



Comparative evaluation of ammonia sensor principles for SCR applications: From laboratory characterization to real exhaust operation

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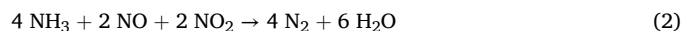
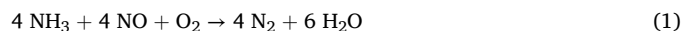
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ABSTRACT

Precise detection of ammonia (NH₃) in the exhaust gas of SCR systems is crucial for complying with emission limits and for optimizing exhaust gas aftertreatment systems. In this work, two different high temperature sensor principles for detecting NH₃ intended for use as slip sensors in SCR systems were investigated. One sensor concept is based on the selective adsorption properties of a zeolite towards NH₃, while the other sensor type is based on mixed potential principle. First, a comprehensive pre-characterization of the sensors was carried out in a laboratory test setup to determine their basic sensitivity to NH₃ and various cross-sensitivities to typical exhaust gas components such as NO_x, H₂O, CO, and CO₂. The sensors were subsequently tested under realistic operating conditions at an engine test bench as NH₃ slip sensors. The results of the lab tests could be successfully applied to the measurements during engine operation, thereby enabling continuous monitoring of NH₃ slip. Post-characterization measurements demonstrated that the ammonia sensing properties of both sensor variants remained stable over the measurement duration. Overall, both sensor principles showed promising results with respect to ammonia sensitivity, response time, and aging behavior.

1. Introduction

Selective Catalytic Reduction (SCR) is the current primary technology for reducing Nitrogen Oxide (NO_x = nitrogen monoxide (NO) and nitrogen dioxide (NO₂)) emissions in lean-burn engines used in light-duty and heavy-duty vehicles [1,2]. In this method, an aqueous urea solution (AdBlue®, DEF or AUS 32) is injected into the hot exhaust gas, where it decomposes to NH₃. This reduces NO_x with a SCR catalyst to nitrogen (N₂) and water (H₂O) according to the following reactions [2]:



These reactions differ in their kinetics, with the SCR reaction involving NO (Eq. 1) proceeding more slowly than the one involving NO and NO₂ (Eq. 2). Modern SCR systems typically include a diesel oxidation catalyst (DOC) to reduce hydrocarbon (HC) and carbon monoxide (CO) emissions while simultaneously shifting the NO/NO₂ ratio in order to favor the faster SCR reaction (Eq. 2), as the raw exhaust typically

exhibits a NO/NO₂-ratio of around 90/10 [3,4]. Significant development effort is still dedicated to identifying suitable SCR catalyst materials that offer high NO_x conversion over a wide temperature range (200–500 °C) and long-term stability. Zeolite-based and vanadium-tungsten-titania (VWT) catalysts have become particularly dominant [5,6].

Detecting ammonia in exhaust gas is essential for optimizing NO_x reduction and to prevent unnecessary NH₃ injection. Ammonia slip is regulated under legislation such as Euro VII due to air quality concerns, with acceptable limits in the low ppm range [7–9]. Beyond their use in diesel SCR systems, SCR catalysts are also used to reduce NO_x emissions in CO₂-neutral biomass combustion. Moreover, they are expected to play a significant role in future low-carbon engine technologies such as hydrogen- or ammonia-fueled combustion engines [10]. Ammonia is being considered as a carbon-free fuel for internal combustion engines [11]. In hydrogen-powered engines, NH₃ may be present in the exhaust gas either as a fuel impurity or as an additional reducing agent for NO_x reduction [12,13]. Consequently, robust ammonia sensors will be essential not only for optimizing current SCR systems but also for

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enabling future zero-CO₂ combustion concepts.

Up to now, SCR systems are typically controlled using NO_x sensors located upstream and downstream of the catalyst, in combination with model-based algorithms that consider ambient and engine parameters [14,15]. The required amount of AdBlue® is calculated in a closed-loop control system that also integrates NH₃ storage behavior of the SCR catalyst material [16]. However, as the NO_x sensors exhibit cross-sensitivity to NH₃, they only enable indirect estimation of ammonia slip [17–19]. This limitation has recently accelerated the development of model-based approaches to enhance NH₃ slip monitoring, including advanced estimation strategies for SCR systems [20]. This can still be problematic when model-based operation is not possible or when errors occur.

For these reasons, it is expected that selective ammonia sensors will become more important. Recent review articles underline that ammonia sensing remains an active research field, focusing on improved selectivity, lower detection limits, and enhanced long-term stability under realistic operating conditions. In this context, high-temperature solid-state sensors are considered especially promising for exhaust-related applications because they combine thermal robustness with direct NH₃ detection [21]. The literature describes various sensor technologies capable of operating under harsh exhaust gas conditions, such as metal-oxide semiconductor (MOS) sensors. These types of sensors detect NH₃ based on the change in electrical resistance caused by adsorption [22,23]. Typical materials include zinc oxide (ZnO), tin dioxide (SnO₂) or tungsten trioxide (WO₃), whose selectivity and stability can be enhanced by doping with metals such as Cu or Pt [24]. These sensors are typically operated between 200 °C and 400 °C and show high sensitivity to NH₃ in the ppm range, but they suffer from cross-sensitivities to water vapor and hydrocarbons that can be reduced by using special coatings or catalytic filters [25–27]. In addition, the long-term stability in real exhausts is not sufficient [28].

Another approach involves optical systems based on absorption or infrared spectroscopy that make use of characteristic NH₃ absorption bands. While these systems offer high selectivity and allow direct, continuous, non-contact measurement, they are expensive and technically complex. Despite their excellent analytical performance, optical and spectroscopic approaches are still mainly restricted to laboratory or stationary applications, whereas compact solid-state sensors remain more attractive for mobile exhaust systems [29–31].

Further approaches, such as photoacoustic spectroscopy and fiber-optic sensors, offer high precision and fast response times but are not yet suitable for automotive applications due to issues regarding robustness and costs [29,32].

Commercial ammonia sensors are already used in some diesel exhaust aftertreatment systems. They are typically electrochemical multilayer sensors based on yttrium-stabilized zirconia (YSZ). They exhibit cross-sensitivities to H₂O and O₂, which partly offset each other, while their responses to NO_x, CO, and HC are negligible. An integrated heating structure and temperature monitoring provide high robustness to variations in exhaust flow and pressure [33]. A related electrochemical planar sensor concept is also examined in this work [34]. In parallel, recent studies on YSZ-based mixed-potential ammonia sensors have demonstrated considerable progress in terms of sensitivity, selectivity and stability when the sensing electrode composition and sensor architecture are adapted for SCR-relevant conditions. For example, Ag-modified FeVO₄ electrodes and double-sensing-electrode designs have been reported to improve NH₃ response while reducing interference from typical exhaust gas components [35,36].

In this study, two sensor concepts, both built on a common planar ceramic substrate but using fundamentally different sensing mechanisms, are investigated and compared. The first type is a capacitive zeolite-based sensor that uses its selective NH₃ adsorption properties. The other one is a mixed-potential sensor based on YSZ with an SCR-active functional film consisting of a vanadia/titania catalyst material (VT). Both concepts are thermally robust and use materials proven in

exhaust environments. However, a fundamental difference exists between these two sensor concepts regarding their principles and the mechanisms of signal generation. The aim of this study is to compare these two approaches in terms of their suitability for NH₃ detection in the SCR system of an internal combustion engine.

2. Experimental

The following section describes the structure and operating principles of the two sensor types, focusing on sensor properties relevant for gas measurements under laboratory and engine test bench conditions.

2.1. Sensor designs and functionality

Both sensor variants are based on the same platform, consisting of an alumina substrate (CeramTec™ Rubalit 708S) with a heater structure applied to the reverse side. The structure is identical for both sensor concepts and is shown in Fig. 1a. It is made of platinum (Heraeus™ LPA88–11S) and manufactured by screen printing onto the sensor substrate. A glass-ceramic cover layer (DuPont™ QM42) is applied on top to protect the heater and prevent unintended reactions between the reactive platinum and exhaust gas components.

The heater itself is realized in a four-wire configuration, allowing its resistance to be measured specifically within the region beneath the functional area of the sensor. Using a pre-determined temperature–resistance calibration curve, the sensor temperature can be controlled during operation [37]. The heater layout was designed to maintain a homogeneous temperature distribution across the functional films, even under varying exhaust gas flow conditions. Despite the elevated operating temperatures, the power consumption remains moderate due to the localized heating design, with typical values in the range of approximately 3.5–6.5 W, depending on the operating temperature and flow conditions.

Subsequent processing differs, depending on the sensor principle used. It is described in the following sections.

2.1.1. Capacitive zeolite-based sensor

The capacitive ammonia sensor contains an interdigital capacitor (IDC) structure on the front side. The electrodes, with finger width and spacing of 20 μm each, are laser-structured from a full-area gold film (DuPont™ 5744 R) using a picosecond laser (LPKF™ Protolaser R4). A functional zeolite film is located on top of this IDC structure. Both, the gold film and the zeolite functional film above, are realized via thick-film screen-printing [38]. Fig. 1b illustrates the sensor front layout.

