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**Development of an analysis method to measure  
airborne acrylate in UV-cured motor vehicle  
repair coatings**

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## EXECUTIVE SUMMARY

UV-curable coatings are widely used in a growing number of industries such as paints, contact glues, printing inks and furniture coatings. The main reasons for this are their low or solvent-free formulations, high cure speeds, low-temperature processing and preferential finish properties such as scratch and chemical resistance. The potential cost advantage of their application to the automotive refinish market are substantial in terms of energy savings and throughput from the rapid curing process.

UV-automotive coatings (ostensibly primers and undercoats, but some clear coats e.g on headlight lenses and fillers) are usually composed of three major components; an acrylate prepolymer (e.g urethane acrylate, polyester acrylate), a multifunctional acrylate monomer (e.g pentaerythritol tetraacrylate) and a photoinitiator system (e.g benzophenone). These products may contain up to 60% solids or up to 90% organic solvents. Other essential components with important roles in the product may include UV-absorbers and radical scavengers.

Few automotive products have come to market in the past 5 years and these have not been successful because of their high cost, limitation to refinish repairs and reticence in the uptake of new technology however most of the worlds leading coatings manufacturers are actively researching and developing UV-coatings and curing technology. It is likely that additional coatings will be introduced in the near future and the number of refinish products is expected to surge as the technology becomes more widespread.

Multifunctional acrylates (MFA) are well known skin contact irritants and sensitisers and can induce respiratory hypersensitivity leading to asthma. Allergic reactions from acrylates have been found in a variety of products and occupations. Increased use of MFA in automotive spraying applications may result in increased worker exposure. To-date, sampling and analytical procedures have not been extensively studied. Sensitive methods for monitoring worker exposure are important as target exposure levels are likely to be of the order of  $0.1 \text{ mg/m}^3$ . In this report an outline method, suitable for sampling and analysis of a wide range of MFA (aerosol and vapours), has been validated with 2 compounds already used in automotive UV-coatings.

### Objectives

- Develop a sampling and analysis method for monitoring multifunctional acrylate aerosol.

### Main Findings

- The GC/MS procedure adopted for the analysis of MFA was highly sensitive and effective for the identification and quantitation of the MFA investigated.
- Further investigation of chemical ionisation techniques may yield more robust speciation of specific compounds.
- The high sensitivity of the analytical procedure may facilitate the used of short-term sampling periods which is beneficial when studying motor vehicle repair (MVR) applications.
- The precision of replicate analyses for MFA was typically 2.8 - 3.4%.

- The limits of detection for HDDA and PETA of  $0.29 \mu\text{g}/\text{m}^3$  and  $0.14 \mu\text{g}/\text{m}^3$  respectively are less than 1% of the potential target value of  $100 \mu\text{g}/\text{m}^3$ .
- Recovery of the two MFA from the OSHA versatile sampler (OVS) sampler was quantitative with a range of 95.6 – 111 % .
- The sampler demonstrated potential for use for monitoring a wide range of MFA.
- The retention efficiency of the OVS sampler was effective for the compounds studied.
- The stability of the MFA on the OVS sampler upon storage at room temperature for 10 days was demonstrated.

### **Recommendations**

- The method described should be adopted for the measurement of MFA aerosol in air.
- The procedure described should be evaluated in a MVR workplace using UV-cured coatings.
- Further method development using chemical ionisation should be carried out to be able to effectively speciate MFA.

# 1 INTRODUCTION

## 1.1 AUTOMOTIVE USE OF UV-CURABLE COATINGS

UV-curable coatings are widely used in a growing number of industries such as paints, contact glues, printing inks (e.g compact disk manufacture) and furniture coatings. The main reasons for this are their low or solvent-free formulations, high cure speeds, low-temperature processing and preferential finish properties such as scratch and chemical resistance. The potential cost advantage of their application to the automotive refinish market are substantial in terms of energy savings (no baking) and throughput from the rapid curing process (~ 2 minutes) although the extensive use of solvents is unlikely to diminish rapidly because of the properties required in the finished coating.

UV-automotive coatings (ostensibly primers and undercoats, but some clear coats e.g on headlight lenses and fillers) are usually composed of three major components; an acrylate prepolymer (e.g urethane acrylate, polyester acrylate), a multifunctional acrylate monomer (e.g pentaerythritol tetraacrylate) and a photoinitiator system (e.g benzophenone). These products may contain up to 60% solids or up to 90% organic solvents. Other essential components with important roles in the product may include UV-absorbers and radical scavengers.

Few automotive products have come to the market in the past 5 years and these have not been successful because of their high cost, limitation to refinish repairs (due to the short range and line-of-application of UV-curing lamps) and reticence in the market place in the uptake of new technology. However, most of the worlds leading coatings manufacturers (BASF, Akzo Nobel and Dupont) are actively researching and developing UV-coatings and curing technology for the automotive market. It is likely that additional coatings will be introduced into the marketplace in the near future and likewise the number of suppliers offering UV-coatings and related refinish products is expected to surge as the technology becomes more widespread and applicable to automotive refinish procedures.

