# Photodetectors based on GaInAsSb/GaAlAsSb heterostructures for the practical tasks of precision diode laser spectroscopy

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The paper considers the uncooled photodetectors based on GaInAsSb/GaAlAsSb heterostructures, which can be applied in precision diode laser spectroscopy. The spectral sensitivity range of photodetectors with a photosensitive area diameter of 1.0 mm and 2.0 mm is 1.0−2.4 $\mu$m. The current monochromatic sensitivity at the wavelength of 2.1 $\mu$m has a value of 1.0 A/W without bias. The capacity reaches 375 pF with a photosensitive area diameter of 1.0 mm and 800−5000 pF with 2 mm. The modern gas analyzers based on diode lasers and developed photodetectors for medical screening diagnostics by analyzing the gas compositions of exhaled air, for control of impurity gases in the process of rectification of inorganic hydrides, control of methane leaks in gas pipelines, as well as for registration of exhaust gases of a moving car are presented.

**Keywords:** photodetector, heterostructure, diode laser spectroscopy, gas analyzer.

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## 1. Introduction

At present, the development of modern technology requires minimization of the size of analytical equipment and high accuracy of measurements. Diode laser spectroscopy (DLS) is one of the most dynamically developing areas of laser physics as applied to problems of analytical chemistry and gas analysis. Progress in the technology of diode lasers and photodetectors in the near and mid-IR ranges, as well as the development of methods for highly sensitive IR absorption spectrometry, have led to the creation of a new generation of gas analytical systems. Such systems are characterized by high sensitivity to the measured component, as well as unprecedented spectral resolution and operation speed.

For gas analytical measurements, both diode lasers with radiation output into a single-mode fiber, operating in the near-IR range [1], and quantum-cascade diode lasers, generating in the mid-IR range [2]. In particular, semiconductor lasers based on AlGaInAs/InP [3,4] and InGaAsP/InP MQW structures [5] have been developed for wavelengths of 1.3 and 1.55 $\mu$m. For the range of 1.25−1.65 $\mu$m, lasers based on group III nitrides and their GaInNAs, GaInNAsSb, AlGaN nitrides [6–9] were created.

Registration of laser radiation in the near- and mid-IR ranges is one of the most important problems in laser physics. Semiconductor photodetectors based on lead chalcogenides, the operation speed of the best of which is 3−5 $\mu$s [10], are of little use for operation with diode lasers, which require much higher fast operation speed.

In the near-IR region of the spectrum, photodetectors based on InGaAs/InP heterostructures are widely used, in the active region of which a direct-gap solid-state solution $In_{0.53}Ga_{0.47}As$ [11] is used. In the work [12] it was reported about the creation of high-speed InGaAs/InP photodetectors with bandwidth of up to 20 GHz with the sensitive area diameter of 18 $\mu$m. In turn, in the work [13] the authors showed that when the area of the sensitive area of multilayer InGaAs photodetectors is reduced to 24 $\mu m^2$, the bandwidth can reach > 100 GHz. However, for some applications, reducing the size of the sensitive area is undesirable, as this leads to losses in sensitivity.

At present, photodetectors based on InSb [14,15] are used for gas analysis in the IR range of the spectrum. The work [16] reported on the use of a series of uncooled InSb photodiodes for sensors of such gases as $CO_2$, CO and $NO_x$. However, many InSb photodiodes require cooling down to 80−200 K.

The photodetectors based on narrow-bandgap compounds $A^{III}B^{V}$ for the near- and mid-IR ranges of the spectrum have been created in the IR Optoelectronics Laboratory of the Ioffe Physical-Technical Institute. The Department of Diode Laser Spectroscopy of the Prokhorov Institute of General Physics of the Russian Academy of Sciences (IGP RAS) has developed devices based on diode lasers and photodetectors designed to solve analytical and diagnostic problems of ecology, medicine, process control, etc.

