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Progressing TDLAS instrumentation for SI-traceable measurements of nitrous oxide in maritime applications

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Abstract: Nitrous oxide (N_2O) is gaining interest in maritime applications for several reasons, primarily related to engine performance enhancement, emissions reduction, and auxiliary power supply. While N_2O can enhance combustion efficiency and reduce certain emissions, uncontrolled use could result in increased NO_x formation and other pollutants. Effective N_2O monitoring is critical in maritime applications for ensuring operational safety, regulatory compliance, and optimal performance. We introduce an innovative first-principles N_2O spectrometer designed specifically for maritime applications, such as monitoring emissions during and after combustion processes. This TDLAS based spectrometer targets the P29e line at 4.55 µm spectral band and provides direct absolute mole fraction measurements, eliminating the necessity for prior or routine calibration.

Keywords: TDLAS, SI-traceable, N_2O, maritime, pollutants formation, TILSAM

German title: **Weiterentwicklung der TDLAS-Instrumentierung für SI-rückführbare Messungen von Lachgas in maritimen Anwendungen**

Keywords: TDLAS, SI-rückführbar, N_2O, maritim, Schadstoffbildung, TILSAM

1 Introduction

Maritime transport plays a crucial role in global trade but is a significant contributor to greenhouse gas (GHG) and pollutant emissions. Among these, nitrous oxide (N_2O) has garnered increasing attention due to its potent greenhouse effect and role in ozone depletion. N_2O is approximately 300 times more effective at trapping heat in the atmosphere than carbon dioxide (CO_2) over a 100-year period, making it a critical driver of climate change [1]. Moreover, it is the most significant ozone-depleting substance emitted in the 21st century, underscoring the need for accurate monitoring and regulation [2]. In response to the environmental impact of N_2O, regulatory bodies such as the European Union (EU) and the International Maritime Organization (IMO) have implemented measures to mitigate emissions. The EU has incorporated N_2O into its Emissions Trading System (ETS), subjecting maritime transport to emission caps and trading mechanisms starting in 2026 [3]. Additionally, the EU's FuelEU Maritime initiative establishes stringent targets for reducing the GHG intensity of fuels, including N_2O, using a Well-to-Wake (WtW) approach to ensure comprehensive lifecycle accountability [4]. On an international scale, the IMO's 2023 Strategy on Reduction of GHG Emissions from Ships mandates annual reporting and monitoring of N_2O emissions as part of broader decarbonization efforts [5]. While MARPOL Annex VI primarily focuses on nitrogen oxides (NO_x), regulatory discussions are increasingly emphasizing direct N_2O emission control, highlighting the necessity for precise measurement technologies.

Despite its significance, accurately quantifying N_2O from ships remains challenging due to limitations in measurement technologies and the absence of standardized, SI-traceable instrumentation. Current emission inventories often rely on estimates rather than direct measurements, introducing uncertainties regarding the true environmental impact of maritime transport. Enhanced measurement capabilities are essential for regulatory compliance, emissions mitigation

strategies, and a more comprehensive understanding of N_2O's contribution to global warming and atmospheric chemistry.

Several analytical techniques have been employed for N_2O measurement, each with distinct advantages and limitations. Gas chromatography (GC) with electron capture detection (ECD) is a widely used laboratory method known for its high sensitivity and specificity [6]. However, GC-ECD requires extensive sample preparation and is unsuitable for real-time, in-field measurements. Infrared absorption spectroscopy, including Fourier-transform infrared spectroscopy (FTIR), enables continuous monitoring but may suffer from interference by other gases [7]. Chemiluminescence-based techniques, primarily used for nitrogen oxide (NO_x) monitoring, can be adapted for N_2O detection, though they necessitate additional conversion steps and calibration procedures [8]. Mass spectrometry (MS) provides high precision and selectivity for N_2O analysis; however, its complexity and cost limit its widespread application outside laboratory environments [9]. Emerging optical techniques, such as cavity-enhanced absorption spectroscopy (CEAS) and off-axis integrated cavity output spectroscopy (OA-ICOS), offer high sensitivity and real-time detection, making them promising candidates for in-situ N_2O measurement [10].

