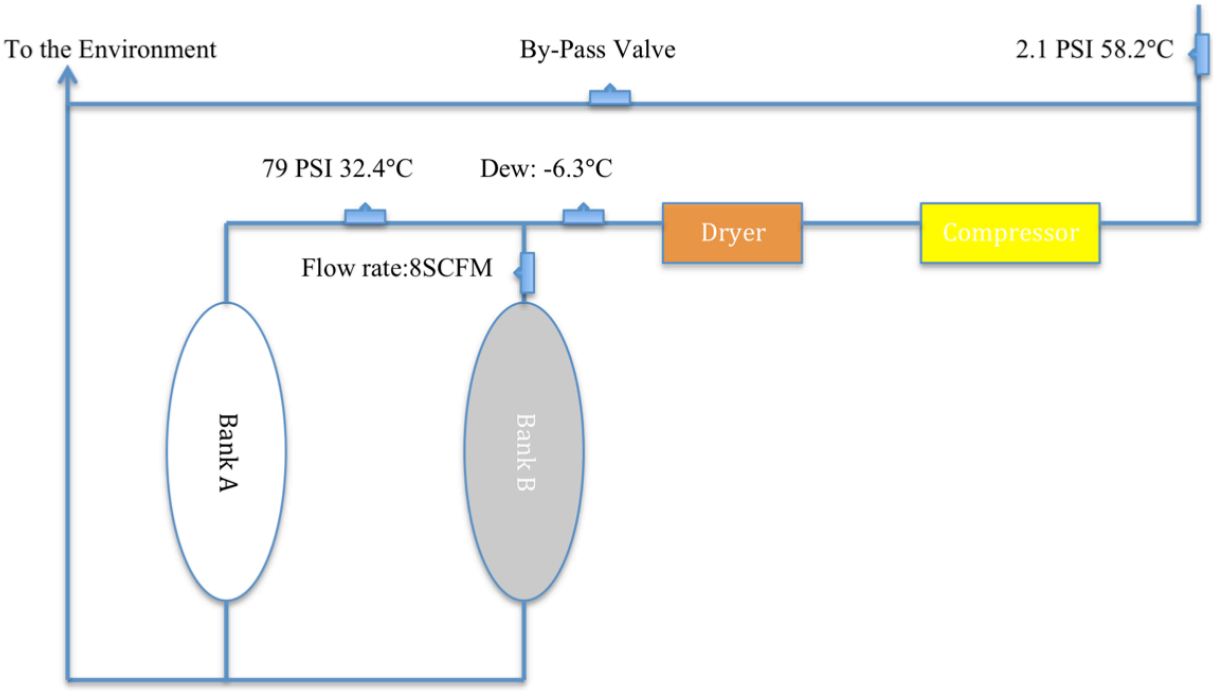

Figure 2-31: The overview of the Grand River Hospital Halogenated Drug Recovery System(HDR system)

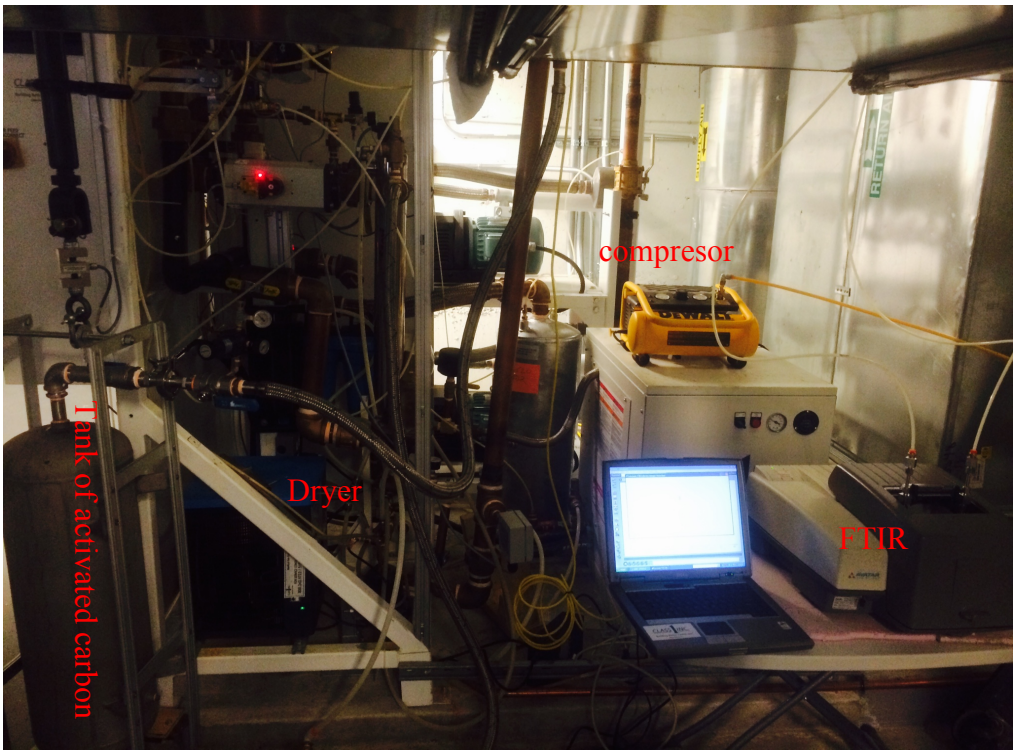

Figure 2-32: the Grand River Hospital Halogenated Drug Recovery System (HDR system)

2-10 FTIR Calibration

The Nicolet Avatar 360 FT-IR was purchased by Class 1 Inc. as an easy to use instrument to set up at the hospital to monitor, online, the composition of the combined gases, Sevoflurane, Isoflurane and Desflurane, in the input and output streams of the adsorbent tank.

The first step was to calibrate this instrument for the three anesthetic gases. For calibration of the instrument, a specific wave number or range of wave numbers was selected, unique to each of the anesthetic gases, such that the concentrations of the individual gases could be determined. The selected peak or peaks should be constant for each gases at different concentrations.

