N_2O detection by gas filter correlation technique using a mid-infrared light emitting diode

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Gas Filter Correlation (GFC) technique [1] is an established sensing approach that was developed at the early thirties of the 1900's The operational principle is based on the quantification of the infrared radiation absorption at specific wavelengths. The technique is very sensitive to a target gas due to the use of a serial combination of filters that can cut off and pass radiation in a particular wavelength band from an incoherent broad band infrared source.
Even if it is an old technique, there are currently several commercial devices based on GFC for N_2O sensing like the Thermo 46i and the Teledyne GFC700T due to its low cost, sensitivity and selectivity performance. Figure 1-a shows the typical configuration and the main components (infrared source, gas filter reference cell, band pass filter, sample cell and detector) of a spectrometer based on gas filter correlation. The most common infrared sources are filaments with their black body e.m. emission spectrum (i.e. Kanthal), since they are cheap, small and requires low energy to achieve high emission even if in a broad spectrum. The reference gas cell (also known as filter wheel) is a key component that consist in a closed chamber contained both the target gas to be measure (N_2O) and a transparent infrared gas (N_2). The bandpass filter is used to cut off and transmit the broad radiation emission over a well-defined wavelength band. The sample cell usually has the White configuration (3 passes with internal gold mirrors). Finally, the photodetectors that work in the IR are commercially available Peltier cooled MCL photovoltaic junctions with good sensitivity.

GFC is also known as self-reference technique, in a simple operation (see fig. 1-b), the light pass through the reference cell as well as the sample cell and once the filter wheel (which is located between the IR source and sample cell) rotates, the IR energy is transmitted in the case of N_2 (scenario I in fig. 1-b) and, it is attenuated by N_2O (scenario II in fig. 4-b) contained in the reference cell. As a result, the difference in energy is inversely proportional to the amount of the species in the sample cell (scenario III in fig. 4-b). This has the advantage of providing more sensitivity at lower measured concentrations. Interferent species present in the gas sample attenuate both the sample and reference signals equally and will not be measured (scenario IV in fig. 1-b).

In our work, we used a typical GFC configuration with a commercial gas filter wheel (containing N_2 and N_2O) as reference cell, a white cell as sample cell, a band pass filter and a photodiode as sensor. A Filament and a mid-infrared LED where both used as different infrared sources. Fig. 1-c shows the experimental set up of GFC using LED emitting at about 4.5 μm. The commercial LED46-TRW model, with a maximum of emitting wavelength about 4.40 - 4.66 μm (FWHM 800 - 1100 nm) that operates in Quasi Continuous Wave mode (4 - 12 μW) mounted in TO-5 package, was provided by ROITHNER LASERTECHNIK GmbH, Austria. The vacuum in the sample cell was obtained by using a double stage rotary pump. A less expensive diaphragm pump is sufficient for a flow detection.

