



# PROJECT FINAL REPORT

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## 2 Final Publishable Summary Report

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### A bio-mimicry enabled artificial sniffer

#### 2.1 Executive Summary

Dogs are known for their incredible ability to detect odours. This makes them precious assets for a broad variety of security applications. However, dogs can only be trained to a limited set of applications, get tired quickly and for many border control applications are poorly accepted by the public. This has created an increasing drive to develop artificial sniffers able to replace or complement dogs and cover a broader range of applications.

Many technological approaches have been tried out but without a resounding success to-date. This can be explained by the complexity of the problem at hand:

- Sensitivity / Selectivity: the need to extract a few molecules from a larger set of molecules
- Environmental complexity: the often challenging “odorant” background
- Moving targets: the challenge of detecting odours from moving targets
- Practicality: the cost and lack of portability of the technologies employed
- Usability: the often need for the intervention of especially trained staff for the use of the technologies employed

Current approaches to "artificial sniffing" are constrained in terms of applicability and require extensive training for each new odour. Therefore most of the current efforts aim at integrating complementary technologies, which makes it difficult to propose upgradable inexpensive devices.

The SNIFFER project proposes a very different concept offering a comprehensive one-stop shop solution to this need. The key idea is to couple selective binding elements from biological systems to highly sensitive diamond sensor technologies. SNIFFER addresses a broad range of border security challenges by delivering an innovative set of highly effective, flexible, low-cost and portable solutions, integrating, adapting and improving advanced sensor technologies, thereby leveraging and complementing the capabilities of dogs. During its 40 months, the SNIFFER project has achieved the following objectives:

1. **Provide a range of practical border security solutions to complement dogs and leverage their capabilities.** SNIFFER has focused on enabling solutions for specific usage contexts:
  - Body-scanning people at airports and at border crossing points
  - Scanning open luggage
  - Scanning the interior of cars for illegal substances and explosives
  - Scanning containers at harbours
  - Scanning closed luggage on a luggage belt

These examples illustrate the breadth of the SNIFFER approach. Several of these solutions were prototypes and evaluated in laboratory tests and in small field trials.



2. **Develop artificial sniffer technologies.** SNIFFER has delivered significant progress for a range of technologies and corresponding SNIFFER sub-systems as building blocks for future solutions:
- *Collection & Sampling:* a new generation of sampling techniques makes it possible to sniff not only gas molecules but also particles
  - *Preconcentration:* micro-preconcentration technologies based on porous silicon micro-channels have been improved and especially adapted to SNIFFER sensing
  - *LBP - engineering:* tailored odorant binding proteins (OBP) bind specific complementary families of molecules
  - *Biosensors:* sensors attained sensitivities in the 1 ppb range by mimicking olfactory mechanisms from nature; they immobilise the OBPs on the current generation of diamond sensors
  - *Learning systems:* multi-parametric training methods are applied to arrays of LBP-enriched diamond sensors support to match macro-patterns against a previously constituted database; this database has been built by exposing the sensor array to a large sample set
  - *Self-diagnostic:* advanced self-diagnostic features allow for the detection of “sensor fatigue” when the sensor has been contaminated and is no longer sensitive enough

These technologies have a broad application potential in many areas such as food safety, environment, industrial control and health.

### 3. Optimise efficiency and impact of SNIFFER:

The project has actively carried out *dissemination activities* to engage with third parties. It built a community of users and experts interested in SNIFFER solutions in the European border security community, in particular by interacting regularly with FRONTEX.

The project has prepared *future exploitation* not only by achieving its technological objectives but by coordinated exploitation planning and IPR management.

The SNIFFER RTD roadmap worked in 2 iterations to produce 2 sets of devices to support a practical dialogue with the users and solve the integration problems early in the project life:

- A first generation set "V1" of SNIFFER devices for a first assessment based on concrete metrics provided by the SNIFFER users (including advice from the UAG members);
- A second generation "V2" built from the assessment of V1 at project end. V2 integrated even more powerful technologies illustrating the full potential of the SNIFFER approach from the user perspective on an additional set of usage cases.

At project end, SNIFFER Partner CEA won the Wolfgang Gopel Memorial award at the 16<sup>th</sup> International symposium ISOEN on Olfaction and Electronic noses held in France early July 2015. ISOEN is the only global event dedicated to E-senses (E-nose, E-tongue and E-eye) and to odor generation/transmission. It awarded Emmanuel Scorsone from CEA, SNIFFER Project Coordinator, with the Wolfgang Gopel Memorial award for: “Diamond micro-cantilevers as transducers for olfaction receptors-based biosensors: application to the receptors M71, OR7D4 and OR1740” presentation.



## **Work performed since the beginning of the project**

**WP1 “Security Usage Cases, Metrics & Validation, Societal & Ethical Aspects”** delivered to the SNIFFER Consortium solid and reliable user requirements and functional specifications of the envisioned devices. WP1 was originally structured by six concrete usage cases. These requirements were validated after a dedicated meeting with the SNIFFER User Advisory Group (UAG) in Manchester in July 2012. During the following 12 months, WP1 continued to provide guidance and feedback to the technical partners on usage issues for the future SNIFFER solutions.

Between November 2012 and January 2013 WP1 examined the details and set-up of the midterm field trials and their possible implications. A field trials preparation plan was prepared with valuable contributions from the user partners. Work then started to identify the necessary clearances and authorisations for these trials. The field trial description presented the Lab Testing protocols and the Laboratory Assessment Methodology proposed by ST(SI)<sup>2</sup> and CREL. During the Munich meeting in spring 2013, lab trials were also planned to ensure the prototype would be functional and safe to be used in real life conditions.

After the project midterm review the consortium decided to limit the use cases to 3, letting aside Use Case 5 (UC5) which involved containers and human odours detection. The project scope focused on the rest of the use cases in order to develop 2 devices – instead of 4 originally anticipated – and to broaden the range of substances to detect.

Furthermore, instead of running full-scale field trials in the mid-term of the project, a task that required overcoming a number of logistics and authorisations problems, the device was extensively tested in a specialised lab and plan a “close to real life conditions” field test at the last months of the project.

Partner 3DSA, WP leader, consolidated the report on National Ethical Approvals that covered the EU privacy and Data Protection Legal Framework and presented the outline for the Trials Procedure Protocols. Initially, extensive work took place to investigate the different ethical and societal issues regarding pursuing security through the detection of odours which became irrelevant once the scope of the project refocused. Furthermore, recommendations were provided in order to ensure the compliance of the SNIFFER devices with ethical and fundamental rights standards by technological design. 3DSA also investigated the required actions and necessary authorisations for the implementation of the final field trials according to the trial protocols that would be developed. To simplify things and avoid time consuming authorisations, the field trials were restricted to the members of the consortium and a few stakeholders (Greek Police, Athens International Airport authorities). These field trials took place in Athens International Airport between 10-12<sup>th</sup> of March and thanks to the involvement and support from both the AIA’s authorities and the Greek Police, were very successful.

### **WP2 “SNIFFER Device Integration” integrated, delivered and supported operational SNIFFER sensor system prototypes.**

The integration work package proceeded iteratively with two cycles of specification, integration and testing. Early into the project it delivered refined specifications of functionalities and system design architecture for SNIFFER devices 1 and 2 focusing on body scanning devices and searching technology at open luggage and cars.

At project midterm system design in sampling, preconcentration and detection device developments for the V1 version was completed. All related and relevant mechanical and electronic sub-elements had been constructed, integrated and tested. Interface definition, electrical and flow design between sampling, pre-concentrator and detector devices were aligned with all involved system partners. Integration of the sub element units were already going to start and field test design and planning were on a mature level. Major system integration aspects between partners had been gathered. The



functionalities of the different sub-systems were aligned and the systematic interaction to achieve the device integration were ensured. All sampling and detection submodules, including the Automatic Trace Collector module, the ESP sampler modules, the pre-concentrator and the main SNIFFER detector module, were completed by the various partners involved and could be integrated altogether successfully. A slight delay in the delivery of the Automatic Trace collector module was observed but the device was finally delivered and this delay had no impact on final integration. Integration was led by CEA with a strong involvement with many partners, and in particular UNIMAN, AIRBUS, ARMINES/EPFL, and TTS.

Based on the experience built from the first version V1 testing, WP2 provided a more detailed specification and description of the devices for the V2 developments, in particular as related to container inspection and luggage belts investigations in airports. This work was carried out in conjunction with the work on use cases in WP1 and resulted in an updated list of devices to be built within the course of the project. Following the feedbacks received from laboratory trials of V1 and end-users observations and comments, a different integration approach to V1 was undertaken in V2. Here the choice was made to sample narcotics or explosives particles using either the ESP devices or the Automatic Trace collector module onto commercially available filters normally used with IMS machines. The trapped particles were then vaporised off the filter using a thermal desorber embedded directly into the SNIFFER detector submodule. Hence a new dedicated desorber was developed by AIRBUS and integrated during the second term of the project. The general integration scheme between the pre-concentrator and the detector device remained unchanged between the V1 and V2. The HMI was developed successfully in parallel to integration work and was first assessed in laboratory conditions and then further validated in field operation at Athens International Airport.

Within **WP3 “Sub-system for sampling, pre-concentration and pre-treatment of target analytes”**, starting from the key functional requirements for the SNIFFER devices and the physico-chemical properties of the SNIFFER target analytes, the overall functionality and the architecture of the SNIFFER sampling and pre-treatment system was developed at the end of the first project period.

The focus of the second half of the project was the new set of enhanced sampling technologies which proved highly relevant towards the end of the project. With the new desorbing design, collected traces of particulates matter now can fast and effectively desorb into the final OBP detection system. New fluidic components were included based on the pre-SNIFFER experiences with regards to high stickiness of explosive and drug residuals on the wall of sensors gas inlet. Heated glass based sensing inlet and desorbing outlet tubes were used to overcome these issues. Sampling material of the same dimensions and characteristics in addition facilitated matching measurements against different standard airport trace detection systems (e.g. Ionscan 400).

Sustaining the airports and car detection operational requirements a first (ESP) sampling development for version 2 devices was further functionalised towards a more hand-held based sampling system, with battery driven functionalities and COTS based light weight construction figures. The two WP3 device groups developed during the second period of the project, together with the odour binding detection system, demonstrated their performance at Athens airport very successfully.

**WP4 “Sensor Sub-System & Array Engineering”** initially included the development of a first version of the sensor subsystem and the following activities:

- The hardware integration of the biosensors
- The multi-parametric training of the biosensors to optimise the recognition potential from the sensor array
- The integration of software and hardware into the Sensor Sub-System.



These developments of the VA sensor subsystem were achieved at project midterm and during the second period of the project, a version V2 of the sensor sub-system was built within WP4. In this second version, minor changes were made respect to V1, to address some issues that arose during V1 characterisation. These issues compromised the success of the project by reducing considerably the limit of detection of the sensor system. The major changes carried out were:

- The sample inlet path was shortened by removing the pre-concentrator from the system;
- Glass tube was used for the inlet path instead of stainless steel;
- An heater and a temperature controller were added to the inlet line to keep it at 100 °C
- USB communication protocol was develop to implement a user interface based on the tablet technology.

Other modifications were carried out within the directly related WP7 that contributed to improve the sensor sub-system performance:

- The modularity of the sensor chamber was increased;
- Corruption of SPI bus data between the sensor board and main board was solved by reducing the communication baud-rate between the main board and the sensor board and reducing the length of the cable to the minimum acceptable size.
- I2C communication problems between the sensor microcontroller and the main microcontroller of SNIFFER device were solved.

Finally, a USB communication protocol was developed to implement a tablet user interface developed by CEA.

**WP5 “Biosensor Development”** focused on the development of the sensor sub-module, involving the development of single microcantilever diamond sensors combining Ligand-Binding proteins selective coatings.

During the first 18 months period, the work had involved the development of diamond microcantilever transducers with optimum performances in terms of mechanical dynamic properties. Moreover chemical grafting procedures were developed and characterized in order to immobilize covalently the bioreceptors onto the diamond surface of the transducers. The sensors were integrated into a gas cell V1 specifically developed for the project’s needs. A sensor array including 8 sensors was designed. A strategy was developed to embed high S/N read out gauges onto the diamond sensor chips and to actuate all cantilevers together using a single piezo-actuation cell. The sensors are held in a gas cell specifically developed for the project’s needs. This cell has been interfaced with the acquisition electronic and was shown to be operational under laboratory conditions. The integration of 8 diamond cantilever sensors grafted successfully with different proteins and integrated into the gas sensor cell constitutes the V1 biosensor.

During the second term of the project, the diamond micro-cantilevers were developed further. In particular, the piezo resistive gauges integrated in the first version of micro-cantilevers features gauge factors that were too low with respect to the target S/N ratios expected to reach the necessary sensitivity of the sensors. Therefore a new cantilever structure was designed in collaboration between ESIEE-Paris and CEA so as to integrate polysilicon gauges into the diamond structures. The grafting procedures developed for V1 in order to immobilise the proteins over the diamond transducers surfaces were used again as they were found to be very successful in V1. But this time new proteins with better affinities with target narcotics and explosives were grafted on V2 cantilevers, following the development work on proteins in WP6, in particular by UNIPD, UNIMAN and GTP. In parallel to the work on sensors, a new gas sensor cell was designed based on the original development of the V1 cell. The performances of the V1 cell were acceptable but the major drawback was the fabrication cost associated with the complexity of the design and the resulting machining constrains. Hence efforts were spent on the V2 to simplify the design, without losing



performances, in order to cut off the fabrication cost. This was achieved successfully and the V2 version was produced with a reduction of fabrication cost by a factor 4 (based on the fabrication of three units). The new cell can accommodate 8 sensors in parallel as in version 1.

In **WP6 “Protein Engineering”** a first batch of OBPs was produced by UNIMAN. It was composed of four different OBPs (1PDOM from the wasp and F88W from the pig were characterised). Moreover, functional recombinant Major Urinary Proteins MUPs (rMUP 8 and rMUP 20) were expressed by UNIPD. These were implemented in the V1 prototype. Site directed mutations of MUPs have been designed using an *in silico* strategy. Four target genes have been synthesised. Scale-up procedures and large scale production of 3 OBPs and 2 MUPs were achieved in the first period of the project. For development of the V2 instrument, six wild type (WT) mosquito (*Anopheles gambiae*) OBPs, WT *Locusta migratoria* OBP (LmigOBP1), WTs GOBP2 and POBP1 from *Bombyx mori*, were expressed and their binding affinities towards the target analytes determined. Three V1 OBPs produced by GTP, WT pigOBP1, mutant PigF88W, and insect wasp 1PDOM, were included. The above OBPs were screened for their binding properties towards V2 target explosives plus some other potential explosives and their derivatives, and they were also screened towards V2 drug targets. In addition, 5 new MUP proteins were synthesised and characterised against explosives and drug. Scale up of production of OBPs and MUPs was carried out by partner GTP. This was not without problems, but for several OBPs quantities between 100 mg to gram quantities were successfully achieved. Selected proteins were then incorporated into the V2 device to produce two types of sensor arrays – one targeted to explosives and the other to drugs.

**WP7 “Self-diagnostic capabilities”** was mainly concerned with the electrical interface and the acquisition of the resonance frequency of each sensor of the array. It also checked that the sensor array was working in the best possible conditions by mean of self-diagnostic and calibration processes. During this first part of the project an electronic architecture had been designed that answers the three main requirements of the work package: readout, calibration and self-diagnostic.

During the first phase an important design effort was made to design an analogue front-end that is both highly sensitive and low noise. The electronic prototype that had been fabricated was able to extract a signal in the  $\mu\text{V}$  range from the 4 micro-cantilever sensors and extract the resonance frequency of each cantilever from it. Results showed that resonance frequency (typically within the range 50kHz – 80kHz) can be obtained with a minimum 10Hz standard deviation under stable conditions. This electronic prototype was fully integrated with the gas analysis chamber developed in parallel in the project. All elements of the signal processing chain had been chosen to that they would evolve in the second part of the project toward auto-calibration and self-diagnostic. At the same time, equipment had been developed using virtual instruments to serve as a reference characterization bench for the sensors that would be produced during the project.

During the second period of the project, this electronic design had been scaled up to an array of 8 cantilever sensors and design correction and optimization had been carried. The V2 subsystem was answering all the requirement linked to the real-time exploitation of diamond resonant sensors resonance frequency. Important design efforts were made to design an analogue front-end that is both highly sensitive and low noise. The main challenge of this sub-system was tracking the resonance frequency of 8 cantilevers at a time while providing real-time diagnostic based on self-characterization data. The final system was able to compensate before each measurement for the drift in electrical and mechanical characteristic and provide the user alerts and information when the sensor is operating outside normal conditions. This was thanks to the evolution of the V1 prototype toward a V2 prototype based on digitally controlled analog-components; ie a mixed signal architecture. A real-time fitting algorithm was integrated into the digital signal processing in order to maximize the immunity of the resonance frequency detection to noise. Results showed that the



resonance frequency (typically within the range 20kHz to 150kHz can be continuously tracked with a minimum 1Hz standard deviation. This was in track with the objective set at the beginning of the project. This electronic prototype was fully integrated in the global SNIFFER system. Extensive communication had been done with the partners responsible for integration so that the mechanical and electrical characteristics of the subsystem were compatible with the fluidics constraints. The mechanical set-up of the electronic systems facilitated the access and replacement of the sensor cartridge. Eventually, innocuous chemical samples suitable for calibration of the sensor arrays were identified and recommended for in-field use.

**WP8 “Dissemination & Exploitation”**, during the first period of the project, was awaiting the first technical results and trials due at the end of the second project year. Thus it focused on the dialogue with users through the SNIFFER User Advisory Group to make sure that the use cases defined by WP1 and the specifications developed in the technical workpackages corresponded well with user needs. A first meeting was organised to present the project and obtain feedback on the documents produced by WP1. Special dissemination activities created awareness through the set-up of a dedicated website, the preparation of dedicated presentation material and the communication about the project to the scientific community.

The second period of the SNIFFER project was the opportunity to fully deploy activities. The project *dissemination* related activities focused on:

- Deepening the dialogue with users through the SNIFFER User Advisory Group. Continuous feedback and advice were sought through meetings and events.
- Further raising awareness through the updates of the dedicated public website, participation and talks in targeted conferences, seminars, symposiums and public events (supported by the dedicated presentation and communication material). The reached audience was not only the scientific community but it also included end-users and international / European institutions interested in border security issues.
- Strengthening contacts with FRONTEX and clustering with other FP7-SECURITY related projects (e.g. DOGGIES, SNOOPY, SNIFFLES, HANDHOLD) through the representation of SNIFFER at their event (e.g. European Day for Border Guard in Varsaw in May 2014) and their invitation to SNIFFER events.
- The organisation of 2 public events, at project year 2 (May 2014) and project end (May 2015), focusing on the presentation of SNIFFER results to a broad range of stakeholders. The first public event had a strong dog handler colour whereas the second public event focused on the variety of potential end-users if the SNIFFER devices.

During the second period of the project, *exploitation* activities were also fully deployed. The exploitation plan was fully established and further updated and improved up to project end while better visibility on project results was made possible with time. SNIFFER related patent, publications, potential commercial products and other dissemination activities are listed in the corresponding document.

In **WP9 “Project Management”** the SNIFFER Project Office, provided by ARTTIC, first supported the set-up of the different project management tools and processes whilst CEA-LIST was focusing on the strategic and contractual lead of the project. The internal project website was launched at project start and was extensively used by the partners. Dedicated deliverable review processes involve the project partners in ensuring the technical excellence and the usability of the SNIFFER project deliverables.

During the second period of the project, the SNIFFER Project Management Office continued to support the set-up of the different project management tools and processes. Project management



tools established at the beginning of the project were kept available to all project partners on the project private website throughout the second half of the project. A risk register was regularly updated throughout the project. These daily actions were carried out until the end of the project and proved successful in ensuring effective day-to-day operational project management. The SNIFFER consortium's General Assembly met face-to-face every 6 months as scheduled, and the Executive Board met remotely every month. Specific project management issues were discussed, especially that leading to Grant Agreement amendments such as the refining of project scope and project extension, as well as changes in the consortium.

## 2.2 Project objectives for the period

The below section details the objectives and the corresponding results emphasizing the second work period.

### *WP1 Security Usage cases, metrics and validation, societal and ethical aspects*

- Provide extensive definitions, user requirements and desired specifications for the final proposed artificial sniffer devices (DL111 & DL121)
- Define the methodology for measuring and validating the results from the operational point of view (DL111 & DL121)
- Integrate findings and requirements with WP2 and plan the application demonstrator (DL122)
- Address Societal and Ethical Aspects of such devices and its use (DL111, DL121 & DL131)

They were all achieved as shown in the corresponding deliverables, especially those mentioned into brackets.

### *WP2 SNIFFER Device Integration*

- V1 system design completed. All subsystem components (sampling, preconcentration and detection) designed, assembled, tested and ready for integration. V1 Integration work initiated.
- V2 submodules completed and integrated

Results for both objectives are specifically shown in DL 242 and DL 262.

### *WP3 Sub-system for sampling, pre-concentration and pre-treatment of target analytes*

- MEMS-based vapour preconcentrators available for integration into V1/2 SNIFFER systems.
- Electrostatic particle precipitators (ESP) modules available for integration into V1/2 SNIFFER systems.

### *WP4 Sensor Sub-System & Array Engineering*

- Define of the functional specifications of the sensor sub-module prototype (v1 and v2)
- Define of the architecture of the sensor sub-module prototype (v1 and v2): Major changes carried out with respect to v1 to address problems encountered for v2 to produce a robust v2 system.
- Low level electronic and firmware interface and selection of microcontroller for low-level operations: The same microcontroller technology adopted for v1 was used. Minor variations in the firmware were introduced to control the sample inlet temperature and to implement a touch screen display (mounted on the sensor device) and a tablet user interface.
- Selection of pneumatics between sensor array and the other sub-modules
- A neural network strategy based on a radial basis function architecture for programming into a microcontroller environment: No variation with respect to V1

All these objectives were achieved.

### *WP5 Biosensor Development*

- Immobilise OBPs and MUPs onto diamond surfaces + transfer immobilisation protocols onto sensor active surfaces
- Optimise diamond microcantilever transducers in terms and sensor actuation and read out
- Validate the whole sensor principle



- V2 of microcantilever including new read-out gauges and structure (DL522)
- V2 of biosensors including the new proteins developed throughout the project and design and fabrication of new gas measurement cell (DL523)

#### *WP6 Protein Engineering*

- Selection of proteins. Tailoring of protein recognition site conformation for selected analytes through selective mutagenesis (V1 and V2 proteins): Achieved, New OBP and MUP proteins were developed during the second part of the project targeting specific analytes.
- Production of proteins by bacterial expression systems and the industrial scale-up of production of proteins: Achieved for both OBPs and MUPs, some proteins were problematic to scale up production.
- Immobilisation of proteins on to diamond coated cantilever substrates: Achieved, robust coverage of proteins on diamond surfaces was obtained.
- Testing of sensitivity and selectivity of immobilised proteins to selected analytes: Achieved, a range of analytes were tested.
- Validation of stability, repeatability and influence of environmental factors: Achieved, data indicate good stability over time.

#### *WP7 Self-Diagnostic Capabilities*

- Define a high sensitivity and agile electronic mixed-signal front end architecture specifically for the diamond microcantilever transducers
- Design, build and verify an electronic board prototype able to extract the resonance frequencies of multiple transducers around the gas cell developed within WP5
- Develop a reference instrument based on virtual instrumentation to serve as a reference characterization platform for the sensor manufactured within the project
- Implement innovative calibration and characterization process to provide reliable and informative operation of the array of diamond resonant sensors.
- Develop and implement digital signal processing tools for pushing frequency detection of resonant diamond cantilever to lab instrumentation level within a mobile, autonomous and field efficient device.
- Develop and implement digital signal processing tools for pushing frequency detection of resonant diamond cantilever to lab instrumentation level within a mobile, autonomous and field efficient device.
- Sensor Calibration Methods: Chemicals selected for in-field sensor calibration

All the above listed objectives were achieved.

#### *WP8 Dissemination & Exploitation*

- Promote SNIFFER project and European RTD efforts and make research results available to a larger community: Achieved, through the set-up of the communication tools – public website, leaflet, poster – and presentation of the SNIFFER Project at various external conferences
- Follow up decisions from advisors: Achieved, Regular and valuable UAG and various end-users input all project long.
- Prepare exploitation of SNIFFER results: Achieved through the establishment of an exploitation plan, updated along with project results
- Develop links with third parties: Achieved, High level of interest from third parties and take-up of the results in a H2020 follow-up project

#### *WP9 Project Management*

- Ensuring the strategic, financial and contractual management of the consortium: Achieved

All contractual documents were established, shared and implemented all project long. Needed amendments process was successfully covered by the project management team. Strategic issues and changes in the consortium were dealt with for efficient project management and resulted in a successfully managed project.



Management procedures and quality plan were defined and agreed upon at project start. They were implemented all project long. Project management tools were easily accessible and available for all in their latest updates on the project private website. A risk register has been established and regularly updated according to the life of the project.

- Setting up the management infrastructure: At project start, the SNIFFER consortium set up its General Assembly, which met twice a year for a consortium meeting, and its Executive Board, which met every month for a project management follow up teleconference. Each project management issue was dealt with at the appropriate project level, with respect to the project hierarchy.



## 3 Description of the main S&T results / Foreground

### 3.1 WP1 Security Usage Cases, Metrics & Validation, Societal & Ethical Aspects

WP number	1		Start date or starting event:	M1	
WP title	Security Usage Cases, Metrics & Validation, Societal & Ethical Aspects				
Activity type	RTD				
Participant ID	3	7	12	13	2
Part. short name	ST(SI) <sup>2</sup>	CSSC	3DSA	INP	UNIMAN
PM total (for 60M)	0	14	30	7	0.80
PM spent in period 1	0	5,50	15,10	4,32	N/A
PM spent in period 2	4	0	12,90	<i>missing</i>	0.40
PM spent total	4	5,50	28	<i>missing</i>	0.40

#### Overview of the progress of the work during the period

The objective of WP1 was to provide the necessary Usage Case (UCs) definitions in 3 distinct UCs (according to the amendment to the original DoW).

Indeed, four devices were initially envisioned to cover the needs of these UCs although the core modules in all devices will be the same:

1. **SNIFFER BODY SCANNER:** A portable scanner able to identify illegal and/or dangerous substances (like drugs and explosives) by detecting their explicit odour. The first envisaged device is a portable (ideally handheld) body scanner that will be used to scan people. The procedure is almost identical to the common procedure of testing a person with a metal detector. That is why ideally this scanner should be integrated into a metal body-scanner, standard equipment in many security sensitive environments (critical infrastructures, airports, etc.).
2. **SNIFFER PORTABLE SCANNER:** A device that will be able to identify illegal and/or dangerous substances (like drugs and explosives) by detecting their explicit odour. The envisaged device is a portable (ideally handheld) scanner that will be used to scan open luggage and cars (interior).
3. **SNIFFER PORTABLE CONTAINER SCANNER:** A SNIFFER device will be able to detect clandestine and illegal immigrants, hiding in a container or a truck by detecting their explicit odour. The envisaged device is a medium size scanner that will be used to scan containers.
4. **SNIFFER PORTAL SCANNER:** A SNIFFER device will be able to identify illegal and/or dangerous substances (like drugs and explosives) by detecting their explicit odour. The envisaged device is a portal size scanner that will be used to scan luggage on a running belt.

