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1. Introduction

This document gives additional background information on methods for detecting total volatile organic compounds (TVOC) with the ZMOD4410 Metal Oxide Gas Sensor Module and answers typical questions for individual VOC and TVOC detection. The ZMOD4410's 3 Sensor "S," sensitivity, selectivity, and stability are discussed.

TVOC is the sum of all volatile organic compounds (VOC), and it is referred as a typical indicator for air contaminants, especially for indoor air quality¹. Many technologies for VOC detection have been established in the last decades with different emphasis

Examples:

- Highest possible sensitivity for one specific VOC
- Precise selectivity to distinguish between individual gases
- Extremely accurate measurements, e.g. for research
- Robust technology for harsh environments, e.g. industrial applications
- Low-cost effective methods for mass market

Each user must find a compromise to meet the user's specific measurement requirements. IDT's gas sensor modules are chemiresistors using a catalytic reaction of the gas air molecule on a metal-oxide (MOx) surface. The advantages of this technology are high sensitivity to both TVOC and individual VOCs, a robust design, a small mechanical size, and low cost production in high volume as well as other advantages.

Usually the TVOC concentration is given as either

- Molar ratio: ppm (parts per million) or ppb (parts per billion)
- Physical concentration: mg/m³ (mass per volume)

It is easy to convert from ppm to mg/m³ as seen in Equation 1:

$$c \text{ [ppm]} = \frac{V}{M} * c \left[\frac{\text{mg}}{\text{m}^3} \right] * 10^3 \quad \text{Equation 1}$$

For an ideal gas at ambient temperature (25°C) and atmospheric pressure (1013 mbar), c is the concentration in ppm or mg/m³, V is the molar volume (0.024465 m³·mol⁻¹), and M is the molecular mass of the individual VOC in g·mol⁻¹. Table 1 gives some conversion examples for typical VOC gases. More trivial is the conversion from ppm to ppb since 1ppm equals 1000ppb.

Table 1. Concentration Conversion for Typical VOC Examples

VOC Name	Chemical Formula	Molar Mass [g·mol ⁻¹]	Molar Ratio [ppm]	Concentration [mg/m ³]
Ethanol	C ₂ H ₅ OH	46.069	1	2.0057
			0.5311	1
Acetone	C ₃ H ₆ O	58.080	1	2.3740
			0.4212	1
Formaldehyde	CH ₂ O	30.026	1	1.2273
			0.8148	1
Toluene	C ₇ H ₈	92.141	1	3.7662
			0.2655	1

¹ ZMOD4410 White Paper – Overview of TVOC and Indoor Air Quality; available at www.idt.com/zmod4410.

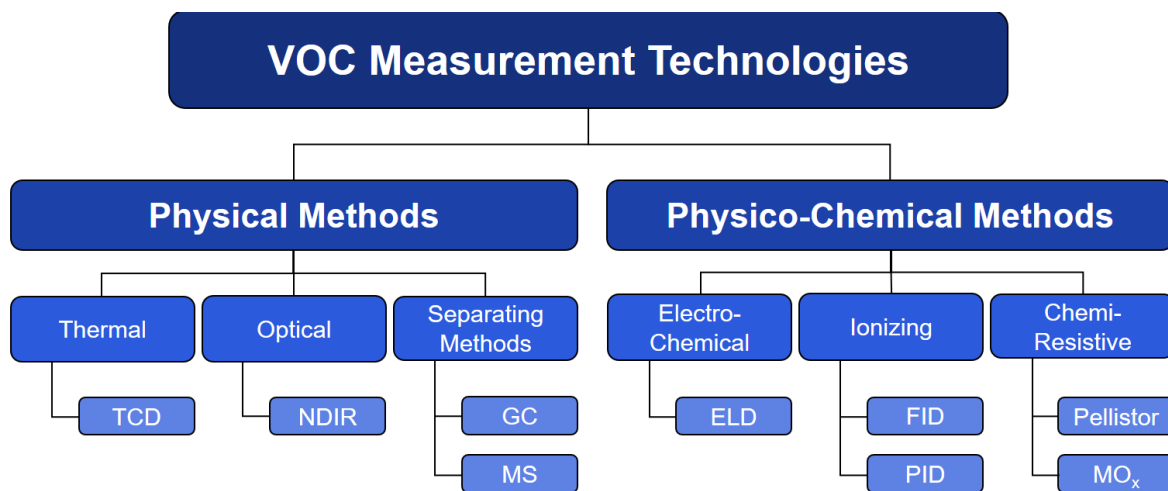
2. Methods for VOC and TVOC Sensing

2.1 Volatile Organic Compounds (VOCs)

In recent years, volatile organic compound (VOC) measurements have gained increasing importance due to the widespread occurrence of VOCs and the need to monitor VOCs in comfort, environmental safety, automation, health, and medical care applications. Depending on the application, a single VOC or the sum of all organic compounds, referred to as the total VOC (TVOC), may be present.

To fit the demands of these applications, a multitude of VOC measurement techniques have been developed within the last decades. Figure 1 gives an overview of the most significant techniques and detectors: thermal-conductivity detectors (TCD), non-dispersive infrared (NDIR) sensors, gas chromatography (GC), mass spectrometry (MS), electro-chemical electrolyte detectors (ELD), flame ionization detectors (FID), photoionization detectors (PID), pellistors, and metal oxide (MO_x) sensors. Depending on their size and complexity, the instruments, which are introduced in the following section, might also be differentiated as portable or stationary devices. Here the demand in inexpensive, miniaturized, portable, and highly sensitive sensors is driving the current industrial sensor development.

