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Detection of Liquid Explosives and Flammable Agents in Connection with Terrorism

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Andrey Kuznetsov

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Detection of Liquid Explosives and Flammable Agents in Connection with Terrorism

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Series B: Physics and Biophysics

Detection of Liquid Explosives and Flammable Agents in Connection with Terrorism

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Preface

The organization of an Advanced Research Workshop with the title "Detection and Disposal of Liquid Explosives and Flammable Agents in Connection with Terrorism" was motivated by international findings about activities in this field of application.

This ARW followed a meeting about the "Detection of Disposal Improvised Explosives" (St. Petersburg, 2005). Both items show the logistic problems as one of the lessons, terrorists have to overcome. These problems are connected with the illegal supply and transport of explosives and fuels and as counter-measure the detection of these materials.

The invention of liquid explosives goes back to the middle of the 19th century and was used for special purposes in the commercial field of application. Because of the high sensitivity of liquid explosives against mechanical shock, caused by adiabatic compression of air-bobbles producing "hot spots" as origin of initiation the commercial application was not very successful. Because of this high risk, liquid explosives are not used in military or commercial application with some exceptions.

In the commercial field explosives as slurries or emulsions consisting of suitable salts (Ammoniumnitrate etc.) and water are used to a large extend because of their high insensitivity. In many cases these slurries or emulsions were unfit for terrorist actions, because of their low sensitivity, large critical diameter and using in confinement.

In the military field liquid explosives are used in World War I and II as bomb-fillings.

The large danger of spontaneous detonation was avoided by the storage of two liquids in the bomb, who were not detonable for their own, but create a large power if the liquid are mixed. Only after dropping the bomb the liquids were mixed by a propeller-driven stirrer and than initiated.

In opposition to the tendency of safe handling, experiences have shown, that terrorists ignore to a large extent the danger by handling liquid explosives because of different advantages using liquid explosives: Available commercial product or produced by simple processes, mixing from small portions to large ones and easy to initiate with very high performance.

September 11th has shown also, that beside explosives also flammable fluids reacting with air can be used for catastrophic effects. In military applications these combinations are called "Fuel-Air-Explosives".

In principle liquid explosives can be detected like solid ones, because they contain the same functional groups and are suitable of trace and vapour detection.

Because of handling liquid explosives must be kept in containers of different materials.

Therefore only those detection methods can be applicated which are transparent through the distinct container-wall. These cirumstances reduce the choice of methods to a large extend. If the outside of containers are not absolutely clean, vapour and trace detection can be used, if you have a negative result, you may be not sure if there is explosive material.

For flammable fuels, well known detections methods can be used, if an access to the fluid is possible. There is no detection method known about the composition of the fluid if the liquid is absolutely closed in a container. There is only the possibility to confirm, that there is material in the container.

Co-chairmen
Hiltmar Schubert
Andrey Kuznetsov

Acknowledgement

The international Advanced Research Workshop (ARW) was organized in St. Petersburg by my co-chairman, Dr. Andrey Kuznetsov and his crew from the Khlopin Institute.

I thank my co chair for his initiative to propose and organize this workshop, which was the fourth event in a series of workshops dealing with detection of explosives in connection with terrorism. Special thanks should be given to Dr. Kuznetsov and his crew for the excellent local organization leading to a very successful event.

Thanks also to the director of the Khlopin Institute, Professor Dr. A. Rimski-Korsakov, for his interest and support.

The large international participation of scientists in this upcoming item of detection of liquid explosives has shown the relevance of this item, and I thank all colleagues from NATO and Partner countries for their contributions and discussions.

I thank also the Programme Director of the Collaborate Programmes Section of NATO, Dr. F. Pedrazzini, for the support and interest in this ARW.

Hiltmar Schubert

Contents

The Terrorist Pallet of Liquid Explosives and Flammable Fuels

H. Schubert

Abstract The paper presents a general picture about liquid explosives and flammable fuels starting with the different advantages using these materials for terrorism.

An overview is dealing with the properties of the different substances and mixtures from the chemical point of view including handling, performance and possible methods of detection.

Keywords Liquid explosives, flammable fuels, detection methods

1 Introduction

Explosives as liquids, slurries or as emulsions are well known by experts after nitric acid esters like nitroglycerine are investigated and produced in larger amounts since the second half of the 19th century. In general liquid explosives are much more sensitive against shock than solid comparable substances. (The reason is the possibility of an adiabatic compression of air in the liquid material creating so called “hotspots”.)

Advantages of using explosives in a liquid phase are the simplicity of mixing different components together (binary systems) and to dose or combine into different portions for specific applications. On the other hand to transport, store or handle liquid explosives, containers are necessary. In the commercial industry very often slurry – and emulsion explosives containing mainly suspended inorganic salts like nitrates or chlorates, who are less sensitive, were pumped. Because of the easy mixing of different components together, the pallet of these explosives is large and obscure belonging to detection.

Some assaults of terrorists with liquid explosives have failed in the past, nevertheless intensive research in detection of this kind of explosives will be necessary to counter these terrorist intensions.

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September, 11 has shown that disasters with flame- and pressure waves may be possible by flammable fuels creating by an accident or crash or by means of special additional explosive devices. A special kind of these types are the – so called – fuel air explosives.

2 Components of Liquid Explosives

2.1 *Single Compounds*

In principle liquid explosives have in most cases the same chemical composition like solid explosives, that means they contain functional groups like Nitro-, Nitrato- or Nitramino-Groups.

Detection-methods may be used as we applicate for solid explosives. The difference may be the usage of the explosive material in a container which must be transparent for the measurement. Examples of liquid explosives with a very high performance are:

- Nitric acid esters like Nitroglycerine, Nitroglycol etc.
- Nitro compound like Nitromethane, Nitroethane etc.
- Plasticizers with Nitro- and Fluorin-groups.

Beside these nitrogen compounds, Peroxide-containing compounds are used like Triacetontriperoxide (TATP) and its derivatives.

2.2 *Multiple Systems*

2.2.1 Solutions

Binary or multiple systems are combinations of oxygen containing compounds and fuels in liquid solutions or mixtures to influence the properties (performance and safe handling) of these systems.

Typical mixtures are:

- Nitric acid/Nitromethane
- "PLX": Nitromethane/Ethlyendiamine (5%)
- "Panklastite": Nitrogentetroxide/Nitrobenzene, Dinitrotoluene etc.
- "Bolerone": Nitric acid/Dinitrobenzene, Dinitrotoluene or Cabon-hydrogen fuels
- Solutions with Hydrogenperoxid

Adding phlegmatizers may be possible for additional phlegmatization. For masking, small amounts of additives to change colour, viscosity or odour may be used.

2.2.2 Emulsions and Slurries

These systems have a heterogeneous composition and consist of compounds, which have a high explosive behaviour only in combination. These relative safe explosives were used as slurries or emulsions in commercial applications.

Examples are:

- Astrolite G (Ammoniumnitrate/Hydrazine, composition 2:1)
- Astrolite A (Astrolite G + Aluminiumpulver)
- Kinepak (Ammoniumnitrate/Nitromethane)
- Detagel (Hexamine-nitrate Slurry-Water-Gel) and
- Perchlorate containing mixtures

3 Detection –Methods

In principle all detection methods can be used, which are applied for solid explosives. For details an exchange of information about practical experiences are necessary. The relative high vapour-pressure of most of the Nitrogen containing liquid explosives makes the detection process more easy, if the explosive is accessible.

On the other side we have to take into account, that liquid explosives must be stored or transported in containers of different material, which are different transparent against the various detection-methods.

Like the Nitrogenoxid based explosives also commonly used peroxides like TATP develop vapour pressures and can be detected.

Because liquid explosives have in most cases a high vapour pressure, vapour and trace detection are important, provided that the used containers are not absolutely closed or traces will be found on the container material.

Unlike to these liquid explosives, emulsions and slurries based on water with salts like Ammoniumnitrate, Sodiumnitrate or Ammonium- or Potassiumperchlorates have no vapour pressure and must be only detected by suitable bulk detection methods.

The different detection methods may be distinguished into

- Trace and Vapour Detection and
- Bulk Detection

Methods are for instance:

- Using animal- and artificial noses
- Electromagnetic methods
- Neutron-technology and backscattering
- Nuclear quadropole resonance
- Laser techniques etc.

The detection of flammable liquids gives in principle no problems, because they produce vapour pressure. The transparency of container-material belonging to detection methods is the same with liquid explosives.

4 Application

For terrorist actions, the application of liquid explosives have large advantages, that is the easy supply of the material as commercial products, the separate and therefore safe transport of binary systems and the simplicity of mixing, the combination of different portions and the application of relative low energy for the initiation of the explosive charge down to relative small diameters.

Some disadvantages have to be taken into account:

These are the necessity to transport the material in closed containers, the relative high vapour pressure of the material and the high sensitivity against shock and coarse handling.

Advantages and disadvantages by using emulsions or slurries are different. The manufacture of these materials must be done in special devices, performance will be in the medium range and detonation is in most cases only possible in larger diameters and under confinement by strong initiation. The advantages are the safe handling of these materials.

During the last years we observe an improvement of knowledge in the application of explosives by terrorists. Of course we cannot generalize this statement, because origin and training of terrorists are different. Organized terrorist groups are supported by professionals mainly from the near-east and there will be a danger to be confronted in future with new ideas and improvements of terrorist actions.

Commercial and military explosives must be initiated by detonators, which must be in general produced by specialists. The commercial distribution is limited by law.

The manufacture of detonators is expensive and needs special knowledge. In some cases “home-made detonators” were found and some types of liquid explosives can be ignited without detonation caps. These caps consist of tubes made from aluminium or cupper and filled with primary explosives. The manufacture of primary explosives needs special knowledge and is very dangerous in a home-made process.

Because of the metal tube, detonators are very easy to detect with usual metal detectors. But we know from the land-mine detection, that detonators are commercial available without plastic-tubes.

5 Flammable Fuels

If a flammable fuel is distributed in air and ignited, a gas explosion creating a pressure wave is possible. This phenomenon is used in military application known as “Fuel-Air-Explosives”. To create a fast reaction, a fine distribution of the fuel-particles in air is necessary. This fuel distribution may be produced by an explosive charge or by an accident.

Flammable fuels consist usually of carbonhydrates of different fractions without any functional groups. According to the usage the fuel must have a low ignition temperature and a high vapour pressure to react with oxygen of the surrounding air. Different sensors are commercial available. For the measurement, you have to gather a sample of the fuel. There is no method available to identify a flammable fuel on line of inspection in a closed container from outside. Of course, the container itself can be identified.

A fire in a cabin of an airplane is very dangerous, because the fire consume oxygen, which is limited and produce heat and poisonous Carbonoxyd and Carbondioxyd.

6 Conclusion

The paper presents an overview about the composition of liquid explosives and their combinations including liquids, emulsions and slurries, which may be used by terrorists. From the aspect of terrorist actions the advantages and disadvantages regarding aspects of logistic and application of these explosives are described and detection-methods are mentioned.

Because liquid explosives have to be kept and transported in containers, the wall of the container has to be transparent for the detection methods. For this reason, suitable detection methods are reduced. Trace and vapour detection may be useful, if traces would be present after filling the container.

To detonate a liquid explosive charge a detonator or only an igniter is necessary depending on the composition. Very often the detection of the detonator or igniter may be more simple than the liquid explosive in the container itself.

Flammable fuels in a fine distribution in air caused by an accident or by a primary explosion can produce also pressure waves with severe destructions. If samples are available commercial detection methods can be used, container-walls are not transparent for these methods.

Further improvements of terrorist actions may be expected in the future, therefore research activities for the detection of explosive charges in containers are necessary.

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Overview of Liquid Explosives' Detection

A.V. Kuznetsov and O.I. Osetrov

Abstract Different methods of detection of liquid explosives and flammable agents are described. Feasibility and efficiency of different methods of detecting liquid explosives and flammable liquids in different scenarios are discussed.

Keywords Liquids, explosive, flammable, detection methods

1 Introduction

Attempted terrorists' attacks on planes using liquid explosive in Great Britain in August 2006 dragged attention to the problem of liquid explosives' detection. Such explosives have a number of features, which make them different from other materials. Some liquid explosives have low point of ignition (200–250 °C) and can be set on fire easily, with no need in a traditional detonator or fuse. Their burning may sometimes turn into detonation. Such a device could be detected only if the explosive material itself is recognized.

General overview of methods of improvised explosives' detection was published by the authors earlier [1]. This article discusses applicability of different modern explosives detection methods to the detection of hazardous liquids.

2 Types of Hazardous Liquids

The following types of hazardous liquids may be distinguished: (a) mono-component nitro-explosives; (b) multi-component nitro-explosives; (c) explosive peroxides; (d) flammable liquids.

Many different liquid mono-component nitro-explosives are produced for industrial and military use. For example, nitromethane is used as a solvent, as a component

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of binary liquid explosives and as a rocket fuel; nitroglycerine and ethylene glycol dinitrate are common components of different powders and explosives, and are rarely used as explosive materials on their own. These materials are sensitive to fire, flammable, and under certain conditions their burning turns into detonation. Characteristics of some mono-component nitro-explosives are listed in Table 1 [2, 3].

Multi-component nitro-explosives are multi-component mixtures. One of the components is a liquid oxidizer (nitric acid, dinitrogen tetroxide etc.), and the other one is some organic fuel. While separate, components are not explosive (but may be flammable): they become an explosive liquid only when mixed. Such non-explosive components are safer to transport. That leads to the point that not only explosive liquids should be detected, but also their non-explosive components.

Many constituents of multi-component liquid explosives are widely used for industrial purposes.

- Nitrobenzol is used in production of polymers and different paints.
- Nitric acid is widely used in chemical industry.

Some multi-component nitro-explosives and their characteristics are listed in Table 2 [2, 3].

Peroxides are the compounds with -O-O- group. Explosive peroxides have been used for terrorist purposes, since they are:

- Highly explosive (TATP: ~0.7 of TNT).
- Can be produced in a home lab from many widely available chemical components.
- Production does not take much time.

Table 1 Some mono-component nitro-explosives and their main features

Item	Chemical formula	Density (g/cm^3)	Vapor concentration (ppm)	Z_{eff}
Nitromethane	CH_3NO_2	1.137	36,630	7.033
Tetranitromethane	$C(NO)_2)_4$	1.640	10,900	7.592
Nitroglycerine	$CHONO_2(CH_2ONO_2)_2$	1.595	0.306	7.344
Ethylene glycol dinitrate	$(CH_2ONO_2)_2$	1.489	71.4	7.316
Diethylene glycol dinitrate	$(CH_2CH_2ONO_2)_2O$	1.380	5.1	7.233

Table 2 Some multi-component nitro-explosives and their characteristics

Item	Chemical formula Состав	Z_{eff}
Helhofite	Nitrobenzol ($C_6H_5NO_2$) (28%) and nitric acid (HNO_3) (72%)	7.32
Anergit	Dinitrobenzol ($C_6H_5(NO_2)_2$) (50%) and nitric acid (HNO_3) (50%)	7.22
Panclastit	Dinitrogen tetroxide ($(NO_2)_2$) (65%)2 carbon disulphide (CS_2) (35%)	
KD-mixture	Nitric acid (45%), dichloroethane (30%)2 oleum (25%)	

Table 3 Some explosive peroxides and peroxide nitro-explosives and their characteristics

Item	Chemical formula	Density (g/cm^3)	Z_{eff}
Triacetone triperoxide (TATP)	$C_9H_{18}O_6$	1.0–1.2	6.549
Hexamethylene triperoxide diamine (HMTD)	$C_6H_{12}O_6N_2$	0.6–0.9	6.769
Methyl ethyl ketone peroxide (MEKP)	$C_8H_{18}O_6$	1.0	6.486

Table 4 Different types of flammable liquid (carbon hydrates) and their characteristics

Item	Chemical formula	Density (g/cm^3)	Vapor concentration (ppm)	Z_{eff}
Gasoline	C_7H_{16}	0.76		5.375
Alcohol	C_2H_5OH	0.78	130,000 ppm (40 °C)	6.043
Acetone	C_3H_6O	0.79	234,000 ppm (35 °C)	6.034

Some widely used explosive peroxides and peroxide nitro-explosives and their characteristics are listed in Table 3 [2, 3].

Flammable liquids are widely used for everyday purposes. Their main features are:

- Flammability.
- High volatility and high level of evaporation (thousands ppm).
- Some flammable liquids can be used as components of multi-component liquid explosives (e.g. nitrobenzol).
- Some flammable liquids can be used as components of peroxides (e.g. acetone).

Characteristics of some flammable liquid (carbon hydrates) are listed in Table 4.

Liquid explosives generally have the following characteristics:

- Many liquid explosives have low density (about 1 g/cm^3).
- Usually have high volatility and high level of evaporation.
- High viscosity (similar to that of oil).
- Some have low stability, and cannot be kept for long period of time.
- Some can be made at a home lab out of available chemical materials.
- Some can be easily set on fire with a lighter, their burning may turn into detonation.
- Have Z_{eff} = 6.5–7.5, same as common organic materials.

3 Detection Methods

An explosive device based on liquid explosives may not have traditional fuse and detonator, and this makes their detection a serious problem. Besides that, liquid explosives and their components can be concealed in luggage and on human body.

It is important to have equipment capable of inspecting luggage as well as people. To sort out a suicide bomber it is necessary to have secretly working equipment.

Liquid explosives can be detected by use the following methods:

- Vapor and trace detection
- Electromagnetic methods
- X-rays
- Neutron methods

Vapor and trace detection methods detect vapors and traces of explosives. These methods can have various technical implementations – biosensors, electronic and electro-chemical sensors: ion drift spectroscopy, mass spectroscopy, field ion spectroscopy, thermo-redox, gas chromatography etc. Advantages of vapor and trace detection methods are:

- High selectivity to highly volatile liquid explosives
- Well-known methodology
- Detection of a wide range of commercially-produced explosives
- Can be used on people

Disadvantages of vapor detection are:

- Cannot detect sealed explosives, masking can be used
- Vapors from common materials can cause alarms
- Cannot localize the source and determine mass of explosives

Electromagnetic methods for explosives' detection use electromagnetic radiation with megahertz (nuclear quadruple resonance), tens of gigahertz (microwave radars), 100 GHz–10 THz (terahertz waves) frequencies, and radiation of optical frequency range (Raman spectroscopy etc.). Table 5 shows some advantages and disadvantages of electromagnetic methods.

Table 5 Electromagnetic methods for detection of explosives

Technology	What is detected	Advantages	Problems
Nuclear quadruple resonance	Resonance radiation of NO_2 group	High selectivity to nitrogen, one-side access	Insensitive to peroxides Do not recognize explosive in metal covering
Raman spectroscopy	Vapors and traces of explosives	High selectivity	Cannot detect sealed explosives
Terahertz waves	Transmission spectra	High selectivity	Do not recognize explosive in metal covering
Microwave radars	Dielectric properties	Stand off inspection, selectivity	Do not recognize explosive in metal covering

X-ray methods are widely used for detection of explosives. They allow one to obtain an image of inspected object and determine its density and effective charge. Advantages of X-ray methods are:

- High resolution
- High penetration ability
- Can inspect people
- Selective to some peroxides with high densities and effective charge

At the same time these methods cannot reliably distinguish explosives from other widely used liquids. Canadian company Optosecurity claims to have a method for processing X-ray images, which allows one to determine not only density and Z_{eff}, but also viscosity of inspected liquids. This may help to distinguish liquid explosives from common liquids (water solutions).

Neutron methods Presently the most advanced of all neutron-based methods is Associated Particles Technique (APT)/Nanosecond Neutron Analysis (NNA) [4]. The Inspected object is irradiated with a tagged neutron flux with energy 14 MeV. Secondary radiation, which appears when neutrons interact with the nuclei of the object's material, is detected. This method allows one to detect carbon, oxygen, nitrogen, aluminum, sulfur and many other elements, and to reconstruct the chemical formula of material of the object.

The main advantages of the method are:

- High selectivity
- High penetration ability
- Spatial resolution
- Low background
- Portability
- One-side access

APT/NNA method could be used for detection of nitrogen-containing explosives, as well as for non-nitrogen explosive peroxides. On Fig. 1 one can see a relation of partial densities of carbon, oxygen and nitrogen in different nitrogen-containing materials:

- Explosive (TNT, RDX, black Powder, PETN, tetryl, HMX, Comp-B, C3, C4, ammonium nitrate etc.)
- Non-explosive (wool, silk, dakron, orlon, nylon, cotton, paper, alcohol, oil, barley, soy beans, wood, soap, chocolate etc.)
- Liquid explosives (Nitromethane, Tetranitromethane, Nitroglycerine, Nitroglycol, Diethyleneglycoldinitrate)
- Explosive peroxides (HMTD)

These explosives have the following chemical features, which make them different from non-explosive materials:

- In explosives the mass of oxygen is larger than the mass of carbon.
- In explosives the mass of nitrogen in not less than 20% of oxygen mass.

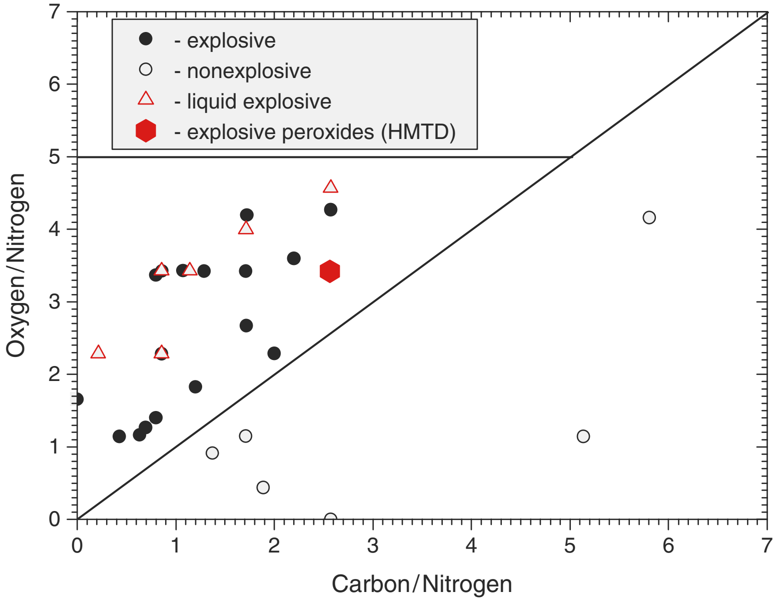

Fig. 1 Relations of partial densities of carbon, oxygen and nitrogen in various nitrogen-containing materials: explosives, non-explosives, liquid explosives, and explosive peroxides

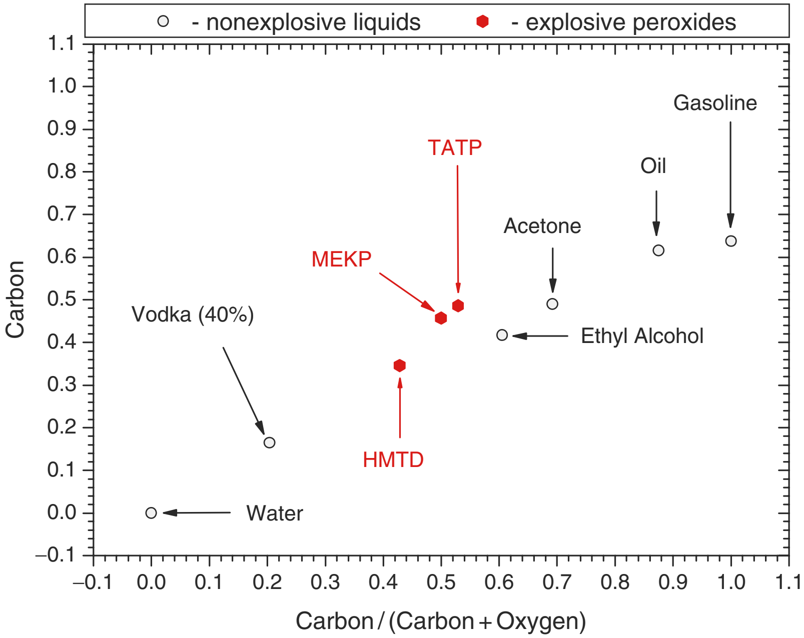

Fig. 2 Partial density of carbon and relation of partial densities of carbon/(carbon + oxygen) for non-explosives and explosive peroxides

Figure 2 shows possibilities to detect non-nitrogen explosive peroxides. It shows partial density of carbon versus relation of partial density of carbon to the one of oxygen and carbon (carbon/(carbon + oxygen)) for non-explosive materials (water, vodka, ethyl alcohol, acetone, oil, gasoline) and explosive peroxides (TATP, HMTD, MEKP). Small values of C/(C + O) correspond to common materials and objects containing a lot of oxidizer (oxygen) and little fuel (carbon). That means that such materials cannot burn or detonate. Explosive organic peroxides have approximately equal amount of oxygen and carbon. Materials with high C/(C + O) contain a lot of fuel and little oxidizer. They cannot explode but burn intensively using atmospheric oxygen. Experimental results on identification of liquids by NNA/APT are presented in [5].

4 Conclusion

1. Unsealed liquid explosives and flammable liquids can be detected by vapor detectors.
2. Sealed mono- and multi-component explosive nitrocompounds can be detected by methods used for detection of solid military and industrial explosives (for example, CT X-Ray) since they have high density, characteristic Z_{eff} and contain NO_2 –groups in their molecules.
3. Sealed peroxides and flammable liquids cannot be detected using methods used for detection of solid military and industrial explosives, since they have low density (~1 g/cm^3), Z_{eff} similar to that of organic materials, and do not contain NO_2 –groups.
4. Sealed peroxides, flammable liquids (in metallic and other boxes or containers) and nitrogen-containing explosives can be detected by APT/NNA neutron method by their C/O/N ratio.

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Chemistry and Properties of Liquid Explosives

P. Mostak

Abstract Liquid explosives are classified in several groups, and groups of liquid explosives characterised. Special attention is given to properties of liquid explosives based on the mixtures of hydrogen peroxide and alcohols and mixtures of hydrogen peroxide with solid fuels. Some specific aspects of explosive behaviour of liquid explosives are discussed. Important procedures for the detection of liquid explosives and limits for detection of these products by detection techniques are considered. Some Countermeasures against misusing of liquid explosives to bomb attacks has been proposed.

Keywords Explosives (Liquids, Suspensions, Emulsions), hydrogenperoxide mixtures

1 Introduction

Liquid explosives are an interesting group of explosive systems. These explosives are in many aspects very similar to solid explosives, on the other hand, they have also some specific properties arising from their physical state.

Several cases of misusing of liquid explosives in terrorists bomb attacks attracted the interest to the question of suitability of these products for this purpose and to efficient countermeasures against IEDs containing liquid explosives.

The evaluation of potential risk led to dramatic measures in the civil aviation transport, which limited substantially the possibility to carry any liquids by persons to cabin of civil planes.

The aim of this contribution is to present the classification of liquid explosives, characterise the main compounds and explosive system and, present the aspects in which liquid explosives differ from solid explosives. The assessment of the detection of liquid explosives, and some possible countermeasures decreasing the risk of misusing of liquid explosives is proposed [1].

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2 Classification of Liquid Explosives

In the beginning, it seems to be reasonable to propose some classification of liquid explosives. As liquid explosive, a broad range of components may be considered, including individual chemical compound having explosive properties, mixtures of such compounds, mixtures of components, which as such have no explosive properties, but as a mixture, they are strong explosives. The liquid explosives can differ in consistency, starting from clear liquids, to suspensions and emulsions and ending by pasty, semi-solid systems.

Another approach is the classification based on the origin of explosives, very interesting is a group of home-made explosives prepared for the use in criminal or terrorist bomb attacks.

Therefore, the liquid explosives should be divided into two main groups:

2.1 Classification in Relation to Chemical Composition and Physical Properties

- Individual liquid explosive compounds
- Mixtures of chemical compounds
- Suspensions
- Emulsions

2.2 Classification According to the Origin of Explosive

- Industrially produced explosives
- Explosive components of industrial explosives
- Home-made explosives

The classification presented above is apparently not the only possible way how differentiate liquid explosives, but it is a logical system, that should be used for the characterisation of these products.

3 Individual Liquid Explosive Compounds

3.1 NG, EGDN

Nitroglycerin and glycol dinitrate are strong explosives, cap sensitive, having the high explosive strength. These explosives are used as components of industrial explosives (dynamites) and smokeless powders. These liquid nitrates are produced industrially in big quantities, but they are not easy to gain and they are very sensitive to mechanical impulses. Nevertheless, nitroglycerin was used in several cases as explosive mini-charge in “brief bombs”.

3.2 Methyl Nitrate

Methyl Nitrate is a strong explosive used in mixtures with some further components as effective explosive system.

3.3 TMEN

Trimethylolethane trinitrate is a strong explosive with the explosive strength 140% TNT, cap sensitive, the procedure for preparation is at disposal in open literature.

3.4 NIBT

Nitroisobutylglycerol trinitrare is a very strong explosive, with the explosive strength 205% TNT, cap sensitive. The preparation of this compound is rather complicated.

3.5 Hydrazonium Nitrate

Hydrazonium nitrate is a very strong explosive having the explosive strength 190% of TNT, cap sensitive. This explosive is produced industrially as Astralit G and with addition of 20% Al as Astralit A, which has even higher explosive strength. Preparation of hydrazonium nitrate from hydrazine and nitric acid is easy. Therefore, hydrazine should be considered as precursor.

3.6 Nitromethane

Nitromethane is not explosive, but is used as industrial chemical for various purposes. Nitromethane can explode only in big quantity and in strong confinement. In combination with some further components, nitromethane is the important part of very strong, cap sensitive explosives. Therefore, nitromethane is an easy accessible precursor for preparation of strong home-made explosives.

3.7 Tetranitromethan, Trinitromethane

Both compounds are strong explosives, their explosive strength can be increased by addition of suitable fuels, usually organic liquid compounds.

3.8 Hydrogen Peroxide

Hydrogen peroxide is not an explosive, but can explode by strong initiation impulse in strong confinement. This compound is precursor for preparation of solid explosive peroxides, or liquid explosive mixtures in combination with organic liquid fuels.

Liquid explosives mentioned above are not a complete list, but only examples of most important products used in technical praxis and of some interesting explosives, which can be rather easily prepared in improvised conditions.

4 Mixtures of Liquid Chemical Compounds

4.1 Nitromethane and Amines

Amines catalyse the explosive reaction, this catalysis results in the higher detonation velocity, detonation pressure, and explosive strength of modified nitromethane. For instance the explosive PLX developed by the Piccatiny Arsenal containing 95% of nitromethane and 5% of diethylenediamine is a strong explosive having the detonation velocity 6,165 m/s.

4.2 Nitromethane and Oxidisers

Mixtures of nitromethane with oxidisers, such as nitric acid or ammonium nitrate enables to achieve a high energy evolved at explosion and also a high detonation velocity and pressure.

4.3 Nitromethane and Some Further Additives

Admixture of Al powder to nitromethane increases the sensitivity and explosive strength of this compound, some further additives should have the similar effect.

4.4 Mixtures of Trinitromethane and Tetranitromethane with Fuels

Such mixtures have usually higher explosive strength and detonation parameters than individual compounds, some mixtures have the detonation velocity near to 7,000 m/s.

4.5 Nitric Acid and Fuels

Mixtures of nitric acid with organic liquid compounds are strong explosives, cap sensitive and they can be prepared very easy from components, which are at disposal on the market. The mixture of nitric acid with nitrobenzene is one of the known mixtures.

4.6 Hydrogen Peroxide and Alcohols

Mixtures of hydrogen peroxide with various alcohols are strong explosives. The explosive properties are strongly dependent on the water content in these systems. Explosives containing hydrogen peroxide and organic fuels will be discussed later in a special part of this contribution.

4.7 General Characteristics of Mixtures

It is typical, that explosive liquid mixtures have usually the high explosive strength. This is caused by the fact, that the mixture of two or more liquid components enables to prepare the system having the optimum composition with the oxygen balance near to zero. Such mixture enables to achieve full oxidation of organic components in which the maximum detonation energy is evolved. More exact definition should be suggested, that the composition, giving the maximum energy in explosive reaction, can be easily prepared.

The liquid consistency of mixtures gives to reacting molecules the intimate contact and enables to achieve the high velocity of explosive reaction and, consequently, high detonation parameters.

The explosive behaviour of mixtures is similar to individual explosive compounds having the same composition. The detonation of most mixtures is characterised by the narrow reaction zone, high detonation velocity and pressure, and by high detonation strength.

5 Explosive Suspensions

Suspension is a mixture of solid and liquid components, which contains various solid explosives and non-explosive components, water is usually the homogenous phase. This class of explosives is often known as "slurry" explosives.

The main components of these explosives are:

- Ammonium Nitrate or other oxidiser
- Explosive or/and sensitising component
- Additives
- Water

The following components can be used as sensitising component:

- TNT or other high explosive
- Smokeless Powder
- Methylamine Nitrate
- Microballons
- Small air bubbles developed by chemical reaction

The broad scale of slurry explosives is produced as commercial products. The cap sensitive slurries, having the critical diameter under 30 mm, are produced in cartridge form. Booster sensitive types were developed for blasting in open pits and quarries, some of them have consistency enabling the filling of boreholes by pumps.

Slurry explosives can be prepared by a simple improvised procedure.

6 Emulsion Explosives

The emulsion is a liquid explosive, in which the homogenous phase is oil, and the water phase, containing ammonium nitrate solution, is emulsified. This explosive is sensitised by microbaloons, expanded perlite, small air bubbles are also used as a sensitising component.

The main components of emulsion explosives are:

- Ammonium nitrate
- Oil
- Emulsifier
- Thickener
- Sensitising component
- Water

Emulsion explosives are industrially produced in big quantities, similarly as slurries with various sensitivity, explosive strength and physical form. The assortment of emulsion explosives starts with cap sensitive, rather stiff explosive in cartridges with the explosive strength near to dynamite and ends with pumpable types characterised by the low sensitivity to initiation impulse destined for blasting works in open pits and quarries. Emulsion explosives are often prepared on the place of use.

They may be prepared very easy by improvised procedure, necessary components can be gained in the open market.

7 Mixtures of Hydrogen Peroxide

7.1 Hydrogen Peroxide – Water

Hydrogen peroxide is able to detonate under heavy confinement and by strong impulse in concentrations 86–100%. At big quantity, (hundreds kilogram) the detonation limit, under conditions mentioned above, is approximately 80%.

Explosive strength of hydrogen peroxide is low, the energy evolved at explosion is only 690 cal/g.

7.2 Hydrogen Peroxide – Water – Alcohols

The properties of the mixture having the composition with balanced equilibrium in relation to complete oxidation of components were studied many years ago in detail [2].

It was found, that the mixture of hydrogen peroxide with ethanol is a cap sensitive explosive the explosive strength is higher than TNT (mini Trauzl test is higher than 45 g/ccm).

Most liquid explosives detonate with two or three different detonation velocities, (individual compounds and mixtures) and these velocities can substantially influence the explosive strength and further detonation characteristics of liquid explosives. Mixtures of hydrogen peroxide – water – alcohol detonate in the two detonation regimes with the detonation velocities 6,800 m/s and 2,200 m/s and, also the third, the very low detonation velocity of 750 m/s was proved.

The detonation velocity is dependent on the water content in the mixture, the high detonation velocity is slowly decreasing with increasing water content and, at approximately 70% the velocity is changed by jump to the low detonation regime. This behaviour is influenced also by further conditions, especially by size of the explosive charge and confinement. The transition points, from high to low detonation velocity is presented in the Table 1.

Table 1 Transition from high to low detonation velocity

Transition point % H_2O_2	Dia of charge mm	Confinement tube wall
70	21	2 mm Al
85	8	1 mm glass
97	4	1 mm glass

The very similar explosive behaviour was found in mixtures of hydrogen peroxide with methanol, ethanol, and glycerol, these changes in detonation velocity are also typical for many other liquid explosives.

The mixture of hydrogen peroxide with glycerol was patented in USA already in 1948 as a system suitable to be used as industrial explosive [3]. Explosive limits of these mixtures are characterised in the three triangular component graph, which is presented in the Fig. 1.

The explosive properties of liquid explosives are very dependent on the composition. Very important is the content of diluent. With raising of this content, the detonation pressure decreases and the failure diameter is increasing.

This dependence estimated at nitromethane-acetone mixture is demonstrated in the Fig. 2 [4].

When the detonation pressure falls under initiation pressure, the mixture loses the ability to detonate.

Interesting results has been achieved in the investigation of A. Schreck et al., in which the thermal explosion and detonation of mixtures containing hydrogen peroxides and different alcohols was studied [5]. The detonation behaviour was similar to results of previous works Substantial differences were found in the development of thermal explosion at some alcohols based apparently on the different mechanism of the alcohol decomposition.

7.3 Hydrogen Peroxide – Water – Solid Fuels

These explosives contain instead of liquid fuel solid organic materials in the form of fine particles dispersed in hydrogen peroxide. Such explosives were patented in the USA in 1962 as cap sensitive explosives for industrial use for similar application as

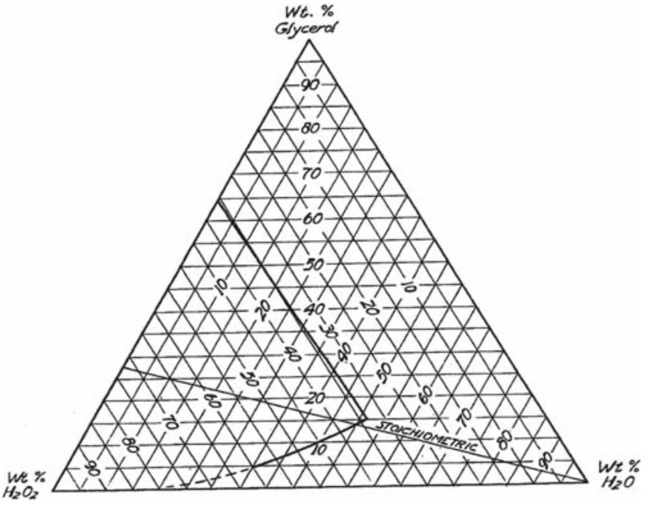

Fig. 1 Detonation limits of the hydrogen peroxide – glycerol – water mixture

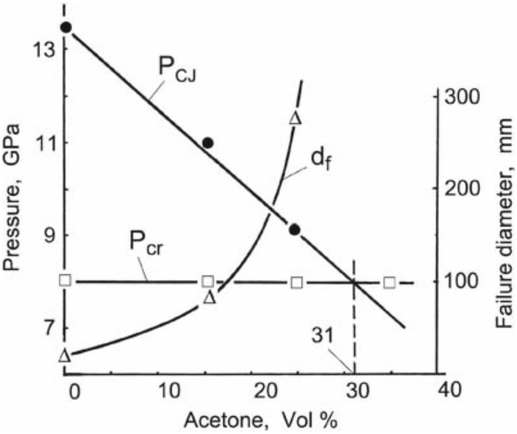

Fig. 2 Dependence of the detonation's pressure (P_{CJ}), failure diameter (d_f) and critical shock pressure (P_{CR}) to initiate the detonation on the concentration in the nitromethane-acetone mixture

are used dynamites and ammonites [6]. The inventors preferred to use the hydrogen peroxide with the concentration between75% and about 90%, but explosives with the concentration of hydrogen peroxide as low as 60% and 70% were also prepared.

Broad range of organic materials was tested as a fuel. For example: fine wood saw dust and wood mill, grain hulls, straw and flours, pits and shells, synthetic resins. The use of flours or starch enables to produce gelatinous consistency of explosive.

The optimum concentration of the fine organic fuel in said explosives was usually in the range of 20–30%.

Hydrogen peroxide should be stabilised in such explosive mixtures by some chemicals as acetanilide, phosphoric acid or phosphates.

8 Liquid Improvised Explosives Based on Hydrogen Peroxide

Explosive mixtures of hydrogen peroxide and liquid or solid organic fuels can be prepared relatively easy from components, which can be gained in the open market.

Mixture of hydrogen peroxide with flour was used in bomb attacks performed unsuccessfully by terrorists in the UK 2 years ago.

Large amount of hydrogen peroxide was found in hands of terrorist group in Germany in 2007.

Properties of these mixtures were studied in the UK and USA, results were published on the 9th ISADE in Paris in 2007.

The improvised explosives based on hydrogen peroxide present a real danger taking into account that the mixtures of hydrogen peroxide are strong explosives, component for preparation are easy at disposal and the procedure of producing such explosives rather simple.

On the other hand, the preparation and use of these explosives is not quite easy as it looks at first sight. The chemical stability of these mixtures is not high and can be impaired by the presence of some impurities. Metals catalyse the decomposition of hydrogen peroxide and this chemical reaction can lead to self-ignition or even explosion of improvised explosives based on mixtures of hydrogen peroxide with liquid or solid organic fuels. Even mild decomposition of explosive, usually accompanied by bad smell, is decreasing the concentration of hydrogen peroxide in the explosive and consequently also explosive properties.

9 Assessment of Liquid Explosives as Component of IED

It is evident, that liquid explosives present a broad scale of products having different compositions, explosive strength and accessibility. Some of them (emulsions, slurries) are produced industrially in hundreds thousands tons/year, some can be prepared easy from products available on the market.

In many aspects, the properties of liquid explosives are near to solid explosives. Differences are in vapour pressure, which is usually higher at liquid explosives. Liquid explosives can be used only in closed containers, which should be significant in detection by imaging technologies.

The liquid explosives are not, in fact, the new threat, but the new threat is the attention of terrorists to these materials.

10 Detection of Liquid Explosives

The detection of liquid explosives is limited by several factors:

- Liquid explosives are filled in tight containers, this lowers the efficiency of vapour detection.
- Some liquid explosives are mixtures without explosive component and therefore, out of focus of standard detection techniques.
- Detection techniques in operational use is not very effective in detection of most liquid explosives.

11 Specific Countermeasures

Liquid explosives require some specific steps in checking procedures:

- Qualified training of personal working in security checking about risk of liquid explosives.
- Careful checking of bottles and other containers with liquids in places of potential risk especially in transport means.

- Control over sales and distribution of most important precursors used in preparation of strong liquid explosives (hydrogen peroxide, hydrazine, nitromethane, concentrated nitric acid).
- Special attention to initiation chains used in IEDs.

12 Conclusions

Liquid explosives present a broad spectrum of products, many of them are produced on industrial scale, liquid mixtures or suspensions offer huge number of combinations with very different properties.

Most of liquid explosives are characterised by high explosive strength, precursors enabling improvised preparation of liquid explosives are accessible in the open market.

Detection technologies in operational use are not effective in some conditions. Therefore, the further research and development of more efficient detection procedures is of imminent importance.

It should be very reasonable to implement adequate control of sales and distribution of some important precursors to complicate the access of terrorists or criminals to components suitable for preparation of improvised liquid explosives.

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What's Special About Liquid Explosives?

J.C. Oxley

Abstract Liquid Explosives do not differ substantially from solid explosives in their density or perfomance. Naturally they are closer to the gaseous state so they are expected to have a strong vapour signature. The unique features of liquid explosives are ascribed to their homogeneity versus inhomogeneous solids. This uniqueness affects their initiation and propagation, but for those concerned with detection of liquid explosives should not be significant. Herein is a discussion of the basic principles of detonation, particularly as it impacts liquids, and a review of various liquids which have been used as explosives.

Keywords Liquid explosives, detonation, homogeneous explosives

1 Basics of Detonation

Since the thwarted English terrorist plot of August 2006, those fighting terrorism have become concerned with liquid explosives. The thrust of this talk is that liquid explosives are no different from solid explosives in aspects related chemistry and performance. However, they do differ from solid explosives in the manner in which they are initiated and in which they propagate detonation. With that in mind, it is appropriate to understand some details about detonation. To undergo detonation, a material must decompose with the release of heat and gas; the latter becomes the working fluid. The reaction is differentiated from a fast burn (deflagration) in the rapidity of the reaction. The power of the detonation is that the energy release is rapid enough to support a shock front. (see Table 1)

Detonation is initiated by heat of shock compression. Energy released from rapid chemical reaction supports the shock wave. Energy loss occurs in the expansion of the detonation gases, mainly at the rear of the charge. Side losses are negligible in

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Table 1 Detonation versus burn

Type of reaction	Reaction rate (m/s)	Energy output (cal/g)	Power output (W/cm^2)
Detonation	7×10^3	$\sim 10^3$	$\sim 10^9$
Deflagration	~1	$\sim 10^3$	$\sim 10^6$
Mild burn	$\sim 10^{-3}$	$\sim 10^3$	$\sim 10^3$
Fuel/air burn	$\sim 10^{-6}$	$\sim 10^4$	~10

large charges, but significant in smaller ones. Detonation velocity decreases linearly with inverse diameter from the theoretical maximum at infinite diameter d_{inf} down to a ***critical*** or ***failure diameter*** (d_{cr}). At d_{cr} side losses are so significant that detonation cannot propagate [1]. While d_{cr} is characteristic of the explosive, it is also dependent on confinement, density, particle size, temperature and impurities. Critical diameter is measured experimentally in conical or stacked hat-box charges. Observation of detonation may be by witness plate, velocity probes, or both. In solids, initiation is by shock compression which collapses the explosive bed, forming a plug. Soaring pressure pulse causes SDT. This mechanism, Shock (S) that Transitions to Detonation (D), is called SDT. In accidents it is often Deflagration that Transits to Detonation (DDT) [2].

In heterogeneous explosives, the high local temperatures necessary for detonation propagation are achieved in hot spots that are linked to porosity, voids, defects, or other low-density regions. When all porosity has been squeezed out of an explosive, such as by the passage of a strong, but non-initiating shock front, then initiating detonation in the material becomes very difficult, if not impossible. This is the origin of "dead pressing" of solids. Liquids differ from solids in being homogeneous, unless bubbles or other inhomogeneities are somehow introduced. If cavities are introduced into the liquid explosive, it becomes much easier to initiate; thus, "don't drop the nitroglycerin" is a reasonable precaution.

2 Similarities and Differences Between Liquid and Solid Explosives

Detonation performance of liquids is similar to solid explosives of comparable density and energy content. They resemble solids because they have comparable densities. This is seen in their detonation velocities plotted against density in Fig. 1. In general, liquid explosives have vapor pressures higher than solid explosives, but this is expected for lower molecular weight materials (Fig. 2).

The unique features of liquid explosives are ascribed to their homogeneity versus inhomogeneous solids. Dremin has reviewed the aspects of liquid detonation. He wrote that liquid explosives differ from solid explosives in the following aspects:

1. The detonation velocity of liquid explosives does not change dramatically with decreasing diameter (it may decrease as little as 1% before critical diameter is reached).

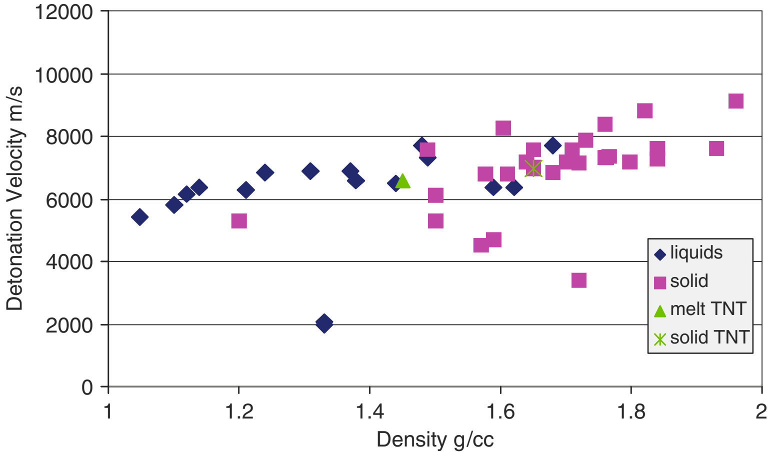

Fig. 1 Detonation velocity as a function of density for liquid and solid explosives

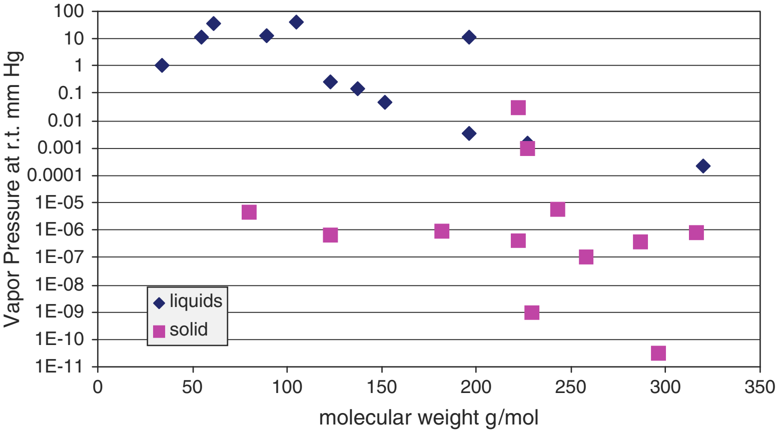

Fig. 2 Vapor pressure as a function of molecular weight for liquid and solid explosives

2. Critical diameter depends strongly on energy content; thus, diluting liquid explosive with inert material dramatically reduces critical diameter, and addition of fuel to an oxidizing liquid explosive dramatically increases performance.
3. Detonation is attenuated transitting from narrow to large confinement [3].

In heterogeneous solids, two mechanisms support wave propagation – hot spots and homogeneous burn. Only the latter exists in homogeneous materials, i.e. liquids; therefore, critical diameter is larger for liquids than solids [1].

Due to their homogeneity, detonation of liquids more closely resembles the detonation of gases rather than solids. Like detonating gaseous mixtures, detonating liquid explosives develop a cellular structure due to transverse waves traveling along the detonation front and reflecting off the side confinement. These are lateral or transfer *compression* waves, not release waves, because side walls are stiffer than shock impedance of detonation. The transverse *compression* waves interact with detonation shock (a two-way interaction) and produce a higher temperature state along trajectory. This strengthens the compression along the front rather than weaken it, as release waves do. When transverse waves moving in opposite directions across the shock front diameter meet, an extra-strong, three-way interaction forms. Then, transverse waves continue in both directions, across the front producing the observed cellular structure. Cell size is determined by reaction kinetics. The most sensitive (reactive) mixtures have smallest cell sizes. The abrupt loss of propagation (d_{cr}) observed for liquid explosives is attributed to loss of the transfer waves interactions [4]. Shock-wave profiles in homogeneous explosives differ from those of heterogeneous explosives. The induction delay in development of detonation behind the shock front in homogeneous explosives produces super-detonation in the shock-compressed region. After overtaking the shock, super-detonation relaxes to the normal C-J detonation; e.g. in nitromethane the initial velocity is 10.4 mm/us but this settles to the C-J velocity of 6.3 mm/us. In heterogenous explosives, the deflagration reaction behind the shock front produces a pressure hump that overtakes and strengthens the shock front. The wave then grows quickly to C-J detonation.

Liquids also exhibit the property of low velocity detonation (LVD). A LVD is a shock wave propagating through a liquid explosive at constant speed just above speed of sound in the liquid. The typically LVD is about 2,000 m/s. High-velocity detonation (HVD) (CJ detonation) occurs at rates between 6,000–8,000 m/s. Both LVD and HVD can occur simultaneous, depending on confinement, charge diameter, and initiation conditions. An LVD can become HVD but not vise versa. A stable LVD can form if the speed of sound in the walls of the container is greater than LVD so that the shock-front can propagate through the walls producing zones of cavitation *ahead* of the LVD. These cavities (vapor bubbles) serve as hot spots to support the LVD.

3 Use of Liquid Explosives

Historically liquid nitrate esters have been used for oil well fracturing (NG); inserting a break in a forest fire; mine field clearance; and liquid landmines. Their common applications today are as plasticizer and as mono- or bi-propellants. Some nitrate esters find application as sensitizers for ammonium nitrate (AN). Kinepak is a commercial product where the user adds nitromethane (CH_3NO_2) to AN. Formulations of AN with hydrazine were patented as Astrolite G (the binary; detonation velocity claimed as 8,600 m/s [5]) and Astrolite A with AN, hydrazine and aluminum (detonation velocity claimed as 7,600 m/s [5]). Although these liquid explosives containing hydrazine are no longer sold commercial; they are described to the home chemist on the Web. A number of the nitrate esters, e.g. NG and EGDN, and even hydrogen peroxide have been used to create a paste or solid explosive by gelatinizing nitrocellulose. These materials usually lack long-term thermal stability and must be used within a few days. The variety of liquid explosives which have been made and tested are shown in Table 2.

