

Transfeu WP2 Subtask 2.1.2 Development of small-scale test method for fire effluents

Step 5 : Development of calibration method

Smoke chamber test – FTIR analyser calibration procedure

Collection of training spectra using standard concentration gas bottles

According to DoW

► Step 5: Development of calibration method

Partners: LSFIRE, LNE, RATP, VTT, SP, CUR

The optimum calibration procedures will be carried out in parallel by LSFIRE, LNE, RATP, VTT, SP and CUR. These procedures will be established by checking of thermal attack and by measurements of gases, with particular attention to the quantitative measure for the expected kind of gases.

15. Forward

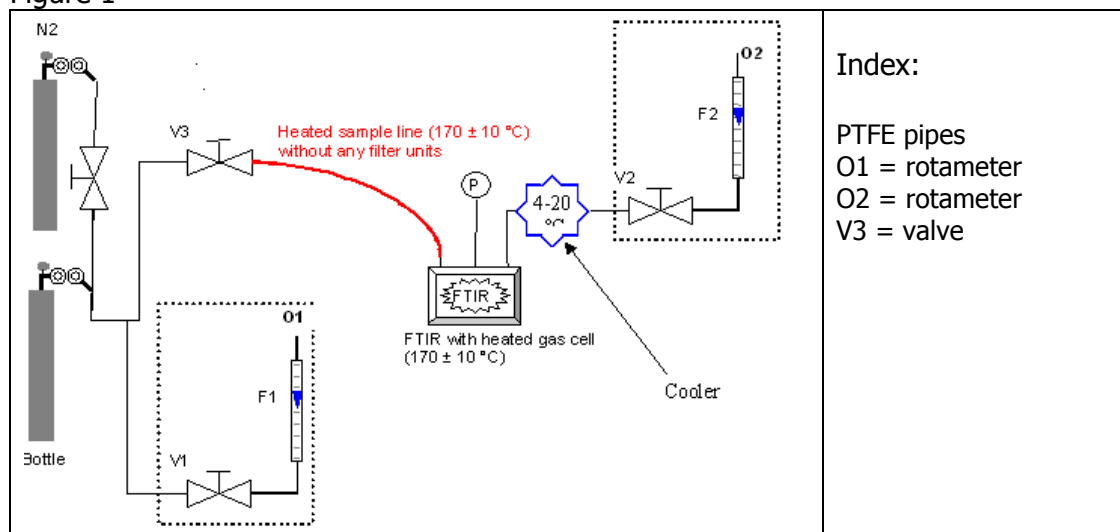
The principle of this calibration is to collect a series of absorbance spectra (training spectra) for each gas species to be analysed in the project using standard bottles gas mixture (certified) in order to obtain a minimum quantitative spectral library to be able on calibration method building according to existing several chemo metrical techniques.

The number of standard bottles (different concentrations) to be chosen for each gas species is based on considerations concerning the concentration range for prediction and the behaviour of the gas (linear or non-linear). 5 to 6 concentrations for non-linear gases and 3 to 4 concentrations for linear gases is a good optimum.

16. Set up of apparatus

The collection of training spectra shall be done using the same experimental settings (cell path length, resolution, temperature of the transfer line, ...) with which you will collect real smoke gas spectra. The scheme of the calibration procedure to be used is shown in figure 1.

Figure 1



Recommendations:

- The length of pipe between the calibration bottles and V3 shall be as short as possible (max 200mm) to avoid the condensation of halogen gas species (HCl, HBr) before the heated sample line inlet point.
- The rotameters O1 and O2 shall be the max flow rate range of 4 l/min with a minimum reading steps of 0,1 l/min.
- The pressure transmitter (P) should be able to real time read the cell absolute pressure output values with a sensitivity of mbar unit (working range from vacuum to 3bar absolute press.). Its sensor shall not be a extension probe with a volume not more than 2% of the gas cell volume. It shall be able to working at temperature $\leq 200^{\circ}\text{C}$.
- The gas cell shall have a volume $\leq 0,4$ liters to avoid a too long response time (not more than 15s with a sampling flow rate of 1.5 l/min) and a minimum IR beam pathlength of 3m.
- The spectral resolution to be used shall be in the range between 0.5 and 2.0cm^{-1} .
- The Mid-IR spectral range shall be set at $600\text{-}4400\text{ cm}^{-1}$.
- The number of scans per spectra shall be as high as possible (noise reduction) in order to have a spectrum collection time not more than 10s.

17. Calibration procedure

- a. Wait that all parts in the scheme of figure 1 reach the temperature steady-state for a minimum time of 30 min.
- b. Keeping close the standard gas mixture and nitrogen bottles, open totally valves V1, V2 and close valve V3.
- c. Open nitrogen bottle and adjust F1 to 2 l/min.
- d. Open V3 slowly until atmospheric pressure looking P output monitoring.
- e. Adjust F2 with V2 to 1.5 l/min.
- f. Wait 1 minute for flow rate and pressure (P) stabilization
- g. Collect background spectrum and measure the pressure P during collection
- h. Close nitrogen bottle and valve V3
- i. Open totally valves V1 and V2
- j. Open standard gas mixture bottle and adjust F1 to 2 l/min.
- k. Open V3 slowly until atmospheric pressure looking P output monitoring.
- l. Adjust F2 with V2 to 1.5 l/min.
- m. Wait 1 minute for flow rate and pressure (P) stabilization
- n. Collect absorbance spectrum (training spectrum) and measure the pressure P during collection
- o. Close the standard gas mixture bottles and purge all the line introducing nitrogen for a minimum period of 1min.
- p. Proceed successively with other standard mixture bottles (from step "h" to "o") using the same background spectrum collected in the step from "c" to "g".

Recommendations:

- The use of the same background is limited to the one daily calibration period. Each day when the calibration process will be started, a background spectrum (nitrogen sampling) shall be collected firstly. The background as also absorbance spectra are strictly related to the absolute pressure value measured in the cell (including atmospheric pressure).
- Each gas species spectra series (different concentration standard bottles mixture) shall be collected at the same absolute pressure (cell) with a maximum variation of ± 10 mbar.
- The type of gas species in nitrogen mixture, the concentration range and minimum nr. of points (different concentrations for training spectra collection) are shown in the table 1.

Table 1 - Gas concentrations for standard bottles (in nitrogen)

Gas	Concentrations range (ppm/%)	Nr. Of Bottles (nr. of points)
CO + CO ₂ / N ₂	From 50 ppm + 0.005% To 5000 ppm + 3.5%	5
HCN / N ₂	From 10 ppm To 400 ppm	5
SO ₂ / N ₂	From 20 ppm To 300 ppm	3
HCl / N ₂	From 50 ppm To 5000 ppm	5
HF / N ₂	From 50 ppm To 500 ppm	4
HBr / N ₂	From 50 ppm To 1000 ppm	5
Acrolein / N ₂	From 20 ppm To 250 ppm	3
Formaldehyde / N ₂	From 20 ppm To 250 ppm	3
NO / N ₂	From 10 ppm To 200 ppm	3
NO ₂ / N ₂	From 20 ppm To 750 ppm	3
Ammonia / N ₂	From 20 ppm To 300 ppm	3

18. Use of calibration results on test data.

To be completed

19. Report

To be completed

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