This work is devoted to uncooled photodetectors based on GaSb/GaInAsSb/GaAlAsSb heterostructures and their practical application in diode laser spectroscopy for gas

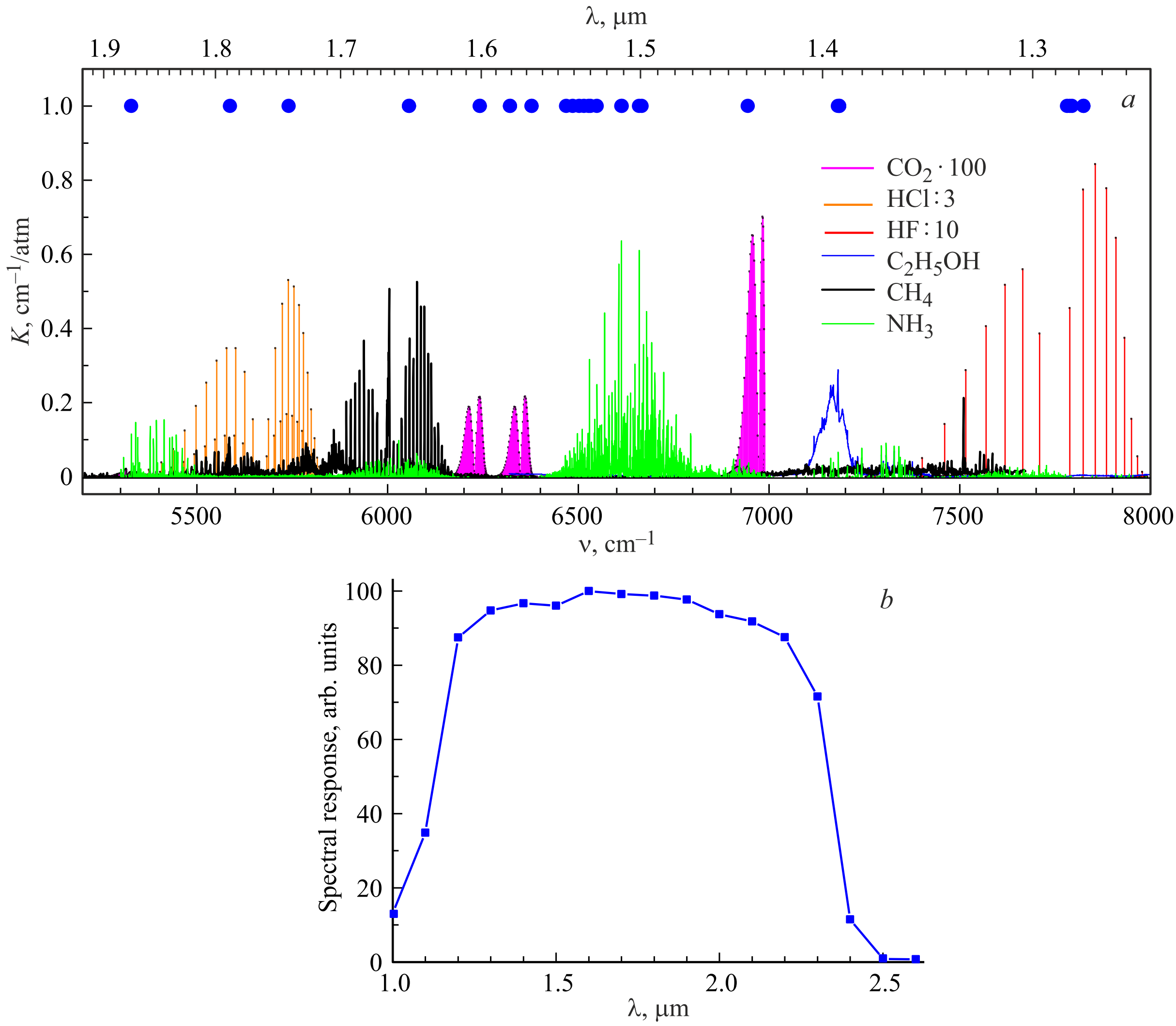


**Figure 1.** Spectral characteristics in the near IR range: *a* — absorption bands of light molecules (blue marks — spectral regions for which diode lasers are commercially produced); *b* — photoconductive response of GaSb/GaInAsSb/GaAlAsSb receiver at room temperature. (Colored version of the figure is presented in electronic version of the article).

analysis problems in medicine, ecology, electronic and optoelectronic industries.

