Development and Implementation of a Two Channel System to Measure the Response of Quartz Crystal Resonator Gas Sensors using an FPGA

José Lorenzo Muñoz-Mata[1], Severino Muñoz-Aguirre[1], Humberto González-Santos[1], Georgina Beltrán-Pérez[1], Juan Castillo-Mixcóatl[1],

[1] Facultad de Ciencias Físico-Matemáticas, Benemérita Universidad Autónoma de Puebla
Av. San Claudio y Rio Verde, Col. San Manuel, CU. C.P. 72570, Puebla, Puebla, México

Abstract. The development of gas sensors based on quartz crystal resonators requires the use of a frequency counter to measure the sensor response. However, the commercial frequency counters have at most 2 channels and in odor sensory systems (Electronic Noses), there are used arrays of at least 8 sensors. Furthermore, it is also desirable that the signal processing and data analysis stage can be included in the same system. FPGAs can provide a solution to this kind of problems. The present work involves the development of a high resolution frequency counter using a FPGA for quartz crystal resonators, which is capable of processing and storage the data in 32-bit registers. The data transfer and acquisition was performed by a microcontroller. The virtual instrumentation software LabVIEW was used for the sensor response display and data storage. Tests were performed using gas sensors to obtain real data. The system was scaled to two channels verifying that it behaves in a correct and stable way.

Keywords: FPGA, Frequency Counter, Gas Sensor.

1 Introduction

Current technology on electronic noses has had substantial progress in the recent years [1]. Sensor arrays that respond to a wide range of compounds, as well as advanced pattern recognition and artificial intelligence techniques, which allow the user to extract relevant and reliable information, have been used. An electronic nose has been defined as a system that detects and identifies odors and vapors, typically by using chemical sensing with signal processing and pattern recognition subsystems [2].

The development and utilization of gas sensor arrays (commonly called Electronic Noses, EN) have received a high importance in the field of the scientific research due to the necessity of working on detection, recognition and discrimination of gases. This is a high interest area owing to the enormous variety of gas sensors as well as the diversity of feasible materials to be used as sensing films. Likewise, there are many applications of the system such as air quality monitoring, quality control in the food industry and beverages, cosmetology, biotechnology, etc. [3].

The quartz crystal microbalance (QCM) has been used in sensor applications such as gas-mass detectors and in recent years its applications have been extended since

© C. Delgado, C. Gutiérrez, R. Velázquez, H. Sossa (Eds.)
Advances in Computer Science and Electronic Systems
Research in Computing Science 52, 2011, pp. 235-244

Received 23/01/11
Accepted 20/02/11
Final version 07/03/11

scientists realized that it can be operated in contact with liquids and viscoelastic deposits [4]. Moreover, applications of QCM as gas sensors are widely used due to their high correlation with the human nose.

The QCM gas sensor operation principle is based on the fact that when the gas molecules interact with the sensing film, its mass increases and the resonance frequency decreases owing to the mass loading effect. This frequency shift is proportional to the amount of mass that interacts with the sensing film and it is known as the sensor response [5, 6].

The frequency shift is described by the Sauerbrey equation (Eq. 1) [7], which relates the mass changes in the sensing film per unit area at the QCM electrode surface with the observed change in the crystal oscillation frequency.

$$\Delta f = -2.3\times10^{-6} \cdot F^{2}\frac{\Delta m}{A} \qquad (1)$$

Where Δf is the frequency shift (Hz), -2.3 $\times$ 10^{-6} is a constant obtained from the quartz density (ρ_q) and the shear modulus(μ_q), Δm is the mass (g) of the adsorbed gas molecules, A is the coated area (cm^2) and F is the fundamental resonance frequency (Hz) of the crystal.