Figure 1. Overview of the Most Significant Gas Sensing Technologies



2.2 Physical Methods

These instruments rely on physical measurement principles and do not change the chemical composition of the test gas.

2.2.1 Thermal-Conductivity Detectors (TCD)

Thermal-conductivity detectors use a comparison between the thermal conductivity of the test gas and a reference gas. For this method, the cooling powers of two heater elements (having one heater in the test gas and the other in the reference gas) are compared. An increased gas concentration will give an increased thermal conductivity of the test gas, which lowers the temperature and resistance of the heating element. This resistance change is measured and compared to the reference gas.

Due to the simplicity of this technique, it can be applied to both organic and inorganic gases, where sensitivities of up to 1ppm are achievable. However, the lack of selectivity makes the sensor most suitable for well-defined process-control applications or use with other separation techniques.

2.2.2 Non-Dispersive Infrared (NDIR) Sensors

NDIR instruments use infrared (IR) absorption spectroscopy to infer the composition and concentration of individual gases within a test gas. For this, an infrared beam from an IR source is passed through the test gas and the absorption is measured for each wave length in a spectrum analyzer. The recorded spectra are compared to reference spectra of the individual gas components. By applying the Lambert-Beer law, it is possible to infer the concentration of each component within the test gas and thus its composition.

Due to its working principle, an NDIR sensor can only detect infrared-active compounds. These are mostly heteronuclear or polar molecules such as CO, CO₂, hydrocarbons, ammonia, and many more. Homonuclear molecules, such as most pure gases, cannot be detected as their excitation frequencies lie outside of the spectral range used for NDIR. Major drawbacks of this technique are that gases with similar absorption spectra are hardly distinguishable from each other and NDIR sensors can only provide information on known gas components.

2.2.3 Separating Measurement Techniques

Separating technologies have in common that by using the principle of separation (either by chemical reaction or by mass) the instrument becomes very complex, large in size, and expensive. Due to very low detection limits and extremely high precision, these instruments are handled as a gold standard. However, there are no sensors available using this technology approach and only stationary instruments are available in highly specialized labs.

Gas Chromatography (GC)

Gas chromatography is based on the difference in diffusion velocity of gases through a separation tube (chromatography column). A sample of the test gas is injected into the chromatography column together with a continuous flow of carrier gas. Inside this column, the individual components of the test gas are differently strongly bounded to the filling material and column walls. Thus, stronger-bound molecules are slowed. This leads to a separation in time of the individual gas components at the exit of the chromatography column, where they can be measured individually. A very common method for collecting VOC gas samples in the field is the use of polymer adsorbents. This sample gas tube is then analyzed by thermodesorption to release the VOC into the GC instrument.

Mass Spectrometry (MS)

Mass spectrometry is a technique relying on the mass and charge separation of ion beams in an analyzing magnetic field. For this, an electron beam ionizes a fraction of the test gas in a high vacuum environment. The resulting cloud of ionized gas is accelerated in an electric field and passed through a strong magnetic field. Within this analyzer field, the ionized gas beam splits into its various components depending on the ratio of molecular mass and charge (m/z ratio). Here the lightest and most heavily charged ions are deflected primarily due to the Lorentz force. The intensity of the resulting fan of sub-beams is measured in an angular array of Faraday cups. Each Faraday cup corresponds to a certain mass-to-charge ratio, and these ratios are specific to chemical compounds and molecules.

By measuring the mass spectrum, it is possible to infer the occurrence and concentration of chemical compounds within the test gas. Due to its highly sensitive and fundamental working principle, MS can be applied to almost any gas and is capable of detecting the smallest amounts of gases, even at sub-ppb levels. A disadvantage of mass spectrometry is that organic molecules in particular are partially fractionalized during the ionization and therefore different gases could result in similar fractions.

2.3 Physico-Chemical Methods

Unlike physical instruments, physico-chemical detectors are based on a combination of chemical reactions and physical measurement techniques. Due to ongoing chemical reactions, the test composition of the test gas is chemically changed during the measurement.

2.3.1 Electro-Chemical Electrolyte Detectors (ELD)

The electrolytes used in ELD sensors can be divided into solid state or liquid materials, which influence the cost, size, and design. Their working principle is based on the ionization of the test gas at one electrode and diffusion of the ionized gas through an electrolyte medium towards the counter electrode. For solid electrolytes, this comes with a high power consumption to heat the element to several hundred degrees Celsius.

The resulting current is proportional to the gas concentration and can be directly measured for the amperometric measurement mode. Although these sensors use a well-established technique, they naturally suffer from large drift, especially the liquid electrolytes, which tend to dry out at operation temperatures above ambient conditions, causing large changes in sensor behavior.

2.3.2 Ionizing Methods

Flame Ionization Detectors (FID)

Flame ionization detectors measure an ion current between a hydrogen burner nozzle and a nearby collector electrode. The test gas is injected into a very hot (2500°C to 3500°C) hydrogen flame, where it is thermally decomposed and ionized. An applied voltage of a few hundred volts between the burner nozzle and collector electrode generates a measureable current.

