Spectronus FTIR trace gas analyser

N_2 purge requirements and correction for CO in N_2

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Preamble

This document addresses two aspects of minimising and correcting for residual CO absorption due to (1) CO buildup in the FTIR instrument and (2) CO in the N_2 purge gas.

1. Left unpurged, CO accumulates in the FTIR spectrometer as it is emitted from the metal walls and components of the interferometer compartment. With a 0-10 LPM rotameter/flowmeter for N_2 purge installed in the early Spectronus, even the minimum setting of 0.1-0.2 SLPM results in a G-cylinder lifetime of about 1 month. This is more than the flow required to maintain an acceptable purge. By reducing the flow to 50-70 mL/min, a G-cylinder should last 2-3 months.
2. Even with N_2 purge, industrial and even high purity N_2 contains a significant amount of CO, typically around 200 ppb in instrument or industrial grade nitrogen. This background CO contributes to absorption in both background and sample spectra. If the background CO in the purge N_2 remains constant with time, the CO absorption cancels, but if it changes the apparent CO amount also changes. This note describes how to measure and correct for changing CO amount in the N_2 purge gas.

Purge rate.

Left unpurged, in a typical UoW analyser CO accumulates in the FTIR spectrometer at a rate of ~ 150 ppb day^{-1}, which is equivalent to ~ 5 ppb day^{-1} in the cell. (The pathlength inside the spectrometer is approximately 0.8 m and the cell pathlength is 26 or 24 m for metal and glass cells respectively, so the cell-equivalent amount is ~ 30 times less than the actual amount in the spectrometer). CO_2 and CH_4 accumulate at lower, usually negligible rates. The CO accumulates in a volume of approx. 10 L, so the actual cell-equivalent accumulation rate is 50 ppb-L day^{-1}. If this were diluted into a N_2 flow of 100 L day^{-1}, the equilibrium CO amount would be 0.5 ppb, and its variability much less. This is acceptable for all but the most demanding clean air measurements.
Note we do not have data for many spectrometers, and take 5 ppb day^{-1} as typical.
A N_2 flowrate of 100 L day^{-1} = 70 mL min^{-1}. A G-size cylinder holds 7.2 m^3 = 7200 L, so the cylinder should last 72 days, or comfortably 2 months with 12 days buffer to schedule regular 2-monthly tank replacements.
Reducing the flow to 50 mL min^{-1} increases the cylinder lifetime to 100 days.

Recommendations

Replace the current 0-10 LPM rotameter if fitted with a 0-100mL rotameter. Recommended purge rate is 70 mL min^{-1} to keep background CO emitted from the instrument enclosure to less than an equivalent of 0.5 ppb, with proportional increase in flow leading to proportional decrease in background CO.
Alternatively use an external independent mass flow controller to control N_2 purge flow. The flow can conveniently be monitored in a spare Spectronus external AI channel. A spare AO channel can be used to set a flow control voltage for the MFC if required; the disadvantage of this option is that the flow stops when Spectronus is not running.
For remote locations, N_2 tank pressures can also be monitored by Spectronus with the use of tank electronic pressure gauges such as Wika model A-10. The gauge readouts can be connected to spare AI channels for logging in the Spectronus database.

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Correction for residual CO in N_2 purge gas

Even when the instrument is purged, the CO content of the N_2 purge gas is not negligible. CO in the purge gas can be measured directly by Spectronus by filling the cell with the purge gas, FTIR spectrum measurement and analysis using a special set of MALT parameter files suited to the low concentrations of other gases. Typically an inlet line can be teed off from the N_2 supply line and connected to a spare inlet of the Spectronus for regular measurements during calibration cycles. A measurement task can be configured to fill the cell with N_2 from this inlet and measure it, load the N_2 MALT analysis parameters, analyse the mole fractions of CO, CO_2, CH_4, N2O and H_2O and return the Spectronus to its standard operating conditions before the next sample. In general only the CO mole fraction is significant.

The required MALT PRM files and a sample task for N_2 measurement are available from the file area of the ECOTECH Spectronus website as part of the example Measure_demo.tsk task, or by contacting ECOTECH.. The task may need minor modification by the user to adapt to individual user setups (for example, inlet number, pressure or flow rate).

The purge gas analysis provides the true mole fraction of CO in the purge gas. For correction purposes the effective CO amount is reduced by the ratio of the cell pathlength (26 m for the metal cell, 24 m for glass cell) to that inside the FTIR (0.8 m), a factor of 32 (metal cell) or 30 (glass cell).This CO absorption adds to both background and sample measurements. If CO-in-N_2 composition does not change with time, this CO absorption cancels between background and sample measurement and no correction to measured CO is required. The additional CO will be added to the intercept term in the linear calibration procedure. However if the CO-in-N_2 drifts with time, or changes step-wise when changing purge gas cylinder, the change should be measured and used to correct measured sample CO amounts or the calibration intercept term.
For example if CO-in-N_2 from two consecutive tanks differs by 50 ppb, the equivalent CO difference between the two tanks is 50/32 = 1.6 ppb. Measured CO in a sample should be corrected by this amount to allow for the change in background CO between tanks.

Recommendations

1. Measure the CO-in-N_2 mole fraction at the beginning and end of the life of each N_2 purge tank, and at the time of each calibration with standards. Measure more often if drift in the CO amount is suspected.
    - The N_2 measurement task can be fully automated and included after each calibration in a routine operation task.
2. The CO amount at the time of background measurement and/or calibration should be subtracted from the CO amount at the time of sample measurement (interpolated between CO-in-N2 measurements) to calculate an additive CO correction.

Revision history

July 2015	V1	Original version
Aug 2018	V2	Revised for website. Includes CO in N2 measurement and correction strategy

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