EXPLOSIVES VAPORS–CONCENTRATING AND OPTOELECTRONIC DETECTION

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Abstract

Detection of explosives vapors is an extremely difficult task. The sensitivity of currently constructed detectors is often insufficient. The paper presents a description of an explosive vapors concentrator that improves the detection limit of some explosives detectors. These detectors have been developed at the Institute of Optoelectronics. The concentrator is especially dedicated to operate with nitrogen oxide detectors. Preliminary measurements show that using the concentrator, the recorded amount of nitrogen dioxide released from a 0.5 ng sample of TNT increases by a factor of approx. 20. In the concentrator an induction heater is applied. Thanks to this and because of the miniaturization of the container with an adsorbing material (approx. 1 cm^3), an extremely high rate of temperature growth is achieved (up to 500 °C within approx. 25 s). The concentration process is controlled by a microcontroller. Compact construction and battery power supply provide a possibility of using the concentrator as a portable device.

Keywords: explosives concentrator, explosive detection, optoelectronic sensors, CEAS sensors.

1. Introduction

It is observed that over the last few years the terrorism threat has increased. Therefore, the demand for reliable and rapid detection methods of explosives is very important. A lot of works focused on improving the current technologies and on developing new ones have been undertaken, e.g. employing X and γ radiation, gas chromatography, infrared and THz technology, etc. [1, 2].

The main factor determining the suitability of the detection system is the ability to detect the very low level of explosives vapors concentrations (ppt[1] level) in relatively short time. The requirements for the detection system sensitivity are determined by the values of explosives vapor pressure which are listed in Table 1.

Generally, explosives are organic compounds built of carbon (C), hydrogen (H), oxygen (O) and nitrogen (N). Most of them contain the so-called nitro groups (NO_2) [3]. Substances like trinitrotoluene (TNT), dinitrotoluene (DNT), nitroglycerine (NG) or ethylene glycol dinitrate (EGDN) are characterized by relatively high value of their vapor pressure, so they can be detected even at ambient temperature. The situation becomes more complicated in the case of a substance

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[1]Parts per trillion

with the vapor pressure below the detection limit of currently available detectors (e.g.: pentaerythritoltetranitrate – PETN or 1,3,5-trinitro-1,3,5-triazacyklohexane – RDX).

Table 1. The properties of some explosives [3].

Explosives	Chemical name	Molecular formula	Molecular weight [g/mol]	Density [g/cm^3]	Vapor pressure [ppb] @ 25 °C
TNT	2,4,6-trinitrotoluene	$C_7H_5N_3O_6$	227.13	1.65	9
RDX	1,3,5-trinitro-1,3,5-triazacyklohexane	$C_3H_6N_6O_6$	222.26	1.82	0.006
HMX	1,3,5,7-tetranitro-1,3,5,7-tetrazacyclooctane	$C_4H_8N_8O_8$	296.15	1.96	0.0001
PETN	pentaerythritoltetranitrate	$C_5H_8N_4O_{12}$	316.14	1.76	0.0005
EGDN	ethylene glycol dinitrate	$C_2H_4N_2O_6$	152.06	1.48	60 000
DMDNB	2,3-dimethyl-2,3-dinitrobutane	$C_6H_{12}N_2O_4$	176.17	1.34	27 000
p-NT	p-nitrotoluene	$C_7H_7NO_2$	137.14	1.39	>50 000

To enhance the sensitivity of the detection system, a special concentration system can be used (Fig. 1). The concentration system allows collection of explosives vapors present in a high volume of air into a low volume of an adsorbing material (adsorbent).
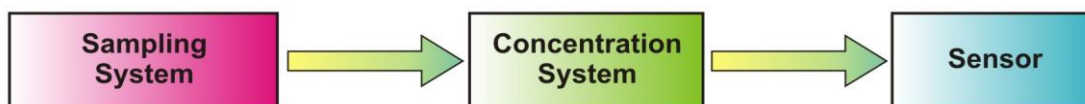

Fig. 1. Block diagram of the explosive vapors detection system [4].

The main task of the sampling system is to deliver the tested air containing explosives vapor into the concentration system. It consists of an explosive vapor concentrator and a heater driver. Explosive particles flowing through the concentrator are adsorbed at room temperature. After the collection procedure, the adsorbing material is heated and a desorption process is performed. In this way explosives vapors are released and sent to the sensor. The concentrator should provide high performance of explosives collection and desorption. Therefore the most important parameter is the concentration factor. The factor is equal to the ratio of substance concentrations in the concentrate and the initial solution [5]. Its value is affected by several factors, such as adsorption material type, desorption temperature and the shape and size of the collection area.

There are various types of concentrators, e.g.:

• metal fibres covered with an adsorbing material [6],

- cylindrical tube coated with thin layer of a resistively heated material [6],
- micro-electro-mechanical structures (MEMS) [7],
- perforated polyimide hotplates [8-12],
- 3D planar concentrator [13, 14],
- chemical devices [15].