Due to the measurement principle, FIDs are non-selective detectors and only measure the sum concentration of ionizable compounds. One of their major advantages is their applicability to a wide range of gases and gas concentrations. These include, first and foremost, the flammable gases and VOCs. However, FIDs are rather insensitive to inert gases and liquids such as H₂O, CO₂, N₂, or Ar.

Photoionization Detectors (PID)

In photoionization detectors, the hydrogen flame of FIDs is been replaced by ultraviolet (UV) light. This UV light could come from a passive (sun light) or active (e.g., LED) source. PIDs are comparable to FIDs in applicability and resolution and are often used as analyzers in gas chromatographs. These instruments can work over a wide range of gas concentrations and have excellent sensitivity for VOC, which is why they are sometimes used for handheld devices.

2.3.3 Chemi-Resistive Methods

Catalytic Combustion Sensors (Pellistors)

Pellistors are catalytic sensors in which the exothermal reaction of flammable gases with oxygen is used to cause a temperature and resistance change in a platinum wire. The platinum wire acts as a heater and thermometer simultaneously and is covered in a catalytic substance. The catalyst enables the exothermal oxidation of flammable compounds, which heat the catalyst and platinum wire. This temperature change is measured as a resistance increase of the platinum wire and is proportional to the gas concentration.

Due to their chemical and physical robustness and capability to detect flammable gases, pellistors are often used in explosion-prone and safety-critical environments. A disadvantage of these sensors is their large power consumption and sensitivity to lead, sulfur, and halogen hydrocarbon poisoning. Furthermore, the lower limit of detection is relatively high (e.g., 100's to 1000's ppm).

Metal Oxide Sensors (MO_x)

This well-known method for VOC detection has been used for years in a wide range of applications. Traditional metal-oxide sensors use an MO_x material deposited onto a ceramic substrate, which leads to good performance and long life, while more modern MO_x sensors take advantage of silicon micromachining to create a robust but thermally isolated micro-hotplate (MHP), resulting in extremely low power consumption, miniature size and low prices due to their small dimension and high volume production. Typical MO_x materials are doped semiconducting materials such as SnO₂, WO₃, and TiO₂, etc. At elevated temperatures, gas molecules can adsorb onto the MO_x surface and generate free charge carriers. The consequential resistance change can be measured at two electrodes. Hence, the sensor raw signal strongly depends on the ambient atmosphere, utilized materials, production technology, time, and temperature. All organic compounds will catalytically react on the MO_x surface and cause a resistance decrease or increase, depending on the gas oxidizing or reducing behavior.

3. TVOC Detection with IDT's MO_x Gas Sensors

Metal oxide (MO_x) based gas sensors have been developed since the 1960's and have already been on the market for nearly 50 years. Key to this technology is the MO_x chemistry as well as a well-designed MEMS structure, reliable technology, and firing process. This section provides a description of IDT's superior MO_x gas sensor technology using the ZMOD4410 Gas Sensor Module as an example.

Detailed information is given for particular critical sensor characteristics, including the following:

- Sensitivity (also referred as accuracy for a specific target gas)
- Selectivity and cross-sensitivities (sensitivity to other gases)
- Stability (long-term behavior)
- Poisoning (resistance against poisoning gases)

Gas sensors can be operated in different modes; e.g., continuous temperature operation or low-power duty cycling. Due to optimization in each mode, the sensor characteristics are not influenced by any of the ZMOD4410 operation modes offered by IDT.

IDT is in a strong position to fulfill all requirements for indoor air quality (IAQ) monitoring and TVOC detection according to the UBA study². Details can be found in the *ZMOD4410 Datasheet* and the *ZMOD4410 White Paper – Overview of TVOC and Indoor Air Quality*. The governmental sensor research Kurt Schwabe Institute (KSI) did extensive independent investigations for IAQ and IDT's ZMOD4410 gas sensor and reported excellent conformance to the UBA study and to EPA standards. In addition to the VOCs mentioned in the UBA study, IDT's gas sensors have also excellent sensitivity to other VOCs and even non-VOC components (e.g., ammonia), which are part of IAQ.

The ISO 16000-29 standard describes test methods for different VOC detectors. However, a calibration according to this standard is not useful for indoor air quality measurements with metal-oxide sensors because it does not reflect the use case or the expected VOC gases and concentrations of interest. Hence, IDT tested a large number of typical indoor VOC gases in a wide concentration range.

3.1 Sensitivity

The ZMOD4410 is designed to be an excellent sensor for all VOC compounds. There are different possibilities for describing the sensitivity to gas sensors and for performing a raw sensor signal calculation:

- Ratio of resistances
- Calculation of slopes

3.1.1 Ratio of Resistances

A very common definition of the sensitivity is the ratio R_{Air}/R_{Gas} , where the sensor's resistance in clean air (R_{Air}) is divided by the sensor's resistance at a specific gas concentration (R_{Gas}). During these tests, other parameters such as the environmental conditions do not have to change. Figure 2 and Figure 3 show an identical set of data: The raw sensor resistance and sensitivity (resistance change ratio) for the reaction over a large concentration range for the ZMOD44100 to ethanol as a VOC representative. This sensitivity description is commonly used to present the sensor signal for a specific concentration range.

² Umweltbundesamt, *Beurteilung von Innenraumluftkontaminationen mittels Referenz- und Richtwerten*, (Bundesgesundheitsblatt - Gesundheitsforschung - Gesundheitsschutz, 2007).