Two products (clear coats) have been found to date which are available in the UK. One product contains >40% solvent, tripropylene glycol diacrylate and an acrylate pre-polymer. There is no evidence of widespread use of this product in the UK. The second contains >35% solvent, pentaerythritol tetraacrylate but no other acrylate resin. Other products such as UV-primers and fillers have also seen some limited use in the UK and US markets.

Acrylates are well known skin contact irritants and sensitisers and can induce respiratory hypersensitivity leading to asthma (Bjorkner, 1984; Piirila et al, 1998). Allergic reactions from acrylates have been found in a variety of products and occupations. The risk of inducing contact allergy depends both on the sensitisation capacity of the chemical and its penetration into the skin which in turn depends on the physico-chemical properties of the substance. Tripropylene glycol diacrylate is the most common multifunctional acrylate used in non-automotive applications (e.g printing) and has a strong sensitising capacity. Its low vapour pressure and water solubility means that it remains on the skin for a relatively long period of time after deposition. Increased use of multifunctional acrylates (MFA) in automotive spraying applications may result in increased worker exposure to airborne contaminants. To-date, sampling and analytical procedures have not been as extensively studied as those for dermal exposure to UV-curable acrylates (Nylander-French, 2000, Surakka et al, 2000). Sensitive methods for monitoring worker exposure to acrylates are important as target exposure levels are likely to be of the order of 0.1 mg/m<sup>3</sup> reflecting the potential health concerns of this class of compounds. Few methods have been published on the sampling and analysis of MFA and existing methods have not been validated at the low levels required (Nylander-French et al,

1994). In this report an outline method, suitable for sampling and analysis of a wide range of MFA (aerosol and vapours) has been validated with 2 compounds already used in automotive UV-coatings; hexanediol diacrylate (HDDA) and pentaerythritol tetraacrylate (PETA).

## 1.2 COMPOSITION OF UV-CURABLE AUTOMOTIVE COATINGS

UV-curable automotive coatings consist of a complex mixture of volatile solvents, reactive chemicals and additives. Isomers and by-products generated during manufacture of the MFA and acrylate prepolymer (Marek and Grollmann, 1999) add significantly to the complexity of the UV-coating formulation. Figure 1.1 shows a GC/MS TIC chromatogram of a commercial clear coat as mentioned in section 1.1. The chromatogram shows a complex mixture of over 30 components (excluding solvents which are not observed under the analysis conditions).

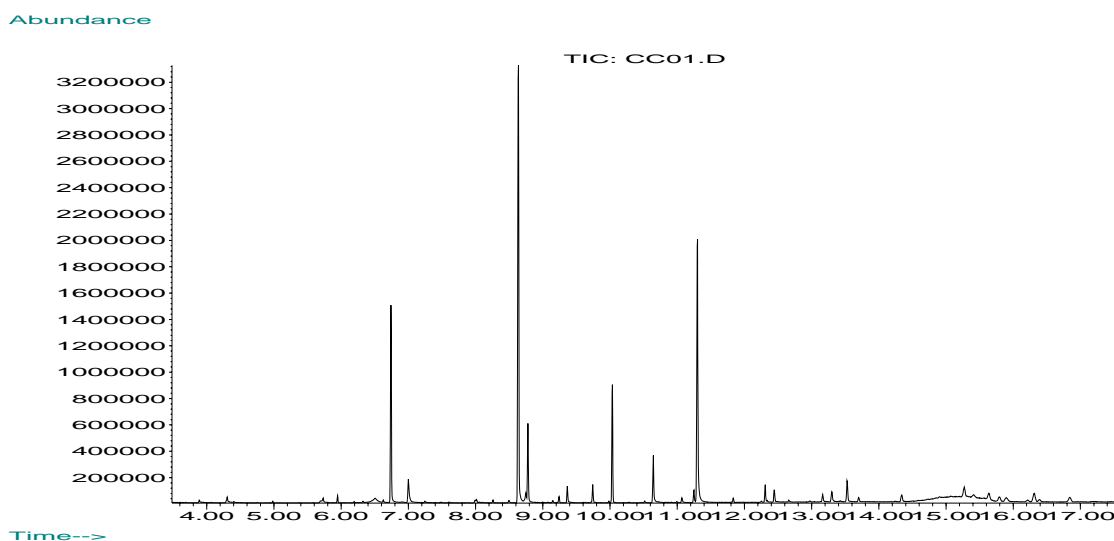


Figure 1.1 TIC chromatogram of a commercially available UV-cured clear coat.

Some of the typical constituents and their role in the coating are given in table 1.1. All of the constituents are harmful to man and the environment.