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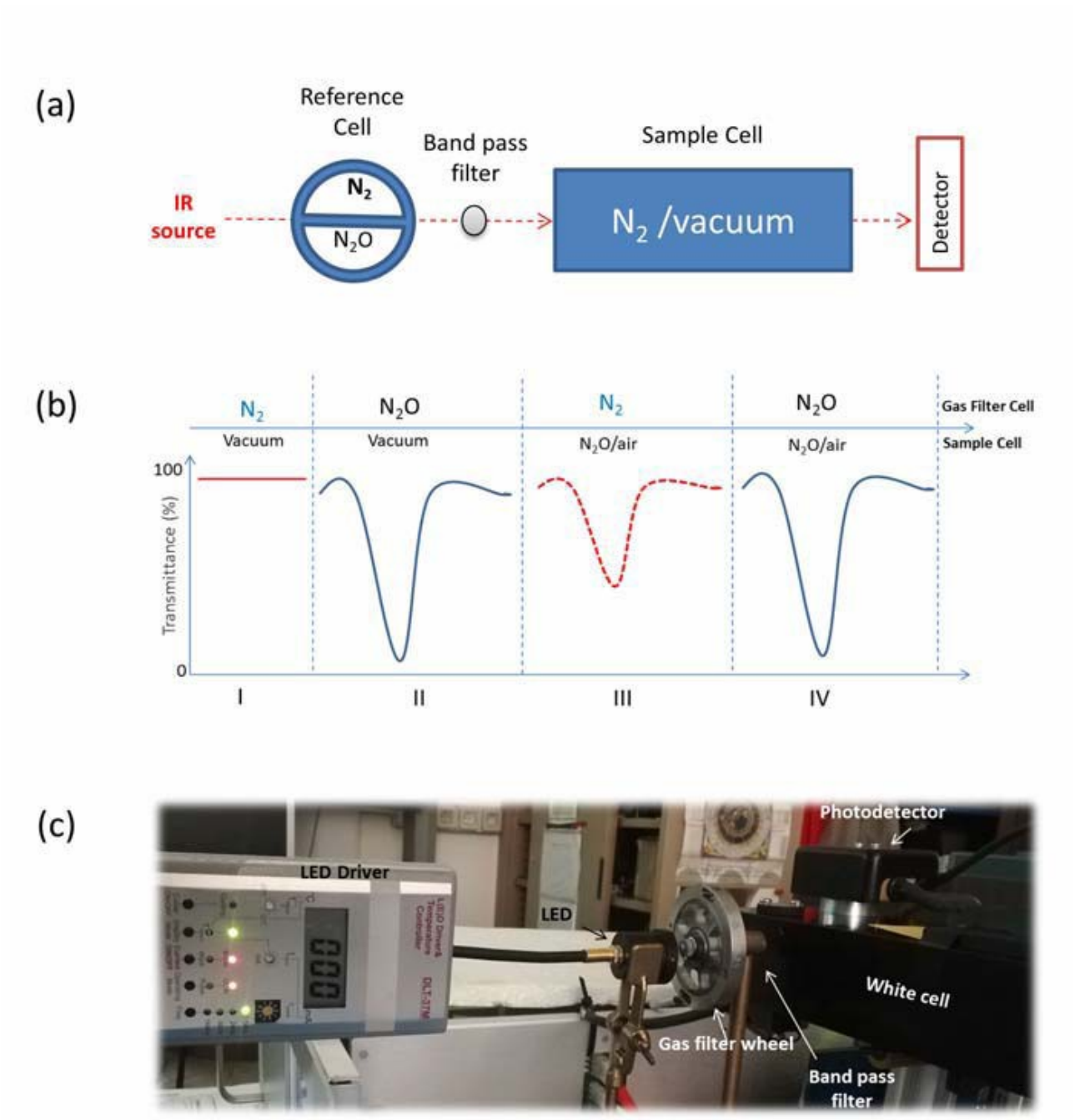


Figure 1. Gas filter correlation (a) typical configuration, (b) signal response at the different configurations (reference/sample cells) and (c) experimental set-up using in the laboratory.

Figure 2 illustrates the GFC response obtained using three different concentration of N_2O in the sample cell (P = 0, 1.6 and 6.5 Torr), a filament as infrared source and an optical chopper with 6 slots in each reference cell. The two different regions of the reference cell are clearly observed at P = 0 Torr (the pressure is reference to the maximum vacuum achieved by the vacuum system) as the black line in the figure. once the pressure of N_2O was increased in the sample cell, an attenuation in the signals (N_2 in reference cell) is observed. The difference is thus proportional to the gas concentration since not attenuation was observed for N_2O. Further increments in the N_2O pressure (P = 6.5 Torr) in the sample cell caused a "saturation" due to attenuation for all signals.

Figure 3 shows the response of the system under different concentration of N_2O; t is evident how the system works very well with linear response for low concentrations (Pressure below 8 - 10

Torr).

