Real Time Calibration and Testing of Chemical Sensors enabled by Precision Micro-dispensing Technology

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ABSTRACT

Precision micro-dispensing based upon ink jet technology has been used in medical diagnostics since the early nineties, and now is moving into a wide range of applications. Ink-jet printing technology can reproducibly dispense micro-droplets of fluid with diameters of 15 to 100 µm (2pl to 5nl) at rates of 0 - 25,000 per second from a single drop-on-demand printhead. The deposition is non-contact, data-driven and can dispense a wide range of fluids. It is a key enabling technology in the development of Bio-MEMS devices, Sensors, Micro-fluidic devices and Micro-optical systems. In this paper, we will discuss the use of this technology for real time calibration and testing of chemical sensors. The technology is based upon test systems developed for olfaction testing which are capable of precisely dispensing chemical aromas in concentration that vary over 6 orders of magnitude. The droplets of each chemical are thermally converted into a vapor that is fed directly into the sensor under test.

Keywords: Sensors, Chemical Sensors, Calibration, Detection, Ink-jet

1. INTRODUCTION

The success of miniature chemical analysis systems in both defense and consumer markets is being limited by the stability and reliability of individual microelectronic chemical sensors and in the cost at the system level of the sensor network. Smart networks can compensate for some of the instabilities in the sensors that make up the network, but the best coach in the world cannot take a team of mediocre ball players to the super bowl. Improving the sensors or keeping them calibrated is required.

There are many different types of chemical sensors and each type has its own set of parameters that influence the final signal. Many of the unwanted parameter changes need to be compensated for by one means or another. A list of some parameters are:

a. Temperature variations
b. Drift due to moisture absorption (relative humidity)
c. Cross-sensitivity with other chemicals
d. Memory / loading effects
e. Non-specific selectivity
f. Drift due to the sensor technology

Variations in theses parameters can be compensated for by one of three means: improved sensor design, parameter measurement / control and real time calibration. This paper will focus on the use of precision micro-dispensing technology to enable real time calibration and testing of chemical sensors.

Figure 1 is a schematic of the sensor / calibrator system The most basic detector array relies on sensing and transducing a "positive" signal into information the end-user can understand. Calibrating the detector array is critical if the output is