## 2. Photodetectors based on GaInAsSb/GaAlAsSb heterostructures for the near- and mid-IR ranges

It is known that overtones located in the spectral range of $1-2\,\mu$m, where many molecules of light gases have absorption bands (Fig. 1, *a*), are characteristic of C–H, O–H, N–H bonds, etc. Photodiodes based on the GaSb/GaInAsSb/GaAlAsSb heterostructure have the spectral sensitivity in the range of $1.0-2.4\,\mu$m, which makes it possible to detect the indicated absorption bands. The maximum sensitivity ($> 90\%$) is observed for radiation wavelengths of $1.45-2.2\,\mu$m (Fig. 1, *b*).

In the work [17] we have demonstrated that GaSb/GaInAsSb/GaAlAsSb photodiodes have sufficiently fast operation speed to detect, without cryogenic cooling, radiation from quantum-well WGM-lasers with a fundamental wavelength modes in the range of $2.2-2.3\,\mu$m. However, the measurement of absorption spectra of light molecules in various physical processes by DLS methods requires the use of spectrally matched photodetectors with parameters (sensitivity, operation speed, etc.) determined for each specific task. For example, photodetectors with smaller area of the sensitive area have the lower level of reverse dark currents and, accordingly, noise, as well as a lower capacitance (higher speed of operation) than devices with the larger area. On the other hand, decrease in the area of the sensitive area leads to decrease in the spectral sensitivity and adjustment difficulties.

**Table 1.** Parameters of GaSb/GaInAsSb/GaAlAsSb photodiodes

| Parameter, units of measure | Parameter value | |
|---|---|---|
| Photosensitive area diameter, mm | 1.0 | 2.0 |
| Surface area of the photosensitive area, $mm^2$ | 0.79 | 3.14 |
| Spectral sensitivity range (> 10%), $\mu$m | 1.0−2.4 | |
| Maximum spectral sensitivity range (> 90%), $\mu$m | 1.45−2.2 | |
| Current monochromatic sensitivity at $\lambda = 2.1\,\mu$m, $U_{rev} = 0$ V, A/W | 1.0 | |
| Differential resistance, kOhm | 1.05 | 0.4−1.5 |
| Dark current at $U_{rev} = 0.2$ V, $\mu$A | 75 | 50−200 |
| Capacitance at $U_{rev} = 0$ V, pF | 375 | 800−5000 |
| Rise and fall time of the photocurrent pulse by level 0.1−0.9 (load 50 Ohm, $U_{rev} = 0$ V), ns | 30−45 | 100−770 |

**Figure 2.** Photodiode array of 5 elements in standard package TO-8.

Table 1 shows the values of the main parameters of GaSb/GaInAsSb/GaAlAsSb photodiodes with a photosensitive area diameter of 1 and 2 mm. The operation speed of the developed devices is 30−45 ns with diameter of 1 mm and 100−770 ns with diameter of 2 mm, which, as will be shown below, is sufficient value for operation as part of the DL gas analyzers.

To stabilize the temperature, the thermoelectric module in the TO-8 package was used.

We also created the photodiode array consisting of five elements with diameter of 1.0 mm (see Fig. 2).

## 3. Principles of detection of light gaseous impurities by DLS methods

Absorption spectroscopy and gas analysis using diode lasers are based on the principles of molecular absorption spectroscopy, the essence of which is to measure the absorption of IR radiation at the vibrational-rotational transition of molecule according to the Bouguer−Lambert law. For these purposes, diode laser radiation is scanned in a strictly selected wavelength range containing the transition frequency of the molecule, which makes it possible to ensure high selectivity for the measurement of the selected molecular component.

The block diagram of a gas analyzer based on diode laser and photodetectors for measuring gaseous impurities in the near-IR range is shown in Fig. 3.

