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MATERIAL ODOUR EMISSION TEST METHODS: REVIEW AND EVALUATION

R E P O R T

Material Odour Emission Test Methods: Review and Evaluation

Final Report to:	Task Force on Materials Emissions c/o Canada Mortgage and Housing Corp. 700 Montreal Road Ottawa, Ontario K1A 0P7
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SUMMARY

This materials odour emission project was conducted for the Task Force on Materials Emissions and four industry sponsors. The objectives of the project were to review material odour test methods, conduct comparative tests of various materials by the most applicable methods and to recommend test methods for materials odour emission characterization. The review identified six test methods which were then compared in a program of odour testing of 10 materials. Two test methods were quantitative odour intensity methods and four methods used perceived intensity and hedonic scales. The materials tested were those considered typical indoor sources of volatile organic compounds (VOCs) and with weak to potentially high odours. These materials included resilient flooring, carpet, counter top, office partition panels, painted trim and plywood. TVOC emissions tests were also performed on the 10 materials using a small dynamic chamber.

The six odour evaluation methods generally were in agreement in identifying the high, moderate and low odour emitting materials. The odour threshold or butanol reference methods could both be used as quantitative materials odour emission evaluation methods. A simpler method which used multi-point perceived intensity and hedonic scales was the preferred non-quantitative method. There was no relationship between odour strength/intensity and TVOC emissions.

SOMMAIRE

Cette recherche sur l'émission d'odeurs par les matériaux a été réalisée pour le Groupe de travail sur les normes d'émission des matériaux de construction et pour quatre parrains de l'industrie. Elle avait pour but de passer en revue des méthodes d'essai visant à quantifier les odeurs émises par les matériaux, de mener des essais comparatifs sur divers matériaux à l'aide des méthodes les plus applicables et de recommander des méthodes d'essai devant servir à la caractérisation des odeurs émises par les matériaux. L'examen a permis de relever six méthodes d'essai que l'on a comparées dans le cadre d'un programme d'analyse des odeurs produites par 10 types de matériaux. Deux méthodes quantifiaient l'intensité des odeurs et quatre méthodes étaient fondées sur l'intensité perçue et sur des échelles hédoniques. Les matériaux mis à l'essai étaient ceux que l'on considère habituellement comme des sources de composés organiques volatils (COV) en milieu intérieur et dont la production d'odeurs peut varier de faible à élevée. Mentionnons par exemple un revêtement de sol résilient, une moquette, une surface de comptoir, des panneaux servant de cloisons dans les bureaux, une menuiserie de finition peinte et du contreplaqué. Des essais visant à déterminer la concentration totale de COV (COVT) ont aussi été réalisés sur les 10 matériaux au moyen d'une petite chambre dynamique.

Les six méthodes d'évaluation des odeurs ont généralement permis d'obtenir des résultats similaires quant au degré d'émission d'odeurs des matériaux (élevé, modéré, faible). Les méthodes à seuil olfactif ou à substance de référence (butanol) pouvaient toutes deux être employées comme méthodes d'évaluation quantitative des émissions d'odeurs par les matériaux. Une méthode plus simple consistant à recourir à l'intensité perçue à plusieurs endroits et à des échelles hédoniques a été considérée comme la méthode non quantitative de prédilection. On n'a constaté aucune relation entre la force ou l'intensité de l'odeur et les émissions de COVT.

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1. BACKGROUND INFORMATION

In 1995 the Task Force on Materials Emissions (TFME) invited proposals for jointly funded emissions research. ORTECH responded to this request by submitting a proposal for a project to review and evaluate test methods for the characterization of odour emissions from materials. Currently employed material emissions testing techniques are used to quantify and characterize volatile organic compound emissions based on the chemical analysis of the chamber air samples obtained. Sampling and analysis is conducted by standard indoor air methods which employ collection on tenax or multi-adsorbent (Carbotrap) tubes with subsequent thermal desorption and analysis by a combination of GC/FID or GC/MSD. The data from this chemical characterization of VOCs has been used successfully to rank emissions from materials, determine which sources are contributing to indoor air contaminants and assist material manufacturers in reducing emissions from their products. The chemical data can also be compared to odour threshold databases to evaluate the potential odour emissions from the materials. However, even though odours are considered an important issue of indoor air quality, there are no widely practiced methods for determining odour emissions from materials.

