

# ***ON THE TEMPORAL EVOLUTION OF MICRO-DISCHARGE SPECTRA AND DETECTION OF ORGANIC VAPORS IN AIR***

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## **Abstract**

This paper reports the use of time resolved pulse discharge spectroscopy for detecting organic vapors in ambient air. The impact of both sample duration and delay is investigated. A pulsed micro-discharge, consuming only 400  $\mu$ J of energy, is struck by applying a high voltage pulse between two closely spaced electrodes patterned on a glass substrate. As a result, the ambient gas in the glow region of the micro-discharge breaks down into molecular and atomic fragments. The time resolved spectrum of the light emitted by these fragments is obtained with a spectrometer, which is triggered a programmed time delay after the discharge pulse. It is observed that better detection is achieved by limiting the sample duration to 2.1 ms and with a delay of  $<1$  ms after initiation of the micro-discharge. This is verified for isopropyl alcohol (100 ppm) and acetic acid (250 ppm) vapor samples.

**Keywords:** Micro-discharge, volatile organic compounds, gas sensor, transient spectroscopy

## **1. Introduction**

In the quest for miniaturized vapor and gas sensors, a variety of transduction methods have been explored. Chemi-resistors, chemi-capacitors, chemi-mechanical sensors, and metal oxide sensors measure changes caused by analytes to thin films [1]. However, film properties can drift and change with contaminants, package stress, and ambient temperature. Some applications require greater speed and specificity than these approaches offer. Miniaturized sensors that use plasma spectroscopy of target species can complement other sensors in many respects, but typically require low operating pressures (and hence a pump) and do not favor an air ambient [2]. Pulsed arcs (micro-discharges) can, however, operate at atmosphere in air, and have been used for spectroscopically detecting inorganic impurities in water [3]. Resulting emissions have also been used for fluorescent detection of bio-chemicals [4]. This paper describes the first use of pulsed discharge spectroscopy (using a micromachined device) for detecting organic vapors in an air ambient at atmospheric pressure. It also describes how the temporal evolution of spectra affects the signal

## **2. Microsystem Design**

The sensor (Fig. 1) utilizes two closely spaced Ti electrodes patterned on a glass substrate. The gas flows through the channel formed between the substrate and two glass spacers bonded to it. The gas molecules are fractionated as they pass through the micro-discharge region [5]. The emitted spectrum, which is characteristic of the fragments, is observed using a portable off-chip spectrometer (Ocean Optics USB2000). The micro-discharge is powered by a capacitor bank under computer control, which provides a current pulse of about 2  $\mu$ s duration (Fig. 2). A shunt switch is used to avoid the damage caused by high currents in the series loop. A trigger pulse (Fig. 3), generated after a programmable delay of time  $t_1$  after the discharge, turns on the spectrometer for 2.1 ms. By varying  $t_1$ , a time resolved spectrum of the micro-discharge is obtained.

## **3. Experimental Results**

Figure 4a shows a baseline measurement of the device in air taken during and immediately following the discharge. Line spectra corresponding to  $N_2$ ,  $O_2$  and  $H_2O$  fragments are superimposed on

a wider background. The temporal evolution of the spectra is shown in Fig. 4b. The line spectra decay rapidly compared to the wider background. Thus, by limiting the observation window to 2 ms after the discharge, greater sensitivity can be obtained.

The time resolved spectra of acetic acid, with 2.1 ms sample time, are shown in Fig. 5a. Similar to the air micro-discharge, it shows a relative decrease of line spectra compared to the wide background spectra. Figure 5b shows the spectrum of acetic acid, taken asynchronously with a 10 ms integration time to capture all the emitted light. It is evident that the spectra with a 2.1 ms sample time taken at  $t=0$ , has a better signal to background (noise) ratio. Thus, by performing a synchronous detection and limiting the integration time, resolution is enhanced.

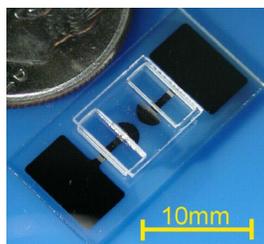
Figure 6a shows the spectrum in the presence of isopropyl alcohol (IPA) vapors, taken synchronously with the discharge and a sample time of 2.1 ms. The IPA concentration was independently measured with a commercial VOC sensor (MiniRAE 2000) to be 100 ppm. Different lines corresponding to fragments of the organic compounds ( $C_2$ , CO, CH) are evident. Figure 6b. shows the spectrum with a longer integration time. Although the overall intensity is higher, the relative intensities of the carbon lines (especially the 560 nm  $C_2$  line) are lower.

It is notable that a pulsed discharge does not consume excessive energy (similar to a camera flash) and is appropriate for low power applications. For example at a sample rate of 1 Hz, our present setup consumes  $<400 \mu W$  for the discharge, which can be reduced further if needed.