Following the project mid-term review, the original list of Usage Cases has been reduced to three concrete usage cases:

- UC3: controlling suspicious open luggage and cars with small portable scanning device (V1)
- UC4: controlling suspicious open luggage and cars with small portable scanning device (V2)
- UC6: systematically scanning closed luggage on a running luggage belt for illegal substances and



explosives (V2). The above corresponding devices covered all described UCs and served as a basis for WP2 and the rest of the technical development.

### Work progress in each task

#### *Task 1.1 & 1.2 – UCs for V1 and V2 technologies*

CSSC's initial contribution consisted in a preliminary analysis of the ethical and societal implications generated by the perspective deployment of SNIFFER devices for security checks at borders and at other critical installations. It also consisted in the elaboration of recommendations for field trials. The above preliminary analysis focused on 4 devices for 6 UCs: UC1 & UC2, UC3 & UC4, UC5, UC6. Unfortunately, CSSC went through voluntary liquidation early 2014. However, the burden of ethics was lessened following the first review meeting as the scope of the project was re-focused.

Moreover, the French ministry of interior as an end-user participated in the elaboration of the UCs. The ST(SI)<sup>2</sup> with the support of CREL from the national police made a field inquiry in the airports with the different bodies in charge of security: civil aviation, specialized gendarmerie, customs, border police, ... The CREL made a great job in the technical definition of the devices that could be developed. It managed to get some specifications from various end-users who shared main practical constraints in the field of detection of dangerous substances on the borders. The CREL prepared a methodology for the future laboratory tests and submitted it to the consortium.

INP's contribution consisted of forming a workgroup with relevant Police Departments such as: The Operations Department, The Department of Investigations & Intelligence, and The Police Legal Adviser. It collected information and requirements from end-user's in the Police Department regarding various aspects such as legal requirements, operational requirements, ethical requirements. It also contributed to the planning and implementation of trials (field trials and lab trials of TTS machine): testing requirements, sites for testing, and legal requirements.

After the 1<sup>st</sup> Project Review and following recommendations and discussions with the PO and reviewers, it was decided to limit 3 Use Cases as stated above. UC3 lead to a lab trial, which allowed the SNIFFER users to assess under lab conditions the dedicated devices delivered by WP2. UC4 and UC6 in turn lead to field trials, which allowed the SNIFFER users to assess under close to real life conditions the dedicated devices delivered by WP2. Each Usage Case task followed the same "definition - trial - assessment" cycle, lasting 24 months and starting at T0 (T0 =Month 1 for the first usage case and T0 = 13 for the second usage case):

1. Define the usage case from the different perspectives (T0 - T0 + 4 months)
2. Regularly stay in contact with users to present progress, obtain additional inputs (information, samples, etc.) and plan deployment of trial (T0 + 9 months; T0 + 26 months; T0 + 37 months)
3. Set up the trial: Receive devices, install, train users, setup demonstration (T0 + months 18 to 25) (T0 +months 25 to 37)
4. Run the field trial, assess and write assessment report (T0 + month 40) with support from technical partners in WP2.

The target compounds for the lab tests and field trials will be limited to the following:

- Explosives: 2,4 DNT, 2,6 DNT, and TNT are required. RDX and PETN are the priorities. HMX, EGDN and ammonium nitrate are optional for V1 and will be included for V2.
- Drugs: Cocaine, Ephedrine, Heroin, THC, Ecstasy (MDMA)
- Other: Atropine (to show the ability to detect other substances)

These tasks were completed with the submission and acceptance of the revised deliverables that limited the use cases to 3 and described a more viable approach to the project.



### *Task 1.3 – Collection of ethical approvals*

CSSC initially assisted WP1 leader in the drafting and reviewing of a detailed information package to be sent to National Data Protection Authorities that describes the project, its main mid-term results and presents the procedure to be followed to organise and implement field trials. In particular, CSSC contributed to the document by presenting an overview of the EU privacy and data protection legal framework and reaffirming the ethical standards to be followed during the field trials that will be held in Greece and Israel. Unfortunately, CSSC went through voluntary liquidation early 2014. However, the burden of ethics was lessened following the first review meeting as the scope of the project was re-focused.

Deliverable 131 “Report on National Ethical Approvals” was rewritten and resubmitted on Dec 2013. That deliverable covered the EU privacy and Data Protection Legal Framework and presented the outline for the Trials Procedure Protocols. 3DSA was also appointed (as the WP leader) to investigate the required actions and necessary authorisations for the implementation of the final field trials according to the trial protocols that would be developed.

To simplify things and avoid time consuming authorisations, the consortium decided to restrict the field trials to the members of the consortium and a few stakeholders (Greek Police, Athens International Airport authorities). This decision eliminated the need for authorisations. Nevertheless 3DSA contacted the Hellenic Data Protection Agency (HDPA) and after some communications and presenting of the case, HDPA ruled that authorisation was not necessary and field trials could take place as planned.

### *Task 1.4 - Independent Ethics Advisor*

Prof. Timothy Stibbs from the University of Manchester, an experienced ethics specialist, oversaw with impartiality the ethical concerns raised by this research involving human beings.

Planning of experiments involving public venues and reports associated were screened by the ethical advisor. Mid-term by the Ethics Advisor were submitted to the EC/REA with the Periodic Report. The same process will be followed concerning final reports.

#### **Significant results:**

- Preliminary ethical and fundamental rights analysis of the UCs together with recommendations for field trials
- Suggestions for modifications and adjustments to suit the real operational requirements and legal limitations
- Establishing lab trials prior to field tests
- Set up and organisation of the Final Field Trials at Athens International Airport
- Successful delivery of all WP DLs, specifically resubmission of DL111 and DL 121, as well as DL 131 according to recommendations from the 1<sup>st</sup> review report (Dec. 2013)

Description of problem / deviation	Suggested / undertaken corrective action	Impact on other WPs, budget/PM plan or work plan
Lack of information concerning the prototypes to be tested at V1 field test scheduled for UC1 and UC3, lack of reports (technical + operational + safety)	Requirements have been sent – ongoing reply Lab/field trials technical meetings (June and September)	Delayed planning of field trials.

	2013)	
Not able to find narcotics at the Athens International Airport Trials (Airports are not interested in these)	Trials took place with explosives. Samples were more than enough to test the protocols and equipment.	None as extensive lab trials have been ran with the above list of narcotics.



## 3.2 WP 2 SNIFFER Device Integration

<b>WP number</b>	2		<b>Start date or starting event:</b>	M1	
<b>WP title</b>	SNIFFER Device Integration				
<b>Activity type</b>	RTD				
<b>Participant ID</b>	2	5	1	11	
<b>Part. short name</b>	UNIMAN	AIRBUS	CEA	TTS	
<b>PM total (for 60M)</b>	30	15,50	17,40	3	
<b>PM spent in period 1</b>	9	10	N/A	N/A	
<b>PM spent in period 2</b>	22	2,16	17,40	missing	
<b>PM spent total</b>	31	12,16	17,40	missing	

### Overview of the progress of the work during the period

- WP2 achievements to M4 was related to detailed specification and definition of functionalities and system design architecture for SNIFFER devices 1 and 2 focusing on body scanning devices and searching technology at open luggage and cars.
- Up to M18 a more detailed specification and description is supplied in device 2, V2 developments. Deliverables 5-6 were focusing on UCs 4-6, which deal with container inspection and luggage belt screening in airports.

### Work progress in each task

System design in sampling, preconcentration and detection device developments (V1+V2) are finished at the end of reporting period. All related and relevant mechanical and electronic sub-elements are constructed, integrated and tested already.

*Task 2.1 – Develop D1 – SNIFFER V1 of an enhanced metal body-scanner with “artificial sniffer” functions*

This task was cancelled with the amendment of the SNIFFER Description of Work at mid-project.

*Task 2.2 – Develop D2 - SNIFFER V2 of enhanced metal body scanner with ‘artificial sniffer’ functions*

This task was cancelled with the amendment of the SNIFFER Description of Work at mid-project.

*Task 2.3 – Develop D3 - SNIFFER V1 of a light portable device for open luggage and cars*

D3 device architecture was defined in the same way as that developed for D1. The main difference between D1 and D3 is in the sampling flows. The consortium selected stainless steel tubing and valves for this prototype. The electronic board for D3 was developed, built and tested. It is the same as for D1. In fact, it allowed selecting the right flows required by this different application. USB, SPI, I2C, Ethernet communications system were integrated the system for communicating with the user and the different submodules.

Keypad and LCD were also implemented as an easy user interface.

*Task 2.4 – Develop D4 - SNIFFER V2 of a light portable device for open luggage and cars*



D4 detector sub-module was modified respect to the V1 version to improve sensor performance.

The device was conceived as an independent sub-module that can be controlled and run by an end-user. When sample detection needs to be performed, the user selects start measurement on the touch screen display and the following procedure is started:

1. The Main microcontroller sends a signal to the thermo-desorber/pre-concentrator communicating that it is ready for starting a measurement;
2. The thermo-desorber/pre-concentrator notifies the detector that it is ready for desorbing the sample and then wait for the start signal from the detector;
3. The detector sucks air at 100 sccm from the inlet for 10s to record the baseline and then sends the “start desorption” signal to the desorber/ pre-concentrator;
4. The sampler/ pre-concentrator starts the desorption while the detector is sucking at 100 scmm. Hence the vaporised sample is delivered to the biosensor. The proteins bind the analyte molecules changing their shape. The transducer converts the binding effects into a variation of the sensor electronic signal read. This step last for 40 sec;
5. The thermo-desorber/pre-concentrator communicates to the master the end of the desorption;
6. The detector sucks air from the inlet to clean the sensor;
7. The sensor data are immediately processed by the radial basis neuronal network software and the results are shown on the touch screen display or a HMI.

The first v2 ESP sampling development (DL321-324) worked effectively and fulfilled all needed requirements. Following several trials it was decided that for Athens Airport field tests a new more practicable device configuration should take place. A handheld sampling device D4 based on corona discharge and electrostatic precipitation of particles targeting sampling of narcotics or explosives with extremely low vapour pressures was developed and delivered on time. The physical principles behind this technology and the technical details of this device can be found in DL242. The device was assessed in laboratory conditions and then in Athens International Airport to sample illicit particles e.g. in passenger open luggage. This sampling device D4 is also suitable for searching cars in particular for particles of narcotics and eventually explosives.

The new configuration in D4 and D6 device operations also required also new thermos-desorption figures which are described more in detail under DL321-324. A new thermos desorption device was designed and configured with regard to the needed operational procedures and figures.

#### *Task 2.5 – Develop D5 - SNIFFER portable device for containers*

This task was cancelled with the amendment of the SNIFFER Description of Work at mid-project.

NB: A macro preconcentrator that can sample large volumes of air was developed by partners ARMINES and EPFL and interfaced to the SNIFFER detector submodule. Such device is particularly suited for search of volatile odorants such as tobacco odours in containers. For obvious practical reasons the device was tested in laboratory conditions for the detection and identification of tobacco.

#### *Task 2.6 – Develop D6 - SNIFFER Monitoring Portal for a luggage conveyor belt*

While D4 needs to scan open luggage, D6 aims at controlling close luggage on a conveyor belt. Hence, they have been designed as two devices having the same sensor array but with a different sampler.

An automatic Trace Collector module was developed by partner TTS for searching particles of narcotics and explosives in closed luggage. The module was tested and validated in Athens International Airport in combination with the other SNIFFER submodules successfully. Although the



device was not directly tested in conveyor belt configuration the technology could easily be adapted to such application *via* reasonably low further engineering efforts. Similar technology has already been demonstrated by partner TTS in conveyor belt configuration. Indeed, it was decided that D6, being only a technology demonstrator did not require the use of a conveyor belt. As a consequence, the device was to be built without one. The requirements for the machine were to be able to accept the largest of hand-carried baggage typical of air travel and extract traces of materials on and within the bag for purposes of Electronic Trace Detection (ETD) methods to be applied to the traces. TTS took the lead for the design and production of this device as it is the world recognized leader of this technology. The design included application of TTS's technology for using aerodynamic flows to accomplish the extraction and collection of the traces. A chamber was devised, somewhat larger than the largest hand-carried baggage in which the aerodynamic devices were placed. The frame and supporting controls and air handling equipment was procured and the device assembled. The device was tested using a number of powders as simulants as well as laptops and few bags to determine the collection capability. The device passed these tests even without optimization. After a few months, the device was taken to the Israeli National Police for testing with live explosives and narcotics. It was fortunate that the test facility was near the INP canine facility and side by side testing was done. Troubles with the air providing devices prevented a full set of tests, but the those accomplished showed that the D6 device was capable of extracting materials even from within closed bags. In this series, the canines proved extremely reliable as well.

Subsequent testing at the Athens International Airport in Greece again proved that the D6 device was capable of extracting even small amounts of explosive materials from within bags which had been zipped close. The amount of materials extracted were sufficient in every case for the SNIFFER device to detect their presence.

#### *Task 2.7 – Final Consolidated Technology Report*

Between the last sampling V2 developments and at the beginning of the field trials at Athens Airport some issues with regard to maturity levels and operational behaviour were identified. Most of them could be solved and at some degree the outcome solution showing up better results as it were expected at the beginning.

*Several outcomes and problems encountered between our last V2 sampling-desorption elements could be discussed in following manner:*

#### Problems regarded to operational procedures

- Originally ESP sampler wasn't small and portable enough and needs power line interconnection, which shows up not very convenient and valuable for the field operation.
- Non testability of TTS luggage belt sampling system befor the field trials
- Different sampling pad sizes and materials from luggage belt and thermos desorption and ESP sampling devices

#### Problems encountered after laboratory trials

- Still contamination of glass inlet tube from desorption chamber
- Solenoid thermo-desorption system fails because of long term overheating and connectivity problems
- Heat stamp cables insulation were scratched after several ours operation time
- Interface needed from V1 sampling system wouldn't be appropriate for the new design

#### Problems with regard to the timeline

- Very short timeline between laboratory trials and field test



- New configuration of interface and controller occupies great amount of time

**Solutions:**

- Heating of inlet block and transfer-lines of sampling inlet
- Design and build new ESP handheld sampling device
- Thermo-desorption-heat protection screen with perforated plate for safe operation
- Refurbishing of heat stamp because the coating of cables were damaged
- Creative and intense communication between WP3 and TTS solving probably interface issues solved very successfully
- Creation of new desorption communication and control interface
- TTS designed new filter holder and also tested our envisaged filter pad materials to fit into the needs with regard to the different devices and usage cases.

**Significant results:**

Major system integration aspects between partners were gathered. A systematic device integration concept of functionalities and integration aspects could be delivered. Functionalities are aligned and systematic interaction in device integration is at a mature level for device integration 1 to 4.

All SNIFFER submodules V2 were delivered and finally integrated on time to be tested. Following laboratory validation, the SNIFFER detection module, the Automatic Trace Collector sampling module and the Electrostatic particle collector were tested together in the same environment in complementarity for the first time in Athens International Airport, in an operational environment: the airport luggage-check area. The SNIFFER results were benchmarked against an IMS (state-of-the-art in airports today). The sampling systems proved to be efficient in collecting particles hidden in back bags (validated by IMS first then SNIFFER): 100 % of hidden particles were detected and therate of identification was assessed to be around 60 %. Those results are very promising and were well received by end-users and stakeholders to whom they were presented at the subsequent SNIFFER workshop.

**Deviations:** None

### 3.3 WP 3 Sub-system for sampling, pre-concentration and pre-treatment of target analytes

<b>WP number</b>	3			<b>Start date or starting event:</b>	M1
<b>WP title</b>	Sub-system for sampling, pre-concentration and pre-treatment of target analytes				
<b>Activity type</b>	RTD				
<b>Participant ID</b>	4	5	6	11	
<b>Part. short name</b>	ARMINES	AIRBUS	EPFL	TTS	
<b>PM total (for 60M)</b>	26	17	36	16	
<b>PM spent in period 1</b>	15	10	15	8,75	
<b>PM spent in period 2</b>	9,03	10	18	<i>missing</i>	
<b>PM spent total</b>	24,03	20	33	<i>missing</i>	
<b>Overview of the progress of the work during the period</b>					
<ul style="list-style-type: none"> <li>• A particle sampling system using electrostatic precipitation (ESP) technique was developed. Sampling tests showed that the system is able to sample selectively allowing preconcentration of target substances.</li> <li>• The ESP approach will be further improved by modifying the sampling electrode (ceramic chip) by adding a conducting layer on top which allows electronic discharge when particles are sampled. This was done using aluminum deposition techniques. Tests will be carried out in the next weeks.</li> </ul>					
<b>Work progress in each task</b>					
<p>An electrostatic particle precipitator (ESP) sampler unit was initially developed at EADS (former AIRBUS) and integrated into a breadboard system. This breadboard setup allows the ESP sampler to be combined with various kinds of detectors to perform full-chain sampling-detection experiments. The sampler on its breadboard unit allows solid particle residue (drugs, explosives) to be extracted from suspect surfaces through hydrodynamic forces (vacuum cleaner). The sampling air stream is then guided through the ESP sampler where the sampled residue becomes electrically charged upon transit through a corona discharge. Depending on the polarity of the discharge needle, either highly electron (explosives) or highly proton-affine (drugs) substances become electrically charged and immobilized on a heatable ceramic chip. After the sampling step the chip with the collected substance is moved outside the particle sampler underneath the SNIFFER detector entrance port. This method of sample transfer minimizes the loss of target substance due to vapor condensation on internal walls of the SNIFFER device. At EADS successful full-chain sampling and detection tests have been performed with TNT (explosive) and Ephedrine (drug) particles and a number of common interfering substances (mineral dust, pollens, food stuff, medical drugs). As SNIFFER OBP-detectors had not yet been available, commercial metal oxide (MOX) or surface ionization (SI) detectors had been used in these preliminary experiments.</p> <p>A news V2 electrostatic particle precipitator (ESP) sampler unit was developed at Airbus with new handheld metrics and figures. This new setup allows the ESP sampler to be combined with various</p>					



kinds of detectors to perform full-chain sampling-detection experiments (IMS / OBP). The sampler unit allows solid particle residue (drugs, explosives) to be extracted from suspect surfaces through hydrodynamic forces (vacuum cleaner). The sampling air stream is then guided through the ESP sampler where the sampled residue becomes electrically charged upon transit through a corona discharge. Depending on the polarity of the discharge needle, either highly electron (explosives) or highly proton-affine (drugs) substances become electrically charged and immobilized on a heatable ceramic chip. After the sampling step the sampling pad with the collected substance is moved outside the particle sampler and feed into the new OBP-thermos-desorption device. This method of sample allows the transfer either from the ESP sampling system as well as from the luggage belt UC6 set up. At Athens Airport successful full-chain sampling and detection tests have been performed with different explosive particles and a number of common interfering substances. As SNIFFER OBP-detectors had not yet been tested under real environmental conditions all test was compared as well with IMS detection devices as well.

### **Handheld aspiration device for the final field tests at Athens airport V2**

A more flexible and more “hand held” based sampling system for the field test with regard to the use case at the Athens Airport was developed (V2) with mayor focus of operability with regard to the use cases.

Sustaining this needs and reducing cost and time we decide to use a commercial of the shelf (COTS) - based vacuum cleaner system in which the required ESP mechanical and electronically parts could be integrated.



*Overview of Handheld Sampler assembly, ESP with particle aspiration and air-sampling system*

### **ESP-Handheld Device (V2) - Structural Changes and New Features**

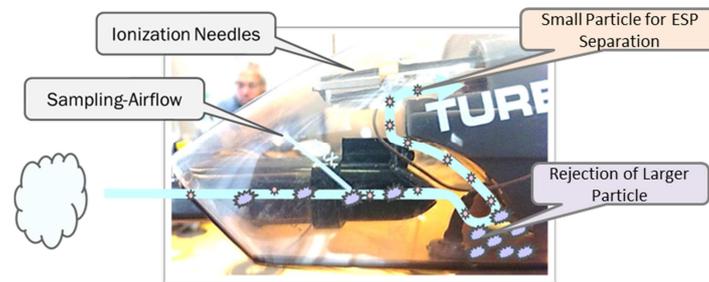
- Following field testing at AIA, the ESP-sampling module needs optimization to be suitable for the operational procedures needed for luggage and car inspections.
- Following lab test, V1 was already rearranged hand-held and working independently from the lab instrumentation (power supplies, measurement instrumentations...)
- Using a battery (NiMH 14,4V) driven vacuum blower configuration to subtract the particles form surfaces of investigations (e.g. seats, luggage, cloth..)
- New design to be based on COTS-based instrumentation including all SNIFFER new developments for a more comfortable usage in conjunction with or operational requirements

### **Inertial Impactor practical Sniffer-Handheld-ESP-Sampling-Design**

In our new handheld based ESP sampling approach the introduction of the impactor technology shows up in a higher sampling efficiency and leaving out most of the unwanted bulkier particles originated from dirt and other air impurities.

To our knowledge this is the first time that the impactor and ESP technology were integrated into a particle sampling system for security screening approaches.

The figure below sketches the sampling- air pathway until it reaches the ESP separation chamber. It could be seen that the larger sized particles (purple) are passed away before charging, only the smaller sized one can reach the area of ionization needles.



*Pre-separation impactor-chamber for extraction of bulky and heavier particle fractions from air streams*

### *Task 3.1 – Development of the testing bench for DNT generation and TPD studies*

ARMINES's contribution mainly focuses on the implementation of a test bench and the optimization of adsorbent materials to fill up the preconcentrator devices whereas EPFL efforts were centred on the microfabrication of silicon and plastic preconcentrators.

Therefore the test bench implemented in project is basically composed of a gas generator from Owlstone Nanotech using permeation tubes, mass flow-meters and a climatic chamber containing the preconcentrator device and the detection system. The climatic chamber is maintained at 40°C to avoid any condensation between the gas preconcentrator and the detector. Different permeation tubes (TNT, DNT, others interfering gases) were purchased and calibrated by weight loss at a given temperature for estimating their permeation rates. According to the vapor pressures, the maximum concentrations which can be obtained at 25°C are 8 ppb for TNT and 482 ppb for DNT.

Meanwhile Four adsorbent materials: a carbon nano-powder, TENAX powder, porous silicon and Carboxpack B were characterized using TPD (Temperature Programmed Desorption) and BET (Brunauer, Emmett and Teller) method for evaluating their affinity toward target gases in the SNIFFER project (explosives and drugs) and also for estimating their desorption temperature. Globally these experiments show high desorption temperature of explosives (desorption between 350 and 400°C) on carbon nano-powder and on TENAX while porous silicon and on carbopack B show low desorption temperature (down to 250°C) of DNT compounds. For this purpose, carbopack B and porous silicon were chosen for the preconcentration of explosives and Tenax is used for the preconcentration of drugs derivatives such as acetophenone.

Three versions of gas preconcentrators were developed through SNIFFER. At the beginning of the project a micro preconcentrator based on silicon technology was implemented for fulfilling the requirement of small size device for the version one of Sniffer handheld detector. However, our partners suggested the development of macro-preconcentrator for large volume sampling for the user case of cargos inspection. Following the development of the macro-preconcentrator, another small size device based on plastic technology was implemented in order to ally small size and large volume sampling since this device possesses adjustable inlet/outlet allowing high flow rates.

### *Silicon micro gas preconcentrators*

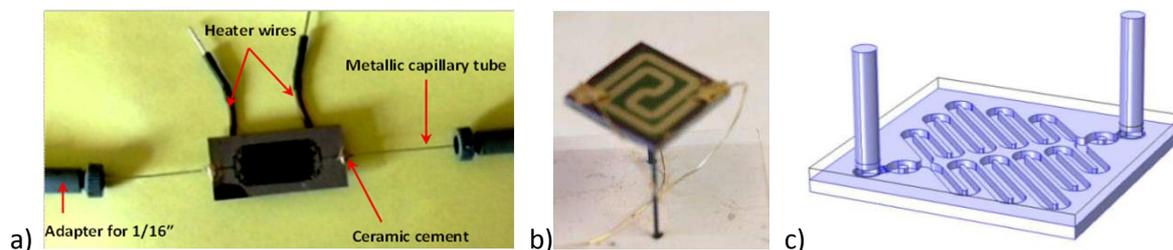
Two different micro-preconcentrators have been developed during the sniffer project. The first ones are based on silicon technology. They are made with a succession of photolithography and DRIE steps and bonded with a glass cover by anodic bonding. The design of channels within the device and the

inlet/outlet vary according to the adsorbent material and the gas to be detected. Some have a preconcentration chamber for receiving a large amount of adsorbent and some are made with a main channel for maximizing the probability for a gas to be detected. Silicon micro gas preconcentrators include a platinum heater at the backside deposited either by lift-off process or by screen printing. Prior the heater deposition, the backside of the micro-devices is coating with 150nm of Si<sub>3</sub>N<sub>4</sub> in to avoid any electrical breakthrough during desorption over 300°C.

Meanwhile, the use of porous silicon as an adsorbent or/and a binding surface was also studied. Indeed, pores were grown within channels and in the preconcentration by anodic etching in HF bath. Besides enhancing the preconcentration performance of the device, the pores allow easy desorption of target gases by lowering their desorption temperatures.

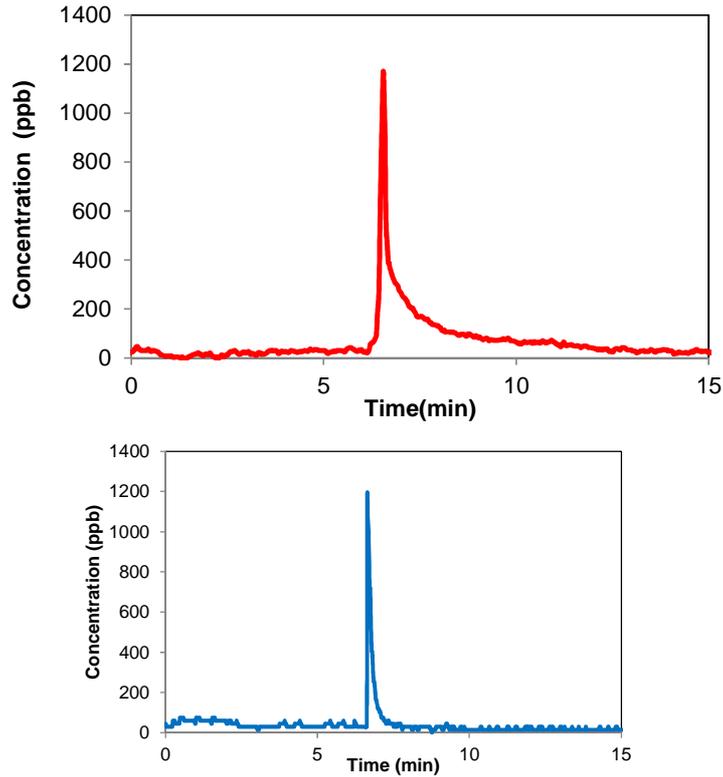
Long used in SPME (solid Phase Micro-Extraction) fibers for gas chromatography, Carboxen B has been chosen as adsorbent for also its affinity towards target gases in SNIFFER context such as DNT, BTX (Benzene, Toluene, Xylene) and Acetophenone. Nevertheless, others adsorbent like carbon nanopowders and Tenax were considered. Adsorbents were inserted in micro-devices in granular form before the sealing of glass cover for preserving its interstitial porosity and thus its high surface to volume ratio.