Table 2 Properties of some liquid explosives

Liquid explosives	Color	MW (g)	Density (g/cc)	m.p. (C)	b.p. (C)	$\%OB_{CO_2}$	velo. (m/s)	($cm^3/10\,g$)
Diglycerol tetranitrate	Yellow oily	346	1.33	none	d	−18.5	2050	470
TMETN, trimethylolethane trinitrate (MTN)	Cream	255	1.47	−3	182 de	−34.5		400
BTTN 1, 2, 4-butanetriol trinitrate	It yellow	241	1.52			−16.6		
TEGDN triethylene glycol dinitrate	It yellow	240	1.33	−19		−66.7	2000	320
NG, nitroglycerin	Clear-yellow	227	1.48	13	218	3.5	7700	620
DEGDN, diethylene glycol dinitrate	Clear	196	1.38	2	160	−40.8	6600	410
PGDN 1,2 propylene glycol dinitrate	Clear	166	1.37	−43		−28.9	6900	540
EGDN, ethylene glycol dinitrate	Clear-yellow	152	1.49	−23	199	21	7300	600
i-Propyl nitrate	Clear	105	1.05	−82	102	−99	5400	165
Ethyl nitrate	Clear	91	1.10	−102	87.5	−61.5	5800	420
Methyl nitrate	Clear	77	1.21	<20	65	−10.4	6600	610
NM, nitromethane, CH_3NO_2	Clear	61	1.14	−29	101	−39.3	6370	
PLX (95/5 NM/ethylene diamine)	Yellow	61	1.12			−48	6165	400
LX-01 NM/TNM/1 Npropane (52/33/15)	Yellow	95	1.24				6840	
NM/TNMe (55.5/44.5)	Yellow	101	1.31				6880	
Tetranitromethane (TeNMe)	Clear-yellow	196	1.62	14	126	49	6360	66
Trinitomethane (TNMe)	Clear-yellow	151	1.59	26		37	6360	
2-Nitropropane	Clear	89	0.98	−93	120	−135		
NB, nitromethane	It yellow oil	123	1.20	6	210	−163		
2-nitrotoluene	It yellow	137	1.16	−3	222	−181		
H_2O_2	Clear	34	1.44	−2	158	47	6500	
MEKPO	Clear, oily	89				−189		
Anilite N_2O_4 + butane							7600	
N_2O_4 + TeNMe (22.5/77.5)		168	1.68				7700	
Nitric acid + nitrobenzene								
LP 1864 (HAN/TEAN/water) (60.8/19.2/20)	Clear aq. mix	55	1.43		124			
Astrolite G (AN + hydrazine)							6800	
FEFO, $C_5H_6N_4O_{10}F_2$		320		14		−15		

4 Survey of Liquid Explosives

Many of the liquids shown in Table 2 can be explosive alone or, if they are oxidizers, mixed with fuel. Sprengel explosives, used in the 1890s, consisted of mixtures of oxidizers, such as nitric acid or nitrogen tetroxide, and fuel (e.g. nitrobenzene, CS_2, petroleum). Some of these formulations had special names, such as Acenina for nitric acid with acetonitile or Dithekite and Hellhofite for various ratios of nitric acid and nitrobenzene or Oxonite for nitric acid with picric acid [6]. Panclastites used nitrogen tetroxide (N_2O_4), the condensed form of nitrogen dioxide. Nitrogen tetroxide (N_2O_4) is liquid over the range (-11–21 °C):

$$N_2O_4 \text{ (clear liquid)} \Leftrightarrow NO_2 \text{ (orangish gas)}$$

Explosives made of nitrogen tetroxide and combustible liquids, such as carbon disulfide, nitrobenzene, nitrotoluene, benzene, gasoline, kerosene, halogenated hydrocarbons, were studied for weapon use as early as the 1880s [6, 7]. The formulations were inexpensive and easy to prepare, but their extreme sensitivity dictated they be mixed just prior to use. The corrosivity of N_2O_4 required special vessels. Some Panclastites performed better than TNT, (e.g. 6,900 m/s for N_2O_4 with nitromethane). Bombs containing N_2O_4 and CS_2 in separate compartments would explode on impact with no fuze required.

The ultimate oxidizer is oxygen. Shortly after Linde developed a method to liquefy gas (1895), liquid oxygen explosives (LOX) were invented. During World War I, the Germans used LOX when supplies of other explosives ran low. LOX found limited application for commercial rock blasting from 1926 into 1960s. These explosives were made by saturating porous combustible materials, e.g. lampblack, with liquid O_2. Liquid O_2 with carbon black is reported to have a detonation velocity of 3,000 m/s; and with acetylene (1/3) a detonation velocity of 6,000 m/s (cf.

Table 3 Exemplary mono- and bi-propellants (From Encyclopedia of Explosives [6])

Mono-propellants
H_2O_2, N_2H_4, CH_3NO_2, CH_3ONO_2 in ethanol $(CH_3)_2N_2H_4$

Components of bi-propellants
H_2O_2, HNO_3, $HCIO_4$, $CH(NO_2)_2$, N_2O_4, CIF_3

Cryogenic oxidizers
F_2, O_3, N_2F_4, CIF_3, N_2F_4, BrF_5, CIO_3F, F_2O, F_2O_2

Formulation
LOX/EtOH; LOX/kerosene; LOX/hydrazine;
  N_2O_4/N_2H_4
  H_2O_2 + hydrazine hydrate (hypergolic)
  nitric acid + aniline
  nitric acid + turpentine, NH_3, or CH_3CH_2OH
  $CH_3NHNH_2 + N_2O_4$
  $N_2H_4 + F_2$
  N_2H_4 catalytic decomposed in thrusters by Ir, Rh, Ru
Otto fuel = propylene glycol dinitrate (77%) + dibutyl sebacate (23%) + 2-nitro-DPA

solid acetylene = 2,270 m/s). There were two major problems that limited the use of LOX. As the oxygen warmed above its boiling point (−183 °C), the materials lost their explosiveness, and they presented an extreme fire hazards. Liquid oxygen, nitric acid, nitrogen tetroxide and more exotic oxidizers have found use over the years in liquid rocket motors and thrusters (see Table 3) [6].

5 Nitrate Esters

The first practical high explosive made was the liquid explosive nitroglycerin (NG). Making nitrate esters is relatively easy. Any alcohol can be nitrated by the slow addition of mixed acid – nitric acid with sulfuric acid as a dehydrating agent – this technique allows the straightforward preparation of a host of nitrate esters, **if** the reaction is kept cold during nitration and **if** excess acid is carefully removed from the product. The synthesis of methyl nitrate ester is shown.

$$CH_3OH + HNO_3/H_2SO_4 \rightarrow CH_3ONO_2$$

If the starting alcohol is a liquid, the nitrate ester produced is likely to be liquid. It would be difficult to list all the nitrate esters which can be made. Common ones include nitroglycerin (NG), isopropyl nitrate (IPN) $(CH_3)_2CH\text{-}ONO_2$, ethylene glycol dinitrate $O_2NOCH_2CH_2ONO_2$ and methyl nitrate ester. Liquid explosive can be as powerful as solid explosives. For example, tetranitromethane mixed 2-to-1 with benzene is reported to have TNT equivalence of 134%; and an oxygen-balanced mixture with toluene has a detonation velocity 8,000 m/s.

In 1994 Ramiz Yousef, master mind of the World Trade Center bombing in 1993, developed a new plot, the so-called Bojinka Plot. It involved three parts:

Assassinating Pope John Paul II during visit to Philippines on January 15.

Flying a private plane with explosives into CIA headquarters in Virginia, USA.

Bombing of 11 airliners flying between Asia and the U.S. on January 21 and 22.

The first device was tested in a mall in Cebu City; there was minor damage. The second was placed in a movie theater in Manila. On December 11, components of a bomb were smuggled aboard Philippines Airline Flight 434 from Manila to Tokyo via Cebu. The device was assembled on the first leg of the journey, and the terrorist deplane at Cebu. The device was too small to take down the plane, but one Japanese passenger was killed, and ten were injured. Yousef's associates were arrested. In that plot the threat was believed to be nitroglycerin; at the time, there were no fielded methods to detect nitroglycerin.

6 Nitromethane

There are several liquid explosives which are commonly used by legitimate industry – nitromethane, methyl ethyl ketone peroxide (MEKPO), and hydrogen peroxide. Nitromethane (NM) is a colorless liquid with boiling point of 101 °C, a TNT equivalence of ~1.1, and a detonation velocity of ~6,000 m/s (metal confinement). It was first prepared 1874 via the reaction of methyl iodide and silver nitrite [8]. Present-day industrial preparation creates nitromethane by the high temperature reaction of methane and nitric acid. After two large tank car accidents in the 1950s, bulk shipments of nitromethane were forbidden in the USA. Nitromethane requires sensitization to initiate it to detonation, but sensitization can be by addition of commercially purchased glass microballoons or a variety of acids or bases. Often diethylene tetramine (DETA) is added at the 5% level; this mixture, called PLX (Picatinny Liquid Explosive), is the red-orange color of DETA. Use of PLX is suspected in the bombing and disappearance of KAL flight 454 from Baghdad to Bangkok via Abu Dhabi (November 29, 1987). In that case, the terrorist device was in carry-on baggage–a radio containing the initiating system and 350 g of C4 and a liquor bottle containing PLX or methyl nitrate ester. Two terrorist planted the bomb and deplaned in Abu Dhabi; the plane with 115 people on board disappeared.

7 Liquid Peroxide Explosives

Peroxides are used industrially primarily because they readily decompose, but this decomposition can be violent, especially if the peroxide is in contact with a fuel. For example, H_2O_2 violent decomposes to water and oxygen with catalytic amounts of lead, silver, manganese or saliva. Pure H_2O_2 decomposes violently above 80 °C and is considered detonable above 85% concentration. A number of hydrogen peroxide explosives and propellants have been reported and some patented (Table 4). (One patented formulation, H_2O_2 with hexamethylenetetramine and HCl, probably makes HMTD, a highly sensitive white solid.)

Recipes on the Internet claim that dilute solutions of hydrogen peroxide can be concentrated simply by heating on the stove, though this is likely to be an operation of some hazard. Hydrogen peroxide of concentrations capable of detonating (85% or greater) are essentially unavailable except for military use. There are a few uses for 70% hydrogen peroxide and more of 50%, but the concentration most widely available to the public, at least in the United States, is 35% for spa cleaning or much lower 6% for hair bleaching or 3% for general disinfectant or mouthwash. Table 5 lists uses of hydrogen peroxide.

The use of H_2O_2 by terrorists has been somewhat confused by the media, and this may not be bad. There are three ways it can be illicitly use:

Table 4 Some H_2O_2 explosive formulations [9]

Formulation	Comments
H_2O_2 (90.7%)	5,500–6,000 m/s
H_2O_2 (35 mol%) in air	6,700 m/s
H_2O_2/ethanol/water	6,700 m/s
H_2O_2 (60%) + paraformaldehyde	Sensitive crystals
H_2O_2 (70%) + diesel + gel agent	Explosive
H_2O_2 (83%) + cellulose	More powerful than TNT

Table 5 Legitimate uses of hydrogen peroxide [10]

Bleach & Decolorizing	Bactericide	Chemistry	Oxidations
Household	Contact lense sterilization	Benzoyl peroxide Alcohol	
Animal feed	Cleaning semiconductors	MEK peroxide synthesis	Amines
Food fibers	Cleanser	Catalyst regeneration	Cyanides
Hair bleaching	Drill mud treatment	Cellulose delignification	Hydrazine
Bone bleaching	Disinfecting	Chlorine reduction	Hydroxylamine
Cotton bleching	Mouthwash	Chemical lazers	Mercaptan
Pulp bleaching	Oral sore medication	Co/Ni separation	Methanol
Detergents	Pool chemicals	Desulfurization	Phenol
De-inking		Epoxidation	Vat dye
Tooth bleaching	**Environmental**	Ester manufacturing	Vanadium
Tooth paste	Air detoxification	Emulsion polymerization	
Tobacco bleach	BOD reduction	Formaldehyde detox	**Miscellaneous**
Wood bleach	Bioleachant	Gold & silver leaching	Propellant
Yam bleach	Bioremediation	Copper etching	Corrosion inhibitor
Decolorizing sugar syrup	COD removal	Peracids	Etchant metals Cu
Decolorizing sulfruic acid	Deodorizing	Polymer initiaion	Etchant circuit boards
	Odor removal	Uranium purification	Fabric desizing
	Sour water treatment	Soybean oil expoxidation	
	TOC removed from H_2SO_4	Vulcanization accelerant	
		Vanillin production	

1. Above 80% pure H_2O_2
2. In the 50–80% concentration range with a fuel
3. As a reagent to make the primary explosives TATP or HMTD

The fuel which is mixed with hydrogen peroxide is often a miscible liquid. Generally chosen are liquids which are not readily oxidized by peroxide, typically an alcohol, glycol or hydrocarbon. Solid fuels such cellulose can be used, but these result in pastes or solids. In all instants, these hydrogen peroxide/fuel mixtures must be regarded as make-and-shoot formulations; they do not possess long-term thermal stability. The British forensic lab report that the explosives found in the attacks of July 21, 2005 were bubbling, yellowish pastes that had to be destroyed in place [9].

Terrorist use of hydrogen peroxide came to public attention in August of 2006 when passengers could no longer carry liquids onto planes. However, terrorists attempted to use liquid hydrogen peroxide as early as March of 2004 in Karachi when 750 l were intercepted outside the U.S. embassy. Since then caches have been found in Jordon (2004); England (2005 and 2006) and Germany (September 2007, 1,5001). This is, indeed, a difficult material to countermeasure.

8 TATP

To make TATP the ingredients acetone and hydrogen peroxide and a catalytic amount of some acid (type not critical) are required. The concentration of H_2O_2 affects the yield of the reaction, as noted on the Web, but otherwise peroxide concentration is not critical. The Web instructions advise the home-chemist to boil away some of the water from the hydrogen peroxide to increase its concentration and the yield of TATP. The white solid, which precipitates from the reaction of hydrogen peroxide and acetone, is a mixture of the trimer (TATP) and the dimer (DADP). The Web notes caution again making DADP, rightly noting that its production can be discouraged by keeping the reaction mixture cold. However, in practice, the DADP appears to be as an effective explosive as TATP and not any more sensitive; thus, having a mixture of the two is not a matter of concern. It is often said that TATP is unstable. Its thermal stability is as good as most organic materials of its melting point (94–95 °C). It is more sensitive to impact that PETN, but not markedly so. [It should be noted that HMTD (hexamethylene diamine triperoxide) is made in a similar fashion substituting hexamethylene tetramine (hexamine) in place of acetone. However, HMTD does not perform as well as TATP and is much more sensitive.] The negative feature of TATP, in terms of handling, is

Table 6 Terrorist use of TATP up to 2001

Incident	TATP/HMTD	Year	Location	Size
Israel has experienced the largest bombs				
UX pipe bomb	T	1980	Israel	Pipe
X & UX pipe bomb	T	1982	Israel	1 kg
Clandestine lab	T	1998	Israel	100’s kg
US had 12 incidents, 9 during bomb prep., 13 injured, 16 recoveri				
UX blasting cap	T	1983	CA	Cap
Blast in prep of pb	T	1989	MD	Initiator
UX pipe bombs (77)	T/H		CO	Initiators
Powder	T	1990	FL	16 g
Recovered from car	H	1999	WA	17 g
England has had about the same level of use as USA				
UX devices & ingredient	T	1995	London	2 devices
Chlorate car bomb	T?	1999	London	Initiator
Ingredients & devices	H	2000	England	
Australia had 6 incidents all pipe bombs, half with juveniles				
Others				
Philippine Airline bomb	T?	1994	Manila to Tokyo	Initiator?
Recovered from apartme	T	1995	Manila	1 kg
American Airline (shoel	T	2001	Paris to Miami	Initiator (1 g)

Table 7 Large-scale terrorist bombings

Year	Location	Type of explosive	Injured	Dead
1983	Beirut Marine & French Barracks	2 trucks, 12K Ib C4?		300
1988	Pan Am 103, Lockerbie Scotland	Semtex RDX/PETN		269
1992	St Mary’s Axe/Docklands, London	1,000’s Ib AN icing sugar		3
1993	World Trade Center, NY	1,200 Ib, urea nitrate	~1,000	6
1993	Bombay 13 car & scooter bombs	RDX?	~1,200	317
1993	Bishops Gate, London	3,000 Ib AN/icing sugar	40	1
1995	Oklahoma City Federal building	5,000 Ib ANFO	~1,000	168
1996	Canary Wharf/Docklands London	3,000 Ib AN/icing sugar	39	0
1996	Manchester, UK	1,000’s Ib AN/icing sugar	~200	0
1996	Khobar Towers, Saudi Arabia	0.5–30 K Ib C4?	372	19
1998	Kenya & Tanzania	2,000 Ib TNT & PETN?	1,000s	224
2000	U.S.S. Cole, Yeman	1,000 Ib TNT?	39	17
2002	Limburg oil tanker	TNT?	12	1
2002	Bali nightclub bombs	Chlorate	209	202
2003	Istanbul, Turkey	2 bombs	450	28
2004	Madrid subway, 10 suicide bombs	gelignite in 4 locations	~600	191
2005	London subway, 4 suicide bombs	Peroxide explosive	~700	56
2006	Mumbai, India railroad	7 explosions	625	190

* PIRA bombs targeted economic loss rather than humanloss; warnings were issued

that it readily sublimes and has a vapor pressure at room temperature between 0.03 torr [11]. It is always hazardous to have an explosive that can unexpectedly move into joints and valves. Use of TATP also makes forensics difficult, but it is a feature which can be overcome by proper sealing of evidence. That this in known to terrorists can be seen in the pictures of confiscated material from the July 2005 bombings in England. TATP is not a new chemical; it was investigated by the U.S. Army in the 1950s and first appeared on the terrorist scene in the 1980s in Israel (Table 6).

Initially, Palestinian terrorists used TATP to make large bombs, but it was quickly realized that this was too risky an operation even for terrorists. However, TATP remains popular with terrorists because it is the easiest route to a primary explosive. Primary explosives are essential to initiate the secondary explosives, the main charge. Typical, primary explosives, such as lead azide and mercury fulminate, are not easy to synthesize. TATP offers the easiest and cleanest route to a primary explosive for illicit use.

9 Conclusion

What are terrorists likely to use? It depends on availability of precursors and terrorist know-how. In 1993, urea nitrate was used in the World Trade Center bombing in New York City, even though ammonium nitrate was widely available and under no controls. Terrorist camps had trained on the synthesis and use of urea nitrate. Thus, we should examine previous use to assess the question of availability of chemical precursors and knowledge. Table 7 reports large-scale terrorist bombings.

The caveat "large-scale" has been added since, typically, a pipe bomb or suicide vest kills no more people than a rifle.

Using Table 7 as reference, we see that control of six chemical precursors (three of them liquids) should provide control most of the current threats: ammonium nitrate; nitric acid; urea; chlorate salts; hydrogen peroxide; and nitromethane. This list is far from exhaustive, and it leaves two important questions unanswered: What concentration is hazardous? What amount is a security risk? Controlling, not banning, the six-chemical threat list would go a long way to preventing illicit preparation of explosives.

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Search of Explosives in Vehicles by Using Tagged Neutrons

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Abstract The search for explosives hidden in vehicles is one of the tasks of counterterrorism application. This task requires the detection of explosive material in car or truck bombs as well as illicit transport of such items. Liquid explosives, especially home made (HME) ones, have been used in terrorist attacks and are thus of great importance in this field. The detection of HME liquid explosives in trucks by using a neutron-in/gamma-out system as the EURITRACK portal is discussed. Moreover the possibility of material recognition by transmission measurements with a tagged ^{252}Cf source is also presented. The latter system might find application in car inspections or in small parcel scanning systems.

Keywords Fast neutron inspection, ^{252}Cf source, transmission measurement, explosive detection, liquid explosives, home made explosives

1 Introduction

Non-intrusive inspection of cars and trucks is a key issue in the fight against terrorism. Moreover, liquid explosives of the home made (HME) type have been employed in several bombing around the world. As a consequence, there is need of detecting HME that can be hidden in cars and trucks and then used as Vehicle Born Improvised Explosive Devices by terrorists. In such case there is certainly no need of masking the explosive contained in a car or truck bomb, especially when the vehicle is driven by a suicide commando. However, in some cases, the transport form one location to another inside a country or from a country to another might be an issue. In this case, masking of illicitly transported explosives is an important fact. As a further complication of the

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problem, precursors employed in the preparation of home made liquid explosives might be illicitly transported from a location to another to prepare attacks.

Liquid explosives (LE) are detectable by using different techniques. In some cases, when LE are transported in metallic containers, one needs to perform analysis of the bulk material using probes effective in penetrating such containers (as is the case of a tanker or in the trunk of a car). In this case X-rays or neutrons are among the possible candidates. Explosives are detected in airports using systems based on advanced X-ray technologies (as dual-energy, diffraction or tomography). The effectiveness of such systems to detect HME remains to be demonstrated. Moreover, material recognition capability of X-ray based techniques is in some case impaired when the photon energy is increased in order to obtain better images in dense and thick objects as is the case of large vehicles and trucks. In such conditions, the exploitation of different technologies, such as the use of neutrons, is an interesting alternative.

Within the proposed neutron technologies, the Tagged Neutron Inspection System (TNIS) concept which uses the neutron source $T(D,n)^4He$ reaction has undergone lab testing in the last few years [1–3]. Moreover the EURITRACK project, funded within the 6th Framework Programme of EU, developed a second generation TNIS for inspection of cargo containers [4]. In this system the tagged neutron probe is used in cascade with a conventional X-ray scanner that determines precisely the position of a suspect volume unit (voxel) inside the cargo. Only such voxels are then inspected with neutron beams, thus minimizing the radiation hazard.

In the first part of this paper we will discuss the possibility of detecting HME (with special emphasis on liquids) in large vehicles as trucks by using the EURITRACK portal. In the second part, the possibility of material recognition by using a simple system based on a tagged ^{252}Cf source will be explored. The latter should be useful in searching small vehicles as cars or small parcels.

2 Detecting Liquid Explosives with a Tagged Neutron Portal

The EURITRACK portal is fully described in reference [4]. In the following, the relevant features of this system are shortly presented. A compact EADS-Sodern sealed-tube neutron generator includes a 8 × 8 matrix of YAP:Ce alpha particle detectors coupled to a multi-anode H8500 photomultiplier [5]. Twenty-two high-efficiency 5″ × 5″ and 5″ × 5″ × 10″ NaI(Tl) detectors from Saint-Gobain Crystals & Detectors are located around the cargo container to detect the neutron-induced gamma-rays. The NaI(Tl) detectors are grouped in three arrays mounted on mobile platforms. Neutron attenuation across the container is measured with a 5″ × 5″ BC501A liquid scintillator detector. All scintillation detectors are equipped with fast photomultiplier tubes to achieve nanosecond time resolution [6]. A dedicated VME front-end electronics from CAEN is used to verify coincidences between any alpha and gamma-ray detectors. Coincidence events are then stored on disk for on-line data analysis [7]. The EURITRACK Information System allows managing all phases of the inspection with the TNIS, using as input the information from the X-ray image in which the suspect voxel has been identified [8].

The partners of the EURITRACK project have developed and tested the TNIS components and sub-systems during the years 2005–2006. Their integration was first performed at Institute Ruder Boskovic (IRB) in Zagreb, Croatia, where a first run of functionality and detection tests was performed. Functionality tests were completed at CEA Saclay, France, where the TNIS active components were assembled on the inspection portal structure. During 2007, the portal was then installed and commissioned at the Port of Rijeka, Croatia, for the final demonstration campaign.

It is important to stress that during the R&D phase, the design and expected performances of the system were continuously studied using Monte Carlo simulation employing the MCNP program [9].

Tests performed in laboratory conditions showed that the TNIS can detect, in 10 min, 100 kg of TNT hidden in a container fully filled with iron freight of 0.2 g/cm^3 mean density, which was defined as the reference case of the EURITRACK project.

The capability of the EURITRACK portal in detecting LE and HME has been studied by Monte Carlo simulations using the same MCNP program that had been validated during the EURITRACK project. The cases of hydrogen peroxide, ANFO and TATP were considered, placing about 100 kg of material at the centre of a 20′ container.

The simulated gamma ray energy spectrum of pure hydrogen peroxide is presented in Fig. 1. In case of oxygen peroxide based explosives, it is mandatory to discriminate such chemicals from common use materials based on water. Discrimination capability of the EURITRACK system between samples of H_2O_2 and H_2O was studied using equal volumes (40 × 40 × 45 cm^3) filled with the two liquids and measuring both the $E_\gamma = 6.1$ MeV yield from oxygen nuclei and the number of 14 MeV transmitted neutrons, the latter being mainly determined by the density of the sample. The simulation results testify the sensitivity of the present technique: the oxygen gamma rays yield increases by about 20% and the neutron transmission decreases by 25% going from normal water to H_2O_2. This proves that a vehicle loaded with peroxide based explosive (truck bomb) can be identified measuring the two quantities described above by means of a tagged neutron portal such as the EURITRACK system.

In case the liquid explosive is hidden among mixed goods, the advantage of a tagged neutron system consists in exploring a single voxel inside the truck volume, thus providing the possibility to measure the oxygen density of the liquid alone. This would allow discriminating between explosives and common materials in containers with miscellaneous content. Transmission measurement seems to be meaningless in this case.

The simulated spectrum from a TATP sample is also presented in Fig. 1, showing the characteristic gamma rays from ^{12}C ($E_\gamma = 4.4$ MeV) and ^{16}O ($E_\gamma = 6.1$ MeV). The chemical formula of TATP is $C_6H_{12}O_4$ with a C/O ratio very close to a variety

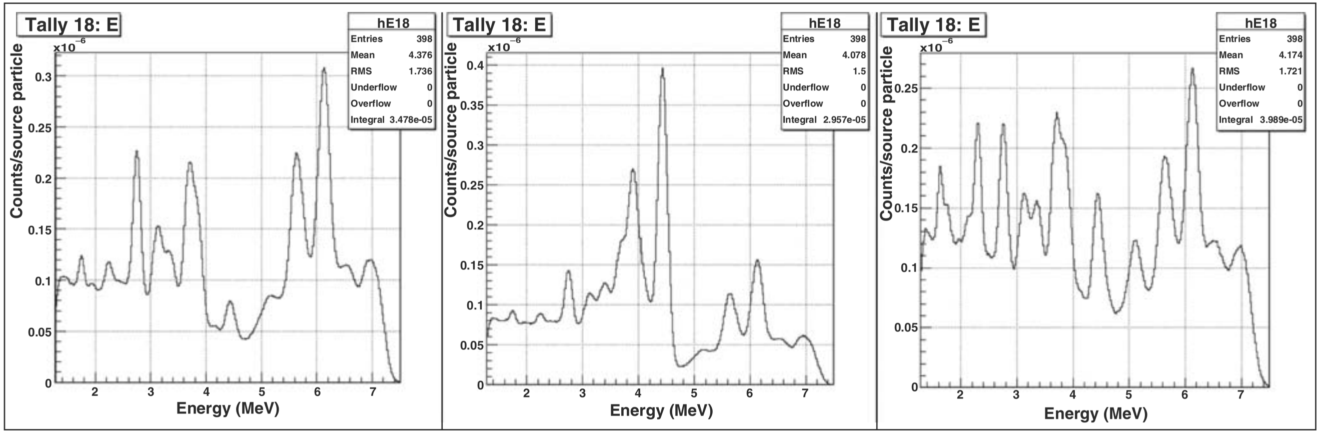

Fig. 1 Simulated gamma ray spectrum from H_2O_2 (left), TATP (center) and ANFO (right)

of common materials. Consequently, it is rather difficult to discriminate clearly TATP using neutron probes which determine only the C/O ratio, when the presence of other common use material can't be a priory excluded.

The case of ANFO is also illustrated in Fig. 1. We used a 95% ammonium nitrate 5% fuel oil mixture characterized by average elemental ratios C/O = 0.10 and C/N = 0.15. In this case discrimination between ANFO and other materials is relatively simple.

3 Material Recognition Using a ^{252}CF Source

As discussed in the previous section, LE and HME can be identified using tagged neutron techniques as implemented in the EURITRACK portal for large volume vehicles. Such technique can be also applied to design smaller inspection systems for cars. In addition to the Tagged Neutron technique using the neutron-in/gamma-out scheme, other methods based on transmission measurements have been recently proposed [10, 11]. One interesting case is the scanner designed by CSIRO and now in operation at the Australian Brisbane airport [12]. Such system performs material recognition making transmission measurement of 14 MeV neutrons (from a D + T generator) and gamma rays from an intense ^{60}Co radioactive source for each pixel of a detector array. The scanner produces a 2-D radiographic image in false colours in which each pixel brings material information. Limits to the system are due to the fact that measured quantities are averaged over all the materials crossed by the radiation. In order to optimize the material recognition identifying single elements in the sample, it has been proposed to measure neutron transmission with different neutron energies and looking to resonances in the total cross section for a number of light elements [10]. Such technique should provide element identification as in the case of the neutron-in/gamma-out techniques.

We have recently explored the possibility of performing material recognition using neutron and gamma-ray transmission measurements, as in the CSIRO case, but employing a tagged ^{252}Cf source. Despite the large difference in the radiation flux of a typical source compared to a neutron generator, ^{252}Cf produces in the fission decay wide energy neutron and gamma-ray spectra that are effective in material recognition. Neutron/gamma discrimination and neutron energy are obtained by measuring the time-of-flight with fast scintillators. In addition to the transmission measurements, the wide neutron spectrum can be used to determine the presence of a given light nucleus (C,N,O) in the sample by looking at the resonances. Moreover, the wide energy distribution for gamma and neutrons emitted from the ^{252}Cf source allows measuring the transmission of the two kinds of radiations with cuts in energy, thus resembling the "dual energy" method often used in X-ray radiography.

The experimental set-up employed for the proof-of-principle of this technique is rather simple: a 10^6 fissions/s ^{252}Cf source was tagged using two 2″ × 4″ liquid NE213 scintillator cells mounted in a close geometry around the source to detect neutrons and gammas emitted in the fission events. In this way the tagging efficiency was almost 100%. The tagged radiation was collimated by a 40 cm long lead-polyethylene collimator. The samples were placed at the exit of the collimator. Finally, a 2″ × 4″ NE213 liquid scintillator cell was placed at a distance of about 160 cm from the tagged ^{252}Cf source. The time resolution of the time-of-flight system was about $\delta t = 2$ ns [FWHM]. A large number of samples have been employed, ranging from low Z (plastics, carbon) to lead. In the data analysis, the measured quantity is the ratio

$$R = \mu_n / \mu_\gamma = \ln(I_n / I_{n,0}) / \ln(I_\gamma / I_{\gamma,0})$$

deduced from the transmission measurements with gamma ray and neutrons. It is worth noting that R does not depend on the sample thickness. Measured R values are reported in Fig. 2 for a large number of pure elemental and mixed samples. It is clear that the R value is varying by more than a factor 10 from low Z to high Z samples. Of special interest for the detection of liquid explosives is the fact that the measured R value from a water sample is R = 6.9, whereas a commercial H_2O_2-H_2O mixture (3% in H_2O_2) is characterized by R = 6.0. This demonstrates the possibility of discriminating between the two liquids.

The very good material recognition obtained by such measurements can be further implemented by looking directly at the transmission as a function of neutron time-of-flight (and thus as a function of the energy). It is well known that the total neutron cross section of some light elements exhibits a well defined resonance structure in the energy range $E_n = 0.1$–5 MeV, characterizing the ^{252}Cf source. This

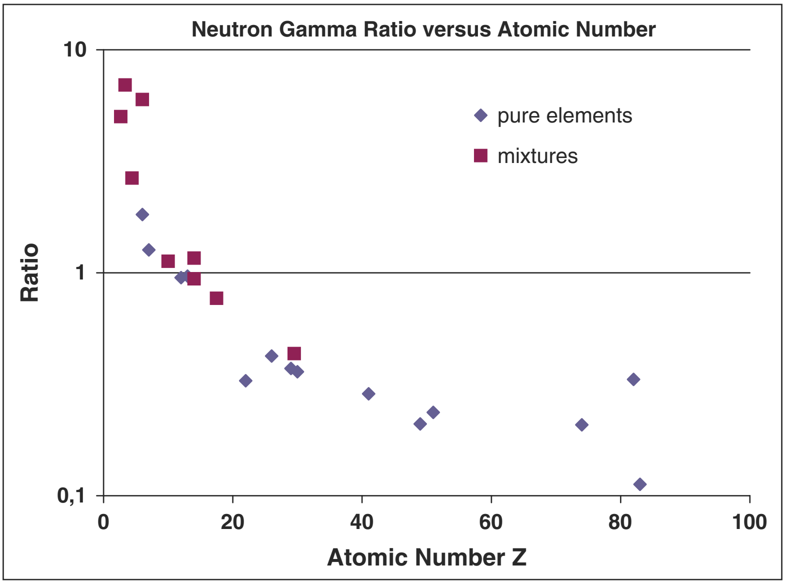

Fig. 2 Ratio values measured with the tagged ^{252}Cf source

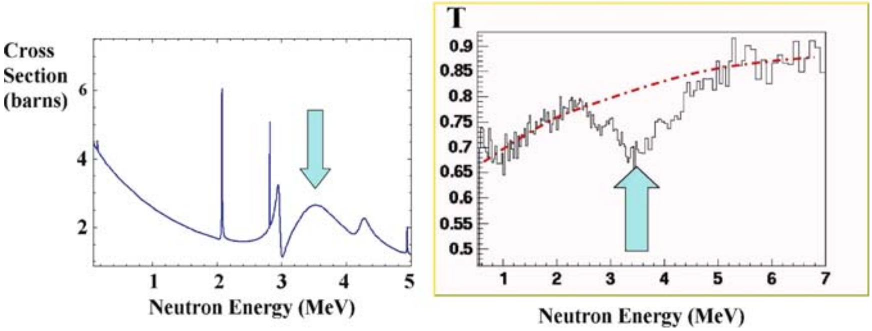

Fig. 3 Measured transmission as a function of the neutron energy for the graphite sample (right) and total neutron cross section for ^{12}C (left)

resonance structure is shown in Fig. 3 in case of ^{12}C nuclei. Particularly interesting are the large resonances in the region $E_n = 2.5–4.5$ MeV. The measured transmission as a function of neutron time of flight, presented in the right panel of Fig. 3, shows clearly a well defined dip in the transmission that corresponds to the resonance structure. On the contrary, the two narrow resonances are not seen because of the experimental resolution of our set-up. Specific fingerprints have been identified also in the measured time of flight spectra for other light nuclei (as ^{16}O) that are important in the identification of explosive materials.

Due to the limitation in the management and tagging of ^{252}Cf sources, the use of a time-of-flight spectrometer is certainly limited to the inspection of light vehicles as a second screening line after a conventional X-ray scan. A second possible application of this technique would consist in scanning small parcels in airports or at the entrance of governmental building or critical infrastructure.

4 Conclusions

Fast neutron inspection is one of the open options in the search for explosives in volumes that can not be accessed directly, as is the case of some vehicles. In these circumstances elemental information on a specific voxel can be obtained by the neutron-in/gamma-out technique by using tagged neutron beams. Such technique has been successfully applied in the EURITRACK inspection portal employed to inspect trucks transporting containers at the seaport exit. The EURITRACK portal is used as second line inspection after the traditional X-ray radiography. Monte Carlo simulations reported in this work demonstrate that it is possible to detect home made (HME) and liquid explosives hidden inside trucks. In this application, the possible interference with other materials has to be taken seriously into account as a source of false alarms.

A second inspection technique based on the use of a tagged neutron source has been developed in laboratory conditions using a ^{252}Cf source with time-of-flight measurements. Determining the attenuation ratio between neutrons and gamma rays as well as the ratio between high energy and low energy neutrons (and gammas), a set of material dependent quantities can be used in a material identification algorithm to deduce the average atomic number of the material <Z>. Moreover, neutron attenuation as a function of energy, show well defined element dependent fingerprints that can be used to extract the elemental composition of the sample. Such technique can be applied to search for explosives, including HME and LE, in light vehicles or in small parcels.

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Optimization of Hardware for Tagged Neutron Technology

M.D. Karetnikov, A.I. Klimov, K.N. Kozlov, E.A. Meleshko, I.E. Ostashev and G.V. Yakovlev

Abstract The possibility of background suppression by spatial and time discrimination of events stipulates the potentialities of the Nanosecond Tagged Neutron Technology (NTNT) for various application, e.g., for remote detection of wide range of explosives and flammable agents. For practical realization of NTNT the time resolution of pulses from gamma-detectors with respect to the associated pulses from alpha-detector should be close to 1 ns. The total intensity of signals can exceed $1 \cdot 10^6$ 1/s from all gamma-detectors and $1 \cdot 10^7$ 1/s from the alpha-detector. The processing of such stream of data without losses and distortion of information is one of challenging problems of NTNT. It is suggested to implement the preliminary "on-line" data processing by hardware. The architecture of data acquisition and control system and examples of realization are considered. Another important problem of NTNT is a choice of proper gamma-detectors to provide sufficient efficiency, amplitude and time resolution of measurements. With this aim, gamma-detectors based on NaI, BaF2, LYSO, and BGO crystals were assembled; the crystals were coupled with fast photomultipliers of similar series for more unambiguous interpretation of measurements. The results of experiments and features of detectors as applied to the NTNT systems are discussed.

Keywords Nanosecond Tagged Neutron Technology, NTNT, hardware, crystal

1 Introduction

One of basic challenges of neutron technologies of remote control is a high background affecting the accuracy of measurements. Last years, the nanosecond tagged neutron technology (NTNT) has been rapidly progressing [1]. This technology provides effective (by 2–4 orders of magnitude) suppression of background by

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spatial and time discrimination of events. NTNT is the most effective for solving such tasks as the detection, identification, and localization of chemical explosives by a relative concentration of key chemical elements (carbon, nitrogen, and oxygen) in the inspected object.

NTNT is based on the following principle. Neutrons are produced at the T(d,n) ^{4}He reaction while deuteron beam bombards the tritium target. Vectors of escape of neutron and associated alpha-particle (^{4}He) are uniquely correlated [2]. A multipixel position- and time-sensitive alpha-detector measures the time and position of incident alpha-particles. It provides the angle and time of neutron escape (the "tags" of neutron). The fast "tagged" neutrons are directed to an object of interest and induce characteristic gamma rays produced through the inelastic scattering of neutrons. Individual nuclei inside the object are identified by recording energy spectrum of emitted gamma-rays by a gamma-detector array.

A data acquisition and control unit traces the number (position) of gamma-detector, gamma-ray energy and recording time, as well as pixel number and recording time of alpha-particle. The speed of 14 MeV neutron is as high as 5.2 cm/ns. Thus, the precision of timing for event localization with the accuracy of several cm should be around 1 ns.

For practical application of NTNT, the multi-detector systems and high intensity (up to $1 \cdot 10^8$ 1/s) neutron generator should be used [1]. The total counting rate can exceed $1 \cdot 10^6$ 1/s for all gamma-detectors and $1 \cdot 10^7$ 1/s for the alpha-detector. The transmission of such stream of data to the computer and its processing might heavily complicate the data transmission interface and computer equipment. Thus, it is reasonable to implement the on-line preliminary data processing by the hardware. The basic criteria of selection of "useful" events are the presence of signals from alpha- and gamma-detectors in a certain time interval (tracking interval), range of gamma-ray energy, and absence of foldover of the signals.

2 Data Acquisition and Control System for NTNT

The flow block of data acquisition and control (DAC) system suggested by the Russian Research Center "Kurchatov Institute" [2] and then realized in several versions is displayed in Fig. 1. The signals from gamma-detectors come to the units of gamma-channels; each unit processes signals from several gamma-detectors. The constant fraction discriminator **CFD** at the input of each gamma-channel generates the logic signal $\boldsymbol{T}_\gamma$ that starts the time-digital converter **TDC** and initiates an encoding of the number of activated gamma-detector ($\mathbf{R}_\gamma$). The shaping amplifier **ShA** provides the required resolution of measurements of signal amplitude by analog digital converter **ADC**.

The alpha-channel unit issues a logic signal $\boldsymbol{T}_\alpha$ (time stamp of alpha-particle recording) and a code of the number of activated pixel of alpha-detector ($\boldsymbol{R}_\alpha$). These signals are transmitted to the timing and address buses of alpha-channels and shared by all units of gamma-channels.

As far as the intensity of signals gamma-detectors is much less than those from the alpha-detector the master is always the signal from the gamma-detector.

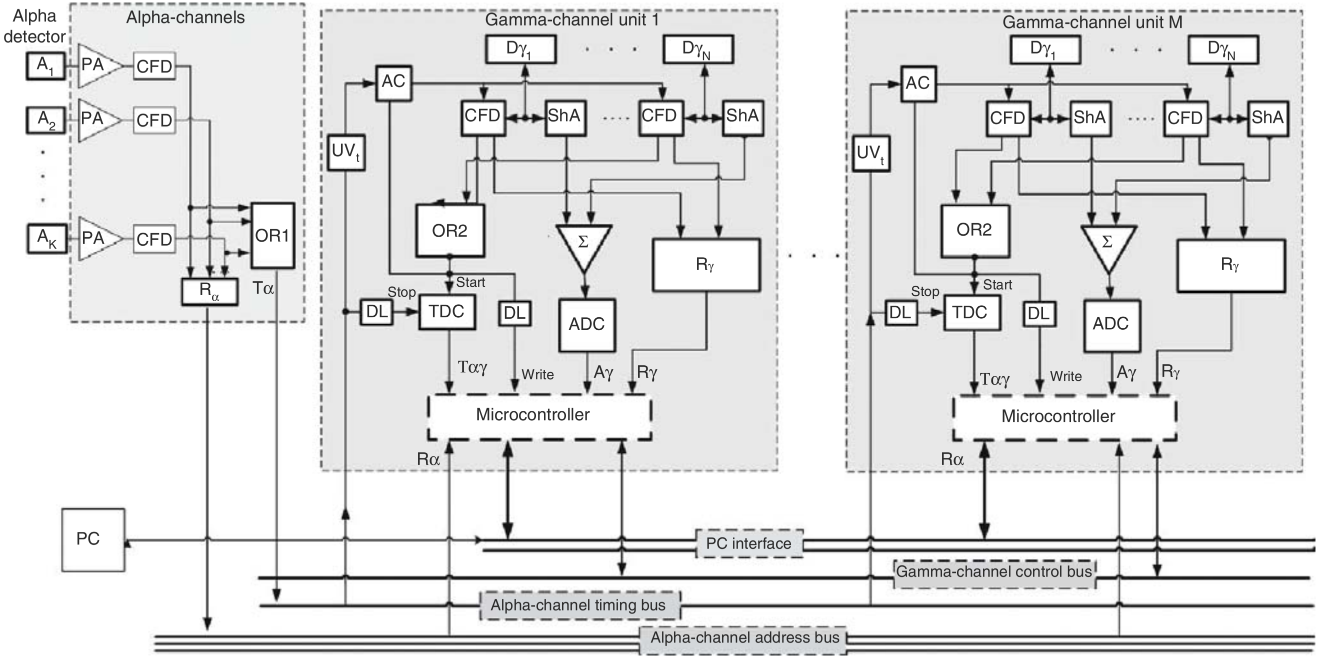

Fig. 1 Flow block of DAC system

However, as far as the signal from the associated alpha-detector comes first, the delay line **DL** (several dozens ns) shifts the time of arriving of the $\boldsymbol{T}_{\alpha}$ signal to the time analog converter **TAC** after the $\boldsymbol{T}_{\alpha}$ signal for proper timing.

The univibrator $\mathbf{UV}_{\mathbf{T}}$ generates the pulse with duration equal to the tracking interval. If the signal $\boldsymbol{T}_{\alpha}$ from the alpha-channel comes during the tracking interval, it initiates **ADC** to measure the amplitude of gamma-signal $\boldsymbol{A}_{\gamma}$ and stops **TDC** that issues the code of time between signals from alpha- and gamma-channels $\boldsymbol{T}_{\alpha\gamma}$. After completing the measurements, the codes $\boldsymbol{A}_{\gamma}$, $\boldsymbol{R}_{\gamma}$, $\boldsymbol{R}_{\alpha}$, and $\boldsymbol{T}_{\alpha\gamma}$. are packed by the microprocessor and sent to the computer **PC**. The anticoincidence unit **AC** and foldover discriminators prevent the writing of the event at the presence of two or more signals from detectors during the tracking interval.

The main features of DAC systems developed by this scheme in the Kurchatov Institute are as follows:

- Quantization of sampling time: 0.075–0.2 ns.
- Intrinsic time resolution of hardware: ≤ 0.3 ns.
- Digit capacity of time code: 8–12 bits.
- Digit capacity of gamma-energy code: 8–12 bits.
- Digit capacity of the code of gamma-detector number: 8 bits.
- Digit capacity of the code of alpha-detector pixel number: 8 bits.
- Options of the interface for data transmission to the computer: Ethernet; RS-422; RS-485; USB; BlueTooth.
- Form-factor: Eurocrate; CAMAC (optionally).

The suggested architecture of DAC system allows an alteration of the number of gamma-detectors, connection of alpha-detectors with various number of pixels, adjustment of scintillation units with different crystals for gamma-ray detection. It makes possible to use unified DAC system with different devices utilizing tagged neutron technology for various applications- from the detection of explosives in sea cargo containers to the inspection of luggage.

3 DAC System Examination

The DAC system was tested with the experimental test-bench included the NG-27 neutron generator (NG) manufactured by Dukhov All-Russian Research Institute of Automatics [1] with the built-in 9-pixel semiconductor alpha-detector. The tagged neutrons were directed to the graphite box (7 cm in height, 5 cm in width, 3 cm in thickness). The gamma-rays induced by neutrons were recorded by the LYSO gamma-detector 50 mm in diameter and 50 mm in height coupled to the XP4372 photomultiplier [3]. A configuration of the test-bench and basic dimensions are given in Fig. 2. The signals from the gamma- and alpha-detectors were processed by the DAC system described above. During the measurements, the intensity of NG was as high as $2 \cdot 10^{7}$ 1/s.

The time spectrum of alpha-gamma coincidences (upper curve) measured by DAC system is given in Fig. 3. The gamma-detector was shielded from the NG by

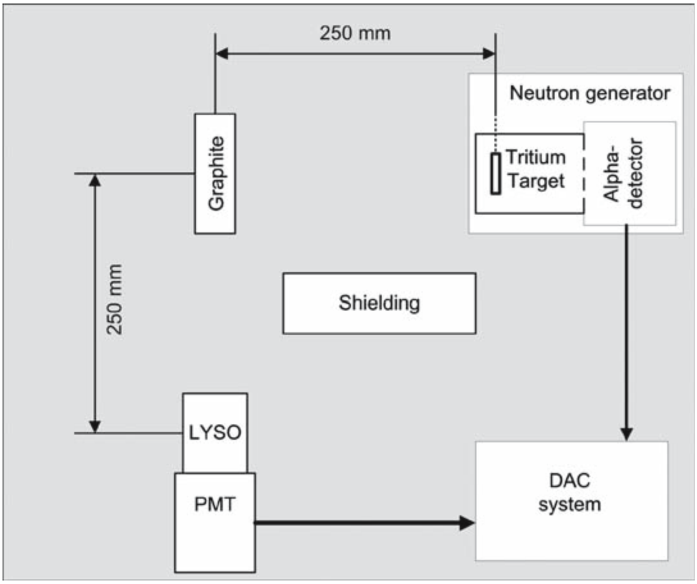

Fig. 2 Test-bench for testing the DAC system

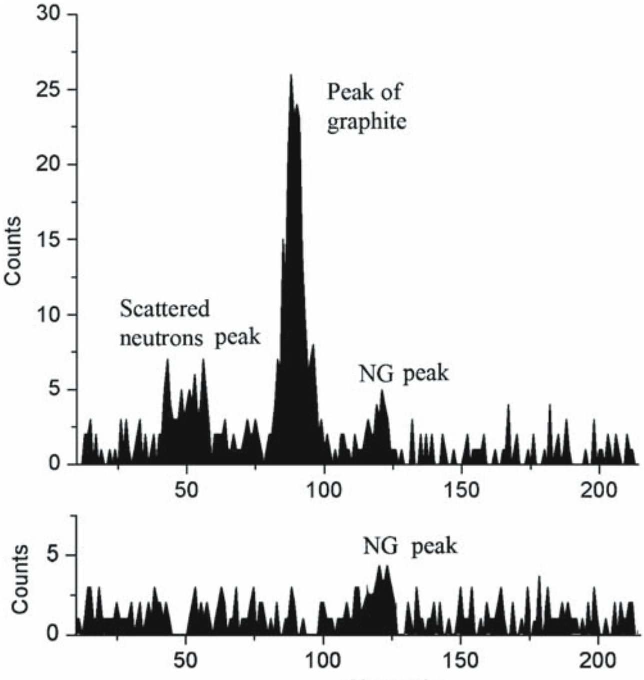

Fig. 3 Time spectrum of alpha-gamma coincidences for the shielded gamma-detector when the graphite box is installed (upper curve) and when the graphite box is removed (lower curve). 1 channel = 0.15 ns

8 cm iron bar. The lower curve displays the background spectrum taken when the graphite box was removed.

A portion of "tagged" neutrons are scattered at the copper holder of target and the casing of NG inducing background gamma-rays. This peak of "correlated" background (indicated in Fig. 2 as **NG peak**) is partially suppressed by the iron shielding. Another component of "correlated" background (**Scattered neutrons peak**) is caused by the elastic scattering of tagged neutrons in graphite towards the gamma-detector.

The "non-correlated" background includes:

- Gamma-rays induced by "non-tagged" neutrons (emitted at the high angles about the "tagged" neutron paths; the associated alpha-particle is not recorded by the alpha-detector).
- Gamma-rays emitted through the reactions other than inelastic scattering (radiative neutron capture, radioactive decay, etc.).

It can be seen a good time separation of peak of "useful" events (**peak of graphite**) from peaks of correlated background. The width (FWHM) of the **peak of graphite** on the time spectrum diagram can be assessed as

$$\tau = \sqrt{\tau_0^2 + \tau_f^2}$$

where τ_0 is a total time resolution of the measuring system, τ_f is a time of 14-MeV neutron flight through the graphite box. As far as $\tau \approx 1.2$ ns and $\tau_f \approx 0.6$ ns, then $\tau_0 \approx 1$ ns. The time between the **peak of graphite** and **NG peak** corresponds to the time required for 14 MeV neutron to pass the distance between the NG and graphite.

As far as the events beyond the tracking interval are rejected by the DAC system, these components of "non-correlated" background are highly reduced as can be seen in Fig. 3 (lower curve).

Figure 4 displays the time spectrum of alpha-gamma coincidences when the iron shielding was removed. It can be seen the increase of **NG peak** and "non-correlated" background, however, the peaks are still well separated, and the time resolution is close to 1 ns. The results of these measurements demonstrate that the NTNT based systems can operate without a massive shielding of a gamma-detector. It provides the opportunity to develop a portable hand-held neutron-based explosives detector with a sufficiently reduced weight.

Several types of other non-organic scintillators promising for NTNT application were also tested by our team. They included BaF_2, NaI, and BGO scintillators with diameter varied from 63 to 75 mm. The crystals were coupled with ultrafast photomultipliers of XP43xx series produced by Photonis Group [3] for more unambiguous interpretation of measurements. The summary of results are summarized in Table 1. The absorption efficiency of 4.4 MeV gamma-ray is assessed for the crystals 6.3 cm in diameter and 6.3 cm in height. The experiments demonstrated that LYSO has the best performance both by time (0.9–1.1 ns) and amplitude (3.6–3.8% for 4.4 MeV gamma-rays) resolusion.

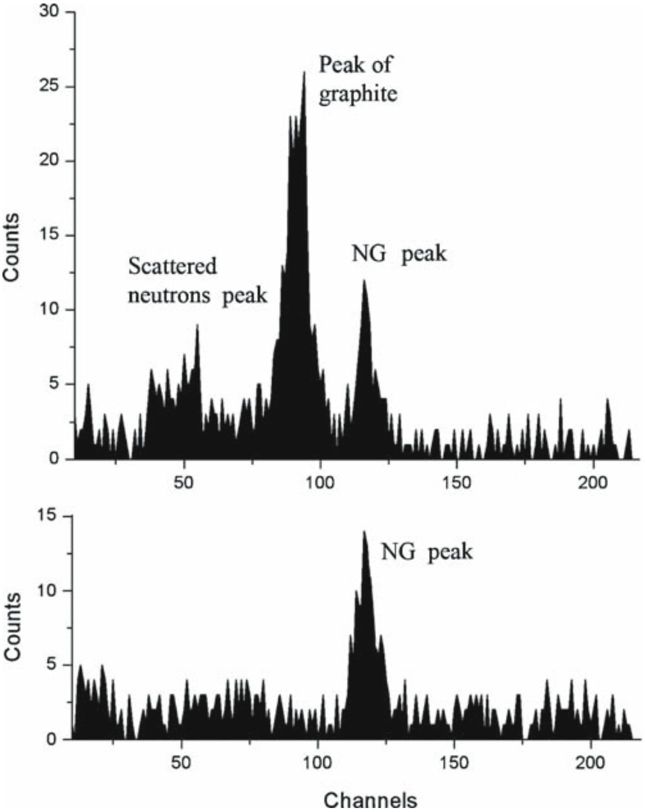

Fig. 4 Time spectrum of alpha-gamma coincidences for the unshielded gamma-detector when the graphite box is installed (upper curve) and when the graphite box is removed (lower curve). 1 channel = 0.15 ns

Table 1 Comparison of non-organic scintillators promising for NTNT application

Type of crystal	Time resolution (ns)	Amplitude resolution for 4.4 MeV (%)	Efficiency for 4.4 MeV (%)	Thermal coefficient (%/deg)	Radiation stability (rad)
NaI(Tl)	1.4–1.6	4.6	55	−0.3	10^4–10^3
BaF_2	0.9–1.1	6	64	−(1 ÷ 2.4)	$>10^7$
$Bi_4Ge_3O_{12}$	2.4–2.6	4.0–4.2	82	−1.2	10^5–10^6
$Lu_{1.8}Y_{0.2}SiO_5$(Ce)	0.9–1.1	3.6–3.8	84	+0.04	10^5–10^6

4 Discussions

It was demonstrated that NTNT provides suppression of background by spatial and time discrimination of events. The efficiency of background suppression increases with the improvement of time resolution. The total time resolution τ_0 can be estimated as:

$$\tau_0 = \sqrt{\tau_i^2 + \tau_{D\gamma}^2 + \tau_{D\alpha}^2 + \tau_{G\gamma}^2 + \tau_{G\alpha}^2}$$

where τ_i is the intrinsic resolution of DAC hardware; $\tau_{\mathrm{D}\alpha}$ and $\tau_{\mathrm{D}\gamma}$ are the time jittering of alpha- and gamma detectors; $\tau_{\mathrm{G}\alpha}$ and $\tau_{\mathrm{G}\gamma}$ are the geometrical factors caused by finite size of alpha- and gamma-detectors. The value of τ_i measured using the signals from external pulse generators was as low as 0.3 ns [4]. For the detectors used in the existing NTNT systems [1], the assessed values of $\tau_{\mathrm{G}\alpha}$ and $\tau_{\mathrm{G}\gamma}$ are less than 0.5 ns.

The jittering of gamma-detector $\tau_{\mathrm{D}\gamma}$ is caused by a PMT transit time difference, fluctuations of charge collection, etc. For small crystals (1 in.), the jittering can be as low as 0.5 ns for fast inorganic scintillators [3]. However, the scintillator for NTNT applications should have sufficient cross-section and height for full absorption of gamma-rays with the energy above 1 MeV. The jittering increases with the scintillator volume and active diameter of PMT.

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Detection of Liquid and Homemade Explosives: What Do We Need to Know About Their Properties?

