DETERMINATION OF FLUID DYNAMIC BEARING (FDB) OIL AND PIVOT GREASE IN HARD DISK DRIVE COMPONENTS VIA GAS CHROMATOGRAPHY-MASS SPECTROMETRY

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RESEARCH REPORT SUBMITTED IN FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF SCIENCE (ANALYTICAL CHEMISTRY & INSTRUMENTAL ANALYSIS)

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ABSTRACT

Fluid dynamic bearing (FDB) oil and pivot grease are used as lubricant in the spindle motor and in the actuator pivot bearing cartridge in the hard disk drive (HDD). The presence of high amount of both FDB oil and pivot grease in hard disk drive may lead to the drive failure. Determination of FDB oil and pivot grease presence in the HDD after 300 hours, 600 hours and 900 hours of testing in reliability chamber was done by extracting the recirculation filter and adsorbent breather filter in the HDD using hexane as the solvent. The presence of FDB oil and pivot grease in the extracted solvent from the recirculation filter and adsorbent breather filter was determined via gas chromatography (GC) and identified by mass spectrometry (MS).

The amount for both FDB oil and pivot grease were found to increase with the HDD test time. At the point of saturation, the internal HDD environment contains 116 ng/sample of FDB oil and 1120 ng/sample of pivot grease based on the recirculation filter extraction. However, this amount of vapors were able to be removed by the adsorbent breather filter as it has yet to reach the maximum amount that it can absorb.

ABSTRAK

Minyak *fluid dynamic bearing* (FDB) dan gris pangsi digunakan adalah pelincir bagi pemutar (*spindle motor*) dan penggerak pangsi kartrij (*actuator pivot bearing cartridge*) di dalam pemacu cakera keras (HDD). Kehadiran minyak FDB dan gris pangsi dengan kuantiti yang tinggi di dalam pemacu cakera keras boleh menyebabkan kegagalan pemacu. Dalam eksperimen ini, minyak FDB dan gris pangsi dalam HDD selepas 300, 600 dan 900 jam operasi telah ditentukurkan dengan teknik pengekstrakan yang menggunakan heksana sebagai pelarut pada penapis edaran (*recirculation filter*) dan penapis penyerap pernafasan (*adsorbent breather filter*) di dalam HDD. Kehadiran minyak FDB dan gris pangsi dalam pelarut diekstrak daripada penapis edaran dan penapis penyerap pernafasan ditentukan melalui kromatografi gas spektrometri jisim (GCMS).

Jumlah kedua-dua minyak FDB dan gris pangsi didapati meningkat dengan masa operasi HDD. Udara persekitaran dalaman HDD pada ketika tepu mengandungi minyak FDB sebanyak 116 ng/sampel dan gris pangsi sebanyak 1120 ng/sampel. Keputusan eksperiment juga menunjukkan bahawa jumlah wap yang dapat dikeluarkan oleh penapis penyerap pernafasan masih belum mencapai jumlah maksimum yang ia boleh diserap.

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TABLE OF CONTENTS

		Page
TITLE PAGE		**i**
ORIGINAL LITERARY WORK DECLARATION FORM		**ii**
ABSTRACT		**iii**
ABSTRAK		**iv**
ACKNOWLEDGEMENTS		**v**
TABLE OF CONTENTS		**vi**
LIST OF FIGURES		**viii**
LIST OF TABLES		**ix**
LIST OF SYMBOLS AND ABBREVIATIONS		**x**
LIST OF APPENDICES		**xi**
CHAPTER 1 INTRODUCTION		**1**
1.1	Introduction to Hard Disk Drives (HDD)	1
1.2	Hard Disk Drive Components	2
1.3	Magnetic Recording Process	4
1.4	Emerging Trends in Hard Disk Drive Industry	6
1.5	Spindle Motor	7
1.6	Actuator Pivot Bearing Cartridge	10
1.7	Contamination in Hard Disk Drive	12
1.8	Filters	13
1.9	Objective	15
CHAPTER 2 EXPERIMENTAL		**16**
2.1	Chemicals	16
2.2	Standard Solutions	16
	2.2.1 FDB Oil	16

2.2.2 Pivot Grease 16

2.2.3 Anthracene-D10 Spike Standard Solution 17

2.3 Samples 17

2.4 Sample Preparations 18

2.4.1 Recirculation Filter 18

2.4.2 Adsorbent Breather Filter 19

2.5 Quantification of FDB Oil and Pivot Grease 20

2.6 Recovery Study 21

2.6.1 Recirculation Filter 21

2.6.2 Adsorbent Breather Filter 21

2.7 GC-MS Settings 21

CHAPTER 3 RESULTS AND DISCUSSION 23

3.1 FDB Oil 23

3.2 Pivot Grease 28

3.3 Recovery Study 33

CHAPTER 4 CONCLUSION 35

4.1 Conclusion 35

REFERENCES 37

APPENDICES 38

LIST OF FIGURES

Figure		Page
1.1	3.5 inch HDD (bottom) and 2.5 inch HDD (top)	2
1.2	Basic HDD components	3
1.3	Schematic diagram of head-disk interface	5
1.4	Magnetic recording process (taken from HGST, 2007)	5
1.5	Ball bearing spindle motor and FDB spindle motor (taken from HGST, 2012)	7
1.6	Actuator pivot bearing cartridge (taken from Hanke, 2013)	11
3.1	(a) Gas chromatogram for FDB oil and (b) mass spectrum for peak retention time 18.347 min (Decanoic acid-hexadecyl ester)	24
3.2	Plot of the measured total concentration of FDB oil detected in recirculation filter (n = 3)	25
3.3	Plot of the measured total concentration of FDB oil detected in adsorbent breather filter (n = 3)	27
3.4	(a) Gas chromatogram for pivot grease and (b) gas chromatogram for pivot grease signature hydrocarbon hump	29
3.5	Plot of the measured total concentration of pivot grease detected in recirculation filter (n = 3)	30
3.6	Plot of the measured total concentration of pivot grease detected in adsorbent breather filter (n = 3)	32
3.7	Gas chromatogram of 2 ppm anthracene-d10 standard	33

LIST OF TABLES

Table		Page
2.1	HDD combination and sampling run time	18
2.2	Samples for FDB oil and pivot grease analysis	20
2.3	HP 6890-5973 GC-MS instrument set-up	22
3.1	Total concentration of FDB oil detected in recirculation filter	25
3.2	Total concentration of FDB oil detected in adsorbent breather filter	26
3.3	Total concentration of pivot grease detected in recirculation filter	30
3.4	Total concentration of pivot grease detected in adsorbent breather filter	31
3.5	Recovery of anthracene-d10 standard	33

LIST OF SYMBOLS AND ABBREVIATIONS

amu	Atomic mass unit
ESD	Electrostatic charge
eV	Electron volt
FDB	Fluid dynamic bearing
GC-MS	Gas chromatography-mass spectrometry
HDD	Hard disk drive
HP	Hewlett Packard
IBM	International Business Machines Corporation
MBA	Motor base assembly
m/z	mass-to-charge ratio
n	Number
ND	Not detected
NIST	National Institute of Standards and Technology
NRRO	Non repeatable run out
ppm	Part per million
PTFE	Polytetrafluoroethylene
rpm	Revolutions per minute
U.K.	United Kingdom
U.S.A.	United States of America
USB	Universal Serial Bus
VCM	Voice coil motor

LIST OF APPENDICES

Appendix		Page
1	Total concentration of FDB oil detected in each recirculation filter	38
2	Total concentration of FDB oil detected in each adsorbent breather filter	39
3	Total concentration of pivot grease detected in each recirculation filter	40
4	Total concentration of pivot grease detected in each adsorbent breather filter	41
5	Individual anthracene-d10 standard recovery	42
6	Gas chromatogram of 200 ppm anthracene-d10 standard	43
7	Gas chromatogram for recirculation filter	44
8	Gas chromatogram for adsorbent breather filter	45
9	Gas chromatogram of recirculation filter extraction for test time 900 hours for motor from Supplier A and pivot from Supplier X	46
10	Gas chromatogram of adsorbent breather filter extraction for test time 900 hours for motor from Supplier A and pivot from Supplier X	47
11	Gas chromatogram of recirculation filter extraction for test time 900 hours for motor from Supplier A and pivot from Supplier Y	48
12	Gas chromatogram of adsorbent breather filter extraction for test time 900 hours for motor from Supplier A and pivot from Supplier Y	49
13	Gas chromatogram of recirculation filter extraction for test time 900 hours for motor from Supplier A and pivot from Supplier Z	50
14	Gas chromatogram of adsorbent breather filter extraction for test time 900 hours for motor from Supplier A and pivot from Supplier Z	51

CHAPTER 1

INTRODUCTION

1.1 Introduction to Hard Disk Drives (HDD)

Hard disk drives are widely used in computers as the main data storage device for storing and retrieving digital information. Hard disk drive is also known as disk drive or hard disk and abbreviated as HDD. The hard disk drive was first introduced in 1956 by IBM which used fifty pieces of vertically stacked disks measuring 24 inches in diameter, to store merely five megabytes of data (Jacob *et al.*, 2008). Since its introduction, HDDs have been evolved in terms of increment in its storage capacity, reduction in its physical size, lowering the cost of manufacturing while improving its performance and reliability. The evolution of HDDs is due to the advancement in technology and consumer demands over the decades.

HDDs utilized the magnetic recording technology to store or retrieve data by using read and write heads to detect and induce a magnetic field from or onto the disks. Both the heads and disks are sealed in an enclosure for contamination prevention purpose. The HDDs can be classified based on the dimensions of its external enclosure which refers to the width of the sealed disk drive unit. The HDDs size that commonly available are either 3.5 inch or 2.5 inch which are usually used in desktop computers and notebooks respectively (Jacob *et al.*, 2008). Figure 1.1 illustrates a 3.5 inch and a 2.5 inch HDDs.

Even though majority of HDDs are used as internal hard drives, many consumers also used external hard drives. Internal hard drives are placed inside the computer system, while external hard drives are usually portable and stored in an enclosure that protects the drive at the same time allowing it to interface with computer using a USB port. External hard drives are often used to backup data on the computer and expand the total storage capacity available. Today, usage of HDDs is no longer limited in computer industry. HDDs can be found in televisions, media players and other entertainment systems.

Figure 1.1 3.5 inch HDD (bottom) and 2.5 inch HDD (top)

1.2 Hard Disk Drive Components

A HDD is made up of several mechanical and electronic components internally which are controlled by electronic boards externally. The most important components of the HDD are the magnetic heads and media or disks where they are involved in the magnetic recording process. The HDD enclosure is made up of a base casting and a top cover which houses all the components inside a HDD. Figure 1.2 shows the components inside a HDD.

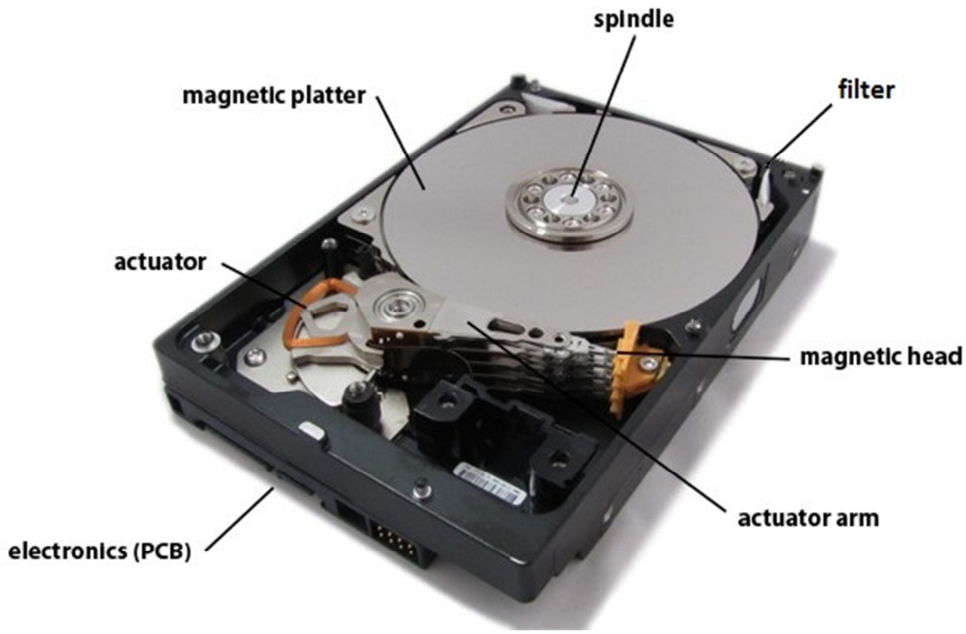

Figure 1.2 Basic HDD components

The HDD may contain either a disk or a stack of disks which is stack on a spindle motor in the radial direction relative to housing. The disk made up of aluminium or glass that coated with thin layer of magnetic film on both sides. Disk spacers are use to separate the disks when there is a stack of disks. The disks are hold in placed using a disk clamp. Spindle motor is mounted on the base casting which is known as motor base assembly (MBA). The spindle motor operates as the axis and rotates the disk or disks (Gao *et al.*, 2008).

The magnetic head made up of a slider and a read-write transducer is mounted on a suspension which then mounted an actuator assembly. The actuator assembly can consists one or more actuator arms. The actuator assembly is represented in the form of a comb when a number of actuator arms are present and is pivotally mounted on base.

Hence with this assembly, the heads are positioned in between the disk surfaces and enabling them to perform data storing and retrieving across the disk surfaces. The voice coil motor (VCM) functions as an electro-magnetic positioning magnet which holds and rotates the actuator about the pivot.

Other smaller components such as filters, seals and plastic parts also play essential functions in the overall drive operations. All of these parts are sealed completely in an enclosure to protect them from contaminations.