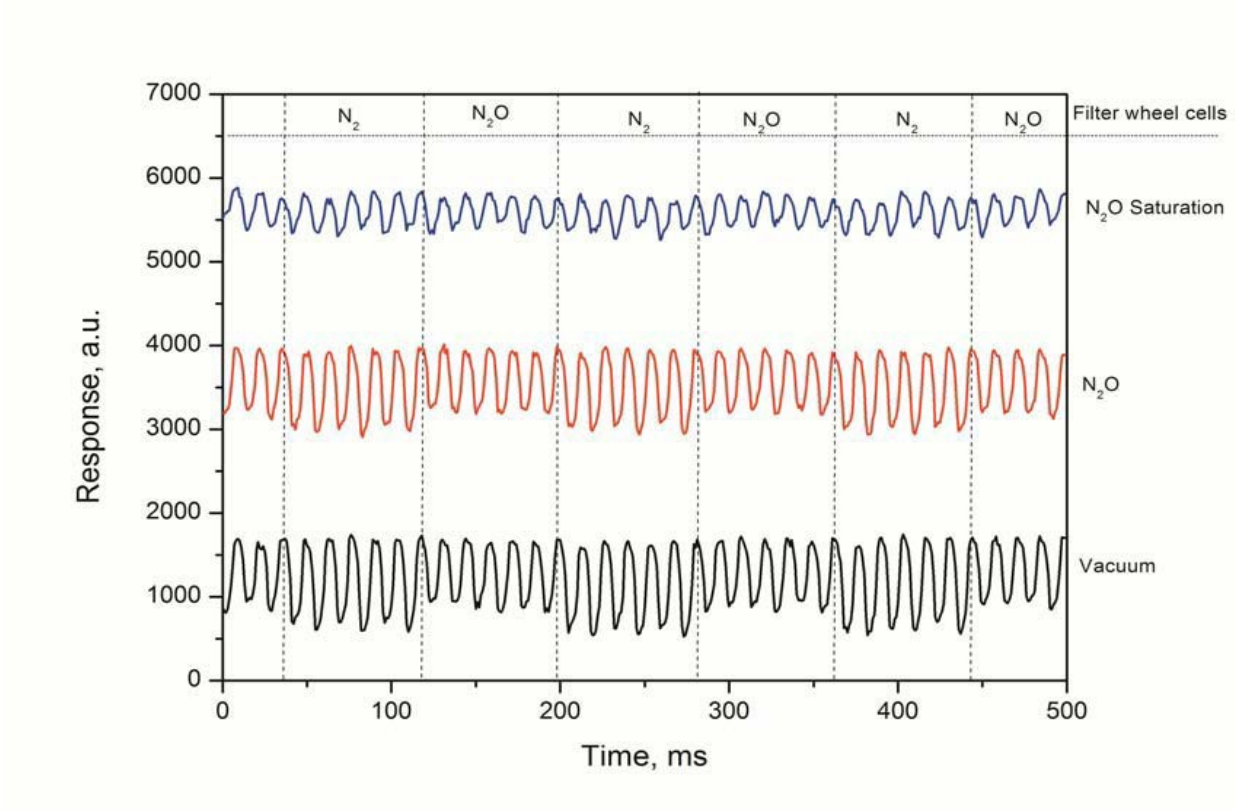


Figure 2. N_2O gas filter correlation response under (a) vacuum, (b) 1.6 Torr and (c) 6.5 Torr of N_2O in the sample cell.