Some concentrators are able to operate in a cascade configuration. Such a setup consists of a first trap with a high volume followed by a second trap with a lower volume. Thanks to this, a significant improvement in selectivity and sensitivity can be obtained [15].

So far, explosive vapors concentrators are not commercially available. Most of them operate as a laboratory or prototype device [16]. Some examples of such concentrators are listed in Table 2.

Table 2. Examples of concentrator arrangements.

Concentrator design	Adsorbing material	Temp. & desorption time	Dimensions	Comments
Metal filter	No data	200 °C	No data	Used in portable device IMS sensor [17]
Stacked filters coated with an adsorbent	Polymer (Tenax or PDMS), tytanium, carbon nanotubes	No data	No data	Two-stage concentrator to improve sensitivity and selectivity [15]
Cylindrical metal tube	Silicone rubber (dimethyl and methylvinylsiloxane copolymers), thickness less than 1mm	No data	No data	Heating element made of a thin layer of resistive material [6]
Planar microstructure, Si substrate, Si_2N_3 membrane	Sol-gel layer	200 °C in 4 ms	2.2 mm × 2.2 mm	Integrated in the µChemLab system, platinum heater, power consumption100 mW [18]
Planar microstructure, polyimide membrane	Polymer	120 °C in less than 120 ms	9.5 mm ×8.5 mm	Tested in the Vapor Tracer II system [8]
Planar microstructure, SiO_2membrane	Polymer	180 °C in 40 ms	4 mm ×4 mm	Tested in Vapor Tracer II system [12, 19]
Planar microstructure, Si substrate, Si_2N_3 membrane	carbon nanotubes, sol-gel layer	200 °C	No data	3D concentrator, with flow perpendicular (power consumption 150mW but higher concentration efficiency& parallel to the substrate surface (power consumption 0.6 W) [13, 14]
Tube with a SiO_2 inner layer	No data	No data	Measuring head of [20×10]cm	System using gas chromatography, NO detector [20]

2. Application of NO$_x$ optoelectronic sensor to explosives detection

Preliminary studies with a designed nitrogen dioxide (NO$_2$) sensor showed that it is possible to detect such explosives as nitrocellulose, nitroglycerin or TNT [21]. However, the sensor sensitivity was insufficient for other explosives (more stable and with much lower value of vapor pressure) [21]. Therefore, a unique concentration system to increase nitrogen oxide concentration was developed. The proposed setup combines two functions. The first one is the concentration of the vapors emitted by the explosives, and the second one is thermal decomposition of adsorbed explosives vapors.

The operation idea of the explosives vapor detection system with the NO$_x$ sensor is presented in Fig. 2. In the first step, the air surrounding the test object is filtered, dried and then transferred to the next stage, where the concentration of explosives vapor takes place. During this process the explosive vapors present in the air are collected in the adsorbing material. Then, the material is rapidly heated to the thermal decomposition temperature. As a result the concentration of nitrogen oxides (e.g. NO, NO$_2$, N$_2$O) increases. This rise can be measured by NO$_X$ sensors. In the experiments, the optoelectronic sensors developed at the Institute of Optoelectronic MUT were used.
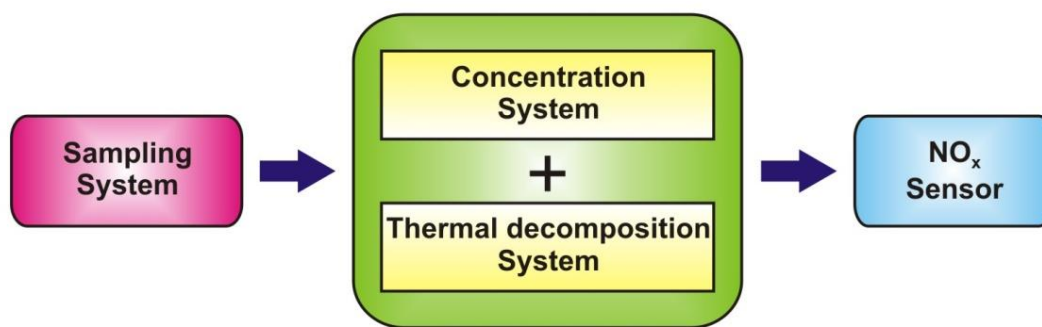

Fig. 2. Block diagram of the explosives vapor detection system with NO$_x$ sensor.