1.3 Magnetic Recording Process

Data are store in HDD on the surfaces of the disks in the form of binary bits. The bits are written closely-spaced in the form of concentric, circular tracks on the disk surface as the disk rotates by the spindle motor. The motion between the magnetic head and magnetic disk enables the magnetic recording process. The disk rotates at high speed by the spindle motor (currently up to 10,000 revolutions per minute (rpm)) forms airflow that generates air bearing enabling the slider to fly across the disk surface with a gap between them. The gap that separates between the slider and the disk surface is known as the flying height as shown in Figure 1.3.

The read-write transducer consists of two separate elements called the read and write heads which write data to and read data from the disk or the recording medium. When current is applied to the inductive write element which consists of windings of electromagnet during writing, a tiny magnetic field is created and thus magnetizing the magnetic disk. The direction of the magnetization depends on the direction of the current applied to the write element.

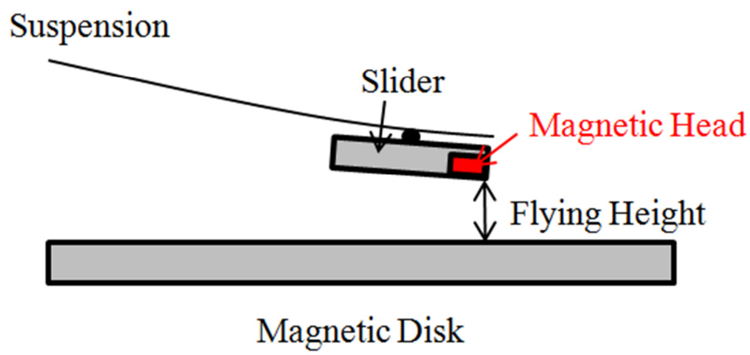

Figure 1.3 Schematic diagram of head-disk interface

The read element exhibits the magnetoresistive effect where it changes its resistivity in the presence of magnetic field. During reading, when the magnetic head passes over the magnetized bit, a current is sent through the read element. Figure 1.4 demonstrates the magnetic recording process.

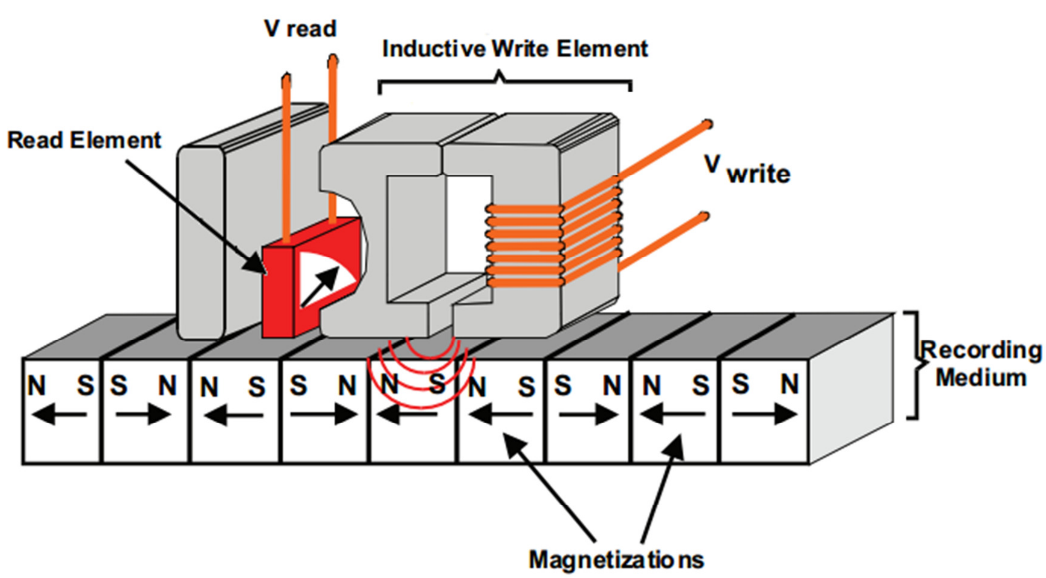

Figure 1.4 Magnetic recording process (HGST, 2007)

1.4 Emerging Trends in Hard Disk Drive Industry

The use of HDDs not only in consumer electronic devices but also in modern computer systems such as cloud storage and as high-capacity servers in data centers which require large storage capacity has driven the evolution of the hard disk technology. Modern HDD integrates the latest technology in magnetic recording, material science, and digital signal processing to achieve greater storage capacity (Strom *et al.*, 2007).

The storage capacity of a hard disk is determined by the areal density of a disk, which is the number of bits recorded in per square inch of the disk surface. The total capacity of the hard disk is then calculated based on the number of disk it contains. In order to enable higher areal density, various technologies have been incorporated in a HDD as follows (Jacob *et al.*, 2008):-

- Thinner magnetic coating which improves the magnetic properties
- Head design, i.e. read-write elements
- Flying height of the head
- Accuracy of head positioning servo, enabling tracks to be closer

One of the most critical factors which determine the areal density of the HDD is the separation distance between read-write elements and the disk where it directly affects both signal strength and resolution (Strom *et al.*, 2007). Therefore, the flying height decreases as magnetic recording densities increases where current lowest flying height is about 10 nanometers. Besides that, the closer the data tracks the higher density can be recorded on a disk surface.

1.5 Spindle Motor

The data in HDD are recorded and reproduced on concentric tracks. Hence, the data transfer rates are determined by the rotational speed of the spindle motor. As the tracks densities become higher and higher, it is necessary to reduce the off-tracks errors of heads. Besides that, other requirements for the spindle motor are high reliability, increased rotation speed, low noise and low power consumption (Matsuoka *et al.*, 2001). Figure 1.5 depicts both the ball bearing spindle motor and fluid dynamic bearing (FDB) spindle motor.

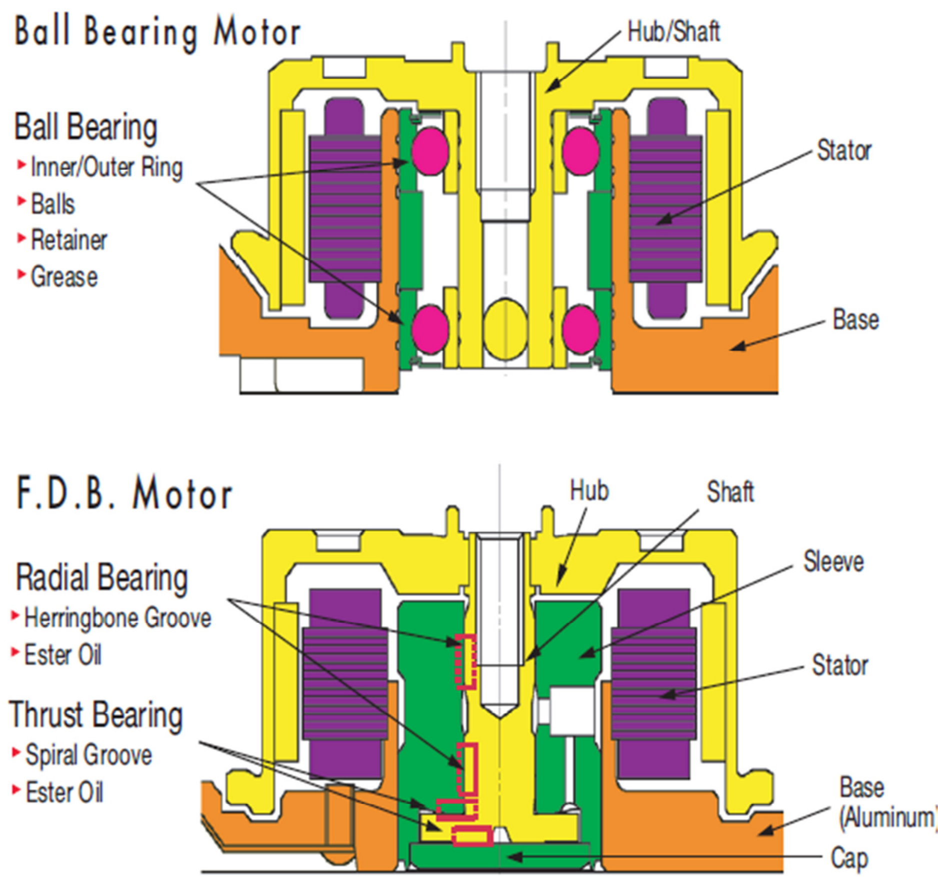

Figure 1.5 Ball bearing spindle motor and FDB spindle motor (HGST, 2012)

Ball bearings have been used in spindle motor for many years to rotate the disks. However, it can no longer meet the requirements of the current HDD performance as it generates high amount non repeatable run out (NRRO) which cause the disks to wobble. NRRO is the major cause of track misregistration where the head failed to follow the indented track, producing errors in positional information (Matsuoka *et al.*, 2001). Besides that, the tracks are not in perfect circular when written on the disk due to this factor which directly reduced the number of bits recorded in per square inch of the disk surface. This condition occurred when the ball bearings are not shaped into complete smooth spheres and the mechanical contact between the ball bearing and the raceway surfaces distorted due to preload. Tighter tolerance and inspections were implemented during production of ball bearing to minimize NRRO. Nevertheless, the ball bearing design spindle motor has already reaches its upper limit where it can no longer prevail over the NRRO issue at higher areal densities requirement. Other problems with the use of ball bearings are the noise produce during revolution and the bearing lifetime.

In order to fulfill the demanding HDD requirements for the spindle motor, a fluid dynamic bearing (FDB) has been incorporated into the spindle motor. In FDB spindle motor, there is a free of contact between the shaft and bearings in rotation which is surrounded with liquid in this case oil or lubricant. FDB oil or lubricant is crucial to allow the fluid dynamic motor to operate as per the requirements. With this feature, the lower level of rotation noise is generated and the life span of the spindle motor can be extended. However, there are some disadvantages about FDB spindle motors (Matsuoka *et al.*, 2001):-

- Wear of bearings caused by metallic contact at start and stop
- Deterioration of bearing performance and contamination of heads and disks caused by scattering and evaporation of lubricant
- Deterioration of lubricant (due to heat, moisture and etc.)
- Increase in load torque at start and during steady rotation caused by higher viscosity of lubricant at low temperatures
- Positional variations in axial direction of shaft or bearings

FDB oil or lubricant is crucial to allow the fluid dynamic motor to operate as required. The FDB life span and the design of bearings are influenced by the selection of lubricant. The desired lubricant properties are as follows (Matsuoka *et al.*, 2001):-

- Low degree of temperature dependence of viscosity
- Low volatility and aging rate
- Excellent heat resistance
- No generation of outgas that can cause contamination
- Excellent compatibility with bearing materials

FDB oil has been developed and is a combination of both base oil and additives in order to meet the specific needs. About 90% or more of the total amount of the FDB oil is made up of base oil which can be either synthetic hydrocarbon, ethers or esters. The base oil determined the fundamental properties of the FDB oil. The additives consist in a small amount (about less than 5%) of the overall FDB oil where it performs specific functions. The additives are categorized into a few classes depending on its function such as fluid lubrication, boundary lubrication and oxidation inhibitors to prevent deterioration (Kim *et al.*, 2001; Matsuoka *et al.*, 2001). As the spindle motor spins at the speed as high as 10,000 rpm, the oil temperature increases and it influenced the performance of the FDB spindle motor. In addition, heat will speed up the evaporation rate and chemical change of the FDB oil. As the consequences, FDB oil will be deteriorated and at the same time due to the evaporation effect, the FDB oil can contaminate other disk drive components which can reduce the life time of the HDD.

1.6 Actuator Pivot Bearing Cartridge

The actuator assembly is made up of actuator arms assembled with suspension and magnetic head which is pivotally attached to the base of the disk drive. For the actuator assembly to pivot the magnetic heads across the surface of the disks, the actuator assembly is installed with an actuator pivot bearing cartridge. The pivoting of the actuator assembly is limited to a certain angular range by the relative position of other disk drive components. The actuator pivot bearing cartridge has the similar design as the ball bearing spindle motor where it uses ball bearing for the pivoting function. The ball bearings are lubricated such as grease for them to rotate smoothly between the inner and outer ring (Hanke, 2013). Figure 1.6 illustrates the actuator pivot bearing cartridge.

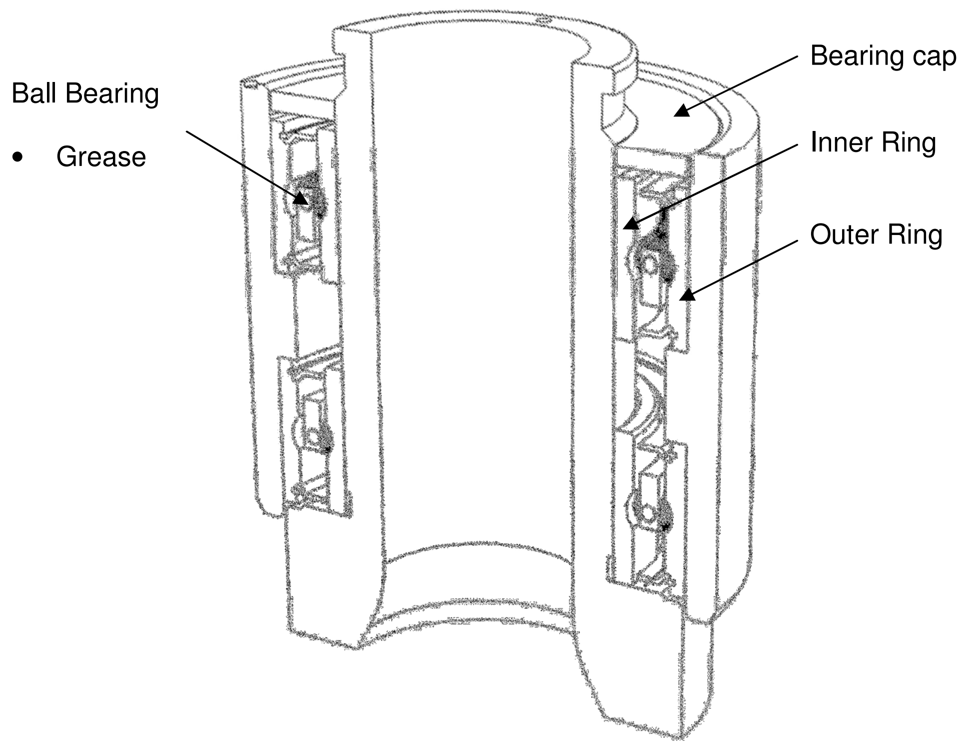

Figure 1.6 Actuator pivot bearing cartridge (Hanke, 2013)

For convenience, the grease used in actuator pivot bearing cartridge will be termed as pivot grease. Pivot grease has the similar composition as the FDB oil where the major composition is made up of base oil and a small amount of additives. As the HDD operates at high temperature based on the mechanics, the migration of the pivot grease via evaporation from the actuator pivot bearing cartridge to other locations in the HDD may occur. This will cause contamination on other components in the HDD and therefore reducing the life time of the HDD.

