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Deliverable D2.4

Validation of the first version of LOTUS Prototype

Lead: UNI EIFFEL

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Project Deliverable

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D2.4 Validation of the first version of LOTUS prototype -public summary

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Abstract

This deliverable reports on the validation steps carried out for the first version of LOTUS prototypes (described in deliverable D2.1) from August 2020 up to January 2022 within the framework of Task 2.2. This task has been heavily impacted by COVID (6 months delay) and by technical difficulties which caused an additional 6 months of delay. Despite these challenges, important progress were made and results at the state of the art are discussed in the deliverable.

First the deliverable provides an overview of the validation plan to be implemented, then discusses the end-to-end integration (Sensor node; Lotus box); then the lab validation in deionized water (Temperature and conductivity; Chlorine, pH, Magnesium, glyphosate, nitrate and arsenic; Time stability of the CNT inks) and finally the lab validation in drink water (linear monoparameter calibration in active chlorine; Bayesian biparameter calibration providing pH or ClO^- prediction). Facilities used for the work are described in Appendix.

Regarding validation of the end to end integration, the sensor node mechanical package is waterproof up to 8bar or 1m/s in a drink water pipe. Its electronic cards have sufficient repeatability and low enough noise for use in lab settings, but suffer interferences and high noise in realist environment, which has motivated the redesign of the electronic card, currently completed and under test (at M38). The sensor chips wirebonded to PCB have demonstrated 2 months survival in lab tap water, but still suffer from occasional sensor death due to fragile wirebondings (different optimization pathways are available). To mitigate this, the sensor nodes will be deployed in the field through a patented flow adaptor system.

The LOTUS box designed for wireless field deployment of LOTUS node has also been validated on the hardware and software front. Integration of the LOTUS node to the LOTUS box has been demonstrated, including data display on the dashboard supported by a FIWARE context broker. Next stage of validation will be deployment in realist (Sense-City) and field conditions, as soon as the flow adaptor and the updated electronic card are ready.

Regarding lab validation in deionized water, the results of chemical sensitivity screening experiments show clear chemical differentiation brought by the FFUR ink compared to pristine CNT for chlorine (x2), arsenic (x1.7), magnesium (more than x3), glyphosate (x3.5), nitrate (opposite trend). Detection limits estimated for pH (0.2pH unit), arsenic (2µg/L) and chlorine (0.08mg/L) are particularly relevant for drink water use cases. Magnesium detection limit (10mg/L) is relevant for irrigation water.

Temperature and conductivity sensors also respond well in deionized water, and calibration models can be proposed, although error metrics are still high because of the high noise level (best MAE at 0.9°C and 44µS/cm). CNT sensors show clear sensitivity to pH even when conductivity and temperature are varying. However, temperature needs to be accounted for in their calibration model.

Regarding chemical sensing in deionized water, the two main next steps are 1) systematic testing of the sensitivity in the presence of interferents to prepare for use in varying water matrices, and 2) preparation of tri-parameter sensing by the addition of a third functional ink on chip, with focus on arsenic. First devices with three inks are already available for characterization.

Regarding drink water validation, calibration of three sensor chips in drink water according to the same protocol has allowed to determine the best variable to use for sensor calibration. A correction factor has also been developed which allows to compensate for chip ageing. Two algorithms for calibration are proposed, one based on standard inversion of a linear monoparameter calibration model, the other on Bayesian multiparameter calibration including measurement uncertainties.

Active chlorine can be predicted in tap water with pH freely ranging from 6 to 8 with high reproducibility between devices and chips, but no difference between pristine and FFUR CNTs. Best achieved MAE (with Bayesian monoparameter calibration) is 0.047mg/L of HClO for HClO below 1mg/L (chip 68). The detection limit is estimated in average at 0.012mg/L. Sensitivity is 5 to 10 times stronger than observed in deionized water.

Bayesian calibration allows to predict both ClO^- and pH. For ClO^-, best MAE 0.08mg/L with chip 68 (MAE on HClO 0.11mg/L). For pH, best MAE 0.36 pH unit with chip 73 (MAE on HClO 0.11mg/L). All three chips can be used for ClO^- prediction, only chip 68 and 73 for pH prediction. Sensitivity to pH is comparable to what is observed in deionized water (around 1%). Sensitivity to ClO^- is stronger than sensitivity to pH.

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For a research point of view, these results are state of the art in terms of Bayesian calibration algorithms and CNT sensor performances for drink water. However, they rely on a reduced dataset with high measurement uncertainties on reference quantities. For further applications within LOTUS, the different algorithms proposed here need to be validated by long time characterizations in flowing water in lab and field, monitored continuously with high accuracy chlorine and pH sensors. The testbenches will be available for that early in 3rd period at IITG and Uni Eiffel (Sense-City).

Keywords

Sensor; pH; chlorine; temperature; conductivity; calibration; characterization; integration; specifications

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The LOTUS Project

LOTUS is a project funded by DG Environment under the European Union Horizon 2020 Research and Innovation Programme and by the Indian Government. It brings together EU and Indian prominent organisations with the aim to co-create, co-design and co-develop innovative robust affordable low-cost sensing solutions for enhancing India's water and sanitation challenges in both rural and urban area.

The LOTUS solution is based on an innovative sensor and includes tailor-made decision support to exploit the capabilities of the sensor as well as a specific approach to co-creation. LOTUS aims to be co-designed and co-produced in India, and have a wide, diverse and lasting impact for the water sector in India due to intense collaborations with commercial and academic partners in India.

Based on the low-cost sensor platform, solutions for the early detection of water quality problems, decision support for countermeasures and optimal management of drinking and irrigation water systems, tailored on the functionalities of the new sensor, will be developed and integrated with the existing monitoring and control systems.

This sensor will be deployed in five different use cases: in a water-network, on ground-water, in irrigation, in an algae-based waste water treatment plant and in water tankers. The packaging of the sensor, as well as the online and offline software tools will be tailored for each of the use cases. These last will enable to test the sensors and improve them iteratively.

The project is based on co-creation, co-design and co-production between the different partners. Therefore, an important stakeholder engagement process will be implemented during the project lifetime and involve relevant stakeholders, including local authorities, water users and social communities, and will consider possible gender differences in the use and need of water. Broad outreach activities will take place both in India and in Europe, therefore contributing to LOTUS impact maximisation.

The further development and exploitation (beyond the project) of the novel sensor platform will be done in cooperation with the Indian partners. This will create a level playing field for European and Indian industries and SMEs working in the water quality area.

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Acronyms and Definitions

Acronyms	Defined as
CNT	Carbon nanotubes
CNT/FF-UR	CNT functionalized with polymer named FF-UR
AFE	Analog Front End (see D2.1)
LOD	Limit of detection
TRL	Technology Readiness Level

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1 Executive Summary

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2 Description of LOTUS 1st prototype

The composition of LOTUS 1st version prototypes is described in deliverable D2.1 and summarized in Figure 1 to Figure 4. All the building blocks and naming convention are reminded position of LOTUS 1st version prototypes is described in deliverable D2.1 and summarized in Figure 1. The core of the technology is the sensor chip, which contains 2 temperature sensors, 3 conductivity sensors, and 20 chemistors split into 2 sets of 10 device each, each set carrying a different type of carbon nanotubes-based coating (Figure 2, left). The sensor chip is wirebonded on a sensor PCB, and the wirebonding are waterproofed and mechanically protected by a resist coating (Figure 2, right). The sensor PCB is packaged into a sensor head, then connected to its analog front end (AFE). The AFE is placed into the sensor housing, and the sensor head is screwed to one end of the sensor housing (Figure 3). The AFE is either connected to a PC, for lab testing (Figure 3), or to a LOTUS box (Figure 4), for field operation.

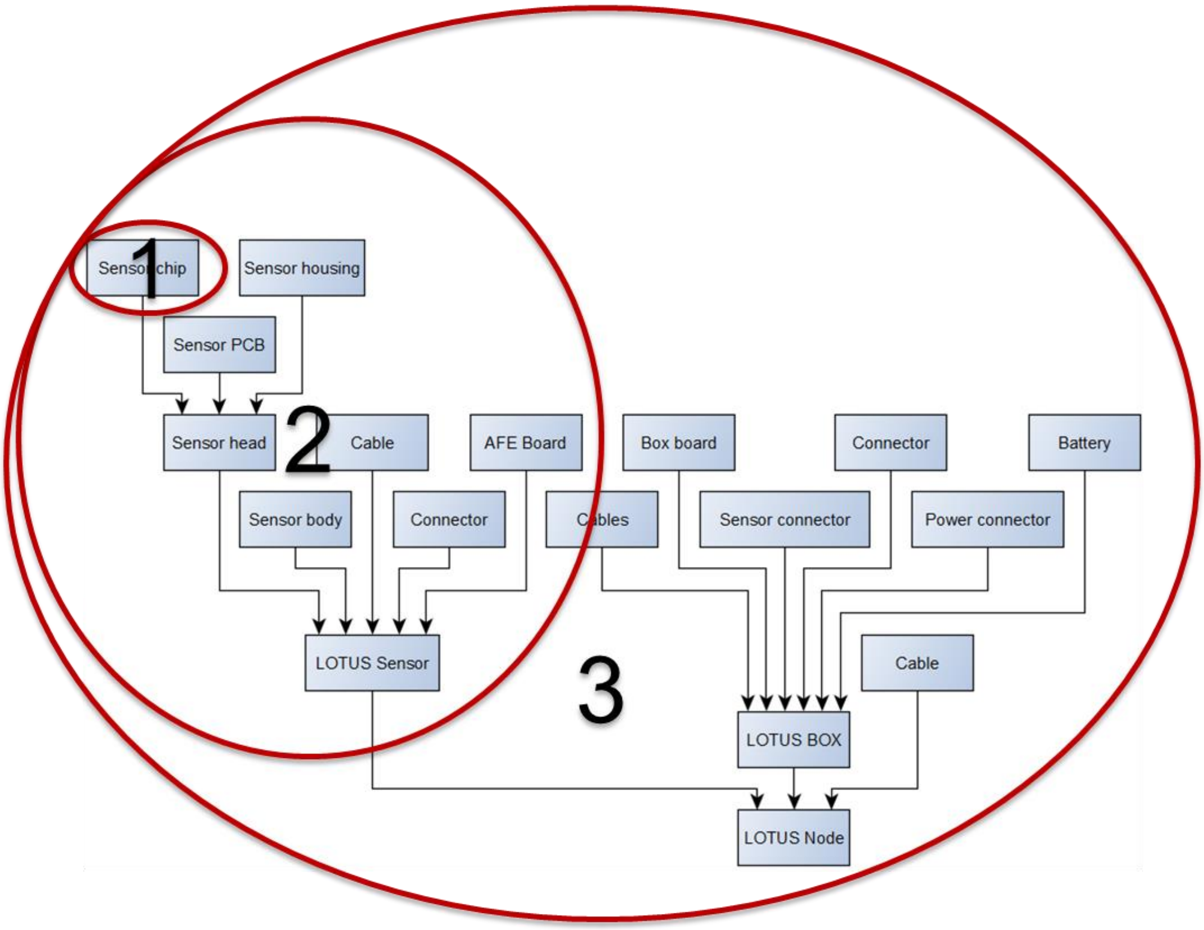

Figure 1: Architecture of LOTUS hardware solution. Step 1: Sensor chip – core sensing component. Step 2: LOTUS sensor – wired sensing solution. Step 3: LOTUS Node – wireless, autonomous sensing solution.

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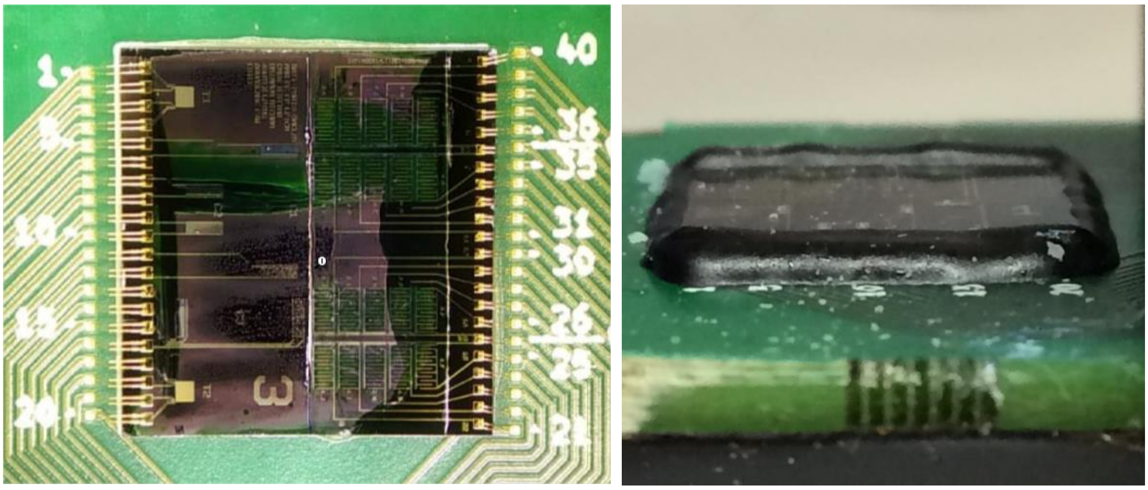

Figure 2: Left - Wirebonded sensor chip before globtop. Right: sideview of the sensor chip with globtop layer.

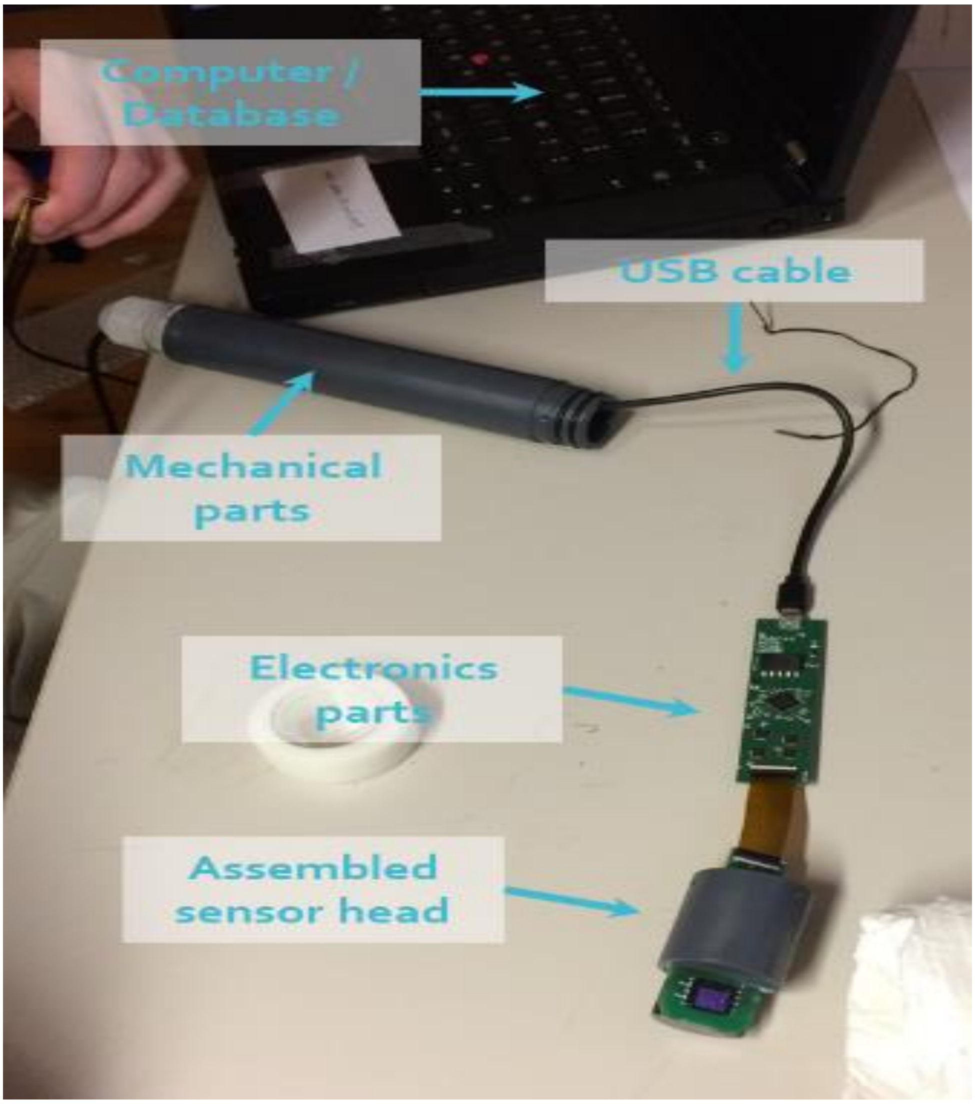

Figure 3: Assembled sensor.