The use of a frequency counter is essential for the measurements of gas sensors based on quartz resonators, as it was mentioned above. Therefore, in order to characterize QCM sensors is necessary to observe the shift rate in the sensor frequency. Furthermore, coating the QCM with different types of sensing films it will be possible to obtain different characteristics and to observe the tendency of each sensor achieving more precise measurements for many gases or vapors [5]. Then it is very important the use of a frequency counter in order to measure the frequency variation of one or various sensors. Although some commercial frequency counters are commercially available, they only have one or at least two input channels; however in the electronic nose it could be necessary to use a large number of sensors. Therefore, it is necessary to develop frequency counters with more than two input channels, which must have the characteristics of commercial systems. Virtually any frequency counter commercially available can measure *5 Vpp* square wave frequencies with 50 Ω output impedance provided by the QCM [8].

The present work reports the development of a frequency counter, which is part of a characterization system to measure quartz crystal resonator gas sensors. The counter was designed in an FPGA and it has been connected with an acquisition card, which in turn was in communication with the computer. The virtual instrumentation software LabVIEW was used for the software development in order to display and storage information. Tests were performed using real sensors to obtain information of real measurements. The system was scaled to two channels verifying its stability for one as well as two channels. Finally as a future work the system will be upgraded at least to four channels in order to perform a characterization of various gas sensors, simultaneously.

2 Experimental Set-Up

The implemented experimental set-up is shown in Figure 1. The frequency counter was designed using an Altera Field Programmable Gate Array (FPGA) from the Cyclone II family [9] which has a 50 MHz clock inside. A function generator (AFG3102, Tektronix) was used to evaluate the counter performance. An oscilloscope (TDS-3034, Tektronix) was used to observe the measured signals and a data acquisition card (USB-DAQ) was used to send the information to a computer. In order to verify the frequency counter logic correct performance, it was used a logic analyzer 1582AD (Agilent) [10].

2.1 Frequency Counter Design

The direct frequency counting method was used for the frequency counter design. This method performs the frequency counting from the sensor and the oscillator circuit during a timebase period of one second, as shown in Figure 2.

To perform the frequency count Equation 2 was used.

$$Frequency = \frac{Pulse\ Number}{t_0}. \tag{2}$$

Therefore, if the timebase is one second, the frequency is simply equal to the counted pulse number.

The frequency counter firmware was developed using the software Quartus II. The implemented logic was designed using blocks in order to develop the frequency counter for one channel, which is shown in Figure 1. The blocks are described below.

Timebase. A state machine was designed, which is in charge of initialize the timebase of one second period. The state diagram implemented is shown in Figure 3a. Once the start operation signal is received from the computer via the microcontroller the enable signal is generated, which is also the frequency counting activation signal. Moreover, it is used to enable the data storage in the corresponding block. If the start operation signal is not present the state does not change, otherwise this signal will be automatically generated by this block each second starting another timebase period until a stop signal is received from the computer.

Frequency Counter. It was developed a circuit that verifies the arrival of the enable signal, which was produced by the timebase block. This means that when a timebase is initiated, the frequency counting starts. The counting is performed using a counter circuit, which consists of an array of 32 enable type flip-flops. The clock signal of the counter circuit is taken from the oscillation circuit, which would be the signal generated by the QCM sensor, as is shown in the dataflow diagram in Figure 3b. The stored data are deleted after they are transmitted to the microcontroller by a clear signal.

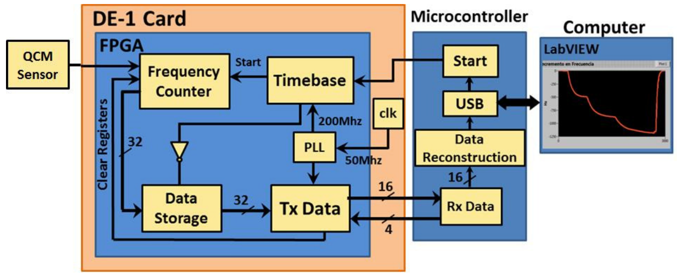


Fig. 1. Block diagram of the implemented system.