Using the IDC geometry, the dielectric properties of the zeolite film can be measured. Previous publications by Wöhrl et al. provide a more detailed description of the fabrication process and the influence of zeolite composition and sensor operation [38,39]. The fundamental sensing mechanism relies on the reversible, temperature-dependent adsorption of NH₃ molecules in the microporous structure of the zeolite [40]. In particular, NH₃ is selectively adsorbed at the acidic sites of the zeolite framework, where its basic character leads to strong interactions with the surface [41]. The adsorption of NH₃ molecules within the zeolite pores leads to an increase in the effective permittivity of the functional film [42]. This results in a capacitance change ΔC that can be detected with the help of the IDC structure [43]. At an operating temperature of T_{H, cap} = 400 °C, obtained with the heater structure on the reverse side of the sensor, a balance between adsorption and desorption is established, leading to a concentration-dependent signal [44]. With increasing NH₃ concentration, the loading of the zeolite increases accordingly. However, due to the presence of different adsorption sites within the zeolite framework, a linear response behavior is not expected [41]. The zeolite used here is a proton-exchanged ZSM-5 with a SiO₂/Al₂O₃ ratio of 27 (ALSI-Penta Zeolithe GmbH), offering a high density of acidic sites and therefore high sorption capacity for ammonia

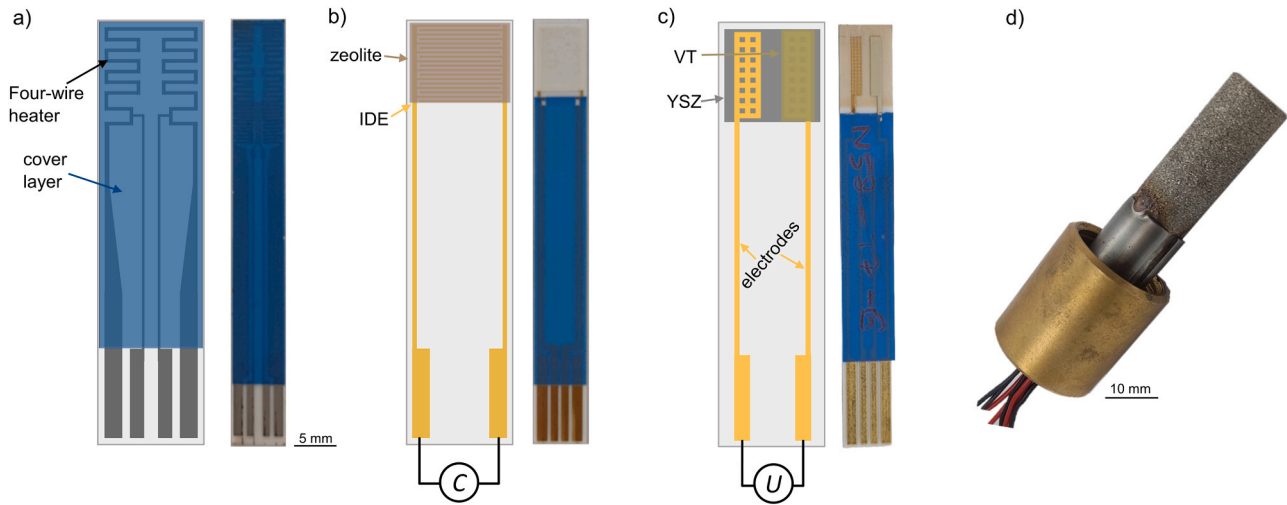


Fig. 1. Basic structure of two sensor concepts for ammonia detection. a) Rear heater structure in four-wire technology and insulation film. b) Capacitive sensor with interdigital capacitor and a functional zeolite film on top. c) Mixed-potential sensor with YSZ as electrolyte and VT as functional films. d) Sensor housing for gas measurements in the laboratory and in the engine exhaust on the test bench.

[45]. The proton-exchanged form also minimizes catalytic activity, which helps to reduce possible cross-sensitivities [46,47].

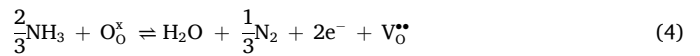
Compared to previous works, this study avoided the use of expensive temperature controllers and impedance analyzers. Temperature control was implemented using a four-wire heater regulation based on an Arduino™, which adjusts heater power in response to changes of the heater resistance due to varying exhaust flow conditions. Capacitance was measured using a Sigma-Delta capacitance-to-digital converter (CDC), commonly used in MEMS devices. The Analog Devices™ EVAL-CN0552-PMDZ module operates at an excitation frequency of 32 kHz (square wave, 1.2 V) [48,49]. A second Arduino™ controlled the capacitance readout independently from the heater control.

2.1.2. Mixed-potential sensor

The mixed-potential sensor contains a front-side film of yttria-stabilized zirconia (YSZ, Tosoh™ TZ-8YS), which is applied using the powder aerosol deposition (PAD) process [50]. PAD is a vacuum-based coating technique in which ceramic powders are dispersed in a carrier gas (here oxygen), accelerated through a nozzle and deposited at room temperature. As the particles hit the surface, they crack and form a dense, nanocrystalline film [51]. The YSZ film serves as the solid electrolyte and, due to the high density enabled by PAD, offers improved ionic conductivity compared to screen-printed films [50].

Two porous gold electrodes are applied on top of the YSZ, one of which is coated with a catalytically active VT film (vanadia/titania). The high selectivity of the VT-covered electrode towards NH₃ is closely related to the catalytic properties of vanadia–titania materials known from SCR applications [52]. Under the chosen operating conditions, NH₃-related surface reactions dominate at the coated electrode. As a result, the mixed potential is mainly influenced by ammonia-dependent processes, leading to the pronounced NH₃ selectivity of the sensor [53, 54]. These films are produced by screen printing. The porous morphology of the electrodes enhances the triple-phase boundary (TPB), which is essential for the mixed-potential formation [55,56]. Fig. 1c shows the sensor structure. A more detailed description of the fabrication process is given by Donker et al [50].

The sensing mechanism is based on the mixed-potential principle, in which competing electrochemical reactions occur at the electrodes. In this context, the sensor signal results from the imbalance of the anodic and cathodic reaction rates at the two electrodes [57]. This leads to the formation of a mixed potential under open-circuit conditions. For ammonia detection, the dominant reactions are [58]:



Here, V_O^{••} denotes a double positively charged oxygen vacancy, O_O^x an oxide ion on a regular lattice site according to the Kröger-Vink defect notation [59], and e[−] a free electron. Due to the catalytic VT film, the reaction equilibrium at the coated electrode shifts selectively in the presence of NH₃, leading to the formation of a mixed potential relative to the uncoated reference electrode [60]. The resulting potential difference depends on the relative contribution of these reactions. It is influenced by the NH₃ concentration at the sensing electrode. As NH₃ promotes reaction (4), the mixed potential shifts accordingly, leading to a sensor signal. The signal exhibits a typical semilogarithmic dependence on NH₃ concentration [53].

Voltage measurements were performed in open-circuit mode using a PalmSens 4 (PalmSens BV™) electrochemical analyzer. The back-sided heater was operated at a temperature of T_{H,mix} = 550 °C using a Keithley™ 2400 Sourcemeter. The sourcemeter is controlled by a custom LabVIEW™-program, to control the sensor temperature.

Both sensor variants, the capacitive and the mixed-potential sensor, also contain an additional cover layer over the feedlines on the sensor side to prevent possible short circuits caused by soot deposits or other reactions with the exhaust gas environment. This can be seen in the depiction of the real sensors in Fig. 1b) and c) by the bluish layer.

The sensor concepts examined in this work were chosen due to their implementation on a common planar platform featuring identical substrate architecture and an integrated heater structure. This provides comparable thermal and geometric conditions. Furthermore, both sensing approaches use materials that are established in exhaust gas aftertreatment and are known for their thermal and chemical robustness under SCR-relevant conditions. Their respective sensor signals are driven by either sorption-related equilibrium in the zeolite film or electrochemical reactions at the YSZ-based solid electrolyte. This makes them particularly suitable for a comparative investigation of two fundamentally different NH₃ sensing mechanisms.

2.2. Measurement setup

The sensors were mounted in a modular sensor housing suitable for laboratory and engine test bench measurements. It consists of a

clamping mechanism for the sensors to enable electrical contact, a threaded fitting for installation in the measuring chamber, and a sintered metal protective cap [61]. The sintered metal has a pore size of 50 μm . The use of this protective cap is based on experience from previous work, as there is no entry of larger particles with sufficiently high permeability of ammonia [62]. The complete setup is shown in Fig. 1d.

2.2.1. Laboratory test bench

For pre- and post-characterization of the sensors, a laboratory gas mixing test bench was used that allowed simultaneous evaluation of NH_3 sensitivity and cross-sensitivities. The setup is shown schematically in Fig. 2.

The gas supply consists of two mass-flow-controller (MFC) lines. The upper line is used to supply the various basic gas components N_2 , O_2 , CO_2 , and gaseous H_2O . The gas composition can be varied within the concentration ranges typical for real engine exhaust gas. The lower line contains additional MFCs, that can be used to dose other special gases that are of interest in low concentrations for synthetic exhaust gas. In this case, this included the target gas NH_3 , which was dosed from a gas cylinder containing 10 vol% NH_3 (rest N_2). Other gases, such as NO (2 vol% in N_2), NO_2 (3 vol% in N_2), and CO (100 vol%), were available for investigating possible cross-sensitivities (all gas cylinders were provided by Rießner-Gase GmbH).