The signal recorded by the photodetector (Fig. 3, block *4*) after the passage of laser radiation through the gas under study can be represented in the form

$$I = I_0 T(\nu) = I_0 \exp[-\sigma(\nu) P_0 C L] = I_0 \exp[-K(\nu)], \quad (1)$$

where $I_0$ is laser radiation intensity, $T(\nu)$, $K(\nu)$ are transmission and absorption spectra respectively, $\sigma(\nu)$ is absorption cross section, $P_0$ is pressure of the studied gas mixture, $C$ is concentration of the detected molecule, $L$ is optical path length in the cell with the studied gas.

The diode laser was excited by periodic trapezoidal current pulses. The duration of the pumping current pulse was 1−2 ms, the repetition frequency was 40 Hz. The power emitted by the laser, as well as the signal recorded by the photodetector, had a shape close to trapezoidal. The duration of the pumping current pulse was 1−2 ms, the repetition frequency was 40 Hz. Change in the pumping

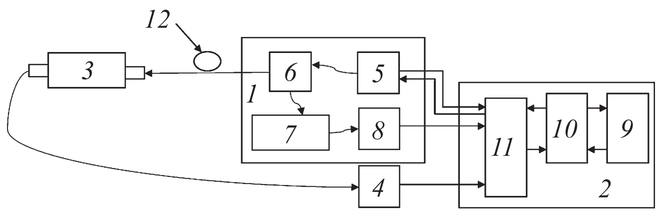


**Figure 3.** Block diagram of a gas analyzer based on diode laser and a near-IR photodiode for measuring gas impurities: *1* — laser radiation unit, *2* — control unit, data reception and processing, *3* — analytical cell with fiber input, *4* — analytical signal photodetector, *5* — diode laser module, *6* — fiber splitter, *7* — reference cuvette with Fabry−Perot interferometer, *8* — reference signal photodetector, *9* — programmable digital module, *10* — DAC and ADC modules, *11* — analog signal converter module, *12* — fiber optic cable.

