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ABSTRACT

Interband cascade lasers (ICL) are high output power mid-infrared light sources with low power consumption, serving as a viable alternative to more commonly applied quantum cascade lasers. The integration of ICLs with thin-film waveguides facilitates miniaturized sensing platforms based on the principles of evanescent field absorption toward tailorable high-fidelity portable mid-infrared (MIR) sensing solutions for on-site chemical analysis. The analytical performance of a MIR sensing system is presented combining a tunable interband cascade laser emitting at a wavelength range of 5.88–6.09 μm (1702–1641 cm^{-1}) with a 6 μm GaAs/AlGaAs thin-film waveguide as the active transducer for customized evanescent field absorption analysis of food contaminants. A comparison of the analytical performance of the developed tICL-based system vs conventional Fourier-transform infrared spectroscopy is presented for the exemplary analysis of the *Fusarium* mycotoxin deoxynivalenol, a persistent food contaminant resulting from fungal infection. The thin-film waveguide enhances the sensitivity compared to conventional attenuated total reflection systems, revealing improved detection limits. The compact design of the system, along with the favorable analytical figures-of-merit and automated data processing, confirm the potential of the developed tICL-based spectrometer for on-site detection of food contaminants at various stages along the food supply chain.

I. INTRODUCTION

In recent years, on-site analytical applications have been increasingly demanded by the food and feed industry and raw material suppliers. Regulations on food contaminants have been tightened due to the increasing evidence of their public health impact, environmental considerations, and globalization of food trading linked to the harmonization of international standards.[1,2] In response, efforts are focused on the development of in-field analytical solutions to monitor food contaminants directly at crop-lands or during transportation, storage, and processing of goods. In-field conditions are usually demanding for operating analytical devices, as they may be exposed to dust, temperature fluctuations, and mechanical impact/vibrations. In addition, the analytical workflow and the representation of the obtained results should be user-oriented with a focus on non-specialized operators.[3] In summary, it is evident that on-site analytical tools are moving toward compact, robust, and user-friendly technologies, facilitating their ubiquitous usage.[4,5]

The mid-infrared (MIR) electromagnetic window ranging from 3 to 20 μm is an emerging spectral window for food safety/quality applications, as the vibrational molecular signatures of food matrices result in pronounced MIR spectra providing detailed—albeit highly convoluted—chemical, structural, and compositional information. Next to appropriate data mining and interpretation strategies, a main focus has been the development of next-generation miniaturized yet efficient MIR light sources such as interband cascade lasers (ICL), which are suitable for integration into portable spectrometers. Portable field-deployable devices demand low-threshold currents and, therefore, a low overall power consumption along with properties such as room temperature operation, high optical output power operation within the 3–6 μm spectral regime, and wavelength tunability.[6,7] These advantageous properties have led to a range of applications, including but not limited to gas sensing, medical diagnosis and environmental pollution monitoring.[8]

While the properties of the light source are crucial, an optical sensing system also requires a transducer, i.e., an interface for intimately and reproducibly interacting photons with sample constituents. The combination of miniaturized light sources with appropriate waveguide technology facilitates lab-on-a-chip concepts based on an increasing number of suitable materials and configurations.[9] Using well-established microfabrication processes for deposition and structuring, Mizaikoff and collaborators[10] pioneered the development of semiconductor thin-film waveguides based on the GaAs/AlGaAs material system. The spectral window of these waveguides propagates radiation up to a wavelength of 13 μm and can be tailored to ideally meet the emission characteristics of ICLs toward maximizing the signal-to-noise ratio (SNR) during evanescent field absorption measurements. The sample, present at or close to the surface of the single-mode thin-film waveguides, is reached by the evanescent field of the light, which is leaking a few micrometers into the sample. If the evanescence field interacts with MIR absorbing molecules, the compound-specific attenuation of the IR radiation results in discriminatory IR spectra comparable to conventional macroscopic IR-attenuated total reflection (ATR) spectroscopy.[11,12]

In the present study, a miniaturized continuous wave (CW) broadly tunable MIR ICL (tICL) has been combined with a GaAs/AlGaAs thin-film waveguide and a thermoelectrically cooled mercury–cadmium–telluride (MCT) detector. The utility of this chemical sensing platform for food contaminant analysis has been demonstrated by characterizing its analytical performance for the exemplary analyte deoxynivalenol (DON), a regulated secondary fungal metabolite (a.k.a., mycotoxin) commonly found in cereals and cereal based food. Recent data demonstrate that up to 60% of food crops contain detectable concentrations of DON.[13]

The aim of the present study is the performance comparison of the developed sensing system with a conventional benchtop Fourier-transform infrared (FTIR) spectrometer using a single-reflection ATR assembly to highlight the further potential of the developed portable sensing system for the on-site detection of mycotoxins. This characterization is the starting point for further studies on the combination of technology with preconcentration strategies to reach on-site mycotoxin detection at regulatory limits.