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Figure 4: The LOTUS box with its electronic cards and cell batteries

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3 Validation plan for LOTUS 1st prototypes

The ultimate goal of LOTUS validation procedures is to build the future commercial datasheet for LOTUS sensors, with information such as

- Operating conditions : range of acceptable pressure, flowrate, temperature…
- Operating modes: wireless, powerless…
- Operating range for the target parameters
- Sensing performances: sensitivity, limits of detection, accuracies, response times, acquisition frequency
- Reliability: time between calibration, service time

Toward this, the validation procedures carried out in Task 2.2 and reported in deliverable D2.9 aim at

- showing the level of operationality and performances of the first version of LOTUS prototypes compared to the requirements summarized in deliverable D2.1.
- understanding the sensing and ageing features depending on fabrication and data acquisition parameters, in view of optimizing performances.
- defining, then optimizing, the calibration algorithms and the calibration protocols.
- fine-tuning the specifications and design of LOTUS second version prototypes.

As per LOTUS work program defined in LOTUS first amendment, the 1st LOTUS validation report includes the following content:

- T2.2a Lab validation of the sensing capability of the chemical sensor array: Validation of sensing capability on dedicated sensor calibration bench, in view of optimizing sensor fabrication, sensor firmware, establishing cross sensitivity and ageing models.
- T2.2b Validation in model environment and real environment: in realist setup (in Sense-City or equivalent EU facility), EGM demonstrates full system operation and EP validates calibration and reliability data. EGM, DFM and NEERI achieve field tests in relevant use case conditions in EU first and then in India.
- T2.2.c Systematic calibration of all prototypes before use in real environment, including development of calibration protocol (proposed by EGM and EP, adapted to Indian facility by IITG and DFM), development of automated calibration test bed in India (IITG and DFM with EP support, implementation of protocols (DFM with IITG support for analytical chemistry), exploitation of calibration data to provide calibration data sheets (DFM with EP support).

As reported in the 2nd period report, developments in WP2 task 2.2 have been slowed down by several factors:

- Activities in WP2 require physical presence in the labs and direct interaction, which was slowed down by the COVID crisis. Over about 1 year of “heavy” pandemic, activities at EP underwent

about 3 months of direct delay due to lab closure and 3months of indirect delay due to reduced physical presence.
- Two major technical difficulties were encountered – instability of the globtop and of the analog front end – which caused an additional 6 month delay. These difficulties are described in the deliverables.

As a consequence, the three subtasks outlined above did not allow to reach the expected validation level for LOTUS 1st prototype by the end of Task 2.2. The limitations of the current solution will be addressed and mitigated in Task 2.3 and deliverable 2.5. In advance of task 2.3a, the deliverable also includes a list of issues to be solved in LOTUS 2nd version prototype as well as the proposed solutions.

To cover both the success and the limitation of the 1st version of LOTUS prototype, as well as to explain the root causes of these limitations, the deliverable is organized as follows:

1. End-to-end integration (Task 2.2b – toward TRL5 – enabling field deployment)
    a. Sensor node
    b. LOTUS box
2. Lab validation in deionized water (Task 2.2a –TRL3 – understanding in view of optimizing)
    a. Temperature and conductivity
    b. Chlorine
    c. pH
    d. Magnesium, glyphosate, nitrate, arsenic
    e. Time stability
3. Lab validation in drink water (Task 2.2c – TRL4)
    a. Linear monoparameter calibration: performance in active chlorine alone
    b. Bayesian calibration: Performance combined in chlorine, pH and ClO^-

Facilities used for the work reported in this deliverable are described in Appendix 1.

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4 Validation of end-to-end integration

4.1 Outline

This section presents the validation results for the end-to-end integration of the sensor node: validation of the sensor node (mechanical packaging, electronics, stability in water) and on the other hand of the LOTUS box (validation of hardware and software, connectivity to the sensor node).

4.2Sensor node

4.2.1 Stability of the mechanical packaging

Figure 5, left, shows an assembled sensor version 1.0 (details are provided in Deliverable D2.1). The mechanical robustness and waterproofness of the system was tested under realistic pressures and flowrates in Sense-City in July 2020. Figure 6 shows the installation of the sensor into the water loop, and Figure 5, right, shows the sensor chip on PCB after testing under water flow.

Figure 5: Left - assembled sensor; Right - Zoom on the sensor chip after exposure to water flow.

Figure 6: Insertion of the sensor into Sense-city drink water loop

The compatibility of the sensor node design with the water loop was first demonstrated: the sensor can be properly inserted into the water loop, the insertion point remains dry. Following this test, minor design optimizations were proposed to facilitate insertion (now implemented in all the prototypes).

- To facilitate the insertion of the sensor head into the LNODE support, a chamfer is added around the circumference of the sensor head (Figure 7(a));
- To ensure the proper orientation of the sensor with respect to flow, and to facilitate manipulation of the sensor, the initially perfectly cyclinder-shaped sensor body now features two flats (Figure 7(b)).

(a) (b)

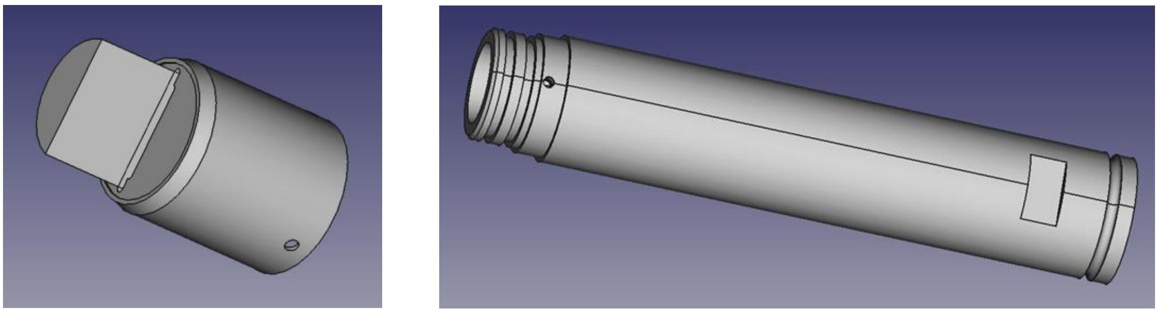

Figure 7: Modification of: (a) the sensor head and (b) the sensor body.

Then, the mechanical resilience of the sensor under pressure and flow was proved:

- Three positions of the sensor were tested in the loop under various water flow and water pressure: back to the flow, parallel to the flow and facing the flow of water. Each set of water flow, pressure, sensor orientation was tested for about 20 min. After 20 min, one verifies that the internal part of the sensor head is still dry.

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- At flow rate of 0.1 m/s, the pressure was increased up to 8 bar. For the positions facing the flow and parallel to the flow, no water leak was observed up to 8 bar and up to 0.1 m/s.
- For the position back to the flow, the test was only carried out up to 5 bars. No water leak was observed. Figure 5, right, shows that after pressure and flow test, the chip and the globtop appear undamaged.
- Above 5 bars, the node was ejected from the loop. The ejection of the node – rather than leakage of water through the sensor body - suggests the failure was due to the insertion setup, not the sensor node itself.

These results show waterproofness of the sensor node and mechanical resilience of the chip and globtop up to 5 bars notwithstanding the position of the sensor head with respect to flow. Resilience up to 8 bar is confirmed for 2 out of 3 positions of the sensor head.

4.2.2 Electronic read out by the analog front end (AFE)

Content is confidential

4.2.3 Stability of the baseline resistance

Content is confidential

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4.2.4 Toward increased sensor node reliability : Flow adaptor

A major issue that affects the sensor performance and usage life is the effect of fouling. This can be attributed to the presence of minute sediments and micro-organisms in the water. Also, as discussed in the previous section, electrical contacts (wirebondings) feature instability, which is expected to be amplified at high flow. These problems can be solved by the use of the developed membrane-based flow adapter as sensor protection system. Moreover, the systematic use of the adaptor in the use cases will reduce the impact on calibration of the different installation conditions.

This system developed by IITG as part of the testbench, is housed in a bypass structure and connected to recirculation line. The outline of the complete structure is shown in Figure 8.

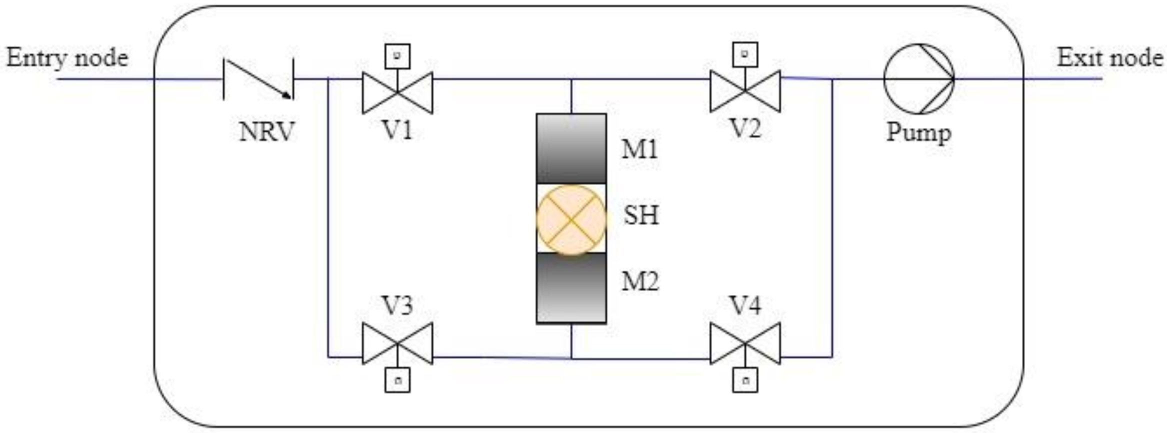

Figure 8: Bypass structure with flow adaptor system

The above system functions as follows: the entry node and exit node are connected to the recirculation line (in the IITG testbench, same can be adapted to connect to distribution lines in the field). The water enters the system through the entry node and the no-return valve (NRV) restricts the back flow of water to main line via entry node. The valves V1, V2, V3 and V4 are controlled by a microcontroller and are turned on and off in periodic fashion. For a set period valves V1 and V4 will be turned on while V2 and V3 will be off. After the period, the states of the valves are reversed. This periodic state reversal changes the water flow direction through the sensing chamber marked by M1-SH-M2, wherein M1,M2 refers to filter caps fitted with semi permeable membranes and SH houses the LOTUS sensor. During the period when the valves V1 and V4 are turned on, the water flow follows the path: Entry -> NRV -> V1 -> M1 -> SH -> M2 -> V4 -> Pump -> Exit; while when the valves V2 and V3 are turned on, the water flow follows the path: Entry -> NRV -> V3 -> M2 -> SH -> M1 -> V2 -> Pump -> Exit. The pump in the structure is employed to pressurize the water enabling it to exit and join the main line. In this way the membranes in the filter caps are alternatively in active filtering and backwash conditions, thereby reducing the need for regular maintenance/cleaning of membranes.

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4.3 LOTUS Box

4.3.1 Objectives

LOTUS sensors can be operated through computer, as shown in the previous section, but this does not transfer to field situations. The LOTUS box (Figure 4) provides the required field-compatible communication and power supply to LOTUS sensor so that it can be deployed in real environment. LOTUS node validation includes proving data collection from the LOTUS sensor into the LOTUS box (with the same data quality), then operation of the system in quasi-real, then real conditions (T2.2b).

In this section we present:

- How the LOTUS box hardware was tested, fixed, and validated.
- How its software was designed and validated.
- How the LOTUS sensor was connected to the LOTUS box to get reliable data.
- How the AFE was integrated into the whole acquisition and processing chain.

4.3.2 Validation and correction of LOTUS box hardware

4.3.2.1 Lotus Box Hardware

There are 17 functional requirements for the LOTUS Box listed in D6.1. The design of the electronics, the choice of the components, and the supported technologies were done to fulfill them as much as possible. This led to the choice of the hardware components listed below.

STM32L462VeT microcontroller (STMicroelectronics)

This is a very low power integrated circuit on a processor named Arm Cortex, the latter has a 32-bit RISC architecture, a flash memory of 512 Kbytes, and an SRAM memory of 160 Kbytes. The functionalities of this MCU are detailed in Figure 9.

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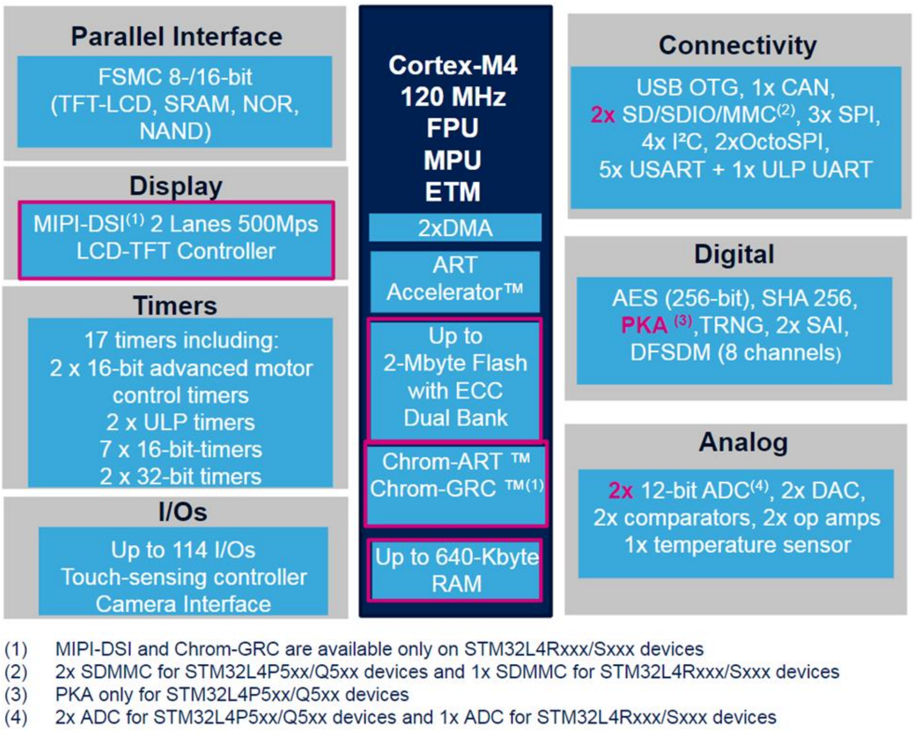

Figure 9: STM32L462VeT microcontroller

This chip contains:

- An analog-to-digital converter (ADC) and a digital-to-analog converter (DAC) with 12-bit resolution and high speed.
- A low-power real-time clock (RTC).
- 16–32-bit timers that can be used as timers, command generation/capture and PWM (Pulse Width Modulation) signals...
- Standard and advanced communication interfaces, namely four I2C, three SPI, three USART, one UART and one low power UART, one SAI, one SDMMC, one CAN, one full speed USB device, these interfaces constitute the drivers of the board and provide the basic functionality of our system to be generic enough to integrate any sensor, use any technology to send the processed data to the periphery and control any actuator.

Accelerometer

An accelerometer is a sensor that measures the vibration or acceleration of an object's movement. It is used largely for detection of motion and shocks that the card may undergo. The selected accelerometer for the Lotus Box is the LIS2DW12. It is a low-power accelerometer with 16-bit resolution and communication using the I2C and SPI interfaces.

Temperature-humidity sensor

This sensor measures water vapor and heat/coolness and convert them into a readable unit. The selected sensor HTU21D has typical accuracy of 2% (humidity), 1% (temperature) with an optimized

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operating range of range of 5% to 95% RH for humidity and -30 to 90°C for temperature. This sensor operates at 3 to 5 (volts) and communicates using an I2C link.

