

BRIAN MILLIGAN Associates

Consultants in Occupational & Environmental Hygiene

**MEASUREMENT OF EMISSIONS
FROM A CYLINDER WASHER AT
FABDEC LTD.
MAY 2017**

Report prepared for:

Mr Brian Gough
Fabdec Ltd.
Grange Road
Ellesmere
Shropshire SY12 9DG

Survey carried out and reported by:



Brian D. Milligan, Chartered Occupational Hygienist

Report date: 27th May 2017

Total number of pages: 6

EXECUTIVE SUMMARY

A survey of gaseous emissions to atmosphere from a cylinder washing machine was carried out on the 3rd May 2017. The aim was to determine whether emissions of hydrogen fluoride and oxides of nitrogen complied with the concentration limits set out in official guidance.

The methodology was based on the method previously set out in Appendix 2 of PG 4/1(04) : *Measurement of total oxides of nitrogen* (now superseded) . The emission limit values below are those appearing in DOE Process Guidance Note PG 4/1(13)

Oxides of nitrogen	200 mg/m ³
Hydrogen fluoride	5 mg/m ³

One sample was taken over one complete wash cycle. The concentrations of oxides of nitrogen (1.7 mg/m³) and hydrogen fluoride (<0.1 mg/m³) were both below the maximum permissible limits.

In the following pages, a more detailed account of the work undertaken, and of the calculations employed to derive the results, is presented.

INTRODUCTION

This report concerns measurements carried at Fabdec Ltd, Grange Road, Ellesmere, Shropshire SY12 9DG on the 3rd May 2017, at the request of Mr A. McFarlane. The aim of the measurements was to determine the emission concentrations of acid mist/vapour from a cylinder washing machine which contained a mixture of nitric and hydrofluoric acids and was exhausted through a ducting system to a fan and hence to outside the building above roof level.

One sample was taken, over a complete wash cycle of 75 minutes (including the fixed 45 minute wash/rinse cycle and further loading/unloading time).

EMISSIONS FROM METAL TREATMENT PROCESSES

Emission Standards

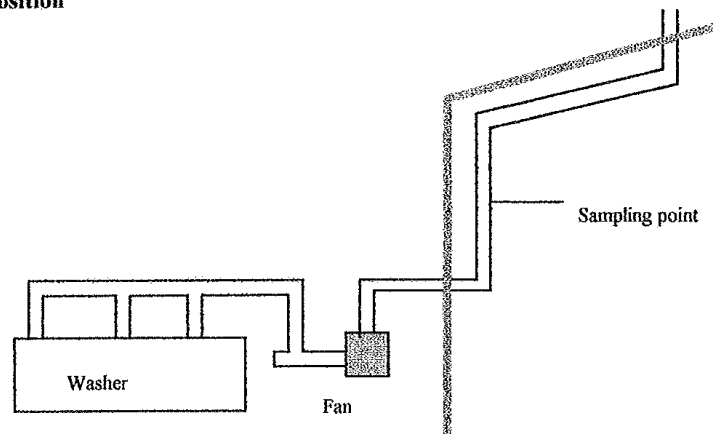
This process is listed as a part B process with Local Authority control under the Environmental Protection Act 1990 and the Pollution Prevention and Control Act 1999.

DEFRA has published Process Guidance Note PG 4/01(13) setting out emission limits as follows:

Oxides of nitrogen	200 mg/m ³
Hydrogen fluoride	5 mg/m ³

All measurements are to be calculated at standard temperature and pressure (0°C, 101.3 kPa), without correction for water vapour.

Sampling Position



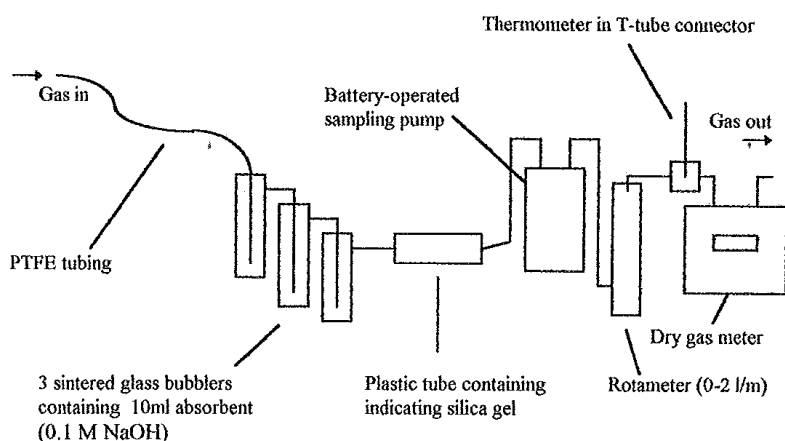
Method

The sampling method is based on that previously printed in Appendix 2 of PG 4/1(04) Although this guidance has been superseded, the methodology complies with the requirements of the current guidance. There are some deviations from the published method.