D. Menning and H. Östmark

Abstract Improvised Explosive Devices (IEDs) pose a threat to both military forces deployed in foreign regions and to civil society. The threat from military and commercial explosives used in IEDs has shifted to homemade explosives (HMEs). Examples of liquid explosives, solid explosives and explosive mixtures used as HMEs are presented. Properties of importance for detection of these energetic species are listed and described. Some examples are provided. Vapour pressure, performance, density, elemental composition, detonability, ignitability, sensitivity, stability, chemical reactivity, UV absorbance, mass spectrum, IR and Raman absorbance, dielectric constant, purity, viscosity, particle adhesion and particle size are factors affecting the probability of detecting an explosive compound and have to be considered when developing and designing novel detection methods and instrumentations, but also when improving existing techniques and methods for explosives detection. Disposal personnel (military IEDD/EOD,[1] police bomb disposal) also have to consider many of these properties when handling HMEs, in order to reduce risks during transportation and disposal.

Keywords Homemade Explosives, Improved Energetic Materials/IEM/HME/ liquid explosives, properties, vapourpressure, perfomance

1 Introduction

Today, Improvised Explosive Devices (IEDs) pose a serious threat to the Western military forces in Iraq and Afghanistan [1]. In the military conflict zones, military explosives are frequently being used in IEDs, but the threat from homemade explosives

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[1] Improvised explosive device disposal/explosive ordnance disposal

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(HMEs), however, is always present. In civilian society, IEDs has shifted from commercial and military explosives to the use of HMEs. The latest events in London in July 2005 [2] and the terrorist plot arrests in UK in August 2006 [3] have showed that there is a significant threat from liquid and homemade explosives. Organic peroxides have been used in attacks with HMEs in Scandinavia. Methyl Ethyl Ketone Peroxide (MEKP) is believed to have been used in Vantaa, Finland, in 2002, when an IED was detonated in a shopping mall, killing the perpetrator and six other, innocent people [4]. In Sweden and Denmark, Triacetone Triperoxide (TATP) is troubling the police bomb disposal personnel when found at miscellaneous crime scenes [5]. The way perpetrators use liquid or solid explosives or explosive mixtures is widening and the challenge for society is to detect these energetic compounds before they can pose a threat to civilians, rescue services, police or military personnel.

In order to set requirements and specifications for detection equipment and to design suitable, capable and robust detection systems, it is of outmost importance to know the properties of the detected species. Properties of interest for detection are listed and described in this paper. However, it is important to realise that due to the nature of its definition, an HME can be made in an unlimited number of ways, comprising all explosives known, which makes it very difficult to detect every specific energetic compound possible. Fortunately, there are a limited number of energetic materials which are more likely to be used as HMEs, due to their ease of manufacture, availability, costs of raw materials etc. This paper lists a number of examples of energetic materials used as liquid and solid homemade explosives or in homemade explosive mixtures.

2 Energetic Materials Used as Liquid and Homemade Explosives or in Explosive Mixtures

A huge number of different energetic compounds have been used and may be used as homemade explosives (liquid or solid) in explosive devices. A limited number of examples are presented in this paper. Important to have in mind is that the term ‘liquid explosive’ is a relative one (as for the term ‘solid’). In general, ‘liquid explosive’ refers to an explosive which is in liquid state at ambient temperature (room temperature, surrounding temperature, outdoor temperature etc.). Often, in a military context, the temperature range in which an explosive is to be used in is specified to be between $-50\,°\mathrm{C}$ to $+70\,°\mathrm{C}$, depending on region, country, military etc.

Examples of liquid and homemade explosives and energetic materials used in liquid and solid explosive mixtures are listed below (Table 1).

Table 1 lists energetic compounds that have been or may have been used as HMEs in IEDs, either in pure form or in mixtures, or both. Examples given for liquid explosive mixtures have, or may have, also been used for solid explosive mixtures and vice versa.

Nitroglycerine and EGDN are two liquid explosives that have been used in commercial explosives (dynamites) for a very long time. They are highly shock-sensitive

Table 1 Examples of liquid and solid homemade explosives and explosive mixtures

Liquid explosive
Nitroglycerine (NG), $C_3H_5N_3O_9$
Ethyleneglycol dinitrate (EGDN), $C_2H_4N_2O_6$
Nitromethane (NM), CH_3NO_2

Homemade explosive (HME)
Triacetone triperoxide (TATP), $C_9H_{18}O_6$
Hexamethylene triperoxidediamine (HMTD), $C_6H_{12}N_2O_6$
Nitrourea, $CH_3N_3O_3$
Urea nitrate, $CH_5N_3O_4$

Energetic material used in liquid explosive mixtures
Ammonium nitrate (AN), NH_4NO_3
Sodium nitrate, $NaNO_3$
Hydrogen peroxide, H_2O_2
Nitric acid, HNO_3
Methyl ethyl ketone peroxide (MEKP), $C_4H_8O_2$
Isopropyl nitrate (IPN), $C_3H_7NO_3$
Nitrobenzene, $C_6H_5NO_2$

Energetic material used in solid explosive mixtures
Nitrocellulose (NC), $[C_6H_7N_3O_{11}]_n$*
Potassium chlorate, $KClO_3$
Sodium chlorate, $NaClO_3$
Ammonium perchlorate (AP), NH_4ClO_4
Potassium perchlorate, $KClO_4$
Ammonium nitrate, NH_4NO_3
Sodium nitrate, $NaNO_3$
Potassium nitrate, KNO_3

* n = 1,000–3,000 [6]

in pure form and therefore production rates of the two explosives have declined during the past decades, being replaced by other, less sensitive AN-based explosives. In attacks, dynamites have been used in explosive devices since the bombings of the Russian tsar Alexander II, ending up killing him in 1881 [7, 8]. The latest, most known examples where dynamite explosives have been used are the train bombings in Madrid, in 2004. In other words, there is still a strong need for detection of these substances, which, to some extent, also relate to their widely spread use in gun propellants.

Nitromethane is a liquid explosive that is frequently being used as a fuel for racing cars, as a rocket propellant etc. It has not been widely used as an explosive, due to its high volatility and relatively low performance (in military applications).

TATP and HMTD are two solid, organic peroxide based explosives which have been known for more than a hundred years, but are too sensitive and exhibit too poor properties to have found practical use in military and civilian applications. However, HMTD, is patented as a primary explosive for detonators [9]. Both explosives are easy to manufacture, which have made them very popular for use as HMEs. TATP sublimes readily at room temperature. HMTD reacts with many metals and is thermally unstable. Both explosives are extremely sensitive towards impact, friction and electrostatic discharge, making them very dangerous to handle at all times.

Nitrourea is a thermally unstable, hygroscopic explosive, which is decomposed by water. Urea nitrate is a chemically unstable explosive, with very acidic properties. The unfavourable properties of both nitrourea and urea nitrate have made them unsuitable for commercial and military use.

Hydrogen peroxide is a very common oxidiser in the modern world of today, used in bleaching and disinfection applications, as a preservative, curing agent etc. It is being used in everyday consumer products in diluted form, in order to reduce the dangerous and energetic properties of the substance. However, when concentrated, hydrogen peroxide is used in HME mixtures.

Nitric acid is a very strong acid and oxidiser which is used in large quantities in the modern society as a base reagent or catalyst for chemical reactions at an industrial scale. It is widely available and is used as a nitrating agent in the manufacture of explosives.

MEKP is an organic peroxide and a commonly used curing agent, in diluted form, for monomers undergoing radical polymerization to produce plastics, i.e. polyesters. MEKP is classified as an explosive in concentrations higher than 52 wt%, according to international transport regulations [10]. MEKP is one example of over hundred different organic peroxides used at an industrial scale in the European Union at present time. Not all of these represent explosive properties, but are potential energetic materials that may be used in homemade explosive mixtures.

IPN is used as a fuel in specific applications and has a sweet odour. It is a flammable agent exhibiting some explosive properties. It has been used in thermobaric compositions.

Nitrobenzene is used for the production of aniline and has a characteristic odour of bitter almond. It is highly toxic and is an energetic material. It should be considered to be of interest when referring to liquid homemade explosive mixtures.

Potassium and sodium chlorate are two common oxidisers used in the pyrotechnics field. Both energetic materials are highly friction and impact sensitive. They have been used in different HME mixtures.

Ammonium and potassium perchlorate are two oxidisers exhibiting somewhat lower sensitivity when compared to chlorate based oxidisers. AP is a very common component in solid rocket motors, while potassium perchlorate is used in the pyrotechnics field. AP is believed to have been used in the Bali terrorist attacks, in 2002.

Ammonium and sodium nitrate are common components of commercial liquid explosives due to their low cost, very low sensitivity and positive oxygen balance. To a large extent, ammonium nitrate is also used in commercial solid explosives (ANFO, dynamites, powder explosives etc.). Ammonium nitrate was used in the attack against the FBI building in Oklahoma City, in 1995.

Potassium nitrate is used as a preservative and a fertilizer and is the energetic component of one of the oldest explosives known: black powder. Black powder is often a physical mixture of sulphur (10–12 wt%), charcoal (12–15 wt%) and potassium nitrate (74–78 wt%) [11] and is very easy to manufacture or to acquire. In some types of black powders the potassium nitrate is replaced by sodium nitrate (which reduces costs). Black powders are friction sensitive and very sensitive to electric discharge. Due to their ease of manufacture and high availability, black powders are frequently used as HMEs.

NC is an explosive in pure form, but it is generally modified and coated with graphite to form single base gun propellants, or used in different mixtures for double or triple base gun propellants. NC is highly sensitive to electrostatic discharge and it is impact sensitive.

A liquid or solid explosive mixture often contains flammable materials, such as fuels or solvents. Examples are: petrol, diesel, kerosene, alcohols, ethyl acetate, acetone and other ketones, alkanes etc. Metal powders are often used as fuels. Examples are: aluminium, magnesium and zirconium. A flammable metal powder composition can reach very high burning rates (explosion) if the metal particle sizes are small enough, due to the large surface to volume ratio, which accelerates the reaction.

3 Physical and Chemical Properties of Liquid and Homemade Explosives of Interest for Detection

In order to increase the probability of detecting explosive compounds, knowledge of their chemical and physical properties is essential for success. Depending on which detection method is used – bulk or trace, specific properties of an energetic compound are of interest.

Detection systems for liquid and homemade explosives, both in military and civilian applications, need to be fast, robust, accurate, selective and provide low false alarm rates. There is a strong need for stand-off solutions, for a number of reasons. Safety and personal integrity (at crowded places, i.e. airports etc.) are two of them. The properties of the explosives to be detected are of vital importance, due to the many practical aspects affecting the detection result, such as sampling technique (vapour and/or particles), pre-concentration of sample, safety issues etc. There are also significant differences between bulk and trace detection, considering sensitivity, selectivity, sampling etc.

Frequently, when an IED incident takes place, the explosive is not exposed; it is either completely hidden or encapsulated in some sort of container, in order to avoid detection. The concealment complicates the detection situation considerably, compared to easier (possibly optimized?) environmental conditions in a laboratory or at a test facility. This fact limits the number of potential methods to be used in real situations (i.e. methods “to be fielded”) and the properties of the explosive become even more important, in order to distinguish characteristic features of a suspected explosive substance, or several suspected explosive substances, from a complex background.

Properties of value for explosives detection are many, but some properties are more apparent and are addressed in this paper. It is of fundamental interest to know the basic physical and chemical properties (melting point, boiling point etc.), in order to address the problem correctly. Then, it is possible to further enhance a detection method and reduce the false alarm rates by knowing and utilizing

Table 2 Properties of liquid and homemade explosives of interest for detection

Property
Vapour pressure
Performance
Density
Elemental composition
Detonability (critical diameter)
Ignitability
Sensitivity
Stability
Chemical reactivity
UV absorbance
Mass spectrum
IR absorbance
Raman absorbance
Dielectricity constant
Purity
Viscosity
Particle adhesion
Particle size

additional, properties, relevant for the detection. Properties of importance for explosives detection are listed (Table 2) and further described below.

3.1 Vapour Pressure

Vapour pressure is a property of vital importance for detection methods based on trace detection, such as stand-off detection techniques. A high vapour pressure ensures an increased probability of finding a hidden or partially enclosed explosive (or a fully enclosed explosive, encapsulated in a gas permeable casing). Some liquid explosives and energetic materials exhibit high, or very high, vapour pressures. TATP, which is a solid homemade explosive at ambient temperature, distinguishes itself from other commonly used solid explosives (TNT, tetryl, PETN, RDX and HMX) by exhibiting a relatively high vapour pressure. Examples of vapour pressures for HMEs and energetic materials are illustrated below (Fig. 1).

3.2 Performance

As a basic characteristic of an explosive, performance is important to characterise in order to determine or to estimate the damage effects caused by a homemade explosive. A high explosive (HE), compared to a low explosive (LE), is a shattering explosive, exhibiting brisance, which is one effect strongly influencing the outcome of an explosive attack.

In a military context, performance is highly related to the shattering and metal acceleration ability, while performance in civilian applications relate to the ability

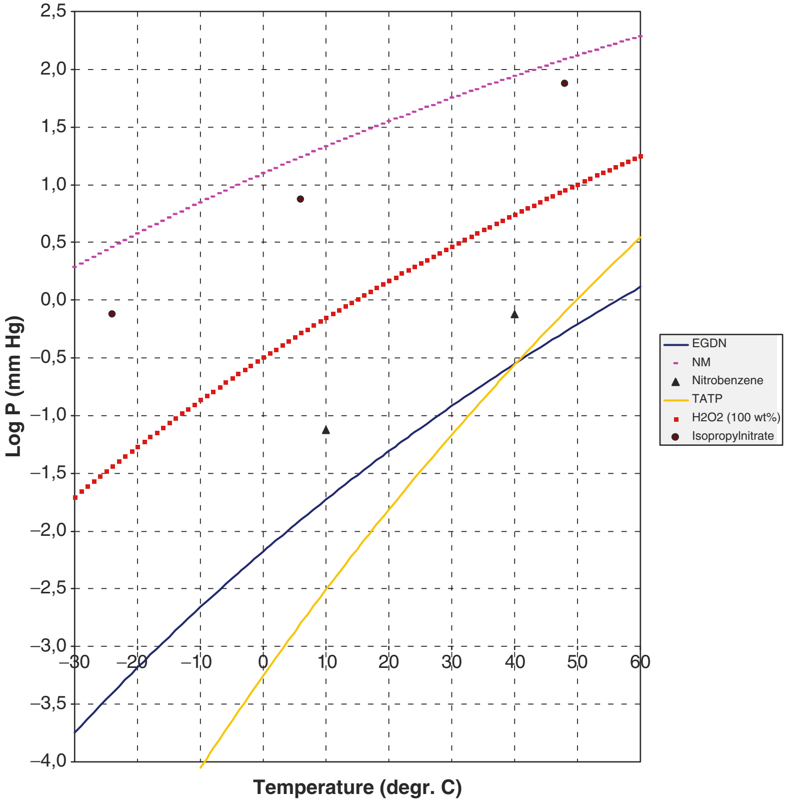

Fig. 1 Vapour pressures for different liquid and solid energetic materials. References for EGDN [12], NM [13], nitrobenzene [14], TATP [15], H2O2 (100 wt%) [16] and isopropyl nitrate [14] are approximated values (Data curves represent referenced equations, derived from empirical data. Data points represent referenced values)

to produce work (gas production). Performance is crucial to determine the hazardous danger area for a specific explosive charge, in order to protect people, estimate damage on vehicles and buildings, to assess the risks of secondary dangers etc. It is of interest to detection for safety reasons, in case of a detonation or deflagration reaction occurs during operation.

Performance is frequently determined experimentally, where detonation velocity and detonation pressure are measured parameters. However, in order to avoid experimental setups, demanding relatively large amounts of possibly highly hazardous and unstable explosive material, it is possible to estimate the performance of an explosive, or explosive mixture, by calculating the theoretical values, using thermochemical code. By comparing the calculated results for an explosive with unknown properties with the results of a known explosive, such as PETN, it is possible to estimate the relative performance of the explosive. This is illustrated below, by performed calculations on a number of energetic materials, using Cheetah, Version 2.0 [17] (Table 3).

Table 3 Performance data results, as calculated by Cheetah [18]

Explosive	Detonation velocity (m/s)	Detonation pressure (GPa)	$V/V_0 = 6.50$ (rel. PETN, $\rho = 1.76\,g/cm^3$)	Enthalpy of formation (kJ/kmol)	Density (g/cm^3)
Isopropyl nitrate, $C_3H_7NO_3$	4,996	7.08	0.32	−229.79 [19]	1.034 (19 °C) [20]
Urea nitrate, $CH_5N_3O_4$	7,128	16.42	–	−546.47 [18]	1.69 (20 °C) [20]
Nitrobenzene, $C_6H_5NO_2$	4,474	5.80	0.25	12.47 [21]	1.204 (20 °C) [20]
Nitromethane, CH_3NO_2	6,292	12.46	0.50	−107 [18]	1.16 [18]
Nitric acid, HNO_3, 70 wt%	5,423	10.29	–	−174 [18]	1.50 [18]
Teflon, C_2F_4, 30 wt%				−810 [18]	2.25 [18]

The thermodynamical code solves the thermodynamical equations to find equilibrium between reaction products. The Chapman-Jouguet state is possible to predict by using correct input data and by using detonation theory. Cheetah can (from the CJ-state) predict essential parameters and course of events for a detonating explosive. It also has the capacity of determining the shock wave effect in different types of transport media, which is essential to estimate the posed threat from a specific explosive, or mixture of explosive materials.

Thermochemical code can be used to calculate performance estimations, but there is always a cause for caution, due to the limitations simulations based on theoretical assumptions form. Sometimes experiments are inevitable.

3.3 Density

Density is a property of vital importance for present, x-ray based detection methods. X-ray based methods utilize density differences to differentiate between liquid and solid compounds and current systems are highly advanced, providing enormous amounts of data for image presentation. Density is also crucial for determining other properties of explosive, such as performance, detonability, ignitability etc.

3.4 Elemental Composition

In general, explosives contain mainly nitrogen and oxygen, which is a feature utilized in detection methods determining elemental composition in unknown materials. X-ray and nuclear based methods (gamma-rays, neutrons) use this feature for stand-off bulk detection [22].

3.5 Detonability (Critical Diameter)

A safety related property of a homemade liquid or solid explosive is the detonability of the compound. It is usually performed by a number of different tests (cylinder tests, steel sleeve tests, wedge tests etc.) in order to determine under what circumstances (what the critical diameter is etc.) the specified explosive is detonable or not. HMEs and liquid explosives may not be suitable for these types of tests, due to their incompatibility problems, poor stability and sometimes high handling sensitivity. Other properties may also affect the detonability result, such as composition of the explosive, its purity and density.

3.6 Ignitability

Ignitability is a property related to safety and important to evaluate, due to the fact that HMEs frequently are used as primary explosives in IEDs, either combined with a main charge explosive, or alone. Ignitability is a property depending on the amount of activation energy available for current explosive to decompose by deflagration to detonation transition (DDT).

3.7 Sensitivity

Sensitivity of explosives is a much debated issue and is divided into several aspects, such as handling sensitivity (impact, friction, electric discharge), thermal and shock sensitivity, combined with chemical compatibility and stability aspects when the explosive is ageing or affected by heat or temperature variations over time (environmental influences).

In some cases, the sensitivity of the liquid or solid homemade explosive is so high, that an inappropriate detection method may cause, or accelerate, a decomposition reaction, which can result in initiation and detonation, or deflagration, of the explosive. This risk may be a reality when detecting species, such as highly sensitive organic peroxides (TATP, HMTD).

The sensitivity of HMEs is illustrated by some examples of performed small scale sensitivity tests on Triacetone Triperoxide (TATP), synthesized at FOI (Table 4).

Impact sensitivity was determined by using a ERL/Bruceton Apparatus. The sensitivity was measured with a 2.5 kg drop weight and testing was performed by using an up-and-down method on both sides of the 50% probability level. A total of 25 tests were made per sample and an average value was calculated on a 95% confidence level (when applicable).

Table 4 Friction and impact sensitivity properties of TATP, according to the *up-and-down*-method, synthesized at FOI during 2006–2007

Explosive	Form	Friction sensitivity, BAM (kp/cm^2)	Impact sensitivity, ERL (J)	Std.dev. (J)	Temp./RH (°C/%)
TATP (FOI), 2006-08-24	Needle-like crystals	<1.0	<1.2	–	21/74
TATP (FOI), 2007-01-19	Fine powder	<0.5	<2.0	–	18/27
TATP (FOI), 2007-02-13	Fine powder	<0.5	1.9	0.9	19/26
TATP (FOI), 2007-02-22	Crystals	<0.5	<1.2	–	17/25
TATP (FOI), 2007-02-26	Fine powder	<0.5	2.3	0.3	19/28

The friction sensitivity was determined by using a Julius Peters (BAM) apparatus. The testing was performed by using an up-and-down method on both sides of the 50% probability level. A total of ten tests were made per sample.

As the example shows, homemade explosives sometimes are too sensitive for commonly used test equipment for military and commercial explosives, when determining handling sensitivity.

3.8 Stability

Thermal stability, chemical stability and ageing of explosives all affect the risks of handling a specific explosive. Many HMEs are too unstable to be evaluated in commonly used stability tests (this is one of several reasons why many of the homemade explosives haven't been thoroughly tested in the first place) and it is important to be aware of the risks when using detection techniques which may increase the temperature of an explosive or cause a chemical interaction.

3.9 Chemical Reactivity (Colour Reactions)

Reactivity of an explosive towards other chemical compounds is of interest, when referring to chemical colour reactions for identification. Colour reaction tests for common military explosives (TNT, tetryl, RDX, HMX and PETN) have been available for many years [23, 24], while tests for organic peroxides, such as TATP and HMTD are relatively new. However, these methods still call for manual sampling and sometimes pre-treatment of the explosive to be analyzed [25].

3.10 UV Absorbance

UV absorption is a property which most explosive substances exhibit, which makes it an attractive feature in stand-off, laser based detection methods. However, for some types of HMEs (i.e. organic peroxides), UV absorption leads to decomposition and can therefore be dangerous in bulk detection situations.

3.11 Mass Spectrum

Mass fragmentation of a compound in a mass spectrometer provides a unique mass spectrum, which makes mass spectrometry a very powerful and effective method for identifying unknown explosive species. However, development of efficient sampling, pre-separation and ionization techniques (i.e. EI, CI, laser etc.) remains a challenge. Bulk detection systems call for pre-treatment of unknown samples in order not to overfeed the detection system. Explosives metastable condition also calls for suitable separation methods to avoid pre-decomposition of the species to be analyzed.

3.12 IR and Raman Absorbance

IR absorbance of an explosive compound is important for the identification of functional groups of an unknown explosive, which can indicate a type of explosive and/or complement other identification methods. It is possible to a certain extent to receive an IR 'fingerprint' of an explosive compound due to the different absorbance patterns in the so called 'fingerprint region' of an IR spectrum, combined with specific bands in the lower and higher wavenumber regions [26]. Today, it is possible to perform fast analyses, without using additional solvents or matrix materials, by using the Attenuated Total Reflectance (ATR) technique, which is illustrated by examples from performed analyses at FOI on IPN, nitrobenzene, HMTD and TATP (Figs. 2–5).

Analyses were performed with an Alpha FT-IR Spectrometer from Bruker Optics (Bruker Optik GmbH), equipped with an ATR platinum Diamond 1 Refl. Evaluation software used was Bruker Optics, Bruker Opus, Version 6.5 (2007). Instrument parameters: resolution was set to $4\,\mathrm{cm}^{-1}$, atmospheric compensation was selected and sample scan time was set to 1 min. A background run (62 scans) was performed before each sample was run. Sixty-two scans were performed per sample analysis and ten samples per explosive/energetic material were analysed. No sample preparations were needed and no solvents were used. Sample amount used was in the range of a few milligrams, for both liquid and solid samples.

IR analysis performed by using ATR is in practical use today for explosives detection [27] and is still improving. However, the detection method still has the disadvantage of forcing the operator to physically take a sample from the explosive material for analysis.

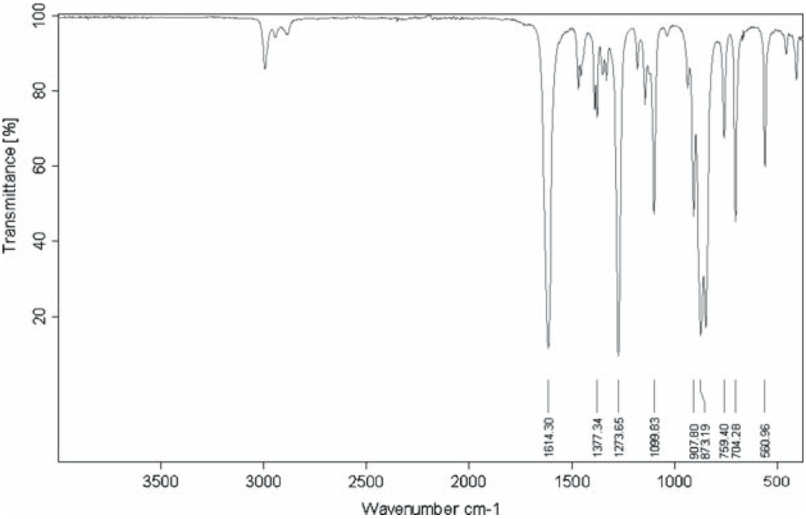

Fig. 2 IR spectrum (ATR) of isopropyl nitrate (technical purity), showing symmetric NO_2-strech (1,273 cm^{-1}) and asymmetrical NO_2-stretch (1,614 cm^{-1}) [26]

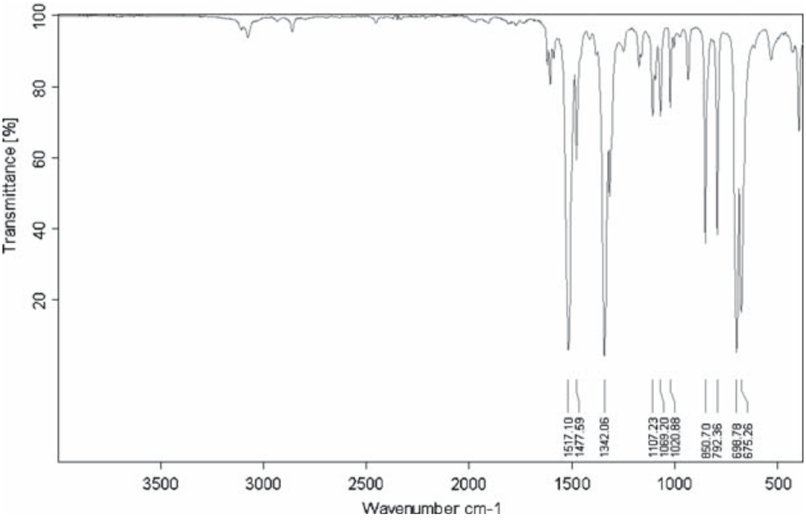

Fig. 3 IR spectrum (ATR) of nitrobenzene (TCI, pro analysi), showing various C-NO_2 (1,517 cm^{-1}) [26]

IR and Raman spectroscopy are complementary methods, where Raman spectroscopy provides information on functional groups of the explosive molecules by probing the vibrational transitions.

Raman uses a monochromatic excitation source, i.e. a laser, and can thereby be applied for stand-off detection. Raman spectroscopy is a method with low sensitivity as compared to resonant spectroscopic techniques, but sensitivity may be increased

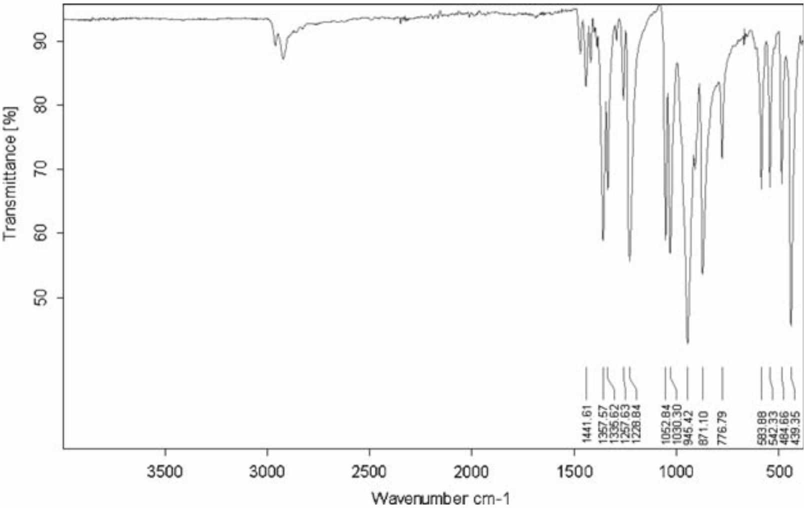

Fig. 4 IR spectrum of HMTD (synthesized at FOI, 2007, from laboraty chemicals, PA purity)

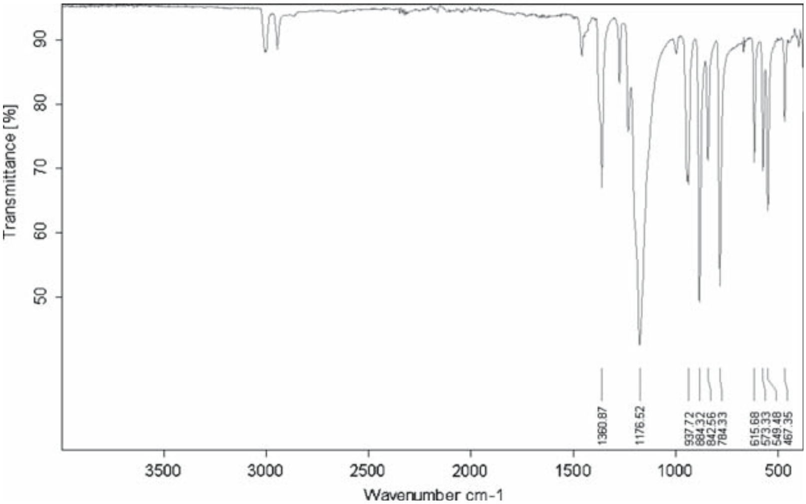

Fig. 5 IR spectrum of TATP (synthesized at FOI, 2007, from household chemicals, technical quality)

by applying resonance Raman. Resonance Raman is not currently available in detection instrumentation, but is a promising method for future trace stand-off explosives detection solutions.

3.13 Dielectricity Constant

The dielectric constant (relative permittivity) is a temperature- and density-dependent property, which can be utilised to selectively detect different types of explosives without opening sealed containers. It is defined as the capacitance ratio of a sample material filled condenser to the capacitance of an empty condenser, having only vacuum between its plates.

It is possible to use this property to establish empirical relationships for several component explosive materials.

3.14 Purity

Explosives exhibiting high purity increase the probability of correct identification, but impurities (reagents, by-products) can also enhance the probability of identifying an explosive. It is also possible to further determine the explosive properties of the current composition if the impurities can be identified.

3.15 Viscosity

Altered viscosity of a liquid explosive affects the evaporation rate of volatile components or the rate of evaporation of the explosive itself. Increased viscosity of a liquid explosive may impose decreased evaporation rates, which makes it more difficult to detect it by trace detection methods. Therefore, a thorough understanding of the relationships between viscosity, evaporation rates and other properties, such as vapour pressures of explosives, will help to improve an existing detection method, or the development of a new technique.

3.16 Particle Adhesion

For solid explosives, the adhesion of particles to different surfaces is of vital interest for the development of novel trace detection methods. As for TATP, which sublimes, it is most probable to find residue trace amounts on cold surfaces nearby an IED containing the explosive. It is of interest to evaluate how explosive particles adhere to clothes, skin, metal surfaces, building materials, paints etc. and also how they can be dislodged. Particle adhesion relates to other properties, such as particle size distribution, particle porosity, polarity etc. It is of importance to examine these relationships in order to be able to address the problem correctly.

3.17 Particle Size

Particle sizes of solid explosive residues affect the probability of finding the explosive by trace detection methods. Larger particles are easier to find, but smaller particles adhere better to electrically charged surfaces, due to the electronegative parts present in the explosive or energetic material (nitro-, nitrato-, peroxo-, perchloro-groups etc.). Sampling techniques depend on particle size, especially when vaporization, or heating to increase evaporation, of the particles is one step of the process. Porosity may also be of importance, because high porosity provides a larger surface exposed to heating, which results in a faster evaporation of the explosive.

4 Disposal of HMEs – A Delicate Task for Military EOD/IEDD Teams and Police Bomb Disposal Personnel

Disposal personnel assigned with transport and discarding of HMEs frequently expose themselves to major hazards. Properties of importance to disposal of HMEs often coincide with properties important for detection, such as: sensitivity, stability, performance, ignitability, detonability etc. Disposal personnel also have to consider other properties of homemade explosives, such as toxicity and compatibility with other substances. Therefore, repeated training and a “hands on”-perspective are key factors to minimize handling risks, combined with an efficient explosives detection system.

5 Summary

Homemade explosives, both in liquid and solid form, are an increasing threat. It is important to address this threat by improving existing detection methods and developing new and more efficient detection methods for explosives. To accomplish that, knowledge of the properties of explosives of interest to detection, is crucial for success. Today, there is a strong need for improved knowledge of many HMEs and HME mixtures regarding properties such as: performance, vapour pressure, detonability, ignitability, sensitivity, stability, particle adhesion etc.

Properties of importance to detection often coincide with the properties important for disposal of HMEs, which may facilitate synergy effects in future research.

There is a strong need for determining suitable detonability, ignitability and sensitivity tests for HMEs, in order to minimize handling risks and to support disposal personnel with “hands on”-knowledge.

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Detector of Hazardous Substances Based on Nanosecond Neutron Analysis

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Abstract Feasibility of Nanosecond Neutron Analysis/Associated Particle Technique for detection of explosive and flammable liquids has been experimentally demonstrated. Experimental results with imitators and real liquids are discussed.

Keywords Liquids, explosive, flammable, neutrons, associated particles

1 Introduction

Recent advances in the development of portable neutron generators with built-in detectors of associated α-particles [1] and in high-speed digital data acquisition electronics paved way for compact devices based on Neutron Nanosecond Analysis/Associated Particle Technique (NNA/APT) [2, 3]. NNA/APT is the most advanced of all "neutron in, gamma out" neutron interrogation techniques [4], in which chemical (isotopic) composition of the inspected material is determined from the analysis of energy spectra of prompt γ-rays produced by the incident neutrons in the material of the inspected object.

In NNA/APT some 14 MeV neutrons produced inside a DT neutron generator in the reaction $d + t = n + \alpha$ are "tagged" by detecting the associated α-particle with a position-sensitive detector built into the sealed vacuum tube. The position sensitivity of the α-particle detector in combination with time-of-flight analysis allows one to obtain energy spectra of prompt γ-rays separately from each 3D element ("voxel") of the investigated volume. Analysis of these spectra yields a 3D distribution of chemical elements in the inspected volume, allowing one to detect a small object (e.g. an explosive) hidden among other objects (e.g. inside luggage) [5].

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Since NNA/APT is sensitive to the elemental composition of the object, and since neutrons and high-energy γ-rays can easily penetrate the walls of any bottle irrespective of their material, an NNA/APT device can be used as a confirmation sensor working as an alarm resolution tool after some anomaly detector (e.g. an X-ray device).

The main goal of this work was to check whether the ability of NNA/APT to determine concentrations of carbon, nitrogen, and oxygen in liquids inside hermetically sealed bottles can be used to achieve automatic discrimination between benign liquids, explosives (both those containing nitrogen and nitrogen-free), and flammable liquids.

2 Experimental Procedure

The experimental device consisted the following parts:

- Portable neutron generator NG-27 by VNIIA, Moscow, with built-in 36-pixel (6 × 6 matrix) α-particle detector by APSTEC.
- Three γ-ray detectors based on 3″ × 3″ BGO scintillating crystals and Hamamatsu 6233-01 photo multipliers.
- APSTEC's modular digital data acquisition system (DAQ).
- Compound shielding (lead + polyethylene) of BGO crystals from 14 MeV neutrons.
- Custom-made frame.

These components were arranged as shown on Fig. 1. The upper half of the broad "tagged" neutron beam defined by 18 out of the 36 pixels of the associated α-particle detector was directed towards the inspected bottle. The lower part of the beam was not used in the analysis, and its neutrons were absorbed in the shielding and did not reach the BGO detectors. The active 18 pixels (matrix 3 × 6), which defined the upper portion of the "tagged" neutron beam, were joined into three groups, so that the "voxel" corresponding to the central group covered most of the inspected bottle, and

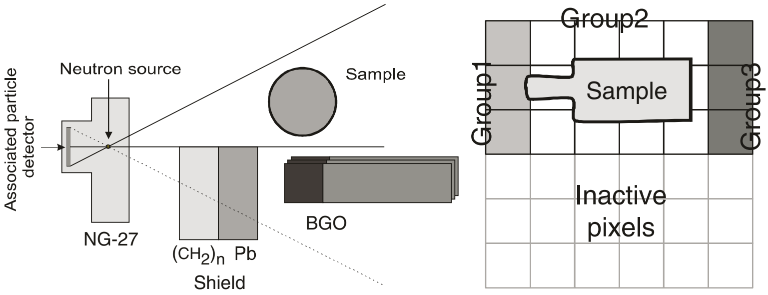


Fig. 1 Schematic drawings of the experimental set-up

the other two "voxels" contained mostly air, and were used as a reference. Joining pixels was used to increase the counting statistics, since for homogeneous materials such as liquids breaking them into separately analyzed "voxels" makes no sense. Gamma-ray detectors were located in a row right under the inspected samples.

The distance between the target of the neutron generator and the inspected sample was about 25 cm. Intensity of the neutron generator was about 2×10^7 n/s into 4π, about 500,000 n/s of which were directed towards the liquid inside the bottle.

The data acquisition system was placed on top of the frame, which housed the neutron generator, γ-ray detectors and the shielding (Fig. 2). It included a built-in computer, which accumulated and stored the experimental data and then transferred them via Ethernet cable to a remote computer for analysis. The operation of the device was fully automatic: it included data collection for 1 min, automatic energy calibrations of all γ-ray detectors, construction of energy spectra of γ-rays for each of the three groups of "voxels", determination of elemental concentrations from these spectra, and their analysis by a "fuzzy" logic decision-making engine [6].

The list of samples used in experiments is given in Table 1.

These samples represented four groups of materials:

- Standard solid nitrogen-containing explosives (TNT, C4).
- Nitrogen-free explosives (TATP).
- Flammable liquids and sprays (deodorant, acrylic paint, kerosene, benzene, solvent, white spirit).
- Benign liquids (cola, shampoo, toothpaste, shaving foam, yogurt, vodka, calendula solution).

Fig. 2 Experimental setup and investigated samples

Table 1 List of investigated substances

	Item	Quantity
1	TNT imitator in plastic bottle	200 g
2	C4 imitator in plastic bottle	200 g
3	TATP imitator in plastic bottle	200 g
4	Kerosene in plastic bottle	1,000 ml
5	Benzene in plastic bottle	1,000 ml
6	Organic solvent in plastic bottle	1,000 ml
7	White spirit in plastic bottle	1,000 ml
8	Acrylic paint in metallic bottle	<250 ml
9	Deodorant in metallic bottle	150 ml
10	Cola and cola light in metallic cans	330 ml each
11	Shampoo in plastic bottle	200 ml
12	Toothpaste in plastic tube	100 ml
13	Shaving foam in metallic bottle	200 ml
14	Yogurt in plastic bottle	100 ml
15	Vodka in glass bottle	500 ml
16	Calendula tincture in glass bottle	40 ml

Explosive substances were imitated by mixing melamine, water and graphite so that the C/N/O ratios of the mixtures were the same as for the corresponding explosives. Masses (volumes) of the samples ranged from 40 ml for calendula to 1,000 ml for kerosene and other flammable liquids.

Each 1 min-long measurement was repeated five times to make sure that the device produced stable results.

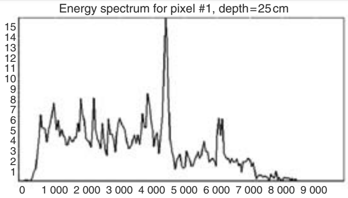
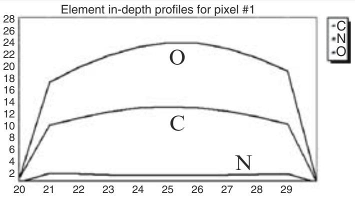
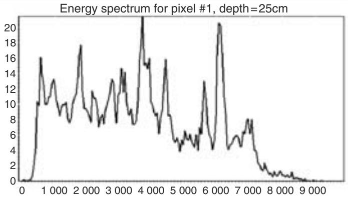
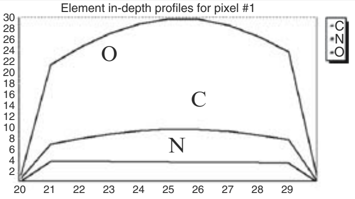
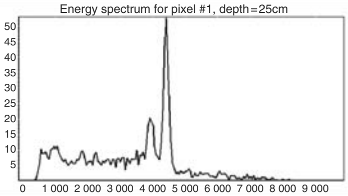
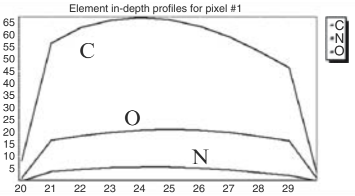
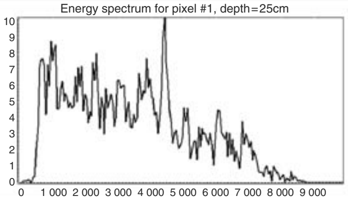
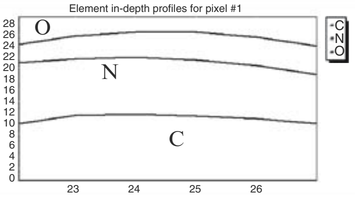
3 Experimental Results

The major advantage of NNA/APT over X-ray-based detection methods is its ability to determine the spatial distribution of many chemical elements in the inspected volume. These concentrations can be then automatically analyzed to produce a simple “yes/no” decision.

Table 2 shows examples of energy spectra of γ-rays measured in 1 min, and distributions of concentrations of carbon (C), nitrogen (N), and oxygen (O) along the depth axis for TATP, vodka, kerosene, and C4. The energy spectra are shown for the depth coordinate corresponding to the location of the samples (maxima of the concentration curves).

The experimentally obtained concentrations of carbon and nitrogen are rather close to their real concentrations in the samples. The concentration of oxygen is proportional to its real content in the samples, but is systematically overestimated for all samples. This reason is that some incident 14 MeV neutrons scatter in the material of the sample towards γ-ray detectors, and may react there with the oxygen of the $Bi_3Ge_4O_{12}$ (BGO) crystal, producing γ-rays that are detected with high efficiency by the same crystal. In the further analysis the obtained concentrations of oxygen were corrected for this effect.

Table 2 Energy spectra of γ-rays and distributions of concentrations of C, N, O along the depth axis for TATP, vodka, kerosene, and C4

	Energy spectrum of γ-rays	C/N/O distribution along depth
TATP		
Vodka		
Kerosene		
C4		

Distribution of all measured samples in the coordinates N/(C + O) versus C/(C + O) is shown on Fig. 3. These coordinates were chosen in order to remove the dependence of the results on the mass of the sample (normalization), and to accommodate the extreme cases when the sample contains either no carbon (e.g. water) or no oxygen (kerosene). The expected locations of hexamethylentriperoxiddiamin (HMTD) and nitroglycerine (NG), which were not among the measured samples, are shown by crossed symbols.

One can see from Fig. 3, that standard nitrogen-containing explosives (C4, TNT etc.) can be separated from innocuous liquids by C/(C + O) and N/(C + O) coordinates, even though error bars ("cloud" sizes) for small samples like for 40 g calendula solution may be quite large.

Flammable liquids containing almost no oxygen (kerosene etc.) or little oxygen (acrylic paint etc.) are well separated from innocuous liquids, which are for the most part water solutions. TATP with its C/(C + O) ~ 0.5 lies in between, and is also

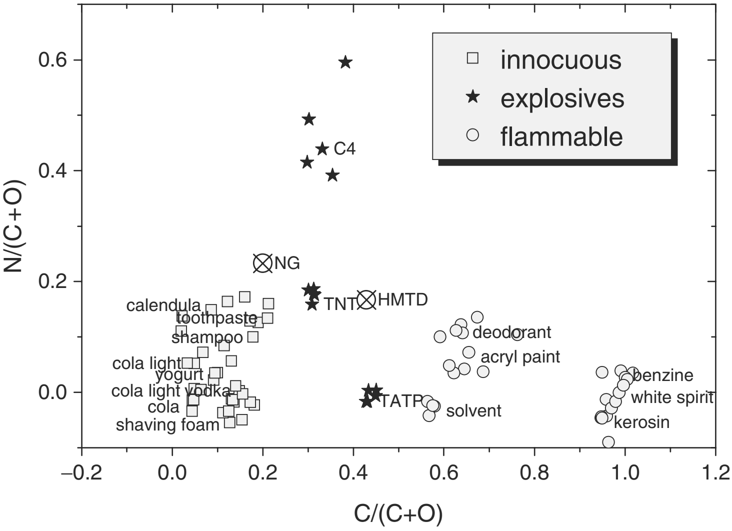


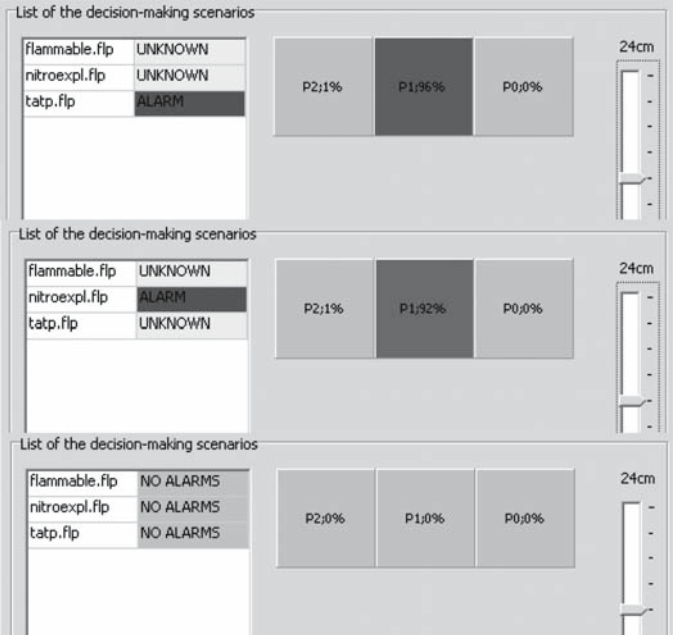
Fig. 3 Experimentally measured relative concentrations of nitrogen and carbon in the samples listed in Table 1. Each point represents a single 1 min-long measurement. Five measurements carried out for each sample form a 'cloud' of points; the size of this 'cloud' corresponds to experimental errors for the given sample

well separated from innocuous liquids. Although there are many innocuous materials, which are composed of carbon and contain little or no nitrogen and oxygen (e.g. many plastics, cotton, etc.), having such a substance in the liquid form almost guarantees that it is flammable. In opposite to that, common liquids containing oxygen and very little carbon are for the most part water solutions, which are not flammable or explosive (except hydrogen peroxide).

Figure 3 was used to define parameters of the automatic decision-making algorithm based on "fuzzy" logic, which analyzed elemental concentrations and produced an alarm if the measured concentrations matched one of the three scenarios: nitrogen-containing explosives, flammable liquids, or TATP.

Examples of the decisions made for TATP, C4, and vodka are shown on Fig. 4. The "fuzzy" logic scenarios were tuned so, that they included among possible decisions "Alarm", "Unknown", and "No alarm". These decisions in turn corresponded to red, yellow, and green color of the "semaphore", which represented the alarm level associated with each of the three "voxels" of the sensitive volume (see Experimental procedure section). The depth coordinate at which the highest alarm level was obtained is shown on the slider to the right of the "semaphore".

After the decision-making procedures were taught on a small subset of the training data, it was able to correctly identify 100% of all samples as innocuous, flammable, or explosive.



List of the decision-making scenarios

flammable.flp	UNKNOWN
nitroexpl.flp	UNKNOWN
tatp.flp	ALARM

List of the decision-making scenarios

flammable.flp	UNKNOWN
nitroexpl.flp	ALARM
tatp.flp	UNKNOWN

List of the decision-making scenarios

flammable.flp	NO ALARMS
nitroexpl.flp	NO ALARMS
tatp.flp	NO ALARMS

Fig. 4 "Semaphores" shown for TATP (top), C4 (middle) and vodka (bottom). Colors on this black-and-white figure are shown by brightness: from dark gray (red, ALARM) to light gray (green, NO ALARMS)

4 Conclusions

The device based on Nanosecond Neutron Analysis/Associated Particle Technique (NNA/APT) can quantitatively analyze concentrations of carbon, nitrogen, and oxygen in liquid and solid samples in sealed glass, metallic and plastic bottles.

The precision that can be reached in 1 min-long measurement was shown to be enough to distinguish between innocuous, flammable, and explosive samples weighting from 40 to 1,000 g.

The decision-making procedure based on "fuzzy" logic was tuned to make detection fully automatic.

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Some Detection Procedures for Liquid Explosives

M. Stancl and M. Kyncl

Abstract The work is focused on the possibilities to minimize the danger of misusing liquid explosives for terrorist attacks. Several detectors of explosives are described here, including their working principles, capable of detecting some liquid explosives. The results of experiments accomplished using several detection procedures are presented, reliability and detection limits are considered.

Detection of liquid explosives by means of the agents producing colour reactions on contact with explosives was studied and the results obtained with some improvised liquid explosives were evaluated. Special attention was paid to liquid explosives consisting of a mixture of nitro groups containing aliphatic compounds, or nitrates with other components and, as another group, emulsion and slurry explosives. The colour reactions seem to be able to provide warning signal for a wide range of such liquid explosives, where standard detection techniques often are not very effective.

Keywords Liquid explosives, detectors of liquid explosives, detection procedures, security technologies, First Defender, Detection Kit DETEX

1 Annotation

The work is focused on the possibilities to minimize the danger of misusing liquid explosives for terrorist attacks. Several detectors of explosives are described here, including their working principles, capable of detecting some liquid explosives. The results of experiments accomplished using several detection procedures are presented, reliability and detection limits are considered.

Detection of liquid explosives by means of the agents producing colour reactions on contact with explosives was studied and the results obtained with some improvised liquid explosives were evaluated. Special attention was paid to liquid explosives

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Explosia a.s., Research Institute of Industrial Chemistry, Pardubice – Semtín, Czech Republic

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consisting of a mixture of nitro groups containing aliphatic compounds, or nitrates with other components and, as another group, emulsion and slurry explosives. The colour reactions seem to be able to provide warning signal for a wide range of such liquid explosives, where standard detection techniques often are not very effective.

2 Introduction

Facing a permanent threat of terrorist attacks of various extremist groups the countries worldwide are forced to spend huge funds on improving the protection of human life and possessions. The extremists keep looking for still new means for realization of the attacks. Weakness of the existing protective measures came out again in August 2006, when a terrorist plot was revealed to blow up ten airliners departing Great Britain for the United States by means of liquid explosives [1]. The attack was fully foiled but that attempt showed urgent necessity to improve detection system, especially in the field of explosive liquids, flammable liquids and the liquids which can be used as explosives precursors (e.g. nitric acid of concentration at least 68%, hydrogen peroxide of concentration at least 30%). The danger of misuse of flammable liquids or liquid explosives appears in the 1980s (hijack incidents). Probably the earliest well documented use of liquid explosives is dated back to January 1987 when bottles with methylnitrate were discovered and seized at Frankfurt Airport [1]. At that time the study started of X-ray, electric, electromagnetic, nuclear magnetic, optical, acoustic and nuclear methods enabling to distinguish "innocent" liquids from liquid explosives and flammables. On the basis of the results achieved, many types of detectors were developed capable of detecting dangerous liquids differing, however, in their performances in terms of e.g. number of right and false alarms, the possibilities to detect liquid explosive in a sealed container or acceptable detection time.

The survey of some commercial devices designed for detection of liquid explosives is given in the next chapter.

3 Some Commercial Devices Capable of Detecting Liquid Explosives

Many companies all over the world (and particularly in the U.S.A.) have liquid explosives detectors in development or ready for deployment. One of such devices is *the First Defender (Ahura Corporation)*, a handheld liquid explosives detector, capable (with certain limitation) of detecting liquid explosives in sealed containers. The First Defender operates on the principle of Raman spectrometry – a contactless and non-destructive method for identification and characterization of chemical composition and structure of the material. The Raman effect originates at the moment of incidence of monochromatic laser beam on the material investigated. The light scattered by this material displays frequency changes corresponding with the frequency of vibrations of atoms in the material. Analysis of frequency shift of the light spectrum indicates chemical composition and structure of the investigated material. The measured scattering patterns (Raman spectra) of unknown samples are compared with reference spectra database. As from September 2006 the First Defender can detect the scattering patterns of 2,500 substances, including all known liquid explosives and dangerous stuffs (e.g. chemical weapons including sarin or mustard gas).

The above mentioned limitation concerns the incapability of laser beam to penetrate opaque materials, i.e. if the explosive is housed in e.g. ceramic or metallic container, the First Defender will not detect it.

Weight of the First Defender is 1.6 kg and the price is $30,000. Ahura Corporation plans to develop even smaller, cell-phone size device.

A series of measurements was accomplished with the First Defender detector and the results are given in the Experimental Work.

There are also softwares working in coordination with X-ray machines to analyze substances pixel by pixel and alert the personnel when the analysis of an object in a carry-on bag has the "pixel signature" of an explosive material [2].

The DefenderTech International Solutions PROTADAS™ is the detector working on the basis of passive millimetre wave, capable of identifying the types of containers that might be used to house liquid explosives, even if concealed beneath clothing or "body suits". The Protadas™ threat detection system camera provides further, more comprehensive defense against liquid explosives placed in containers made of plastic, composite, ceramic, metal or non-ferrous metal. Further, the system would detect devices of the size and materials most commonly used to construct non-liquid explosive-based bombs. This detection happens automatically at full motion speeds as a subject passes through the camera's view. The users can specifically target and isolate persons carrying objects that may be liquid explosives or other types of suicide bombs, before those persons can harm people or damage property [3]. Some other security devices (mostly in development) that can analyze contained liquids are based on such methods as radio wave and microwave bombardment. But these methods have also certain shortcomings.

