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**Optimisation of a Carbon Bed
Adsorber Plant using FTIR**

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Executive Summary.

As part of a DTI funded project led by NPL, Protea Limited conducted a programme to investigate the applicability of portable Fourier-Transform Infra-Red (FTIR) spectroscopy to monitoring process and emission concentrations. The technique was employed to monitor a carbon-bed recycling system used to recover a mixture of solvents eluted from the manufacture of a key agrochemical at a large manufacturing centre.

Two BOMEM 9100 FTIR spectrometers were used to study the concentrations of selected species across the carbon-bed system. The spectrometers used proved capable of semi-continuously profiling all key process components simultaneously. In addition the monitoring identified two further components not previously known to be present.

Portable FTIR Spectroscopy was demonstrated to be a suitable analytical technique for the simultaneous and semi-continuous measurement of solvents. The technique coped well with the process conditions of temperatures between 20 and 140°C and water concentrations of up to 30%.

The study demonstrated that the efficiency of the carbon-beds in trapping key process components was not optimal, as significant losses to atmosphere were observed during ‘Steam Blow-off’ of the regeneration bed. The detailed process information obtained during the study has enabled effective prioritisation of improvement plans. Potential savings include improved solvent recovery (£60k / year) and optimisation of carbon-bed replacement (beds are currently replaced every year at a cost of £15k).

The flexibility offered by FTIR technology suggests that the methodology used in this study would be readily transferable to other recovery systems e.g. cryogenic condensing systems.

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Approved on behalf of the Managing Director, NPL
by Stuart Windsor, Business Leader, Quality of Life Division

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Introduction.

As part of the DTI sponsored VAM Project 1.6 'Validation of Novel Spectrometric Techniques', Protea Limited were commissioned to undertake case studies on the applicability of portable Fourier-Transform Infra-Red Spectroscopy to the monitoring of industrial processes. One of the case studies chosen was a carbon-bed recovery system.

The system is designed to recover a solvent, which is also used as a process raw material, and closely related compounds. Emissions from the system are of dual importance; as well as the commercial value of lost solvents, emissions from the process are regulated by the Environment Agency through an Integrated Pollution Control authorisation.

Conventional monitoring techniques have previously been applied to the system, however the complex mixture of compounds within the stream has limited the applicability of such techniques.

Background.

In addition to the process raw material (Solvent A), it was recognised that decomposition products and impurities may be present within the recovery system. Also expected were water, carbon dioxide and carbon monoxide.

The monitoring task required measurement of these compounds under the range of conditions encountered under normal process operation:

- Stack temperature 20-140°C.
- Stack pressure – nominal ambient.
- Water concentrations of up to 30% at temperatures above 100°C.
- Range of concentration of target compounds – 1 to 50000 mg/m³.

Process Operation.

All waste gas streams from the manufacturing process are routed to the carbon adsorption bed system for solvent recovery. The system consists of three carbon beds with two beds operated in series whilst the third bed is regenerated. The beds are regularly cycled with the primary bed going for regeneration, the secondary bed becoming primary and the regenerated bed becoming the secondary bed (Appendix 1: Figures 1 – 3).

Regeneration is accomplished by steam stripping the carbon and recovering the desorbed solvents. Prior to changeover the bed is dried by a 'cooling fan', which drives remaining steam from the bed (Steam Blow-off).

Sampling points are provided on the inlet stream, at the breakthrough points (three points combined into one sampling line) and in the exhaust stream.

Project Objective.

The primary aim of the study was to demonstrate the applicability of Fourier-Transform Infra-red (FTIR) spectroscopy in the quantitative profiling of a complex mixture of solvents in process streams subject to variable conditions.

Secondary study aims were to:

- Quantify emissions to atmosphere of determinands regulated under the plant's IPC authorisation thereby enabling the significance of losses to vent to be established versus total losses as calculated by mass balance.
- Determine the efficiency of operation of the primary and secondary carbon beds.
- Correlate emission profiles with individual process operations.

Monitoring and Analysis Methods.

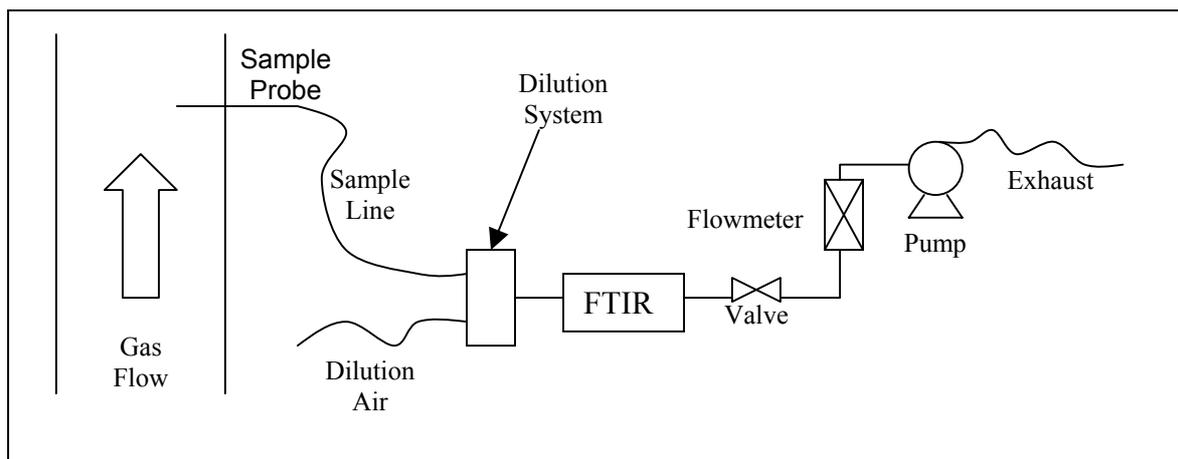
Measurement of Concentration.

Throughout the study Protea employed two BOMEM 9100 FTIR analysers fitted with 6.4m path-length gas cells operating at 120°C. Prior to operation, the spectrometers and gas cells were thoroughly purged with dry nitrogen gas and instrument background spectra were recorded.

During monitoring periods spectra were collected at 1 cm⁻¹ resolution, using Bomem CAAP data acquisition software. Spectral data were averaged and recorded over 16 spectra acquired approximately every 2 minutes. Data were interpreted post collection using GRAMS32 software.

Sampling positions and protocols were varied during the study to assess the impact of heated versus non-heated lines and to allow concentrations from various process positions to be determined. Where appropriate, dilution systems were set up directly prior to the spectrometers to bring determinand concentrations within the range of the analysers. The dilution systems used consisted of two rotameters feeding sample gas and dilution (ambient) air into a mixing chamber, adjustment of the rotameters enabled an appropriate dilution ratio to be set (typically 4:1) – all results quoted in this report have been adjusted to account for the dilution factor set. It is recognised that the dilution systems employed were not ideal and increased the uncertainty of the determined values. This increase in uncertainty is not considered to significantly impact on the validity of data obtained for the stated purposes of this study, however if accurate absolute values are required it is recommended that a more robust, validated dilution system be employed.

The sampling set-up is illustrated below:



A PTFE heated sampling line, operating at 120°C, was temporarily installed onto the exhaust stream close to the unheated sample line permanently installed to serve existing analysers.

Determination of Analyte Concentration.

Absorption bands of all components suspected to be present in the process were pre-chosen for quantitative analysis. Due to the high range of measured concentrations it was necessary to use different absorption bands for individual components dependent upon the concentration of these components present in sample gas. Quantitative library spectra were used to identify and quantify the compounds in the sample spectra. These spectra were generated from gas cylinders of certified concentration.

Detection limits for known components are considered to lie between 1 and 5 mg/m³ under ideal conditions. Detection limits will be higher in the presence of high concentrations of interfering components, thus the limit of detection for any individual component will vary over the study period dependent upon the type and concentration of other components present at the time of measurement. Limits of detection will further be impacted by the sample dilution factor applied.

Unknown species were detected during the study period. It is believed that these comprised two components, an additional solvent and an organic aldehyde. Comparing residual spectra with library spectra identified the additional solvent. Searches against Protea's in-house database as well as several (NIST Standard Reference Database, Thermo Galactic 'Spectra Online' and USEPA vapour phase FTIR library) alternative commercial databases failed to identify the suspected aldehyde. The identified solvent was quantified using a response factor typical of a related compound. It is recognised that there will be significant error associated with this assumption, more accurate quantification could be attained by calibrating with a reference standard of the solvent, however no such suitable reference material could be sourced within the timeframe of this study.

Monitoring of Flow, Temperature and Pressure.