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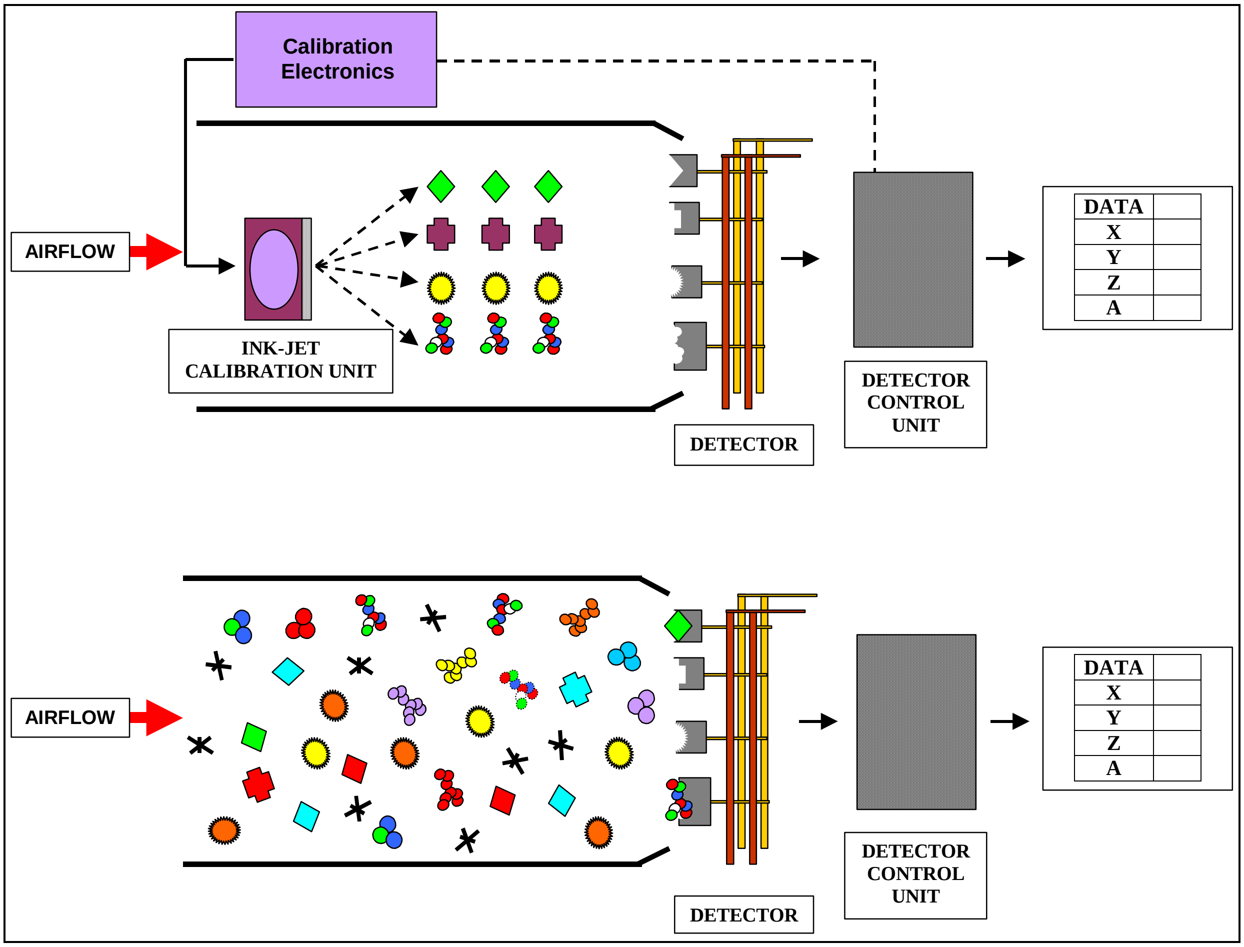


Figure 1: Schematic of a Sensor / Calibrator System

to have real value. Values for "positive" signals can originate from a database or be made in real time when discrete test substances are available. This figure describes how real time calibration can be performed.

The upper half of Figure 1 shows an ink-jet calibration unit at the input end of a hypothetical detector unit. This unit can disperse discreet amounts of substances of interest and convert them into vapor. This programmable, precisely controlled vapor enables the calibration of sensors in real time. The control unit can send signals to the calibrator electronics to alter the inputs. Once the sensor is calibrated, the unknown gas is switched to the input end of the detector unit and impinges on the sensor's active surface. The signals received in the control unit are compared to the database for species determination. Calibrating on site increases the flexibility of the detector by adapting the system to be most sensitive to a given environment and to eliminate many of the unwanted parameter changes that occur with actual use. Real time calibration built into a self-contained sensor unit will enable sensor networks of the future.

2. BACKGROUND

2.1. Ink Jet Technology

In demand mode ink-jet printing systems, a volumetric change in the fluid is induced either by the displacement of a piezoelectric material that is coupled to the fluid,[1] or by the formation of a vapor bubble in the ink, caused by heating a resistive element.[2] This volumetric change causes pressure/velocity transients to occur in the fluid and these are directed so as to produce a drop that issues from an orifice.[3,4] A droplet is created only when it is desired in demand mode systems. Demand mode ink-jet printing systems produce droplets that are approximately equal to the orifice diameter of the droplet generator. Figure 2 shows a single channel MicroJet device generating 50µm diameter drops of ethylene glycol from a device with a 50µm orifice at 2,000 per second.