The final gas mixture (6 l/min) passes first through a chamber containing a lambda probe (Bosch LSU 4.2) to verify the O_2 concentration. The sensor prototypes were installed in a subsequent measuring chamber. All pipes and chambers were heated above 50 $^\circ\text{C}$ to prevent condensation and to minimize adsorption effects, particularly of ammonia. The final component was an FTIR spectrometer (MKS Multi-Gas 2030 FTIR Analyzer, mksinstTM) for continuous analysis of the dosed gas components (except N_2 and O_2). All the measured values served as a reference for the sensors to determine the characteristic curves.

2.2.2. Engine test bench

Real exhaust gas measurements were performed on a test bench with a Mercedes-BenzTM OM651 diesel engine (4-cylinder, 2.1 L displacement, compression ratio 16.2:1, 150 kW). Different load conditions can be achieved by the connection to an eddy current brake. During the measurement series, the engine was primarily operated at a constant speed of 1850 rpm with a load of 45%, corresponding to a torque of approximately 246 Nm. The engine includes a turbocharger and was operated without EGR during the tests to maintain sufficiently high exhaust temperatures for urea injection and SCR reactions.

A NO_x sensor upstream of the DOC measured the raw NO_x emissions. The DOC serves both to oxidize unburned carbon compounds (HC, CO) and to oxidize a part of the raw NO emissions to NO_2 . This leads to a shift

in the NO/NO_2 ratio to NO_2 , thereby enhancing the fast SCR reaction from Eq. (2) and consequently increasing the NO_x conversion.

For the AdBlue[®] dosing, a thermocouple located in the exhaust gas ensures that a sufficiently high temperature of approx. 200 $^\circ\text{C}$ is present for the conversion of the injected urea solution to ammonia. Dosing was carried out using a single-fluid injector for light vehicles, whose injection quantity was controlled via a pulse width modulated signal and its pulse duration. The control and adjustment of the AdBlue[®] quantity to be injected was carried out manually using a pulse generator.

A cordierite mixing unit also ensured improved conversion of AdBlue[®] to ammonia and homogeneous mixing with the exhaust gas. A copper-exchanged zeolite was used as the SCR catalyst. Additional temperature monitoring took place up- and downstream the catalyst. After the entire exhaust gas treatment process, the sensors and analytics used in the laboratory were installed. The only difference here is the additional pump with an integrated filter element between the exhaust pipe and the FTIR spectrometer, which provided a constant flow of approx. 12 l/min for the analytics during the measurement runs. A schematic drawing of the entire setup can be seen in Fig. 3.

The ammonia sensors therefore served as slip sensors downstream the SCR catalyst to detect an overdose of ammonia. The installation position in the rear section of the aftertreatment system also has the advantage that possible cross-sensitivities due to unburned hydrocarbons or NO_x emissions have less influence on the sensor signal due to the low concentrations that occur at this location.

3. Results and discussion

The first subsequent measurements were used to determine the basic characteristics of the sensors. Measurements of NH_3 sensitivity and several cross-sensitivity tests were carried out in the laboratory setup in order to obtain characteristic curves for the real exhaust gas measurements on the engine test bench. At the end, after the series of measurements on the engine test bench, the sensors were measured again in the laboratory test bench to track any possible aging of the sensors.

3.1. Laboratory measurement

The laboratory measurements form the basis for the investigations conducted at the engine test bench. Characteristic curves for each sensor were recorded under different exhaust-like gas compositions so that these could later be used as a reference for comparison with the post-characterization measurements.

3.1.1. Basic sensor characteristics

The initial characterization of the sensors was performed using a

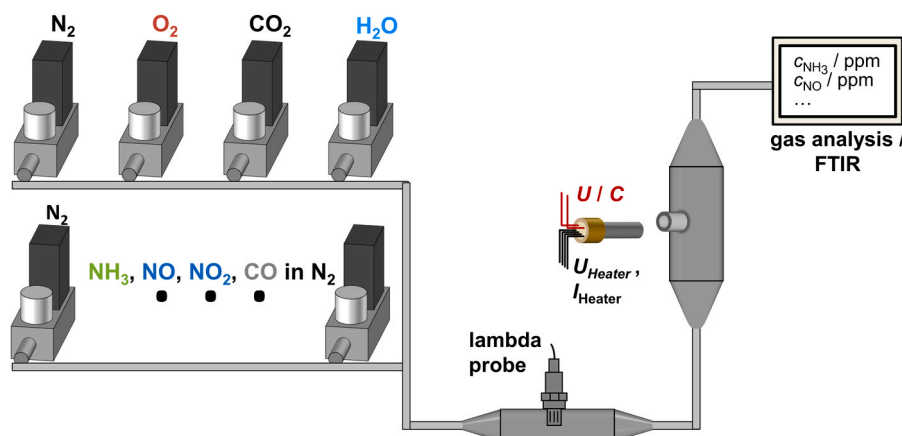


Fig. 2. Schematic layout of the test bench, consisting of MFCs, sensor chamber, and gas analysis using FTIR spectrometer and lambda probe, for characterizing the sensors in terms of NH_3 sensitivity and cross-sensitivities.

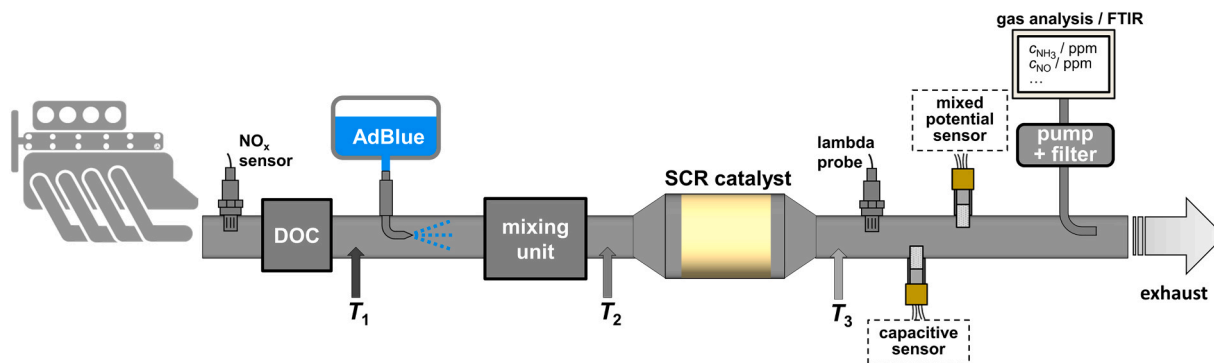


Fig. 3. Schematic layout of the exhaust gas line of the engine test bench with SCR system, including sensor mounting and gas analysis.

fixed base gas composition of 5 vol% O_2 , 5 vol% H_2O , and 3 vol% CO_2 . During this measurement, the ammonia concentration was gradually increased up to 300 ppm. Fig. 4a-c show the time-dependent raw data from both sensors and from the FTIR spectrometer, along with the corresponding NH_3 -dependent sensor characteristics (Fig. 4d and e) derived from these data.

In general, both sensors followed the various steps of ammonia dosing and returned to their initial values once the dosing was stopped. Nevertheless, due to their respective operating principles, the sensors exhibited certain characteristic differences.

The capacitive ammonia sensor displayed a baseline capacitance of approximately 99 pF in the base gas without ammonia. This baseline originates from contributions of the substrate and the zeolite film, defined by the permittivity of the materials used [63]. Additionally, partially unshielded measurement leads to an increase in the

measured total capacitance. NH_3 sorption changes the dielectric properties of the zeolite film, while the baseline capacitance remains constant. For further evaluation, the change in capacitance ΔC with respect to the base value is preferred. This can be achieved through calibration in the installed state.

The NH_3 -dependent characteristic of the capacitive sensor shows the highest sensitivity at low NH_3 concentrations (< 100 ppm), with decreasing sensitivity at higher concentrations and an almost linear trend. This behavior corresponds well with adsorption isotherms known from literature for zeolites [64–66].