After the adsorbent deposition, the micro gas preconcentrator is then decorated with micro-capillaries as fluidic connections. In the first proposed designs the fluidic capillaries are laterally connected to the device whereas the new design of micro gas preconcentrator offer the possibility of using large capillaries since inlet/outlet are directly dug in the glass cover (Figure 1). The devices allow flow rates up to 20L/h with a pressure drop down to 2bars and could reach a heating rate of 60°C/s thanks to their small thermal mass.

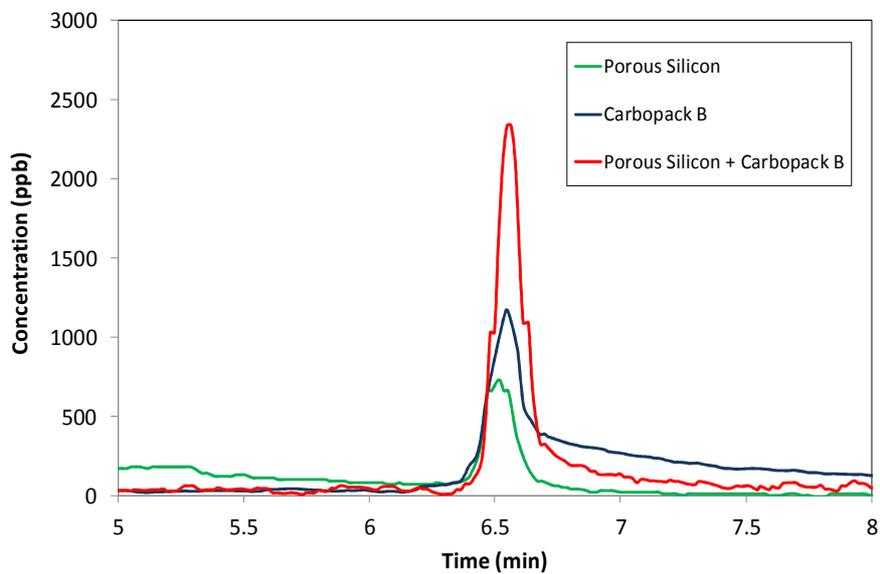


*Images of a) First design of silicon preconcentrator at the final stage with lateral fluidic connections and heating element b) Second design of silicon device platinum heater at the backside c) 3D view of the second design with fluidic connections on the glass cover.*

In overall, the combination of the new preconcentrator design with porous silicon and carbopack B shows the highest preconcentration performances with a complete desorption in comparison with the old design and/or without porous silicon when tested at the same experimental conditions.



*DNT peak after an adsorption of 200ppb@5min at 20L/h and a desorption at 250°C with a flow of 10L/h obtained with a old design (left) and new design (right) of gas preconcentrators with filled 1mg of carbopack B.*



*Comparison between carbopack and Porous silicon tested separately and in combination after adsorption of 200ppb@5min of DNT at 20L/h and a desorption at 250°C with a flow of 10L/min.*

**Macro-preconcentrators**

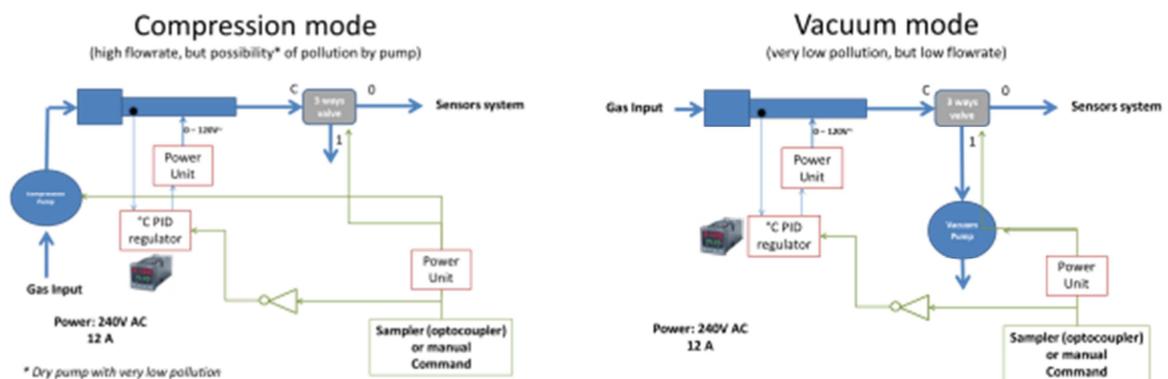
The macro-preconcentrator is basically made with a metallic tube filled with carbopack B and



wrapped up with a heating element. The macro-preconcentration is incorporated with pump for defining flows, a fan as a cooler and PCBs for obtaining an autonomous operating system. A photograph of the macro-preconcentrator system is shown in Figure 4. The submodule can work either in compression mode or vacuum mode (Figure 5). In the compression mode the pump is upstream the pre-concentrator; this mode allows the higher flowrates up to 18 L/min, but there is a risk of contamination by the pump (even if the pump is a “low pollution” dry piston pump). In the vacuum mode the pump is downstream the preconcentrator and then sucks the gas in. This mode eliminates any risk of contamination but allows lower flowrate (8 L/min). The current system is delivered with the vacuum mode. Tubing modifications could allow switching easily to the compression mode.

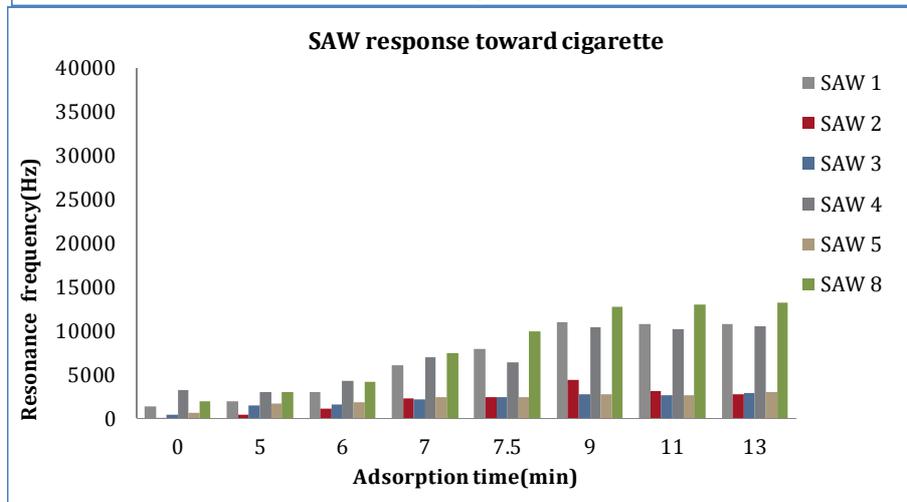
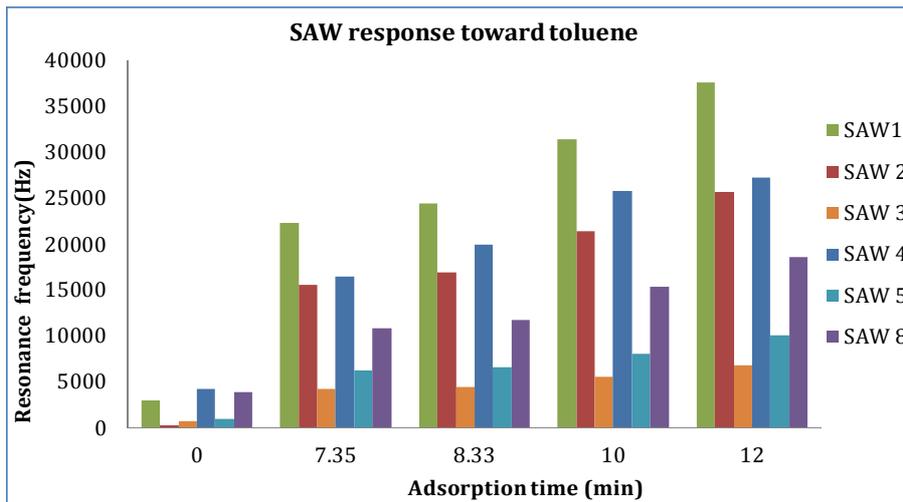


Photographs of the macro pre-concentrator

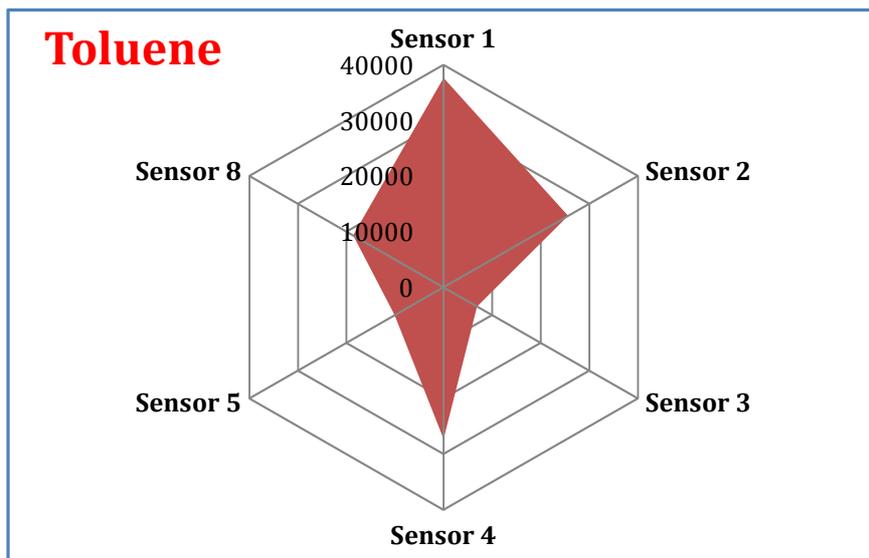


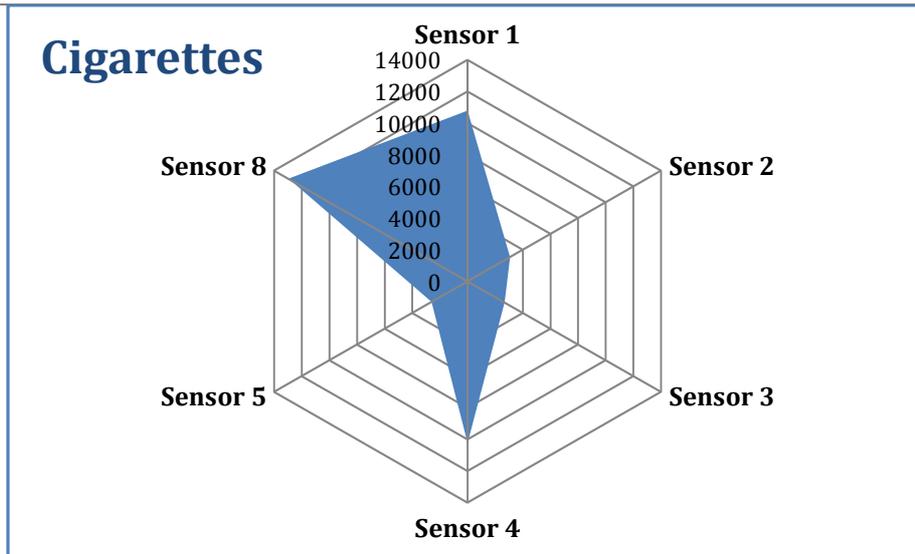
Two modes of operation of the macro-preconcentrator submodule. Right: Compression mode, Left: vacuum mode

The macro-preconcentrator device was tested by our CEA partners under toluene and cigarettes. These results have shown an increase of the sensitivity of SAW sensors array when operating with a preconcentration at different adsorption time (figure 6). Besides, the pattern recognition algorithm developed in Sniffer project allows separating aromatic compounds such as toluene from cigarettes which is one of the main interfering compounds in context of explosives and drugs detection (figure 7).



Response of an array of SAW sensors toward toluene (left) and (cigarettes (right) adsorbed at 450mL/min and desorbed at 250°C with a flow of 350mL/min.

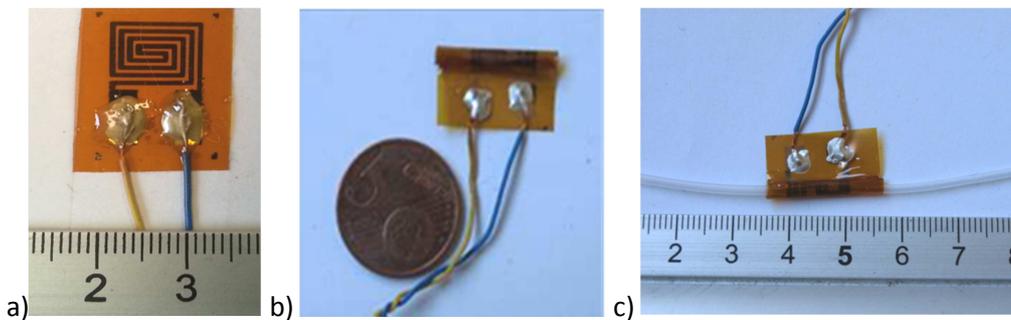




*Fingerprint of toluene (left) and cigarettes (right) with an array of 8 SAW sensors for adsorption time of 10min.*

**Foil gas preconcentrators**

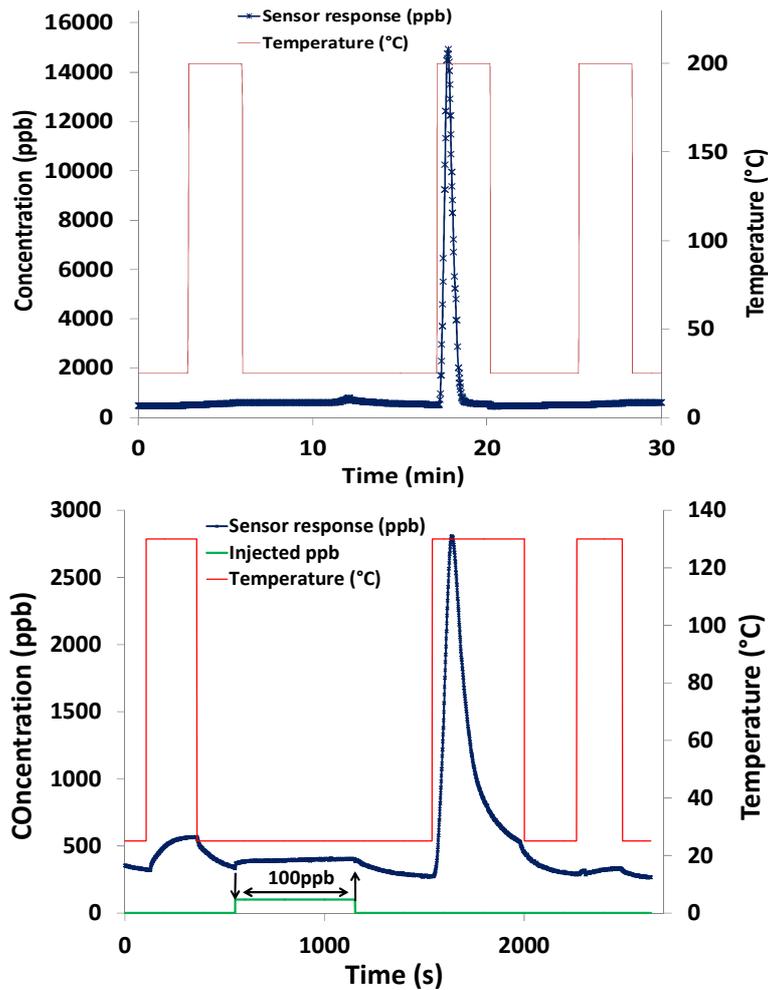
The second small devices developed in this project were based on plastic foil technology. These foil gas preconcentrators are made by rolling up a gold micro-hotplate printed on foil substrate before filling it with the desired adsorbent. This concept allows easy fabrication of gas preconcentrators with adjustable inlet/outlet diameter depending on the adsorbent to insert and the flow rate to be imposed. Two chosen adsorbents (carbopack B and Tenax TA) were inserted within the rolled hotplates in granular and blocked with glass wool. The fluidic and electrical connections are then sealed with ceramic cement and conductive glue, respectively (Figure 8). Foil devices are usable at flow rate up to 100L/h and at temperature up to 350°C.



*Implementation process of the foil gas preconcentrator: images of a) a printed hotplate with electrical wires b) The rolled up micro-hotplate compared to 5cent and c) The final Foil preconcentrator.*

The foil gas preconcentrators are successfully tested under benzene, acetophenone and nitrobenzene (figure 9). These results show preconcentration performances better than silicon devices thanks to the use of high adsorption flow rates, up to 80L/h (20L/h for the silicon).

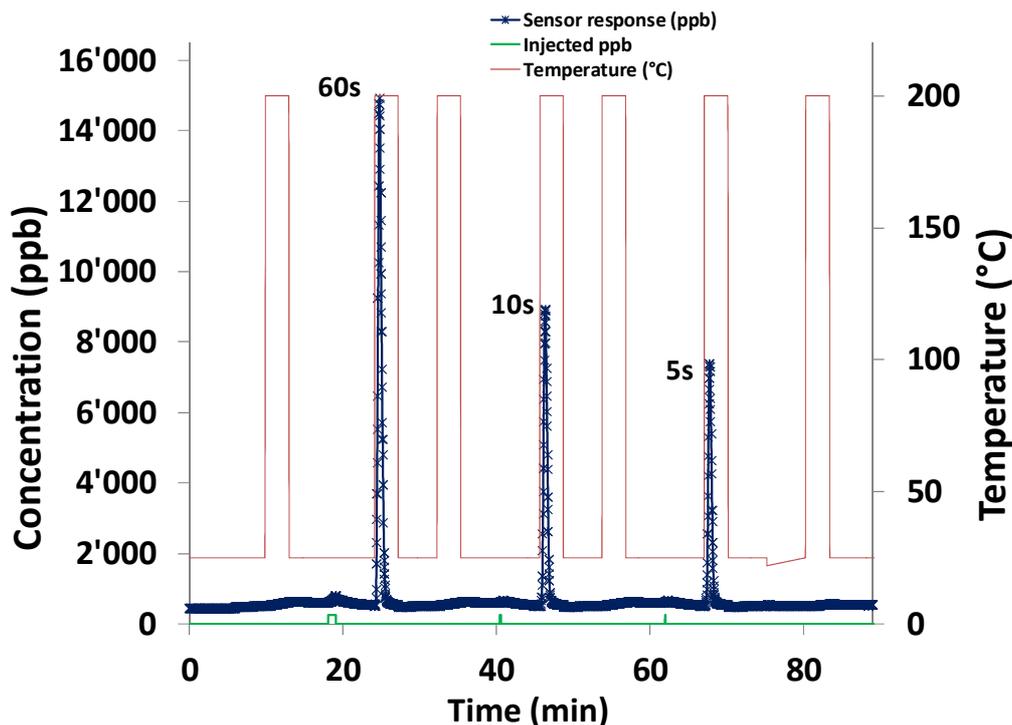




*Sensor response for a foil gas preconcentrator filled with (left) 1mg of carbopack B when exposed to 250ppb@1min of benzene at 40L/h and desorbed at 200°C with a flow of 4L/h and right) 1mg of Tenax TA when exposed to 100ppb@10min at 20L/h of acetophenone and desorbed at 130°C with a flow of 2L/h.*

In addition, foil micro gas preconcentrators permit also operating with a duty cycle down 30s (crucial in case of explosives detection) while conserving its high preconcentration performances (figure 10).





*Benzene peak for a FGP filled with 1mg of carbopack B after a adsorption of 250ppb during 60, 10 and 5s at 40L/h and desorbed at 200°C with a flow of 4L/h.*

### *Task 3.2 - Design and fabrication of new devices (IMT4) and feasibility study on flexible substrates*

Concerning the development of pre-concentrator devices on plastic, preliminary temperature tests are currently realized by TPD in order to determine the range of temperatures which can be used with the different polymers. On the other hand, for the OBP deposition in the device, it is expected to use inkjet printing. The adaptability of the inkjet machine of ARMINES and the determination of the deposition parameters are the objectives of the next period.

New preconcentration devices (IMT4) have been designed and fabricated in silicon technology. These devices are smaller than the IMT3 ones and have a serpentine design for maximizing the adsorption rate. In opposition to previous devices, IMT4 devices have a fluidic connection on the glass cover which allows no limitation on the capillary size since inlet/outlet are not dip in the silicon As IMT3, these new micro-channels include also a platinum micro-heater on the backside made by micro-fabrication process (Lift-off). These IMT4 devices are under optimization using FEM model for thermal and fluidic simulation for maximizing the adsorption capacity and the heating efficiency.

### *Task 3.3 – Module for automatic trace collection on hand-luggage for device D*

#### *Further Development of Trace Extraction Methodologies*

The current methods of removing particles and vapours from within closed carry bags and from the external surfaces were examined for improvement. For internal extraction, it remains to be sufficient to cyclically pressurize and depressurize a small chamber containing the examined package. Studied was the pressurization amplitude and frequency. A new data base is being developed for various types of packages so that the most effective pressure variation can be part of a program for the operator. This data base will be used in the future. For now a very good scheme has been developed for both particles and vapours. For external contamination (particles only), a series of jets have been shown to be effective at loosening pieces of contaminants to join the airborne particles and vapours

during the extraction phase. Jet timing and speed are important parameters in this process.

#### *Further Development of Trace Concentration Methodologies*

The particles and vapours extracted from the above phase are now distributed throughout the chamber and must be concentrated and collected. This should be done thoroughly to avoid chamber contamination in the event of the presence of contamination. Collection and direction methods were improved for particles as small as ½ microgram. Specialty jets were developed to take advantage of the special nature of airborne particles as they begin to fall and for the larger particles as they impact the floor and walls of the chamber. The jets cause the particles to be directed toward a target collector which currently is a fiber-glass filter capable of holding particles down to 1 microgram. These particles can later be vaporized if desired to provide vapours from them. Vapours in the chamber naturally follow the exhaust air and are sampled upon exit from the chamber.

#### *Design of Device for collecting trace vapours and particles from hand-carried*

In this task a device suitable for the SNIFFER program was completely designed with all engineering work including ergonomic considerations completed. The device includes conveyor means to automate placing the inspected articles in the testing chamber. For the field trials, it should be easy enough to provide the basic system without the conveyors to save costs.

Testing of using heating to improve concentration of extracted particles resulted in stickier particles rendering the method ineffective at improving extraction > Limit heating to 35°C.

Testing of collection concepts through a diaphragm: the idea resulted in a more complex diaphragm design rendering it more expensive. It further could not be made to be effective for the all-important larger particles which would get stuck on the walls > Corrective action was to collect vapours and particles from the floor of the chamber. This was the current practice so this was no problem to use.

#### **Significant results:**

- Flexible preconcentrator + OBPs as an absorbent material
- Optimized devices obtained
- Macro-porous silicon preconcentrator fabricated
- Under integration in finite device
- Optimization of DNT generation and process for study of adsorbent materials by TPD
- Validation of porous silicon and Carbopack B for the preconcentration of DNT
- Validation of Si3N4 insulating film for heater and process for capillaries sealing

With the involvement of a new “view” perspective within integration process a more complete integration progress is coming into progress.

- Development of high reliability method for trace particle extraction developed and for trace vapour extraction;
- Improved aerodynamic means of particle control and concentration and of concentrating vapours;
- Complete system for collecting trace particles and vapours from hand-carried packages designed.

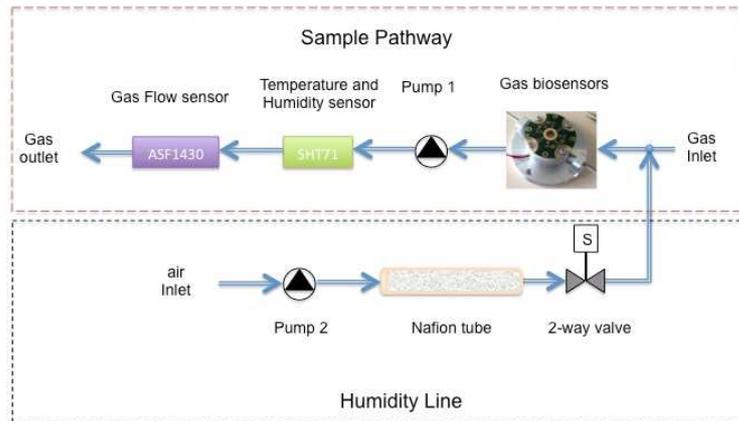
**Deviations:** None



## 3.4 WP 4 Sensor Sub-System & Array Engineering

<b>WP number</b>	4	<b>Start date or starting event:</b>	M1
<b>WP title</b>	Sensor Sub-System & Array Engineering		
<b>Activity type</b>	RTD		
<b>Participant ID</b>	2		
<b>Part. short name</b>	UNIMAN		
<b>PM total (for 60M)</b>	21		
<b>PM spent in period 1</b>	9		
<b>PM spent in period 2</b>	1,40		
<b>PM spent total</b>	10,40		
<b>Overview of the progress of the work during the period</b> <ul style="list-style-type: none"> <li>• The hardware integration of the biosensors into a small portable sensor sub-module</li> <li>• The multi-parametric training of the biosensors to optimise the recognition potential from the sensor array</li> </ul>			
<b>Work progress in each task</b> <ul style="list-style-type: none"> <li>• All related DL submitted</li> <li>• Functional Specifications and Design of the V2 system was achieved</li> <li>• Parametric software for pattern recognition was written and tested</li> <li>• V2 sensor-sub-module was integrated and the system went through lab and field trials</li> </ul> <p><i>Task 4.2 V2 of Sensor Sub-system</i></p> <p>The work in this work package included:</p> <ul style="list-style-type: none"> <li>• The hardware integration of V2 biosensors</li> <li>• The multi-parametric training of the biosensors to optimise the recognition potential from the sensor array</li> <li>• The integration of software and hardware into the V2 Sensor Sub-System</li> </ul> <p>Four deliverables were produced: DL421, which defines the functional specifications and design of architecture of V2 Sensor; DL422, defining the parametric software algorithms for pattern recognition, DL423, aimed at the description of the version 2 of the sensor sub-module and DL424, that reports on technical support and problems encountered during the V2 development.</p> <p>The second version of the Sensor Sub-System was built and integrated. It is composed of 3 main parts: pneumatic system, main electronic board, and sensor system.</p> <p><b>Pneumatic system</b></p> <p>V2 pneumatic system was modified respect to the V1 version. The new arrangement is shown below:</p>			





The V2 delivery system is composed of two main parts: Sample pathway and Humidity line.

### Humidity line

This line provides on-demand humid air to the biosensors. It is composed of a 2-way valve, a small glass bottle containing distilled water and a nafion humidifier tube and a pump that control the flow through the nafion tube.

### Sample Pathway

To reduce the probability of sample condensation on the inlet pathway, the inlet was made as short as possible, was built with glass tube, and was provided with a controlled heating system that fixes its temperature at 100 °C. This temperature can be easily changed by an end user, thanks to the touch screen display interface mounted on the front panel of the sub-module.

The sensors temperature is also controlled by means of a thermoelectric module, underneath the sensor chamber, controlled by the main microcontroller.

The temperature and humidity level of the biosensors and the airflow passing through the sensor chamber are constantly monitored by means of two sensors collocated at the output of the sensor chamber.

Pump 1 fixes the airflow rate passing through the sensor chamber. Its velocity is controlled by the microcontroller and can be easily changed by an end user.