Figure 2. ZMOD4410 Resistance Response to Ethanol

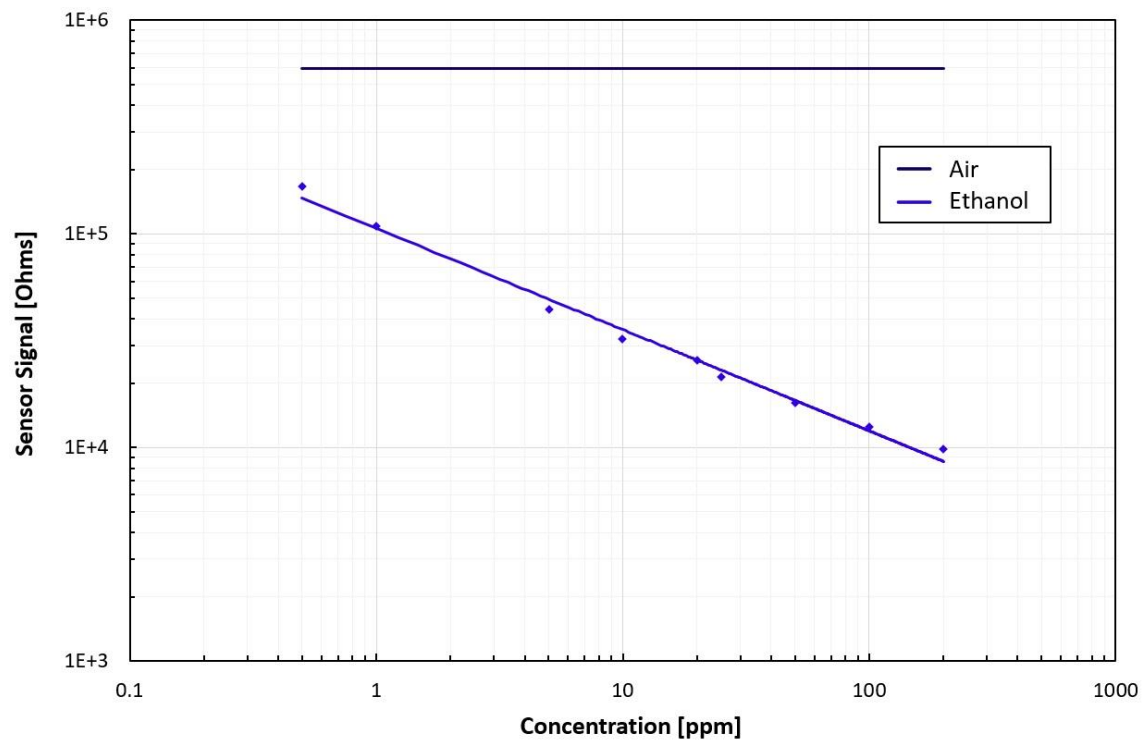
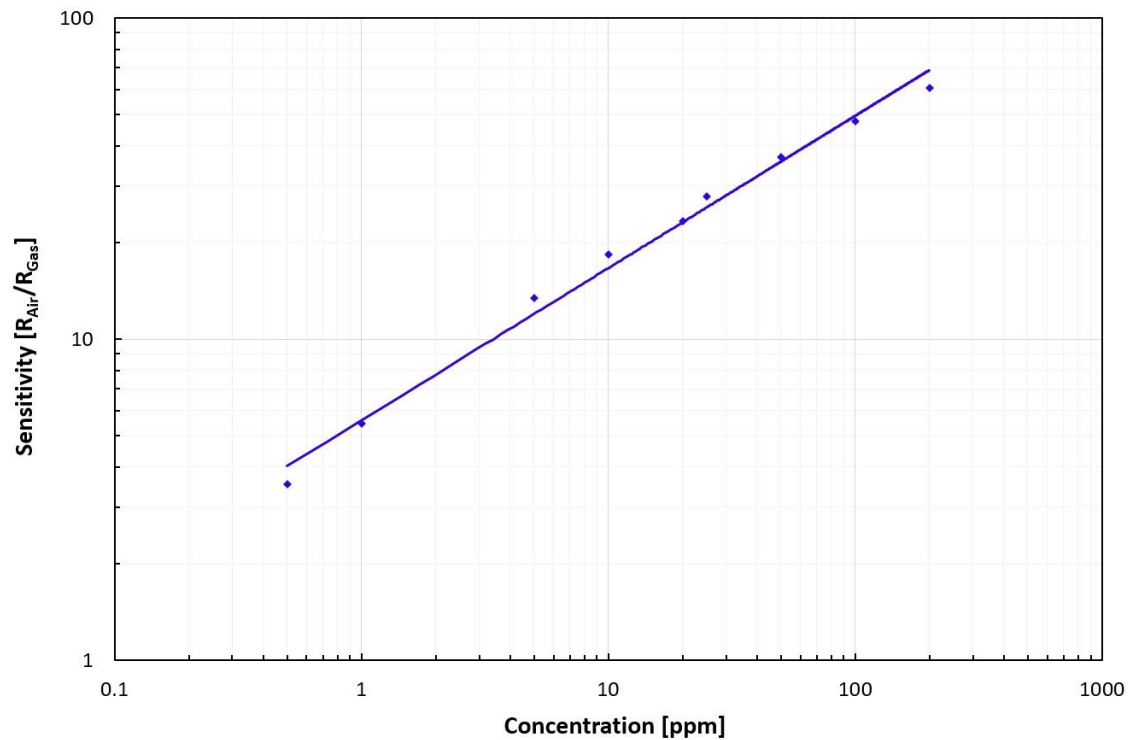