The mixed-potential sensor initially exhibited a voltage close to 0 mV. Since identical reactions occur at both electrodes in the base gas, they maintain the same potential. This resulted in a very low baseline voltage. Minor temperature inhomogeneities between electrodes can lead to small differences in activity or thermoelectric effects, which also

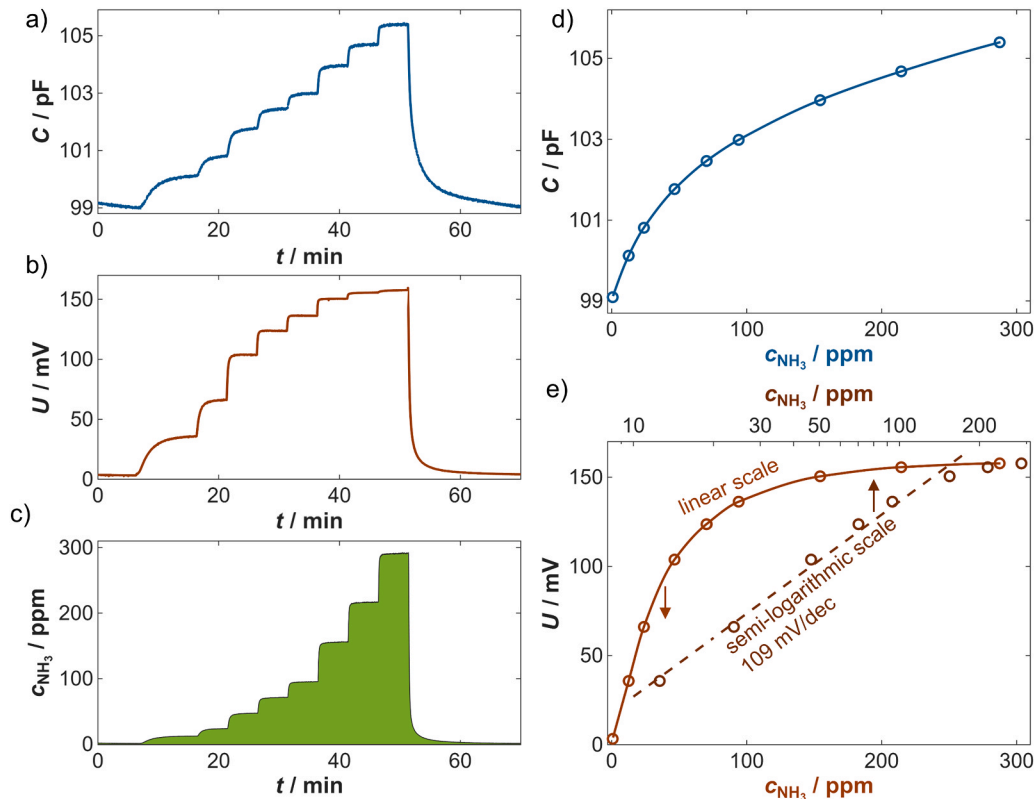


Fig. 4. Characteristic behavior of both sensors. a) Time curve of the capacitive sensor (blue) at an operating temperature of $T_{H,cap} = 400$ °C. b) Time curve of the mixed-potential sensor (red) at an operating temperature of $T_{H,mix} = 550$ °C. c) Time curve of the FTIR spectrometer as a reference with increasing ammonia dosage (0–300 ppm). d) NH_3 -dependent characteristic curve of the capacitive sensor. e) NH_3 -dependent characteristic curve of the mixed-potential sensor on a linear (bottom) and logarithmic scale (top). Base gas composition: 5 vol% O_2 , 5 vol% H_2O , 3 vol% CO_2 in N_2 .

can be minimized by determining the voltage change as response.

The addition of ammonia resulted in a shift in the kinetics of the competing electrochemical reactions at the two electrodes. Due to the asymmetric VT coating at one electrode, a measurable potential difference occurs [54]. At increasing NH_3 concentrations, a saturation-like behavior is visible, which appears nearly linear in a semi-logarithmic representation (see Fig. 4e). The slope was approximately 109 mV/decade NH_3 , consistent with earlier results reported by Donker et al [50]. Compared with recently published solid-state mixed-potential NH_3 sensors, the sensitivity obtained here is within the typical range reported in the literature. Depending on the electrode material and operating temperature, sensitivities between approximately 35 and 90 mV/decade NH_3 have been reported for comparable sensor concepts [35,36,54].

Due to time delays caused by the piping between the sensors and the FTIR spectrometer, information on the response times of the sensors can only be provided to a limited extent. This means that a signal change occurred in some cases before a change in the NH_3 concentration was detected by the FTIR spectrometer, indicating a sensor response time below a few seconds. This is in the same order of magnitude as values reported in the literature, where response times ranging from a few seconds to a few tens of seconds are found, again depending on the sensor principle and the operating temperature [36].

3.1.2. Cross-sensitivities

Further laboratory investigations focused on evaluating cross-sensitivities that might affect the sensors during the real-exhaust measurements. These measurements included variations in the base gas concentrations of O_2 , H_2O , and CO_2 as well as additional gases such as NO , NO_2 , and CO . All cross-sensitivity experiments were performed simultaneously with NH_3 dosing so that possible changes in the NH_3 characteristic curves could be observed. Fig. 5 shows the obtained sensor characteristics under variations of the H_2O and O_2 concentrations.

The capacitive ammonia sensor exhibited a cross-sensitivity to changes in water content (see Fig. 5a). Increasing the H_2O concentration from 1 to 5 and finally 10 vol% resulted in a nearly constant offset towards higher capacitance values. This behavior is attributed to zeolites' well-known adsorption capacity for water, making them a widely used drying agent or humidity sensor film [67–69]. However, no dependency of the capacitance signal on the oxygen concentration was observed.

An opposite trend occurred for the mixed-potential sensor. Increasing the oxygen concentration from 5 to 10 and finally 15 vol% led to a reduced sensor response across the entire NH_3 concentration range of 0–300 ppm (see Fig. 5b). Higher O_2 levels promote the oxygen reaction described in Eq. (3), reducing the contribution of ammonia to the sensor voltage [50,53]. As a result, the sensor sensitivity towards

NH_3 decreased. In contrast, changes in water concentration had an only negligible effect on the sensor signal.

Further investigations involved NO and NO_2 at various concentrations and mixing ratios, with the maximum NO_2/NO_x ratio limited to 10%, consistent with realistic conditions downstream the SCR catalyst where NO dominates. The fast SCR reaction (Eq. 2) leads to rapid conversion of NO_2 . Fig. 6 shows the sensor responses during simultaneous dosing of NH_3 and NO_x .

The capacitive sensor showed a stronger dependency on the NO_x concentration compared to the mixed-potential sensor. This behavior can be attributed to the SCR activity of the zeolite [70]. Although the proton-exchanged zeolite used here exhibits significantly reduced catalytic activity compared to Fe- or Cu-zeolites, it cannot be completely suppressed. As a result, part of the adsorbed ammonia reacts with NO_x and is therefore not detectable via the capacitive signal. Consequently, increasing total NO_x concentration and NO_2 fraction enhanced this effect according to the Eqs. (1) and (2) (see Fig. 6a).

In contrast, the mixed-potential sensor exhibited only minor shifts in its NH_3 characteristic upon addition of NO_x (Fig. 6b). Possible electrochemical reactions with NO_x contribute only weakly to potential formation compared to ammonia. This is likely due to selective NH_3 conversion of the VT film, which is assumed to remain fairly inactive towards NO_x at 550 °C.

Furthermore, NO and NO_2 are known to generate mixed potentials with opposite signs for this sensor type when applied individually. Since NO_2 was present only as a minor fraction of the total NO_x concentration in the present study, its contribution likely partially compensated the NO -induced signal, resulting in only a small overall shift of the NH_3 -dependent characteristic [50].

Additional cross-sensitivities are shown in Fig. 7. No measurable sensitivity to changes in CO_2 content (3–5 vol%) was exhibited by the sensors, indicating that CO_2 does not influence NH_3 adsorption of the zeolite or mixed-potential formation.

However, different behaviors appeared for dosing at 195 ppm CO (Fig. 7c, d). The zeolite-based sensor showed no response to CO , whereas the mixed-potential sensor exhibited an increased voltage across the entire NH_3 concentration range. Similar mixed-potential sensor architectures have been used for CO detection in the literature, as the strongly reducing nature of CO significantly affects the electrochemical equilibrium, producing an additional voltage offset that interferes with the NH_3 signal [60].

For reasons of clarity, Fig. 8 provides an overview of the cross-sensitivities investigated for the two sensor variants described. To this aim, the relative deviation was calculated that results at a constant ammonia concentration of 100 ppm when the concentration of the other gas components changes. Using the example of an increase in oxygen

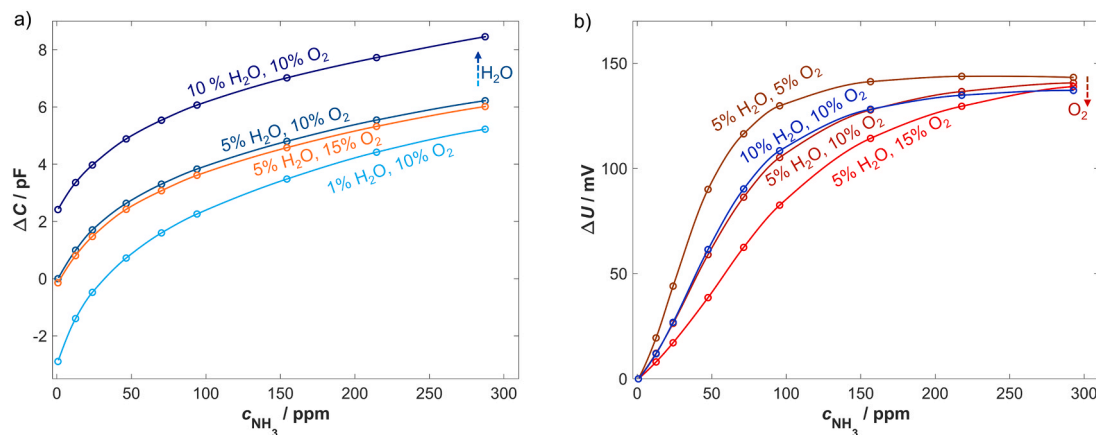


Fig. 5. Characteristic curves of the capacitive sensor at (a) and of the mixed-potential sensor at (b) in cross-sensitivity tests with variation of the base gas concentrations of O_2 and H_2O at a constant CO_2 concentration of 3 vol% in N_2 , sensor temperatures: $T_{\text{H,cap}} = 400$ °C and $T_{\text{H,mix}} = 550$ °C.