### **Main electronic board**

A custom-made master board based on the ARM microcontroller technology is used to run D4. It is similar to that developed for V1 and consent to:

- Drive the 2 pumps;
- Control the 2-way valve;
- Manage a SD memory card to save sensor data;
- Manage 2 FRam memories;
- Control the inlet heating system;
- Control the sensor thermoelectric module by means of RS232 communication bus;
- Communicate with the sensor board by means of an I2C bus;
- Drive the samplers and the pre-concentrator;
- Implement a user interface by means of a touch screen display driven by a SPI bus;
- Implement several communication protocols (USB, RS232; Bluetooth, SPI, Ethernet);
- Run the detector system through the different measurement phases;
- Launch the multi-parametric software for sensor data recognition.

**Sensor array**

The sensor array comprises 8 diamond biosensors mounted into a circular sensor chamber. The biosensors were developed within WP5 and WP6. A custom-made sensor electronic board was developed within WP7. It allows sensor acquisition, calibration and self-diagnosis.

**Significant results:**

- Individual submodule tested: electronics and software validated;
- Low level software – written and tested – activation and scanning software for the microcantilevers tested and functional.
- Sensor interface electronics and software designed, constructed and tested;
- Master controller interface to WP2 functional were verified;
- Firmware for running the sensor sub-module was written and tested;
- Parametric pattern recognition software written, tested and implemented in the master controller of WP2;
- V2 built and tested in field trials in Athens airport in March 2015

**Deviations:**

Description of problem / deviation	Suggested / undertaken corrective action	Impact on other WPs, budget/PM plan or work plan
Delays in testing individual components – redesign necessary in some cases	Corrective action taken in all cases	Slight delay possible in delivery of V1 instrument
Condensation of the sample on the inlet pathway having the effect of reducing sensor sensibility, reproducibility and increases the limit of detection of the system.	Corrective action taken - inlet pathway as short as possible, Inlet glass tube, heating of the inlet path	None



## 3.5 WP 5 Biosensor Development

<b>WP number</b>	5	<b>Start date or starting event:</b>	M1
<b>WP title</b>	Biosensor Development		
<b>Activity type</b>	RTD		
<b>Participant ID</b>	1	9	
<b>Part. short name</b>	CEA	ESIEE	
<b>PM total (for 60M)</b>	60	42	
<b>PM spent in period 1</b>	27,29	27,84	
<b>PM spent in period 2</b>	32.7	27,82	
<b>PM spent total</b>	60	55.66	

### Overview of the progress of the work during the period

The work carried out during this first period led to the following achievements:

- Successful immobilisation of OBPs and MUPs onto diamond surfaces
- Optimisation of diamond microcantilever transducers in terms of sensor actuation and read out (OK)
- Transfer of OBPs and MUPs immobilisation protocols onto sensor active surfaces
- Validation of the whole sensor principle

### Work progress in each task

#### *Task 5.1 V1 of Biosensors*

The technologies developed within SNIFFER included some sampling as well as detection sub-systems. The scientific background of the sampling steps has already been described in deliverable D311 and the development of V1 sub-systems for the sampling and pre-concentration of target analytes were presented in DL314.

WP5 has dealt with the development of single microcantilever diamond sensors combining the odorant proteins selective coatings. This work has involved the development of diamond microcantilever transducers with optimum performances in terms of mechanical dynamic properties, which are linked directly to the sensitivity of the sensors. This work has involved also the development of chemical grafting procedure in order to immobilize covalently the bioreceptors (MUPS and OBPs) onto the diamond surface of the transducers. The specifications of such sensors have been defined in D511. Those technologies were then exploited in WP4 for the sensor array development which takes also in consideration the problematic associated with multiplexing the cantilevers.

The tasks that have been involved in V1 of biosensors included:

The development of diamond microcantilever chips

The development of protein covalent immobilization procedure on diamond surfaces

Assessment of the performance of the immobilized proteins using pre-existing surface acoustic wave available in the CEA laboratory.

Transfer of the immobilization procedures onto actual diamond microcantilever transducers

Development of a gas cell that can accommodate several sensors together in view of their integration into a sensor array.



### *Task 5.2 V2 of Biosensors*

During the first 18 months of the project a prototype V1 of biosensor has been specified and developed. The specifications of such sensors have been reported in DL5.1.1 and the results from the work and validation of V1 described in details in DL5.1.2. Furthermore, specifications of the version V2 of biosensors have been reported in DL5.2.1 which covers all aspects of biosensor development including the transducers, the biological sensitive coatings immobilization (OBPs and MUPs) as well as the multisensor array gas cell.

In the version V2, the main improvements on the microcantilever are as follows. Firstly the read out metal gages were replaced by polysilicon gages featuring much better gage factor. Secondly, the metal used for contacting to the sensors was changed to insure better resilience during the protein immobilisation process. Finally, the cantilever geometrical parameters were modified in order to improve sensitivity. Two different approaches have been considered in V2: bulk diamond cantilevers and diamond coated silicon cantilevers.

An efficient protocol to immobilise both MUPs and OBPs covalently onto the diamond surface of the cantilevers was proposed with V1. This protocol proved to be highly efficient therefore it was also used in v2 to immobilised new proteins on diamond sensor .

A V1 gas cell was designed according to the SNIFFER project specifications of biosensor development. In collaboration with ESIEE-Paris the sensors in the cell could be interfaced electronically to the acquisition board to display and validate that the complete sensor array system was operational. The development of the V2 multi-sensor gas cell was a follow up of the first cell that was developed in the first part of the SNIFFER project and patented. It was described in DL523. V2 multi-sensor gas cell takes into account the feedbacks received from the work experience and assessment gained with the first gas cell. However most of the improvements made were mostly targeting a reduction of fabrication cost since the performances of the first cell were already acceptable.

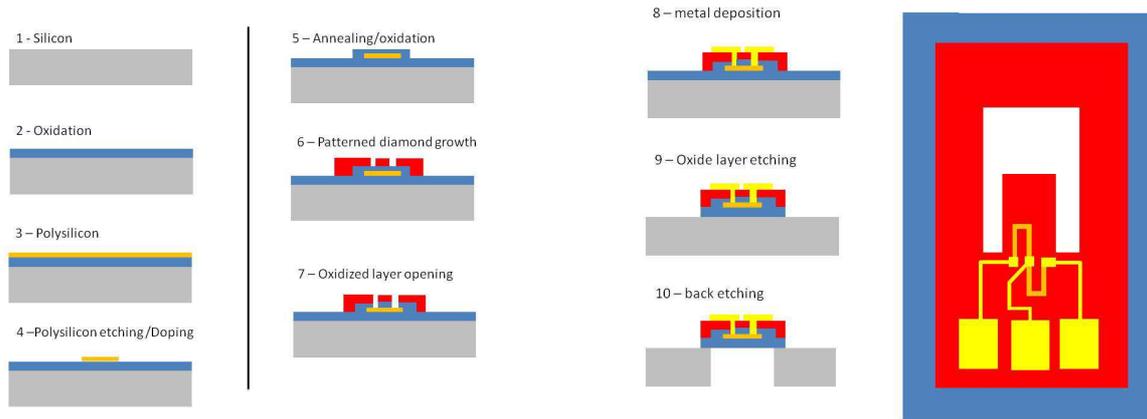
Finally a range of new OBPs and MUPs have been provided by partners UNIMAN UNIPD and GTP that were characterised either using reference diamond SAW transducers or diamond cantilevers.

## **1. V2 of microcantilevers**

### **1.1. Bulk diamond microcantilevers**

The strategy elaborated to actuate and read-out the sensor signals was described in DL5.1.1. It consists of actuating all cantilevers in the gas cell array using a single external piezo-transducer located underneath the different sensor chips. The sensor signal is thus obtained from the measurement of the strain resistance of gages placed at the anchor on the cantilevers. The optimum gages parameters were simulated and reported in DL5.1.1. The following gage characteristics were obtained: gage factor > 20 and resistance value < 1 k $\Omega$ . Neither metal nor boron doped diamond gages used in the V1 of cantilevers were found not to satisfy those conditions. Therefore it was decided for V2 to use polysilicon gages. Indeed polysilicon gages have proven to be efficient in the area of silicon MEMS with gage factors in the order of 100.

Hence the new strategy consists of embedding polysilicon gages into the diamond microstructures. Thus a process to include polysilicon piezoresistive gages into diamond micro cantilevers was developed as described in Fig.1.

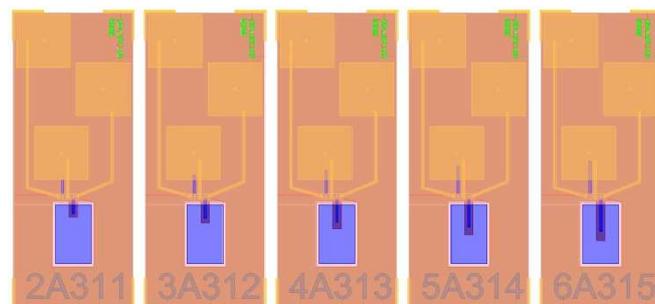


*Process flow to the fabrication of diamond micro cantilevers including embedded polysilicon piezoresistive gages*

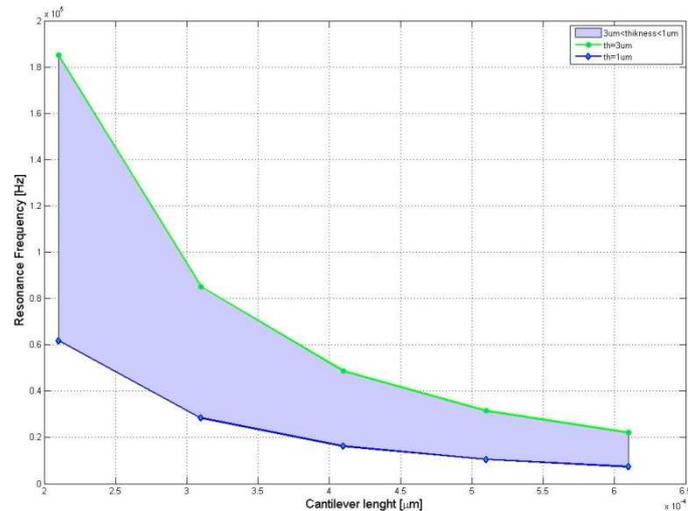
For mass detection, we demonstrated that the higher the resonance frequency of the cantilever, the higher the detection sensitivity. However, high resonance frequency cantilevers are geometrically short and mechanically rigid cantilevers therefore reducing the electrical signal at the output of the piezo-resistive strain gages. The main drawback with this design is that the level of signal delivered by the gages is also low. Even though with the electronic amplification it is possible to follow the frequency resonance of the cantilever, the signal strength is of the same order of magnitude as the noise level. To improve the electrical S/N at the output of the gages, it was decided to explore 5 different lengths of cantilever. The dimensions and theoretical resonance frequency (first mode) of each design is indicated in Table 1. Fig. 2 gives a graphical representation of 5 cantilever designs. Additionally, Fig. 3 presents the resonance frequency variation as a function of cantilever length and thickness.

*Dimension and theory frequencies of new cantilevers design*

Design	Cantilever characteristic (length / width)	Gage dimensions on the cantilevers (length / width )	Theoretical Frequency(Hz)	
			e=1 μm	e=3 μm
1	210 μm /140 μm	150 μm / 10 μm	61700	185300
2	310 μm /140 μm	225 μm / 10 μm	28300	85000
3	410 μm / 140 μm	300 μm / 10 μm	16200	48600
4	510 μm /140 μm	375 μm / 10 μm	10400	31400
5	610 μm /140 μm	450 μm / 10 μm	7300	22000

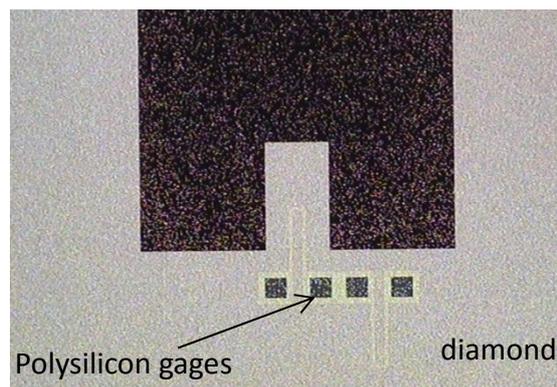


*Fabrication masks design for the five cantilever lengths*



*Resonance frequency as a function of Cantilever length and thickness*

Diamond microcantilevers were fabricated according to the above descriptions. Figure 4 shows an optical microscope image of a cantilever during the fabrication process.



*Optical microscope image of a polysilicon gage during the fabrication process in step 7*

### 1.2. Diamond coated Silicon microcantilevers

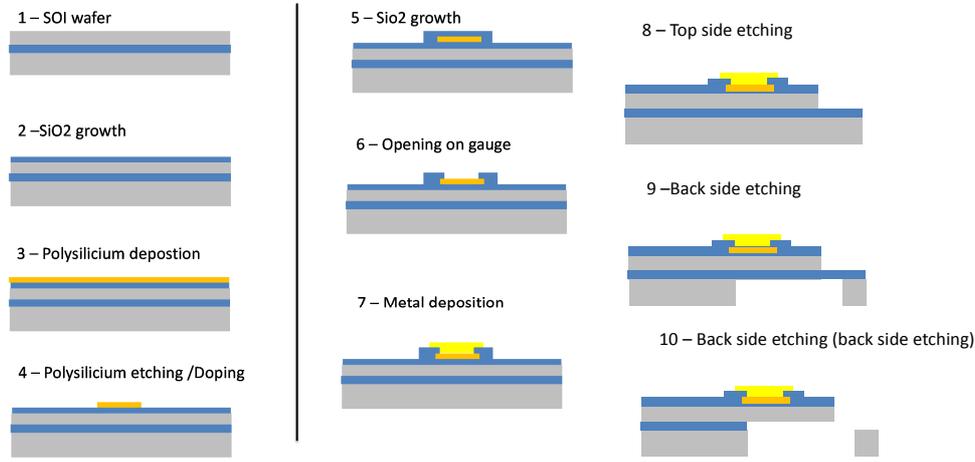
CEA-LCD developed a specific process to graft OBPs and MUPs on diamond surfaces. The process was validated by chemical surface analysis (XPS, EIS, fluorescence imaging) and for sensing performances using diamond coated SAW devices. Diamond coated SAW sensor is a technology that was developed prior to the Sniffer project by CEA-LIST. Here the process involves spreading diamond nanoparticles over the sensor substrate and then fix them by a short CVD plasma growth in order to obtain at the end a thin polycrystalline diamond coating over the surface.

Based on the same approach, silicon cantilevers were fabricated with embedded polysilicon gages and coated with diamond. This approach enables high control of the whole fabrication process using previously developed processes on silicon while still offering all the advantages of the diamond coating for chemical grafting and stability. Hence the two approaches (bulk diamond and diamond coating) will be later assessed in terms of sensing performances.

The silicon cantilever process uses a specific silicon substrate call SOI: Silicon-On-Insulator. These

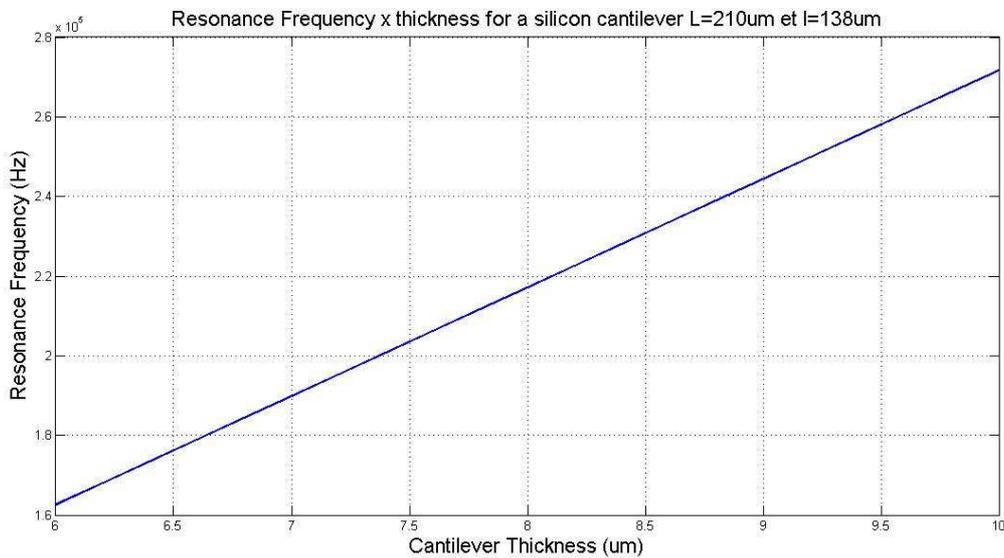
substrates are composed by 3 layers: a first silicon layer (Device), a silicon dioxide layer, and a second silicon layer (Handle). When compared to standard plain silicon wafers, SOI allows high accuracy control over the cantilever thickness and the integrated silicon dioxide layer is naturally acting as a stop layer during backside etching and release of the resonant structure.

The process to manufacture silicon cantilever using SOI wafer is summarised in Fig.5.



*Description of process for Silicon cantilevers*

Simulations were performed to determine the thickness of the silicon cantilevers leading to resonance frequency within the same range as the diamond cantilever already used in the project. As shown in Fig. 6, a silicon layer of 6.5µm thickness is adapted to the resonance frequency range targeted by this project.

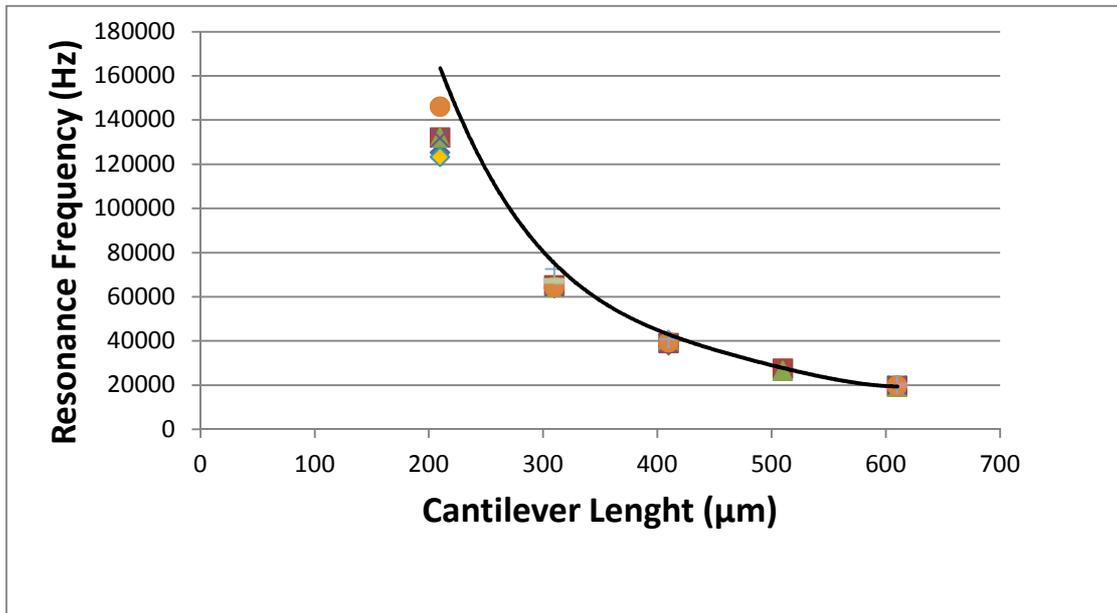


*Silicon cantilever resonance frequency vs. thickness*

Silicon cantilevers were fabricated using the SOI process described in figure 5. A Micro Scanning Laser Vibrometer was used for characterizing the frequency response of the designed cantilevers. This equipment consists of a laser source emitting between 620 nm and 690 nm, an optical fibre interferometer OFV511, a demodulator OFV3001 (Polytec GmbH) and a network analyzer Agilent 89410A. Figure 7 shows the frequency measured for each cantilever of different length (represented

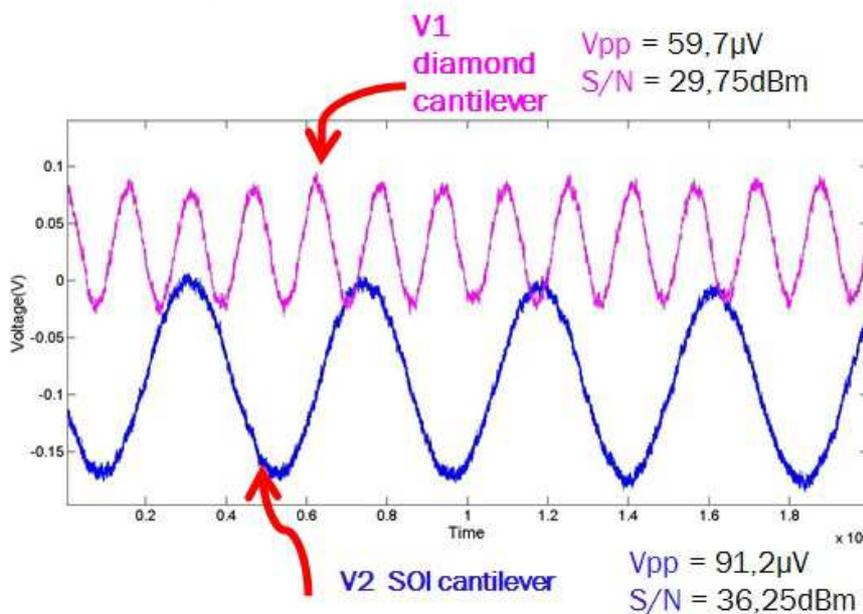


by points) and the computed theoretical curve of frequencies (represented by a black line). The results show that the measured frequencies match well the expected values from calculation.



Resonance frequency measured (points) and computed (black line) as a function of Cantilever length

Cantilevers were then electrically connected in order to measure the S/N ratio out of the strain gages using the read-out electronic developed for V1 Sniffer device. Fig. 8 shows the typical response for a V1 and a V2 cantilevers with the same length (here 210 µm). Here the S/N ratio was improved by 25% hence leading to better sensitivity of the resulting sensors. Example of spectral responses are summarised in Table 2 for V1 and V2 cantilevers, again confirming those results.



Example of response measured using SNIFFER V1 read out electronic for a V1 and a V2 cantilever with the same length 210 µm

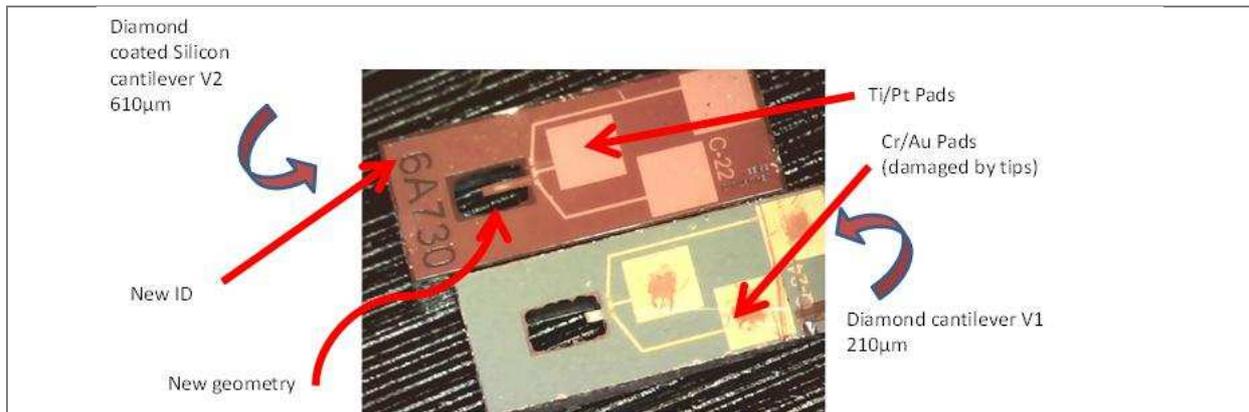


Examples of spectral responses for both V1 and V2 cantilevers

Channel	ID	Length (um)	Freq (KHz)	S/N	Resistance R1 (cantilever) and R2 (fixe) (KΩ)	Spectrum
1	SOI 2-6A515 (V2)	610	20,700	40.0	R1 = 61,59 R2 = 62,85	
3	SOI1-2B721 (V2)	210	131,770	40.0	R1 = 26,55 R2 = 27,84	
4	Diamond Q24 (V1)	210	63,870	29.75	R1 = 23,16 R2 = 22,29	

Finally the metal contact used in V1 proved not to be performing well. This was due to the lack of robustness of the Cr/AU layers when the cantilevers were placed in the diamond growth reactor to undertake plasma treatment in view of hydrogen terminate the surface, a step that is necessary in order to later graft the ligand binding proteins. Hence those contacts were replaced in the V2 by Ti/Pt contacts that were found to withstand perfectly the same plasma conditions. Fig. 9 shows comparative images of V1 and V2 generation cantilevers.

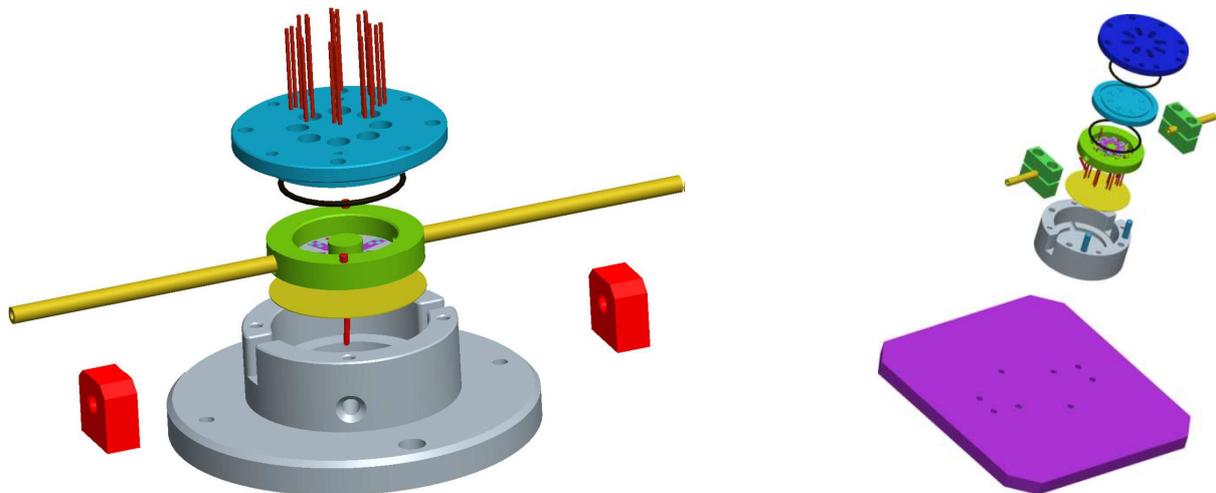




*Microscope image of two cantilevers, V1 and V2, summarizing all improvement implemented in V2 when compared to V1.*

## 2. V2 of multisensory gas cell

The development of the V2 multi-sensor gas cell is a follow up of the first cell that was developed in the first part of the SNIFFER project and patented. It was described in DL512. V2 multi-sensor gas cell takes into account the feedbacks received from the work experience and assessment gained with the first gas cell. The improvements that have been made are still in line with the claims made in the first patent; therefore this new cell is also protected by the same patent. A schematic view of this new cell is shown in Fig.10 (left). As a reminder and for comparison purpose a view of the former V1 gas cell is also shown (Fig.10 right). These schematics show clearly that V2 of the gas cell has been simplified when compared to the V1.