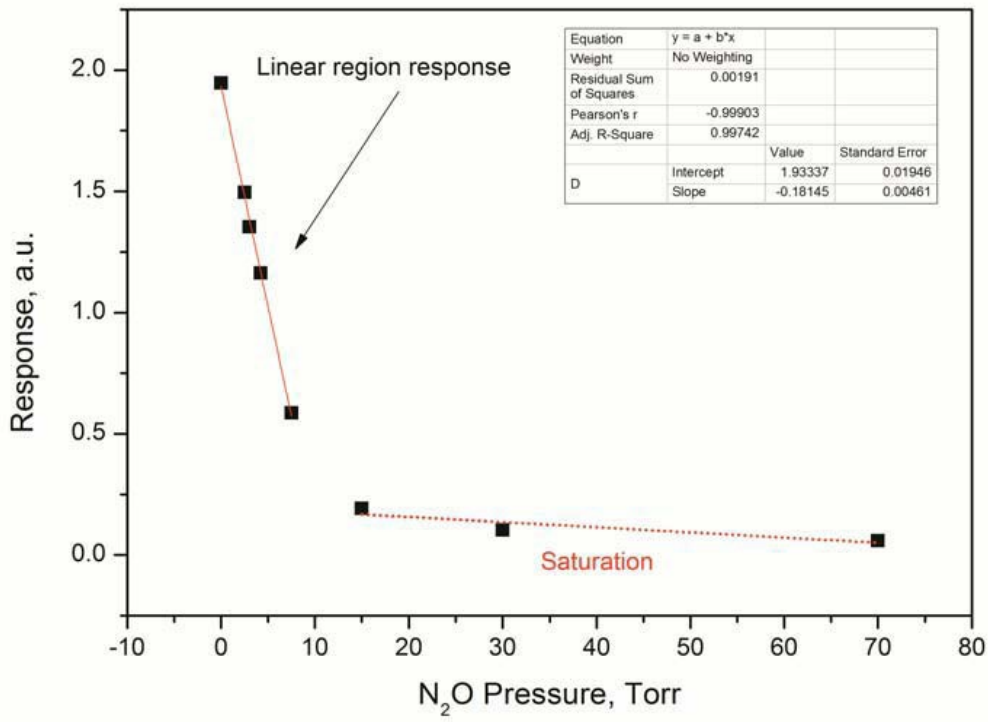


Figure 3. N_2O gas filter correlation response as function of N_2O pressure in the sample cell. Linearity is observed at low pressure regime.

The GFC response as a function of N_2O pressure using LED as infrared source is illustrated in Figure 4. The *negative filtering* signals were obtained at room temperature and using 200 mA in a quasi-continuous wave mode at 512 Hz as LED working conditions. Under such conditions, the systems is able to detect N_2O concentration about 100 ppmv. In order to explore real sensing conditions, the interference of vapor water contained in environmental air was explored.