This submission on evaluating and reviewing materials odour test methods was approved and this report presents the activities and findings of the project. In addition to the partial funding provided by the TFME, additional funding was provided by Armstrong World Industries, Interface Flooring, Teknion and ORTECH.

2. PROJECT TERMS OF REFERENCE

The objectives of the project were:

i) To review and present odour evaluation techniques which are applicable to materials emissions,

ii) To conduct comparative testing of various materials by the most applicable methods, and

iii) To recommend test methods for materials odour emission characterization.

3. ODOUR EVALUATION TECHNIQUES

A variety of approaches have been developed to address the problem of evaluating odours and odour nuisances. These approaches are normally tailored to a specific type of odour problem, for example, the assessment of the potential impact of an odorous air emission on the relative acceptability of a material or product. A number of different odour "dimensions" may be quantified and there may be several ways of determining a given "dimension". Some examples are as follows:

Odour Dimension	Measurement Approach
Detection Threshold	• Static dilution, triangle forced choice • dynamic dilution, triangle forced choice • dynamic dilution, free choice • extrapolation from butanol referencing data
Other Threshold	• recognition threshold • complaint threshold
Perceived Intensity	• magnitude estimation scaling • category scaling • butanol referencing olfactometer
Hedonic Scale	• category scaling, verbal categories • category scaling, visual categories
Odour Character	• standardized descriptor profiling • direct comparison profiling

These examples are by no means exhaustive, and even within a given measurement approach there may be a number of very different implementations. Dilution to threshold approaches have been used widely for air pollution issues, and for the determination of detection thresholds of pure compounds. The design of olfactometers, including flowrates, presentation method, number of panelists, panelist selection and a number of other variables have been, and continues to be the subject of much research and debate. Within the intensity and hedonic scaling methods there are a number of different methods, many tailored to specific materials or products, and often requiring special preparation of the samples before evaluation.

Despite the variety in the approaches, and the general debate over methods, sensory assessment techniques have found a place in a variety of fields and are used on a routine basis to provide useful data which cannot be supplied by chemical analysis. The variation in response between individuals in a population need not be a hindrance to a reliable evaluation - indeed, certain methods are predicated on this. The utility of odour evaluations is that they focus on the key issue - sensory nuisances are best evaluated by sensory means.

The review of odour evaluation techniques involved on-line searches of databases, and accessing in-house odour evaluation technology and project files. From this review, the following methods were identified as the most applicable to materials odour emissions.

3.1 Methods ASTM C665 / ASTM C739

ASTM C665 and ASTM C739 are general test methods for evaluating the performance of mineral-fiber blanket (C665) and cellulosic fiber loose fill (C739) thermal insulation[1,2]. Both methods have similar clauses which outline methods for evaluating odour emissions. Prior to evaluating the odours, the samples are conditioned for 24 hours and then heated to 65°C for 30 minutes. The odours are then evaluated according to 3 ranking scales. The methods do not specify whether the panel members are trained or untrained.

3.2 Method SAE J1351

SAE J 1351 is a test method to evaluate and compare the odour characteristics of various automotive trim, insulation materials and composites[3]. Prior to evaluating the odours, the samples are conditioned for 24 hours and then heated to 65°C for 30 minutes. The odours are then evaluated according to one 5-point ranking scale. The method does not specify whether the panel members are trained or untrained.

3.3 Automobile Interior Fitting Ranking

This method is used by an automobile manufacturer to evaluate the odour emissions from interior fitting components and parts. Samples of the materials are placed in Tedlar bags, evacuated with nitrogen, heated in an oven for one hour, cooled and then diluted with nitrogen prior to the odour evaluation. The odours are then evaluated according to two ranking scales. The method does not specify whether the panel members are trained or untrained.

The scores were then plotted on a two-axis chart and the score compared to criteria areas (Good, Acceptable, Unacceptable) of the chart. An example of this chart is presented in Figure 1.

3.4 Danish Indoor Climate Labeling (DICL) Scheme

DICL is a scheme for labeling building products according to their impact on the indoor air quality which takes into account the emission decay of volatile organic compounds from new building materials[4]. A time value is determined which is a measure of the duration in which a new material may cause increased exposure and enhance the probability of increased indoor air quality problems. The chemical chamber decay data is evaluated to determine when a given VOC is below a threshold based on odour and mucous membrane irritation. This threshold is presently determined based on chemical emissions testing. Sensory testing is also conducted as a check to validate the threshold time value predicted by the chemical emissions testing.