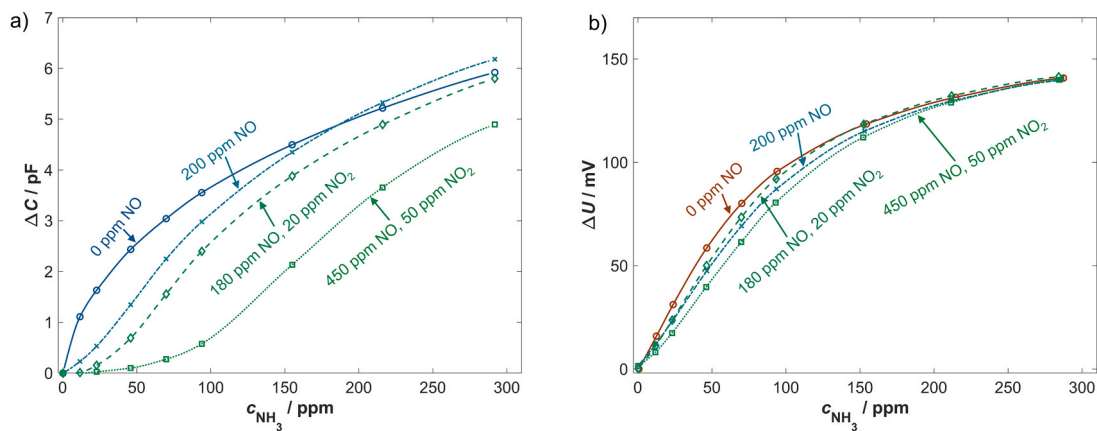


Fig. 6. Characteristic curves of the sensors in case of the capacitive sensor (a) and of the mixed-potential sensor (b) when adding different NO_x concentrations with simultaneous dosing of ammonia. Base gas composition: 10 vol% O_2 , 10 vol% H_2O , 3 vol% CO_2 in N_2 , sensor temperatures: $T_{\text{H,cap}} = 400^\circ\text{C}$ and $T_{\text{H,mix}} = 550^\circ\text{C}$.

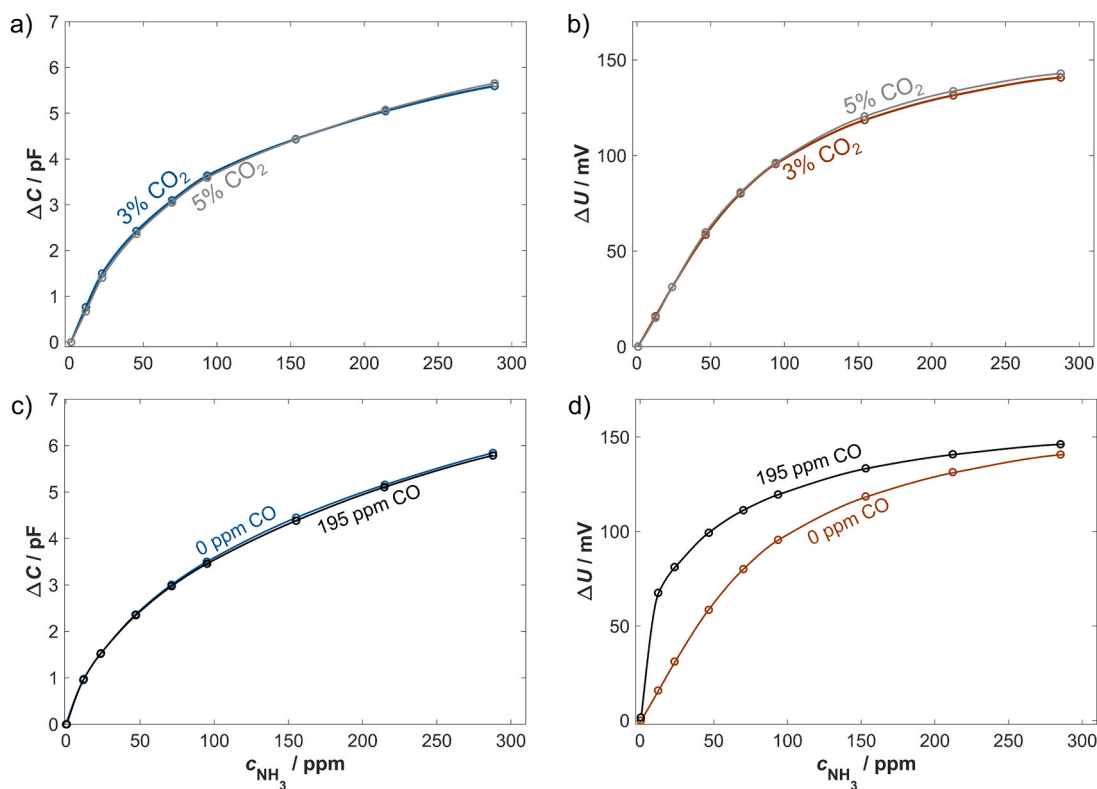


Fig. 7. Characteristic curves of the sensors when the base gas CO_2 is changed in the range from 3 to 5 vol% (a) for the capacitive sensor at $T_{\text{cap}} = 400^\circ\text{C}$ and (b) for the mixed-potential sensor and 195 ppm CO is added (c) for the capacitive sensor and (d) for the mixed-potential sensor. Base gas composition: 10 vol% O_2 , 10 vol% H_2O , 3 vol% CO_2 in N_2 , sensor temperatures: $T_{\text{H,cap}} = 400^\circ\text{C}$ and $T_{\text{H,mix}} = 550^\circ\text{C}$.

content of 2.0 vol% during measurement, a relative deviation of approx. 23% would occur with the mixed-potential sensor if the characteristic curve initially used at 5 vol% O_2 remains in use. While the capacitive ammonia sensor would show almost no deviation here, an increase in water content of 2.0 vol% would result in a relative deviation of approximately 30%.

For NO dosing, the capacitive sensor exhibited the most significant reduction in response, with increasing NO concentrations leading to a higher deviation in NH_3 determination. In contrast, the mixed-potential sensor exhibited notable dependency for CO, resulting in an overestimation of NH_3 concentration.

Although these experiments do not cover the full range of gas components present in real diesel exhaust, they provide a general overview

of potential influences and help to interpret behavior during following engine-bench measurements. A comparison with a commercially available ammonia sensor developed for closed-loop SCR control shows that application-relevant cross-sensitivities are also present in established sensor systems. The electrochemical YSZ-based sensor reported by Wang et al. exhibits a semilogarithmic NH_3 response and shows measurable influences of H_2O and O_2 , although these effects are partly compensated under diesel engine conditions due to their opposite trends with changing air-fuel ratio. In contrast, the effects of NO, CO, and HC were reported to be comparatively small, while NO_2 requires dedicated compensation in the commercial sensor system. Overall, the cross-sensitivity levels observed for the two sensor concepts investigated here are therefore comparable to those reported for application-oriented

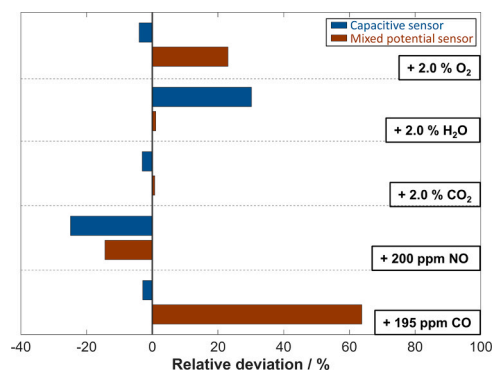


Fig. 8. Summary of cross-sensitivities of both sensors, based on the relative deviation at an NH₃ concentration of 100 ppm and a concentration increase of the base gas components by 2 vol% and dosing of the process gases NO of 200 ppm and CO of 195 ppm in N₂. Sensor temperatures: $T_{H,cap} = 400$ °C and $T_{H,mix} = 550$ °C.

NH₃ sensor concepts already described in the literature [33].

3.2. Real exhaust gas measurements

The measurements described in this section were conducted under nearly stationary engine-operating conditions. This approach was

chosen because the AdBlue® dosing was performed manually, which does not allow sufficiently fast adjustment of injection quantity during rapid load changes. Therefore, the sensor behavior was investigated under conditions where the AdBlue® dosing was incrementally increased until ammonia breakthrough occurred, which the sensors were expected to detect. Several measurements with two sets of sensors were conducted for different NH₃ concentration ranges. Both capacitive sensors and both mixed-potential sensors were pre-characterized in the laboratory setup and exhibited the behavior described above.