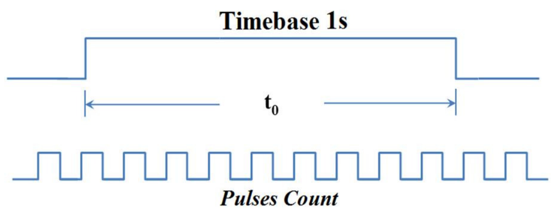


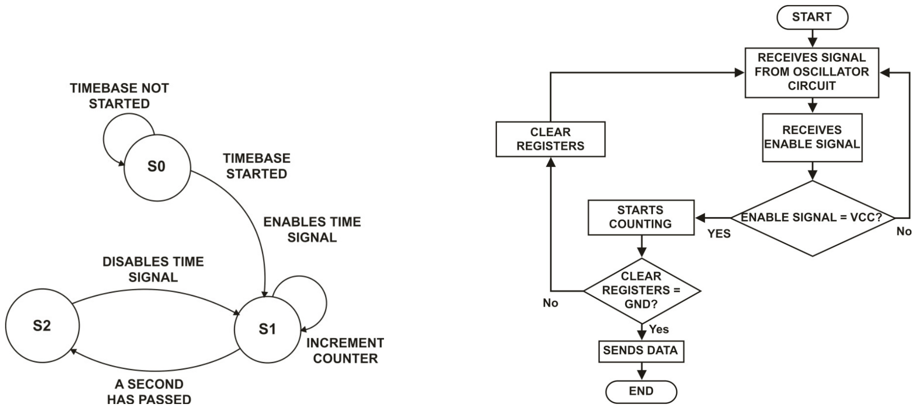
Fig. 2. Frequency counter operation principle.

Data Storage. The data storage block was designed using a circuit implemented with latch devices, which store the information sent by the frequency counter block when the timebase finishes. This means that one second has passed and then the enable signal is activated in order to capture the information (See Figure 4a). The obtained data from the sensor are stored in 32-bit registers in such way that they can be sent to the computer through an interface card via USB.

Data Transmission. For the data transmission, a strategy was designed to send the data from the FPGA in 16-bit packets. The data flow is controlled by a microcontroller using various control bits. When the microcontroller is ready to receive the data, it sends a control signal in order to ask for the first 16 bits packet, in this case the most significant bits (MSB). Once the microcontroller receives the first data packet, it sends the next control signal to receive the next 16-bit packet (LSB) as is shown in Figure 4b.

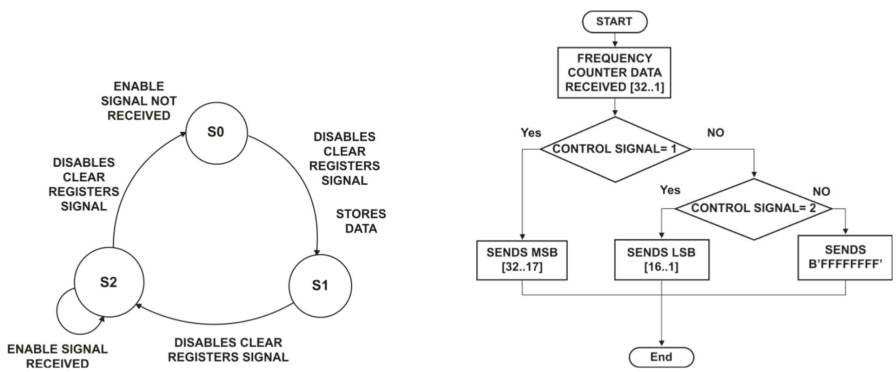
Acquisition Card Design. To perform the data acquisition from the FPGA, it was fabricated an acquisition card (USB-DAQ) using the microcontroller PIC18F4455 (Microchip) [11], which contains the Universal Serial Bus (USB) communication module. The printed circuit (PCB) was designed using the Altium Designer 6.0 software.

