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**SUBSTRATE EFFECT ON VOC EMISSION CHARACTERISTICS
FROM CARPET ADHESIVE ASSEMBLIES
– AN EXPERIMENTAL INVESTIGATION**

by

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**A thesis submitted to
the Faculty of Graduate Study and Research
in partial fulfilment of the requirements for the
Degree of Master of Engineering**

**Department of Mechanical and Aerospace Engineering
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Ottawa, Canada

June, 1997

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FROM CARPET ADHESIVE
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Submitted by

Xiangdong Li

in partial fulfilment of the requirements for
the degree of Master of Engineering

Dr. R. Bell, Chair, Department of Mechanical
and Aerospace Engineering

Dr. E. G. Plett, Thesis Supervisor

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June, 1997

Abstract

A comprehensive study of the emission behavior of VOCs from carpet adhesive during its curing process was conducted at NRC. Two kinds of substrate and three kinds of adhesive with different solvent contents were tested to investigate the effect of substrate on the VOC emissions from the adhesives. The adhesive application methods were evaluated and a container adhesive application method was developed that provided a better control of the amount of the adhesive applied than the traditional trowel method. The effect of the adhesive amount applied was also studied based on the container adhesive application method. All the tests were conducted in a conditioned well-controlled small scale test chamber. An electronic balance along with conventional VOC measurement techniques was used during the tests. Both VOC weight loss data and concentration decay data were obtained through corresponding data acquisition systems. Thermal adsorbent tubes were used to collect air samples from the test chamber and then the GC/FID/MS method was used to identify the volatile compounds in the test sample. A double exponential model was used to analyze the test result.

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Nomenclature

A'	surface area of one adhesive stripes for samples prepared by the trowel method, m^2
A_e	effective adhesive emitting area, m^2
A_s	area of the emission surface, m^2
ACH	air change per hour
C	concentration of VOC inside the test chamber, mg/m^3
C_s	concentration of the VOC concentration at the surface, mg/m^3
$C_s(0)$	the initial VOC concentration at the surface, mg/m^3
D	dimension of the trowel notch, inch
$E(t)$	emission rate, mg/h
FID	flame ionization detector
GC	gas chromatograph
GC/MS	gas chromatograph / mass spectrometry
H	height of adhesive stripes for samples prepared by the trowel method, m
H_x	GC peak height of one component
K	interfacial mass transfer coefficient, m/h
k_1	first order decay rate constant for the fast decaying fraction, h^{-1}
k_2	first order decay rate constant for the slowly decaying fraction, h^{-1}
k_s	decay constant for the concentration at the material surface
L	chamber loading, m^2/m^3 , $L=A_s/V$
L_s	length of the substrate, m
N	number of air changes per hour, ACH, $N=Q/V$

N_{eff}	GC column efficiency
N_s	number of adhesive stripes on the substrate for samples prepared by the trowel method
Q	supply air flow rate, m^3/h
R	emission rate per unit area of the emission surface, mg/m^2h
R_{GC}	GC resolution
R_o	hot wire probe resistance at 0 °C
R_{100}	hot wire probe resistance at 100 °C
R_{op}	operating resistance of hot wire probe
RT_a	retention time of inert gas
RT_x	retention time of one component in GC column
RT_x'	time difference between the retention time of one component and the RT_a
THC	total hydrocarbon
T_{int}	turbulence intensity, %
t	time, h
t_1	end of the fast decay period
t_2	beginning of slow decay period
T_{op}	hot wire probe operating temperature
V	net air volume inside the outer test chamber, m^3
V_a	initial volume of adhesive applied, m^3
V_a'	volume of one stripe of the adhesive for samples prepared by the trowel method
VOC	volatile organic compound

W	weight of adhesive, g
$W_{1/2}$	GC peak width of one component at its half height
W_0	initial adhesive application amount on the substrate, g
W_{24}	adhesive weight remained on the substrate after 24 hours of application, g
W_{32}	adhesive weight remained on the substrate after 32 hours of application, g
ΔW_{24}	adhesive weight loss after 24 hours of application, g
ΔW_{32}	adhesive weight loss after 32 hours of application, g
ΔW_{t1}	adhesive weight loss after time t_1 , g
ΔW_{t2}	adhesive weight loss after time t_2 , g
α	GC separation factor
ρ	density of the adhesive, g/m ³

Chapter 1

Introduction

1.1 Objectives and Significance

A recent survey indicates that people spend 80% of their time indoors. The indoor air quality (IAQ) appears very important to people's health. "Sick building syndrome", a group of symptoms including sleepiness, irritability and inability to concentrate, has occurred with increasing frequency among office workers in new energy efficient buildings in recent years. Relevant research has discovered some volatile organic compounds (VOCs) in new buildings which contribute to the "sick building syndrome". Those VOCs are emitted from many sources, such as paint, carpet, adhesive, furniture and office machines (Mohave, 1982; Girman *et al.*, 1984). The VOC concentration is usually very low inside the buildings. However, the risk to human health is not as low as expected. The risk of one volatile organic compound to human health is proportional to its hazard and exposure. The hazard of different compounds are different, such as benzene may cause cancers and methyl alcohol will cause liver disease. The

concentration of VOC vapor in air is the exposure when people are exposed. It is believed that the sensory reaction of the human body to odorous substances in our surroundings in many cases is proportional to the logarithm of the exposure to the specific irritant (Berglund *et al.*, 1985).

It is essential to quantify the emission characteristics of volatile organic compounds (VOCs) from commonly used building materials and products in order to predict and control the contaminant levels in buildings. The VOC emission behavior is very complicated and is subject to many factors. Air velocities and turbulence levels (Low, 1996; Zhang and Haghighat, 1995), humidity levels (Sollinger and Levsen, 1994), temperatures (Sollinger and Levsen, 1994), and overall ventilation conditions (Sollinger and Levsen, 1994; Tichenor and Mason, 1988) have been studied to discover their effects on the VOC emission characteristic from building materials. Some mass transfer models have been developed to predict VOC concentration levels in buildings (Guo *et al.*, 1996; Zhang *et al.*, 1996; Christianson, 1993; Hanna *et al.*, 1993; Tichenor *et al.*, 1993; Clausen *et al.*, 1993; Colombo *et al.*, 1990). However, to date, no experimental method or analog model can predict or fit completely all the real situations. Efforts are still needed to find the emission mechanisms.

Since there are a lot of possible factors affecting the VOC emission, such as temperature, humidity, air exchange rate and turbulence conditions, it is important to consider all the possible factors and to include all the possible contamination sources and their interactions in a practical model. However, the emission mechanism is still not fully understood and the complexity of each model is limited by the knowledge of the emission mechanism and the capacity of computing facilities. Thus each emission source has to be

treated individually and assumptions are needed to simplify each model. This causes discrepancies between the models and reality.

A carpet and adhesive assembly is an important contaminant source in buildings. Some chemical compounds emitted from carpet and adhesives are irritants to people. Rothweiler *et al.* (1992) has found that the VOCs from the carpet and adhesive would last for a very long period. People are conventionally thinking that the major source of contamination comes from carpet itself. However, recent research (Black *et al.*, 1990; Mohave, 1982) showed that most of the VOCs are emitted from adhesive instead of the carpet. It is of great interest to find the adhesive emission behavior.

The adhesive emission rate is affected by both the adhesive composition, such as the VOC content, the moisture content, as well as the age of the adhesives, and the ambient conditions, such as the ambient temperature, humidity, velocity and air exchange rate. The effects of temperature, humidity and velocity have been studied by researchers. However, no attention was observed paid to the effect of substrate (contact surface of adhesive) on VOC emission behavior in the literature. In practical carpet installation processes, some VOC contents in adhesives may penetrate into the substrate materials, such as wood and concrete, and are kept inside the substrate materials for quite a long time. Thus the general VOC emission rates are also affected by the adsorption and desorption rates of VOC components in substrate materials. This kind of effect needs to be studied to provide more realistic experimental data for mathematical models.

An interesting phenomenon is observed that the test results always vary from laboratory to laboratory and even from test to test in the same laboratory. It is observed that the

preparation method of a test sample contributes its role to the test repeatability. In the carpet-adhesive assembly study, the adhesive application method was found affecting not only the amount of the adhesive applied, but also the contact surface area of the adhesive to the ambient air. The contact surface area of the adhesive will change the loading factor inside the test chamber which is quite important to the VOC emission rate as will be discussed in the literature review. The amount of adhesive applied was also found to severely affect the test repeatability in this study. It is critical to develop a standardized application method to achieve reasonable test repeatability and reproducibility.

The objectives of this thesis work are:

- (1) Investigate the effects of substrate on VOC emission behavior in a carpet/adhesive assembly
- (2) Evaluate and develop adhesive application methods to provide good test repeatability
- (3) Investigate the effect of the amount of the adhesive applied on VOC emissions
- (4) Provide data for further modeling work.

1.2 Literature Review

1.2.1 Adhesive Study

Previous studies have indicated the potential of building materials to contribute to poor indoor air quality by the emission of volatile organic compounds (Miksch *et al.*, 1982; Mohave, 1982). Molhave (1982) tested 42 building materials and identified adhesives as a particularly important source of VOC in indoor air. The highest concentration of VOCs

from the 42 materials was obtained from adhesives. However, no continuous concentration changing with time was monitored.

Some comprehensive adhesive tests were conducted by Girman *et al.* in 1986. 15 adhesives were selected for study based upon a broad range of application and were formulated for bonding of carpet, vinyl floors and sub-floor assemblies. In the first phase of their study, vacuum extraction with cryogenic trapping of volatile compounds was used as a screening procedure for the qualitative and semi-quantitative determination of emissions of VOC from adhesives. In the second phase of their study, a simple exposure apparatus was used to determine emission rates of VOC from adhesives. The study by Girman *et al.* (1986) confirmed that adhesives contribute to poor indoor air quality. Girman *et al.* (1986) also realized that their study only represented an initial probe of adhesive emissions and did not address the potentially important effects of temperature and aging on emission. Their study was just to provide data for architects and builders to form an objective basis for the selection and safe utilization of architectural materials in new or remodeled office buildings. Their study also did not provide sufficient data to predict the concentration level in buildings. Another drawback of their study is that they let test specimens be exposed to room conditions for at least 7 days and then started the measurement. Some important information may be lost during this period.

Black *et al.* (1990) tested four kinds of adhesives and SBR latex-backed carpet. One of adhesives was synthetic "low VOC" adhesive. The other three multipurpose adhesives displayed similar TVOC decay patterns with time whereas the "low VOC" adhesive showed considerably lower TVOC than the other three adhesives. Unfortunately, Black *et al.* did not identify individual compounds inside each adhesive. Some dangerous

compounds may appear in the “low VOC” adhesive. The carpet and adhesive study showed that VOC contribution was definitely dominated by the adhesive. This conclusion supports previous researches. One strategy that Black recommended to control indoor pollutant exposure is to use low-emitting materials while simultaneously providing adequate ventilation to the indoor space.

A carpet and adhesive assembly was studied to find the concentration distribution during carpet installation (Davidson *et al.*, 1991). Test data of concentration versus time was measured. A small environmental chamber was used during the study. Four kinds of carpet and one kind of “low VOC” adhesive were tested. Concentration peaks occurred at 2.4 exposure hours. An interesting phenomenon was observed that some compounds that appear in the individual adhesive test did not appear in the carpet/adhesive assembly tests. It was also found that the adhesive appeared to cure more rapidly ($k=0.042$) when it was evaluated independently than when it was a component in the carpet assembly ($k=0.0086$). These observations may indicate that the carpet was behaving as a resistor for the adhesive VOC emission and thus delayed the emission process. Davidson *et al.* (1991) also compared the emission rate of the adhesive, the carpet and the carpet-adhesive assembly. They found that the emission rate of the adhesive was the highest while the emission rate of the carpet was the lowest.

All studies mentioned above emphasized the identifications of chemical compounds emitted from building materials. Few efforts were put to predict long or short-term concentration variation associated with the ambient conditions. Especially when adhesives were tested, no attention was paid to the effect of substrate on the emission rate. As the carpet will play a role as a resistor above the adhesive, the substrate will act

as an absorber underneath the adhesive. It is very necessary to discover how much the substrate will affect the VOC emission rate from the adhesive.

1.2.2 Testing Methodology

1.2.2.1 Test Facilities

The facility used to test VOC emissions from building materials is the small-scale environmental test chamber at the NRC. With this facility, the test conditions can be controlled precisely so it can simulate quite accurately real indoor conditions thus allowing estimation of the contribution by a given product to the overall emission rate or how a given condition will influence the overall indoor concentration. A GC/MS method was used to identify each individual compound in the test samples and a GC/FID method was used to quantify those compounds. Thermal desorption tubes were used to collect gas samples. Table 1.1 shows a list the typical facilities used by others as reported in the literature.

Table 1.1 Typical Test Facilities Used in Literature

Author	Year	GC/FID	GC/MS	Thermal Desorp. Tubes	Chamber	Volume of Chamber
Mohave	1982	No	Yes	Yes	Yes	1000 l
Girman <i>et al.</i>	1986	Yes	Yes	No	Yes	3.8 l
Tichenor <i>et al.</i>	1988	Yes	Yes	N/A	Yes	166 l
Davidson <i>et al.</i>	1991	N/A	Yes	No	Yes	N/A
Black <i>et al.</i>	1991	N/A	Yes	No	Yes	N/A
Black <i>et al.</i>	1993	N/A	N/A	N/A	Yes	50 l
Nagda <i>et al.</i>	1993	Yes	N/A	N/A	Yes	53 l
Guo <i>et al.</i>	1996	N/A	N/A	N/A	Yes	53 l
Low	1996	Yes	Yes	Yes	Yes	400 l
Zhang <i>et al.</i>	1996	Yes	Yes	Yes	Yes	400 l

1.2.2.2 Chamber Test Conditions

For studies of the type under consideration here, test conditions are very critical to test results, with different test conditions leading to different test results. Temperature, relative humidity, air exchange rate, loading factor, air velocity and turbulence intensity are the most important factors which affect VOC emissions from carpet adhesive assemblies. Table 1.2 shows some typical test condition settings reported in the literature. The range of conditions used may lead to differences among results even if the same material is tested. Researchers are trying to develop acceptable standardized test conditions to achieve reasonable test repeatability and reproducibility. At the same time, researchers are trying to alter each individual condition to see how the change will contribute its effect to the general VOC emission behavior.

Table 1.2 Typical Test Conditions for Small Test Chambers

Air Exchange Rate (ACH)	Temperature (°C)	Relative Humidity (%)	Author	Year
0.04	21.1	35 – 40	Mohave	1982
14	23 – 25	40	Girman <i>et al.</i>	1986
0.54 – 3.61	25	50	Tichenor <i>et al.</i>	1988
1.0 ± 0.05	25 ± 0.2	50 ± 2	Davidson <i>et al.</i>	1991
1 ± 0.05	25 ± 2	50 ± 2	Black <i>et al.</i>	1991
1 ± 0.05	23 ± 1.0	50 ± 5	EPA	1991
1 ± 0.05	23 ± 1	50 ± 5	Black <i>et al.</i>	1993
1.0	25	50	Nagda <i>et al.</i>	1993
0.98 – 1.0	22.8 – 23.5	46.5 – 50.2	Little <i>et al.</i>	1994
0.5	23	50	Guo <i>et al.</i>	1996
1 ± 0.05	23 ± 1	50 ± 5	Low	1996
0 – 3	15 – 35	20 – 80	Zhang <i>et al.</i>	1996

1.2.2.1 Air Exchange Rate

Tichenor and Mason (1988) conducted an experiment to develop a correlation between the VOC emission rates and air exchange rate. They used a 166 l chamber and tested with air change rates of 0.36 and 1.84. They found that it was helpful to increase the air change rate to reduce the VOC concentration level. However, the measured concentration was not inversely proportional to the air change rate as expected. The increased air exchange rate increased the concentration gradient between the VOC source and air, thus leading to a higher mass transfer rate. It can be concluded that it is helpful to increase the air exchange rate at the beginning period of the source material installation so the VOC concentration in the source material can be rapidly reduced. Figure 1.1 shows one of their test results. Sollinger and Levsen (1994) conducted another carpet test with air exchanger rate to be 0.5, 0.9 and 1.9. Sollinger and Levsen's result supported the conclusion of Tichenor and Mason.