Flow measurements in the exhaust stream were provided by the plant operator, in m³/hr, from an on-line flow meter. Temperature and pressure data are not routinely monitored and were not collected for the purposes of this study.

Flow is not routinely measured at any point in the process other than at the exhaust, it was therefore not possible to calculate mass transfer rates at either the inlet or first carbon bed breakthrough section.

Experimental.

Monitoring was conducted between 19/6/02 and 3/7/02. Appendix 2 (Table 1) details the location and duration of the various monitoring exercises undertaken.

With two spectrometers available to monitor from three positions (inlet, first carbon bed breakthrough and exhaust) it was necessary to monitor for correlations between the sampling positions separately (i.e. Breakthrough/Exhaust, Inlet/Exhaust and Inlet/Breakthrough). Additional monitoring was undertaken to assess the efficiency of the existing unheated sampling line versus a line maintained at 120°C to avoid condensation of water or analytes.

Results.

All results are presented in Appendix 3.

Discussion.

Flow.

Exhaust flow measurements for the whole monitoring period are shown in Figure 4, with an expanded time axis shown in Figure 5 for clarity.

Over the entire monitoring study the average exhaust flow was 1049 m³/hr with a range between 102 – 2720 m³/hr. Statistical information on flow for each period when emissions from the exhaust vent were monitored is given in Table 2.

The flow changes periodically with high flow being associated with the Steam Blow-off from the regeneration bed at the end of the regeneration process, this association was confirmed by visual observation of the emission of a steam plume coinciding with periods of maximum flow.

The statistical data suggests that the maximum flow periodically exceeds the maximum flow measurable by the on-line flow meter. Figures 4 and 5 show that such exceedances are transitory and suggests that maximum values will not be significantly greater than those quoted. In this report, for the purposes of mass emission calculations, the measured flows are taken as being accurate.

Comparison between Heated and Unheated Sample Lines.

To assess the efficiency of existing sampling lines a heated line was installed on the exhaust vent on 27th June. The heated line was a standard 10 m electrically heated PTFE tube inlet and the inlet was located close to the existing sample line inlet. The line was maintained at 120°C to ensure that all emitted components would be maintained in the vapour phase.

Comparative monitoring commenced at 11:20 on 27th June and ceased at 12:22 on 28th June. As can be seen from Figure 6 emission profiles for Solvent A from heated and unheated sample lines follow almost identical trends, however significant differences were recorded in absolute concentrations with the average Solvent A concentration from the unheated line being 47% below that from the heated line (see Tables 3 and 4).

Whilst most other components show a similar trend as Solvent A the water profiles differ considerably (Figure 6). The smoothing of the water emissions graph is almost certainly due to condensation occurring in the unheated line with this condensate being transported to the analyser over time; note that the average water concentration from the unheated line is only 1.4% lower than from the heated line. The differences in percentage loss between water and Solvent A are not readily explained. Whilst a potential reason is preferential loss through sampling line joints, further studies would be required to investigate this thoroughly.

The differences between heated and unheated lines in terms of mass emission are significantly different from the concentration variations. This is due to the correlation between analyte concentration and flow and is dealt with in detail later.

The water profiles from the heated line show that water concentration spikes are occasionally present between spikes associated with the steam blow-off process (see below). There does not appear to be a clear correlation between these spikes and Solvent A concentrations, however there is correlation with Solvent B concentrations (Figure 7) although the concentrations of Solvent B emitted coincidentally with these intermittent water spikes are not as high as the concentrations associated with the Steam Blow-off.

Exhaust Emissions.

Summary statistics for emissions from the exhaust vent are given in Table 5. In line with the original monitoring plan exhaust emissions were monitored throughout the study except during a period between 26/6/02 and 27/6/02 when the analysers were employed to study correlations between inlet and breakthrough values.

Figure 8 clearly demonstrates the relationship between flow and total concentration of solvent emissions, with the sharp increase in flow associated with the Steam Blow-off from the regeneration bed, as previously described, being accompanied by even sharper rises in total solvent concentration.

Table 5 summarises all data obtained from monitoring the emission vent. As it has been previously shown that significant sample losses occur when using an unheated as opposed to heated sample line only data obtained using heated sample lines is considered in the following discussion.

Table 6 summarises the significance of emissions in general and the significance of emissions associated with the Steam Blow-off process specifically. Note that ‘emissions associated with the Steam Blow-off process’ have been taken as a 12 minute peak with peak integration starting with the periodic step change in vent flow.

The summary statistics demonstrate the considerable variability between the two monitoring periods with average solvent losses per cycle of 2.51 kg between 27th and 28th June but only 0.30 kg between 28th June and 3rd July. Whilst sampling / analysis problems cannot be entirely ruled out, the cause of this variability is probably process related. A distillation process, during which process chemists have indicated that they would anticipate the most significant losses, was operating for the majority of the former monitoring period but not at all during the latter. Throughout all exhaust vent monitoring (including monitoring via the unheated line) seven peaks of > 20,000 mg/ hour were recorded, all of these coincide with distillation.

The relative abundances of individual components also showed marked variation between monitoring periods (Table 7). In particular Solvent C was twice as abundant from 28th June to 3rd July versus 27th to 28th June whereas Solvent A dropped in relative abundance from 72% to 57% over the same period.

Comparison between Inlet and Breakthrough Concentrations.

Figure 9 demonstrates the efficiency of the carbon beds in adsorbing and recovering Solvent A with concentrations of over 60,000 mg/m³ at the inlet to the beds producing breakthrough concentrations of less than 600 mg/m³.

To a limited extent breakthrough concentrations are related to the time a bed has spent in the primary adsorber position, this is demonstrated by the sharp falls in breakthrough concentrations at various points in the monitoring period (e.g. 15:53 and 19:14 on 26th June) – these times correspond to bed changes.

Any relationship between inlet and breakthrough concentrations appears to be less significant than differences between beds themselves. Breakthrough from Bed 2, which was in-line on 26th June from 15:56 – 17:26 and 20:58 – 22:28 and on 27th June from 01:54 – 03:31 and 06:57 – 08:21, is consistently lower than from Beds 1 and 3 (with Bed 3 breakthrough being lower than Bed 1).

Recovery of Solvent D is much less efficient than recovery of Solvent A, with intermediate recovery of Solvent C as shown in Figures 10 and 11. There are marked changes in recovery efficiency – the maximum inlet concentration for Solvent A over the monitoring period being approximately 64,000 mg/l versus a maximum breakthrough concentration of approximately 560 mg/l; the equivalent figures for Solvent C and Solvent D being 25000 / 1300 mg/l and 3400 / 2300 mg/l respectively.

The changes in recovery efficiency correlate well with material boiling points, which range from >40°C for Solvent A down to <5°C for Solvent D.

Comparison between Breakthrough and Exhaust Concentrations.

It is not possible to draw any conclusions about any potential correlation between component concentrations at bed breakthrough and final exhaust due to interference from emissions during Steam Blow-off as described earlier. The extent to which Steam Blow-off emissions mask any potential background is illustrated in Figure 12

It is also, therefore, impossible to ascertain the impact of running two carbon beds in series on background emissions from the process. Should it prove possible to eliminate (or at least significantly reduce) emissions associated with the Steam Blow-off it would be practical to re-examine the efficacy of the secondary carbon bed.

Comparison between Inlet and Exhaust Concentrations.

No correlation between inlet and exhaust concentrations has been considered as masking of any such correlation by Steam Blow-off emissions is to be expected as above.

Advantages of Profiling Methodology.

Profiling gaseous concentrations through use of technology such as portable FTIR can be immediately seen to offer significant advantages from a developmental perspective in that it allows concentrations to be tightly correlated with production activity. The detailed information given by profiling helps engineers and chemists to gain a better understanding of the process, in turn enabling the process to be better controlled.

From an environmental monitoring perspective profiling offers an additional advantage in instances where both flow and analyte concentration are variable. When sampling liquid effluents the default position is to use a flow-proportional sampler to ensure any subsequent analytical results are representative of the stream sampled. Use of automated isokinetic sampling equipment that provides a link between vent flow and flow through the sampling system also provides flow-proportional sampling for the monitoring of particulate or aerosol based matter in gas vents.

However, when sampling gases that are believed to be homogenous within vents potential correlations between vent flow and analyte concentration are frequently ignored. This is possibly as a result of vent monitoring methodology being derived from occupational hygiene type methods where results are assessed versus a time-weighted concentration limit value.

Provided either the vent flow or the concentration is constant, time-weighted sample results can be multiplied by the total flow to give a mass flow that is accurate within the limitations of the method. When flow and analyte concentration are variable throughout the monitoring period flow-proportional sampling or simultaneous continuous monitoring of both flow and analyte concentration is required to give an accurate measure of emitted load.