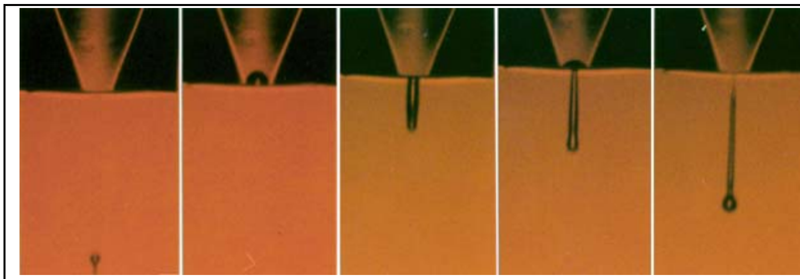


Figure 2: 60µm droplets of alcohol generated at 4kHz.

The high precision and reproducibility of this type of micro-dispensing has been demonstrated with many applications over the years. Two examples will be highlighted here. Highly uniform sized (less than 1 µm standard size deviation) paclitaxel-loaded microspheres were manufactured by ink-jet technology (Figure 3)[5]. The uniformity of the individual microspheres enabled them to self-assemble into the close-pack pattern shown in this figure. The measured 1 µm standard deviation was limited by our ability to measure. We believe the actual numbers are much better than this.

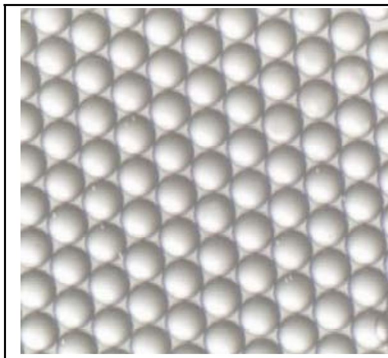


Figure 3: Microspheres loaded with anticancer drug (paclitaxel) of high drug content (PLA to PGA ratios of 65:35).

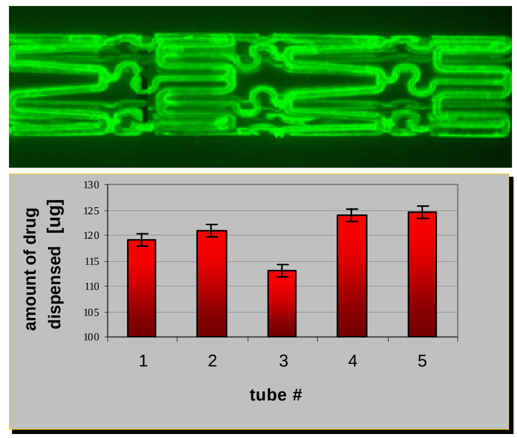


Figure 4: Top: cardiac stent coated with a polymer-drug solution by ink-jet printing (a fluorescent dye was added for visualization); Bottom: Repeatability of the stent coating process via ink-jetting.

Ink jet technology also enables high precision in the amount of drug jetted on cardiovascular stents. The standard deviation of the amount of drug dispensed on coated, stent-like cylindrical tubes was below 5% (Figure 4), a low number compared to over 30% variation obtained with other coating technologies such as dipping or spaying (Figure 4 indicates the amount of drug recovered from jetted stents). Our research partners, Abbott Laboratories, presented data showing CV's as low as 2%.[6]

Figure 5: Piezo-micro-dispensing olfactometer.

2.2. Olfactometer

The original test and calibration research performed at MicroFab was aimed at developing a medical diagnostic instrument to measure a persons smell thresholds.[7] In this case we were developing a clinical instrument to test the human nose. Impairment of odor detection and scent identification has been reported to occur presymptomatically for Parkinson's both Disease and Alzheimer's Disease.[8,9] Olfactory testing has been considered as a component of an early detection screening for these diseases. Using a piezo-

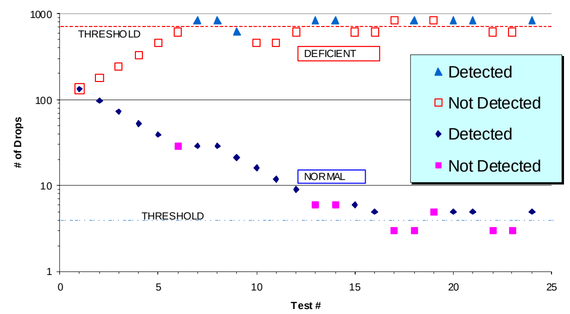


Figure 6: Protocol for the determination of the olfaction threshold.