3.2.1. Low concentration range < 150 ppm NH₃

During engine operation, the reducing agent was overdosed until a maximum concentration of around 150 ppm of NH₃ occurred downstream of the SCR catalyst measured by the FTIR spectrometer. Based on the laboratory results, this range corresponds to the region in which both sensor types exhibit their highest sensitivity. Fig. 9 shows the time-dependent raw data of the measurement.

Plots a) and b) show the raw data from the capacitive and mixed-potential sensors. Plot c) shows the time-dependent behavior of NO_x and NH₃ downstream of the SCR catalyst. The raw NO_x concentration upstream of the DOC was measured using the commercial NO_x sensor with an output signal of about 1500 ppm at constant conditions. NO, NO₂, and NH₃ downstream of the SCR catalyst were measured using an FTIR spectrometer. At the start of the measurement, when AdBlue® dosing was not yet active, the same total NO_x concentration was also

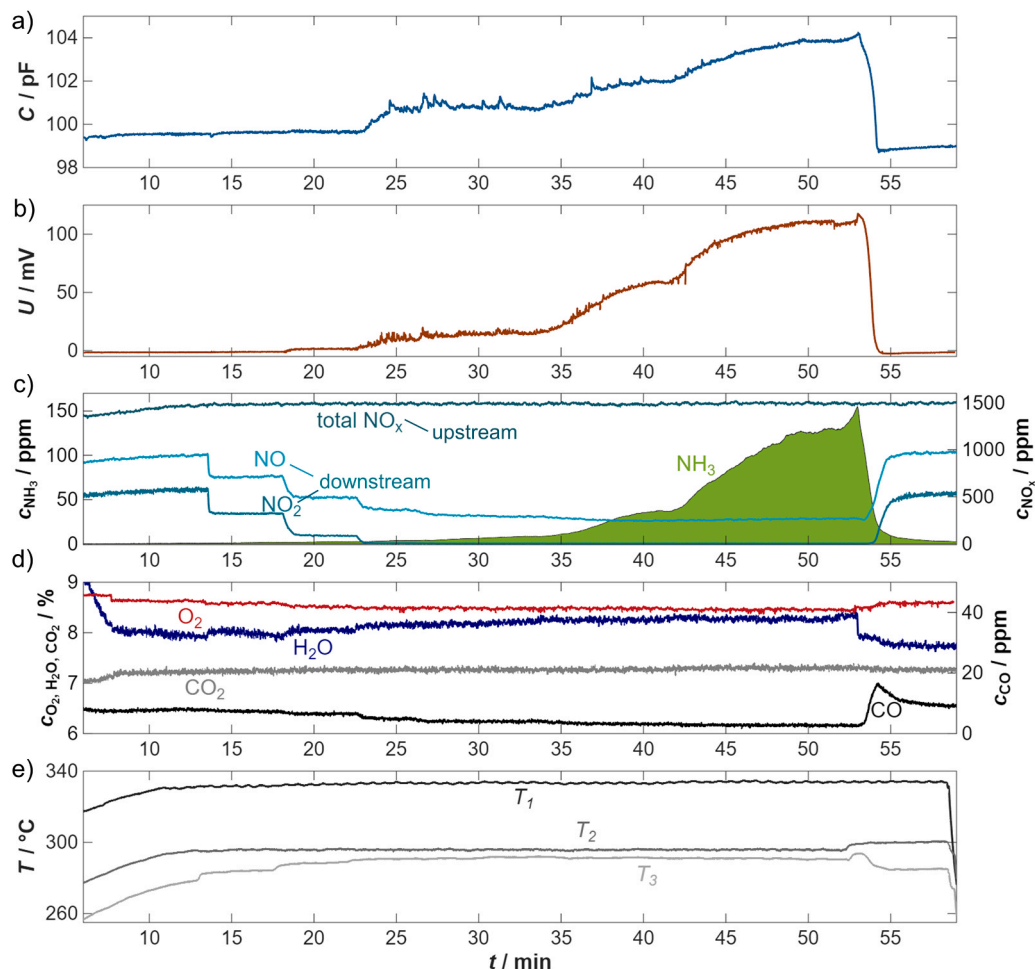


Fig. 9. Time curve of raw data during a real exhaust gas measurement on an engine test bench with an increase in AdBlue® dosage. a) Data of the capacitive sensor. b) Data of the mixed-potential sensor. c) Upstream NO_x concentration measured by the commercial NO_x-sensor, downstream NO, NO₂, and NH₃ concentration measured by the FTIR spectrometer. d) O₂, H₂O, CO₂, and CO downstream concentrations. e) Temperatures along the exhaust gas path. Sensor temperatures: $T_{H,cap} = 400$ °C and $T_{H,mix} = 550$ °C.

determined by FTIR. The NO/NO_2 ratio here was 62:38 due to the upstream DOC.

Incremental increases of the AdBlue® injection (e.g., at 13 and 18 min) led to a reduction of the NO_x concentration downstream of the SCR catalyst due to the reactions described by Eqs. (1) and (2). At this stage, no NH_3 slip was observed. With further NH_3 dosing, the NO_x conversion reached a maximum of approximately 82%, while NO_2 was completely reduced through the fast SCR reaction. About 260 ppm NO remained in the exhaust gas. Continued intentional overdosing resulted in an NH_3 concentration of up to 150 ppm downstream of the catalyst.

Other exhaust parameters remained largely stable due to the stationary operation. The water content increased slightly from 8.0% to 8.4%, and the oxygen content decreased from 8.8% to 8.5%. These minor changes are attributable to SCR reactions consuming O_2 and producing H_2O . After NH_3 dosing ended at approximately 54 min, the exhaust conditions returned to their initial state.

CO emissions remained very low (below 20 ppm) due to the DOC, as illustrated in Fig. 9d. Thus, potential cross-sensitivities were generally negligible. Exhaust temperatures remained relatively constant as well. Due to the uninsulated exhaust pipe, the temperature decreased from 335 °C upstream (T_1) of the SCR to roughly 290 °C (T_2 , T_3) at the sensor location (Fig. 9e).

The raw data of both sensor variants exhibited a time-dependent behavior consistent with the FTIR-measured NH_3 concentration. As in the laboratory measurements, the capacitive sensor exhibited a baseline capacitance near 100 pF. Therefore, the analysis focused on the absolute change of the capacitance. Both sensors showed stable baseline signals before ammonia breakthrough (until approx. 23 min). Variations in NO_x concentration did not significantly affect both sensors. The capacitive sensor would only show NO_x -induced changes in the presence of NH_3 as observed in the lab, and the mixed-potential sensor showed only minor NO_x cross-sensitivities.

After stopping AdBlue® injection, both sensors returned to their initial baseline values. For evaluation, the laboratory-derived calibration curves (from comparable base gas compositions) were used without additional corrections. Fig. 10 compares the calculated NH_3 concentrations from the sensor outputs with the FTIR measurements.

Plots a) and c) of Fig. 10 compare the time-dependent NH_3 concentrations, using calibration curves derived from measurements of 3.1. Both sensors agreed well with the FTIR data, with rapid responses to

NH_3 changes, particularly to the drop at around 52 min.

At low NH_3 concentrations, both sensors detected the onset of ammonia slip earlier than the FTIR. Several factors may contribute to this. The increasing water content or decreasing oxygen content in the exhaust gas during measurement would lead to a slight increase in the sensor signal for both sensor variants and thus to an overestimation of the NH_3 concentration. Another possible reason for the deviation could also be attributed to the piping to the FTIR. Although the pipes were heated to a temperature of approx. 150 °C, sorption processes can still occur in the pipes and the pump located between them, which can lead to an increased time delay at the FTIR spectrometer, especially in the case of ammonia. This circumstance is particularly evident when the AdBlue® dosing is switched off at about 52 min. There, both sensor responses returned significantly faster to the baseline signal than the FTIR spectrometer detects 0 ppm of NH_3 . This indicates that small concentrations (< 50 ppm) in particular could be detected much faster by the sensors in direct contact with the exhaust system than by the FTIR spectrometer (with its long pipes). Accordingly, only a limited statement can be made here regarding the response time of the sensors. In the case of rapid, significant changes, such as shortly before the 52-minute mark, for example, the response time of both sensors is in the range of a few seconds.

At higher concentrations, both sensors continued to show a high degree of agreement with the FTIR. This was also shown by the direct comparison of the NH_3 concentrations determined by the sensors and the FTIR spectrometer in Fig. 10 b) and d), evaluated during the entire increase in NH_3 concentration up to 52 min. This confirmed that both sensors slightly overestimated NH_3 at low concentrations but aligned increasingly well with FTIR data at higher levels.

3.2.2. High concentration range > 150 ppm NH_3

A second test run was conducted under similar operating conditions using the second set of sensors, but the NH_3 concentration was further increased, reaching up to 320 ppm. This is consistent with the upper concentration range examined in the laboratory. Fig. 11 shows the NH_3 concentrations already calculated from the sensor signals using the laboratory calibration curves as well as the raw data.