Tunable Diode Laser Absorption Spectroscopy (TDLAS) has emerged as a leading technique for real-time, high-precision gas analysis. TDLAS provides high selectivity, rapid response times, and low detection limits, making it particularly suitable for monitoring trace gases such as N_2O [11]. Studies have demonstrated the efficacy of TDLAS in detecting N_2O across diverse environments, including laboratory setups, industrial emissions, and atmospheric monitoring [12]. Compared to traditional methods such as GC and chemiluminescence, TDLAS enables a non-invasive, in-situ approach with the potential for continuous monitoring. Advances in laser technology, optical sensing techniques, and spectral data processing have further enhanced the sensitivity and accuracy of TDLAS for N_2O detection [13]. However, to facilitate its deployment in maritime applications, further improvements in instrumental design, calibration methods, and data validation are necessary to ensure traceability to the International System of Units (SI). Addressing these challenges will support reliable and standardized N_2O measurements in the shipping industry.

The MaritimeMET project [14], selected for funding under the European Partnership on Metrology, is co-financed by the European Union's Horizon Europe Research and Innovation Programme and participating states. This project aims to advance measurement techniques for controlling and monitoring ship engine emissions through SI-traceable measurements of pressure, temperature, and emission compositions in real-world maritime applications. In this context, we investigate whether tunable diode laser absorption spectroscopy (TDLAS) can be developed and optimized to provide SI-traceable measurements of nitrous oxide (N_2O) in complex maritime environments. This study specifically focuses on advancing TDLAS instrumentation to address challenges such as sensitivity, selectivity, and stability. The system is designed to comply with the TILSAM protocol [15] (traceable infrared laser-spectrometric amount fraction measurements), which ensures high-precision, SI-traceable gas amount fraction measurements using infrared (IR) laser spectroscopy and comprehensive uncertainty evaluation. This approach supports robust and reliable data generation for both regulatory compliance and scientific research.

2 Methodology

In TDLAS, the wavelength (λ) of a tunable laser is swiftly scanned across a narrow segment of the optical spectrum, encompassing one or more electronic, vibrational, or rotational-vibrational absorption lines. The intensity of the laser light, denoted as $I(\lambda)$, that is focused onto a photodetector after traversing the sample containing absorbers can be characterized by the extended Lambert-Beer law [16].

$$I(\lambda) = E(t) + I_0(\lambda) \cdot T_r(t) \cdot \exp[-S(T) \cdot g(\lambda - \lambda_0) \cdot N \cdot L] \quad (1)$$

The analysis involves the background emission $E(t)$ at a specific time t, the initial laser intensity $I_0(\lambda)$, and the broadband transmission losses $T_r(t)$, all of which are derived synchronously from the individual raw signals and absorption profiles. The exponential term incorporates the absorption line strength $S(T)$ at the gas temperature T, the normalized line shape function $g(\lambda-\lambda_0)$ (which is centered at the wavelength λ_0), the absorber number density N, and the optical path length L.

By integrating Equation (1) with the ideal gas law, we can determine the gas concentration, expressed as the amount fraction x [17].

$$x = \frac{k_B \cdot T \cdot A_{\text{line}}}{S(T) \cdot L \cdot p_{\text{total}}} \tag{2}$$

where k_B is the Boltzmann constant, p_{total} is the total pressure of the gas sample, and A_{line} is the line area determined by spectral integration of the measured absorption line over the wavenumber axis. The dynamic wavelength tuning (wavenumber axis) coefficient of the laser has to be determined experimentally. This is extracted from the Airy-signal when the laser light is transmitted through an optical etalon.

