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Abstract: The single diffraction grating-based spectrometer has been integrated to a prototype, together with the multipass cell, the supercontinuum source, the camera, and the control electronic. It is basically the integration from the laboratory breadboard-based spectrometer to a compact prototype. Preliminary result on CH ₄ gas monitoring at <50 ppm will be presented, as well as the first measurements outside the laboratory, thus validating the correct operation of the system under real conditions.			

Document History

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Executive Summary

This document reports on Task 4.8 of the FLAIR project, concerned with the integration of spectrometer to a compact prototype and the test on the vital functionalities of the air quality monitoring. The light source of this prototype is the supercontinuum source with a maximum emission at 3.25 μm from NKT Photonics which is particularly well suited for the spectroscopic detection of methane, a potent greenhouse effect gas. The specification of the gas sensing system such as spectral resolution and one-shot spectral range has been evaluated with a single-species CH_4 at various concentrations.

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List of Acronyms

Acronym	Meaning
FLAIR	FLying ultrA-broadband single-shot InfraRed Sensor
IR	Infrared
MIR	Mid-infrared
MFC	Mass flow control
MPC	multipass cell
ppm	Particle per million
SNR	Signal-to-noise ratio
UAV	Unmanned aerial vehicle

Table 1 – List of acronyms.

1 Introduction

FLAIR is aimed at flying trace gas sensor to remotely monitor the air quality in the vicinity of polluted regions. So, the main objective of the project is to develop a compact and lightweight spectrometer, so that the whole sensing system can be suitable to be deployed in a UAV (unmanned aerial vehicle). FLAIR sensor system consists of two main parts: hardware for embedded system and software for data processing algorithm, as depicted in Figure 1.

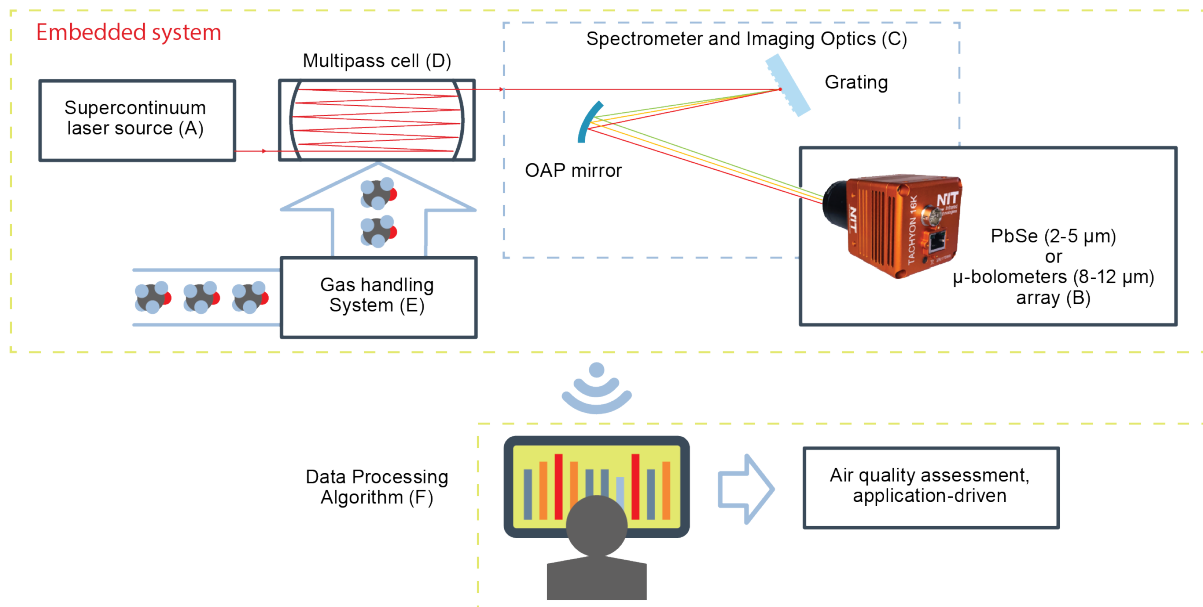


Figure 1 – Block diagram showing the position of NKT's light source in the FLAIR system.