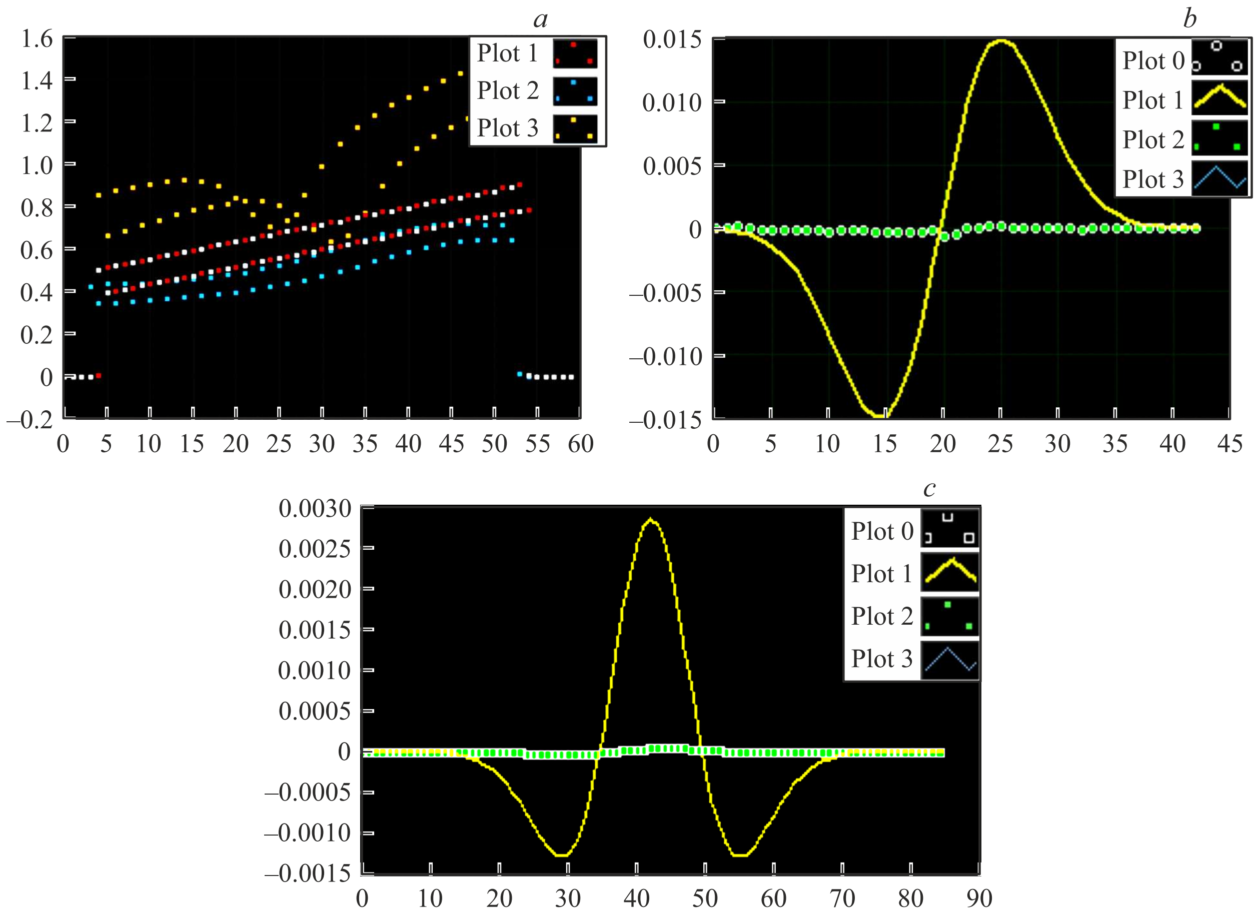


**Figure 4.** Diode laser pulse signals recorded by photodetectors in the reference (upper dependence) and analytical channels (*a*); logarithms of the frequency-shifted signal components, *a* (*b*); autocorrelation and cross-correlation convolution of signals *b* (*c*). (Colored version of the figure is presented in electronic version of the article).

current during the pulse is accompanied by a change in the radiation frequency.

The pumping current pulse of the diode laser is modulated in amplitude with a frequency equal to the frequency of the DAC/ADC converter (Fig. 3, block *10*). The frequency is optimized for the bandwidth of the preamplifiers of the photodetectors of the analytical and reference channels (Fig. 3, block *4*, block *8*) and is 111.111 kHz. The modulation depth is selected in such a way as to provide a frequency shift in the generation mode by the half-width of the absorption lines of the studied gas and to obtain the maximum absorption contrast.

Figure 4 shows the diode laser signals visualized on the laptop screen.

To determine the concentration of the studied gas, mathematical processing of amplitude-modulated signals is carried out (Fig. 4):

1 — Registration of transmission signals in the reference and analytical channels (Fig. 4, *a*). The signals are shifted by the amount of modulation depth.

2 — Logarithmation of the frequency-shifted transmission signal components in the reference and analytical channels in accordance with the Bouguer–Lambert–Burr law and obtaining the absorption coefficients (Fig. 4, *b*). It should be noted that the shape of the signals is similar to the first derivative of the transmission signal.

3 — Autocorrelation $R_{xx}$ and cross-correlation convolution of the absorption of the reference signal, cross-correlation convolution $R_{xy}$ of the reference signal with the analytical one. This procedure provides additional filtering of the useful signal, which is noisy (Fig. 4, *c*).

4 — Linear regression of $R_{xy}$ on $R_{xx}$ and finding the coefficient of coincidence for functions $\alpha$, which is defined as $\alpha = R_{xy}/R_{xx}$.

The concentration of the studied gas $C$ (in ppm, one part per million) is proportional to the value $\alpha$ and is calculated by the formula

$$C = \frac{\alpha \cdot P_R \cdot L_R}{P_A \cdot L_A} \cdot 10^6, \qquad (2)$$

where $\alpha$ is coefficient of coincidence of functions $R_{xy}$ and $R_{xx}$, $P_R$, $P_A$ are gas partial pressure in the comparison channel (reference) and in the analytical channel, respectively, $L_R$, $L_A$ are optical lengths in the cell of the reference channel and analytical cell, respectively.