Laser spectrometry enables the quantification of traceable amount fractions of gas species in gas mixtures, as outlined in the TILSAM protocol in which it defines the different terms used to describe the spectrometric measurement process, the model used to process the raw data and the subsequent estimation of uncertainties associated to the results of this method. The data processing algorithm based on TILSAM is illustrated in Fig. 1. The quality of being free-of-calibration with reference gas standards has been proved in EUROMET project 934 (EURO 934) [18]. First, the absorption line area (in the wavenumber domain, cm^{-1}) is determined using the measured laser intensity and tuning characteristics, which are evaluated through an etalon experiment. In addition to the spectrometric measurements themselves, TILSAM also relies on the measurement of the gas parameters, pressure and temperature. The respective measurements have to be performed with calibrated devices in order to make the results traceable. Subsequently, the amount fraction is calculated following Equation (2).

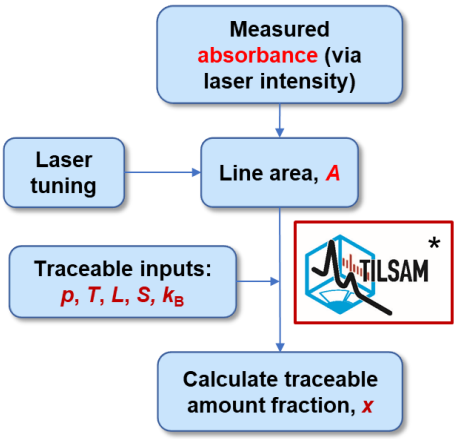

Fig. 1: The flow chart of TDLAS data processing algorithm based on TILSAM. *p*: pressure; *T*: temperature; *L*: path-length; *S*: line intensity; k_B: Boltzmann constant.

3 Experimental setup

To evaluate and quantify N_2O absorption lines, a custom-built laser absorption spectroscopy (LAS) system was developed. A schematic representation of the experimental setup is provided in Fig. 2. N_2O measurements were conducted in a static configuration within a gas cell. A continuous-wave distributed-feedback interband cascade laser (CW-DFB-ICL, Nanoplus) centered at 2196.7 cm^{-1} was employed to probe the P29e transition of N_2O. The laser wavelength was precisely controlled using a laser diode controller (PRO8000, equipped with LDC8002 and TED8020, Thorlabs) in conjunction with a function generator (KEYSIGHT, 33500B). A triangular current ramp with a tuning frequency of 140 Hz was applied. Prior to measurements, dynamic laser tuning was calibrated using a Germanium etalon (76.244 mm in length), traceable to the Physikalisch-Technische Bundesanstalt (PTB) length standard. The etalon measurements enabled conversion of the spectral *x*-axis from time to wavenumber. The laser beam was collimated and directed through a 32.52 cm stainless steel gas cell with wedged CaF_2 windows before being focused onto a photodetector (PVI-4TE-5, VIGO). Signal acquisition was performed at 16-bit resolution with a sampling rate of 600 kHz using a data acquisition card (USB-6363, National Instruments). Gas pressure and temperature were monitored using a capacitance manometer (0–1000 mbar, MKS Baratron) and a Pt100 resistor, respectively. Both

sensors were calibrated and traceable to the International System of Units (SI) through PTB's national pressure and temperature standards.

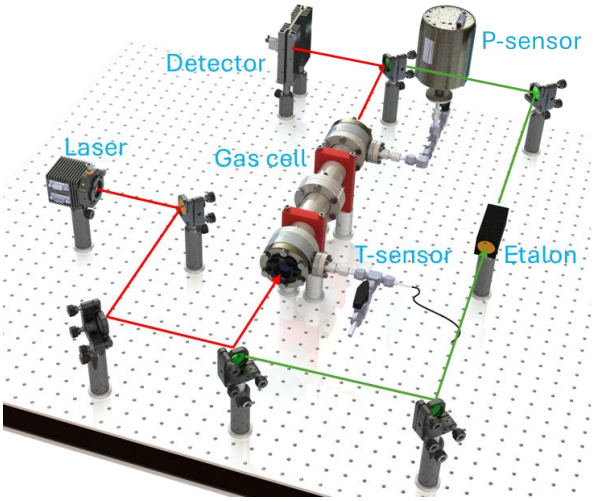

Fig. 2: Schematic of the experimental N_2O TDLAS setup. P-sensor: pressure sensor; T-sensor: temperature sensor.

