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Evaluation of calibration transfer strategies between Metal Oxide gas sensor arrays

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Abstract

Inherent variability of chemical sensors makes necessary individual calibration of chemical detection systems. This shortcoming has traditionally limited usability of systems based on Metal Oxide (MOX) sensor arrays and prevented mass-production for some applications. Here, aiming at exploring transfer calibration between electronic nose systems, we exposed five identical 8-sensor detection units to controlled gas conditions. Our results show that a calibration model provides more accurate predictions when the tested board is included in the calibration dataset. However, we show that previously built calibration models can be extended to other units using a reduced number of measurements. While baseline correction seems imperative for successful baseline correction, among the different tested strategies, piecewise direct standardization provides more accurate predictions.

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1. Introduction

Although the initial expectations for electronic noses –systems composed of a chemical detection platform coupled with machine learning algorithms to recognize gas samples–, such systems have not found massive use beyond laboratory settings and are far from fulfilling industry requirements. Transfer calibration between systems has been identified as one of the main technological difficulties that burden the road from research environments to industry [1]. Previous research studies focused on calibration transfer between MOX sensor arrays were based on sensors sharing the same conditioning electronics, on only two independent platforms, or on one gas as reference [2-4]. Here, in the search for efficient calibration transfer methodology, we generated a dataset with 5 twin sensing platforms exposed to four analytes with several repetitions of each concentration level distributed in time.

2. Experimental setup and data generation

We implemented 5 independent sensing platforms based on identical designs. Each platform included 8 MOX sensors and integrated custom-designed electronics for sensor control and signal conditioning. In particular, each sensing platform included 4 different types of commercially available sensors (Figaro USA Inc., Glenview, USA) to generate multivariate responses to the different presented stimuli. The control electronics allowed to set the operating temperature of each sensor individually. Each sensor array included two units of the following sensors: TGS2602, TGS2610, TGS2611, TGS2612. In order to increase the diversity of the detection platform, the replicas of each sensor type operated at two different temperatures set by the voltage in the heater: 5.0V and 5.65V. Hence, due to the sensor diversity and different operating temperatures, the 8 channels of any detection platform will provide different responses to the presented stimuli.

Variations in the composition of gas mixtures induce changes in the MOX sensor's conductivity. We developed an experimental setup to acquire continuously the conductivities of the developed detection platforms while the gas conditions were controlled. The sensor array was placed in a 60 ml measurement chamber, where the gas sample was injected at a constant flow of 400 ml/min. Finally, the resulting mixture circulated continuously through the measurement chamber and was collected by the exhaust system. The sensors' conductivities were acquired continuously at 100 Hz throughout the complete experiment. Figure 1 shows one of the detection units with 8 MOX sensors in the measurement chamber.

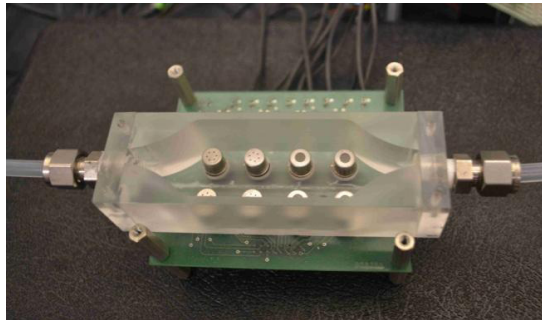


Fig 1: We developed 5 chemical detection platforms following the same design. Each platform included 8 MOX sensors and was placed in a measurement chamber where gas samples flowed at 400 ml/min. The lid of the chamber was removed for the picture.

We followed a reproducible experimental protocol to measure the response of the chemical detection units. Each day, a different unit was tested, which included the presentation of 40 different gas conditions. In particular, the unit under test was exposed to 10 concentration levels of Ethanol, Methane, Ethylene, and Carbon Monoxide (see Table 1). The design of the experiment was the same for the four tested volatiles: First, a constant flow of air (carrier gas) circulated through the sensing chamber for 50 s. Second, the carrier gas was mixed with the selected volatile at the desired concentration level and the resulting gas mixture circulated during 100 s. Finally, the vapor was purged out from the test chamber by re-circulating only clean air during the subsequent 450 s. Then, once the recovery phase was complete, the concentration phase of a new measurement could restart. Hence, the total duration of the experiment was 600 s. Moreover, the sensory units were tested several times (two or four) over a period of 22 days. As a result, the generated dataset included 640 different measurements.

Table 1. Tested analytes and concentration levels.