The presented procedure for calculating the concentration of the studied gas significantly limits various low-frequency (vibration, flicker) noises of the receiving-recording path of the gas analyzer. It is linear to the measurement of gas concentrations at low absorption values (background measurements).

## 4. Gas analysis by DLS

The main advantage of DLS in the gas analysis of molecular subjects is the high measurement accuracy combined with high operation speed. DL gas analyzers with fiber output are compact, self-powered and can be integrated into any gas analysis system (distillation columns, centrifuges for isotope selection, etc.) and installed on mobile platforms (car, helicopter, unmanned aerial platforms, etc.).

DL gas analyzers based on GaSb/GaInAsSb/GaAlAsSb photodiodes, developed in the IR Optoelectronics Laboratory of the Ioffe Physical-Technical Institute, were created in the DLS Department of the IGP RAS.

### 4.1. Screening DLS diagnosis of diseases by exhaled air

Early diagnosis of various diseases makes it possible to avoid the development of irreversible changes in the human body, complications, decrease in the quality of life, and in some cases save his/her life. At the present time, special attention is paid to screening diagnostics. Screening assay in medicine is understood as a set of measures aimed at identifying diseases in a large group of patients in the absence of marked symptoms. The main requirements for a screening test are simplicity, non-invasiveness and safety of testing procedures, as well as high speed of data processing and the ability to detect diseases at an early stage.

Multichannel analyzer for non-invasive screening and biomedical research was developed on the basis of near-IR diode lasers manufactured by „NTT Electronics" (Japan) and GaSb/GaInAsSb/GaAlAsSb photodiodes described above. The instrument allows you to measure such biomarkers of exhaled air as carbon dioxide isotopometric molecules $^{12}CO_2$ and $^{13}CO_2$, molecules of methane $CH_4$, ammonia $NH_3$, water $H_2O$ and hydrogen sulfide $H_2S$. The concentration of molecules was measured in a multi-pass cell with a base length of 40 cm and a volume of 1.8 L, built according to the Herriot scheme. The total length of the optical path in such a cell reached 26 m. Three fiber-coupled diode lasers were used in this work. $CH_4$ detection was carried out at wavelength of 1.665 $\mu$m, measurements of $^{12}CO_2$, $^{13}CO_2$ and $H_2S$ were carried out near 1.602 $\mu$m, and $NH_3$ and $H_2O$ — near 1.512 $\mu$m. All measurements were taken in real time.

Clinical trials of the diode laser spectrometer were carried out at the Moscow City Clinical Hospital after V.M. Buyanov (CCH № 12, Moscow) [18]. A total of more than one hundred patients were examined. The study was conducted in accordance with the Declaration of Helsinki (2013) and approved by the Ethics Committee of the Moscow City Clinical Hospital after V.M. Buyanov of the Moscow Department of Health and the Ethics Committee of the Central Clinical Hospital of the Russian Academy of Sciences. Informed voluntary consent was obtained from all participants involved in the study.

**Table 2.** Specifications of the multichannel DL gas analyzer

| Parameter, units of measure | Parameter value |
|---|---|
| Registration wavelength, nm: | |
| $CH_4$ | 1625 |
| $NH_3$, $H_2O$ | 1512 |
| $^{12}CO_2$, $^{13}CO_2$, $H_2S$ | 1602 |
| Detection limit, ppm: | |
| $CH_4$ | 0.1 |
| $^{12}CO_2$ | 20 |
| $^{13}CO_2$ | 20 |
| $H_2S$ | 0.4 |
| $NH_3$ | 0.03 |
| $H_2O$ | 100 |
| Operating mode setting time after switching on, min | 10 |
| Single sample measurement time, s | 30 |
| Operating temperature, °C | from +10 to +45 |

The tests included the measurement and study of biomarkers $^{12}CO_2$, $^{13}CO_2$, $CH_4$, $NH_3$, $H_2O$ and $H_2S$ in the exhaled air of patients with various diseases. The conducted studies have shown the effectiveness of the developed DL spectrum analyzer for analyzing the components of exhaled air in order to identify functional disorders in various diseases of the digestive system, cardiorespiratory system, diseases caused by a violation of the nitrogen-separating function of the kidneys, etc.