*Schematic view of - Left: the V2 multi-sensor gas cell, - right: the V1 multi-sensor gas cell*

Several changes have been made over version V1 in particular to improve both cost efficiency and performances. In particular:

- The 2-parts base of the device (holder + embodiment) has been replaced by a single part (in grey colour in the V2 schematic). Since this part of the device does not see gas the material has been replaced from stainless steel to aluminium for cost efficiency.
- The cantilever holder (green part on both figures) has been simplified in version 2. Indeed in version 1 the inside of this part had to be micro-machined in order to be able to position the sensors with high accuracy so as to align the 24 contacting pin with the metal contact pads of the 8 cantilevers. This operation contributed to increasing very significantly the fabrication

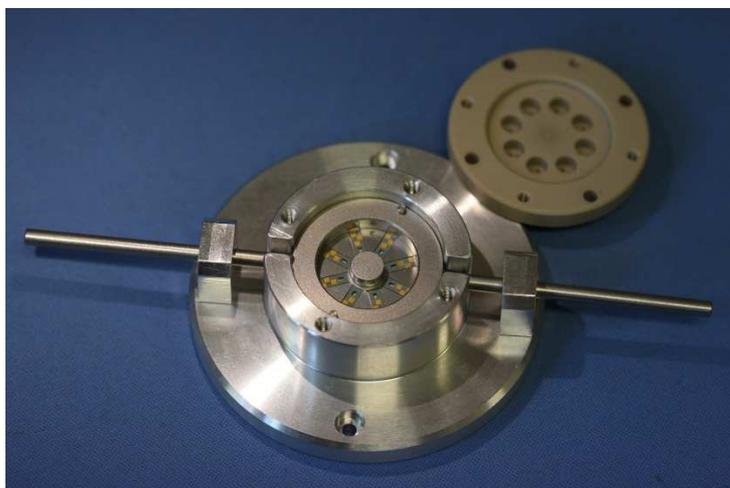
cost. Therefore in the version2 of the cell, micro-machining of this part was not done. Instead a new part was fabricated from a sheet of stainless steel that was laser cut. A photograph of this new part is shown in the following figure.



*View of the part designed to hold and align the cantilevers inside the gas cell*

Finally, the lid of version 1 which was also made of two parts has been simplified to one part. This facilitates significantly the replacement the opening/closing of the cell and therefore the replacement of sensors. The design of this lid was also modified to improve sealing of the cell to ensure a perfectly gas tight chamber.

The above modifications allowed reducing the fabrication cost by typically a factor 4 (for small batches processing) and reducing very significantly the size and weight of the overall cell. A photograph of the finished cell is shown in the following figure.



*Photograph of the V2 of multi-sensor gas cell*

### **3. Selection of proteins as selective coatings**

Based on previous results obtained by the UNIMAN and UNIPD on one side while performing competitive binding experiments in the liquid phase, and on the results from the first series of laboratory trials, a shortlist of LBPs has been drawn that will be used to perform the final testing. These proteins are listed in the following table.

*List of OBPs and MUPs to be used for the final lab trials and field trials*

<b>OBPs</b>	<b>MUPs</b>
GTP_pigWT	MUP3 (wt)
GTP_pigF88W	MUP20 (wt)
GTP_AgamOBP1	MUP20L88Q
GTP_AgamOBP4	MUP20L124V
GTP_AgamOBP5	MUP20Y103R
GTP_AgamOBP19	MUP20Y103D
GTP_agamOBP47	MUP7
GTP_AgamOBP1_S82P	MUP20N107L
GTP_AgamOBP1_K93H	
GTP_LmigOBP1	

The proteins will be grafted onto the diamond sensors using the chemical protocol developed in version 1 of sensors consisting of grafting a NTA-Ni radical onto the diamond surface followed by chelating to the Nickel via the 6His tags of the proteins. This approach was found to give the best results in terms of detection. When wild type proteins with no 6His tag will be used the other approach involving attachment of hexanoic acid radical followed by EDC/NHS coupling will be used, as described previously.

#### 4. Sensor integration into the measurement gas cell

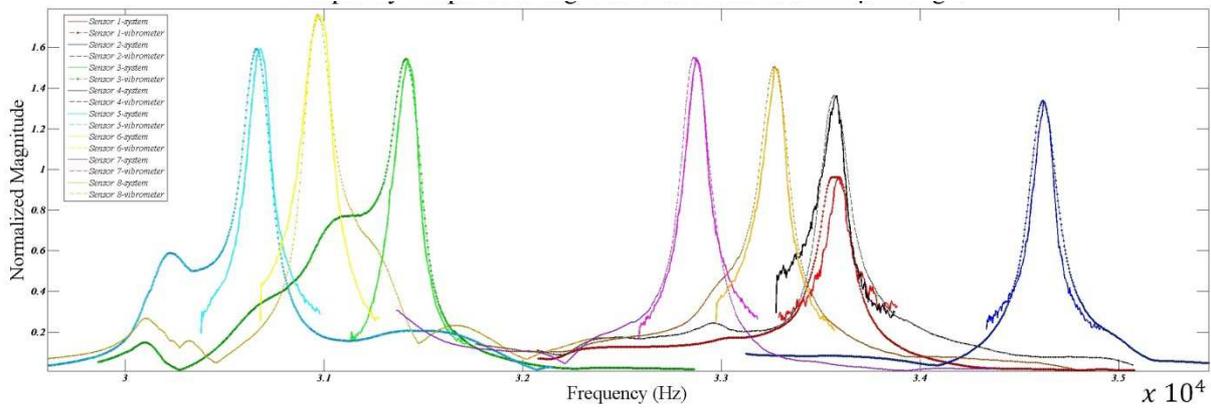
The transducers developed of the V2 of biosensors have been described specifically in DL5.2.2. Their description details and fabrication steps can be found in this deliverable. Fig.13 shows a picture of eight diamond micro-cantilevers placed in the gas cell.



*View of a V2 micro-cantilever placed in the gas cell*

In order to validate the V2 of biosensors, the cantilevers were characterized in the cell gas and configured to work on the standard test conditions. The performances of the system were assessed using different cantilever lengths. A micro-scanning laser vibrometer (Polytec OVF511 and OVF3001) and a spectrum analyzer (Tektronix RSA3303B) were used to characterize each sensor in terms of resonance properties and the data were compared to the resonance profiles extracted by our autonomous SNIFFER system. Frequency profiles were measured and normalized within a bandwidth

of 3 kHz around each resonance frequency. An example of comparison between the two methods is depicted on Fig. 14. Table 4 presents the experimental figures measured to compare the optical reference data with the electrical measurement with the SNIFFER readout system.



Frequency Response of eight cantilever of 510µm length.

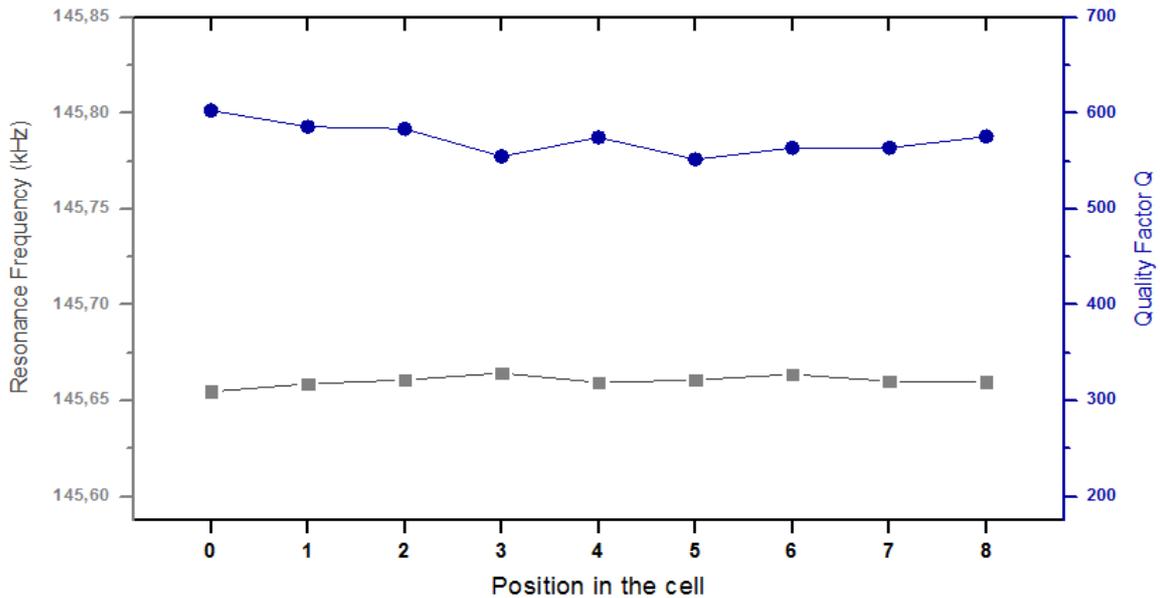
Comparison of the optical reference measurements with the electrical measurements

Sensor number	Characteristics			Sensor number	Characteristics		
	Δ Frequency(Hz)	Δ Q. Factor	RMSE		Δ Frequency(Hz)	Δ Q. Factor	RMSE
1	26.31	101.75	0.108	5	22.17	44.93	0.154
2	6.61	100.22	0.091	6	0.50	8.28	0.090
3	9.72	77,89	0.110	7	10.00	52.27	0.111
4	12.31	37.82	0.057	8	2.59	48.56	0.098

Note: Root mean square error (RMSE) is evaluated using the difference between the two frequency responses corresponding to the two measurement methods presented in this work (evaluated using the normalised magnitude above -8dB of the centre frequency).

Optical and electrical measurements were also carried out for each cantilever placed in the gas cell to analyse the influence of the relative location on the piezoelectric cell on the measured resonance frequency and quality factor. The results are presented in the following figure.





*Resonance Frequency and Quality Factor of a V2 micro-cantilever placed in different positions in the gas cell*

From these preliminary tests we were able to quantify the influence of the closed gas cell and the electronic circuit on the resonance frequency measurements. Further experiments were performed in order to validate the operation of the sensors and to demonstrate the performance of the whole system, including gas cell and electronics, to detect sensors frequency shifts.

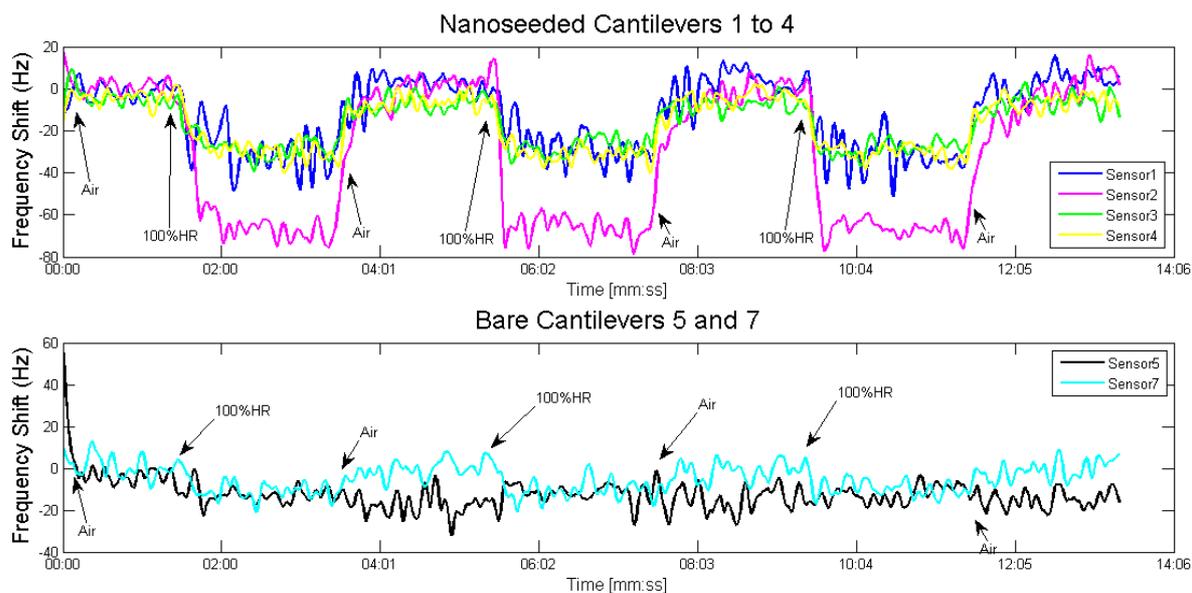
A set of eight silicon cantilevers of 410µm length were selected to be tested. The below table lists the sensors characteristics, as well as the post-treatments received by each sensor, respectively.

*Sensors characteristics and treatment in experiment 1*

Cantilever	Unique code	Resonant Frequency(KHz)	Treatment	Cantilever	Unique code	Resonant Frequency (kHz)	Treatment
1	4B118	54.368	Diamond nanoseeded	5	4B113	49.353	Bare cantilever
2	4B23	58.981	Diamond nanoseeded	6	4B38	48.234	Bare cantilever
3	4B318	47.304	Diamond nanoseeded	7	4B813	52.646	Bare cantilever
4	4B218	49.742	Diamond nanoseeded	8	4B58	47.410	Bare cantilever

In order to verify the influence of the humidity on the sensors, the gas (nitrogen) flow in the cell was set to 500 sccm/min. After stabilization of the system (regarding temperature, flow, etc.) we proceed to the injection of humid air at 100%. The results of this experiment are presented in figure 16.





*Frequency shift of sensors subjected to cycles of dry air and moist air*

In this experiment, sensor 7 and 8 could not be electronically compensated and the output signal was discarded in this analysis. In the first graph of figure 17 we can identify a frequency shift of some tens of Hertz for the nanoseeded cantilevers. This demonstrates that the frequency shift depends on the surface conditions of the cantilevers.

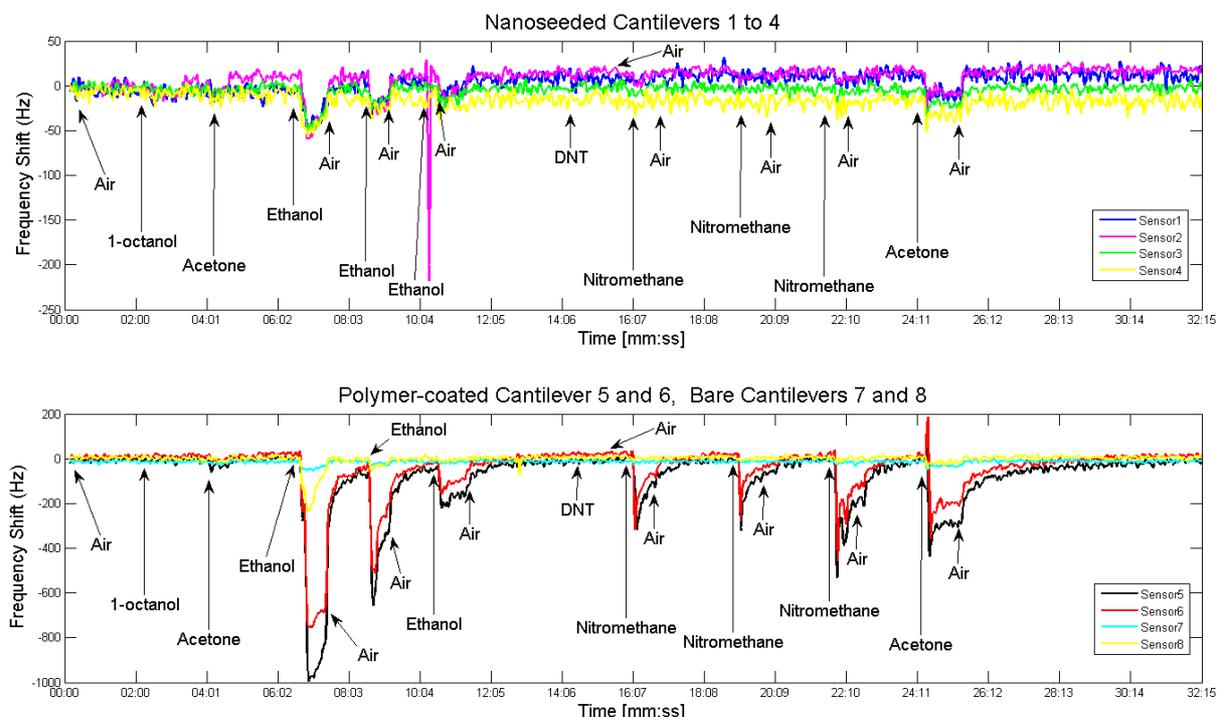
In the following experiment, using the same set of eight cantilevers described in table 5, cantilever 5 and 6 were coated with PMMA polymer in order to obtain sensitivity to chemicals. The characteristics of sensors are listed in the table below.

*Sensors characteristics and treatment in experiment 2*

Cantilever	Unique code	Resonant Frequency(KHz)	Treatment	Cantilever	Unique code	Resonant Frequency (kHz)	Treatment
1	4B118	54.368	Diamond nanoseeded	5	4B113	49.580	PMMA
2	4B23	58.981	Diamond nanoseeded	6	4B38	48.335	PMMA
3	4B318	47.304	Diamond nanoseeded	7	4B813	52.646	Bare cantilever
4	4B218	49.742	Diamond nanoseeded	8	4B58	47.410	Bare cantilever



After being polymer-coated, cantilever 5 and 6 have changed their resonance frequency due to the added mass. Before injecting solvent vapours on the gas cell, the gas flow was set at 500sccm/min (nitrogen) and we waited for two minutes in order to stabilize the measurement. The chemicals used in this experiment are: 1-octanol, acetone, ethanol, DNT and Nitromethane (a precursor used to prepare nitroaromatic explosives). The resulting sensor responses during this experiment are presented below.



*Frequency shift of sensors subjected to cycles of different chemicals*

From the results presented in figure 17, we can clearly observe that sensors 5 and 6 have shown the best responses to ethanol, acetone and nitromethane. For the detection of ethanol, the frequency shift can reach up to 1 KHz and for nitromethane we can observe several hundred of Hz of variation. Bare cantilevers (7 and 8) respond only to ethanol while the nanoseeded cantilevers (1 to 4) respond to acetone and ethanol with a frequency shift of about 40Hz.

Improvements on the functionalization steps can be done to achieve higher sensitivity and selectivity. Further lab trials are to be carried out on diamond cantilevers as well as silicon cantilevers, using LBPs as sensitive coatings.

### Significant results:

Two procedures have been developed and characterised in order to immobilise OBPs and MUPs onto diamond surfaces. The first procedure enables covalent attachment of the proteins with random orientation over the surface. The second approach allows orientation of the proteins over the surface through the use of histidine tag.

Both protein attachment procedures were transferred successfully to diamond microcantilevers. The second approach appeared to give significantly better sensing performances and will be chosen for the V1 prototype.

A gas cell was designed according to the SNIFFER project needs. The cell can accommodate 8 sensors in parallel. In collaboration with ESIEE-Paris the sensors in the cell could be interfaced electronically

to the acquisition board to show that the complete sensor array system is operational.

Sensor performance assessment was carried out with chemical simulant (DNT, 2-methylpyrazine). A test campaign with real drugs and explosive has been carried out in July at CREAL (France) to validate the sensors with substances of interest for the SNIFFER applications.

A set of improvements has been implemented in version 2 of cantilevers. They can be summarised as follows:

- Change of piezo gage material from BDD to polysilicon with much higher gage factor. A dedicated process had thus to be developed in order to embed such gages into bulk diamond cantilevers.
- Change of geometry of both the cantilevers and strain gages in order to increase S/N ratio. This change was shown to pay off since an increase up to 25% in S/N could be observed with the V2 cantilevers
- Change of metal contacts to increase robustness, in particular during the proteins immobilization process, and long term stability of the sensors.

Moreover an alternative process based on the fabrication of Si cantilevers from SOI wafers and then coating of the cantilevers with diamond has been proposed.

A new version of gas cell was also developed.. This new gas cell can accommodate up to 8 individual sensors that can be actuation simultaneously by a single piezo-actuator embedded into the gas cell. The gas cell has been simplified considerably over the first version in order to reduce fabrication cost and practicality of replacing the sensors in the cell.

- The cantilevers were functionalised with various coatings in order to verify the chemical sensitivity of the complete sensing module. The sensors were sensitive to various vapours including acetone, ethanol or nitromethane, a precursor for many explosives.

**Deviations:** None

## 3.6 WP 6 Protein Engineering

<b>WP number</b>	6		<b>Start date or starting event:</b>	M1
<b>WP title</b>	Protein Engineering			
<b>Activity type</b>	RTD			
<b>Participant ID</b>	2	8	10	
<b>Part. short name</b>	UNIMAN	UNIPD	GTP	
<b>PM total (for 60M)</b>	15	36	12	
<b>PM spent in period 1</b>	9	22	8,48	
<b>PM spent in period 2</b>	23	28	7,64	
<b>PM spent total</b>	32	50	16,12	

### Overview of the progress of the work during the period

The major objectives were :

- Selection of LBPs
- Tailoring of protein recognition site conformation for a selected analytes through selective mutagenesis
- Production of proteins by expression and the industrial scale-up of LBP productions
- Immobilisation of proteins on to diamond coated cantilever substrates
- Testing of sensitivity and selectivity of immobilised proteins to selected analytes
- Validation of stability, repeatability and influence of environmental factors.

#### *Task 6.1 – V1 of proteins*

Task 6.1 concerned selection of suitable proteins for Version 1 of the SNIFFER device. Close interactions between UNIMAN, UNIPD, GTP as well as the user partners CEA and ESIEE were established to achieve the goals. This involved selection of LBPs, followed by in-house protein expression, then industrial scale production by P10 (GTP), then testing the binding properties of the produced proteins towards the targeted analytes. Initial experiments were performed by UNIMAN and UNIPD aimed at carrying out TASK 6.1 'V1 of Proteins'. A first batch of OBPs was produced by UNIMAN using a bacterial expression system. It was composed of four different OBPs. Moreover, a functional recombinant MUP (rMUP) was expressed by UNIPD. Affinities and selectivities of the selected OBPs and the rMUP for target analytes were investigated. Processes were developed for producing and purifying the OBPs and MUPS using appropriate molecular biology technique, to produce test batches in small quantities. Fluorescent binding experiments were performed to test the affinity of the mutant pOBP-FW and the rMUP to target explosives and drugs. The gene sequences of selected proteins were transferred to partner GTP, who commenced the process of scale up for large scale production of the ligand binding proteins.

#### *Task 6.2 – V2 of proteins*

Task 6.2 was concerned with the preparation for V2 of proteins (OBPs and MUPs) for the second stage of the project. This involved the design of methodology for cloning, expressing and modification of the selected proteins, as well as the quality control criteria for assessing the viability of the produced proteins. It involved the definition of the boundaries of acceptable performance



taking into account dynamic range, affinity, selectivity etc. In conjunction with partner GTP, compatibility with industrial production methods and application-focused needs such as response time, robustness and influence of environmental variables were optimised.

### Work progress in each task

#### *Task 6.1 – V1 of proteins*

This involved close interaction between UNIMAN, UNIPD, GTP as well as the user partners CEA and ESIEE. Consultant Prof. Paolo Pelosi from University of Pisa and his laboratory were heavily involved in the initial stages and provided genetic material to UNIMAN and practical help to both UNIPD and UNIMAN. This involved selection of LBPs, followed by in-house protein expression, then industrial scale production by P10 (GTP), then testing the binding properties of the produced proteins towards the targeted analytes.

Initial experiments were performed by UNIMAN and UNIPD.

This task included:

- Setting up recombinant protein expression at UNIPD and UNIMAN
- rOBP and rMUP expression and purification at UNIMAN and UNIPD
- Evaluation of rMUP ligands by GC/MS - UNIPD
- rMUP binding assay in two independent Labs - (UNIPD and Prof. Pelosi Lab in Pisa)
- *In silico* screening of natural MUP mutants - UNIPD
- Selection of alternative natural MUP isoforms - UNIPD
- Setting up gene sequences for OBPs and MUPs – UNIMAN, UNIPD
- Engineering of expression plasmids for OBPs and MUPs –UNIMAN, UNIPD
- Bacterial transformation and protein Expression of OBPs and MUPs –UNIMAN, UNIPD
- Characterisation of newly expressed recombinant proteins – UNIMAN, UNIPD
- Binding assays to characterise the proteins against target analytes, UNIMAN, UNIPD

A first batch of OBPs was produced by UNIMAN. It was composed of four different OBPs (1PDOM from the insect wasp and F88W from the pig were characterised). Moreover, a functional recombinant MUP (rMUP 8) was expressed by UNIPD. Affinities and selectivities of the selected OBPs and the rMUP for target analytes were investigated. Processes were developed for producing and purifying the OBPs and MUPS using appropriate molecular biology technique, to produce test batches in small quantities. Fluorescent binding experiments were performed to test the affinity of the mutant pOBP-FW (UNIMAN) and the rMUP (UNIPD) to target explosives and drugs. Samples of proteins developed were sent to CEA for immobilisation on to diamond substrates and subsequent characterisation of immobilised protein. The gene sequences of selected proteins were transferred to partner GTP, who commenced the process of scale up for large scale production of the ligand binding proteins.

### Major Urinary Proteins

Engineered yeast (*Pichia Pastoris*) expressing recombinant mouse Major Urinary Protein (rMUP) was kindly provided by Dr. Elena Ferrari (University of Parma – Italy). Heterologous expression of rMUP was carried out according to a protocol previously described [Ferrari et al., 1997].

The optimal volume of biomass for the best rate of protein production was determined and rMUP was expressed, purified and concentrated. A first evaluation on possible endogenous ligands, naturally present in the purified rMUP, was performed by Solid Phase Micro Extraction (SPME), followed by GC/MS analysis according to a previously described procedure [Redaelli et al., 2006]. The data were processed using the software MS data Review (Varian, Italy) in comparison with our in



house spectra library and the NIST Mass Spectra Database (version 06, NIH 2006). Binding assays against target analytes were performed in two independent facilities (UNIPD and Pisa Lab).

[Ferrari E, Lodi T, Sorbi RT, Tirindelli R, Cavaggioni A, Spisni A. Expression of a lipocalin in *Pichia pastoris*: secretion, purification and binding activity of a recombinant mouse major urinary protein. *FEBS Lett.* 1997. 401(1):73-77.]

#### Task 6.2 – V2 of proteins

This involved close interaction between UNIMAN, UNIPD, GTP as well as the user partners CEA and ESIEE. Work done in this task has been reported in detail in DL6.21, DL6.22 and DL6.23.

- A number of OBPs and MUPS mutants were selected through *in silico* screening towards target analytes via ligand docking experiments and subsequent data analysis.
- Methods of protein expression were optimised and selected mutants of OBPs and MUPS were expressed, and their affinities to target analytes tested, together with stability and repeatability of performance in detecting and recognising sniffer target analytes.
- Quality control considerations have been analysed and steps taken to ensure the performance of manufactured proteins.
- The scale-up of protein production was carried out by partner GTP.
- Characterisation of the GTP produced proteins (both WTs and mutants) in terms of their affinities, and stability of performance in detecting and recognising sniffer target analytes was carried out at UNIMAN and UNIPD.
- Proteins were sent to Partners ESIEE and CEA to be immobilised onto the transducers.
- The characterisation of the proteins once immobilised onto the transducers was carried out in partner (CE and CREAL) and that have the appropriate licenses for target compounds.