FRAM

A FRAM (ferroelectric Random-Access Memory) is a non-volatile random access memory non-volatile memory with low energy consumption. This memory combines the advantages of conventional non-volatile memories (such as Flash and EEPROM) and fast static RAM (SRAM and DRAM), so it offers a very fast writing speed writing speed while saving energy.

SD card reader

An SD (Secure Digital) card is a removable memory. This mini component is present on many smartphones and digital cameras, it has a high data transfer rate with a low data transfer rate with low battery consumption, it uses flash memory to provide non-volatile storage, making it unnecessary to use a power source. The Lotus Box card offers its user the ability to store data through its SDMMC interface, which allows for writing up to 48 MHz.

Transceiver RS232/RS485

As its name indicates, this component allows to transmit data easily on a RS232/485 by converting the received signals into a simple UART (Universal Asynchronous Receiver Transmitter) type data frame.

MikroBUS slots

MikroBUS™ (Figure 10) is a standard adopted by many companies; it was created by MikroElektronika, a manufacturer specializing in the development of hardware and software tools for embedded systems.

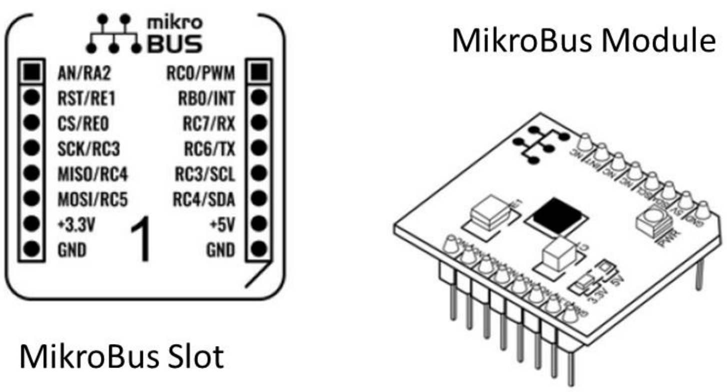

Figure 10: MikroBUS™ module

A mikroBUS™ standard module includes a pair of 1 × 8 female connectors. This module has a pinout always arranged in the same order, it contains the basic pins of the 3 communication buses (SPI, UART and I2C), six additional pins (PWM, interrupt, analog input, reset) and two power supply groups (+3.3 V and 5V).

The Lotus Box has three mikroBUS™ slots allowing to connect small expansion boards. To date, there are more than 1000 different types of such modules on the market allowing to add options to the box in terms of connectivity, of sensors or of actuators.

These microbus slots have been integrated into the Lotus Box to provide the most generic IoT board. These slots make the development process and the creation of proof of concept for additional function easier and faster.

4.3.2.2 Lotus Box hardware validation

The 17 hardware requirements defined in D6.1 were used to validate the design and to elaborate a test plan for the boards. The Table 1 shows, for each requirement, the reason for its design validation.

Table 1 : Design validation of the Lotus Box hardware

#	Expression	Criterion	Validation
FP0	Computation	Must have an internal computation	The board integrate a STM32L4 MCU.
FP1	Modularity	The overall device functionalities must be changed or extended according to customer need	The board is based on off-the-shelf modules and add-ons that can also be customized to meet even the most specific needs of a particular IoT application, while removing time-to-market and financial risk related to the development of an IoT, a solution from scratch. We chose MikroElectronika's MikroBUS™ standard because it offers maximum expandability with the lowest pin count. Moreover, it is an open standard adopted by many MCU manufacturers. The LOTUS box has three MikroBUS™ slots. To date, there are more than 1237 different modules, so an almost infinite possible combination.
FP2	Sensor input	One or more sensors must be attached to the device. Could be done by FP1	The LOTUS box integrates an industrial sensor interface (Dual full duplex RS232, RS485/422 and UART), but also integrates other communication buses such as SPI, I2C and BLE5 thanks to a MikroBUS click.
FP3	Actuator output	Must operate a device like a valve	We can count on versatility and modularity with the 3 MikroBUS™ slots where we can connect, for example, a relay module.

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FP4	Communication output	Device must transmit data from the sensors to the cloud. Could be done by FP1	This hardware is an industrial module with Dual full duplex RS232, RS485/422 and UART on the board. It is possible to add other types of wireless or wired communication thanks to some specialized MikroBUS modules (e.g. Lora, Ethernet, BLE5, Iridium, NB-IoT, LTE-M, 3/4G, Ethernet…).
FP5	Data logging	Must record all data from connected sensors	The board has a data backup (On-board RAM + µSD slot).
FP6	Real time clock	Must add a timestamp to recorded data	The device has a permanent real time clock (RTC).
FP7	Internal t°/hum	Must provide internal health status for internal diagnostic	Internal t°/hum sensor on the board for diagnostics.
FP8	Autonomy	May have several hours/days of autonomy	In the event of low sunlight or a power outage, the hardware can operate autonomously thanks to a LiFePO4 battery pack which coupled with the low power mode can bring an autonomy up to several months.
FP9	Internal charging unit	May charge an internal battery, in conjunction with FP8	The LOTUS box has on-board battery charging and management.
FP10	External power	Must be externally powered	The device can be powered with external AC/DC power supply 12V (or any other DC power adapter), industrial power supply 24V, Car battery 12V or USB.
FP11	Energy harvesting	May source energy from environment	External solar panel can be plugged to the board which contains a battery charge controller.
FP12	Core power	Must provide power supply to all internal component	The card has a central power which provides electrical power to all internal components regardless of its power source (battery, 12V, 24V, USB...).
FP13	Power supply output	Must supply power to the sensor (see FP2)	The card can power devices with 5V and 12V, but also any other power supply voltage with a MikroBUS module (24V for example...).
FP14	Power control	Must control each module (see FP1) power supply to shut them down	The LOTUS box can switch off 5V and 12V power supply and can also switch off the power supply for each MikroBUS slot individually.

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FP15	Security	Must encrypt data or similar	The microcontroller has an Advanced Encryption Standard hardware accelerator. The AES interface is widely used for cryptographic applications.
FP16	Updatable	Must be externally updatable (OTA updates)	User can update the software from USB. An OTA-ready bootloader is also available.
FP17	HMI / configuration	Human machine interface. Must be externally or internally configurable	The user can configure the card through buttons and a screen or a touch screen but also through a shell interface.

Moreover, the test plan, which was executed on each produced board, is show on Table 2

Table 2: Lotus Box boards test plan

Function	Expression	Tests executed
FP0	Computation	• Alimentation test • Upload an example of code
FP1	Modularity	• Alimentation test • Software test for communication protocol (SPI, I2C, UART...)
FP2	Sensor input	• Alimentation test • Software test for communication protocol (SPI, I2C, UART...) • Software test with some sensors
FP3	Actuator output	• Software test for MikroBUS Relay click
FP4	Communication output	• Software test for wireless and wired communication with three different MikroBUS click connected simultaneously (LoRa, Wi-Fi, Ethernet)
FP5	Data logging	• Read and write software test for FRAM and µSD
FP6	Real time clock	• RTC software test with reboot after board power off
FP7	Internal t°/hum	• Software test of the internal Temp/Hum sensor
FP8	Autonomy	• Alimentation test • Software test with different low power mode and a simple application to estimate the autonomy
FP9	Internal charging unit	• Alimentation test with different power source (USB, Solar panel, External power 12/24V
FP10	External power	• Alimentation test
FP11	Energy harvesting	• Alimentation test • Battery charging power test with Solar panel
FP12	Core power	• Alimentation test • Inspection of all voltages on the board

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FP13	Power supply output	• Alimentation test • Inspection of 5/12V voltages on the board
FP14	Power control	• Alimentation test • Shutdown test of MikroBUS slots
FP15	Security	• Test data encryption and decryption software
FP16	Updatable	• Application update test by USB cable
FP17	HMI / configuration	• Test of the shell and interaction with the board (on/off of Led and power supply...)

4.3.2.3 Tests results on first hardware version

The first batch of boards produced using the V1 of the LOTUS box design suffered from some problems that were identified during the execution of this plan. They were:

- The component used to activate/deactivate the MikroBUS power supply was not working properly (always on or always off).
- We identified an overconsumption of the board due to two diodes that had a high leakage current.
- Sometimes the LSE clock of the microcontroller did not work reliably.
- On some cards, the Temp/Hum sensor returned zero or wrong values.
- The relay pins were reversed due to the wrong footprint library.
- The FRAM did not work.
- Sometimes the read/write functions of the microSD card were not working properly.

4.3.2.4 Modifications made to the board

To solve these problems, we have updated the design and provided a V2 of the board. The modifications are as follow:

- We changed the component which manage the powering of the MikroBUS slots with another reference while respecting the routing instructions (ground plane, capacitors on the input/output...).
- We replaced some diodes with standard ones having a very low leakage current.
- We have corrected the orientation of some components in the footprint library.
- We have modified the routing to have a more precise ground plan and minimize the distance between the Crystal and the capacitors
- The values of the pull-up resistors on the I2C communication bus are very low, we have increased the values to avoid any possible disturbance.
- We corrected the schematic/routing of the FRAM component which was incorrectly connected.
- We have changed the ESD protection component with another one and internal pull-ups.

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4.3.2.5 Validation of hardware V2

A first batch of 20 board was made with the V2 design. The same test plan was executed on each of them. They all passed the test successfully. This validated the final design of the LOTUS box hardware.

4.3.3 Software of the LOTUS box

4.3.3.1 The LOTUS box SDK requirements

The functional requirements discussed in the previous section dedicated to the hardware are also imposing constraints on the software design. In particular, the expected extreme modularity and diversity of external devices (sensors, actuators, communication cards, …) expressed in FP1 to FP7 lead to design the software in the same modular way. This software must also help to support all the other functionalities like data logging, very low power management, embedded RTC clock, data encryption or HMI management.

To minimize the time-to-market and the financial risk of the development of an IoT solution, the software architecture distinguishes an *application* part which is use-case specific from an *SDK* part which contains the reusable code.

This SDK is divided into submodules (called here *Controllers*), each responsible for controlling different parts of the hardware or other high-level functionalities. The controller requirements are listed in the Table 3.

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Table 3: Lotus Box SDK controller requirements

Functionality	Description
Sensors Controller	- Lotus Box must configure and communicate with sensors to get appropriate measurements and data from different sensors
Actuators Controller	- Lotus Box must configure and control the different system and actuator that will be plugged in. - Lotus Box must provide multiple interfaces and protocols that can communicates with the different industrial systems
Power Controller	- Lotus Box must supply and control power for the different internal components and add-ons - Lotus Box must control his power consumption to optimize the battery life or his main functional. - Lotus Box must provide multiple Power indicators for the main actors.
Data Controller	- Lotus Box must analyze and decode data, if necessary, using the appropriate approach or specification of clients - Lotus Box must store data if needed or when he cannot reach the cloud.
Network Controller	- Lotus Box must use the appropriate network module to transfer data. - Lotus Box must manage the change of network module if needed
Configuration Controller	- Lotus Box must store his metadata and internal configuration - Lotus Box must provide multiple solution to manage and update his configuration by the different main actors.
Firmware Update Controller	- Lotus Box must be able to be updated through different approach and different level using his different components
Shell Controller	- Lotus Box must provide a shell or command line interface to ensure monitoring and configuration for the main actors
Display Controller	- Lotus Box must show the indicators and parameters for the main actors through the configured display module
Geolocation Controller	- Lotus Box must use the appropriate and pre-configured technology to get his position in his environment.

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Security Controller	- Lotus Box must alert if there any anomaly in his internal components - Lotus Box must alert if there any problem with the add-ons - Lotus Box must protect stored data - Lotus Box must protect stored configuration - Lotus Box must ensure security communication if needed - Lotus Box must protect the software and hardware from internal and external attacks - Lotus Box must ensure many levels of security privilege when the system will be managed in accordance with the user role.
Monitoring Controller	- Lotus Box must monitor his internal Components - Lotus Box must monitor all the add-ons and the external system - Lotus Box must ensure an exception handler and a critical handler if there a problem with software
Buttons Controller	- Lotus Box must execute tasks on demand of the main actors

The non-functional requirements help to assess the proper functioning of the system rather than its behavior. In our case, we have identified the following ones:

Footprint: Because devices are constrained, we expect Lotus Box software to have low memory, power, and processing requirements. Overhead due to Lotus Box software should be minimal.

Portability: Lotus Box software isolates applications from hardware specifications. Typically, Lotus Box software is ported to different hardware platforms and interfaces with the board support package (BSP) in a standard way, such as using abstract interface calls.

Modularity: Lotus Box software has a mandatory core. All other functionalities can be included as add-ons if required by the application.

Connectivity: Lotus Box software supports various connectivity protocols, such as LPWAN, Wi-Fi, BLE, etc.

Scalability: Lotus Box software must be scalable to any device type. This means that developers and integrators need to become familiar with a single Lotus Box software for all sensors, network modules and other add-ons.

Reliability: Often, devices are in remote locations and must operate for years without failure. Reliability also means that Lotus Box software must meet certifications for certain applications.

Security: Lotus Box software has add-ons that bring security to the device through secure boot, SSL support, encryption components and drivers.

Test unit: Lotus Box software has multiple components and features. Therefore, we need to ensure that each of them is working properly and that the main function of Lotus Box is working.

4.3.3.2 SDK design and architecture

The architecture of the Lotus Box SDK design is largely inspired by the other SDKs available on the market for other platforms but cannot be a simple implementation of one of them because of its very specific hardware target, its dedicated hardware components, and some flexibilities we want to allow (like to reallocate the pins of the mikroBUS in a way not specified by the standard).

Through the Lotus Box SDK, we aim at simplifying development of an embedded IoT application by

- Allowing the developers to use a user-friendly API and programming interfaces for essential drivers such as SPI, I2C and the serial port and these drivers will be adapted to the constraints of Lotus Box hardware.
- Allowing a simple evolution of the SDK to any evolution of the hardware board.
- Simplifying the porting of a third-party middleware (like the ones provided by Arduino or STMicroelectronics).

To address this challenge, it has been chosen to use object-oriented programming. This paradigm is already strongly present in the world of modern embedded programming and focuses on three fundamental terms (encapsulation, inheritance, and polymorphism) which, combined to good design practices lead to:

- Enhance the expressivity of the code and helps to provide developers with an SDK allowing them to focus on their application.
- Ease the reuse and recycling of the code.
- Save time in terms of code modification and maintenance.

This naturally led to use the C++ language to implement this SDK.

The STM32 libraries provided by the manufacturer offers two programming interfaces to facilitate the programming of its microcontrollers. These interfaces are called HAL (Hardware Abstraction Layer) and LL (low layer). HAL is a set of high-level functions that allows coding any STM32 target, it allows the user to easily use the microcontroller without worrying about the technical details and architecture of the latter, this library can be used for applications that do not require memory and speed optimization. LL is a set of functions that facilitate access to operations on predefined registers of the microcontroller. To be able to use it the user must know all the details that the designer has previously provided on the data sheet of the module.

The Lotus Box SDK uses the LL library to enable optimization of the code for memory usage and execution speed. Figure 11 shows the layers of this SDK, they are further described in Table 4.

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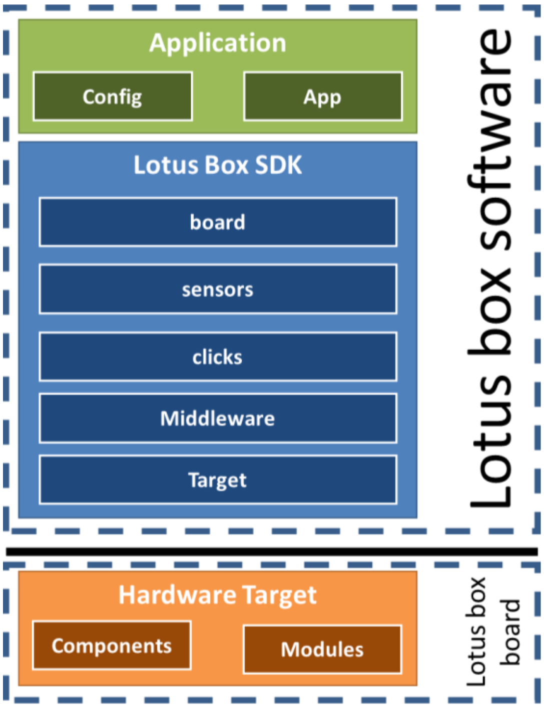

Figure 11: The Lotus Box software layers

Table 4: Description of the software layers of the Lotus Box

Module / Sub-module		Description
Application		• Includes the main application functions and configuration. • Write user application functionalities. • Lotus Box board components configuration. • Execute the functions of the SDK controllers.
	Config	• MikroBUS communication/pins configuration. • Lotus box board universal Extension pins/communication configuration. • Buttons/LED configuration and each hardware component configuration.
	App	• Define the functions of the user application. • Define the functions of the global application and controllers.
Lotus Box SDK		• Includes the Lotus Box driver classes and some drivers related to the STM32 chip, MikroBUS modules driver, and code documentation.
	Board	• Lotus box supported components driver's class. • Peripherals classes definition.