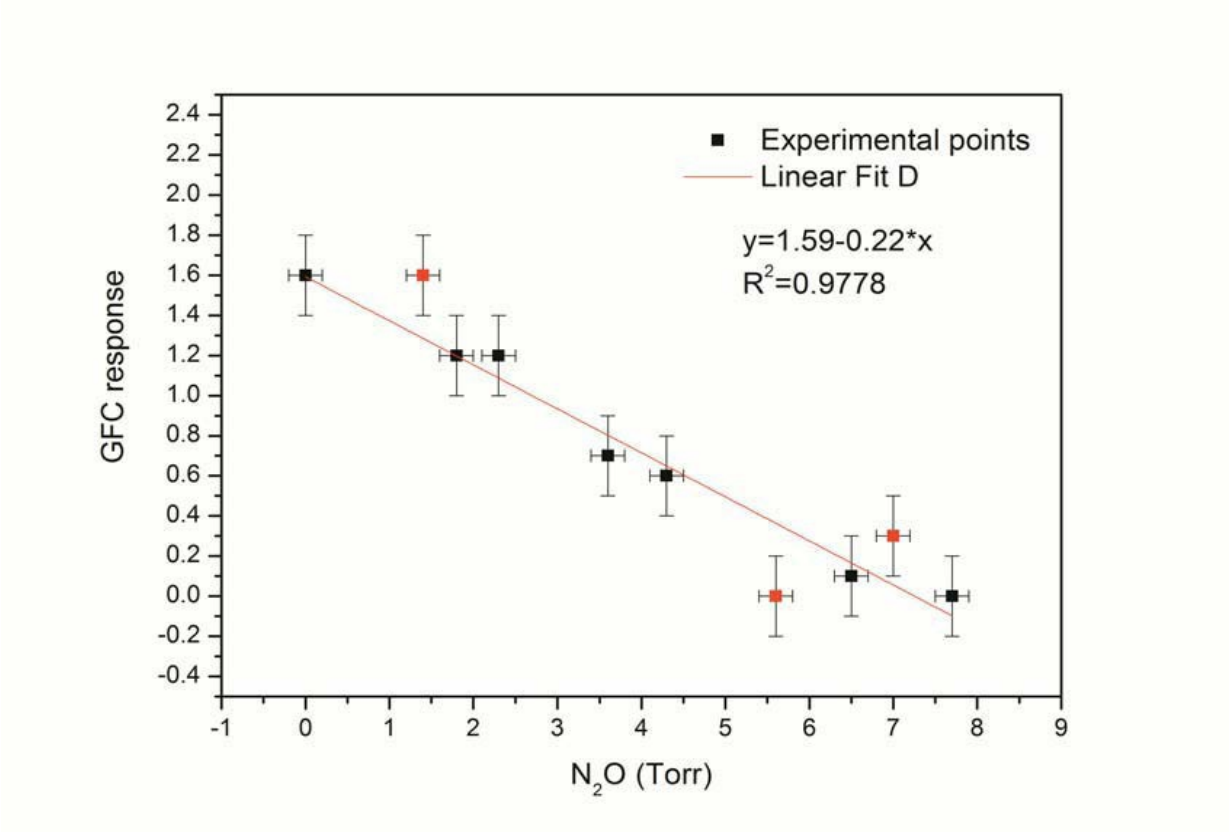


Figure 4. GFC response as function of N_2O pressure in the sample cell using LED as infrared source.

Figure 5 shows the GFC response at different N_2O concentration (up to 3 mTorr) in an air + N_2O mixture with humidity levels up to 60%. Under such conditions, the sensitivity of the detection systems is in the order of about 20 ppmv.