GaSb/GaInAsSb/GaAlAsSb photodiodes with a sensitive area diameter of 1 mm were used to detect diode laser radiation in the analytical and reference channels (see Table 1). The detectability of such photodiodes was $D^* = 1 \cdot 10^{11}\,W^{-1} \cdot cm \cdot Hz^{1/2}$.

The technical characteristics of the developed multichannel DL spectrum analyzer for non-invasive screening and biomedical research are given in Table 2.

### 4.2. Control of impurities in the process of obtaining high-purity hydrides

High purity hydrides $NH_3$, $PH_3$, $AsH_3$, $SiH_4$, $GeH_4$ are widely used in the electronic and optoelectronic industries. The concentration of impurities in the final product of hydrides should not exceed tens of ppb (one part in a billion). Real-time control of impurities in the process of distillation purification of hydrides is a task that gas analysis technology based on diode lasers successfully copes with. At present, for FSUE ≪NPP „Salyut"≫ (Nizhny

**Table 3.** Impurities detected using DL gas analyzers in the process of distillation purification of inorganic hydrides

| Impurity | Wavelength of registration, nm | Hydrides | Sensitivity, mol% |
|---|---|---|---|
| $H_2O$ | 1.391 | $NH_3$, $PH_3$ | $1 \cdot 10^{-5}$ |
| $NH_3$ | 1.512 | $AsH_3$ | $5 \cdot 10^{-5}$ |
| $C_2H_2$ | 1.635 | $SiH_4$ | $2 \cdot 10^{-4}$ |
| $CH_4$ | 1.651 | $SiH_4$ | $4 \cdot 10^{-5}$ |
| $CO_2$ | 2004 | $PH_3$, $SiH_4$, $AsH_3$ | $1 \cdot 10^{-4}$ |
| $H_2S$ | 1.601 | $AsH_3$ | $1 \cdot 10^{-3}$ |
| $C_2H_2$ | 1.531 | $PH_3$, $SiH_4$ | $1 \cdot 10^{-4}$ |

Novgorod) the line of instruments based on a fiber-coupled diode laser for continuous monitoring of impurities $H_2O$, $CO_2$, $CH_4$, $NH_3$, etc. in the process of distillation of high-purity hydrides, has been created, tested and is in operation. Gas analyzers are integrated directly into distillation columns, which allows for continuous monitoring of impurities during the purification of hydrides [19]. The cleaning time depends on the physicochemical properties of the hydrides and is > 10 h. The control of impurities in the „on-line" mode by DLS methods makes it possible to optimize the hydride purification time. Table 3 contains the list of detecting impurities by DLS methods. GaInAsSb/GaAlAsSb photodiodes with a sensitive area diameter of 2 mm were used as the device receiving radiation (see Table 1).

### 4.3. Methane concentration monitoring

The monitoring of methane concentration $CH_4$ in industrial hazardous areas is the extremely important task in the coal, oil and gas industry. The minimum explosive concentration of $CH_4$ in air is no more than 5%. Gas analyzers that measure the pre-explosive level of methane should not have electrical contacts, while operating at remote distances and have high sensitivity over the wide temperature range (from −40°C to +50°C) at high humidity and dust content. Together with the enterprises of the group of companies JSC ≪AK „Transneft"≫ the gas analyzer [20] was developed, designed to detect methane by absorption spectroscopy using the diode laser in the near-IR range and a remote sensor — single-pass optical cell 50 mm long with the fiber input and radiation output, with a distance of > 50 km. The main element of the sensor is optical cells with fiber input and output, which are placed in a dust- and moisture-proof housing for operation in real climatic conditions.