1.7 Contamination in Hard Disk Drive

Contaminants have been present all the time in HDD which may lead to drive failure. Contaminants can originate from both internal and external of the HDD enclosure where it can enter during the assembly process or from the component of the HDD. As the HDD becomes smaller and smaller due to the technology and consumer demands, the size of the HDD components also reduces which leads to the spacing reduction between the components. Hence, contaminants which were once present in HDD at a harmless level are causing the drive to fail. Generally there are three classes of contaminants which are organic, particulate and ionic contaminants.

There are several types of mechanism which organic contamination can cause failures in the HDD. Stiction is one of most severe organic contamination where it is due to the adhesion of the head to a disk during stationary resulted from thin film of condensed phase organic material on the head surface when the drive cools down. At drive operating temperatures where heat is generated due to the disk rotation by the spindle motor, liquid materials used in the drive components can be vaporized and accumulated on the head surface. Example of organic contaminant sources of in the HDDs are adhesives, oils, greases, and organic coatings.

Head crash is another serious contamination related failure of HDD. This phenomenon can occur when particles enter the head disk interface. Submicron-sized particles have become a problem with the higher density drive which has lower flying height or reduced spacing between the head and disk during the operation and disks rotating at 5400 rpm or greater. The drive can failed to operate when the head crash into the particle or the disk after flying over a particle. Particulate contaminant could be came from the glove powder or maybe HDD components that contain loose particle inclusion.

Besides that, corrosion of components within the drive can also caused drive operation failure. Corrosion is usually due to ionic contamination such as anions which can be chloride, nitrate or sulphate that react in the presence of moisture to form their corresponding volatile hydrochloric, nitric and sulfuric acids. Heads and disks are sensitive components in the drive which can be corroded due to the presence of high vapor pressure corrosive compounds. One of the possible sources of ionic contaminants is the ionic surfactants such as sulphate use in cleaning remained in the HDD components when the parts were not thoroughly cleaned.

1.8 Filters

The hard disk drives must be protected from the contaminants from the surrounding environment and also internally. Various strategies have been made available to prevent contamination such as assembling the HDD in control environment and thorough cleaning of the HDD components. Another measure that the HDD manufacturers took was designing a filtration system in the HDD to ensure the HDD enclosure able to be self cleaned and also to prevent external contaminants entering the assembled HDD.

There are two types of filters in the HDD which are recirculation filter and breather filter. Recirculation filter is made of pieces of filter media such as expanded PTFE membrane laminated to a polyester non-woven backing material, or "pillow-shaped" filters containing electrostatic filter media (Dauber, 2001). These filters are placed inside the disk drive assembly, typically in the active air path created by the rotating disks. The purpose of the recirculation filter is to trap particulate contamination which is generated inside the HDD or enters the HDD from the external environment.

Breather filter is placed over the breather hole which is designed into the base or the cover of the HDD. During the operation of the HDD, the air to enter and exit the drive via the breather hole allowing the drive to “breathe”. The breather filter is mounted and sealed over the breather hole to filter the particulates from the air entering the drive. Breather filter is usually made up of high-efficiency filter media, a low-outgassing adhesive and a silicone-free release liner. The filter media used is similar to the material used in recirculation filter. The breather filter is sealed to the base or the cover using a low-outgassing pressure sensitive adhesive while the silicone-free release liner is to secure and allow the ease the dispensing of the breather filters.

Both the recirculation filter and breather filter use filter media which are effective in removing the particulate contaminants generated from internal and external environment of the HDD but unable to address the vapor phase contaminants. Hence, adsorbent materials are incorporated in the filters design especially with the breather filter to address the problem where impregnation activated carbon is commonly used. By using the adsorbent material, both organic and corrosive vapors from the external environment and the internally generated organic vapors can be adsorbed. The construction of the adsorbent material into the breather filter is somewhat similar except that the adsorbent material is encapsulated between the layers of filter media and covered with the PTFE membrane making them waterproof.

1.9 Objective

The main objective of this study was to determine the level of FDB oil and pivot grease present in HDD assemblies after tested in the reliability chamber at 60 °C for a predetermined hours, with the run time of after 300 hours, 600 hours and 900 hours via GC-MS. Two different filters in the HDD assemblies, recirculation filter and adsorbent breather filter were extracted with organic solvent to quantify the amount of FDB oil and pivot grease. The HDDs were also built with three different pivot manufacturers for comparative studies.

CHAPTER 2

EXPERIMENTAL

2.1 Chemicals

FDB oil and pivot grease were obtained from a motor and a pivot supplier respectively. These chemicals are manufactured specifically for the motor and pivot manufacturers.

Commercially available chemicals used were analytical grade hexane and methylene chloride purchased from J.T. Baker (U.S.A.). Anthracene-d10 was obtained from Sigma-Aldrich (U.S.A.).

2.2 Standard Solutions

2.2.1 FDB Oil

50 ppm of FDB oil standard was prepared by weighing 5 mg of the compound and diluting it with 100 mL of methylene chloride in a volumetric flask. This standard is used to identify the FDB oil peak via GC-MS.

2.2.2 Pivot Grease

50 ppm of pivot grease standard was prepared by weighing 5 mg of the compound and diluting it with 100 mL of methylene chloride in a volumetric flask. This standard is used to identify the pivot grease signature via GC-MS.

2.2.3 Anthracene-D10 Spike Standard Solution

200 ppm of anthracene-d10 spike standard was prepared by dissolving 20 mg of the compound using 100 mL of methylene chloride in a volumetric flask. 1 mL of the 200 ppm anthracene-d10 was then diluted with 100 mL of hexane in volumetric flask to prepare 2 ppm of spike standard.

The spike standard solutions were prepared and mixed thoroughly by shaking and inverting the volumetric flasks repeatedly. The standard solutions were sealed and stored at 4 °C in a refrigerator not more than three months. This is to ensure that the standard solutions are not deteriorated when use.

2.3 Samples

A total of 27 HDDs built with combination of single spindle motor manufacturer and 3 different pivot manufacturers were tested in reliability chamber at 60 °C. The disk drives were powered up and performed the normal operation of read/write throughout the tested time. The disk drives were then removed and sampled at predetermined run time for analysis of the FDB oil and pivot grease within the drive by extracting the recirculation filter and adsorbent breather filter using hexane. The combination of disk drive assemblies and the run time for sampling selected are summarized in Table 2.1.

Table 2.1 HDD combination and sampling run time

Motor Supplier	Pivot Supplier	Run Time (h)
Supplier A	Supplier X	300
		600
		900
	Supplier Y	300
		600
		900
	Supplier Z	300
		600
		900

2.4 Sample Preparation

Hexane was used in performing extraction of FDB oil and pivot grease from both the recirculation filter and adsorbent breather filter. Even though there are many different types of solvents available, hexane is one of superior solvents used in extracting oil and grease for its ability to dissolve hydrocarbon.

2.4.1 Recirculation Filter

The recirculation filter in the drive was transferred into a clean 20 mL glass vial with PTFE lined cap. 1 mL of hexane was added into the vial and was capped. The vial was swirled for 1 minute to ensure that the recirculation filter was soaked. The filter was then left to soak for additional 4 minutes. The extract was collected in to a 1.5 mL GC vial and was dried using nitrogen gas purging. 100 µL of hexane was then added into the dried GC vial to re-dissolve the residue, and the solution was transferred completely to a 250 µL GC vial. The extract in the GC vial was once again dried using nitrogen gas purging before 50 µL of 2 ppm anthracene-d10 standard was added into the GC vial. The vial was capped tightly to ensure no evaporation occurs and followed by GC-MS analysis.

A total of 3 combinations of drives sampled at 3 different run time with three replicates each were prepared as shown in Table 2.2. A method blank was prepared following the same procedure without any recirculation filter for extraction.

2.4.2 Adsorbent Breather Filter

A cleaned blade and a cleaned pair of forceps were used to remove the PTFE layer of the adsorbent breather filter. The carbon layer was then placed into a clean 20 mL glass vial with PTFE lined cap and extracted in 5 mL of hexane for 16 hours. 1 mL of the 5 mL extracted solvent was then drawn and transferred into a 1.5 mL GC vial. Finally, 10 μL of 200 ppm anthracene-d10 standard was added into the GC vial. The vial was capped tightly to ensure no evaporation occurs and followed by GC-MS analysis.

A total of 3 combinations of drives sampled at 3 different run time with three replicates each were prepared as shown in Table 2.2. A method blank was prepared following the same procedure without any carbon for extraction.

Table 2.2 Samples for FDB oil and pivot grease analysis

Motor Supplier	Pivot Supplier	Run Time (h)	Number of Replicates
Supplier A	Supplier X	300	3
		600	3
		900	3
	Supplier Y	300	3
		600	3
		900	3
	Supplier Z	300	3
		600	3
		900	3

2.5 Quantification of FDB Oil and Pivot Grease

The peaks obtained were integrated using the Chemstation software after the GC-MS data acquisition. Both the FDB oil and pivot grease peaks are identified by subjecting the standards prepared to GC-MS analysis. The amount of the compound of interest was calculated with reference to the peak area of anthracene-d10 standard internal standard using the following equation:

$$Amount\ of\ interested\ compound = \frac{Peak\ area\ of\ interestedcompound}{Peak\ area\ of\ internal\ standard} \times xy \quad \text{Equation 1}$$

where x = Concentration of internal standard

y = Volume of internal standard

For pivot grease, the pivot grease signature hump is integrated. Peaks on the top of the hump were integrated using valley-to-valley method. Peaks area such as phthalate and FDB oil were subtracted from the overall integrated hump peak area.

2.6 Recovery Study

2.6.1 Recirculation Filter

50 μL of 2 ppm anthracene-d10 standard were spiked on the 3 different new recirculation filters and were left to dry prior to sample extraction to check the accuracy of the extraction method. The samples were then extracted as described above in section 2.4.1 except addition of 50 μL of 2 ppm anthracene-d10 standard into 250 μL GC vial before GC analysis was not performed.

2.6.2 Adsorbent Breather Filter

10 μL of 200 ppm anthracene-d10 standard were spiked on the 3 different new adsorbent breather filters and were left to dry prior to sample extraction to check the accuracy of the extraction method. The samples were then extracted as described above in section 2.4.2 except addition of 10 μL of 200 ppm anthracene-d10 standard into 1.5 mL GC vial before GC analysis was not performed.

2.7 GC-MS Settings

GC-MS system from Hewlett Packard (HP) 6890 GC system coupled with 5973 mass selective detector and equipped with 7673 auto injector was used to determine the amount of the FDB oil and pivot grease. Prior to sample run, standard spectra tuning was performed to ensure the system was in good condition by determining the percentages level of air and water. HP-5MS column from Agilent Technologies, poly(5%-diphenyl-95%-dimethylsiloxane) fused silica column with 30 m length, 0.25 mm inner diameter and 0.25 μm film thickness was used for separation. Helium was used as the carrier gas in this system. Table 2.3 shows the GC-MS settings used for the RD910 and pivot grease analysis.

Table 2.3 HP 6890-5973 GC-MS instrument set-up

Parameter	Setting
Injector temperature	300 °C
Injection mode	Splitless
Sample injection volume	3 µL
Septum purge flow	1 mL/min
Column flow	1 mL/min
Column mode	Constant flow
Initial column temperature	30 °C
Initial Time	1 min
Temperature ramp	15 °C/min
Final temperature	300 °C
Solvent delay	4.5 min
Total run time	Approximately 40 min
MS interface temperature	300 °C
Ion source	Electron ionisation
Electron energy	70 eV
Ion source temperature	230 °C
Mass analyser	Quadrupole
Quadrupole temperature	150 °C
Mass range	33 – 700 amu
Scan time	1.14 scan/s
Mass detector	Electron multiplier

CHAPTER 3

RESULTS AND DISCUSSION

3.1 FDB Oil

Prior to the identification of the FDB oil peak from the extracts of recirculation filter and adsorbent breather filter, a 50 ppm FDB oil standard solution was subjected to GC-MS analysis. From the total ion chromatogram (TIC), there are four main peaks which were observed at retention time of 18.070, 18.347, 18.757 and 19.683 min. The chromatogram and the mass spectra corresponding to peak at the retention time of 18.347 min are shown in Figure 3.1.

As stated in section 1.5, FDB oil consists of 90% base oil which is synthetic hydrocarbon or ester. Hence, the peak with the retention time 18.347 min which is attributed to decanoic acid-hexadecyl ester with *m/z* of 155, 173 and 244 was the most prominent peak. This peak was chosen to determine and quantify the presence FDB oil in the extracts from the recirculation filter and adsorbent breather filter.

