



Sheffield Hallam University

Sampling of Exhaust Gases from Purex Filtration Equipment

Final Report

**Work carried out by Advanced Chemical Services,
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Introduction:

A project has been initiated by Hi-Tech U.K. Ltd, Dinnington Sheffield, to examine the composition of exhaust gases from Purex Filtration equipment as fitted to a pilot laser etching plant. The samples submitted for this analysis are as follows:

- Polypropylene
- Polyacrylonitrile – butadiene – styrene (ABS)
- Polystyrene
- Polyethylene – terephthalate (PET)
- Polyvinylchloride – unplasticised (uPVC)
- Bonded Paper
- Glass

The samples submitted were analysed for the following analytes:

- Methylmethacrylate
- Esters
- Benzene
- Hydrogen Chloride
- Ammonia
- Silicates
- Organic Acids
- PAH's (Polynuclear Aromatic Hydrocarbons)
- Alkanes

Experimental

All analyses were performed in triplicate.

a). Laser etching

Samples were ablated with the laser for varying lengths of time depending on the NIOSH method used (see Appendix 1) with manual adjustment of the ablated sample area, using the sample presentation stage as supplied.

b). Sampling

Sampling was carried out according to the appropriate NIOSH method (see Appendix 1). In summary an adsorbent tube was placed at the outlet of the exhaust pipe of the Purex filtration equipment. Eight litres of air was drawn through this tube using a castella pump. In the case of the silicates however a filter unit was used to collect the exhaust gas and four hundred litres of air were drawn through this filter using the same set-up.

c). Analyses.

An analyses were performed according to the relevant NIOSH method (see Appendix 1)

Results and Discussion.

1). Methyl Methacrylate

The analysis was carried out using NIOSH method 2537 (see appendix 1) Calibration data for methylmethacrylate in the range 0.001 mg/ml to 0.5 mg/ml was performed using standards. This is equivalent to a range of levels in the air samples taken of 0.25 – 5 ppb.

The calibration data for methylmethacrylate obtained from the gas chromatographic analyses of standards is shown as Figure 1. As can be seen a good linear calibration curve was obtained. The established limit of detection for the methodology employed was equivalent to 0.25 ppb for an air sample.

Methylmethacrylate was not detected in any of the samples analysed and the data is summarised in table 1.

Table 1: Data obtained from the analysis of exhaust gases of the Purex filtration unit for methylmethacrylate.

Substrate	Methylmethacrylate (ppb)
PET	<0.25
ABS	<0.25
Polystyrene	<0.25
Polypropylene	<0.25
Glass	<0.25
uPVC	<0.25
Bonded Paper	<0.25

2). Hydrochloric Acid

The analysis was carried out using NIOSH method 7903 and analysed using a Dionex Ion Chromatograph and using standards equivalent to the range of 0.01 to 5 mg/m³ for a 50 litre air sample. The limit of detection for this method is 0.2 mg/m³. Small amounts of hydrochloric acid were found in all but one of the samples (PET) and this could be a contributing factor to the pungent smell given off by the laser etching. The results of these analyses are summarised in table 2.

Table 2: Data obtained from the analysis of exhaust gases of the Purex filtration unit for Hydrochloric Acid.

Substrate	HCl (mg/m ³)
PET	<0.2
ABS	0.75
Polystyrene	1.03
Polypropylene	0.88
Glass	0.76
uPVC	0.47
Bonded Paper	0.95

3). Esters

The analysis of esters was carried out using NIOSH method 1450 (see appendix 1). The calibration data for the t-butyl acetate and propyl acetate obtained from the gas chromatographic analyses of standards is shown as Figure 1 and 2. As can be seen good linear calibration curves were obtained. The established limit of detection for the methodology employed was equivalent to 1.7 ppb for an air sample. No esters were detected in any of the exhaust gas samples. These data are summarised in Table 3.

Table 3: Data obtained from the analysis of exhaust gases of the Purex filtration unit for esters.

	1	2	3	4	5	6	7
n-amyl acetate	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb
n-butyl acetate	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb
sec-butyl acetate	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb
t-butyl acetate	<0.005 ppb	<0.005 ppb	<0.005 ppb	<0.005 ppb	<0.005 ppb	<0.005 ppb	<0.005 ppb
2-ethoxy ethyl acetate	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb
iso-amyl Acetate	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb
iso-butyl acetate	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb
n-propyl acetate	<0.005 ppb	<0.005 ppb	<0.005 ppb	<0.005 ppb	<0.005 ppb	<0.005 ppb	<0.005 ppb
ethyl acetate	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb	<1.7 ppb

KEY

- 1 PET
- 2 uPVC

- 3 ABS
- 4 Bonded Paper
- 5 Glass
- 6 Polystyrene
- 7 Polypropylene

4). Ammonia

Ammonia was analysed using NIOSH method 6701 using a Dionex Ion Chromatograph with a working range of 2 to 110 ppm and a limit of detection of 1 ppm. Ammonia was not found in any of the samples analysed. These data are summarised in Table 4.

Table 4: Data obtained from the analysis of exhaust gases of the Purex filtration unit for ammonia.

Substrate	Ammonia
uPVC	<1ppm
Paper	<1ppm
ABS	<1ppm
PET	<1ppm
Polystyrene	<1ppm
Polypropylene	<1ppm
Glass	<1ppm

5). Organic Acid (Acetic Acid)

Acetic acid analysis was carried out using gas chromatography following NIOSH method 1603 (see appendix 1). The limit of detection for this method is 0.1 ppm. No acetic acid was found to be present in any of the samples analysed. These data are summarised in Table 5.