#### Significant results:

Successful site directed mutagenesis techniques have been developed by UNIMAN and UNIPD involving *in silico* modelling of the binding sites and docking screening approaches. The approach has been used to design mutant OBPs and MUPS leading to new proteins with enhanced binding properties towards Sniffer target analytes.

Eleven 11 OBPs (eight Wild Type proteins and three mutants) were produced in industrial scale by GTP (Partner 10) and then characterised; these proteins were used to develop SNIFFER devices.

Ten MUPS (three WTs and seven mutants) were produced by UNIPD and GTP and then characterised; these proteins were used to develop SNIFFER devices.

The scale-up of protein production by partner GTP means that OBPs and MUPS can be produced in 100mg to gram quantities. This is a significant technical success that would not have been possible without the SNIFFER project to drive this development.

#### Major result:

We consider that the development of new and novel Ligand Binding Proteins for the SNIFFER project has been an extremely successful part of the project and this has provoked innovative and novel technical advances that have also generated significant new intellectual property applicable to the wider biosensor field apart from the objectives of the SNIFFER project. The incorporation of these proteins into arrays has never been achieved before. The characterisation of the proteins once immobilised onto the transducers carried out in partner laboratories (CE and CREAL), indicate good sensitivities to target analytes. The recent field trial carried out in Athens has confirmed that the LBPs developed in this project are indeed performing well in the recognition of the sniffer target analytes.

**Deviations:** None



## 3.7 WP 7 Self-Diagnostic Capabilities

<b>WP number</b>	7	<b>Start date or starting event:</b>	M1
<b>WP title</b>	Self-Diagnostic Capabilities		
<b>Activity type</b>	RTD		
<b>Participant ID</b>	2	9	
<b>Part. short name</b>	UNIMAN	ESIEE	
<b>PM total (for 60M)</b>	6	18	
<b>PM spent in period 1</b>	2	13,35	
<b>PM spent in period 2</b>	0,3	3,50	
<b>PM spent total</b>	2,3	16,85	

### Overview of the progress of the work during the period

WP7 is completely dedicated to self-diagnosis and calibration.

- Task 7.1, Self-Diagnostic Sub-System, aims at the development of the electronics for performing the self-diagnosis. The objective of the first period was to develop a prototype that can be interfaced with the gas cell to extract the resonance frequency of 4 cantilevers in real time. This working V1 prototype was then refined during the second period of the project toward an electronic that can address a full array of 8 diamond resonant sensors and bring full self-diagnostic and self-characterization feature. The objective of reaching precise resonant frequency tracking while providing the real-time information of the characteristics and health of the sensors has been successfully reached. Moreover self-characterization information are used to calibrate the electronic and the digital signal processing algorithm developed to improve overall system sensibility and robustness. .
- Task 7.2, Reference Samples for Calibration, is involved in the definition of the calibration procedure and reference samples to be used for the calibration procedure. The objective of this task are to define the calibration process for the prototype as well as the reference samples and the rationale that led to their selection.

### *Task 7.1 – Self diagnostic sub-system*

#### **5. Sensor mixed signal sensor read-out architecture Architecture**

Within the SNIFFER project, WP7 is mainly concerned with the electrical interface and the acquisition of the resonance frequency of each sensor of the array. It also checks that the sensor array is working in the best possible conditions by mean of self-diagnostic and calibration processes. Those tasks have been integrated in an electronic sub-module that is interfaced on one side to the sensor array and on the other side to the global system controller (or supervisor).

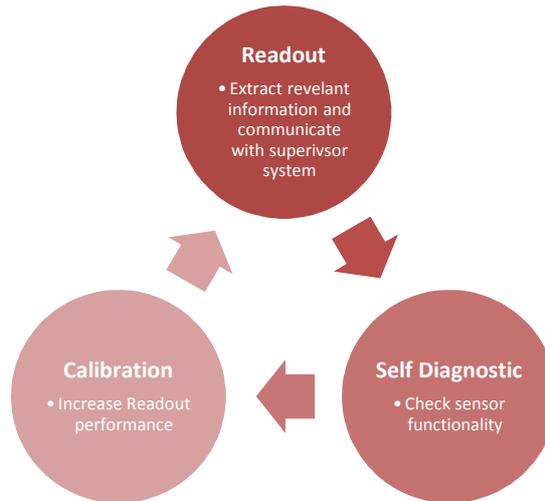
The architecture that has been defined will provide the 3 main following functionalities :

6. **Readout:** Design of electronic interface that provide an accurate readout of the useful values from each cantilever of the sensor array. The parameters that are measured are the resonance frequency of each cantilever as well as the relative amplitude at resonance. Information on each sensor of the array is communicated to the supervisor system.
7. **Self-Diagnostic:** Automatically characterization of each sensor of the array by getting its full spectral response to a monotonic harmonic excitation at different moments of the fabrication and during the life-cycle of the sensor. This is to detect any fabrication fault and

monitor the sensor’s behaviour during time with the perspective to inform the user that the sensor is not usable anymore. Furthermore the electrical characteristics of the sensor are also measured so that they can be compensated in the calibration sequence. This information is also indicative of the aging of the sensors.

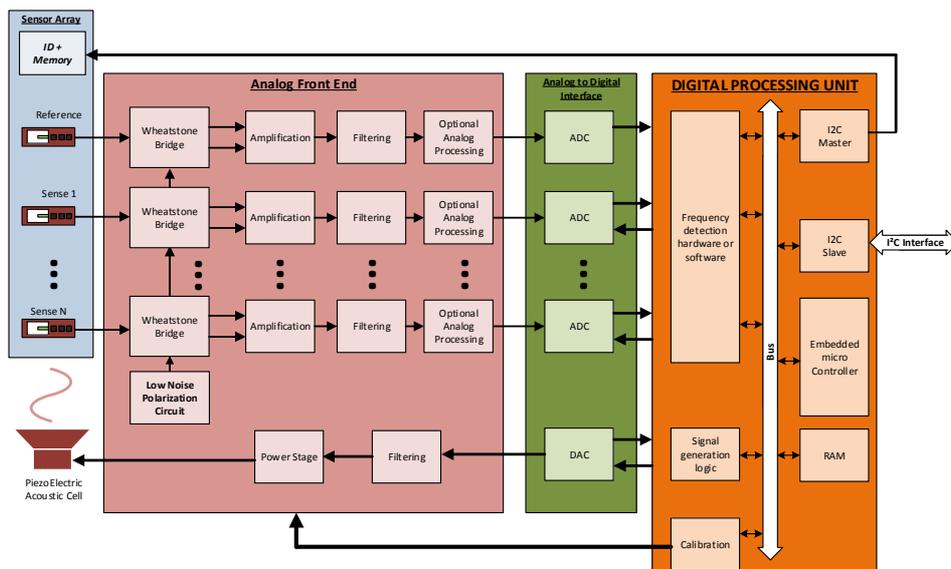
- Calibration:** Using the information from the self-diagnostic function to calibrate the electronic interface to get the best possible (highest SNR) readout from the sensor

These goals can be related to each other as shown in **Error! Reference source not found.** It is expected from this process to calibrate the whole system to get the best possible readout by using the information gathered during self-diagnostic.



*Interaction between the 3 main functionalities of the sensor electronic sub-system*

The block diagram presented shows the general architecture used to implement the sensor analog front end architecture :



*General architecture of the electronic sensor interface sub-module*



Each cantilever of the sensor array is connected to a Wheatstone bridge structure that is polarized using a dedicated low noise polarization circuit to insure maximum signal to noise (SNR) ratio. The output of the Wheatstone bridge is amplified by a low noise instrumentation amplifier (1.5 nV/√Hz at the operating frequency range and gain) before being filtered to focus on the frequency band of interest.

Optional analog processing can also be implemented to directly extract information in the analog domain. This might prove to be useful if the digital counterpart of such processing is too expensive in terms of size, power consumption or development time.

This signal is digitized by an Analog to Digital Converter before reaching the digital processing unit. The digital processing unit is a Cortex M3 processor implemented in a LPC1768 microcontroller from NXP. A coprocessor unit based on Cortex M4 with floating point calculation capability has also been successfully interface with the sub-system. It is programmed to fulfil the functionalities described previously. It also creates a communication link with the supervisor system to receive instructions and provide the resonance frequency of each sensor.

The cantilevers are excited all together by a single piezo-electrical acoustic cell driven by a high current amplifier stage. The excitation signal is directly generated by the Direct Digital Synthesiser (DDS) controlled by the digital processing unit and then low-pass filtered.

The digital processing unit is be able to control parameters from the Analog Front End (AFE). By this mean it will individually optimize the acquisition of the resonance frequency of each cantilever.

## 9. V2 Sensor electronic interface prototype design and validation

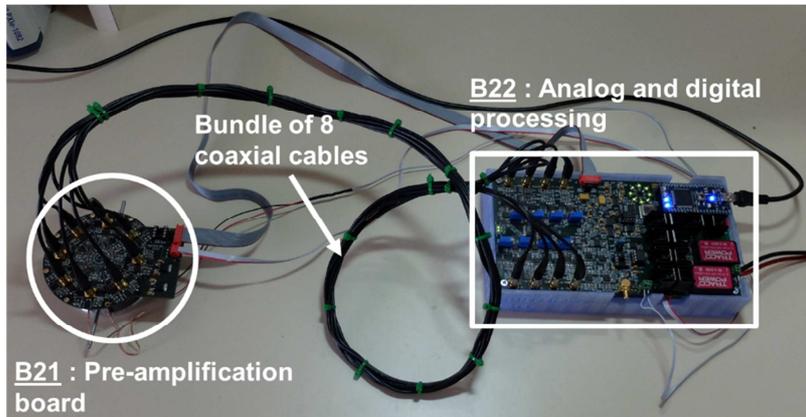
The generic architecture described in the previous section has be declined in 2 versions: V1 for the first period of the project and V2 for the second period. The V1 prototype of the subsystem has brought the proof of concept of the ability of this architecture to correctly address an array of diamond resonant sensors. Considering the challenges of the project and to reduce design risks, it has been decided to work iteratively on successive electronic prototypes. Each of them have implemented and validated each functionality of the sub-module. The V1 prototype architecture is shown on **Error! Reference source not found.**. It was only able to work with an array of 4 sensors and all the trimming and calibration steps were done manually. The cantilever-based sensors are inserted in a Wheatstone bridge structure with a very low noise polarization circuit. The difference at the output of the bridge is amplified by a first low noise amplification stage with a static gain of 2000 due to very low signal level ( $\mu\text{V}$  range). This is followed by a second variable gain amplification aimed to trim the signal amplitude to a level useable by the rest of the acquisition chain. The signal from each sensor is then bandpass filtered, its RMS value extracted and digitized before being processed by the NXP LPC1768 microcontroller. This microcontroller is also controlling the DDS that generates the signal used to drive the piezo acoustic cell below the gas cell. Separating the V1 architecture in three boards is aimed to simplify the validation of each functional block and to improve reusability of the boards over the whole project. This approach proved to be inefficient as assembling the 3 boards within the full integrated device on top of the gas analysis cell was difficult. V1 subsystem was also prone to power supply noise injection form the rest of the system into the low noise analog part of the circuit.



disassembly work.

- Oversize the heat dissipation elements of the power supply circuit to cope with the system internal temperature.

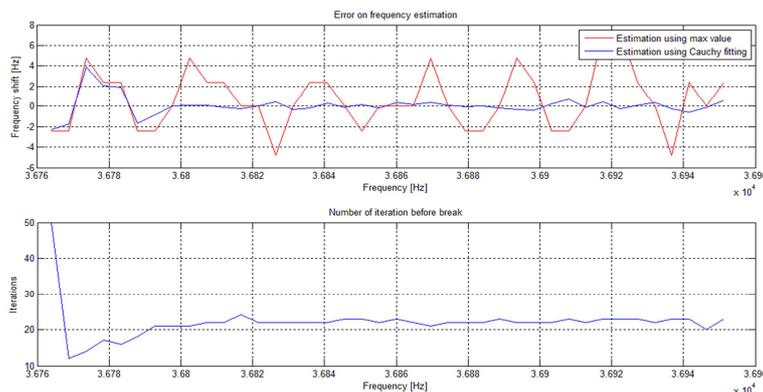
The assembly of the gas cell, contact board (B20), pre-amplification board (B21) and signal processing board (B22) is shown on **Error! Reference source not found.**



Picture of the V2 sub-system

### 12. Digital signal processing algorithm for resonant sensor frequency detection

On the V1 prototype of the sub-system, the detection of the resonance frequency was done by detection of the maximum of the extracted profile. This method has proven to be working but the noise level on the frequency shift detection (at baseline) was between 10Hz to 30Hz depending on the quality of the sensor response. In the second period, beside from the optimization of the hardware, we have focused on developing a digital signal processing algorithm based on function fitting to filter and optimize the frequency shift detection. The solution that has been chosen is based on fitting the frequency profile curve to a Cauchy function. The approach has been validated theoretically and implemented in real time on a Cortex M4 co-processor. **Error! Reference source not found.** shows the noise level on the frequency shift can be brought below 1Hz using this method.



Comparison of frequency shift error for a cantilever sensor resonance frequency shifting from 36,76kHz to 36,95kHz.

More practically, this digital signal processing sequence bring to the system the ability to exploit efficiently signals from sensors with a signal to noise as low as 5dB. **This a tremendous 22dB gain compared to V1.**

From a system functionality point a view, the whole V2 sub-system is able to reliably follow the resonance frequency of up to 8 sensors at a time with a resolution of a 1Hz and with 1 reading a



second. An example of sensor array response is shown on **Error! Reference source not found..**



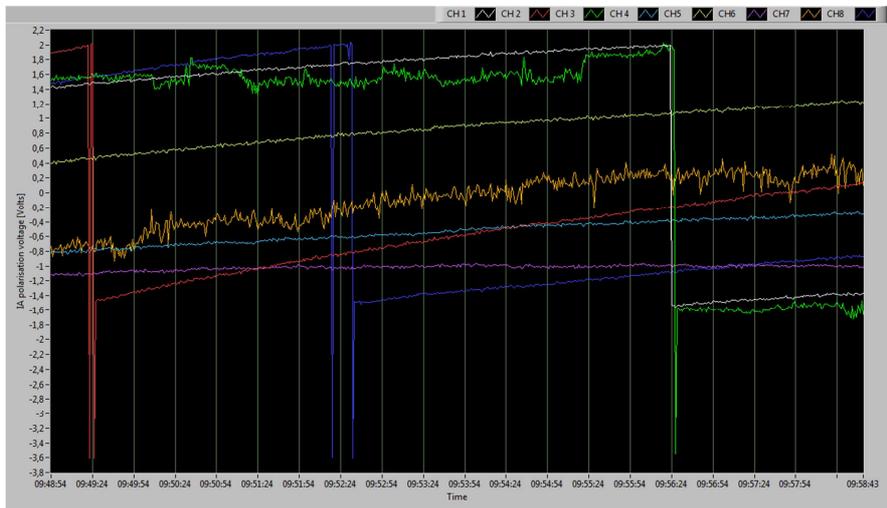
Example of sensor array response detection using V2 electronic sub-system

**13. Solutions for calibration and self-diagnostic**

During the second period of the project, we introduced digitally controllable elements in the analog front end that brought the following functionalities:

**14. Bridge compensation:** Continuous balancing of the Wheatstone bridges.

This brings robustness to the measurement because the electrical equilibrium of the sensors is re-adjusted before every measurement (i.e. every second). The modifications of temperature or humidity in the gas analysis cell due to the sampling processing are not more corrupting the signal from the sensors. An example of this continuous calibration is shown on **Error! Reference source not found..**



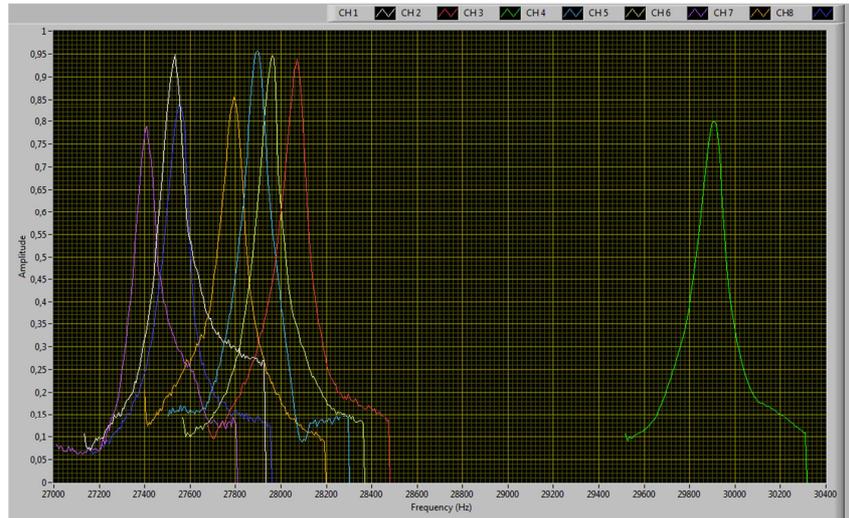
Example of cantilever array calibration voltages. Even if the bias voltage at the output of a sensor is drifting because of environmental parameters modification ( $T^\circ$ , humidity, ...), sensor polarization is continuously re-calibrated

**15. Amplification gain setting:** Continuous adjustment of the amplification stage for a 1V normalized signal.

Each cantilever sensor in the array has a different electrical response because of the



manufacturing dispersion. The amplitude of the sensor can also be modified by temperature changes or mechanical dispersion during the mounting/unmounting of the array in the analysis chamber. By using a digitally controlled amplifier in the analog front end of the detection circuit, the amplitude of the signal at resonance of the sensor of the array can be normalized between 0,8 and 1V. The challenge in designing this feature lies in the introduction of digital control signal in the very sensible low noise analog front-end.



*Frequency profiles of the 8 resonant sensors of the array. Each sensors is individually amplified with a specific gain so that the global response of the array is normalized to between 0,8V and 1V.*

**16. Bandpass filter setting:** Continuous adaptation of the low-pass filters to reject any noise above the resonance frequency

Each of those calibration functionalities are possible because the characterization sequence occurring at system start up.

*Task 7.2 – Reference samples for calibration*

The calibration process, as developed in SNIFFER, is an innovation in border security applications. The actual systems currently employed in this field often require to be handled by experts and it is practically impossible to understand, easily and on-line, if they are working properly and to resolve malfunctions.

The calibration that has been defined will use a self-diagnostic module, implemented in the sniffers, to carry out an easy optimisation of the sensor performances in case degradation is detected. The evaluation of the sensor performances is achieved by acquiring the full spectra response of the array of the reference samples and comparing them with the responses of the same substances acquired after grafting of the OBPs, stored in a database and used as sensor fingerprints. Substances with similar molecular characteristics as the ones targeted by the V2 prototype are needed for the calibration but also need to be selected so that they are not dangerous to work with.

Several substances have been selected as potential reference samples for the calibration of V2 devices, and are summarised in the table below.

Reference substance	Type
2-nitro-p-cymene	Similar to TNT



Musk ketone	Similar to TNT
Piperonal	Precursor of Amphetamines
Safrole	precursor of MDMA
Procaine	similar structure to cocaine
p-nitrophenol	odorant
Carvone (-)	odorant
Carvone (+)	odorant
3-octanol	odorant
IBMP	odorant
o-nitrophenol	odorant

*Reference samples for the calibration of V2 devices*

#### Significant results:

- Definition of an evolutive architecture that lead to the V2 prototype with readout, characterization and calibration elements. It is a highly agile architecture that can be declined to other kind on resonant sensor and for a very large range of applications.
- The challenge of provide a mechanical and electrical solution to access an array of 8 resonant sensors within a gas analysis cell has been successfully solved by the design of an innovative gas analysis cell with dedicated electrical interface.
- A robust, agile and fully functional prototype of the V2 prototype is now used for provide sensor data from sensors within a whole system designed for trace detection based on biomimetic odor detection.
- Characterization and self-diagnostic hardware and procedure have been defined and implemented. They are the key elements in providing information on the reliability of the sensor array as well as for correcting in real time any drift in there operation.
- Furthermore, a specific digital signal processing algorithm has been developed. It allows the systems to monitor the resonance frequency of each sensor of the array with a resolution of 1Hz while keeping the measurement rate up to 1 sample per second (8 resonant sensors data/sec).

**Deviations:** None

## 3.8 WP 8 Dissemination & Exploitation

<b>WP number</b>	8		<b>Start date or starting event:</b>	M1
<b>WP title</b>	Dissemination & Exploitation			
<b>Activity type</b>	OTHER			
<b>Participant ID</b>	1	3	12	14
<b>Part. short name</b>	CEA	ST(SI) <sup>2</sup>	3DSA	ARTTIC
<b>PM total (for 60M)</b>	2	4,75	3	15
<b>PM spent in period 1</b>	1	3	0	5,44
<b>PM spent in period 2</b>	1	1,50	3,10	12,80
<b>PM spent total</b>	2	4,50	3,10	18,24

### Work progress in each task

#### *Task 8.1 Dissemination activities*

The SNIFFER partners took advantage of their network to spread news about the project, developed technologies as well as the European FP7 funding and collaboration frame. To support their actions, a few months after project launch, the project dissemination plan, from which all dissemination actions were implemented, including the communication strategy, was defined by all partners.

The SNIFFER Project dissemination strategy relied on diverse communication materials. It included the items listed below.

- The project logo was drawn at earlier stages of the project. 4 options were presented to the partners who voted for the one to be reproduced on every SNIFFER document, including this deliverable (at the foot of the page). The project identity was then built around this logo, its spirit and colors. It resulted in the production of the project presentation / communication templates to be used by all partners when communicating about SNIFFER.
- The project public website ([www.sniffer-project.eu](http://www.sniffer-project.eu)) which was launched to the public July 6<sup>th</sup>, 2012.
- The project printed material covered a standard poster and leaflet. Their finalisation and printing were delayed due to the fact that the dissemination team waited for promising technical results to be included in their content. They were distributed to partners to support their speech at conferences at mid-project.

During the course of the project and along project findings and promising results, partners made research developments and outcomes available to the scientific community through 6 scientific publications, mainly in peer-reviewed journals. The list of publications is available in the final reporting of the project.

Besides the information given through the SNIFFER public website, the general public was also informed of SNIFFER outcomes and research domain through 2 press releases in widely spread journals.

Specific actions were taken to reach one specific target, the end-users, beyond task 8.3. During the last year of the project in May 2014, SNIFFER attended the FRONTEX European Day for Board Guard



in Warsaw. This event was the opportunity to meet with FRONTEX national representatives, “member states”, and to raise awareness among them of the SNIFFER developments and results. Beyond the networking opportunity with end-users, the ED4BG was also the opportunity to meet with other represented FP7-SEC projects. The final field trials in the airport environment were eventually the opportunity to further reach the end-users. They took place at Athens International Airport during March 2015 for 3 days. They revealed a genuine interest by airport authorities and the police for commercial devices based on SNIFFER technologies.

Also, SNIFFER partners were encouraged to participate in conferences and events related to the field covered by SNIFFER: specific technical bricks (e.g. proteins); artificial sniffing in general, border security, etc. At the end of the project, 16 of such participations have been counted all around Europe. Talks and lectures at national and international conferences were supported by the above described communication material.

Eventually, collaboration with other RTD projects was investigated from the start of the project. Early efforts paid off at project midterm when the SNIFFER Project built stronger links with 2 FP7-SEC “twin” projects (DOGGIES and SNIFFLES) who attended the first SNIFFER public event. Regular contact was made, as well as further networking for clustering with other RTD Projects. SNIFFER was represented at FRONTEX European Day for Border Guard in Warsaw in May 2014, which was a great opportunity to investigate further interactions with end-users and other projects. It resulted in strong links with FP7-SEC projects SNOOPY and HANDHOLD, which actively participated in the SNIFFER final public event. Furthermore, SNIFFER investigated other interactions with RTD projects such as OLAF Rats II, which deals with border security issues and illegal substances detection through rats.

#### *Task 8.2 Exploitations and IPR Management*

The IPR team was assembled based on General Assembly members’ votes. It never met as there was no IPR conflict.

Concerning Exploitation, first discussions on the exploitation plan have begun at project mid-term and are reflected in D821. This initial exploitation plan was further discussed up to project end and led to promising exploitation perspectives. DL 822 reflects that, at project end, the exploitation activities related to SNIFFER results have already started or will do so in the near future. One patent has been issued 04 April 2014 under the reference “FR1000235135” related to Gas sensing cell with remote piezo- actuation of the cantilever sensors. Another patent related to Diamond MEMS with Polysilicon gauges is to be submitted in the near future. 9 major avenues for commercial exploitation are listed in the document as well as non-technical results.

#### *Task 8.3 User Advisory Group*

The UAG was established at project start with the objective to gather experts coming from organisations involved in operational security interested in the SNIFFER technology. It first met in Manchester in July 2012. Some representatives from Athens airport (Greece) and the French customs were invited to give their input in the project. Each of them presented their organisation and the different devices they were used to develop for security on the airports (See DL 831).

The ST(SI)<sup>2</sup> identified some new partners to join the UAG. SNIFFER made contact with Frontex (European agency dealing with security on borders) which is interested mainly by the detection of human beings (illegal migrants) but also the German police and the UK border unit.

The UAG was then understood in broad considerations: it did not only cover the UAG members *stricto*



*sensu* (involved on a regular basis during the 3 years of the project) but also the overall group of end users that, somehow, got punctually involved in the SNIFFER Project by providing a specific input during an informal conversation, punctual help to cover specific project tasks, or simply followed the SNIFFER project through its public events.

During year 3 of the project, another UAG-specific physical meeting did not seem bringing added-value to the project developments. Regular informal meetings, email and phone exchanges between the UAG (in its broad understanding) with some SNIFFER Partners as interfaces for the SNIFFER consortium proved sufficient. The SNIFFER end-user partners were greatly involved in the project. The Greek partner 3DSA, the French Ministry of Interior (ST(SI)<sup>2</sup>) and its police laboratory (CREL) as well as the Israel National Police (INP) proved being an important relay of information towards a broader range of end-users. Also partner ARTTIC, in charge of dissemination activities, made regular contact with its end-user data base built along SNIFFER in order to create and maintain awareness of the SNIFFER Project and in view of optimised attendance of the final SNIFFER public event. These actions were successful as proved by *ad hoc* solicitations from stakeholders in the field to join the SNIFFER UAG.

The outcome of this regular feedback is reflected by the orientation taken by the SNIFFER version 2 developments (e.g. selectivity of the sensors). It is also reflected by the extensive lab trials ran during the second half of the project, and the final field trials held at Athens International Airport at project end. Concerning this final event, the involvement of UAG member AIA, namely Mr Nikas Anastasos, is to be highlighted.

Eventually, the SNIFFER final public workshop organised at project end in May 2015 in Paris was a meeting point between the project and the end-users that followed project developments as well as others interested in the SNIFFER technology. The main outcomes, the informal exchanges, followed the SNIFFER results presentations (questions asked to the presenters but also informal discussions). Each SNIFFER partner had the opportunity to discuss its specific part with the concerned end-user during the breaks and poster sessions inserted for this purpose in the programme between presentations. The two panel discussions also targeted the positive confrontation between research and end-users, encouraging feedback, comments, suggestion and debate. This closing event was also the opportunity to discuss future perspectives for upcoming research projects. Indeed, the closing panel discussions aimed at considering broader perspectives with regards to artificial sniffing and its applications in the security domain: both the research and the end-user aspects were presented.