II. MATERIALS AND METHODS

A. Mid-infrared tunable interband cascade laser (tICL)

The MIR tICL was developed by nanoplus Advanced Photonics Gerbrunn GmbH (Gerbrunn, Germany). It consists of a tunable IC laser diode mounted in a laser package, temperature-stabilized with a thermoelectric cooler, and a collimating lens at the beam exit. The package was integrated with a compact 5 × 5 × 5 cm heat sink. The tICL is controlled by four printed circuit boards (PCBs) purchased from Meerstetter GmbH, Germay: three PCBs driving the operation current and one PCB as a temperature controller to stabilize the laser chip temperature. The tICL emission wavenumber is controlled electronically by changing these currents and chip temperature. The shift in emission wavenumber is modulated by the current's joule heating and the temperature dependence of the refractive index in the different sections of the laser diode. The underlying principle is based on different detuned grating structures and the Vernier tuning principle, as described by Edlinger *et al.*[14] A detailed study on the tICL control will be published in a future article. This tuning was controlled by bespoke software (IRIS Technology Solutions, Spain), which transferred the programmed currents and temperatures to the PCBs. Through this approach, the spectrally single-mode emission wavenumber of the tICL was switched between six characteristic bands covering the spectral window of 1702–1641 cm^{-1}, while offering a high precision tuning (21 wavenumbers) around those specific bands by precise electronic control of the diode currents. Limiting the spectral range avoids the use of bulky components, additional optics and electronics, and cooling systems in comparison with multiple-laser commercial QCLs. The tailored MIR spectral window of the tICL covers the carbonyl stretching (C=O), typical in amide I bands of proteins and carboxylic acids of lipids, highlighted in Fig. 1. The spectral range was specifically selected to cover the absorption of the carbonyl group at C-8 of the DON molecule for its detection.

B. tICL coupling to the GaAs/AlGaAs thin-film waveguide

An optical system was designed and optimized to facilitate efficient coupling of the IR radiation emitted by the tICL into the

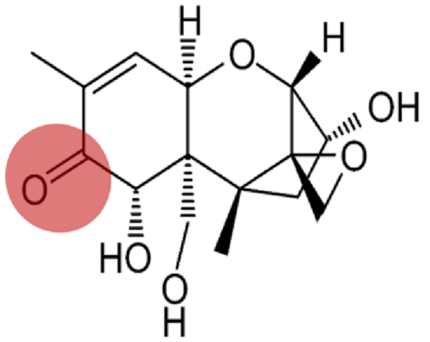

FIG. 1. Chemical structure of deoxynivalenol (DON), with the carbonyl group (C=O) at carbon atom eight highlighted in red.

6 μm GaAs waveguide layer fabricated on top of a Al$_{0.2}$Ga$_{0.8}$As optical buffer layer. The overall waveguide chip dimensions are 10 × 5 mm^2 mounted within a stainless-steel assembly (a.k.a., MIRa-Guide^{TM}) for analyzing 30 μl of liquid sample. Details on the waveguide technology have been published previously.[10,15–17] To guide and focus the IR beam onto the waveguide, two kinematic-mounted gold mirrors and a zinc selenide (ZnSe) 1/2″ lens (Thorlabs Inc., U.S.A.) were used. The waveguide assembly was incorporated to the top of an XY manual stage (Newport Corporation, U.S.A.), facilitating precise waveguide (re-) alignment. A pair of 1/2″ ZnSe lenses was then used to focus the radiation emanating at the distal end of the waveguide chip onto a thermoelectrically cooled MCT detector (Vigo Photonics, Poland). The system was designed for portability, enabling on-site analysis with a dimension of 48 × 30 × 10 cm (L × W × H), as shown in Fig. 2.