<b>Table 1.1 Major constituents of UV-curable coatings</b>			
Constituent and typical concentration (%)	Typical examples	Function	Typical health and environmental hazards
Solvent (30-90)	Ethyl acetate, acetone	Controls viscosity	F, Xi; R11, R36, R66, R67
Photo-initiator (1-5)	2,4,6 -trimethylbenzophenone	Polymerisation initiator	N; R50/53, R43
UV-stabiliser (1-5)	Methyl 1,2,2,6,6-pentamethyl-4-piperidyl serbacate	Prevents light degradation for outdoor applications	N; R50/53, R43
Acrylate prepolymer (1-5)	Isobornyl acrylate	Forms flexible coating when polymerised	Xi, N; R36/37/38, R51/53
Acrylate monomer (0-5)	Hexanediol diacrylate	Reactive diluent/adhesion promoter	Xi ; R36/38, R43

## 2 EXPERIMENTAL AND RESULTS

### 2.1 MULTIFUNCTIONAL ACRYLATES CHOSEN FOR STUDY

Two MFAs (see table 2.1), 1,6-Hexanediol diacrylate (HDDA) and Pentaerythritol tetraacrylate (PETA), having significantly different boiling points and viscosities were selected to represent the range of typical compounds encountered in the workplace and have already been used in automotive products.

MFA	CAS number	Molecular Mass	Boiling Point (°C)	Chemical Structure
1,6-Hexanediol diacrylate	13048-33-4	226.27	107	$[\text{H}_2\text{C}=\text{CHCO}_2(\text{CH}_2)_3]_2$
Pentaerythritol tetraacrylate	4986-89-4	352.34	>> 220	$(\text{H}_2\text{C}=\text{CHCO}_2\text{CH}_2)_4\text{C}$

According to COSHH essentials (HSE, 2003) chemicals such as MFA carrying a sequence of risk phrases such as R36/37/38 and R43 (hazard group C) should have a workplace exposure limit value in the range 0.01 to 0.1 mg/m<sup>3</sup> (although this strictly applies to dusts rather than aerosols). This target concentration range was adopted in the work described here and equates to approximately 10 – 100 µg/sampler for an 8-hour pumped sample at 2 l/min.

### 2.2 MATERIALS AND METHODS

#### 2.2.1 Chemicals

The multifunctional acrylates (technical grade: 80-90% purity) and hexylacrylate (HA) used as an analytical internal standard were purchased from Sigma-Aldrich.

Figures 2.1 –2.3 show the GC/MS total ion chromatograms (TIC) and mass spectra of HDDA, PETA and HA respectively. None of the 3 compounds exhibits a molecular ion in their mass spectra. Several major impurities are shown in each of the MFA standards (additional peaks in the chromatogram). The mass spectra of all three acrylates is dominated by the most abundant fragment at 55 amu (atomic mass units) which corresponds to the moiety  $(\text{H}_2\text{C}=\text{CHCO}^+)$ . In order to achieve the analytical performance required to measure low concentrations of MFA the fragment at 55 amu must be utilised for quantitation. Several other fragments in each compound are suitable as confirmatory ions for identification purposes.