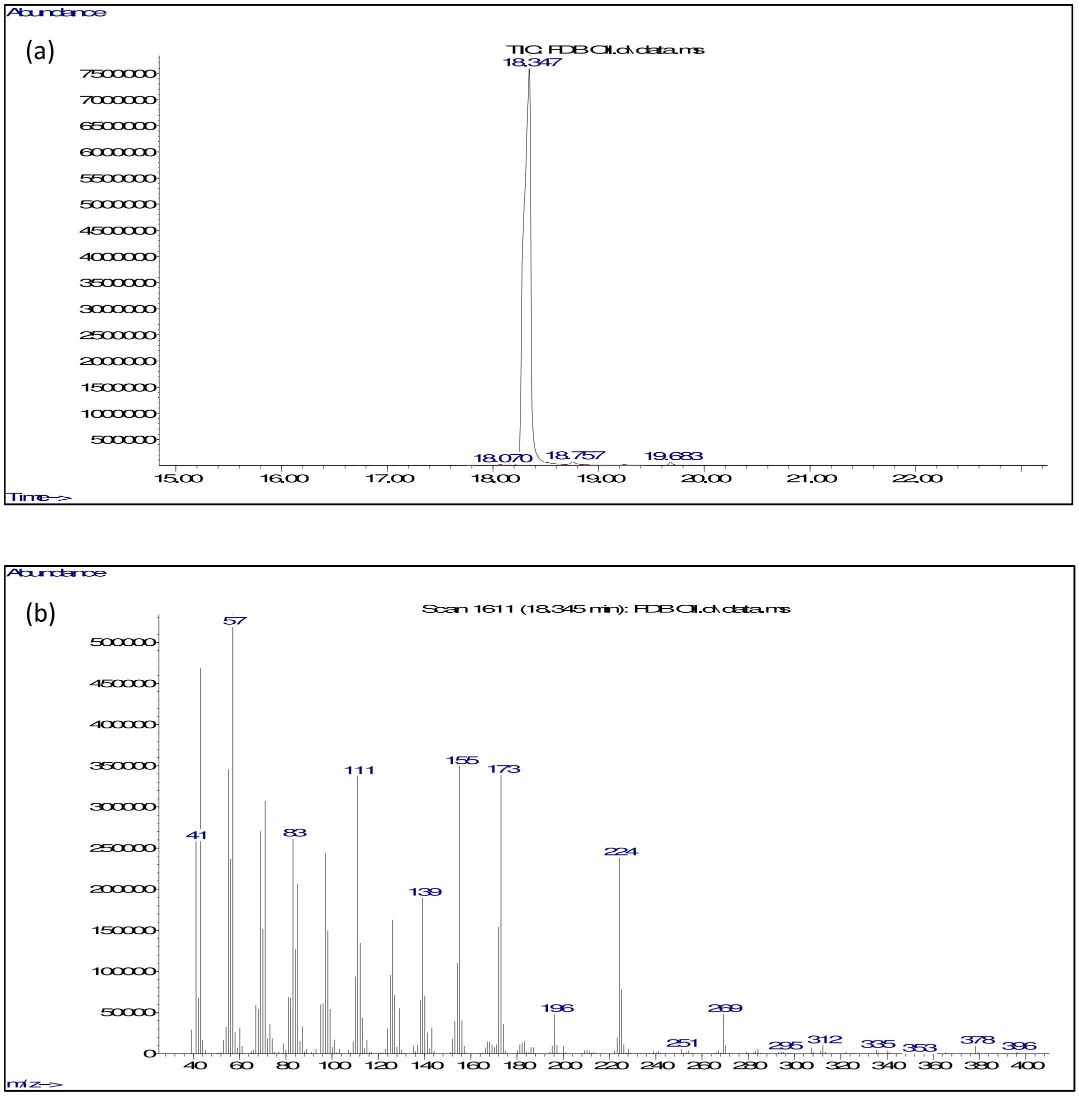

Figure 3.1 (a) Gas chromatogram for FDB oil and (b) mass spectrum for peak retention time 18.347 min (Decanoic acid-hexadecyl ester)

The FDB oil level in the recirculation filter and adsorbent breather filter was analyzed using GC-MS after the extraction with hexane solvent. The amount of the FDB oil was quantified with reference to the peak area of anthracene-d10 standard in the chromatograms according to equation 1. The results are tabulated and plotted in Table 3.1 and Figure 3.2 for recirculation filter and Table 3.2 and Figure 3.3 for adsorbent breather filter. The results were plotted into individual plot for the extracts obtained from recirculation filter and adsorbent breather filter as the HDDs were built using single spindle motor manufacturer and with the assumption of the spindle motor spins at the same rpm throughout the operation during the run time. Based on the results tabulated and plotted, the amount of FDB oil detected from both the extracts of recirculation filter and adsorbent breather filter was found increase with the run time.

Table 3.1 Total concentration of FDB oil detected in recirculation filter

Motor Supplier	Pivot Supplier	Run Time (h)	Total concentration of FDB oil (ng/sample), n = 3
Supplier A	Supplier X	300	$(0.9 \pm 0.6) \times 10^2$
		600	$(1.1 \pm 0.1) \times 10^2$
		900	$(1.2 \pm 0.3) \times 10^2$
	Supplier Y	300	$(0.5 \pm 0.2) \times 10^2$
		600	$(0.9 \pm 0.1) \times 10^2$
		900	$(1.0 \pm 0.4) \times 10^2$
	Supplier Z	300	$(0.5 \pm 0.1) \times 10^2$
		600	$(0.6 \pm 0.1) \times 10^2$
		900	$(0.8 \pm 0.3) \times 10^2$

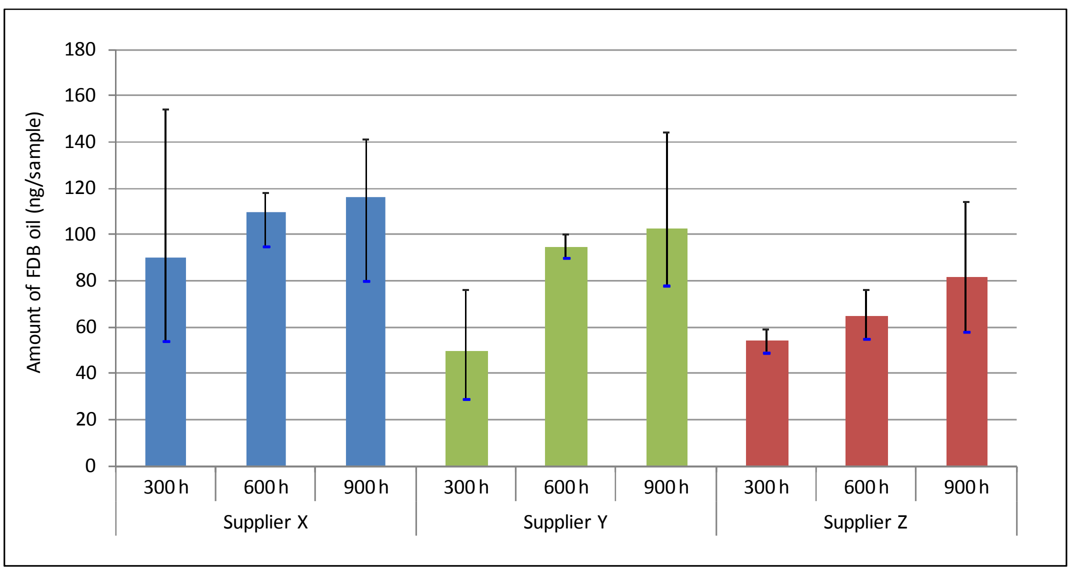

Figure 3.2 Plot of the measured total concentration of FDB oil detected in recirculation filter (n = 3)

Figure 3.2 showed the rate of FDB oil vapor collected in the recirculation filter as the increasing HDD test time. However, it is observed that there was not much changes in the concentration of FDB oil extracted from the recirculation filter from the HDDs built with pivot from Supplier X and Supplier Y between run time after 600 hours and 900 hours as compared to Supplier Z. These results indicated that the recirculation filter is reaching to its saturation point in collecting the FDB oil vapor in the HDDs. In another point of view, the maximum amount of FDB oil in vapor form in the air in the HDD is equal to the amount of FDB oil collected in the recirculation filter which is 116 ng/sample.

Table 3.2 Total concentration of FDB oil detected in adsorbent breather filter

Motor Supplier	Pivot Supplier	Run Time (h)	Total concentration of FDB oil (ng/sample), n = 3
Supplier A	Supplier X	300	$(1.5 \pm 1.2) \times 10^2$
		600	$(2.3 \pm 0.6) \times 10^2$
		900	$(3.4 \pm 0.9) \times 10^2$
	Supplier Y	300	$(0.5 \pm 0.2) \times 10^2$
		600	$(3.0 \pm 0.1) \times 10^2$
		900	$(4.4 \pm 0.7) \times 10^2$
	Supplier Z	300	$(0.5 \pm 0.1) \times 10^2$
		600	$(1.5 \pm 0.2) \times 10^2$
		900	$(2.2 \pm 0.8) \times 10^2$

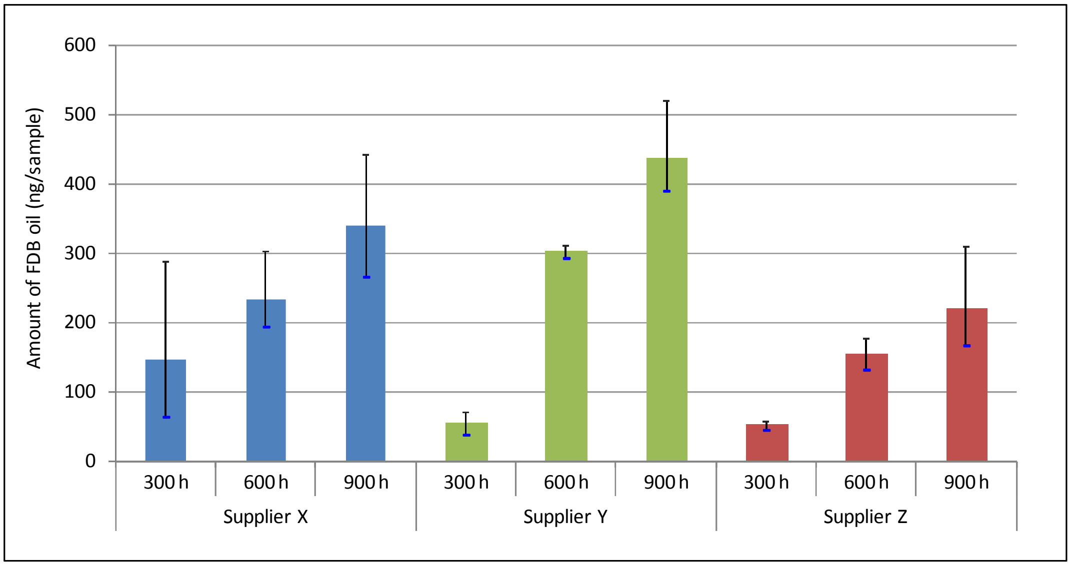

Figure 3.3 Plot of the measured total concentration of FDB oil detected in adsorbent breather filter (n = 3)

Figure 3.3 showed the rate of FDB oil vapor collected in the adsorbent breather filter with the increasing HDD test time. The concentration of the FDB oil extracted from the adsorbent breather filter increased in a constant rate. It did not show the similar trend as observed in the FDB oil extracted from the recirculation filter. The adsorbent breather filter is effective in removing the FDB oil vapor in the HDD with the presence of the impregnated activated carbon.

The standard deviations between three replicates for each of the HDD combinations and test run time from both recirculation filter and adsorbent breather filter showed high sample to sample variation. This may be due to the temperature of the drive during operation inconsistent across all the HDD which leads to the inconsistency of the evaporation rate of the FDB oil.

3.2 Pivot Grease

A 50 ppm pivot grease standard solution was subjected to GC-MS analysis prior to the identification of the pivot grease from the extracts of recirculation filter and adsorbent breather filter. From the total ion chromatogram (TIC), there are a few signature peaks which are observed in the pivot grease chromatogram such as amine compounds and hydrocarbon hump (Figure 3.4 (a)).

The pivot grease signature hydrocarbon hump which starts from 18.00 min to 19.50 min was selected for the determination and quantification of the pivot grease extracted from the recirculation filter and adsorbent breather filter. The retention time for the pivot grease signature hydrocarbon hump does not extend to retention time 32.00 min as the pivot grease's hydrocarbon hump has some overlapping with the recirculation filter hydrocarbon hump. The pivot grease signature hydrocarbon hump from 18.00 min to 19.50 min is shown in Figure 3.4 (b). The amine peaks were not chosen for the quantification for pivot grease as the peaks were present in low abundance or were not present at all in some extracts.

The pivot grease level in the recirculation filter and adsorbent breather filter was analyzed using GC-MS after the extraction with hexane as solvent and was quantified by integrating the signature hydrocarbon hump and excluding the phthalate and FDB oil with reference to the peak area of anthracene-d10 standard in the chromatograms according to equation 1. The results are tabulated and plotted in Table 3.3 and Figure 3.5 for recirculation filter and Table 3.4 and Figure 3.6 for adsorbent breather filter.