Figure 1.1 Effect of Air Exchange Rate on VOC emission

(Reproduced from Tichenor and Mason 1988)

1.2.2.2 Temperature Effect

Sollinger and Levsen (1994) used a small test chamber (1.0 m³ stainless steel) to check the temperature effect on the VOC emission rate from textile floor coverings. They concluded that temperature had a large impact on the concentrations as can be seen from figure 1.2. Especially in the high temperature range, concentration and VOC emission rate vary faster than in the low temperature range. However, in most of the office buildings where temperature does not vary as much as relative humidity does, the effect of temperature may not be significant.

Figure 1.2 Temperature Dependence of the Emission
(Reproduced from Sollinger and Levsen 1994)

1.2.2.3 Humidity and Loading Factor

The humidity effect on VOC emission is not very significant, although some compounds are strongly dependent on humidity under steady conditions according to Sollinger and Levsen (1994). Recent research at National Research Council demonstrated that relative

humidity significantly affected the “wet” materials, such as paint, varnish and adhesive, especially when these “wet” materials are applied to absorbent substrate, such as wood and concrete. Relative humidity control is critical to test repeatability. The effect of humidity on VOC emission lies in two respects: first, affecting the formation of “dry skin” on the surface of “wet” material; second, changing the moisture content inside the substrate material. Further effort needs to be applied to quantify the effect of humidity on VOC emissions. Chamber loading can affect the value of concentration peak dramatically as can be seen in figure 1.3. It is always a good approach to reduce the loading factor in order to reduce the concentration level. However, in practical applications where loading factors are normally constant, the dependence of concentration on loading factor is not a major consideration.

Figure 1.3 Time dependent concentration of the 2,2,6,6-tetramethyl-4-methylideneheptane for two different chamber loadings (1 m^3 test chamber) (reproduced from Sollinger *et al.*, 1993)

1.2.2.4 Air Velocity and Turbulence Intensity

Zhang and Shaw *et al.* (1996) studied the VOC emissions from a wood stain using a small test chamber under two different air velocity and turbulence intensity conditions. They concluded that the mass transfer coefficient and hence the emission rate increased with the air velocity over the material surface (see figure 1.4). The turbulence intensity will increase the emission rate in a initial certain period after sample application but this influence on emission rate will decrease with time. In the test of Zhang and Shaw (1996), because the two turbulence level were both very low and quite close, the effect of turbulence was not obvious. The difference between the emission rates was mainly attributed to the difference of the air velocities.

Figure 1.4 TVOC emission Rate at Two Different Velocities U and Turbulence Intensities T_{int}

(Reproduced from Zhang and Shaw *et al.*, 1996)

1.2.3 Sink Effect

Adsorption of the test substances by surfaces such as walls, known as the sink effect, is often negligible when using Electro-polished stainless steel small-scale or large-scale environmental chambers even though the concentration level of the test substance is relative high. However, when the VOC concentration becomes low as in the later test

stages, the sink effect may appear more important. This is especially so when experimental data are being extracted to predict practical situations where sink effects are normally much stronger than that in the laboratory. Because of the sink effect, the measured concentration level in the early test stage will be lower than it would be in the absence of sink effects, and in the later test stages it is expected to be higher than it would be otherwise. Sink effect also contributes to extending the time over which VOC emissions occur.

1.2.4 Modeling

Table 1.3 Selected Models for VOC Emissions

Name	Expression	Type	Mainly Used For	Ref.
First-Order Model	$R = M_0 k e^{-kt}$	Empirical	Evaporation	Clausen (93)
Second-Order Model	$R = R_0 / [1 + (k/\lambda)tR_0]$	Empirical	Both Mechanisms	Clausen <i>et al.</i> (93)
Double-Exp. Model	$R = R_1 e^{-k_1 t} + R_2 e^{-k_2 t}$	Empirical	Both Mechanisms	Colombo <i>et al.</i> (90)
VB Model	$R = k_m (C_v M / M_0 - C)$	Mass Transfer	Evaporation	Tichenor <i>et al.</i> (93)
Diffusion Model	$R = 0.632 / \lambda M_D (D/t)^{1/2}$	Mass Transfer	Source Diffusion	Hanna <i>et al.</i> (93)
Diffusion Model	$R = \pi^{-1/2} / \lambda M_{D0} (D/t)^{1/2}$	Mass Transfer	Source Diffusion	Christianson (93)
IRC Model	$R(t) = KC_s(0) [(N - ks) * e^{-ks} + LK e^{-(LK + N)k}] / (LK + N - ks)$	Mass Transfer	Both	Zhang <i>et al.</i> (96)
Diffusion Model	$R = M_v k \exp(-kt) + \alpha f_D M_{D0} \exp(-2f_D t^{1/2}) / t^{1/2}$	Mass Transfer	Source Diffusion	Guo <i>et al.</i> (96)

This study is part of the indoor air quality research project at NRC. One of the final goal of the research project at NRC is to find a suitable model that can predict the concentration variation in buildings so the contaminant level can be controlled effectively. Because the emission rate of VOCs from carpet and other building material is

associated with both heat and mass transfer and affected by a lot of unknown factors, a commonly accepted model is still not available. The present models are all based on local conditions while some assumptions and simplifications are necessary. Little et al. (1993) started from a simple one dimensional diffusion equation and assumed that the emissions of VOCs from new carpets originated predominately in a $\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial x^2}$ uniform slab of polymer backing material. Guo and Tichenor (1992) used a vapor pressure and boundary layer (VB) model to predict organic emission rates from oil-based indoor architectural coatings. An assumption they made was that the vapor pressure during the aging period is proportional to the mass of VOC left. They claimed this model provides a better fit to chamber-derived emission data than the empirical first-order model. However, their assumption needs to be validated. Zhang (1996) et al. developed an IRC model basing on an assumption that the gas concentration at the emission surface can be described by the first order decay equation, that is $C_s(t) = C_s(0)e^{-k_s t}$. The advantage of the IRC model is that it can be used to derive k_s (decay constant) and K (interfacial mass transfer coefficient) directly from test data without mathematical regression and the model prediction fit the test data reasonably well. Table 1.3 shows a list of some selected models.

1.2.5 Conclusion based on review of the literature

From the literature it can be seen that there are two kinds of possible strategies to control the VOC concentration level in buildings. The first is to develop low VOC emitting material; the second is to control the environmental conditions to minimize VOC production. The first strategy is to be preferred. Some so-called “environmental friendly” or “VOC free” materials can already be found on the market. However those materials

are still too expensive compared to normal building materials to be widely used in industry. It has also been observed that although the total hydrocarbon level is low in the emission from those materials, some dangerous compounds were still found. It is still a big challenge for researchers and manufacturers to produce safer and cheaper building materials. The second strategy is more passive, but it is still an effective way to reduce the VOC concentration levels in buildings.

A carpet and adhesive assembly is an important VOC contamination source. It is found that the effect of substrate material on VOC emission from carpet and adhesive assemblies was ignored by previous the researchers when they did the studies on carpet and adhesive. As will be discussed in this thesis, the substrate will have the effect of absorbing VOC contents in the initial adhesive application period and thus delay the total VOC emission rate. As a result, long term emissions are always expected.

It is also observed that the difference caused by different adhesive application methods was also ignored by previous researchers. Different application methods lead to different test conditions being used and thus poor test repeatability. This thesis work will concentrate on the effect of substrate material on the emission rate from carpet adhesives and try to develop a standardized test method for adhesive application. This thesis work is also going to investigate the effect on VOC emissions caused by the amount of the adhesive applied and to compare the behavior among different adhesives.

1.3 Basic Mass Transfer Theory

Assuming the mixing inside the test chamber is complete, then the mass balance of the VOC component within the test chamber can be described as:

$$V \frac{dC(t)}{dt} = AR(t) - QC(t) \quad (1)$$

Based on the interfacial mass transfer theory, the emission rate can be modeled as:

$$R(t) = K[C_s(t) - C(t)] \quad (2)$$

Assuming the gas concentration at the emission surface follows a first order decay, then

$$C_s(t) = C_s(0)e^{-k_s t} \quad (3)$$

Substituting equation (2) and (3) into equation (1) and rearranging results in

$$\frac{dC(t)}{dt} + (LK + N)C(t) = LKC_s(0)e^{-k_s t} \quad (4)$$

Equation (4) is a linear, non-homogeneous differential equation. If at time zero, the concentration inside the test chamber is zero, the solution to equation (4) becomes

$$C(t) = \frac{LKC_s(0)}{LK + N - k_s} (e^{-k_s t} - e^{-(LK + N)t}) \quad (5)$$

with the following substitution,

$$a = \frac{LKC_s(0)}{LK + N - k_s} \quad (6)$$

$$b = k_s \quad (7)$$

and

$$c = LK + N \quad (8)$$

equation (5) may be written as

$$C(t) = a(e^{-bt} - e^{-ct}) \quad (9)$$

Then using a nonlinear regression technique, the coefficients a, b and c can be determined from the measured data of concentration as a function of time C(t). The three unknowns $C_s(0)$, k_s and K, can be calculated as:

$$C_s(0) = \frac{a(c - b)}{c - N} \quad (10)$$

$$k_s = b \quad (11)$$

$$K = \frac{c - N}{L} \quad (12)$$

These can then be substituted into equation (5) and (2) to permit the emission rate to be calculated as:

$$R(t) = \frac{KC_s(0)}{LK + N - k_s} [(N - k_s)e^{-k_s t} + LKe^{-(LK + N)t}] \quad (13)$$

Chapter 2

Experimental Design

This chapter describes the design of the experiments performed in this study. The test design includes the setup of the test facilities, the choosing of the test conditions, the choosing of the test materials and associated test plan.

2.1 Choosing of Substrate and Adhesive

The effect of the substrate on VOC emissions from the carpet adhesive assembly was to be studied. To achieve this goal, two kinds of substrate and three kinds of adhesive were chosen. The substrates were concrete and glass, designated substrates I and II. The reason to choose concrete substrate was that concrete is most commonly used as a floor material in large buildings. The reason to choose glass substrate was that glass can be considered as a kind of non-absorbent material and this characteristic can be used to find out the effect of concrete substrate on VOC emission behavior. To make the test result more comprehensive, three different solvent content adhesives were chosen. They were Roberts 900, Roberts 3850 and ECO 900, designated adhesives I, II, III. Adhesive I and

source to the indoor air quality. The quantities of those solvents decide the VOC contaminant levels in buildings. To make the test results more comprehensive, three adhesives with different solvent contents were chosen. They were Roberts 900, Roberts 3850 and ECO 900, designated adhesives I, II, III. Adhesive I and II are synthetic latex based adhesives made by the same manufacturer. Adhesive III is a so-called “environmentally friendly” solvent-free adhesive from another manufacturer. Adhesive I consists of 3% solvent content. Adhesive II consists of 5-10% solvent content.

A stainless steel substrate (substrate III) was also used to develop a better adhesive application method. Based on this method, the amount of adhesive applied can be controlled at three levels: low, medium and high. The effect of the amount of the adhesive applied was checked based on the stainless steel substrate.

2.2 Test Plan

All tests were designed to be done in a small-scale stainless steel environmental test chamber. This chamber was designed to provide a well-controlled test environment. A schematic diagram of the testing system setup is shown in figure 2.1. A detailed description of each piece of equipment is found in chapter 3. Temperature, humidity and airflow rate could be controlled accurately in the test chamber. The electro-polished stainless steel chamber wall would minimize the possible sink effects provided by containing walls. An electronic balance (maximum range 600 g and accuracy ± 1 mg) was chosen to measure the weight loss versus time of VOC content from the adhesive. The VOC content emitted by each test specimen was analyzed by a Meloy hydrocarbon analyzer. Multi-adsorbent tubes were used to take a gas sample from test chamber and the gas sample was analyzed by a GC (gas chromatograph) to identify chemical components

Figure 2.1 Schematic of System Setup

in the vapors emitted by the adhesive.

Two different substrates were tested using adhesive I, II & III to find the effect of substrate on the VOC emissions. Adhesive I was also used on the substrate III to test the effect of the amount of the adhesive applied on VOC emissions. Three adhesives were tested on the substrate II to comprehend the test result. Each test was followed by repeat tests to verify the test consistency. The test plan used is shown in table 2.1.

Table 2.1 Test Plan

Test Group	Substrate	Adhesive	Remarks
1	I	I, II	Initial Tests
	II		
2	I	I, II	Repeat Tests for Group 1
	II		
3	I	III	Enhanced Adhesive Tests
	II		
4	III	I	Method Development
5	III	I	Effect of adhesive amount

* Adhesive I — Roberts 900

Adhesive II — Roberts 3850

Adhesive III — ECO 300

Substrate I — Concrete

Substrate II — Glass

Substrate III—Stainless Steel

2.3 Test Conditions

All experiments were done in a well-controlled environmental chamber which has the dimensions 1.0m × 0.8m × 0.5m. Table 2.2 shows the test conditions inside the test chamber. The 23 °C temperature, 50% relative humidity and 1ACH air change rate are

widely accepted as the standard test conditions for the tests in the small scale test chamber. The air change rate is the ratio of the volume of air brought into the chamber hourly to the chamber volume measured in identical units (typically expressed in air changes pre hour — ACH). The temperature in the test chamber was controlled by the environmental controller, air conditioning system and heating unit. The relative humidity and airflow rate were controlled by the environmental controller. The velocity over the specimen surface was controlled by the mixing fan power supply unit. The pressure inside the test chamber was controlled by an exhaust pump and a compressor.

Table 2.2 Test Condition inside the Test Chamber

Temperature	$23\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$
Relative Humidity	$50\% \pm 5\%$
Relative Pressure	$10\text{ Pa} \pm 5\text{ Pa}$
Air Exchange Rate	$1\text{ ACH} \pm 0.05\text{ ACH}$
Air Velocity	$0.05 \pm 0.01\text{ m/s}$
Turbulence Intensity	1.24%

Chapter 3

Experimental Facilities

This chapter introduces the facilities that were used in the experiments. Those facilities include the test chamber and associated controlling system, measuring devices, data acquisition devices, chemical analysis devices, air and water supply system, sample taking devices and other accessories. Each piece of equipment is introduced and a detailed equipment list is given in appendix D.

3.1 Test Chamber

A small-scale environmental test chamber (1m×0.8m×0.5m) was used for the testing work. It is made of stainless steel following ASTM's Small-Scale Chamber Guidelines to minimize the possible sink effect. The small-scale chamber consists of an inner chamber and an outer chamber. The schematic diagram of the small chamber is shown in Figure 3.1. The top plate and a glass window at the front panel of the outer chamber can be opened to load the specimens. The glass window is convenient for loading small specimen when no change to the inner chamber setup is needed. To load large specimens or to remove the inner chamber, the top plate must be opened. The top plate and the front window are fastened to the body of the small chamber by screws. In order to prevent

possible leakage, a Teflon gasket is used to seal the gap between the top plate and chamber body and between the glass window and chamber body.

The inner chamber is placed in the middle of the outer chamber supported by two legs. The inner chamber allows air to flow over the specimen at a constant speed. The speed of air velocity is controlled by the speed of the mixing fan. The turbulence level over the test specimen can be altered by changing the end screen of the inner chamber.

Figure 3.1 Photograph of Small Scale Test Chamber

3.2 Test Room

The small test chamber was placed in the center of a well-controlled test room. The temperature was controlled within $\pm 1^\circ\text{C}$ and the relative humidity was controlled within $\pm 5\%$. The temperature inside is controlled both by a Nortec air conditioning system and a heating system. The humidity is controlled by a humidifier.

3.3 Measuring System

3.3.1 Electronic Balance

In order to measure the weight loss of adhesives accurately, a Sartorius electronic balance is used. The balance has a resolution of ± 1 mg. The power is provided by a 9V DC adapter. The balance has a Liquid Crystal Display (LCD) at the front and also has a communication port at its back. This communication port allows the balance to transfer the weight signal to a computer. A computer using an Intel 286 chip is used to collect the signal from the electronic balance. The data of weight versus time is saved as files. Figure 3.2 shows a photograph of the balance and the position of the balance in the small-scale test chamber.