Solvent A vent emissions in this report can be used as an excellent example of the type of errors that can be introduced through indiscriminate time-weighted sampling:

During monitoring of the vent exhaust to atmosphere from the carbon adsorbers via heated line 28th June to 3rd July 2002 the average vent flow was 1048 m³/hour. Over the same period FTIR analysis showed the average emission of Solvent A to be 67.37 mg/m³, by time-weighted average sampling the mass Solvent A emitted was therefore:

$$1048 \times 67.37 / 1000 = 70.6 \text{ g/hour}$$

As has previously been demonstrated a significant proportion of Solvent A losses to atmosphere occur during the Steam Blow-off, a part of the operating process that is also associated with high vent flow rate. As both the vent flow and the Solvent A concentration were simultaneously recorded it is possible to profile mass loss of Solvent A to atmosphere by multiplying the two figures for each time point. Doing so and then summing the losses and dividing by number of hours sampled to give the same units as above produces a result of:

$$98.7 \text{ g/hour}$$

Time-proportional sampling would therefore underestimate losses of Solvent A to atmosphere by over 28% if applied to the vent from the carbon bed adsorbers.

Conclusions.

This case study has demonstrated the coupled benefits of FTIR technology that are on-line and real time monitoring. The plant operator has been provided with species profiling data which when correlated against production activity will allow, amongst other benefits, identification of process activities that lead to high and low periods of solvent emissions. In addition, the benefit of the multi-species capability of FTIR spectroscopy was demonstrated in that the existence of a previously unseen solvent was discovered. Through comparison of the measurements to reference library spectra strong evidence was found as to the identity of the species.

It was demonstrated that approximately 60% of the exhaust emissions originated from the Steam Blow-off of the regenerating bed. There are clear environmental and cost benefits to be gained from reducing this contribution to the emissions and consequently increasing the plant recovery efficiency. Perhaps the most obvious method by which this might be realised would be to increase the length of time of the regeneration cycle with the aim of stripping a greater proportion of the trapped solvents from the carbon bed. The plant operator has estimated that improved solvent recovery would save ~£60k per annum. Coupling this with further savings through the optimisation of the carbon bed replacement programme which currently costs ~£15k per annum (bed lifetime is correlated to the frequency of regeneration) highlights the significant cost savings that process measurements of this type can yield. Also in this study there was a correlation found between relative breakthrough abundance and component boiling point, specifically, the higher the boiling point of the species the greater the efficiency with which it appeared to be recovered. Lastly, it was also found that using an un-heated sample line lead to artificially low concentrations of the target analytes.

Recommendations.

Some specific recommendations can be made relating to the plant monitored. These provide examples of the type of information that FTIR measurements can provide to plant operators.

- Solvent loss reduction initiatives should initially be strongly focused on eliminating the emission spikes associated with the ‘Steam Blow-off’ of the carbon bed being regenerated.
- The specification of the existing on-line flow monitor associated with the carbon bed vent should be re-evaluated and a system capable of monitoring flow, to known uncertainty, across the full range of flows encountered should be installed.
- All future samples taken from the carbon bed system for extractive analysis (at- or off-line) should be taken via heated line (minimum temperature 120°C)
- All monitoring of mass emissions from the carbon bed vent should utilise simultaneous profiling of flow and analyte concentration or flow-proportional sampling.

APPENDIX 1: Process Schematics.