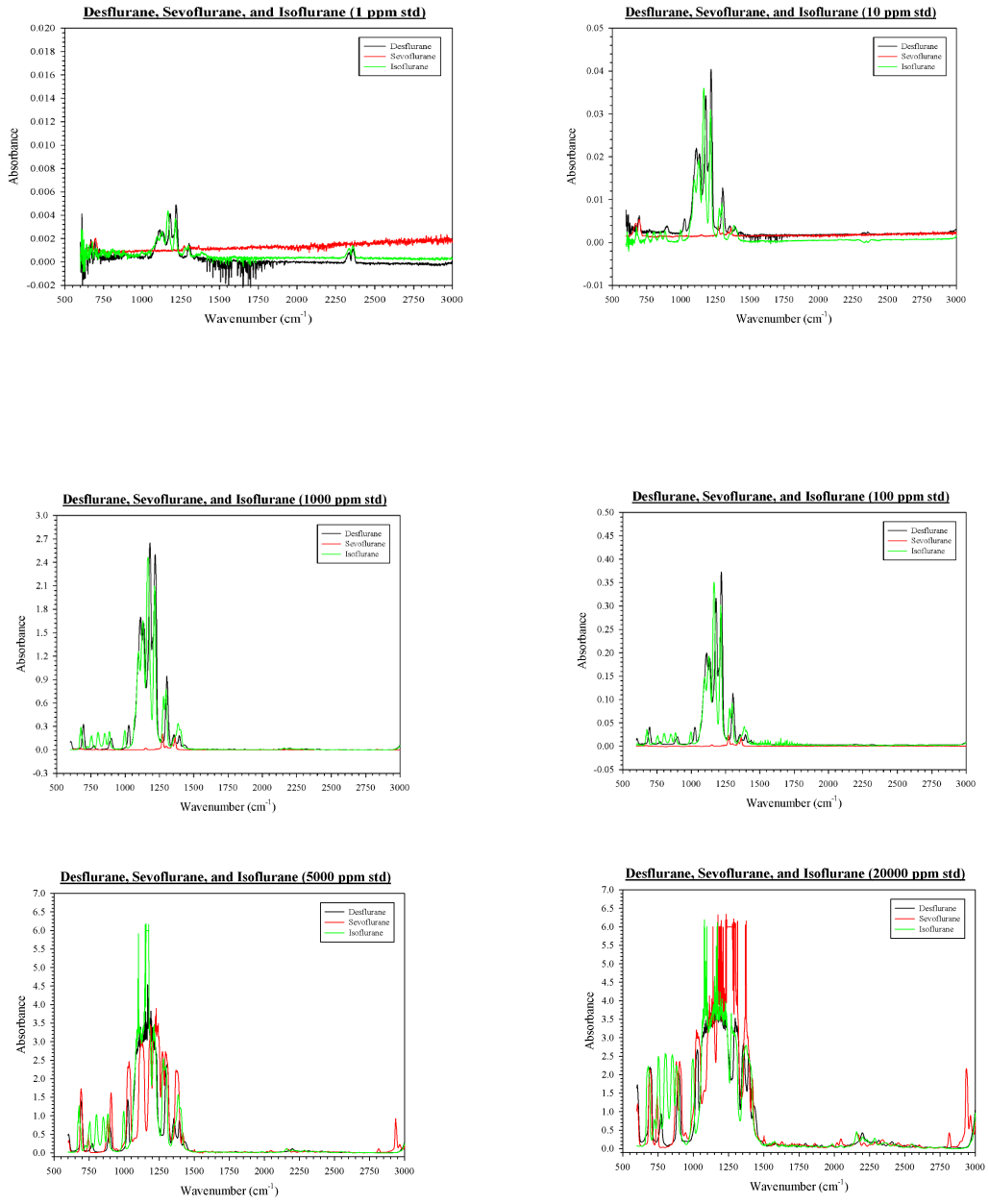

Figure 2-33: Absorbance in different wavelengths for three anesthetics in various concentrations in FTIR

There were two problems in this area that needed to be resolved before confidence in the concentration data could be achieved . First, the high and low concentrations of the anesthetic gases display different profiles (refer to Figures x and x). For example, the 1 ppm isoflurane absorbance spectrum demonstrates a very sharp peak at 1166 cm^{-1} whereas at 20000 ppm, the detector response in this area is saturated. A peak at 802.28 cm^{-1}, which was not observed to be

present for either the desflurane or the sevoflurane, was selected as the best substitution for Isoflurane at 2000 ppm. Second, the Desflurane peaks always overlapped with the Isoflurane or the Sevoflurane or both of them.

To solve the first problem, the 5000 and 20000 ppm calibration data were neglected as the typical concentration of anesthetic gases at the hospital is lower than 1000 ppm . These gases are assumed to be diluted with a high amount of air in the hospital compressor before entering the tank (this assumption was verified using GC analysis of different samples of hospital gases collected in tedlar bags at different times). The 1 ppm calibration data were removed from the instrument set up because it could not be distinguished from background noise. Consequently, the regions listed in Table (2-16) were used in the calibration method for the instrument:

Table (2-16): Selected wave number ranges and applicable concentration for each gas

Anesthetic gas	Wave number(cm-1)	Concentration(ppm)
Desflurane	1191 to 1204	10, 100, 1000
Sevoflurane	2927 to 2950	10, 100, 1000
Isoflurane	869.78 to 899.67	10, 100, 1000

The FT_IR measurement accuracy was assessed through the use of GC analysis as an alternative method. The continuous flow of anesthetic gases passed through the FT-IR and samples were collected for the GC sampling before the input stream entered the FT-IR . The results are shown in Table (2-17):

Table (2-17): Comparison of FTIR and GC analysis results for each gas

	GC results			FTIR Results		
Sample number	Isoflurane (ppm)	Sevoflurane (ppm)	Desflurane (ppm)	Isoflurane (ppm)	Sevoflurane (ppm)	Desflurane (ppm)
1	0	201	350	35	293	462
2	0	206	341	31.99	284	447
3	0	197	314	39	289	422

As can be observed in table (2-17) there was a good correlation between the GC and FT-IR results for Sevoflurane and Isoflurane. For Desflurane, the concentration is a little higher in the FT-IR, however, that was judged to be not a significant problem for the purpose of the

industrial measurements and monitoring purpuses.

2-11 Depressurizing

After approximately three weeks, the tank at the hospital did not show any significant aditional gain in weight. Therefore, it was concluded that it had reached its maximum adsorption capacity and it was exchanged with the second tank. The full tank was sealed and held at its pressure (88 Psi) during transportation to the lab. The total weight that the tank gained was 6.35 kg. In the first step for regeneration, it was depressurized with the known flow rates that were demonstrated in Table(2-18). The concentration of the desorbed anesthetic gas was measured using the GC .

From 88 psi to 44 psi the concentrations of desorbed Desflurane and Sevoflurane were approximately constant regardless of different flow rates. Therefore , after the tank reached a pressure of 44 psi the depressurization was continued using a constant flow rate of 112 l/min for around two hours. The total weight that the tank lost due to the depressurization process was 0.1679 g of anesthetics (0.0786 gr Desflurane+0.089 g Sevoflurane) which is very low in comparison with the gained weight (6.35 kg). The lost amount was estimated by multiplying the flow rate, time and measured concentration of anesthetics that exited from the tank. This experiment was repeated for this tank after a cycle of heated desorption and additional adsorption at the hospital. The second time the total lost weight due to the depressurization was 0.038 g of anesthetics, that is approximately in the same range as the first cycle. This suggests that the adsorption/desorption cycle is repeatable, and that desorption due to depressurization is a very minor amount.