Odour samples are obtained from small chamber tests in which material off-gases are collected in Tedlar or Rilsan bags and then presented to an untrained panel for evaluation. The odours are evaluated using the following two ranking scales:

1 Odour Intensity.................................. 0 to 5
2 Odour Acceptability......................... -1/0/1

If the odour intensity is greater than 2 and the acceptability is less than 0, then the sensory tests determine the time value for the material and not the chemical emission predicted time value.

3.5 Odour Threshold Method

The determination of the odour detection threshold of the material off-gas samples was by means of dynamic dilution olfactometry using a nine member odour panel. The samples were collected in Tedlar bags from a small dynamic materials off-gas chamber. Panelists are considered to be representative of the normal population.

3.6 Butanol Reference Method

The intensity of a gaseous sample is assessed by matching the intensity to concentrations of a reference odorant, n-butanol.

4. COMPARATIVE TESTS OF ODOUR EVALUATION METHODS

4.1 Test Procedures

The test program involved evaluating up to 10 materials by six odour evaluation techniques plus a small chamber VOC emission test. The materials included resilient flooring, carpet, counter top, office paneling, painted trim and plywood. These materials were considered typical indoor sources of VOCs and odours.

4.1.1 Methods ASTM C665 / ASTM C739

Samples were evaluated with a five-member panel. The panel members were drawn from a pool of people who routinely participate in olfactory evaluations and have been screened for odour sensitivity.

Samples were initially conditioned at 21°C and 50% RH for 24 hours. After conditioning each sample was placed in a stainless steel container, closed and placed in an oven at 65°C for a period of 30 minutes. The panel of five members then open the containers and sniff the odours in the test containers. The odours were evaluated using the following three ranking scales:

1 Odour present........................ Yes/No
2 Odour Quality........................ Objectionable/Pleasant/Neutral
3 Odour Strength..................... Weak/Strong/Very Strong

4.1.2 Method SAE J1351

Samples were evaluated with a five-member panel. The panel members were drawn from a pool of people who routinely participate in olfactory evaluations and have been screened for odour sensitivity.

Samples were initially conditioned at 21°C and 50% RH for 24 hours. After conditioning, each sample was placed in a stainless steel container, closed and placed in an oven at 65°C for a period of 30 minutes. The panel of five members then open the containers and sniff the odours in the test containers. The odours were evaluated using the following ranking scale:

Odour Ranking:

1 No noticeable odour
2 Slight, but noticeable odour
3 Definite odour, but not strong enough to be offensive
4 Strong offensive odour
5 Very strong offensive odour

4.1.3 Automobile Interior Fitting Ranking

Samples were evaluated with a five-member panel. The panel members were drawn from a pool of people who routinely participate in olfactory evaluations and have been screened for odour sensitivity.

Samples (100 mm x 100 mm) were placed in 3 litre Tedlar bags and the bags sealed. The bags were evacuated and then two (2) litres of nitrogen injected into the bags. The sample bags were placed in an oven at 80°C for a period of one hour. The sample bags were then cooled to room temperature (22°C). The air from the sample bags was then diluted into other 3 litre bags with nitrogen (ratio of nitrogen to sample bag air is 11:1).

Each panel member evaluates each sample by opening a valve on the bag and sniffing the contents. The odours were evaluated using the following two ranking scales:

a)	**Odour Strength**	*1*	Not perceivable
		2	Barely Perceivable
		3	Perceivable
		4	Easily Perceivable
		5	Strong
		6	Extremely Strong
b)	**Odour Discomfort**	*-4*	Excellent
		-3	Very Good
		-2	Good
		-1	Slightly Good
		0	Average
		1	Slightly Bad
		2	Bad
		3	Very Bad
		4	Extremely Bad

The scores were then plotted on a two-axis chart and the score compared to criteria areas (Good, Acceptable, Unacceptable) of the chart.

4.1.4 Danish Indoor Climate Labeling (DICL) Scheme

A dynamic small chamber materials emission testing system was used to deliver odour samples into the tedlar bags. Odour samples were taken after the materials had been in the small chambers for 24 hours. Panel member were asked to sniff the undiluted odour samples and report whether an odour was or was not detected.

Samples were evaluated with a five-member panel. The panel members were drawn from a pool of people who routinely participate in olfactory evaluations and have been screened for odour sensitivity.