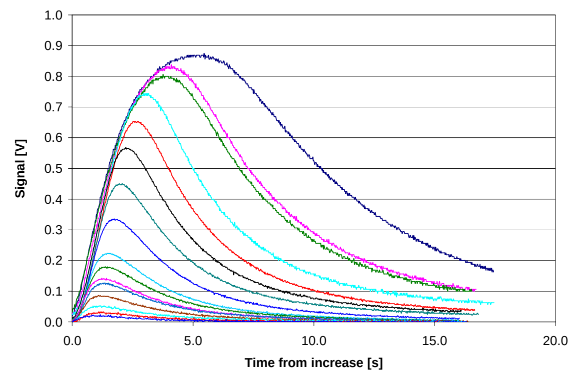


Figure 7: Sensor response for various number of drops. The bottom curve corresponds to 6 drops and the top curve corresponds to 618 drops.

micro-dispensing olfactometer (Figure 5) we performed threshold sensitivity tests for phenyl ethyl alcohol (PEA) and lemon extract. Subjects included healthy controls, Parkinsonian syndromes, Alzheimer's Disease, groups "At Risk" for Parkinson and Alzheimer diseases based upon family history, and a group at risk for Alzheimer's Disease based on mild cognitive impairment classification. Figure 6 outlines the protocols followed for the test.

Using a PID sensor one output profile for the olfactometer is shown in Figure 7. The bottom curve measures the chemical output for 6 drops dispensed at a dispensing frequency of 1,000/sec. The top curve is for 618 drops at the same dispensing frequency. In this figure the actual shape of the curves has more to do with the response of the sensor than the actual time profile of the vapor.

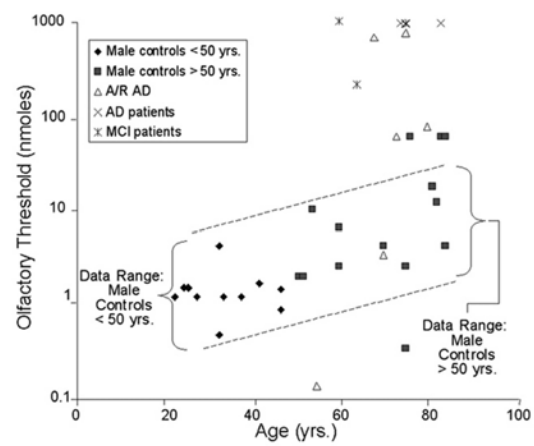


Figure 8: Scatter plot showing olfactory sense threshold (male controls with Alzheimer's related group).

The final figure in this section shows results from tests on actual patients at the Human Performance Laboratory of Presbyterian Hospital of Dallas. Figure 8 is a scatter plot which represents about 20% of the people tested. It shows olfactory sense thresholds as a function of age for "rose" (PEA). Male controls with Alzheimer's-related groups are represented. To our knowledge, this is the first olfactory study in which the stimulus generated and the olfactory thresholds recorded have been measured in terms of precise mass of odorant delivered.

2.3. Other Applications

Digital controlled generation of aromas or odorants can be used in many human interface applications. Figure 9 shows a prototype gaming unit that brings one more of the senses into video games.[10] This unit can dispense eight different aromas in response to what is going on in the game. If the player shoots a gun, he smells smoke; if he goes into the woods he smells the pine; etc.

Figure 9: Prototype gaming unit.

Figure 10 is a prototype of a perfume kiosk. In this application a person can design a perfume by programming the precise amounts of 16 different ingredients and then, when the button is pressed, the combination fragrance is dispensed. Since this unit is controlled by a computer, a fragrance can be designed in one geographical location and created and dispensed at different and remote locations. A perfumer in Paris could design the fragrance and a person in Orlando would experience it.