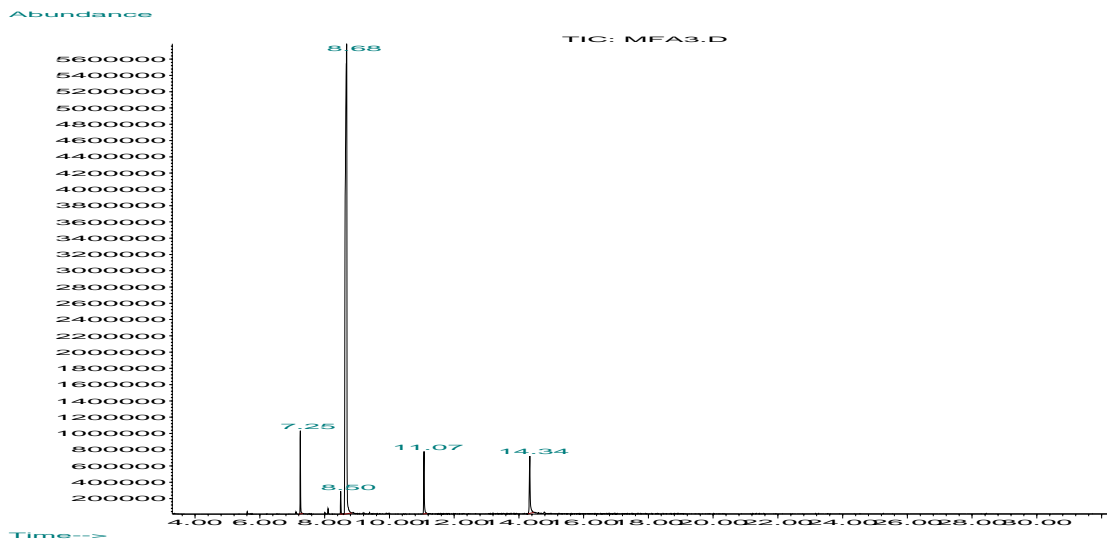


Figure 2.1a. TIC chromatogram of HDDA standard

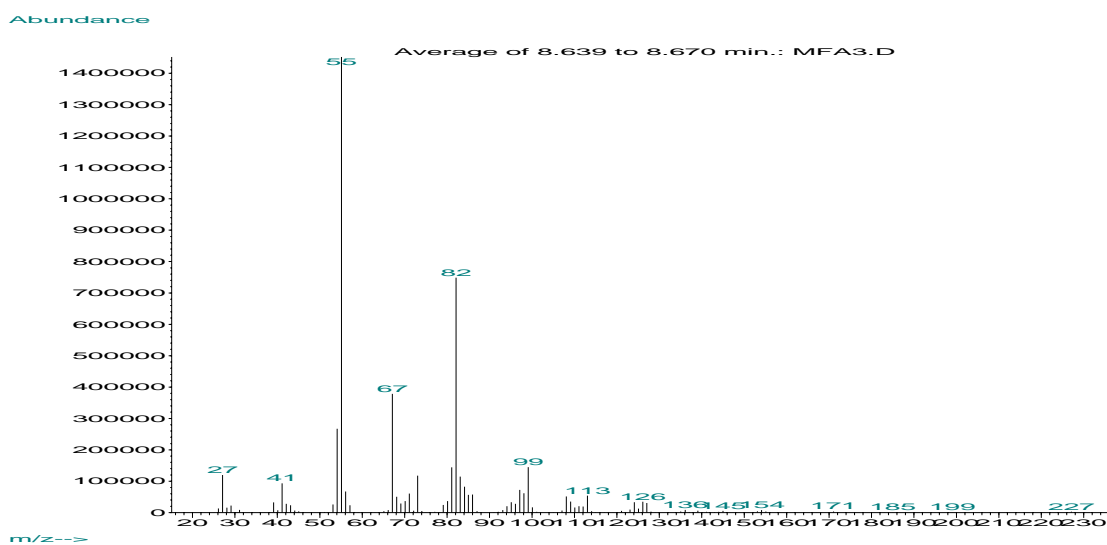


Figure 2.1b. Mass spectrum of HDDA.