After the successful demonstration of a breadboard-based FLAIR sensor system (referred to deliverables report *D4.6 2D spectrometer and imaging optics*), it has been integrated to a compact prototype. The CAD design and the integrated system of FLAIR prototype during it's integration phase are illustrated in Figure 2(left) and (right), respectively.

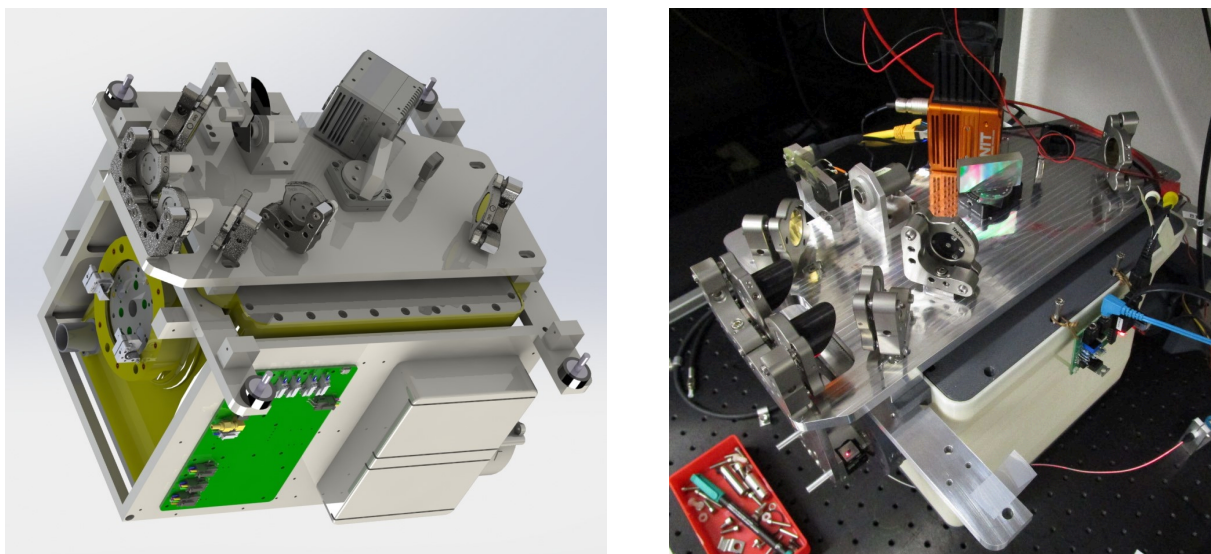


Figure 2: Prototype. (left) CAD design and (right) during integration

2 Test on sensing system performance under laboratory conditions

2.1 Laboratory gas handling system

The laboratory gas handling system is nearly same as the previous one that is used in deliverables report D4.6. 2, except two main differences. First, since a 10 m-long optical pass length gas cell (MPC) is integrated a low concentration CH₄ bottle at 50 ppm was utilized to evaluate the minimum detectable concentration of the sensing system. Second, an integrated ventilation fan was used as air vacuum pump to circulate the ambient air through the MPC. Since the MPC volume is ~1.5 liter, the minimum capacity of the ventilation fan was designed to be ~1.3 liter/sec. Due to the limited capability of laboratory, the maximum mass flow control (MFC) for the test gas was only 0.83 liter/minute (or 0.014 liter/second). However, this gas handling system could allow us to evaluate the preliminary performance of the prototype.