A photograph of the developed concentrating system is presented in Fig. 3a. It was constructed in cooperation with the Electric Power Research Institute at Warsaw University of Technology. The device provides a possibility to obtain an adsorbent temperature of about 500 °C within 25 s. Experimental results of adsorbent heating for different gas flow rates are presented in Fig. 3b.
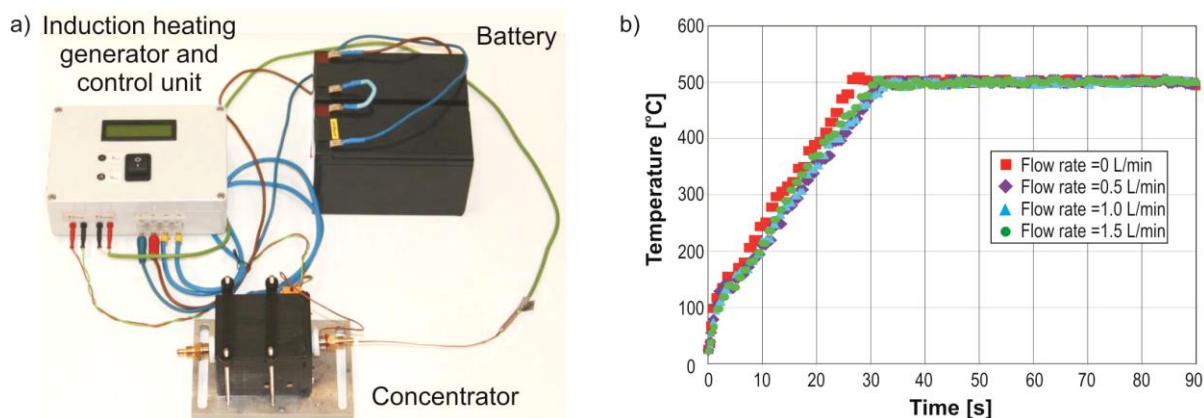

Fig. 3. Photograph of the developed concentrating system (a) and adsorbent temperature changes during the heating process for different gas flow rates (b).

The concentrator consists of an inductor, mounting plate assembly, temperature sensors and a replaceable container with an adsorbing material (Fig. 4). The container is equipped with an adsorbent temperature sensor as well. Applying a special construction, multiple exchange of the adsorbing material is possible. The adsorbent is heated by an inductor and its temperature is controlled by an induction heating generator and control unit. The unit provides container temperature stabilization (Fig. 3b).
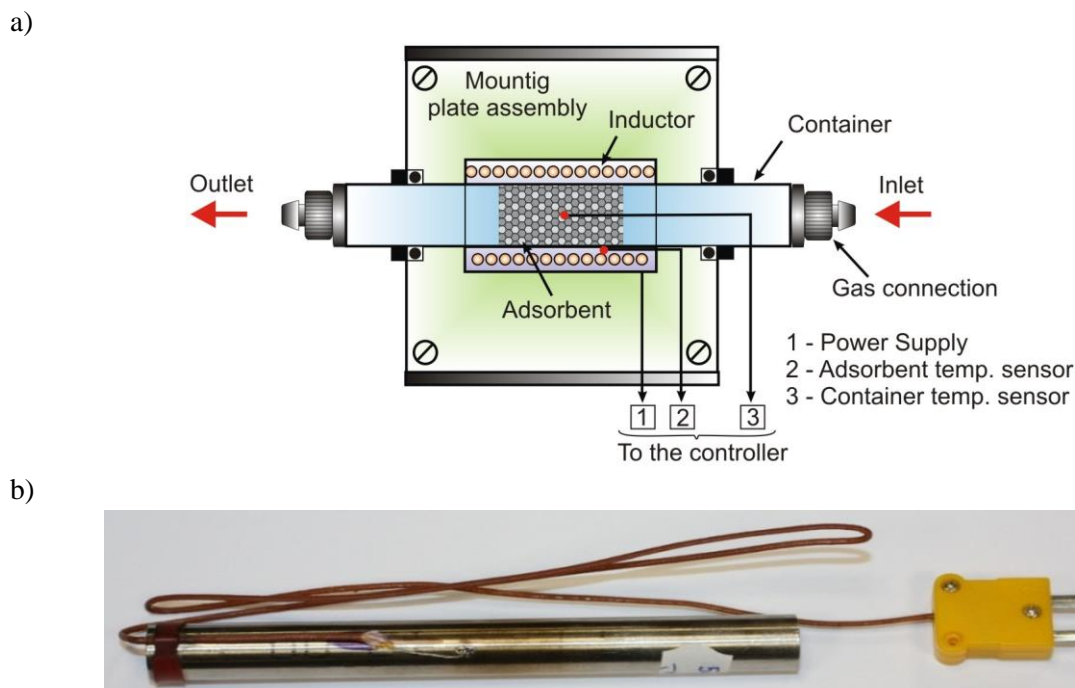
a)

b)

Fig. 4. Construction of concentrator (a) and photograph of adsorbent container (b).