4 Results

Figure 3 (left) presents a typical transmitted laser signal recorded after passing through the gas cell containing the N_2O–air mixture. The measured signal reflects the combined effects of absorption by N_2O molecules and any baseline variations introduced by the optical components and laser source. The observed spectral features correspond to the characteristic absorption of N_2O at the target wavelength range, while the overall signal intensity is influenced by factors such as laser power stability, optical alignment, and detector response. This transmitted signal serves as the basis for further spectral analysis, including baseline correction and absorption profile fitting, to accurately determine the gas mole fraction and spectroscopic parameters. To accurately determine the absorbance line area (A_{line}), the spectrometric measurement must be performed by spectrally sweeping the laser emission wavelength across the target absorption line. This process ensures the full capture of the absorption profile necessary for precise quantitative analysis. Since the laser wavelength sweep is often controlled and recorded in the temporal domain (i.e., as a function of time), it is essential to establish a reliable method for converting this sweep into the wavenumber domain. This transformation is crucial for accurate spectral fitting and for ensuring consistency with reference databases such as HITRAN [19]. The conversion typically requires calibration based on an external frequency reference, such as an etalon or known absorption features, allowing precise mapping of the laser's time-dependent tuning to wavenumber units. By implementing an accurate wavelength-to-wavenumber conversion, the integrated absorbance can be evaluated with high precision, enabling robust spectroscopic analysis of N_2O and other trace gases. Figure 3(right) shows a typical measured etalon signal (FSR 0.016 cm^{-1}). It was used to convert the x-axis of the measured spectra from the time to wavenumbers domain by extracting the Etalon peak and performing 3rd polynomial fit to get the relative wavenumber.

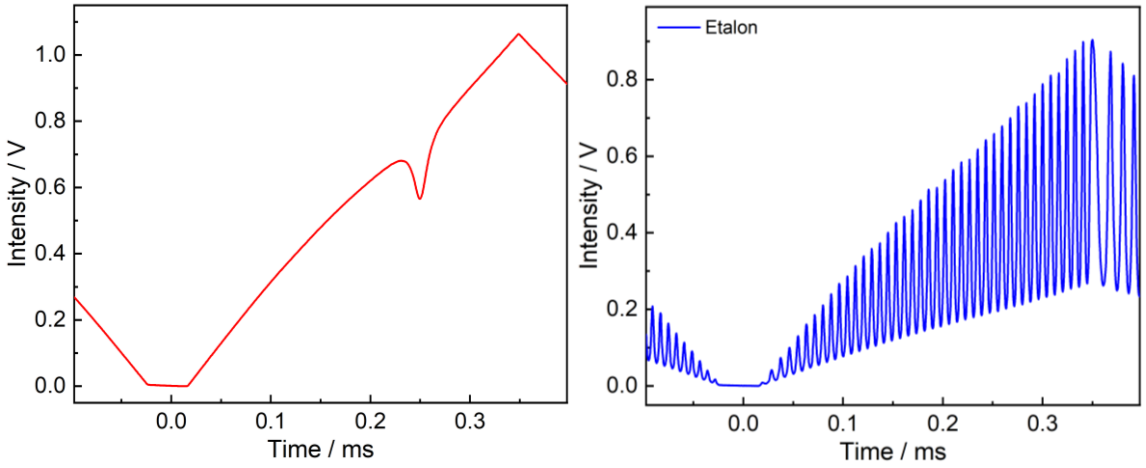

Fig. 3: Left: the measured typical laser intensity with N_2O absorption peak; Right: the measured Etalon signal.

To validate the wavenumber scale measured using the etalon, the obtained relative wavenumber values are compared with reference data from the HITRAN database, as illustrated in Fig. 4. In this analysis, the position of the third N_2O absorption peak (indicated by the dashed line in Fig. 4) is used as a reference point, with its wavenumber assigned according to HITRAN. The deviations between the measured wavenumber values and those reported in HITRAN are found to be within approximately 10^{-3} cm^{-1}, demonstrating a high degree of accuracy in the measurement.