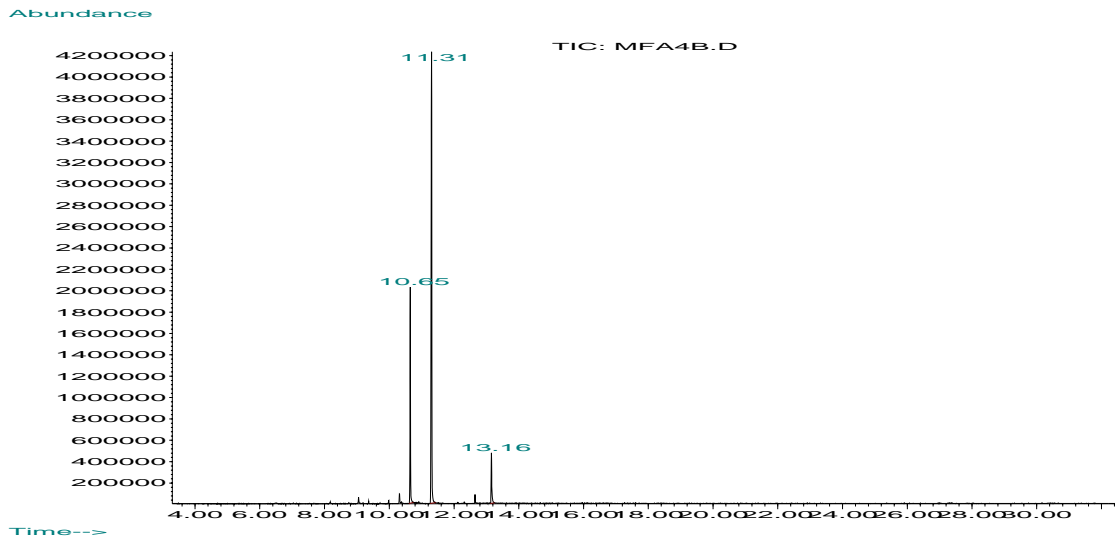


Figure 2.2a. TIC chromatogram of PETA standard

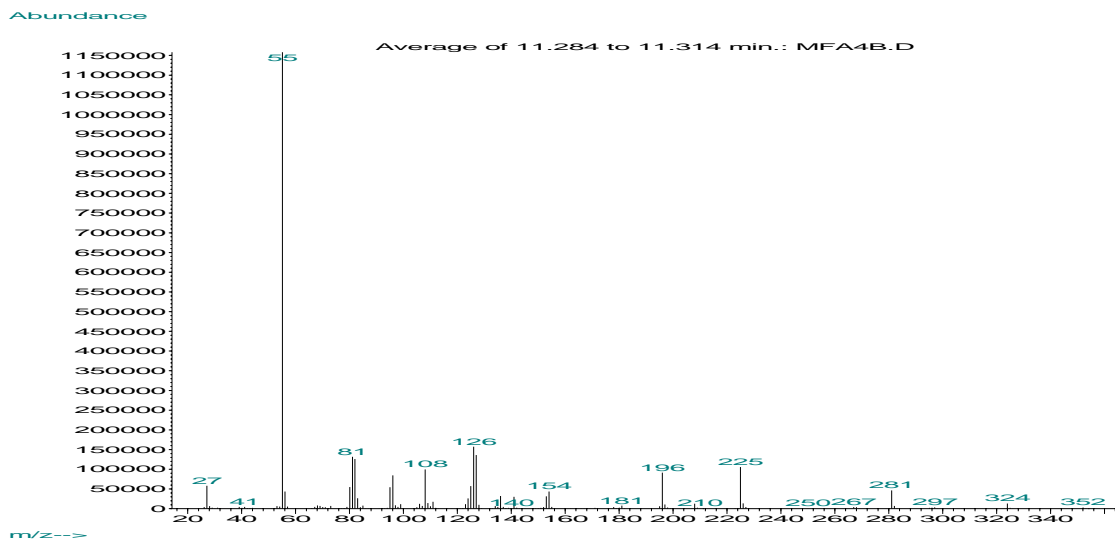


Figure 2.2b. Mass spectrum of PETA.

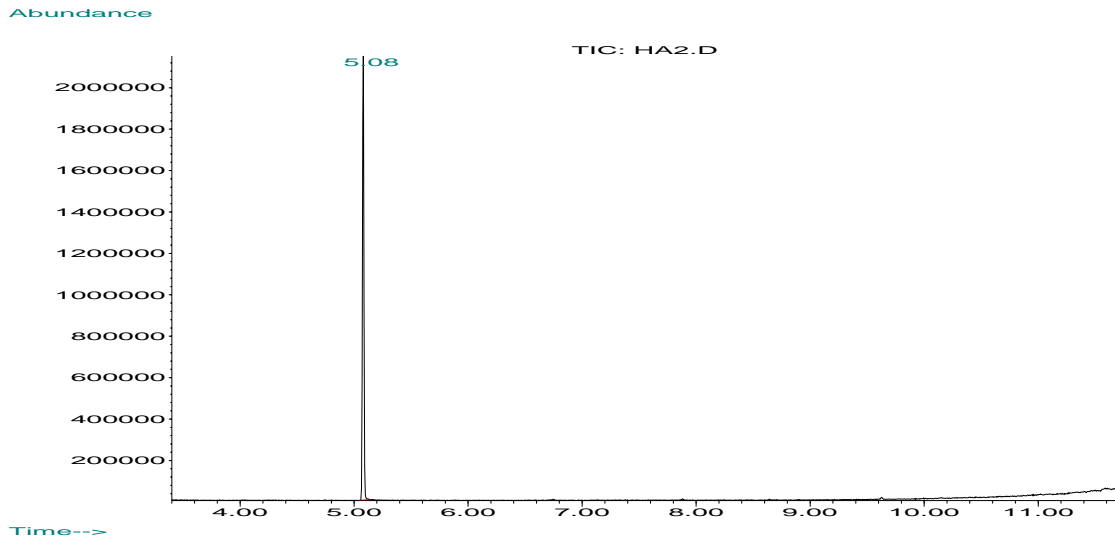


Figure 2.3a. TIC chromatogram of HA internal standard

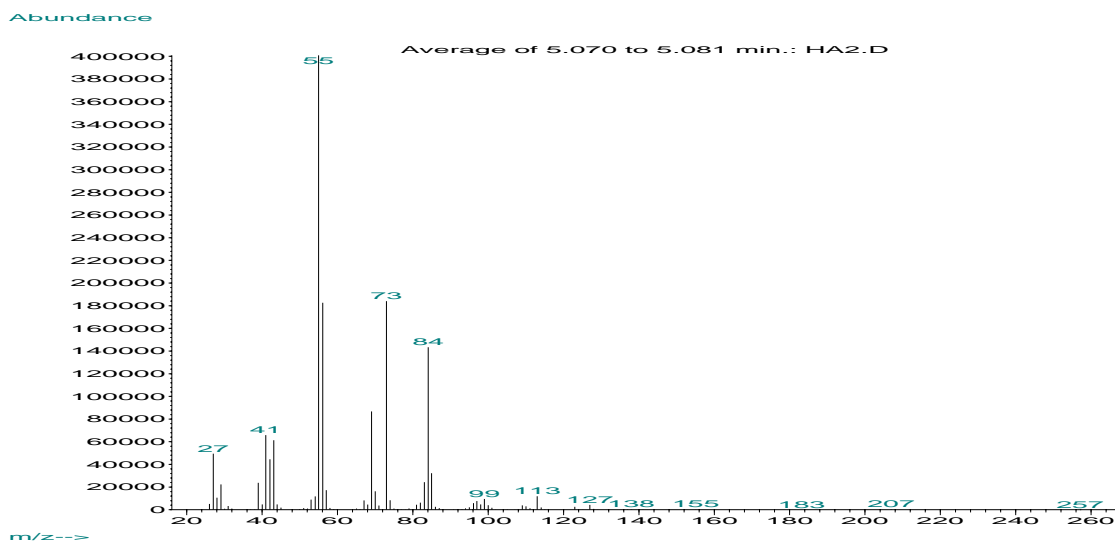


Figure 2.3b. Mass spectrum of HA internal standard.

