

TATP and TNT detection by mid-infrared transmission spectroscopy

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ABSTRACT

Sensitive and fast detection of explosives remains a challenge in many threat scenarios. Fraunhofer IPM works on two different detection methods using mid-infrared absorption spectroscopy in combination with quantum cascade lasers (QCL). 1. stand-off detection for a spatial distance of several meters and 2. contactless extractive sampling for short distance applications.

The extractive method is based on a hollow fiber that works as gas cell and optical waveguide for the QCL light. The samples are membranes contaminated with the explosives and real background. The low vapor pressure of TNT requires a thermal desorption to introduce gaseous TNT and TATP into the heated fiber. The advantage of the hollow fiber setup is the resulting small sample volume. This enables a fast gas exchange rate and fast detection in the second range. The presented measurement setup achieves a detection limit of around 58 ng TNT and 26 ng TATP for 1 m hollow fiber. TATP – an explosive with a very high vapor pressure in comparison to TNT or other explosives – shows potential for an adequate concentration in gas phase under normal ambient conditions and thus the possibility of an explosive detection using open path absorption of TATP at 8 μm wavelength. In order to lower the cross sensitivities or interferences with substances with an absorption in the wavelength range of the TATP absorption the probe volume is checked synchronously by a second QCL emitting beside the target absorption wavelength. In laboratory measurements a detection limit of 5 ppm*m TATP are achieved.

Keywords: explosives, quantum cascade laser, absorption spectroscopy, TATP, triacetone triperoxide, TNT, trinitrotoluene

1. INTRODUCTION

A high level of security against terrorism is for the public benefit. At public places like railway stations or airports the detection of explosives are important parts of preventive measures. But there is no perfect device or method which covers all the following necessary key issues concomitantly: sensitive, reliable, fast and specific.

Other requirements of the applicators are to check non-invasively the explosive contamination of people or baggage and with the smallest amount of manpower. So, the most promising systems are multi-sensor platforms and stand-off explosive detection. The presented hollow fiber setup is done in the framework of the GOSPEL (General Olfaction and Sensing Projects on a European Level) project TASSE (Trace Automatic Sampling and Sensing for Explosives) to develop two complementary methods for a multi-sensor platform: laser-based ion spectrometry (IMS) and the optical absorption spectroscopy based on quantum cascade laser. In many scenarios stand-off-detection would be preferred compared to contact sampling systems.

By stand-off detection hidden screening of areas of interest is possible and in case of an alert first measures can be initiated without knowledge of the suspect. However, no reliable technical solution exists up to now for distances larger than a few m. In this work we show a promising detection scheme for stand-off detection for explosives with high vapor (Fraunhofer program OFDEX).

X-rays employing different energies can be used to detect suspicious materials but cannot confirm that they are explosives. Dogs are used extensively for cargo screening but they are not appropriate for 24 h assignment. The most popular detection methods are gas chromatography (GC) and ion mobility spectrometry (IMS). The GC is a very powerful and versatile laboratory instrumentation designed for fixed-facility use and for the detailed analysis of complex mixtures. The IMS devices are portable and easily transportable and intended for field use for the detection of specific target compounds.

However, these techniques have some drawbacks: insensitivity to some key compounds, masking by interferences, practical deployment concerns etc. The GC needs a special sample preparation and shows the result in several seconds. Due to this relative long respond time it cannot apply on moving targets. The nature of the threat from explosives is also increasing by the developments in improvised explosive devices (IEDs) which use explosives manufactured from common domestic chemicals rather than those produced commercially. One example is TATP (triacetone triperoxide) which was used in the London transport bombings. Explosives such as the commercial available TNT, RDX are easily detected with an IMS and a GC system. In a save and complete detection scheme other explosives such as TATP or other IEDs need to be effectively detected at a security checkpoint, too. These threat compounds are not easily seen in a normal, single mode IMS system.

So, a new setup operating on TATP and TNT explosives was investigated to have a complementary method to the IMS. The absorption spectroscopy in the mid-infrared is a strong and specific tool for analysing chemical compounds. TATP and TNT have broad, characteristic and strong absorption bands in the mid IR. Fourier Transform Infrared (FTIR) spectroscopy is the appropriate standard method for an optical gas analysis^{1, 2, 3, 4}, but the conventional FTIR devices work too slow and are not suitable in field use. Our approach is to setup an absorption spectrometer with a quantum cascade laser (QCL) as light source and a hollow fiber as gas cell. The laser light and the gas are guided in the hollow fiber. The explosives are evaporated in the desorbition unit and the gas is introduced into the fiber. Normally only a small amount of explosives is available for detection i.e. several nanograms. This requires an extreme small sample volume to avoid dilution. A hollow fiber is an optimum gas cell for this application and enables an efficient overlap with the probe light of the QCL.