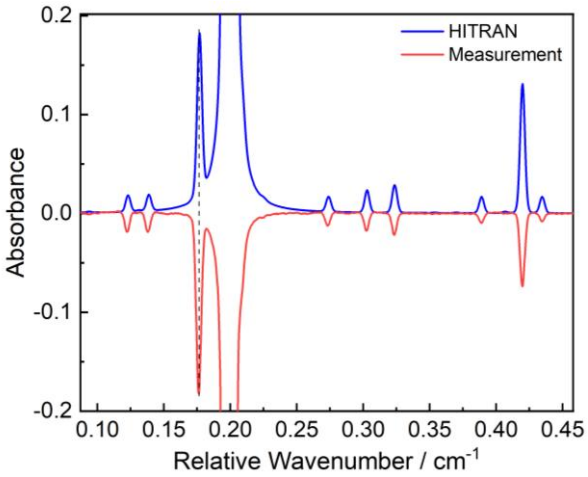

Fig. 4: The line positions between measured and HITRAN database.

A gas mixture of N_2O and air was prepared in the gas cell following a systematic procedure to ensure consistency. The preparation steps were as follows: 1) The entire system, including the gas cell, gas manifold, and all connected components up to the gas cylinder, was evacuated to remove any residual gases. 2) The gas cell was flushed with pure N_2O and subsequently evacuated to eliminate any remaining impurities. 3) Step 2 was repeated three times to further ensure the purity of the N_2O within the system. 4) A small amount of pure N_2O was retained in the gas cell at a pressure below 0.05 mbar. 5) The buffer gas, air, was then introduced to achieve the desired gas mixture pressure. The total pressure within the gas cell was controlled by adjusting the gas cell outlet valve and the exhaust pump. Figure 4(a) presents three cases of measured absorption spectra for the N_2O–air mixture at different pressures. The laser signal was averaged over ten scans to enhance measurement stability and reduce noise. The baseline was fitted using a third-order polynomial, while the absorption profile was modelled using the Voigt function, fitted through a nonlinear Levenberg–Marquardt optimization algorithm. The residual between the measured and fitted data, shown in Fig. 4, is approximately $1\sigma = 1\times10^{-3}$ in optical density. This corresponds to a high signal-to-noise ratio (SNR) of approximately 300, indicating the reliability and precision of the measurement.

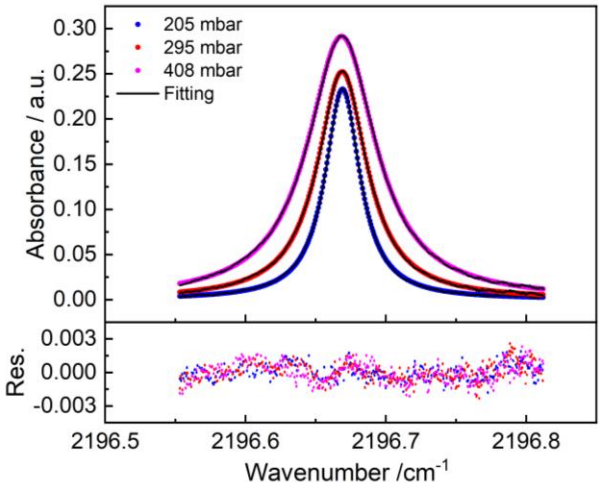

Fig. 5: Measured N_2O absorption spectra (10 scan averaged) in air mixture at pressures from 205 mbar to 408 mbar. The fitting residuals between measured and fitted data are shown below.