### 2.2.2 OVS sampler

The OVS sorbent tube validated for sampling of the MFA in air is shown in figure 2.4. This sampler has already been used for the measurement of several MFA (Nylander-French et al, 1994) at higher concentrations than the target value of 0.01 to 0.1 mg/m<sup>3</sup>. The sampler consists of a 13 mm OD glass tube fitted with a GFA pre-filter followed by two Tenax sections separated by a polyurethane foam section (SKC, 226-30-4).

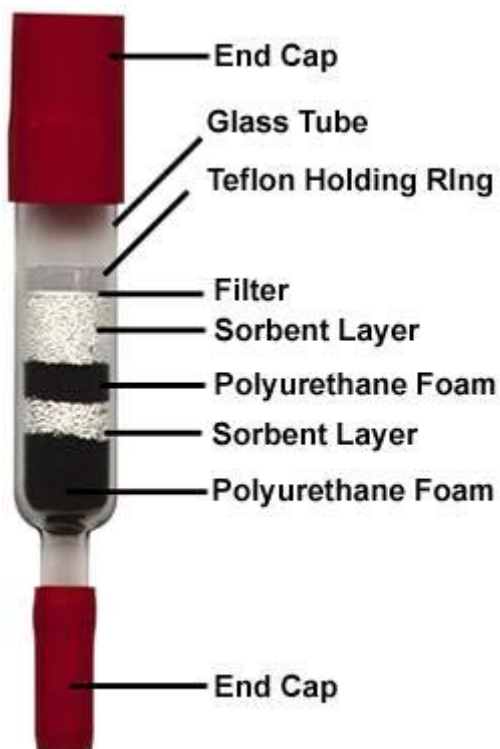


Figure 2.4 OVS sampler

### 2.2.3 Sample preparation

After spiking the sampler with MFA the front Tenax sorbent section, Teflon ring and GFA filter are placed in a 4 ml amber vial with PTFE lined screw cap and acetone (2 ml) added. The first polyurethane foam section and back Tenax sorbent section are placed in a separate 4 ml amber vial with PTFE lined screw cap and acetone (2 ml) added. Both sections are sonicated for 30 min. A 1 ml aliquot of the sample is removed and 50 µl of hexylacrylate internal standard added. The samples, calibration standards and quality control samples are analysed using the MFA method described in section 2.2.4. Each sample and quality control sample was analysed in triplicate with quality control samples analysed every 6-9 samples.

### 2.2.4 Analysis

The instrumental conditions for the GC/MS analysis of MFA extracted from the OVS sampler are summarised in table 2.2. In order to ensure analytical robustness the internal standard, (HA), was added to all standards and sample extracts. This internal standard was effective for the compounds under study as it did not co-elute with HDDA, PETA or any of their impurities and its most abundant ion also occurred at 55 amu.

Table 2.2. Agilent Technologies 5973 - GC/MS conditions for analysis of MFA	
<b>Injector :</b>	
splitless	
Injection volume	1 $\mu$ l
<b>Temperatures (<math>^{\circ}</math>C)</b>	
injector	350
detector	230
<b>Oven :</b>	
Initial temperature	50
Initial hold time	1
Ramp rate	30 $^{\circ}$ C/min to 280 $^{\circ}$ C
Hold time	2 min
Run time	14.5 min
<b>Column:</b>	HP5: 30 m x 0.25 mm x 0.25 $\mu$ m film thickness
<b>Retention times (min) and SIM ions (a.m.u.):</b>	
Hexylacrylate (internal standard)	5.6 min, 55, 73, 84 amu.
HDDA	9.0 min , 55, 67, 82 amu.
PETA	11.7 min, 55, 81, 126 amu.

### 2.3 ANALYTICAL PRECISION

Ten replicate injections of a standard containing HDDA (24.79  $\mu$ g/ml) and PETA (20.59  $\mu$ g/ml), a concentration equivalent to an 8-hour sample at 2 l/min at 0.05 mg/m<sup>3</sup>, were made to estimate the precision of the GC/MS analytical procedure. The statistical results are given in table 2.3.