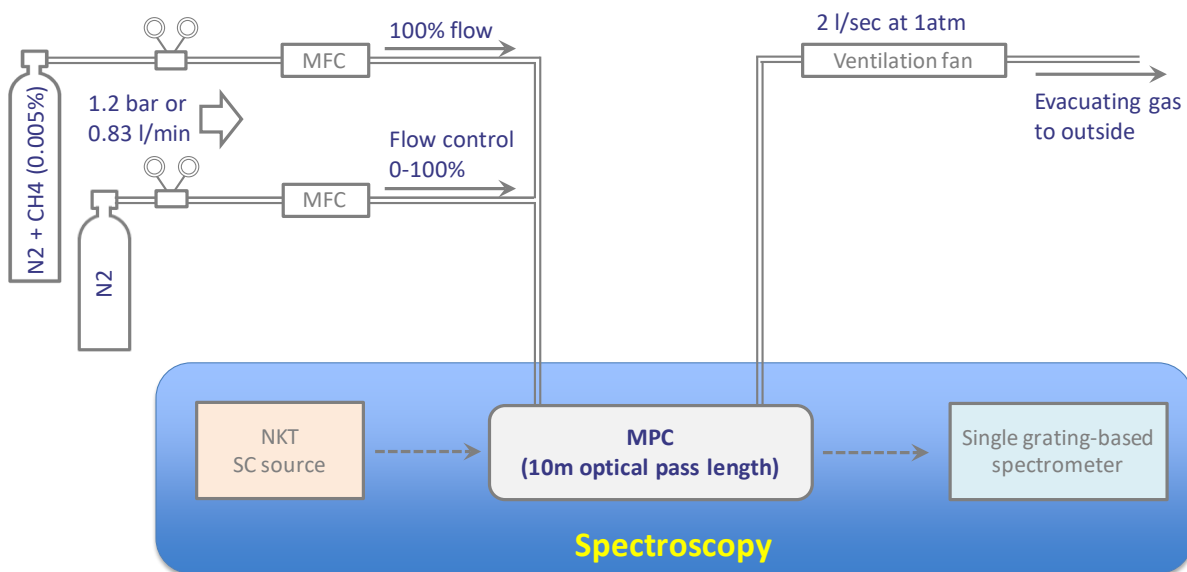


Figure 3: Schematic diagram of controlling the CH₄ concentration inside the gas cell

2.2 Sensing system performance

Prior to the methane (CH₄) filling into the MPC, the MPC was purged by nitrogen (N₂), simply by opening only the N₂ bottle for 3 minutes. This way any presence of the water vapor absorption peaks in the spectrum was expected to be sufficiently removed. In turn, the prototype measurement was continuously performed over 16 seconds while the methane flowing into the cell was started at time = 7 seconds and ended at time = 12 second. The measurement conditions were as follows:

- Camera acquisition, 1000 fps
- Measurement time, 1 second
- Lock-in modulation, 127 Hz
- Methane concentration, 50 ppmv
- Methane flow rate, 0.831 liter/minute
- Air circulation, ~1.3 liter/second

The first 5 measurements in the absence of methane were averaged to be used as reference (See Figure 4(left)), while Figure 4(middle) is an exemplary demodulated image when the gas cell started to be filled in methane. At first glance, the two images look obviously similar due to

the low gas concentration. However, after the signal processing (more details in deliverable report *D4.8 Data processing algorithm*), the gas absorption feature with a high signal-to-noise ratio (SNR) was visible in the normalized transmission spectral profile, as shown in Figure 4(right).

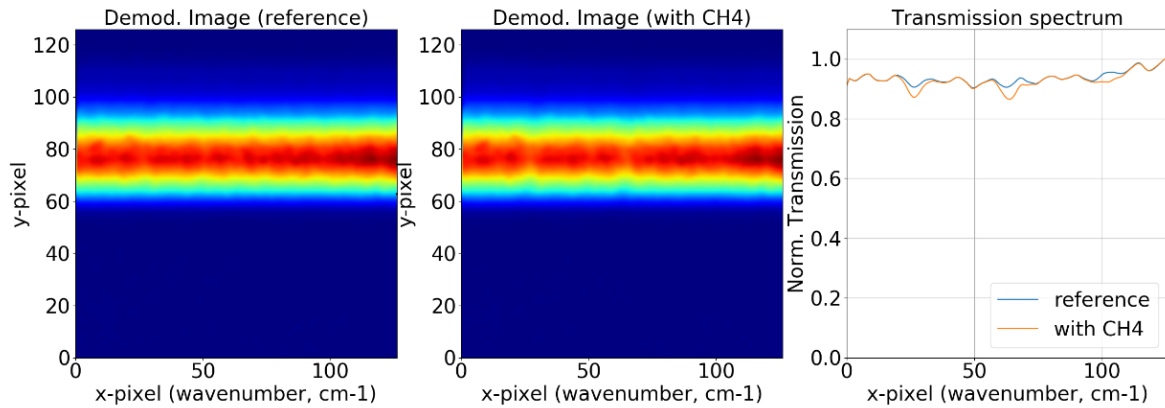


Figure 4: Demodulated 128x128 pixels images with and without methane, and vertically averaged line plots