In the unit, a pulse driver with half-bridge circuit transistors was applied [22]. The N-MOSFET switch is triggered by the electronic circuit shown in Fig. 5. The control process is carried out in two stages: heating with automatic selection of near-resonant frequency and maintaining a specific temperature with over-resonant frequency. The pulse repetition frequency is adjusted using an analogue phase-locked-loop (PLL). The comparator analyzes the signals from the current transformer and from the voltage controlled oscillator (VCO) located in the PLL circuit. The output signal from the comparator is filtered by a low-pass filter and is coupled to the input of the VCO. The VCO switching transistors with a frequency close to resonance, keeping the main circuit in the resistive-inductive range. If the temperature reaches the required value, the adder reduces power by increasing the frequency. In the developed system the frequency changes from 0.8 to 3 times of the resonant frequency.

All operational parameters of concentrator, i.e. temperature and time settings and the record of the heating process are monitored using a built-in microcontroller of the AVR series. It makes it possible to display operating data on the LCD screen, to monitor the heating process parameters (temperature and time) by pressing buttons, to switch on and off the heating process, to control the signal frequency, to carry out measurements of actual values of temperature and time, to communicate with external devices via an RS232 or USB interface (e.g. PC). The microcontroller software allows to

set the temperature in the range of 60 °C to 500 °C with a resolution of 1 °C and to measure temperature with a accuracy of 0.5 °C.
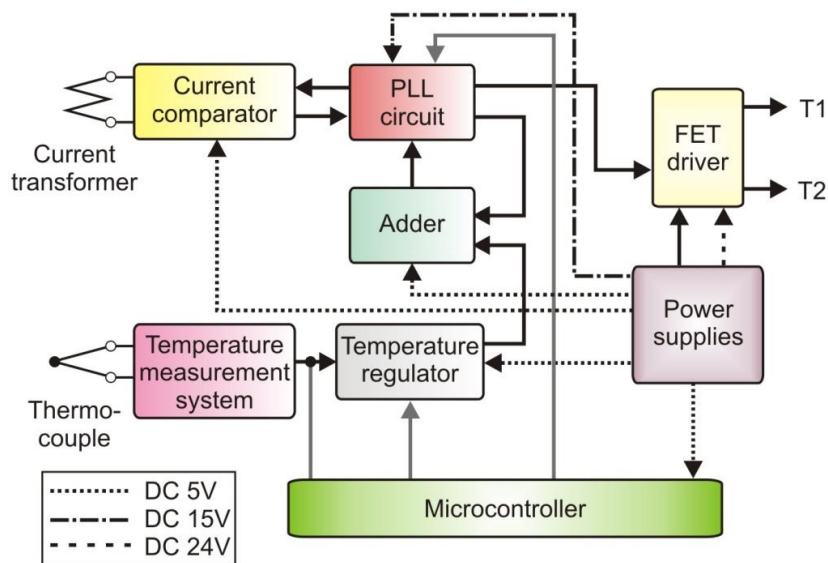

Fig. 5. Block diagram of the induction heating generator and control unit.

Investigations of static and dynamic characteristics of the developed device were performed (Fig. 6). Static characteristics, which are dependent on the demanded temperature, on power consumed by the heater, were determined in heat-set conditions. The heater was supplied with a voltage with a value from 0 V to 10 V. The static characteristics are for the adsorbent container made from metal (m – dashed line) and from metal-glass (s– solid line). In case of metal containers, the dynamic characteristics were performed for different power values (P1 = 150 W; P2 = 275 W; P3 = 500 W). Static and dynamic characteristics were used to determine the basic parameters of the induction heating generator.
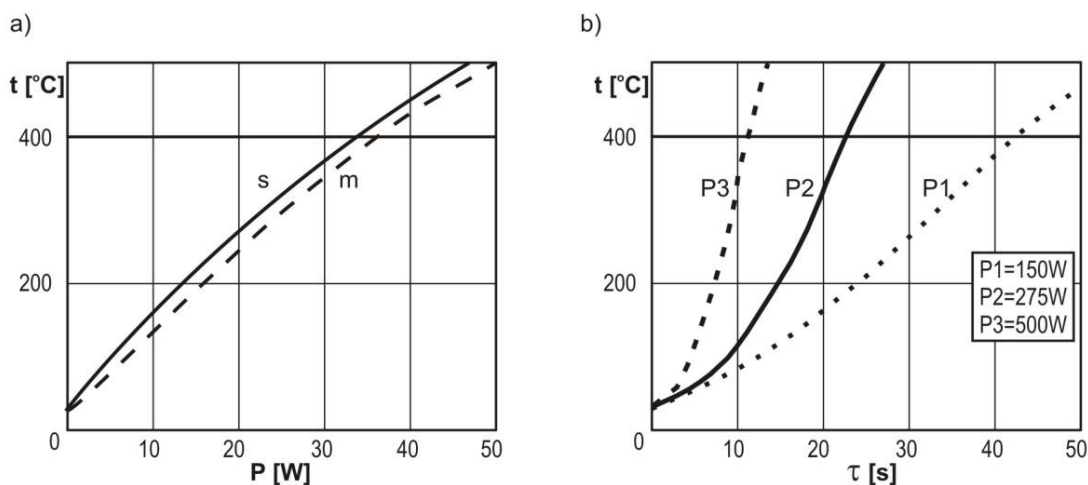

Fig. 6. Temperature characteristics of the concentrator, static (a) and dynamic (b).