4.1.5 Odour Threshold Method

The odour samples were evaluated within 24 hours after collection into Tedlar bags using the dynamic dilution olfactometer in ORTECH's odour test facility. The odour test facility is a specialized room designed to provide an odour-free environment for accurate evaluations. A dynamic small chamber materials emission testing system was used to deliver odour samples into the tedlar bags. Odour samples were taken after the materials had been in the small chambers for 24 hours.

The olfactometer is a binary port system operated in a non-forced choice mode, and is shown schematically in Figure 2. The sample bag was pressurized in a pressure vessel, and the resulting flow was metered through an electronic mass flow controller at a predetermined rate. The sample was diluted with flow-controlled odorless air, and was passed to the panel members through one of two sample ports. A three-way valve allowed the operator to direct the sample through either of the two ports.

Each evaluation began at a high dilution level, which was lowered in a step-wise sequence by a factor of 1.41 at each step. At each dilution level, the panelists registered their responses by entering, on a micro terminal, the letter of the port at which they detected the odour. The range of dilution ratios of ORTECH's odour test facility is from 5,793 times to 8 times.

The panelist responses were processed by an IBM-PC based data acquisition system, which determined the odour threshold value for the sample. This was done by a regression analysis of the log of the dilution level versus the probit value of the percent of the panel responding. The point at which statistically 50% of the panel could just detect the odour was recorded as the ED_{50} (effective dilution to 50% response) or the odour threshold value (OTV). The odour threshold value is a dilution factor and therefore has no units. For convenience, however, the OTV may be expressed in odour units (ou).

A nine-member panel was used for all evaluations. They were drawn from a pool of people who routinely participate in this type of work. They have all been tested for odour sensitivity and are considered to be within the normal range.

4.1.6 Butanol Reference Method

Off-gas samples were collected from a small dynamic materials emission chamber. Samples were collected in 30 litre Tedlar gas sample bags. A dual port referencing system was used, whereby the sample was metered through one sample port, and a variable concentration of butanol was metered through the other. Flowrates through both ports were the same, 5 litres per minute. The air sample was delivered from a pressure vessel, to minimize loss or contamination by pumps. The butanol reference flow system consisted of a calibration gas mixture of 10 ppm butanol in nitrogen and a gas blender with two mass flow controllers to allow metering of butanol mixture with clean air.

The operating procedure involved sniffing the sample stream, then sniffing the butanol stream. The concentration of butanol was gradually increased until the intensities were considered to be the same. This was done in a series of steps, taking precautions to prevent olfactory fatigue. Once the intensities were considered similar, the operator rested for about one minute, and then compared the odours again, and adjustments were made to the butanol concentration, if necessary. The concentration of butanol, in ppm, was then recorded. This value was reported as the odour intensity.

4.1.7 Materials VOC Emissions Tests

Materials off-gassing tests were conducted using the ASTM document "Standard Guide for Small-Scale Environmental Chamber Determinations of Organics from Indoor Materials/Products, D5116-90" as a guide[5]. The flow through the chamber was set at 0.5 airchanges per hour. Temperature of the chamber was at ambient temperature, 23°C ± 1°C, and humidity was 40% ± 5% RH.

The material emissions test method also requires the calculation of a materials loading ratio (m^2/m^3), which is the ratio of the test specimen area to the chamber volume. For the carpet, resilient flooring and plywood the loading ratio was 0.41 m^2/m^3. For the painted trim and the counter top the loading ratio was 0.15 m^2/m^3. For the office panel the loading ratio was 0.85 m^2/m^3.

The samplers were received at ORTECH in their wrapping and appropriately labelled. Samples were unwrapped and cut to the size required for the loading ratios. Prepared specimens were placed in the chamber and conditioned for 24-hours prior to emissions testing. Testing consisted of a combination of collection of chamber air on Carbotrap multi-adsorbent tubes with thermal desorption and GC/MS VOC analysis and collection in a sodium bisulphite solution and analysis by the chromatropic acid method for formaldehyde. Total volatile organic compound emissions are the sum of the VOC analysis and the formaldehyde analysis.

5. COMPARATIVE TEST RESULTS AND DISCUSSION

The results of the comparative tests are summarized in Table 1. Materials are identified by number and not by material type (ie,. carpet, plywood etc.). The purpose of the study was to evaluate odour evaluation methods, not to determine odour emission characteristics of materials. Only 10 samples of six materials were tested and the results are only for the samples tested and are not representative of the material types.

