Gas mixing apparatus for automated gas sensor characterization

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Abstract
We developed a computer-controlled gas mixing system that provides automated test procedures for the characterization of gas sensors. The focus is the generation of trace gases (e.g. VOCs like benzene or naphthalene) using permeation furnaces and pre-dilution of test gases. With these methods, the sensor reaction can be analyzed at very low gas concentrations in the ppb range (parts per billion) and even lower. The pre-dilution setup enables to cover a high concentration range (1:62 500) within one test procedure. Up to six test gases, humidity, oxygen content, total flow and their variation over time can be controlled via a LabVIEW-based user-interface.

Keywords: gas mixing, trace gas, permeation, gas dilution, air quality monitoring

Introduction
Since gas sensors in their applications are often exposed to a multitude of different chemical substances and environmental conditions, the characterization of gas sensors and gas measurement systems requires a versatile gas mixing apparatus (GMA). Gas sensors such as metal-oxide gas sensors (MOX) or gas-sensitive field effect transistors (FETs) are sensitive to several oxidizing and reducing gases as well as humidity. The development of application specific gas sensor systems, therefore, requires extensive test procedures, which can be performed by this automated test device. In the literature, different approaches of gas mixing systems can be found [1–3].

This GMA is based on dynamic volumetric methods described in ISO 6145 [4] and has different fields of application due to its versatility.

One primary goal is the evaluation of the suitability of gas sensors for indoor air quality monitoring which is mainly based on the ability of the sensors to selectively detect relevant volatile organic compounds (VOC). The regulatory concentration limits for hazardous VOCs have been decreased successively: recommended maximum exposure limits for formaldehyde and naphthalene are now in the ppb range [5]. Hence, the generation of defined gas mixtures in this concentration range is necessary to reproduce typical indoor atmosphere scenarios to study sensitivity, stability and selectivity of gas sensors to harmful gases and therefore their suitability for air quality monitoring.

Another field of application is the simulation of typical exhaust gas mixtures from combustion processes, e.g. automotive emissions or emissions from power plants. Here, gas sensors based on silicon carbide (SiC-FETs) were successfully tested. Silicon carbide has a wide band gap of 3.2 eV which allows operating temperatures up to 700 °C without losing its semi-conductive behavior. In addition, silicon carbide is chemically inert, making it suitable as a substrate material for use in harsh environments, e.g. directly in the exhaust gas stream [6]. For this application, the GMA is able to generate gas mixtures with typical exhaust gases such as carbon monoxide (CO) and nitrogen oxides (NOₓ) with varying oxygen levels.
Figure 1. Schematic diagram of the different gas lines of the GMA. In the actual setup, two permeation gas lines and one dilution and bottle gas line are used.

Figure 2. Mechanical setup as CAD model: the permeation gas generation with two permeation ovens (1), MFCs and pressure regulators (2) is placed below the main platform (570 × 380 mm²), the other MFCs (5), valves and gas mixing chamber (3) and sensor test chamber (4) are placed above.

Hardware

The GMA can be divided into five parts (i)–(v): gas mixing, humidification, permeation gas generation, gas dilution and gas measurement (figure 1).

(i) The system (figure 2) is equipped with 11 mass flow controllers (MFCs), which control the mass flow to a set-point value independent of pressure and flow resistance variations. Two types of MFCs (Christian Bürkert GmbH & Co. KG, Ingelfingen, Germany) are used working with different thermal anemometer sensors. For test gases, we use a Type 8715 MFC with a bypass capillary sensor that shows a slow reaction when the flow changes but is also suitable for high concentrations of aggressive gases [7]. It has an accuracy of ±1.5% of rdg (reading) and ±0.3% FS (full scale). Here, the defined switching of gas concentration after a pre-ventilation phase is realized via a 3/2-way valve (Bürkert, Type 6624) with the FFKM seal material. The carrier gas flows (e.g. synthetic air or nitrogen) are controlled by MFCs with a micromechanical sensor (type 8713) that allows a flow set-point regulation within a few 100 ms and an accuracy of ±0.8% of rdg and ±0.3% FS [7]. Both types of MFCs have a high repeatability (0.1% FS) and are calibrated and certified by the manufacturer. The same carrier gas is used for test gas mixing, permeation gas generation and test gas dilution, so the influence of pollution of the carrier gas on sensor response can easily be determined and discriminated from the target test gas. We use synthetic zero-air with a purity level of 5.0 which guarantees a sum concentration of impurity gases like CO, H₂ and CH₄ lower than 10 ppm. Note that in order to be able to measure low concentrations of a target gas, the same carrier gas has to be used throughout the system, which thus detects the influence of the target gas on the sensor baseline in background similar to a sensor in real operating conditions. The relative error of the ratio between test gas flow and total gas flow is the sum of the relative errors of both flows. Since the test gas flow is much lower than the total gas flow, the error can be simply calculated as the sum of relative error of test gas MFC (type 8715) and carrier gas MFC (type 8713). At the set-point of 10% FS of both MFCs involved, the relative error of the gas mixing layer is below 8.3%.