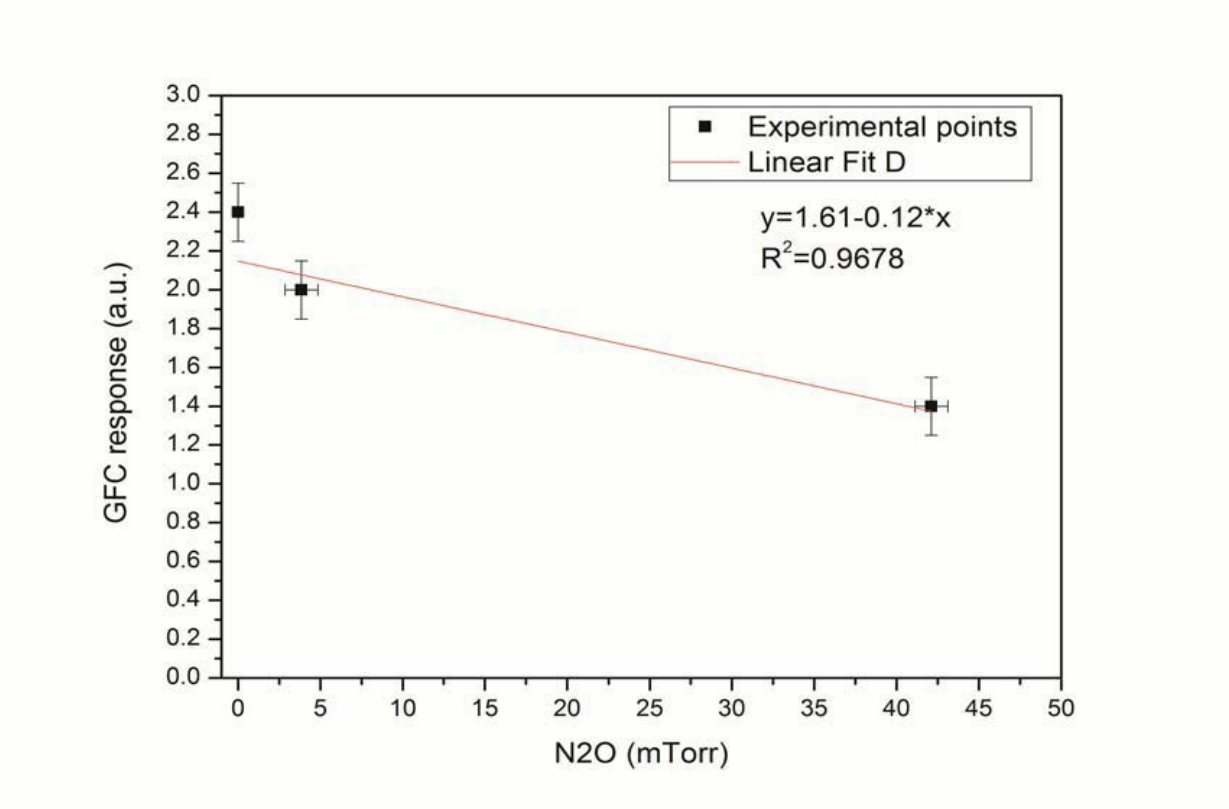


Figure 5. GFC response using atmospheric water in air at low pressure of N_2O.

For a comparison between the system with MID IR LED sources made in the laboratory and the N_2O Thermo Scientific 46i detector system, the nominal measurements of this device has been read according to the pressure of the pure N_2O in the measuring cell and they are shown in the next figure.