Data Acquisition Firmware Development. For the data acquisition from the FPGA to the computer it was developed the microcontroller firmware using PIC C Compiler 4.0 [12].



a) b)

Fig. 3. a**)** Timebase state diagram. b) Frequency counter flowchart. Both implemented in the FPGA



a) b)

Fig. 4. a) Data storage state diagram. b) Data transmission flowchart. Both implemented in the FPGA

First of all, the communication parameters setting is performed, i.e. to define the type of communication to be performed, in this case the bulk communication [13], the size of memory reserved for data transmission, the variables declaration as well as the in-out port configuration. The microcontroller USB module is initialized and it verifies if it is connected to the computer in order to start the device enumeration process. Then an endless loop begins in which the device waits for being enumerated by the computer. Once the device (in this case the microcontroller) has been enumerated by the computer, it checks if some data has been received. If this is true the data are stored in a buffer called *endpoint*, which will contain such information until the device decides to accept these data or data packet [14]. The Figure 5 shows the flowchart with the implemented algorithm for the data acquisition.

To be able to perform the data transfer control among the FPGA, the microcontroller and the computer, it was used a multiplexer, which receives the con-

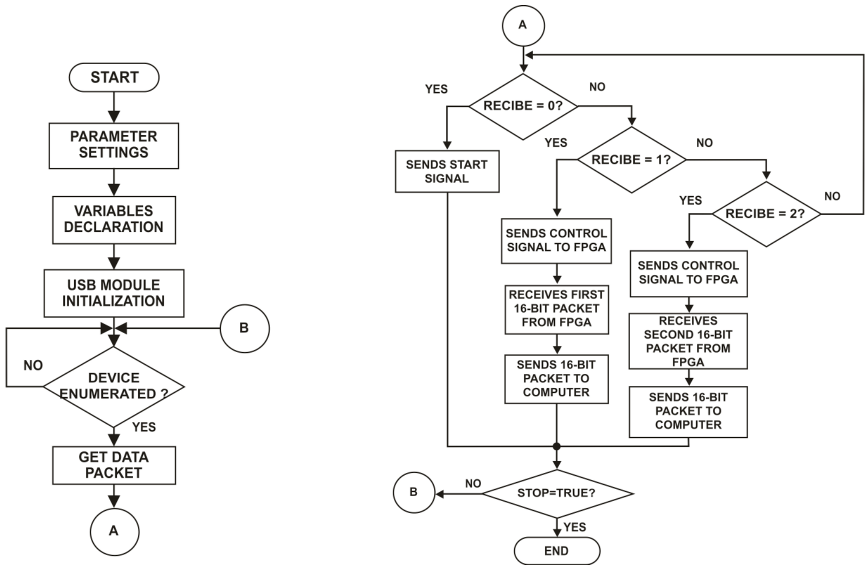


Fig. 5. Acquisition data flowchart of the implemented algorithm in the microcontroller.

trol signal from the computer. When the microcontroller receives the first control signal, it sends the start signal to the frequency counter timebase block inside the FPGA in order to start the frequency counting. The second control signal from the computer indicates to the microcontroller that it must send the corresponding control signal to the FPGA data transmission block to obtain the first 16 MSB data packet from the 32-bit register storage. The maximum number of bits available to receive in parallel by an input port in the microcontroller is 8 bits; therefore the 16-bit packet is received through two 8-bit ports to be able to achieve the acquisition in parallel. Once the frequency counter data are received, the information is recovered in a 16-bit register and these data are sent to the computer. The following 16 bits are sent to the computer where the complete information (32 bits) is reconstructed by software.

Software Development. The software development was carried out with the instrumental software LabVIEW v9.0 [15]. The flowchart for the implemented software is shown in Figure 6.