Figure 1

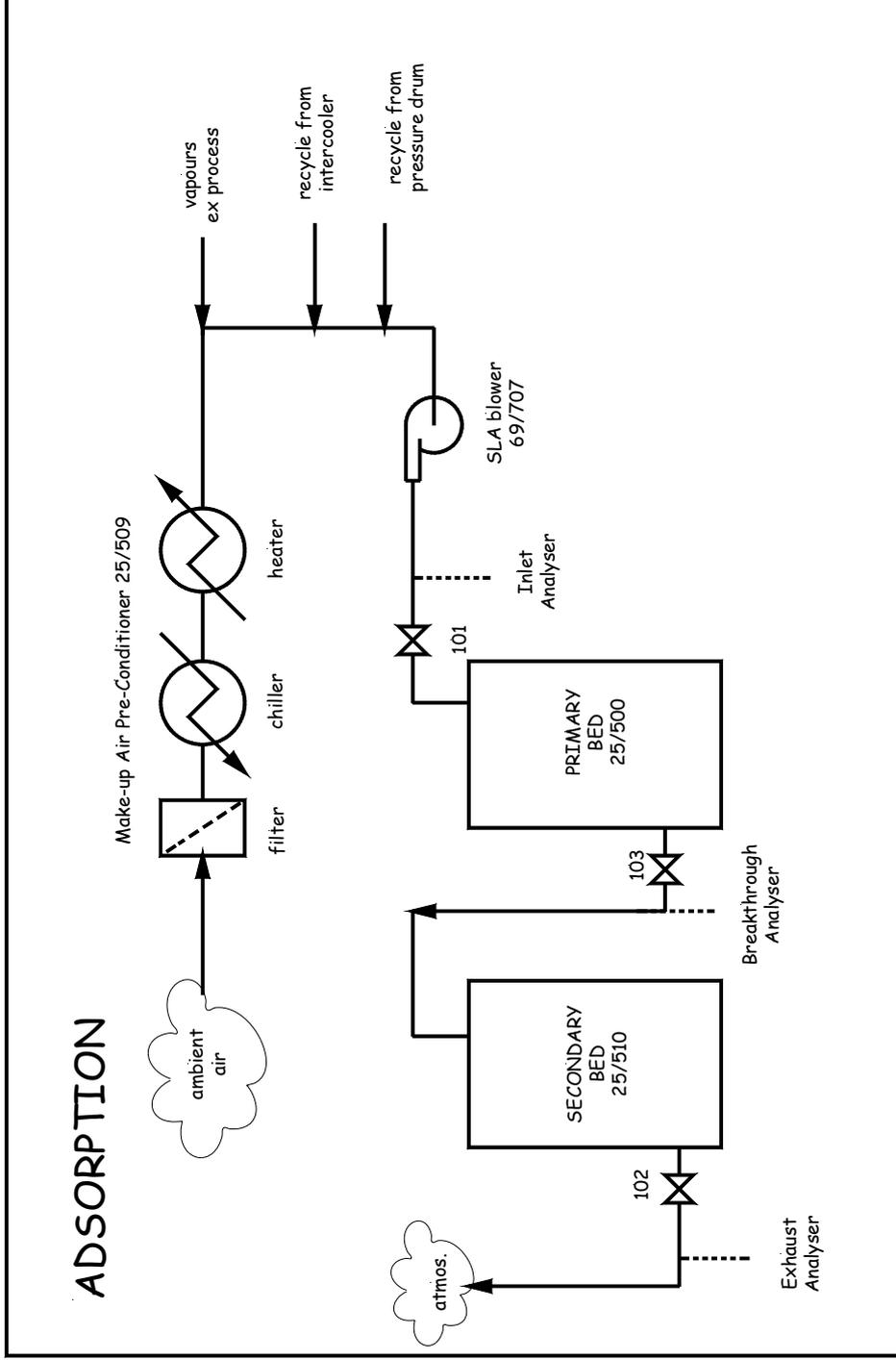


Figure 2

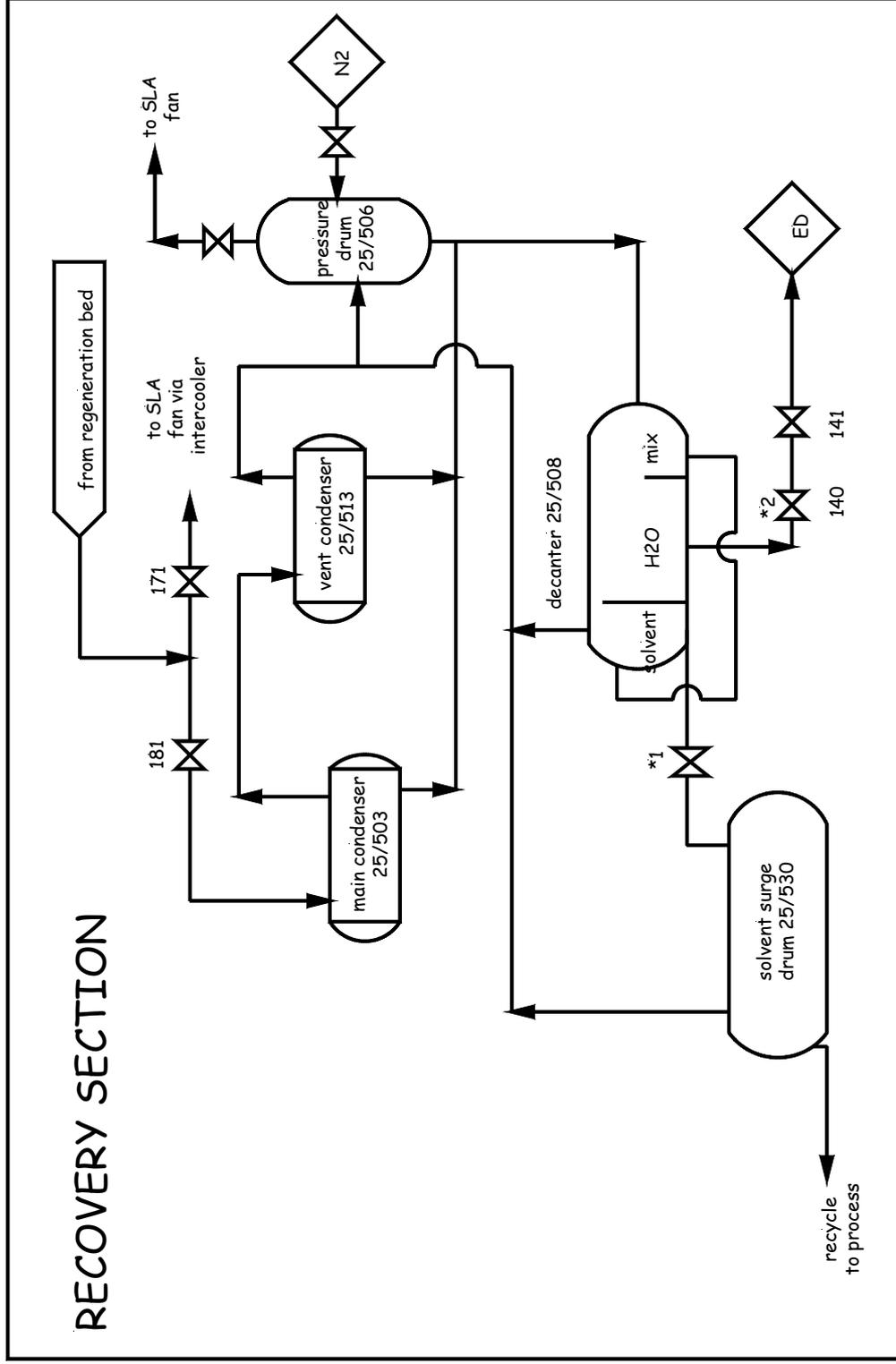
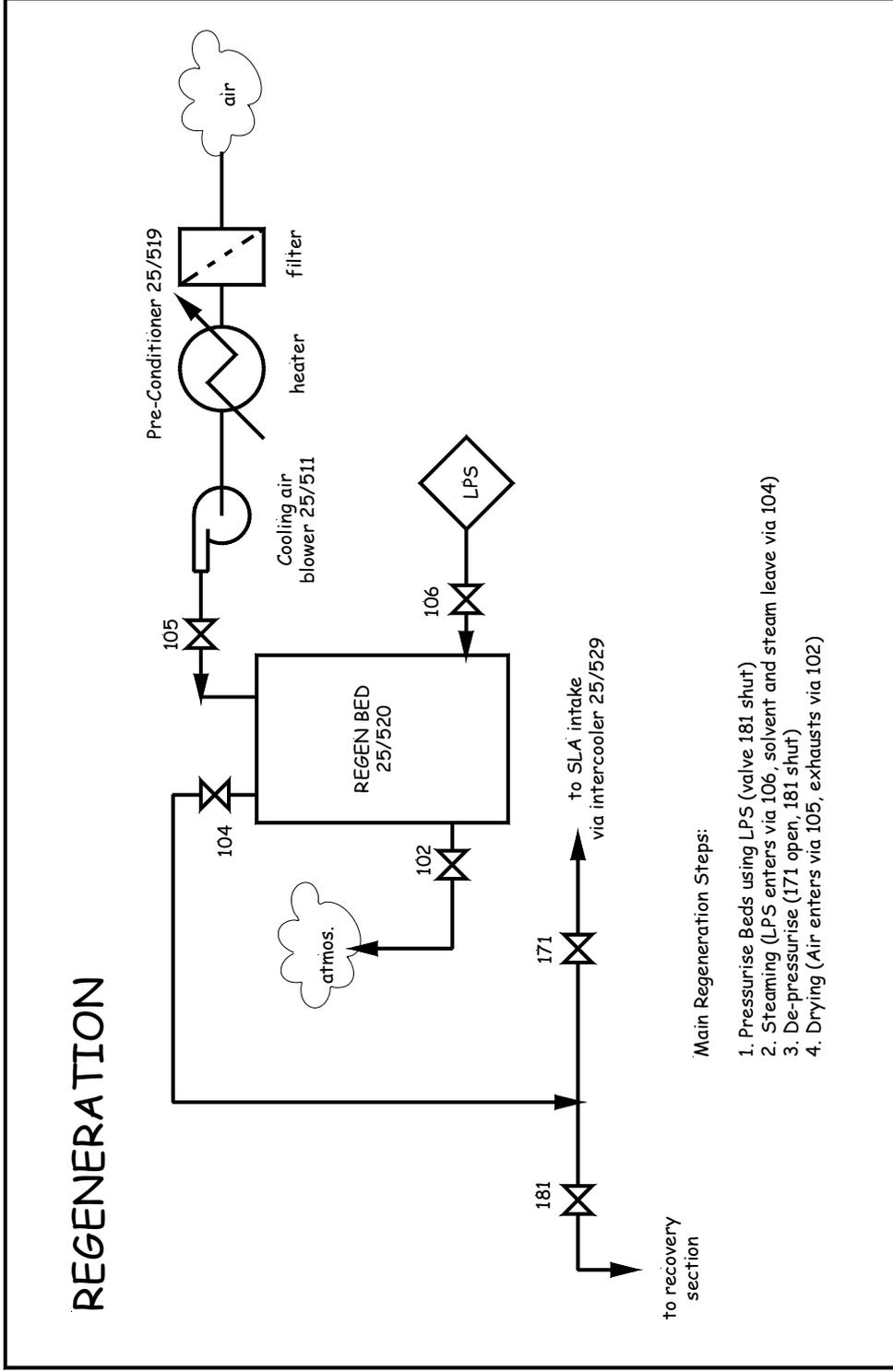


Figure 3



APPENDIX 2: Monitoring Periods.

Table 1 – Monitoring Summary

Duration	Monitoring Points	Spectrophotometer Used
20/6/02 16.58- 25/6/02 9.44	Breakthrough	N
	Exhaust (unheated line)	A
25/6/02 15.19-26/6/02 12.31	Inlet	N
	Exhaust (unheated line)	A
26/6/02 12.52- 27/6/02 10.51	Inlet	N
	Breakthrough	A
27/6/02 11.20- 28/6/02 12.22	Exhaust (heated line)	A
	Exhaust (unheated line)	N
28/6/02 12.44- 3/7/02 10.01 Regeneration time extended	Breakthrough	N
	Exhaust (heated line)	A

APPENDIX 3: Concentration & Flow Data.