UNI Dai-Ichi Shoji Co. Ltd. offers a portable type of screening device for liquids contained in plastic bottles. This detector is capable of distinguishing safe, water-based drinking liquids (e.g. mineral water, coffee etc.) from dangerous, flammable or explosive liquids even if only a small quantity of the liquid is at disposal. The detection method consists in microwave bombardment of an unknown liquid material (contained in a plastic bottle) and its identification through evaluation of specific changes in the wavelength of reflected microwaves, caused by dielectric constant or electric conductivity of the liquid tested.

Certain drawback of this method is that microwaves cannot penetrate metal containers (aluminium cans to some extent only) [4].

Haz Mat ID portable chemical identifier is a highly specific tool that measures how unknown liquids, powders or solids interact with infrared light. Each substance has its own unique infrared finger print which, when analyzed by the *Smiths Detection Company's Haz Mat ID*, is compared against an onboard library database to provide identification. Analysing a sample is easy, a drop or a few grains of the unknown substance are placed on the small diamond sensor. A simple touch-screen program walks the responder through a complete analysis in less than 20 s [5].

Pacific Northwest National Laboratory in Richland has created a device which uses non-invasive ultra sound (ultrasonic waves) to rapidly and accurately identify any suspicious liquids with the detection time of about 3–4 s. The device is patented under name *HAZAID* (Hazardous Material Acoustic Inspection Device) [6].

ICx Nomadics has developed a detector based on specially formulated chemically sensitive films, fluorescing under certain conditions. The film coats inside of a glass tube, through which vapour is drawn. The glass walls of the tube guide the light from the film to the light sensor, which registers changes in light intensity. In addition to that, hollow fibers have been developed that can guide the light much better than the glass tubes used, and also light detectors capable of incorporating directly into these fibers. The company is also developing polymers for detecting a variety of toxic industrial chemicals.

The ICx Nomadics detectors, working by sensing minute amounts of vapours only, represent a novel, potentially faster and more accurate way to detect liquid explosives [7].

Scanna's hand held liquids detector Liquidscan is a unique innovation in the field of contactless detection of flammable and explosive liquids contained in sealed containers made of plastic or glass and other non-metallic materials (out of metal materials only aluminium).

The detector utilizes the principle of quasistatic electrical tomography. This technique estimates the spatial distribution of electric properties of the unknown medium and can determine the characteristics of a liquid irrespective of the size of container. Electrical properties of the liquid (permittivity and conductivity) determine whether the liquid is either flammable or explosive. The device is completely electronic and does not contain any radioactive or microwave source or any other potentially dangerous parts.

Liquidscan is very easy to use, it is only necessary to simply hold the sensor of the device close to surface of the checked container below the level of a liquid and press the button.

Green light of the indicator shows that the liquid is not flammable, red signal indicates potential danger of the contents (explosive or flammable liquids).

The *Liquidscan* is a small device of dimensions 135×70×24mm and weight 110g [8].

The next method used for detection of liquid explosives is nuclear magnetic resonance (NMR), providing detailed spectral information on the molecular level. Low-resolution NMR provides substantial chemical information regarding liquids. Specific parameters available from low-resolution NMR include the signal amplitude (A_0), the spin-lattice relaxation time (T_1), the spin-spin relaxation time (T_2), the diffusion constant (D), and the spin-spin coupling constant (J). Sequences of RF pulses can be designed to respond to one or more of these parameters and therefore unique NMR signatures for various liquids can be defined.

NMR Liquid Explosives Screening System for liquid explosives capable of scanning unopened bottles has been developed [9].

This system can detect specific explosives directly, or to verify the labelled or bar-coded content of a container. NMR produces extremely rich data set and many parameters of the NMR response can be determined simultaneously. As a result, multiple NMR signatures may be defined for any given set of liquids, and the signature complexity then selected according to the level of threat [10].

A totally different kind of scanning technology that has already begun to emerge is backscatter screening. Backscatter screening is much like traditional X-rays, except that the system sends more, but weaker X-rays at an object. They cannot penetrate skin, but can reveal items under someone's clothes such as a hidden bottle of liquid. Major problem is that the view is so powerful that an individual's private parts can be seen, which forced the TSA to delay tests while vendors tweaked the machines' programming to distort or mask bodily images. Backscatter systems still leave it up to a human screener to recognize a suspicious item [11].

Chinese producer *NUCTECH* introduces the newly developed machines to spot liquid explosives at China's 147 civil airports to protect travellers during 2008 Beijing Olympic Games from terrorist attacks. The detector *TH SCAN LS 8016* is sold at $200,000 a unit on the international market [12].

Very cheap, fast and simple method of detection of liquid explosives is detection system based on colour reactions of detection solutions with the unknown liquids examined one of such systems is the *Verifier*™(CHemSee Inc.). The determination if a liquid is flammable and/or can be used as explosive is accomplished by simple putting 2–12 droplets of the sample on the Verifier ™ and looking for a colour change. The colour changes in 2–12 s. Red colour forms if the sample contains a flammable material (acetone, gasoline), black colour forms if the sample contains e.g. hydrogen peroxide. The function of the *Verifier*™ system was tested using many flammable liquids, common liquids containing e.g. hydrogen peroxide, acetone or nitromethane and also many common innocent liquids to ensure that the system does not produce false responses. Non–hazardous liquids such as water and lemonade do not cause the colour to change. *The Verifier*™ set can contain also *Gel-Ox*™ *Tabs* (detect hydrogen peroxide in gels and thick liquids) or *On The Spot*™ *Tabs* which detect traces of nitrates and nitrites [13].

Similar system for easy detection and identification of explosives called *DETEX* is produced by the Czech company *Explosia a.s*. The kit contains six small bottles with detection solutions enabling detection of common military, commercial and improvised explosives, including liquid explosives and explosives based on organic peroxides (TATP, HMTD) through chemical colour reactions. Big advantage of this method is its cheapness if compared with other detection systems. Price of the *DETEX* kit is about 160 Euro (2007) [14].

The results of experimental testing of *DETEX* kit in terms of possible detection of liquid explosives are given in the chapter Experimental Work.

4 Development Trends in the Field of Security Technologies

Radical shifting has been perceptible in the last 5 years in the methods and technologies used for protection of airlines terminals and aircraft against terrorist attacks. Relatively complex solution in this field offers American Transportation Security Administration (TSA), dealing with the questions of safety in transportation, related with the all ICAO member states accepted standard 4.4.8, containing the provision about the requirement for 100% luggage check since 01. 01. 2006. No less important questions are protection against suicide bombers and detection of liquid explosives, of course with utilization of up to date security technologies. The detection methods for explosives, used in the last decades (physical inspections, detection by means of sniffing dogs or conventional X-ray apparatuses) are replaced with new technologies, where as the primary method has been adopted computer tomography (CT) based on automated detection systems of explosives (EDS). Only in the U.S.A. more than 1,500 of these certified systems have been utilized since 2001 for luggage inspection in the main metropolitan airports. With regard to high financial costs for implementation of these systems, less expensive systems are used for smaller airports, of electronic detection of traces of explosives (ETD). Approximately 6,000 of such equipments are in operation.

The part of EDS are sophisticated portals combining detection of traces of explosives and metals, or new, above mentioned CT technologies which enable, through the utilization of multiplied sources, either to X-ray the luggage from three various directions or to use the apparatus with X-ray diffraction. The luggage can be then imaged in three dimensional virtual model with colour differentiation of the materials in question. The detectors of this type called RAPISCAN SYSTEMS RTT 120 CT are tested in UK airports at operation rate of about 1,500 pcs of luggage per hour.

Another step to increase the safety in dangerous zones is detection of explosives prior to entering the detection systems. This requirement for the so called Stand Off Detection is, for the time being, limited by utilization of millimeter waves technology, capable of detecting of weapons or explosives hidden under clothing even from the distance of 25 m, as the case may be the programmes can be used analysing behaviour of suspicious persons or to utilize biometric systems. There has been intensive research ongoing in this field, which is documented also by the information from the 16th meeting of Ad Hoc Group of ICAO specialists in September 2006. The research is aimed at examination of electromagnetic spectra, by means of which remote identification is possible of hidden anomalies. By using AM radio frequencies the explosives can be detected according to their quadrupole resonance. This technology does not belong directly to “Stand Off Detection”, because the object has to be placed in a chamber similar to telephone box for a few seconds. When shifting to higher wavelengths (to the area of mm waves) approximately 94 GHz, we obtain imaging system, capable of penetrating through clothing and working in active or passive mode. When shifting to even higher frequencies up to terahertz (300–3,000 GHz) the spatial image of the object gets even better. There is a potencial for terahertz spectrometers and it could lead to extraction of specific spectral characteristics of explosives and their differentiation from common objects. This technology is attractive primarily for the fact, that in terahertz area reduction could be assumed in the price of radiation source and detectors. When examining the areas with even shorter wavelengths, i.e. in infrared area, the human body appears to be a strong heat source and so it is possible to gain image by means of relatively cheap cameras. Penetration through clothing in this wavelength area is however rather problematic. In visible spectrum the clothing is still non-transparent, however, potentially this method is feasible with the aid of active interaction with utilization of either remote Raman scattering or laser, but this technology requires more intensive external source that can endanger human sight [1].

5 Experimental Work

5.1 FirstDefenderXL

5.1.1 Introduction

Raman laser spectrometer FirstDefenderXL (Ahura Corporation) utilizes the state-of-the-art technologies from the field of microelectronics and telecommunication technologies. In addition to that the optics used, with very low Stray Light, enables also measuring in the proximity of excitation laser wavelength. Quite a new mechanism of substances identification was also designed for this spectrometer – search in spectral databases, significantly suppressing the errors of positive identification (false signal of positive identification), capable even of managing mixtures of compounds (up to five main components).

The spectrometer makes it possible to measure directly powder materials, liquids, suspensions as well as foils, to identify material of plastic containers etc., but in many cases it is possible to realize measurement through the packing (e.g. glass bottles, also coloured etc.). It is not possible to measure gases and ion compounds (primarily diatomic, e.g. NaCl, KBr, etc.).

This spectrometer enables detection of more than 3,500 dangerous substances, explosives, drugs, plastics, white powders etc., also in their mixtures. The database of the apparatus is easy to extend by further substances, necessary to be identified. The main merit of Raman spectrometry is the fact that there is no water interference and that it is thus possible to analyse also solutions or wet samples.

The measurement can be realized in three ways: contactless measurement from the distance of approx. 1.5 cm (focal length), utilization of the cell with sample, or the use of optical probe connected with the apparatus by means of 1 m long optical cable. The probe is of robust construction and can be easily mounted, removed and decontaminated. No further change of adjusting is required after connecting to the apparatus.

The probe is suitable primarily for:

- Fast measurements on difficult to reach places
- Identification of compounds in vessels, drums and waste containers
- Accurate screening of heterogeneous mixtures

The probe can be easily connected to laser head of the First Defender, with the same measurement accuracy and reproducibility being ensured as in standard measurement modes. The probe is suitable for measurements in the places difficult to reach or for safe measurement of explosive compounds. The probe operates in point-and-shoot regime and utilizes micro-optics, filters and the cover with special surface treatment. The device is marked by a long-term stability of measurement.

In April 2–8, 2007 the tests were accomplished in cooperation of Explosive Ordnance Disposal Division of Police Presidium of the Czech Republic and Population Protection Institute Lazne Bohdanec, of explosives and auxiliary materials (primary high explosives, explosives, propellants, military and commercial explosives, auxiliary materials added to explosives). The objective was to ascertain the measure of utilizability of Raman spectrometer for the needs of criminological-technical expertise investigation – fast identification of explosives in the scene of the crime (house searches, withheld booby-trap system, rests of explosives after explosion of booby-trap system, etc.)

5.1.2 Test Conditions

Altogether 129 samples were tested of explosives and auxiliary materials used in industry, IED or loaded in ammunition to be found in the territory of the Czech Republic, namely: 2 samples of primary high explosives (organic peroxides), 7 samples of liquid explosives, 38 samples of chemical individua, 50 samples of military and commercial explosives, 10 samples of unknown or not specified explosives, 3 samples of propellants, 4 samples of pyrotechnic compositions or their components and 15 samples of auxiliary materials.

Measurement of samples was accomplished twice, with various levels of laser output according to the type of examined sample (50, 150, 300 mW). Firstly the examined materials were always measured using external sensor through the container, predominantly of transparent laboratory glass. Second measurement was accomplished either in the cell or by adjusting the laser output.

Preparation of the apparatus for measurement takes approximately 1 min. Laser irradiation of samples is discontinuous, no significant heating occurs – except for the materials of black colour or if the measurement time is very long, a great number of the samples were measured in up to 1 min time. In case of a sample with supposed measurement time varying above 40 min, such a measurement did not lead to successful identification and was repeatedly tried with the sample being placed into the cell, under compacting, by transfer to solution etc. Only exceptionally such a sample was successfully identified.

In case the sample had been placed in a cell to the height of approximately 0.3–1 cm the measurement was faster and the spectra were more identifiable and matched more accurately to the data in database. With reduced laser output the measurement time got longer at some samples, but not by more than several tens of seconds or several minutes.

5.1.3 Results

When measuring primary high explosives the spectrometer operated accurately and very fast. Even in case of samples, the compositions of which were not saved in database, i.e. that it was not possible for the spectrometer to define the unknown substance. Even the spectra of the substances not saved in the database were readable. The only not identified material was nitrocellulose.

Inaccuracies in evaluation occurred, however, when measuring military and commercial explosives (percent of substances in the sample, not defined main component, especially ammonium nitrate, detection of compounds not present in the sample). Measurement time varied predominantly up to 1 min except for some plastic explosives, where measurement time grew longer up to 30 min.

Other nitrates – most often silver nitrate and thallium nitrate – appeared in the result at a number of commercial explosives, which however follows from the closeness of spectra of individual nitrates.

At some plastic explosives the identification was not possible (Semtex 1 H, PlNp 10, PlHx 30, Semtex 2, PL U-EP 14, TKN 10). There are several probable reasons: presence of plasticisers, dye, content of aluminium or carbon black.

Number	Sample	Characteristics	Result		Evaluation
Chemical individuum					
1.	HMTD	Dry in vial	HMTD	50 mW < 1 min	
2.	TATP	In solution H_2O and ethanol	TATP	150 mW < 1 min	
3.	NG	40% in ethanol	NG	50 mW < 1 min	
4.	Methylnitrate	20% in ethanol	Not found	300 mW < 1 min	not in database
5.	Nitromethane		Nitromethane	150 mW < 20 s	
6.	DMDNB	2,3-dimethyl-2,3-dinitro- butane	2,3-Dimethyl-2,3-Dinitrobutane	50 mW < 1 min	
7.	p-MNT	p-mononitrotoluene	p-Mononitrotoluene	300 mW < 30 s	
8.	2,4 DNT	2,4-dinitrotoluene	2,4,DNT	300 mW < 20 s	
9.	TNT	2,4,6-trinitrotoluene	2,4,6-TNT	300 mW < 20 s	
10.	Tetryl		Tetryl	300 mW < 1 min	
11.	Pentrite K		Pentrite	50 mW < 20 s	
12.	Hexogene		Hexogene	50 mW < 1 min	
13.	Nitrocellulose dynamite		Not identified	300 mW too long measurement time	Not identified
14.	Ammonium nitrate		AN	300 mW < 1 min	
15.	Nitroquanidine		Nitroquanidine	300 mW < 1 min	

(continued)

(continued)

Number	Sample	Characteristics	Result		Evaluation
Military and commercial explosives					
16.	PERMONEX V 19		TNT 32% DA 23%	300 mW < 1 min	
17.	Perunit 28	NGG 28% TNT 4.4% DNT 4% DA 60% NC 1.3% wood flour 2.3%	DA 62% TiNO$_3$ 23%	300 mW < 1 min	
18.	Semtex 1A	PETN 83% binder 17% (butadiene rubber mineral oil)	PETN20	300 mW < 1 min	
19.	Semtex 10 SE	PETN 85.5% binder 14.5% (acrylonitrile rubber dibutylphthalate)	PETN	300 mW < 1 min	
20.	Semtex 1H	RDX 50% PETN 35% binder 15% (butadiene-styrene rubber, mineral oil)	Not identified		Not identified
21.	PlNp 10		Not identified		Not identified
22.	PlHx 30		Not identified		Not identified
23.	Semtex S30		Na$_2$CO$_3$ 56% PETN 10%	300 mW < 1 min	
24.	A-IX-1	RDX 94% wax 6%	RDX	300 mW < 1 min	
25.	A-IX-2	A-IX-1 80% Al 20%	Not identified		Not identified
26.	C4 (USA)		RDX	300 mW < 1 min	

(continued)

(continued)

Number	Sample	Characteristics	Result		Evaluation
Propellants					
27.	Black powder		Burn up	300 mW < 30 s	BURN UP !!!
28.	Ng powder	Granulated thermostable	Burn up	300 mW < 1 min	BURN UP !!!
29.	NC powder		Burn up	300 mW < 1 min	BURN UP !!!
Additives for commercial explosives					
30.	Dibutylph thalate	Through container	Dibutylphthalate	300 mW < 1 min	
31.	Ammonium nitrate crystalline		AN	300 mW < 1 min	
32.	Calcium carbonate		$CaCO_3$	300 mW < 3 min	

At the materials of black colour absorption occurs of laser radiation and heating of examined sample
Not identified – too long measurement time or no spectrum
Not in database – spectrum defined, identification of the substance impossible, not in database
BURN UP – the material initiated in the course of analysis

When measuring propellants, all examined samples burned up (1 sample of black powder, 2 samples of thermostable nitrocellulose and nitroglycerine powders). In spite of the fact that no sample burned up when the laser output was reduced to 50 or 150 mW, positive identification was not accomplished. Unfeasibility of measurement and subsequent identification of these materials corresponds with the above mentioned limitation of the apparatus. This method is not suitable for identification of these materials.

Tables with some results of measurements are given in the following chapter.

5.1.4 Survey of Results

5.1.5 Conclusion

The results of the tests proved the capability of very fast and unambiguous identification of explosives, especially chemical individua, except for propellants and some plastic explosives. The rate of measurement of examined materials varied predominantly up to 1 min. This method will not replace the necessity of further investigation in the field of chemistry, but for the first, in situ accomplished identification of unknown materials it renders invaluable service.

Successfulness of measuring and identification for primary high explosives and chemical individua of explosives was 97.8%. The only unidentified material was nitrocellulose.

Successfulness of identification at military and commercial explosives was 80%. Identification was very problematic especially at plastic explosives (Semtex 1 H, PlNp 10, PlHx 30). There are several probable reasons: presence of plasticiser, dye, content of aluminium or carbon black.

At propellants (various types of black powder and smokeless powders), the burn up occurs after a very short time especially when measuring in the cell. When measured with external probe the burn up occurred at laser output 300 mW. At lower output values no burn up occurred, but the materials were not identified. For the above mentioned reasons the other prepared samples of propellants were withdrawn from further testing.

Total successfulness at measured samples (including the repeated ones) was 82.9%, out of 129 samples, 22 were not measurable and identifiable.

5.2 Detection Kit Detex

The kit contains six bottles with detection agents used for detection of unknown samples either individually or in combination (always to start with agent No.1). DETEX kit is shown in Fig. 1.

Fig. 1 Detection Kit DETEX

- **Agent No. 1** – NaOH ethanol solution – on the basis of Janowsky reaction with aromatic polymetanitrocompounds this agent produces intensively coloured compounds of quinoid structure, known as Meisenheimer complexes. The colouring formed depends on the structure of aromatic nitrocompound and also on its concentration.
- **Agents No. 2 and 3** – active and passive component of Griesse agent. By saponification of nitroesters or nitramines by means of inorganic hydroxide (agent No.1) nitrite anion is released which, after addition of agent No.2, diazotates aromatic amine and, subsequently, after addition of agent No.3 coupling reaction occurs with the coupling agent present under formation of colour diazocompound. The reaction produces pink colouring. It is necessary to mention that the tint of the colour formed is absolutely independent of the type of the nitroester or nitramine analyzed. It is solely defined by the type of the active and passive component used. However, this reaction cannot – if compared with determination of presence of various types of nitrocompounds by the agent No.1 – specify if the material examined contains nitroester or nitramine.
- **Agent No. 4** – water suspension of powder Zn – serves to detect inorganic nitrate, which does not produce any colour reaction after stepwise additions of agents No. 1, 2 and 3. Only after addition of agent No.4 reduction occurs of inorganic nitrate to nitrite and the nitrite causes, on the basis of Griesse reaction, formation of pink colouring, the chemical composition and also the tint of which are identical with the colouring produced by nitroesters and nitroamines with agents No. 1, 2 and 3.
- **Agent No. 5** – diphenylamine solution in sulphuric acid – with inorganic chlorates (in higher concentrations also with perchlorates) produces intensive blue colouring.
- **Agent No. 6** – diphenylamine solution in sulphuric acid with ferrous-ammonium sulphate for detection of peroxides.

5.2.1 Detection of Liquid Explosives

The possibilities were tested to detect liquid explosives with the following findings:

(a) **Nitromethane** – with detection agents DETEX No. 1, 2 and 3 produces pink to red colouring, the intensity of which increases with time.
(b) **Methylnitrate** – with detection agents DETEX No.1, 2 and 3 produces the same pink colouring as other nitroesters and nitramines based explosives, it reacts instantaneously.
(c) **Hydrogen peroxide 3% solution** – as other organic peroxides, with detection agents DETEX No. 5 and 6, hydrogen peroxide produces blue colouring.
(d) **Hydrogen peroxide 30% analytical grade with alcohol in ratio 1:10** – after application of detection agents DETEX No. 5 and 6 blue colouring appears.
(e) **Emulsion explosives** – containing as detectable additive a mixture of inorganic nitrates, produce red colouring with agents DETEX No. 1, 2, 3 and 4.
(f) **Liquid nitroesters** – (nitroglycerine, etylenglycoldinitrate) produce standard pink colouring with detection agents DETEX No. 1, 2 and 3.

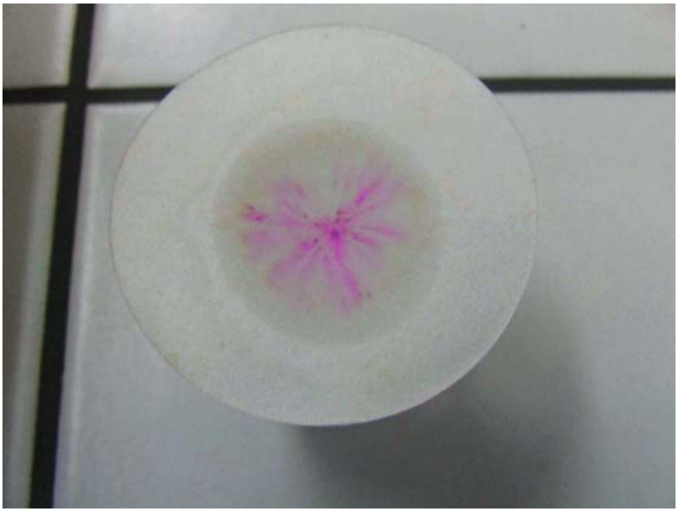

Fig. 2 Colouring produce by nitromethane, methylnitrate, emulsion explosives and liquid nitroesters with DETEX agents

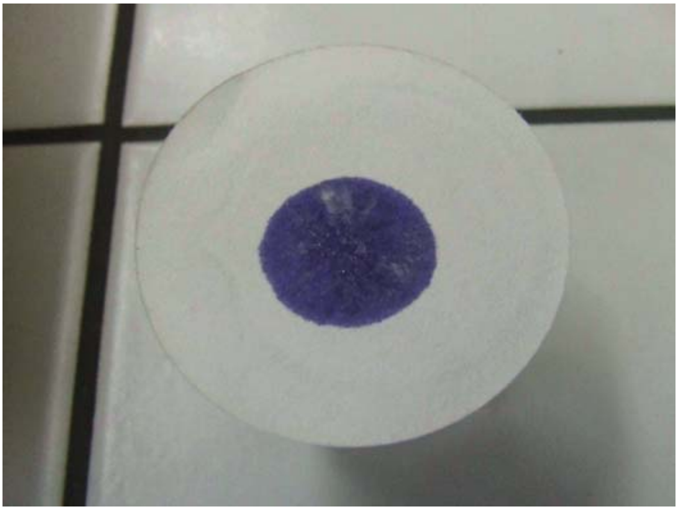

Fig. 3 Colouring produce by hydrogen peroxide with DETEX agents

(g) **False signals test –** test samples: gin and whisky – at both samples no colouring appeared after application of detection agents DETEX No. 5 and 6.

As experimentally verified, at all above explosives the amount sufficient to produce colour reaction with DETEX agents is 5.10^{-7} g of the explosive detected. Samples of explosives (a), (b), (e) and (f) produce colouring shown in Fig. 2, samples (c) and (d) produce colouring shown in Fig. 3.

5.2.2 Automatic Evaluation Device

A new, miniaturized and very simply controllable automatic evaluation device has been developed in the Research Institute of Industrial Chemistry (VÚPCH), processing the spectra of colour compounds of samples of unknown explosives with detection agents of DETEX system in visible region (Fig. 4). The spectrum of the colour compound (dependence of absorbance on wavelength within 400–700 nm) is automatically compared with colour spectra reference database, saved in the device memory, and several seconds after measurement the result is visualized on LCD display.

To illustrate the detection method utilizing the measurement of colour reactions in solutions by means of spectrophotometer, 1 ml of 3% water solution of hydrogen peroxide was placed in the beaker, 1 ml of detection agents DETEX No. 5 and No. 6 were added under formation of blue colouring (Fig. 5). The solution was transferred into the cell (1 cm) and the colouring was measured. The spectrum (dependence of absorbance on wavelength) is shown in Fig. 6.

6 Conclusion, Future Work

On the basis of results of experimental work it can be stated, that the detector tested, "First Defender" and detection kit DETEX offer certain possibilities of detection of liquid explosives even if either means is of different level (in terms of technology, methodology as well as economy).

As obvious from the above mentioned, utilization of some areas of electromagnetic spectra for detection of bombs or explosives (including liquid explosives) hidden under clothing appears to be quite real at present or in the near future.

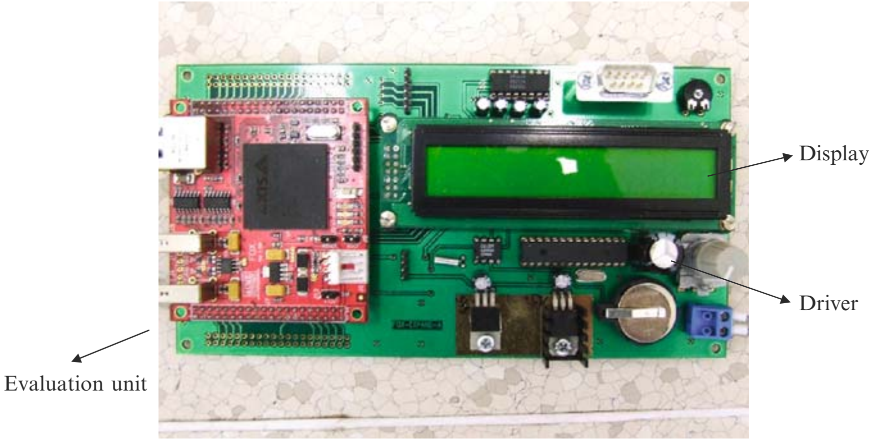

Fig. 4 Automatic Evaluation Device processing the spectra of compounds

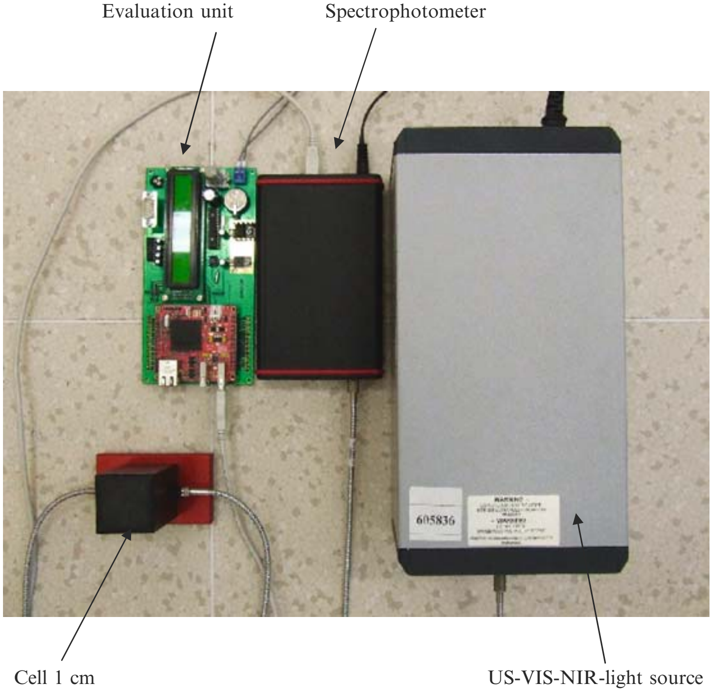

Fig. 5 Automatic Evaluation Device processing the spectra of compounds

It is however necessary to concentrate the effort on gaining optimal radiation sources and high performance detectors covering the whole area of interest. Automated system should be created, enabling identification of the object with subsequent decision on further action.

So, the detection system developed, based on evaluation of electromagnetic spectra, shall be above all feasible, functional, reliable and safe, but, with regard to supposed utilization, also of reasonable price.

It can be said that for the time being there is no absolute method of detection of contained liquid explosives, but permanent intensive development in this field holds promise that such detection method could be on the horizon.

Acknowledgments This work has been accomplished in Explosia a.s., Research Institute of Industrial Chemistry with support of the Ministry of Industry and Trade, Czech Republic.

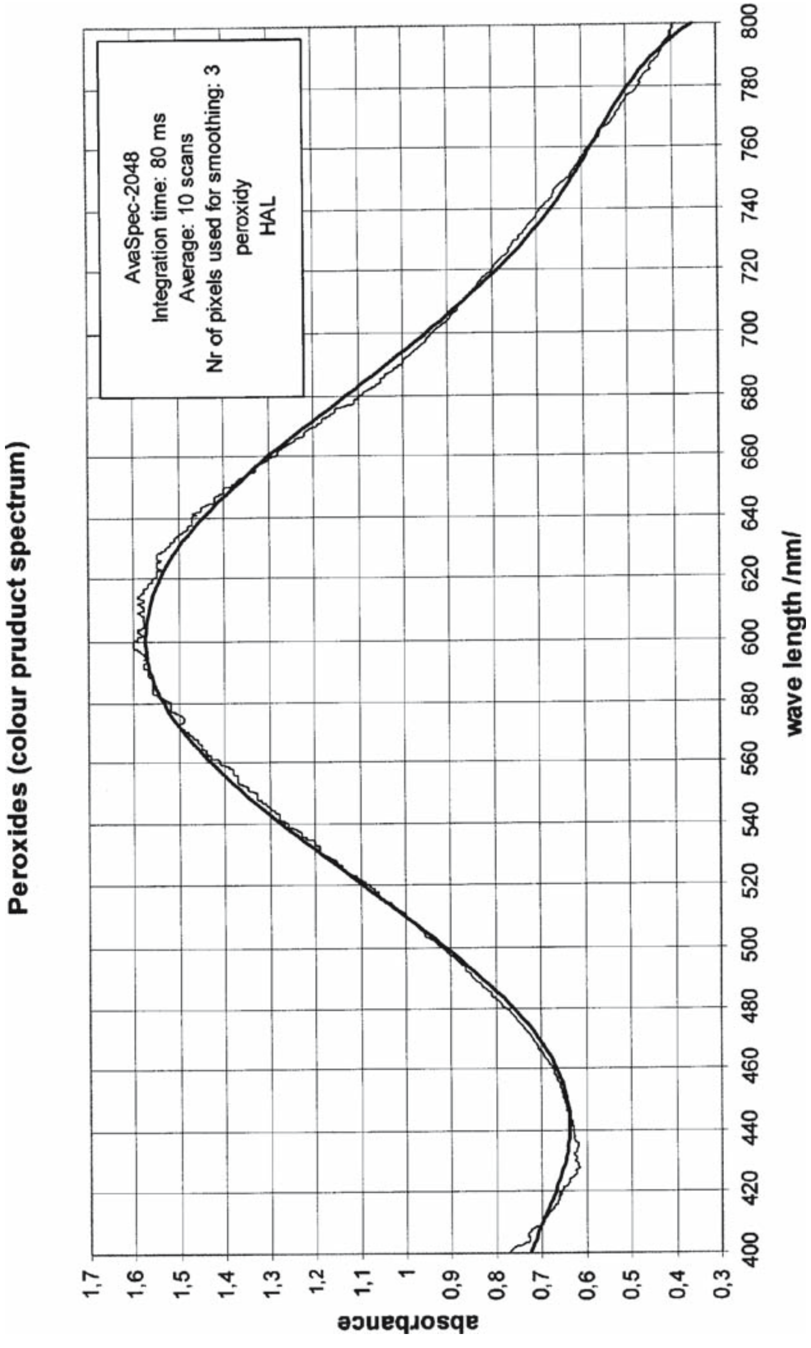

Fig. 6 The spectrum of 3% water solution of hydrogen peroxide

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Detection of Explosives Using Nuclear Resonance Absorption of Gamma Rays in Nitrogen: A Russian/US Collaboration

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Abstract A summary of the work performed to date is presented. The project brings together accelerator scientists in the US and Russia to develop high-current charged-particle accelerators for applications to explosives detection. These include an electrostatic tandem accelerator of novel design and a compact cyclotron and storage ring with energy compensation and electron cooling.

Keywords Explosives detection, compact cyclotron, proton storage ring, electron beam cooling, vacuum insulted tandem accelerator, Gamma-ray Resonance-absorption (GRA), Nuclear Resonance-absorption (NRA), Pulsed fast Neutron Analysis (PFNA)

1 Introduction

Recent technical developments encourage us to revisit a nuclear-based explosive detection technology that demonstrated great potential in the early 1990s but lacked the advanced accelerator technology required for practical applications. The gamma ray resonance absorption (GRA) method provides highly penetrating gamma rays from a point source that are strongly and preferentially absorbed in nitrogen, a major constituent of most high explosives [1]. Pending the advancement of accelerator technology to meet demands for high-quality, high-intensity proton beams, however, GRA has remained a laboratory curiosity for the most part.

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The Putin/Bush summit of May 2003 provided an incentive for US/Russian collaboration to combat terrorism. In June 2003 Russian and US scientists convened a workshop in Moscow under the auspices of the Department of Energy's Nuclear Warhead Safety and Security Exchange (WSSX) agreement. The workshop identified technologies relevant to counterterrorism for which cooperation could lead to mutual advantage. Among other technologies, Russian accelerator expertise was recognized as among the most advanced in the world.

Accelerator scientists and nuclear physicists from Russian institutes and American national laboratories are now working together to develop several accelerator variants capable of producing high-quality, mega-voltage, multi-milliampere proton beams. Participants include the All Russian Institute for Technical Physics (VNIITF) in Snezhinsk, the Joint Institute of Nuclear Research (JINR) in Dubna, the Budker Nuclear Physics Institute (BINP) in Novosibirsk, the Research Firm IFI of Moscow, and Los Alamos National Laboratory.

In this paper we summarize the progress achieved in the several accelerator technologies selected to address these requirements. The technologies include a high-current electrostatic variant, the Vacuum Insulated Tandem Accelerator (VITA) at the BINP; a compact cyclotron at JINR; and a storage ring, possibly with an internal target, energy compensation, and electron cooling, at BINP and JINR. Supporting this work and coordinating the scientific efforts is the Research Firm IFI of Moscow.

The extension of the project to include the detection of liquid explosives based on pulsed fast-neutron analysis (PFNA) is explored.

2 Background and Rationale

The detection of explosives is a serious challenge to all proposed technologies regardless of their physical basis. This is primarily because the elemental composition and atomic properties of explosives are similar to other commonly occurring materials. This results in high false alarm rates and/or missed detections in those technologies that rely on atomic properties such as electron density derived from x-ray tomographic inspections. Similarly, high-resolution x-ray radiography is also compromised when the shape of the explosive device is atypical, either in thin sheets or cloaked to mimic other innocuous shapes.

The x-ray systems now deployed at airports and other sensitive portals, however, do not detect nitrogen or any other explosive-specific property. X-rays lack the energy to probe beyond the atomic electrons and must rely on a determination of electron densities and characteristic shapes, neither of which is specific to explosives, resulting in high false positive rates. Despite shortcomings, these systems are attractive due to their cost, size, and operational simplicity and are deployed on a massive scale for lack of anything better. In the meantime, advanced systems with greater specific sensitivity for explosives and reduced sensitivity to backgrounds still need to be pursued to address the next generation of threats. These methods, of necessity, include nuclear-based active interrogation methods based on probing gamma rays and neutrons.

There are, fortunately, several explosives-specific indicators that can be exploited for more reliable detection. These indicators are manifested at the nuclear level. One such indicator in most explosives is the relatively high percentage (by weight) of nitrogen. Other indicators include the ratios of nitrogen to carbon and oxygen. Technologies that can specifically and sensitively detect and quantify nitrogen, carbon, and oxygen are, therefore, expected to produce better results than methods that rely on atomic properties alone.

Methods based on determining nuclear properties, however, are more complicated and although feasibility has been demonstrated in the laboratory for several nuclear-based methods, they inevitably depend on the development of other enabling technologies, such as charged-particle accelerators, to achieve field-operational status. Unfortunately, the sponsors of research and development of explosive detection technologies in the US have been unable to initiate and sustain a comprehensive, long-term approach that includes the development of these enabling technologies. As a result, nuclear-based methods with demonstrated feasibility remain laboratory instruments.

3 Nuclear-Based Active Interrogation Techniques

Several active interrogation techniques based on neutron probes are capable of penetrating dense overlying structures to identify explosives by either stimulating high-energy gamma-ray emissions in the elemental components of explosives or by being resonantly absorbed in them. Techniques such as thermal neutron activation (TNA), PFNA, and neutron resonance radiography (NRR) exploit the bulk nuclear properties of explosives that are sensitive to neutron radiation. The feasibility of these methods was demonstrated in the 1980s, and they have been extensively developed. However, both an irrational fear of neutron irradiation in the general public, and the size, cost, and complexity of these systems have prevented their acceptance and deployment.

Gamma-ray methods have also demonstrated feasibility to detect explosives. Gamma ray resonance absorption (GRA) utilizes a prominent and narrow (128 eV) gamma-ray resonance in nitrogen,

$$N^{14}\ (\gamma,p)\ C^{13}$$

at precisely 9.17 MeV. The method originated with Israeli scientists from Soreq Nuclear Research Center in the mid 1980s, and several variants of the method were successfully demonstrated in the early 1990s.

The method depends critically on the production of a very narrow spectrum of gamma rays with a width of several hundred electronvolts, centered at 9.17 MeV. One way to produce such a spectrum is to use the inverse resonant reaction,

$$C^{13}\ (p,\gamma)\ N^{14},$$

in which protons at precisely 1.75 MeV are captured in carbon-13 to form the 9.17-MeV excited state of nitrogen-14, which subsequently decays in flight, ejecting a 9.17-MeV gamma ray. Depending on the angle of emission with respect to the recoil momentum of the nitrogen-14, the gamma ray will be Doppler-shifted in energy. At 80.6 degrees with respect to the recoiling nitrogen-14, the gamma-ray energy is Doppler-shifted by the precise amount of energy to compensate for the recoil energies lost during emission and at its subsequent resonant absorption in nitrogen.

In spite of the low yield of gamma rays from C^{13} (p,γ) N^{14} (approximately 10^{-8} gamma rays/proton), proof-of-principle experiments were carried out with low-current proton beams from electrostatic accelerators. These experiments demonstrated clearly the sensitivity of the method for detecting and imaging explosives.

The electrostatic accelerators used for proof-of-principle experiments provide the proton beam quality required for resonance absorption. However, the available currents and the resulting gamma-ray fluxes are woefully inadequate for use in airports and other sensitive portals. On the other hand, accelerators based on radio-frequency methods capable of higher currents could not meet the beam quality requirements to exploit the sensitivity of the method.

Accelerator technology in the 1980s was not sufficiently advanced to produce high-current, high-quality charged-particle beams for a practical implementation based on the method. Interest in the method continued into 1990s both in the US and abroad but with a reduced level of support.

4 Advanced Accelerator Technologies for Explosives Detection by Nuclear Resonance Absorption

4.1 Development of a Compact Cyclotron

Investigations [2] of the beam dynamics in an earlier version cyclotron with an external H^- source had the following characteristics:

Maximum current (~1.8 MeV)	2.2–2.5 mA
Transverse emittances	150–300π mm·mrad
Energy spread, ΔE/E	±8%

Since these beam parameters do not meet the requirements for nuclear resonance absorption, a proposed solution suggested a cyclotron with a reduced current of ~200 μA and the transverse emittances and the energy spread reduced by an order of magnitude. This beam could then be injected into a small storage ring in which the desired final beam parameters could be achieved.

To simplify the design and reduce costs, an internal ion source was examined. It was expected that the source would be able to deliver 6 mA of H^- beam into the continuous region of the cyclotron. Due to the 30-fold decrease of the average captured beam current for acceleration (from 6 mA to 200 μA) achieved with a diaphragm on the first turn, it became possible to reduce the transverse emittances and the energy spread. Calculations were performed taking into account the 3D distributions of the electric fields of beam space charge as well as the effects of the accelerating system.

The configuration space diagram of the ion beam in the cyclotron is shown in Fig. 1, where extensive scraping of the H^- beam is required to achieve a high-quality proton beam at the expense of a much reduced current. In Fig. 2, calculations of the bunched phase of the beam during each turn in the cyclotron are shown.

The axial motion of ions is shown in Fig. 3. The amplitude of the axial oscillations does not exceed 5 mm during the entire acceleration. Positions of ions on the phase planes and on the plane radius-energy are shown in Figs. 4 and 5. The final rms values ($\pm 2\sigma$) of the beam transverse emittances are $\varepsilon_r = 50\pi$ mm·mrad, $\varepsilon_z = 9\pi$ mm·mrad. The ions are distributed inside within ±10 mrad and ±5 mrad in the horizontal and vertical planes, respectively. The average energy of the beam is 1.82 MeV, and the energy spread is ±2.5%.

Thus, the internal ion source makes it possible to decrease the radial and axial emittances and energy spread by factors of 5, 15, and 3, respectively, compared with a cyclotron with an external ion source and with a current of 2 mA.

4.2 A New Concept for a Low-Energy Proton Storage Ring of High Luminosity with Electron Cooling and an Internal Target

4.2.1 Space Charge of Proton Beam

The most evident limitation on the accumulated proton current at 1.75 MeV is the space charge. The intensity limitation for protons in the coasting beam is

$$N = \varepsilon_x \Delta Q_x \frac{4\pi \beta^2 \gamma^3}{r_p}, \tag{1}$$

where ΔQ_x is the maximal allowed betatron tune-shift and $\varepsilon_x = \theta^2_x\, \beta_x = \sigma^2_x / \beta_x$ is the rms beam emittance. It is possible to estimate the maximal value of $\Delta Q_{max} = 0.25$ for the beam rapidly lost on a target. If proton beam energy is $E = 1.75$ MeV, the total orbit length is $P = 870$ cm, the average orbit radius is 140 cm, the revolution frequency is $f_0 = 2.1$ MHz, and the maximal possible angular spread in proton beam is $\theta_{max} = 10^{-3}$ rad, then the size of proton beam is $a_p = \beta_{max} \times \theta_{max} = 0.14$ cm, the maximum number of particles and beam current are

$$J_{\max} = eN_{\max} f_0 = 3.5\text{mA},\ N_{\max} = 10^{10}, \tag{2}$$

and the beam emittance is 1.4 mm·mrad.

Two methods are proposed for solving the space charge problem. The first is to neutralize of the beam space charge with the help of accumulation of ions from the

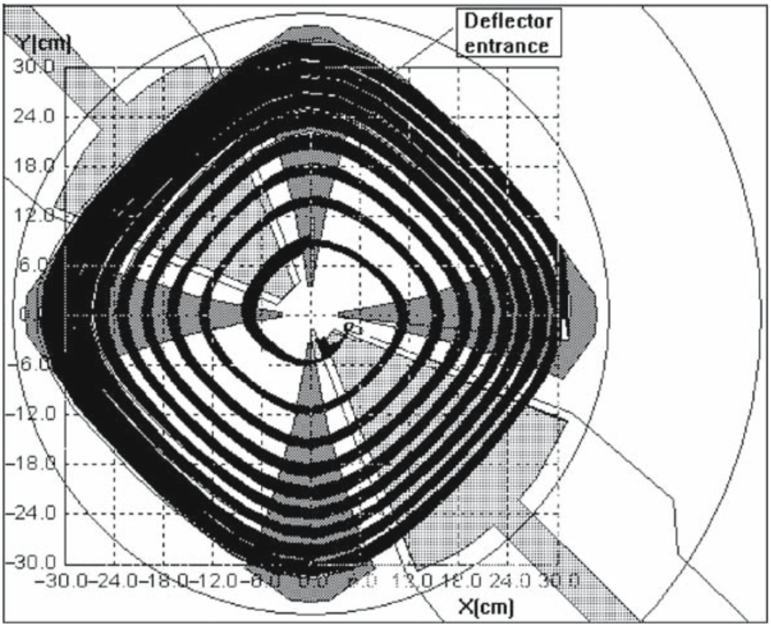

Fig. 1 Plan view of the cyclotron acceleration region

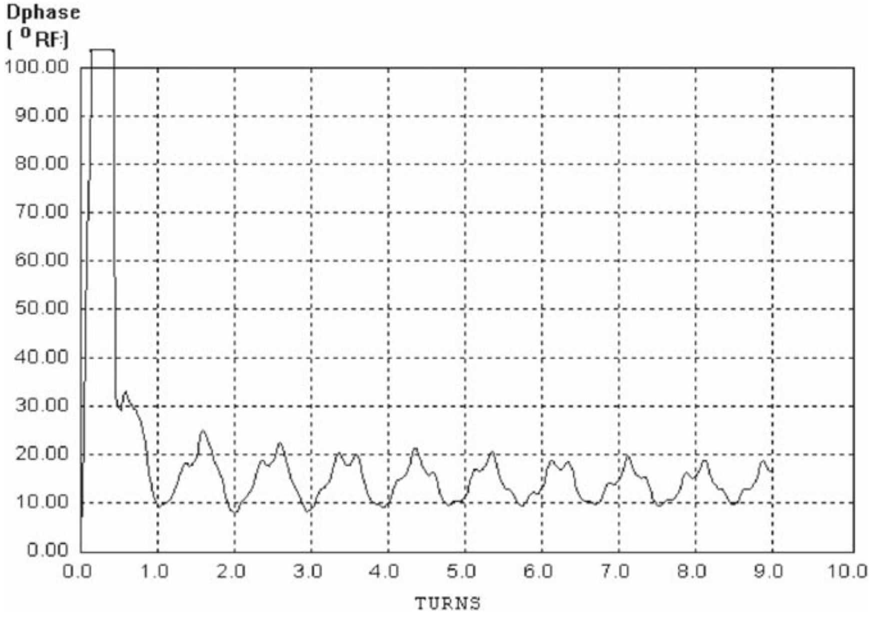

Fig. 2 Bunch phase width versus turn number of acceleration

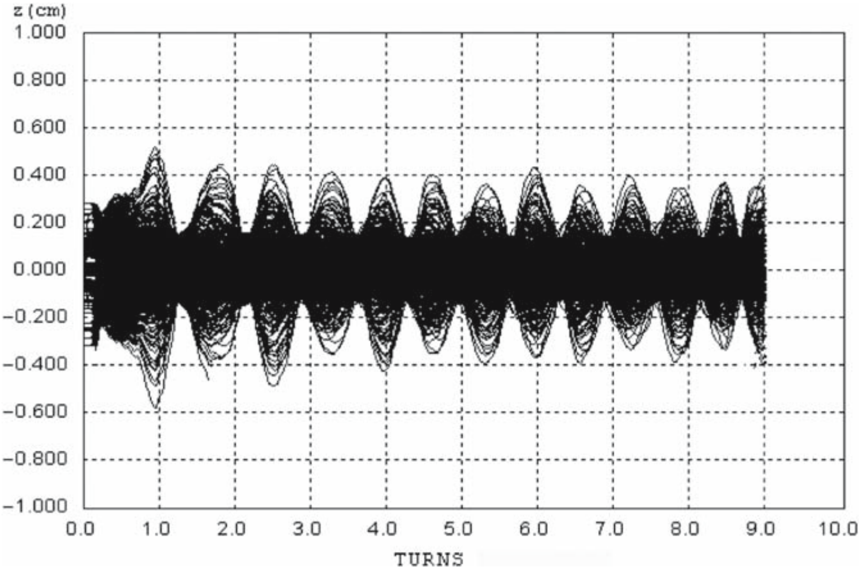

Fig. 3 Ion axial motion versus acceleration turn number

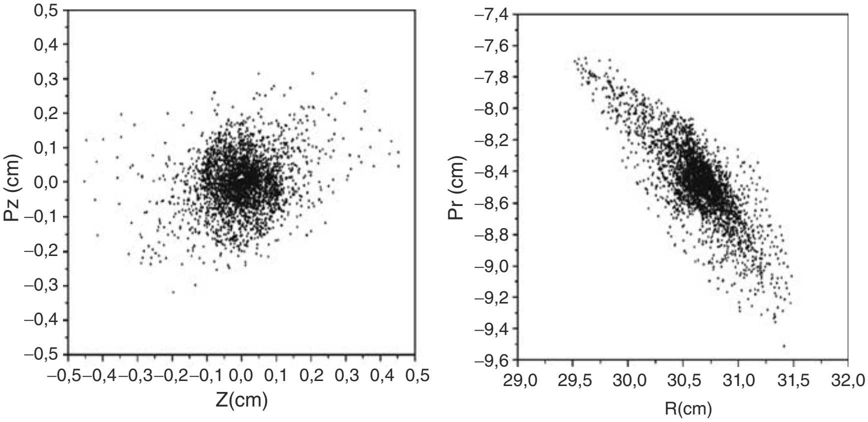

Fig. 4 Position of 3,000 ions on the transverse phase planes at the azimuth of the deflector entrance: radial plane (left) and axial plane (right)

residual gas and/or target ionization [2, 3]. The second is to use a special magnetic structure with strong longitudinal magnetic field resembling a tokamak or a stellarator [4]. In this case the protons are magnetized and move along the force lines of the longitudinal magnetic field. The motion of proton separates into two types formally. The first motion is a fast cyclotron rotation around the force line, and the second is a slow drift from one magnetic force line to another. Such dynamics of a proton beam can be described by means of two beta functions. One of them is responsible for strong focusing and has a small value

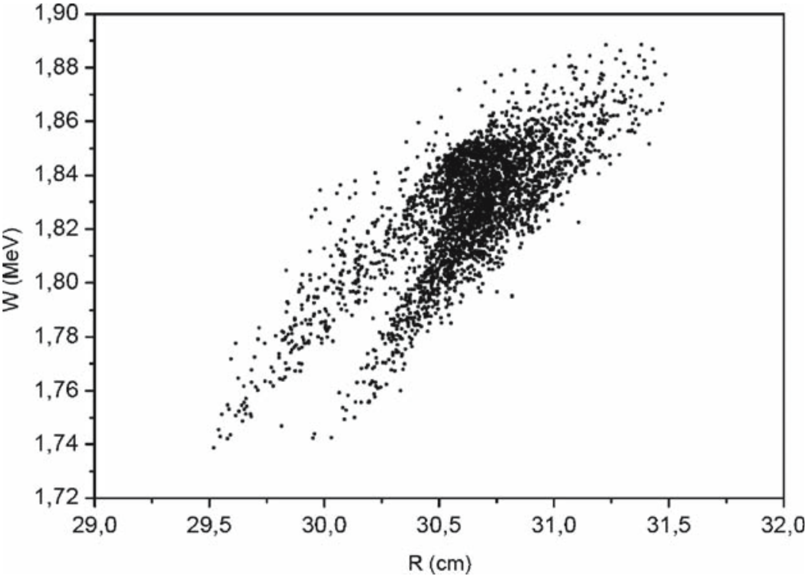

Fig. 5 Position of ions on the plane radius-energy at azimuth of the deflector entrance

$$\beta_s = \frac{\gamma \beta m_p c^2}{eB}. \tag{3}$$

This β-function is $\beta_s = 20\,\text{cm}$ at the magnetic field $B = 10\,\text{kG}$.

The other β-function has a bigger value $\beta_{max} = R_{av}/Q_{\perp}$, where $Q_{\perp}$ is a drift frequency. It describes the slow drift around the magnetic axis. To estimate, we take $Q_{\perp} = 0.2$ and a value for the maximal beta-function of about $\beta_{max} = 700\,\text{cm}$. The proton beam will look as if it consists of many small-size beams but with large size of the whole beam. In this case, for an angular spread ~1 mrad, the rotation radius around the force line at 10 kG magnetic field will be $a_p = \beta_s \cdot \theta_{max} = 0.2\,\text{mm}$ and the total proton beam size $\beta_{max} \cdot \theta_{max} = 0.7\,\text{cm}$. Actually, the proton beam at the same density will contain $(0.7/0.02)^2 \approx 1000$ beams.

4.2.2 Electron Cooling and Target Processes

The second problem is the degradation of the proton beam quality induced by the interaction with the gamma-ray production target. Electron cooling is required to solve this problem [5, 6]. Figure 6 shows the result of a Monte Carlo simulation of the combined processes in the target and electron cooler. Figure 7 shows the interaction of the proton beam with the target without cooling. It is shown that cooling enables a practically “quasi-stationary” situation without essential degradation of the beam quality. The absence of cooling, on the other hand, leads to the fast degradation of the proton beam at small target density.