The measured spectral window corresponds to P(4), P(5) and P(6) absorption lines of methane as shown in Figure 5(left). This measurement window was intentionally selected for the spectrum calibration purpose, since more absorption lines can provide a better accuracy for the calibration in terms of the spectral resolution and range. Moreover, in real measuring condition, this region has very few water absorption lines, which eases the interpretation of the signal through the algorithm. Figure 5(right) illustrates the result of the calibration. The reference spectrum that is theoretically calculated from HITRAN database is precisely fitted to the measured absorbance spectrum. This way, the spectral resolution and the one-shot spectral range of the FLAIR gas sensing system have been unambiguously evaluated to be 0.7 cm^{-1} (equivalent to 0.7 nm or 21 GHz) and 35 cm^{-1} (from 2940 cm^{-1} to 2975 cm^{-1}), respectively. One-shot measurement refers to a measurement for a fixed angle of the diffraction grating. Since the grating is placed on a rotational stage operated by a motor, simply by adjusting the grating angle the spectral window of measurement can be readily shifted towards upper or lower frequency while the spectral resolution remains nearly intact.

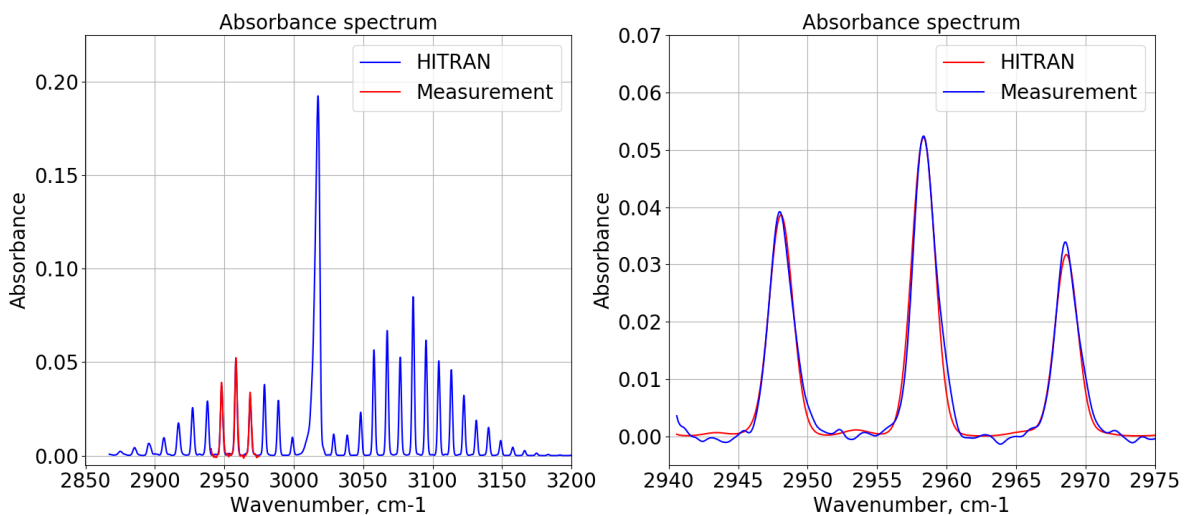


Figure 5: Absorbance spectrum comparison between measurement and reference from HITRAN

Figure 6 shows the general view of the results for the feasibility test of the FLAIR prototype performed under laboratory conditions. Figure 6(left) depicts a spectrochronogram, which

represents the temporal evolution of the measured methane absorbance spectrum at each measurement time (i.e., 1 second for this test). Then, each spectrum was fitted to a reference via non-negative least square regression; hence, computing in real-time the concentration of the methane inside the gas cell, as shown in Figure 6(right). The measurement result has a good agreement with the operation conditions of the gas flow. The monitored methane concentration started to rapidly increase at time = 7 seconds when the gas bottle was opened. Then, the concentration reached a peak at time = 12 seconds before it started to decrease, which was temporally well synchronized to the closure of the gas bottle.