Figure 3. ZMOD4410 Sensitivity to Ethanol



3.1.2 Calculation of Slopes

The mathematical function that describes the sensor reaction is a logarithmic function. It is commonly used for a general descriptions and mathematical modelling of the sensor behavior for precise concentration calculations. The sensor response to a gas is given by (Equation 2):

$$\log(R) = \log(A) - \alpha * \log(c) \quad (\text{Equation 2})$$

Where

R = Resistance

A = Intercept

α = Slope

c = Concentration

In this description, the slope (α) is an indication of the sensitivity. For a constant environment, α is a function of operation temperature and usually shows a maximum due to a chemical equilibrium of adsorption, reaction, and desorption.

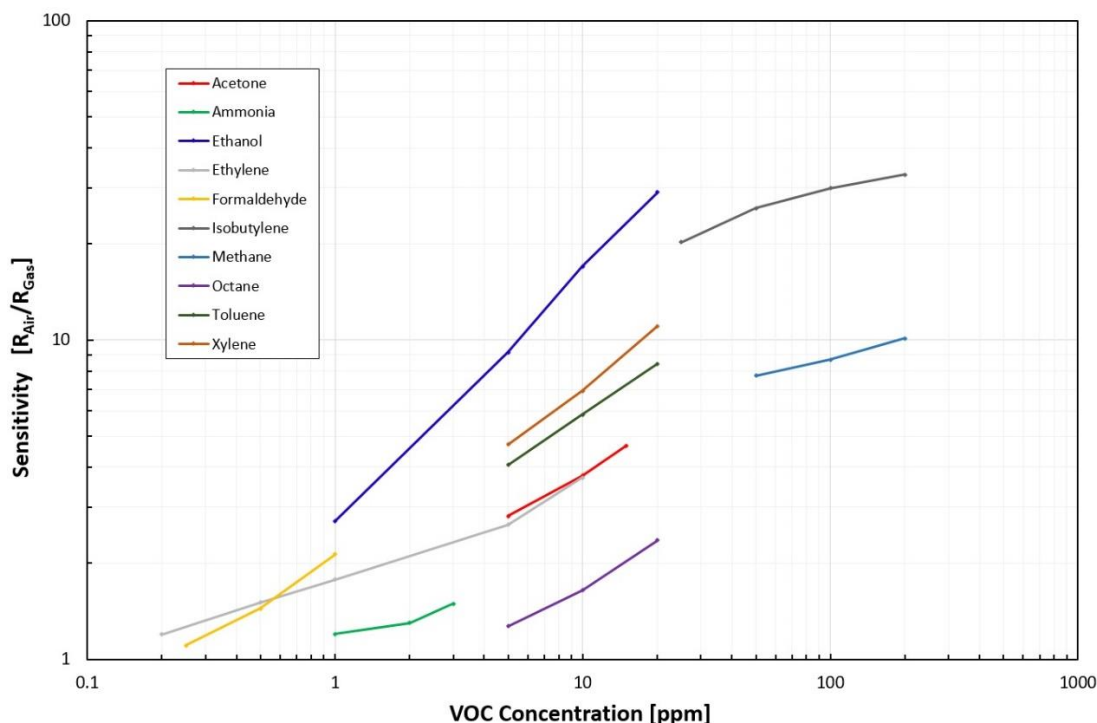
3.2 Selectivity

Selectivity is a response to a target gas species while cross-sensitivities are unwanted interfering reactions. Like most other gas detection methods, MO_x sensors show an interference reaction in the case where several gases are present in the ambient environment.

3.2.1 Selectivity to VOC and Non-VOC

The sensitivity towards typical air quality components as well as other VOC gases and some non-organic gases is shown in Figure 4. These characteristics are valid for all operation modes, such as standard operation and low-power operation. The most common cross-sensitivities in a home environment include sulfur gases, including hydrogen sulfide (H_2S) and other volatile sulfur compounds (VSC), and humidity, although some applications will consider these gases a component of air quality. The ZMOD4410 is also able to detect safety-relevant gases for indoor air, such as carbon monoxide (CO); however, the sensor is not designed to detect these interferants reliably and therefore should not be, and is not approved for use in any safety-critical or life-protecting applications. IDT disclaims all liability for such use.