The first control signal is generated by the computer and it is sent to the microcontroller, which sends the start signal to the FPGA. Then the second control signal is sent to the microcontroller which, in turn, sends its respective control signal and receives the first data packet form the FPGA. After receiving the first data packet from the microcontroller, these are sent to the computer and it performs the same process for the second data packet. When the computer obtains both data packets they are reconstructed and displayed on an indicator, plotted and stored. The same process is repeated in a cycle of approximately three minutes, which is in order to wait for the sensor to attain stability. After that, the software asks if tree minutes have passed to be able to capture a data and to make the baseline zero and these data are displayed, plotted and stored, respectively. This process is performed to have a better understanding of the interaction mechanism between the gas molecules and the sen-

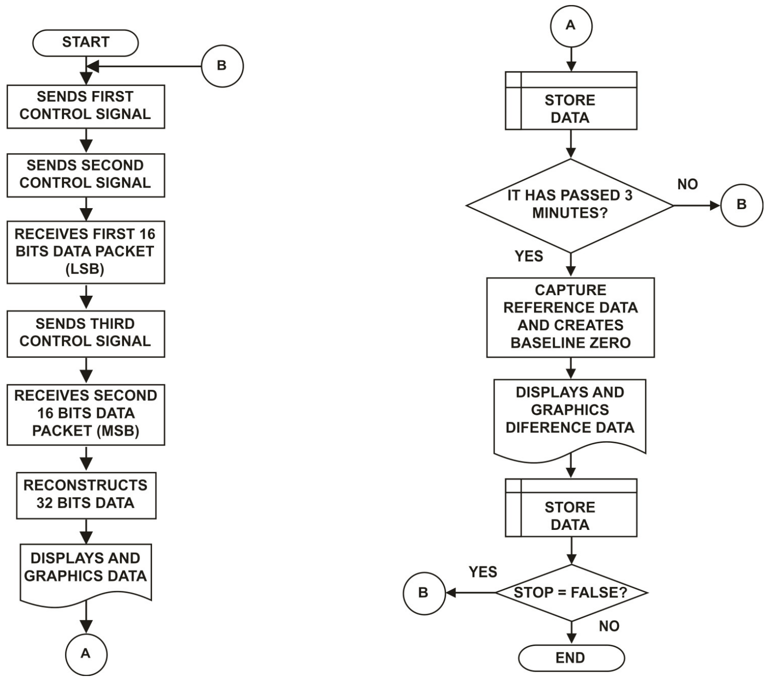


Fig. 6. Acquisition Data flowchart of the software algorithm in the computer.

sing film, which is related to the decrease in QCM resonant frequency by applying a gas to the sensor.

3 Experimental Results

Once the USB-DAQ card for the data acquisition, the frequency counter with the FPGA and the computer software were achieved, the connection of the whole system was performed. A test firmware was developed to produce an incremental data within the FPGA using the same information transmission strategy. The preliminary experimental set-up was implemented using a function generator, an oscilloscope, an Altera development board DE-1 connected with the acquisition card USB-DAQ for the purpose to verify that the frequency counter data were correctly obtained by the computer. The preliminary experimental set-up is shown in the Figure 7.

A 100 KHz square signal was applied to the system taking the function generator as pattern. The obtained information in the computer generated by the FPGA frequency counter corresponds quite well with the input frequency. Subsequent, higher frequencies (1 MHz, 10 MHz, 20 MHz and 50 MHz) were tested with satisfactory results and it can be said that the system has a stable behavior similar to the function generator (1ppm). Until then the software was only able to display the data, plots and store information. Then, it was performed a test using real sensors with controlled temperature. The experimental set-up is shown in Figure 8. In order to perform such test, additionally it was used a refrigerated bath (RTE-10, Neslab) in order to keep the sensor temperature controlled. Also, it was used a measurement chamber made of Teflon which is capable of resisting many organic solvents (See Figure 8). The sensor was placed inside the measurement chamber. In this case a 12MHz sensor coated with an ethyl cellulose sensing film of 29 KHz (Δf) was used. The response was measured applying three 7600 ppm concentration steps of ethanol and the sensor response was measured and stored. For this experiment the entire software was used for a single channel where the response of the sensor can be visualized in the computer.