Set-points below 10% FS evoke a warning by the control software. The typical concentration uncertainty of a bottled gas like CO is ±2%, so the overall relative concentration error in this case can be estimated to be below 10.3%.

A mass flow meter (MFM) measures the flow through the sensor chamber and detects deviations of the total flow, which points to leakages in the system. The tubing material is stainless steel due to its favorable chemical properties.

(ii) Humidification is required providing selectable but constant levels of absolute humidity. The level can be selected by the ratio between the volumetric humid carrier gas flow \( V_{\text{humid}} \) and total flow \( V_{\text{total}} \). The humid carrier gas (air/nitrogen) is saturated with water vapor in a glass bubbler, which is temperature-
regulated by a thermostat (LAUDA RE307). The set-point temperature is slightly below room temperature to avoid condensation in the tubing. The absolute humidity of the humid carrier gas $AH_{hum}$ can be calculated from the saturation vapor pressure according to the ideal gas law. In the total gas flow, the absolute humidity can be calculated by

$$AH_{total} = \frac{V_{hum}}{V_{total}}AH_{hum}. \quad (1)$$

As default setting the software will display the humidity as a fraction of the saturation humidity at thermostat temperature. Due to the cooling of the glass bubbler, we achieve in theory a maximum relative humidity (RH) in the gas sensor chamber of approx. 95%. The relative humidity and temperature of the test gas flow blend are monitored via the combined temperature and capacitive humidity sensor Driesen+Kern SHT75 (accuracy ±2% RH FS in the range 10–90% RH).

(iii) The generation of low concentration VOCs is based on the principle of permeation. Permeation is defined as the mass flow $m$ of a gas through a solid state membrane (of thickness $d$). The mass flow is proportional to the area $A$, the molar mass $M$, the material- and gas-dependent permeation constant $P$ and the partial pressure difference $P_1 - P_2$. It is reciprocal to the thickness $d$ of the membrane:

$$m = A \cdot M \cdot P \cdot \frac{P_1 - P_2}{d}; \quad P_2 \ll P_1. \quad (2)$$

$P$ depends linearly on the diffusion constant $D$ and the sorption constant $S$, which are both strongly temperature dependent [8]. The diffusion can be calculated using Fick’s first law, where the diffusion constant $D$ is proportional to $e^{\frac{\Delta H}{RT}}$ (with $E_D$ being the activation energy and $k_B$ the Boltzmann constant). The sorption constant $S$ according to Henry’s law is proportional to $e^{\frac{\Delta H}{RT}}$ (with $\Delta H$ being the enthalpy of sublimation from the sorbent). The mass flow $m$ (equation (2)) can therefore be controlled by the temperature $T$:

$$m \sim P = D_0 \cdot S_0 \cdot e^{\frac{\Delta H}{RT}}. \quad (3)$$

We use a permeation oven with a temperature range of 30–110 °C and an accuracy of 0.01 °C (VICI Dynacalibrator 150 [9]). The carrier gas flows around the permeation tube inside an inertized (glass-coated stainless steel) oven chamber.

The concentration of the test gas is given by the ratio of permeation and carrier volume flows [8]:

$$\text{conc.} = \frac{\text{molar volume} \cdot \frac{m}{V_{carrier}}}{\text{molar mass}}. \quad (4)$$

The accuracy of the permeation line is given by the error sum of the permeation rate and flow tolerances. There are several factors that influence the permeation rate, e.g. length and thickness of the tube and temperature (equation (3)). For the very precisely temperature controlled permeation ovens, the contribution to the total concentration error is negligible (<1%). The permeation rate can be calibrated by the mass loss of the permeation tubes. For low permeation rates (e.g. 10 ng per minute), the calibration is time consuming and requires an accurate weighing process. Therefore, the error for these devices is typically high (more than 10%).

The worst case relative concentration error resulting from MFCs is ±12.1% (±3.8% carrier gas generation and ±8.3% gas mixing, supposing MFC set-points of 10%).