Figure 3.2 Position of Balance in Test Chamber

3.3.2 Hydro-carbon Analyzer

Figure 3.3 Photograph of Data Acquisition System

When the exhaust air flows out of the test chamber, a part of it would be sucked into a Meloy HC500/2C hydrocarbon analyzer. A photograph of the Meloy HC500/2C is shown in Figure 3.3. This hydrocarbon analyzer analyzes the total hydrocarbon in the exhaust gas sample. In other words, it measures the concentration in the small chamber. The analyzer has a FID (flamed ionization detector) which burns the hydrogen. A purified hydrogen cylinder is therefore necessary. The analyzer has five ranges: 10 ppm, 50 ppm, 100 ppm, 500 ppm and 1000 ppm. To calibrate the analyzer, two extra cylinders are needed which are zero gas (purified compressed air) and span gas (cyclohexane). A Matheson 8240 mass flow controller and a transducer are used to control the zero gas

flow rate. A Miller Nelson MFC-1113 mass flow controller and a transducer are used to control the span gas flow rate. The Matheson and Miller Nelson mass flow controllers can only show the voltage readings. They can not show the mass flow rates directly. They need to be calibrated to get the real mass flow rate. Because the resolution of these two mass flow controllers is not high, the voltage signals are transferred to high resolution HP (Hewlett Packard) 3468A multimeters which can give a resolution as high as 10^{-6} V. The calibration of these two mass flow controllers is based on the voltage readings from the HP multimeters. A sketch of the analyzer setup is given in figure 3.4.

Figure 3.4 Schematic of Hydrocarbon Analyzer Setup

- 1: Meloy Hydrocarbon Detector
- 2: Hydrogen Cylinder
- 3: Zero Gas Cylinder (Compressed Air)
- 4: Span Gas Cylinder (Cyclohexane)
- 5, 6: Transducer
- 7: Matheson HC500/2C S/N8A165 Mass Flow Controller
- 8: Miller Nelson MFC-1113 Mass Flow Controller
- 9, 10: Hewlett Packard 3468A Multimeter

—→: Gas Flow Direction

—: Signal Line

Once the hydrocarbon analyzer is properly calibrated, the TVOC signal can be sent to the data acquisition system. The TVOC value will be shown on the monitor software VIEWDAC and will also be saved as data files. Refer to appendix C for detailed analyzer calibration procedure.

3.3.3 Hot Wire Anemometer

A hot wire anemometer was used to measure the velocity of air over the surface of specimen. The hot wire probe operating temperature is 100 °C. Resistance at 0 °C and 100 °C are 5.64 Ω and 7.13 Ω respectively. A detailed hot wire anemometer calibration procedure is found in appendix C.

3.3.4 Temperature and Humidity Sensor

A Vaisala HMI33 temperature and humidity sensor was placed in the corner of the test chamber. This sensor was connected to the Miller–Nilson HC-301 mass flow controller. The mass flow controller thus could sense the temperature and relative humidity inside the test chamber. This sensor also sends the temperature and relative humidity signals to the data acquisition system where they can be recorded by the data acquisition software. Figure 3.6 shows the photograph of the hot wire probe. A detailed temperature and humidity sensor calibration procedure is described in appendix C.

3.3.5 Micromanometer

An Oxford AP270S micromanometer was used to measure the pressure inside the test chamber. This manometer uses ambient pressure in the test room as reference pressure and it measures the pressure difference between the test chamber and test room. The pressure in the test chamber was kept slightly above the ambient pressure. This positive pressure differential prevents the chamber from sucking in ambient air that will cause contamination of the test chamber. An exhaust pump was used to maintain the positive pressure difference inside the test chamber. This exhaust pump can overcome the long tubing resistance from the chamber to the exhaust gas outlet and thus keeps the exhaust gas flowing out of the test chamber smoothly. At the same time, the suction rate was set slightly lower than the inlet flow rate of fresh air thereby maintaining a positive pressure differential.

3.4 Test Condition Controlling System

The test condition controlling system includes both the devices that control the test room conditions and the devices that control the conditions inside the test chamber. The test condition in the test room was controlled by a heating system, an air conditioning system, a dehumidifier and the ventilation system. An environmental controller was used to control the temperature, humidity and air exchange rate inside the test chamber.

3.4.1 Environmental Controller

Test conditions are critical to test results. A well-controlled environment was required to achieve good test results and test repeatability. An HCS-301 Miller-Nelson

environmental controller was used to control the test conditions inside the test chamber. A completely automated system consisting of three controlling mechanisms and three sensors has been designed to precisely control the flow, temperature and humidity of air. The panel lights on the control module can indicate if appropriate water pressure, water level, and air flow are available. The internal switches can automatically deactivate the humidity and temperature controllers and associated heaters when conditions are not adequate for safe operation of the system. The environmental controller was designed to achieve $\pm 2\%$ of airflow accuracy, $\pm 3\%$ of humidity accuracy and $\pm 1^\circ\text{C}$ of temperature accuracy. A photo of the environmental controller is shown in figure 3.5.

Figure 3.5 Photo of the Environmental Controller

The environmental controller, usually called the blue box, collects the temperature and humidity signals through a sensor placed in the corner of the test chamber. On the front panel of the controller, there are three setting knobs which are used to control the

temperature, relative humidity and air flow rate. A schematic diagram of the controller is shown in figure 3.6. A detailed schematic of the control system is shown in figure 3.7.

Figure 3.6 Block diagram of flow-temperature-humidity control system

Figure 3.7 Schematic diagram of flow-temperature-humidity control system

There are three indicator lights on the front panel which indicate the air flow, water level and water pressure status. If any panel light is not lighted, the air and water heaters will not activate and hence no control will occur.

Because the small chamber was not insulated, the temperature control in the small chamber depended on the control of the room temperature. This was because the air

heater in the controller could only provide a limited amount heat to the chamber. If the room temperature was low, the heat loss from the chamber to the room was much larger than the heat provided to the chamber. The room temperature control thus turned out to be very important. Since the volume of the room was much larger than the volume of test chamber, it brought extra difficulties to the temperature controller.

3.4.2 Air Conditioner and heater in test room

The room temperature was controlled by a Nortec air conditioning system and a heating system. The temperature of the test room was balanced by the two systems. The evaporator of the air conditioning system is located in the ceiling of the test room and provides cold air to the test room through the air duct. The condenser, compressor and controlling units are outside the test room. Environmental air is used to cool down the condenser. A fine and a coarse adjusting knob are used to set the room temperature. A heater is used to compensate the room temperature. A temperature sensor and adjusting knob were connected with the heater. If the room temperature went below the setting temperature, the heater would start automatically to provide heat to the room. If the temperature went higher than the set point, the heater would be shut down. The temperature was cooled down by the air conditioning system. The temperature in the test room can be controlled within $\pm 1^{\circ}\text{C}$.

3.4.3 Humidifier

In order to keep a relatively stable humidity in the test room, a humidifier was used. This humidifier consists of a sensor, an adjusting knob, a water sink, a heater and a fan. The humidifier works automatically. The room humidity can be set by the adjusting knob. If

the humidity goes below the set value, the heater starts to work and vaporizes the water in the sink. The fan will blow the water vapor into the room. If the humidity is high enough, the heater will be shut down automatically. Distilled water was supplied to the water sink continuously. The humidity in the room was designed to be controlled within $\pm 5\%$.

3.5 Air and Water Supply System

3.5.1 Air Supply System

The fresh air for the test chamber came from the outdoor air. It is first compressed to overcome the resistance of the piping system and the positive pressure inside the chamber. The air is then dried as it passes through a desiccant dehumidifier. Before the air flows into the test chamber, it passes an oxidizer to eliminate any possible organic (carbon) contamination. This guarantees that only “zero” gas can enter the test chamber. A sketch of the air supply system is given in figure 3.8.

Figure 3.8 Schematic of Air Supply System

1--pump

6--regulator

2--compressor

7--environmental controller

3,4--desiccant dehumidifier

8--drain water

5--oxidizer

3.5.1.1 Dehumidifier

In order to control the humidity inside the test chamber, dry clean air must be supplied to the controller so the controller can mix the appropriate amount of the dry air and water vapor to produce the desired humidity. Two desiccant dehumidifiers were used to dry the outdoor air. These two dehumidifiers can not both be shut down when a test is running, at least one should be kept operating to ensure the desired flow rate and quality of air into the small test chamber. To replace exhausted desiccant, the two dehumidifiers should be replaced alternatively. See Figure 3.9 for reference.

Figure 3.9 Dehumidifiers and Oxidizer

3.5.1.2 Oxidizer

After being dehumidified, the air goes into the catalytic oxidizer (figure 3.9). This catalytic oxidizer guarantees that only “zero” gas can enter the test chamber. This oxidizer can effectively remove carbon monoxide and hydrocarbons in the air. It also provides low cost ambient air replacing high priced air from compressed air cylinders.

The oxidizer operates at a temperature about 340 °C and a flow rate of 0.24 to 7 l/min. With these operating conditions, this oxidizer can remove 99% of the carbon monoxide and hydrocarbons.

3.5.1.3 Compressor

The compressor consists of a pump and high-pressure container (see figure 3.10). The pump operates automatically, starting and stopping based on sensing the pressure inside the container. This high pressure allows air to overcome the friction force along the piping to the test chamber and the positive pressure inside the test chamber.

Figure 3.10 Compressor and Container

3.5.2 Water Supply System

In order to avoid possible contamination of the small test chamber, purified water is used to provide humidity to the test chamber. The source water comes from tap water and is purified by a carbon and deionizing filter. Carbon and possible ions in the tap water can

be eliminated leaving distilled water to be provided to the test chamber. The water supply tubing is connected to both the environmental controller which provides humidity in the test chamber and the Nortec heater which provides humidity to the test room. Both the relative humidities inside the chamber and inside the test room are set to 50% to eliminate possible vapor pressure difference between the test room and test chamber. This guarantees that the humidity inside the test chamber is kept at a constant level when the test chamber is opened to load test specimens. It also provides a well-controlled environment to pre-condition test specimens, especially concrete.

3.6 Calibration Devices

3.6.1 Hydrocarbon Analyzer Calibration Devices

The hydrocarbon analyzer is a very important component of the test facilities. It directly provides the instantaneous total hydrocarbon level inside the test chamber. It was found that the “zero point” of the Meloy hydrocarbon analyzer was shifting with time so a periodic calibration is required to verify its output reading. Some auxiliary equipment is also needed for the calibration. A detailed calibration procedure for the Meloy hydrocarbon analyzer can be found in appendix C.

3.6.1.1 Transducers

Two Matheson mass flow transducers were used in the calibration process for the hydrocarbon analyzer. The transducers can sense and control the gas flow rate. To measure gas flow rate, each transducer uses a small stainless-steel sensor tube. Two external, heated resistance thermometers are wound around the sensor tube. When gas is flowing, heat is transferred along the line-of-flow to the downstream thermometer,

thereby producing a signal proportional to the gas flow rate. The higher the flow rate, the greater the differential between the thermometers. This signal is compared to a command voltage from the mass flow controller. From the comparison, an error signal is generated which adjusts the valve power to change the gas flow until the desired set point is reached. Figure 3.11 shows the structure of the transducer.

Figure 3.11 Mass Flow Transducer Outline

3.6.1.2 Multimeters

Mass flow controllers were used to control the gas flow rates. In calibration of the mass flow controllers, the voltage output is needed to plot the calibration curve. However, the voltage outputs of the two mass flow controllers are not precise. Two multimeters are thus connected parallel to the two mass flow controllers to measure the voltage output of each. These multimeters have a resolution of $\pm 10^{-6}$ V.

3.6.1.3 Gas Producers

Two gas producers were used in the hydrocarbon analyzer calibration process. These gas producers are placed in front of the flow transducers to ensure that only gas will flow into the transducers. Therefore the transducers can measure precise flow rate for an accurate calibration.

3.6.1.4 Mass Flow Controllers

In order to control the zero gas and span gas flow rates, two mass flow controllers were used. These two mass flow controllers are connected to the two flow transducers. The flow rate can be controlled by adjusting flow rate controlling knobs on the controllers. Through adjusting the knobs, each controller sends different comparison signals to its corresponding transducer and the transducer will adjust its needle valve to allow corresponding flow to pass.

3.6.2 Hot Wire Anemometer Calibration Devices

The hot wire probe should be calibrated regularly to achieve accurate output because the hot wire probe is sensitive to ambient pressure and temperature. Calibration for every test is recommended. The calibration of the hot wire probe was done in a calibration lab using a TSI 8390 wind tunnel.

3.6.2.1 TSI 8390 Wind-Tunnel

A TSI 8390 wind tunnel was used to calibrate the hot wire anemometer. This TSI 8390 wind tunnel can be used to calibrate a velocity transducer in the range of 0.15m/s to 45

m/s. The velocity was established by using a laser Doppler velocimeter which can be regarded as a primary standard. Thus the accuracy of the wind tunnel is traceable to the accuracy in measuring the diameter and rotational speed of the rotating disc. The velocity level is controlled by a DART 2500 controller.

3.6.2.2 Manometer

A manometer was used to measure the difference between the total pressure inside the wind tunnel and ambient static pressure and thus the velocity inside the wind tunnel can be calculated. This manometer gives a reading accuracy of 0.01mm H₂O. Ambient temperature and pressure will also affect the offset of this manometer and this offset should be subtracted in calculating the air velocity. A data sheet should be prepared before the calibration and the manometer readings should be calculated basing on different velocity levels. These velocity levels can be achieved by adjusting the DART 2500 controlled to let the reading on the manometer match the calculated values.

3.6.3 Temperature and Humidity Calibration Device

A chilled mirror temperature and humidity probe was used to calibrate the temperature and relative humidity inside the test room and test chamber. This chilled mirror probe can give an accuracy of $\pm 0.1^{\circ}\text{C}$ in temperature and $\pm 0.1\%$ in relative humidity.

3.6.4 Flow Rate Calibration Devices

Two flow meters, ADM3000 intelligent flow meter and Humonics bubble flow meter, were used to calibrate the gas flow rate in order to achieve accurate readings.

3.6.4.1 ADM3000 Flow Meter

The ADM3000 intelligent flowmeter was computer calibrated by the manufacturer periodically. It gives a flow range from ± 1.0 to 750 ml/min, an accuracy of $\pm 3\%$ and a temperature range from 0°C to 45°C.

The ADM also prevents the influence of moisture in the sample air. The normal bubble type flowmeters add water vapor to the gas being measured, which introduces error into the flow rate measurement. The reading error arises from relatively high concentrations of water vapor present in the soap bubble apparatus. At room temperature, water vapor can raise readings by nearly 4%. This unfortunate property is amplified by the effect of temperature. Since the ADM3000 is bubble-free, this error is not introduced into the flow rate measurement. On the other hand, the ADM3000 can give a very quick reading and this is very critical in the sample taking procedure because the concentration in the test chamber changes very fast in the first several hours of the test. The concentration level decides the sampling volume and thus the sampling rate. The fast changing of concentration requires the quick detection of the flow rate.

3.6.4.2 Humonics Bubble Flowmeter

The Humonics bubble flowmeter is calibrated to operate with a completely wet bubble meter. It has a sensor block and a microprocessor. The sensor can sense the movement of bubbles and the microprocessor can automatically adjust the resolution of the display to the gas flow rate. The microprocessor can also do the auto-averaging to increase the accuracy of the reading. A moving flow average of up to fifteen individual flow results

will be displayed. Flow range for the Humonics flowmeter is from 0.1 l/min to 25 l/min.

It is suitable for a large flow rate calibration, such as chamber supply air.

3.7 Data Acquisition System

A computer based data acquisition system is used to collect the temperature, relative humidity and concentration data from the test chamber. It includes a Keithley 500A measurement and control system, IFA 100 intelligent flow analyzer, several computers and corresponding software.

The Keithley 500A measurement and control system collects all the necessary data signals from the testing system, including temperature and humidity signals inside the test chamber from a Vaisala temperature and humidity probe, the THC signal from hydrocarbon analyzer, the pressure signal from the micromanometer, the velocity signal from the hot wire anemometer, and the air flow rate signal from the environmental controller. Then the Keithley 500A sends all the signals to a DELL 466 computer through interface software "VIEWDAC". Each signal is displayed on a different channel on a monitor. All the calibration equations should be input into the software beforehand.

The velocity signal is also sent to another collecting software, "ROOMAIR", in another computer. "ROOMAIR" is especially useful to control the movement of the hot wire probe. The hot wire probe can be attached to a traverse device driven by a step motor. This traverse device allows the hot wire probe to move vertically to measure the velocities along the vertical direction. "ROOMAIR" is useful to do the dynamic calibration for the test chamber. "ROOMAIR" is also used when doing the hot wire calibration.

Another data collecting software is "STAR". "STAR" collects the data analyzed by GC/FID. "STAR" saves test data into a Digital 486 computer. "STAR" also controls the operation of the GC and multi-tube desorber. A method file needs to be built up and the GC calibration data needs to be input to the software. Detailed operation procedures are found in the relevant manuals.

3.8 Chemical Analysis Devices

3.8.1 Gas Chromatograph

The analysis of the VOC concentration and identification of the chemical components are done by a GC system. This system includes a Varian GC Star 3400, a multi-tube desorber and a multi-tube conditioner.