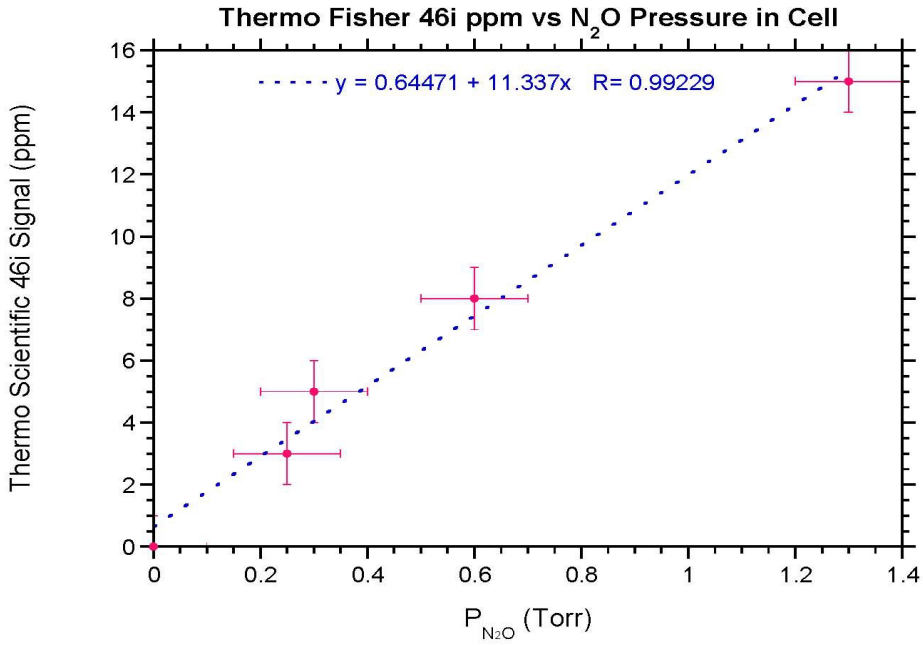
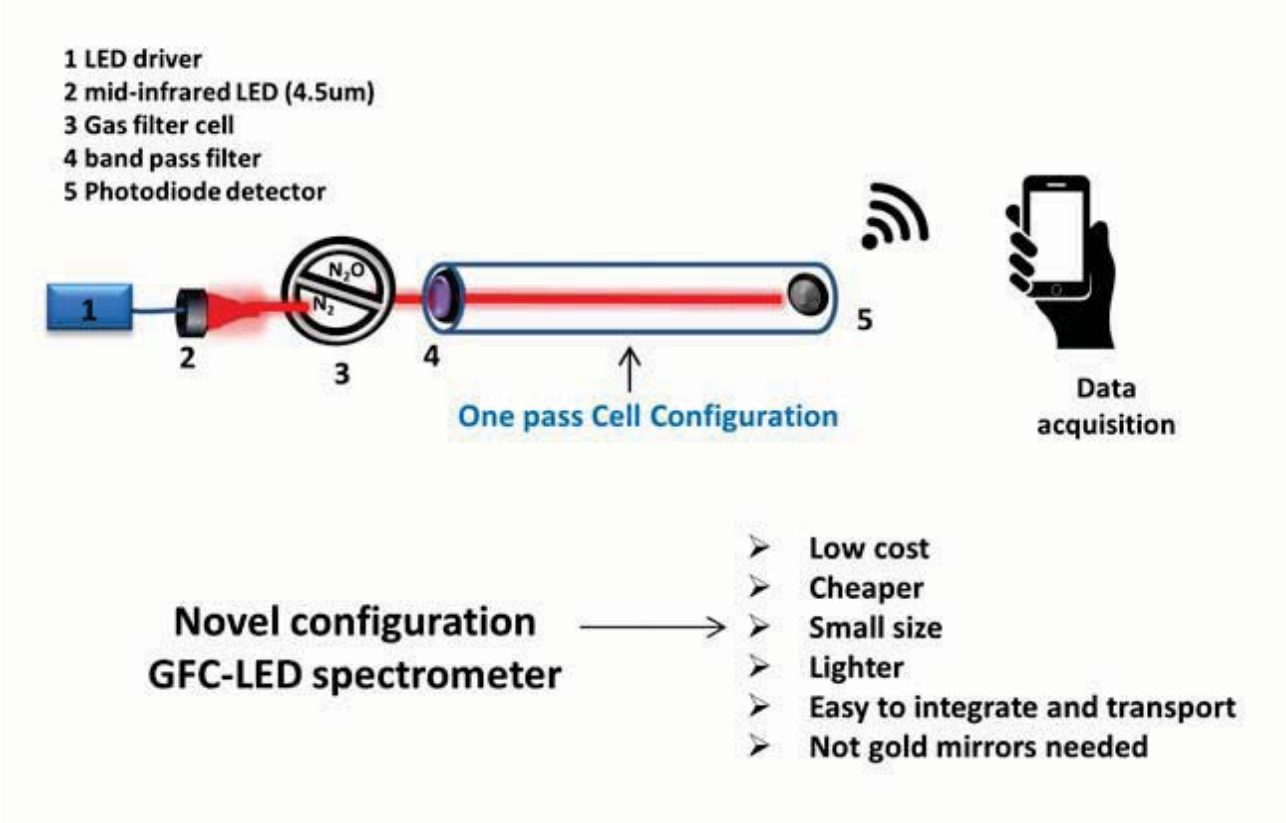


Figure 6. Response of the Thermo Scientific 46i detection system varying the N_2O pressure inside the measurement cell.

In summary, the advantages of using LED as infrared source in the GFC technique, name low cost, easy to modulate and easy to couple to lock in detection systems, small size and low energy consumptions, pose them as novel infrared sources particularly useful for the development of portable sensing devices. In fact the choice of appropriate IR detectors, as well as measuring cells of dimensions 60 - 70 cm length and 1" diameter are compatible with a single operator transportable system, all controlled via software and with reduced energy requirements with a scheme as the one proposed in the last figure.



Reference

[1] T. Dinh, I. Choi, Y. Son, J. Kim, A review on non-dispersive infrared gas sensors: Improvement of sensor detection limit and interference correction, Sensors and Actuators B: Chemical **231**, 529-538 (2016), (doi:10.1016/j.snb.2016.03.040).