C. Sample preparation, data collection, and data processing

A 10 mg/ml DON standard (Romer Labs, Tulln, Austria) solution in methanol was diluted to concentrations of 2.5, 0.5, 0.25, 0.1, 0.05, and 0.025 mg/ml in methanol. 10 μl of each solution was deposited onto the waveguide surface, ensuring complete surface coverage. The measurement was started once the solvent was evaporated. After each measurement, the waveguide was cleaned with isopropyl alcohol. Deposition of each standard solution was replicated thrice, and each replicate was measured in triplicate to address the measurement and pipetting repeatability and the analytical precision of deposition on the sensing system.

The software controlling the laser also collects the voltage signal from the MCT detector via a portable digital oscilloscope (PicoScope Series 5000, Pico Technology, U.K.). Since the tICL is operated in CW mode and the oscilloscope requires modulated light for periodic signal detection and noise reduction, an optical chopper system (Thorlabs, U.S.A.) operating at a frequency of 150 Hz was used. The average peak-to-peak amplitudes (V) from each emitted

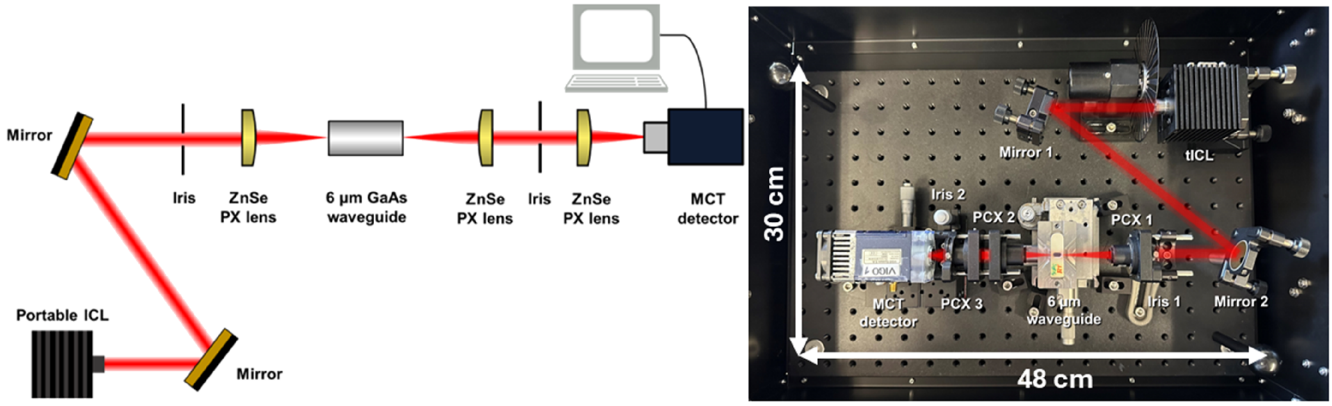

FIG. 2. Schematic (left) and photograph (right) of the tunable interband cascade laser (tICL)-based sensor system.

FIG. 3. Analytical workflow, including sample application, measurement, and data processing workflow for the comparative evaluation of the IR devices.

wavelength are calculated automatically within an interval of 140 ms. Absorbance spectra were then calculated via the Beer–Lambert law for each wavelength following A = −log(I/I$_0$), with I corresponding to the light intensity after sample interaction and I_0 the background light intensity. The analytical performance of the system was determined via establishing a calibration function and analyzing the relationship between the areas under the curve (AUC) for the characteristic DON peak vs the concentration. The performance of the tICL-based system was compared to a conventional single-bounce ATR assembly using a diamond crystal and a thermal detector (DLaTGS) combined with a FTIR spectrometer (Bruker Tensor II system, Bruker Optics GmbH, Germany). A common analytical workflow was used, ensuring comparability of the results obtained at both devices, as summarized in Fig. 3.

III. RESULTS AND DISCUSSION

A. Noise level evaluation and DON measurements

For determining the noise floor of the developed sensing system, triplicate measurements of air vs air (i.e., as a sample and as a background) were executed and averaged. The root mean square (rms) noise of the tICL system (1702–1640 cm^{-1}) was 0.013 61 units vs 5.77 × 10^{-5} units for the FTIR device within the same spectral window. It should be noted that the noise floor of the tICL-based system results from a single-scan and six characteristic spectral bands, while the noise of the FTIR spectrometer derives from averaging 512 scans with a spectral resolution of 4 cm^{-1}. The time per measurement of the t-ICL-based spectrometer and FTIR spectrometer was ~3.5 and ~4 min, respectively.

Despite the higher rms noise, the tICL-based measurements revealed comparable SNR to the conventional FTIR spectrometer with absorbance values ~1000 times higher. As anticipated, using thin-film waveguide technology provides an increase in evanescent field intensity for a tailored wavelength regime and an enhanced light-sample interaction leading to a comparable SNR.