3.8.2 Multi-Tube Desorber

Figure 3.12 Photograph of Multi-tube Desorber and Conditioner

The multi-tube desorber is the first step in the analysis of the gas samples. The desorber has 16 heating jackets and each jacket can hold one Tenax tube. Therefore it can process a maximum 16 tubes at one time. The tubes will be heated up to 230 °C to vaporize all VOC contents inside and Helium carrier gas will take those contents to traps to concentrate them before letting them go into the GC. Figure 3.12 shows a photograph of the multi-tube desorber.

3.8.3 Multi-Tube Conditioner

The Tenax tubes must be cleaned to be re-used after being run on the GC. This job is done by a multi-tube conditioner (see figure 3.12). The conditioner heats up the tubes to 275 °C and lets Helium gas pass through the tubes to carry away any VOC contents inside. This conditioner has 6 heating jackets and can clean a maximum of 6 tubes each time. Each jacket has individual valves to control the Helium flow rate and operating temperature. A detailed cleaning procedure can be found in appendix B.

3.9 Sampling System

In order to collect gas samples in the small test chamber for further GC analysis, a sampling port was set up outside the test room. Teflon tubing was connected to the exhaust air tubing from the test chamber. A schematic diagram is given in figure 3.13.

Figure 3.13 Schematic Diagram of Sampling Port

- 1: Transducer – Matheson 8272-0432 Mass Flow Transducer
- 2: Suction pump – CAST DOA-V111-AA
- 3: Mass Flow Controller-- Matheson Dyna-Blender 8280 Mass Flow Controller
4. Calibration Flow Meter –ADM3000 Intelligent Flowmeter
5. Tenax Adsorption Tube

A CAST suction pump was used to suck the sample air into the TVOC tube. The sampling rate is controlled by the Matheson mass flow controller.

3.9.1 Mass Flow Controller and Transducer

The sampling rate is adjusted by a Matheson 8280 mass flow controller. The adjustable range is 0–300 ml/min. If necessary, a larger range mass flow controller can be chosen. An adjusting knob on the front panel of the Matheson mass flow controller is used for convenient adjustment and an LCD display shows the current flow rate. The display signal is sent back by the Matheson 8280 mass flow transducer. The transducer is suitable to operate under normal room temperature and has an inlet pressure of 30psig and outlet pressure which is the atmospheric pressure. To allow a better TVOC adsorption, the sampling rate should be controlled to less than 250 ml/min.

3.9.2 Sampling Tube

Figure 3.14 Schematic of Thermal Desorption Tubes

Tenax sampling tubes were used to take gas samples from the test chamber. Figure 3.14 shows a schematic diagram of it. The maximum adsorption capacity is 12 μg of hydrocarbon, and 10 μg sampling amount is recommended.

3.10 Accessories

There are several other accessories such as gas cylinders, fan motor and dogmatic indicator and processor. Please refer to appendix D for a complete list.

Chapter 4

Adhesive Application Method Development

In testing the VOC emissions from carpet-adhesive assemblies, the amount of adhesive applied is very critical to the test results. The amount of the adhesive applied is controlled by the application method. A good application method should provide repeatable test specimens to achieve repeatable test results. A test specimen in this study refers to a piece of geometry-fixed substrate on which a certain amount of adhesive is applied following some application method. One of the commonly used methods is the trowel method proposed by EPA in which a trowel is used to apply the adhesive onto the substrate surface. This method will be called the “trowel method” hereafter. It is easy to operate but it is difficult to control the accuracy of the amount of the adhesive applied. The “V”-shaped notch of the application trowel also results in a non-uniform adhesive surface that introduces difficulties in the mass transfer modeling. It is necessary to develop a standardized adhesive application method that is easy to follow and can provide a good control to the amount of the adhesive applied. Three adhesive application methods, the trowel method, the applicator method and the container method, were developed and evaluated in this study.

4.1 Development of Adhesive Application Method

The trowel adhesive application method is a commonly used method proposed by EPA (1991). The advantage of trowel method is that it is easy to operate and it is very similar to the current practice. However, this method introduces difficulties in the control of the amount of the adhesive applied. The “U” or “V”-shaped notches of the trowel also produce non-uniform adhesive layers that are very difficult to simulate in the mass transfer models. To achieve a uniform adhesive layer above the substrate, two other adhesive application methods were developed—the application method and the container method. The procedures of these three adhesive application methods are described below.

4.1.1 Trowel (EPA) Method

Figure 4.1 Position of Substrate Using Trowel (EPA) Method

Figure 4.2 Dimension of Trowel Notch

Figure 4.3 Photograph of EPA Trowel

Figure 4.4 Photograph of Substrate Holder

In the trowel test method, a 1/8"×1/8"×1/8" trowel (see figure 4.2 & 4.3) was used to apply the adhesives onto a piece of 12cm×12cm glass substrate. The glass substrate was placed on a same size (12cm×12cm) base with 4 adjustable screws that can adjust the level of the glass substrate (see figure 4.1 & 4.4). The following procedures were followed in the trowel method.

- a) Weigh the initial mass of the substrate. Tare the balance with the substrate on.
- b) Remove the substrate from the balance and place the substrate on the application base.
- c) Adjust the height of the substrate. Level the substrate with the surface of the container.
- d) Place excessive amount of the adhesive along one edge of the substrate taking no more than 30 seconds to do so. Use the trowel to make a single pass across the substrate at a speed of approximately 0.5 – 0.7 cm/s, then make another pass perpendicular to the first one at approximately the same speed. The trowel should be held at an angle of 45° to the substrate surface.
- e) Visually check the adhesive surface. If uniform adhesive strips are formed and no big area of defect is observed, then go to the next step. Otherwise, reject the sample.
- f) If the sample passes the step e), remove the substrate from the container and check the weight of adhesive. If the weight is more than 15% different from the target amount, reject the sample.
- g) If the sample passes step f), place the sample in the test chamber

The time from taking the adhesive out of its container to putting the test sample into the

test chamber should be less than five minutes. The zero time was set when the sample was placed in the test chamber.

4.1.2 Applicator Method

In the applicator method test, an applicator with an adjustable accuracy of 0.025mm was used to apply the adhesives onto a piece of 12cm×12cm glass substrate. The glass substrate was placed into a same size (12cm×12cm) base holder which had 4 adjustable screws that can adjust the level of the glass substrate (see figure 4.5). The following procedures were followed in the applicator method.

- a) Weigh the initial mass of the substrate with the electronic balance. Tare the balance with the substrate on.
- b) Place the substrate into a base holder that is fixed on a flat base.
- c) Use the applicator to set the initial substrate height. See figure 4.5.
- d) Adjust the micrometers of the applicator to leave a space that is the required thickness of adhesive between the substrate and the bottom of the sliding block.

Figure 4.5 Position of Substrate Using the Applicator Method

- e) Place excessive amount of adhesive along one edge of the substrate taking no more than 30 seconds to do so. Move the applicator from the adhesive side across to the other side at a speed of approximately 0.5 – 0.7 cm/s.
- f) Lift the applicator and move it back to its starting point. Make another pass across the substrate at approximately the same speed.
- g) Visually check the adhesive surface. If uniform adhesive strips are formed and no big area of defect is observed, then go to the next step. Otherwise, reject the sample.
- h) If the sample passes the step g), remove the substrate from the base holder and check the weight of adhesive. If the weight is more than 15% different from the target amount, reject the sample.
- i) If the sample passes step h), place the sample in the test chamber.

The time of taking the adhesive out of its container to putting the test sample into the test chamber should be less than five minutes. Zero time for the purpose of later reading was set when the sample was placed in the test chamber.

4.1.3 Container Method

Figure 4.6 Photograph of the 0.4mm Container

A container method was developed for a better control of the adhesive amount applied. A photograph of the 0.4 mm container is shown in figure 4.6. A schematic diagram of 0.4mm thickness container is shown in figure 4.7. The container was made of stainless steel to prevent the possibility of the adhesive being absorbed by the surface. To verify the effect of the amount of adhesive on the emission rate, three containers with different recess thickness, 0.4 mm, 0.7 mm and 1.0 mm were made. The adhesive application method using this container is described as follows:

Figure 4.7 Photograph of the 0.4 mm Container

- Weigh the initial mass of the container with an electronic balance with a resolution of 1 mg. Tare the balance with the container on.
- Place the container into its base holder.

- c) Level the applicator with the container top surface as shown in figure 4.8.
- d) Place excessive amount of adhesive along one edge of the container taking no more than 30 seconds to do so. Pass the applicator slowly from the adhesive side across to the other side at a speed of approximately 0.5 – 0.7 cm/s.
- e) Turn the applicator around and pass it back to the starting point at the same speed.
- f) Turn the applicator around and pass the applicator to the other end again.
- g) If a uniform layer is observed, go to step h); otherwise, reject the sample.
- h) Use the balance to check the weight of the adhesive applied.
- i) Place the sample in the test chamber

4.2 Evaluation of the Adhesive Application Methods

Ten adhesive application tests were conducted for each application method using adhesive I. The initial weights of the adhesive applied were measured. Table 4.1 shows the test results. The standard deviation of the average amount of adhesive applied for the trowel method is 0.645 g which is more than 10% of the average amount (6.147g); the standard deviation for the applicator method is 0.617g which is equivalent to 9.7% of the average amount. The container methods tests show much smaller standard deviations as shown in table 4.1. The standard deviations in the 0.4mm, 0.7mm and 1.0mm container tests are 3.2%, 2.5% and 2.4% of their average amounts respectively. It shows that the container method gives a much more accurate control of the adhesive application amount. Figure 4.9 –4.13 show the distribution of adhesive application amount in the repeatability tests.

Test Method		Trowel	Applicator	Container		
		1/8"1/8"1/8"	0.4mm	0.4mm	0.7mm	1.0mm
T e s t N o	1	5.632g	5.458g	3.876g	6.748g	9.853g
	2	6.656g	6.954g	3.861g	6.631g	10.427g
	3	6.998g	5.437g	3.797g	6.704g	10.464g
	4	6.098g	5.448g	3.642g	6.401g	10.123g
	5	6.054g	6.233g	3.640g	6.826g	9.822g
	6	6.899g	5.773g	3.868g	6.612g	9.940g
	7	5.107g	5.733g	4.002g	6.765g	10.362g
	8	6.730g	6.709g	3.876g	6.773g	10.186g
	9	5.972g	5.329g	3.992g	7.048g	10.214g
	10	5.456g	5.103g	3.822g	6.801g	10.440g
Ave. Weight		6.160g	5.818g	3.838g	6.731g	10.183g
Std. Deviation		0.645g	0.617g	0.122g	0.167g	0.245g1
% Error		10.5	10.6	3.185	2.483	2.404

Figure 4.9 Distribution of adhesive application amount in the Trowel method repeatability test

Figure 4.10 Distribution of Adhesive application amount in applicator method repeatability test

Figure 4.11 Distribution of adhesive application amount in the 0.4mm container repeatability test

Figure 4.12 Distribution of adhesive application amount in the 0.7mm container repeatability test

Figure 4.13 Distribution of adhesive application amount in the 1.0mm container repeatability test

The repeatability tests showed that the container method gave a better control of the amount of adhesives applied than did the other two methods. To check the repeatability of the emission from samples prepared by the container method, five chamber tests were conducted under the standard test conditions. The ratios of the adhesive weight remained on the containers after 24 hours (W_{24}) and 32 hours (W_{32}) to the initial adhesive weight are listed in table 4.2.

Table 4.2 Repeatability test of Container Method			
Test No.	$W_n(g)$	W_{24}/W_n	W_{32}/W_n
1	6.704	0.5798	0.5783
2	7.128	0.5775	0.5762
3	6.797	0.5866	0.5858
4	6.795	0.5879	0.5866
5	6.366	0.5807	0.5793
Average	6.758	0.5825	0.5812
Std. Deviation	0.272	0.00451	0.00467
Rel. Std. Dev.	4.0%	0.7%	0.7%

Five chamber tests were also conducted for samples prepared by the trowel method under the standard test conditions and a comparison between the results obtained with samples prepared by the trowel application method and container application method is given in table 4.3.

Table 4.3 Comparison between the Trowel and the Container Application Methods

Method	W_n (g)	ΔW_{24} (g)	ΔW_{32} (g)
Trowel	5.912 ± 0.313	3.560 ± 0.269	3.585 ± 0.269
Container	6.758 ± 0.272	3.940 ± 0.030	3.926 ± 0.032

Chapter 5

Testing Procedures and Results

Three areas were studied using a small-scale environmental test chamber under standard test conditions in this study: (1) the influence of the adhesive amount applied on the VOC emissions in the carpet-adhesive assemblies; (2) the effect of the substrate on the VOC in the carpet-adhesive assemblies; (3) comparison of different adhesives in the carpet-adhesive assembly. The weight loss data and concentration decay data were measured in each test. The gas samples from the test chamber were collected and were analyzed by the GC/FID/MS method to identify VOCs and quantify the VOC concentration levels in the test chamber. This chapter will discuss the testing procedures and results.

5.1 Testing Procedures

The testing procedures included the equipment set-up and calibration, test sample preparation, test data collecting and gas sampling taking procedures.

5.1.1 Equipment set-up and calibration

The equipment set-up and calibration were performed at least one day before the test. The equipment set-up procedures included the equipment connection and warming-up, test

chamber cleaning and test room and chamber pre-conditioning under the standard test conditions. Not all the equipment needed to be calibrated for every test. However, the hydrocarbon analyzer needed to be calibrated immediately before the testing began. The step-by-step equipment set-up and calibration procedures can be found in appendices B and C.

5.1.2 Chamber Background Concentration Measurement

After all the test equipment were properly set up, the test chamber was closed and fresh air was flushed into the test chamber for at least 3 hours at a rate of one air change per hour. Then the chamber background concentration was measured. The chamber background should not be greater than 0.3 ppm. If the chamber background concentration is larger than 0.3 ppm, longer flushing period is needed until the background concentration is smaller than 0.3 ppm. This background concentration value should be subtracted later from the measured VOC concentration values to get rid of the influence of the chamber background concentration.

5.1.3 Test sample preparation

The test sample preparation procedures included the adhesive preparation, the substrate preparation and the adhesive application. The adhesives were filled into small containers before a group of tests began. The glass, stainless steel and concrete substrates were placed in the test room for pre-conditioning under the standard test conditions. The pre-conditioning of the substrates allowed the temperature and relative humidity of the substrates to reach the test conditions in the test chamber. The glass and stainless steel substrates were pre-conditioned for 2-3 hours before testing. The concrete substrate was pre-conditioned for at least one week before testing.

A test specimen was then prepared following one of the three adhesive application methods described in chapter 4. The time to prepare a test specimen was less than 5 minutes. When a test specimen was ready, it was placed on the electronic balance in the test chamber and then the chamber was sealed.

5.1.4 Test Specimen Loading

A test specimen was placed on an electronic balance with a resolution of 1mg. The electronic balance was placed in the middle of the inner test chamber. The relative position of the balance in the inner chamber is shown in figures 3.2 and 5.1

Figure 5.1 Schematic Diagram of the Balance Position in the Test Chamber

5.1.5 Test Data Collecting

Once the test specimen was placed in the test chamber, the inner chamber and the outer chamber were sealed and the data collecting began. The temperature, relative humidity,

VOC concentration, pressure in the test chamber, air velocity over the test specimen and chamber air exchange rate data were collected every 30 seconds by the VIEWDAC system. The weight decay data were collected every 10 seconds in the first half-hour, every 20 seconds from the half-hour to one hour and every 30 seconds after the first hour.

5.1.6 Gas Sample Taking

Before the test specimen preparation began, a gas sample was taken from the sealed empty test chamber to determine the initial chamber background VOC concentration. In the testing period, the samples were taken as follows:

Day one:

The samples were taken every 15 minutes for the first hour, very 30 minutes for the next 4 hours and every hour after 5 hours.

Day two and three:

Three samples were taken:

the first at approximately 9:00am, the second at approximately 12:00am and the last one at approximately 6:00pm.

Day four and after:

two samples were taken:

one at approximately 9:00am and the other at approximately 6:00pm.

5.2 Quantifying Weight Loss Data Using a Double Exponential Model

The weight decay data versus time were measured in each test. To analyze these data, a double exponential model was used to describe the weight decay trend:

$$W(t) = a \exp(-k_1 t) + c \exp(-k_2 t) \quad (1)$$

The emission rate can be calculated by:

$$E(t) = -d[W(t)]/dt = a k_1 \exp(-k_1 t) + b k_2 \exp(-k_2 t) = E_1(t) + E_2(t) \quad (2)$$

Where $W(t)$ is the weight of the adhesive remaining on the substrate, g;

a , b , k_1 and k_2 are constants determined by the regression analysis.