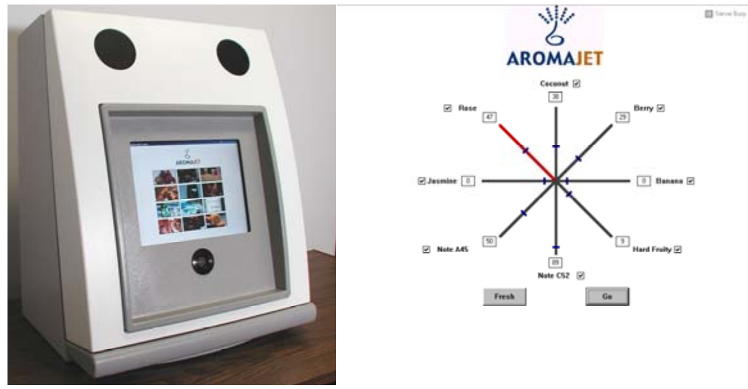


Figure 10: Prototype perfume kiosk.

3. SENSOR CALIBRATOR

3.1. Prototype and principle of operation

This system was built as a test bed for investigating the feasibility of using inkjet technology for vapor metrology. More specifically, it is used to produce vapor containing trace amounts of high explosives so that, ultimately, the performance of explosive vapor detectors may be traced to known standards. Ultimately it will be used to calibrate sensors which detect other chemical species.

Figure 11 shows the first prototype. It is PC controlled, has a six jet capacity, and has the ability to create pulses of vapor as well as a continuous flow. In the prototype system the jets were designed to have a drop size of 60pl. In burst mode the number of drops can be specified anywhere from 1 to 10,000 drops. The delivery rate, in either burst or continuous mode, can be set to anywhere from 1 to 2 kHz. Higher drop rates can be achieved, up to around 10 kHz, but resonance effects play a pronounced role, so operation in this range is more complicated. By using different dilutions of the material under test, a much broader range of vapor concentrations may be achieved. A range of six orders of magnitude is possible in some cases.

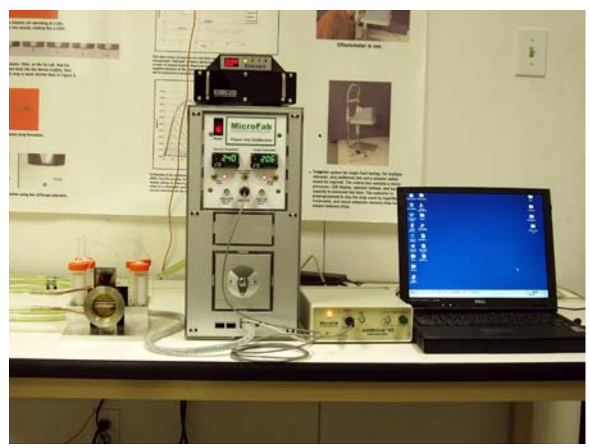


Figure 11: Prototype of the calibration sensor.

In Figure 11**,** going from left to right, there is the dispense head, the Main unit, on top of the Main unit is the Cooler, the Jet Drive III, and the host PC. The eventual product for testing airport security will be handheld and battery operated. Further miniaturization is possible through MEMS technology. It is possible to build a system on a chip which would be incorporated as part of the sensor for the purpose of periodic self-testing. The corresponding block diagram for this Table Top Vapor-Jet Calibrator is shown in Figure 12.

The dispense head sub-system consists of the Microjet device, heaters, reservoirs, temperature controls and interconnects. This sub-system is shown in Figure 13.

Figure 14 shows, schematically, the system's key elements and operation. The Jet is supplied by a reservoir (not shown) and ejects drops as determined by the operator. The fluid is vaporized and then directed to the sensor under test. Heated walls insure that the fluid does not condense on the walls. A mixer may be added downstream of the Vaporizer to make sure that the output is homogeneous.