At lower concentrations, the same behavior observed in the measurement of Fig. 10 appeared. Both sensors detected the rise earlier than the FTIR. At concentrations above 200 ppm, the capacitive sensor

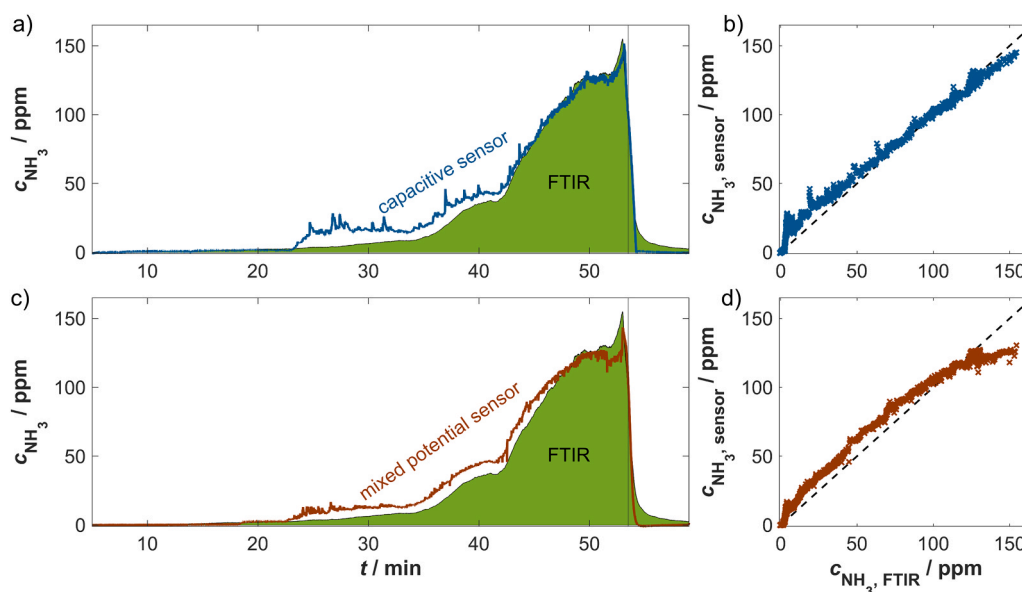


Fig. 10. Comparison between the NH_3 concentration determined using an FTIR spectrometer and the sensors, converted using the characteristic curve determined in the laboratory (refer to Fig. 4). a) and c) Representation of the time courses. b) and d) Direct comparison of the NH_3 concentration determined by the sensors and FTIR spectrometer. Sensor temperatures: $T_{\text{H,cap}} = 400$ °C and $T_{\text{H,mix}} = 550$ °C.

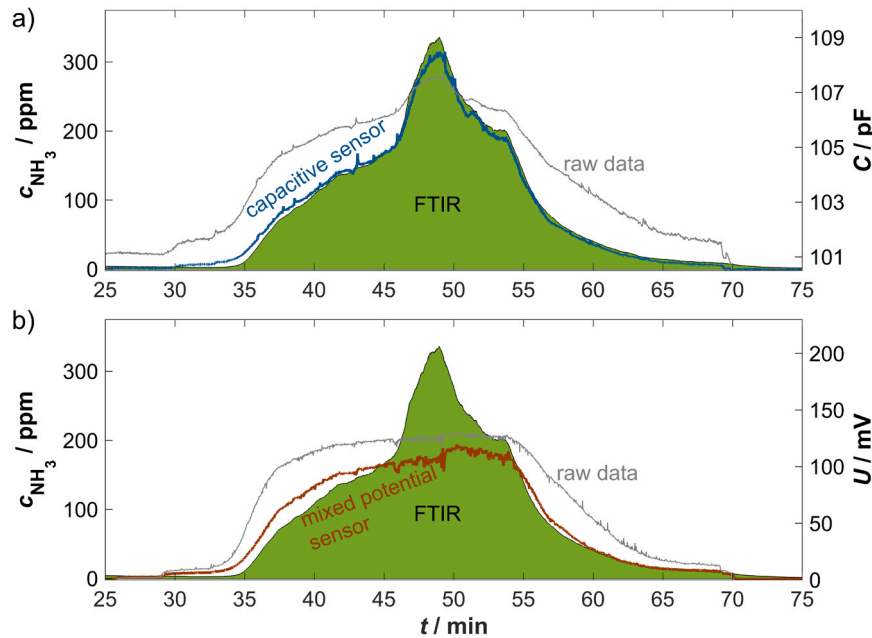


Fig. 11. Time curves of the sensor and FTIR signals with an increased overdose of AdBlue®, resulting in ammonia concentrations of up to a maximum of 320 ppm. a) Raw and converted data of the capacitive sensor. b) Raw and converted data of the mixed-potential sensor. Sensor temperatures: $T_{H, \text{cap}} = 400^\circ\text{C}$ and $T_{H, \text{mix}} = 550^\circ\text{C}$.

continued to match the FTIR readings well, whereas the mixed-potential sensor exhibited saturation at around 180 ppm. Consequently, higher concentrations could no longer be detected. This saturation behavior was already observed in the laboratory studies. Due to the semi-logarithmic response characteristic, the resolution decreases at high NH_3 concentrations. Additionally, engine operation may cause increased sensor cooling due to the high exhaust flow velocities. Despite adjusting the heater power based on the resistance of the back-sided heater, an increased temperature difference between the front and rear

side of the sensor may occur. According to [50], lower front-side temperatures can shift the electrochemical equilibrium and cause earlier saturation of the mixed-potential signal. The cooling effect may not have such a strong impact on the capacitive sensor, as it operates at a lower operating temperature of 400°C and showed lower temperature dependence in preliminary work [38].

To address this effect of the mixed-potential sensor more in detail, an additional measurement was conducted at an elevated sensor temperature of 580°C instead of 550°C . Previous studies on this sensor principle

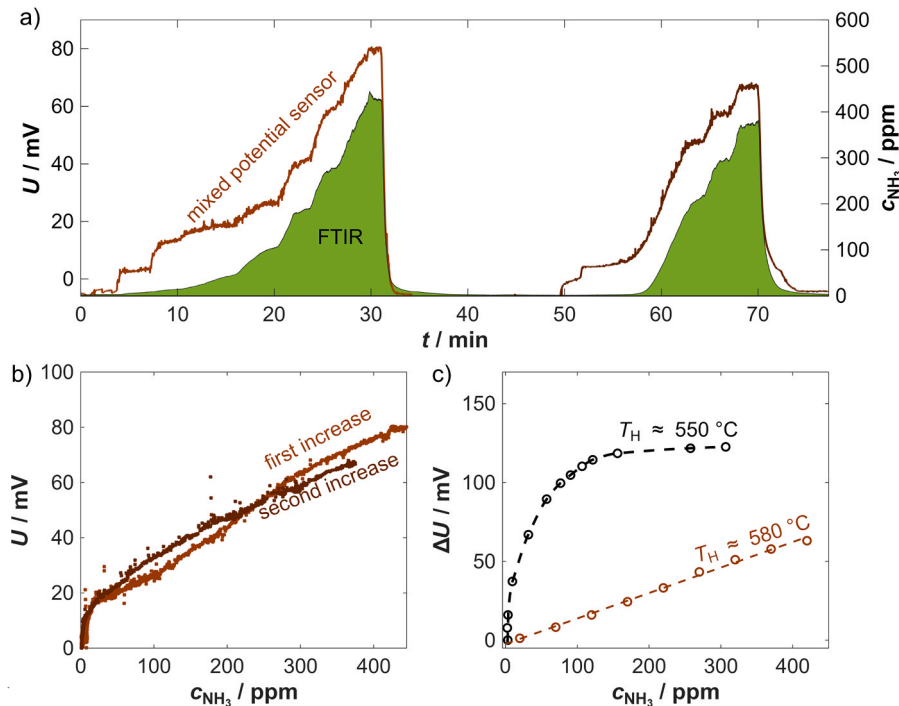


Fig. 12. Further investigation of the mixed-potential sensor for detecting elevated ammonia concentrations at a sensor temperature of $T_{H, \text{mix}} = 580^\circ\text{C}$. a) Time curve of two consecutive engine runs with increasing NH_3 concentration in the exhaust gas (raw data). b) Characteristic curves during the two engine runs. c) Comparison of the characteristic curves during engine operation at $T_{H, \text{mix}} = 550^\circ\text{C}$ and 580°C .

have shown that an increase in sensor temperature causes a change in the characteristic curve, which could improve the resolution of higher NH_3 concentrations. The raw data from this engine experiment are shown in Fig. 12, as no corresponding laboratory pre-characterization at 580 °C was conducted and therefore no calibration curve was available.

Under these conditions, NH_3 concentrations up to 400 ppm were reached. Two engine operating points were tested, causing slight differences in the water content (7.2 → 6.1 vol%) and in the oxygen content (8.5 → 10.2 vol%) between the first to the second increase of ammonia dosage. Throughout the entire measurement period, the sensor responded to increases in AdBlue® dosing across the entire 400 ppm NH_3 range (Fig. 12a).

The characteristic curves obtained from the engine measurements are shown in Fig. 12b and c. These reveal that increasing the sensor temperature caused a shift in the characteristic curve, with the sensor response moving from a semilogarithmic to a nearly linear dependency. This means that although the voltage changes are smaller at 580 °C than at 550 °C, higher NH_3 concentrations can be resolved when required simply by adjusting the sensor temperature.