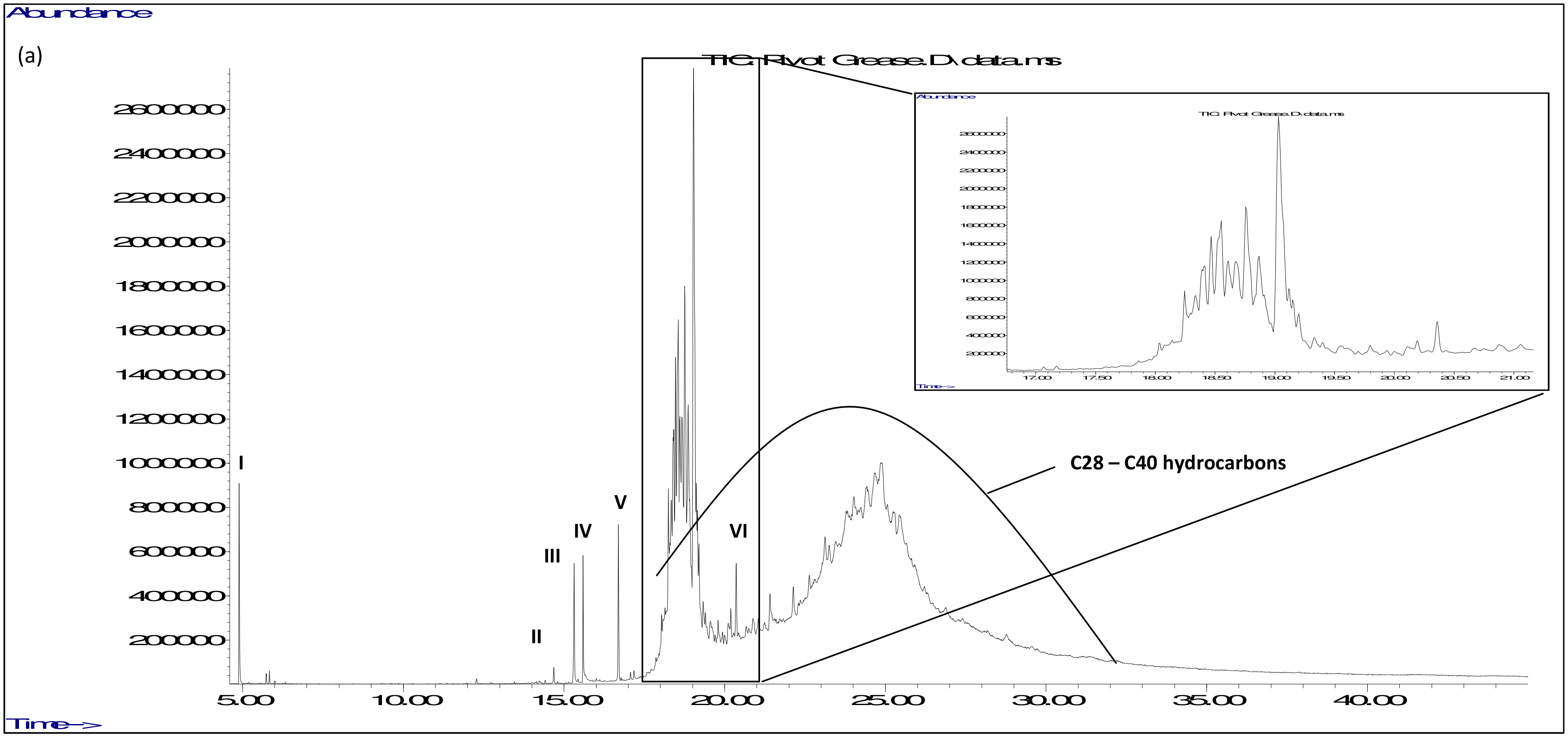

Figure 3.4 (a) Gas chromatogram for pivot grease [Cyclohexyl amine (I), Butyl diphenyl amine (II), Urea, N,N'-dicyclohexyl (III), Methylene diphenyl diisocyanate (VI), Octyl diphenyl amine (V), Bis-(octylphenyl) amine (VI)] and (b) gas chromatogram for pivot grease signature hydrocarbon hump

Table 3.3 Total concentration of pivot grease detected in recirculation filter

Motor Supplier	Pivot Supplier	Run Time (h)	Total concentration of pivot grease (ng/sample), n = 3
Supplier A	Supplier X	300	$(0.74 \pm 0.04) \times 10^3$
		600	$(0.94 \pm 0.07) \times 10^3$
		900	$(1.07 \pm 0.02) \times 10^3$
	Supplier Y	300	$(1.07 \pm 0.25) \times 10^3$
		600	$(1.13 \pm 0.39) \times 10^3$
		900	$(1.10 \pm 0.08) \times 10^3$
	Supplier Z	300	$(0.83 \pm 0.01) \times 10^3$
		600	$(0.83 \pm 0.04) \times 10^3$
		900	$(1.02 \pm 0.08) \times 10^3$

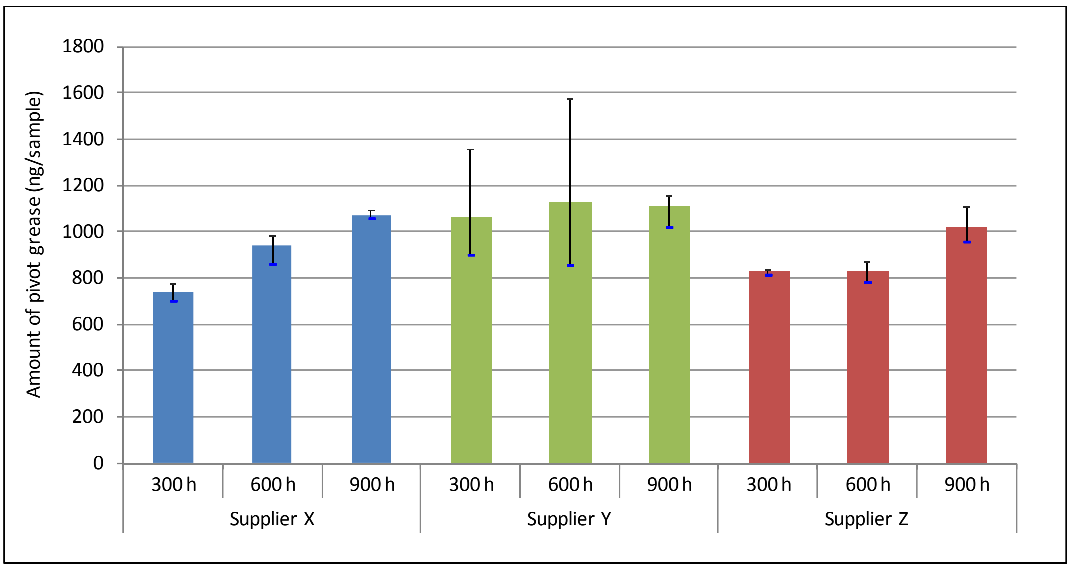

Figure 3.5 Plot of the measured total concentration of pivot grease detected in recirculation filter (n = 3)

Figure 3.5 showed the pivot grease collected from the recirculation filter against the HDD test time. Based on the results, pivot from Supplier Y generated the highest amount of pivot grease vapor in the HDD throughout the test time while pivot from Supplier X and Supplier Z showed the similar amount of pivot grease vapor generated.

Besides that, there was not much increased in the amount of pivot grease extracted from the recirculation filter from the HDDs built with pivot Supplier Y. This showed that recirculation filter has reached to its maximum amount of pivot grease that can be collected. This indirectly indicated that is maximum amount of pivot grease vapor form in the air in the HDD at the point of HDD in operation mode was about 1120 ng/sample.

Table 3.4 Total concentration of pivot grease detected in adsorbent breather filter

Motor Supplier	Pivot Supplier	Run Time (h)	Total concentration of pivot grease (ng/sample), n = 3
Supplier A	Supplier X	300	$(2.7 \pm 0.8) \times 10^3$
		600	$(5.1 \pm 1.5) \times 10^3$
		900	$(7.9 \pm 0.2) \times 10^3$
	Supplier Y	300	$(2.2 \pm 1.2) \times 10^3$
		600	$(6.6 \pm 1.4) \times 10^3$
		900	$(10.3 \pm 0.8) \times 10^3$
	Supplier Z	300	$(2.1 \pm 0.7) \times 10^3$
		600	$(3.7 \pm 1.5) \times 10^3$
		900	$(5.9 \pm 0.5) \times 10^3$

Figure 3.6 showed the amount of pivot grease generated with the HDD run time collected in the adsorbent breather filter. Based on the results, similar observations were observed in the amount of pivot grease vapor collected in the recirculation filter. The rate of the evaporation of pivot grease from Supplier Y was fastest compared to Supplier X and Supplier Z. When compared between recirculation filter and the adsorbent breather filter for pivot from Supplier Y, the amount of pivot grease vapor collected in recirculation filter reaches the plateau state while in the adsorbent breather filter the amount of pivot grease vapor collected was still increasing. From this observation, it can be concluded that the adsorbent breather filter effectively removes the pivot grease vapor from the recirculation filter and from the air in the HDD.

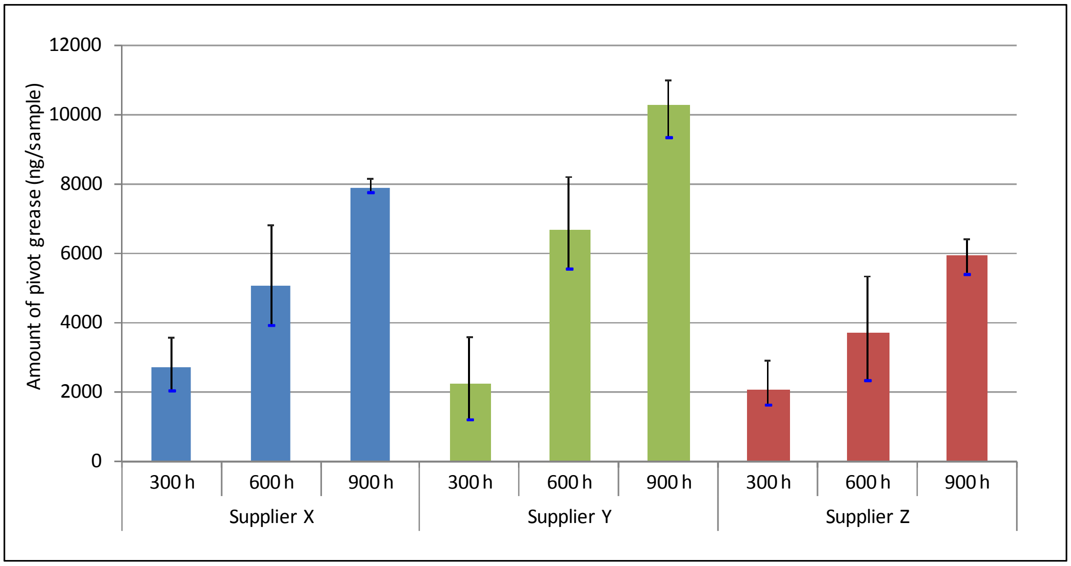

Figure 3.6 Plot of the measured total concentration of pivot grease detected in adsorbent breather filter (n = 3)

High sample to sample variation was observed based on the standard deviations between three replicates for each of the HDD combinations and test run time from both recirculation filter and adsorbent breather filter. This may be due to the inconsistent temperature of the drive during operation which leads to the inconsistency of the evaporation rate of the pivot grease. Besides that, the design of the pivot also affects the evaporation rate of the pivot grease as higher standard deviations is observed for pivot from Supplier Y as compared to the other two suppliers, Supplier X and Supplier Z.

3.3 Recovery Study

3.3.1 Recirculation Filter

The accuracy of the extraction method was examined by the recoveries of 50 µL of 2 ppm anthracene-d10 standards which were spiked onto new recirculation filters and 10 µL of 200 ppm anthracene-d10 standard were spiked on new adsorbent breather filters prior extraction. The recoveries for the adsorbent breather filter were quantified against the 2 ppm anthracene-d10 as the final concentration of the anthracene-d10 in the extracts is 2 ppm. The 2 ppm anthracene-d10 standard peak has the retention time of 13.563 min with the peak area of 8264047 (Figure 3.7). The recoveries for both recirculation filter and adsorbent breather filter were summarized in Table 3.5.

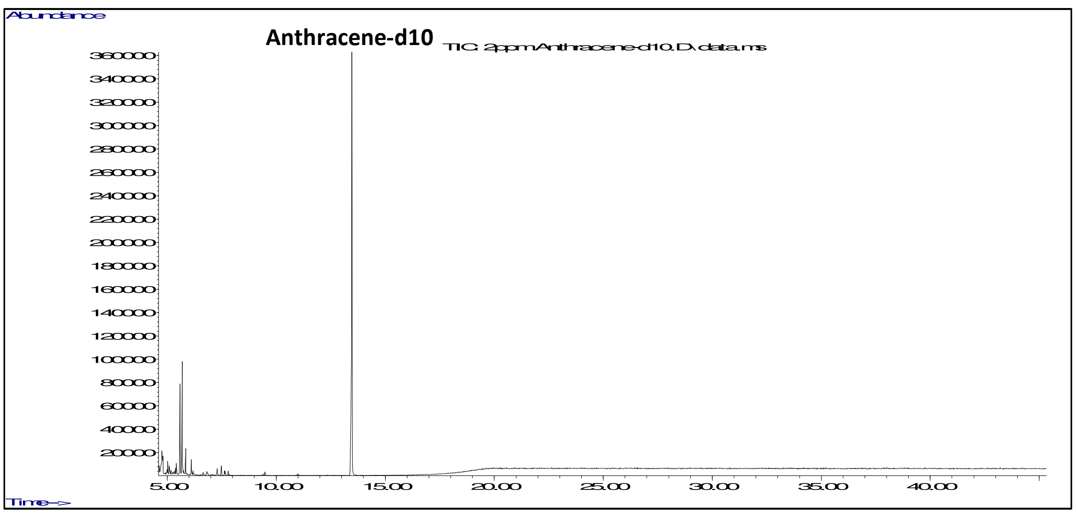

Figure 3.7 Gas chromatogram of 2 ppm anthracene-d10 standard

Table 3.5 Recovery of anthracene-d10 standard

Component	Recovery (%), n = 3	RSD (%)
Recirculation filter	131 ± 11	8.6
Adsorbent breather filter	102 ± 12	12.3

Generally, the effectiveness of the extraction method for both the samples was acceptable. The recirculation filter samples showed recovery of 131% which is slightly higher than the acceptable recovery percentage. While the adsorbent breather filter showed recovery of 97% which was satisfactory. The low recovery of the standard could be affected by the errors which might have been introduced during the sample extraction steps that cause some loss of the sample. Evaporation of solvent during the extraction method at the soaking step or the improper GC vial capping could lead to higher recovery obtained.

CHAPTER 4

CONCLUSION

4.1 Conclusion

The presence of the FDB oil and pivot grease used in the HDD were able to be determined and quantified using hexane extraction of the recirculation filter and adsorbent breather filter in the HDD by using GC-MS method.