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	Sensor	• Unified interface for sensors, sensor data and interface definition for all supported sensors.
	Clicks	• MikroBUS modules drivers and interfaces.
	Middleware	• Contains all the third-party library and the supported Features like SD card, USB, Serial commands, Displays, etc.
	Target	• STM32 target drivers, configuration files and target main file

4.3.3.3 Class Diagram

To illustrate the modularity of the Lotus Box software as well as the other requirements, the following section will focus on the Sensor class hierarchy which has the responsibility to control and communicate with the different sensors, to retrieve their data properly and process them to finally get the physical measurements. This class belongs to the group of classes called *Controllers.* It allows to define a unique operating mode for any type of sensor linked to the electronic board.

Figure 12 shows a part of the Sensor class hierarchy with internal temperature, humidity, accelerometer, and battery voltage sensors of the Lotus Box. Sensor is an abstract class, derived to define components representing specific types of sensors. Its attributes are shown on Table 5. These subclasses follow the whole concept of polymorphism, each class defines its own implementation of the basic functions of Sensor. Moreover, a derived class sensor can be associated with other communication classes like I2C, SPI, to easily associate the communication protocol of the sensor.

The sensors of the SDK have all the same usage pattern and follow the same state machine shown on Figure 13. This state machine is managed by the `Run()` method of the Sensor class which manages the states and their transitions. These transitions are done by calling abstract functions that return an error. In case of an error, an exception handler is launched to handle it.

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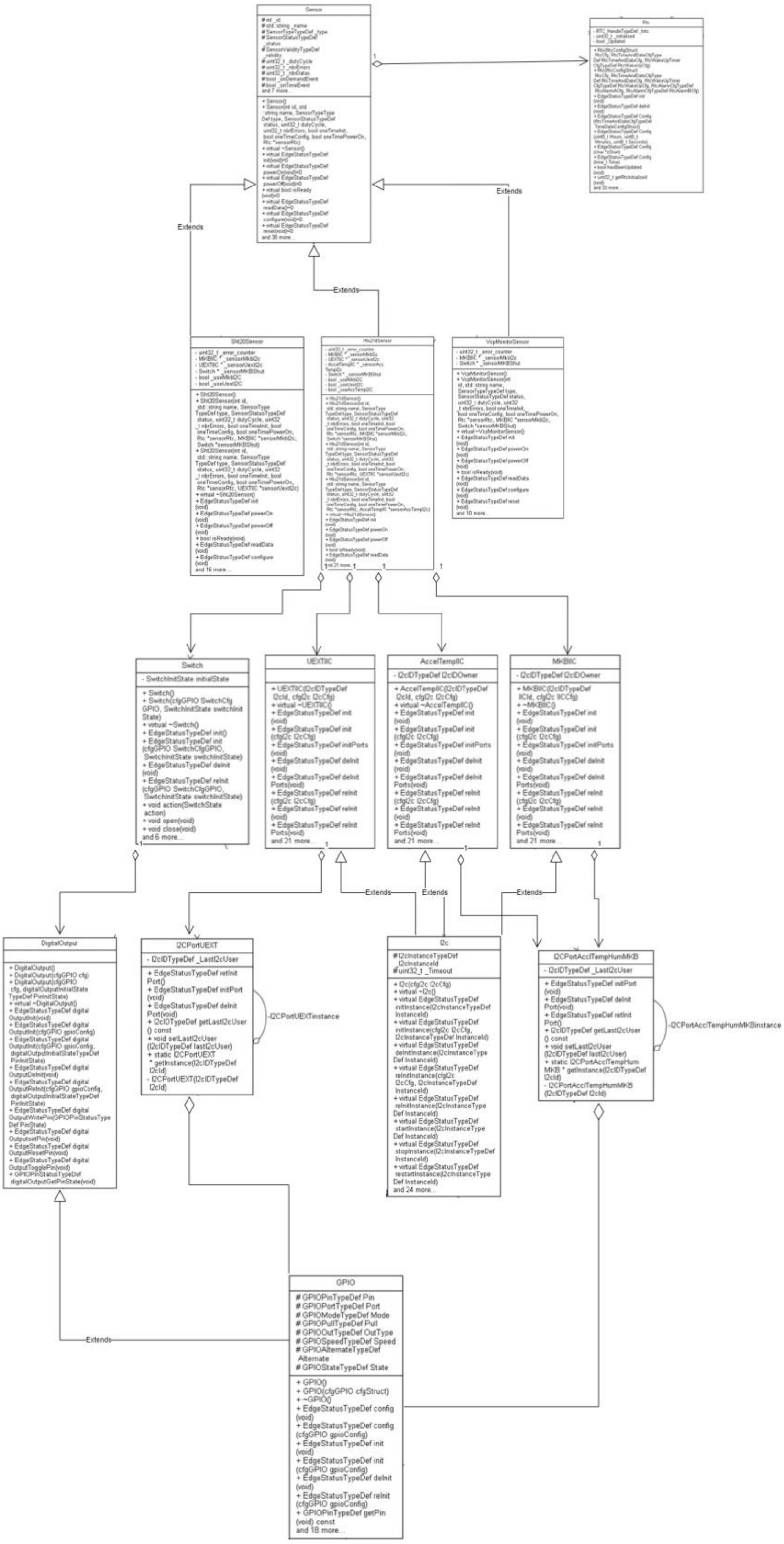

Figure 12 Sensor class hierarchy

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Table 5: Attributes of the Sensor class

Attribute	Description	Example
Id	Sensor Index	1
Name	Sensor name	"HumiditySensorS0"
Type	Sensor task type	MONITORING
Status	Sensor activation status	SENSOR_ACTIVE
Validity	Sensor Lifetime Status	SENSOR_FAILURE
dutyCycle	Period between two measurements in seconds	600
nbrErrors	maximum number of errors tolerated	2
nbrData	number of data read since the activation of the sensor	6
onDemandData	Sensor operating in asynchronous mode	false
onTimeData	Sensor operating in periodic mode	true

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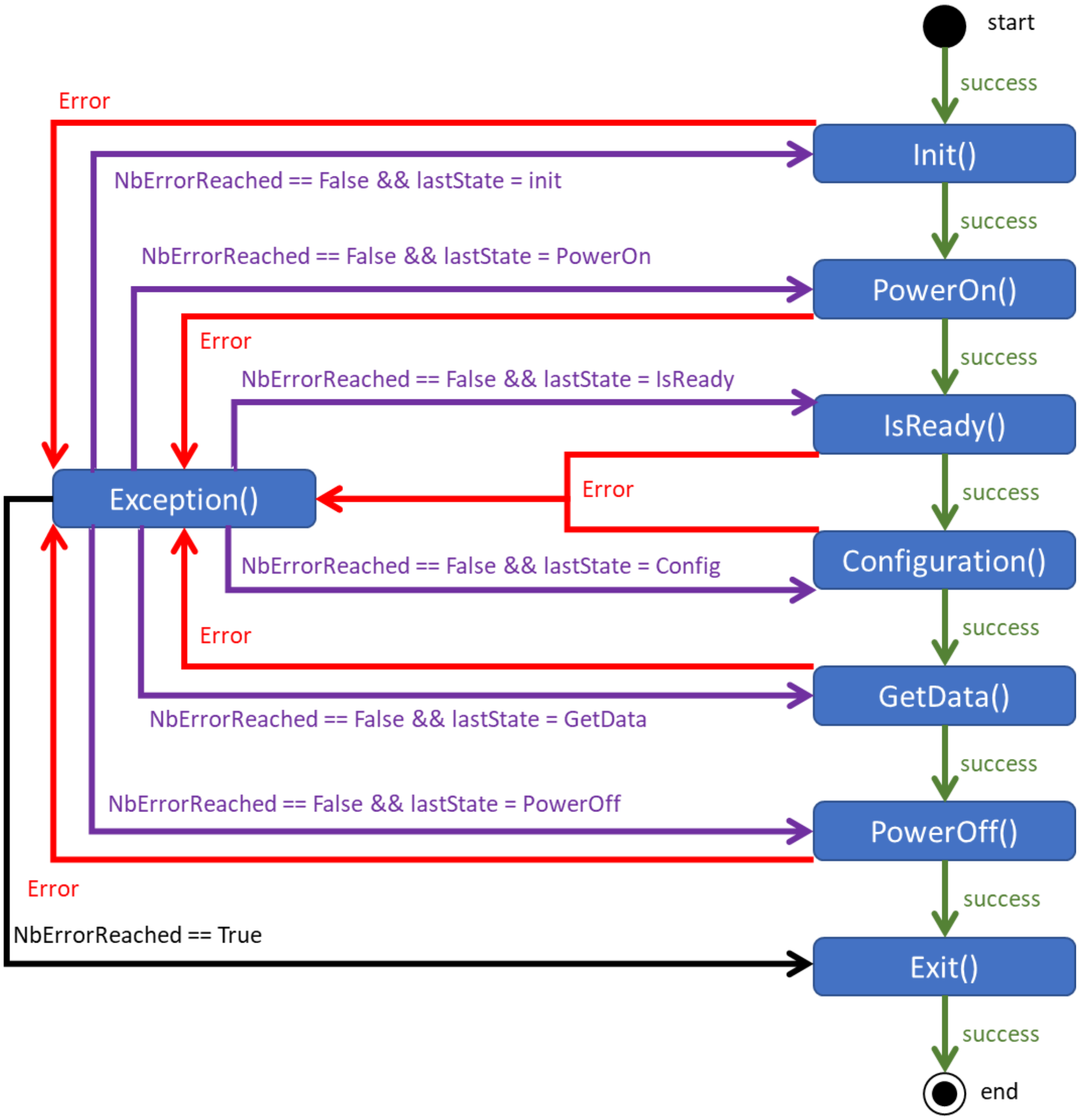

Figure 13: State machine of the Sensors class

4.3.3.4 The LOTUS box application

The Lotus Box application has been developed at the same time as the SDK. For this validation step, it contains a simple infinite loop that read the AFE sensor data and send them through the network.

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4.3.4 LOTUS sensor connection to LOTUS box

4.3.4.1 Validation of communication between the LOTUS box and the AFE

4.3.4.1.1 Communication protocol test setup

The testing of AFE connectivity to the Lotus Box uses a 5V UART interface. Data is then collected from the Lotus Box though another interface such as LoRaWAN, Wi-Fi, or UART. A programming interface based on AT commands is supplied with the AFE.

To test the Lotus Box with the AFE device, the first steps were conducted with a preliminary version of the Lotus Box. The test procedure was:

- Connect the AFE (power and serial bus) to the Lotus Box.
- Power on the Lotus Box.
- Connect a FTDI (USB to UART converter) cable between the laptop and the Lotus Box.
- Run a python script that allows to read the data from the monitor port and store it in a .csv file. This code also displays the curves of these measurements.
- Collect results as a .csv file containing the data of all the sensors.

The acquisition process from the AFE is as follow: a read command is sent with the duration N of the reading window as an input parameter. During that time, measurement points are pushed over the UART interface. The AFE currently limit the reading window N to about 1s. The number of measurement samples sent during that period depends on the queried sensor: the temperature sensor would send 112 samples whereas the conductivity one would send 13 samples.

4.3.4.1.2 Communication protocol validation

The initial phase of the testing plan is to focus on the most innovative part and to validate the connectivity between the AFE and the Lotus Box. Then the stability of measurements is analysed and optimum measurement parameters (i.e., waiting time for measurements stabilisation) are determined.

Table 6: Validation of AFE / Lotus Box communication protocol

ID	Purpose	Process	conditions
1	Check that AFE to Lotus Box connection is established	Execute the python script and check data exchange is taking place	
2	Lotus Box can check if the AFE is ready or not	Query the version of the AFE	
3	Lotus Box can collect data from one sensor of the AFE	Query one raw value from one sensor	One period of measurement (1 second)

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4	Collect data from one sensor of the AFE every N second	Query one raw value from one sensor each N seconds at least	N >=2 seconds. 1 second to collect the measures. 1 second as a pause (power off) for the sensor.
5	LotusBox is able to collect data from all the sensors of the AFE	Scan all the available sensors and get their values	One period of measurement (~1 minute)
6	collecting data from all the sensors every N minute	Scan all the available sensors and get their values each N minutes.	N>=2 minutes 1 minute to finish the measures. Restart the AFE before scanning again the sensors.
7	Identify parameters for stable measurements	Take series of measurement until output stability is better than S % over local moving average	S < 1% over 10 points moving average

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4.3.4.1.3 ID1 and ID2

The initial test plan described in the previous section was carried out. Main results obtained after the connection has first been established (which validates Id1 & Id2) are presented below.

4.3.4.1.4 ID 3: 1 sensor data collection

The curves below show the obtained measurements of the two temperature sensors (T1 and T2):

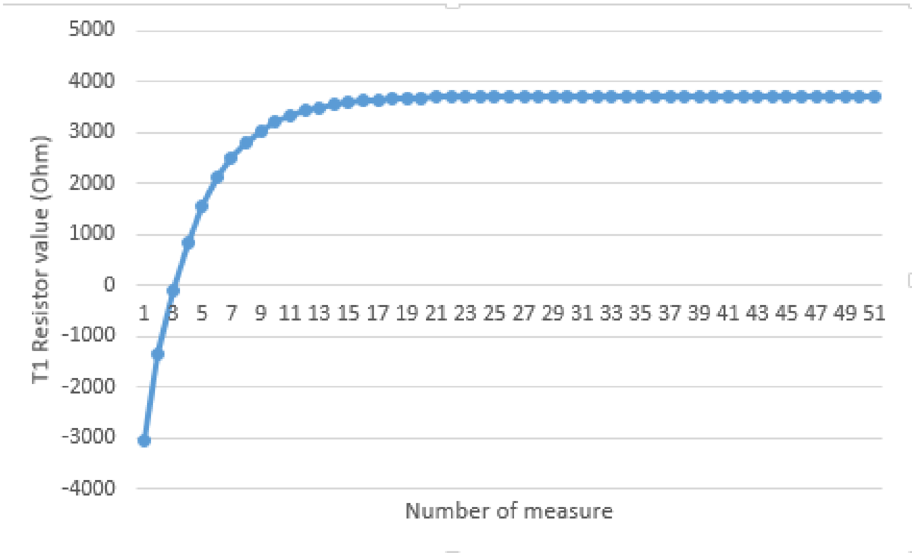

Figure 14: resistor value of the temperature sensor T1 – sampling rate: 10ms

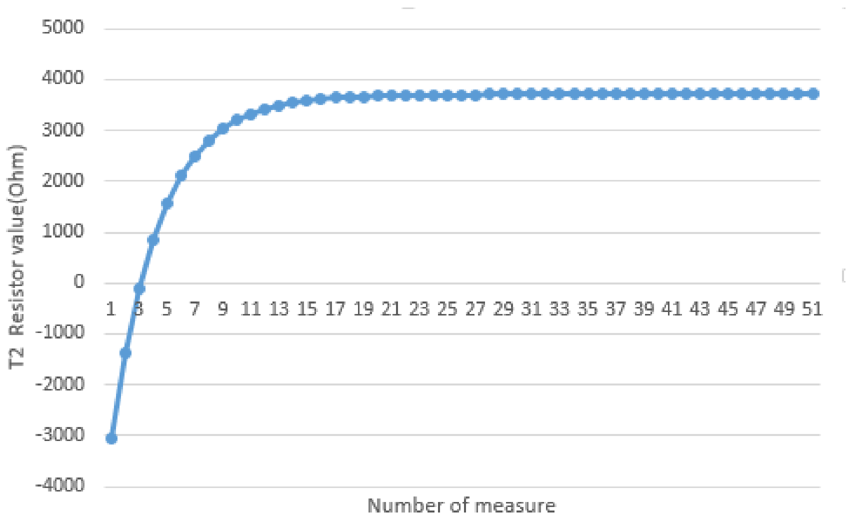

Figure 15: resistor value of the temperature sensor T1 - – sampling rate: 10ms

We notice that the measurements begin to stabilize approximatively from the twentieth measurement. This is further analysed in test Id7.