N_2O is generally considered to exhibit low surface adhesion (or "sticky" effects) compared to highly adsorptive molecules such as ammonia (NH_3) [20]. However, minor adsorption can still occur, particularly on untreated surfaces or under specific experimental conditions. Despite the fact that the inner surface of the gas cell was passivated according to the preparation steps outlined earlier, a prolonged period was still required to reach equilibrium after sealing the gas cell. This extended stabilization time can be attributed to two primary factors: 1) Degassing from Surface Adsorption – N_2O molecules initially adsorbed onto the passivated inner surface of the gas cell gradually desorbed into the gas

phase, contributing to slow equilibration. Even after passivation, residual adsorption sites may still be present, leading to a delayed release of N_2O into the gas mixture. 2) Gas Homogenization Time – The time required for the gas mixture to become fully homogeneous within the cell could also contribute to the observed delay. This effect may be exacerbated by diffusion limitations, minor thermal gradients, or weak convective mixing within the gas cell. These observations suggest that while N_2O adsorption is relatively weak compared to NH_3, residual surface interactions and gas mixing dynamics can still influence measurement stability and equilibration time.

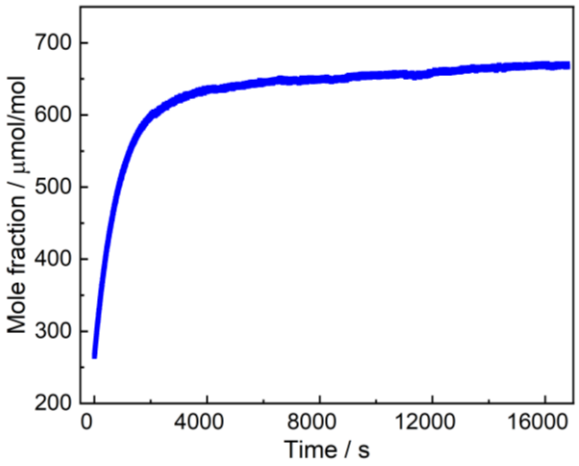

Fig. 6**:** Long term concentration measurements.

To assess the precision and repeatability of the N_2O-TDLAS system, a long-term measurement study was conducted after the gas mixture reached equilibrium. The measured mole fraction over time is presented in Fig. 7 (left), demonstrating the stability of the system under continuous operation. To further analyze the statistical distribution of the measured values, Fig. 7 (right) displays a histogram of the recorded mole fractions. The data exhibit a well-defined Gaussian distribution, indicating that the measurement noise is primarily stochastic in nature. A Gaussian function was fitted to the histogram, and the precision of the N_2O-TDLAS system was quantified using the half-width at half-maximum (HWHM) of the fitted curve, as described in [21]. The resulting HWHM value was determined to be 0.346 µmol/mol (0.2% in relative) at an integration time of 1.3 seconds, providing a quantitative measure of the short-term precision of the system. These results highlight the high measurement stability and reliability of the N_2O-TDLAS system, making it well-suited for precise trace gas analysis applications. Further improvements in precision may be achievable by increasing the integration time, as suggested by the Allan deviation analysis.