Table 2.3 Analytical precision of MFA (peak area counts/1000) at ~ 0.05 mg/m <sup>3</sup> (simulated 8-hour sample)		
Replicate	HDDA (24.79 µg/ml)	PETA (20.59 µg/ml)
1	1071	354
2	1140	359
3	1125	361
4	1079	349
5	1101	355
6	1138	372
7	1124	352
8	1119	348
9	1180	376
10	1114	384
<b>Mean</b>	<b>1119</b>	<b>361</b>
<b>Standard Deviation</b>	<b>31.3</b>	<b>12.3</b>
<b>RSD(%)</b>	<b>2.8</b>	<b>3.4</b>

## 2.4 DETECTION LIMITS FOR MFA

The limit of detection (LOD) estimated from a low-level standard of HDDA and PETA at 2.0 µg/ml in acetone are given in table 2.4. The LODs are in the region of 0.1-0.3% of the target concentration of 0.1 mg/m<sup>3</sup>.

Table 2.4 Estimated Limits of detection for HDDA and PETA		
MFA	LOD (SN: 3:1) (µg/sample)	8-hr TWA concentration (µg/m <sup>3</sup> )
HDDA	0.145	0.29
PETA	0.071	0.14

## 2.5 RECOVERY OF MFA FROM THE OVS SAMPLER

### 2.5.1 Acrylate working standards

Details of the HA and MFA in acetone standard solutions used throughout this work for calibration and sampler spiking are described in table 2.5. The spiking levels are approximately the amount of MFA expected on the OVS tube after sampling for 8-hours at 2 l/min at the target concentrations of 0.01 and 0.1 mg/m<sup>3</sup>.

Table 2.5 Details of MFA in acetone calibration and spiking solutions used in method development				
Acrylate	Mass of Acrylate (µg in 10ml)	[Acrylate] (µg/ml)	Mass of acrylate in 50 µl spike (µg)	
			High level	Low level
HDA	26580	2658	132.9 <sup>3</sup>	132.90 <sup>3</sup>
HDDA	24790	2479	123.95 <sup>1</sup>	12.40 <sup>2</sup>
PETA	20590	2059	103.0 <sup>1</sup>	10.30 <sup>2</sup>

<sup>1</sup> High level stock solutions <sup>2</sup> Stock solutions diluted by a factor of 10. <sup>3</sup> Internal standard 50 µl spike in all samples and standards

### 2.5.2 Determination of recovery efficiency of MFA from the sampler

The following procedure was carried out to determine recovery efficiency:

Six replicate tubes were spiked with 50 µl of each stock solution directly onto the GFA filter of the OVS sampler whilst drawing air through the sampler at 2 L/min. Similarly, six low level replicate tubes were also spiked with the low level stock solutions. Each spiked tube had a total of 60 l of air at 2 l/min drawn through it to deposit the MFA efficiently. Each sampler was then prepared and analysed as described in section 2.2.3 and 2.2.4.

The results for the determination of recovery efficiency are given in table 2.6.

Table 2.6 Recovery of MFA from the OVS sampler				
Replicate	Spiking level (µg/sample).			
	HDDA (12.4)	PETA (10.3)	HDDA (123.7)	PETA (103.0)
1	13.7	10.48	118.4	100.48
2	14.24	11.08	119.04	93.3
3	13.8	10.96	117.28	88.98
4	13.14	9.74	119.22	98.16
5	14.3	11.06	132.03	108.36
6	13.4	10.15	132.08	101.6
<b>Mean</b>	<b>13.76</b>	<b>10.58</b>	<b>123.0</b>	<b>98.48</b>
<b>Recovery %</b>	<b>111</b>	<b>102.6</b>	<b>99.4</b>	<b>95.6</b>
<b>Standard Deviation</b>	<b>0.47</b>	<b>0.55</b>	<b>7.04</b>	<b>6.77</b>
<b>RSD(%)</b>	<b>3.3</b>	<b>5.2</b>	<b>5.7</b>	<b>6.9</b>

## 2.6 SAMPLING EFFICIENCY OF THE OVS SAMPLER FOR MFA

An effective sampler should retain all the collected analytes efficiently for the full duration of the sampling period. The following procedure was carried out to simulate sampling efficiency:

Six replicate tubes were spiked with 50 µl of each stock solution directly onto the GFA filter whilst drawing air through the sampler at 2 l/min. Each tube was pumped for 4 hours until 240 l of air at 2 l/min had been drawn through it. The samplers were then prepared and analysed as described in section 2.2.3 and 2.2.4. The results for the determination of sampling efficiency are given in table 2.7.

Table 2.7 Sampling efficiency of MFA on OVS sampler (%)				
	HDDA (123.7 µg/sample)		PETA (103.0 µg/sample)	
Replicate	Front section	Back section	Front section	Back section
1	98	0	99	0
2	96	0	94	0
3	97	0	102	0
4	103	0	106	0
5	103	0	108	0
6	100	0	103	0
<b>Mean</b>	99.5	-	102	-
<b>Standard Deviation</b>	3.0	-	5.0	-

## 2.7 STORAGE STABILITY OF MFA ON THE OVS SAMPLER

Six replicate samplers were prepared as described in section 2.6 and 60 l of air was drawn through the tubes after spiking. The samplers were then wrapped in aluminium foil and stored in the dark at room temperature for 10 days prior to analysis. The samples were analysed as described in section 2.2.3 and 2.2.4 to determine whether either component was unstable under the storage conditions adopted. The results are given in table 2.8.