Figure 4. Sensitivity to Different VOC Gases

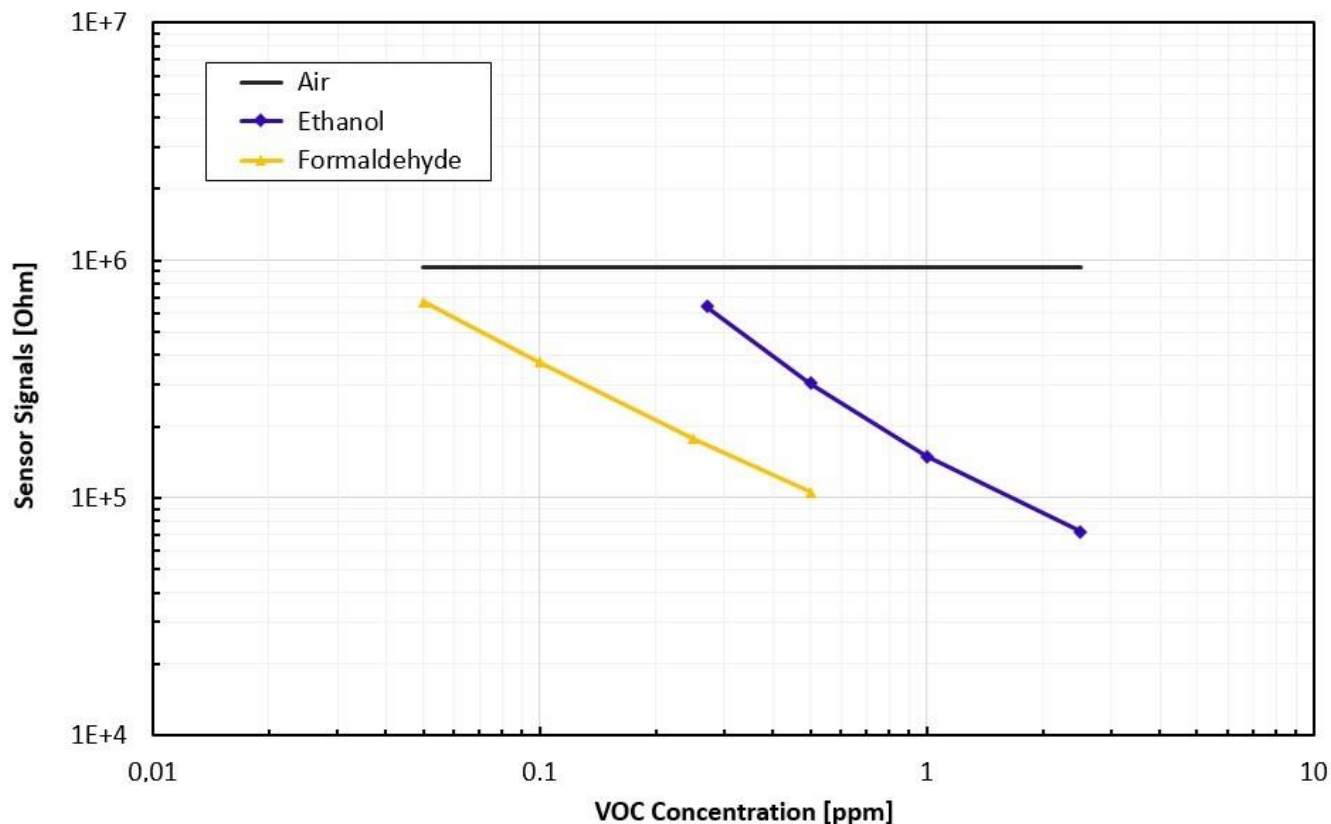


3.2.2 Sensitivity towards Formaldehyde

Formaldehyde (CH_2O) is also referred to as methanol and the simplest of the aldehyde molecule class. One of its main applications is use in the production of wooden products and coatings. Formaldehyde has a boiling point of -19°C and that is the reason why it usually occurs as vapor and can cause outgassing in home furniture. Due to its toxicity in the ppb range, it is one of the VOCs getting the highest concern from customers.

Although the ZMOD4410 is designed to detect all the different classes of VOC, a focus is often pointed towards formaldehyde. The ZMOD4410 can detect concentrations down to 50 ppb (see Figure 5).