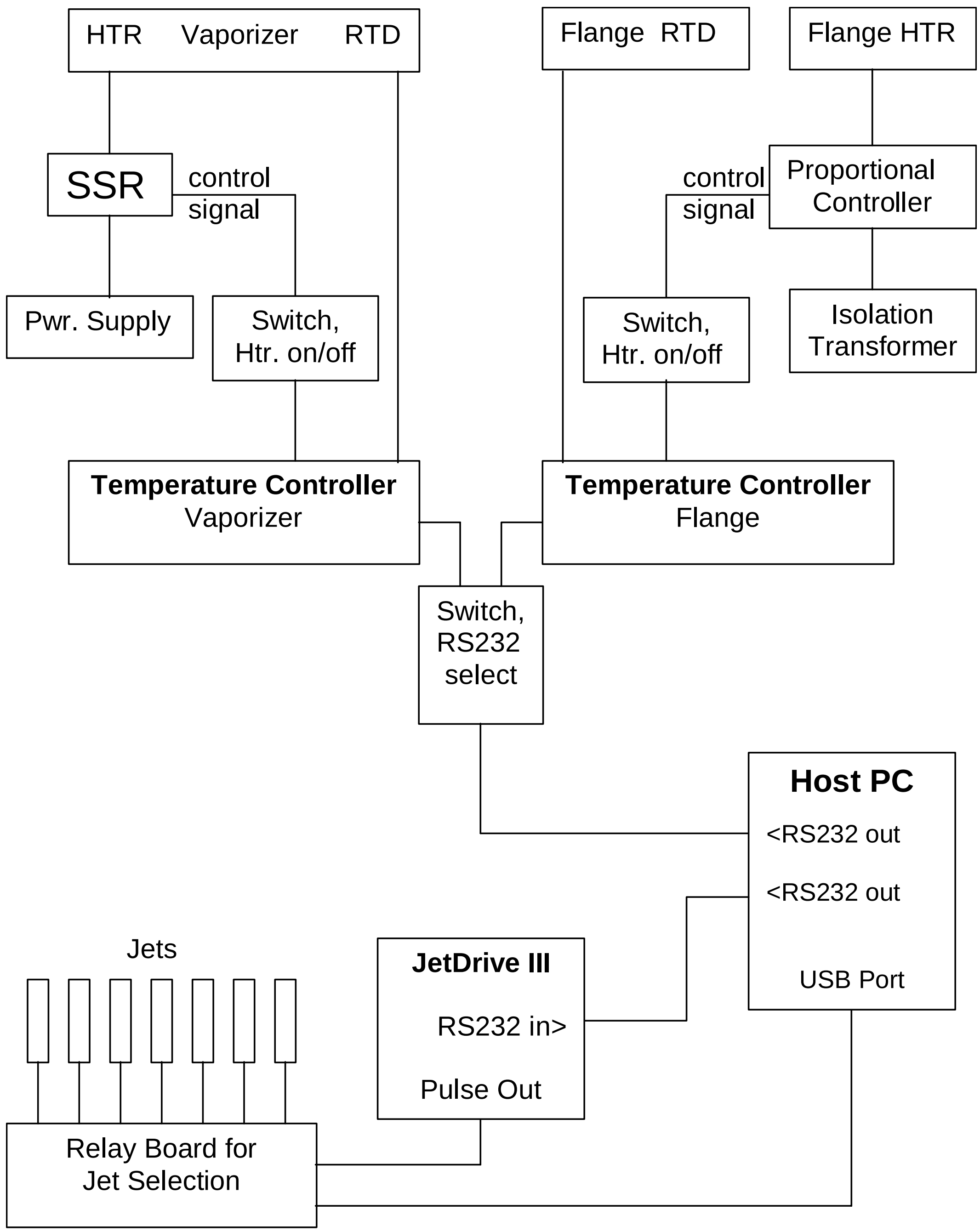


Figure 12: Electrical block diagram.

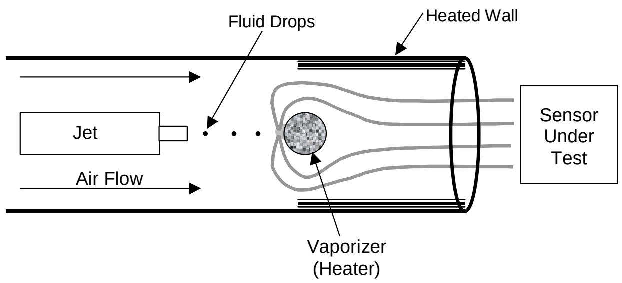


Figure 14: System schematic and key components.