How can be used such a hollow fiber and QCL based detection system in the field? A new approach, items are placed within an air-tight chamber (Fig 1). The target objects, i.e. Baggage or persons, have to pass this chamber with an induced draft fan. Then, induced particles in the airflow are collected by a membrane. This works like a preconcentrator, the membrane is heated and the collected explosives evaporate and are introduced into the spectrometer (Fig. 1). Ray Detection's DiscoveryCERT[®] (Contaminants Enhanced Release and Transfer) system is an example of this approach. The CERT technique coupled with GC has been successfully demonstrated in a number of field trials at the Tel Aviv International Airport. The tests covered detection of real explosive devices placed in a bag, luggage or cardboard packaging and concealed within genuine shipments on pallets.

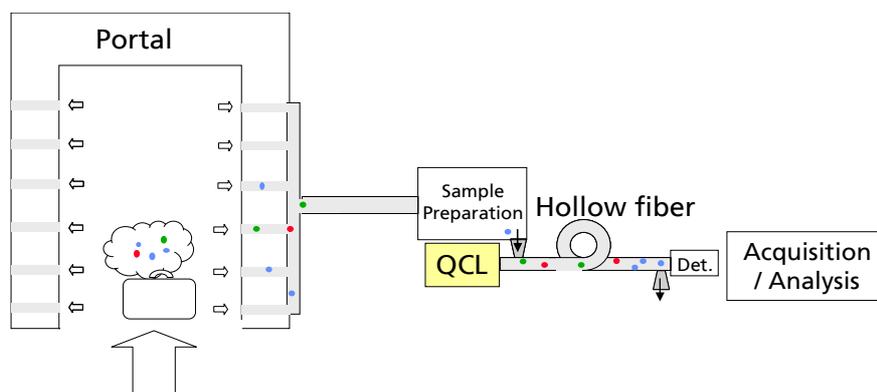


Fig. 1. The explosive detection system could be applied in combination with an airtight chamber. The sample preparation could work like DiscoveryCERT[®] system: the explosive particles are extracted from the portal, collected, concentrated and desorbed in the sample preparation and detected in the hollow fiber QCL spectrometer.

The basic concept of a QCL open path setup for explosive detection is shown in Fig 1a. It consists basically of QCL and detector modules with long distance optics on a tripod, and a cornercube reflector. To cope with cross sensitivities a setup with two QCL modules for sensing and reference wavelengths will be employed. In a realistic scenario, the plume of the evaporating explosive material e.g. TATP is diluted and disturbed by movements and air flow. The material could be encapsulated or hidden below clothing, or only traces are left on the surface. Thus gas phase concentrations probably will be much lower than the equilibrium values in table 1. Hence we first have to determine our detection limits with well defined samples and conditions in the laboratory.

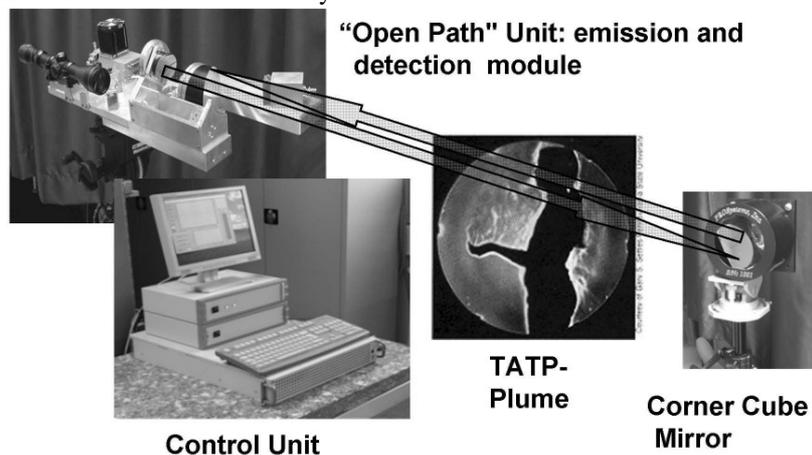


Figure 1a: Basic concept of open path setup for explosive detection.

1.1 TATP and TNT

In Table 1. Here the common explosive ingredients and their vapor pressures at 25°C are listed^{5, 6, 7}. These compounds are often used in combination, for example the commercial explosive Semtex is composed of the active ingredients RDX and PETN along with plasticizers.

Explosive	Vapor pressure at 25°C (ppt)	Source	Classification
TATP- triacetone triperoxide	430,000,000	improvised explosive	Potentially high vapor pressure
EGDN - ethylene glycol dinitrate	100,000,000	commercial / military	Potentially high vapor pressure
NG – nitroglycerine	500,000	commercial / military	Potentially high vapor pressure
DNT – dinitrotoluene	56,700	commercial / military	Low vapor pressure
TNT – 2,4,6 trinitrotoluene	9,400	commercial / military	Low vapor pressure
PETN – pentaerythritol tetranitrate	16	commercial / military	Very low vapor pressure

Table 1 shows that detection in the gas phase of many of these explosives is not feasible by simple ‘sniffing’ of the target object. However use of the desorption methodology allows particles of the explosive to be captured, concentrated and thermally desorbed enabling the use of gas phase detection. Explosives like TATP and TNT have different absorption bands in the mid infrared. As an example in Fig. 2 a FTIR spectrum of TATP is shown.