At the beginning of the test the sensor response is let to stabilize for approximately three minutes to capture the baseline data, while the sensor response is plotted and stored. Once the baseline data is captured, a difference is made between this value and the response of the sensor to obtain the absolute response (Δf); all these data are plotted and stored as is shown in Figure 9. In the left chart the sensor raw frequency data are shown, while in the right chart the frequency decrement of the sensor (Δf) is plotted. It could be observed the response of the sensor when the ethanol sample was injected as well as the frequency decrement of approximately 492 Hz after 15 minutes of stabilization. A second sample was injected, which provoked a frequency decrement of approximately 381 Hz for a total decrement of 873Hz after stabilization, 14 minutes after the second injection. The third sample was injected and the sensor had a frequency decrement of 303 Hz for a total decrement of 1176Hz after 17 minutes. Finally the measurement chamber was purged pumping air and the sensor response recovery can be observed. The test finishes after 50 minutes. All these data were displayed, plotted and stored into a file. The plotted response is quite stable and smooth since the sensor stability is approximately ±1Hz. Therefore, it can be said that the frequency counter was able to measure with such stability.

Finally, the system was scaled to two channels and it was tested using real sensors exposed to ethanol vapor at room temperature. The results are shown in Figure 10, where can be observed that the system can register the frequency response of both sensors at the same time. The system can register frequency variations that take place in a time period of one second.

4 Conclusions

A frequency counter using an FPGA was developed to measure the response of gas sensors based on quartz crystal resonators. Tests on the communication, among the FPGA, the data acquisition card and the computer were performed, generating an internal data within of the FPGA and verifying the effectiveness of the system. There were performed tests on the whole system integrated using as input data a function generator and a stability similar to this instrument (1ppm) was obtained. It was achieved a software upgrade in order to process the information gathered from the sensors, which means to obtain the raw frequency of the sensor, as well as the frequency decrement (Δf). In order to verify the system performance, there were performed tests using a gas sensor and applying three concentration steps of ethanol vapor. The obtained results showed a smooth behavior of the response with a stability of approximately ±1Hz for 12 Mhz sensors. The system was scaled to two channels

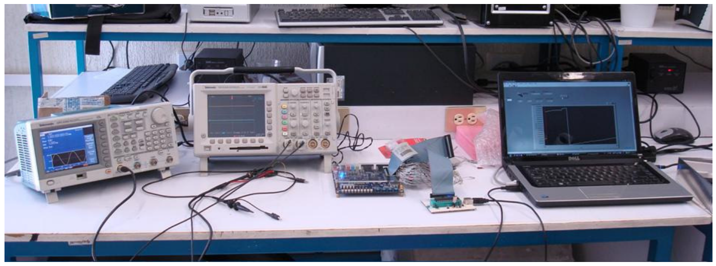


Fig. 7. Experimental set-up to measure frequency with a function generator.

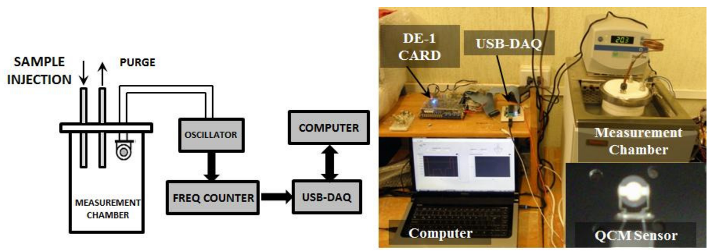


Fig. 8. Experimental set-up using gas sensors at controlled temperature.

using a preliminary software version and performing tests for two gas sensor exposed to ethanol gas at room temperature.

Currently a work under progress is on the information process to achieve a system robust enough in order to obtain a good sensor characterization and be able to automate the whole system, as well as a four channels system expansion.

Acknowledgments

This work was financially supported by the project Conacyt Jovenes Investigadores 61126.