3. Investigation of various adsorbing materials

Low pressure of vapors emitted by the majority of the explosives significantly limits the use of many detection systems. Suitable adsorbing materials provide effective concentration of target chemical compounds. The analysis shows that the adsorbents should be characterized by low thermal resistance, acid resistance, high sorption selectivity, and short sorption-desorption processes [23, 24].

The described concentrator system is designed to perform two functions: explosives vapor concentration and their thermal decomposition. Thus, the adsorbing material should be characterized by both high concentration factor and high temperature of destruction. The main parameters of the most popular adsorbents are listed in Tab. 3.

Table 3. Main properties of selected adsorbents.

Adsorbent	Formula	Molecular weight [g/mol]	Density [g/cm^3] @ 20 °C	Character	Temp. limit [°C]	Particle size	Surface area [m^2/g]	Desorption temp. [°C]	Comments
Zeolite molecular sieves (eg. 5A, 13X)	Al & Si compound	-	~0.65	Granules	600-800		-	250 - 350	The ability to selective adsorption of explosives molecules
Cerium oxide (IV)	CeO$_2$	172.11	7.13 @ 25 °C	Nano-powder	-	<25 nm	-	180-250	-
Aluminium oxide	Al$_2$O$_3$	101.96	4,00	Powder	-	<50 nm	>40	-	Highly hygroscopic, insoluble in water
Carboxen 1000 (carbon molecular sieves)	C	12.1	0.25-0.6	Powder, granules	400	60-80 mesh*	~1200	225 - 300	Inflammable, insoluble in water
Tenax TA	polymer resin	-	~0.25	Powder	350	60-80 mesh*	~35	180 - 300	Useful in the case of sorption in high humidity condition

number of mesh per inch

During investigations, the optimal parameters of the heating system, like maximum temperature, heating and cooling time for selected adsorbents were determined.

3.1. **Procedure of experimental sample preparation**

For the investigations the following adsorbents were chosen: Tenax TA, Carboxen 1000, aluminium oxide, and cerium oxide. During the study, two techniques were used to prepare samples for chromatographic analysis. In the first case a glass sorption tube (ampule, cartridge) filled with an adsorbent was prepared (Fig.7). The volume of the adsorbent was approx. 0.25 cm³ (0.1 g). To remove adsorbed pollutants, the ampule was heated in a stream of argon at a temperature of 200 °C to 250 °C.
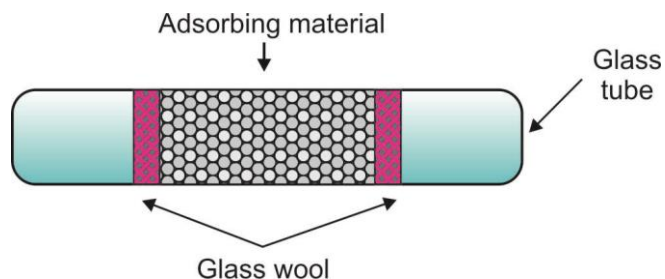

Fig. 7. Sorption tube filled with the selected adsorbent material.

In the second technique, *Solid-Phase Micro Extraction* (SPME) was applied. In this method a syringe equipped with quartz fiber coated with thin sorption layers is used (Fig. 8). These layers form a cylindrical shape on the outer fiber surface. It facilitates the mass exchange during the sorption and releasing process of trapped compounds. It also eliminates the problems associated with deposit clogging. The applied technique can be divided into two stages. During the first stage, the fiber contacts with the sample in the gas phase (or in the liquid phase) and a sorption process of analytes is performed. During the second stage, the fiber is exposed to high temperature inside the hot dispenser of a gas chromatograph. Released analytes are transferred to the chromatographic column. In the column, splitting and quantification of these analytes are carried out. For sorption of explosives vapor, coatings made from polydimethylsiloxane/carboxen and polydimethylsiloxane were used.
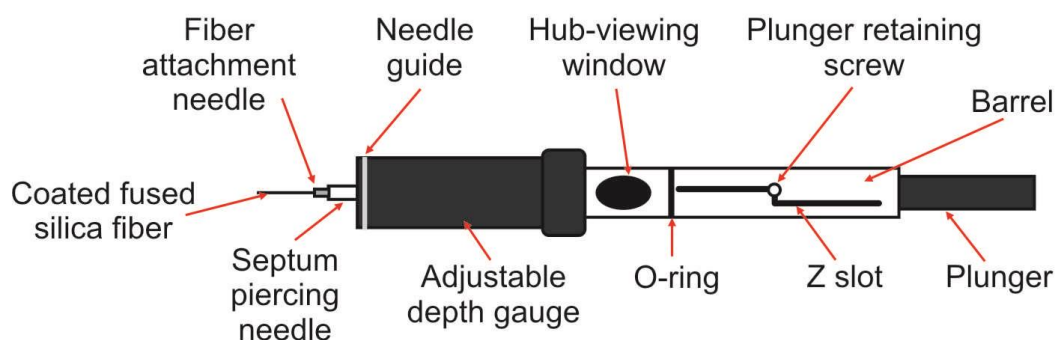

Fig. 8. Scheme of a syringe used in the SPME technique.