Table 2.8 Storage stability of MFA on the OVS Sampler		
Replicate	HDDA (123.7 µg/sample)	PETA (103.0 µg/sample)
1	120.44	98.66
2	117.74	95.4
3	115.48	94.94
4	121.86	97.98
5	125.26	100.74
6	121.46	96.72
<b>Mean</b>	<b>120.37</b>	<b>97.41</b>
<b>Recovery %</b>	<b>97.3</b>	<b>94.6</b>
<b>Standard Deviation</b>	<b>3.4</b>	<b>2.2</b>
<b>RSD(%)</b>	<b>2.8</b>	<b>2.3</b>

## **3 DISCUSSION OF RESULTS**

### **3.1 ANALYTICAL METHOD**

The GC/MS procedure adopted for the analysis of MFA was highly sensitive and effective for the identification and quantitation of the MFA investigated. The high boiling points of these compounds enables elution of the chromatographic peaks free of interferences such as VOCs and low molecular weight impurities, several of which have acrylate functionality. However, due to the lack of molecular ions or a large characteristic fragments the quantitation and speciation of the MFA is achieved by the use of the 55 amu fragment ion, typical of the acrylate group, and the retention time of the compound. Further investigation of chemical ionisation techniques rather than the electron impact ionisation adopted here may yield larger fragments or molecular ions which could be more diagnostic of the MFA and of greater benefit in speciation of unknown compounds.

The high sensitivity of the analytical procedure may facilitate the used of short term sampling periods which is beneficial when studying MVR applications.

### **3.2 ANALYTICAL PRECISION AND LIMITS OF DETECTION FOR MFA**

The coefficient of variation, an estimate of precision, for HDDA and PETA for 10 replicate determinations was 2.8% and 3.4% respectively (table 2.3). This data demonstrates effective analytical precision within the analytical run.

The limits of detection for HDDA and PETA (table 2.4) of 0.29 and 0.14  $\mu\text{g}/\text{m}^3$  are less than 1% of the potential target value of 100  $\mu\text{g}/\text{m}^3$  and demonstrate the high sensitivity of the analytical method.

### **3.3 RECOVERY OF MFA FROM THE OVS SAMPLER**

Recovery of the two MFA from the OVS samplers was quantitative with a range of 95.6 – 111% for the two spiking levels (table 2.6). The high recoveries and low coefficients of variation between replicate spiked samplers suggest the sampler type and extraction procedure are fit for purpose.

Removal of the 2 sorbent sections within the sampler followed by sonication in acetone (2 ml) was more effective than the recoveries of 85 – 86% obtained by elution of the whole tube with solvent (15 ml) described by other workers (Nylander-French et al (1994)).

The sampler also demonstrates potential for a wide range of MFA since recoveries of typical di- and tetra-functional MFA were quantitative.

### **3.4 SAMPLING EFFICIENCY OF THE OVS SAMPLER**

Although the procedure of spiking liquid components onto the sampler to simulate workplace exposure of the MFA aerosol is not ideal it does provide evidence of the potential retention efficiency and capacity of the sampler. It is likely that the sampler is effective for at least 4-hours at a flow rate of 2 l/min as no breakthrough (no MFA found on the back sorbent section) was observed during this period. It is likely that the sampler has potential for the collection of higher loadings over sampling periods greater than 4-hours.

### **3.5 STORAGE STABILITY OF OVS SAMPLERS**

Storage of samplers wrapped in aluminium foil at room temperature for 10 days was effective and showed no evidence of degradation of the MFA. The low volatility of the two MFA is advantageous and exclusion of light avoids UV degradation.

## 4 CONCLUSIONS

- The GC/MS procedure adopted for the analysis of MFA was highly sensitive and effective for the identification and quantitation of the MFA investigated.
- Further investigation of GC/MS analysis utilising chemical ionisation techniques may yield more robust speciation of MFA compounds through the presence of characteristic molecular ions.
- The high sensitivity of the analytical procedure may facilitate the use of short term sampling periods which is beneficial when studying MVR applications.
- The precision of replicate analyses for HDDA and PETA at 2.8% and 3.4% respectively was fit for purpose.
- The limits of detection for HDDA and PETA of  $0.29 \mu\text{g}/\text{m}^3$  and  $0.14 \mu\text{g}/\text{m}^3$  are less than 1% of the proposed target value of  $100 \mu\text{g}/\text{m}^3$  and demonstrate the high sensitivity of the analytical method.
- Recovery of the two MFA from the OVS sampler was quantitative with a range of 95.6 – 111 % .
- The sampler demonstrated potential for use for monitoring a wide range of MFA.
- The retention efficiency of the OVS sampler was effective for the compounds studied with no breakthrough onto the backup section of the sampler observed.
- The stability of the MFA on the OVS sampler upon storage at room temperature after 10 days was demonstrated.
- Validation of the sampling and analysis procedure is required under workplace conditions

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