The six odour evaluation methods generally were in agreement in identifying the high, moderate and low emitting materials. Quantitatively, odour thresholds of greater than 10 ou/m^3 were associated with materials with strong, objectionable or offensive odours. Materials with odour thresholds at the detection limit of 8 ou/m^3 exhibited perceived moderate odours which were not as objectionable. Materials with odour thresholds of less than 8 ou/m^3 exhibited weak odour strengths. These material odour threshold trends are an agreement with odour threshold trends for community nuisance odours. There was no apparent relationship between odour strength/intensity, odour thresholds and TVOC emission values (see Appendix A).

For the methods which used perceived intensity scales and hedonic scales to rank the odour emissions (DICL, auto trim, ASTM C739 & C665, SAE J1351) variations occured in the odour ranking. This variation can be attributed to the different sample preparation and conditioning requirements employed prior to the sensory evaluations. Ranking scales also vary from 3 point to 9 point.

Methods which involve heating and no dilution of the air sample could be considered worst case or unrealistic. Those which involve moderate conditioning heating and air sample dilution may be considered most representative of actual indoor conditions. Methods which use a small dynamic emission chamber incorporate dilution in the sample preparation and are equivalent to the TVOC emission test procedures.

For the perceived intensity and hedonic scale methods, the automotive interior fitting ranking method which uses multi-point perceived intensity and quality scales was the preferred non-quantitative test method. This method involves dilution in sample preparation and ranks the odours both on intensity and quality scales.

The odour threshold and butanol reference methods could be used as quantitative odour emission evaluation methods. The butanol reference method appears to exhibit greater advantages in that it produces comparative data and has greater sensitivity. However, further comparative evaluation tests should be performed to verify this.

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3. SAE, Designation J 1351, Hot Odor Test for Insulating Materials.

4. Wolkoff, P., P. Nielsen, "Indoor Climate Labeling of Building Materials: The Experimental Approach for a Prototype", Characterizing Sources of Indoor Air Pollution and Related Sink Effects, B. Tichenor - Editor, ASTM Publication Code No. 04-012870-17, 1996.

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Table 1

Materials Odour Test Method Summary

Material No.	Odour Threshold (ou)	Butanol Equivalent Threshold (ppm)	DICL	Auto Trim	ASTM C739 & C665	SAE J1351	TVOC Emission (mg/m^2·h)
1	16	6	yes	unacceptable	objectional & strong	strong offensive	1.03
2	<8	--	no	good	objectional & strong	definite odour	<0.01
3	8	--	yes	good	objectional & weak	definite odour	0.33
4	<8	--	no	good	objectional & weak	definite odour	0.89
5	<8	--	no	acceptable	objectional & strong	definite odour	0.89
6	<8	--	no	acceptable	objectional & weak	definite odour	1.28
7	10.5	1.5	yes	unacceptable	objectional & strong	very strong offensive	0.15
8	10	1.5	yes	acceptable	objectional & weak	definite odour	0.01
9	14	3	yes	--	--	--	0.52
10	--	--	--	acceptable	pleasant & weak	definite odour	0.05