#### *Task 8.4 SNIFFER User Workshops*

The SNIFFER Project organised two public workshops at early year 3 of the project and at its end. They were both successful. The first public event focused on the rationale of such a SNIFFER R&D Project, from the dog handler to the research point of view; whereas the second event – presenting final results of the project – targeted the end-users and “client” of the SNIFFER Project. Specific reports for each event have been written and cover their organisation and outcome in detail.

#### **Significant results:**

- Dissemination plan successfully implemented
- Regular feedback and input from UAG (considered in its broad understanding): all UCs were covered by the collected feedback
- 2 Public Workshops organised
- Exploitation defined and promising for a strong SNIFFER impact

#### **Deviation**

<b>Description of problem / deviation</b>	<b>Suggested / undertaken</b>	<b>Impact on other</b>
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	<b>corrective action</b>	<b>WPs, budget/PM plan or work plan</b>
Public Workshop postponed (waiting for concrete results – field trials and integrated V1 prototype)	Postponed event, organisation to be started now to reach a broader audience	None
Overconsumption of human resources at ARTTIC	Partner internal shift from “other direct costs” envelop to “personal costs”	None

## 3.9 WP 9 Project Management

<b>WP number</b>	9		<b>Start date or starting event:</b>	M1
<b>WP title</b>	Project Management			
<b>Activity type</b>	MGT			
<b>Participant ID</b>	1	14		
<b>Part. short name</b>	CEA	ARTTIC		
<b>PM total (for 60M)</b>	1	17		
<b>PM spent in period 1</b>	0,56	8,15		
<b>PM spent in period 2</b>	0,44	12,70		
<b>PM spent total</b>	1	20,85		

### Overview of the progress of the work during the period

ARTTIC, the Project Office, and CEA, the Project Coordinator, work closely together and shared project management tasks and responsibilities.

At project launch, the Project Office put into place web-based tools for partners to exchange information, be informed of what is expected from the consortium, and be continually updated on project progress. The collaboration between partners was excellent and partners were very involved and active in preparing scientific deliverables.

Suitable management bodies and procedures for the project were set-up. The General Assembly (GA) has met six times as planned, and the Executive Board (EB) held a teleconference mostly every month. All meeting minutes and deliverables were delivered on time except for a few justified delays. Contractual documents have been agreed upon and signed by all partners, and shared with the EC when needed.

### Work progress in each task

#### *9.1 Strategic management and financial administration*

All contractual documents were established, signed and shared with all project partners and the EC if needed during the first months of the project.

The pre-financing and interim-payment shares were distributed to all partners according to the agreed timeline and breakdown and in line with the Grant and the Consortium Agreements rules.

In terms of strategic management, at project start, the SNIFFER consortium set up its GA, which met twice a year for a consortium meeting, and its EB, which met every month for a project management follow-up teleconference. SNIFFER had all tools needed for success however faced some issues along its course leading to necessary evolution of the work plan and partners' responsibilities:

- Amendment of the DoW following the midterm review: taking the most of the allocated time and budget through a focused scope and realistic implementation
- Partner CSSC's voluntary liquidation at the start of project year 3
- A major technical issue: Partner TTS's risk of being defaulting party and not delivering UC6 sampling device
- Partner EADS's change of strategy resulting in less integration responsibilities for SNIFFER
- Numerous difficulties leading to a 4-month project extension

Thanks to the efficiency of project management process, these difficulties were identified early enough to be dealt with in the appropriate manner and timing. They were all overcome and eventually did not harm the project success at its end, however leading to quite time and budget consuming activities.

### 9.2 Project Management Office

Project management guidelines were agreed upon and implemented at project start. They defined action items for each objective to be dealt with on a daily basis or as necessary. These action items were adjusted as needed during the project to match the project requirements and learn from past lessons.

Besides the initial set up of the project management process, methods and tools, regular milestones were to be met during the project to assess the efficient project management. The calendar as well as the scope of the project were slightly redefined at project midterm in order to better fit to what was realistically possible, considering the status of the SNIFFER developments at this stage (e.g. specific focus on 2 usage cases), and not to lose time with little added-value actions (e.g. replacing midterm field trials by extensive lab trials). Regular feedback was put in place leading to regular updates of the implementation plan and the corresponding action lists for both the managing team and the partners.

By doing so, calendar, quality and scope were respected despite difficulties such as the voluntary liquidation of a partner or the change of strategy of another one. Due to internal restructuring and challenging engineering feats for integration, the consortium and European Commission Project Officer agreed upon a 4-month extension to the project.

Eventually, as the SNIFFER project came to its end with successful technical results, promising exploitation and fruitful further collaboration in between partners, it is to be noted that efficient project management was successfully achieved during the project duration.

#### Significant results:

The most significant result of WP9 is demonstrated in the fact that the collaboration between partners was very good, and sometimes even excellent.

The SNIFFER project management was carried out on a day-to-day basis, relying on web-based tools available at all times to all partners. The Project Management guidelines including a management plan were established at project start and agreed by all partners (D921). The management bodies were elected and meet on a regular basis. More precisely, to monitor the project, a risk register has been established and is regularly updated (first iteration shared with the EC through D922). Internal reporting is monthly done.

#### Deviations

Description of problem / deviation	Suggested / undertaken corrective action	Impact on other WPs, budget/PM plan or work plan
Technical results needing a re-focus of project scope for efficient use of allocated time and budget	Discussions at consortium level, GA and then during the mid-term review meeting with EC PO and reviewers	See DoW Amendment #1
CSSC voluntary liquidation	Reconsideration of ethical aspects, also with regards to scope refocus	Extra budget for technical work and field trials

Partner TTS's risk of being defaulting party and not delivering UC6 sampling device	Intense project management, delayed interim payment upon evidence of work, web demonstration of the developed device working + internal testing at partner INP	Potential failure of UC6
AIRBUS (Former EADS) change of strategy	Involvement of a new laboratory of CEA in the project: CEA-DACLE + further involvement of UNIMAN with regards to integration work	Delay in the technical developments and integration work + Impact on future exploitation
Delay in technical results	Project extension of 4 months	Failure to provide final assessment of the fully integrated devices

### **3.10 Potential impact (including the socio-economic impact and the wider societal implications of the project)**

Many sensitive controls are carried out at border checks by trained dogs, which cannot be neither replaced, nor be expected to operate on a 24/7 basis. SNIFFER devices cover the variety of border security situations (contexts and targets) where dogs are used today. The devices, once developed as market products, will be able to operate on a 24 hours/7 days per week basis and allow dogs to be used in situations where they can potentially make a difference.

SNIFFER, in particular with the V2 technology developed during the 2<sup>nd</sup> period of the project, proved being able to go farther than dogs as the sensitivity of SNIFFER devices will keep increasing. Dogs are also unable to push air onto objects to collect particles or gas molecules as is intended for the automatic artificial sniffer portal proposed by SNIFFER to control suitcases on luggage belts. Through these achievements, border security would be significantly enhanced as the difficulty for illegal traffics of all kinds (drugs, tobacco, illegal immigration...) to cross border lines, especially in airports, would dramatically increase. At the same time, border security would be improved as regards to possible terrorist attacks. Although SNIFFER focused on specific usage cases leaving aside container checking and human odour detection, the project results are quite promising with regards to these potential exploitation fields. It is to be noted that the SNIFFER technology will be further developed through the H2020 Project C-BORD where some of the SNIFFER submodules will be adapted and tested for the detection of illicit substances in containers. In general, the expected impact of SNIFFER will contribute to the overall field of security. Many security applications would benefit from the high-sensitivity low-cost technology provided by SNIFFER (in comparison with COTS), for example: the search of casualties after a disaster, the detection of dangerous gases close to an industrial facility or the search of drugs by police forces. Indeed the SNIFFER instrument developed is much promising and a mature result. As a sensing electronic submodule capable of sensing substances it can be exploited in many markets such as that of the access control in every critical infrastructure and use of the equipment from law enforcement agencies.

Furthermore SNIFFER will contribute to many other application fields such as: health (breath analysis, other body odours, faeces), food safety (e.g., to detect problems with recycled bottles) and environmental monitoring (e.g., air and water quality). SNIFFER results obtained during the process development of OBPs may be helpful in the case of development of other OBPs for other applications such as pharmaceuticals, diagnostic, environment, biotechnology and nanotechnologies, industrial monitoring. Moreover, the development of MUP targeted to different analytes can lead to the development of sensors based on different technologies, which may be used in various fields (pharmaceuticals, environmental). Also, the innovative characteristics of the SNIFFER work sits in the durability and re-usability of the sensors which has shown the potential for evolution of the system for other applications. Another field of application will be the detection of volatile organic compounds for medical diagnostic and air/water monitoring. At industrial level, broader potential and outcome from the OBP-sensing- and ESP-sampling developments can be envisaged. The technology by its own could also be used in testing the air quality as well as to test health and safety issues in structure and manufacturing processes for instance.

Specific individual plans for exploitation of project results are summarized below.

Major avenues for commercial exploitation include the following list of SNIFFER enabled products:

- The **microcantilever based Multisensor chemical detection module (CEA, UNIMAN, ESIEE)** addresses the market of multiple sensor applications (potential of 500k€ in 5 years). As it reached TRL 5 at the end of the SNIFFER Project, time to market is estimated to 3 years. Competitive assets sit in the complementary and concurrent design of both the sensors, the support electronic and the odour detection algorithm.
- The **diamond MEMs with Polysilicon gauges (ESIEE)** address the market of integrated sensor industrial for specific application. It involves innovative and complementary process between partners CEA and ESIEE. The potential is evaluated to be 2M€ in 3 year-time from now.
- The **recombinant proteins (UNIMAN)** reach pharmaceutical, environmental, security sectors for which the size of market is still to be defined. Potential has however been estimated to 1M€ in 3 years, up to 5M€ in 5-10 years. Current competitive assets are the novel, stable biorecognition elements as well as SNIFFER partner available facilities for production.
- The **SNIFFER Instrument (UNIMAN)** could reach pharmaceutical, environmental and security markets in 3 year-time. Potential has been estimated to 1M€ in 3 years, 2M€ in 5 years and up to 5M€ in 10 years.
- The **pre-concentrator components (ARMINES, EPFL)** address the market of small detection devices for specific applications (environment, security). The main barrier to market sits in getting the preconcentrators to be manufactured. A major competitive asset is the acquired expertise on the manufacturing and the user of pre-concentrator components.
- With regards to **OBPS, Ligand Binding Proteins (GTP)** will address the pharmaceuticals, diagnostic, environment, biotechnology and nanotechnologies and industrial monitoring market in 2 to 3 years. Potential is estimated to 1M€ in 5 years. A major asset is the new protein biosensors. **Major Urinary Proteins (UNIPD)** will address the pharmaceutical, environmental and security markets, also possibly generating 1M€ in 5 years. The major asset is the custom-tailored proteins for biosensors.
- The **ESP-sampling and thermos-desorption system (Airbus Group Innovations)** will reach new fields of application beyond the security field through the detection of failure or safety issues with regards to carbon fibre manufacturing processes.
- The **SNIFFER Monitoring portal (TTS)** reaches the major transportation hubs, shopping centres, government buildings, sports and other entertainment arenas markets. Its integration into 4 TTS products – ShoeSafe, CompactSafe, CarrySafe, and CargoSafe – would generate revenues from \$375 000 in 3 years for ShoeSafe to \$40 000 000 in 10 years for CarrySafe and CargoSafe. There are almost no competitors with the same level of technology for particle extraction. With the use of an activated carbon filter, it is also possible to extract vapors from the inspected baggage.

There are also SNIFFER non-technical results developed with intention to exploit following the project, in order to optimise the SNIFFER impact. In particular, this includes the potential for partner 3DSA to take on a role as consultant for SNIFFER users following the project. This will include consulting and training services, as well as optimisation of security protocols and policies for SNIFFER devices. It also includes partner ARTTIC activity, as involved in other FP7 and H2020 projects as part of its core business, which intends to keep up the work carried out during SNIFFER dissemination activities. The objective will be for the community built around SNIFFER (end-users, research, institutions interested in artificial sniffing and/or detection for border security) to benefit from its network in future projects / events in the field.

With regards to patent, patent on “Microcantilever based Multisensor chemical detection module” has been filed during the project between CEA/ESIEE-Paris/UNIMAN. This could be exploited through

licensing to a third party of through the creation of a start-up company supported at least by CEA depending on the results from market study.

Partner UNIMAN also envisages to develop new biosensors based on OBPs, build further EU project proposals in the security area based on SNIFFER developments to take TRL levels to TRL 6 and above and publish scientific papers with partners CEA, UNIPD on new developments on OBPs and MUPs for chemical sensing. Partner EPFL is approaching companies developing portable gas analyser systems for technology transfer of the gas preconcentrator developed on silicon and envisages the development of polymeric based disposable gas detection systems based on printing technology.

Eventually, the technology developed by CEA and other partners within SNIFFER will be further developed through the H2020 project C-Bord.

## 4 Use and dissemination of foreground

### 4.1 Section A

#### 4.1.1 List of scientific (peer reviewed) publications

#	Title	Main author	Title of the periodical or the series	Number, date or frequency	Publisher	Place of publication	Year of publication	Relevant pages	Permanent identifiers <sup>1</sup> (if available)	Is/Will open access <sup>2</sup> provided to this publication?
1	One nose, one brain: contribution of the main and accessory olfactory system to chemosensation.	<b>Mucignat-Caretta C</b> , Redaelli M, Caretta A	Frontiers in Neuroanatomy	6		online	2012	46	1. doi: 10.3389/fnana.2012.00046	yes
2	Biomimetic Olfactory Sensors	Persaud, K.C.	IEEE Sensors Journal	Vol 12 (11)	IEEE		2012	3108-3112		No

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- 1 A permanent identifier should be a persistent link to the published version full text if open access or abstract if article is pay per view ) or to the final manuscript accepted for publication (link to article in repository).
- 2 Open Access is defined as free of charge access for anyone via the internet. Please answer "yes" if the open access to the publication is already established and also if the embargo period for open access is not yet over but you intend to establish open access afterwards.

#	Title	Main author	Title of the periodical or the series	Number, date or frequency	Publisher	Place of publication	Year of publication	Relevant pages	Permanent identifiers <sup>1</sup> (if available)	Is/Will open access <sup>2</sup> provided to this publication?
3	From Sniffer Dogs to Emerging Sniffer Devices For Airport Security: An Opportunity to Rethink Privacy Implications?	Matteo E. Bonfanti	Science and Engineering Ethics.	Forthcoming in 2013	Springer	...	2013	...	...	No
4	Development and characterization of an electrostatic particle sampling system for the selective collection of trace explosives	S. Beer	Talanta		Elsevier		2012		Talanta. 2012 Jan 30;89:441-7. doi: 10.1016/j.talanta.2011.12.059. Epub 2011 Dec 28.	
5	Structure and biotechnological applications of odorant-binding proteins.	Pelosi, Paolo, et al	<i>Applied microbiology and biotechnology</i>	98.1, 2014	Springer		2014	61-70		

#	Title	Main author	Title of the periodical or the series	Number, date or frequency	Publisher	Place of publication	Year of publication	Relevant pages	Permanent identifiers <sup>1</sup> (if available)	Is/Will open access <sup>2</sup> provided to this publication?
6	Grafting odorant binding proteins on diamond Bio-MEMS	Raafa Manai, E Scorsone, L Rousseau, F Ghassemi, M Possas Abreu, G Lissorgues, N Tremillon, H Ginisty, JC Arnault, E Tuccori, M Bernabei, K Cali, KC Persaud, P Bergonzo	Biosensors and Bioelectronics	Vol.60	Elevier	Journal	2014	311-317	NA	No

#	Title	Main author	Title of the periodical or the series	Number, date or frequency	Publisher	Place of publication	Year of publication	Relevant pages	Permanent identifiers <sup>1</sup> (if available)	Is/Will open access <sup>2</sup> provided to this publication?
7	Major Urinary Proteins on nanodiamond based resonators toward artificial olfaction	Emmanuel Scorsone, Raafa Manai, Maria Jimena Ricatti, Marco Redaelli, Philippe Bergonzo, Krishna C. Persaud, Carla Mucignat	Submitted to the review "ACS Nano"							
8	Preconcentration Modeling for the Optimization of a Micro Gas Preconcentrator Applied to Environmental Monitoring	Malick Camara	Analytical Chemistry	87	American Chemical Society		2015	4455-4463	DOI: 10.1021/acs.analchem.5b00400	

### 4.1.2 List of dissemination activities

Activity no.	Type	Title	Exact Date	Location	Impact observed (*)	Contribution to the project	Presentations by SNIFFER partners, who presented	Partners involved
1	International Conference	The International Conference on Diamond and Carbon Materials,	Sep 03-06, 2012	Granada, Spain	Scientific Community	Presentation to experts on synthetic diamond based sensors and acknowledgment of the SNIFFER project.	Title of Presentation: "Odorant binding proteins for chemical sensing" Raafa Manai	CEA
2	International Conference	Conference: IUMRS-ICEM 2012	23-28 September 2012	Yokohama, Japan	Scientific Community	Presentation to experts on synthetic diamond based sensors and acknowledgment of the SNIFFER project.	Oral presentation "Resonant diamond MEMS biosensors for trace detection in liquids" Emmanuel Scorsone	CEA

Activity no.	Type	Title	Exact Date	Location	Impact observed (*)	Contribution to the project	Presentations by SNIFFER partners, who presented	Partners involved
3	International Conference	The Amsterdam Privacy Conference 2012	8 October 2012	Amsterdam	Scientific Community/Civil Society/Policy Makers	Official presentation and acknowledgment of the SNIFFER project.	Title of presentation: "Do Sniffer Dogs Respect Individuals' Privacy more Effectively than Emerging Sniffer Technologies for Security Checks at Borders and at other Critical Installations?" Matteo Bonfanti	CSSC

Activity no.	Type	Title	Exact Date	Location	Impact observed (*)	Contribution to the project	Presentations by SNIFFER partners, who presented	Partners involved
4	International Conference	Biometric 2012	30 October 2012	London	Scientific Community/Civil Society/Policy Makers	Official presentation and acknowledgment of the SNIFFER project.	Title of presentation: "The future is now: fundamental rights and ethical implications of human odour-based biometrics for law enforcement and security purposes." Andrew Rebera	CSSC
5	International Conference	15th International Symposium Meeting on Olfaction and Electronic Nose Chemical Sensor IMCS 2012	20-23 May 2012	Nürnberg	Scientific Community	Obtained a positive feedback from expert colleagues on the method of electrostatic precipitation developed for the SNIFFER project.	Scientific Presentation: "A Particle Sampler for Trace Detection of Explosives" Sebastian Beer, Gerhard Müller, Jürgen Wöllenstein	EADS

Activity no.	Type	Title	Exact Date	Location	Impact observed (*)	Contribution to the project	Presentations by SNIFFER partners, who presented	Partners involved
6	International Conference	The 17th International Conference on Solid-State Sensors, Actuators and Microsystems.	June 16-20, 2013,	Barcelona. Spain	Scientific Community	Participation to leading international conference on chemical sensing. Technical discussions with other SNIFFER partners attending the conference.		CEA, ARMINES, EPFL, UNIMAN, ESIEE
7	International conference	XVI International Symposium on Olfaction and Taste	23-27 June 2012	Stockholm, Sweden	Scientific Community	Meet other SNIFFER Partners and other colleagues working in the Odorant Binding Protein field, to share concepts and ideas about the engineering of the proteins to be used for the SNIFFER Project, and to present to them our project.	"OBP biosensors for detecting Volatile Organic Compound in vapour phase" presented by Krishna Persaud, UNIMAN	UNIMAN (Krishna Persaud), UNIPD (Carla Mucignat)

Activity no.	Type	Title	Exact Date	Location	Impact observed (*)	Contribution to the project	Presentations by SNIFFER partners, who presented	Partners involved
8	International Conference	15th International Symposium Meeting on Olfaction and Electronic Nose Chemical Sensor	2-5 July 2013	Daegu	Scientific Community	Presentation to experts in artificial olfaction and acknowledgment of the SNIFFER project.	Scientific Presentation: "Immobilisation of Ligand Binding Proteins onto diamond surfaces for biosensing" Raafa Manai (CEA), oral presentation	CEA
9	International conference	UKSN meeting (UK semiochemistry Network)	30-31 July 2013	Cambridge	Scientific Community	Obtained a positive feedback from expert colleagues on the method of in silico validation developed for the SNIFFER project	Presentation of the SNIFFER project "Beyond the Biological Olfaction: a step towards the artificial sniffer"	UNIPD (Ricatti MJ, Redaelli M, Mucignat C)
10	Congress Abstracts	ECRO Congress	26-29 August 2013	Leuven	Scientific Community	Obtained a positive feedback from expert colleagues on the method of in silico validation developed for the SNIFFER project	SNIFFER Poster: Odorant binding protein activity prediction: the pathway for in silico screening	UNIPD (Ricatti MJ, Redaelli M, Mucignat C)

Activity no.	Type	Title	Exact Date	Location	Impact observed (*)	Contribution to the project	Presentations by SNIFFER partners, who presented	Partners involved
11	International Conference	IMCS 'International Meeting on Chemical Sensors' meeting: <a href="http://www.imcs2014.org/">http://www.imcs2014.org/</a>	16-19 March 2014	Buenos Aires, Argentina	Scientific Community	Preconcentrator development	EPFL :lecture (M. Camara) ARMINES: poster (C. Pijolat)	EPFL , ARMINES
12	International Conference	FRONTEX European Day for Border Guard (ED4BG)	22-23 May 2014	Warsaw, Poland	Create awareness on SNIFFER, network and interact with end-users		CEA: 1 person (E. Scorsone) for 2 days ARTTIC: 1 person (T. Deheunynck) the first day	
13	Symposium	Carbon materials: surface chemistry and biomedical applications of the E-MRS 2014 Spring Meeting	26-30 May 2014	Lille, France		Oral presentation of the paper published in "Biosensors and Bioelectronics" Journal	CEA: 1 person (R. Manai)	

Activity no.	Type	Title	Exact Date	Location	Impact observed (*)	Contribution to the project	Presentations by SNIFFER partners, who presented	Partners involved
14		UNIPD's Department Scientific meeting	30 May 2014	Padua, Italy		Brief presentation of SNIFFER in a broader presentation of the laboratory work at UNIPD	UNIPD: 1 person (C. Mucignat)	
15	Article in general press	The Huffington Post	26 June 2014	online		Un nouvel outil pour détecter explosifs et drogues dures	Press release	CEA ARTTIC
16	Congress	ECRO Congress	10-14 September 2014	Dijon France		Abstract: Major Urinary Proteins tailored to detect selected compounds as tools for developing biosensors	UNIMAN: 1 person (K. Persaud)	
17	Conference	EUROSENSORS 2014	7-10 September 2014	Brescia, Italy	Scientific Community	Preconcentrator development	EPFL :lecture ( M. Camara) ARMINES : Poster (C.Pijolat)	EPFL, ARMINES

Activity no.	Type	Title	Exact Date	Location	Impact observed (*)	Contribution to the project	Presentations by SNIFFER partners, who presented	Partners involved
18		CPEXpo: The EC security research conference - SCR 2014	9-10 December 2014	Genova, Italy	Create awareness around SNIFFER		UNIPD: 1 person (C. Mucignat)	
19	Conference	IEEE MEMS conference	18-22 January 2015	Estoril, Portugal	shared results on micro electro mechanical systems			ESIEE
20	Meeting	Presentation of the SNIFFER Technology to Athens International Airport and Hellenic Police	10-12 March 2015	Athens, Greece	Interest from both AIA authorities and Police Chemical Labs		All (3DSA Organised the event)	CEA, UNIMAN, TTS, 3DSA
21	Conference	CBRN	16-18 mars 2015	Nice , France	Scientific Community	Preconcentrator development	ARMINES :lecture (C. Pijolat)	EPFL, ARMINES
22	Project Workshop	DOGGIES Public Workshop	20-21 May 2015	Münster, Germany	create awareness around the project		presentation of SNIFFER made by Bruno Gérard	ARTTIC, CEA

Activity no.	Type	Title	Exact Date	Location	Impact observed (*)	Contribution to the project	Presentations by SNIFFER partners, who presented	Partners involved
<b>Upcoming events</b>								
23	International Conference	Transducers 2015 18 <sup>th</sup> International Conference on Solid-State Sensors, Actuators and Microsystems	21-25 June 2015	Alaska, USA			ARMINES EPFL	
24	International Conference	6th International symposium ISOEN on Olfaction and Electronic noses	29 June – 01 July 2015	Dijon, France		Emmanuel Scorsone from CEA, won the Wolfgang Gopel Memorial award	Lecture : Emmanuel Scorsone "Diamond micro-cantilevers as transducers for olfaction receptors-based biosensors: application to the receptors M71, OR7D4 and OR1740"	CEA

Activity no.	Type	Title	Exact Date	Location	Impact observed (*)	Contribution to the project	Presentations by SNIFFER partners, who presented	Partners involved
25	International Conference	European Chemoreception Organisation Congress (ECRO 2014)	01-05 September 2014	Dijon, France	Positive feedback from delegates of the SNIFFER project	Ricatti, M. J., M. Redaelli, K. Persaud, and C. Mucignat. "Major Urinary Proteins tailored to detect selected compounds as tools for developing biosensors." in <i>CHEMICAL SENSES</i> , vol. 40, no. 3, pp. 256-256. GREAT CLARENDON ST, OXFORD OX2 6DP, ENGLAND: OXFORD UNIV PRESS, 2015. Cali, Khasim, Mara Bernabei, and Krishna Persaud. "Development of mutant Odorant Binding Proteins (OBPs) for Security Applications: Detection of Explosives and Drugs." in <i>CHEMICAL SENSES</i> , vol. 40, no. 3, pp. 266-267. GREAT CLARENDON ST, OXFORD OX2 6DP, ENGLAND: OXFORD UNIV PRESS, 2015.	UNIMAN (Khasim Cali) UNIPD (Jimena Ricatti, Carla Mucignat)	UNIMAN UNIPD
26	Conference	EUROSENSORS 2015	6-9 September 2015	Freiburg, Germany	Scientific community	Preconcentration development	EPFL (M. Camara)	EPFL, ARMINES
27	Exhibition	Milipol 2015	17 November 2015	Paris, France	Media, policy makers, industry, military		Poster	CEA, ST(SI) <sup>2</sup>

## 4.2 Section B

### 4.2.1 Part B1

LIST OF APPLICATIONS FOR PATENTS, TRADEMARKS, REGISTERED DESIGNS, ETC.			
Type of IP Rights: Patents, Trademarks, Registered designs, Utility models, etc.	Application reference(s) (e.g. EP123456)	Subject or title of application	Applicant (s) (as on the application)
Patent	FR1000235135	Dispositif de détection et/ou de dosage d'au moins un composé chimique et enceinte destinée à former un tel dispositif (Gas sensing cell with remote piezo- actuation of the cantilever sensors)	CEA, UNIMAN, ESIEE, namely Emmanuel Scorsonne; Bertrand Bazin; Farbod Ghassemi; Gaelle Lissorgues; Rafea Manai; Krishna Persaud; Lionel Rousseau