Figure 4

Carbon Adsorption System - Exhaust Flow Rate, m3/hr

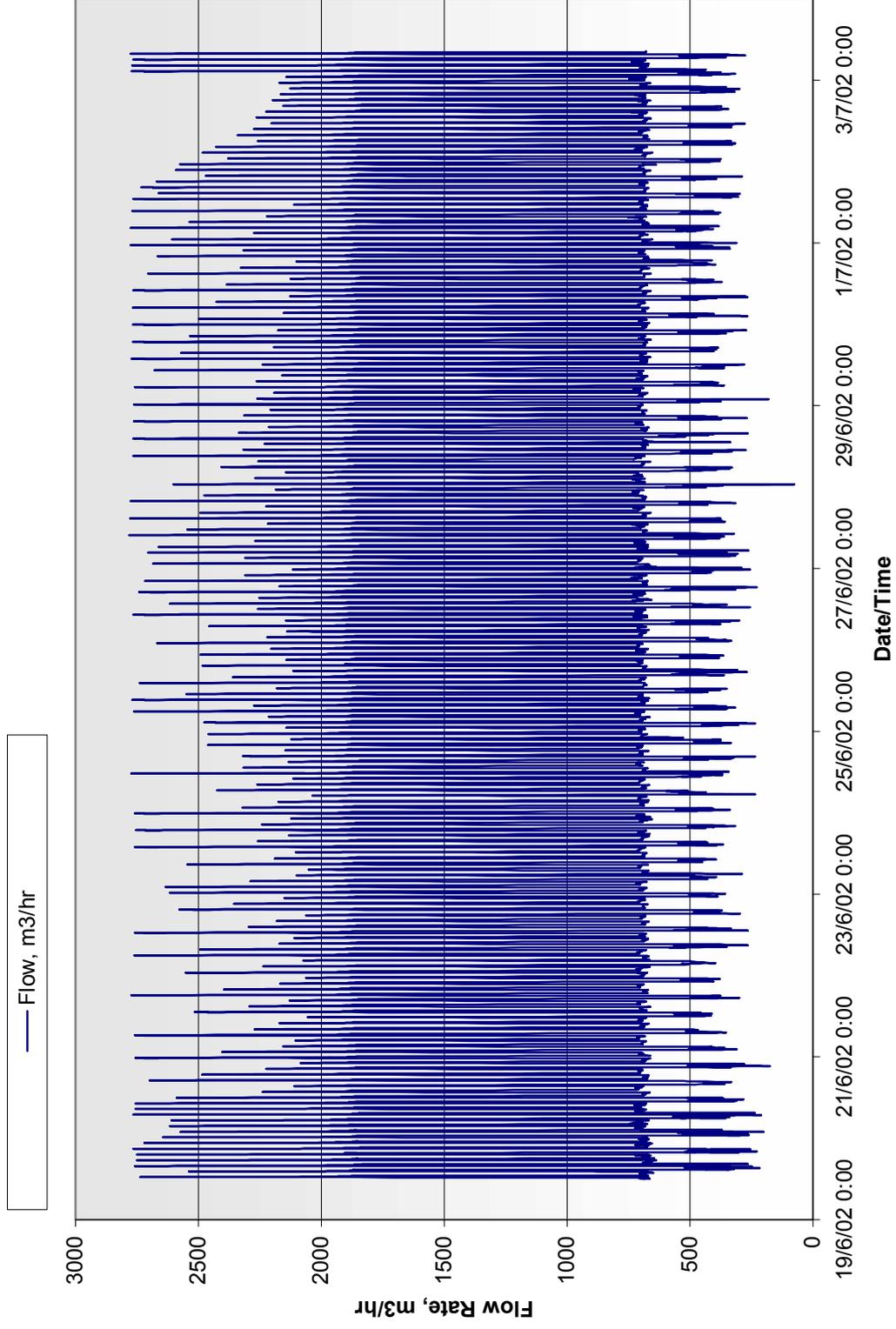


Figure 5

Exhaust Flow 21st June 2002

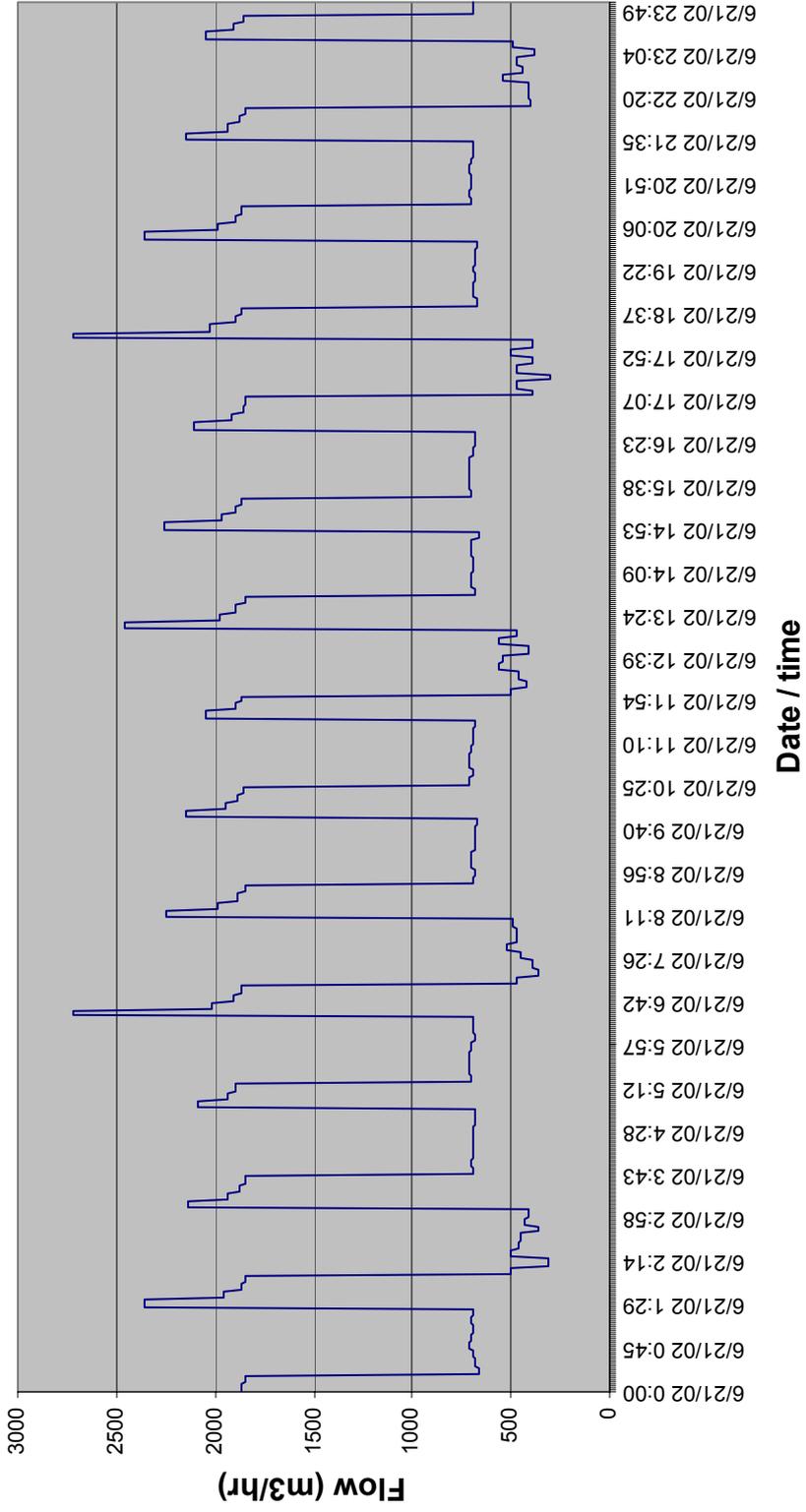


Table 2 – Exhaust Flow Statistics

Monitoring Period	Total Flow (m3)	Maximum Flow (m3/h)	Minimum Flow (m3/h)	Average Flow (m3/h)
20/6/02 16.58- 25/6/02 09.44	119309	2720	182	1058
25/6/02 15.19- 26/6/02 12.31	22684	2720	269	1070
26/6/02 12.52- 27/6/02 10.51	Exhaust emission not monitored			
27/6/02 11.20- 28/6/02 12.22	26454	2720	102	1058
28/6/02 12.44- 03/7/02 10.01	122875	2720	199	1048