-- test not conducted

Figure 1

Intensity - Hedonic Scale

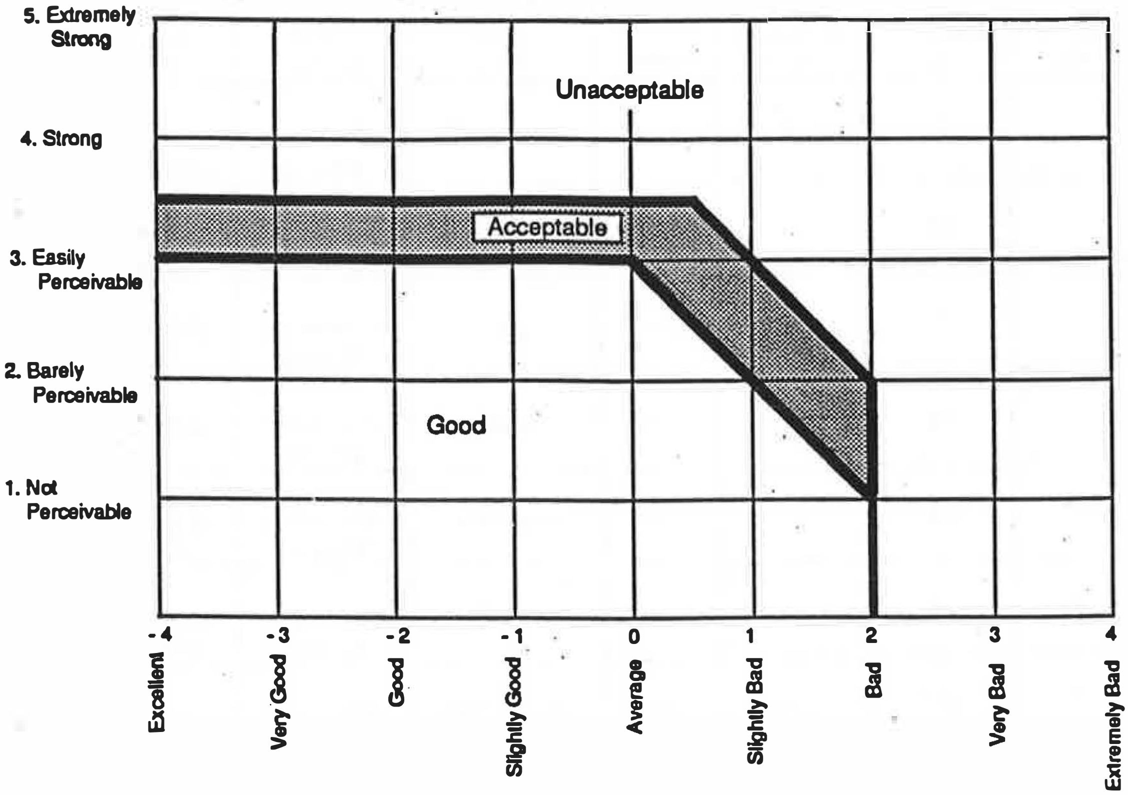
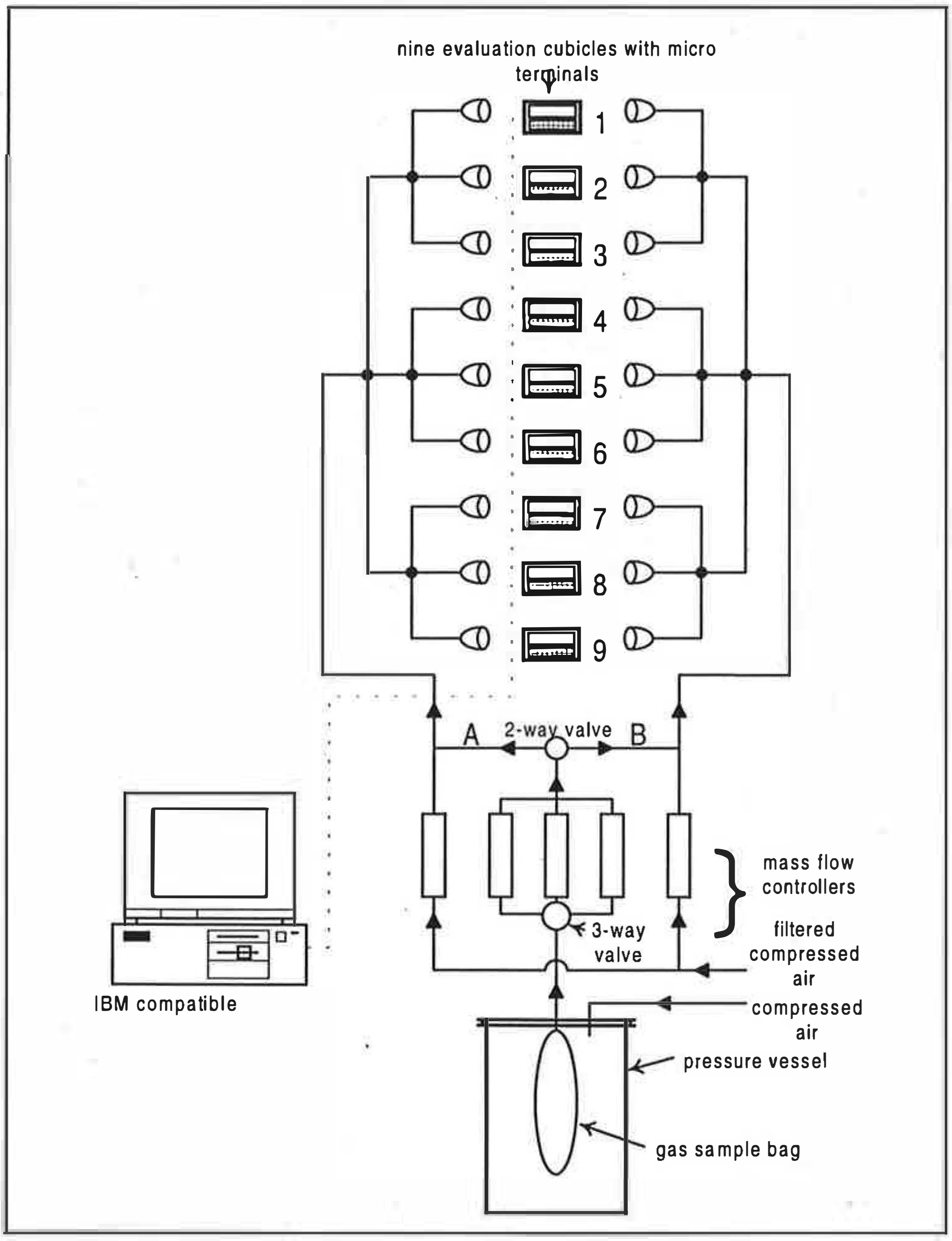