To achieve a constant temperature of the sorption process, a glass container containing a given explosive was placed in a thermostat. This set makes it possible to determine the influence of temperature on the explosives sorption process for various adsorbents. To establish a balance between the over-surface phase and explosives, the container had been thermostated before the measurements. Adsorption of explosives was started after equilibrium between the gas phase and solid was

established. The scheme of the explosive vapor collection system is shown in Fig. 9. Into the container with an explosive material, a stream of air was introduced with a flow of 100 mL/min. When the sorption tube was saturated with explosive vapours (after about 1 min.), it was placed for 20 min. in an oven preheated at 100 °C. After that, a chromatographic analysis of the released gases was performed.
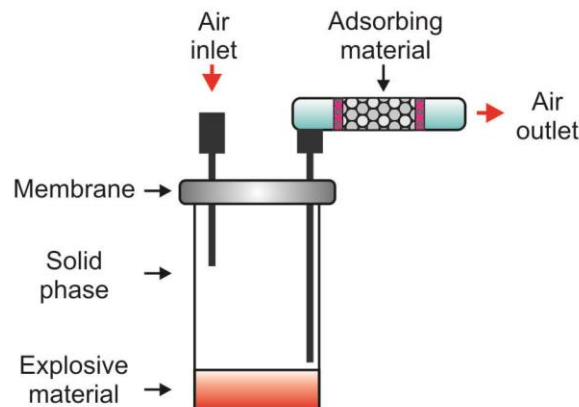

Fig. 9. Scheme of the collection system using sorption tubes.

During the experiments, collection of explosive vapors using stationary phase micro-extraction was used as well. This greatly simplifies the entire process and eliminates several intermediate steps (e.g. it does not require preparation of sorption tubes).

The sealed containers with explosives were placed in a thermostat preheated at a given temperature (25, 30, 35, 40, 50, or 60 °C) for approx. 20 min. After that, SPME fiber was introduced (through a seal membrane) into the container. The adsorption lasted for approx. 10 min. Next (finally), the fiber was inserted directly into the injector of a gas chromatography system and the extracted vapours of explosives were thermally desorbed and analysed.

3.2. Experimental results

Two explosive materials (TNT) and (RDX) have been used in this research and their purity was determined using the TLC method described in literature [25].

Samples of the explosives vapors were analysed by the use of a HP 6890 gas chromatograph with an HP G2350A atomic emission detector from Hewlett-Packard (Wilmington, DE, USA). Software Chemstation HP 35920A was used for equipment operation and for collection of measurement data as well as to process the recorded data.

Helium was used as the carrier gas at a flow rate of 1.0 mL/min in the constant flow mode. An atomic emission detector has been used on carbon and nitrogen selective channels. During the chromatographic analyzes the temperature program has not been used but the capillary column temperature was constant at 30 °C.

Fig. 10 shows the dependence of detector response for selected adsorbents and explosives (TNT and RDX) as a function of temperature.
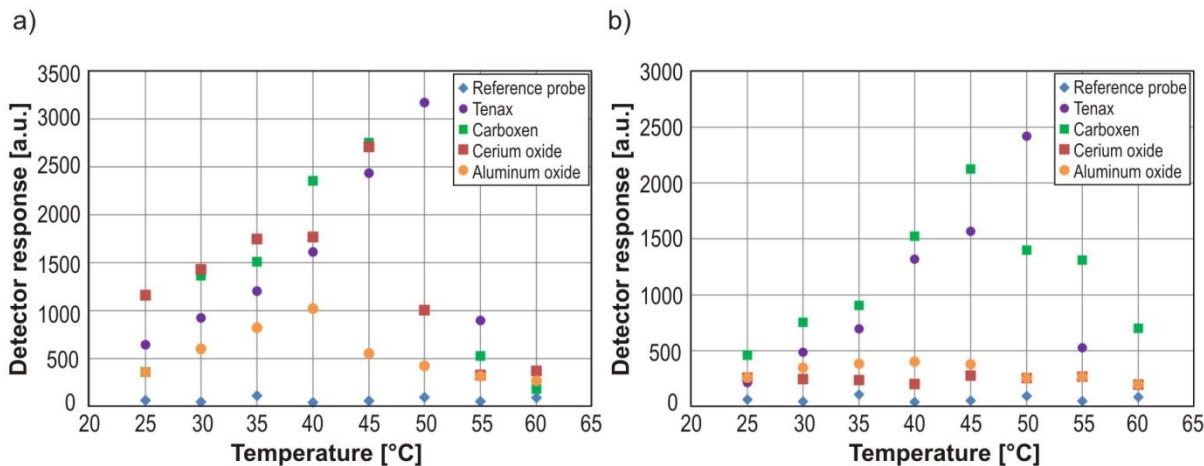
a)

b)

Fig. 10. Dependence of detector response on selected adsorbents temperature for TNT (a) and for Hexogen (b).