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4.3.4.1.5 ID 4: Repeated measurements

This part shows that it is possible to sequentially query a sensor by switching it off between measures and get repeatable values.

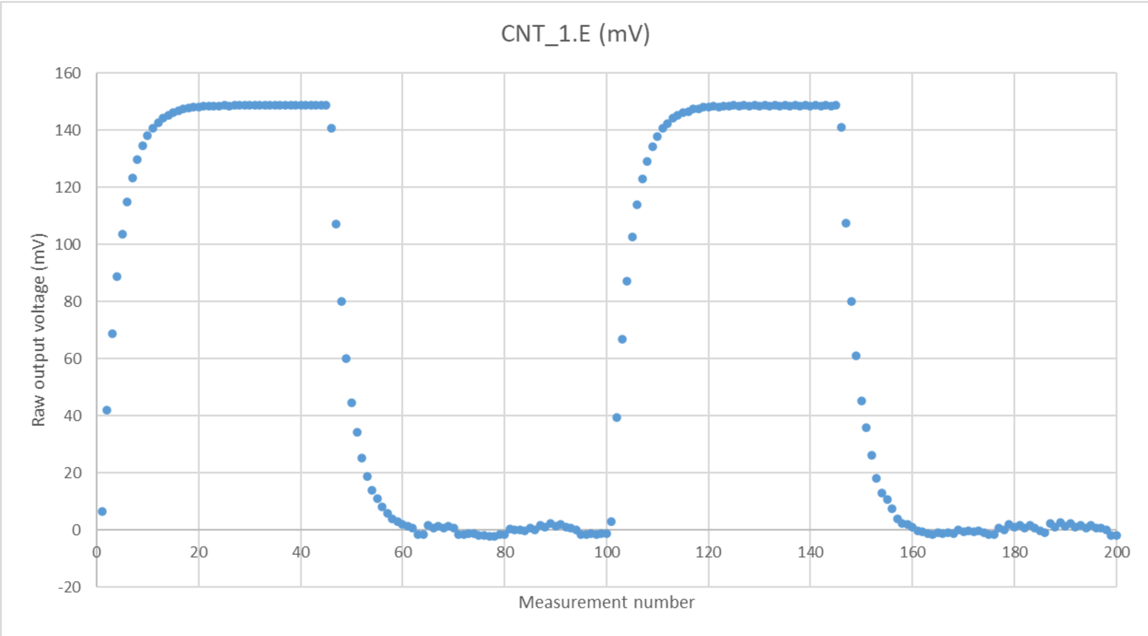

Figure 16: Periodic measurement of a sensor – sampling rate : 10ms.

4.3.4.1.6 ID 5: The measurements of all CNT sensors

In that test, all sensors have been read sequentially one after each other. These have been superimposed on Figure 17. The experience demonstrated the need to power off the AFE between subsequent measurements.

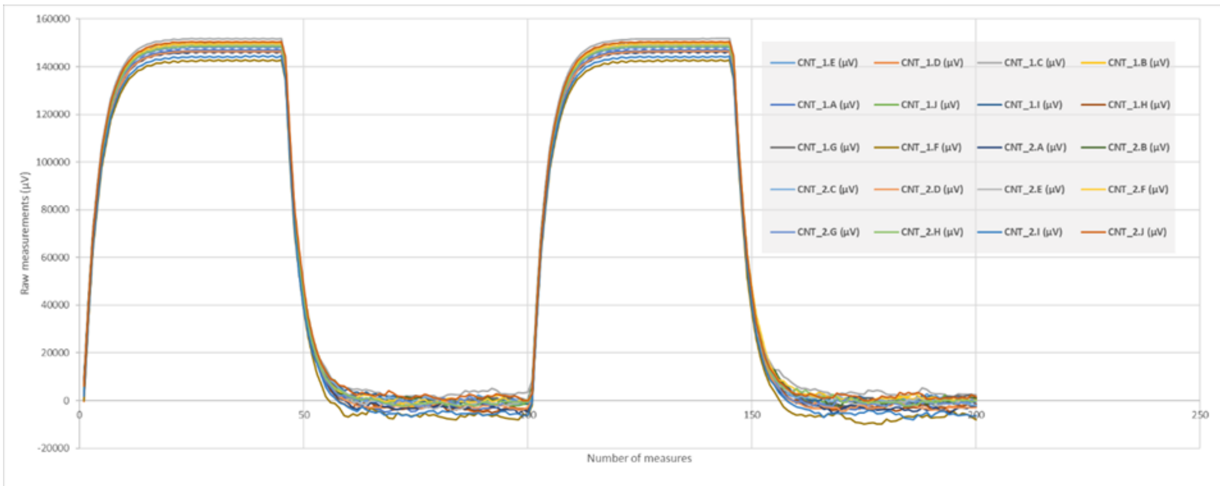

Figure 17:Ssequential measurement of all CNT sensors – sampling rate : 10ms

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4.3.4.1.7 ID 6: Periodically get a measurement from all sensors

The periodic scan of the sensor is currently failing as it requires a manual restart of the AFE between the sequence. To be solved, the ability to switch off/reset the AFE must be implemented as an AT command or as a Lotus BOX capability.

4.3.4.1.8 ID 7: Stabilisation parameters

All measured sensors exhibit a comparable behaviour with a stabilisation period. Once a read command is sent to the AFE, data are collected until the measurement process is stopped. Comparing a measurement with the average of the 10 preceding points show that after 25 measurements, the stability is better than 1%. There are still some small variations which seems to be of periodic nature and need investigation.

For initial configuration of CNT measurement parameters, the following procedure is then suggested:

- Initiate the data collection process
- After 25 values provided by the AFE, collect the 5 next one
- Provide the average of the 5 values as the sensor measurement.

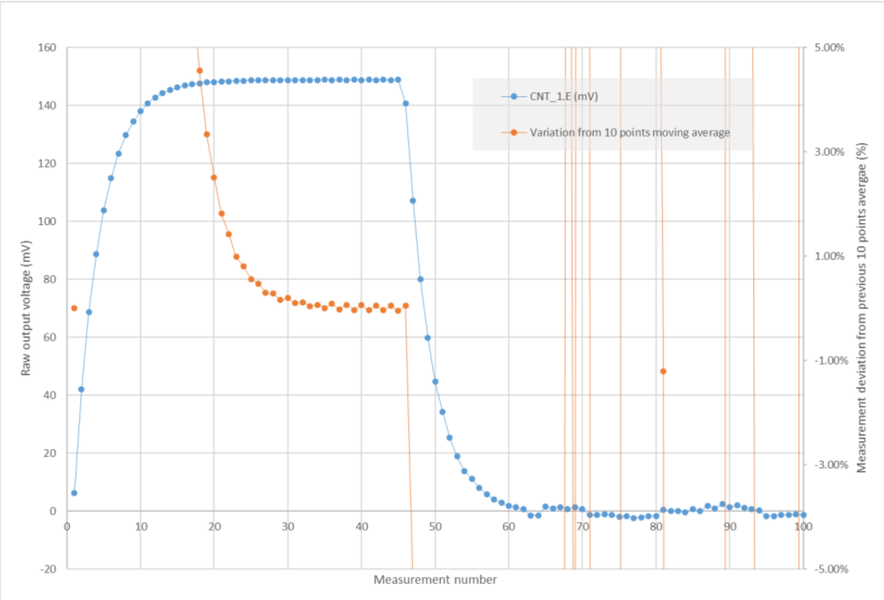

Figure 18: Measurements stabilisation analysis – sampling rate : 10ms – duty cycle 50%.

4.3.4.2 Device driver

A full device driver for the AFE is not yet available since its AT set is not yet stabilized. But, as we saw on the previous sections, everything is in place for its development.

4.3.5 Integration of AFE in the acquisition and processing chain

A full integration setup has been tested to demonstrate the feasibility of a full acquisition chain. (see Figure 19). The Lotus sensor and the AFE communicate with the Lotus box via a serial interface (UART). The acquisition protocol is the one explained in section 4.3.4 that has been implemented in the embedded software, the AFE driver only implementing the AT commands. The result of the data acquisition is sent to a FIWARE context broker used to display a dashboard.

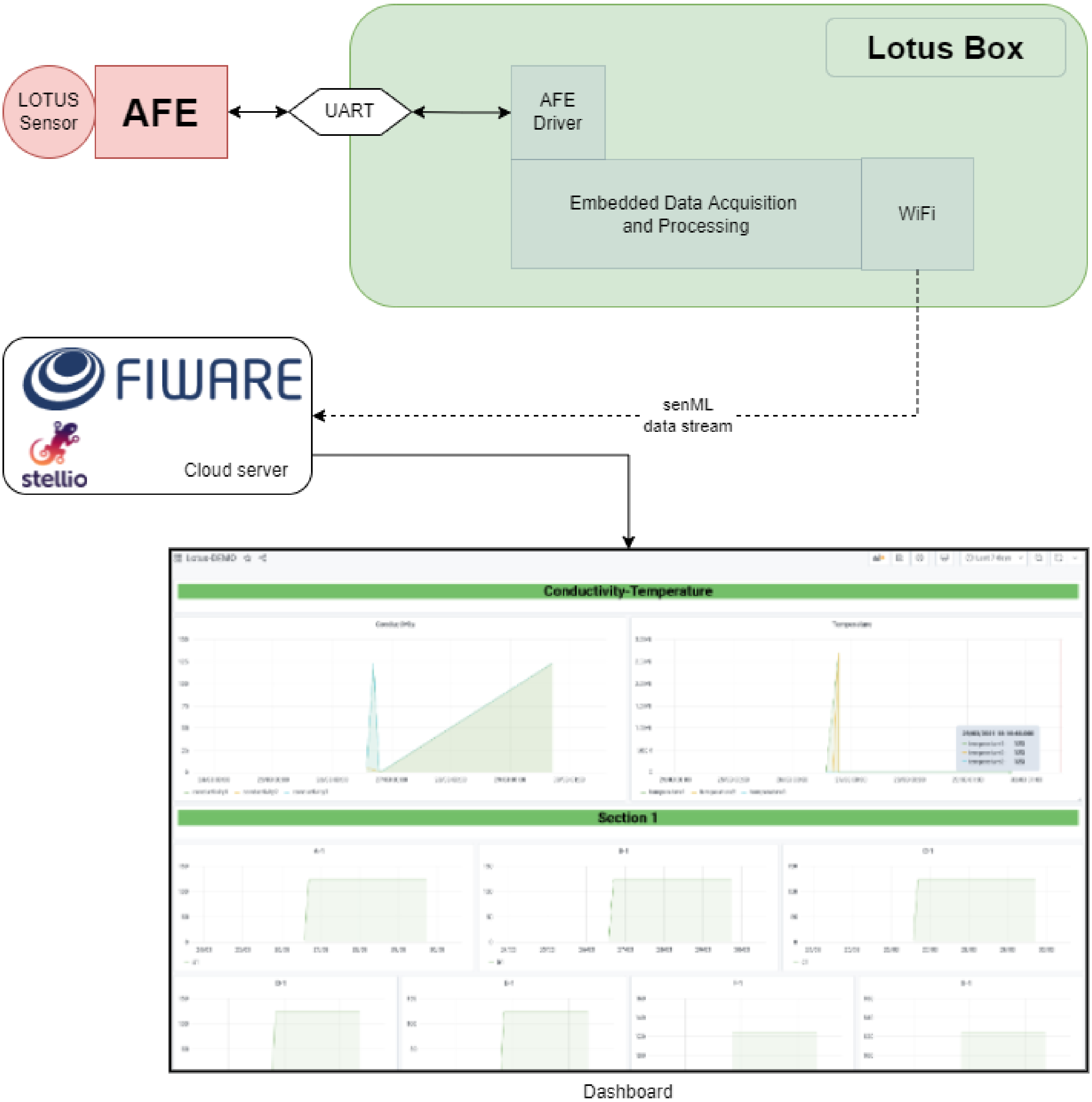

Figure 19: Synoptic of the setup for testing the full acquisition chain.

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The whole chain has been demonstrated to work as expected. A screen shot of the dashboard can be seen on the Figure 19.

4.3.6 Deployment in Quasi-real configurations

Once the validation of the LOTUS sensor to LOTUS Box connection will be completed, the system will be deployed in Sense-City. The testing plan will mostly focus on validating (and if necessary, optimizing) the acquisition frequency and the connection to the cloud, as well as the energy harvesting and energy management capabilities. The sensor performances will be summarily tested to verify that the use of the LOTUS box does not modify existing computer-based calibration, but it will not be the main point of these experiments, as this will have been carried out in the LOTUS sensor validation part.

4.3.7 Configurations for deployment in the field

Depending on the outcomes of the Sense-City LOTUS node validation results, the need for field validation will be investigated. If Sense-City validation data are complete enough, it may be decided to move directly to use case testing (WP6). Alternately, an appropriate test site will be determined close to IITG.

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4.4 Success and challenges in end-to-end integration

Regarding the sensor node, the following results are reported:

- The mechanical package is waterproof up to 8bar or 1m/s in a drink water pipe.
- The electronic cards have
  - very good repeatability in measurements but needs individual calibration (i.e. relationship between measured and actual resistance)
  - noise level at 0.1% in lab setting, but regularly 1% in noisy environment
  - possible partial interferences between neighbouring CNT devices
- The packaging of the sensor chips on the sensor PCB (wirebonding+globtop)
  - After optimization, has demonstrated survival in tap water in lab for two months
  - Still requires initial stabilization and still feature events of sudden device death
- Two mitigations have been developed
  - Redesign of the electronic card is completed
  - A pipe adaptor has been designed and patented to protect the sensor chip from the flow and thus extend the lifetime of its wirebondings.

Regarding the LOTUS box

- LOTUS box validation
  - Two generation of hardware have been tested; the second-generation correcting bugs observed in the first version. The second generation passed fully the validation plan.
  - A suite of operating software has been developed, which successfully passed its validation plan
- Integration of the sensor node to the LOTUS box
  - The LOTUS node can be successfully controlled by the LOTUS box
  - Data is sent to a FIWARE context broker used to display a dashboard

Field deployment has not been validated yet, awaiting the optimized version of the electronic card and the completion of flow adaptor prototypes. It will be carried out early during 3rd period of LOTUS.

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5 Validation of sensing performances

5.1Outline

This section presents first TRL 3 (deionized water) sensitivity screening experiments for LOTUS chips, then systematic TRL 4 (drink water) characterization experiments. To exploit these drink water experiments, different calibration and ageing algorithms are then introduced and their performances detailed.

5.2Sensing performances in deionized water

5.2.1 Objectives

The results reported in this section were acquired in deionized water in lab setting (TRL3). They are not meant to provide exhaustive sensitivity analysis but were designed as screening experiments before testing in drink water to provide insight into main trends and interferents, and to evaluate differences in sensitivity between inks.

5.2.2 Summary of results in deionized water

The results of chemical sensitivity screening experiments in deionized water are summarized in Table 7. They are sorted from lowest to highest LOD. The conclusions are following:

- Clear chemical differentiation brought by FFUR for chlorine (x2), arsenic (x1.7), magnesium (more than x3), glyphosate (x3.5), nitrate (x -1.3)
- Detection limit to pH, chlorine, arsenic is particularly relevant for drink water use cases.