Figure 6

Comparison between Heated and Unheated Sample Lines

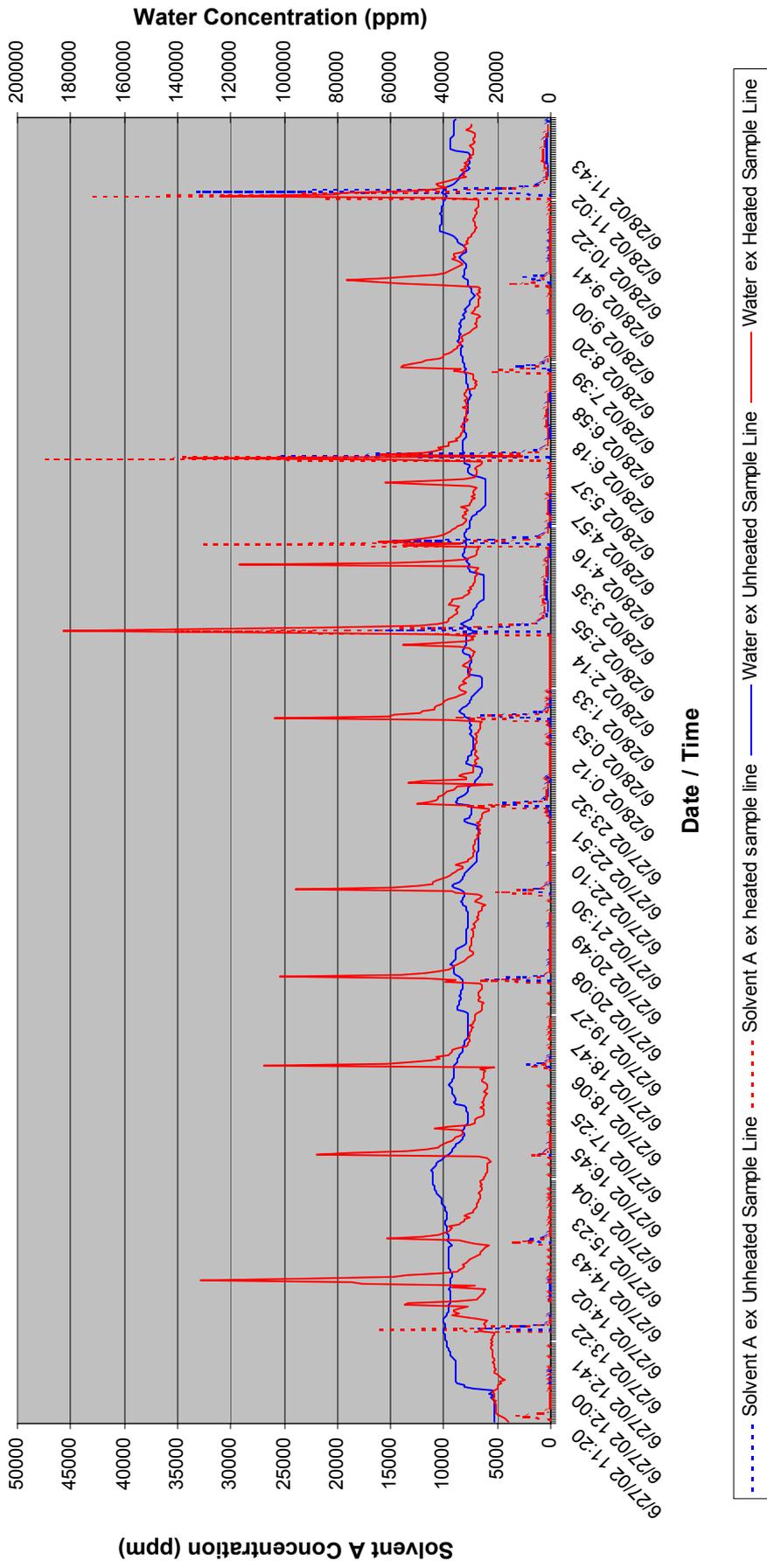


Table 3 – Average Exhaust Gas Concentrations Sampled Through Heated and Unheated lines 27 – 28th June 2002

Component	Heated Line Average Concentration	Unheated Line Average Concentration	% Loss (Unheated line versus heated line)
Solvent A	879 (mg/m ³)	470 (mg/m ³)	47
Solvent B	68 (mg/m ³)	46 (mg/m ³)	32
Solvent C	96 (mg/m ³)	76 (mg/m ³)	21
Water	26870 (mg/m ³)	26488 (mg/m ³)	1.4
Carbon monoxide	11.4 (mg/m ³)	7.0 (mg/m ³)	39

Table 4 – Average Exhaust Gas Mass Emission Rates Sampled Through Heated and Unheated lines 27 – 28th June 2002

Component	Heated Line Average Emission Rate	Unheated Emission Rate	Line Average Rate	Average	% Loss (Unheated line versus heated line)
Solvent A	1084 (g/hr)	546 (g/hr)	546 (g/hr)		50
Solvent B	91 (g/hr)	53 (g/hr)	53 (g/hr)		42
Solvent C	171 (g/hr)	111 (g/hr)	111 (g/hr)		35
Water	31.4 (kg/hr)	27.9 (kg/hr)	27.9 (kg/hr)		11
Carbon monoxide	63 (g/hr)	36 (g/hr)	36 (g/hr)		43