Table (2-18): Anesthetic gas concentrations in the air exiting the tank as it was depressurized

time(min)	GC desflurane concentration(ngr/ml)	gc sevoflurane concentation(ng/ml)	flow rate(ml/min)	tank pressure(psi)
0				88
90	1243	1588	96.8	86
120	1214	1581	83	84
150	1151	1573	69	80.5
210	1183	1577	68	80
255	1184	1580	62	78
300	1094	1567	58	75
360	1109	1570	38	73
444	1187	1579	36	70
630	1180	1578	30	66
720	1176	1575	27	62
780	1162	1578	17	62
840	1252	1591	16.8	60
900	1261	1592	15	58
1032	1263	1593	12	57
1110	1283	1596	9	55
1200	1230	1589	8	52
1260	1245	1590	6.7	51
1530	1313	1602	2	48
1620	1262	1593	1.9	44
1630	1608	1617	112	43
1637	1501	1640	112	34
1642	1667	1671	112	28
1652	1899	1702	112	21
1658	2101	1720	112	19
1664	2245	1724	112	17
1672	2290	1750	112	15
1686	2459	1757	112	13
1692	2505	1826	112	11
1713	2408	1798	112	8
1729	2709	1732	112	0

The second time depressurizing experiment results are provided in Table (2-19):

Table (2-19): Anesthetic gas concentrations in the air exiting the tank as it was depressurized, after a second cycle of adsorption at the hospital

Time(min)	Desflurane concentration(ng/l)	Sevoflurane concentration(ng/l)	Isoflurane concentration(ng/l)
2	364	510	301
12	454	629	234
28	269	376	182
34	246	304	83
45	448	537	108
61	293	378	116
67	332	410	104
72	357	439	83
77	229	288	77
85	549	681	138
96	229	358	90
150	212	258	41
170	294	387	54
190	693	929	244
210	504	654	148
230	334	543	132
250	383	487	78
270	248	423	44
290	365	523	112
310	148	298	21
330	292	348	86
350	288	532	56
370	230	432	48

Based on the above information, the amount of anesthetic gas that was lost due to depressurizing is relatively low in comparison to the remaining anesthetic compounds. With regard to safety problems related to transportation of a pressurized tank it should be noted that if the desorption process takes place outside the hospital, the tank can be depressurized and transferred easily with losing only a very small amount of the adsorbed material.

2-12 Desorption

Primarily, air at the room temperature was passed through the depressurized tank to monitor the desorption behavior. The amount of Sevoflurane and Desflurane desorbed after 9 days is shown in Table 2-20:

Table (2-20): Desorbed anesthetics with passing air at room temperature through the tank

Day	desorption hour per day	Average Desflurane concentration (ng/ml)	Average Sevoflurane concentration (ng/ml)	flow rate (lit/min)	total des desorbed(g)	total sevo desorbed(g)
1	5	2953	1690	10	8.859	5.07
2	7.5	2831	1693	10	12.7395	7.6185
3	7	2676	1696	10	11.2392	7.1232
4	24	1712	1617	10	24.6528	23.2848
5	24	1316	1589	10	18.9504	22.8816
6	24	1035	1555	10	14.904	22.392
7	24	953	1512	10	13.7232	21.7728
8	24	836	1487	10	12.0384	21.4128
9	24	824	1452	10	11.8656	20.9088

The total desorbed anesthetic gases after 163 hour was 281.4 g of which 129 g was Desflurane and the remaining amount was Sevoflurane. Since the tank gained approximately 5.9 kg during the adsorption cycle, this suggests that a large amount of anesthetic gases still remained adsorbed to the carbon. This also advises that, if the desorption process was continued in the same way, it would take a long time to desorb all or most of the anesthetic. Thus, heating as an auxiliary factor was added to the desorption process.

Adding heat to the desorption:

There are two ways to add heat to the desorption process. First, the flow gas (air) can be heated and then passed through the tank but it was difficult to produce hot air to heat the carbon with the

available equipment. Therefore, it was more effective to heat the tank directly, especially for the small diameter tank (24 cm diameter and 69 cm height) used in this work.

Figure 2-34: Absorber tank with electrical heating tape wrapped around for heating purpose

To heat the tank, it was wrapped with an electric heating tape (the heating rate:620 Watts)and heated until around 111°C, as measured by a probe inserted in the middle of the tank. Since only a short probe was available in the lab, the temperature was measured only in the outer layer of the tank. This is obviously different from the middle of the tank, which had a 26 cm diameter. In addition, it takes time for the heat to be conducted to the centre of tank. Thus time and position should be noted in the heat transfer process. The flow rate in this process was a constant amount of 972 ml/min.

Table (2-21): Time, temperature profile and anesthetic gas concentration in the air existing the tank during thermal desorption

Time (min)	temperature©	Desflurane Concentration(ng/ml)	Sevoflurane concentration(ng/ml)	Isoflurane concentration(ng/ml)
3	22	747	1535	17
8	22.2	820	1538	32
15	22.4	803	1542	21
21	29	884	1549	39
27	47	812	1544	23
34	66	833	1540	38
41	80	1401	1627	67
105	84	1504	1643	70
112	71	1760	1681	65
130	89	2012	1724	8
140	95	2284	1754	103
150	99	3500	1805	137
166	100	4432	1941	170
171	102	8354	2050	227
180	105	10900	2151	551
187	105	10474	3438	109
208	109	20667	5227	112
216	112	159019	32117	67
256	112	217570	39864	98
266	112	283910	67589	106
300	112	262234	54923	107

It was observed that it takes around 3.5 hours for the heat to penetrate in to the tank and during this time the desorption amount increased significantly. The concentration of Desflurane increased more rapidly than Sevoflurane as its boiling point($23.5°C$) is less than half of Sevoflurane ($58.6°C$). In addition, using heat in the desorption process resulted in 351 and 44 times faster desorption for Desflurane and Sevoflurane, respectively.

2-13 Condensation

For recovery of anesthetic gases , a two step condensation process was designed to recover as much desorbed gases as possible. The melting point of Sevoflurane and Desflurane were reported to be -67 ° C and -101° C by Berry .[27]

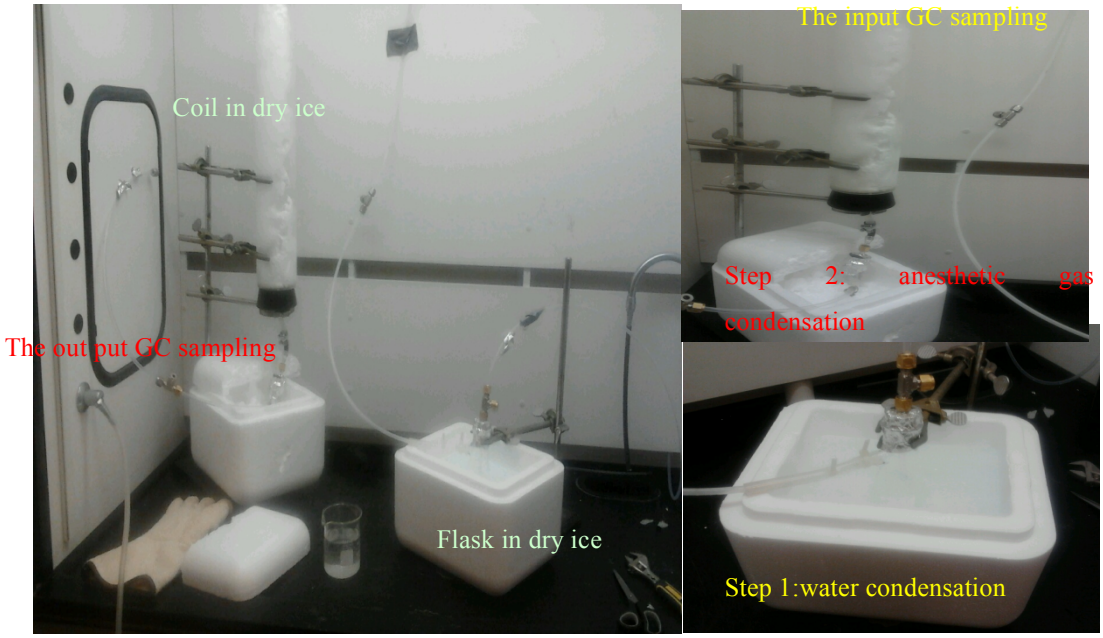

Figure 2-35: The condensation system with two steps process for recovery of the anesthetics

In the first step , water was condensed in a flask on dry ice. In the next step, a glass coil immersed in dry ice (with surface temperature of -78 °C) was selected to capture all anesthetic gases from the tank. The flow rate of the carrier gas was set at 1 L/min since it was observed that a higher flow rate resulted in water escape from the first condensation flask and blockages due to ice formation in the coil. It was observed that a very good condensation of the gases occurred from the dry ice coil system with surface temperature of -78°C. In the flask positioned before the coil , a significant amount of water and a little insoluable (oily) liquid was detected .