From the recirculation filter extraction, the amount of FDB oil and pivot grease that were present in the HDD at the point of saturation are 116 ng/sample and 1120 ng/sample. The amount of both FDB oil and pivot grease from the extracts of the adsorbent breather filter on were found to increase with HDD run time. It was also observed that even though the recirculation filter has reached the maximum amount that it can collect for both FDB oil and pivot grease vapors, the adsorbent breather filter was able to clean up these vapors in the HDD.

With the presences of the recirculation filter and adsorbent breather filter, the HDD are able to self clean the FDB oil and pivot grease vapors which are introduced by the components. Consequently, the contamination causes by the organic vapors are able to be reduced.

Nevertheless, further studies could be performed on the adsorbent breather filter using the demonstrated extraction method to determine its saturation point. This study showed that even after 900 hours of test time, the amount of both FDB oil and pivot grease vapors have yet to reach the maximum volume that they can evaporate. Hence, the saturation point for the adsorbent breather filter is valuable information as once it reaches to the point that it unable to clean up the organic vapors generated by the components which will then lead to the HDD failure.

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APPENDICES

Appendix 1: Total concentration of FDB oil detected in each recirculation filter

Motor Supplier	Pivot Supplier	Run Time (h)	Total concentration of FDB oil (ng/sample)	Total concentration of FDB oil average (ng/sample)
Supplier A	Supplier X	300	0.5×10^2	$(0.9 \pm 0.6) \times 10^2$
			1.5×10^2	
			0.6×10^2	
		600	1.0×10^2	$(1.1 \pm 0.1) \times 10^2$
			1.2×10^2	
			1.2×10^2	
		900	1.4×10^2	$(1.2 \pm 0.3) \times 10^2$
			1.3×10^2	
			0.8×10^2	
	Supplier Y	300	0.3×10^2	$(0.5 \pm 0.2) \times 10^2$
			0.4×10^2	
			0.8×10^2	
		600	1.0×10^2	$(0.9 \pm 0.1) \times 10^2$
			0.9×10^2	
			0.9×10^2	
		900	0.9×10^2	$(1.0 \pm 0.4) \times 10^2$
			1.4×10^2	
			0.8×10^2	
	Supplier Z	300	0.5×10^2	$(0.5 \pm 0.1) \times 10^2$
			0.6×10^2	
			0.5×10^2	
		600	0.6×10^2	$(0.6 \pm 0.1) \times 10^2$
			0.8×10^2	
			0.6×10^2	
		900	0.7×10^2	$(0.8 \pm 0.3) \times 10^2$
			0.6×10^2	
			1.1×10^2	

Appendix 2: Total concentration of FDB oil detected in each adsorbent breather filter

Motor Supplier	Pivot Supplier	Run Time (h)	Total concentration of FDB oil (ng/sample)	Total concentration of FDB oil average (ng/sample)
Supplier A	Supplier X	300	0.6×10^2	$(1.5 \pm 1.2) \times 10^2$
			2.9×10^2	
			0.8×10^2	
		600	2.0×10^2	$(2.3 \pm 0.6) \times 10^2$
			3.0×10^2	
			1.9×10^2	
		900	4.4×10^2	$(3.4 \pm 0.9) \times 10^2$
			3.1×10^2	
			2.7×10^2	
	Supplier Y	300	0.4×10^2	$(0.5 \pm 0.2) \times 10^2$
			0.5×10^2	
			0.7×10^2	
		600	3.1×10^2	$(3.0 \pm 0.1) \times 10^2$
			2.9×10^2	
			3.0×10^2	
		900	3.9×10^2	$(4.4 \pm 0.7) \times 10^2$
			5.2×10^2	
			4.0×10^2	
	Supplier Z	300	0.6×10^2	$(0.5 \pm 0.1) \times 10^2$
			0.5×10^2	
			0.5×10^2	
		600	1.3×10^2	$(1.5 \pm 0.2) \times 10^2$
			1.6×10^2	
			1.8×10^2	
		900	1.8×10^2	$(2.2 \pm 0.8) \times 10^2$
			1.7×10^2	
			3.1×10^2	

Appendix 3: Total concentration of pivot grease detected in each recirculation filter

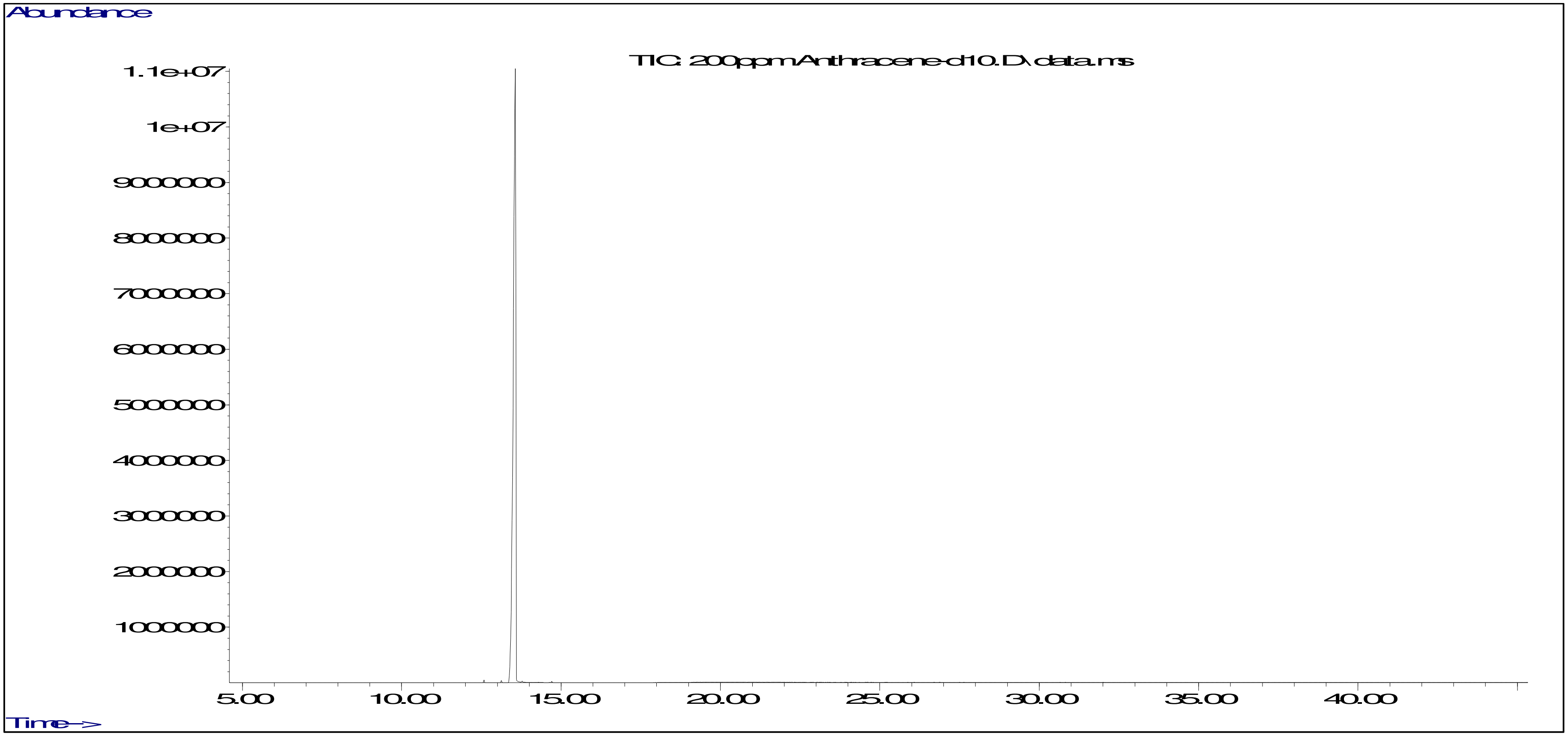
Motor Supplier	Pivot Supplier	Run Time (h)	Total concentration of pivot grease (ng/sample)	Total concentration of pivot grease average (ng/sample)
Supplier A	Supplier X	300	0.70×10^3	$(0.74 \pm 0.04) \times 10^3$
			0.74×10^3	
			0.77×10^3	
		600	0.98×10^3	$(0.94 \pm 0.07) \times 10^3$
			0.98×10^3	
			0.86×10^3	
		900	1.07×10^3	$(1.07 \pm 0.02) \times 10^3$
			1.06×10^3	
			1.09×10^3	
	Supplier Y	300	0.94×10^3	$(1.07 \pm 0.25) \times 10^3$
			0.90×10^3	
			1.35×10^3	
		600	1.57×10^3	$(1.13 \pm 0.39) \times 10^3$
			0.86×10^3	
			0.95×10^3	
		900	1.02×10^3	$(1.10 \pm 0.08) \times 10^3$
			1.15×10^3	
			1.15×10^3	
	Supplier Z	300	0.83×10^3	$(0.83 \pm 0.01) \times 10^3$
			0.82×10^3	
			0.84×10^3	
		600	0.78×10^3	$(0.83 \pm 0.01) \times 10^3$
			0.87×10^3	
			0.84×10^3	
		900	0.99×10^3	$(1.02 \pm 0.08) \times 10^3$
			0.96×10^3	
			1.11×10^3	

Appendix 4: Total concentration of pivot grease detected in each adsorbent breather filter

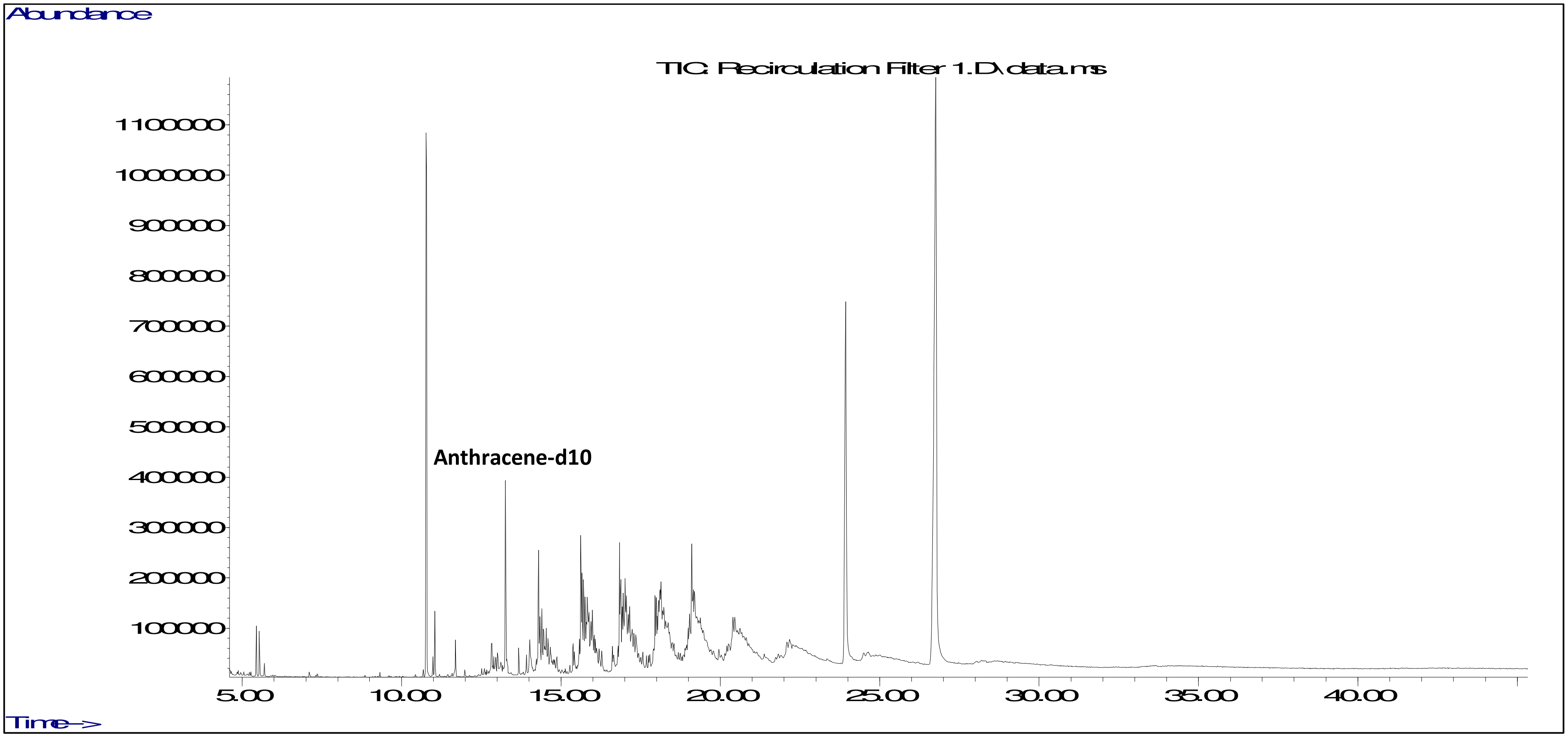
Motor Supplier	Pivot Supplier	Run Time (h)	Total concentration of pivot grease (ng/sample)	Total concentration of pivot grease average (ng/sample)
Supplier A	Supplier X	300	2.5×10^3	$(2.7 \pm 0.8) \times 10^3$
			3.6×10^3	
			2.0×10^3	
		600	3.9×10^3	$(5.1 \pm 1.5) \times 10^3$
			6.8×10^3	
			4.5×10^3	
		900	8.1×10^3	$(7.9 \pm 0.2) \times 10^3$
			7.8×10^3	
			7.8×10^3	
	Supplier Y	300	3.6×10^3	$(2.2 \pm 1.2) \times 10^3$
			1.2×10^3	
			1.8×10^3	
		600	8.2×10^3	$(6.6 \pm 1.4) \times 10^3$
			5.6×10^3	
			6.2×10^3	
		900	11.0×10^3	$(10.3 \pm 0.8) \times 10^3$
			10.5×10^3	
			9.3×10^3	
	Supplier Z	300	1.6×10^3	$(2.1 \pm 0.7) \times 10^3$
			1.7×10^3	
			2.9×10^3	
		600	2.3×10^3	$(3.7 \pm 1.5) \times 10^3$
			3.4×10^3	
			5.3×10^3	
		900	6.0×10^3	$(5.9 \pm 0.5) \times 10^3$
			5.4×10^3	
			6.4×10^3	