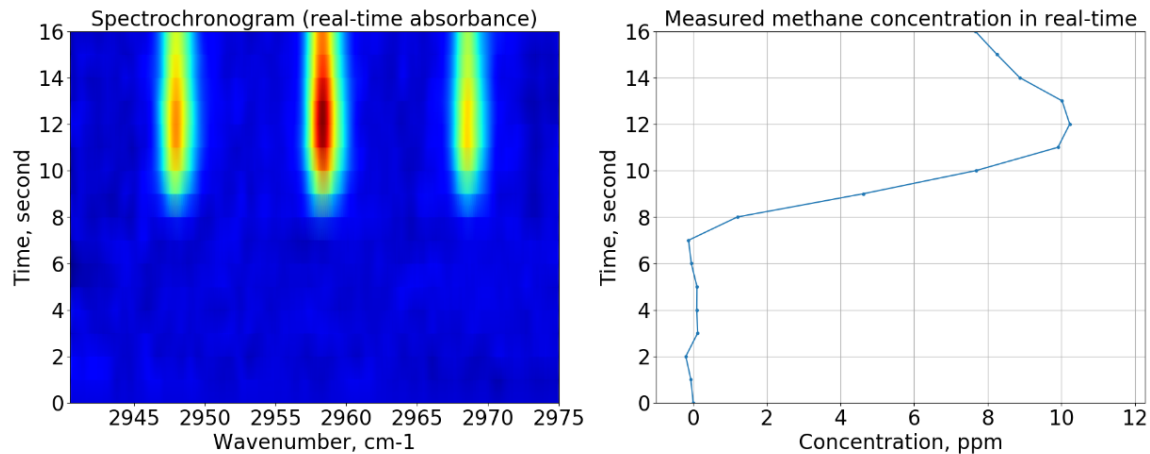


Figure 6: Results on the real-time methane monitoring by FLAIR sensor system

The gas bottle that was used for the test contains a 50 ppm of methane while the rest is filled in nitrogen. However, the maximum concentration retrieved from the measurement was only ~10 ppm. This mismatching can be partially attributed to the gas handling system, since it was not able to evacuate the cell (down to vacuum) and there might be some dead volume inside the cell. Another possible reason would rely on the low mass flow of the gas. In general, the amount of gas filling in the cell is exponentially proportional to the time, so that ~10 minutes of filling time would be required to completely fill in the gas cell. Following this rule, it can be estimated that only 15% of the cell volume was filled at time = 12 seconds, which corresponds to 7.5 ppm, close to the measurement value of ~10 ppm.

2.3 Mismatch between measured and expected concentrations

Subsequent analysis of this issue showed that it was caused by a leak in the metallic enclosure of the multipass cell mirror frame. 2 threaded holes for M5 had been machined but were finally not used to support the spectrometer to the MPC. For experiments where the fan was placed after the MPC, ambient air from the laboratory could enter the MPC directly from those holes. Once the cause had been found (between the calibration tests at EMPA and the Zeppelin flights), the holes had been sealed, and the measured concentration matched the expected values

2.4 Sub-ppm limit of detection for methane in laboratory conditions

As mentioned earlier, the grating is mounted on a precision rotating platform that allows to select any measurement window. It has been tuned to match the strong Q-branch absorption line (the intense line in the middle of the absorption spectrum shown in Figure 5 (left)). In this configuration, concentrations below the ppm level could be detected under laboratory conditions (as visible in Figure 7)), i.e. methane in dry nitrogen, with no water vapor.