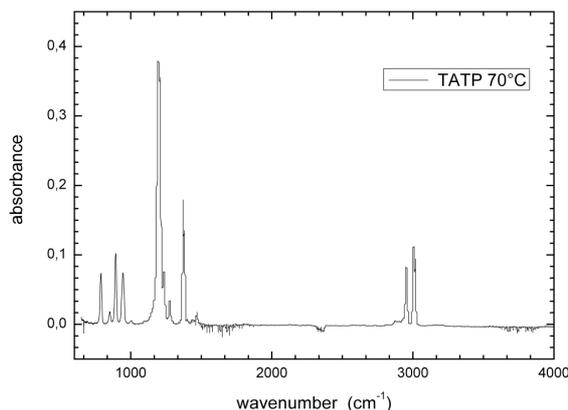


Fig. 2. The FTIR spectrum of a rather high concentration of 0.15g/l TATP in the gaseous phase at 70°C in 10 cm cell shows a characteristic absorption at 1190 cm^{-1} with a maximum absorbance of 0.38.

2. EXPERIMENTAL

2.1 Quantum Cascade Lasers



Fig. 3. IPM-FhG basic QCL-measurement system: QCL driver, detector driver, PC for data acquisition ⁸.

The QCL is an intensive, compact and wavelength selective light source in comparison to thermal emitters used in FTIR. The wavelength of the QCL matches the absorption band of the explosive. So no dispersive element or spectral analysis in such setup is needed. For the TNT detection measurements the QCL emits at 7.4 μm and for the TATP experiments at 8.2 μm . TATP and TNT have broad absorption bands (>100 nm) (Fig. 4), they are not really suitable for QC laser spectroscopy because the spectral pulse width is smaller than 0.02 nm and the tuning range of around 4 nm is not sufficient to scan the absorption band. But the broad absorption bands are on the other side an advantage, because it provides the application of high power multi mode laser in pulse operation. The multimode emission bands cover the main spectral range of the absorption band. In the preferred pulse mode of operation the manufactures (NanoPlus GmbH, Fraunhofer IAF) offers devices with an average power of more than several milliwatts with sub amp threshold current and possibility of duty cycle >1%.

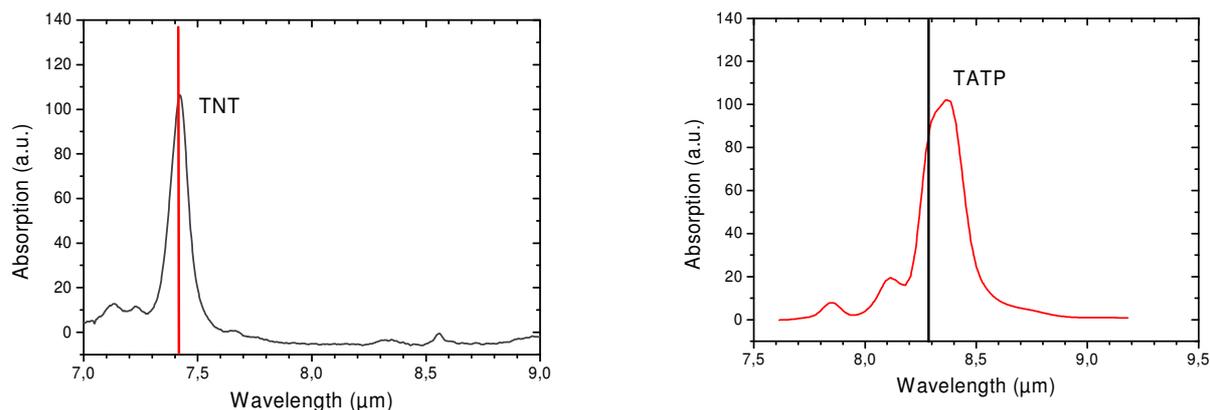


Fig. 4. Absorption bands and the laser emission wavelength. TNT has its prominent absorption band at 7.4 μm , TATP at 8.4 μm . The absorption bands have a spectral width > 16 nm. The bars in the graphs show the emission wavelength of the QCLs.