Figure 7

Correlation Between Solvent B and Water Emissions

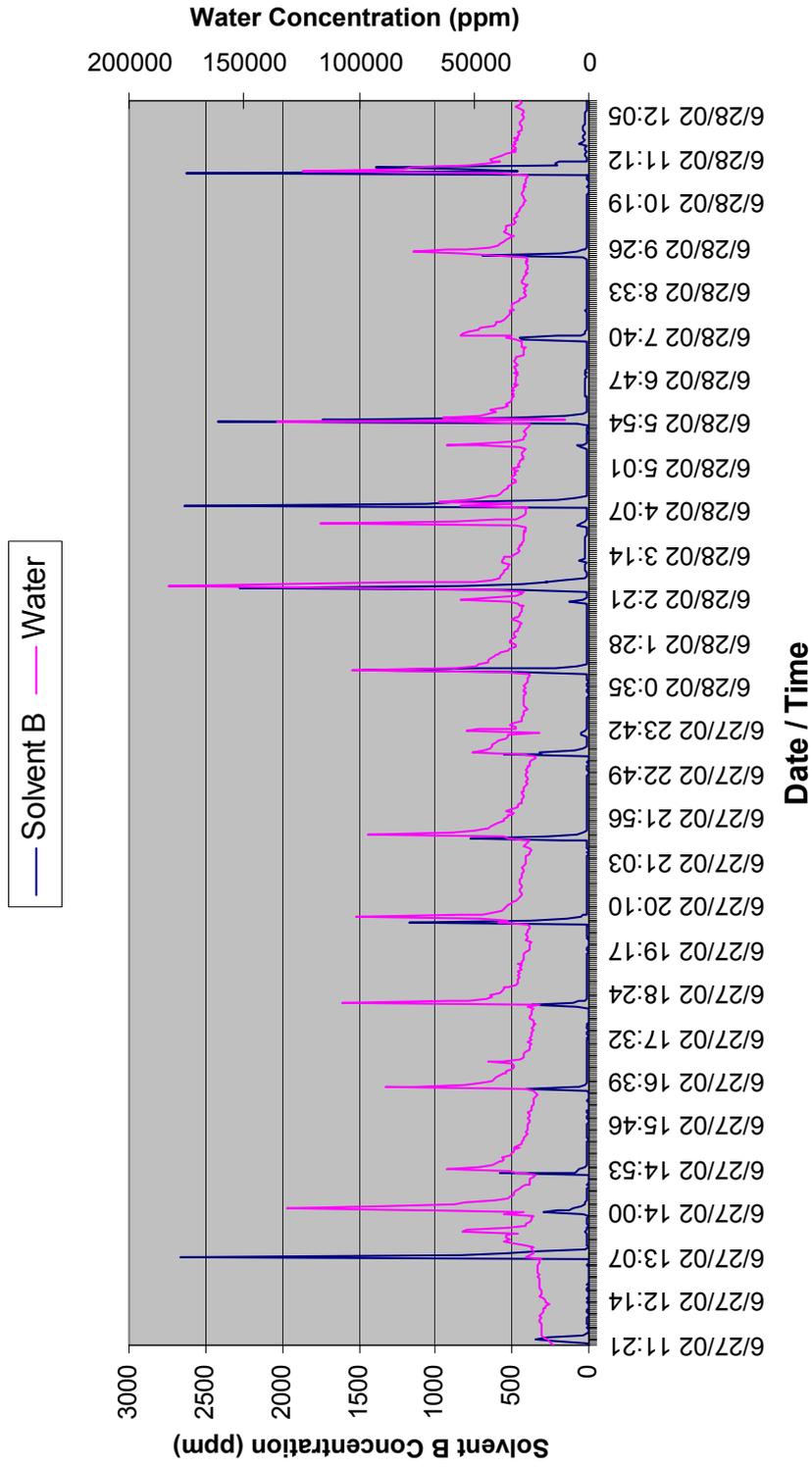


Figure 8

Total Solvent Emission Concentrations Versus Flow 27th - 28th June 2002

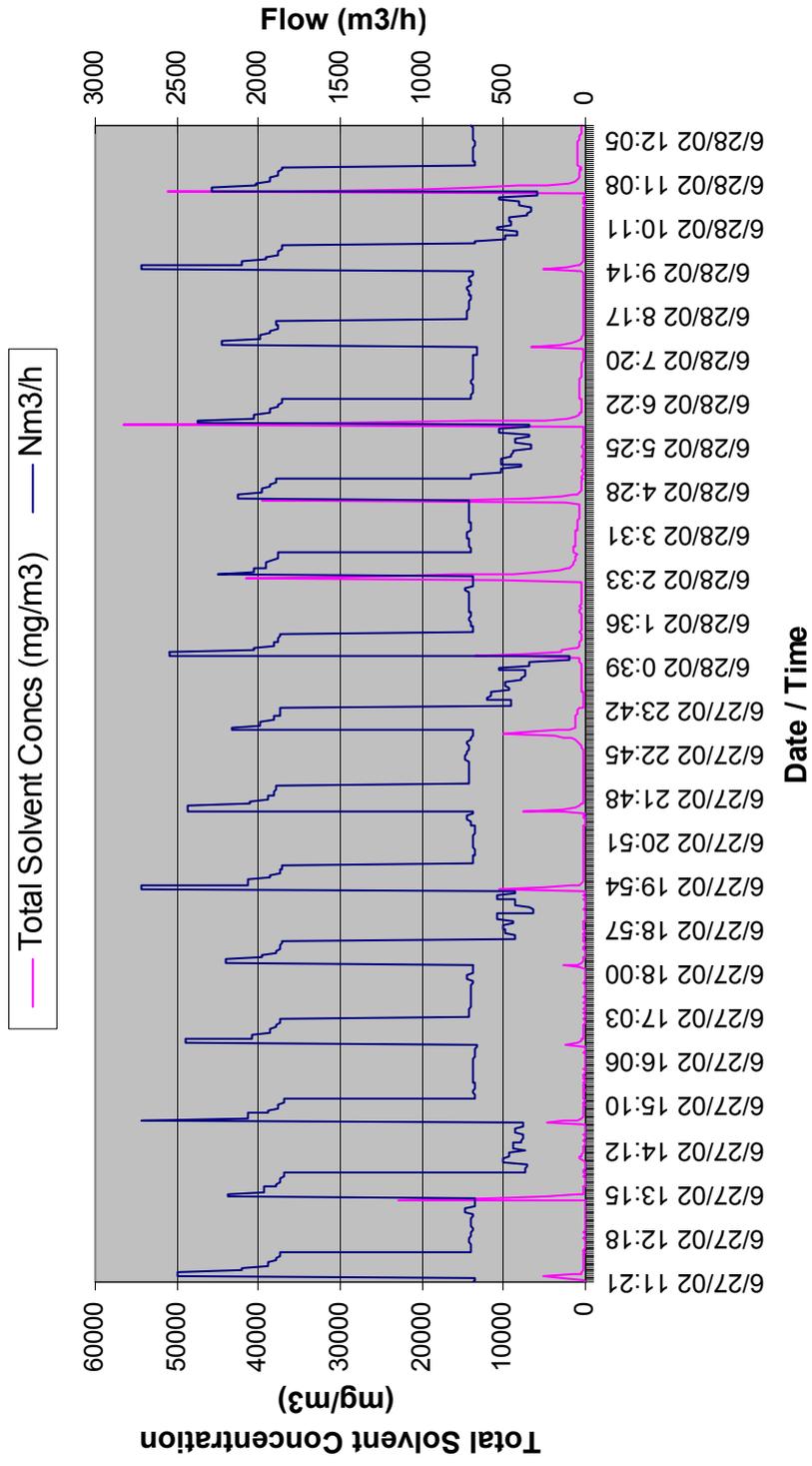


Table 5 – Exhaust Emission Summary

Values across monitoring period	Component			
	Solvent A	Solvent B	Solvent C	Solvent D
20/6/02 16.58- 25/6/02 9.44 (Unheated Sample Line)				
Average Concentration (mg/m3)	60	19	22	13
Maximum concentration (mg/m3)	1550	2830	3420	873
Average Mass Flow (kg/hr)	0.016	0.025	0.029	0.009
Maximum Mass Flow (kg/hr)	0.876	7.50	9.07	1.13
25/6/02 15.19-26/6/02 12.31 (Unheated Sample Line)				
Average Concentration (mg/m3)	275	27	26	17
Maximum concentration (mg/m3)	15100	1620	2040	829
Average Mass Flow (kg/hr)	0.307	0.033	0.028	0.019
Maximum Mass Flow (kg/hr)	23.7	1.78	1.91	0.753
27/6/02 11.20- 28/6/02 12.22 (Heated Sample Line)				
Average Concentration (mg/m3)	879	68	96	121
Maximum concentration (mg/m3)	50500	2855	1820	3000
Average Mass Flow (kg/hr)	1.08	0.091	0.171	0.157
Maximum Mass Flow (kg/hr)	73.3	4.39	5.72	5.31
28/6/02 12.44- 3/7/02 10.01 (Heated Sample Line)				
Average Concentration (mg/m3)	67	12	25	14
Maximum concentration (mg/m3)	1200	1450	1490	655
Average Mass Flow (kg/hr)	0.099	0.016	0.039	0.021
Maximum Mass Flow (kg/hr)	3.08	1.87	3.77	1.23

Table 6 – Significance of Exhaust Emissions

Monitoring Period	27/6 – 28/6	28/6 – 3/7
Total Mass emission (kg)	37.6	20.6
Number of bed regenerations	15	68
Average mass emission per regeneration cycle (kg)	2.51	0.30
Solvent emission associated with Steam Blow-off (kg)	23.1	12.2
% mass emission associated with Steam Blow-off	62	59

Table 7 – Relative Exhaust Emission Abundance

Component	Solvent A	Solvent B	Solvent C	Solvent D
27/6 – 28/6 Average mass emission (g/hour)	1084	91	171	157
27/6 – 28/6 Relative abundance (%)	72	6	11	10
28/6 – 3/7 Average mass emission (g/hour)	99	16	39	21
28/6 – 3/7 Relative abundance	57	9	22	12