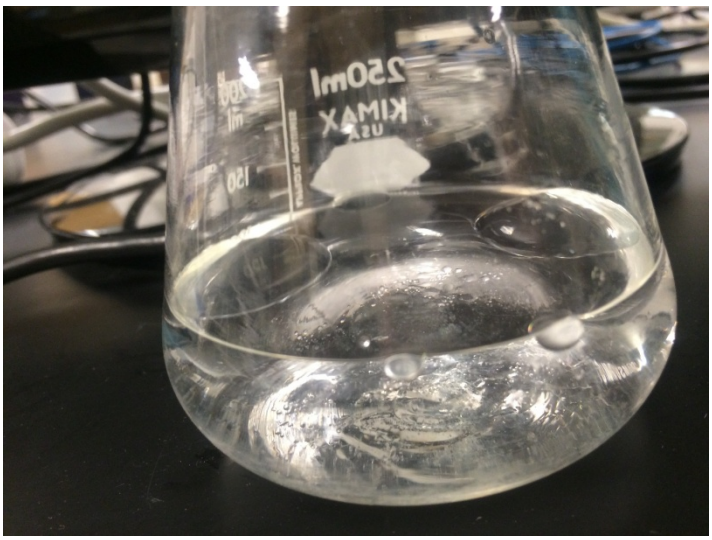

Figure 2-36: The captured water from the first condenser (ice) with oily phase visible on top

After the coil, the captured liquid displayed two peaks with retention times that were approximately in the same region as Sevoflurane and Desflurane. Therefore, this confirmed that the Sevoflurane and Desflurane were recovered through the dry ice condensation process. In a review of historical literature, condensation with liquid oxygen at -150°C was suggested as a capture method. Another interesting result was that analyzing gas samples collected before and after the coil with the GC illustrated an extra peak in the output gas from the coil condensation in the range near the Desflurane retention time. Thus , this extra peak and the oily captured liquid, with a density less than water (all anesthetics have density of more than water) suggested that degradation of these gases was occurring.

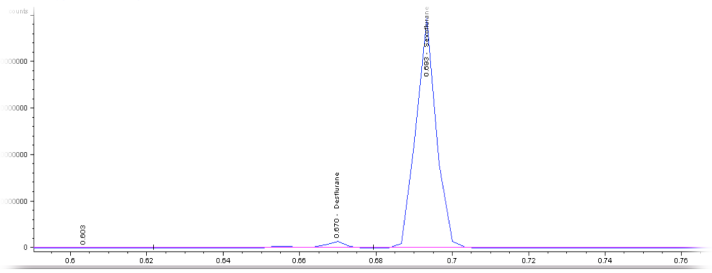

Figure 2-37: The gas chromatography of the desorbed gas from the tank before entering the condensation part

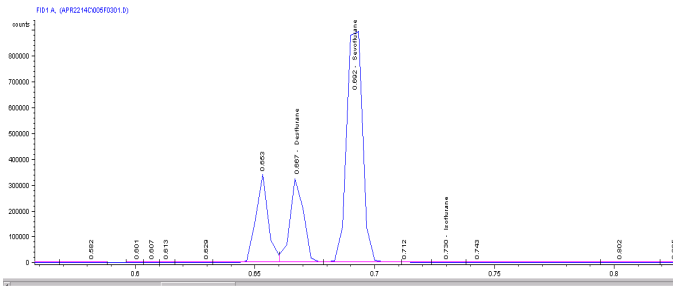

Figure 2-38: The gas chromatography of the desorbed gas from the tank after leaving condensation part

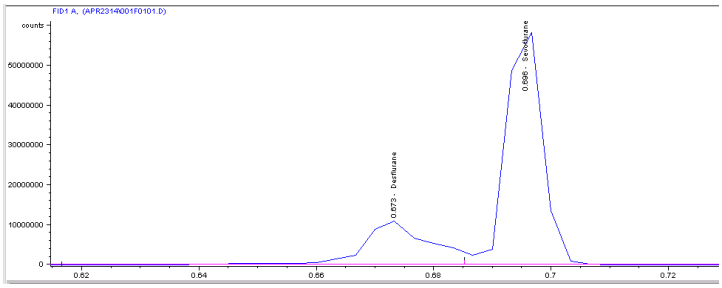

Figure 2-39: The gas chromatography of the captured liquid that obtained from the coil of dry ice(second step of condensation)

The capture efficiency in the two runs was calculated through measuring the composition of gases at two points (labeled in Figure (2-35) with yellow and red color): before and after the dry ice coil condensation. In the first run in which the whole of the coil was covered with dry ice, Sevoflurane and Desflurane, the capture efficiency was 98% and 95%, respectively. In the second run, in which more than half of the coil was covered with dry ice , the Sevoflurane and desflurane capture efficiency was decreased to 96% and 68%, respectively. Therefore, the condensation process efficiency strongly depended on the coil length immersed in the dry ice.

Table (2-22): Condensed anesthetic gases amounts (calculated and measured values)

Captured compound	Density(g/ml)	Average desorbed gas concentration(ng/ml)	Flow rate(ml/min)	Time (min)	Captured anesthetic gas weight(g)	Calculated products
Sevoflurane	1.522	104926	1000	50	14.35	5.2463
Desflurane	1.465	44811	1000	50	1.96	2.150928
Water+ oily liquid	1+less than 1	----	1000	50	27.777	----

The results obtained(Table (2-22)) found that the captured liquid was more than what was expected. This means that one of assumptions is not correct. The flow rate is the only factor that could cause this problem as the GC data was measured accurately. Measuring flow rate in different places of the condensation process showed that the flow rate measured after the dry ice coil dropped from 1000 to 333.3 L/min. Therefore a new calculation for measuring captured liquied is provided below in the Table (2-23). It can be observed in Table(2-23), that masses of 4.78 g and 0.65 g of Isoflurane and Sevoflure, respectively, were obtained from the coil of dry ice. The calculated masses of Isoflurane and Sevoflurane determined by multiplying the flow rate and time with the concentrations collected by GC measurement were 5.25 g and 2.15 g .