Appendix 5: Individual anthracene-d10 standard recovery

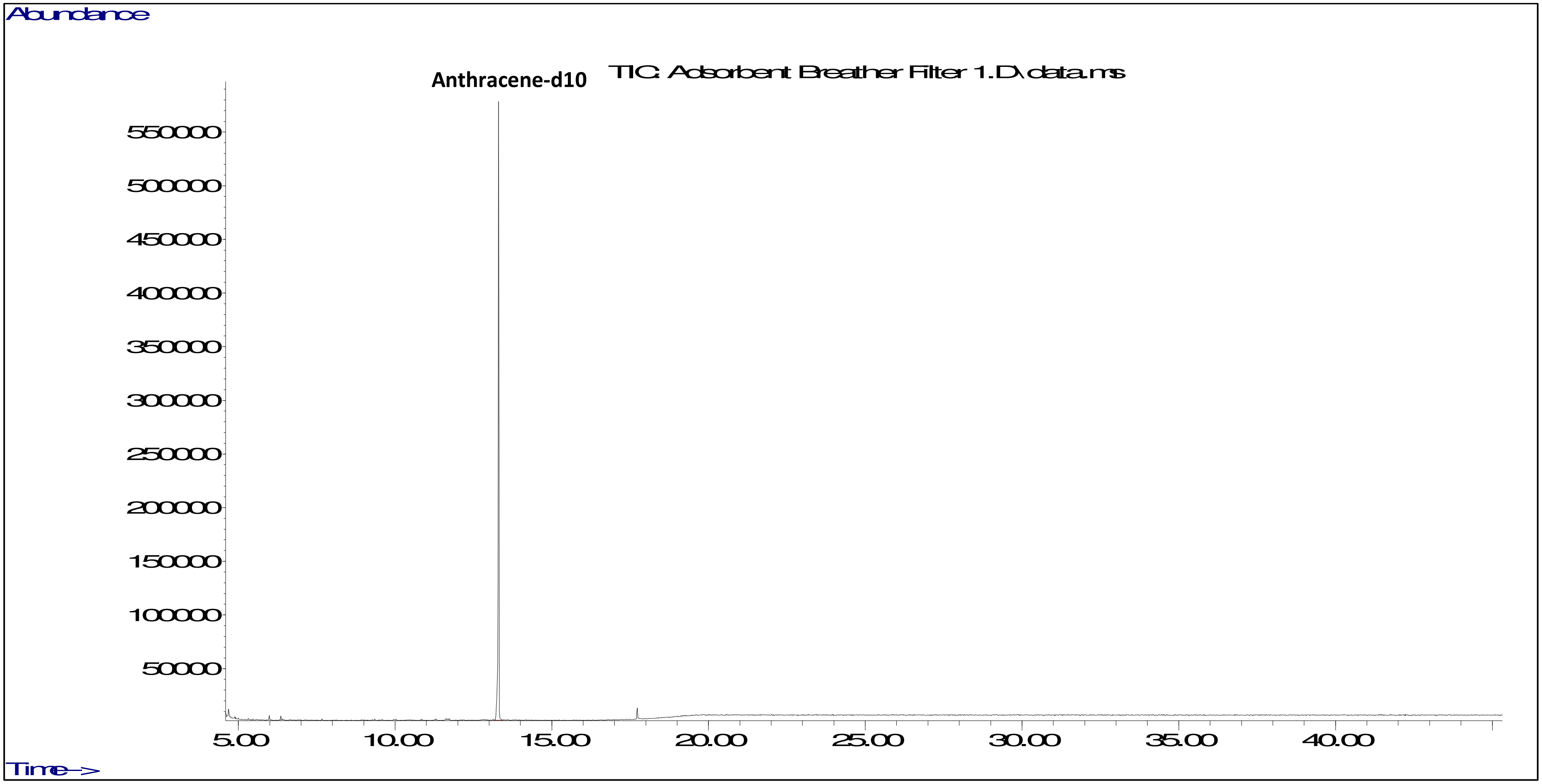
Component	Area of anthracene-d10	Recovery (%)	Recovery average (%)	RSD (%)
Recirculation filter	11918235	144	131 ± 11	8.6
	10166669	123		
	10503910	127		
Adsorbent breather filter	9607165	116	102 ± 12	12.3
	7832469	95		
	7797245	94		



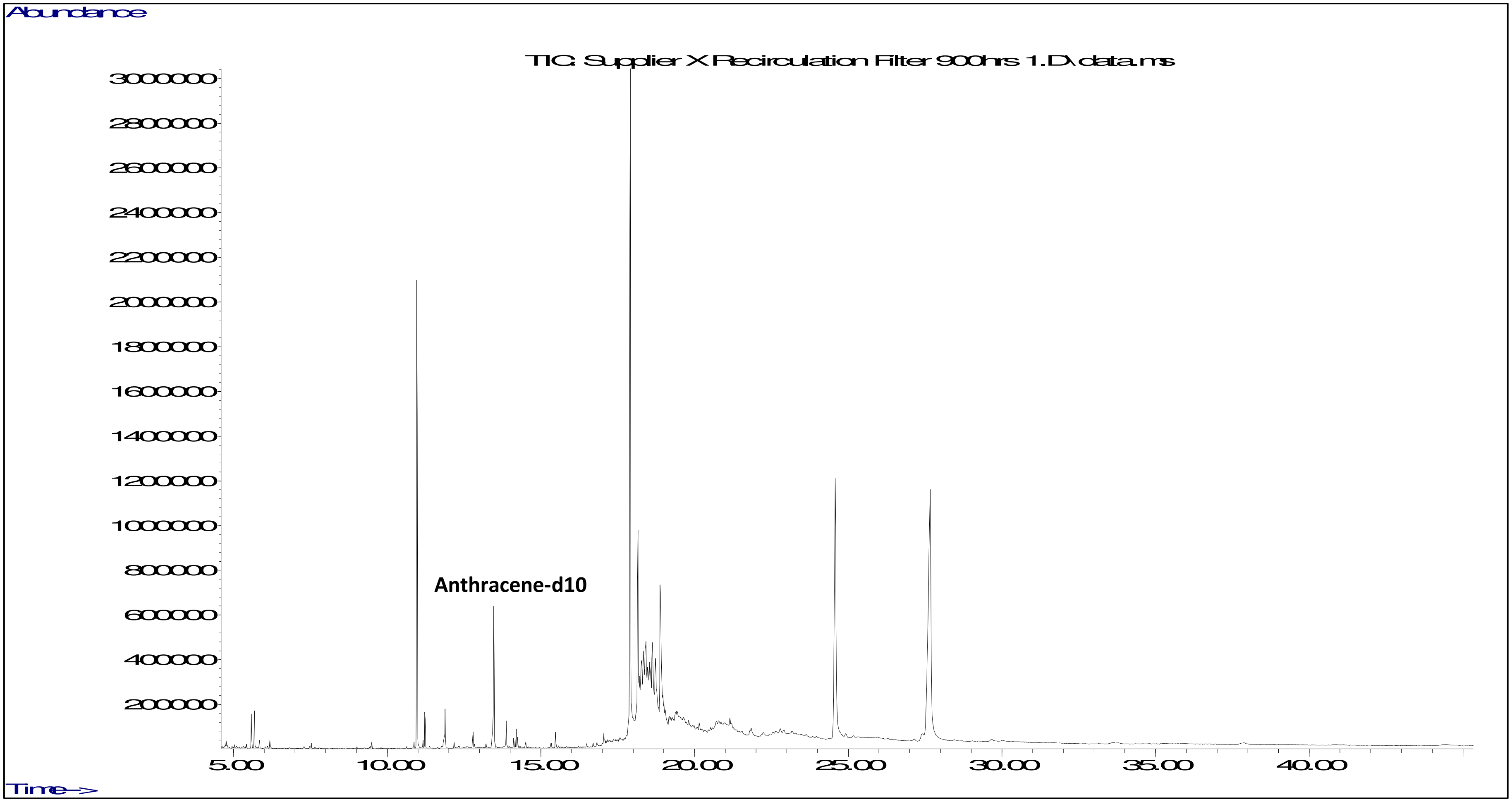
Appendix 6: Gas chromatogram of 200 ppm anthracene-d10 standard



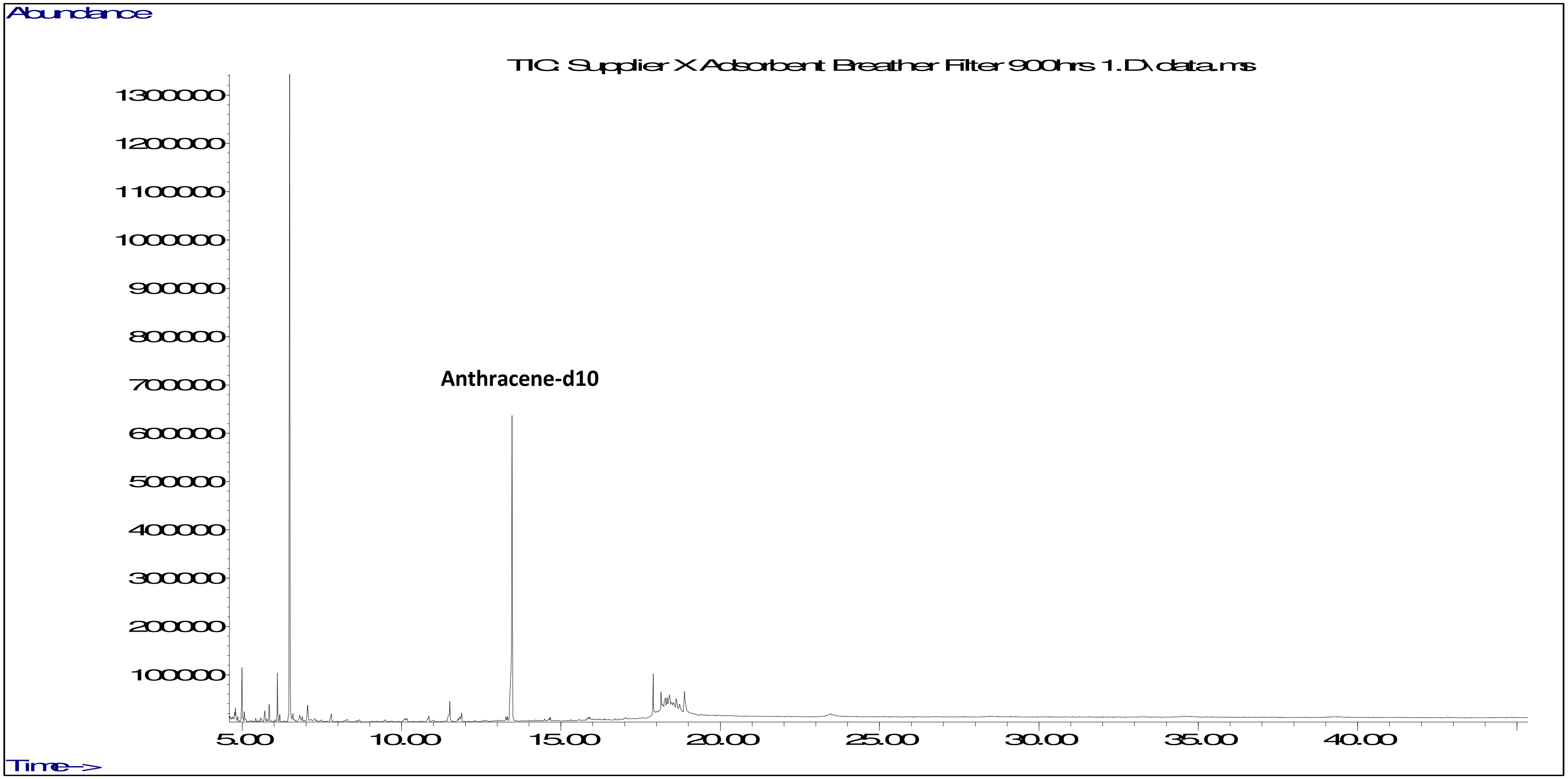
Appendix 7: Gas chromatogram for recirculation filter