Exemplary DON spectra from the evaporated solutions are shown in Fig. 4 illustrating the relationship between solution concentration and absorbance of the carbonyl group of DON. Spectra of standard solutions at concentrations of 0.05 mg/ml or lower were not discernible from the noise threshold.

In order to determine the analytical performance of the tICL-based device, calibration functions were established. They were derived from the evanescent field absorption spectra providing analytical figures-of-merit, i.e., concentration range (mg/ml), coefficient of correlation (R), coefficient of determination (R^2), limit of detection (LOD), limit of quantification (LOQ), and relative standard deviation (RSD). The LOD and LOQ were calculated in the following equations:

$$LOD = 3.3\frac{SD\ of\ intercept}{slope}, \tag{1}$$

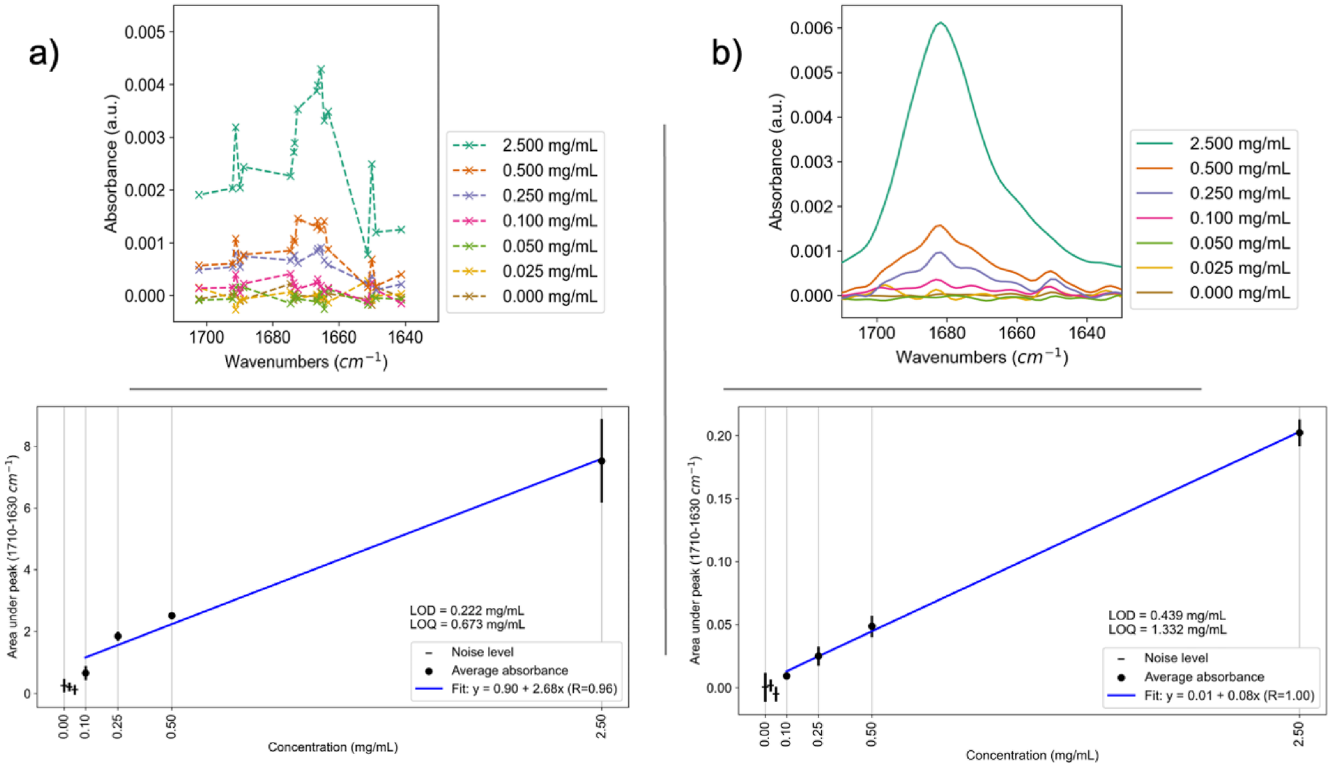

FIG. 4. IR spectra of DON residue after evaporation of methanol and derived calibration functions (0–2.5 mg/ml) for (a) tICL-based system and (b) FTIR spectrometer.

$$LOQ = 10\frac{SD\ of\ intercept}{slope}. \tag{2}$$

B. Performance comparison of tICL-based portable system and FTIR spectrometer

The analytical performance metrics for both systems are summarized in Table I. The range of DON concentrations was 2.5–0 mg/ml, with the lowest detectable concentration of 0.1 mg/ml for both systems. Therefore, the calibration function range was limited to DON concentrations in the bracket of 0.1–2.5 mg/ml, ensuring the accuracy and reliability of the established calibration models.