Table (2-23): Condensed anesthetic gases amounts (calculated and measured values) with the correction of flow rate

	Density(g/ml)	Average desorbed gas concentration(ng/ml)	Flow rate(ml/min)	Time (min)	Captured anesthetic gas weight(g)	Calculated products(g)
Sevoflurane	1.522	104926	333	50	4.78	5.25
Desflurane	1.465	44811	333	50	0.65	2.15
Water+ oily liquid	1+less than 1	----	1000	50	27.777	----

2-14 White By-Product Sample

After several days of heat addition to the desorption process, a white solid powder was observed exiting the tank, which blocked the outlet tube (Figure (2-40)). Interestingly, this powder only accumulated outside the tank in the fitting connection and tubing when heat was added to the process as well. There was no sign of it inside the tank of activated carbon. In addition, the inside of the collection flask that captured the water from tank was appeared to be pitted, which could be a sign of HF (hydrofluoric acid) production . To analyze this sample, a series of experiments was done to discover the identity of this decomposed product:

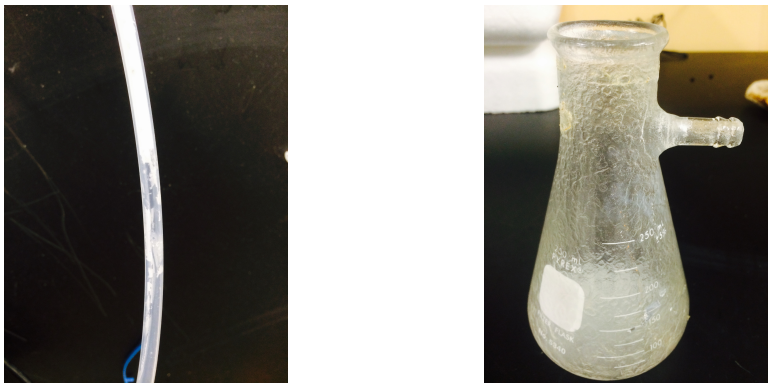

Figure 2-40: Photograph of white solid material collected in the outlet tube (left) and condenser flask (right) after several days of thermal desorption

Analysis of White By-Product Material:

Thermogravimetric analysis was used on a sample of the white powder in an attempt to determine its nature and composition. Upon thermogravimetric analysis, the white powder lost mass from room temperature until 80C. Therefore, this product is very volatile or unstable and can easily transfer to the gas phase. In addition, at the end of the TGA test, nothing remained in the sample tray, which suggests the absence of any metal elements or presence of a strong heat resistance bond in the solid sample.

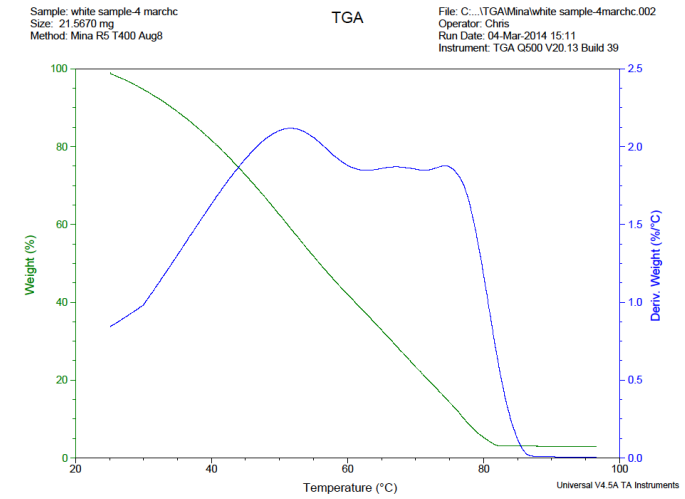

Figure 2-41: Thermogravimetric analysis (TGA) of the White By-Product powder

Next, solubility tests were performed to provide further information on the chemical nature of the White By-Product. Different polar and non polar solvents were selected to find an appropriate solvent to dissolve the white solid so that other various analytical methods could be tested.

Table (2-24): Solubility of the white by-product in different solvents

Solvent name	Solubility of white by-product
ether	___
Methanol	A little
water	A little
ethanol	___
Dimethylformamide (DMF)	___
aceton	___
Dicloromethane	___
Hexane	___
Benzene	___
Toluene	___
Ethyle accetate	___

Sevoflurane is miscible with ethanol, ether, and benzene but the white sample did not display any solubility in the same solvents. Compound A, which is one reported degradation product of Sevoflurane, is soluble in ethyl acetate [28] but the white sample did not show the same behavior, suggesting it is not compound A. Therefore, analyzing this solid with Mass spectroscopy on a liquid sample would not be feasible. The headspace method was suggested as a solution but the accuracy of this method is questionable.

All GC_MS experiment were conducted with a Varian Model CP 3800 GC and Saturn model 2000 MS detector. As no solvent could be found for the white powder, the head space method, where a gas sample was collected from the top of the solid, was selected to find more information about this solid. The sample was pre heated in the oven before the analysis and then a gas sample was collected from the top of the vial which contained the white sample . Also, the same samples were collected from the pure Sevoflurane and Desflurane to compare results .There were no matches found in the library of the GC_MS device (Nist 2000) for the white sample mass spectroscopy.