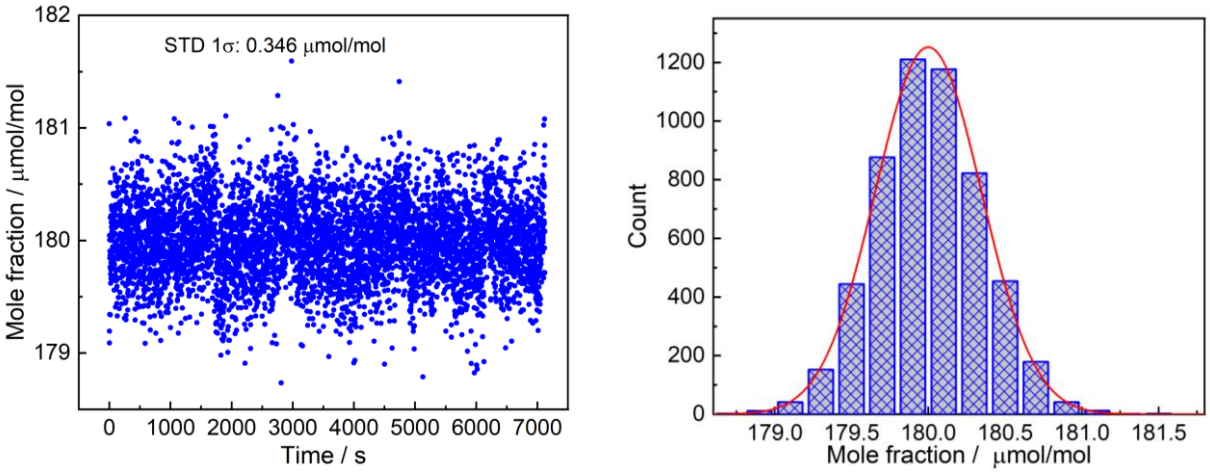

Fig. 7**:** Left: Long term mole fraction measurement results under the equilibrium condition. Right: Histogram of the measured mole fractions distribution and the Gaussian fit contour.

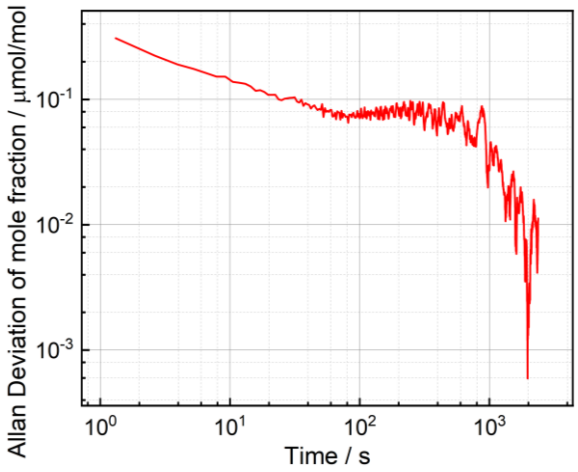

Fig. 8: Allan deviation plot for the time series shown in Fig. 7.

The precision and long-term stability of the spectrometer were evaluated using the Allan deviation analysis [22], as illustrated in Fig. 8. The Allan deviation quantifies measurement stability over time, revealing how precision improves with increased integration time due to the suppression of white noise through signal averaging. For short averaging times, the precision improves as expected. However, for longer averaging periods (ranging from 100 to 1000 s and beyond), the improvement is limited by long-term instabilities, such as thermal drifts and other systematic noise sources. At an integration time of 10 s, the precision of the spectrometer was determined to be 0.14 µmol/mol for N_2O. By extending the averaging time to 100 s, the precision was further improved to 0.07 µmol/mol (equivalent to 70 nmol/mol), demonstrating the high sensitivity of the instrument. Given that the mole fraction of the N_2O-air gas mixture in the experiment was 180 µmol/mol, this level of precision corresponds to a signal-to-noise ratio (SNR) of approximately 2570. These results highlight the spectrometer's capability for high-precision trace gas detection, making it well-suited for applications requiring accurate and stable N_2O measurements over extended periods.

5 Conclusions

[1]. This TILSAM protocol is a recommendation for using the principles of spectrometric methods to measure amount fractions of molecular species in a given gas mixture. According to this, in the paper we present a novel first principles N_2O spectrometer especially for applications in maritime e.g. during and from combustion processes. It is operated on the P29e line in 4.55 µm band and offers an absolute mole fraction response without a need for previous or regular calibration exercise. We tested and validated the new spectrometer under well-controlled lab conditions, i.e. detection limits. The precision, stability, and absorption characteristics of an N_2O-TDLAS system were investigated. The precision and repeatability of the N_2O-TDLAS system were further assessed using long-term stability measurements and Allan deviation analysis. The results showed that increasing the integration time reduced white noise, with a precision of 0.14 µmol/mol at 10 s and 0.07 µmol/mol at 100 s, yielding an excellent signal-to-noise ratio (SNR) of approximately 2570. Furthermore, statistical analysis of the measured mole fraction distribution confirmed a Gaussian noise profile, with a HWHM of 0.346 µmol/mol at 1.3 s integration time, demonstrating high short-term precision. These insights provide a solid foundation for further refinement of TDLAS-based N_2O sensing systems, with potential applications in environmental monitoring, industrial process control, and atmospheric science.

6 Acknowledgement

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