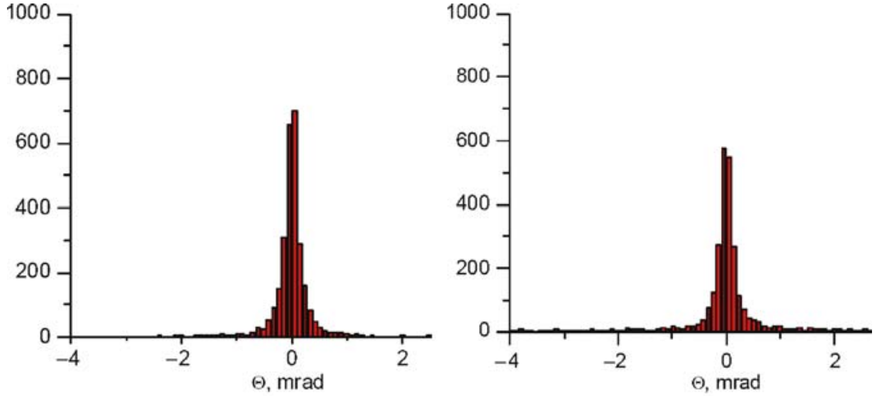

Fig. 6 Angular spread of the proton beam under the cooling and target processes. Target thickness is $4.0 \cdot 10^{15}$ cm^{-2}. The initial proton beam is cooled down. The left picture corresponds to time $\Delta t = 147\,\mu$s, and the right picture corresponds to $\Delta t = 750\,\mu$s. The electron current is 1.5 A, and the radius of the electron beam is 0.4 cm. Scattering is described by the Rutherford model

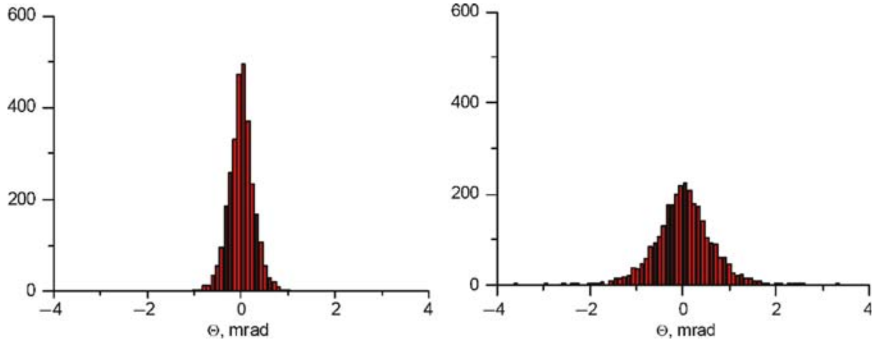

Fig. 7 Angular spread of the proton beam due to the target process only. Target thickness is $1.0 \cdot 10^{15}$ cm^{-2}. The initial proton beam is cooled down. The left picture corresponds to time $\Delta t = 73\,\mu$s, and the right picture corresponds to $\Delta t = 370\,\mu$s. Scattering is described by the Rutherford model

If the electron cooling effectively suppresses the target effect then the luminosity of such a device can be determined

$$L = \frac{I_p}{e} n_a = 1.6 . 10^{34}\,\text{cm}^{-2}\text{s}^{-1}$$

at the target density $n_a = 5 \cdot 10^{15}$ cm^{-2} and the proton current $I_p = 0.5$A. The corresponding gamma-ray flux is about $5.5 \cdot 10^{8}$ s^{-1} ($\sigma_\gamma = 3.5 \cdot 10^{-26}$ cm^{-2}). Certainly, this optimistic estimation of the ultimate luminosity demands detailed R&D programs and the investigation of prototypes of many elements.

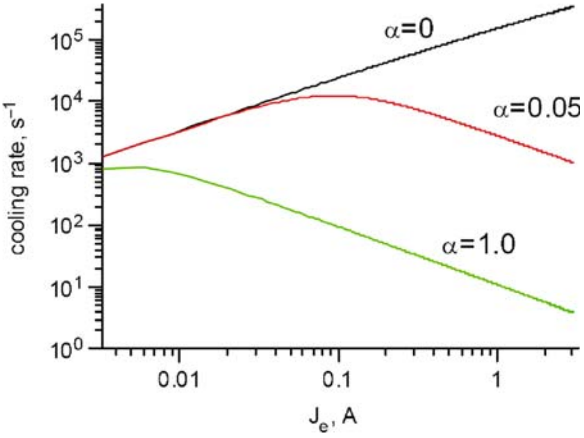

Fig. 8 Cooling force versus electron current at different values of the neutralization factor (α = 1.0 means no neutralization)

The space charge effect is very strong for the electron cooling process as well. The current requirement is large, and the electron beam energy is low. The potential sagging is

$$\Delta\varphi = \frac{30.I}{\beta} = 100\,\mathrm{V}$$

at the electron current 0.2 A that is unfit for operation. Consequently, neutralization is required to eliminate space charge effects in the electron beam. The secondary ions not only decrease the effect of space charge field on the electron motion but also allow electron beams with higher intensity. Figure 8 shows the comparison of the cooling rate at the different levels of the neutralization parameter. If the non-neutralized electric field is even 0.2% from maximum value then there are significant problems in obtaining the maximal parameters.

One of the limitations to obtaining a stable compensated state is the appearance and progress of different beam-driven instabilities. The rough criterion of compensated state stability is a small value of oscillation phase advance of the electron drift motion during time of flight through the cooling region $\omega_d \frac{1cool}{\beta c} < 1$, which gives the limit of maximum possible electron beam density:

$$n_e < \frac{B\beta}{2\pi e\, l_{cool}} = \frac{2.10^4 \cdot 6.5\times10^{-2}}{2\pi \cdot 4.8\times10^{-10} \cdot 100} \approx 4\times10^9\,\mathrm{cm}^{-3} \tag{4}$$

Although this condition is not absolute, its realization leads to a short length of the electron beam and a large value of the magnetic field. The coherent processes determine the compensation stability. The experience at BINP of operating with compensated proton and electron beams [7] has shown that stable states are possible, but this problem can be finally solved only in experiments.

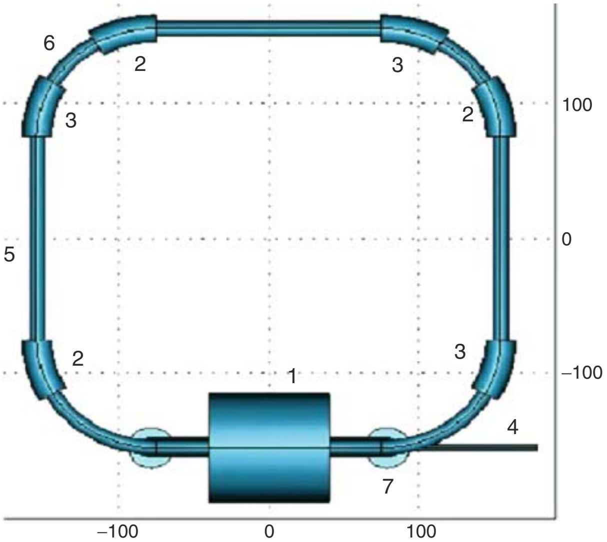

Fig. 9 Sketch of storage ring vacuum chamber: (1) target for the generation of gamma rays (at the same time it is the target for charge exchange injection), (2) the electron gun section, (3) the collector section, (4) injection section, (5) cooling section, (6) bending section, and (7) pumping section

4.2.3 Storage Ring Structure

Figure 9 shows the structure of the storage ring with the longitudinal magnetic field. The negative ions H^- are produced by the cyclotron and stripped to neutral atoms of H^0. These neutral atoms are injected to the strong magnetic field along the injection line (4). The target (1) for charge exchange from H^0 to protons H^+ is the same that generates the resonance x-rays. The protons move clockwise. The electron beam is produced in the electron gun areas (2). The electrons and protons move together in the sections (5). So, there are three sections for the cooling process. The electron beam is absorbed to the collector in regions (3).

The motion in the dipole magnet is used for separation of the proton and electron beams. The example of such a separation scheme is given in Fig. 10 and Table 1. The transverse magnetic field in the dipole shifts the magnetic force line from the magnetic axis. The proton beam moves in the orbit plane since the transverse (dipole) field is compensated by centrifugal force $\gamma \beta m_p c^2 / R$, and electrons, being 1,836 times lighter, move upward along the force line. At the same time the electron beam for cooling in the next straight section can be brought in from below.

The electron cooler in the storage ring with internal target can be used to strongly suppress the degradation of the proton beam quality, giving hope that a flux of resonant x-rays of about $5.0 \cdot 10^8$ quanta per second can could be produced. This effective cooling process is possible with space charge neutralization of the proton beam by the electron beam if a stable state of such beam-plasma system can be established through

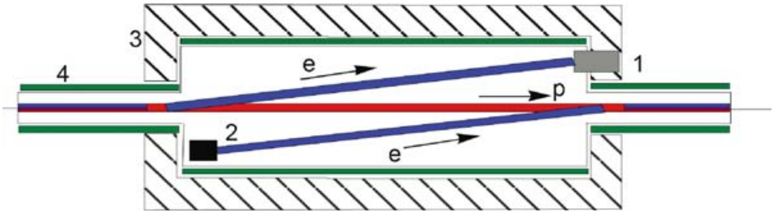

Fig. 10 Separation scheme of the electron and proton beams in the dipole magnet. The electron beam moves from the collector to the proton orbit. In the cooling section the electron and proton beam move together. Finally, in the next bending magnet the electron beam moves to the collector and is absorbed by it. The preliminary parameters of such a storage ring are shown in the Table 1

Table 1 Preliminary parameters of the proposed storage ring

Proton energy	1.75 MeV
Circumference (R = 80 cm, L = 150 cm)	11.0 m
Length of straight sections	150 cm
Average radius	1.75 m
Revolution frequency	1.66 MHz
Total length of cooler sections	About 420 cm
Electron current	About 1.0 A
Radius of bending magnet	80 cm
Transverse field in bending magnet (R = 80 cm)	2.4 kG
Field index	0.5
Longitudinal magnetic field	25 kG
Electron beam radius	The same as proton beam radius
Aperture of vacuum chamber Ø	5–10 cm
Proton current	100 mA–1 A
Injection current (DC)	100–300 μA
Injected particle	H^0
Injection method	Stripping on the internal target
Initial angle spread at injection	3 mrad
Initial energy spread of proton beam	±30 Kv
Target	CH_4 (methane)
Target density, cm^{-2}	Up to 10^{16}
Average energy losses in target	Up to 40 V per turn
Luminosity	Up to 10^{35} cm^2 s^{-1}

careful designs. The requirement on the large proton current and small angular spread can lead to the inadmissible value of the betatron tune shift. However, judicious choice of ring structure with the longitudinal magnetic field may help to solve this problem.

4.3 The BINP Vacuum-Insulated Tandem Accelerator (VITA) for the Detection of Explosives

The "standard" electrostatic tandem accelerator has limited beam current [8]. Therefore, BINP scientists decided to design a new type of high-current proton tandem accelerator: the Vacuum Insulation Tandem Accelerator (VITA). Such a tandem accelerator is capable of producing the very stable 10 mA, ~1.75 MeV proton beam required for GRA. VITA has advantages compared with other electrostatic accelerators because the ion source is at ground potential and the required operating voltage is only half of that required in other schemes.

VITA essentially eliminates the accelerating columns inside the tandem accelerator. In a conventional tandem, two accelerating columns based on ceramic tubes are connected by the high-voltage parts with the stripping target located in between. This scheme for high beam current (a few tens of milliamperes) has major disadvantages:

- The necessity of pumping the gas from the stripper through accelerating columns.
- The inevitable current emission of secondary electrons and ions from the high current beam passage region to the inner surfaces of high-voltage electrodes and ceramic insulators.

In the VITA scheme, high voltage is applied through an electrical feedthrough insulator (which can be arbitrarily remote from the accelerator beam passage region) to the cylindrical potential electrode with the charge-exchange target, located within the vacuum tank. The pumping of gas from the stripper is realized not through the accelerating columns but through regions without solid insulation at high-voltage potential.

An additional advantage of VITA is a wider range of choices of high-voltage gradient for the elements of the feedthrough insulator in comparison with the choice of high-voltage gradient for accelerating columns. The high-current beam tandem accelerator with vacuum insulation provides higher reliability compared to tandem based on accelerating columns with ceramic insulation.

Figure 11 shows the full-scale design of the GRA complex based on the vacuum insulated tandem accelerator. A H^- beam is formed by an ion source (3) and injected into the accelerator through the low-energy beam tract (4). After charge-exchange of the H^- ions in the stripper tube in the center of the high-voltage electrode (5), a proton beam forms at the exit. The beam is accelerated to twice the voltage of the high-voltage electrode. Then the full-energy proton beam forms inside high-energy beam tract (7) and interacts with the gamma-ray production target (8).

The high-voltage electrode of the tandem is surrounded by a system of shields at different potential providing homogeneous distribution of the potential and preventing full voltage effect.

The most important component of the accelerator is the high-voltage feedthrough insulator (2), which connects the high-voltage power supply (1) inside a tank filled with SF_6 gas (0.8 MPa) with vacuum part of the accelerator (Fig. 12). The high-voltage feedthrough insulator can be placed far from the accelerated beam passage region. The high-voltage electrode is placed on the vacuum part end of the high-voltage feedthrough insulator on the metal flange, which is vacuum-tightened by the tightening pipe passing along its axis connected to another metal flange placed on the gas part end of insulator. This metal flange is connected to the high-voltage source. The gas part of the through-pass insulator (placed in SF_6) is made of a stack of ceramic rings separated by metal rings for the potential distribution. The vacuum part of the insulator is made of glass rings, which are also separated by metal rings. Inside the insulator (under an SF_6 gas pressure 0.3 MPa) around the tightening pipe, thin-wall pipes of various lengths are

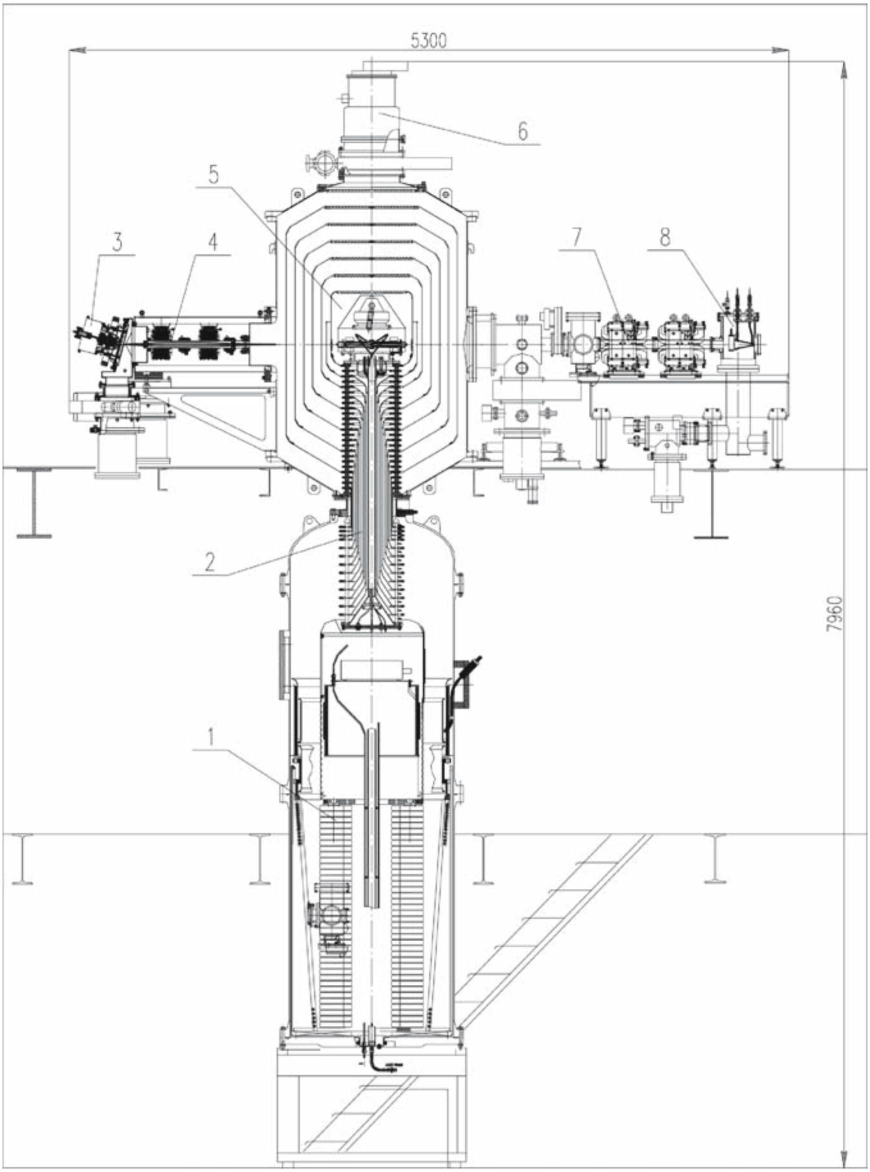

Fig. 11 VITA based GRA complex

concentrically located to connect the respective metal rings of different potential on the gas part of the insulator and its vacuum part. The voltage is applied to these rings from a resistive divider, which distributes potential homogeneously along the insulator.

The electrostatic field intensity of the separate insulators of the vacuum part of the high-voltage feedthrough insulator is 15 kV/cm and 18 kV/cm for the insulators in the gas part. Electrostatic intensity at the six accelerating gaps is 33 kV/cm. The

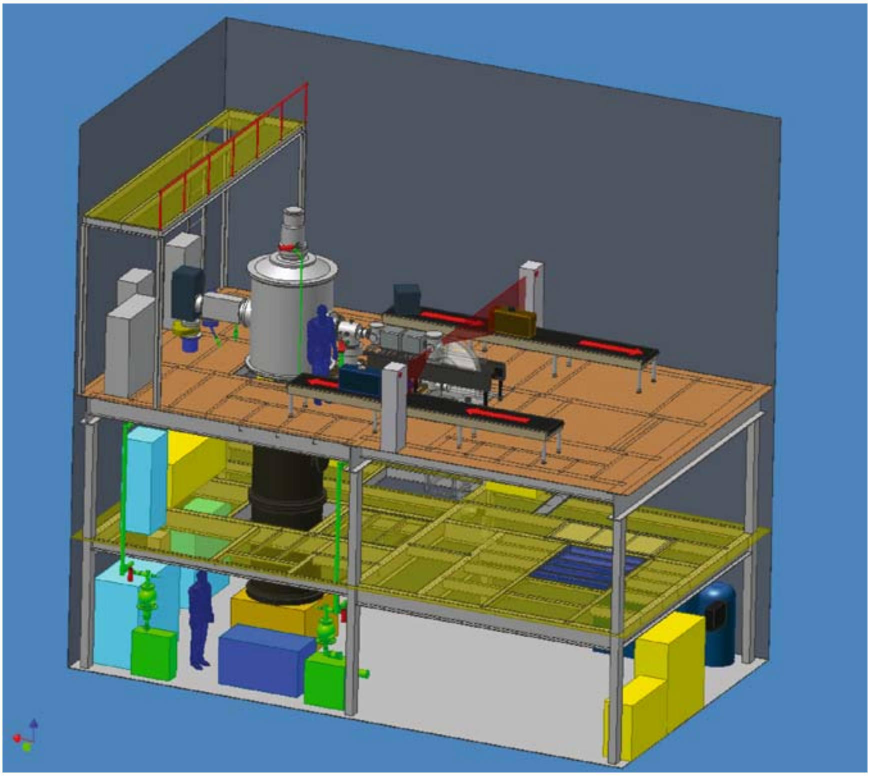

Fig. 12 GRA explosives detection system based on VITA accelerator

stored energy in each high-voltage vacuum gap is limited to 30 J. Vacuum pumping is carried out through heads of potential shields by a cryogenic pump (6).

The walls of vacuum tank, high-voltage electrode, and the shields have coaxial round holes for beam passage. Since the thin-wall shields are placed along the equipotential surface, the electrostatic field contributes slightly to focusing.

A gas target was chosen for stripping the negative ion beam. The stripping target is a pipe 10 mm in diameter and ~400 mm in length. In the center of the pipe, gas is supplied at a rate to provide stripper density of 3×10^{16} mol·cm^{-2} for 99% stripping efficiency. Several target designs were considered:

(i) Gas target with outer pumping through the heads of potential shields.
(ii) Gas target with recycling turbo-molecular pumping inside the high-voltage electrode.

The high-voltage conditioning procedure, as well as high-voltage strength of vacuum gap, depends on the energy that was stored in corresponding capacities before breakdown. These parameters were tested on a prototype vacuum insulation tandem accelerator. Table 2 shows status as of September 2005. Experiments with the proton beam are planned for the second quarter of 2006.

Table 2 Status of GRA complex

1	The sectionalized rectifier from industrial ELV-type electron accelerator as a powerful source of high voltage	Manufactured and tested up to 1.4 MV
2	The feedthrough insulator	The shell of feedthrough insulator is manufactured and tested under inner and outer pressure. The inner electrodes are planned to be manufactured this quarter
3	The negative hydrogen ion source	Pilot version of the source produces 10 mA, 25 kV beam. Measured normalized emittance is about 0.2π mm·mrad
4	Low energy beam line	Low-energy line components were fabricated
5	Gas stripping target, placed in the center of high-voltage electrode, surrounded by potential shields	The vacuum tank and high-voltage electrode of tandem are manufactured. The target and potential shields fabrication is planned for 2005
6	Cryogenic pump	Exists
7	High energy beam line	Manufacture of the high energy tract components is continued
8	Gamma generating target	The work on the design of gamma-targets is continued

The development of the accelerator has been partially supported by ISTC (collaboration of BINP, IPPE, MRRC, RFNC ITP – grants #1484, #2569, Europe) and also by US DOE through IPP grant (BINP collaboration with Brookhaven National Laboratory and Brookhaven Technology Group, Inc.)

5 Detection of Liquid Explosives

Detecting liquid explosives is more complicated than detecting bulk explosives. Many liquid-explosive compounds have little or no nitrogen, as is the case of peroxides. Consequently, a more comprehensive method is required to determine the densities of all the major components of explosives, namely, carbon, nitrogen, oxygen and other elements such aluminum, iron, and chlorine.

PFNA as implemented by Rapiscan Neutronics and Advanced Technologies (RapNAT), formerly Ancore Corp, of Sunnyvale, CA, USA, has been demonstrated to meet the requirements for liquid explosives detection.

PFNA utilizes a pulsed deuteron beam to produce an 8-MeV neutron beam from deuteron-deuterium interaction at 6 MeV. In the Rapiscan implementation of the technique, the neutron interrogation beam is collimated to a $5 \times 5\,\mathrm{cm}^2$ rectangular spot that is scanned across the object, producing gamma rays from neutron inelastic scattering in C, N, and O by the inelastic interaction (n, n' γ). These characteristic gamma rays are identified and quantified by sodium iodide detectors using gamma-ray spectroscopy. To complete the three-dimensional image of the elemental composition, neutron time-of-flight is measured for the detected gamma rays from each $5 \times 5 \times 8\,\mathrm{cm}^3$ voxel.

The characteristic inelastic gamma-ray spectra derived from this method are depicted in Fig. 13 for carbon, nitrogen, and oxygen, individually, and in composite

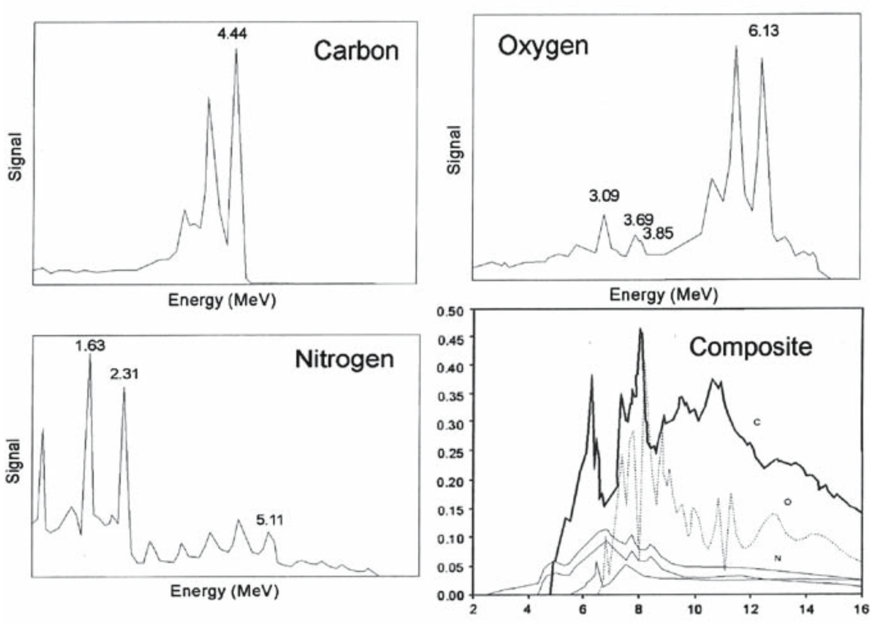

Fig. 13 Gamma-ray spectra produced by PFNA

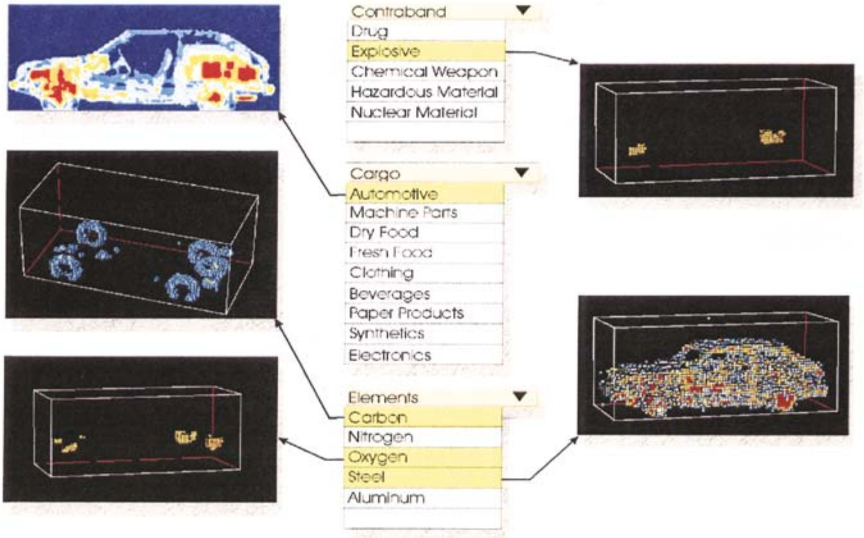

Fig. 14 PFNA images of an automobile containing stimulant explosives

form as would be the case for an explosive. Similar spectra are generated for each voxel produced by the scanned beam in combination with the associated neutron time-of-flight to produce the three-dimensional image of an automobile containing simulated explosives in the engine compartment and in the rear compartment (Fig. 14).

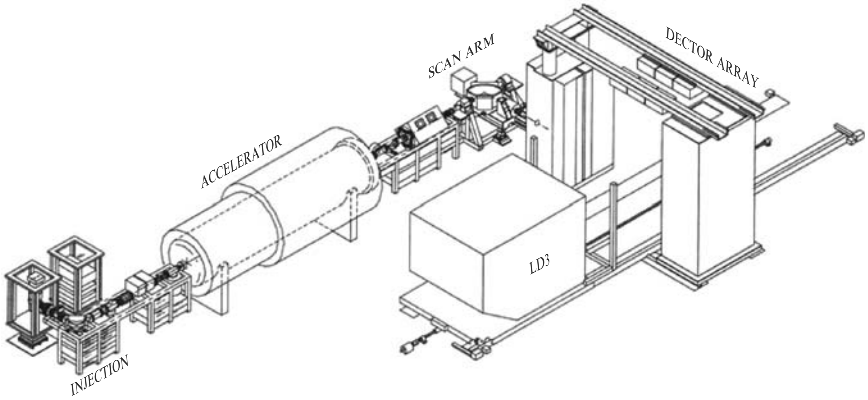

Fig. 15 Rapiscan PFNA cargo inspection system based on a Van de Graaff accelerator

The PFNA neutron interrogation beam is produced by 140-µA, 6-MeV deuterons accelerated by a pulsed Van de Graaff accelerator with 2-ns wide pulses at a 3.5 MHz repetition rate (Fig. 15). Neutrons produced in a high-pressure, cooled deuterium target are kinetically-focused in the forward direction and collimated to form a rectangular spot which is scanned vertically.

The reaction ^{2}H (d,n) ^{3}He produces a quasi-monoenergetic 8.5-MeV neutron-interrogation neutron with the same time structure and scanning feature as the deuteron beam.

As a result, the smallest voxel that can be interrogated is ~$5 \times 5 \times 8\,cm^3$.

The Van de Graaff-based PFNA implementation is consequently limited by several factors. The low deuteron current results in lengthy interrogations when the interrogated volume is large. Similarly, the 2-ns wide beam pulse limits longitudinal resolution to ~8 cm. In addition, the cost of the accelerator system is large ($1–2 million) as is the physical footprint.

However, many of the PFNA accelerator requirements are similar to those that we have been pursuing for the GRA method, and we believe that improvements to the present PFNA accelerator source are possible. In particular, the JINR four-sector cyclotron design proposed for GRA could be modified to provide increased average deuteron beam current, up to 2 mA, which would result in an order of magnitude reduction in interrogation times. Improved longitudinal resolution would also be possible with the cyclotron, which is capable of reducing the pulse width (<2 ns) with reduced wings.

Initial cost estimates for the cyclotron-based system suggest a reduced cost over the Van de Graaff with a reduced footprint that could be achieved by increasing the cyclotron magnetic field. Turnkey operation would also be an available feature with the cyclotron.

6 Conclusions

The WSSX project continues to support the development of high-current charged-particle accelerators for explosives detection in Russia. Both a cyclotron/storage ring system and a vacuum-insulated tandem accelerator are being explored. The results to date have been reported here.

The results achieved thus far include successful operation of the tandem accelerator and a determination of resonance absorption in nitrogen at 1 mA proton current. High-power target designs and upgrades to the low-energy beam transport channel are underway to extend the useful operational current to 5 mA. Future experiments will include a measurement of the effective resonant absorption in simulated explosives.

The design parameters of the cyclotron/storage ring concept have been determined, but budget constraints have delayed the project from proceeding into the fabrication stage. The characteristics of the extracted cyclotron beam required for efficient injection into a storage ring for continuous operation have been determined. The requirement for large proton current with small emittance leads to an unacceptable betatron tune shift that may require a storage ring with longitudinal magnetic field and space-charge-neutralization to mitigate the problem. Emittance growth of the proton beams due to beam-target interactions can be effectively compensated with electron cooling. In this way the generation of resonant gamma-ray intensities in excess of 10^8/s will be possible.

To extend the usefulness of the accelerators under development, application to the detection of liquid explosives and flammables using PFNA based on the four-sector cyclotron design have been investigated. Based on preliminary estimates, it would be feasible to improve the voxel resolution of the present PFNA system while reducing interrogation times by an order of magnitude in a compact system amenable to turnkey operation.

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A Device for Detecting Concealed Explosives

V.A. Teryokhin and Yu.I. Chernukhin

Abstract The paper discusses a device which increases sensitivity to and improves detection reliability of concealed explosives, including cases of humanitarian de-mining. The device consists of an emitter of thermal neutrons and high-effective large-aperture heterogeneous scintillation detector (HSD) of hard gamma-rays. The detector is a set of gamma-radiation converting layers and adjacent scintillating layers, which detect electrons and positrons produced in the converting layers. Layer thicknesses are selected so that to allow charged particles generated from gamma-quanta in the converting layers to be detected in two or more scintillating layers. Each scintillating layer and all of them as a whole are connected to the data processing system.

Keywords Device, neutrons, layers

Abbreviations HE – high explosive; HSD – heterogeneous scintillation detector; EXPLODET – EXPLOsive DETection; PMT – photomultiplier

Neutron-radiation method, i.e. detection of concealed HE through detecting nitrogen contained in almost all HE types by bombarding HE with thermal neutrons and recording secondary gamma-radiation with the energy of 10.8 MeV, is well-known and can be used in mobile complexes for the purposes of humanitarian de-mining as well as in the stationary complexes to inspect carry-on and large-size luggage [1–4].

In 1998 the Project EXPLODET (EXPLOsive DETection) [2] started in Italy under the IAEA Coordination Program “Nuclear Technology Application for Antipersonnel Mine Detection”. Major part of EXPLODET efforts focused on the HE detection with the neutron-radiation method using thermal neutrons and recording gamma-quanta with the energy of 10.8 MeV born in the reactions of thermal neutron capture by ^{14}N nuclei. The relevant papers were published in EXPLODET REPORT 1998–2002.

Russian Federal Nuclear Center - All-Russia Research Institute of Technical Physics, Snezhinsk, Chelyabinsk area, 456770, Russia

H. Schubert and A. Kuznetsov (eds.), *Detection of Liquid Explosives and Flammable Agents in Connection with Terrorism*, 117–121.

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Figure 1 shows an experimental set-up modeling search for concealed HE in the field conditions [3].

A thermal neutron emitter (1) is a polyethylene cylinder 19 cm in radius and 26.5 cm high, which houses a lead sphere (2) with the radii of 1.5 ÷ 4.5 cm having inside a ^{252}Cf source (3) of neutrons with the intensity of ~10^7 n/s. Distance between the ^{252}Cf source and bottom end-face of the emitter is $\boldsymbol{d}_3 = 6.5$ cm. The emitter is located at the distance of $\boldsymbol{d}_1 = 20$ cm from the ground (5).

Specimen (4) modeling chemical composition of conventional HE is made of 800 g of melamine ($C_3H_6N_6$) and is buried at the depth of d.

Scintillation gamma-spectrometer (6) with the cylindrical NaI(Tl) crystal 7.5 × 7.5 cm in size is used as a detecting unit. The scintillator is located at the distance of $\boldsymbol{d}_2 = 15$ cm above the ground surface (5) and is shielded with 2 cm-thick lead (7) from the emitted gamma-quanta.

Typical gamma radiation spectra obtained in the experiments are shown in Fig. 2.

It is obvious from the above data that valid signal is relatively small if compared to the background, therefore sensitivity and reliability of detection of the concealed HE are limited.

All devices for HE detection with the neutron-radiation method use a scintillation unit for gamma-radiation detection based on large inorganic scintillators of NaI(Tl), CsI(Tl), and BGO types. To improve efficiency of gamma-radiation recording, several scintillators can be used. A basic drawback of such devices for HE detection is caused by characteristics of the detecting unit, namely, high sensitivity to soft gamma radiation, significant contribution to which is made by scintillator activation with thermal neutrons. This leads to high traffic in the recording channel and superposition of pulses from gamma-rays resulting from neutron interactions with detector material and environment. This results in a low signal-to-background ratio (~10^{-1}) in the 9–11 MeV energy range of gamma-quanta. Therefore, according

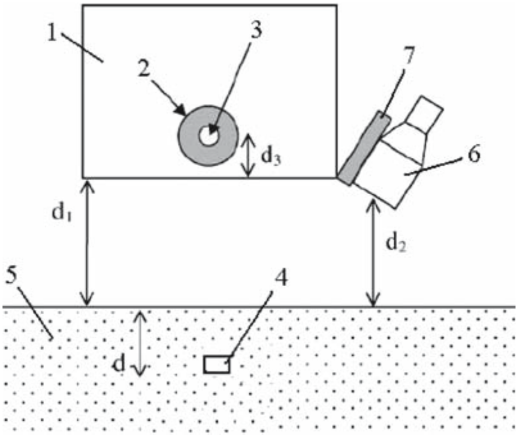

Fig. 1 Experimental set-up

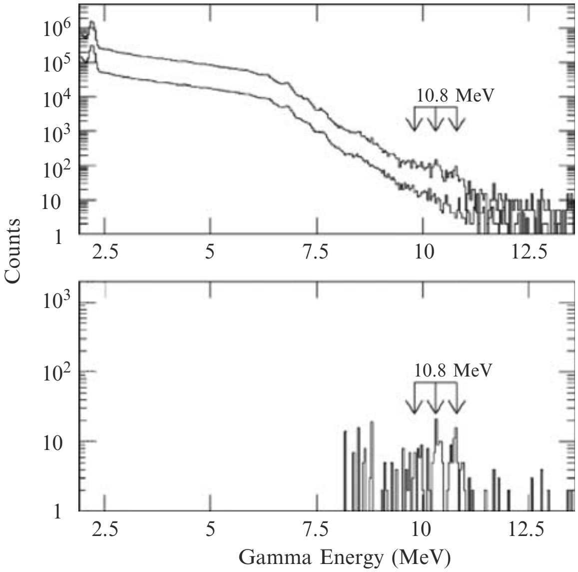

Fig. 2 Typical gamma radiation spectra. The upper plot shows the measured melamine specimen spectrum (×5) and background (×1). The lower plot illustrates spectrum after background deduction. Arrows indicate peaks corresponding to gamma-radiation with the energy of 10.8 MeV. Measurement time is 500 s

to the estimates [3], the considered method will give HE detection sensitivity of ~1 kg in several minutes for such application as humanitarian de-mining, and this would not allow detection of antipersonnel mines containing about 40 g of HE.

The objective is to develop a simple mobile device capable of detecting antipersonnel mines and employing a new high-efficient wide-aperture unit for hard gamma radiation detection based on a heterogeneous scintillation detector [5], which has low sensitivity to neutrons and low-energy gamma-quanta, increases strength of the neutron source and improves signal-to-background ratio in the gamma-quanta energy range of 9–11 MeV. Feasibility of such solution is confirmed by simulations and qualitative comparison of the following characteristics of the heterogeneous scintillation detector (HSD) and inorganic scintillators: energy dependence of efficiency of gamma radiation recording, speed of response, measuring channel throughput, and sensitivity to background including a neutron one.

The proposed device for HE detection incorporating a thermal neutron emitter, gamma-radiation detection unit and data processing unit employs a novel detection unit based on a high-efficient wide-aperture heterogeneous scintillation detector of hard gamma radiation. This detector is designed as a set of gamma-radiation converting layers and adjacent scintillation layers, which detect electrons and positrons emerging in the converting layers. Thicknesses of layers are determined so that to detect the charged particles born from gamma-quanta in the converting layers in two or more scintillation layers. Each scintillation layer and all of them as a whole are connected to the data processing unit.

This design eliminates the effect of neutrons and gamma-quanta background on detector signals and improves the HE detection sensitivity due to the use of the high-intensive (≥10^8 n/s) neutron source and the wide-aperture (~1 m^2) heterogeneous detector.

Configuration of the thermal neutron emitter and its positioning with respect to the ground are identical to those shown in Fig. 1. The thin (1–3 cm) flat heterogeneous scintillation detector containing M alternating converting and scintillation layers is located between the emitter and the ground.

HE detection happens when the detector records gamma-quanta with the energy of E = 10.8 MeV, which result from irradiation of nitrogen contained in the buried HE specimen with the thermal neutrons. Thicknesses of the converting layers are selected so that the background gamma-quanta (with the energy of <2 MeV) give counts in one scintillation layer only, whereas "hard" gamma-quanta producing fast electrons and positrons give counts in several scintillation layers at a time. Signals from the $\boldsymbol{i}$-th scintillation layer of the detector are recorded by a PMT $\boldsymbol{N_i}$ ($\boldsymbol{i}$ = 1, 2, …M, M is a number of scintillation layers in the detector), and one more PMT N_D records all scintillation layers as a whole. Information on the energy spectrum of incident gamma radiation can be then drawn from the comparison between $\boldsymbol{N_D}$ and $\boldsymbol{N_\Sigma} = \sum_i \boldsymbol{N_i}$.

The data processing unit of the heterogeneous detector allows operation with the use of the high-intensive (≥10^8 n/s) neutron source with suppression of background and extraction of valid signal, which is hard gamma-radiation spectrum (~10.8 MeV).

For example, consider a flat HSD model consisting of ten converting layers of 0.3 mm thick lead and ten layers of scintillator 1.4 mm thick, total detector thickness being ~2 cm.

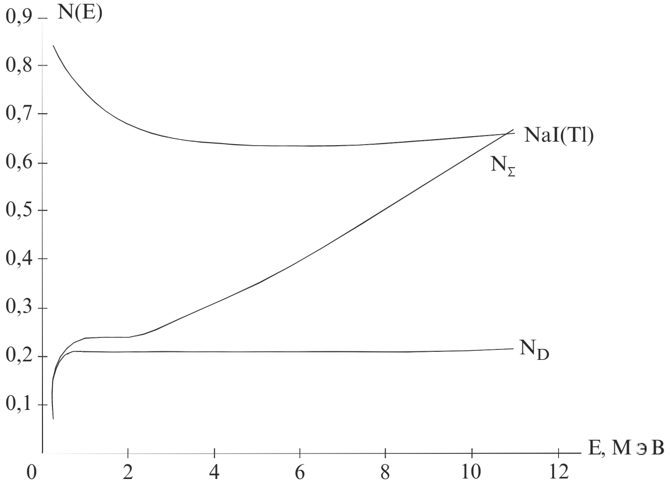

Fig. 3 Energy dependence of efficiency of HSD and NaI(Tl)-based detector

Figure 3 shows Monte Carlo calculations of detector counts per incident quantum from a plane-parallel source as a function of energy $\boldsymbol{N}(\boldsymbol{E})$ for NaI(Tl)-based detector 10 cm high and 15 cm in diameter as well as $\boldsymbol{N}_{\boldsymbol{D}}(\boldsymbol{E})$ and $\boldsymbol{N}_{\Sigma}(\boldsymbol{E})$ versus energy for the above HSD model.

It is obvious from Fig. 3 that in the HSD model under consideration, gamma-quanta with the energy of $\boldsymbol{E} > 10$ MэВ produce coinciding counts in three scintillation layers making it possible to discriminate background gamma radiation with the energy of $\boldsymbol{E} < 10$ MeV and extract valid signal with the scheme of triple coincidences (scheme of double coincidence ensures detection threshold of gamma-quanta with the energy of $\boldsymbol{E} \sim 6$–7 MeV). An expected efficiency of HSD detection of gamma-quanta with the energy of $\boldsymbol{E} = 10$ MeV is comparable to that for large-size NaI(Tl)-based detectors (Ø15 × 10 cm).

Unlike NaI(Tl)-based detector, HSD has a large aperture ($\sim 10^4$ cm^2), therefore sensitivity of the proposed unit for gamma-quantum detection can be improved by 10–20 times.

Further increase of the proposed unit sensitivity to hard gamma-quanta can be reached by increasing a number of heterogeneous layers in HSD.

Small HSD thickness (~2 cm) simplifies design of a mobile system for HE detection.

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Trace and Bulk Detection of Explosives by Ion Mobility Spectrometry and Neutron Analysis

D. Rondeshagen*, G. Arnold, S. Bockisch, K.-P. Francke, J. Leonhardt and A. Küster

Abstract In the paper we present the results on measurements of traces of explosives (like TNT, TATP, RDX, C4, PETN and SEMTEX) by means of an ion mobility spectrometer (IMS). Due to the excellent sensitivity of the IUT ion mobility spectrometer which is in the lower nano gram range the device is best suitable for the explosive inspection.

Keywords Explosives, ion-mobility spectrometry, IMS, neutron based trace detection, bulk detection, neutro-test, bottle scanner, shell-inspection, neutron backscattering

In a second part we report about the development of devices, based on neutron backscattering. These devices are used for inspection of shells or to decide, if the content of a bottle is possibly hazardous or not.

1 Trace Detection of Explosives by Ion Mobility Spectrometers (IMS)

IUT is an independent German developer and manufacturer of ion mobility spectrometers (IMS). Since 1992 IMS devices have been produced primarily for the detection of chemical warfare agents (CWA) and other toxic gases (TIC). These devices with a classic IMS design are used in many stationary and mobile systems in governmental facilities in Germany but also in industrial applications worldwide. One of the main references is the complex protection system against CWA and TIC in the German Parliament building (Reichstag) installed in 1997/98.

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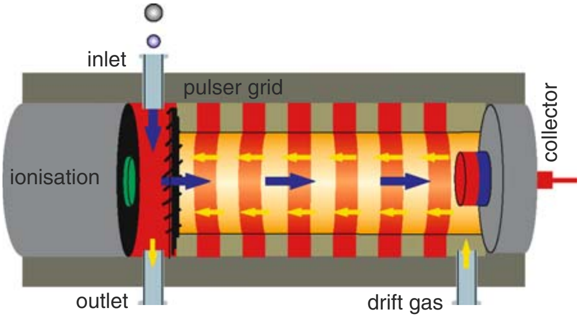

Fig. 1 Principle of the IMS cell

Fig. 2 IMS device for the detection of explosives

The IMS based on the different mobilities of ions of different size and composition in an electric field. The sensitivity is in the lower ppb-range with response times of some seconds. Air and other molecules are sucked into the IMS and are ionized. In the drift cell the different ions are separated from each other and transfer their charge to the collector. The IMS operates under ambient pressure. No vacuum system and no carrier gas are required. This reduces/avoids additional cost during operation (Fig. 1).

Recently a new generation of portable IMS devices was developed combining the high technical standard which was already reached in the past with a new inlet system and an IMS detector operating at higher temperature necessary for the detection of low volatile substances like explosives.

The new IMS tubes are characterized by a high resolution power and sensitivity due to the specific design of the reactant ion chamber containing an especially performed tritium radiation source, and their drift region. Besides of this the device is equipped with a removable sampling and thermo desorption unit. By means of this unit traces of explosives can be easily wiped off by a special wipe material from surfaces of suspicious objects, as shown in Fig. 2. After finishing the sampling process and connecting the unit to the heated inlet system of the IMS the sampled

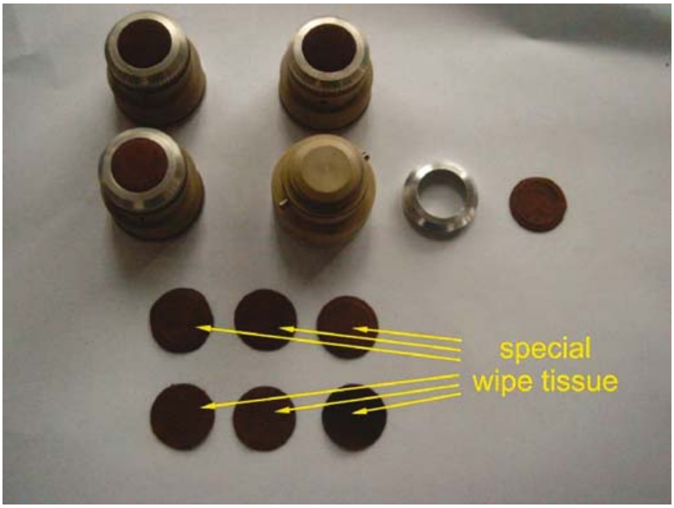

Fig. 3 Wipe test unit with and without Teflon substrate material

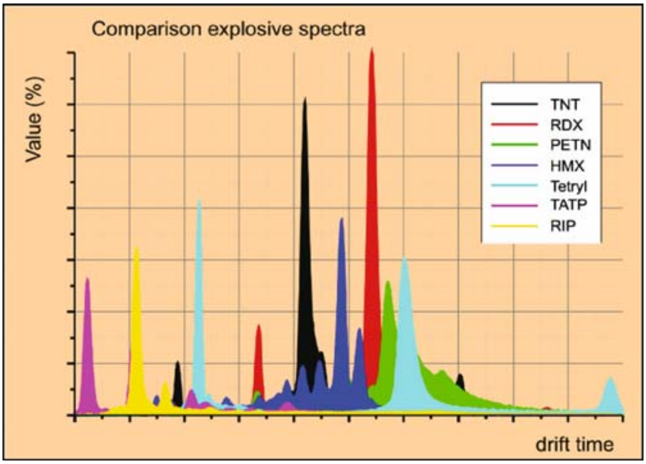

Fig. 4 Comparison of the IMS spectra of different explosives

explosive particles are vaporized and detected. The whole detection process including sampling, thermo desorption and following substance identification can be performed within few seconds.

The IMS device for explosive detection is shown in Fig. 2.

As swab/wipe material a special Teflon textile is used. Figure 3 shows the wipe test unit with and without substrate material.

The ion mobility spectra of different explosives were recorded. Based on these measurements the spectra identification library was created. Figure 4 shows spectra of the different explosives in comparison.

As examples the ion mobility spectra of TATP, TNT, RDX (another name is Hexogen), C4 (mixture of Hexogen and plastifying chemicals), PETN (another name is Nitropenta), and SEMTEX (mixture of Nitropenta and Hexogen) will be presented and discussed in more detail.

Due to the need of improved control of the distribution of explosive material used for terrorist attacks the detection of the so-called homemade explosives like Triacetone Triperoxide (TATP) is obviously of increasing interest. TATP is an explosive which is characterized by a relatively high vapour pressure under normal conditions. At 25 °C the concentration of TATP in air is about 40 ppm. That makes it easy to detect TATP by IMS even without any sampling or pre-concentration process. Investigations were carried out in order to understand the ion mobility spectra of this substance. In dependence on the acid used during the synthesis of TATP the spectra are different. It can be concluded that the best identification is possible through the ion molecular peak in the positive mode independently on the type of synthesis. TATP made by means of HCl originates besides of the molecular ion peak in the positive mode a negative ion peak which is typical for chlorinated hydrocarbons. This negative ion peak does not appear in the case when H_2SO_4 is used as catalyst during the manufacturing of TATP.

Figure 5 shows the ion mobility spectra of TATP in the positive and negative modes.

Figure 6 shows the spectrum of 500 ng TNT wiped off from a table surface. The estimated detection limit is about 10 ng.

C4 is an explosive material mainly used in the US army. It is a mixture of Hexogen and plasticising chemicals. Therefore it can be identified by detecting RDX as the main compound of C4 in the negative mode of the IMS or by detecting the plasticising components in the positive mode. Figure 7 shows the spectrum of pure RDX (Hexogen). The detection limit is about 50 ng.

In Fig. 8 the positive spectrum of real C4 is shown.

In Fig. 9 the spectrum of pure PETN is shown. The detection limit is about 50 ng.

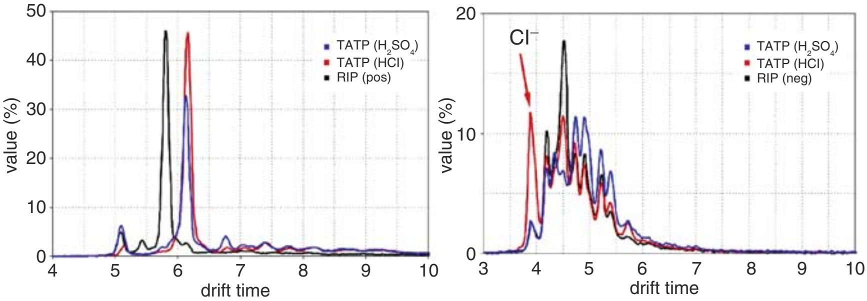

Fig. 5 Positive and negative ion mobility spectra of TATP

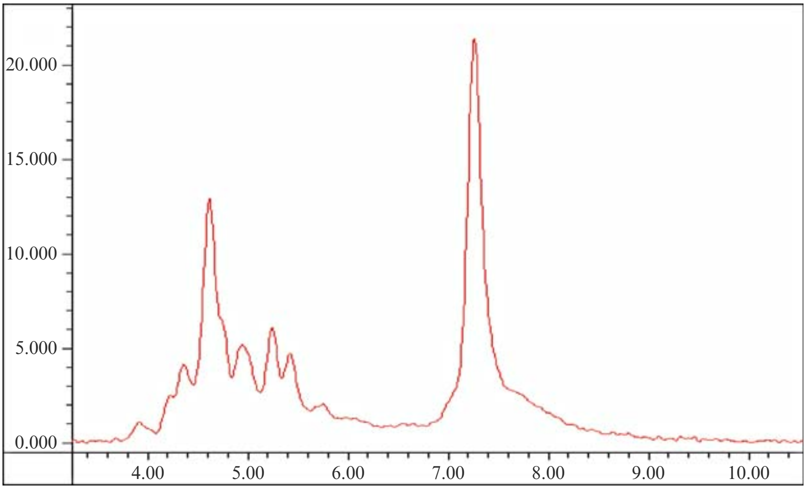

Fig. 6 Spectrum of 500 ng TNT wiped from a table surface

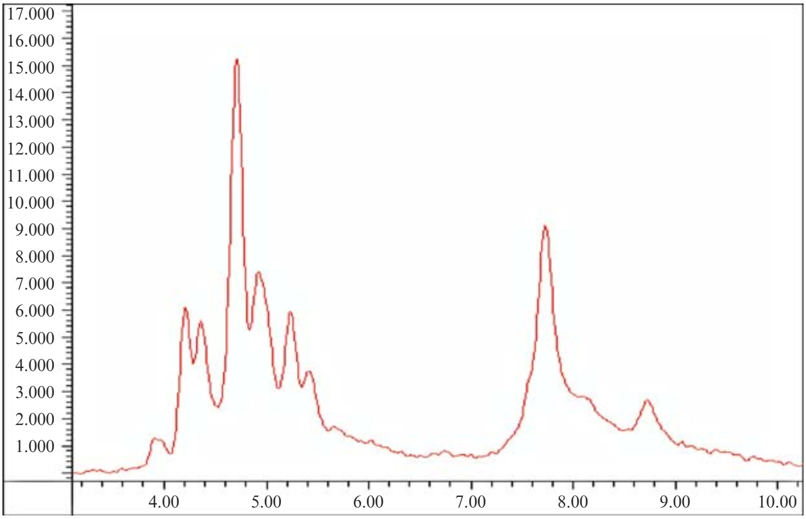

Fig. 7 Spectrum of RDX, wiped from a table surface

The mixture of PETN and RDX plus plasticizing additives and wax gives SEMTEX. The spectrum of SEMTEX is shown in Fig. 10. It is seen that both compounds are clearly separated.