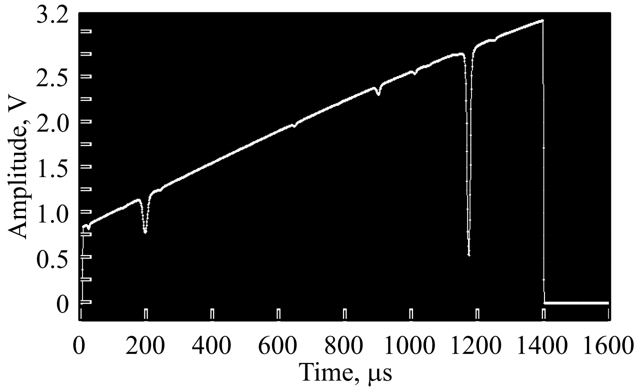


**Figure 5.** Absorption spectrum of methane recorded at wavelength of 1.65 $\mu$m.

Each instrument uses two GaSb/GaInAsSb/GaAlAsSb photodetectors with a sensitive area diameter of 2 mm (see Table 1). Figure 5 shows the absorption spectrum of methane recorded with the gas analyzer at a wavelength of 1.65 $\mu$m.

The diode laser pulse duration was 4.5 ms; the frequency tuning length was 2.3 $cm^{-1}$. Receivers with the sensitive area of a relatively large size (2 mm) made it possible to register a weak signal from the optical path.

**Table 4.** Parameters of the remote sensor for recording the exhaust gases of a moving vehicle

| Parameter name, units of measure | Parameter value |
|---|---|
| Dimensions of receiving-transmitting unit, mm | 400 × 500 × 400 |
| Mass of receiving-transmitting unit, kg | 14.8 |
| Dimensions of hardware-software block, mm | 400 × 300 × 500 |
| Weight of hardware-software blocks, kg | 9.0 |
| Voltage and frequency of the mains, V, Hz | 230, 60 |
| Consumed power, W | 146 |
| Operating mode setting time after turning on the power, min | 10 |
| Temperature, °C | from +10 to +45 |
| Analyzed medium | Moving vehicle exhaust |
| Object speed, km/h | Not more than 60 |
| Sensitivity to exhaust gas components, vol% | |
| $CH_4$ (1651 nm) | 0.002 |
| CO (1578 nm) | 0.5 |
| $CO_2$ (2004 nm) | 0.02 |
| NO (5260 nm) | 0.01 |

### 4.4. Registration of exhaust gases of a moving vehicle

It is known that many modern devices for measuring the concentration of gaseous substances do not involve working with moving sources. One of the latest joint developments of the DLS Department of the IGP RAS and the IR Optoelectronics Laboratory of the Ioffe Physical-Technical Institute is the remote sensor, the main advantage of which is the ability to detect exhaust gases $CH_4$, $CO_2$, CO and NO of a moving car [21].

The sensor consists of three main parts: transceiver optical system, electronic control and registration unit, and reflection unit. Transmitter-receiver system and the reflection unit must be at the level of the vehicle exhaust pipe. The device is based on a diode laser, GaSb/GaInAsSb/GaAlAsSb photodiodes with a sensitive area diameter of 2 mm, and a 5-element photodiode array (see Fig. 2). The use of such array made it possible to effectively receive laser radiation scanned over space and thereby increase the coverage area during „on-line“ registration of exhaust gases from cars moving at a speed of no more than 60 km/h. The sensor parameters are given in Table 4.

## 5. Conclusion

The work presents the parameters of GaSb/GaInAsSb/GaAlAsSb photodetectors developed at the Ioffe Physical-Technical Institute and presents promising developments of the Prokhorov Institute of General Physics of RAS, based on data from photodetectors and diode lasers. The created gas analyzers are used in medical screening diagnostics, in systems for monitoring methane leaks in gas pipelines and impurity gases during rectification purification of inorganic hydrides, as well as for remote registration of exhaust gases from a moving vehicle.

In the future, the development of new technologies for creating lasers with pico- and femtosecond generation pulses duration in the near- and mid-IR ranges will expand the area of analytical applications of DLS. Such systems will make it possible to study the dynamic pattern of fast processes, such as the internal motion of molecules, elementary stages of chemical reactions, relaxation of photoexcited electrons in semiconductors, primary stages of light conversion in photosynthetic and visual pigments, etc. Thus, fast-working photodetectors for operation in the spectral window $1-3\,\mu$m are becoming more and more in demand.

### Conflict of interest

The authors declare that they have no conflict of interest.

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