Figure 5.2 Typical Weight Decay Curve of Adhesive on Glass Substrate

A mass transfer process is controlled by the diffusion mass transfer mechanism and convective mass transfer mechanism. When a “wet” material like adhesive is just placed into the test chamber, the diffusion mass transfer rate is higher than the convective mass transfer rate because VOC liquid is presented at the surface of the adhesive. Since convective mass transfer cannot carry away the mass brought by the diffusion mass transfer, the general mass transfer rate is limited by the convective mass transfer rate in this period. With the depletion of the VOCs at the adhesive surface and the formation of a dry skin layer at the surface of the adhesive, the diffusion mass transfer rate decreases very quickly and becomes smaller than the convective mass transfer rate. The general mass transfer rate is thus limited by the diffusion mass transfer rate in later period. Generally, the mass transfer rate in the diffusion-controlled period is much smaller than that in the convective-controlled period. Because of the decrease of the general mass transfer rate, the weight decay process can be divided into three periods accordingly: fast decay, transition and slow decay. Figure 5.2 shows a typical adhesive weight decay curve on the glass substrate. The double exponential model is used to fit the weight decay data. The maximum fitting deviation is about 0.0565 g and the standard fitting deviation is only 0.0168 g.

The double exponential model discussed above can be consider as the addition of two first order decay models, fast decay and slow decay, according to Zhishi Guo, 1996. To characterize the three periods, two characteristic times, t_1 and t_2 , are defined. t_1 is defined as the time when $E_1(t) = E_2(t)$. That is, $a \cdot k_1 \cdot \exp(-k_1 t_1) = b \cdot k_2 \cdot \exp(-k_2 t_1)$
 t_2 is defined as the time when $E_2(t) = 100E_1(t)$. That is,

$$100 \cdot a \cdot k_1 \cdot \exp(-k_1 t_1) = b \cdot k_2 \cdot \exp(-k_2 t_1)$$

t_1 can be considered as the end of the fast decay period and beginning of the transient period. t_2 can be considered as the end of the transient period and the beginning of the slow decay period. Note that this double exponential model is an empirical model, it cannot explain the mass transfer process physically. However, this double exponential model does give very good curve fitting to the weight loss data and can be used to characterize the test data preliminarily in particular the length of each period.

Although the same size of the substrate was used for samples prepared by the trowel method, the contact surface area of the adhesive to the ambient air is different from the areas in the other two methods because of the particular shape of the trowel notch. The difference of the contact surface area results in the difference in the loading factor that is known as an important factor influencing the VOC emission rate. In order to compare the characteristic time t_1 and t_2 between samples prepared by the trowel method and samples prepared by the other two methods, a specific term, effective emitting area (A_e), is used instead of the area of the substrate. Effective emitting area hereby refers to the contact area of the adhesive to the ambient air. The effective emitting area of a test specimen using the container or applicator application method is the area of the substrate itself. To calculate the emitting area of a test specimen prepared by the trowel application method, two assumptions are made: (1) All the adhesive strips are even. (2) No adhesive exists on the substrate other than the adhesive strips. The following calculation method was followed:

a) Calculate the number of the adhesive strips.

If the length of the substrate is L_s cm and the dimension of the trowel is $D'' \times D'' \times D''$, the number of adhesive strips (N_s) is calculated as: $N_s = \text{ROUND}(L_s / (2.54 * 2D))$

“ROUND” means the rounding of the calculation value to the nearest integer.

b) Calculate surface area of one strip of the adhesive.

The volume (V_a) of the adhesive applied is calculated first: $V_a = W_0/\rho$ (cm^3)

Where ρ is the density of the adhesive and W_0 is the initial weight of the adhesive. The volume of one adhesive strip (V_a') is calculated in order to calculate the height (H) of it.

$$V_a' = V_a/N_s \text{ (cm}^3\text{)}$$

$$H = V_a'/(1/2 * L * D * 2.54) \text{ (cm)}$$

Once the height of the adhesive strip is achieved, the surface area of the adhesive strip (A') can be calculated as: $A' = L * 2 * \text{Sqrt}((D/2)^2 + H^2)$

c) Calculate the effective emitting area (A_e) $A_e = A' * N_s$

Table 5.1 Comparison between the Trowel and the Container Application Methods

Method	W_0 (g)	t_1 (hour)	t_2 (hour)	W_{t1}/W_0 %	W_{t2}/W_0 %
Trowel	5.912 ± 0.313	7.790 ± 0.306	13.971 ± 0.615	62.7 ± 7.3	61.6 ± 7.4
Container	6.758 ± 0.272	9.599 ± 0.317	15.774 ± 0.263	60.0 ± 4.0	58.5 ± 4.0

Using this double exponential model and the concept of the effective emitting area, the characteristic times t_1 and t_2 of the container methods tests and trowel methods tests in chapter 4 can be calculated and plotted. Table 5.1 shows the characteristic times t_1 and t_2 for samples prepared by trowel method and container method respectively. The weight remaining on the substrates after t_1 (W_{t1}) and t_2 (W_{t2}) are also listed in table 5.1. From table 5.1 it can be seen that smaller percent the standard deviations of weight decay were observed for samples prepared by the container method. The comparisons of t_1 and t_2 versus W_0/A_e are shown in figures 5.3 and 5.4.

From the comparison in figures 5.3 and 5.4, it can be seen that small t_1 and t_2 were

obtained from samples prepared by the trowel method. This may be explained as that the samples prepared by the trowel method have smaller effective emitting areas than those prepared by the container method. Therefore the samples prepared by the trowel method dry up faster than those prepared by the container method do. The fast decay period and transition period thus become shorter for samples prepared by the trowel method.

Figure 5.3 Time t_1 Comparison between Trowel and Container Methods

Figure 5.4 Time t_2 Comparison between Trowel method and Container Method

5.3 Test Results

5.3.1 Effect of Adhesive Amount Applied

The influence of the amount of adhesive applied on the VOC emission was tested. Three pieces of 12cm×12cm stainless steel with different recess thickness (0.4mm, 0.7mm and 1.0mm) were used following the container adhesive application method. Adhesive I was used in all the tests. All tests were conducted in a 1.0m×0.8m×0.5m small-scale test chamber under standard test conditions. An electronic balance with a resolution of 1 mg was used to measure the adhesive weight decay data in the small test chamber. The hydrocarbon analyzer was used to collect the concentration data inside the test chamber. As discussed in chapter 4, the container method was developed for a better control of the amount of the adhesive applied. Thus the amount of the adhesive applied was controlled at three levels: low, medium and high. The testing procedures were described in chapter four.

Figure 5.5 shows the comparison of the VOC concentration decay data in the test chamber comparison for the 0.4, 0.7 and 1.0mm adhesive layers. The initial adhesive weights of the three tests were 4.29g, 6.704g and 9.749g. For comparison, each set of concentration decay data was normalized by its highest concentration peak value. It is seen from figure 5.5 that the increasing amount of the adhesive applied leads to a shift of the concentration peak to the right, which means that the concentration peak occurs later with a higher adhesive application amount and thus a longer emission time is expected. It is also observed that after one hour from the initial adhesive application, an inflection period occurred and this inflection period was elongated with an increase of the adhesive application amount. Figure 5.6 shows the emission rates of these three tests.

Corresponding to the concentration decay curves in figure 5.5, there are initial decreases of the emission rates followed by the increases and then decreases again as can be seen in figure 5.6. This may indicate that the dry skin begins to form after the adhesive application and the formation of the dry film has an impact on the emission rate. The initial emission rate decreases may be caused by the VOC contents at the adhesive surface being almost depleted and the VOCs underneath the dry film need to diffuse through the film before emitting to the air, resulting in a decrease in emission rates. The emission rates began to increase again at about 40 minutes, 60 minutes and 80 minutes for the 0.4, 0.7 and 1.0 mm specimens, respectively. This may be caused by some micro-cracks formed on the dry skin because the drying process caused the surface stress to increase, which commonly happens to rubber materials. The VOC vapor therefore could break through the dry film layer from the cracks. The reason why a longer inflection period was observed in thicker adhesive layer is still unclear.

Figure 5.5 the Effect of the Amount of the Adhesive Applied on Concentration Decay

Nonane, decane and undecane are selected as three major compounds to represent the volatile organic compounds from the low to high molecular weight. Figure 5.7 shows a gas chromatogram analysis of the VOCs from the carpet adhesive in the 1.0mm container test after 5 minutes of application. Figure 5.8 shows the concentration decay of the three compounds. The decay trends of these compounds appeared to be the similar to the decay trend of the TVOC. The inflection periods are also observed. As expected, the concentration peak time is proportional to the molecular weight. It means that it takes longer time for higher molecular weight compounds to escape from the adhesive layer. Figures 5.9-5.11 show the comparison of the concentration decay of each single compound for different thickness specimens. For all three compounds, it is found that the concentration peak time of a compound is proportional to the thickness of the adhesive layer.

Figure 5.6 Emission Rate Comparison among Different Thickness Containers

Figure 5.7 Gas Chromatogram of the Carpet Adhesive after 5 Minutes of Application

Figure 5.8 the Concentration Decay Comparison of Nonane, Decane and Undecane on 1.0 mm Container

Figure 5.9 Nonane Concentration Decay Comparison Corresponding to Different Thickness Containers

Figure 5.10 Decane Concentration Decay Comparison corresponding to Different Thickness Containers

Figure 5.11 Undecane Concentration Decay Comparison corresponding to Different Thickness Containers

The double exponential model was used to fit the weight decay data of the container tests. Characteristic time t_1 and t_2 were listed in table 5.2. It can be seen that the characteristic time t_1 and t_2 increase with the adhesive application weight. Figures 5.12 and 5.13 show the changing of t_1 and t_2 with the application weight (W_0) over the effective emitting area A_e . W_{t_1} and W_{t_2} represent the weights remaining in the adhesive layer at time t_1 and t_2 .

Table 5.2 Parameters in the Containers Tests

Test id	W_0 (g)	t_1 (hour)	t_2 (hour)	W_0/A_e (g/m ²)	W_{t_1} (g)	W_{t_1}/W_0	W_{t_2} (g)	W_{t_2}/W_0
ctn-04-1	4.29	6.668	10.639	297.9167	2.551	0.595	2.536	0.591
ctn-07-1	6.704	9.525	15.783	465.5556	3.941	0.588	3.906	0.583
ctn-07-2	7.128	9.314	15.669	495	4.182	0.587	4.141	0.581
ctn-07-3	6.797	9.905	16.021	472.0139	4.024	0.592	3.999	0.588
ctn-10-1	9.749	13.376	22.783	677.0139	5.866	0.602	5.8	0.595
ctn-10-2	10.92	13.581	23.481	758.3333	6.596	0.604	6.513	0.596

Figure 5.12 Change of t_1 with W_0/A_e Figure 5.13 Change of t_2 with W_0/A_e

Thicker adhesive layers can be used in the future to verify the inflection phenomenon and to check the dry film forming time at the surface of the adhesive layer. From the previous test of M. Low (1996), no such phenomenon was observed in the test of whole carpet-adhesive assembly. There are three possible reasons: the first is that the effect of the carpet resistance weakens the inflection phenomenon; the second is that the carpet has an impact on the formation of the dry skin layer at the adhesive surface; the third possible reason is that the adhesive layer is too thin to show the inflection phenomenon. Further tests with the carpet covered on the top of the container are recommended.

5.3.2 Effect of Substrate

A commonly used sub-floor in office buildings, concrete, was studied to discover the effect of the substrate on VOC emission behavior. A glass substrate of the same size was also studied for the comparison purpose because glass is considered as the non-absorbent material. The trowel adhesive application method was used for both substrates. All tests were conducted in the small-scale test chamber under the standard test conditions.

Table 5.3 shows the weight decay comparison between the glass and concrete substrates. The weights remained on the substrate from the initial application amount after time t_1 , t_2 , 24 hours and 32 hours are listed in table 5.3. Figure 5.14 also shows the adhesive weight decay curve comparison between the glass and concrete substrate. Each of the weight decay curves in figure 5.14 was normalized by its initial adhesive application amount to eliminate the possible influence of the amount of the adhesive applied. From table 5.2 and figure 5.14 it can be seen that less of the adhesive was emitted from the concrete substrate in the same test period. This can be explained as the adsorption effect of the concrete substrate. A part of the adhesive that was applied on the concrete substrate was

adsorbed by the concrete substrate after the adhesive application and a sharper adhesive weight decay trend was found on the glass substrate in the fast decay period. However, a faster weight decay rate was found on the concrete substrate in the slow decay period probably because some of the VOC contents absorbed by the concrete substrate began to diffuse out. The characteristic time t_1 and t_2 were also calculated and compared in figures 5.15 and 5.16. From figures 5.15 and 5.16 it can be seen that t_1 and t_2 are smaller on the concrete substrate than the values on the glass substrate if the same amount of adhesive per emittable area is applied.

Table 5.3 Weight Decay Ratio Comparison between the Concrete and Glass/Steel Substrate

Substrate	$\Delta W_r/W_0$	$\Delta W_g/W_0$	$\Delta W_{st}/W_0$	$\Delta W_{st}/W_0$
Glass	0.626 ± 0.016	0.615 ± 0.017	0.602 ± 0.020	0.600 ± 0.019
Concrete	0.694 ± 0.006	0.680 ± 0.006	0.661 ± 0.011	0.627 ± 0.018

Figure 5.14 Weight Decay Comparison between the Glass and the Concrete Substrate

Figure 5.15 Effect of Substrate on t_1 Figure 5.16 Effect of Substrate on t_2

Figure 5.17 Gas Chromatogram of the Adhesive on Glass Substrate

Figure 5.18 Gas Chromatogram of the Adhesive on Concrete Substrate

Figure 5.19 Nonane Concentration Decay Comparison

Figure 5.20 Decane Concentration Decay Comparison

Figure 5.21 Undecane Concentration Decay Comparison

Figure 5.22 TVOC Concentration Decay Comparison

The Gas Chromatograms of adhesive I on the glass substrate and the concrete substrate are given in figures 5.17 and 5.18. Three single compounds are selected to compare the concentration decay between the glass and the concrete substrate. The three compounds are nonane, decane and undecane. The molecular weights of the three compounds are 128, 142 and 156 respectively and represent the range from low to high molecular weights. The concentration decay comparisons of these three compounds between the glass substrate and the concrete substrate are shown in figures 5.19 –5.21. The TVOC comparison is shown in figure 5.22. The initial adhesive weights on the concrete and the glass substrates were 7.128 g and 7.613 g respectively. Although a larger amount of adhesive was applied on the concrete substrate, a lower TVOC concentration was found for the concrete substrate. It was also found that the concrete adsorbing levels to the different compounds were different. The concrete substrate showed a higher adsorbing ability to a lighter molecular weight compound. The concentration peak values of undecane were observed almost the same on both the glass and the concrete substrate whereas the concentration peak of nonane on the concrete substrate was 17% less than that on the glass substrate.

5.3.3 Comparison of Different Adhesives

There is a large variety of the adhesives available in the market and different adhesives behave differently because of different VOC contents in each adhesive. Not only the total emittable VOC amounts are different, but also the concentration decay trends and the emitted compounds are different. These bring difficulties to the mass transfer models in predicting the VOC concentration level in the office buildings. To achieve a comprehensive understanding of the VOC emissions from the adhesives, three different

adhesives were studied. As described in chapter 2, adhesive I and II are synthetic latex-based adhesive with different solvent contents and adhesive III is a so-called environmental-friendly adhesive.

The adhesives were applied on the 12cm×12cm glass substrates following the trowel application method. All tests were conducted in the 1.0m×0.8m×0.5m small-scale test chamber in which test conditions were well controlled. An electronic balance with a resolution of 1 mg was used to check the amount of adhesive applied on the substrate and to measure the adhesive weight decay data in the small test chamber. A hydrocarbon analyzer was used to measure the TVOC concentration values inside the test chamber. Multi-adsorbent tubes were used to collect the gas samples from the test chamber and the gas samples were analyzed by GC/MS/FID to identify and quantify the individual VOC compounds.

Figures 5.23-5.25 show the gas chromatograms of the volatiles emitted by these three adhesives. Gas chromatograph patterns presented by these adhesives were absolutely different. Lists of identified compounds for the three adhesives are given in table 5.4. Most compounds that are identified in adhesive I and II are middle range boiling point compounds that are typically used as solvents to control the drying time of the adhesives by the manufacturers. Because solvent contents in these adhesives were different, the TVOC concentration peak levels showed great differences as can be seen in figure 5.26. Adhesive III is the solvent-free adhesive and it showed a much lower concentration peak compared to the other two adhesives. Adhesive II which contains 5% solvent showed the highest concentration peaks among of the three adhesives tested in this study.