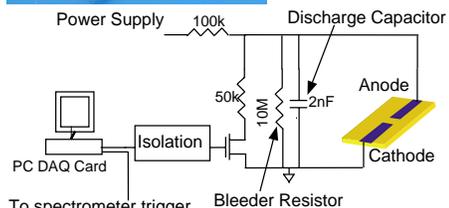
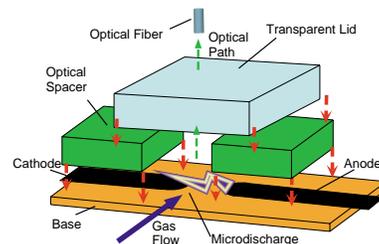
#### 4. Conclusion

The use of time resolved pulsed discharge spectroscopy for detection of organic vapors has successfully been demonstrated. The signal to noise ratio is seen to decrease with time as the wide background radiation decays slower than the line spectra. Hence, by limiting the spectrometer on time and synchronizing the spectrometer trigger to the discharge much higher resolution is obtained.

#### Figures

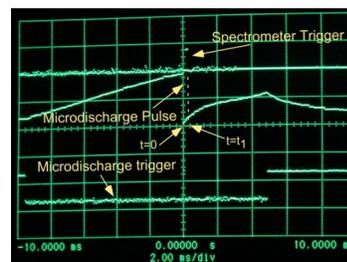


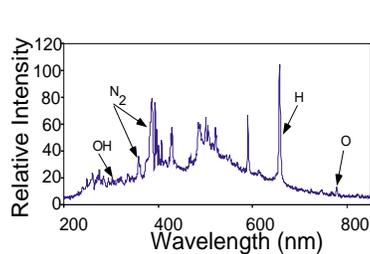
**Fig 1 (a-left):** Photograph of the final device shown against a US dime. **1 (b-right):** Schematic of the sensor, showing the gas flow and the optical path of the emitted spectra.



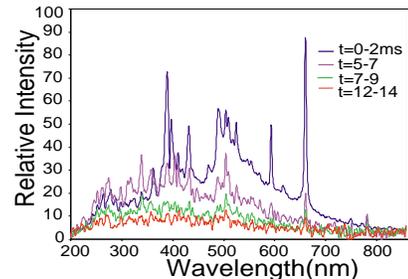
**Fig. 2 (top):** Schematic of the pulsed micro-discharge circuit.

**Fig 3 (right):** Captured waveform showing the discharge pulse and spectrometer trigger.

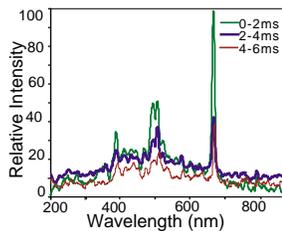




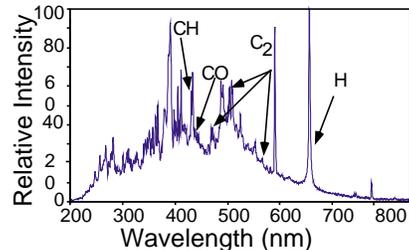
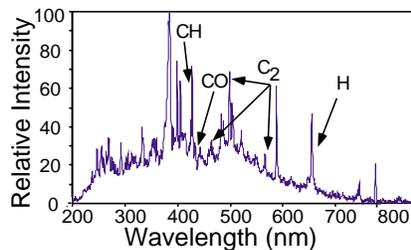
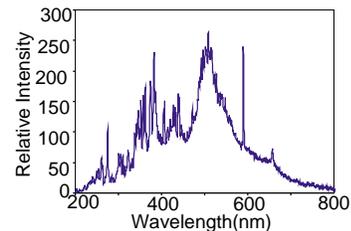
**Fig. 4 (a-left):** Spectra of the micro-discharge in air. Line spectra corresponding to the nitrogen and water vapor are seen superposed on a continuous spectrum.



**4 (b-right):** Time resolved spectra of air. The emitted light is sampled for 2.1 ms, at different points after a micro-discharge pulse. Although the intensity is on a relative scale, the readings are taken with the same setup, so a comparison on of intensities can be made.



**Fig. 5 (a-left):** Temporal evolution of the spectra of acetic acid **5 (b-right):** Emission spectra of acetic acid with a long (10ms) integration time. The signal to noise ratio is decreased by the longer sample time.



**Fig. 6 (a-left):** Spectra of the micro-discharge in air with 100 ppm IPA taken at  $t=0$ , and a sample time of 2.1 ms. Spectra corresponding to fragments of carbon compounds are evident. **6 (b-right):** Spectra of IPA vapors in air ambient with a 10 ms time. Although the overall signal is stronger, the lines corresponding to the hydrocarbon fragments are smaller.

**References:** [1] D.S. Wilson, et al. "Chemical sensors for portable, handheld field instruments," *IEEE Sensors Journal*, 1(4), Dec. 2001, pp 256-274 [2] J. C.T. Eijkel, et al., "A Molecular Emission detector on a chip Employing a Direct Current Microplasma", *Anal. Chem.*, 71(14), July 1999, pp 3600-2606 [3] C. G. Wilson ET. al, "LEd-SpEC: Spectroscopic detection of water contaminants using glow discharge from liquid electrodes," *IEEE Transactions on Electron Devices*, 49(12), Dec 2002, pp. 2317-2322 [4] L. Que, et al., "Dye-Fluorescence LEd-SpEC: A Battery-Operated, On-Chip, Wavelength-Tunable Optical Source for Detection of Biochemicals", *MicroTas*, Oct. 2003 [5] R.S. Bramal, et al. "Direct current discharge spectral emission type detector", *Anal.Chem.*,40,(1), Jan 1968, pp 95 -106