Table 7: Sensitivity to various chemicals in deionized water

Beaker of deionized water with no stirring. ***Lab noise level: ~0.1%***	**Sensitivity** **Pristine CNT**	**Sensitivity** **FF-UR CNT**	**Estimate of lab detection limit** **3xNoise/Sensitivity**	**Expected level in drink water**
pH 6 to 8	1.6%/pHu	1.8%/pHu	0.2pH unit 0.5µg/L H_3O^+ at pH 7	6-8 Preferably <0.1pH unit
15 to 300µg/L **Arsenic(III)**	0.1%/µg/L	0.17%/µg/L (approx. same with CF-OX)	2µg/L	Measured: 1 to 100µg/L Legal threshold: 10 to 50µg/L
Chlorine from 0 to 5mg/L in PBS at pH 7	-1.4%/mg/L	-3.9%/mg/L	0.08mg/L	0 to 5mg/L

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				Preferably < 0.1mg/L
20mg/L Glyphosate	0.1%/mg/L	0.35%/mg/L	0.9mg/L	Measured: <1µg/L Legal threshold: 0.1mg/L to 1mg/L
100mg/L Magnesium(II)	<Noise	0.03%/mg/L	10mg/L	Measured: 1 to 30mg/L (NB: Irrigation water: 30 to 50mg/L)
1000mg/L Nitrate	+0.3%/g/L	-0.4%/g/L	750mg/L	Measured: 1 to 100mg/L Legal threshold: 10mg/L
Stability (transistor devices)	>9months	>3 months <9 months		

Regarding temperature and conductivity, the main results are summarized in **Erreur ! Source du renvoi introuvable.**, **Erreur ! Source du renvoi introuvable.** and **Erreur ! Source du renvoi introuvable.**. Temperature and conductivity sensors respond well to respectively temperature and conductivity variation, and calibration model can be proposed. CNT sensors show clear sensitivity to pH even when conductivity and temperature are varying. However, temperature needs to be accounted for in the temperature model.

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5.3Chemical sensing in drink water

5.3.1 Objectives

This section presents the first available TRL 4 characterization data of LOTUS sensor, namely characterizations of the sensor chip with its target acquisition chain and in non-ideal water (tap water). The deliverable presents also how these characterizations are exploited to provide calibration of the sensors and then an error estimation of the sensor outputs. These data are to be used to build the system datasheet.

5.3.2 Description of protocol and dataset

Content is confidential

5.3.2.1 Tested chips

5.3.2.2 Characterization protocol

5.3.2.3 Reference (external) sensors

5.3.2.4 Acquisition of sensor chips data

5.3.2.5 Datasets building

5.3.3 Active chlorine prediction in drink water at varying pH leve

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5.3.3.1 Data processing and KPIs

5.3.3.2 Accuracy on chlorine prediction

5.3.3.3 State of the art reproducibility in active chlorine sensing

5.3.3.4 Comparison to state of the art

Our review paper "Electrical and Electrochemical Sensors Based on Carbon Nanotubes for the Monitoring of Chemicals in Water—A Review" (acknowledging LOTUS) proposes an exhaustive status (up until mid 2021) of existing carbon nanotube sensors for water quality monitoring (90 papers analyzed). There are only 3 papers on chlorine sensing, and only one of these discussing HClO, compared to which the results presented here are better (factor 10 in sensitivity, factor 2 in detection limit, testing at different pH). The batch reproducibility of the sensors is not discussed in these papers, and very rarely addressed in the literature on CNT sensors[1]. To the best of our knowledge (this will be confirmed for the publication in preparation), this level of reproducibility in CNT chemical sensor (gaz or liquid sensing) is state of the art

Table 8: State of the art, by mid-2021, of CNT-based chlorine sensors

Type of CNT	Functionliza tion	Form of chlorine	Limit of detecti on	Sensitivi ty	Transduct ion mode	Processin g of CNTs	Electrode material	Substrate	Selectivi ty	Ref
MWC NT	**Pristine**	**Free chlorine (hypochlor ous acid form)**	**~12ppb**	**1.5µA/p pb**	**Chemistor**	**Ink-jet printing**	**Pt**	**Si3N4**	**Tap water, pH from 6 to 8**	**This work**
MWC NT	Pristine	Hypochlori te ion	<5ppb	Logarith mic 39% /decade * (0.03~8 ppm)	Chemistor	Dielectro phoresis (aligned MWCNT)	Cr/Au	Glass	No informati on about selectivit y, pH informati on not provided	1
MWC NT	**Epoxy EpoTek H77A (non covalent)**	**Hypochlor ous acid at pH 5.5**	**20 ppb**	**0.15 µA/ppb (0.02~4 ppm)**	**Voltamm etry**	**Paste poured into tube and thermally cured**	**Epoxy /MWCNT composit e**	**Not provided (tube)**	**Validate d in real water matrices (tap water and swimmi ng pool)**	**2**
SWCN T	Phenyl capped aniline tetramer	Free chlorine	<60ppb	92 nA/deca de	Chemistor	Drop casting	Au	Glass	Non selective to different	3

1 Highly reproducible, hysteresis-free, flexible strain sensors by inkjet printing of carbon nanotubes, Michelis et al., https://doi.org/10.1016/j.carbon.2015.08.103

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	(covalent)			(0.06~60 ppm (linear up to 6ppm))					oxidants – list of oxidants not provided Regeneration possible	

[1] L. Hsu, P. R. Selvaganapathy, E. Houqe and P. Kruse, "A carbon nanotube based resettable sensor for measuring free chlorine in drinking water," in *Proc. IEEE Sensors*, 2014.

[2] L. Yang, M. Li, Y. Qu, Z. Dong and W. J. Li, "Carbon nanotube-sensor-integrated microfluidic platform for real-time chemical concentration detection," Electrophoresis, vol. 30, p. 3198–3205, 2009.

[3] R. Olivé-Monllau, A. Pereira, J. Bartrolí, M. Baeza and F. Céspedes, "Highly sensitive CNT composite amperometric sensors integrated in an automated flow system for the determination of free chlorine in waters," Talanta, vol. 81, p. 1593–1598, 2010.

5.3.3.5 Limitation of the study and next steps

These results show very high sensitivity to HClO in tap water under varying pH, which in turn yield very good sensing performances that are reproducible from device to device and repeatable over several days.

There are two important topics regarding HClO not addressed by the study and that will be investigated further:

- Response time: in the absence of real-time measurement of chlorine, response time cannot be evaluated.
- Response under decreasing and randomly varying chlorine: this has not been assessed either.

5.3.4 Bayesian calibration – Active chlorine and pH prediction in drink water

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5.3.4.1 Including measurement uncertainties in calibration to derive sensitivity to secondary parameters

5.3.4.2 Principle of Bayesian calibration

5.3.4.3 Application to the dataset

5.3.4.4 Baseline and ageing management

5.3.4.5 Application to active chlorine prediction

5.3.4.6 Bi-parameter prediction with temperature correction

5.3.4.6.1 Methodology

5.3.4.6.2 Linear model in HClO and ClO^-, both below 1mg/L

5.3.4.6.3 Linear model in HClO and pH with HClO and ClO^- below 1mg/L

5.3.4.7 Discussion on the outcomes of bi-parameter calibrations

5.3.4.7.1 Success of the method

5.3.4.7.2 Further exploration of these datasets

5.3.4.7.3 Future calibration protocols

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5.3.5 IITG Water lab - Test bench

IITG Water Lab is a laboratory 80L water loop under construction at Indian Institute of Technology, Guwahati. Its purpose is to conduct extensive mono-parametric and multi-parametric experiments on LOTUS sensors under continuous water flows and in different water models. It will contribute notably to understanding the sensor performances and setting up advanced calibration protocols (T2.2b).

Apart from the Test bench setup, high end analytical instruments are also available at IITG Water lab: Ion Chromatography, Mass Spectrometer, Atomic Spectrometer, and HPLC; the lab is also equipped with High end computer systems for the heavy computational works and data analysis.

The simplified outline structure of the testbench is visualized in Figure 20. It has 4 major blocks, marked as B1, B2, B3 (red dashed boxes) and 80L tank. Each block serves a unique purpose in the testbench, as detailed below:

5.3.5.1Analyte introduction, B1

The block B1, consists of 6 analyte containers (with the option for extension) that houses 6 different salt solutions. These solutions are transferred to the 80L tank via the dispensing unit which consists of peristaltic pumps, each connected to each of the analyte containers. The volume of the analytes to be transferred to the 80L tank is controlled by the peristaltic pumps which are controlled by a central PC (via UI).

5.3.5.2 Water source, B2

The block B2, supplies water from water source (typically from tap) to fill the 80L tank. This water is then recirculated and sensor measurements are taken. The block also houses a turbine flow sensor (FC 01) which measures the volume of water transferred to 80L tank and stops the flow once desired volume is reached (tank is filled).

5.3.5.3 80L tank

Once the water from water source is filled and analytes are added; the stirrer inside the tank is utilized to mix the contents homogenously. The 80L tank also consists of temperature control system (TC 02) to regulate the temperature of the solution inside (to maintain it at the desired level set by the user via UI).

The tank also has a drain valve, which is used to remove the solution form the tank once the experiment is completed.

5.3.5.4Sensing block, B3

The solution from the 80L tank (after mixing for a set period defined via UI) is pumped by pump VFD_P at a predefined pressure (to maintain desired flowrate, as set by user via UI) and recirculated back to the tank continuously for a set duration (set via UI). The pump VFD_P is equipped with VFD to manipulate the flowrate. The bypass structures connected to the recirculation pipeline houses the LOTUS sensors along with the flow adaptor. Based on the requirement up to 5 LOTUS sensors can be

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connected in the recirculation line via bypass structures. Other analog sensors including but not limited to temperature, pH, chlorine, conductivity are also connected in the recirculation line in the sensing block B3.

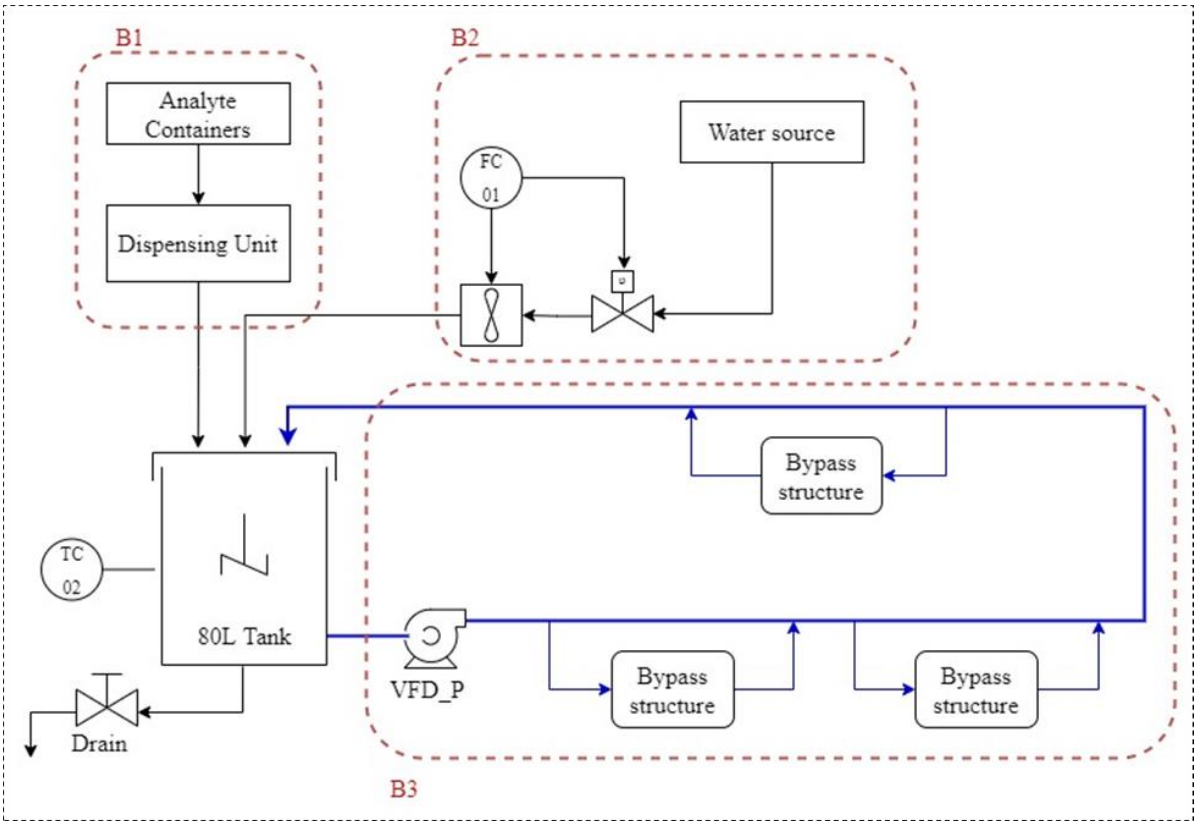

Figure 20: IITG Water lab testbench outline

The 3d design of the test bench is visualized in Figure 20. A central PC is utilized to monitor as well as control the complete testbench. Data collected from LOTUS sensors as well as other analog sensors are continuously saved into a database in the same PC.

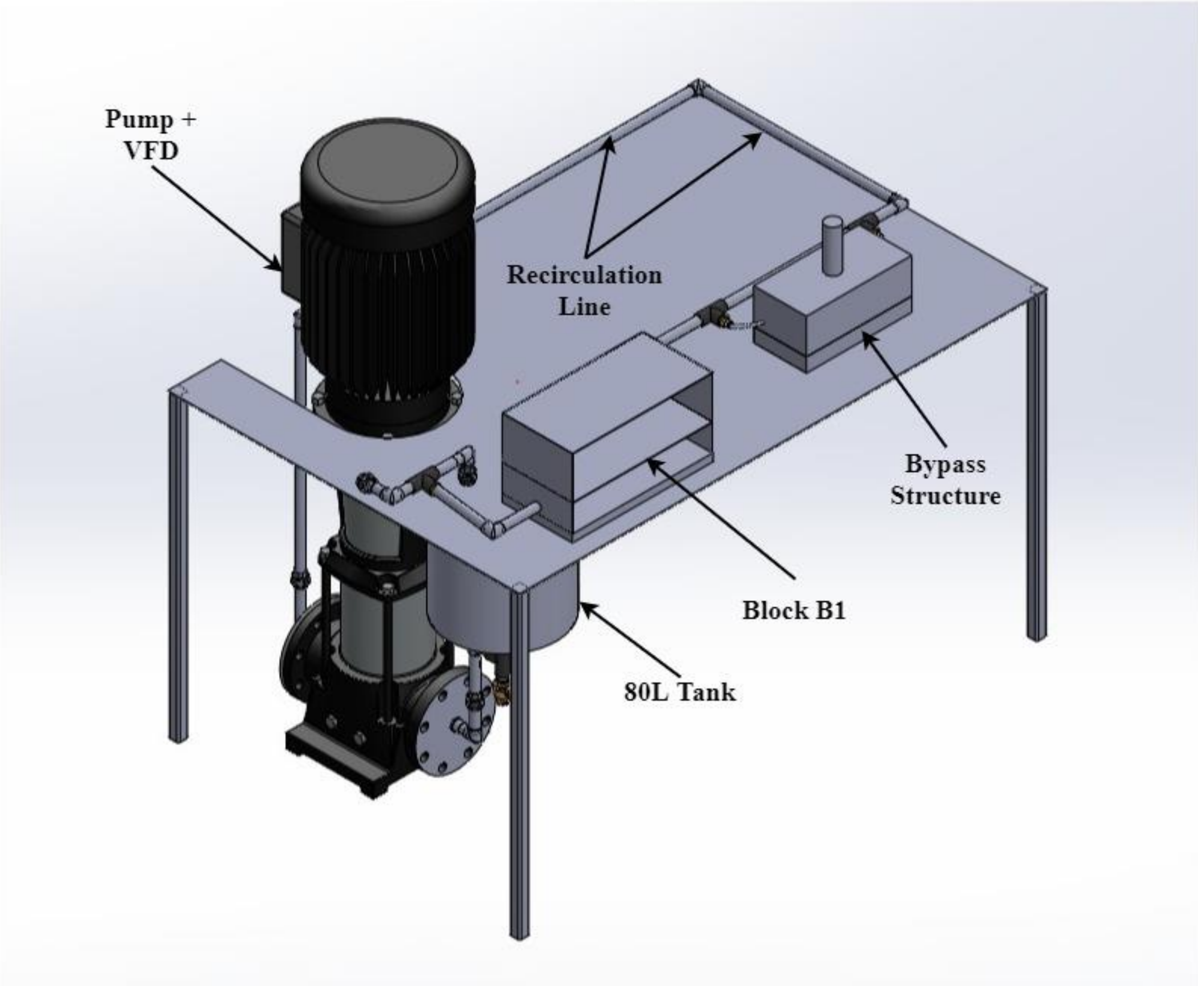

Figure 21: 3D design of the IITG water lab testbench

5.3.6 Calibration transfer techniques for sensor mass production

5.3.6.1 Calibration transfer

Calibration of a multi-sensor system such as LOTUS sensor is time consuming and resource hungry task. Due to minute deviations between the sensor chips of different LOTUS sensor units and differences in the environment, the calibration model developed with one LOTUS sensor (primary sensor) can not be used as is with another LOTUS sensor (secondary sensor) or in a different environment. There would be differences in the performance of the calibration model developed with the primary sensor when used with secondary sensor. One way to overcome this issue is to recalibrate the secondary sensor (or primary sensor in the new condition/environment) which is not efficient. An alternative to this recalibration is calibration transfer. Calibration transfer also aids in mass calibration of mass-produced sensor units.