Figure 5. Sensitivity to Formaldehyde

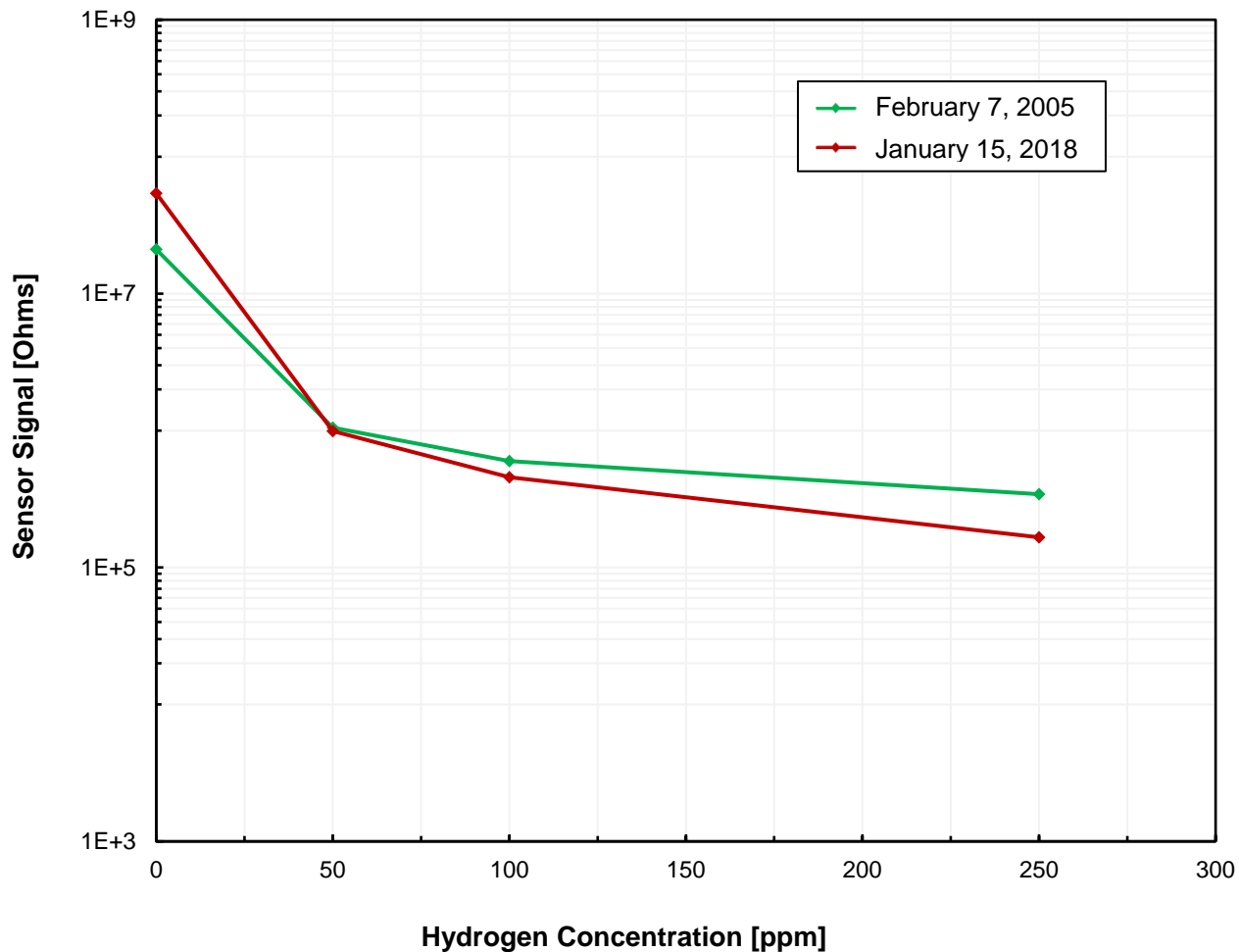


3.3 Stability

Nearly all sensor and measurement instruments suffer from drift; i.e., shifting readings and sensitivity over time. Thus, during development, a priority must always be to minimize drift by optimizing the sensor technology itself. This includes design of materials and fabrication methods, adequate sintering technology, and optimal operation conditions. The goal must be to obtain a long lifetime while achieving high accuracy and minimizing or avoiding the need for recalibration.

IDT has a history of developing metal-oxide materials for nearly 20 years. Although, the sensor and deposition method used for TVOC sensing is new, it has been developed based on the experience of older MO_x sensors, which are still on the market and in operation for long-term testing. Figure 6 shows example resistance data for the developed MO_x material, which has been in operation for more than 14+ years for hydrogen (H_2) detection and is still in operation. The sensor responses from 2005 and from 2018 are very similar and show excellent sensitivity even after 13 years of operation.

Figure 6. Example for IDT Long-Term Stable MO_x Materials



3.4 Poison Resistance

Some gases have the ability to damage the sensor permanently. They react with the sensor surface and passivate the catalytic active centers of the metal oxides. Depending on adsorption behavior and chemical bonding, some gases desorb again at elevated temperatures. A treatment at temperatures above the operation temperature may be useful to “clean” the MO_x surface. However, thermal stress resulting from excessive cleaning may lead to long-term reduction of the sensor performance.

A common ambient gas, which passivates the MO_x surface permanently and leads to damage of the sensor, is siloxanes. These chemicals occur in several household products; e.g., silicon oil, antifoaming agent, rubber materials, food preservation, personal care products, and many more. Siloxanes are VOCs that include a silicon structure; for example, octamethylcyclotetrasiloxane (D₄) has the chemical formula C₈H₂₄O₄Si₄. At elevated temperatures, especially above 300°C, chemical cracking takes place at the catalytic surface and silica (SiO₂) is formed³. Silica is a well-known sensor poison because of its glazing. Because of the wide usage of siloxanes in home environments, it is essential to prove that these VOC gases do not lead to sensor signal drift or loss in sensitivity for indoor air quality applications.

IDT's ZMOD4410 Gas Sensor Module has been exposed to different siloxanes to prove poison resistance. High concentrations of siloxanes have been applied to the metal oxide successively for several weeks to simulate the sensor module's lifetime.

Most siloxanes are noncritical and non-toxic to the environment⁴ and concentrations above 0.0014ppm⁵ do not represent use cases for an average indoor air contamination. See Table 2 for typical expected siloxane concentrations for indoor air⁵. Hence, applying higher concentrations yields an accelerated lifetime test and will give information on long-term behavior for MO_x sensor operation.

Table 2. Typical Expected Siloxane Concentration for Indoor Air

Chemical	Guidance Value Indoor Air [µg/m ³]	Guidance Value Indoor Air [ppb]
Octamethylcyclotetrasiloxane (D ₄)	7	0.57
Decamethylcyclopentasiloxane (D ₅)	22	1.4

IDT has performed several tests with multiple gas sensor modules to characterize their siloxane resistance in detail. A summary is shown in Table 3. During siloxane exposure, tests with different ethanol concentrations have been repeated to monitor the sensitivity. Figure 7 gives an example of the sensor signal change accuracy for exposures with 10 ppm and 20 ppm ethanol for Low-Power Operation Mode. It shows the average of 15 sensor modules working in parallel, including their standard deviation for a 25-day period of exposure to siloxanes.