Technical Parameters of the IMS device:

- Dimensions: 25 cm × 14 cm × 36 cm
- Weight: 6 kg including rechargeable battery
- Input flow: 200 ml/min

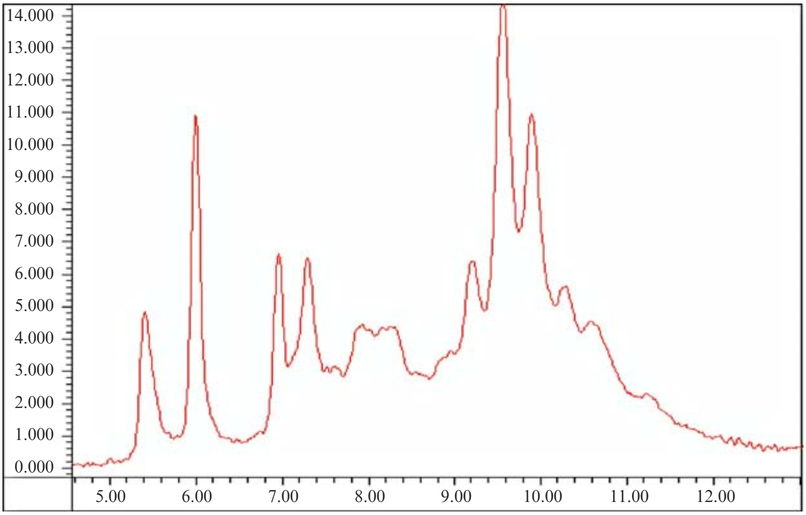

Fig. 8 Spectrum of plasticizing additive of C4

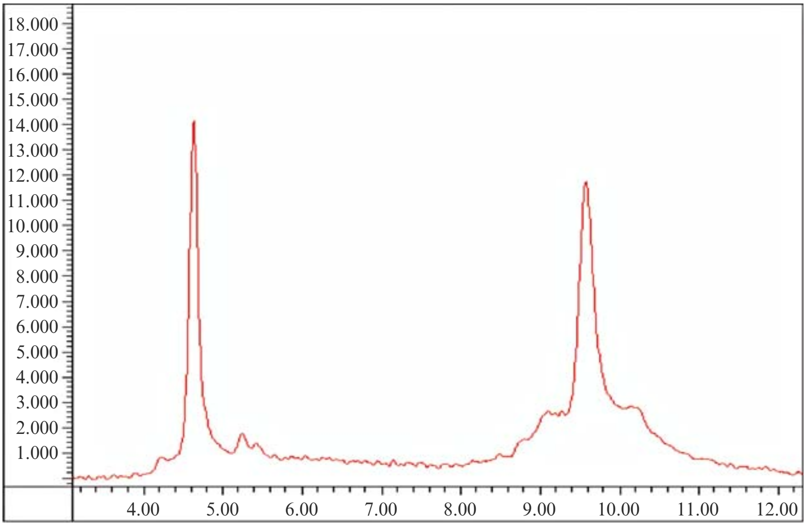

Fig. 9 Spectra of 1,000 ng PETN (basic explosive of SEMTEX), wiped from a table surface

- Operation temperature: −10–50 °C
- Air moisture: 0–90% relative humidity
- Power supply: 110 V/220 V, 12 V rechargeable batteries
- Power consumption: 50 W
- Response time: few seconds
- Warm-up time: 5 min

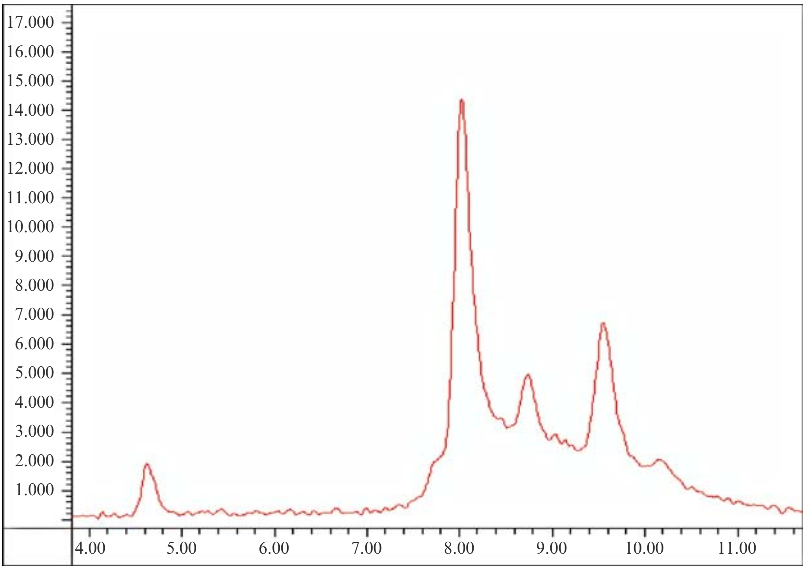

Fig. 10 Spectrum of SEMTEX, wiped from the table surface after contaminating it with traces of SEMTEX

- Running time with rechargeable battery: several hours
- Consumables: particle and molecular sieve filters

The device does not need any chemical reactive (dopants) and does not use a membrane. The inner circuit filter must be changed every 6 months. Due to the good selectivity of the device the rate of false alarms is lower than 4%.

Currently the device should be mainly operated when connected to power supply. The battery runtime is still limited due to the consumption of 50 W by the heated probe and the detector unit.

The detector is equipped with a small radioactive ionisation source, which activity is under the free limit activity according to the radiation regulations of the EU, and does not require special clearance or educated persons.

Actually this device is under testing at different German and Italian companies and authorities.

2 Bulk Detection of Explosives by Neutron Analysis

At IUT several devices were developed on the basis of neutron and gamma radiation technology. One of the simplest and low cost methods used in the field of bulk detection is the neutron backscattering technology, which gives good results in certain cases. The basic principle is that fast neutrons emitted from an isotope source or from a neutron generator irradiate the object, where they are slowed down

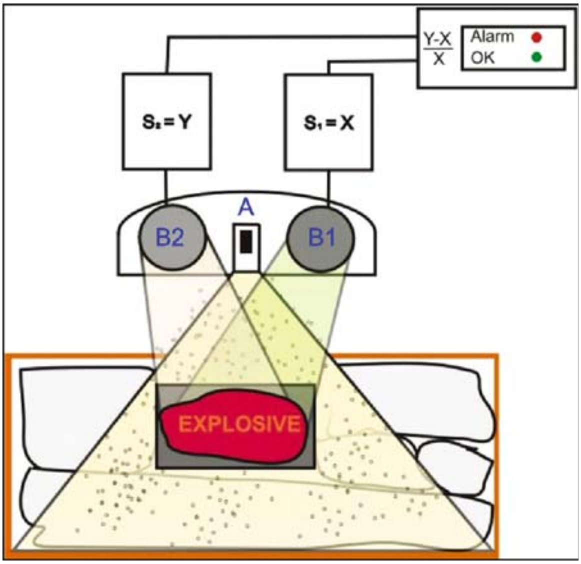

Fig. 11 Principle setup of the so-called NeutroTest device

(thermalized) and backscattered by light elements, mainly hydrogen. These backscattered neutrons which contain information about the composition of the investigated object are detected by a special arrangement of neutron counter tubes. The so-called NeutroTest device is a non-destructive measurement device. Neutrons are able to penetrate even centimeters of steel or lead without any problem. NeutroTest is best suitable for the detection of hidden substances like explosives.

In Fig. 11 the scheme of such a device is shown. The detector unit contains the neutron source (A) and two ^{3}He neutron counter tubes (B). The electronic units of each neutron counter tube consist of a preamplifier, an amplifier, a high voltage circuit and a circuit for data processing. The backscattered neutron fluxes cause in neutron counter tube B1 the signal S1 = X and in tube B2 S2 = Y. In case of ^{3}He neutron counter tubes the signals are mixed: backscattered fast and thermal neutrons are registered. To separate fast and thermal neutrons one of the neutron counter tube is surrounded by cadmium shielding, which absorbs thermal neutrons and therefore the signal reflects fast neutrons only. In case of calibrated and identical neutron counter tubes the difference Y-X gives the part of thermal neutrons, if tube 1 is cadmium shielded. The ratio of thermal to fast neutrons is equal to R = (Y-X)/X, which is independent of the geometry and gives extra information about the type of material.

The ratio of thermal to fast neutrons allows to cancel out uncertainties mainly coming from geometrical factors. Since the number of particle in a given solid angle depends on the distance with $1/r^2$, the counting rate measured by the neutron tubes depend even with $1/r^4$ on the distance since the distance source-object and object-detector has to taken into account. As shown in the following formula the geometry factors are cancel out since they are the same for fast and thermal neutrons, and the ratio depends only on properties of the material of the object which scattered the neutron back. With the ratio the result is independent of the distance, in first approximation.

$$Ratio \sim \frac{N0(source)\cdot Gl\_th\cdot G2\_th\cdot H\_th(Object)}{N0(source)\cdot Gl\_f\cdot G2\_f\cdot H\_f(Object)}$$

$$Ratio \sim \frac{H\_th(Object)}{H\_f(Object)}$$

N0(source) → activity source
G1 → Geometry factor, describing the distance between source and object
G2 → Geometry factor, describing the distance between object and detector
H → scatter properties of the object
f → fast neutrons; th → thermal neutrons

The described technology was prepared with certain modifications for two applications. The first application is the identification of the content of shells, which is necessary for the decomposition of old ammunition. The second application is the classification of the content of bottles, spray cans, thermos flasks and similar containers of liquids for safety reasons, for example during the control of luggage of passengers on airports or visitors of public buildings.

In both applications a neutron source is used which consists of a composite of the isotope ^{241}Am (emits α-rays) and ^{9}Be. By means of this source about 10^6 neutrons per second are generated through the reaction ^{9}Be (α, n) ^{12}C. The emitted neutrons penetrate the wall of the object being under investigation and interact with the atomic nuclei of the substances inside the object. During this interaction the neutrons change their energetic characteristics. The backscattered neutrons which again penetrate the wall are then detected by neutron counter tubes filled with ^{3}He under a pressure of 810 kPa (8 at). In dependence on the analytical task the diameter and the length of the neutron tubes have to be optimized. The signal processing is carried out by an electronic unit which has to be adapted to the chosen neutron tubes. By means of sophisticated evaluation software it is possible to reach good results in determination of the content of shells. Before the beginning of the measurements teach-inprocess has to be carried out with objects of known content.

The apparatus can be used effectively in the case when a big amount of shells has to be analyzed. The transport of shells to the sensor unit is realized by means of special transport mechanism (Fig. 12).

The second application for the detection of liquid explosives in sealed container like bottles is the so-called BottleScanner (shown in Fig. 13).

By means of this BottleScanner a big variety of containers with different contents was tested. It came out that this relative simple apparatus is able to discriminate between drinking water, beverages or food and liquid explosives like nitro methane.

In this apparatus the geometrical arrangement of the neutron source and the neutron counter tubes differs from the previous shell detection device in order to

Fig. 12 NeutroTest apparatus for the inspection of shells

Fig. 13 BottleScanner for the liquid explosives detection and representation of inspected objects

make the measurement and subsequent decision more or less independent from geometry and size.

3 Conclusion

The presented IMS and neutron devices have shown their ability to answer questions about possible hazards connected with hidden explosives or unknown content of shells, containers or vessels. Thus the described methods and devices can help to protect people from terror attacks or professional hazards.

Possibilities of Liquid Explosives Countermeasures at Airports

J. Turecek

Abstract In this article the possibilities of liquid explosives revelation during routine checking of person and baggage are discussed. Both the containers filled with liquid explosives and assembled IEDs can be detected easily. There are a few possibilities to distinguish the kind of liquid by common x-ray system at checkpoints. Apart from some particle detectors the explosive and flammable liquids can also be sniffed out by dogs. The more stringent regulations relating to liquids in the cabin baggage and people profiling is just another possibility. There is a requirement for more reliable detection methods based on dielectric and spectral measurements (glass and plastic containers), thermal characteristics (metal containers) and smaller and cheaper devices based on neutron activation of gamma radiation (especially metalized plastic or metalized paper).

Keywords Liquid explosives, luggage screening, handheld detection, detection dogs

The air terrorist plot in London in August 2006 in a large extent medialized the threat of using explosive and flammable liquids by suicide bombers on board the aircraft. It exerted a sudden intensive political pressure to solve this long-term problem. As it is analyzed later, explosive and flammable liquids do not pose a significant complication during the security screening of check-in luggage and passengers regarding other states of matter forms of these dangerous matters. The main problem consists in the possibility of smuggling explosive or flammable liquids in cabin luggage brought by passengers on board the aircraft and on the assumption that a suicide attack might be carried out. This problem can be solved in several ways the best of them is the use of a suitable detection technology.

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1 Liquid Explosives in Check-In Luggage

The detection of improvised explosive devices (IEDs) containing liquid explosives in check-in luggage intended for an aircraft cargo space is not, in comparison with the detection of IEDs using other forms of explosives, more difficult. More to the contrary. The problem with check-in luggage consist in the fact that an IED is necessary to have been assembled ahead. It means that the ignition system must be also installed – there has to be a kind of a detonator with electric wires (or a detonation tube etc.) leading from it to an electronic system with a battery. All of this are markers for x-ray systems operators. Plastic explosives can be shaped into different common objects and first of all it is possible to plant in them a detonator invisible for x-ray systems operators. To design an equivalent of a detonator invisible for x-rays in a liquid explosive, i.e. a liquid detonator, is impossible. On the contrary – a detonator in a liquid explosive would be a very significant marker. Thus there is an assumption that an ignition system could be put either directly into the casing of a liquid explosive (a container – in this case it would be difficult to disguise it) or in its close vicinity. It would be extremely difficult to design an IED well disguised for x-ray systems.

In every case liquid explosives do not pose a hightened threat level to check-in luggage with regard to other state of matter forms. Both solid and plastic forms of explosives pose a similar threat.

2 People Screening and Liquid Explosives

In most security screenings people screening is still based only on metal detectors – on walk-through metal detectors with additional hand-held metal detectors and sometimes boot metal detectors. The detection of trace particles with the manual taking of samples is used mostly for a higher level of the screening and portal detectors of explosive vapours are not wide spread either. The method of people screening based only on metal detectors is excellent for the detection of hand grenades, mines with metal casing etc., but in fact it is really insufficient for the detection of the explosives themselves which are not electrically conductive or ferromagnetic. It does not apply only to liquid explosives but also to all state-of-matter forms of explosives.

The solution could be x-ray scanners that have been offered for many years and first of all newly developed people scanners using millimetre waves of electromagnetic spectrum. Mass application of these scanners will enable the detection not only of non-metal weapons, but also of all bulk objects hidden under the clothing of the screened people. Explosive or flammable liquids or liquids suitable for the preparation of an explosive are also detected by these scanners. Liquids needy to be stored in a container will be on the other hand difficult to disguise as a part of clothing. Microwave scanners or back scatter x-ray systems are not able to identify exactly the kind of a material. In this case of people screening in air transport it is not absolutely necessary to identify the kind of a material. These scanners namely check whether the passengers really took all objects out of their pockets and presented them to cabin luggage screening.

For a higher reliability and speed of the screening it would be suitable to use a combination of devices using other physical principles, for instance vapour detection, quadrupole resonance or terahertz spectrometry. Portal detectors of explosive vapours or detectors of trace particles of explosives with manual taking of samples by sucking vapours and wiping will generally raise the probability of explosives detection and the extent of deterrence of possible attackers. The portals combining the metal detection on people and the detection of explosives in boots by the method of quadrupole resonance can be, especially in case of their mass production, less expensive, and by integrating two different principles it saves space, a number of security personnel and especially the quickness of the screening. And moreover, passengers need not take off their boots and put them on the x-ray belt.[1] Liquids, however, do not seem to be the most suitable state-of-matter form to be used in boots. The promised terahertz wand could be a necessary means for people screening – especially for the screening of a higher level, because its price can be assumed rather high. But it seems to be unreplaceable for the screening of handicapped people in wheel chairs etc.

The detection of explosives on people is a problem solvable for the present security screenings. The liquid form of explosives does not change too much.

3 Liquid Explosives and Cabin Luggage Screening

The problem with the detection of explosive and flammable liquids, appropriately with the detection of liquids which, in case of making a mixture of them, create an explosive, arises during the security screening of cabin luggage. The principle of the problem consists in several points. The volume of a liquid (cream and gel) falling on one passenger is counted in kilograms without any security restrictions. There are a lot of kinds of them, their consistence and effective proton numbers have a wide spectrum of values, these values are rather variable even for one type of the declared volume[2] (e.g. English X-mas pudding?). Regarding the substances that are searched for the process is complicated by the improvised character of liquid explosives and therefore by a wide spectrum of their composition and by a high-level difficulty of the security screening of their production, the absence of their labeling etc. The main problem is a suicidal character of the attack, so that an IED may not be made up, and the inability of x-ray systems at terminals to identify

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[1] This improvement is not even degraded too much by a necessity for some passengers to take off their boots containing a high volume of metal particles. These metal particles might be a disguised weapon and therefore such boots are mostly checked by x-ray systems anyway.

[2] What ingredients does for example "English X-mas pudding" consist of exactly?

exactly the type of a substance. Security personnel detect a container with a liquid by means of x-ray systems. They will reliably distinguish for instance an empty bottle, a bottle filled with sand, a bottle filled with a liquid. They will also distinguish gel or cream from liquid according to the shape of the volume filling the bottle and dual-energy x-rays (even without automatic detection of explosives) enable their operator to distinguish a liquid containing a higher inorganic content by colours classified according to the liquid consistence.[3] For example considerably salt water[4] has on the x-ray image a greenish touch in comparison with orange colour of drinking water and soft drinks generally. But the operator is not able to identify the kind of liquid, gel or cream more precisely. It is possible to increase the accuracy of identifying the consistence of substances by x-ray dual-energy method, but it is not sufficient. There are a lot of types of chemical composition of liquids, creams and gels that passengers carry with them in their cabin luggage. They are different alcoholic and non-alcoholic drinks, perfumes, cosmetics, medicines, etc. And the fact that there is no ignition system in a liquid in a cabin luggage is not sufficient for the security. An ignition system can be hidden in any type of electronic items.

It is necessary to point out that the same problem also appears with a lot of types of solid organic items whose consistence – more precisely their effective proton number – is the same as with some type of an explosive. Typical examples are for instance chocolate and other food, some types of paper etc. In liquids, besides their big volume per one passenger, there is also a danger of a perfect sealing and cleaning of the surface of the container in which the liquids are kept. It is applicable especially for glass bottles. Opening bottles is appropriate only for higher-level checks – in case of a significant suspicion and inability to identify the content of the bottle by another method. Considering a rather relatively lengthy procedure and elaborateness of the taking of samples for the detection of trace particles it is obvious that it is useful to use this method but it is not sufficient for acceptable solution.

4 Security Restrictions Concerning Liquids in Cabin Luggage

As mentioned in the introduction the revelation of London air terrorist plot in August 2006 exerted a sudden political pressure to tackle the threat of using explosive or flammable liquids by suicide bombers on board the aircraft. An ideal solution would be a technical solution, especially from a long-term standpoint. But at the airports there was no available technology suitable for identifying chemical composition of liquids in an enormous number of containers in cabin luggage. It is the matter for the airports to be equipped with the detection technology, the com-

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[3] More exactly according to proton number.

[4] For example Dead Sea water – a popular souvenir.

mercial offer of this technology, its technical and operational parameters, and first of all it is the economic issue. As there was a considerable political pressure to solve the situation immediately, it resulted in a really simple, but effective and frequently used security measure – ban. On November 6th 2006 new long-term intensive security restrictions were introduced in connection with the transport of liquids in cabin luggage.[5] Each passenger is permitted to bring with him/her only a very small amount of liquids, both in the cabin luggage and on him/her. The liquids have to be packed in small amounts, maximum capacity 100 ml. All containers have to be placed into one clear, closed and resealable plastic bag with a capacity of no more than 1 l per person.

The restriction concerning liquids are applied not only to water and other drinks, soups and syrups, but also creams, gels, cleansing lotions, oils, perfumes, aerosols, gels including shampoos and shower gels, pressure containers including shaving foam, foam setting lotions and other foam formula and deodorants, pastes including toothpastes, mixtures of solid and liquid materials and all other materials of a similar consistence. These restrictions do not apply to liquids bought in airport shops situated behind the passport control. In case the items are bought in a sealed bag it can be open just after the passenger is checked in by security personnel. In case of interlanding flights this check-in is understood the one carried out at the last airport. This almost total ban on liquids is effective, but very embarrassing for the passengers.

5 Passenger Profiling

Passengers profiling is another non-technical security measure that is favoured by some security experts who appeal to use it in a wide scale.

The most severe is the Israeli way of passenger profiling. It consists in inquiring individual passengers and a follow-up risk evaluation. A lot of passengers are cross-examined by another security agent. This method is, however, time consuming and demanding for a number of security personnel and airport premises as well.

The second possible way is surveillance, sometimes called passive profiling. The goal is to identify passengers judged to be suspicious by secret agents or surveillance camera systems at airport premises.

The third way is active profiling. The active profiling means that each passenger is profiled just before the security check. On the basis of this profiling a decision concerning the in-depth check-in of this passenger and his/her cabin luggage is made. Positive profiling is based on assessing those passengers that represent only a small threat posing a narrow probability to carry out an attack on an airplane.

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[5] Restrictions on amount of liquids, gels and substances of a similar consistence transported in cabin luggage of passengers according to the European Union Regulation No.1546/2006.

These passengers are subsequently allowed to undergo just only the technical part of the check-in in a limited range.

Passenger profiling is only an auxiliary means. Nevertheless in this case it is supposed quite successful because a suicide attacker is profiled closely before his/her attack.

6 Specialized Desk-Top and Hand-Held Detectors of Flammable and Explosive Liquids

From the facts mentioned above it is necessary to have affordably priced compact devices (maximum desk-top type) for the detection of flammable and explosive liquids in individual containers. For these purposes it seems quite suitable to use dielectric, thermal or sound characteristics or optical characteristics of liquids. These desk-top and hand-held detectors of flammable and explosive liquids utilize in a large extent the fact that most drinks and cosmetics contain a lot of water. On the other hand explosive and flammable liquids mostly do not contain water at all or only a minimum amount. Dielectric constant of liquids for water analysis in individual plastic or glass containers is used for instance in hand-held or desk-top microwave detectors.[6] In these devices the container with a liquid is irradiated with electromagnetic signal in the microwave area. This signal is partly absorbed by the material of the checked object and partly it is reflected back. It depends on dielectric characters of the material. The operators have first to make a database of typical sizes of a signal for different objects. Then they can check bottles with soft drinks, wine and liquors, whether they may contain an explosive or another chemical substance, whether a drug has been dissolved in the drink or whether water has been substituted with alcohol. The disadvantage of this method is the fact that it cannot be used for metal containers and first of all that the size of the signal depends not only on the dielectric constant of the liquid but also on the size, shape and characteristics of the material of the case – walls of the container. In the database of the device there has to be the size of a signal from a container of the same type and volume and the operator has to compare the size of a signal with an item in the database.

Hand-held or desk-top detectors using quasistatic electric field[7] are also based on the measuring of dielectric constant of liquid. In these devices two or more electrodes are subjoined to the wall of the checked container and dielectric characteristics of the content are measured by means of quasistatic electric field between the electrodes. If more electrodes are installed it enables to eliminate the influence

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[6] For example Micro Spector M600 hand-held detector, Spatial Dynamics, Inc.

[7] For example a hand-held device VETOCH (SKAT), AVK Lab. www.avklab.ru; or Flammable Liquid Tester (Bottle Liquid Checker) SLC-215D, Tokyo Gas Engineering Co.

of the thickness of the container walls, the type of the material of the container wall and the size of the air pocket between the electrodes and the container wall. It is not, of course, possible to check the content of metal containers.

To analyse liquids in metal containers, namely in aluminum tins, devices measuring thermal characteristics of liquids are used.[8] In these devices the aluminum wall of the checked container is slightly warmed and the thermal characteristics of liquids inside the container are measured.

Another group of detectors includes optical spectrometers. These devices use namely laser Raman spectroscopy.[9] Their advantage consists in the fact that they have a potential to analyse substances of interest in different state-of-matter forms. Unfortunately they usually need several seconds to carry out the analysis and they are rather expensive.

Acoustic detectors[10] mostly emit ultrasound pulses at the container with a liquid and they measure speed of sound and acoustic attenuation.

7 Detection Dogs for the Detection of Explosive and Flammable Liquids

At present police pyrotechnists' dogs are trained especially for locating military and industrial explosives. Searching for IEDs, especially those liquid ones as well as flammable liquids has been a little bit left out. Nevertheless the dogs are an excellent tool for recognizing trace amount of vapours of different substances. Police dogs can be, besides routine patrol service, trained for special scent identification work. This can be, besides routine search work, also special scent identification work for criminalistic purposes, locating people (caves-in etc.), explosives, drugs, weapons, human remains and accelerant substances – fire centres at the seat of fire. The last mentioned method is used for the investigation of all causes of fire and it has been in use for about 20 years. These canines are used for the investigation of all fires being suspected of arson. It is based on the fact that arsonists usually use a flammable liquid – accelerant (mostly petrol, oil, diluent etc.). The perpetrator usually pours this liquid on the floor and objects in that area. The liquid soaks into porous materials and it runs into slits and cracks. Because of an insufficient inlet of oxygen it is possible to find, in many cases, imperceptible traces of this liquid in the fire seat. During the follow-up investigation, in practice it is about 1 h after extinguishing the fire, the dog locates and identifies this place. Then this place is analyzed, wipe samples are taken and the accuracy of samples is confirmed. In case

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[8] For example Flammable Liquid Tester (Bottle Liquid Checker) SLC-215D, Tokyo Gas Engineering Co.

[9] For example Ram-On detector, Senspex, Inc.

[10] For example HILDA Bottle Inspektor, www.spearheadinnovations.com

the samples are found positive they are submitted to forensic laboratories. This method was imported to the Czech Republic from the United States and it was for the first time used in the action in Prague.

In the Czech Republic the first canine was trained for this purpose by Mr Ludvík Pinc[11] who began cooperating with fire service investigators in Prague.[12] Subsequently this method became used in the whole of the Czech Republic. These dogs are also used for searching for arsonists that often move around the scene of fire and are contaminated with the flammable liquid. In this case it is important for the dog handler to arrive in the scene of fire as soon as possible and to go in civil clothing round the people standing by. Another possibility is to detect the contamination of a suspect tipped off in another way. For instance a detector dog is able to locate remains of car petrol on a person's hands even after several washings. Practice has confirmed that the dogs trained in this way do not even have problems to locate a flammable liquid hidden in luggage.

Flammable liquids that are possible to be located by detector dogs include namely car petrol that is most used by arsonists. Further among these liquids there are other light products, as cigarette lighter fluid, camping cookers fluid fuels, diluents, ethanol and acetone, middle light products as alcohol mineral, iso-vapours, xylenes, binary motor oils, heavy petrol and turpentine and heavier products as kerosene, crude oil and lamp odourless oils. This list cannot be complete because the range of goods is constantly extending.

Therefore there is no occasion to hesitate that dogs could be able to locate IEDs with flammable liquids. But it is not possible to use the dogs trained for locating accelerants at the scene of a fire for recognizing flammable liquids in IEDs because these dogs are not trained to keep down ferreting about, biting and barking, which could initiate an IED. For these kinds of work it would be necessary to select out of 18 month-old canines, the temperamentally apt ones, and to train them for this purpose. Similarly locating liquid explosives vapours is a matter of a specialized training which is still in its beginnings.[13] Taking into account the maximum continuous work activity of about 20 min dogs are appropriate for the highest levels of mass security checks of people and their luggage. Dogs are excellent tools for locating explosives in emergency security inspections of different areas. As it was mentioned above they are suitable for locating liquid explosives and flammable liquids. As the location consists in detecting trace amount of vapours, some problems with "hoax" alerts may be supposed during the detection of flammable liquids at airport premises.

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[11] Ludvík Pinc *Training and Utilization of Dog for Detection of Accelerants*, Prague: Czech University of Life Science, 2006, 49 p., www.czu.cz, pinc@af.czu.cz

[12] At present already legendary Ashie – has become the best-known bitch among firefighters.

[13] For example Long Island K-9 Service; New York, USA, www.lik-9.com train their own liquid explosives detector canines.

8 Conclusion

Fast and affordably priced desk-top and hand-held devices can represent a good solution for the detection of flammable and explosive liquids in passengers' containers. A wide scope of physical characteristics of unharmful liquids brought by passengers on board the plane will necessarily lead to the fact that some physical characteristics of these unharmful liquids will be identical with some explosives and flammable liquids. Moreover another fact has to be taken into consideration – liquid explosives can be dissolved, which will again lead to changes of their physical characteristics. It will unavoidably result in a higher percentage of hoaxes. That could be reduced by improving devices or even better by combining different methods in one device. A certain number of hoaxes of detection failures have to be taken into account. Substantial security checks should be based on more physical principles. Less percentage of failures is not a sufficient reason not to introduce these devices. Passive passenger profiling – their surveillance in lounges – is used at a lot of airports. And what is the experimentally elicited detection failure percentage of these officers? To carry out higher-level security checking it will be more suitable to use devices based on gamma-ray neutron activation or trace particles detection etc. For occasional searches at airport lounges and other open areas it is pertinent to use trained canines.

Close-Range and Standoff Detection and Identification of Liquid Explosives by Means of Raman Spectroscopy

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Abstract The problem of standoff detection of explosives is discussed and experimental results of detection and identification of explosives at distances up to 55 m are presented.

Keywords Strand-off detection, explosives, Raman spectroscopy

1 Introduction

Improvised Explosive Devices (IEDs) are a common and growing threat to military operations as well as civilian society. In fact, in Iraq, IED attacks is the largest cause of death for US troops (Fig. 1). Although IEDs come in many shapes, sizes and containers, and use many varieties of explosives, initiation methods and detonators they have one thing in common: They are unique in nature because the IED manufacturer has had to improvise with the materials at hand. For this reason, the threat varies in different parts of the world; it varies over time, and evolves as countermeasures are taken for protection against them.

The diverse and varying nature of IEDs poses great demands on the methods for their detection. An IED typically consists of the following components: initiation system or fuze; explosive; detonator; power supply for the detonator; and container. Most of these components may be improvised from common materials and detection of these components will have difficulties to identify them as hazardous or non-hazardous. However, the explosive, as the one in it self dangerous component, is a good indicator for the presence of an IED threat. Therefore, detection of explosives is a key component of countermeasures against the IED threat.

IEDs are used in many different ways and situations. They can be categorized for example as follows: Roadside bombs, Vehicle born IEDs (VB-IED), suicide vests, and other. According to statistics from The Brooking Institution [1], during

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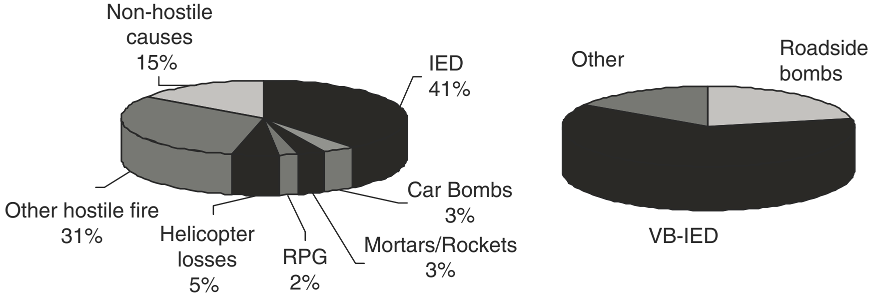

Fig. 1 Cause of death for US troops in Iraq, March 2003- September 2007 (Statistics from The Brooking Institution [1])

Fig. 2 Possible Standoff IED detection scenario

January to September 2007 22% of IEDs with multiple fatalities in Iraq were Roadside bombs, 62% were VB-IEDs and 16% other (e.g. vests).

There are many ways in which a roadside bomb may be located and detonated (Fig. 2). They can be disguised as anything and be hidden among other debris along side the road. To avoid being caught up in an attack, it is of vital importance to spot the IED well before it is passed. This includes also determining that an object is *not* an IED, e.g. under circumstances when an easily spotted but fake IED causes a vehicle or convoy to stop just in the right place to be attacked by well hidden real IEDs.

To this end (spotting the IED), quick and reliable standoff detection of explosives with wide area surveillance capability is required. The capability for detection of IEDs at safe distances is one of the most desired ones. Technologies for standoff detection are currently being researched and developed, but no system with explosives stand-off detection/identification, for distances of tens of meters capacity, are currently deployed or in use.

In the report "Existing and Potential Standoff Explosives Detection Techniques" [3] the authors defined standoff detection of explosives in the following way:

> *Standoff explosive detection involves passive and active methods for sensing the presence of explosive devices when vital assets and those individuals monitoring, operating, and responding to the means of detection are physically separated from the explosive device. The physical separation should put the individuals and vital assets outside the zone of severe damage from a potential detonation of the device.*

The zone of severe damage varies with scenario and bomb type but they chose 10 m for a pedestrian suicide bomber and 100 m for a vehicle based bomb. The zone of severe damage for road-side bombs was not defined. However, it is clear that considering the movement of a vehicle the standoff distance required for detection is quite long and 100 m would be a considerable improvement.

It is important to keep in mind that different threats require different technical solutions and that a solution that is appropriate for one environment and type of situation is not necessary good for another situation. Standoff detection poses requirements very different from a check-point type of scenario such as an airport check-point. However, certain development criteria can be specified as universally desirable, such as lower false alarm rate, higher selectivity and sensitivity and with an increased range of detectable threat substances.

Several technologies have been suggested and investigated for standoff detection applications [2, 3]. The technologies with best potential for being fast, selective, sensitive, able to detect many substances and upgradeable to new threats are laser based spectrometric methods for trace detection. Even though some bulk detection methods also have potential for standoff detection they are generally of imaging types that give less information. They may however make an excellent complement to the more specific trace detection technologies by providing information on the presence of electronic devices (maybe part of an IED) or suspicious, concealed objects under clothing.

One of the detection methods we consider having greatest potential to meet the requirements is Raman spectroscopy. Raman spectroscopy has been used as a standard analytical tool for identification of chemical substances for many years. As such it has excellent capability for high selectivity between substances and building a large reference database of threat and innocuous substances should be possible. This database would be expandable as new threats emerge.

As opposed to Laser Induce Fluorescence (LIF), Raman Spectroscopy is an instantaneous process; an inelastic scattering of photons where some energy is lost to (or gained from) the target molecule, returning scattered light with a different wavelength, the difference corresponding to an energy difference in the molecule. This way, Raman Spectroscopy probes the vibrational modes of the target molecules.

These vibrational modes can be regarded as a fingerprint that uniquely identifies the substance or substances in a sample. Also complex mixtures can often be analyzed using algorithms for pattern recognition.

Raman Spectroscopy has been studied for standoff applications by several research groups for various applications. Standoff Raman spectroscopic systems have been developed for characterization of planetary surfaces [4–8] at distances of about 5–65 m using doubled Nd:YAG lasers at 532 nm.

Carter et al. [9, 10] have studied the identification of 4–8% solid explosives in dry sand at up to 50 m using 532 nm from a Nd:YAG laser.

Sharma et al. [11, 12] have made a portable remote Raman system for monitoring environmental pollution and gases on planetary surfaces up to 100 m using the second harmonic (532 nm) from a Nd:YAG laser. They also measured stand-off spectra of explosives at 10 m distance as well as various liquid explosives and precursors at distances up to 100 m.

A disadvantage with Raman spectroscopy is the small cross sections as compared to infrared (IR) absorption spectroscopy or LIF. Raman scattering (inelastic scattering) is weaker than Rayleigh scattering (elastic scattering) by four to six orders of magnitude. However, just as Rayleigh scattering Raman scattering is proportional to $1/\lambda^4$ giving a significantly higher Raman signal in the ultraviolet (UV) region of the spectrum. Several authors have taken advantage of this fact using lasers in the UV region.

Brookhaven National Laboratory has developed a Mobile Raman Lidar Van (MRLV) [13] for identification of bulk chemical spill (surface contaminations, ~500 g/m^2) at distances of 0.5 km or more using quadrupled Nd:YAG lasers at 266 nm and up to 60 s integration time. They have also performed point UV Raman measurements on some explosives [14] in 0.5–1% concentration using 248 nm laser radiation. At this wavelength they noted a significant near resonance enhancement (*vide infra*) of some of the vibrational modes.

The Raman cross section can be significantly enhanced (by a factor of 100–10^6) [15] by resonance effects when the laser excitation frequency matches or nearly matches an electronic transition in the molecule.

Several papers discuss near resonance Raman spectroscopy on narcotics, explosives and chemical agents. Lacey and Sands et al. [16, 17] studied explosives and drugs with UV near resonance Raman spectroscopy using a frequency doubled CW Ar ion laser. They compare the Raman spectra of thin films of sample on a microscope slide (exposing typically 1 pg to the laser) when using 633 nm with Raman spectra with UV excitation at 244 nm. They conclude that fluorescence interference which can pose severe problems and even make measurements impossible at 633 nm is eliminated using 244 nm. They also observe resonance enhancement of the Raman bands associated with vibrations involving atoms on which the electrons involved in the electronic excitation are located. In some cases the spectra of similar molecules such as 3,4-DNT and 2,4,6-TNT are very similar due to the simplification of the spectra that the enhancement of only some Raman bands gives. However, they also conclude that the molecules can still be unambiguously identified with good enough resolution (in the order of a cm^{-1}). Nagli and Gaft [18] and Christesen et al. [19] have also studied near resonance Raman on explosives and chemical agents.

Hochenbleicher et al. [20] made a laboratory study for a resonance Raman LIDAR system on various gases. They found that absorption of the Raman scattered light by the scattering gas can reduce the observed resonance Raman signal to normal Raman scattering levels. From their study they concluded that the sensitivity of a Raman LIDAR system can only be significantly enhanced by resonance under three specific conditions:

1. A laser with a minimum of 10 mJ at a wavelength coinciding with a strong absorption band of the sought molecule must be available.
2. The absorption coefficient ε of this molecule is of the order of 10^3 $\mathrm{l \cdot mol^{-1} \cdot cm^{-1}}$ or more.
3. The gas is concentrated in a cloud or plume at the emission source and not yet dispersed in a greater distance from the source.

Phillips and Myers [21] studied resonance Raman effects of nitromethane in 5–18 mM solutions in cyclohexane, acetonitrile and water as well as in vapour phase (10 Torr). They observed high overtones in the spectra up to about 15,000 $\mathrm{cm^{-1}}$.

2 Experimental

2.1 Setup

The experiments described in this paper have been made in an experimental setup under development. Hence, the experimental setup has not remained constant throughout all experiments.

An oblique setup (Fig. 3) has been used throughout this work. Two different lasers have been used in the experiments; A pulsed Q-switched Quantel Nd:YAG laser, YG980, with pulse frequency of 10 Hz and 12 ns pulses of 280 mJ at 532 nm; and a pulsed Q-switched Exspla Nd:YAG laser, NT340, with a pulse frequency of 10 Hz and 5 ns pulses of 320 mJ at 532 nm.

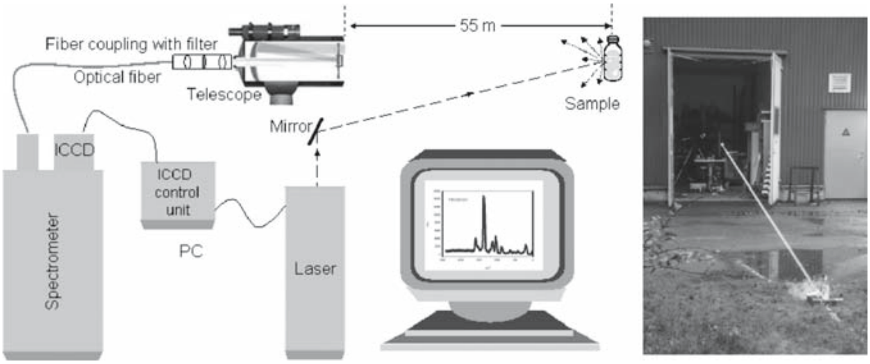

Fig. 3 Left: Experimental setup in oblique geometry. Right: Experiment photo at 12 m

The telescope was a C6-S Celestron, with 150 mm (f/10) aperture of compact Schmidt-Cassegrain construction with peak light transmission of 89% at 520 nm and an adjustable focus. This allowed for easy coupling into a UV fiber with a 400 µm core diameter. An interface designed by Thorlabs was used for this purpose, composed of two achromatic doublets for tighter focus (BK7, f = 45 mm, broadband AR coating centered at 587 nm) mounted in a stackable lens tube with a long-wave-pass filter in between. The filter was a LP03–532RU-25 from Semrock with OD 6 at the laser wavelength but transmitting over 99% for wavelengths above 538.9 nm (corresponding to a Raman shift of ~241 cm^{-1}).

Two different setups have been used. At a distance of 8 m from the laser, the laser beam is deflected by a mirror (Newport HR coated for 532 nm at 45° incidence) at approximately 90°. The laser is placed in an adjacent room at 8 m distance from the mirror used to direct the beam which is mounted next to the telescope in order to make the pathways as collinear as possible. Nevertheless, as the beam is not coaxial to the telescope field of view adjustments for maximum performance had to be made for each new distance and sample location.

It consists of a spectrometer with an intensified charged-coupled device (ICCD), connected to a telescope with an optical fibre and a light collecting interface with a filter and a frequency-doubled pulsed Nd:YAG laser.

A spectrometer from Jobin Yvon, SPEX 500M with a SPEX 500F fiber coupling was used. The grating (Richardson Grating Laboratory with 600 grooves/mm and blazed for 300 nm) allowed depiction of the interval 531–610 nm, corresponding to Raman shifts from –35.4 cm^{-1} to 2403.55 cm^{-1}, on the detector. Spectra were calibrated with seven lines of an Hg lamp and the laser wavelength. Raman spectra were recorded using a gated ICCD (Jobin Yvon CCD-3000i) controlled by SPECTRAMAX software. The detector has an air cooled thermoelectric housing and a maximum dynamic gain of 18.000. The chip measures 1,024 × 256 pixels, each being 26 × 26 µm. The detector was mounted in the spectrometer such that the Raman spectra were dispersed horizontally along the chip. When depicting the spectra in image mode it could be seen that the vertical extension of the spectra was covering no more that 30 pixels in the center of the chip. These pixels were binned together and the rest discarded to reduce electrical noise. The minimum gate achievable with the detector was 100 ns or more although much less according to specifications. This means that it was not possible to match the pulse length of the laser as would have been preferred to minimize electrical noise and the influence of unwanted fluorescence.

Reference spectra were obtained using a Bruker 55 FT-IR with FRA 106 Raman extension. The instrument uses a continuous YAG laser source of 1,064 nm.

2.2 Results

Experiments were performed with the equipment indoors and the samples outdoors in an explosives approved zone (Fig. 4). For laser safety issues the maximum obtainable distance was 55 m. Raman spectra have been measured at 12, 30 and 55 m. The liquid samples were placed in laboratory Pyrex glass bottles.

Fig. 4 The explosives approved zone used with the maximum safe distance, 55 m

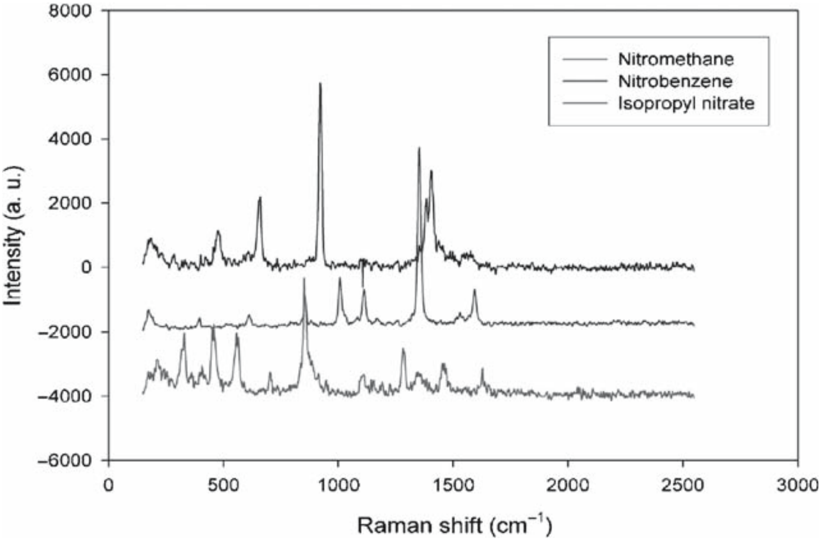

Fig. 5 Raman spectrum of nitromethane (upper trace), nitrobenzene (middle trace) and Isopropyl nitrate (lower trace) measured at 55 m distance with 100 laser pulses (10 s) and 200 µm slit width

Measurements have been performed in various ambient conditions – rain, snow, light fog, at night and during sunny, snowy winter days. Rain and snow do not give noticeably weaker signals. The absorption of 532 nm light in water is negligible. Under the circumstances at hand, the effect from scattering from raindrops and snowflakes does not effect the Raman signal intensity to a noticeable extent. Due to the very weak Raman signal of water, no interfering Raman signal is found in the spectra. Bright light conditions, even sunny winter days with snow, do not affect the quality of the measurements.

Figure 5 shows the spectrum of nitrobenzene, nitromethane and isopropyl nitrate at 55 m distance. The quality of these spectra is good enough to allow for longer

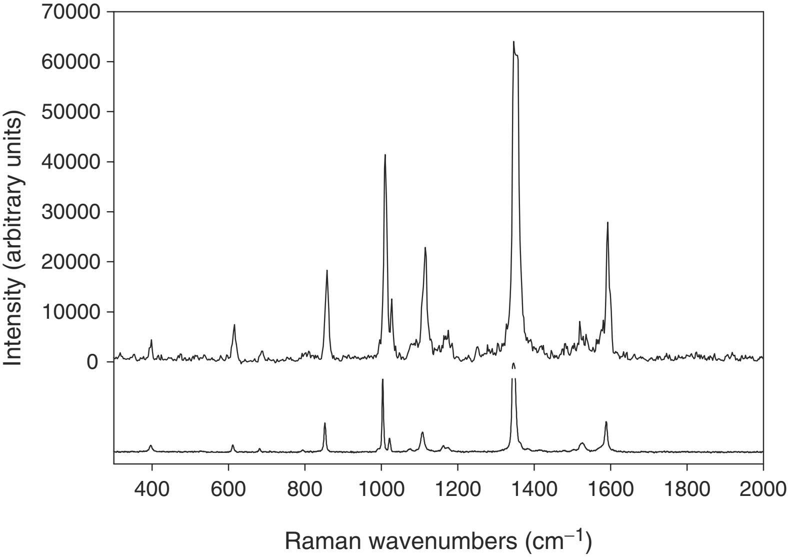

Fig. 6 Raman spectrum of nitrobenzene. Upper trace: Standoff measurement at 55 m using 100 laser shots (corresponds to 10 s). The largest peak has saturated the detector. Lower trace: Reference spectrum from the Bruker instrument (resolution of 2 cm^{-1} acquired using 100 mW laser power and by averaging 20 scans

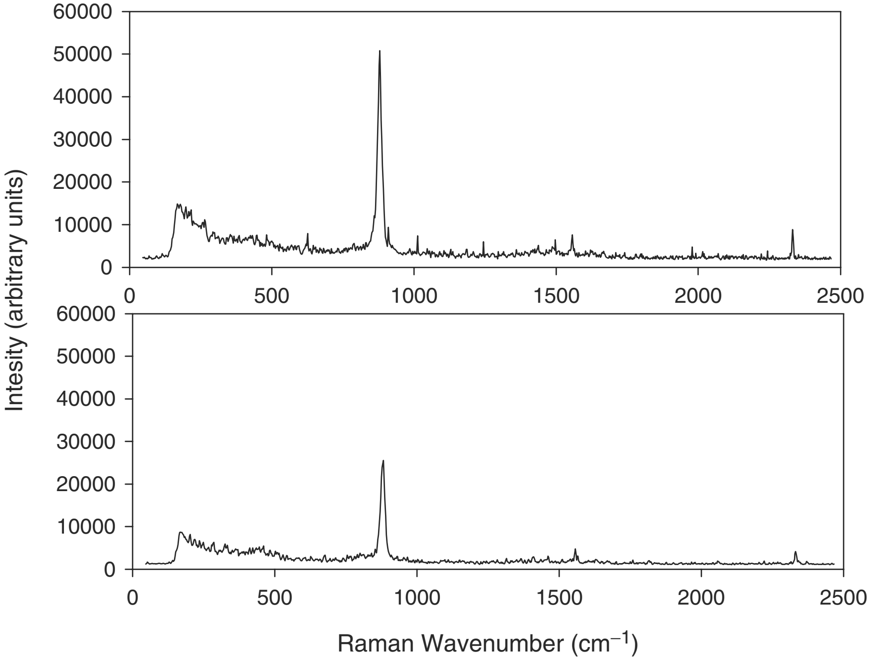

Fig. 7 Raman spectrum of hydrogen peroxide measured at 30 m. Upper trace: 100 shots (10 s) and 30 µm slit width. Lower trace: 10 shots (1 s) and 100 µm slit width

distance or fewer laser shots without compromising the ability to identify the content of the bottle (Fig. 6).

The Raman spectrum of hydrogen peroxide was measured at 30 m (Fig. 7). (The spectrum of hydrogen peroxide was measured using 100 μm slit width and 10 laser shots (1 s). When the measurement time was increased to 100 laser shots (10 s) the slit had to be decreased to 30 μm not to saturate the detector. Raman spectra of nitromethane and nitrobenzene at the same distance are shown in Fig. 8 and Fig. 9.

3 Discussion

Standoff Raman spectroscopy as developed so far lacks full operational capability since it requires line of sight to bulk quantities of the substance. Although this may be very useful under some circumstances it needs the added capability to detect the trace amounts available around the IED to be operationally useful.

There is considerable room for improvement of the detection capability of standoff Raman spectroscopy. This setup is not optimal since some of it is old and does not work to its optimal capacity. It is our assessment that it is possible today to build a detection system for bulk explosives that works over several hundred meters in a few seconds using standard components off the shelf.

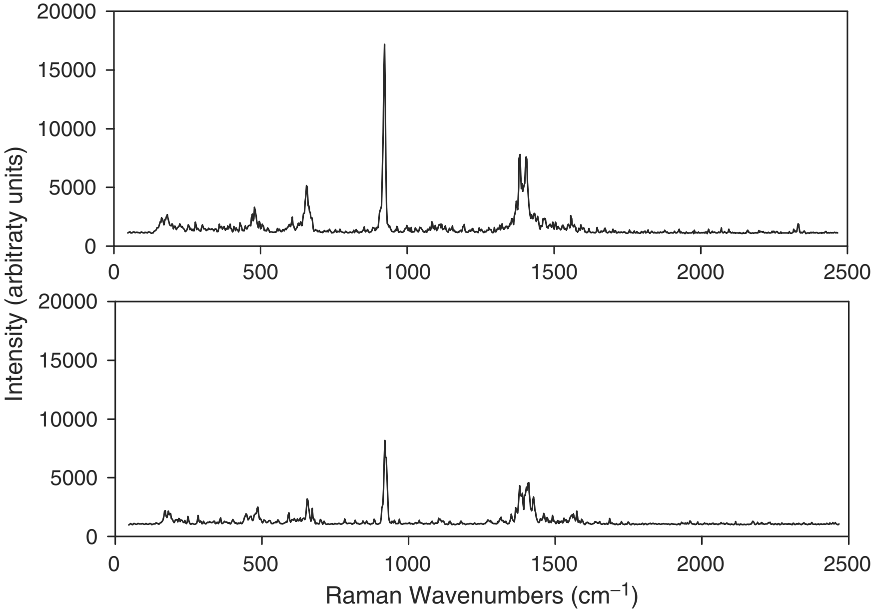

Fig. 8 The Raman spectrum of nitromethane measured at 30 m. Upper trace: Single shot (5 ns) using 100 μm slit. Lower trace: 10 shots (corresponds to 1 s) using 30 μm slit width

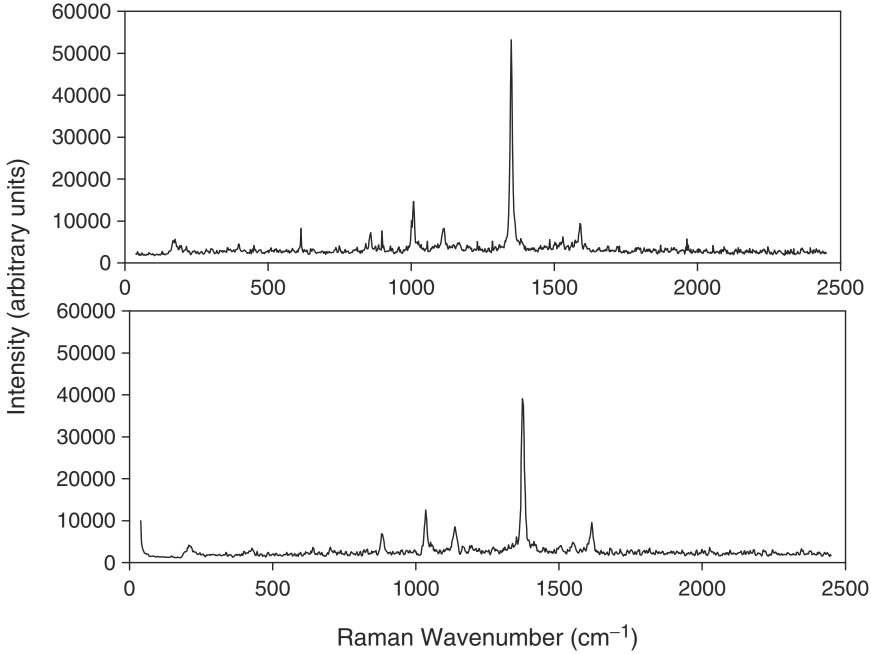

Fig. 9 Raman spectrum of Nitrobenzene at 30 m distance. Upper trace: Using 100 shots (10 s) and 15 µm slit width. Lower trace: Using 10 shots (1 s) and 100 µm slit width

To achieve better detection capabilities, important improvements to this system are:

- The use of UV wavelengths which reduces fluorescence problems and gives a higher Raman scattering intensity
- Shorter gate widths to reduce fluorescence with longer lifetimes than the laser pulse
- Coaxial configuration for easy re-direction of the measurement point
- Better collection optics

In order to use Raman spectroscopy for detection of trace amounts of explosives, e.g. particle traces, it is necessary to improve the sensitivity, probably by orders of magnitude, but also to obtain better knowledge about what we are looking for. How much material are we looking for, in which form and what are the properties of traces?

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Directional Detection of Nitrogen and Hydrogen in Explosives by Use of a DD-Fusion-Driven Thermal Neutron Source

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Abstract Results of 5-year task are described on the research and development of the advanced explosive detection system by using a compact discharge-type fusion neutron source called Inertial-Electrostatic Confinement fusion (IECF) device and directional γ-ray detectors made of BGO and NaI. With $>10^7$ neutrons/s stably produced in CW operation, 10.8 and 2.22 MeV γ-rays from neutron-capture reactions with nitrogen and hydrogen atoms in explosives (TNT, RDX) are detected well, showing promising features for bulk explosive detections.

Keywords Explosive detection, directional gamma-ray detector, neutron source, neutron-capture reaction, thermal neutron, inertial electrostatic confinement fusion

1 Introduction

Described are research and development on bulk explosive detection method by using a compact discharge-type fusion neutron source called IECF (Inertial-Electrostatic Confinement Fusion) [1, 2]. The present detection system makes use of nuclear reactions between the neutrons and explosive materials, by detecting neutron captured γ-rays of specific energies by hydrogen and nitrogen atoms to identify explosives. Also described are the performance test results of a landmine detection system by the use of this technique.