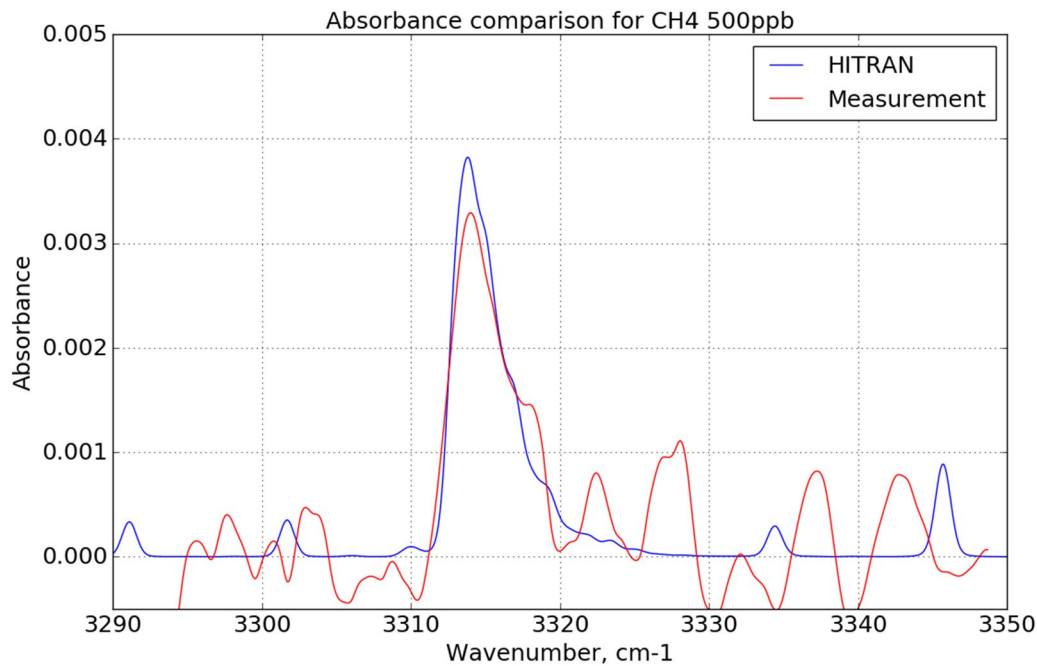


Figure 7: Absorption spectrum of 500 ppb methane in dry nitrogen at the Q-branch location (red) with the corresponding HITRAN simulation (blue)

For all the other measurement situations, the grating has been moved back to the region highlighted in red in Figure 5 (left), where the water absorption is weaker.

3 Preliminary tests in representative conditions

Several tests have been performed in representative conditions. The first has been to make the system measure air sampled from the exterior of the laboratory during a full weekend. This served at testing the robustness and reliability of FLAIR for standalone operations. As lesson learned, we observed that FLAIR detection limit for methane in real conditions is at the same level as the natural occurrence.



Figure 8: The system has left the laboratory and is running on a single battery!

The following week, FLAIR has been taken out of the lab, and operated on a single Li-ion battery. After sampling ambient air, with decided to try the exhaust of a motorbike engine (Figure 9). A broad absorption signal, probably linked to hot residues of hydrocarbons, could be observed on the user interface.



Figure 9: Out-of-lab measurements on the exhaust of the Harley Davidson motorbike of the mechanical designer of FLAIR

The next step was to try FLAIR in a moving vehicle. Figure 10 shows how it has been transferred to the trunk of the car of the lead-engineer. As vibrations from uneven road structures could potentially perturb the measurement stability, we attached 4 Sorbothane feet below the aluminium frame supporting the system.

The wireless link between the system and the operator could also be validated during this test. The right panel of Figure 10 shows the laptop of the operator who is logged to the board computer of the FLAIR system through a 4G network card. There has been no loss of contact during this test.



Figure 10: Out-of-lab measurements tests in a moving vehicle

A GPS tracking system has been integrated to FLAIR. It allowed to record the road taken, as well as the speed of the car, and other parameters, like vibrations and accelerations. The measurements of water and methane could be performed without interruption, and no adverse effect of acceleration or vibration could be observed on this robustly designed, yet lightweight prototype.