Figure 5.23 Gas Chromatogram of Adhesive I on Glass Substrate

Figure 5.24 Gas Chromatogram of Adhesive II on Glass Substrate

Figure 5.25 Gas Chromatogram of Adhesive III on Glass Substrate

Figure 5.26 Concentration Decay Comparison of the three Adhesives on Glass Substrate

Table 5.4 Major Identified Compounds in the Adhesives

Adhesive I	Adhesive II	Adhesive III
acetone	acetone	acetone
1,2,4-trimethyl- cyclohexane	acetic acid	1-butanol
nonane	3-ethyl-2-methyl-heptane	butyl ester acetic acid
3-methyl- nonane	octamethyl-cyclotetrasiloxane	acetic acid
propyl- cyclohexane	octanal	propylene glycol
3-ethyl-2-methyl-heptane	2,5,6-trimethyl-octane	1-ethyl-2-methyl-benzene
4-methyl-nonane	2,4,6-trimethyl-octane	benzaldehyde
2-methyl-nonane	3,7-dimethyl-undecane	1,2,4-trimethylbenzene
1-ethyl-1-methyl-cyclohexane	4,5-dimethyl-undecane	acetophenone
decane	nonane	nonanal
3-Ethyl-Octane	decane	decanal
(2-methylpropyl)-cyclohexane		
undecane		
naphthalene		
dodecane		
decanol		

From the comparison of the concentration decay in figure 5.27, it can be concluded that an effective way to reduce the concentration levels during the carpet-adhesive installation in large buildings is to reduce the solvent content in the adhesives. However, zero solvent content does not necessary mean the zero VOC concentration level because the solvent free adhesive III also showed about 2ppm concentration peak. This part of the VOCs may come from the adhesive material itself.

Figure 5.27 Weight Decay Comparison of the Adhesives on Glass Substrate

It also can be seen that these three adhesives showed different gas chromatograph patterns. The volatile compounds in the adhesive II are very difficult to be separated and they were just classified into several groups therefore. The volatile compounds in adhesive I and III were relatively easy to be separated. Although very few compounds were identified in adhesive III, some volatile compounds in adhesive III were found reacting with each other chemically instead of diffusing to the ambient directly. It is also

observed that peak of dodecane in figure 5.23 was very low in the initial period. However, the peak of dodecane began to increase relatively compared to other compounds after 20 hours. Dodecane has a relatively larger molecular weight and higher boiling point. It seemed quite “sticky” and stayed in the adhesive very stable whereas the concentration peaks of the molecules with lower molecular weights increased faster in the initial period and also decayed faster later. Thus dodecane would stay much longer than the compounds with lower molecular weights.

Table 5.5 shows the weight decay comparison of these three adhesives after 24 and 32 hours of application. Less mass from adhesive III was emitted and a relatively smaller standard deviation in weight loss was shown. Adhesive II showed a large weight decay rate in the slow emission period. A relative larger weight loss ratio from 24 hours to 32 hours after application was found (see figure 5.27).

Table 5.5 Weight Decay Comparison of the Three Adhesives

ADHESIVE	W_{24}/W_0	W_{32}/W_0	$(W_{24}-W_{32})/W_0$
I	0.602 ± 0.020	0.600 ± 0.019	0.002
II	0.726 ± 0.012	0.720 ± 0.011	0.006
III	0.791 ± 0.006	0.789 ± 0.006	0.002

Chapter 6

Conclusions and Recommendations

6.1 conclusions

Based on the experimental results obtained in this study, the following conclusions are drawn:

1. Three adhesive application methods were compared and the container method was found the best on the adhesive quantity control. About 2–3% standard deviation from the average adhesive amount in the container method tests was achieved whereas about 10% standard deviations from the average weights were achieved in the other two methods.
2. The characteristic times t_1 and t_2 increased with the initial adhesive weight normalized by the effective emitting area. In the range of the amount of adhesive applied in this study, the times t_1 and t_2 showed a linear relationship to the weight of the adhesive applied after the weight was normalized by the adhesive effective emitting area.
3. The values of t_1 and t_2 for samples prepared by the container tests are larger than those for samples prepared by the trowel method if the same adhesive amount per unit

effective emitting area is applied.

4. The concentration peak times of the TVOC and individual compound increased with the amount of the adhesive applied.
5. The VOC emission rates decreased initially probably due to the formation of the dry film on the adhesive surface or depletion of volatiles at the surface. This period increased with the thickness of the adhesive layer from about 30 minutes (0.4 mm) to 70 minutes (1.0 mm). The VOC emission rates then began to increase again to higher peak values probably because the VOC vapor broke through the dry film. After the emission rates re-rise to their peak values, they began to decay again.
6. The concrete substrate adsorbed a part of the VOC contents in the adhesive after the application of the adhesive and thus shortened the fast decay period.
7. If the same amount of adhesive is applied on the same effective emitting area, smaller t_1 and t_2 are expected on the concrete substrate than on glass substrate.
8. The degree of absorption by the concrete substrate of different volatile organic compounds are different. The concrete substrate showed a higher adsorbent ability to lower molecular weight molecules than to the higher molecular weight molecules.
9. An effective way to reduce the VOC emission concentration levels from the adhesives is to reduce the solvent content of the adhesive.
10. Different emission patterns were found from different adhesives. Attention needs to be paid to use test data from one specific adhesive to validate the emission models.

6.2 Recommendations

An inflection period in the concentration decay curve for the test sample prepared by the container adhesive application method was observed. It may indicate the formation of the

dry skin at the surface of the adhesive layer after application. Containers that are thicker and/or thinner than those used in this study can be used in the future to verify the formation time of the dry skin. Pieces of carpet can also be used to cover on the top of the adhesives in the containers to verify the general performance of the carpet-adhesive assemblies.

A container adhesive application method was developed to accurately control the amount of the adhesive applied on the non-absorbent substrate in this study. However, because of the difficulties in precisely processing the concrete substrate mechanically, there was not a method developed to accurately control the amount of adhesive applied on the concrete substrate. Farther work can be done in this area.

Appendices

A: Basic Theory of Gas Chromatography

Chromatography is a general term for separation process in which the components of a mixture are repetitively equilibrated between two phases; normally one of these phases is fixed or stationary, and the other is mobile. When the mobile phase is a gas, either a liquid or a solid can be utilized as the stationary phase; these processes are most precisely termed "gas-liquid partition chromatography" or simply "*Gas chromatography*".

A GC/FID has the following advantages:

- Responds to virtually all organic compounds
- Wide linear response range 10^7
- high sensitivity (minimum detectable quantity: 2×10^{-8} mg for alkanes)
- Independence of FID response from temperature, carrier gas, and flow rate
- Easy calibration for relative sensitivity for many components
- Simple operation

When mixed compounds come into a special long column carried by a carrier gas (usually Helium), each component comes out at a different time because of different physical characteristic. Figure A.1 shows the separation procedure for two components.

Figure A.1 Separation of Two Components

As can be seen in figure A.2, gas and liquid phases of one component co-exist in the column. If one component has greater solubility in the liquid phase than the other, it has the lower volatility and will stay in the column longer. Component B comes out later than component A because it has lower volatility than A.

Figure A.2 Gas and Liquid Phase in One Column

The time that one component stays in the column is called the retention time. The retention time of one component depends on how long a time the molecules of the component spend in the liquid phase relative to their time in the gas phase. If the molecules of one species spend less time in the liquid phase, the retention time will be shorter.

Figure A.3 Retention time of one component

Figure A.4 Effect of Column Efficiency on Resolution

Resolution is usually the primary chromatographic goal and depends on column efficiency (N_{eff}) and solvent efficiency α . The efficiency is demonstrated by the narrowness of the peaks and is measured by the number of plates, the height of a theoretical plate (HETP) and the number of plate meter. A plate is essentially the elution distance required for a component to leave and re-enter the liquid phase. N_{eff} is defined as: $N_{eff} = 5.54(RT'_x / W_{1/2})^2$

Separation factor is defined as: $\alpha = RT'_{xA} / RT'_{xB}$ (see figure A.3)

The Resolution of GC is calculated as: $R_{GC} = [k' / (k' + 1)] [(\alpha - 1) / \alpha] N_{eff}^{1/2} / 4$

Where $k' = RT'_x / RT_a$

Figure A.5 shows the separation of two components with different resolutions.

Figure A.5 Comparison of Different Resolution on Components Separation

Figure A.6 Typical Structure of a Typical GC

Figure A.6 shows the typical structure of a Gas Chromatograph. The detector identifies and quantifies the volatile compounds. A commonly used detector is the flame ionization detector (FID) as shown in figure A.7. Usually a FID uses H_2 as the flame gas. The H_2 is broken into hydrogen atoms. The hydrogen atoms crack the hydrocarbon molecules and ions are released. The response of the FID to the ions is linear with the number of the ions, which are proportional to the number of the hydrocarbon molecules. A sample stoichiometric process is shown below:

Figure A.7 Flame Ionization Detector

Figure A.8 shows a typical gas chromatogram.

Figure A.8 A Typical Gas Chromatogram

A table of identified compounds from figure A.8 is shown in table A.1. A Varian Star 3400 GC is used in this study. The structure and front panel of the Varian Star 3400 are shown in figure A.9 and A.10 respectively.

Table A.1 Identified compounds in Figure A.8

1. Carbondisulfide	14. Aniline
2. Diethylamine	15. 2-Ethylhexanol
3. n-Hexane	16. p-Dichlorobenzene
4. Methylcyclopentane	17. n-Undecane
5. Toluene	18. n-Dodecane
6. Ethylbenzene	19. Naphthalene
7. m/p-Xylene	20. Benzothiazol
8. o-Xylene	21. n-Tetradecane
9. Styrene	22. 4-Phenylcyclohexene
10. 2.2.4.6.6-Pentamethyl-3-heptene	23. n-Pentadecane
11. n-Propylbenzene	24. 1.2-Dihydro-2.2.4-trimethylquinolin
12. N.N-Diethylformamide	25. 2.6-De-tert.butyl-4-methylphenol
13. 2.2.6.6-Tetramethyl-4-methylidenheptane	26. n-Hexadecane

Figure A.9 Schematic Diagram of Varian Star 3400

Figure A.10 Front Panel of Varian Star 3400

B: Experiment Procedures

Test procedures are very crucial to test results. A good test procedure is the necessary condition to guarantee the good test repeatability and reproducibility. In this study, the following test procedures were followed at various stages in each test.

B.1 Test Material Preparation

A test specimen includes the testing adhesive and its substrate. The preparation of a specimen is associated with the preparation of adhesive and substrate. Three different kinds of substrates, glass, concrete and stainless steel, were used in this study. Because these three kinds of substrate have different physical characteristics, two different preparation procedures are required for them. This section introduces both the adhesive and substrate preparation procedures for test specimens.

B.1.1 Adhesive preparation

All the adhesives were sealed in large barrels when they arrived at the lab. However, only a very small amount of adhesive is needed in each test. Because of the effect of deposition in a larger volume, the adhesives may not distribute evenly in the barrels. In order to get identical adhesive samples in all tests and to avoid opening the barrels very often (this may lead to the escape of some VOC contents from the adhesive), the following procedure is developed:

1. Prepare a batch of clean glass jars of about 20 ml volume. The number of the glass jars depends on the number of associated with this kind of adhesive.
2. Open the adhesive barrel. Stir the adhesive quickly with a long trowel.
3. Fill the glass jars with adhesive.

4. Seal the adhesive barrel.

5. Seal the glass jars

* The adhesive samples in the glass jars should be rejected if not being used within two months (for safety consideration).

B.1.2 Substrate preparation

B.1.2.1 Glass Substrate and/or Stainless Steel Substrate

- 1) Use lab detergent to wash out the glass or steel substrates. (Toluene can be used first if necessary)
- 2) Rinse the substrate with clean water.
- 3) Dry up the substrate with low-lint lab tissue.
- 4) Put the substrate in the test room. Pre-condition it for 30 – 60 minute under the standard test conditions to let the temperature and relative humidity of the substrate reach the test conditions.

B.1.2.2 Concrete Substrate

- 1) Wash concrete substrate surface with clean water.
- 2) Dry up the concrete substrate surface with low-lint lab tissue.
- 3) Pre-condition the substrate for at least one week in test room before using.

B.2 Instrument Preparation

B.2.1 System Setup Verification

Check all the equipment to see if they are connected correctly (refer to figure 2.1 for the schematic diagram of system setup). Ensure no wrong connection is made. Check all the connection points to find any possible leakage. If no error is found, go to next step.

B.2.2 Equipment Calibration

As stated in chapter 4, all the equipment needs to be calibrated before using. Detailed calibration procedures can be found in chapter 4. Table 5.1 shows how frequently each should be calibration.

Table B.1 Recommended Calibration Period

Calibration	Period
Test Room Temperature	3 – 6 Months
Test Room Humidity	3 – 6 Months
Test Chamber Temperature	3 – 6 Months
Test Chamber Humidity	3 – 6 Months
Environmental Controller	3 – 6 Months
Fresh Air Flow Rate	3 Months
GC	6 Months
Hydrocarbon Analyzer	Every Test
Hot Wire Anemometer	2 – 4 Weeks
Air Velocity	6 Months
Mass Flow Controllers	3 – 6 Months

B.3 Test Procedure

B.3.1 Test Chamber Pre-conditioning

- 1) Turn on the test room air conditioning system. Adjust knob setting on the air conditioner according to corresponding calibration curve to let the room temperature be 23°C.
- 2) Turn on the test room heating system. Adjust knob setting in the test room to let the room temperature be 23°C.
- 3) Turn on the Nortec humidifier. Adjust knob setting according to corresponding calibration curve to let the room relative humidity be 50%.
- 4) Turn on the test room ventilation fan.

- 5) Pre-condition the room for at least 24 hours before any test to let it reach the test conditions.
- 6) Start the data acquisition software "VIEWDAC". Monitor the room temperature and relative humidity using "VIEWDAC".

B.3.2 Equipment Warming Up

- 1) Turn on the hydrocarbon analyzer. Let it warm up for 30 minutes.
- 2) Open the valve on the front of the dehumidifier to let the compressor work.
- 3) Turn on the environmental controller. Adjust the knob settings for temperature, relative humidity and air flow rate to their corresponding calibration curves and let the chamber temperature, relative humidity and air change rate be 23°C, 50 % and 1 ACH respectively. Let the environmental controller warm up for 30 minutes.
- 4) Turn on all data acquisition computers.
- 5) Restart the data acquisition software "VIEWDAC". Enter all calibration equations into their corresponding channels.
- 6) Turn on the Catalytic oxidizer and let it warm up for 30 minutes.
- 7) Open the valve for the distilled water supply.
- 8) Place the inner chamber to its right position and place the electronic balance into it.
- 9) Connect the balance signal line to its data acquisition computer.
- 10) Connect the power supply to the electronic balance and let it warm up for 30 minutes.
- 11) Seal the test chamber and connect the fresh air line, exhaust line and pressure line.
- 12) Turn on the power for mixing fan.
- 13) Start the exhaust pump.
- 14) Turn on the IFA intelligent flow analyzer and let it warm up for 30 minutes.

15) Turn on the power for all the mass flow analyzers

16) Calibrate the hydrocarbon analyzer. Refer to appendix C for details.

B.3.3 Specimen Preparation

1) Substrates pre-conditioning

Concrete substrate should be pre-conditioned in the well-controlled test room for at least one week to let the temperature and relative humidity of the concrete substrate reach the test conditions. Glass substrate should be pre-conditioned for at least one hour in the test room.

2) Applying Adhesives on the substrates

The step-by-step adhesive application methods were described in the chapter 4.

B.3.4 Specimen Loading

1) Place the test specimen on the electronic balance inside the test chamber.

2) Seal the side door of the chamber.

3) Record the initial time.

4) Start the balance controlling software.

5) Start the hot wire anemometer controlling software "ROOMAIR".

B.3.5 Sample Taking

1) Record the concentration value in Testing Chamber

Take the concentration reading on channel 5 on data acquisition software, VIEWDAC.

The reading is in ppm.