Since major explosives include C, N, H and O in specific fraction, interaction with neutrons, such as, neutron backscatter and neutron-induced γ-rays could be made use of as innovative methods for identification. Neutron emitters such as ^{252}Cf or AmBe of the intensity on the order of 10^5 neutrons/s, and a D-T tube, i.e., a small

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accelerator-driven fusion device, are being used for neutron emitters. Isotopes or tritium-included devices are, however, restricted due to radioactive isotopes, or due to operational mode, i.e. pulsed or DC neutron fluxes. From these viewpoints, a D-D fusion driven IECF device could be an ideal neutron source both from safety and pulse/CW dual mode operation.

We thus utilize thermal neutron capture reactions by the use of D-D IECF neutron source for detecting N and H anomaly, though C, N and O can be detected if it runs by D-T through inelastic collisions between fast D-T neutrons and those atoms. An important advantage of the latter scheme is that the associate-particle technique can be made use of for the position sensitive detection of the birthplace of neutron-induced γ-rays [3–5], while this technique cannot be applied for the former because the mandatory thermalization processes make the incident neutrons diverge. Instead, we have developed a directional γ-ray detector in order for the position sensitive detection of explosives.

The IECF neutron source shown in Figs. 1 and 2 consists basically of a spherical gridded cathode at the center of a spherical vacuum chamber (serves as an anode) filled with a D_2 fuel gas. A glow discharge takes place between them, thereby, produced ions accelerated toward the center through the gridded cathode undergo D-D fusion reactions through beam-beam or beam-background gas collisions. Thus it utilizes beam-gas colliding fusion, and therefore its long lifetime and long-term dc operation capability are the most advantageous features against conventional beam-target-type fusion neutron sources. The IECF neutron source also has advantages of robustness and easy operation owing to its extremely simple configuration, all of which are essential for the practical explosive detection application.

Actually, by this very simple device, D-D neutrons, and D-^{3}He protons both in excess of 10^8 s^{-1} in CW mode at UW [6, 7], and 6.8 × 10^9 D-D neutrons/sec in pulsed mode at TIT [8] have been achieved. This concept was first proposed

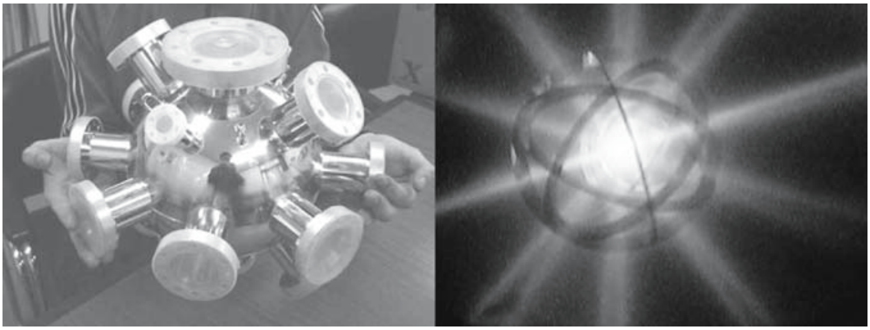


Fig. 1 An Inertial-Electrostatic Confinement fusion (IECF) device and a glow-discharge plasma within a gridded cathode held at the center of the device. The spherical chamber serves as the anode

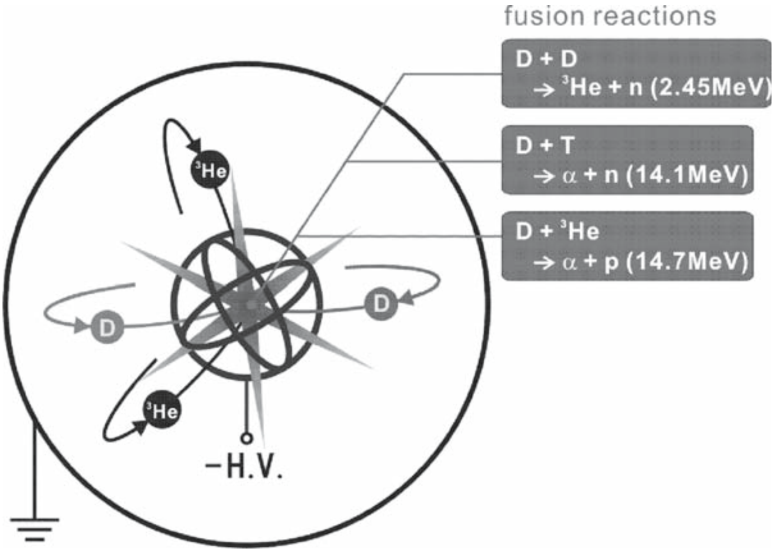


Fig. 2 Schematic cross-section of an IECF device. The ions produced by the glow-discharge are accelerated toward the transparent cathode and undergo fusion reactions through beam-background gas and beam-beam collisions

in 1950s aiming at the future fusion power plant, which is basically a beam-beam colliding fusion device with an extremely compact and simple configuration. Working with Fransworth at ITT laboratories, Hirsch obtained record neutron output of approximately 10^8 D-D neutrons/sec, and 10^{10} D-T neutrons/sec, respectively, in 1967 from a gridded IECF device driven by six ion guns [1]. After a long pause of the research, the present new concept came out, and studied for the versatile industrial applications. Among them, positron emitter production for cancer detection, boron neutron capture therapy (BNCT) for cancer treatment, and explosive detection, i.e. security and/or humanitarian demining application would be three representative application candidates. Although the first two will have huge market in the future, the third one is really urgent issue to be achieved.

For the landmine detection application, we have developed an extremely compact IECF device of 200 mm in diameter with a titanium getter pump as the main exhaust pump to endure the vibration when it is installed at automobile. A water jacket has been designed for both cooling the device and focusing the D-D neutrons toward the target, i.e. the minefield to enhance the incident thermal neutron flux into the mine. For the directional detection of the resultant γ-rays from the mine, we have developed a BGO/NaI-combined scintillation detector and the data processing system based on the anti-coincidence method.

2 Water-Cooled IECF Neutron Source

For the detection of the landmines, it is essential to run the neutron source as stable as possible in a high-flux CW operation. In addition, the enhanced fusion neutron flux towards the minefield is strongly preferred, although emission of fusion neutrons in the IECF device is isotropic. To meet these requirements, a new IECF device of 200 mm inner diameter was manufactured as shown in Fig. 3, which is equipped with a 5 cm-thick water jacket over the upper area for both cooling and reflection of neutrons, while the cooling channel thickness facing the minefield is 1 cm.

The developed device with the water-coolant has shown very stable CW neutron production as expected, without falling off due to arcing as seen in Fig. 4. Though the neutron yield is seen still decreasing gradually as the water temperature increases for constant bias voltage, cathode current, and gas pressure (see Fig. 4), it is also found that a lower chamber temperature by the water-cooling leads to a higher neutron flux as shown in Fig. 5, while the reason for this is left unclear. The water-cooled IECF device can thus produce 10^7 D-D neutrons/s stably in CW operation for 80 kV and 80 mA.

The envisaged D-D neutron flux focusing downward through the thin water channel at the bottom (see Fig. 3) was evaluated by both experiments and simulations by the use of MCNP-4C code [9] with the data library ENDF/B-VI [10]. Figure 6 shows experimental and numerical neutron count rate distribution around the IECF device. It is to be noted that again the count rate by the ^{3}He neutron counter can be regarded reasonably as the thermal neutron flux density. In comparison,

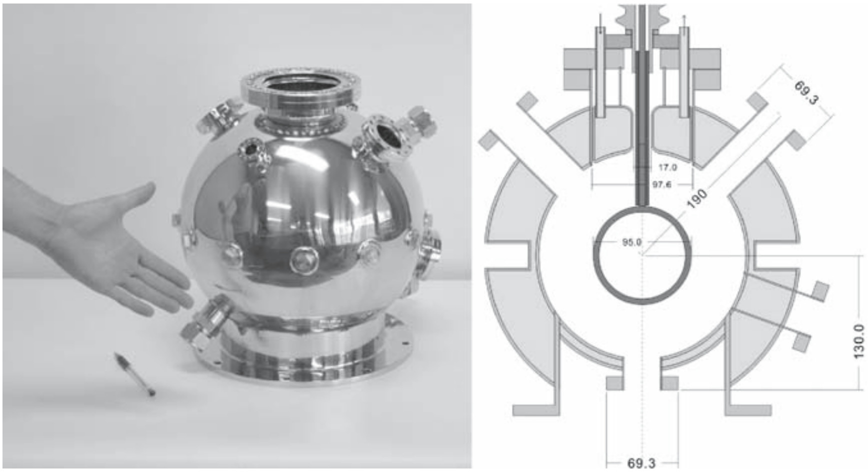


Fig. 3 Photo and cross-section of the water-cooled IECF neutron source. Water coolant is expected to serve as neutron modelator and reflector as well in order for downward focusing of neutrons

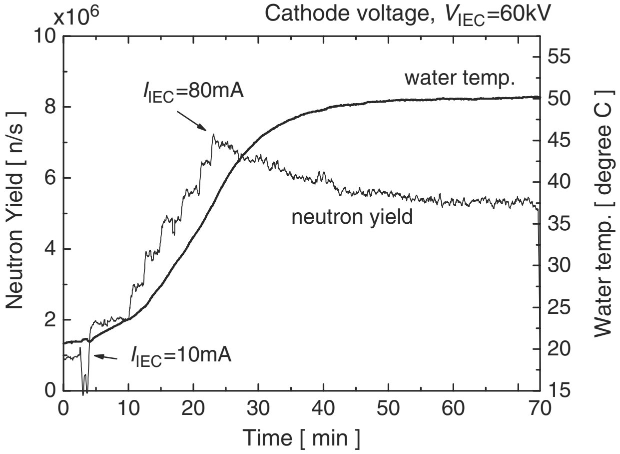


Fig. 4 Time evolution of neutron yield by the water-cooled IECF device and the water coolant temperature during continuous 70 min run. After reaching a constant discharge current of 80 mA, a gradual decrease of the neutron yield is observed due to increasing chamber temperature

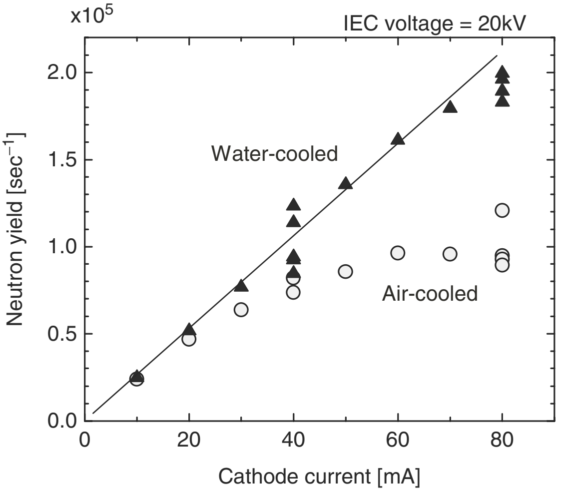


Fig. 5 Neutron yields as functions of discharge current comparing the water-cooled IECF and an conventional air-cooled one. The water-cooling is seen to result in an enhanced neutron flux especially for high current inputs

Fig. 7 shows the calculated distribution of the neutron flux density not excluding fast neutron component. These results show that the contrast between horizontal and downward thermal neutron fluxes is ~20% in Fig. 6, while for the fast neutrons in comparison, the contrast is much clearer, showing a factor of ~2 in Fig. 7.

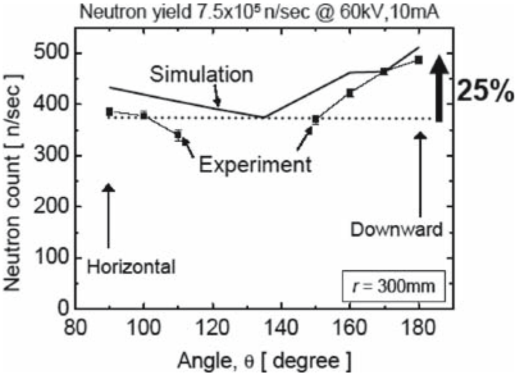


Fig. 6 Comparison of numerical and experimental neutron count rates by the ^{3}He proportional counter as a function of angle with respect to the vertical direction at a constant distance of 300 mm from the IECF neutron source center. In the calculation by the MCNP simulation code, the energy dependence of the ^{3}He counter sensitivity is taken into account

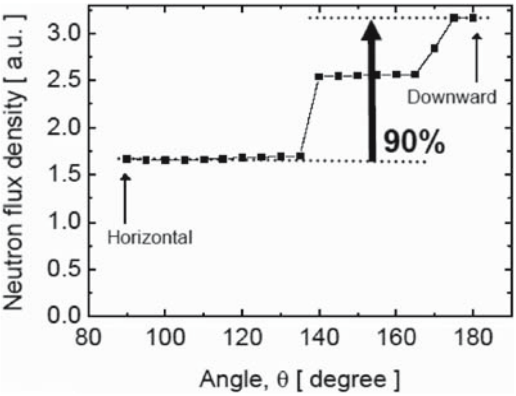


Fig. 7 Calculated neutron flux density distribution around the IECF neutron source

3 Directional Gamma-Ray Detector

For a well-collimated detection of the highly energetic γ-rays from N and H atoms in the explosive, we have developed a BGO/NaI (Bismuth-Germanium-Oxyde/Sodium Iodide)-combined scintillation sensor shown in Fig. 8.

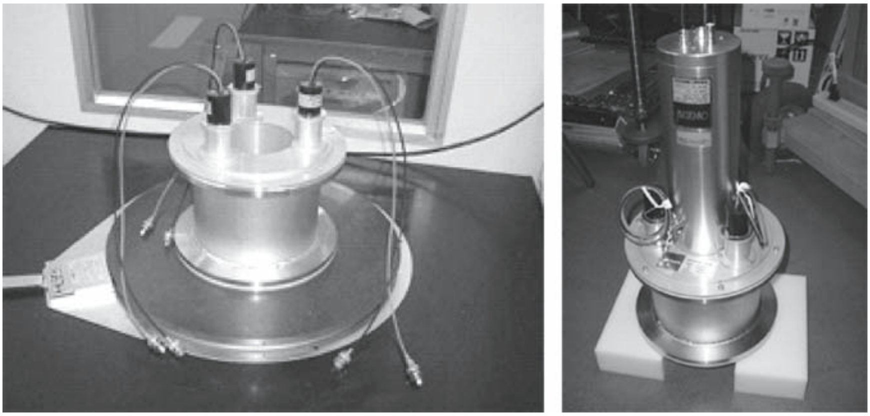


Fig. 8 A BGO/NaI combined scintillator for collimated γ-ray detection by the use of anti-coincidence method

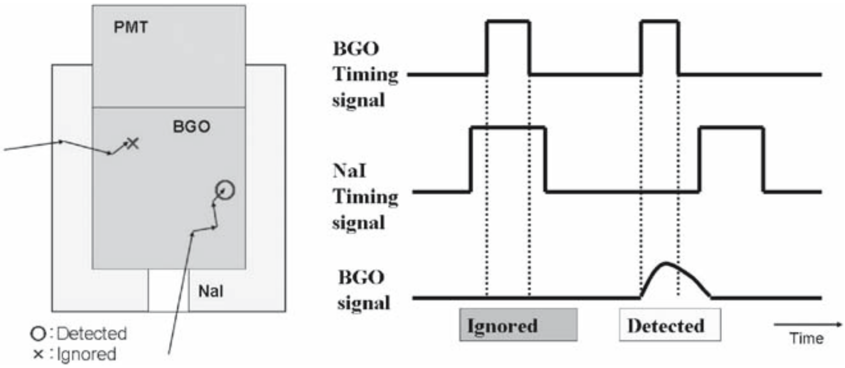


Fig. 9 Schematics of anti-coincidence method by the BGO/NaI combined scintillator

As is schematically shown in Fig. 9, the cylindrical BGO scintillator is placed in the center hole of the annular-shaped NaI scintillator. γ-rays injected into the BGO scintillator through surrounding NaI scintillator are rejected through anti-coincidence scheme, while those through the center hole of annular NaI scintillator are measured. The detection is thus collimated, and background level of γ-rays can be decreased. Though we have not evaluated the spatial resolution, it would be roughly several centimeters at several tens centimeter distance from the detector.

Furthermore the coincidence method is applied at the same time. A portion of the γ-rays through the center hole of annular NaI scintillator tend to escape from the BGO, while the escaping γ-rays mostly show a specific energy of 511 keV corresponding to pair production process. Thus the detection efficiency of the 10.8 MeV γ-rays through the hole can be enhanced by applying the coincidence scheme between a 10.3 MeV photon detected by the BGO and a 0.5 MeV photon by the NaI.

4 Landmine Detection System

We have developed a landmine detection system with the above mentioned IECF neutron source. The photo of the prototype system and the sectional top view are shown in Figs. 10–12. The landmine detection system is equipped with three sensors whose angle with respect to the minefield can be remotely adjustable to corresponding landmine depth. To avoid activation of the NaI scintillators, 5-cm thick powder LiF and a thin lead plate were placed between the IECF neutron source and the detectors for neutron and γ-ray shielding, respectively.

We conducted tests to examine the performance of the prototype system in July through September, 2007 by the use of wax-diluted TNT and RDX as the landmine simulant. The explosives were buried in a soil box set under the detection system as shown in Fig. 13. A typical γ-ray energy spectrum is shown in Fig. 14. One can clearly see peaks of the capture γ-rays of 2.22 MeV from H and 7.63 MeV from Fe,

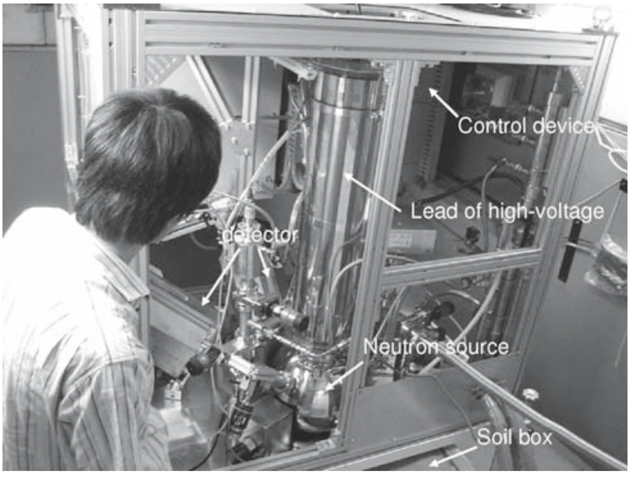


Fig. 10 Landmine detector prototype

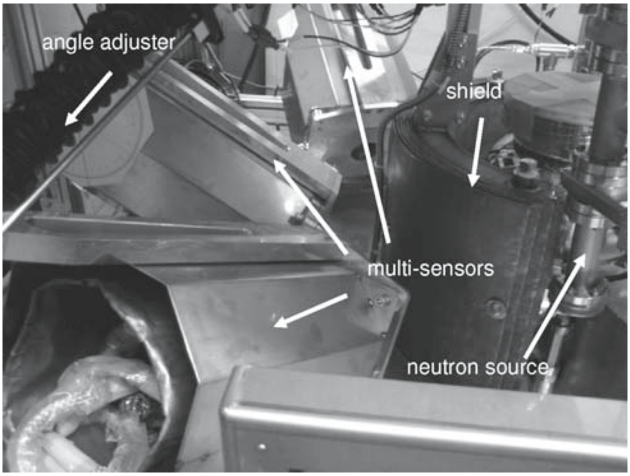


Fig. 11 Three directional γ-ray sensors in the landmine detector

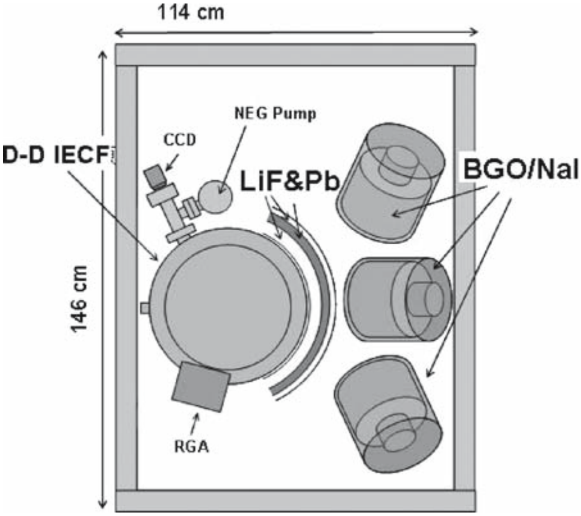


Fig. 12 Sectional top view of the landmine detection system

which are to be used for the energy calibration. Though the 10.83 MeV peak from N is not so clear, the enhancement of the γ-ray count by the existence of the explosive is seen in the magnified view around 10.83 MeV.

The detection tests were done for several cases:

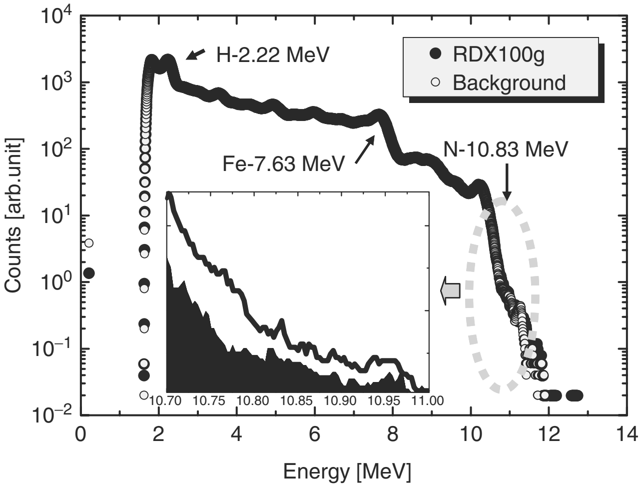
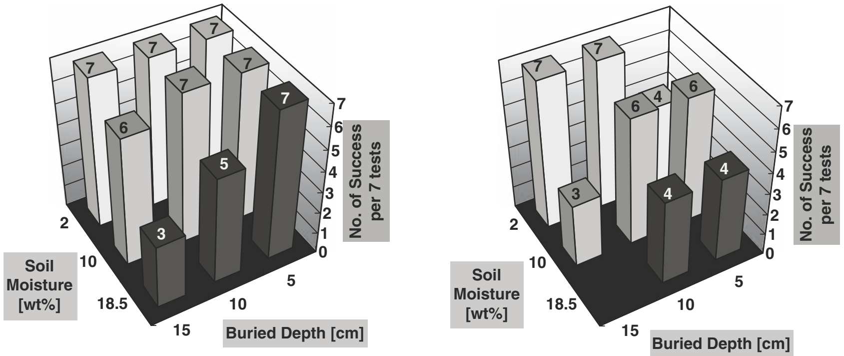


Fig. 13 γ-ray energy spectra with and without a landmine stimulant containing 100 g of RDX

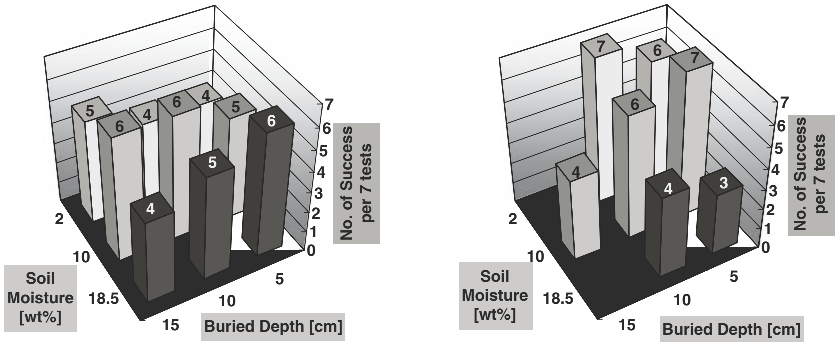
Fig. 14 Setup of the landmine simulant

(i) TNT 240 g, TNT 100 g, RDX 100 g, RDX 29 g
(ii) Depth of explosive: 5 cm, 10 cm, 15 cm
(iii) Soil moisture: 2 wt%, 10 wt%, 18.5 wt%

The D-D neutron production rate by the IECF neutron source was ~1 × 10^7 s^{-1} DC. The judgment criteria of the landmine stimulant existence corresponds to a 50% false alarm rate.



a 240 g TNT **b** 100 g TNT



c 100 g RDX **d** 29 g RDX

Fig. 15 Performance test results of the prototype landmine detector, showing number of successful detection out of seven trials for various conditions

The results are summarized in Fig. 15. The vertical axis shows the number of successes out of seven trials. In total, 179 successes were made out of 231 trials, which is encouraging for the first prototype.

5 Summary

The present scheme using a D-D IECF device as a thermal neutron source in combination with directional γ-ray detectors has shown good performance in detecting nitrogen and hydrogen anomaly. A tentative detection probability by the prototype landmine detection system was found to be 77% in average, and in excess of 80% for arid soil, while further performance improvement is needed especially to reduce the false alarm rate for the practical use.

Acknowledgement The authors would greatly thank Japan Science and Technology Agency (JST) for supporting this work for 5 years.

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Discrimination of the Explosives from Other Materials by Using the Tagged Neutron Beam

D. Sudac* **and V. Valkovic**

Abstract Measurements made with the 100 kg of the explosive Semtex 1 a and 64.4 kg of the paper show that there is a very poor difference in the gamma ray spectra between the explosive and the paper. The nitrogen peak at 5.1 MeV is overlapped with the second escape peak from the 6.1 MeV oxygen line while the peak at the 2.3 MeV is not recognizable at all. By using the triangular diagram the way was found to separate the signature of the Semtex 1 a from that of the paper. The triangular diagram coordinates are the number of counts in carbon peak, the number of counts in oxygen peak and the number of counts in transmitted neutron peak.

The measurements made with the 2.5 kg of the explosive PETN and the various sea sediments and stones show that the whole spectrum signature should be used for the identification of the materials investigated.

In both cases targets were irradiated with the tagged 14 MeV neutron beam, characteristic gamma ray spectra were collected and associated alpha particle method was used. Position of the investigated target material was found in the time spectrum and the chemical composition of the target in the gamma ray spectrum.

Keywords Explosive detections, fast neutrons, container inspections, sensor

1 Introduction

In the past few year many effort have been put into developing a new system for contraband container inspections. It was believed that the efficiency of the commercial x-ray or γ-ray devices for finding the explosives could be significantly improved by using the fast tagged neutrons. The so called "associated alpha particle method" was planed to used for checking the parts of the sea container previously scanned by the x-ray device [1].

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Coastal regions in southern Europe, south-east Asia and elsewhere are contaminated by different ammunition left on the sea bottom after war activities. This represents a constant threat to the sea traffic, fishermen, tourists and local population. Adequate devices, which probably incorporate several physical method, has to be developed in order to map the GPS positions of the unexploded objects on the sea bottom.

In both cases discrimination of the explosives from the other kind of materials is needed. System has to be capable to found the explosive devices and discriminate it from the surroundings.

2 Experimental Arrangements

Two experimental arrangements are used in this study. The first one is represented in Figs. 1 and 2, showing the positions of the container, neutron source, gamma detectors and shields. Target is put in the middle of the container. Target is the iron

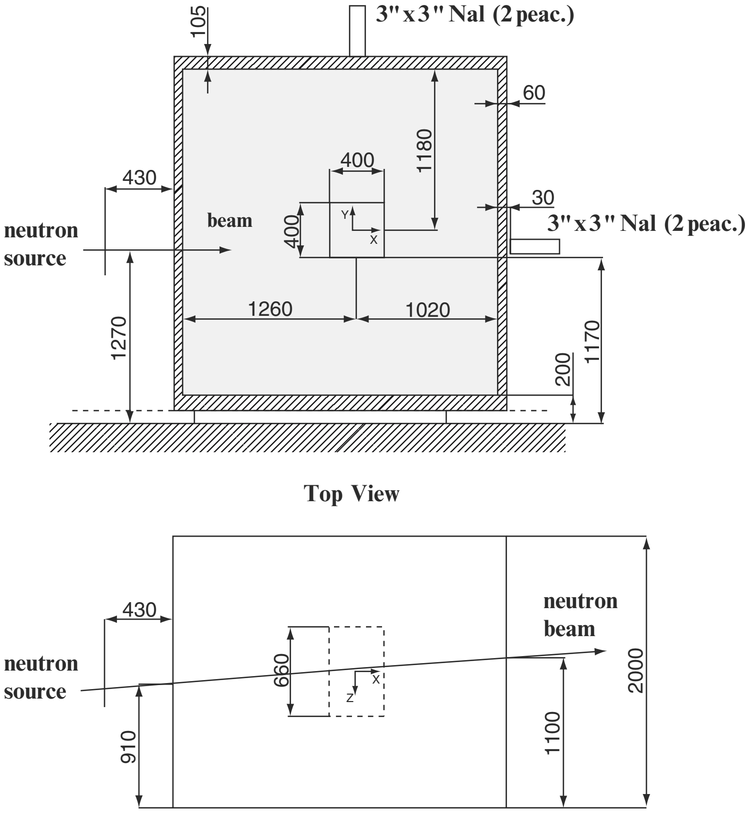


Fig. 1 Experimental set-up 1 (units in mm)

Fig. 2 Paraffine blocks over the neutron source and top detectors on the roof of the container (left) and transmission detectors (right)

box with the dimensions $40\times40\times66\,\text{cm}^3$ and mass 9.2 kg, fill with the 64.4 kg of paper or 100 kg of semtex 1 a, except in the carbon case. Coordinate system is put in the middle of the target. Neutron beam enter the container from the left at the position (−126 cm **x** −10 cm **y** +10 cm **z**) and exit the container at the position (102 cm **x** −14 cm **y** −10 cm **z**), respectively. Two 3″ × 3″ NaI(T1) were put at the top position over the container, on the roof of the container. Two 3″ × 3″ NaI(T1) were put at the transmission position in the cone of the tagged neutrons beam. Each detector was shielded with the 5 cm lead shield. Over the neutron source is paraffin blocks with the volume $40\times40\times40\,\text{cm}^3$ to protect the top detectors from the neutrons. For the same purpose one $10\times10\times30\,\text{cm}^3$ carbon block was put in front of the lead shield of the top detectors. Alpha detector (YAP) is 15 cm from the tritium target. Before the YAP is quadratic collimator 1.8 cm in length. Figure 3 show the electronic set-up. Four fast output from the four-segmented PMT connected to the YAP were fed through the octal constant fraction discriminator and fan-in fan-out to the STOP of the time to amplitude converters, TACs. Outputs from the sodium iodide detectors were fed through the constant fraction discriminators and “or” logic units to the START of the TACs. Slow signals from the gamma detectors were fed through the amplifiers, quad linear gates, and delay amplifiers to the acquisition system.

The second experimental arrangement is presented in the Figs. 4 and 5 showing the relative positions of the neutron generator, API-120, with the detection of associated alpha particles, 3″ × 3″ NaI(T1) gamma detector and the conical shield. As a target on the Fig. 5, explosive PETN was shown.

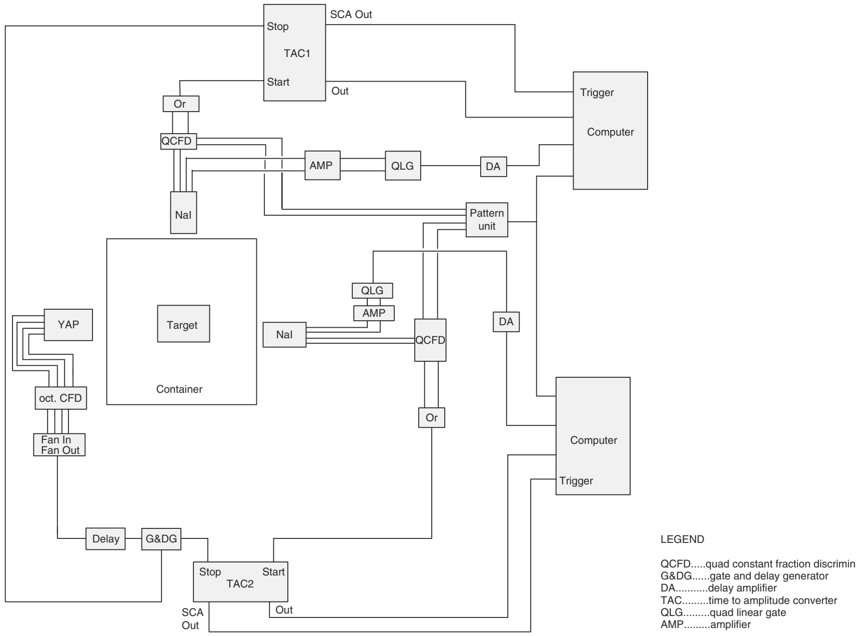


Fig. 3 Electronic set-up for the experimental arrangement 1

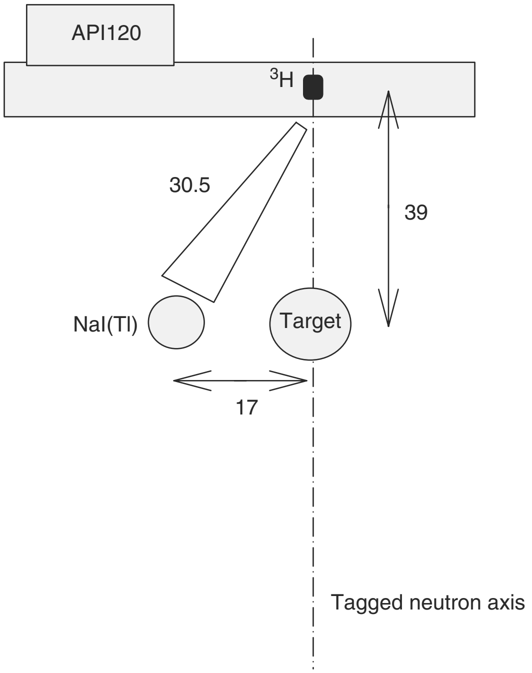


Fig. 4 Experimental set-up 2 (units in cm)

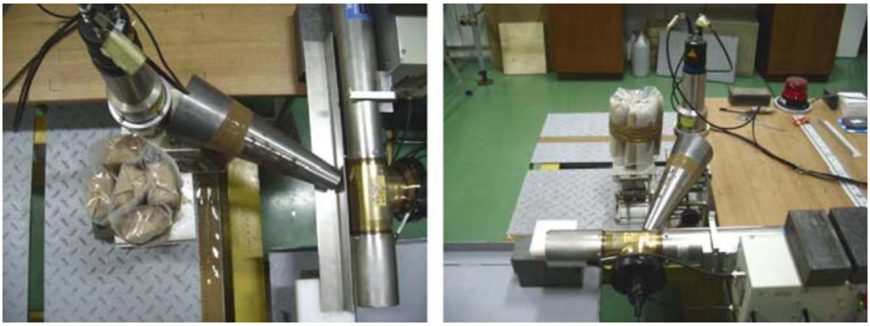


Fig. 5 Explosive PETN (2.5 kg) folded with the nylon et the target position of the experimental arrangement 2

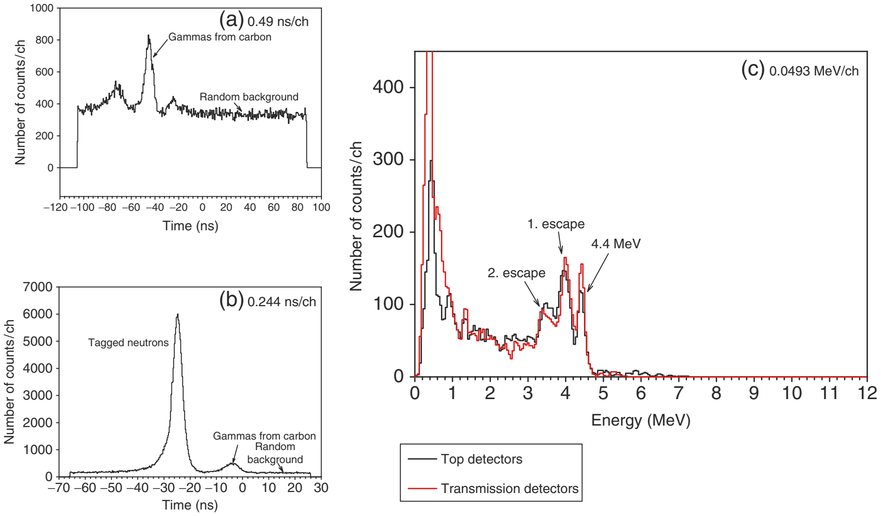


Fig. 6 The time spectrum (a) for the top detectors and (b) for the transmission detectors of the 48.6 kg of carbon. The gamma ray spectra (c) of carbon with the appropriate time window on gamma peaks. (Target volume = 30 × 30 × 30 cm^3, 1.6 × 10^8 tagged neutrons, elapsed time = 10,113 s, average intensity = 1.4 × 10^7 n/s)

3 Experimental Results

First will be discuss the results made with the experimental set-up 1. Performance of the system was checked by using carbon as a target. Figure 6 shows the time spectra and the gamma ray spectra for the top and transmission detectors. The gamma ray spectra were obtained by selecting the appropriate time window which contain the peak belonging to the gammas coming from the carbon (random background was subtracted). Main peak at 4.4 MeV, associated first and second escape peaks are clearly seen in both cases. Measurements have been performed with a real explosive Semtex 1 a and plain paper (see Fig. 7). Density of the explosive was found experimentally to be (1.275 ± 0.04) g/cm^3. There were 100 packages, 1 kg each with the volume approximately 4.7 × 12.2 × 14.3 cm^3. Each package was folded with the paper and nylon. Density of the paper was found experimentally to be (0.74 ± 0.008) g/cm^3. There were 25 packages, with the volume approximately 21 × 29.7 × 5.5 cm^3. Each package was folded with the nylon. Figure 8 show the time spectra (a) and the gamma ray spectra (b) of the paper and the Semtex a 1 for the top detectors, respectively. There is no measurable difference between the paper

Fig. 7 Iron box full with the 100 kg of the explosive Semtex1a (up) and iron box full with the 64.4 kg of paper (down)

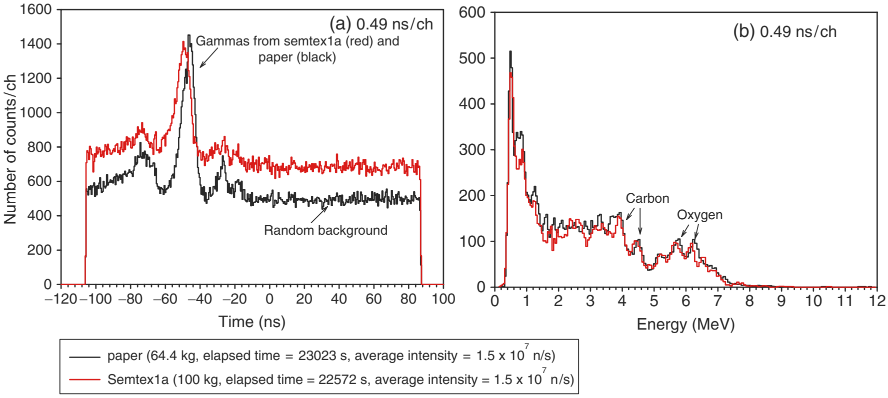


Fig. 8 The time spectra (left) and the gamma ray spectra (right) of the paper and semtex1a. Target volume = $66 \times 40 \times 40\,\mathrm{cm}^3$, 4×10^8 tagged neutrons, Top detectors

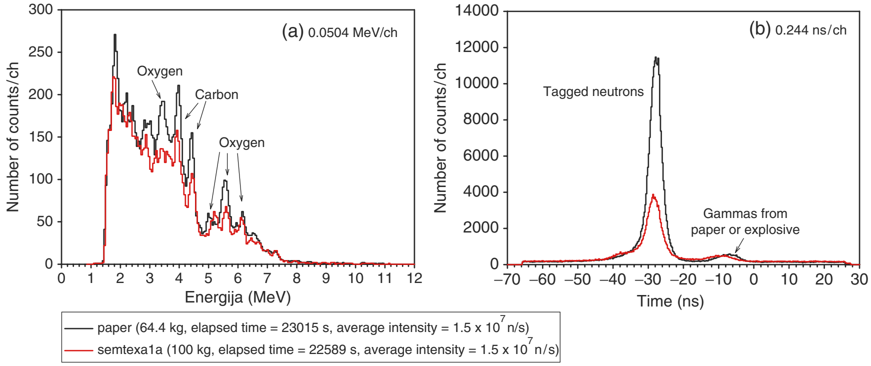


Fig. 9 The time spectra (left) and the gamma ray spectra (right) of the paper and semtex1a. Target volume = $66 \times 40 \times 40\,\mathrm{cm}^3$, 4×10^8 tagged neutrons, Transmission detectors

gamma ray spectrum and the semtex 1 a gamma ray spectrum. The nitrogen peak in the gamma ray spectrum of explosive at 5.1 MeV is overlapped with the second escape peak from the 6.1 MeV oxygen line while the peak at the 2.3 MeV is not recognizable at all. Similar spectra for the transmission detectors were shown on the Fig. 9.

By measuring the C/O ratio we are not in possibility to discriminate the explosive Semtex 1 a from the paper. Besides the carbon "C" and oxygen "O", one more parameter is needed for the successful identification of the target material. Explosives are made mostly from the carbon, oxygen, nitrogen and hydrogen. Nitrogen is poorly seen in our gamma ray spectrum of the Semtex1a and hydrogen could be found only in the random background. Some explosives do not contain nitrogen et all (e.g. TATP). Explosives usually have bigger density then other common materials with the similar chemical composition [2]. Total number of the tagged neutrons "N" depend on the density [3]. So, density is found as a third parameter. By using the triangular diagram the way was found to separate the signature of the Semtex1a from that of the paper. The triangular diagram coordinates are the number of counts in carbon peak, the number of counts in oxygen peak and the number of counts in transmitted neutron peak.

$$Ro* = \frac{\left(\frac{N}{\bar{N}}\right)^2}{\left(\frac{N}{\bar{N}}\right)^2 + \left(\frac{C}{\bar{C}}\right)^2 + \left(\frac{O}{\bar{O}}\right)^2}$$

$$C* = \frac{\left(\frac{C}{\bar{C}}\right)^2}{\left(\frac{N}{\bar{N}}\right)^2 + \left(\frac{C}{\bar{C}}\right)^2 + \left(\frac{O}{\bar{O}}\right)^2}$$

$$O* = \frac{\left(\frac{O}{\bar{O}}\right)^2}{\left(\frac{N}{\bar{N}}\right)^2 + \left(\frac{C}{\bar{C}}\right)^2 + \left(\frac{O}{\bar{O}}\right)^2} \tag{1}$$

$\bar{N}$, $\bar{O}$ and $\bar{C}$ are average values, "C" is the sum of the 4.4 MeV main peak and his first escape, "O" is the sum of the 6.1 MeV peak, his first and second escape. Figure 10 show the triangle diagram. Vertical axes in triangle diagram is ρ^* and horizontal axes is 0.57735 (1 + C* – O*). Bidirectional errors were found using the propagation of errors formula.

Next will be discuss the results made with the experimental arrangement 2. Figures 11 and 12 show the gamma ray spectra of the sea stone with mass 3.25 kg and volume 1,250 cm^3, sea sediment (sample 171), sea sediment (sample 173) and the sea sediment (sample 47) respectively. All sediments were housed inside the metallic box. They were collected from the different locations inside the Adriatic sea. Total number of the tagged neutrons in all measurements was 5.25×10^7, elapsed time vary from 1,872 to 2,442 s depending on the neutron beam intensity which vary in the range $(0.5 - 0.7) \times 10^7$ n/s. The measured spectra are characterised by lines corresponding to the chemical elements carbon, oxygen, calcium and silicon. Almost all calcium line are overlapping with the oxygen lines with the exception of 0.755 MeV line. It was noticed that there are two type of sediments:

(a) Sediments which contain silicon, carbon and oxygen
(b) Sediments which contain calcium, carbon and oxygen

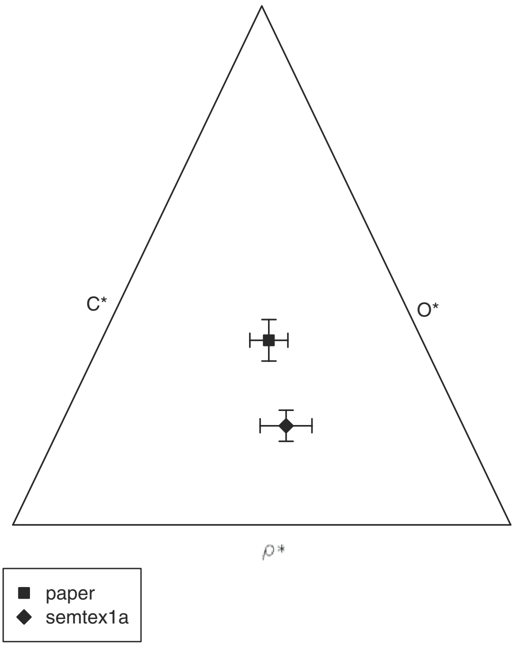


Fig. 10 Triangle diagram

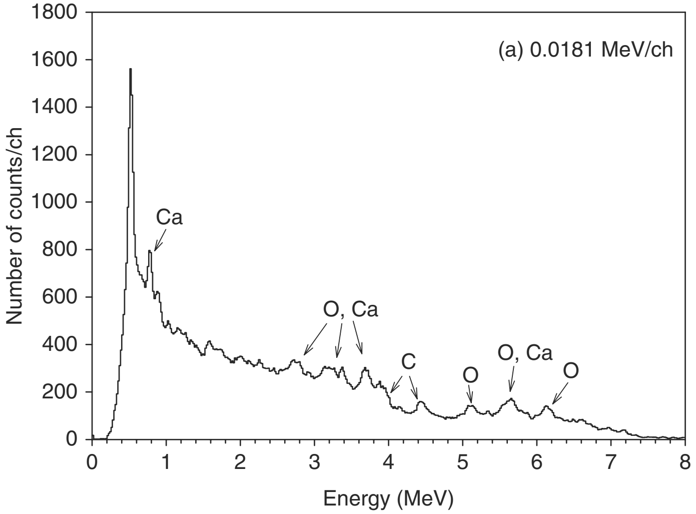


Fig. 11 Gamma ray spectrum of the sea stone with mass 3.25 kg and volume 1,250 cm^3 (up) and the gamma ray spectrum of the sea sediment (sample 171) in the metalic box (down)

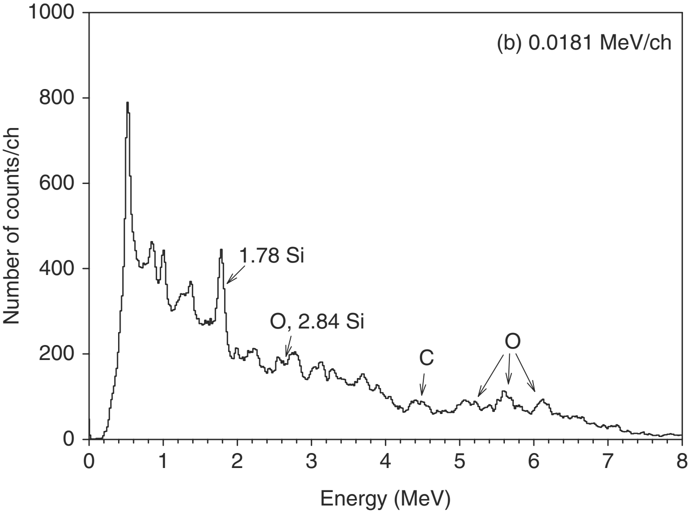


Fig. 11 (continued)

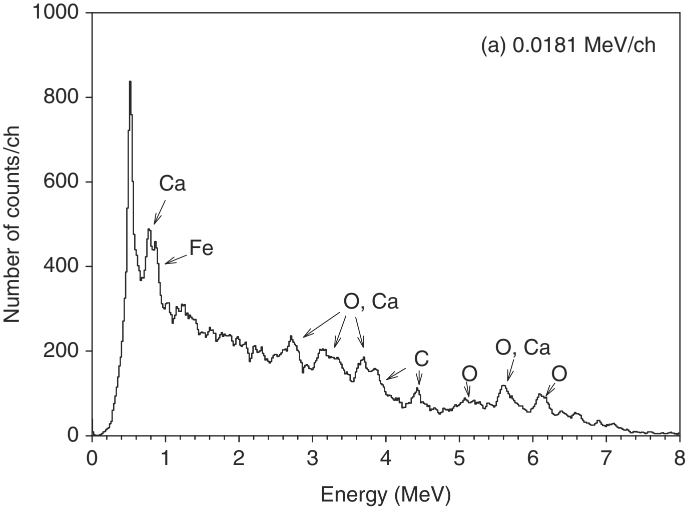
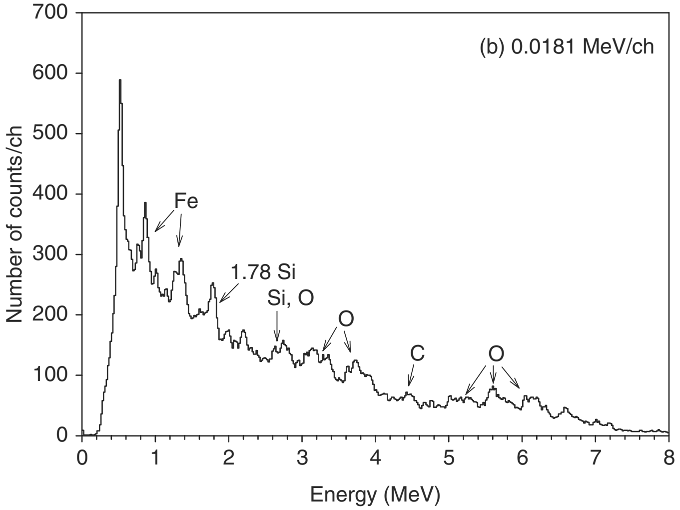


Fig. 12 Gamma ray spectrum of the sea sediments #173 (up) and of the sea sediments #47 (down)



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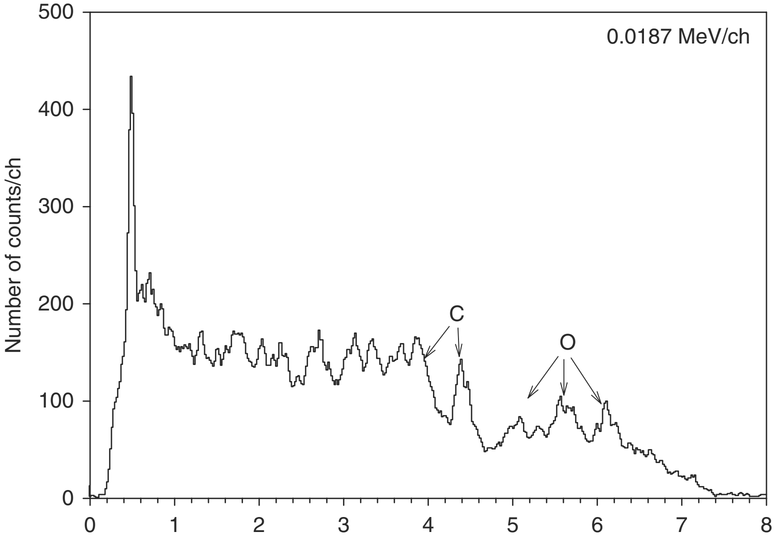

Fig. 13 Gamma ray spectrum from the sample of explosive PETN

Figure 13 show the gamma ray spectrum of the explosive PETN (chemical formula $C_5H_8N_4O_{12}$) with the mass 2.5 kg. The measured spectrum is characterised with the carbon and oxygen lines, but not with the nitrogen one.

4 Conclusions

Semtex1a and paper are well separated in the triangle diagram. Some recent measurements show that this is the case even in iron matrix [3], or in organic matrix [4], if the matrix density is around 0.2 g/cm^3.

Both kind of sediments could be discriminate from the explosive PETN by the silicon line 1.78 MeV or by the calcium line at 0.755 MeV which are not present in the gamma ray spectrum of explosive. Whole spectrum signature also could be used for the identification of the materials investigated.

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Detection of Liquid Explosives and/or Flammable Liquids by Different Techniques

G. Bunte, W. Schweikert, J. Deimling, F. Schnürer and H. Krause

Abstract Not least due to recent terrorist attempts to attack aviation traffic or mass transportation systems, e.g. in London in July 2005 or in August 2006, the secure and sensitive detection of liquid explosives is of major concern. Current presentation will deal with chemical and physical properties of possible liquid explosives used for military or civil applications or misused in improvised explosive devices. Besides a short look at state-of-the-art explosives detection techniques, this presentation addresses e.g. Raman spectroscopic and gas chromatographic measurements of typical liquid explosives and flammables. General challenges for improved detection techniques and the applicability of the addressed analytical techniques will be discussed in relation to different detection scenarios.

Keywords Explosives detection, liquid explosives, ANFO, SPMI, RAMAN, stand-off detection

1 Introduction

Unfortunately, terrorist attacks using liquid explosives have a long tradition/history. Some examples are the bombings of the World Trade Center in New York in 1993 and that of the Oklahoma City Federal Building in 1995. For both attacks vehicle bombs with ANFO (ammonium nitrate with fuel oil) were used. Recent examples are the mentioned attempts in UK where terrorists wanted to use liquid explosives for attacking aviation security. Luckily these plans have been prevented. Another example is that recently in Germany in September 2007, where the BKA found twelve barrels with hydrogen peroxide, H_2O_2, which terrorists intended to use for their attacks.

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Objective of the presented paper is to describe typical liquid explosives and their properties, the threats posed by them, state-of-the-art and challenges of suitable detection techniques as well as to give an overview about current research activities and results at ICT related to detection of liquid explosives and flammables.

2 Typical Liquid Explosives and Flammables

The whole pallet of liquid explosives is big. Starting from explosives such as nitroglycerine, nitromethane or nitroglycols and other nitrate esters, which are liquids at room temperature, there are a lot of pumpable slurries/mixtures based on ammonium nitrate or potassium nitrate with i.e. organic solvents, gasoline, fuel oils or kerosine. The nitrate-based slurries normally serve as borehole explosives or blasting agents i.e. for tunnel construction works and need a booster to explode. Another example mainly used for military applications is the use of mixtures of i.e. dimethylhydrazine with nitric acid (Astrolites), which are mixed shortly before their use due the high sensitivity of the mixtures. Looking at the sensibility especially nitroglycerine is very sensitive to impact and friction so that it is normally only used if gelled with nitrocellulose.