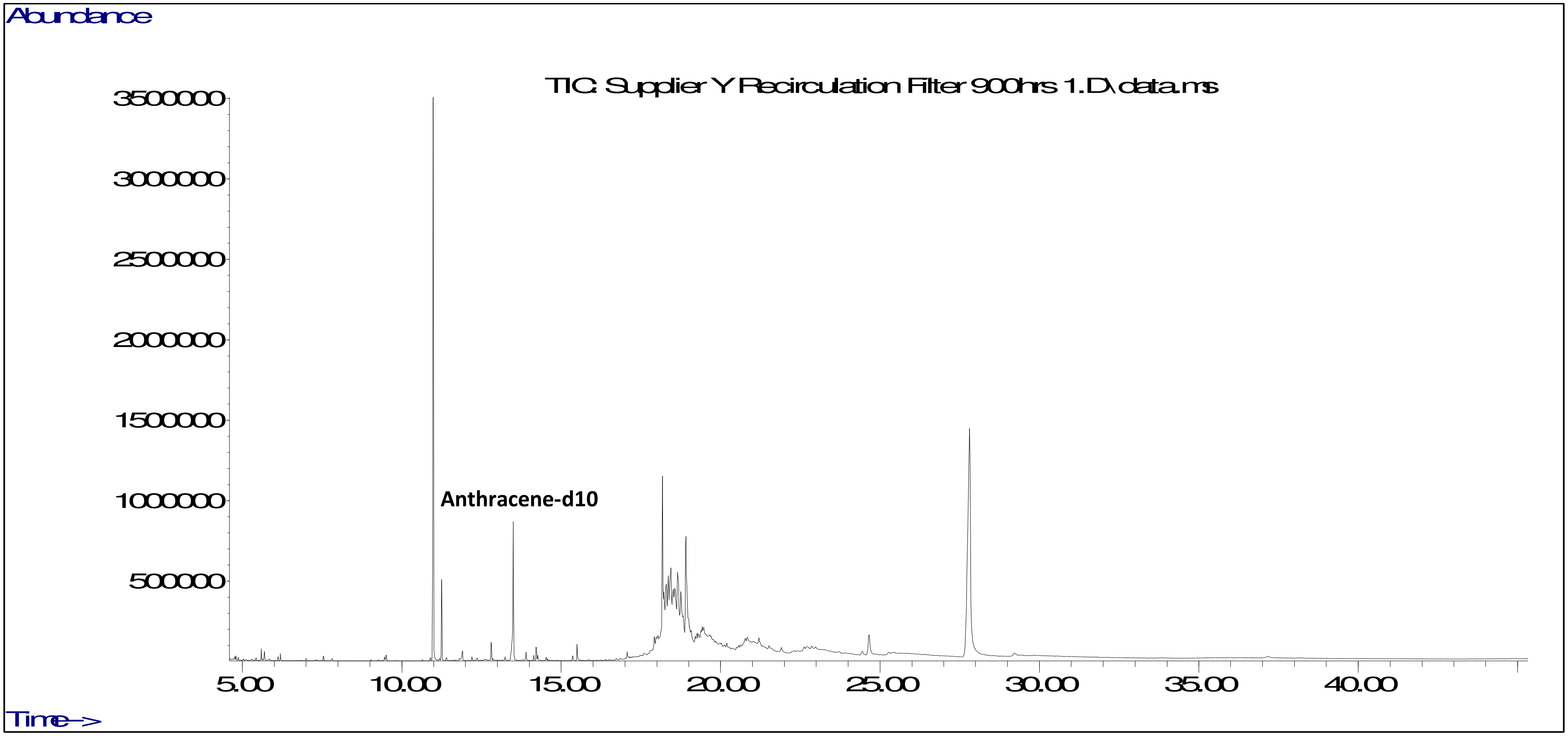
Appendix 8: Gas chromatogram for adsorbent breather filter



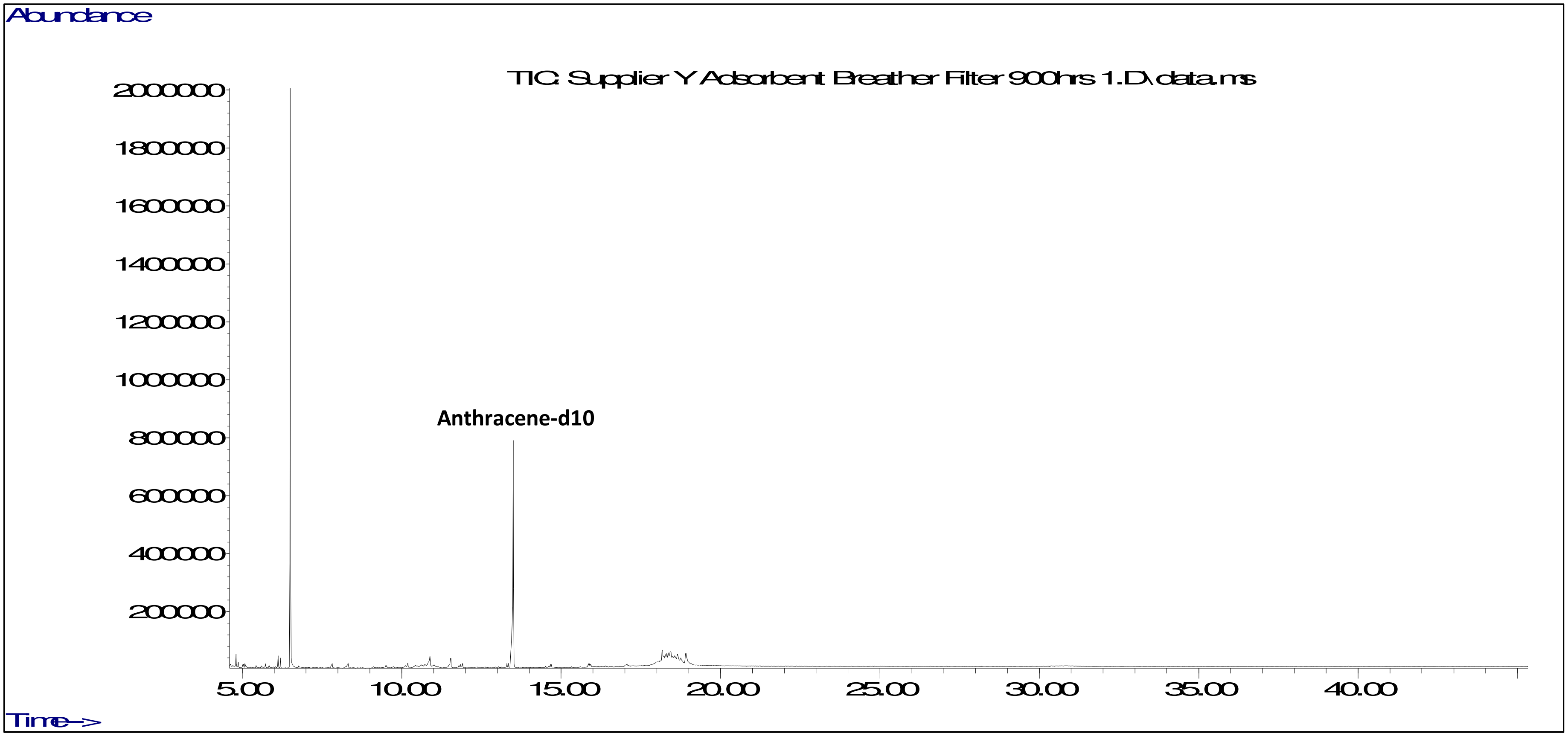
Appendix 9: Gas chromatogram of recirculation filter extraction for test time 900 hours for motor from Supplier A and pivot from Supplier X



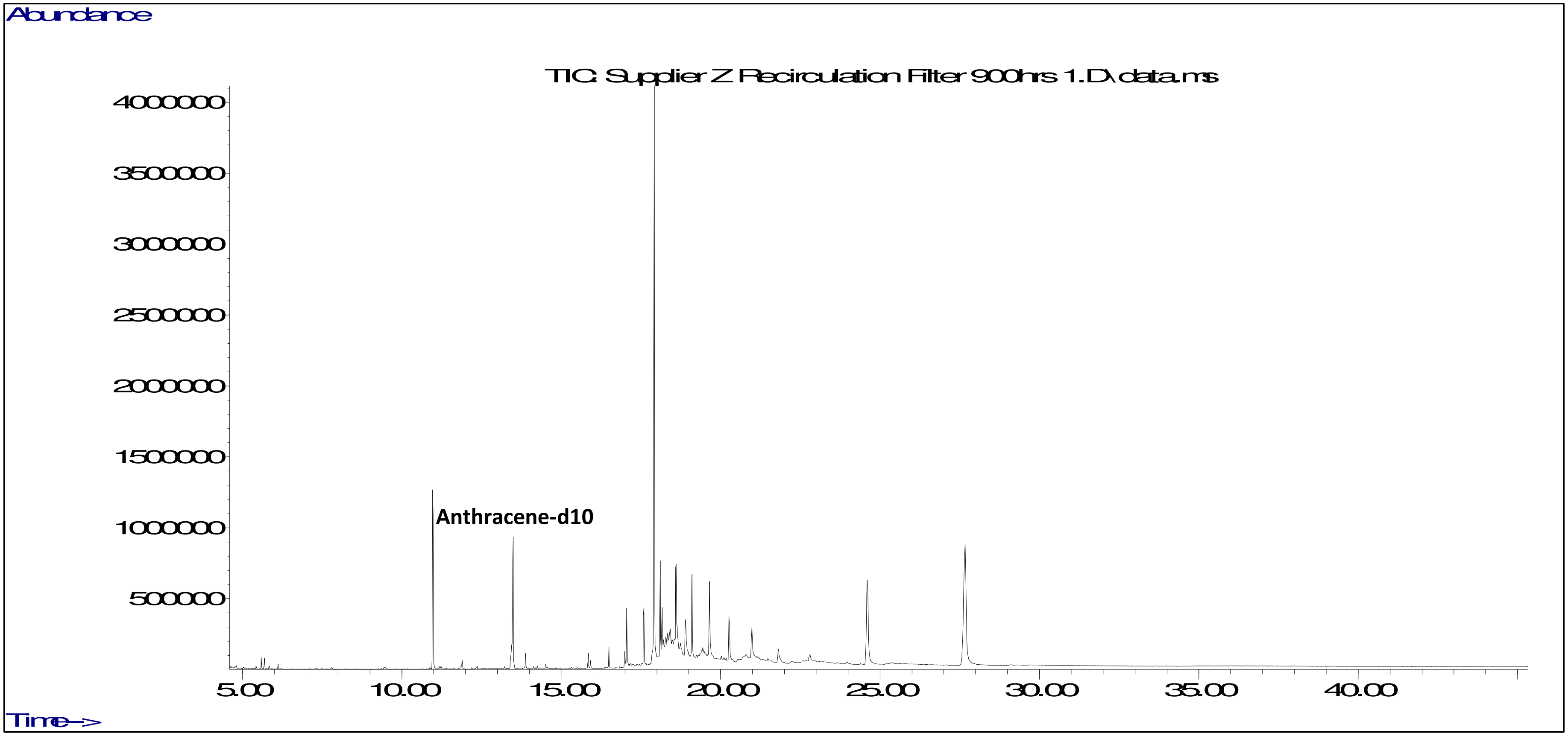
Appendix 10: Gas chromatogram of adsorbent breather filter extraction for test time 900 hours for motor from Supplier A and pivot from Supplier X



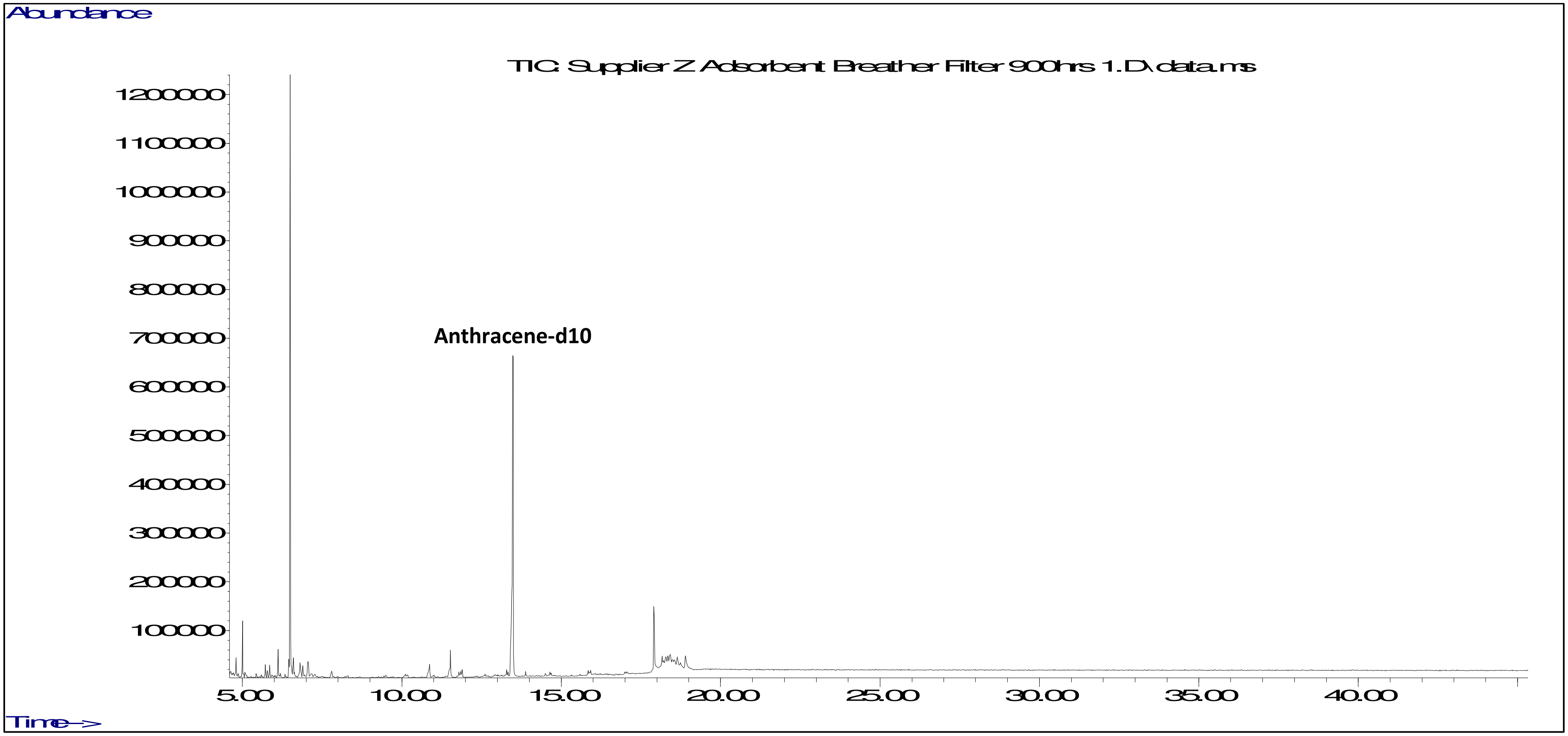
Appendix 11: Gas chromatogram of recirculation filter extraction for test time 900 hours for motor from Supplier A and pivot from Supplier Y



Appendix 12: Gas chromatogram of adsorbent breather filter extraction for test time 900 hours for motor from Supplier A and pivot from Supplier Y



Appendix 13: Gas chromatogram of recirculation filter extraction for test time 900 hours for motor from Supplier A and pivot from Supplier Z



Appendix 14: Gas chromatogram of adsorbent breather filter extraction for test time 900 hours for motor from Supplier A and pivot from Supplier Z