Figure 9

Solvent A Inlet versus Breakthrough

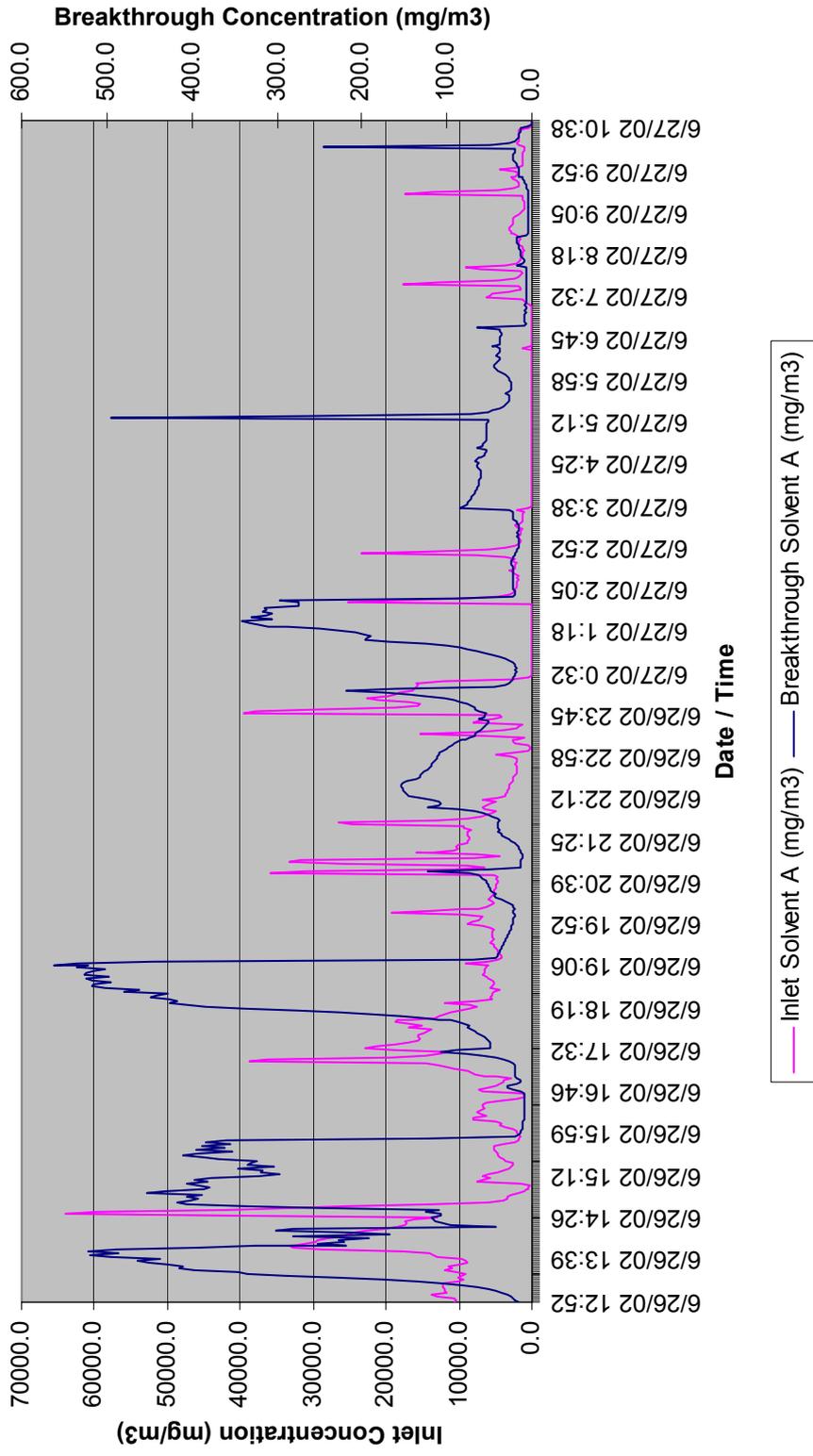


Figure 10

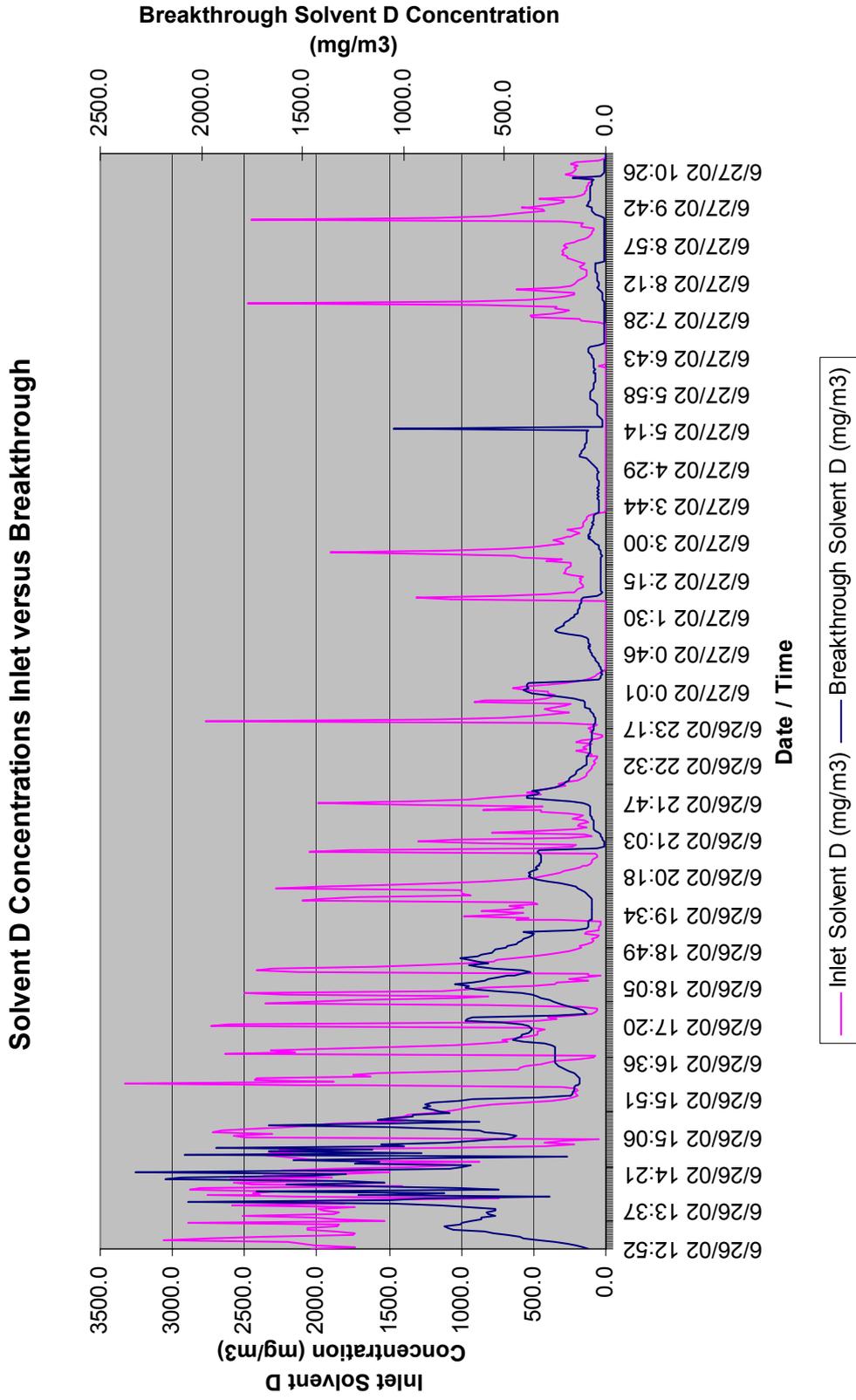


Figure 11

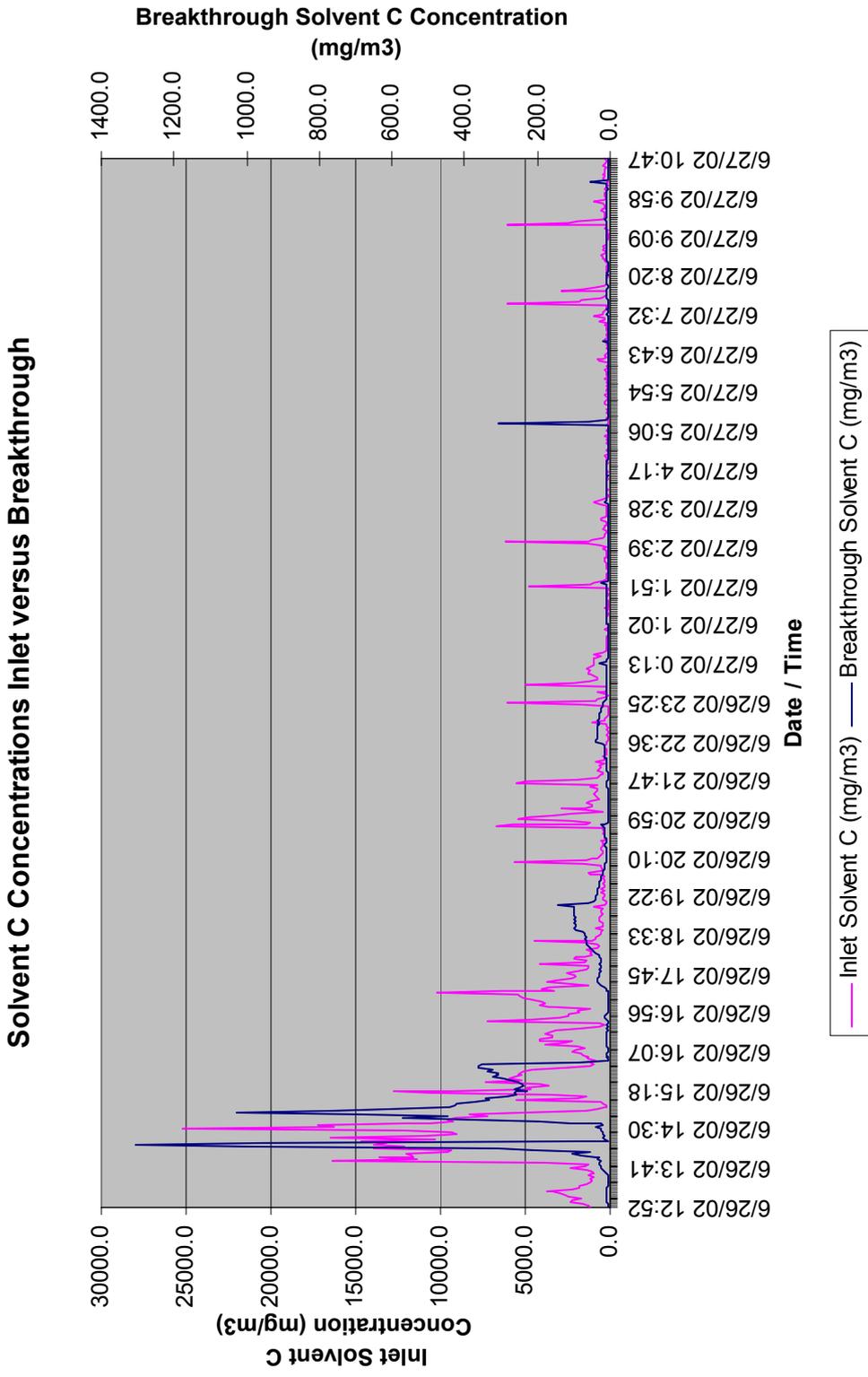


Figure 12

Solvent A Concentrations Exhaust versus Breakthrough