The home build QCL drivers (Fig. 3) provide two different operation modes of the QCLs at room temperature: the short and long pulse mode. The TATP measurements in the hollow fiber were done with the short pulse operation. This means that the QCL pulse width is 100 ns and the repetition rate at 50 kHz, so a duty cycle of 0.5% is achieved. In this operation mode the laser emits up to 30 mW. A fast TE cooled mid IR detector (Vigo Ltd.) is adapted to such a bandwidth of 100 MHz. A 200 MHz oscilloscope records and averages the transients and is read out by a computer. For the TNT measurements in the hollow fiber the QCL operates in the long pulse mode: 10 μs pulses at 700 Hz repetition rate (0.7 % duty cycle). Because of the long pulses a pyro detector (Infratec LME353) is suitable and a digital lock in amplifier (Perkin Elmer) could be used. This is a very cost effective mid IR detection scheme.

The QCLs for open path detection operates in the long pulse mode. But in this case Vigo detectors (PVI-2TE-9) for an increasing signal to noise ratio were used instead of the pyro detectors.

Both operation modes were compared and analyzed. The short pulse mode works with most of the QCLs because the thermal load on the QCL chip is lower than in the long pulse operation. But the improvements in last years enables that the new QCLs can operate with the long pulses up to cw. The noise equivalent detection limit of the transmission is in both cases around 0.0015. But the long pulse mode enables the use of conventional electronics with bandwidths < 100 kHz. But the transmission resolution is limited by the disturbance of the heating electronics and not by the detection scheme.

2.2 Hollow fibers

An important point is the required probe gas volume and the corresponding sample mass. The 10 cm cell used for the measurement in Fig. 2 has a volume of 57 ml which requires 5 mg TATP for saturation with a vapor pressure 1400 Pa at 70°C. Thus reduction of the cell volume for a given optical path length is crucial for the detection of trace quantities and dilution of the sample in the infrared setup has to be avoided.

A huge step can be achieved by the use of hollow fibers as gas cell^{9, 10, 11}. These waveguides – unlike common optical fibers – have an air core where the radiation is guided by reflection at a mirror layer that covers the inner side of the fiber-cladding. At the same time a gas can flow through the air core and absorb the infrared radiation specifically. Since the core diameters of these fibers are in the range of 300 to 1000 μm a relative long optical path can be obtained for a small gas volume. Such fibers are commercially available at Polymicro. In the system setup a 50 cm long fiber with 750 μm hollow core is used. This means that the gas cell has a volume of 0.22 ml.

The HWCA fiber type has the low attenuation of 1.3 dB/m in the 7-8 μm wavelength range. The cross-sections of the two fiber types are shown schematically in Fig. 5 together with the corresponding scanning electron microscope (SEM) pictures (right sides). The in coupling losses of the hollow fiber are very weak, because the core has a large diameter and is filled with air. The applied fiber with the 90° bend has a transmission of around 50 %.

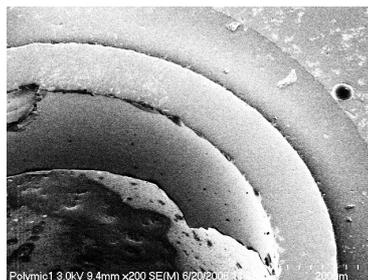
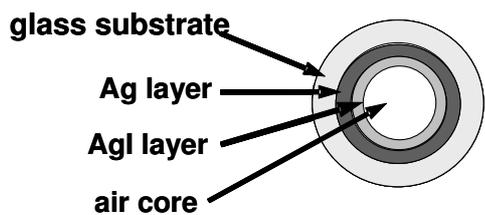


Fig. 5. Design of a hollow Polymicro fiber (left) and SEM-picture of cross section (right) showing Ag layer and due to preparation partly delaminated AgI film.

2.3 Hollow fiber setup

The Fig. 6 shows the desorbption unit, where TATP or TNT samples are evaporated at 90°C and if the equilibrium vapor pressure is approximately obtained the chamber gas is pressed into the hollow fiber by a weak N₂ flow. TATP and TNT have different sample types (Fig. 7). The small closed metal cans filled with TATP crystals. It has been packaged like this to avoid evaporating during storage. The TATP is provided by the Fraunhofer ICT in the framework of the Fraunhofer project OFDEX (Optische Ferndetektion von Explosivstoffen). The cans were opened by a stitch and immediately put into the desorbption unit (Fig. 6). In contrast the TNT samples preserve the amount because of the low vapor pressure. The TNT samples are a mixture with sand and are weighed in the mg range and evaporated in the sample chamber. The TNT sand is provided by the Fraunhofer ICT, too.

The project partner Ray Detection Technologies provides membranes from trace automation sampling (Fig. 7). These membranes were investigated to show the function close to the real application. The membranes were contaminated with TNT and the additional real background contamination. So there is no quantitative information about the TNT amount.

In the Figures 8 and 9 the setup is shown. The light of the QCL is focused into the fiber by a mirror objective with 140 mm focal length. A beam splitter reflects 10% on a reference detector for monitoring the laser intensity. The fiber ends and the adapters have a standard FC coupling (Fig. 9). The adapters contain a 25mm-FC-adapter and packaged in a cylindrical mount with laser window and tight gas channel. In front of the fiber facets and with few millimeters distance the optical windows are fixed. The volume of the two hollow parts in the adapters (<1 ml), the inner chamber volume (<8 ml) and the fittings (<1 ml) are added to the fiber core volume (0.2 ml), so a total volume of around 10 ml arises. The in coupling adapter has a direct and short channel to the sample chamber. At the second fiber adapter the light and the gas is coupled out. An objective is focusing the light on the signal detector.