Figure 2

Odour Evaluation Panel Schematic



APPENDIX A

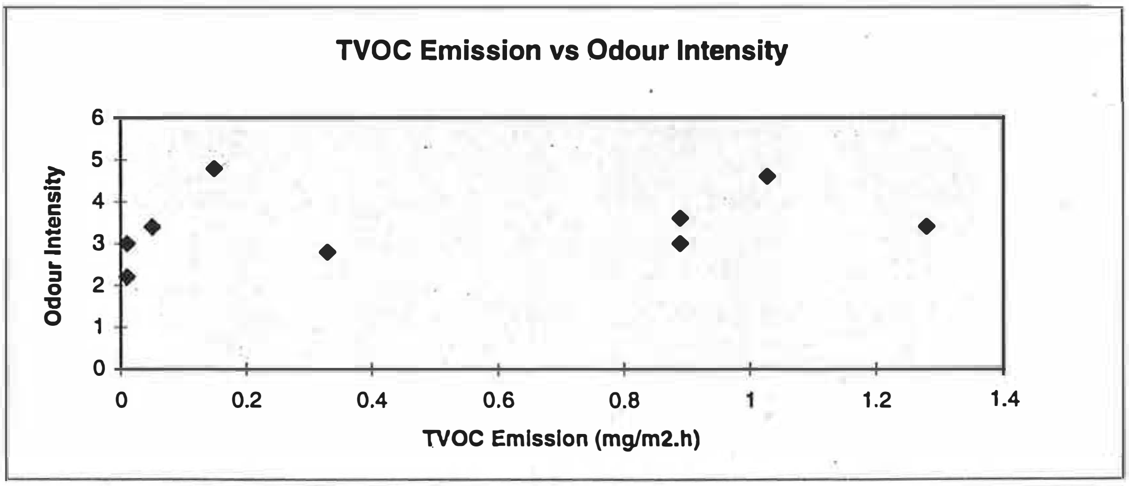
Odour - TVOC Relationships
(2 pages)