### 4.2.2 Part B2

LIST OF EXPLOITATION FOREGROUND									
Nr	Type of Exploitation Foreground	Description of exploitation foreground	Confidential	Foreseen embargo date	Exploitation products or measures	Sector of application	Timetable, commercial or any other use	Patents or other IPR exploitation (licences)	Owner & Other Beneficiary (s) involved

LIST OF EXPLOITATION FOREGROUND									
Nr	Type of Exploitation Foreground	Description of exploitation foreground	Confidential	Foreseen embargo date	Exploitation products or measures	Sector of application	Timetable, commercial or any other use	Patents or other IPR exploitation (licences)	Owner & Other Beneficiary (s) involved
1	General advancement of knowledge and Commercial exploitation of R&D results	<b>Microcantilever based Multisensor chemical detection module</b> Chemical vapour detector, gas cell containing a pre-amplifying electronic	Yes	2017	License or other means via negotiation with interested companies	Security Quality control Food industry Q86 : Human health activities	3 years	Patent Licensable technology	CEA UNIMAN ESIEE
2	General advancement of knowledge and Commercial exploitation of R&D results	<b>Diamond MEMS with Polysilicon gauges</b> CVD diamond based MEMS and Polysilicon gauges	Yes	2018	Possible Licensing, start-up  Publications and technology transfer already done	G46.1.2 - Agents involved in the sale of fuels, ores, metals and industrial chemicals	4 years	Possible licensing, start-up	ESIEE CEA-List
3	General advancement of knowledge and Commercial exploitation of R&D results	<b>Recombinant Proteins</b> Ligand binding proteins	Yes		License potentially with company interested in specific target chemicals  Scientific	Biosensors	Ongoing	License	UNIMAN UNIPD

LIST OF EXPLOITATION FOREGROUND									
Nr	Type of Exploitation Foreground	Description of exploitation foreground	Confidential	Foreseen embargo date	Exploitation products or measures	Sector of application	Timetable, commercial or any other use	Patents or other IPR exploitation (licences)	Owner & Other Beneficiary (s) involved
					publications  Grant applications in security areas				
4	General advancement of knowledge and Commercial exploitation of R&D results	<b>SNIFFER Instrument</b> Sensor electronics			Publications and Platform for further sensor developments	Chemical sensors			UNIMAN
5	General advancement of knowledge and Commercial exploitation of R&D results	<b>Preconcentrator Components; systems</b> Foil preconcentrator: special issue in Sensors and Actuators B following the conference Eurosensors 2015 Optimised silicon preconcentrator			Publications and technology transfers  Service: design and knowhow for manufacturing prototypes, to be exploited <i>via</i> publications and showcases				ARMINES and EPFL

LIST OF EXPLOITATION FOREGROUND									
Nr	Type of Exploitation Foreground	Description of exploitation foreground	Confidential	Foreseen embargo date	Exploitation products or measures	Sector of application	Timetable, commercial or any other use	Patents or other IPR exploitation (licences)	Owner & Other Beneficiary (s) involved
6	General advancement of knowledge and Commercial exploitation of R&D results	<b>Ligand Binding Proteins (LBPs)</b> Odorant Binding protein production-purification process			Kits LBPs for other applications	Industrial, measurement and diagnostic.	3 years	No	GTP
7	General advancement of knowledge	<b>Major Urinary Proteins Production and purification</b>	Yes / No		Publications in peer reviews scientific journals (field of biochemistry/biosensors)		Upcoming months		UNIPD
8	General advancement of knowledge and Commercial exploitation of R&D results	<b>ESP-sampling system</b>			License or other means via negotiation with interested companies	Cabin air quality and health diagnostic	2 years	New IPR may follow	AIRBUS
9	Commercial exploitation of R&D results	<b>Use of aerodynamics to control particle extraction and trajectory</b>	Yes	2016	prototypes to be developed into marketable devices	Public/commercial transportation hubs, shopping centers, governmental facilities	upcoming months numerous demonstrations to potential investors	Company confidential documents, patent applications	TTS

## PROJECT OUTCOME #1

Leader / Exploiting partner: CEA, UNIMAN, ESIEE

Description: Microcantilever based Multisensor chemical detection module

Status at project end: A major outcome of the SNIFFER project has been the development of a multisensory chemical gas cell. The cell can accommodate 8 micro cantilever sensors that can be electronically address individually in parallel using a dedicated electronic circuitry developed also during the project. This cell has been patented by CEA/UNIMA/ESIEE-Paris. The TRL of the cell is assessed to be around 6 to 7. This gas sensor cell was integrated into the main sensor module. However its integration into an explosive and narcotic detection instrumentation involves other constrains hence the overall prototype TRL is estimated to be TRL5. In order to rise the TRL of the explosive/narctotic detector, the technology will be further developed by CEA/ESIEE-Paris and UNIMAN through the H2020 project C-BORD. In parallel, all three partners will prospect for other potential application of the multisensory chemical detection module. Targeted applications (other than security) are for instance the food industry, health and safety, or environmental control.

Potential upcoming exploitation: No exploitation at this stage

IPR issues: Patent, Licensing – patent protection filled

Business perspectives and market outlook: Potential start-up

## PROJECT OUTCOME #2

Leader / Exploiting partner: ESIEE

Description: Diamond MEMS with Polysilicon gauges

Potential upcoming exploitation: Patent to be submitted

IPR issues: No patent issued yet

Business perspectives and market outlook: Potential start-up. This process could be used to develop a large range of diamond components like cantilevers, bridges,.. and could be exploited in a wide range of applications: biology, chemistry,...

## PROJECT OUTCOME #3

Leader / Exploiting partner: UNIMAN, UNIPD

Description: Recombinant Proteins

Potential upcoming exploitation: Different papers defining the methodology for production, the binding capacity and the use of recombinant proteins in sensors are in preparation. Recombinant proteins will be used for implementation of sensors targeted to different analytes.

Business perspectives and market outlook: The biosensor market is global and growing rapidly every year. Pharmaceuticals, environmental, and security sectors are the target market.

## PROJECT OUTCOME #4

Leader / Exploiting partner: UNIMAN

Description: SNIFFER Instrument (sensing electronic submodule)

Potential upcoming exploitation: None yet, licensing and distribution possible.

Business perspectives and market outlook: The security and environmental markets are growing rapidly every year. Pharmaceuticals, environmental, and security sectors are the target market.

## PROJECT OUTCOME #5

Leader / Exploiting partner: ARMINES and EPFL

Description: Preconcentrator Components

Status at project end: A test bench and 3 types of preconcentrators systems developed (1 macro devices and 2 microdevices, 1 on silicon the other on plastic foil support)

Potential upcoming exploitation: Publications and showcases to demonstrate the potential of the pre-concentrator components for any trace detection devices.

IPR issues: Transfer of knowledge will be granted on a case by case basis.

Business perspectives and market outlook: The pre-concentrator components can be used for various other detection systems and devices (pollution control, industrial processes). Contact have been or are going to be made with SMEs working on miniaturized gas detection systems such as MicroSens SA in Switzerland and Apix Analytics in France. More specifically concerning environmental applications, APIX's miniaturized and portable Gas Chromatography system enables direct and on site measurements for real time air quality analysis, VOCs emissions or water and soil pollutions down to ppb level. With regards to security applications, APIX portable and modular gas analyzers offer quick response time and high specificity for on-site measurements of any chemical threats (warfare agents, toxic compounds or explosives detection).

## PROJECT OUTCOME #6

Leader / Exploiting partner: GTP

Description: Ligand Binding Proteins

Status at project end: Production of LBP is still in progress. But the kit development appears more complicated and risky than predicted. The selection of specific LBP appears more complicated than predicted. More time than expected is required for the development of LBP for a given target. Also, it seems that this development will be highly specific to each target and might impact the cost of each product at the end.

Potential upcoming exploitation: None yet as it depends on the success of envisaged following developments of the SNIFFER project, and the identification of the right target/LBP couple.

IPR issues: None

Business perspectives and market outlook: Not yet defined

## PROJECT OUTCOME #7

Leader / Exploiting partner: UNIPD

Description: Custom-tailored proteins – Major Urinary Proteins (MUPs) – for biosensors

Status at project end: Achieved.

Potential upcoming exploitation: Pharmaceutical, environmental

IPR issues: At present are still being explored by UNIPD.

Business perspectives and market outlook: 5 years: 1M€

## PROJECT OUTCOME #8

Leader / Exploiting partner: AIRBUS DEFENSE

Description: ESP-sampling and thermos-desorption system

Status at project end: Achieved



Potential upcoming exploitation: new fields of application (none security) are in major Airbus focus now. The SNIFFER technology on its own could be used for the detection of failure or safety issues with regard to carbon fibre manufacturing processes.

IPR issues: Depending from new planned operational field, still in exploring phase

Business perspectives and market outlook: Not yet defined

## **PROJECT OUTCOME #9**

Leader / Exploiting partner: TTS

Description: A SNIFFER Monitoring Portal

Status at project end: The UC6 device was completed and was successful at all trials. Commercial devices based on the UC6 design are now being designed. In-house data has suggested a number of efficiency improvements to reduce airflow and time of sampling. This is important for less power consumption per test and faster throughput of inspected articles.

Potential upcoming exploitation: Devices can be designed for various applications based on size and intricacy of the articles to be examined.

IPR issues: Licenses will be granted on a case-by-case basis.

Business perspectives and market outlook: The SNIFFER Monitoring Portal can be integrated into existing TTS products. In particular, ShoeSafe, CompactSafe, CarrySafe, and CargoSafe, are existing TTS products with potential for integration of the Monitoring Portal. The market outlook is very promising, as the potential client base is huge: major transportation hubs, shopping centres, government buildings, sports and other entertainment arenas.

## 5 Report on societal implications

<b>A General Information (completed automatically in ECAS)</b>	
Grant Agreement Number: 285203	
Title of Project: SNIFFER	
Name and Title of Coordinator: Emmanuel Scorsone	
<b>B Ethics</b>	
<b>1. Did your project undergo an Ethics Review (and/or Screening)?</b> <ul style="list-style-type: none"> <li>If Yes: have you described the progress of compliance with the relevant Ethics Review/Screening Requirements in the frame of the periodic/final project reports?</li> </ul> <p><b>Yes</b></p> <p>Special Reminder: the progress of compliance with the Ethics Review/Screening Requirements should be described in the Period/Final Project Reports under the Section 3.2.2 'Work Progress and Achievements'</p>	<b>Yes</b>
<b>2. Please indicate whether your project involved any of the following issues (tick box) :</b>	
<b>RESEARCH ON HUMANS</b>	
• Did the project involve children?	<b>No</b>
• Did the project involve patients?	<b>No</b>
• Did the project involve persons not able to give consent?	<b>No</b>
• Did the project involve adult healthy volunteers?	<b>No</b>
• Did the project involve Human genetic material?	<b>No</b>
• Did the project involve Human biological samples?	<b>No</b>
• Did the project involve Human data collection?	<b>No</b>
<b>RESEARCH ON HUMAN EMBRYO/FOETUS</b>	
• Did the project involve Human Embryos?	<b>No</b>
• Did the project involve Human Foetal Tissue / Cells?	<b>No</b>
• Did the project involve Human Embryonic Stem Cells (hESCs)?	<b>No</b>
• Did the project on human Embryonic Stem Cells involve cells in culture?	<b>No</b>
• Did the project on human Embryonic Stem Cells involve the derivation of cells from Embryos?	<b>No</b>
<b>PRIVACY</b>	
• Did the project involve processing of genetic information or personal data (eg. health, sexual lifestyle, ethnicity, political opinion, religious or philosophical conviction)?	<b>No</b>

• Did the project involve tracking the location or observation of people?	<b>No</b>	
<b>RESEARCH ON ANIMALS</b>		
• Did the project involve research on animals?	<b>No</b>	
• Were those animals transgenic small laboratory animals?	<b>No</b>	
• Were those animals transgenic farm animals?	<b>No</b>	
• Were those animals cloned farm animals?	<b>No</b>	
• Were those animals non-human primates?	<b>No</b>	
<b>RESEARCH INVOLVING DEVELOPING COUNTRIES</b>		
• Did the project involve the use of local resources (genetic, animal, plant etc)?	<b>No</b>	
• Was the project of benefit to local community (capacity building, access to healthcare, education etc)?	<b>No</b>	
<b>DUAL USE</b>		
• Research having direct military use	<b>No</b>	
• Research having the potential for terrorist abuse	<b>No</b>	
<b>C Workforce Statistics</b> <b>(to be filled in once answers are received from all partners)</b>		
<b>3. Workforce statistics for the project: Please indicate in the table below the number of people who worked on the project (on a headcount basis).</b>		
<b>Type of Position</b>	<b>Number of Women</b>	<b>Number of Men</b>
Scientific Coordinator		1
Work package leaders	3	2
Experienced researchers (i.e. PhD holders)	2	14
PhD Students	2	3
Other	11	10
<b>4. How many additional researchers (in companies and universities) were recruited specifically for this project?</b>	<b>3</b>	
Of which, indicate the number of men:	2	

<b>D Gender Aspects</b>		
<b>5. Did you carry out specific Gender Equality Actions under the project?</b>		Yes (UNIPD)
<b>6. Which of the following actions did you carry out and how effective were they?</b>		
	Not at all effective	Very effective
<input checked="" type="checkbox"/> Design and implement an equal opportunity policy	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input checked="" type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input checked="" type="checkbox"/> <input type="checkbox"/>
<input checked="" type="checkbox"/> Set targets to achieve a gender balance in the workforce	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input checked="" type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input checked="" type="checkbox"/> <input type="checkbox"/>
<input checked="" type="checkbox"/> Organise conferences and workshops on gender	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input checked="" type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input checked="" type="checkbox"/> <input type="checkbox"/>
<input type="checkbox"/> Actions to improve work-life balance	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>
<input type="radio"/> Other: <input style="width: 200px;" type="text"/>		
<b>7. Was there a gender dimension associated with the research content – i.e. wherever people were the focus of the research as, for example, consumers, users, patients or in trials, was the issue of gender considered and addressed?</b>		
<input type="checkbox"/> Yes- please specify <input style="width: 150px;" type="text"/>		
<input checked="" type="checkbox"/> No		
<b>E Synergies with Science Education</b>		
<b>8. Did your project involve working with students and/or school pupils (e.g. open days, participation in science festivals and events, prizes/competitions or joint projects)?</b>		
<input type="checkbox"/> Yes		
<input checked="" type="checkbox"/> No		
<b>9. Did the project generate any science education material (e.g. kits, websites, explanatory booklets, DVDs)?</b>		
<input type="checkbox"/> Yes		
<input checked="" type="checkbox"/> No		
<b>F Interdisciplinarity</b>		
<b>10. Which disciplines (see list below) are involved in your project?</b>		
<input type="checkbox"/> Main discipline: NATURAL SCIENCES		
<input type="checkbox"/> Associated discipline: Chemical sciences (chemistry, other allied subjects);	<input type="radio"/>	Associated discipline: Biological sciences (biology, botany, bacteriology, microbiology, zoology, entomology, genetics, biochemistry, biophysics, other allied sciences, excluding clinical and veterinary sciences)
<b>G Engaging with Civil society and policy makers</b>		
<b>11a Did your project engage with societal actors beyond the research community? (if 'No', go to Question 14)</b>		Yes
<b>11b If yes, did you engage with citizens (citizens' panels / juries) or organised civil society (NGOs, patients' groups etc.)?</b>		

<input checked="" type="checkbox"/> No <input type="checkbox"/> Yes- in determining what research should be performed <input type="checkbox"/> Yes - in implementing the research <input type="checkbox"/> Yes, in communicating /disseminating / using the results of the project					
<b>11c In doing so, did your project involve actors whose role is mainly to organise the dialogue with citizens and organised civil society (e.g. professional mediator; communication company, science museums)?</b>	No				
<b>12. Did you engage with government / public bodies or policy makers (including international organisations)</b>					
<input type="checkbox"/> No <input checked="" type="checkbox"/> Yes- in framing the research agenda <input checked="" type="checkbox"/> Yes - in implementing the research agenda <input checked="" type="checkbox"/> Yes, in communicating /disseminating / using the results of the project					
<b>13a Will the project generate outputs (expertise or scientific advice) which could be used by policy makers?</b>					
<input type="checkbox"/> Yes – as a <b>primary</b> objective (please indicate areas below- multiple answers possible) <input checked="" type="checkbox"/> Yes – as a <b>secondary</b> objective (please indicate areas below - multiple answer possible) <input type="checkbox"/> No					
<b>13b If Yes, in which fields?</b>					
<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 25%; padding: 5px;">Airport security expertise for consulting services</td> <td style="width: 25%;"></td> <td style="width: 25%;"></td> <td style="width: 25%;"></td> </tr> </table>	Airport security expertise for consulting services				
Airport security expertise for consulting services					
<b>13c If Yes, at which level?</b>					
<input type="checkbox"/> Local / regional levels <input type="checkbox"/> National level <input checked="" type="checkbox"/> European level <input type="checkbox"/> International level					

<b>H Use and dissemination</b>		
<b>14. How many Articles were published/accepted for publication in peer-reviewed journals?</b>		<b>8</b>
<b>To how many of these is open access<sup>3</sup> provided?</b>		<b>3</b>
<b>How many of these are published in open access journals?</b>		<b>3</b>
<b>How many of these are published in open repositories?</b>		<b>0</b>
<b>To how many of these is open access not provided?</b>		
<b>Please check all applicable reasons for not providing open access:</b>		
<input type="checkbox"/> publisher's licensing agreement would not permit publishing in a repository <input type="checkbox"/> no suitable repository available <input checked="" type="checkbox"/> no suitable open access journal available <input type="checkbox"/> no funds available to publish in an open access journal <input type="checkbox"/> lack of time and resources <input type="checkbox"/> lack of information on open access <input checked="" type="checkbox"/> other <sup>4</sup> : choice not to do so		
<b>15. How many new patent applications ('priority filings') have been made?</b> ( <i>"Technologically unique": multiple applications for the same invention in different jurisdictions should be counted as just one application of grant.</i> )		<b>1</b>
<b>16. Indicate how many of the following Intellectual Property Rights were applied for (give number in each box).</b>	Trademark	<b>0</b>
	Registered design	<b>0</b>
	Other	<b>0</b>
<b>17. How many spin-off companies were created / are planned as a direct result of the project?</b>		<b>0</b>
<i>Indicate the approximate number of additional jobs in these companies:</i>		
<b>18. Please indicate whether your project has a potential impact on employment, in comparison with the situation before your project:</b>		
<input checked="" type="checkbox"/> Increase in employment, or <input checked="" type="checkbox"/> Safeguard employment, or <input type="checkbox"/> Decrease in employment, <input checked="" type="checkbox"/> Difficult to estimate / not possible to quantify	<input checked="" type="checkbox"/> In small & medium-sized enterprises <input type="checkbox"/> In large companies <input type="checkbox"/> None of the above / not relevant to the project	

<sup>3</sup> Open Access is defined as free of charge access for anyone via Internet.

<sup>4</sup> For instance: classification for security project.



## 6 Report on the distribution of the EU Financial contribution

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*NB: This table will be completed once financial final reporting is over.*

Name of beneficiary	Final amount of EU contribution per beneficiary in euros
CEA	
UNIMAN	
ST(SI) <sup>2</sup>	
ARMINES	
AIRBUS	
EPFL	
CSSC	
UNIPD	
ESIEE	
GTP	
TTS	
3DSA	
INP	
ARTTIC	

## 7 Annexes: Deliverables and milestones tables

### 7.1 Deliverables

All deliverables have been submitted to the EC through the electronic tool. A few of them were delayed but, overall, these delays did not jeopardise the project.

D#	Title	Comments
DL 1.1.1	Usage Case Definitions for V1 technologies (UC1, UC3)	submitted
DL 1.1.2	Report on mid-term Usage Case trials (UC1 and UC3) and assessments (UC1, UC3)	submitted
DL 1.1.3	Lab trial validation methodology for V1 technologies (UC1)	submitted
DL 1.2.1	Usage Case Definition for V2 technologies (UC2, UC4, UC5, UC6)	submitted
DL 1.2.2	Report on final Usage Case trials and assessments (UC2, UC4, UC5, UC6)	submitted
DL 1.3.1	Report on national ethical approvals	submitted
DL 1.3.2	Fiel trials validation methodology for V2 technologies (UC4 and UC6)	submitted
DL 1.3.3	Report on nationale ethical approvals for field trials (V2)	submitted
DL 1.5.1	Final Consolidated User Report on SNIFFER	submitted
DL 2.1.1	Functional specifications and design of architecture of D1	submitted
DL 2.1.2	Operational device D1 with guidelines for its usage	submitted
DL 2.1.3	Report on technical support & problems encountered with D1	Canceled following amendment #1
DL 2.2.1	Functional specifications and design of architecture of D2	submitted
DL 2.2.2	Operational device D2 with guidelines for its usage	submitted
DL 2.2.3	Report on technical support & problems encountered with D2	Canceled following amendment #1
DL 2.3.1	Functional specifications and design of architecture of D3	submitted
DL 2.3.2	Operational device D3 with guidelines for its usage	submitted
DL 2.3.3	Report on technical support & problems encountered with D3	submitted
DL 2.4.1	Functional specifications and design of architecture of D4	submitted

DL 2.4.2	Operational device D4 with guidelines for its usage	submitted
DL 2.4.3	Report on technical support & problems encountered with D4	submitted
DL 2.5.1	Functional specifications and design of architecture of D5	submitted
DL 2.5.2	Operational device D5 with guidelines for its usage	Canceled following amendment #1
DL 2.5.3	Report on technical support & problems encountered with D5	Canceled following amendment #1
DL 2.6.1	Functional specifications and design of architecture of D6	submitted
DL 2.6.2	Operational device D6 with guidelines for its usage	submitted
DL 2.6.3	Report on technical support & problems encountered with D6	submitted
DL 2.7.1	Final Consolidated Technology report	submitted
DL 3.1.1	Functional specifications and design of architecture of V1 Sub-System	submitted
DL 3.1.2	Components for Sampling, preconcentration and pre-treatment of target analytes	submitted
DL 3.1.3	Modules for Sampling, preconcentration and pre-treatment of target analytes	submitted
DL 3.1.4	V1 of Sub-system Sampling, preconcentration and pre-treatment of target analytes	submitted
DL 3.2.1	Functional specifications and design of architecture of V2 Sub-System	submitted
DL 3.2.2	Components for Sampling, preconcentration and pre-treatment of target analytes	submitted
DL 3.2.3	Modules for Sampling, preconcentration and pre-treatment of target analytes	submitted
DL 3.2.4	V2 of Sub-system Sampling, preconcentration and pre-treatment of target analytes	submitted
DL 3.2.5	Report on technical support & problems encountered	submitted
DL 3.3.1	Functional specifications and design of architecture of automatic trace collector module	submitted
DL 3.3.2	Operational automatic trace collector module	Submitted – strong delay due to the fact that the internal quality assurance team rejected the DL because of the lack of content
DL 4.1.1	Functional specifications and design of architecture of V1 Sensor Sub-System	submitted
DL 4.1.2	V1 of Parametric software	submitted
DL 4.1.3	V1 of Sensor Sub-System	submitted
DL 4.2.1	Functional specifications and design of architecture of V2 Sensor Sub-System	submitted
DL 4.2.2	V1 of Parametric software	submitted
DL 4.2.3	V2 of Sensor Sub-System	submitted

DL 4.2.4	Report on technical support & problems encountered	submitted
DL 5.1.1	Functional specifications and design of V1 Biosensors	submitted
DL 5.1.2	V1 of Biosensors	submitted
DL 5.2.1	Functional specifications and design of V2 Biosensors	submitted
DL 5.2.2	V2 Microcantilevers	submitted
DL 5.2.3	V2 of Biosensors	submitted
DL 5.2.4	Report on technical support & problems encountered	submitted
DL 6.1.1	Functional specifications and design of V1 Proteins	submitted
DL 6.1.2	V1 of Proteins	submitted
DL 6.2.1	Functional specifications and design of V2 Proteins	submitted
DL 6.2.2	V2 of Proteins	submitted
DL 6.2.3	Report on Processes for industrial scale-up of production	submitted
DL 6.2.4	Report technical support & problems encountered	submitted
DL 7.1.1	Functional specifications and design of architecture of Self-Diagnostic Sub-System	submitted
DL 7.1.2	Diagnostic Sub-System	submitted
DL 7.1.3	Report on technical support & problems encountered	submitted
DL 7.2.1	Reference samples for calibration of V1 devices	submitted
DL 7.2.2	Reference samples for calibration of V2 devices	submitted
DL 8.1.1	Dissemination plan	submitted
DL 8.1.2	Project leaflet	submitted
DL 8.1.3	Public website	submitted
DL 8.1.4	Poster	submitted
DL 8.1.5	Final report on dissemination activities	submitted
DL 8.2.1	First version of SNIFFER Exploitation Plan	submitted
DL 8.2.2	Second version of SNIFFER Exploitation Plan	submitted
DL 8.3.1	Report on initial SNIFFER User Advisory Group	submitted
DL 8.3.2	Report on midterm SNIFFER User Advisory Group	submitted
DL 8.3.3	Report on final SNIFFER User Advisory Group	submitted
DL 8.4.1	Report on first user workshop	submitted

DL 8.4.2	Report on second user workshop	submitted
DL 9.1.1	First Management Report	submitted
DL 9.1.2	Mid-term progress Report	submitted
DL 9.1.3	Second Management Report	submitted
DL 9.1.4	Final reports	submitted
DL 9.2.1	Project management guidelines including management plan	submitted
DL 9.2.2	Periodic update of risk register and quality plan	submitted
DL 9.2.3	Periodic update of risk register and quality plan	submitted
DL 9.2.4	Final report on consortium management	submitted

## 7.2 Milestones

WP #	Milestones name	Lead Ben. #	Lead Ben. Name	Due date	Achieved Yes/No	Means of verification
1	MS 1 Usage Cases	12	3DSA	M6 (2012/07/31)	Yes	Accepted by the QA Board and the consortium
2	MS 2 Midterm Technology Delivery of SNIFFER subsystems (V1)	5	AIRBUS	M25 (2013/07/31)	Yes	Lab tests on all kinds of V1 subsystems using reference detectors Lab demo of full-chain sampling detection process
3	Device Integration (V1)	5	AIRBUS	M25 (2013/07/31)	Yes	Full-chain Laboratory -ESP sampling and desorption system test & integration
4	Project Midterm Assessment based on Usage Case (V1)	12	3DSA	M25 (2013/08/31)	Yes	Lab tests on all kinds of V1 subsystems using reference detectors Lab demo of full-chain sampling detection process
5	Technology Develepment and	5	AIRBUS	M35	Yes	Laboratory test at CEA site and integration into OBP-sensor V2 device finalized successfully

WP #	Milestones name	Lead Ben. #	Lead Ben. Name	Due date	Achieved Yes/No	Means of verification
	optimisation (V2)			(2014/01/31)		
<b>6</b>	Final Technology delivery of SNIFFER subsystems (V2)	5	AIRBUS	M37 (2015/03/31)	Yes	Field test at Athens Airport with full functionality of ESP particle sampling system and new thermos-desorption device
<b>7</b>	Final Assessment based on Usage Case (V2) and perspectives	12	3DSA	M40 (2015/05/31)	Yes	Yes (DL122) Field Trials at the Athens International Airport (AIA)