In order to avoid condensation of the explosives at the walls of the fiber a heating jacket for the fiber is developed. A heating wire is bend surround the fiber. The temperature of around 90° is in the operation range of the fiber, no decreasing of the transmission could be observed.

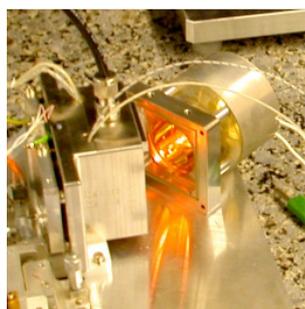


Fig. 6. Open desorbption unit. A lamp heats up the sample and the explosives change into the gas phase. In order to avoid condensation the body of the unit is heated up to 100°C. A nitrogen purge or a pump pushes the gas into the fiber.

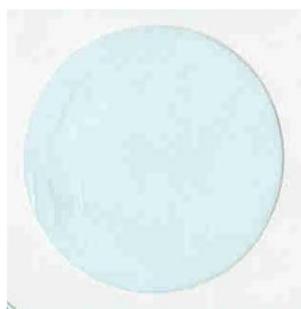


Fig.7. Membrane (left) containing particulate and vapor residue (actual size of membrane approximately 3 cm diameter). the TATP is hermetical enclosed in a metal can (right), the diameter is 5 mm.

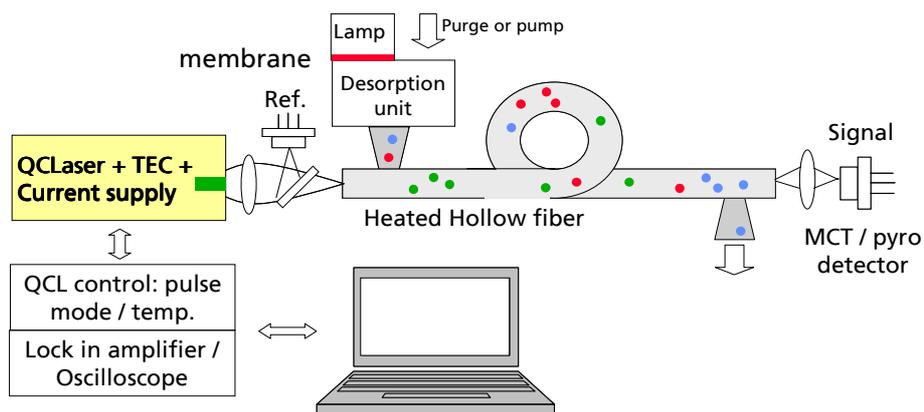


Fig. 8. System setup: The TATP detection operates with the 8.2 μm -QCL, the short pulse mode, the fast MCT detector and the data acquisition with the oscilloscope. Instead of the membrane the small metal can (Fig. 7) is used. The TNT measurements were done with the 7.4 μm -QCL, the lon pulse mode, pyro detector and lock in amplifier.

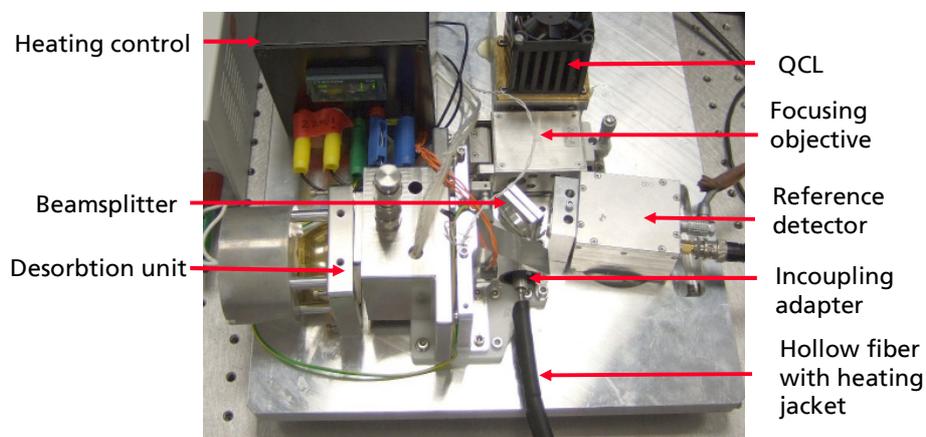


Fig. 9. QCL spectrometer setup with desorption unit and hollow fiber in heating jacket.

2.4 TATP open path detection

The setup to perform laboratory experiments with well defined TATP samples is shown in Figure 10.