2) Determine the Sampling Volume

The maximum sampling capacity for the TVOC tube is 12 μg , with 10 μg sampling

amount recommended. If the sampling volume is Y(ml) and concentration in test chamber is X (ppm), the following relationship should be satisfied:

$$Y \text{ (ml)} * 10^{-6} (\text{m}^3/\text{ml}) * X (\text{ppm}) * 10^{-6} (\text{mol/ppm}) * W \text{ (g/mol)} * 10^6 (\mu\text{g/g}) / V (\text{m}^3/\text{mol}) = 10 \mu\text{g}$$

where W—molecular weight of calibration span gas, cyclohexane, 84.21 g/mol

V—standard volume of air per mole, which is 0.0243 m³/mol at 296K and

0.0246 m³/mol at 300K

thus, the sampling volume can be determined as:

$$Y = 10 * V * 10^6 / (W * X) \quad (\text{ml})$$

take V=0.0244 m³/mol and W=84.21 g/mol, the above equation can be simplified as:

$$Y = 2903.4 / X \quad (\text{ml})$$

Be careful that high sampling volume will cause the GC to crash.

3) Determine the Sampling Rate

In order to find the correct sampling rate, the sampling time t has to be decided first. The typical sampling time t is about 3-5 minutes (depending on the concentration level in the test chamber). The sampling rate Z is then decided as:

$$Z = Y / t \quad (\text{ml/min})$$

Note that ***the sampling rate should not exceed 250 ml/min.*** If the flow rate is too high, sampling time can be increased to reduce the sampling rate.

4) Sampling rate verification

Because of the effect of resistance of the long sampling tubing, the reading on the mass flow controller does not reflect the real sampling rate. The sampling rate needs to be

verified by another flow meter, the ADM3000 intelligent mass flow meter in the case. The flow rate control knob on the Matheson mass flow controller is to be adjusted to let the reading on the ADM3000 be the sampling flow rate wanted. Record the reading on the ADM flow meter.

5) Sample taking

Place the black charcoal side of the sampling tube in the sampling port. Connect the other end of the tube to the mass flow controller. Open the valve on the sampling port. Record the beginning time on the VIEWDAC. After the time t elapses, disconnect the end connected to the mass flow controller first. Then close the valve on the sampling port. Record the finishing time on the VIEWDAC. Take out the sampling tube from the port. Put the sampling tube in its container for further GC analysis.

6) Check the sampling flow rate again using ADM flow rate meter. Record the flow rate again.

7) Record the room temperature and relative humidity. (Read the temperature on VIEWDAC channel 6 and read the room humidity on VAISALA HMI 33).

8) Shut down the suction pump and Matheson mass flow controller if no other sampling is needed.

B.4 Post Processing

B.4.1 Equipment Shut Down

- 1) Close the valve in front of the dehumidifier.
- 2) Shut Down all suction pumps.
- 3) Turn off all mass flow controllers.
- 4) Turn off the hot wire anemometer.

- 5) Turn off the environmental controller.
- 6) Turn off the hydrocarbon analyzer.
- 7) Turn off the micromanometer.
- 8) Open the test chamber and unload the test specimen.
- 9) Shut down the air conditioning system, the heating system and the humidifying system if there is no continuing test.
- 10) Copy all data files to floppy disks and shut down all data acquisition systems.

B.4.2 Test Chamber Cleaning

The test chamber needs to be cleaned after each test to get rid of possible contamination and sink effects. To clean the test chamber, scrub the chamber with lab detergent first; then rinse the chamber with tap water; finally rinse the chamber with distilled water. If any visible adhesive is found attaching on the test chamber, toluene can be used to clean it first then above steps are followed.

B.4.3 GC Operating

- 1) Turn on the air and hydrogen gas cylinders and set the pressures to be 60 psig and 40 psig respectively.
- 2) Check the Helium and Nitrogen gas pressures. Ensure that the pressures should be 80 psig and 80 psig respectively.
- 3) Fully open the valves for air, hydrogen and nitrogen on the GC front panel.
- 4) Ignite the FID detector flame.
- 5) Start the controlling software "STAR".
- 6) Click the system control window and then open the method file. Modify the method file if necessary.

- 7) Under the system control window, open a sample list. Fill in the columns with sample names and types and then save the sample list.
- 8) Place sample tubes in the multi-tube desorber.
- 9) Activate the controlling software "STAR".
- 10) Enter the name of operator.
- 11) Enter the name of sample list.
- 12) Enter the name of testing method.
- 13) Press Start to begin.

B.4.4 Thermal Desorption Tube Cleaning

Although the Envirochem multi-tube desorber can remove almost all the VOC contents from the tubes, the Tenax thermal desorption tubes have to be cleaned again before reuse. This can ensure that VOC residual will not appear in the next sample. If the tubes are not being used for 3 months, they also need to be cleaned again before using them even if they have been cleaned already. The tube cleaning is done by the Envirochem multi-tube conditioner according to the following steps:

- 1) Turn on the Helium valve behind the Envirochem multiple tube conditioner.
- 2) Turn on the individual Helium valves on front of the tube conditioner. There are six valves corresponding to six tube holders. Only turn on the necessary valves.
- 3) Place the tubes to be cleaned in the holders. The charcoal side (black part) of each tube should face the bottom (holder).
- 4) Check the Helium flow rate going through each tube using ADM3000 flow meter. The proper flow rate should be 50-60 cc/min. If the flow rate is not in this range, adjust individual flow control valve to let the flow rate be in this range.

- 5) Turn on the main power.
- 6) After 20 minutes, place the heating jackets over tubes. Turn on individual power switch. Switch the dial on front panel and observe temperature of each tube on LED display. The temperature should be 275 ± 5 C°. If the temperature is not in this range, adjust individual power adjusting dial to let the temperature be in the range.
- 7) After 20 minutes, turn off the power for the heating jackets. Leave heating jackets on the tubes.
- 8) After 20 minutes, observe temperature for each tube. If the temperature has returned to initial temperature, remove its heating jacket.
- 9) Turn off the individual Helium valves.
- 10) Turn the main Helium valve off.
- 11) Remove the tubes and place them in individual sleeves.
- 12) Turn the main power off.

B.4.5 Data Processing

The test data post processing is associated with several processing software because several different data acquisition software are used in each test. The “VOCEMIT” is used to process the “VIEWDAC” data; the “STAR” is used to process the GC data; the balance data are processed by the “EXCEL”. The details of each processing software will be discussed in the following section.

B.4.5.1 VIEWDAC Data

The “VIEWDAC” records the data of time, test room temperature and relative humidity, test chamber temperature and relative humidity, chamber pressure, air exchange rate, velocity of air and total hydrocarbon in an interval of 30 seconds. Test data in one test day

will be saved in one file. It is not necessary to input all the test data into the data processing software such as EXCEL. The purpose of "VOCEMIT" is to process the original data file into several small files and in a larger time interval. Each small file is one parameter mentioned above versus time thus each parameter can be processed without affecting the other ones. Each small file then is input into EXCEL for further processing.

B.4.5.2 GC Data

The Gas Chromatograph uses its software "STAR" to control and collect test data. "STAR" measures all the VOC peaks and their corresponding retention time, calculates area for each peak and gives other necessary information. The major features are summarized in point form below. "STAR" must be used to process the data files it generates. For a more complete description of STAR software please refer to the software documentation.

- Control Envirochem multi-tube desorber
- Control Envirochem concentrator
- Control data acquisition board
- Control temperature scheme
- Control Gas Chromatograph
- Collect test data

B.4.5.3 Balance Data

Balance data files are saved as text files directly. They are processed by Excel afterward.

B.4.5.4 Excel

Excel is the main data processing software. After being processed by each corresponding

processing software, all data files become ASCII files and those ASCII files can be accessed by Excel. A benefit to using Excel is that it can do the mathematics processing and graph plotting of the test data and it can provide a uniform data format for easy storage and re-processing.

B.4.5.5 TABLE CURVE

“TABLE CURVE” not only can convert abstract test data into visual curves, but also can do the curve fitting to the test data. TABLE CURVE provides both linear and non-linear curve fitting techniques and it also supports user defined functions curve fitting for any special requirement. The following data use TABLE CURVE to find their best-fit equations.

- All temperature, relative humidity and air flow rate calibration data
- Hot wire anemometer calibration data
- Concentration calibration data
- All temperature, relative humidity and air flow, velocity and concentration test data
- Weight loss data

B.4.5.6 EASY PLOT

“EASY PLOT” is also a curve plotting software package. Not like TABLE CURVE which is more concentrating on curve fitting technique, “EASY PLOT” is more convenient to plot several curves on one graph so it is more convenient to do the curve comparison. It is a useful supplementary tool for TABLE CURVE.

B.4.5.7 SAS

SAS is an integrated system of software products that enables users to perform

- data entry, retrieval and management

- report writing and graphics
- statistical and mathematical analysis
- business forecasting and decision support
- operations research and project management
- applications development

In this particular study, a non-linear regression method is used to simulate a double exponential model.

C: Equipment Calibration

All equipment must be calibrated before being used because each piece of equipment has its accuracy and resolution. A discrepancy between the measured value and true value may occur, which may change with time and ambient conditions. Some equipment operates stably, so that once calibrated, it will give reliable readings for a quite long time. For example, the GC only needs to be calibrated every 3 months or so. However, some equipment needs to be calibrated very often. Calibration for each piece of equipment also can check if the equipment is working properly. This chapter will discuss the necessary calibration procedures that ensure all tests running properly.

C.1 Hot-wire Anemometer Calibration Procedure

C.1.1 IFA-100 Flow Analyzer Calibration

(I) Equipment Warming up

Turn on the power of the manometer and IFA 100 Intelligent Flow Analyzer. Let them warm up for 30 minutes. Record the initial offset reading of the manometer.

(II) Channel Choosing

Press **CHANNEL** on the IFA flow analyzer, and then select a channel on the number panel. Channel 3 should be selected.

(III) Resistance Measurement

1) Cable Resistance Measurement

Place a null resistance probe in the socket of the probe holder. Press **RES MEAS** and LED displays a reading; Use **OPERATE RES CONTROL** to adjust it to zero. Press **RES**

MEAS again and the LED will display the probe-cable resistance. Press **ENTER** to record the resistance value in memory.

2) Hot Wire Probe Resistance Measurement

Place the hot wire probe in the socket. Press **RES MEAS**; LED displays a reading; Use **OPERATE RES CONTROL** to adjust it to zero. Press **RES MEAS** again, the LED will display the probe resistance; the cable resistance is automatically subtracted.

3) Operating Resistance Setting

First, calculate the proper operating resistance. This resistance is related to the operating temperature. The recommended operating temperature by the manufacture is 250°C. Since the velocity levels in the test chamber are relative low, a lower operating temperature is selected, that is, 100°C -- 120 °C. The calculation of operating resistance is given in following equation:

$$R_{op} = \left(\frac{R_{100} - R_0}{100} \right) T_{op} + R_0$$

where R_{op} = probe resistance at the operating temperature

R_0 = probe resistance at 0 °C

R_{100} = probe resistance at 100 °C

T_{op} = operating temperature

The R_0 and R_{100} values are given by the manufacture on the case of the hot wire probe.

Press **OPERATE RES** and the operating resistance is displayed. Adjust the value on the display with **Operate Res Control** to obtain the desired operating resistance. It is not necessary to press **Enter** again.

Table C.1 Typical Setting of Bridge Compensation

sensor	type	standard 1	high power	1:1
-T1.5	wire	35		188
-P2	wire	83		
-P12.5	wire	115		
-P15	wire	120		
metal	clad	775		
-10A		70	25	
-10		70	28	
-20		115	40	1041
-60		250	85	

(IV) Bridge Compensation Adjustment

An appropriate bridge compensation value should be adjusted based on different probes used. Table C.1 shows the typical settings of bridge compensation. Press **BRIDGE COMP**. Adjust **BRIDGE** knob in the area of **FREQUENCY COMP** and let the reading be the correct value.

(V) Running

Press **RUN/STANDBY**. Now the transducer is ready to work. Press output display in the **SIGNAL CONDITIONER** frame. The magnified voltage is displayed on the LED.

C.1.2 Voltage Measurement

(I) Select a series of velocity that are wanted to be used in the experiment. Calculate the correspond pressure transducer reading (mm Hg).

(II) Adjust the speed of the wind tunnel and let the reading of pressure transducer to be the value wanted

(III) Start the data collecting software ROOMAIR. Choose collecting data at present location. Record the corresponding voltage readings. Table C.2 gives a sample data-

collecting sheet.

Table C.2 Hot wire probe calibration data sheet

Calibration of TSI hot wire/film probe					
- with #2 nozzle plate fitted (0.25" holes) ***					
name :		Xiangdong Li			
date:		97-02-07			
filename:		tsi1li			
model:		1210-T15			
serial:		935095			
TSI ref no.		121002			
Cable resistance :		0.461 Ω			
Hotwire resistance:		6.464 Ω			
Operating resistance :		8.36 (operating temperature: 100 °C)			
configuration:		long coax cable, bulkhead fitting, short cable with short probe holder			
Pressure:		761.2	mmHg		
Temp:		21.5	°C		
density correction factor: K =		0.99978			
transducer offset/zero reading :		0.018	mmHg		
sampling		204.8s @ 20 Hz			
standard velocity S (m/s)	standard pressure P(mmHg)	Desired P S * K (mmHg)	P corr. for trans zero (mmHg)	Output (roomair) (V)	#
standard velocity S (m/s)	standard pressure P (mmHg)	Desired P S * K (mmHg)	P corr. trans (mmHg)	Output (roomair) (V)	#
S (m/s)	P (mmHg)	(mmHg)	(mmHg)	(V)	point
1	5.32	5.319	5.337		12
0.75	2.94	2.939	2.957		11
0.6	1.87	1.870	1.888		10
0.5	1.28	1.280	1.298		9
0.4	0.799	0.799	0.817		8
0.3	0.44	0.440	0.458	4.5	7
0.25	0.302	0.302	0.320	4.4461	6
0.2	0.193	0.193	0.211	4.384	5
0.15	0.108	0.108	0.126	4.3118	4
0.1	0.048	0.048	0.066	4.237	3
0.05	0.012	0.012	0.030	4.1595	2
0.025	0.003	0.003	0.021	4.1265	1
0	0	0.000	0.018	4.1068	0

C. 1.3 Plot Calibration Curve

Save all readings into an EXCEL file. Then use TABLE CURVE to do the best curve fitting. Typical chosen calibration equations are power or polynomial equations. Figure C.1 shows a typical hot wire anemometer calibration curve.

Figure C.1 Hot Wire Anemometer Calibration Curve

C.2 Hydrocarbon Analyzer Calibration Procedure

C.2.1 Choosing of span gas and zero gas.

A typical zero gas is purified compressed air. Cyclohexane can be used as span gas in the hydrocarbon analyzer calibration. According to the range of expected concentration level in each test, different ranges of span gas should be chosen. The concentration of calibration span gas must be greater than the expected concentration level in the test chamber. Double check concentration tags on the cyclohexane cylinder. Inconsistent concentration tags may be posted.

C.2.2 Mass flow controllers Calibration

Two mass flow controllers, Matheson MFC8240 and Miller Nelson MFC 1113, will be used to control the zero and span gas flow rate in the hydrocarbon analyzer calibration process. These two controllers must be calibrated before they are used. The following steps were followed to calibrate the mass flow controllers:

- 1) Ensure the valves behind the transducers are closed.
- 2) Connect the zero gas cylinder to the zero gas transducer.
- 3) Connect the calibrated flow meter to the joint of zero gas and span gas.
- 4) Turn the controlling switch on MFC 8184 from “close” to “control” position.
- 5) Open the valve of zero gas and adjust the knob on the MFC 8284 to maximum (Around 500 ml/min). Record the corresponding voltage values on both MFC 8284 and voltmeter. Record the value on the calibrated gas flow meter.
- 6) Adjust the knob on MFC 8284 to decrease certain amount of flow rate. Record corresponding values.
- 7) Repeat procedure 6 until the flow rate reaches the minimum.
- 8) Turn the switch on MFC 8284 to “close” position.
- 9) Close the valve behind the zero gas transducer.
- 10) Switch the zero gas supply tubing to the span gas transducer.

During this calibration procedure, the zero and span gas will be vented to the room air. Normally the span gas is flammable and hazardous, so the potential danger of using span gas must be decreased as low as possible. Using zero gas instead of the span gas in the calibration procedure is recommended.

- 11) Open the valve behind the span gas transducer.

- 12) Adjust the knob on MFC 1113 to let the reading on the calibrated flow meter be 500 ml/min.
- 13) Record the reading of voltmeter, MFC 1113 (including the knob position) and flow meter.
- 14) Adjust the knob on MFC 1113 to decrease certain amount of flow rate. Record corresponding values.
- 15) Repeat 13 until the flow rate reaches the minimum.
- 16) Close the valve behind the span gas transducer.
- 17) Re-connect the tubing back to their normal positions.
- 18) Save the data into an Excel file.
- 19) Use TABLECURVE to find the best-fit curve. Figure C.2 and C.3 show two calibration curves for span and zero gases respectively.