The influence of the adsorption time on sorption efficiency of Tenax TA and Carboxen 1000 at a temperature of 45 °C was also tested (Fig. 11).

It was noticed that Tenax TA is most efficient for both explosives at a temperature of 50 °C. In the case of Carboxen1000, the highest adsorption efficiency was obtained at a temperature of 45 °C. Investigation with cerium oxide and aluminium oxide showed that these materials have the best performance of adsorption at a temperature of 40 °C for TNT. Regardless of the type of adsorbent, the optimal adsorption time for TNT and hexogen vapor sorption was 5 minutes. A longer exposition of the absorbing material to the explosives vapours did not significantly increase the detector response. Summing up, it was shown that Tenax TA had higher efficiency of adsorption (about 10%) for both explosives as compared to other tested adsorbents.
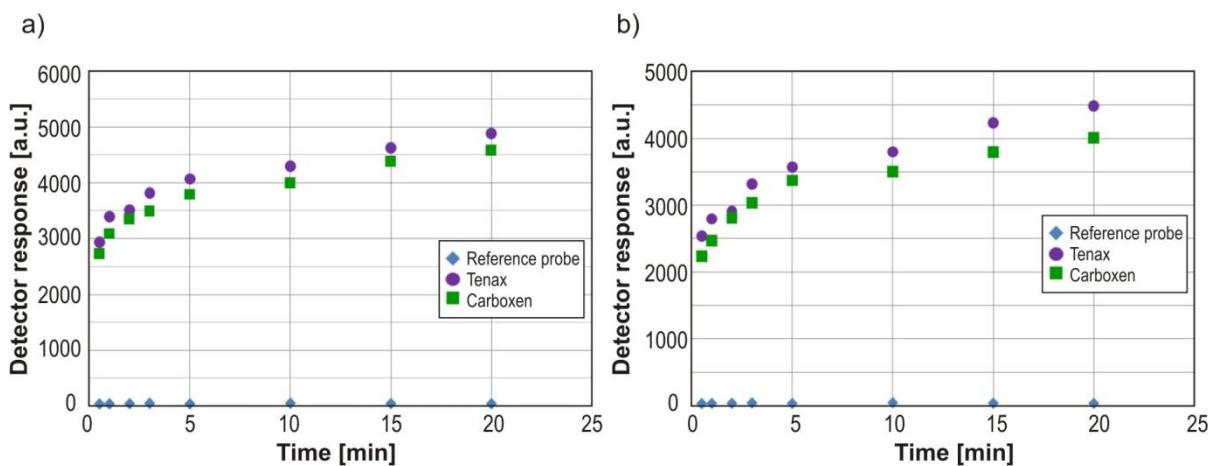
a)

b)

Fig. 11. Dependence of detector response on adsorption time for TNT (a) and for RDX (b).

4. Preliminary research with an optoelectronic sensor

The possibility of explosives detection with the developed concentration and decomposition system was verified using the laboratory setup shown in Fig. 12. In this setup, an optoelectronic dioxide sensor based on *Cavity Enhanced Absorption Spectroscopy* (CEAS) was applied. The sensor allows to measure NO_2 concentration with a detection limit of 1 ppb and uncertainty of 10% [26].
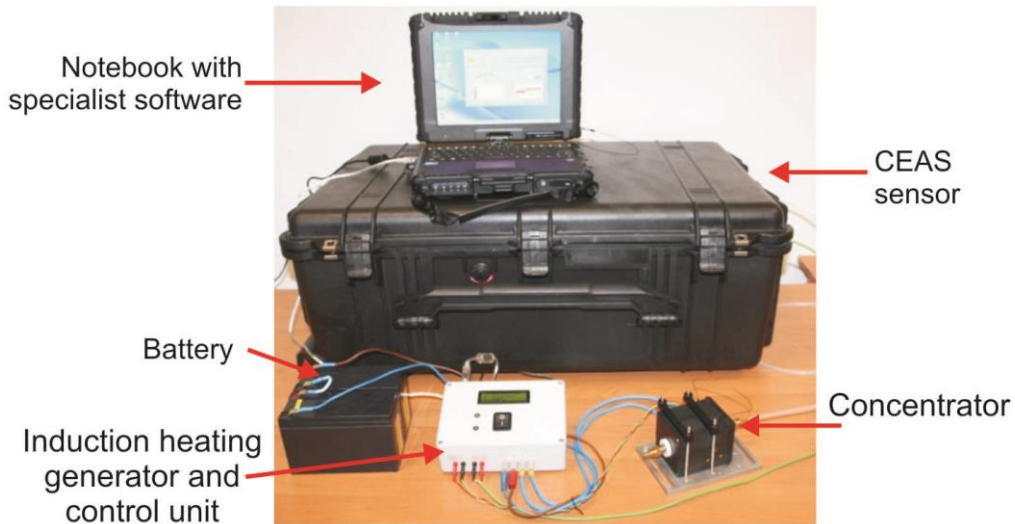

Fig. 12. Photograph of setup for explosives detection.