5.3.6.2 Calibration Transfer techniques

Several techniques exist for calibration transfer between spectroscopic instruments; but these techniques cannot be used as is with the LOTUS sensor as the underlying principles change. Few techniques that perform exceptionally with spectral data (from spectroscopic instruments) may not perform satisfactorily for the non-spectral dataset like that of LOTUS sensor, as each individual sensor in the LOTUS sensor chips, may not necessarily exhibit inter-correlation unlike spectral data.

The most common calibration transfer technique employed with spectral data is Piecewise direct standardization (PDS) which makes use of the adjacent wavelengths and develop a calibration transfer model. On the other hand, for multisensor systems like the LOTUS sensor, adjacent sensors don't have any real significance rather than being placed physically closer on the sensor chip.

From an analysis of a synthetic non-spectral dataset, several calibration transfer techniques developed for spectroscopic instruments were assessed for its performance on such datasets. From this analysis, it was concluded that techniques like PDS, double PLS (partial least squares) and ensemble of extreme learning machines (ELMs) provided satisfactory performance in transfer efficiency. Also, the efficiency of these techniques has significant dependence on the transfer standards chosen.

5.3.6.3 Implementation context for LOTUS sensors

Once a primary calibration model is developed with extensive experimentation for a LOTUS sensor, the same model can be transferred to mass produced LOTUS sensors with comparably reduced experimentation without the loss of performance of the primary calibration model. The same techniques can also be utilized for drift correction of LOTUS sensors, thereby providing as efficient framework for mass calibration and regular maintenance with effective resource usage.

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5.3.7 Summary of achievements in drink water

- Calibration: Two algorithms for calibration are proposed, one based on standard inversion of a linear monoparameter calibration model, the other on Bayesian multiparameter calibration including measurement uncertainties.
- Active chlorine can be predicted in tap water with pH freely ranging from 6 to 8
  - High reproducibility between devices and chips, but no difference between pristine and FFUR CNTs.
  - Best achieved MAE (with Bayesian monoparameter calibration) is 0.047mg/L of HClO for HClO below 1mg/L (chip 68).
  - Sensitivity is 5 to 10 times stronger than observed in deionized water
  - Detection limit is roughly estimated at approx. at 0.012mg/L in average over all device.
- Bayesian calibration allows to predict both ClO^- and pH
  - For ClO^-, best MAE 0.08mg/L with chip 68 (MAE on HClO 0.11mg/L)
  - For pH, best MAE 0.36 pH unit with chip 73 (MAE on HClO 0.11mg/L)
  - All three chips can be used for ClO^- prediction, only chip 68 and 73 for pH prediction.
  - Sensitivity to pH is comparable to what is observed in deionized water (around 1%)
  - Sensitivity to ClO^- is stronger than sensitivity to pH

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6 Conclusions and next steps

6.1 End-to-end integration

Regarding validation of the sensor node integration, the mechanical package is waterproof up to 8bar or 1m/s in a drink water pipe. The electronic cards have sufficient repeatability and low enough noise for use in lab settings, but suffer interferences and high noise in realist environment, which has motivated the redesign of the electronic card, currently completed and under test (at M38). The sensor chips wire bonded to PCB have demonstrated 2 months survival in lab tap water, but still suffer from occasional sensor death due to fragile wire bonding (different optimization pathways are available). To mitigate this, the sensor nodes will be deployed in the field through a patented flow adaptor system.

The LOTUS box designed for wireless field deployment of LOTUS node has also been validated on the hardware and software front. Integration of the LOTUS node to the LOTUS box has been demonstrated, including data display on the dashboard supported by a FIWARE context broker.

Next stage of validation will be deployment in realist (Sense-City) and field conditions, as soon as the flow adaptor and the updated electronic card are ready.

6.2 Sensing in deionized water

The results of chemical sensitivity screening experiments show clear chemical differentiation brought about by FFUR for chlorine (x2), arsenic (x1.7), magnesium (more than x3), glyphosate (x3.5), nitrate (x – 1.3). Detection limits estimated for pH (0.2pH unit), arsenic (2µg/L) and chlorine (0.08mg/L) are particularly relevant for drink water use cases. Magnesium detection limit (10mg/L) is relevant for irrigation water.

Temperature and conductivity sensors respond well, and calibration models can be proposed, although error metrics are still high because of the high noise level (best MAE at 0.9°C and 44µS/cm). CNT sensors show clear sensitivity to pH even when conductivity and temperature are varying strongly. However, temperature needs to be accounted for in their calibration model.

Regarding chemical sensing, the two main next steps are 1) systematic testing of the sensitivity in the presence of interferents to prepare for use in varying water matrices, and 2) preparation of tri-parameter sensing by the addition of a third functional ink on chip, with focus on arsenic. First devices with three inks have already been fabricated.

6.3Sensing in drink water

Calibration of three sensor chips in drink water according to the same protocol has shown that the best parameter to use is the relative resistance variation for each device. A correction factor has been developed which allows to compensate for baseline jumps (e.g. chip ageing).

Two algorithms for calibration are proposed, one based on standard inversion of a linear monoparameter calibration model, the other on Bayesian multiparameter calibration including measurement uncertainties.

Active chlorine can be predicted in tap water with pH freely ranging from 6 to 8 with high reproducibility between devices and chips, but no difference between pristine and FFUR CNTs. Best achieved MAE (with Bayesian monoparameter calibration) is 0.047mg/L of HClO for HClO below 1mg/L (chip 68). The detection limit is estimated in average at 0.012mg/L. Sensitivity is 5 to 10 times stronger than observed in deionized water

Bayesian calibration allows to predict both ClO^- and pH. For ClO^-, best MAE 0.08mg/L with chip 68 (MAE on HClO 0.11mg/L). For pH, best MAE 0.36 pH unit with chip 73 (MAE on HClO 0.11mg/L). All three chips can be used for ClO^- prediction, only chip 68 and 73 for pH prediction. Sensitivity to pH is comparable to what is observed in deionized water (around 1%). Sensitivity to ClO^- is stronger than sensitivity to pH.

For a research point of view, these results are state of the art in terms of calibration algorithms and CNT sensor for drink water. However, they rely on a reduced dataset with high measurement uncertainties on reference quantities. For further application, the different algorithms proposed here need to be validated by long time characterization in flowing water in lab and field, monitored continuously with high accuracy chlorine and pH sensors. The testbenches will be available early in 3rd period at IITG and Sense-City.

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Appendix 1 – Description of testing facilities

Platine Lab

Platine Lab is located at Ecole Polytechnique. It dedicated to nanosensors fabrication, characterization and reliability analysis. This facility - started in 2014 – is being developed within a partnership between Ecole Polytechnique, CNRS and Université Gustave Eiffel. Figure 22 shows the available equipment at Platine Lab. With respect to LOTUS probe validation, the key equipment is the water testbench. It is used to for LOTUS chip and LOTUS sensor validation. Beside equipment in Platine Lab, EP staff has also access to various characterization apparatus such as SEM, AFM, TEM, Raman spectroscopy...

Figure 23 and Figure 24 highlight the key feature of Platine water bench. The system is operated through Labview. The bench also allows for dissolved gas testing (O_2 and N_2) (not shown on the images). Up to 3 assembled sensors or sensor chips on PCB can be tested together with 2 to 4 reference probes at the same time. They can be connected either to their dedicated electronic cards (in LOTUS, an AFE) or high-resolution lab equipment (Keithley double channel sourcemeter in series with Keithley 64 channel multiplexer). Two types of reference probes are available, from Aqualabo (pH/temperature and conductivity/salinity/temperature) and from Atlas (temperature, pH, conductivity, dissolved oxygen). Regular calibration of the reference sensors is carried out. In addition, sampling can be done from the solutions and specific chemicals may be analysed with reagent-based colorimetry (using Aqualabo Odeon photometer). The acquisition of a ion chromatography system is being investigated.

Platine water bench is well suited for testing in deionized water, model water and tap water with various concentrations of chemicals. Due to the small volume in play, water composition can be switched very quickly – steps of 15min are easily possible. Temperature, conductivity, pH interferences may be studied. Ageing can be studies over time frames of a few days, not easily over weeks on end. Turbidity and flow impacts on performances and ageing cannot be assessed.

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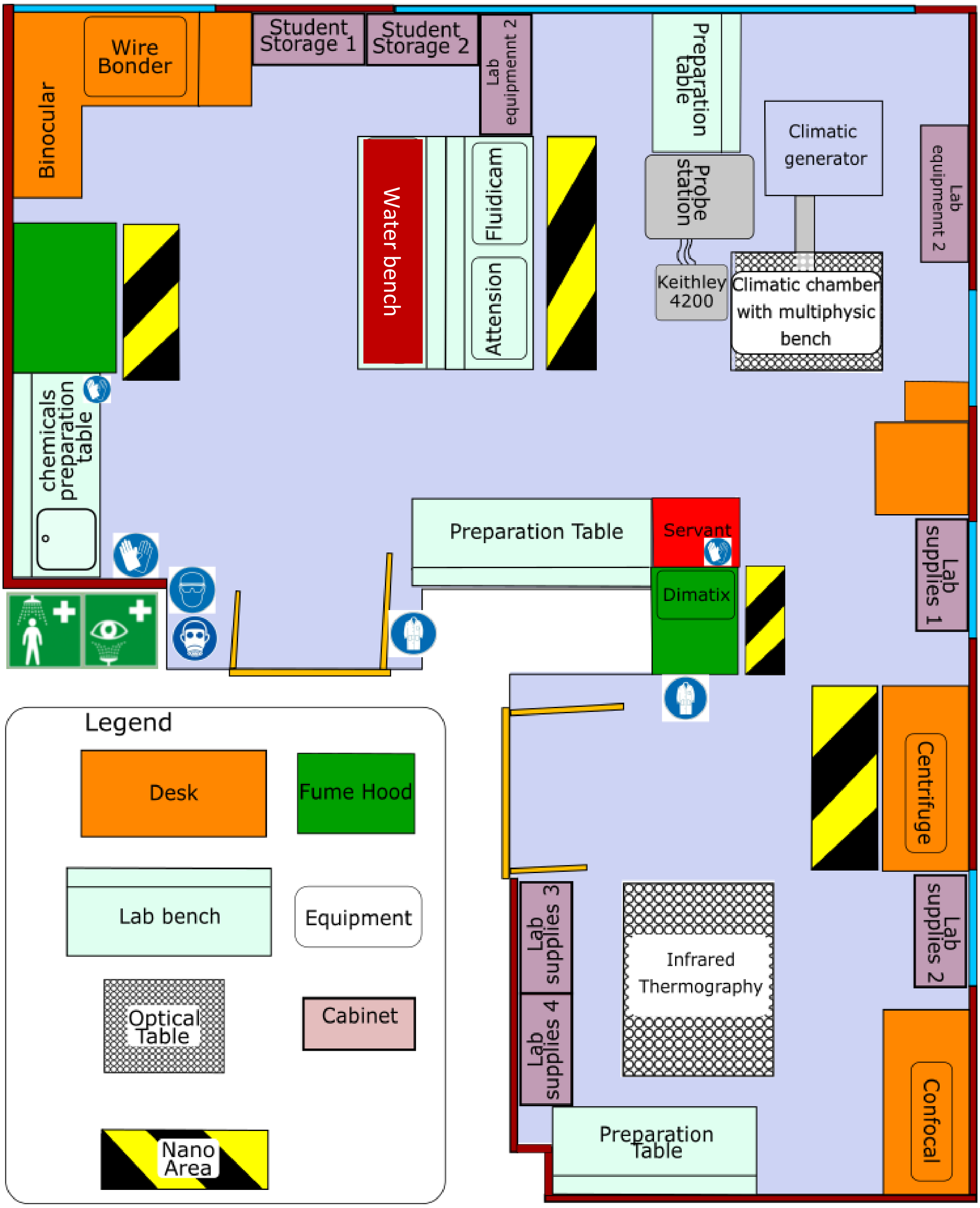

Figure 22: Platine Labs available equipment

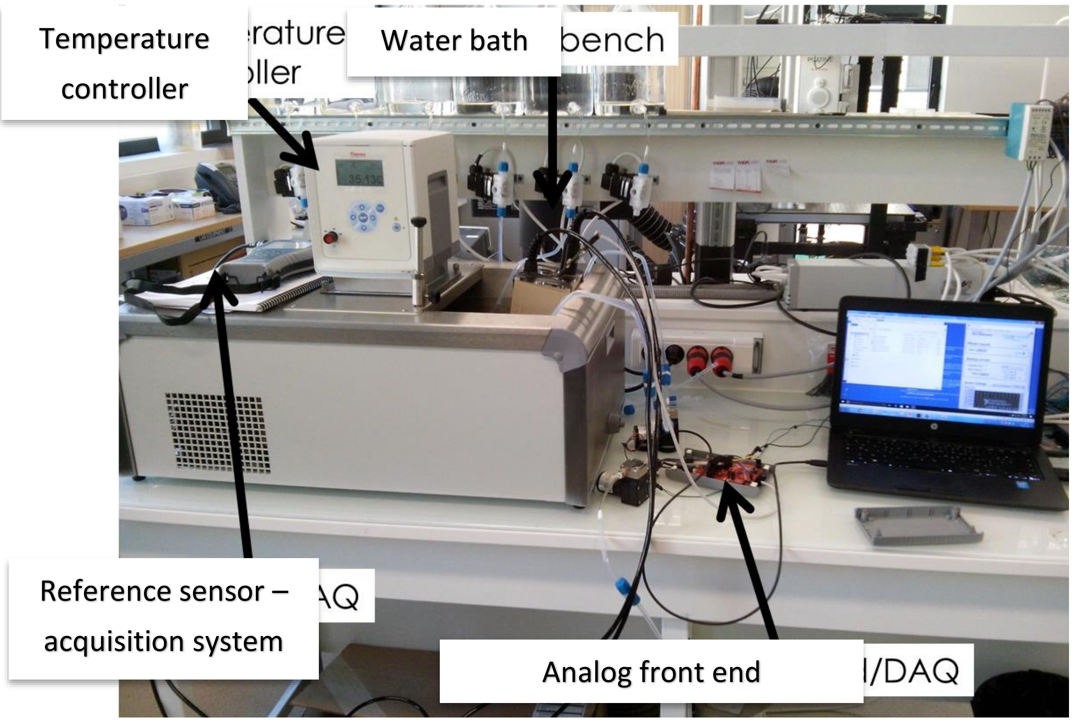

Figure 23: Platine Lab water bench with its water bath placed within a temperature controller. Reference online sensors are placed in the water bath together with the target sensors.