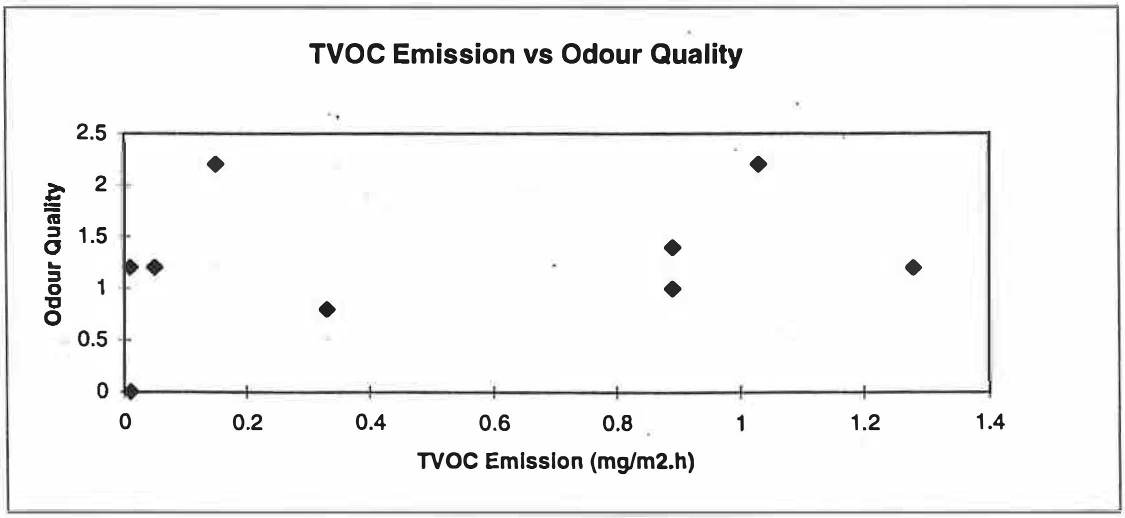
Odour - TVOC Relationships

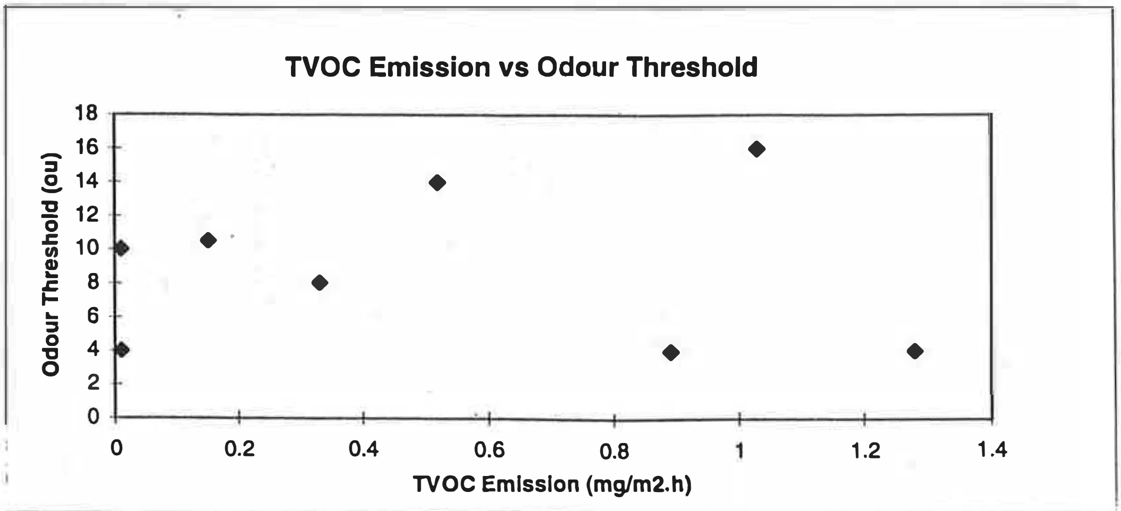
During this study, both odour emissions and total volatile organic compound (TVOC) emissions were determined from tests on the materials. Details of the test methods and the results of these emissions tests are provided in the main text of the report.

The emissions data were examined to determine if relationships existed between odour emissions and TVOC emissions. The automotive trim interior fitting odour test data and the odour threshold method results were used for this comparison. Both odour intensity and odour quality test data of the automotive interior fitting methods and the odour threshold data for the separate materials were compared to the TVOC emissions data. Three graphs comparing these data are presented in Figure A.1. For the test data on the materials, there is no apparent increase in the three measured odour dimensions with the increase in TVOC emission rates. Low and high emission rates were associated with materials with both low and high odour dimensions. Human odour response is to specific chemicals and not to an equivalent total volatile organic compound determination. The sampling and analytical method for TVOC determinations may also not be appropriate to identify potentially odorous compounds such as inorganic compounds and light or very reactive organics which may be emitted from these materials.

Figure A.1


TVOC Emission vs Odour Intensity
Odour Intensity
0
1
2
3
4
5
6
0
0.2
0.4
0.6
0.8
1
1.2
1.4
TVOC Emission (mg/m2.h)


TVOC Emission vs Odour Quality
Odour Quality
0
0.5
1
1.5
2
2.5
0
0.2
0.4
0.6
0.8
1
1.2
1.4
TVOC Emission (mg/m2.h)


TVOC Emission vs Odour Threshold
Odour Threshold (ou)
0
2
4
6
8
10
12
14
16
18
0
0.2
0.4
0.6
0.8
1
1.2
1.4
TVOC Emission (mg/m2.h)