Two explosives samples (TNT) with different concentration levels (50 ng/mL and 0.5 ng/mL) were analyzed. The cartridge heating temperature T_p was set at 150 °C and 200 °C for the first sample, and 200 °C and 300 °C for the second one. The heating time was set to 10 and 20 s. Fig. 14 presents nitrogen dioxide concentration changes at the outflow of the developed concentration system.
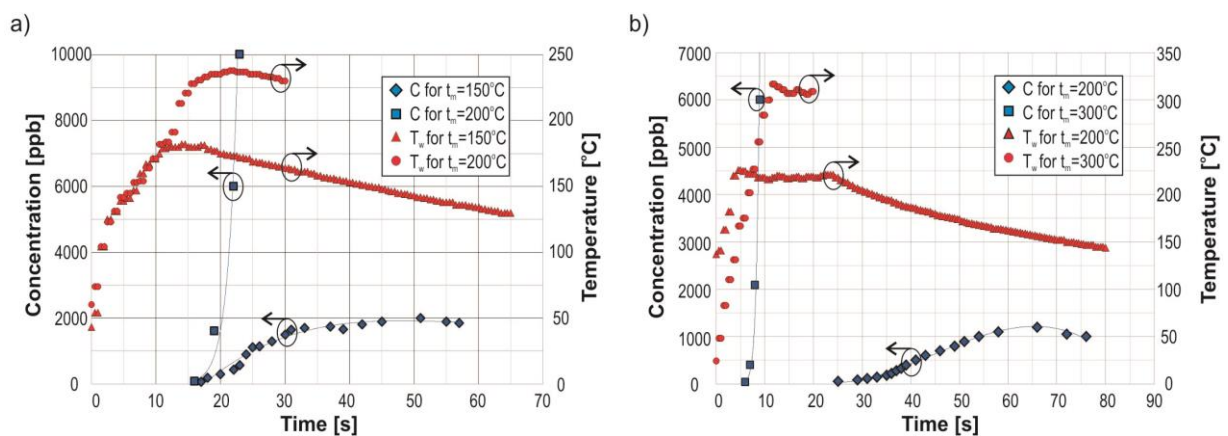

Fig. 14. Experimental results for TNT samples of 50 ng/mL (a) and 0.5 ng/mL (b).

The results presented in Fig. 14a show that both the concentration of NO_2 and its changes with time depend on the heating temperature. A faster increase in the temperature of the cartridge causes a more effective thermal decomposition of explosives. For temperature below 200 °C, NO_2 concentration increases up to 2000 ppb. In the case of temperature of approx. 240 °C, NO_2 concentration exceeds the upper measurement limit of the CEAS sensor (>10 ppm).

In the experiments with TNT samples of 0.5 ng/mL, the tested detector reacted similarly (Fig. 14b). High differences in NO_2 concentration were recorded when the sample was heated to 200 °C and 300 °C. At the latter, the concentration of NO_2 reached 6000 ppb within approx. 10 s.

Results showed that in order to obtain an optimal rise in explosive vapor concentration, the heating temperature is very important. The higher decomposition temperature provides higher nitrogen oxide concentrations [21]. Several other detector parameters should be taken into account as well, especially sensitivity and measurement time. The mass of adsorbent and mass of explosive sample have also significant influence.

5. Conclusion

Trace vapor detection of explosives is an extremely difficult task. The sensitivity of currently constructed sensors is often insufficient. At the Institute of Optoelectronics a concentration and decomposition system of explosives vapors has been developed. This system is designed to work with nitrogen oxide detectors. Preliminary measurements show that using the concentrator, the recorded amount of nitrogen dioxide released from a 0.5-ng sample of TNT increases by a factor of approx. 20. Moreover, a significant increase in the NO_2 concentration can be gained when the desorption temperature is higher than 150 °C.

In the concentrator an induction heater is applied. Thanks to this and because of the miniaturization of the container with an adsorbing material (approx. 1 cm^3), an extremely high rate of temperature growth is achieved (up to 500 °C within approx. 25 s). Additionally, the whole processes of concentration and decomposition is controlled by a microcontroller.

During investigations Carboxen 1000, Tenax TA, cerium oxide and aluminium oxide were applied. Chromatographic analyses showed that Tenax TA gives the highest efficiency of explosives vapour adsorption, when the absorption is carried out at a temperature of 50 °C. As compared to Carboxen, Tenax adsorbs more explosives vapours by approx. 10%.

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