The collimated beam of a QCL passes through a heated open glass tube. Inside the tube a TATP sample was placed onto an extra small heater. When the heater is switched on, TATP evaporated inside the open tube. Condensation on the walls is prevented by heating the tube walls. The tube can be easily rinsed by air flow to recover the baseline signal. Compared to closed cells no uniform gas concentrations values are obtained. However, this setup is more realistic and window effects are avoided.

To study interferences by other atmospheric constituents, a two channel setup was recently assembled, which consists of two QCL modules with sample and reference wavelengths. In this case a closed cell is employed which could be positioned before or behind the open cell for the explosive sample.

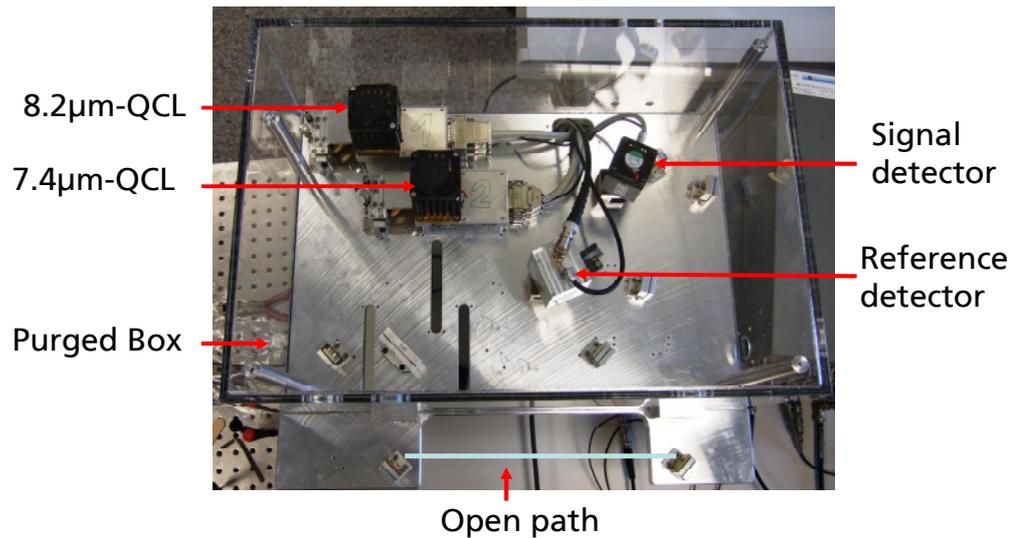


Figure 9: Laboratory setup with two QCL modules for two wavelengths measurements with reference and measurement paths. The path outside the purged box is for explosive detection in the ambient air. In the lab an open tube with an explosive sample is positioned in the path.

3. RESULTS AND DISCUSSION

3.1 Hollow fiber

The TATP measurement was done without heating the fiber. The vapor pressure of TATP is sufficient high to have significant TATP concentrations at room temperature. But to see a fast signal change the heating of the desorbition unit is used to evaporate the TATP in a minute. But due to the cooler fiber the equilibrium vapor pressure corresponds to the fiber temperature of around 23°C. The disadvantage was that the recovering of the signal takes several hours to evaporate completely the condensates TATP. So, for the experiments with TNT a fiber heating was developed to prevent condensation of the TNT at the fiber walls. The result of the TATP measurement is shown in Figure 10. Thus in the desorbition chamber a maximum TATP volume concentration of 11 % is reached. With a fiber length of 50 cm roughly a 50% transmission change is obtained. The fiber has the room temperature (23°C), so the saturated vapor pressure is around 4.3 Pa (Table 1). This means for the estimation of the detection limit a concentration of 43 ppm TATP is in the fiber. The entire probe gas volume (chamber + fittings + fiber) is estimated with 10 ml, so there are a round 3.9µg TATP in the gas phase (molecular weight of TATP is 222 g/mol).

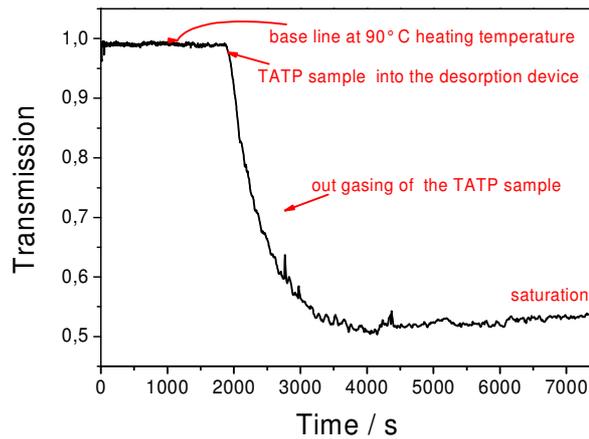


Fig. 10. The TATP sample weight was 1.5 mg. This can was heated up to 90°C, but the fiber has a temperature of 23°C. So for the calculation of the detection limit the vapor pressure from Table 1 is taken into account. The signal did not recover completely because of condensation at the fiber walls. By heating and purging the fiber the TATP desorbs out off the fiber. The TATP signal appears slowly because of the diffusion into the fiber.