- Miniature sintered glass bubblers were used instead of Dreschel bottles so the liquid volumes used were fifteen times smaller.

- b) Instead of a 50 litre/minute mains-operated pump and needle valve, a battery-operated personal sampling pump was used at a flow rate of 1.0 litre/minute.
- c) Since ion chromatography of both nitrite and nitrate is possible, there was no need for any potassium permanganate in the absorber solution.

Before and after the sampling, the stack temperature was determined using an electronic thermometer equipped with a K-type probe, and the stack velocity was determined using a standard pitot tube and electronic micro-manometer (two 10-point traverses at right angles). The sampling train was prepared as shown below.



The prescribed procedure detailed in the Appendix to PG 4/1(04) was closely followed and is summarised below.

1. Check the production conditions to ensure that the sampling period will be representative of typical operational conditions.
2. Leak test the equipment by blocking the probe and observing no air flow through the bubblers or through the rotameter and gas meter. Insert the probe so as to sample from a central point within the stack.
3. Switch on the pump, note the time and gas meter reading. Note the barometric pressure.
4. Adjust the flow to 1.0 l/minute at the rotameter.
5. Periodically check the level of liquid in the bubblers and if necessary top up to the correct level with deionised water. Re-adjust the flow rate to 1.0 l/min as necessary.
6. Periodically note the temperature of the sampled gas as it enters the gas meter.
7. After a complete machine cycle, switch off the pump and remove the probe from the stack. Note the gas meter reading and check the barometric pressure.
8. Empty the contents of the bubblers into a sample bottle. Rinse the probe, PTFE tube and bubblers with deionised water, using a plastic filter funnel and wash-bottle, and add the washings to the sample solution.

The sample solution, together with a sample of blank absorbent reagent, was analysed for nitrate, nitrite and fluoride by ion chromatography [SAL Ltd., Hadfield House, 9 Hadfield Street, Manchester M16 9FE].

CALCULATIONS & RESULTS

1. Determination of Sample Volume

Sampling time	12.41-13.56
Sample volume by gas meter	68 litres
Gas meter calibration factor	1.00
Barometric pressure	102.5 kPa
Mean temp of gas flow at meter	292 K

Hence, volume of gas sampled adjusted to STP $68 \times 1.00 \times 102.5/101.3 \times 273/292$
 $= 64.33$ litres

2. Determination of Concentration

Total volume of absorbent solutions and washings 35 ml

Analyte	Concentration in sample ug/ml	Concentration in blank ug/ml	Total ug collected in 33 ml	As NO ₂ equivalent ug	As HF ug
NO ₃	0.17	<0.05	5.95 }	109.8	-
NO ₂	3.6	0.59	105.35 }		
F	<0.05	<0.05	<2	-	<2

Concentration of NO₂ in gas stream = mass collected/volume sampled
 $= 109.8/64.33$
 $= 1.7 \text{ mg/m}^3 \text{ (@ STP)}$

Concentration of HF in gas stream = mass collected/volume sampled
 $= <2/64.33$
 $= <0.1 \text{ mg/m}^3 \text{ (@ STP)}$

3. Determination of Mass Emission Rate

Internal diameter of stack	0.200 m
Internal area of stack = $\pi \times (0.200/2)^2$	0.0314 m ²
Mean gas velocity at stack temperature and pressure	7.2 m/sec
Stack temperature	290 K
Stack pressure (0.05 kPa above atmospheric)	102.5 kPa

Gas flow rate at STP $= 7.2 \times 0.0314 \times 102.5/101.3 \times 273/290 = 0.215 \text{ m}^3/\text{sec}$

Emission rate of NO₂ $= 0.215 \times 1.7$ **0.4 mg/sec**

Emission rate of HF $= <0.215 \times 0.1$ **<0.1 mg/sec**

SUMMARY OF RESULTS

	Nitrogen Oxides as NO₂ mg/m³@STP	Hydrogen Fluoride mg/m³@STP
Measured concentration	1.7	<0.1
Permissible concentration	200	5

CONCLUSION

1. The concentrations of both nitrogen oxides and hydrogen fluoride were below the permissible concentration set out in the relevant Guidance Note.