Table 5: Data obtained from the analysis of exhaust gases of the Purex filtration unit for acetic acid.

Substrate	Acetic Acid
Paper	<0.1ppm
Glass	<0.1ppm
Polystyrene	<0.1ppm
Polypropylene	<0.1ppm
ABS	<0.1ppm
uPVC	<0.1ppm
PET	<0.1ppm

6). Hydrocarbons and Benzene

Hydrocarbons and benzene were analysed using NIOSH methods 1500 and 3700 respectively. Both were analysed using a gas chromatograph and standards varying in concentration from 0.005 mg/ml up to 0.25 mg/ml. The limit of detection for all hydrocarbons was 0.83 ppb apart from that for pentane which was 8.3 ppb. Trace amounts of hexane were detected in the exhaust gases from the filtration unit following laser etching of; polypropylene, polystyrene and glass. No other hydrocarbons were detected. These data are summarised in Table 6.

Table 6: Data obtained from the analysis of exhaust gases of the Purex filtration unit for hydrocarbons and benzene.

	1	2	3	4	5	6	7
Pentane	<8.3 ppb	<8.3 ppb	<8.3 ppb	<8.3 ppb	<8.3 ppb	<8.3 ppb	<8.3 ppb
Hexane	3.33 ppb	3.33 ppb	<0.83 ppb	<0.83 ppb	3.33 ppb	<0.83 ppb	<0.83 ppb
Heptane	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb
Octane	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb
Toluene	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb
Benzene	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb
Cyclohexane	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb
Cyclohexene	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb
Methyl Cyclohexane	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb	<0.83 ppb

KEY:

- 1 Polypropylene
- 2 Polystyrene
- 3 ABS
- 4 PET
- 5 Glass
- 6 Paper
- 7 uPVC

7). Polynuclear Aromatic Hydrocarbons. (PAHs)

Polynuclear aromatic hydrocarbons were analysed using NIOSH method 5515. The limits of detection for this method are 1ppb. Trace amounts of PAHs were detected in the exhaust gas from the filtration unit following laser etching of; polystyrene, polypropylene, paper, PET and uPVC. These data are summarised in Table 7.

Table 7: Data obtained from the analysis of exhaust gases of the Purex filtration unit for polynuclear aromatic hydrocarbons.

	Glass	ABS	Polystyrene	Polypropylene	Paper	PET	uPVC
PAH							
Acenaphthene	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb
Acenaphthylene	<1ppb	<1ppb	<1ppb	<1ppb	0.06ppm	<1ppb	<1ppb
Anthracene	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb	0.1ppm
Benzo (a) Anthracene	<1ppb	<1ppb	<1ppb	0.44ppm	<1ppb	0.82ppm	1.98ppm
Benzo(a)Pyrene	<1ppb	<1ppb	<1ppb	0.59ppm	<1ppb	0.85ppm	1.72ppm
Benzo(e)Pyrene	<1ppb	<1ppb	<1ppb	1.60ppm	<1ppb	0.88ppm	1.74ppm
Chrysene	<1ppb	<1ppb	<1ppb	1.53ppm	<1ppb	0.41ppm	0.16ppm
Dibenz(a,h)Anthracene	<1ppb	<1ppb	<1ppb	1.55ppm	<1ppb	<1ppb	0.21ppm
Fluoranthene	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb
Fluorene	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb
Naphthalene	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb	0.03ppm
Phenanthrene	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb
Pyrene	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb	<1ppb

8). Silicates

The silicates were analysed using NIOSH method 7602 utilising a Fourier Transform Infra-Red instrument in absorption mode. The samples were collected for four hours onto a filter and then ashed in a muffle furnace. The remaining sample was then analysed. This method has a working limit of detection of 0.025 mg/m³ in air. No silica was detected in any of the samples studied here. These data are summarised in table 8.

Table 8: Data obtained from the analysis of exhaust gases of the Purex filtration unit for silicates.

Sample	Amount of silica (mg/m ³)
Glass	<0.025
ABS	<0.025
Polystyrene	<0.025
Polypropylene	<0.025
Paper	<0.025
uPVC	<0.025
PET	<0.025

Conclusions

The analysis of exhaust from the Purex filtration equipment obtained whilst laser etching of the following substrates has been performed:

- Polypropylene
- Polyacrylonitrile – butadiene – styrene (ABS)
- Polystyrene
- Polyethylene – terephthalate (PET)
- Polyvinylchloride – inplasticised (uPVC)
- Bonded Paper
- Glass

From the data obtained no significantly large amounts of; methylmethacrylate, esters, benzene, hydrogen chloride, ammonia, silicates, organic acids, polynuclear aromatic hydrocarbons or alkanes are being let through the filter currently used. Therefore we can conclude that the filter is quite efficient.

As no significant amounts of any of the above listed analytes were detected it is difficult to pinpoint the cause of the detectable smell given off whilst laser etching was in progress. One cause could be hydrogen chloride which does have a noticeable odour since trace amounts of this compound were detected. The tubes used to collect hydrogen chloride and the inorganic gases for analysis contain washed silica gel. Since this is an adsorbent it could possibly be incorporated into any filters manufactured from hereon.