The tICL-based system displayed superior sensitivity with a LOD of 0.222 mg/ml and a LOQ of 0.673 mg/ml vs the FTIR system, revealing a LOD of 0.439 mg/ml and a LOQ of 1.332 mg/ml. In terms of linearity of the regression, the FTIR spectrometer displayed a slightly higher R^2 of 1 compared to the R^2 of 0.92 obtained with the tICL-based system. As anticipated, the thin-film waveguide provides enhanced sensitivity vs a single-bounce ATR assembly demonstrating a higher slope (2.68 vs 0.08), which results from the increased effective thickness due to the homogeneous evanescent field active along the entire waveguide surface providing an extended interaction pathlength between analyte molecules and the evanescent field.

The repeatability of the tICL-based measurements appears lower when compared at the lowest evaluated concentration of 0.1 mg/ml with an RSD of 26% in comparison with the FTIR device with an RSD of 19%; nevertheless, at higher concentrations of 0.5 mg/ml, the RSD of the tICL-based systems appears two-times more reproducible (i.e., RSD of 7.8%) vs the conventional device (RSD of 19%). It should be noted again that the figures-of-merit underline the enhanced sensitivity of the tICL-based device, proving single-scan spectra vs averaging 512 scans for the FTIR measurement, which also results in extended measurement times. Clearly, miniaturization of FTIR spectrometers is possible, yet it will compromise the device performance when reducing the dimensions of the interferometer. In turn, the system figures-of-merit for tICL-based technologies are not affected by miniaturization strategies.

The obtained analytical performance metrics demonstrate the suitability of the developed compact IR sensing system for the quantification of deoxynivalenol in solution. According to the European Food Safety Authority (EFSA) for monitoring DON in food and feed,[18] the highest levels of DON in 975 samples of unprocessed grains of undefined end-use were found in maize and wheat, with an average concentration exceeding 300 μg/kg and with the 95th percentile revealing levels above 1500 μg/kg. The EU compliance regulatory limit of DON encompasses a maximum level of DON for unprocessed grains at 1000 μg/kg.[19] Considering the regulatory limit and the official methods of analysis of DON from cereals following the AOAC, the LOD required to detect DON at regulatory levels (1000 μg/kg) would be 8.3×10^{-4} mg/ml, being ≈2.25 orders of magnitude lower than the LOD of the tICL-based spectrometer. Several strategies on improving the sensitivity of the presented tICL-based device can be further explored (e.g., using polarimetric balanced detection)[20] and optimized for DON detection in regulatory compliance scenarios. Additional well-established approaches for routine mycotoxin analysis using conventional methods (e.g., HPLC and LC-MS) require preconcentration steps, such as solid-phase extraction (SPE) and immunoaffinity column-based preconcentration and cleanup. Beyond these sample enrichment techniques, the present sensing device enables alternative viable enrichment strategies based on thin-film waveguides, such as surface enhanced IR absorption (SEIRA),[21–23] which can enhance the sensitivity by 2–3 orders of magnitude (100–1000 times). These strategies include, but are not limited to, graphene-enhanced infrared spectroscopy,[24,25] microfluidic-enhanced IR spectroscopy,[26] and molecular imprinted polymers (MIPs).[27–29] The latter strategy allows selectively extracting and preconcentrating mycotoxins in-line or directly at the IR waveguide surface, thereby also reducing the complexity of the sampled matrix. These signal enhancement strategies will effectively allow tICL-based portable sensing systems to serve as a rapid in-field screening solution for on-site detection of mycotoxins in food supply scenarios at the mandated threshold levels.