(iv) To cover a wide concentration range, the GMA is also able to pre-dilute regular pressurized (bottled) test gases. There are different principles for pre-dilution [10, 11] here, we use the continuous volumetric flow mixing approach. With the pre-dilution, a dynamic concentration range of 1 up to 62 500 can be achieved using the following formula, where $c_{set}$ is the concentration set-point, $c_{bottle}$ the concentration of test gas in the bottle, $V_{bottle}$ the MFC flow from the bottle gas, $V_{dilution}$ the carrier flow for predilution, $V_{test}$ the flow of the prediluted test gas and $V_{total}$ the sum flow through the sensor chamber:

$$c_{set} = \frac{V_{bottle}}{V_{bottle} + V_{dilution}} \cdot \frac{V_{test}}{V_{total}}. \quad (5)$$

The characteristic curve for pre-dilution is divided into four different areas, in which one flow is varied linearly with the concentration change and the two other flows are kept constant (figure 3).

This approach is suitable only for gases with high volatility due to the fast changes of the pre-dilution ratio in a measurement procedure with changing concentrations. When working with gases with low volatility, this set-point curve could cause carry-over effects. The worst case relative concentration error of prediluted gas blends can be estimated at ±16.6% (±8.3% gas mixing, ±8.3% gas dilution).

(v) Measurements of the mixed test gas flow are taken in the sensor chamber, which can be adapted to different sensor types and models. The GMA comes with two possible sensor operating modes:
(a) **Constant heating voltage** (corresponding to constant operating temperature in constant gas flow). The sensor signal (can either be voltage, current or resistance) is measured by a digital multimeter (Keithley 2000) with a resolution of $6 \frac{1}{2}$ digits.

(b) **Temperature cycled operation** (TCO) With the Sniffchecker (3S GmbH, Saarbrücken, Germany), gas sensors can be driven in a time-dependent temperature controlled heating mode. The user configures a set of temperature steps or linear ramps, which are continuously repeated (temperature cycle). In this mode, it is possible to extract further characteristics from the sensor response [12] that can be analyzed using multivariate statistics (“virtual multi-sensor”) [13]. The sensor reaction is measured with a non-inverting operational amplifier using a 12-bit ADC with variable pregain.

In figure 4, the control structure of the GMA is shown. Measurements are controlled by a PC that sends the set-points to the peripheral components and receives the measured data via USB in order to guarantee the correct synchronization
between settings (i.e. gas flows adjusted with the MFCs and valve position) and measured data, especially the temperature cycle in the TCO mode. The digital MFC communication has a physical EIA-485 layer and a software protocol that is based on HART [7]. An important component is also the Sniffchecker that has to perform different tasks. It controls the TCO and measurement of up to four gas sensors, switches the 3/2-way valves and provides the timestamp on which the measurement process is based.

**Software**

The developed software is based on LabVIEW® 2011 (National Instruments) and consists of four main configuration parts: gas lines, data acquisition, default values and process configuration.

The gas line configuration (GAS PARAMETER) defines the test gas setup (e.g. used gas lines, bottle concentrations, gas type correction factors for the MFCs) and determines the usable flow, humidity and concentration ranges. Measurement procedures can be configured via a graphical user interface accessing two input clusters: SEGMENTS and LOOPS. In a measurement process configuration (figure 5), SEGMENTS are the base elements and consist of the following values:

- gas concentrations [ppm]
- valve settings
- oxygen content (%)
- relative humidity (%)
- duration (s).

Different concentration set-points can be iterated by a queue of SEGMENTS, which are arranged in an array. The user can add or delete SEGMENTS at any position. The LOOP function allows to automatically create stepwise concentration profiles. Every LOOP modifies one parameter of the SEGMENTS within the configured interval and is defined by a START and END SEGMENT, the number of repetitions and the start and increment values of the parameter being changed. Overall, there are three hierarchical levels of LOOPS: the FLOW LOOP modifies the total flow, the OUTER LOOP influences humidity or oxygen content and the INNER LOOP the gas concentrations (figure 6).

In order to clean the tubing from adsorbed gas, the system can be flushed using a pre-ventilation function.

Due to the reciprocal dependences on concentrations, humidity, oxygen content and total flow, the range of possible values varies, determined by the other inputs. So, the program informs the user about the value range, invalid inputs and shows errors in configuration (figures 7 and 8). The software is designed as an event-driven state machine, which means that the program recalculates the measurement process after every user input. In a first step, the software checks the LOOP configuration for errors such as overlapping. If no error is found, concentration and set-point sequences are generated and shown in a preview diagram. After that, a second check function searches for invalid set-points, i.e. below 0% or above 100%, before the user can start the measurement. Settings in the inaccurate range of the MFCs (below 10%) create a warning message.