The analysis of the noise without TATP signal results that a transmission change of 0.005 ($\sim 3\sigma$) is detectable for such an instrument. For a 1m fiber filled with gaseous TATP a transmission of 25 % is expected. This corresponds to a detection limit of around 26 ng TATP gas at room temperature. By improvements in the system transmission changes in the 0.0001 range are feasible, and the fiber parameters may be further optimized. Hence detection limits in the 1 ng range are within reach.

In the experiment with the 10 mg TNT sand mixture a transmission of 60 % is measured in the 50 cm fiber (Fig. 11). The calculation of the TNT detection limit is similar to the TATP.

The sample contains 2 mg TNT, the rest of the sample is sand. The vapor pressure of 10 Pa corresponds to 100 ppm and this to 741 mg/m³ (molecular weight of TNT is 227 g/mol). This means that 7.41 μ g TNT gas is in the chamber volume of 10 ml, if the saturation is achieved. The spectrometer resolves as smallest transmission change 0.005 (3σ). So for a fiber with 1m the detection limit for TNT gas at 90°C is around 58 ng.

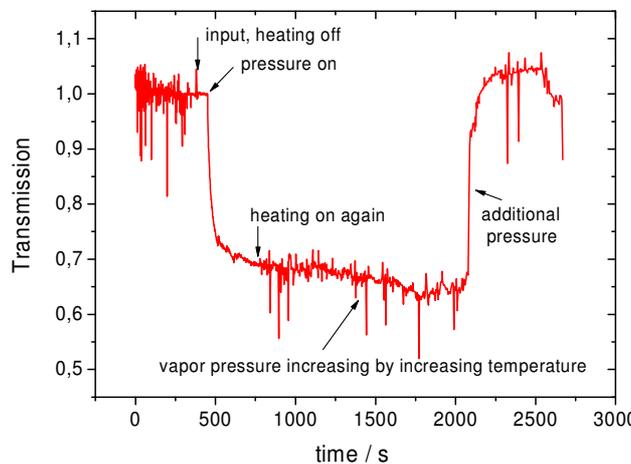


Fig. 11. The 10mg TNT sand (20% TNT+80% sand) sample shows a 40% transmission change. The spikes on the measurement are caused by electric disturbance of the heating control of the desorption unit. This heating was switched off at 300 s in order to have less noisy signal. The heating was switched on again at 750 s. The pressure decreases during the measurement, so an additional pressure step to press all the gas through the fiber. After 2000 s the transmission signal recovers, this shows that the TNT did not condensate

The experiments with the membranes from the automated trace sampling have been shown different results depending on

the contamination level. The measurement campaign includes eight membranes with real contamination background. Four membranes had no or only a low TNT contamination which could not detect. The other four samples show a significant transmission change by 4 % (Fig. 12). The calculation results around a TNT contamination of 600 ng.

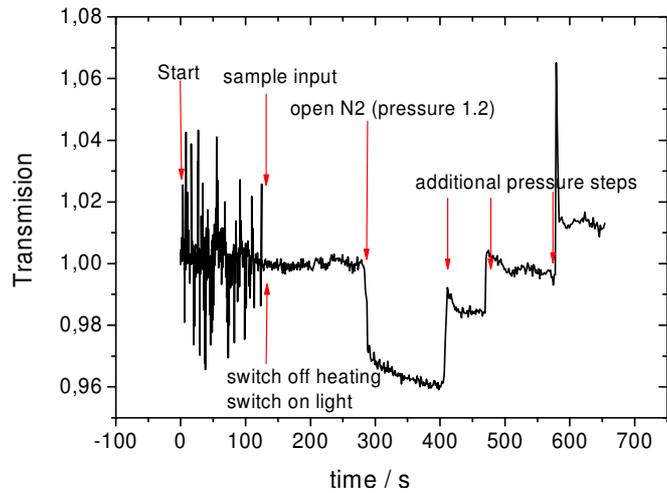


Fig. 12. The graph shows the transient of a measurement of a typical membrane with significant transmission change. At the start the desorbition heating made electronic distribution spikes. The heating is switched off at 150 s, the sample is put in and the desorbition unit lamp is switched on. This lamp has around 30 W and is focused on the membrane. If the lamp is switched on, the membrane is heated up immediately. So the TNT evaporates in several seconds, the pressure hit presses the TNT gas into the fiber. The initial pressure hit was too weak, so an additional stronger hit pushes the TNT gas out off the fiber and the signal recovers completely.