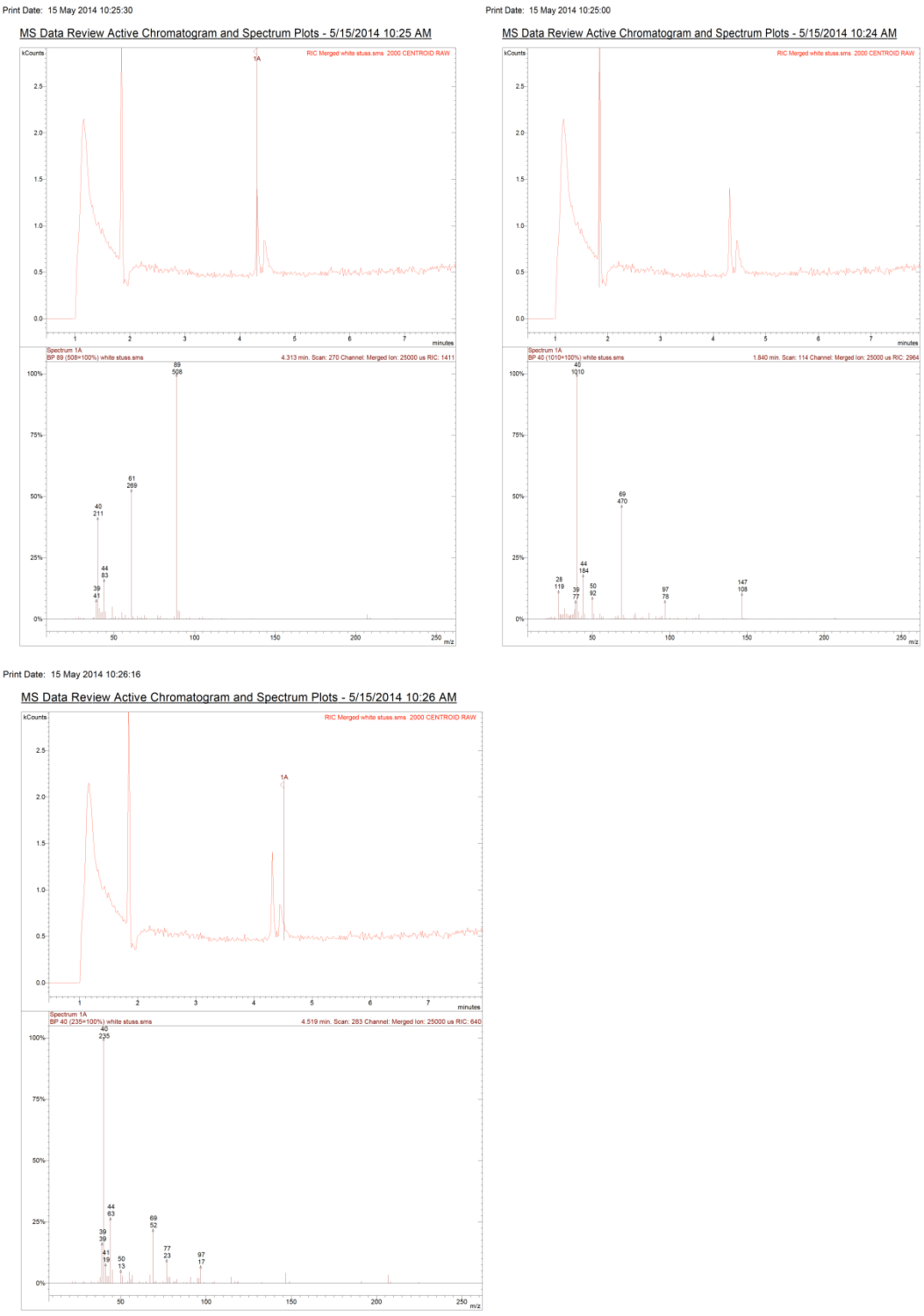

Figure 2-42: The white by-products three detectable main peaks mass spectrum

For Comparsion, mass spectra of two of the pure anesthetic compounds were determined and are shown in Figure (2-43) and (2-44).

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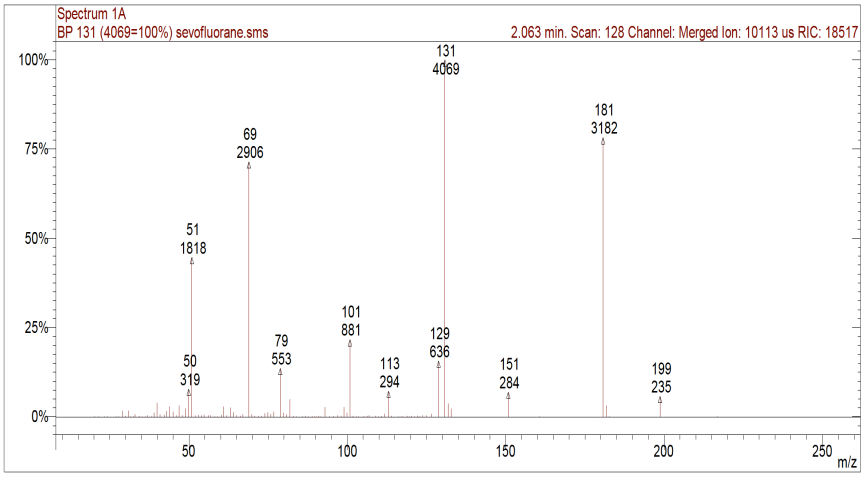

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Figure 2-43: The pure Sevoflurane mass spectrum

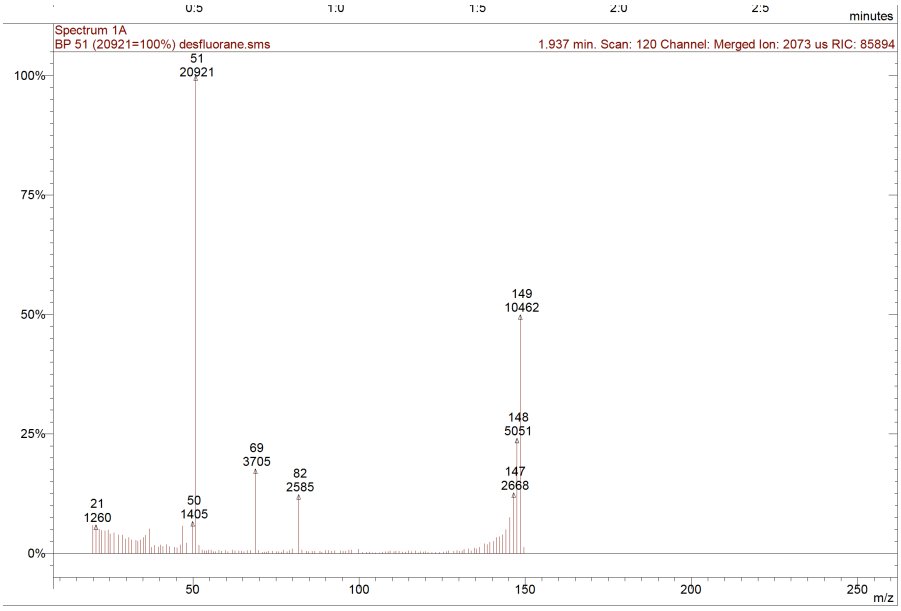

Figure 2-44: The pure Desflurane mass spectrum

It is clear that the white by-product has different total mass and fragments from Sevoflurane and Desflurane. In addition, It is possible that there are some compounds in the white sample which do not evaporate in the head space. Therefore, they are not detected by GC_MS.

FTIR analysis was performed on the white by-product to provide further information about the bands and groups present in the material. The spectrum below is the FTIR absorbance of the white sample at different wave lengths. The peaks from 1000-1300 cm^{-1} were related to C-F band.The peaks around 3000 cm^{-1} represent C-H stretch bands but the shape of them is a little unusul suggesting a possible different kind of band for carbon.[29]

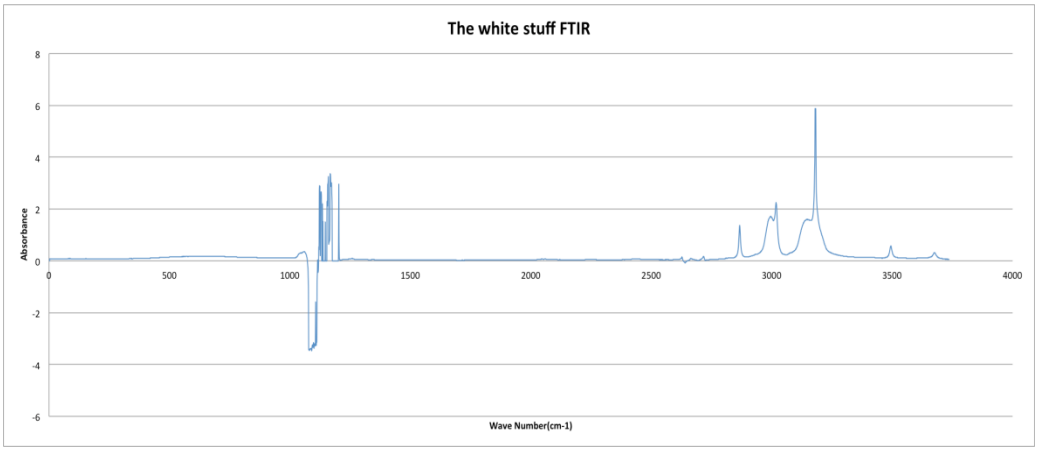

Figure 2-45: The white by-product FTIR spectrum

Furthermore, Nuclear Magnetic Resonace (NMR) analysis was performed to provide aditional information. Deuterated water was selected as a solvent for HNMR of white sample. Three different tests were performed to find Carbon, hydrogen (H) and Fluorine (F). In F19 decoupled experiments, eight numbers of F were detected in the white sample with 2 pairs of them situated very close to each other. In other words, both of them would be bonded to the same carbon or maybe another element. There were 4 signals for hydrogen discovered with two of them very near to each other. A very fast C-13 experiment exhibited only one Carbon. A more expensive experiment would be required detect the final number of Carbons, but this was not undertaken for this work.