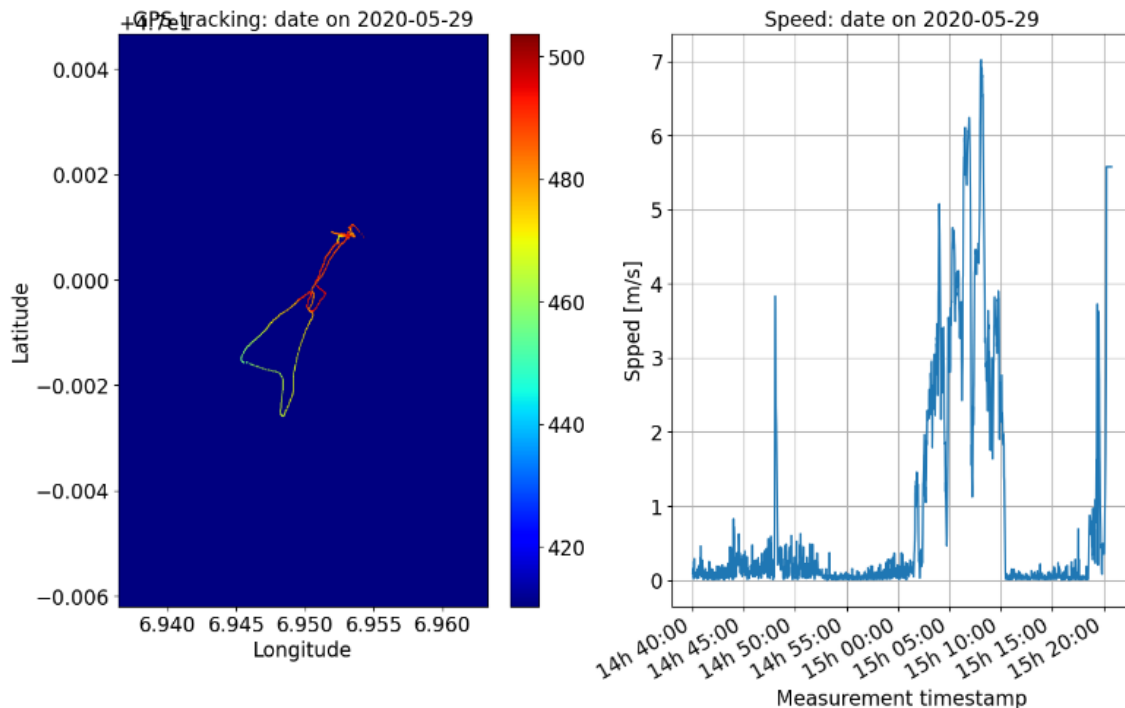


Figure 11 GPS measurements, including the speed of the car, during this first drive test.

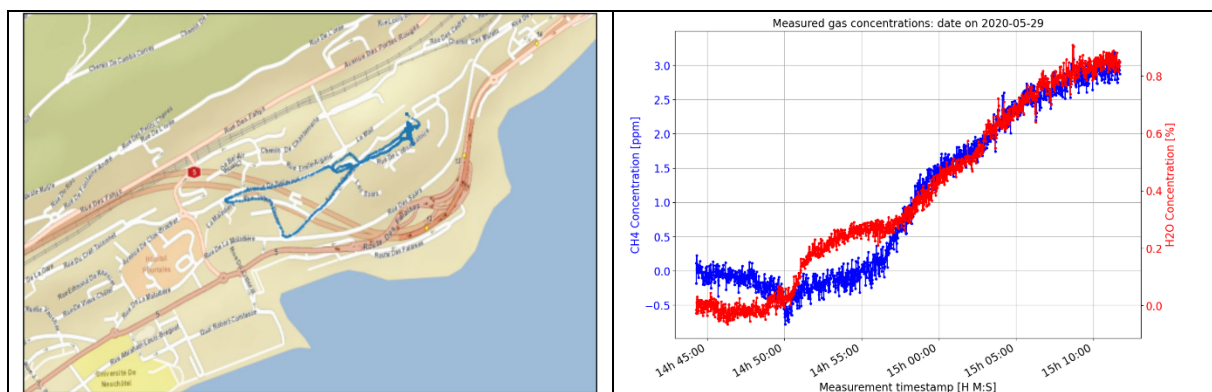


Figure 12 Route taken by FLAIR during its first journey outside the lab (left) and record of the measured concentration of CH₄ and H₂O showing no interruption.

Those preliminary measurements have been made with the purpose to test the transportability and the basic functionality of the system, and check the parameters recorded by the GPS unit. The concentrations measured during the journey (Figure 12 – right) are largely off, probably due to large temperature variation from place in the shade, to the trunk of the car left in the sun.

After those tests, the system has been transferred to EMPA for further calibration and long-term monitoring. The system has been transported by car and has been running seamlessly during all the way on mixed roads and highways. The 4G datalink between FLAIR and the operator's laptop proven robust even at 120km/h speeds.

4 Conclusions

A prototype of the FLAIR sensor system that integrates in a single unit the different parts fabricated by the partners of the consortium has been assembled. After some laboratory validation of its performance (e.g. spectral resolution of 0.7 cm^{-1} over a 35 cm^{-1} window), it has been take outside to check correct operation in an environment setting closer to the validation test that will be performed later during the project. The system passed those tests and has been transferred to EMPA for further calibration experiments prior to the zeppelin and helicopter flights.