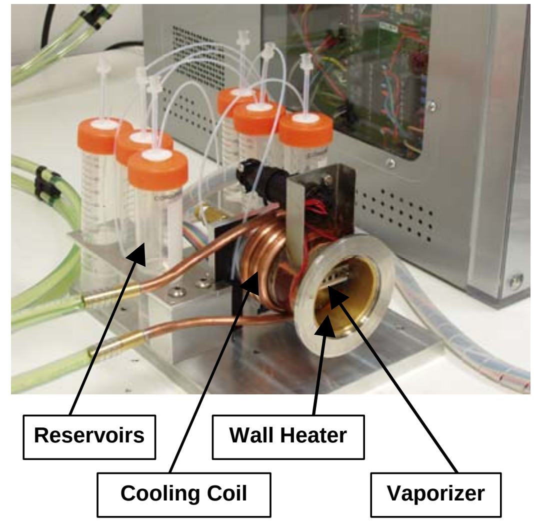


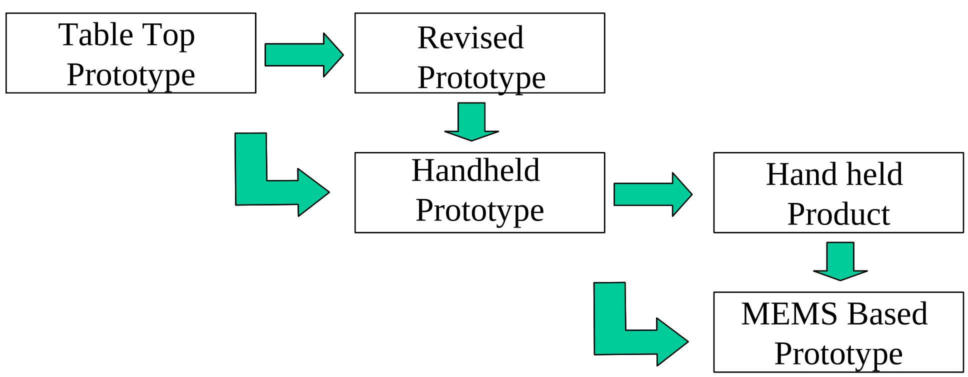
Figure 13: Dispense head sub-system.

3.2. Results

Individual components and subsystems were tested and characterized in MicroFab's research laboratories. Jetting device (6), precision alignment, fluid systems, thermal systems, and electronics sub-systems were all precisely characterized. The overall system performance as a Vapor-Jet Calibrator is presently being evaluated at NIST. Their test results should be available shortly.

4. FUTURE – MICRO-CALIBRATOR

The goal of this research is to accurately demonstrate real time calibration of chemical sensors with a prototype system as delivered to NIST. Improvements in this prototype system will lead to a handheld system the size of a hardback book with the same functions and features. The small gaming unit shown in Figure 9 contained eight different chemicals and was digitally controlled. The ultimate goal of this program is to develop a low cost MEMS based calibrations systems that can be incorporated into the sensor design. It is expected that the low cost micro-system will be sensor dependent and chemical species specific and not have the flexibility of the larger systems. The overall strategy for the development program is illustrated in Figure 15 below.



Status:

- Table Top unit at NIST for Characterization
- Handheld Unit Requirement Spec Complete
- MEMS based unit concepts developed

Figure 15: Pipeline strategy.

5. CONCLUSIONS

We have defined a technology and program for real time calibration of chemical sensors based upon precision micro-dispensing technology. Initial efforts focused on an olfactometer for testing human olfactory thresholds in a clinical setting. This program proved to be very successful. The Vapor-Jet Calibrator is our newest effort and is focused on calibrating sensor and sensor arrays, initially in a laboratory setting and eventually in the field.

ACKNOWLEDGMENTS

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