TABLE I. Analytical figures-of-merit comparison of a FTIR spectrometer with single-bounce ATR assembly vs the developed tICL-based sensing system using a thin-film waveguide for the example of DON.

	tICL-based sensor system	FTIR spectrometer
	Concentration range: 2.5–0.1 mg/ml	
Number of scans	1	512
Time per measurement, min	3.5	4
Equation of the calibration function	y = 0.90 + 2.68x	y = 0.01 + 0.08x
RMS_{noise}	0.013 61	5.77×10^{-5}
R	0.96	1
R^2	0.92	1
LOD, mg/ml	0.222	0.439
LOQ, mg/ml	0.673	1.332
RSD (0.1 mg/ml, N = 3), %	26	19
RSD (0.5 mg/ml, N = 3), %	7.8	19

IV. CONCLUSIONS

In this study, a portable mid-infrared sensing device based on tunable interband cascade lasers and thin-film waveguides tailored for potential applications in food contaminant monitoring was developed and specifically adapted for the determination of deoxynivalenol, the most prevalent mycotoxin in moderate climatic regions of the world. It should be noted that the IR emission window offered by the tICL covers spectral features of a broad range of alternative analytes of interest, confirming the flexible utility of the device within this spectral range. The level of integration of the developed optical setup comprising an on-chip sensing element and a thermoelectrically cooled detector offers significantly improved portability vs conventional FTIR technology and benchtop QCL-based spectrometers. Additionally, the sensing system has the potential for further miniaturization by integrating the tICL, waveguide, and detector into the same integrated circuit chip, opening new possibilities for handheld miniaturization. The comparison of the tICL-based system vs a conventional FTIR spectrometer confirmed that similar analytical figures-of-merit can be achieved with comparable SNR despite the significant difference in the number of spectral scans. In fact, the anticipated sensitivity enhancement of the laser-based system indeed yielded lower limits of detection and quantification while maintaining a compact footprint that may even be further reduced via using a more integrated optical layout. Future strategies for signal enhancement via enrichment and optical amplification schemes will further advance the utility of miniaturized tICL-based systems as highly sensitive analytical solutions for on-site analysis of food contaminants at regulatory limits during storage, transportation, and processing, facilitating informed decision-making regarding potential health risks and economic loss.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

All authors contributed to this work.

A. Femenias: Conceptualization (equal); Data curation (equal); Formal analysis (equal); Investigation (equal); Methodology (equal); Software (equal); Supervision (equal); Validation (equal); Visualization (equal); Writing – original draft (equal); Writing – review & editing (equal). **M. Erdem**: Conceptualization (equal); Data curation (equal); Formal analysis (equal); Validation (equal); Visualization (equal); Writing – review & editing (equal). **P. Fomina**: Conceptualization (equal); Data curation (equal); Formal analysis (equal); Investigation (equal); Methodology (equal); Validation (equal); Writing – review & editing (equal). **J. Scheuermann**: Conceptualization (equal); Data curation (equal); Methodology (equal); Resources (equal); Software (equal); Validation (equal); Writing – review & editing (equal). **U. Blazhko**: Data curation (equal); Methodology (equal); Software (equal); Validation (equal); Writing – review & editing (equal). **S. Freitag**: Conceptualization (equal); Data curation (equal); Investigation (equal); Methodology (equal); Writing – review & editing (equal). **A.J. Bosman**: Conceptualization (equal); Data curation (equal); Formal analysis (equal); Investigation (equal); Methodology (equal); Writing – review & editing (equal). **R. Weih**: Funding acquisition (equal); Investigation (equal); Project administration (equal); Software (equal); Visualization (equal); Writing – review & editing (equal). **J. Koeth**: Funding acquisition (equal); Project administration (equal); Resources (equal); Supervision (equal); Validation (equal); Writing – review & editing (equal). **A. Kohler**: Conceptualization (equal); Funding acquisition (equal); Investigation (equal); Project administration (equal); Resources (equal); Supervision (equal); Validation (equal); Writing – review & editing (equal). **R. Krska**: Funding acquisition (equal); Investigation (equal); Project administration (equal); Resources (equal); Supervision (equal); Validation (equal); Writing – review & editing (equal). **G. I. J. Salentijn**: Conceptualization (equal); Funding acquisition (equal); Investigation (equal); Project administration (equal); Resources (equal); Supervision (equal); Validation (equal); Writing – review & editing (equal). **F. S. Ruggeri**: Funding acquisition (equal); Investigation (equal); Project administration (equal); Resources (equal); Supervision (equal); Validation (equal); Writing – review & editing (equal). **B. Mizaikoff**: Conceptualization (equal); Funding acquisition (equal); Investigation (equal); Methodology (equal); Project administration (equal); Resources (equal); Supervision (equal); Validation (equal); Visualization (equal); Writing – review & editing (equal).

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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