3.2 Open path detection

The changes in the optical transmission expected for detection of TATP vapor are very small. Thus an excellent stability of the laser power and very low noise of the whole signal chain are necessary. By using a single mode DFB laser from Fraunhofer- IAF emitting at 1200 cm^{-1} operating with $100\mu\text{s}$ pulses at a duty cycle of 5% a reasonable stability and noise of the output power was achieved (s. Fig. 13). A relative standard deviation of $1.2 \cdot 10^{-3}$ over 5 min was obtained. The maximum spikes in Fig. 11 amount to $5 \cdot 10^{-3}$.

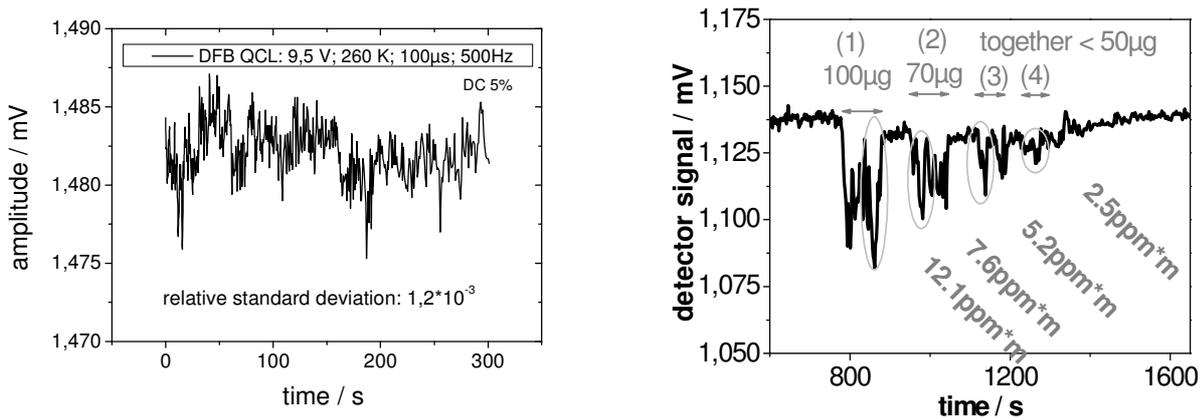


Fig. 13: Laser stability of the QCL-Laser (left) and TATP measurement results using open cell (right).

The result of a TATP measurement with the open cell and a time interval of 10 min is shown in Fig. 13 (right). The TATP grains in the sample holder evaporate subsequently in separate gas bursts. By using spectroscopic data these bursts can be approximately correlated with a certain material mass which is indicated in Fig. 13. The first two bursts correspond to some 100µg material which can be translated to a columnar concentration of 12.1 ppm*m (for a hypothetically 1 m long cell). These first bursts induce a relative signal change of some 4%. The smallest peaks observed correspond to 2.5 ppm*m. After evaporation of all TATP grains the initial signal level was recovered.

As a result open path measurements of TATP columnar concentrations in the 5 ppm*m range is feasible in a laboratory situation. The special dynamics of the TATP evaporation can be observed with fast instruments and may serve as additional information for the detection of the material. In the next step cross sensitivities have to be investigated. However, for realistic standoff- scenarios the expected TATP vapor phase concentrations are often much lower than the currently achieved sensitivity limits of our setup.

3.3 Conclusion

The feasibility of standoff and extractive vapor phase explosive detection of TATP and TNT with mid infrared QCL spectroscopy was investigated. A detection limit of 5 ppm*m for TATP was achieved for open path detection in the laboratory. Due to the broad spectral signatures of TATP and TNT scanning across the characteristic absorption lines by tuning of a single laser was not possible. Thus the detection limit is determined by the intensity noise and drift stability of the QCL. We obtained a relative standard deviation of $1.2 \cdot 10^{-3}$ over 5 min, depending on laser properties and operation parameters.

For long distance standoff scenarios the TATP vapor phase concentrations probably are much lower than the currently obtained detection limits of our setup. However by appropriate beam folding e.g. in an optical portal configuration much higher sensitivities can be expected.

If only very small sample amounts are available, which is the case for extractive trace explosive detection; hollow fibers offer a promising solution. TATP and TNT vapor phase detection was demonstrated with a hollow fiber QCL setup. The key innovation is the use of hollow fibers as compact sampling and gas absorption cell. The small probe gas volume enables compared to classical multipass cells the advantages of fast gas exchange in the sub second range and a sensitivity of TNT and TATP in the nanogram range.

3.4 Further Work

Compared to state of the art systems the proposed QCL spectrometer system can be improved to avoid cross sensitivities by using External cavity Quantum cascade lasers (EC-QCL) or multiple QCL setups. The advantage of the multiple QCLs is a more specific detection of different species can be done because more wavelengths can be covered, so the false alarm could be reduced. The QCLs emits at different wavelength: in the center of two characteristic the absorption bands and one beside. With an EC-QCL the entire spectrum of the absorption band of an explosive can be covered, so the shape of the absorption band could be compared with a data base.

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