Analytes	Concentration levels (in ppm)
Ethylene	12.5, 25, 37.5, 50, 62.5, 75, 87.5, 100, 112.5, 125
Ethanol	12.5, 25, 37.5, 50, 62.5, 75, 87.5, 100, 112.5, 125
Carbon Monoxide	25, 50, 75, 100, 125, 150, 175, 200, 250
Methane	25, 50, 75, 100, 125, 150, 175, 200, 250

3. Results

For each measurement, we acquired the response of the eight sensors exposed to the defined gas conditions. Figure 2 shows that the acquired signals indeed follow the changes in the composition of the gas sample. Using the collected dataset and the model of Clifford-Tuma [5], we estimated sensors' variability from the disparity in the parameters of the fitted models. Figure 3 shows that variability of the models for different gases and different replicas.

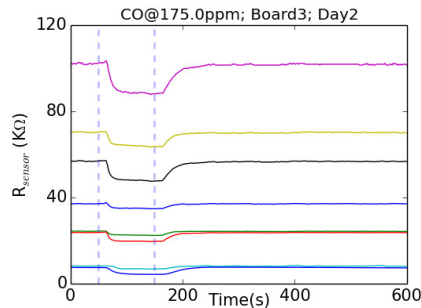


Fig 2: Acquired time series from one detection unit exposed to CO. The sensors are able to follow the changing gas conditions (dashed vertical lines indicate start/stop of selected gas release).

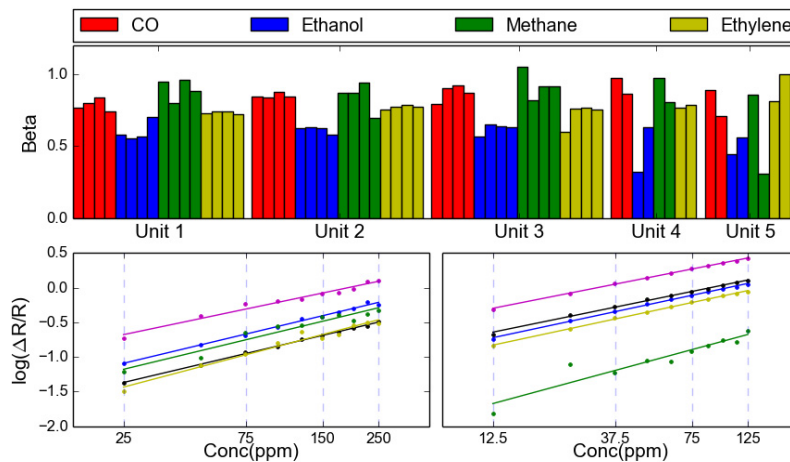


Fig. 3: Sensors' models fitted for five sensors of the same type placed in different units. Parameter β – which is estimated from $\log(\Delta R/R) - \beta \log(\text{conc})$ – is presented for the four gases (grouped in colours) and for the number of repetitions of each gas. Bottom panels show the fitted models and acquired data. Each color represents a different sensor of the same type exposed to CO (bottom left) and Ethylene (bottom right).

We evaluated the ability of calibration models built with one chemical detection unit to predict the gas concentration level measuring with other units. In order to build the calibration models, we concatenated the response of the 8 sensors contained in the same board. The acquired signals were downsampled to 300 samples per measurement and baseline was corrected using the first portion of the acquired signal. Hence, for each measurement, we concatenated 2400 features in a vector.

We trained a Support Vector Regression (SVR) model with two random repetitions selected from the same board. Therefore, the training dataset included 20 measurements of the same volatile at 10 concentrations collected in two different days using the same sensing unit. The metaparameters of the SVR model were selected such that the error in cross-validation (5-fold cross-validation) was minimized. The resulting model was tested with data acquired with the same board but not included in training set, and also with data acquired from the other boards. Table 2 shows the

accuracy of the regression models, with the mean and standard deviation after 40 random repetitions. The accuracy of the models degrades when calibration models are utilized to predict data from units not used in training (compare *Same-board* and *Baseline correction* in Table 2).

To extend calibration models trained with one unit to other units, we explored Direct Standardization (DS), Piecewise Direct Standardization (PDS) and Generalized Least Squares Preprocessing (GLS) [6]. We compared the mentioned approaches with the error provided by processing captured data with other units with no further processing, and the prediction error of the calibrated unit to predict concentration level of measurements not included in training. Table 2 shows that PDS is the strategy that provides more accurate results, extending calibrated models.

Table 2: Approaches tested for calibration transfer and corresponding prediction error.

Methodology	Error in test (in ppm)
Same-board	11.3±3
Baseline correction	32.3±9
Baseline + DS	15.1±5
Baseline + PDS	13.3±4
Baseline + GLS	17.1±3

4. Conclusions

We developed five identical chemical sensing boards with MOX sensors and evaluated the ability of calibration models to be extended to other boards. We found that prediction accuracy degrades significantly if calibration models are directly used for boards not included in training. However, simple strategies such as DS, PDS or GLS enable calibration transfer after baseline correction and when detailed rising and decay transients are captured. These conditions are not met in continuous monitoring scenarios [7-8], making necessary further exploration of calibration transfer techniques.

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