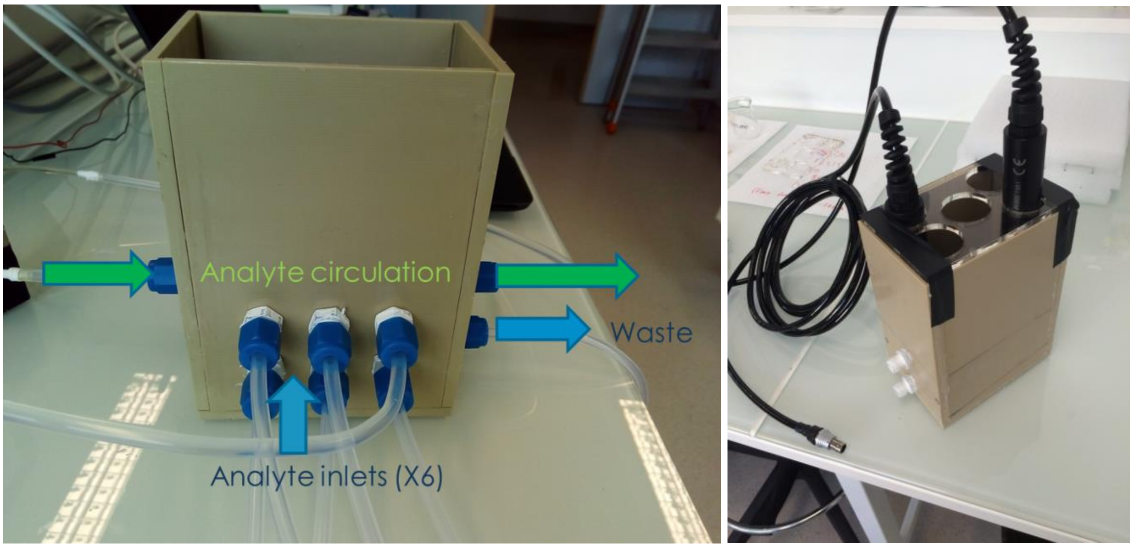

Figure 24: Left - Zoom on the water bath. The water bath is connected to a pump for water cycling (enabling homogenization of analytes) and for reset (emptying to waste and cleaning). Right – Setup to test 3 assembled probes together with 2 reference sensors

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Sense-city water loop

Sense-city[2] 40m/1000L water loop is located at Université Gustave Eiffel, Marne-la-Vallée (East Paris Area). It is an outstanding tool to validate the reliability of water sensors in quasi-real conditions. Successful completion of trials in Sense-City correspond to validating TRL 5 for sensors for drink water network. It will be used to complete LOTUS sensor and LOTUS node validation in quasi-real conditions (T2.2b and T2.2c).

Figure 25 to Figure 29: Water loop sensors show the schematics of the loop within Sense-City chamber. The water loop has four main features:

- Hydraulic setup:
  - 1000L Tank: the tank is filled with tap water – in contact with the heat exchanger of the thermal regulator.
  - The water loop: total length is 44m; among which 38.5m are pressurized. Internal diameter is 93.8mm, for a total volume of water of 310L. Pipes are in PVC/PEDH.
  - A 1m20-deep visit chamber is installed in the middle of Sense-City chamber. Two saddle clamps are available inside the visit chamber to mount two sensors probes with same geometry as Proteus probes. A bypass is available within the visit chamber and could be also outfitted if needed with 1 or 2 saddle clamps.
  - To empty the water in the loop, it can be either released into Sense-City model wastewater network, which empties into Sense-City groundwater reservoir or in the public wastewater network.
- Thermal regulator:
  - It operates by circulating a calorific fluid through the loops of a copper heat exchanger located at the bottom of the water tank.
  - Temperature between 5°C and 40°C can be reached.
- Pressure-flowrate regulator:
  - It controls both the pressure and the flow rate in the chamber
  - Not all pressures and flow rate can be achieved with the current regulator. At low flow rate (10m3/h – 0.1m/s), pressure up to 16bar can be reached. At 1bar, flow rate up to 80m3/h – 0.8m/s is achievable.
- Online monitoring:
  - the reference sensors are listed in Figure 29: Water loop sensors.
  - Continuous data collection into Sense-City supervision system (SCSS) is carried out.
  - Data can be exported in CSV or directly collected from the Sense-City supervision system in real time.

[2] https://sense-city.ifsttar.fr/

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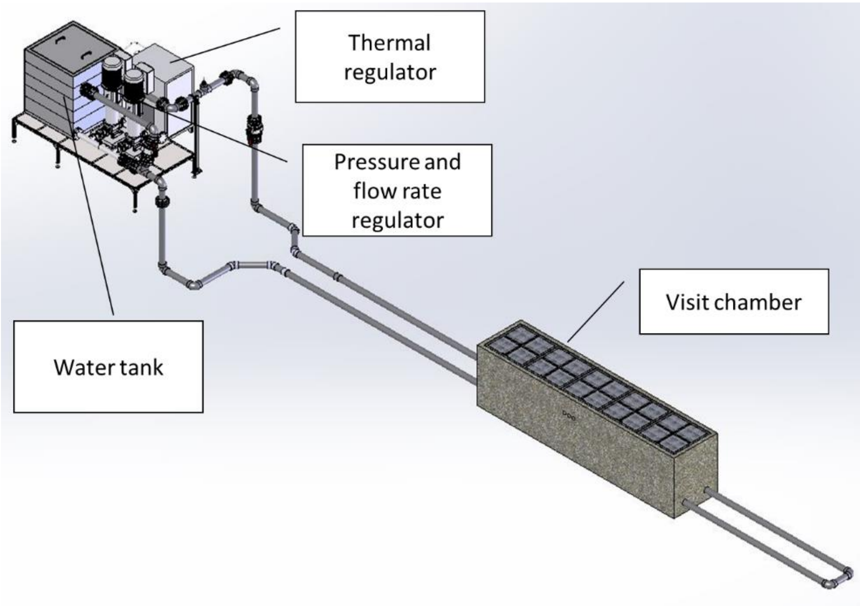

Figure 25: Schematics of the Sense-City water loop with its regulation systems

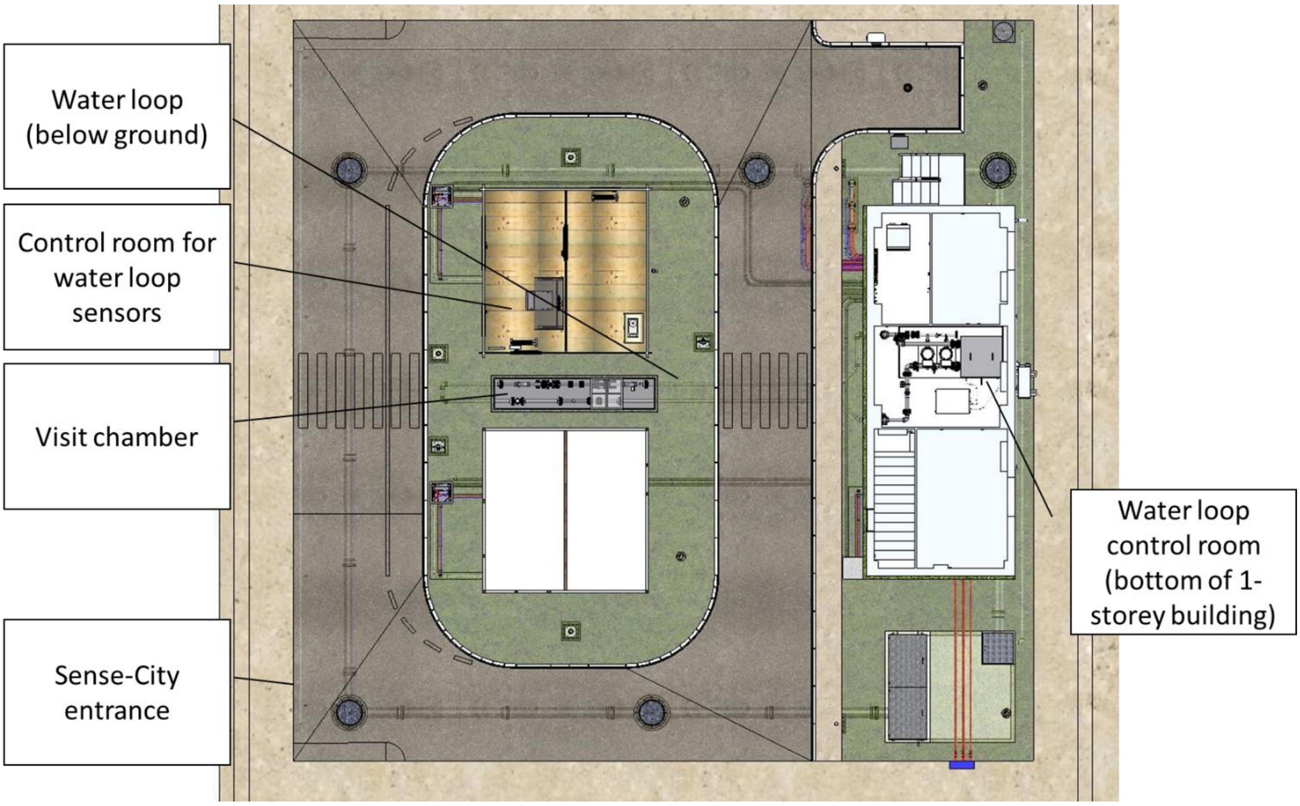

Figure 26: Implantation of the loop within Sense-City chamber – top view

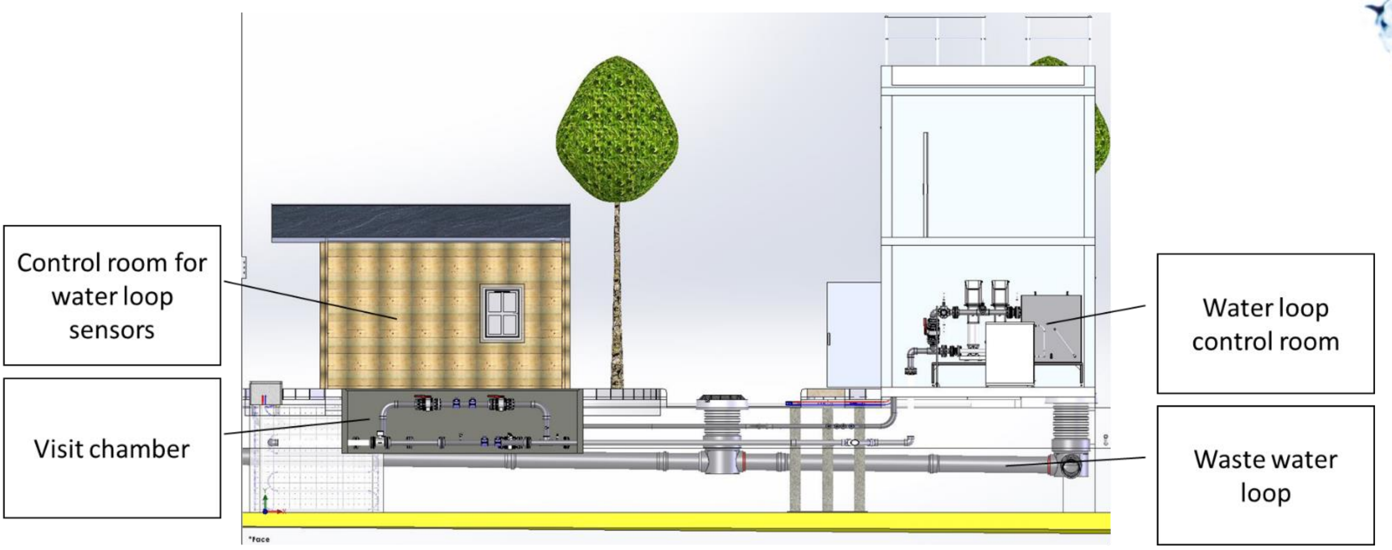

Figure 27: Implantation of the water loop within Sense-City chamber - side view

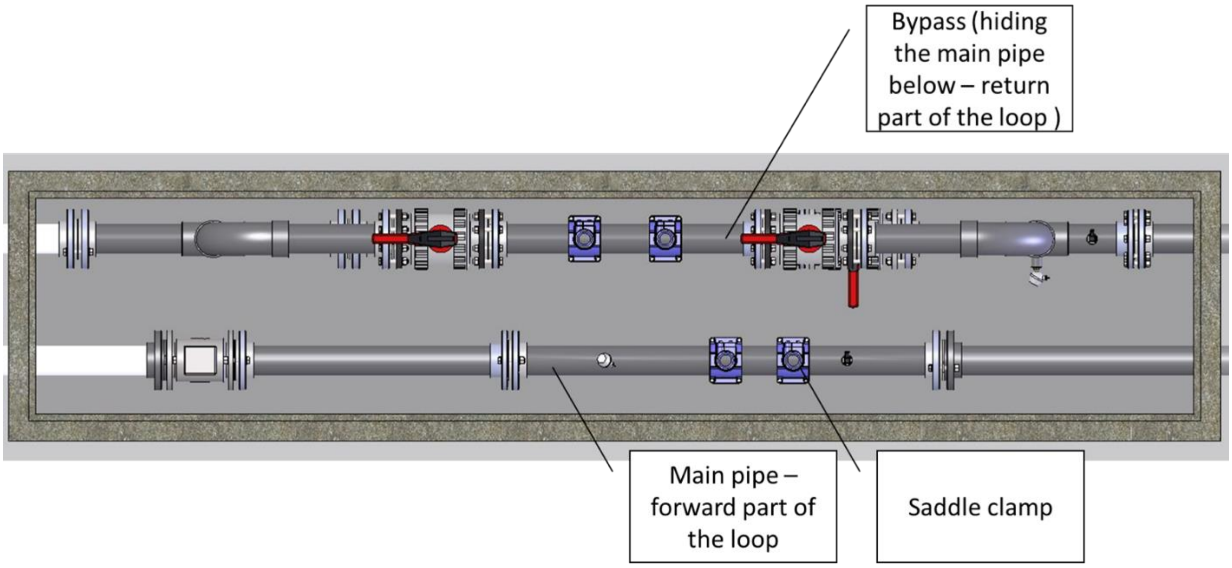

Figure 28: Topview of the visit chamber

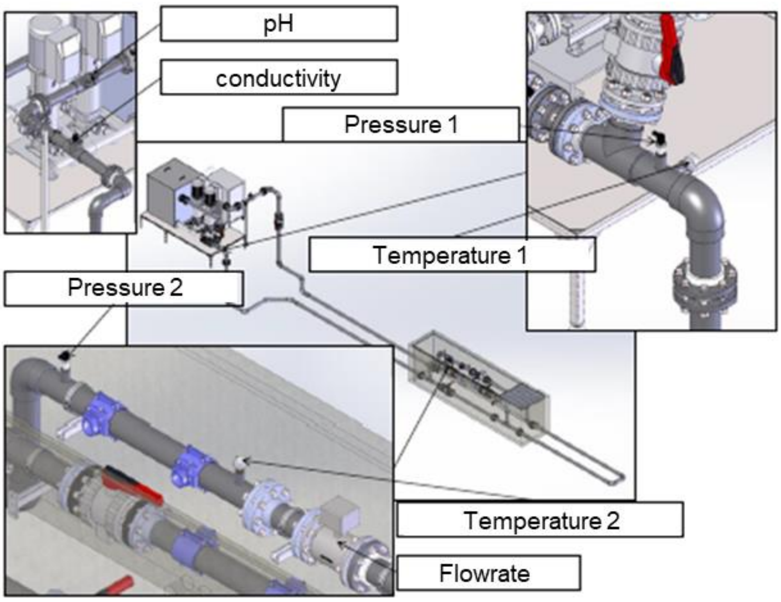

Figure 29: Water loop sensors – positioning in the loop - chlorine sensor is also available but not displayed

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EGM facility

The EGM facility located in Sophia Antipolis, France, is equipped for mechanical and electronical development and integration. It is being used in the LOTUS project for the development of the LOTUS Box, and to contribute to the development of LOTUS sensor mechanic part. Both parts are described in detail in D2.1. The focus here in D2.9 is the LOTUS node validation, namely the integration testing between LOTUS Sensor and LOTUS Box in different functional and environmental conditions.

The EGM lab includes

- Electronic standard equipment for electronic testing of IoT networks and devices
- Series of development boards and gateways covering wide range
- A LoRaWAN network (gateways and LoRaWAN server)
- Oven for mechanical mounting and temperature testing (+10°c above ambient to +250°C)

LOTUS is co-funded by the European Commission under the Horizon 2020 research and innovation programme under Grant Agreement N° 820881 and by the Indian Government, Ministry of Science and Technology.

Figure 30: Top: the glue dispenser and Head holding mechanism with a LOTUS head, Bottom: Head holding mechanism with a LOTUS head in the oven

- Computers with CAD tools: Altium (electronic design), SolidWorks (mechanical design)
- Datacentre with 3 servers, each hosting several virtualised Linux machines running a FIWARE platform in a monitored environment with CI/CD capabilities.
- A sea aquarium for sea water quality sensors development

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Figure 31: LOTUS box with water quality probe