3.2.3. Post-characterization

After the engine test bench measurements, all sensors were re-installed in the laboratory setup for post-characterization to identify potential degradation effects. Fig. 13a and b show the NH_3 characteristics of sensors used in the first engine test, while Fig. 13c and d show those from the second test. Therefore, the characteristic curves of the pre- and post-characterization of the sensor were compared, including a curve

determined during the engine run.

Both zeolite-based capacitive sensors exhibited similar responses compared to their initial characterization, though a slight decrease in sensitivity was observed. This may indicate mild aging of the zeolite film. However, mechanical damage during installation or removal cannot be entirely dismissed, since the zeolite film is sensitive to abrasion caused by shearing forces. This may have influence on the sensor response, leading to a lower capacitance signal.

The mixed-potential sensors showed comparable behavior. Functionality remained intact, and deviations from the pre-characterization curves were minor. Possible aging mechanisms include sintering or deactivation of catalytic sites were not pronounced within the duration of the experiments.

Overall, both sensor variants demonstrated stable behavior over the total test period. Statements on long-term stability over extended operational hours cannot yet be made.

4. Conclusion and outlook

In summary, both investigated sensor principles, the zeolite-based capacitive sensor and the mixed-potential sensor, were found to be suitable for detecting ammonia in the exhaust gas of SCR systems. Within the scope of the laboratory investigations, both sensor types demonstrated consistent and reproducible responses to changes in ammonia concentration, in accordance with their respective functional principles. The findings from the laboratory measurements could be successfully transferred to real exhaust conditions at the engine test

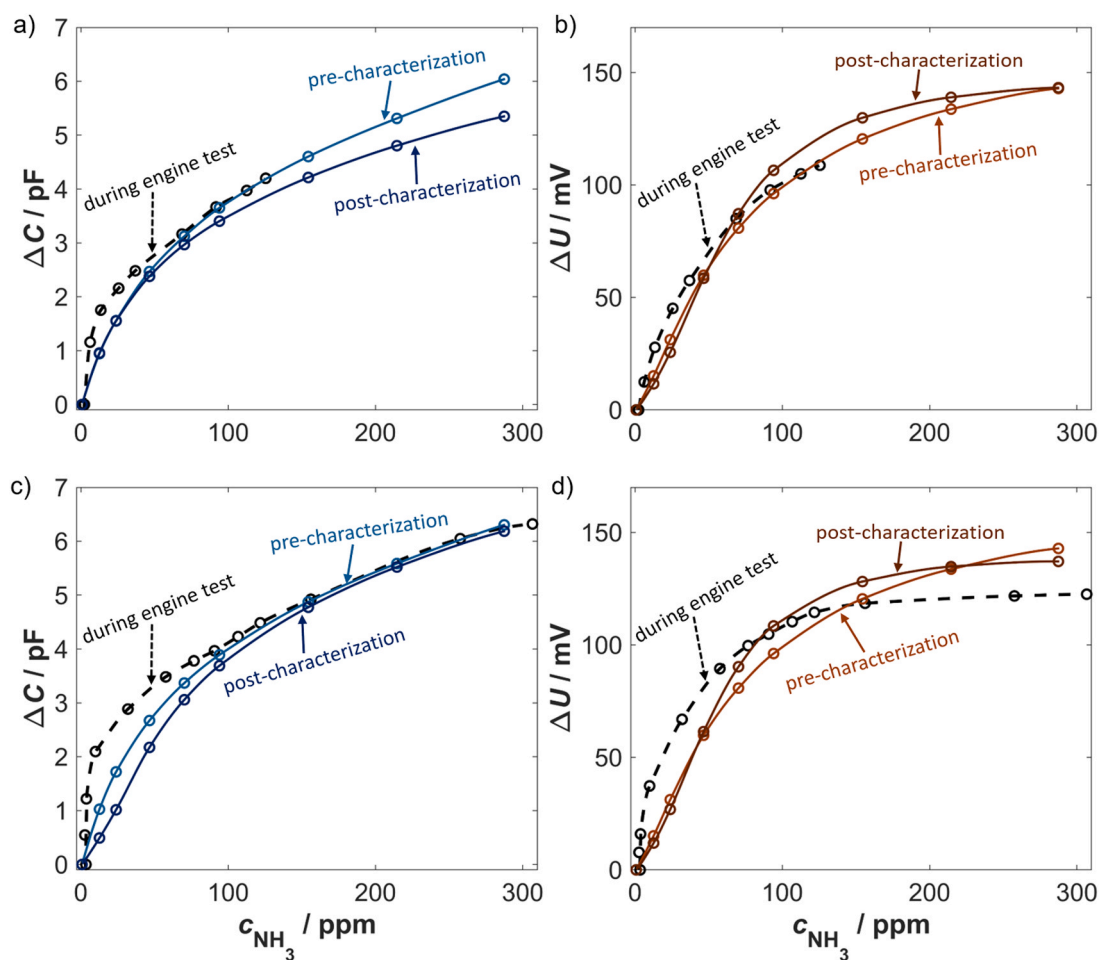


Fig. 13. Comparison of the NH_3 characteristic curve of the sensors before, during, and after measurement on the engine test bench. a) and b) Sensors from the first engine run. c) and d) Sensors from the second engine run. Base gas composition: 10 vol% O_2 , 10 vol% H_2O , 3 vol% CO_2 in N_2 , sensor temperatures: $T_{\text{H,cap}} = 400$ °C and $T_{\text{H,mix}} = 550$ °C.

bench, where the response of the sensor signals could be well correlated with the FTIR spectrometer. The sensors were able to quickly detect increases in ammonia concentration and also exhibited a rapid decrease in signal after the ammonia supply was stopped. By adjusting the temperature of the mixed-potential sensor, it was also possible to cover a higher NH_3 concentration range. In the present study, the use of a protective cap together with active temperature control via the heater resistance was found to effectively stabilize the sensor under varying exhaust gas conditions, with minimal impact on temperature. The protective cap reduced the impact of rapid thermal fluctuations, while the closed-loop adjustment of heater power limited temperature-induced signal drift. Future developments may include flow-optimized mounting position and the integration of the instantaneous power demand into the control strategy in order to further improve sensor robustness under dynamic operating conditions [71].

In addition, the influence of cross-sensitivities on the obtained signals were determined. It was demonstrated which gas components besides ammonia can produce a change in the sensor response, and the extent to which they influenced the ammonia-dependent characteristic curve. The capacitive ammonia sensor was found to be most affected by variations in water content and dosing of nitrogen oxides in combination with ammonia. For the mixed-potential sensor, the most significant influences were found to be oxygen, although carbon monoxide had an influence on the mixed-potential sensor. However, this is negligible when the sensor is used downstream a DOC, where most of the CO is oxidized. Under the real exhaust conditions examined, the influences of the other cross-sensitivities were also minimal.

Characterization after the real exhaust tests revealed that all functional characteristics of both sensor variants remained intact. Slight changes in sensitivity compared to the initial measurements were observed for both principles. Nevertheless, the characteristic curves showed that the typical behavior of both variants were almost unchanged after the operating time on the engine test bench.

This provides a promising basis for future work. Further investigations should focus on the effects of dynamic engine operation, which could not be guaranteed here due to the manual AdBlue® dosing. The results obtained under stationary operating conditions could thus serve as a basis for future controlling of the AdBlue® dosing. In dynamic engine operation, a stronger influence of H_2O and O_2 cross-sensitivity can be expected. Here, a correction of the sensor signal with the help of an existing lambda probe is possible, as has already been investigated for the capacitive sensor in other applications by Wöhrl et al. [62]. There is also further interest in investigations into long-term stability in order to systematically record possible aging mechanisms of the functional films.

In the long term, both sensor concepts could contribute not only to the control and monitoring of diesel SCR systems, but also to future low-emission drive concepts, such as hydrogen- or ammonia-based engines. Therefore, the further development of selective, fast and thermally stable NH_3 sensors is an important component for ensuring conformity with future emission standards and the safe use of alternative energy sources.

CRediT authorship contribution statement

Nils Donker: Writing – review & editing, Visualization, Validation, Software, Methodology, Formal analysis. **Andreas Müller:** Writing – review & editing, Validation, Data curation. **Daniela Schönauer-Kamin:** Writing – review & editing, Validation, Project administration, Conceptualization. **Gunter Hagen:** Writing – review & editing, Validation, Project administration, Conceptualization. **Thomas Wöhrl:** Writing – original draft, Visualization, Validation, Methodology, Formal analysis, Data curation. **Rosa Ehlert:** Writing – review & editing, Visualization, Validation, Formal analysis, Data curation. **Ralf Moos:** Writing – review & editing, Supervision, Funding acquisition, Conceptualization.

Declaration of Generative AI and AI-assisted technologies in the writing process

During the preparation of this work the authors used DeepL and Microsoft Copilot for linguistic refinement and as a reference point for locating relevant literature data. After using this tool/service, the authors reviewed and edited the content as needed and take full responsibility for the content of the published article.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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