The software structure is modular and up to six test gas lines can be chosen. Each test gas line can be user-defined as pressurized (bottle) test gas, permeation oven line or pre-diluted gas. The test gas type determines the number of MFCs involved, which range from one (bottle gas) to three (pre-diluted gas). This results in the maximum possible number of 22 MFCs. Note that an increasing number of MFCs decreases the sampling rate (approx. 100 ms per MFC). The current configuration works with one pre-dilution line, one bottle gas line and two permeation lines (figure 1).

The selected values for total flow, humidity and oxygen content together with the selected gas concentrations determine the various MFC set-points (figure 9). The total flow,
humidity and test gas concentration set-points result directly in a volumetric flow ratio and thus, in a set-point for the MFCs. The situation is different with the carrier gas flows. They depend on the one hand on the user-defined oxygen set-point, and on the other hand on the composition of the test gas mixture. Therefore, the air content must be determined before the carrier gas flows can be computed.

The calculation process from the concentration to the flow set-point consists of the following three phases:

1. Calculation of test gas (bottled gas, permeation gas or pre-diluted gas) and humidity flow set-points (in per cent of maximum flow) through the sensor chamber with the concentration set-point \( c_{\text{set}} \), the test gas concentration \( c_{\text{test}} \), the total flow set-point \( \dot{V}_{\text{total}} \), the maximum MFC flow \( \dot{V}_{\text{MFC}} \) and the gas type correction factor \( k_i \):

\[
\text{set point}[%] = \begin{cases} 
\frac{c_{\text{set}} \cdot \dot{V}_{\text{MFC}} \cdot k_i}{c_{\text{test}} \cdot \dot{V}_{\text{MFC}}} \cdot 100\%, & \text{if valve closed} \\
0\%, & \text{if valve open} 
\end{cases}
\] (6)

2. Calculation of air content in the previous test gas mixture with the set-points \( u_i \) (equation (6)):

\[
c_{\text{air}}[%] = \sum_i \frac{u_i \cdot \dot{V}_{\text{MFC}} \cdot c_{\text{air}}}{\dot{V}_{\text{total}} \cdot k_i}.
\] (7)

Nitrogen content is determined likewise.

3. Calculation of the carrier gas flows for air and nitrogen dependent on the oxygen set-point, here shown for dry air:

Table 1. VOC background of the GMA as evaluated in [14] according to ISO 16000-6 and associated Chemical Abstracts Service (CAS) registry numbers.

| Compound                     | CAS number | \( c (\mu g \, m^{-3}) \) | \( c (\text{ppb}) \) |
|------------------------------|------------|---------------------------|----------------------|
| Benzene                      | 71-43-2    | 0.17                      | 0.053                |
| Toluene                      | 108-88-3   | 0.06                      | 0.016                |
| Chlorobenzene                | 108-90-7   | 0.26                      | 0.056                |
| Camphene                     | 79-92-5    | 0.29                      | 0.052                |
| Benzoaldehyde                | 100-52-7   | 0.2                       | 0.046                |
| Phenol                       | 108-95-2   | 0.3                       | 0.06                 |
| Benzonitrile                 | 100-47-0   | 0.61                      | 0.144                |
| Octanal                      | 124-13-0   | 0.1                       | 0.019                |
| Benzyl alcohol               | 100-51-6   | 0.19                      | 0.043                |
| Acetophenone                 | 98-86-2    | 0.62                      | 0.126                |
| Naphthalene                  | 91-20-3    | 0.24                      | 0.046                |
| Bicyclo[2.2.1]-heptane, 2-chloro-2,3,3-trimethyl | 465-30-5 | 16.2                      | 2.6                  |

Total VOC 24.3
Figure 10. Response of a UST 1000 operated at 250 °C versus humidity and total flow. Comparison between set-points and measured values.

\[
\text{set point}_{\text{air,dry}}[\%] = (c_{\text{air set}} - c_{\text{air actual}}) \cdot \frac{V_{\text{total}}}{V_{\text{MFC}}}
\]

Figure 8 shows an example for a configured concentration profile (used in measurements of figure 12) and the resulting MFC set-points. The concentration diagram takes the valve states into account and, thus, represents the gas concentrations in the gas sensor chamber. The varying flow set-points of the dry air MFC are caused by the set-point algorithm keeping the total flow constant and compensating test gas and humidity flow deviations.

**Evaluation**

Prior to the use of the GMA, potential contaminations of the system were analyzed by taking air samples via TENAX® adsorbent tubes. These samples were analyzed using gas chromatography (GC6890N, Agilent Technologies Inc.) and mass spectroscopy (MSD5973, Agilent Technologies Inc.) [14]. The GC/MS characterization (table 1) shows a low level of usual VOC concentrations except for one contaminant, which is identified as 2-chloro-2,3,3-trimethyl-bicyclo[2.2.1]heptane based on the NIST library [15], albeit with a low degree of confidence. Further tests showed that this compound is emitted inside the system, most likely by the mass flow controllers either as a residue of the production process or emitted from the sealing materials used in the MFCs.