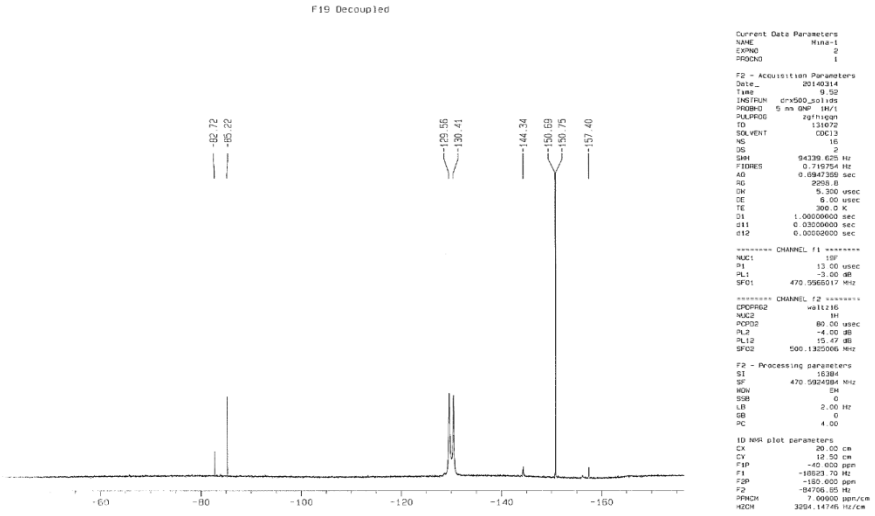

Figure 2-46: Fluorine-19 nuclear magnetic resonance spectrum of by-product

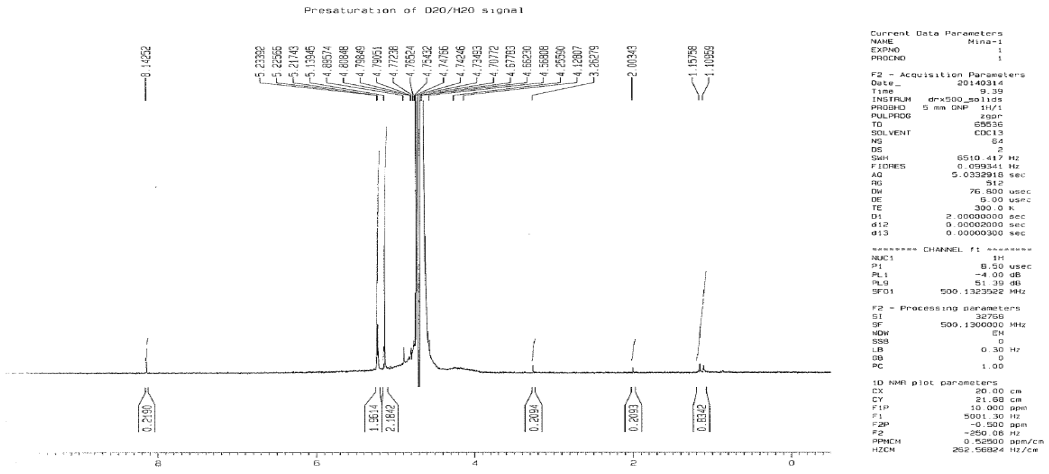

Figure 2-47: Hydrogen nuclear magnetic resonance spectrum of by-product

Table (2-25): Summery of white by-product sample analytical tests:

The test name	results
TGA	It is a volatile compound with no residue remaining after 80 C
Finding Solvent	Does not have same solvent as Sevoflurane or compound A . Not soluble in regular polar or non polar solvents except a little in water and methanol
GC-MS	Different segments and molecular weight from Sevoflurane and Desflurane. No match in library nist 2000
NMR	It definitely has F ,H and C elements. Possible number of eight F and four H
Lewis acid reaction	Degradation happened but no sign of white powders
Changing copper connection	The white sample amounts decreased but not disappeared
FTIR	Possible C-F and C-H band

At this point, it was concluded that analytical analysis of this compound is difficult requiring more advanced instruments and time. A reference [5] was found that described the creation of this white powder in the Sevoflurane bottles in 1996 as a problem in Abbott company products. The Lewis acid degradation of Sevoflurane was explained as the reason for that event but no details were provided about the degradation reactions or products. In this paper, the production of HF and pungent odor were the same as what were observed in our experiments. Three quick experiments were performed to find the white sample in the degradation of Sevoflurane by $FeCl_3$, $FeCl_3^+$ water and KOH. Some signs of degradation were observed such as an immiscible liquid by-products but the white sample was not generated.

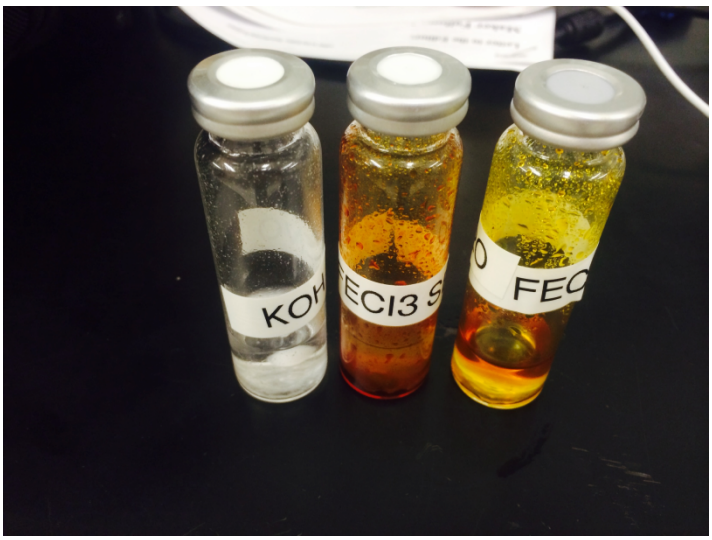

Figure 2-48: From left to right Sevoflurane with KOH, $FeCl_3$ and $FeCl_3^+$water

As the emission system at the hospital is not an isolated process, it is possible that different sources of chemicals (for example from disinfection materials that are used in surgery rooms) can cause this degradation product. Finally, the copper fitting connections on the tank were changed to a stainless steel fitting, with the apparent result that a decreased quantity of white sample was produced. The heat in the desorption process should be controlled precisely to prevent the production of huge amount of this material. It was decided to continue research into the origins of this solid in a future project.