Figure C.2 Miller Nelson Mass Flow Controller Calibration Curve (Span Gas)

C.2.3 Concentration calibration

The total flow rate of zero and span gas should be set to 500 ml/min. The hydrocarbon analyzer uses 250 ml/min of it and the other half is vented away. Although only 250ml/min flow is needed, the total flow rate cannot be set to be the exact 250 ml/min.

Figure C.3 Matheson Mass Flow Controller Calibration Curve (zero gas)

The reason is that the supply flow may fluctuate in a small range and the suction pump will keep sucking gas into the hydrocarbon analyzer at a rate of 250 ml/min. If the supply flow rate is less than 250 ml/min, a small vacuum will be formed. If the system is not perfectly sealed, the analyzer will suck in the ambient gas and thus cause an inaccurate measurement.

If the concentration of the span gas is x and a 0- y ppm calibration range is preferred, then at y ppm, the flow rate of span gas is $a=500*y/x$ ml/min and the zero gas flow rate is $b=500-a$ ml/min. Choose 0 ppm and y ppm as the beginning and end points and choose suitable points between them. (The following example is based on a 116 ppm

cyclohexane gas and a calibration range of 0-100 ppm)

- 1) Prepare the testing data sheet. Choose 3–5 testing points between 0 and maximum calibration range. Calculate corresponding zero gas and span gas flow rates. A sample data sheet is given in table C.3.
- 2) Turn on the power of the hydrocarbon analyzer. Wait for 20 minutes to let it warm up.
- 3) Open the valve of hydrogen cylinder. Adjust the diversion pressure to 45 psi.
- 4) Press the “**IGNITE**” button on the hydrocarbon analyzer. Hold for some seconds and release it. If the ignition light is off, it shows the flame is lit. Try again in case of failure of ignition. Wait 15 minutes to let the analyzer stabilize.
- 5) Turn on the power of zero gas and span gas mass flow controllers.
- 6) Switch range selection knob to the proper scale.
- 7) Check the connecting tubing from the gas cylinders to the mass flow controllers to see if they are connected properly.
- 8) Open the valves of zero gas cylinder and span gas cylinder.
- 9) Check if there is any leakage.
- 10) Start the “**VIEWDAC**” software. Set the computer in the data-collecting mode.
- 11) Switch “mode switch” on the hydrocarbon analyzer to “calibration” position.
- 12) Set the control switch on Matheson mass flow controller to “control” position.
- 13) Open the valve behind the zero gas transducer. Adjust the flow rate to maximum.
- 14) Adjust the zero gas adjusting knob on the hydrocarbon analyzer to let the reading of channel 5 on VIEWDAC be zero (The reading cannot stay very stable; if the reading fluctuates around zero, the setting is OK). The adjustment should allow the relative error be less than 0.1 %.

- 15) Open the span gas valve behind the span gas transducer.
- 16) Adjust the knobs on the mass flow controllers to let the flow rate of zero and span gases are “b” and “a” ml/min.
- 17) Adjust the span gas knob on the hydrocarbon analyzer to let the reading of channel 5 on VIEWDAC be 1.0.
- 18) Close the span gas valve behind the span gas transducer. Turn the switch on the Matheson mass flow controller to the position of “close”.
- 19) Repeat 12-18 one more time.
- 20) Starting from 0 ppm point, measure the voltage on channel 5 corresponding to each point. The measurement procedure should start from a lowest concentration point and increase gradually to the highest point.
- 21) Close the zero and span gas valves. Turn the switch on the Matheson mass flow controller to the position of “close”.
- 22) Close the valve of hydrogen cylinder. Wait until the ignition light on the hydrocarbon analyzer is off.
- 23) Close the valves of zero gas and span gas cylinders.
- 24) Stop the data collection of VIEWDAC.
- 25) Turn the power of hydrocarbon analyzer off.
- 26) Record the position of the knobs of zero and span gas on the hydrocarbon analyzer.
- 27) Use TABLECURVE to find the best-fit curve. Figure C.4 shows a hydrocarbon analyzer calibration curve.

Table C.3 Data Sheet for Hydrocarbon Analyzer Calibration

Hydrocarbon Analyzer Calibration Data Sheet						
Range 0-100 ppm Span Gas 116 ppm cyclohexane Zero Model MFC8240 S/N:85006 Span Model MFC1113 S/N:AA312504 span gas: 116ppm total amount after mixing:500ml/min amount used by hydrocarbon analyzer:250ml/min date: May 31, 1996 Amb Press 758.2mmHg						
concentration ppm	VIEWDAC reading V	%span	zero gas flow rate ml/min	span gas flow rate ml/min	Matheson reading V	Miller Nelson reading V
100	1	86.2069	68.965517	431.03448	0.6392229	1.10575802
80	0.7682	68.96552	155.17241	344.82759	1.5168686	0.89031679
60	0.54824	51.72414	241.37931	258.62069	2.3945143	0.67487556
40	0.34179	34.48276	327.58621	172.41379	3.27216	0.45943433
20	0.14328	17.24138	413.7931	86.206897	4.1498058	0.2439931
0	0	0	500	0	5.0274515	0.02855187

Figure C.4 Hydrocarbon Analyzer Calibration Curve (0–100 ppm)

C.3 Temperature and Humidity Calibration

C.3.1 Room Temperature and Humidity Calibration

As mentioned in chapter 3, the temperature control in the test chamber depends on the temperature control inside the test room and the humidity level in the test room will also affect the initial chamber humidity level. The condition control inside the test room thus turns out to be important. A chilled mirror temperature and humidity probe was used to calibrate the temperature and humidity in the test room. The following procedure was used.

- 1) Turn on the power for the air conditioning system, humidity control system and heater. Pre-condition the test room for 24 hours.
- 2) Turn on the power for the Meloy environmental controller; let it warm up for 30

minutes.

- 3) Start the "VIEWDAC" software to start the data acquisition system.
- 4) Reset the calibration equation on channel 1 of VIEWDAC to " $y=x$ ".
- 5) Reset the calibration equation on channel 2 of VIEWDAC to " $y=x$ ".
- 6) Place the chilled mirror temperature and humidity sensor near the temperature and humidity sensor of the environmental controller.
- 7) Adjust the knob setting of the temperature control to select a series of temperatures which is wanted to be calibrated. Adjust the air conditioning setting to the corresponding settings to keep the room temperature as stable as possible. The time interval between testing points should be greater than 2 hours.
- 8) Record the knob reading and corresponding chilled mirror temperature reading and VIEWDAC output on channel 1 for each temperature setting.
- 9) Plot the calibration curve of knob setting versus temperature and the curve of VIEWDAC reading versus temperature. Figure C.5 shows the room temperature calibration curve of the VIEWDAC.
- 10) Adjust the knob setting of the humidity control to select a series of humidity that is wanted to be calibrated. The time interval between testing points should be greater than 2 hours.
- 11) Record the knob reading and corresponding chilled mirror humidity reading and VIEWDAC output on channel 2 for each humidity setting.
- 12) Plot the calibration curve of knob setting versus humidity and the curve of VIEWDAC reading versus humidity. Figure C.6 shows the room humidity calibration curve of VIEWDAC.

Figure C.5 Test Room Temperature Calibration Curve for VIEWDAC

C.3.2 Chamber Humidity Calibration

- 1) Open air supply valves in front of the dehumidifier to let compressor start to work.
- 2) Turn on the power of catalytic oxidizer. Let it warm up for 30 minutes.
- 3) Turn on Meloy environmental controller. Let it warm up for 30 minutes.
- 4) Place chilled mirror temperature and humidity sensor near the temperature and humidity sensor of the chamber.
- 5) Seal test chamber. Connect fresh air tubing, exhaust air tubing and pressure line to test chamber.
- 6) Turn on the power of chamber mixing fan to allow a good mixing inside the chamber.
- 7) Start "VIEWDAC" software to start the data acquisition system.
- 8) Reset calibration equation on channel 2 to "y=x".

Figure C.6 Test Room Relative Humidity Calibration Curve for VIEWDAC

- 9) Adjust the humidity control knob on Meloy environmental controller to select a set of humidity. Record the knob readings on the controller and corresponding readings on chilled mirror humidity sensor and "VIEWDAC" channel 2.
- 10) Plot calibration curve of VIEWDAC reading versus Humonics reading and the curve of Meloy knob reading versus Humonics reading.
- 11) Enter the calibration equation of VIEWDAC output versus flow rate into channel 5 on VIEWDAC.

C.4 Air Flow Rate Calibration

To calibrate the air flow rate into the test chamber, the Humonics bubble flow meter is required because of the large flow rate. Typical flow rates are from 0.5 ACH to 1.5 ACH.

In this particular study, the airflow rate was fixed to 1 ACH that is equivalent to 6.67 l/min.

- 1) Open the air-supply valves in front of the dehumidifier to let compressor start to work.
- 2) Turn on the power of catalytic oxidizer. Let it warm up for 30 minutes.
- 3) Turn on the Meloy environmental controller. Let it warm up for 30 minutes.
- 4) Start the "VIEWDAC" software to start the data acquisition system.
- 5) Reset the calibration equation on channel 3 to " $y=x$ ".
- 6) Disconnect air supply tubing to the test chamber and reconnect it to the Humonic bubble flow meter.
- 7) Adjust the mass flow control knob on Meloy environmental controller to select a series of flow rate. Record the knob readings on the controller and corresponding readings on Humonics flowmeter and "VIEWDAC".

Figure C.7 Flow Rate Calibration Curve for VIEWDAC

- 8) Plot the calibration curve of VIEWDAC reading versus Humonics reading and the curve of Meloy knob reading versus Humonics reading.
- 9) Select the flow rate wanted and find corresponding knob setting on the environmental controller. Adjust the knob to set the correct flow rate.
- 10) Enter the calibration equation of VIEWDAC output versus flow rate into channel 5 on VIEWDAC.
- 11) Figure C.7 shows a calibration curve of VIEWDAC reading versus flow rate.

D: Equipment List

Function	Equipment	Model	Part No.	Remarks
C h e m i c a l A n a l y s i s	Gas Chromatograph	Varian Star 3400CX	18057	Carrier Gas: Helium, 2.5ml/min @ 80psi Makeup Gas: Nitrogen, 27.5ml/min @ 40psi Flame Gases: Hydrogen, 32.5ml/min @ 45psi Air, 327.5ml/min @ 60psi
	Multi-tube Desorber	Envirochem 8916	T-1028	16-tube capacity
	Concentrator	Envirochem 810X	268242	
	Multi-tube Conditioner	Envirochem	NRC 37430	6-tube capacity
C o n t r o l S y s t e m	Environmental Controller	Miller-Nelson HCS-301	83110	Controlling temperature humidity and air flow rate in test chamber
	Air Conditioner	Neslab EN-850	NRC 083996	Controlling temperature in test room
	Heater	LP3E 1/4	N/A	Controlling temperature in test room
	Fan	LP3E 1/2	N/A	Controlling ventilation in test room
	Humidifier	Condair Res D200	N/A	Controlling humidity in test room. 50%±5% is desired
	Suction Pump	CAST DOL-101-AA	0782	Controlling pressure in test chamber. 10±5 Pa is desired
M e a s u r i n g D e v i c e s	Hydrocarbon Analyzer	Meloy HCA500-2C	8A165	500ml/min sample air is sucked in & 250ml/min is used for analysis; measuring TVOC in test chamber
	Hot Wire Anemometer	TSI 1210-20	935007	Measuring velocity over the surface of test specimen
	Temperature and Humidity Sensor	Vaisala HMI33	833863	Sensing the temperature and humidity in the test room
	Temperature and Humidity Sensor	Vaisala HMP 23 UT	823081	Sensing the temperature and humidity in the test chamber
	Micromanometer	Oxford Ap270S	NRC 084904	Measuring pressure in test chamber
	Electronic Balance	Sartorius LC 621S	51105747	Measuring the weight loss of specimen in the test chamber

Function	Equipment	Model	Part No.	Remarks
Data Acquisition	Measurement and Control System	Keithley 500A	NRC 037430	Collecting signals of temp., relative humidity, air flow rate, pressure and air velocity data in test chamber; also collecting temperature and humidity data in the test room. All the signals are sent to the data acquisition software—Viewdac.
	Computer	Dell 466 VC5	307B5724	Running data acquisition software—Viewdac and saving signals to corresponding files
	Flow Analyzer	TSI IFA-100	NRC 104778	Collecting velocity signals from hot wire probe and transfer it to Keithley and another computer
	Computer	NHC 286	MC-01030-806	Running the software controlling hot wire and its step motor and saving velocity data files
	Computer	IBM AT 5170	AN09SA	Data collection for electronic balance
	Computer	Digital Venturis 5166	NRC 210870	Running GC Star software and collecting GC data
Calibration	Chilled Mirror	Vaisala	MA 410440 probe: 315449	For temperature and relative humidity calibration
	Flowmeter	ADM3000	400-0076	For flow rate calibration Range: 5-750ml/min
	Wind-tunnel	TSI	Ref. 12100	For hot wire anemometer calibration
	Manometer	DP Measurements	NRC 037487	For hot wire anemometer calibration
	Bubble Flowmeter	Humonics	005728	For fresh air flow rate calibration Range: 0.5-10L/min
	Mass Flow Controller	Matheson #240	85006	Controlling zero gas flow rate
	Mass Flow Controller	Miller Nelson SFC-1113	82091	Controlling span gas flow rate
	Multimeter	HP 3468A	2137A04-291	output voltage from zero gas mass flow controller
	Multimeter	HP 3468A	2137A03-634	output voltage from span gas mass flow controller
	Transducer	Matheson 8141	BA406122	Transducing zero gas flow rate to voltage signal Range: 0-500ml/min
	Transducer	Tylan FC-260	AA312504	Transducing span gas flow rate to voltage reading Range: 0-2000ml/min
	Gas Producer	Matheson 6184 T4 FF	N/A	Only allowing gas to pass by the zero gas transducer
	Gas Producer	Matheson 6164	N/A	Only allowing gas to pass by the span gas transducer

Function	Equipment	Model	P/N	Remarks
Air & Water Supply System	Compressor	Magnatek HH2H017 M80218	52E7936-8A	compressing fresh air Max pressure: 100psi Capacity: 1.4 cfm
	Water Purifier	Barnstead 04511	5839308-03128	Purifying the supply water to the test chamber
	Dehumidifier	N/A	N/A	Dehumidifying the fresh air before going into environmental controller
	Pressure Regulator	Xenia	R11-200-KNL	Controlling the pressure of supplying air
	Catalytic Oxidizer	MSA model B	NRC 198067	Purifying and de-ionizing air before going into test chamber
Sample Taking	Mass Flow Controller	Matheson 8280	96014	Controlling the sampling rate
	Suction Pump	CAST DOA-V111-AA	782	Sucking in sample air
	Transducer	Matheson 8272-0432	AU95100-22	Converting sampling flow rate to voltage signal
	Sampling Port	N/A	N/A	Supporting thermal desorption tubes
Gas Cylinders	Compressed Air Cylinder	N/A	N/A	Highly purified Flame gas for GC/FID detector
	Nitrogen Cylinder	N/A	N/A	Makeup gas for GC/FID Highly purified
	Hydrogen Cylinder	N/A	N/A	Flame gas for GC/FID detector Highly purified
	Helium Cylinder	N/A	N/A	Carrier gas for GC Highly purified
	Compressed Air Cylinder	N/A	N/A	Zero gas for Meloy hydrocarbon analyzer; highly purified
	Cyclohexane Cylinder	N/A	N/A	Span gas for Meloy hydrocarbon analyzer; 116ppm
	Hydrogen Cylinder	N/A	N/A	Flame gas for hydrocarbon analyzer detector; highly purified
Other Accessories	Fan Motor	Kepco	NRC 037375	range: 0-25 V DC
	Power Supply	ATE 2-4M		
	Fan Motor	Maxon 2332.96-8-12.2.6-200	N/A	Spinning the mixing fan in test chamber
	Adhesive Spreader	Richard 1/8x1/8x1/8	N/A	Spreading adhesive over the surface of substrate
	Adhesive Applicator	Mitutoyo	N/A	Applying adhesive with a controlled thickness
	Digimatic Indicator	Mitutoyo DP-1HS	5551454	Measuring the thickness of specimen and adhesive layer

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IMAGE EVALUATION TEST TARGET (QA-3)

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