References

1. H. Troy Nagle & R. Gutierrez-Osuna, S. S. Schiffman. The How and Why of Electronic Noses. IEEE Spectrum, September (1998)
2. J. W. Gardner, P. N. Bartlett, Electronic Noses – Principles and Aplications. Oxford Science Publications, (1999)
3. S. Muñoz-Aguirre, T. Nakamoto, A. Yoshino, T. Moriizumi, Odor Approximation of fruit flavors using a QCM odor sensing system. Sensor an Actuators B 105 (2005) 144-149
4. A. Arnau, Piezoelectric Transducers and Applications, Springer, (2004)
5. S. Muñoz Aguirre, "Study of sensing films structure controlled by LB method for QCM odor/gas sensor", Doctoral Thesis, Department of Electrical Engineering, Tokyo Institute of Technology. June (1999)

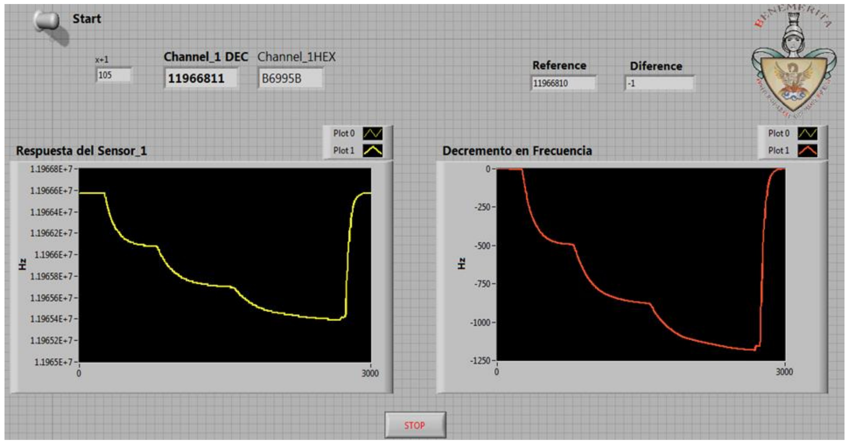


Fig. 9. Sensor response plotted as raw data (left) and frequency decrement (Δf)

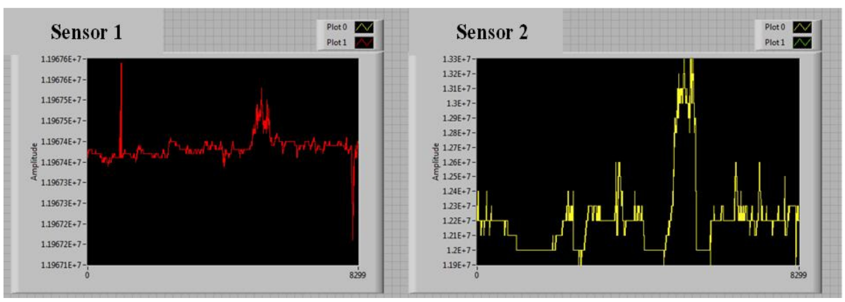


Fig. 10. Data obtained using two sensors tested at room temperature with the two-channel frequency counter.

6. S. Muñoz-Aguirre, TakamichiNakamoto, ToyosakaMoriizumi. Study of deposition of gas sensing films of quartz crystal microbalance using an ultrasonic atomizer. Sensors an Actuators B 105 (2005) 144-149
7. W. H. King. Jr., Piezoelectric Sorption Detector, Analytical Research Division, Esso Research and Engineering Co., Linden N.J. Vo.36, No. 9 Aug (1964)
8. SRS Application Notes, QCM- Quartz Crystal Microbalance Theory and Calibration. Stanford Research Systems. http://www.thinksrs.com/downloads/PDFs/ApplicationNotes/QCMTheoryapp.pdf
9. http://www.home.agilent.com/
10. http://www.terasic.com.tw/cgi-bin/page/archive.pl?Language=English&CategoryNo=53&No=83&PartNo=2#section
11. Microchip. 28/40/44 Pin High Performance, Enhanced Flash USB Microcontrollers DS39617A, (2003)
12. Custon Computer Services. C Compiler Reference Manual. (2009)
13. Eric Lopez Pérez. Ingeniería en Microcontroladores, Protocolo USB (Universal Serial Bus)
14. Derek Liu Asia & Pacific Operation Microcontroller Division. USB Device Development with the MC9S08JM60, Freescale Semiconductors Application Note (2008)
15. http://digital.ni.com/