2-15 Estimation of anesthetic gases adsorbed by the tank

Three differrent ways were used to calculate the quantity of anesthetics in the tank of adsorbent before it reached to the saturation point at the hospital :

Calculating the input stream anesthetic's concentration through the FTIR

Estimating adsorbed amounts by daily measurment of tank's weight

Summation of desorbed anesthetic's values

Unfortunately, Because of some problems in the FTIR, the data were recorded just for some days during the 5 weeks. Regarding the available measurements, as amounts of the consumed anesthetics did not demonstrate any significant change, average adsorbed anesthetics were assumed to be constant during a day. The results are provided in the below table:

Table (2-26): The input stream anesthetic's qualifications measured by the FTIR

The name of compounds	Average daily anesthetic concentration (ppm)	Average daily adsorbed anesthesia (g)
Sevoflurane	589	89
Desflurane	372	24
Isoflurane	2	negligible
Total anesthetics weight	808.63	115

The weight of tank was measured every day through an online instrument at the hospital. The data are demonstrated in Table (2-27):

Table (2-27): Recorded weights of the tank at the hospital

Date	Average weight per day (lb.)	Gained weight per day (lb.)	Gained weight per day (g)
07-08-2014	0.07	3.44	1560.83
07-09-2014	3.51	0.81	365.87
07-10-2014	4.31	0.74	333.70
07-11-2014	5.05	0.85	384.64
07-12-2014	5.90	0.01	2.61
07-13-2014	5.90	-0.01	-4.17
07-14-2014	5.89	0.38	172.26
07-15-2014	6.27	0.46	206.70
07-16-2014	6.73	0.20	89.09
07-17-2014	6.93	0.40	180.63
07-18-2014	7.32	-0.42	-191.69
07-19-2014	6.90	-0.01	-4.29
07-20-2014	6.89	0.01	3.41
07-21-2014	6.90	0.40	180.41
07-22-2014	7.30	3.05	1385.30
07-23-2014	10.35	0.23	104.07
07-24-2014	10.58	0.48	217.08
07-25-2014	11.06	0.27	123.29
07-26-2014	11.33	0.01	2.72
07-27-2014	11.34	0.00	-1.76
07-28-2014	11.33	0.06	25.98
07-29-2014	11.39	0.15	68.55
07-30-2014	11.54	0.37	168.65
07-31-2014	11.91	0.43	195.76
08-01-2014	12.34	0.36	162.46
08-02-2014	12.70	-0.05	-22.50
08-07-2014	12.65	0.00	-1.51
08-08-2014	12.65	0.32	146.36
08-09-2014	12.97	0.00	-1.22
08-11-2014	12.97	-4.05	-1836.20

The highlighted data in the table (2-27) are related to the weekends that the HDR system was automatically turned off. The total weight that was gained by the tank was 12.97 lb. (5.88 kg), at

which point it reached the saturation point and did not gain any additional weight. Except for the 8th and 22th August that show high values, the recorded weights demonstrate a decreasing adsorption rate especially after 3 weeks. This can be explained by the fact that after 3 weeks, the adsorption rate was decreased as some part of the adsorbent's active sites were increasingly occupied by gas molecules. The FTIR measurements on average predicted 115 gr of anesthetics in the input stearm of the tank each day, that is approximately in the same range with the weight numbers. The strange numbers on 8th and 22th August may relate to the water adsorption that was expected to be captured with a dryer in the HDR system.

The desorption process consisted of two steps: depressurization and heated desorption. The depressurization and the heated desorption were explained in (2-10) and (2-11). Desorbed anesthetics in these two steps are organized in the Table (2-28) :

Table (2-28): Desorbed anesthetic values from the tank

The name of compunds	The desorbed due to depressurizing(g)	The desorbed due to heating desorption(g)
Sevoflurane	0.020	555.4
Desflurane	0.014	1000.8
Total	0.214	1556.2

Therefore the activated carbon A48 adsorbed the anesthetics to approximately 25% of its intial weight at the hospital until it reached the saturation point. This result is similar to the q number(26%) that was obtained in the batch laboratory experiments.

3.Conclusions

In this project, an adsorption process was successfully designed and tested to capture three common anesthetic gases (Isoflurane, Sevoflurane and Desflurane) that vent to the environment through a hospital emission system. Gas chromatography with FID detection and an FTIR instrument were calibrated for qualification of these gases in batch and continuous experiments. A specific kind of activated carbon (GAC A48) with a high capacity of adsorption was found to be the best adsorbent in comparison to various relevant adsorbents such as Zeolite, Silica Gel , Molecular Sieve 13X etc. This adsorbent demonstrated the same reversible adsorption characteristics for Isoflurane and Sevoflurane in the temperature range of 35-50$^{\circ}C$.The isotherm graphs of these two gases,as well as halothane, were determinded for the best adsorbent for room temperature. The Freundlich isotherm was fit to them. The Freundlich equation constants were discovered through finding the slope and the intercept of the plot of log q over c_{∞} . A column of adsorbent was used in a laboratory before setting the industrial set up to examine all important factors for the continuous adsorption process. The successful laboratory results were reproduced in an industrial system using an appropriate tank of adsorbent. This tank, containing 14.514 kg of activated carbon, gained 6.35 kg weight before the saturation point for the first installation at a hospital emission system.

The desorption process comprised two steps, depressurization and heated desorption. Less than 1 % of anesthetic gases was lost from the tank in the depressurization step.The depressurization conditions did not have a significant effect on the desorbed amount. The heated adsorption was tested through warming of the column of adsorbent while air was passed through it. For a temperature of around $100^{\circ}C$, the desorption concentration reached a significant amount that was effectively captured in the condensation process. To prevent degradation by-products, higher temperatures are not recommended.

The condensation system which consisted of a flask and a glass coil packed in dry ice effectively captured water and desorbed gases in two steps with an efficiency of more than 95% using a flow rate of approximately 1 L/min.

The primary identification of captured liquid after the coil of dry ice by gas chromatography confirmed the presense of Sevoflurane and Desflurane. Significant amounts of water were

observed in the condensation flask that suggests an inefficiency in the water removal system in the hospital capture system.

Degradation by-products in the form of a white powder and oily liquid, observed during the heated desorption, have completely different physical and chemical characteristics than the anesthetic gases. Reproduction was difficult and accurate identification of these materials was unsucessful using the available laboratory analytical methods.

The resuts of these experiments demonstrated that a cost effective waste anesthetic gas adsorption system using activated carbon with high capacity of adsorption could be introduced into a hospital emission system. The recovery process using a very simple dry ice cryogenic condenser with high efficiency completes this reusable system.

3-1 Recommendations

In order to meet expected regulations in the control of waste anesthetic gas emissions that will be approved in the near future in North America, it is very important to quickly develop this system in an industrial applicable form for installation at Hospitals. For this type of sytem, the following is required:

1- Designing an online heating system to run the desorption process at a hospital
2- Planning an appropriate heat exchanger in the desorption part to selectively condense desorbed gases in an economical way at a hospital. One way to improve the condensation process is to place two heat exchangers with different effective temperatures, one before the tank to better capture all water and the second one after the tank to recover anesthetics in the desorption stage.
3- Using a bigger adsorbent tank to make the replacement time (3weeks) longer depending on system limitations
4- Placing some simple IR sensors (such as those used for CFC detection) instead of an expensive FTIR instrument in different places to make the recovery system smart in the detection of anesthetic gases; for example: an alarm would identify when anesthetic gases are detected in the output tank stream or shut off the input stream when no gas exists
5- Improving the system construction to prevent creating degradation products; for example, replacing any Lewis acid sources such as copper connections
6- Developing the system to capture N_2O in addition to other anesthetic gases

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