We have tested the basics features of the GMA using an SnO₂-based metal-oxide gas sensor (UST-1000, Umwelt-Sensortechnik GmbH, Geschwenda, Germany, with broad sensitivity spectrum versus various reducing gases) at 250 °C. Figure 10 shows the influence of total flow and relative humidity on the sensor reaction. Even though the sensor is temperature controlled, the conductance slightly decreases (approx. 1.3%) with increasing flow from 100 to 500 ml min⁻¹ indicating a slight cooling effect of the air flow. The measured flow also depends on the relative humidity and varies by approx. 2.4% between minimum and maximum humidity. This effect can be explained on the one hand by the increasing mass flow due to the adsorbed water vapor, and on the other hand by the changing physical properties of the gas stream that show a slight effect on the thermal anemometer measurement principle [16]. After changing the humidity set-point there is a running-in characteristic approaching a constant value, and the difference between set-point and the measured value of relative humidity is up to 6% at 500 ml min⁻¹ 30 min after the humidity set-point change.

Figure 11 shows the high dynamic range of the concentration that can be achieved with the pre-dilution system. The concentration values are obtained by the measured flow controllers.
flow of the MFCs (equation (5)); the resulting sensor response to these concentrations is shown at the top of the graph. In the beginning, a running-in characteristic of the gas sensor is observed due to the conductance base-line drift of the sensor. Therefore, the results for increasing concentrations are not evaluated. After 8 h, the sensor is considered to be in equilibrium, since the base-line remains nearly constant. The sensor reaction at descending concentration steps indicates a slight carry-over effect in the pre-dilution system. The concentration of the test gas formaldehyde was varied over four orders of magnitude. Even extremely low concentrations of formaldehyde at $10^{-4}$ ppm (or 0.1 ppb) cause a distinct reaction of the SnO$_2$ gas sensor UST 1000.

In figure 12, measurements assessing the permeation units are shown. The concentrations are calculated by measured gas flows (equation (5)) and the permeation rate of the tubes (equation (4)). Note that the overall uncertainty of the applied gas concentration is dominated by the uncertainty of the permeation rate in the permeation tubes (according to equation (4)) given by the manufacturer that is ±15% for the naphthalene and ±50% for the benzene tubes used in this measurement. The graph above shows the sensor reaction of a WO$_3$-based gas sensor (UST 5000, Umwelt-Sensortechnik GmbH, Geschwenda, Germany) to the test gases benzene and naphthalene that are generated by permeation ovens. This sensor response is extracted from a TCO of the gas sensor heater by selecting and plotting defined data-points of each cycle (quasi-static operation). The concentration profile of the VOCs is repeated for zero-air atmosphere and against a background of 2 ppm ethanol as a typical interfering gas (generated from a bottled test gas). The base value of conductance rises significantly in the ethanol background and the sensitivity to naphthalene (5 ppb) decreases from 17% to 9% at 200 °C.

The test procedure shown in figure 13 simulates exhaust gas emissions. Four oxygen levels from 6 up to 20% were used wherein the CO test gas concentration is varied from 5 up to 45 ppm to reproduce different kinds of combustion. The varying oxygen concentration based on the ratio change between the carrier gases air and nitrogen (equation (8)) affects the sensor response of MOX and FET sensors that are both sensitive to oxygen. For an SnO$_2$ sensor (e.g. the UST 1000), the conductance rises with decreasing oxygen levels due to the changing surface coverage of O$^-$-ions at the grain boundaries.
used SiC-FET, this results in increasing values for $V_{DS}$ with increasing oxygen levels and decreasing CO concentrations.

**Conclusion**

Measurements have shown that the gas mixing apparatus is a versatile instrument for gas sensor characterization. Up to six test gases can be tested simultaneously, gas concentration can be changed over four orders of magnitude with predilution, ultra-low concentrations can be reliably prepared using permeation ovens and carrier gas composition can be varied during the measurement to simulate changing operating environments. The user is able to easily configure complex test procedures with the graphical user-interface which also checks for errors in the settings.

In the field of air quality monitoring, measurements with the novel GMA have already revealed a high sensitivity of metal-oxide gas-sensors [14] and SiC-FETs [20] to different VOCs with detection limits in the ppb or even sub-ppb range. Furthermore, tests have verified that the system is able to simulate exhaust gas mixtures from combustion processes.

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