Table 3. Tests Performed with Siloxanes

Chemical	Method of Operation	Concentration [ppm]	Test Duration [days]	Total concentration [ppm·h]	Signal Drift during Exposure [%]
Octamethylcyclotetrasiloxane (D ₄)	Constant Temperature (Standard)	55	12 days	15800	4.3
Decamethylcyclopentasiloxane (D ₅)		25	25 days	15500	3.9
Octamethylcyclotetrasiloxane (D ₄)	Duty Cycle (Low Power)	25	25 days	15200	4.4
Decamethylcyclopentasiloxane (D ₅)		25	25 days	15500	5.3

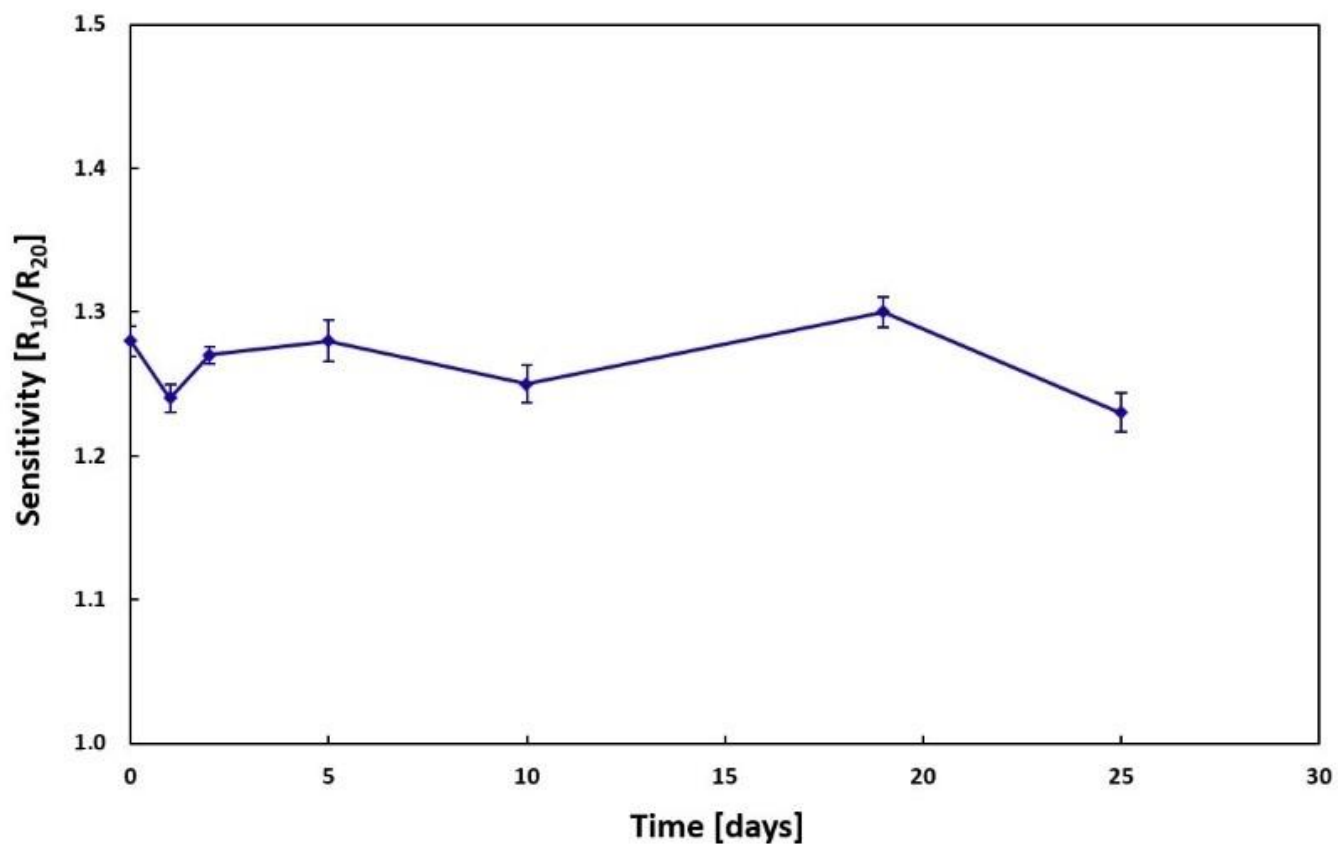
From all testing discussed above, it can be concluded that siloxanes do not harm the ZMOD4410 Gas Sensor Module in any operation mode over its lifetime even at concentrations much higher than expected in an indoor air environment.

³ T. Mundry, *Einbrennsilikonisierung bei pharmazeutischen Glaspackmitteln - Analytische Studien eines Produktionsprozesses*, Dissertation (Berlin: Humboldt University Berlin, 1999).

⁴ ECCC, *Report of the Board of Review for Decamethylcyclopentasiloxane (Siloxane D5) established under Section 333(1) of the Canadian Environmental Protection Act of 1999*, (Ottawa, Canada: 2011).

⁵ Association of Ecological Research Institutes e.V. (AGOEF), *AGÖF Guidance Values for Volatile Organic Compounds in Indoor Air*, (Springe-Eldagsen, Germany: 2013).

Figure 7. Sensitivity Change for the IDT ZMOD4410 Gas Sensor Module during Siloxane Exposure



4. Revision History

Revision Date	Description of Change
March 4, 2019	Update with information on Low Power Operation Mode; addition of sections for formaldehyde and introduction.
June 1, 2018	Initial release.

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