Flammable liquids such as ethanol, methanol, acetone or nitromethane are solvents with very low flash points. They are easily inflammable by an ignition source such as a candle, fire or even an electrostatic spark, transported by a human. These solvents commonly are used by terrorists in combination with fertilizers such as ammonium nitrate or potassium nitrate. A special solvent is acetone, which is one of the educts needed for the synthesis of TATP. The latter is one of the mostly used substances used for homemade improvised explosives devices, mainly applied in the Near East and Europe. Another precursor for TATP is hydrogen peroxide, a further substance, which everybody can buy in commercial stores in different forms, concentrations and/or products.

3 State-of-the-Art Detection Techniques and Challenges

Main threats posed by terrorist attacks and related challenges are the use and hopefully in-time detection of home-made explosive devices based on TATP including the secure and fast detection of concealed precursor substances, H_2O_2 and acetone as well as the substance itself. Furthermore, the secure detection techniques of concealed flammables and fuel oils used together with i.e. nitrate salts are of major concern.

State-of-the-art explosives detection techniques used e.g. in airport portal systems or swapping technologies (Imaging by X-ray, TeraHz-, MMwave, NQR, neutron activation techniques, electronic noses, IMS) are cost intensive equipments. Under test status are also so-called handheld electronic noses. Disadvantage of the IMS-technique is the necessity of high investment costs for the instruments and the use of vapour detection or thermal desorption of sampled particulates by special swaps. X-ray techniques mainly detect metal-containments instead of the explosive substance itself. Concerning the neutron activation techniques as well as for NQR very high investment costs for the instrumentation are compulsory while today only one or two portable systems are commercially available. Moreover, neutron activation and NQR techniques have typically very high detection limits of several grams (e.g. 50–100 g of explosives). TeraHz- and MMwave techniques are new techniques currently under research status. Concealed liquids such as acetone and hydrogen peroxide are not easily to detect due to the fact, that they have comparable densities and/or elemental compositions like typical normal organic products or water. Explosive detectors based on so-called electronic noses are relatively expensive and need a high air-volume for trapping and enrichment of the targeted analyt in order to realize their limits of detection (ppb-range). Current electronic noses are unable to truly identify an explosive substance but use suitable pattern recognition algorithms for the detection and need a person to be driven. If used in an unknown, contaminated atmosphere the calibrated patterns may be disturbed yielding to positive or negative false alarm rates.

Major challenges and requirements for the development of new, inexpensive detection techniques should aim at capabilities of detecting explosives and flammables from secure long distances (stand-off detection) with low detection limits and/or portal independent systems which provide acceptable false/positive rates. A potential lies in the combination of independent methods (orthogonal systems) presumably with proper response on specific explosives/components and a wide application for different explosives and/or flammables. Methods should have a fast response and not be hazardous for people. Most challenging scenarios for the methods are the true stand-off detection of possible threats, the surveillance of large areas and mass transport systems (airports, undergrounds, …), the rapid inspection of passengers, luggage, postal deliveries as well as the surveillance of goods traffic (containers, storage rooms, lockers, …).

4 Current Work at ICT Concerning Improved Detection Techniques

ICT is working on low-cost sensors based on so-called molecularly imprinted polymers, MIPs. MIPs are synthetic, mimic polymers with substance specific recognition properties known from enzymes. Current work relates to particulate adsorbent materials as well as sensor coating materials combinable with low-cost sensor platforms such as quartz crystal microbalances, surface acoustic wave sensors or field effect transistors. Preliminary results showed that some distinct acrylate-based polymers are suitable for an enhanced enrichment/adsorption capability of explosives such as TNT and DNT, showing nearly no cross-sensitivity for both target substances. Further work will concern MIPs selective i.e. for TATP. MIPs look promising for selective low-cost sensors being capable of self-reporting the detected, targeted explosive components in order to achieve an inexpensive surveillance of traffics goods (containers) or large areas with networked low-cost sensors.

Together with other Fraunhofer institutes, ICT is engaged in the so-called initiative OFDEX, where we are working on new optical stand-off detection methods for the unnoticeable detection of suicide bombers and car bombs from distances greater than 10 or 100 m. Studied/envisaged explosives are TATP, TNT, DMNB (a tagging material for high explosives) and ANFO. Tested/applied techniques include absorption spectroscopy with adjustable MIR lasers, UV photo-fragmentation with following NOx detection, laser emission spectroscopy as well as laser desorption from surfaces with following detection. Preliminary results look promising. Expected outcome of the ongoing project will be small scale stand-off detection devices (>10 m).

5 RAMAN Spectroscopy

RAMAN spectroscopy is an analytical technique potentially providing stand-off detection capabilities. Nevertheless, the common used NdYAG-lasers are not eye-safe whereby the application scenarios of this technique are limited. RAMAN spectroscopy is widely used for the analysis of organic reactions in water because water does not interact with the RAMAN light. Most of the energy put into the samples is scattered by the molecules, known as the Rayleigh scattering. The interaction of the RAMAN light with the molecules leads to a shift of the laser light which is, similar to infrared spectroscopy, expressed as wavenumbers, per centimeter. If the molecules are thermally excited, the energy coming out is higher than the energy which was put into the samples, known as Anti-Stokes scattering. More often used is the Stokes scattering for studying the structure of substances. In comparison to infrared spectroscopy, where molecules show IR active bands if the IR light leads to a change of the dipole moment of a bond, molecules show RAMAN active bands if the laser stimulates a change of the polarisability of the bonds. Theoretically, especially symmetrical bonds such as –O-O– and –C-C– should show strong RAMAN bands. Instead of this, strong IR active functional groups such as –OH or –CO should result to little RAMAN band intensities.

After the prevented attacks on London's aviation transport systems in July 2006, there was a high demand for fast, reliable and inexpensive analysis techniques capable of detecting/verifying the liquids passengers are allowed to take into an airplane. Since then passengers are permitted only to carry with them a transparent sack with a volume of 1 l, including small shampoo bottles, cosmetica and others. This still means that a potential threat remains.

At ICT we therefore analysed the Stokes RAMAN shift characteristics of several different liquid explosives, flammable solvents as well as hydrogen peroxide. Shifts were measured in the range between 60–3,400 cm^{-1}, due to filters the effective range was >500 cm^{-1}. We used a laboratory RAMAN spectrometer from Bruker FT-RAMAN RFS 100/S for the measurements (Fig. 1), which is equipped with a

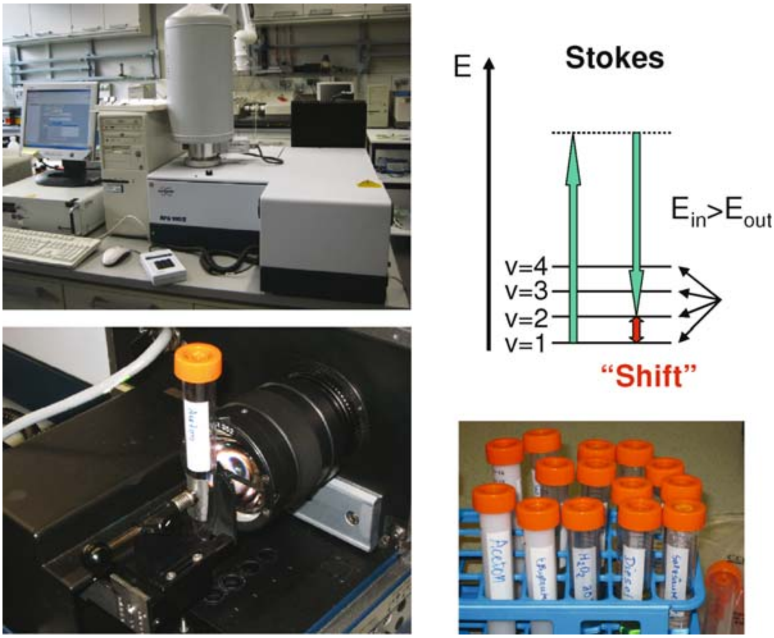

Fig. 1 Photos of the used RAMAN spectrometer, the used measurement technique (Stokes shifts) and the sample vials

NdYAG laser working at 1,060 nm, typically with a power of 500 mW and a sample cross-section of about 0.5–1 mm^2. In order to test which sample container materials are transparent for the RAMAN light, we purchased special sampling vials made of glass, PET and PE, representing the main types of commercial packaging materials. (For metal containers RAMAN is not usable.)

Figures 2–4 exemplarily show the spectra measured in PET vials. Differently to water hydrogen peroxides (Fig. 2) shows a very strong RAMAN shift at about 850 cm^{-1}. Moreover, the intensity of the mentioned band can be used to distinguish between different concentrated solutions. In principle, the content of H_2O_2 in water is detectable if a calibration curve is measured. Spectra using PE and glass sample vials lead to comparable results.

Figure 3 shows the detected RAMAN shifts for acetone, ethanol, ethylacetate and nitro-methane. All four solvents show characteristic Stokes shifts which enable their identification through PET containments. In the case of acetone and ethylacetate the strongest bands are detected in the region of CH valence absorption bands around 2,800 and 3,000 cm^{-1}. For ethanol and nitromethan the most intensive bands are registered in the deformation band region.

Also diesel fuel and ammonium nitrate show very characteristic RAMAN spectra enabeling their identification and detection through PET sample vials (see Fig. 4).

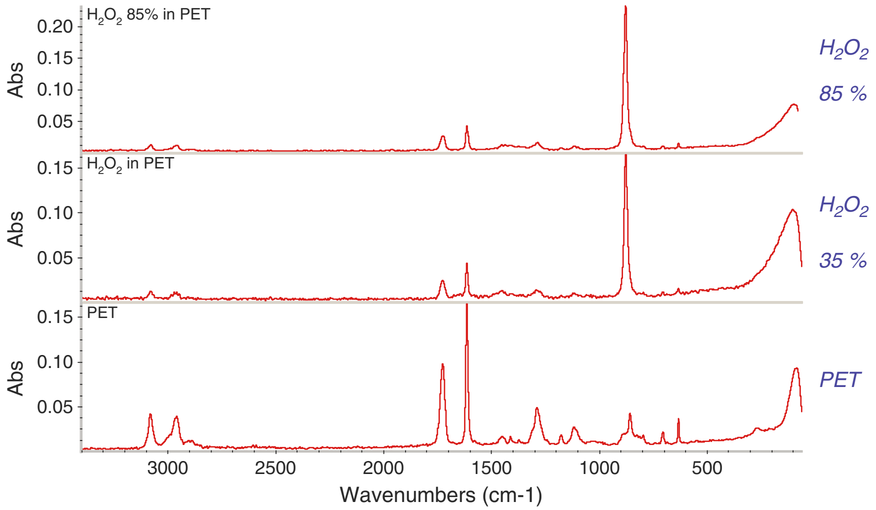

Fig. 2 RAMAN spectra of hydrogen peroxide solutions with different concentration as well as the spectra of the sample containment, PET

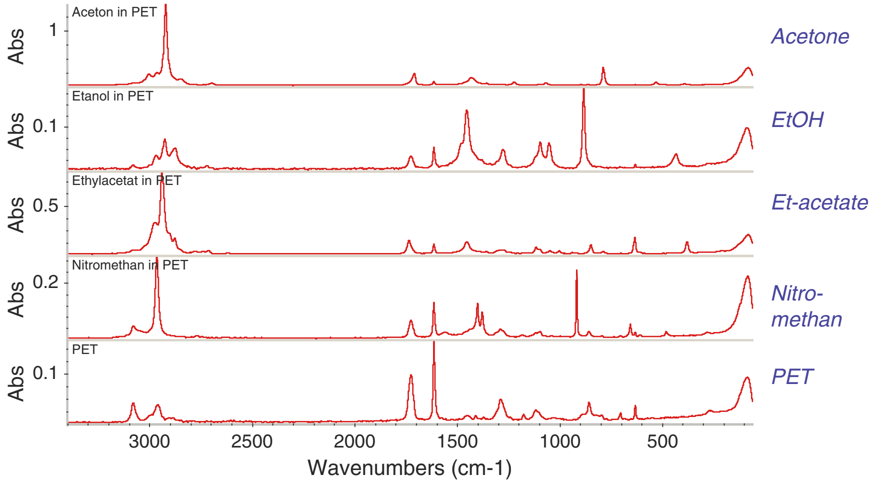

Fig. 3 RAMAN spectra of different liquid explosives/ingredients and flammable solvents as well as the spectra of the sample containment, PET

Not shown in figures are the measurements through glass and PE sampling vials which in principle yielded to the same typical Stokes shifts as for PET vials.

Conclusions from the measured results so far are the following. The RAMAN technique is a suitable method for identifying and detecting a huge range of addressed liquid explosives and flammables. Measurements are possible through

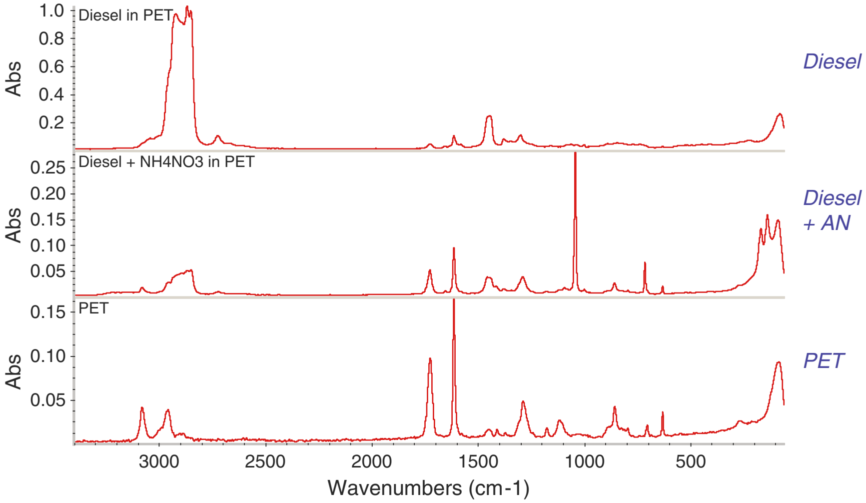

Fig. 4 RAMAN spectra of diesel and a typical ANFO mixture, diesel plus ammonium nitrate, as well as the spectra of the sample containment, PET

glass and plastic containers (PET and PE). In the market there are already some very new portable/handheld RAMAN instruments currently being tested by the aviation security administrations. Drawback for i.e. a wide application at entrance check points is the quite high price (30–40 k€). Nevertheless the technique is suitable for checking concealed liquid explosives and flammables of interest for terrorists.

Challenges for the future are the development of miniaturized, low priced components (lasers, detectors) in order to enable i.e. a more comprehensive surveillance of transported goods or the control of liquids at entrances of public buildings and areas. Potentially the RAMAN techniques also could provide stand-off measurement capabilities, if eye-safe lasers are developed. Future work at ICT will concentrate on achieving extended databases of chemical products, beverages, cosmetics etc and measuring through coloured, dye-containing plastic materials. Together with other research institutes and companies we are looking for new concepts for miniaturized instruments.

6 Solid Phase Micro Extraction, SPME for the Detection of Flammables

A promising technique for the fast detection of flammables and highly volatile liquid explosives such as nitromethane or nitroglycerine is the so called solid phase micro extraction (SPME).

The SPME technique uses micro fibres which are coated with GC-specific separation phases such as polydimethylsiloxane and/or particulate adsorbents such as carboxen or divinyl-benzene (Fig. 5). The fibres are used in the head-space phase above a solid or liquid sample to absorb or adsorb the emittable substances. For liquid samples also a direct immersion of the fibre into the liquid phase is possible. The analysis is done by thermal desorption of the adsorbed components directly into the GC-injector followed by a common GC separation using a mass selective detector for identifying unknown components. Due to the fact that no diluting organic solvents are used the extraction by SPME yields to a strong/effective enrichment of emitted substances. Laboratory measurements were made with a HP GC with mass selective detector (MSD) with a temperature-programmable injector (OPTIC 3) combined with an automatic SPME-sampling device – FOCUS autosampler with head-space oven and needle heater from VARIAN.

Figure 6 exemplarily shows a total ion chromatogram of different analysed solvents. Included into the figure are the measured retention times if analysed on a lab GC with a column of 25 m length. Mentioned in brackets are the retention times for cyclohexane and iso-octane, which were measured in other GC runs.

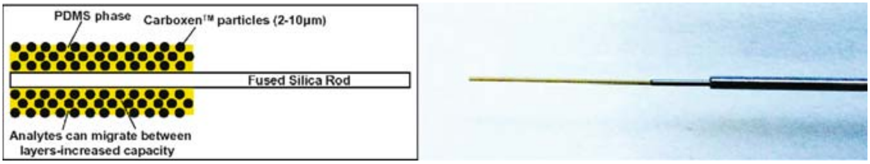

Fig. 5 Schematic view (source: Supelco) and photo of a SPME fibre (CAR/PDMS)

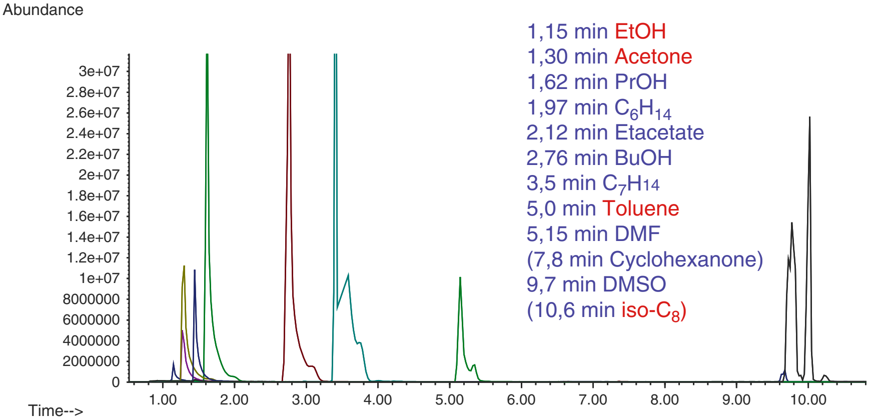

Fig. 6 Total ion chromatogram of different solvents sampled with a SPME fibre CAR/PDMS) and analysed with a GC-MSD

As shown in Fig. 6, flammable solvents such as ethanol, acetone, toluene and iso-octane, which are often used by terrorists, can be measured in 10 min. From application journals of Supelco, we also know that traces of typical fuel oils used in ANFO-based bombs such as kerosene, BTX, gasoline and diesel fuel are detectable by the SPME technique in 10 to 15 minutes. Shorter GC-columns or i.e. micro capillary columns (MCCs) will offer the opportunity of much shorter detection times.

Experiences at ICT with this technique were made in the field of enrichment and analysis of nitro- and aminoaromatic components such as NT, DNT, TNT and their degradation products directly measured in water as well as for the detection of residual solvents in solid explosives such as RDX and HMX. Achievable detection limits were measured to be in the ppb to ppt range. Moreover we successfully used this technique for the analysis of debris to detect possibly used accelerants. So the technique could be used for forensic analysis as well. Measurements so far were made with a laboratory type gaschromatograph, nevertheless the SPME can be used together with mobile GCs in the field. Combining SPME with a mobile FAST-GC or an IMS would provide typical measurement times of 1–2 min. In principle this techniques has also potential for using it as a fast screening test “in the field”, in order to screen suspect transported goods, containments and luggage for detecting flammable liquids.

7 General Conclusions

A lot of state-of-the-art techniques especially for aviation security related scenarios already exist. Improvements are needed for lower false alarm rates and faster detection. Improvised explosives/liquid explosives and flammables are not “catchable” with one universal technique. The suitability of a distinct detection technique is always related to the envisaged scenarios. There is a need for improvements/new developments to achieve increased security of the citizens under selected scenarios. Moreover especially miniaturized, cheap detectors and techniques are highly required as well as suitable stand-off detection capabilities.

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Hilbert Spectroscopy of Liquids for Security Screening

Y. Divin, U. Poppe and K. Urban

Abstract Fast and reliable identification of bottled liquids is of great importance for security screening and, in principle, it can be done by electromagnetic measurements of dielectric permittivity functions of the liquids of concern in a frequency range, where bottles are transparent and liquids have specific dispersions. However, this frequency range is rather extended, from a few GHz to a few THz, and cannot be covered by any single conventional spectroscopy. A concept of liquid identifier, which is based on our new broadband Hilbert spectroscopy and high-T_c Josephson detectors, is presented. The identifier with a Josephson detector in a Stirling cooler will operate at the frequency range from 5 to 1000 GHz with scanning time of around 10 ms and identification time less than 1 second. The first proof-of-principle measurements of reflection spectra from various bottled liquids in the range 40–400 GHz with total scanning time of 1 second have been carried out by Hilbert spectroscopy.

Keywords Spectroscopy, liquids, Hilbert transforms, Josephson effect, security

1 Introduction

After recently uncovered terrorist plots involving the mid-flight detonation of liquid explosives, it became clear that an additional screening of passenger's luggage is required. The goal of this screening should be to find and identify particular sorts of liquids, which might be dangerous themselves or could be used as components for fabrication of explosives on board, e.g. hydrogen peroxide and solvents. The screening should be so fast and specific that it should not disturb a normal flow of passengers and operate with low rate of false alarms.

Among various discussed ways of explosive detection, the techniques using electromagnetic radiation (i.e. microwave and terahertz imaging and spectroscopy), are considered as having a great potential, and intensive research activities

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are recommended in this field by experts of US National Academy [1]. An unambiguous identification of liquids could be realized by electromagnetic measurements of their dielectric permittivity functions $\varepsilon(f)$ [2] in a broad frequency range starting from microwave to terahertz ranges, where the liquids of concern have specific dispersions, but this range cannot be covered by any single conventional technique [3].

The spectral range of a new type of spectroscopy, Hilbert spectroscopy [4, 5] lies in the range of frequencies, where the liquids of concern have characteristic signatures in their electromagnetic response. Total scanning times amount to only several milliseconds, and demonstrators of Hilbert spectrometers were developed to show the potential of the technique in spectral characterization of various terahertz sources and substances including vapors of liquids such as methanol and acetone. Here, we present our approach to liquid identification, based on Hilbert spectroscopy, first proof-of-principle experiments and planned activity in developing a demonstrator.

2 Dielectric Function of Liquids

From the point of view of electromagnetic theory, the electric displacement-field response of any substance to a rapidly varying electrical field is defined by a complex dielectric permittivity

$$\varepsilon(f) = \varepsilon_1(f) + i \cdot \varepsilon_2(f), \tag{1}$$

which is determined by the internal dynamics of the molecules [2]. Therefore a substance can in principle be identified by measuring the dielectric function $\varepsilon(f)$ of this substance over a wide range of frequency f and comparing it with available reference data.

The internal dynamics of liquids can be considered to a first approximation as an alignment of dipoles by the local electric field followed by a Debye relaxation process [6]. In this case the dielectric permittivity function can be written as

$$\varepsilon(f) = \varepsilon_\infty + (\varepsilon_0 - \varepsilon_\infty)/(1 + i2\pi f\tau), \tag{2}$$

where ε_∞ is the “infinite frequency permittivity”, ε_0 is the static permittivity (dielectric constant) and τ is the characteristic relaxation time.

The real and imaginary parts of the dielectric functions of some liquids are shown in Fig. 1. The dielectric relaxation parameters, ε_∞, ε_0, and τ of various liquids, like acetone, water, ethanol etc. were calculated from $\varepsilon(f_k)$-values, measured at some discrete frequency lines f_k at the microwave range and summarized in reference [6]. The data for hydrogen peroxide are calculated from a few points presented in [7, 8] with the assumption that the relaxation time for this liquid is equal to that of water. The values of the main relaxation times τ_1 of different liquids spread over

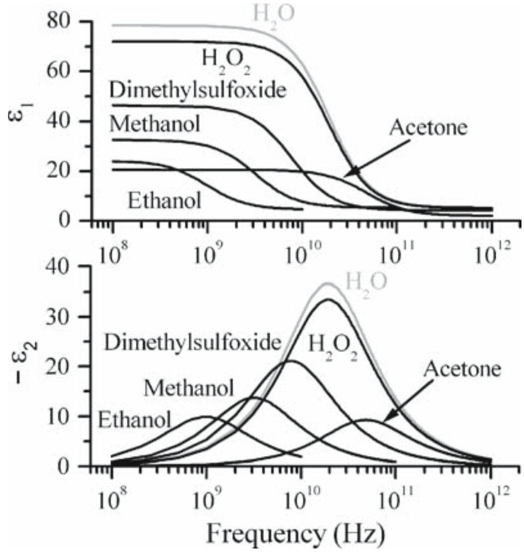

Fig. 1 Real and imaginary parts of the dielectric functions of various liquids at 25 °C, calculated in a Debye approximation with the parameters. (Taken from Chantry [3] Divin et al. [4, 5])

two orders of magnitude, e.g. 3.3 ps for *acetone*, 8.3 ps for *water* and 163 ps for *ethanol*. The characteristic frequency $f_{ch} = 1/2\pi\tau$, where the imaginary part of dielectric function $\varepsilon_2(f)$ exhibits a maximum, ranges from 1 GHz for ethanol to 50 GHz for acetone. To cover a specific dispersion region in $\varepsilon(f)$ only for the main relaxation time τ_1, a frequency dynamic range of more than one order of magnitude around the characteristic frequency f_{ch} is required [6].

Additionally, more dynamic relaxation processes might be involved in liquids, like in ethanol with $\tau_2 = 9$ ps and $\tau_3 = 1.8$ ps [6] or in water with $\tau_2 = 0.17$ ps [9]. Therefore, the frequency range of the dielectric spectroscopy of liquids must be expanded further to hundreds of gigahertz and even to the terahertz (THz) range. It is emphasized in [6] that insufficient frequency coverage is responsible for many unsatisfactory data for dielectric functions of liquids in the literature and that an expansion of the frequency range up to the terahertz-range is required for unambiguous identification and property measurements.

Also, due to the interplay of orientational, intramolecular, kinetic, H bonding, diffusional and migrational modes, the dielectric function $\varepsilon(f)$ of real liquids is more complicated [3, 6, 9], than it is assumed in a simple Debye model. From one side, this circumstance is favourable for detection purposes since the frequency dependence of $\varepsilon(f)$ is more specific to particular liquid. From the other side, it raises an important question of availability of *reliable and precise data base of dielectric functions for pure liquids* in a broadband frequency range from microwave range to terahertz frequencies, which still should be answered.

Liquids of concern, like hydrogen peroxide and solvents, might be used not in a pure form, but as commercially available mixtures with other liquids. For example, hydrogen peroxide-water mixtures are well known and are of great concern. These mixtures present a great challenge for electromagnetic identification. The static dielectric constant ε_0 of hydrogen peroxide-water mixtures as a function of the H_2O_2 concentration has a maximum at the 55 wt% of H_2O_2 and goes down with a further increase of the H_2O_2 content. It reaches the ε_0-values around 80, which is the values of the dielectric constant of pure water at 20 °C, at the H_2O_2 concentrations of around 75 wt% [10].

So, the electromagnetic measurements of static dielectric constant ε_0 alone might not distinguish between harmless water and dangerous hydrogen peroxide-water mixture. We think that application of the spectroscopic techniques covering the whole frequency range of dispersions might give a chance for an identification of liquids and mixtures.

Among the different types of spectroscopic measurements (transmission or reflection), the reflection measurements are indicated as better suited for the identification of highly absorbing liquids [9, 11]. We have calculated the spectral dependences of the reflectance for the same some liquids, shown in Fig. 1 and the results are presented in Fig. 2. The reflectance spectra, when compared with spectra of the real part of the dielectric function (Fig. 1, above), show a tendency for shifting the characteristic frequencies, where reflectance goes down for each liquid, to higher frequencies. This circumstance emphasizes an importance of the measurements starting from microwave range to terahertz frequencies for reliable identification of liquids.

Concerning an identification of liquids, which are held in bottles, we should also first consider the restrictions, implied by a spectral range of transparency of

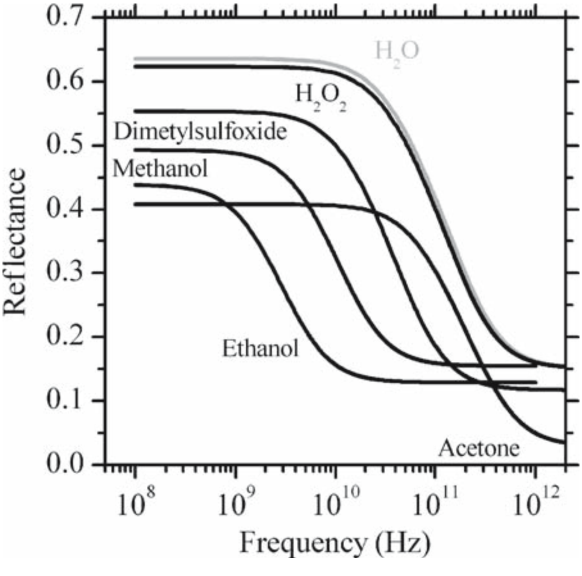

Fig. 2 Reflectance spectra for various liquids, calculated from dielectric functions, shown in Fig. 1

commercial bottles, possible interference in the walls of bottles, additional scattering from the curved surfaces of the bottle's walls, etc. Plastic bottles, made of polyethylene (PE) with the thickness of 1 mm, are quite transparent at the frequency range up to 2 THz and bottles, made of polyethylene terephthalate (PET) with the thickness of 0.5 mm, demonstrated the decrease of the transparency up to 0.5 with an increase of frequency up to 0.5 THz [12]. Glasses of various kinds demonstrate an increased absorption with frequency at the terahertz range, e.g. 2 mm-thick BK7 and Pyrex windows might be considered transparent below 0.5 THz, but windows of the same thickness made of quartz and silica are transparent in broader spectral range up to 2.5 THz [13]. Due to the limited spectral range of transparency of some bottles, the spectral measurements of bottled liquids at the terahertz range above 1 THz might be not informative in general case, when a material of the bottle is not known. So, when developing a general approach for an identification of bottled liquids, we should concentrate at the frequency range below 1 THz.

The second consideration comes from fact that vapor above the liquid in the hermetically sealed bottles is available for spectral transmission measurements. Acetone vapors were found to have significant absorption at the terahertz frequency range from 0.3 to 1 THz, where methanol vapors demonstrated only moderate losses [14]. Additional transmission measurements of vapor of liquids might be considered as a second identification channel, which is importance to create a required orthogonality [1] to detection techniques.

3 Conventional Techniques

The techniques to measure the dielectric properties of liquids in the frequency range of up to about 100 GHz have recently been reviewed [15]. The reflection, transmission and resonator microwave techniques at fixed frequencies are the main conventional approaches in the field. However, due to non-monotonous behavior of the permittivity of two-component liquid mixtures with increase of a concentration of one component [10], the measurement of the dielectric function at fixed frequency, or even at a few preset spot frequencies cannot distinguish unambiguously between some solutions and the liquids of concern. This makes such approaches unreliable, and since the safety system must react in both, harmless and safety-relevant cases, this should give rise to a high rate of false alarms.

The situation can be improved, when the dispersion is measured over an extended frequency range (see Figs. 1 and 2). Such spectroscopic approach can in principle be realized employing commercial network analyzers. However, this approach is hampered by the performance range of commercial network analyzers which, from technical reasons, is usually limited to about 50 GHz maximum. Only at the cost of an exponential increase in price and time required for analysis, a maximum frequency of up to 100 GHz is within reach, which, as described above, is still too low even for such "simple" liquids as acetone and water.

To study fast dynamics in liquids, continuous measurements in the terahertz frequency range have been performed using Fourier-transform spectroscopy (FTS) [3] and time-domain spectroscopy (TDS) [9, 11, 12, 16]. Transmission spectra of some inflammable liquids, like benzene and kerosene, stored in conventional beverage plastic bottles, have been measured in the frequency range from 300 GHz to 1.8 THz [12]. Conventional beverage bottles were found to be transparent up to around 1 THz. No transmission was found for water, and some transmission, decreasing with the frequency, was found for liquids, like benzene and kerosene. From this measurements a crude inspection principle was derived following the rule *"If liquids transmit terahertz radiation, it is dangerous, if not - it is water"*. With this criterion in practice, acetone and hydrogen peroxide would clearly escape detection, due to their large absorption in the terahertz range.

An additional characterization of liquids at lower frequencies might improve the identification procedure, but at microwave range it is done by time-consuming point-by-point measurements at discrete frequency lines. We note also, that terahertz TDS, as well as more conventional FTS, involves extended measurement times, typically of a few minutes, since a mechanically driven optical time-delay line is used for gated operation [9, 11].

4 Hilbert Transform Spectroscopy

4.1 Theory

In contrast to the conventional techniques, Hilbert-transform spectroscopy [4, 5] (in the following abbreviated by HTS) represents a fast technique operating at the frequency range of a few gigahertz to a few terahertz, and thus can make possible an unambiguous identification of dangerous substances within short times. The central part of the Hilbert spectrometer is a nanoelectronic device, a Josephson junction [17].

A Josephson junction, when biased at finite voltage V, is actually a voltage-controlled oscillator with a frequency f_j of oscillations, proportional to the voltage, i.e. $f_j = 2eV/h$. If a Josephson junction is irradiated by monochromatic electromagnetic radiation, the Josephson frequencies f_j, due to the nonlinear dynamics of a Josephson junction, are pulled to the frequency f of external radiation, even if the intensity of external radiation is very low. The result of this frequency pulling is a modification of the static $I(V)$ curve of the Josephson junction around the voltage $V = hf/2e$. The difference between the $I(V)$ curve, modified by radiation, and the unmodified $I_0(V)$ curve is considered as a junction response $\Delta I(V) = I(V) - I_0(V)$, which demonstrates such important analytical features as an odd-symmetric resonance around the voltage $V = hf/2e$ and square-law dependence on the amplitude of the signal [17]. These features of the junction response to monochromatic signals are the basis of Hilbert transform spectroscopy [4].

In the case of an arbitrary spectrum $S(f)$, the response function $H(V) \propto \Delta I(V) \cdot I(V) \cdot V$ was found to be proportional to the Hilbert transform of the spectrum $S(f)$ of the incident radiation [4]. Applying an inverse Hilbert transformation to the measured response $H(V)$ the spectrum $S(f)$ can be recovered as

$$S(f) \propto \left(\frac{1}{\pi}\right) P \int_{-\infty}^{\infty} \frac{H(f_j) \cdot df_j}{f_j - f}$$

where $f_j = 2eV/h$ (h is Planck's constant). The principle of Hilbert spectroscopy is similar to that of Fourier-transform spectroscopy or time-domain spectroscopy. The important distinction, however, is that in Hilbert spectroscopy a direct transformation of the spectrum into an electrical signal is achieved by a nanoelectronic device, the Josephson junction, while in Fourier spectroscopy and time-domain spectroscopy this procedure requires a bulky optical-mechanical device, an interferometer or optical delay line, together with a broadband detector.

4.2 High-T_c Josephson Junctions

The Josephson junctions, which are used in Hilbert spectroscopy, should follow an idealized RSJ model (RSJ - resistively shunted junction) [17]. According to the model, an amplitude I_j of the Josephson current does not depend on the Josephson frequency f_j and equal to the critical current I_c. A quasipartical current I_{qp} follows the Ohmic law $I_{qp}(V) = V/R_n$, where R_n is the junction resistance.

To reach the terahertz range, the junctions should have characteristic voltages $V_c = I_c R_n$ of the order of 1 mV. The junctions with these values of the $I_c R_n$ product are currently produced from high-T_c superconducting materials, like $YBaCu_3O_{7-x}$, in the form so-called grain-boundary junctions [18]. A grain boundary in these junctions is forming a quite transparent tunneling barrier, so low-resistance Josephson junctions, required for Hilbert spectroscopy, might be fabricated.

We have fabricated $YBaCu_3O_7$grain-boundary junctions of various types and optimized their performances for application in terahertz detectors and Hilbert spectroscopy [5, 19–22]. A high-resolution microscopy (HREM) image of a plane-view of one of our [001]-tilt $YBaCu_3O_{7-x}$ grain-boundary junctions is shown in Fig. 3 (left). Perfect cation ordering can be seen in the HREM image up to the nearest vicinity of the grain boundary, situated horizontally in the image.

A light-microscope image of a $YBaCu_3O_{7-x}$ Josephson junction, developed for Hilbert spectroscopy, is shown in Fig. 3 (right). Two micrometer-wide $YBaCu_3O_{7-x}$ bridge (in the center of Fig. 3, right) is crossing the $NdGaO_3$ bicrystal substrate boundary, revealed by a polarization contrast. The Ag broadband log-periodic antenna is attached to $YBaCu_3O_{7-x}$ bridge to convert electromagnetic radiation with the frequencies from few gigahertz to few terahertz into ac currents crossing the $YBaCu_3O_{7-x}$ grain-boundary junctions.

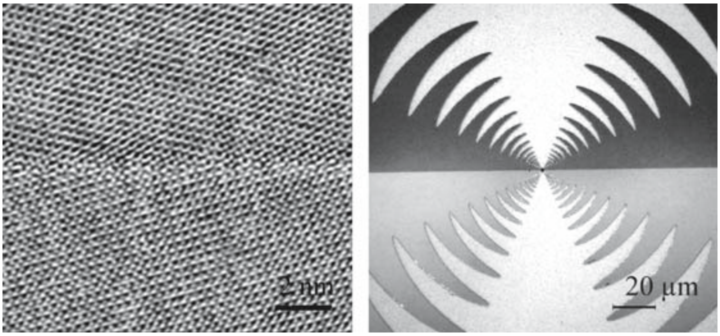

Fig. 3 High-resolution electron microscopy image of a $YBa_2Cu_3O_{7-x}$ bicrystal Josephson junction (left) and micrograph of a 2 μm-wide junction, crossing the bicrystal $NdGaO_3$ boundary, with an integrated broadband Ag antenna (right)

The I_cR_n-values amount to 0.34 mV at 77 K and up to 2 mV at 35 K for [001]-tilt $YBaCu_3O_{7-x}$ grain-boundary junctions [5]. Recently, we have developed the [100]-tilt $YBaCu_3O_{7-x}$ grain-boundary junctions with the record high I_cR_n-values up to 8 mV [20] and we can reach the proximity of the *I(V)*-curves to that of RSJ–model with an accuracy of better than 0.5% at the temperature range 90–50 K in this type of junctions [22].

4.3 Hilbert Spectrometers

Starting from a chip, comprising the $YBaCu_3O_{7-x}$ Josephson junction with the integrated broadband antenna (see Fig. 3 (right), a Josephson detector was fabricated by mounting the chip into a sample holder, attaching electrical contacts and adding a Si hyperhemispherical lens for focusing incoming radiation to the broadband antenna. One of our assembled Josephson detectors, mounted on a temperature-variable cold stage of a liquid-nitrogen cryostat, is shown in Fig. 4 (left).

Various cooling devices were used to maintain the junction temperatures in the range from 35 to 90 K. We have successfully integrated the $YBaCu_3O_{7-x}$ Josephson junctions into a Stirling cryocooler and achieved a stable operation at the temperature range from 35 to 90 K. One of the Hilbert spectrometers with a $YBaCu_3O_{7-x}$ Josephson junction integrated into a Stirling cryocooler was developed [23]. It was quite a time-consuming procedure to arrange an appropriate low level of electromagnetic interferences, magnetic fields, mechanical vibrations and temperature stability, which is required for a successful operation of a Hilbert spectrometer with a Josephson junction, in the case of a Stirling cryocooler, but we have managed to reach all necessary conditions [5, 23].

More simple and cost-efficient cooling device, which we use for fabricating Hilbert spectrometers, is an optical cryostat, filled by liquid nitrogen (LN) with a boiling temperature of 77.3 K at atmospheric pressure. When liquid nitrogen was

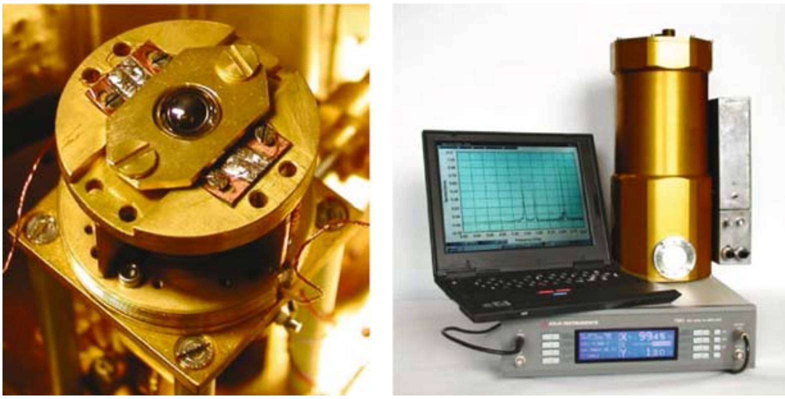

Fig. 4 Josephson detector with a Si hyperhemispherical lens, mounted on the cold stage of a liquid-nitrogen cryostat (left), and one of the demonstrators of Hilbert spectrometers, based on a liquid-nitrogen cryostat (right)

pumped to lower pressures, we were able to reach the junction temperatures as low as 48 K. One of the Hilbert spectrometers, developed by us with a LN cryostat as a cooling device, is shown in Fig. 4 (right). A cryostat with an optical access to the Josephson detector is supplied with low-noise analogue biasing and amplifying electronics, which is placed in a grey box, attached to the cryostat. A commercial lock-in amplifier is used to measure the modulated response ΔI (below cryostat, in Fig. 4, right). The signals, proportional to the response $\Delta I(V)$, the junction voltage V and current I, are digitized by 16 bit, 100 kHz analogue-digital converters and stored in a notebook. A Hilbert spectrometer of this type was used in our proof-of-principle experiments with liquids.

The spectral range of Hilbert spectrometers scales with the I_cR_n-value of the $YBaCu_3O_{7-x}$ Josephson junctions, which increases with a reduction of junction temperature, starting from a critical temperature T_c of around 90 K. For I_cR_n-values of 1.5 mV it reaches terahertz frequencies [5]. We have reached a record high-frequency limit for the ac Josephson effect of 5.2 THz with our high-quality [100]-tilt $YBaCu_3O_{7-x}$ Josephson junctions, which have I_cR_n-values up to 8 mV [21].

When the junction temperature is close to T_c, the spectral range of the ac Josephson effect in the high-T_c Josephson junctions might be right in the range of frequencies, where the liquids of concern have characteristic signatures in their electromagnetic response. Spectral measurements of the responses of a low-resistance [001] $YBaCu_3O_{7-x}$ Josephson junction at two temperatures, close to T_c, are presented in Fig. 5 in a frequency range relevant for the spectroscopy of liquids [24]. At the temperature of 88 K the spectral range, where the odd-symmetric responses $\Delta I(V)$ were observed, is spread from 5 to 100 GHz, and for a temperature of 85 K, we got an extended spectral range from 15 to 1,000 GHz. Optimizing the junction temperature and junction parameters, we can fit the spectral range of Josephson detectors to frequencies from 5 GHz to 5 THz. A spectral resolution $\delta f/f$ of around 10^{-3} was reached for HTS at the terahertz range [25].

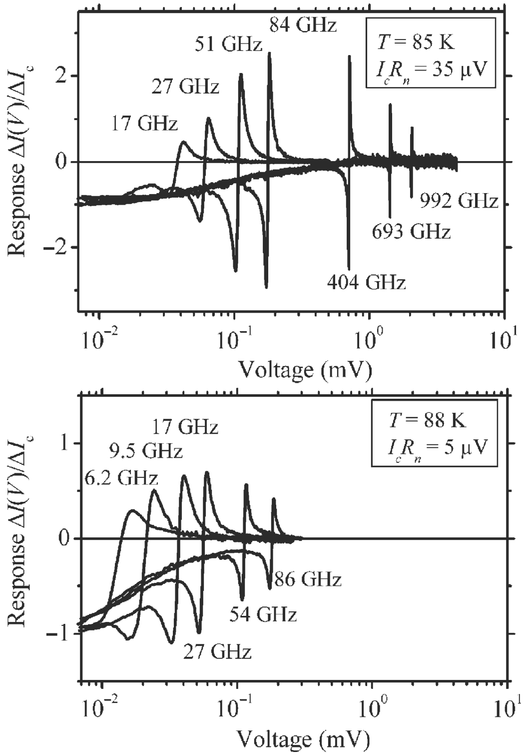

Fig. 5 Two sets of voltage dependences of the responses $\Delta I(V)$ of $YBa_2Cu_3O_{7-x}$ Josephson junction with a normal-state resistance $R_n = 0.5\ \Omega$ to monochromatic radiation with frequencies in the range from 5 GHz to 1 THz. The junction temperatures were equal to 85 K (above) and 88 K (below)

A power dynamic range of more than 50 dB with a noise equivalent power NEP $\approx 10^{-14}$ W/Hz$^{1/2}$ has been demonstrated for Josephson detectors developed by us for HTS [5, 26]. Total scanning times in HTS could be as low as several milliseconds for pulsed sources [27].

The demonstrators of Hilbert spectrometers were developed to show the potential of the technique in spectral characterization of various terahertz sources [5] and gaseous substances [28, 29]. The high-harmonic content of the commercial frequency multiplier was measured by HTS and is shown in Fig. 6. As fabricated, the multiplier was optimized to deliver main power in the range of frequencies 80–110 GHz, which are the sixth harmonics of the microwave pump (15–18 GHz) of the multiplier. This sixth harmonic was selectively suppressed by a polarization attenuator to the level of around -50 dB, and unsuppressed high-frequency part of the radiation was analyzed with HTS. It is clear from Fig. 6, that due to the good sensitivity of HTS, a lot of high-order harmonics could be identified, and, actually, this multiplier can be used as polychromatic source for spectroscopic measurements with HTS. We used this multiplier in our proof-of-principal experiments.

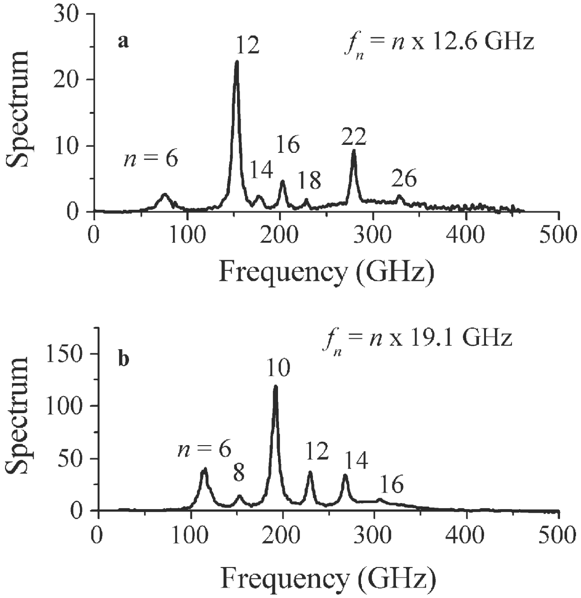

Fig. 6 Emission spectra, measured by Hilbert spectroscopy, from a commercial frequency multiplier, pumped by a microwave oscillator with the frequency f: 12.6 GHz (a) and 19.1 GHz (b). The intensities of the main frequency harmonics with $n = 6$ were selectively suppressed by around 50 dB with the help of a polarisation attenuator

5 Hilbert Spectroscopy of Liquids

5.1 Future Demonstrator of Liquid Identifier

Starting from our previous experience in Hilbert spectroscopy, the schematic of future demonstrator of liquid identifier could be suggested as it is shown in Fig. 7. It can be divided into the following parts: Hilbert spectrometer, consisting of a high-T_c Josephson junction on a Stirling cooler and an analogue electronics, a data acquisition system, a pulsed radiation source, a radiation coupling unit between the source, Hilbert spectrometer and a bottle with the liquid sample, plus a computer with application-oriented software.

The pulsed source delivers a repetitive train of radiation pulses through the radiation coupling unit to a liquid in a bottle. The spectral density of this radiation is indicated as $W(f)$. A train of radiation pulses reflected from the liquid comes to the Hilbert spectrometer with the spectral density of $R(f)\cdot W(f)$, where $R(f)$ is the reflectance of the liquid.

Triggered by a pulsed source, an analogue electronics of the Hilbert spectrometer scans the voltage bias $V(t)$ of the Josephson junction, reaching a new incremental value of the voltage with each trigger pulse. A low-resistance high-T_c Josephson

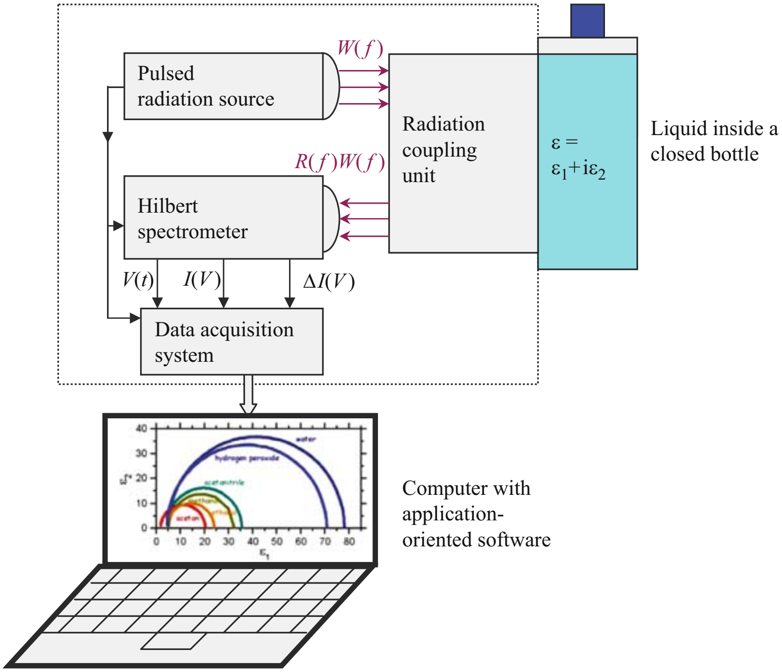

Fig. 7 Schematic of a liquid identifier, based on spectral measurements of reflectance from liquids by Hilbert spectroscopy with a broadband pulsed radiation source

junction is used, which is situated in a cryogen-free Stirling cooler. The resulting modifications in the current $I(V)$ through the Josephson junction and a current response $\Delta I(V)$ of the Josephson junction are amplified by analogue electronics of the Hilbert spectrometer. A set of data $V(t)$, $I(V)$ and $\Delta I(V)$ are digitised by a data acquisition system, triggered from the pulsed radiation source, and stored into the memory of a computer.

With the help of application-oriented software, the measured data $V(t)$, $I(V)$ and $\Delta I(V)$ are first converted to the response function $H(V) \propto \Delta I(V) \cdot I(V) \cdot V$, then to a spectrum of reflected radiation $R(f)$ by Hilbert transformation of $H(V)$ and eventually to the dielectric function $\varepsilon(f) = \varepsilon_1(f) + i\varepsilon_2(f)$.

According to the characterization of various Hilbert spectrometers, made by us, and the requirements, imposed by a substance under study, we may plan to develop a demonstrator of liquid identifier, based on Hilbert spectroscopy, with the following characteristics and main features:

1. Frequency range: 5 GHz–1,000 GHz
2. Scanning time: around 10 ms
3. Identification time: around 1 s
4. Invisible cooling
5. Compact design: housing within a A3 size box

5.2 First Proof-of-Principle Experiments

We have managed to carry out the first proof-of-principle measurements of the reflection from the bottled liquids with the help of an available Hilbert spectrometer. A photo of the set-up, used in these measurements, is shown in Fig. 8. In general, it follows the schematic, presented in Fig. 7. In the center of Fig. 8 one can find a plastic bottle, filled with some optically-transparent liquid. The Hilbert spectrometer, similar to the one shown in Fig. 4 (right), could be found on the left side of Fig. 8. The spectrometer was based on the [100]-tilt $YBaCu_3O_{7-x}$ Josephson junction, which was kept inside LN cryostat at the temperature of 78 K. The resistance of the junction was close to 10 Ω.

A commercial frequency multiplier, mounted on adjustable mechanical stages, with the output, selectively suppressed by a polarization attenuator, can be found on the right side of Fig. 8. The output of the multiplier, depending on the frequency of microwave pump and the observation angle from the optical axis of the multiplier output, consists of several lines of high-order harmonics of the microwave pump oscillator. The high-harmonic content of this multiplier was separately measured by HTS and some spectra are shown in Fig. 6. It was observed that high-order frequency harmonics are spread in the high-order spatial modes and, to maximize the high-frequency content, the output horn of the multiplier was directed in a few degrees tilted direction with respect to the optical axis of the system.

The polychromatic radiation from the multiplier was focused by a gold-plated elliptical mirror on the bottle with the liquid, and the radiation, reflected from the bottle, was focused on the Josephson detector with the help of the second elliptical mirror. The output of the microwave oscillator, pumping the multiplier, was modulated by a PIN switch with a modulation frequency of 50 kHz. The Hilbert spectrometer with the Josephson detector was operated in a voltage bias mode. The junction voltage V was scanned linearly in time with a total scanning time of 1 s. The voltage dependences of the response $\Delta I(V)$ and current $I(V)$ were detected,

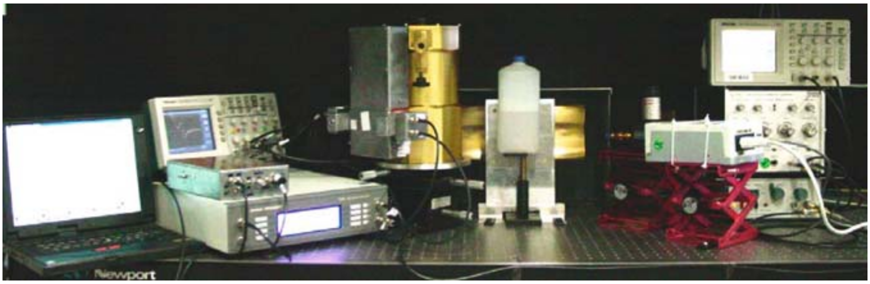

Fig. 8 Photo of the experimental set-up for spectral reflectance measurements of bottled liquids. The Hilbert spectrometer (left side of the photo) was used for spectral measurements of polychromatic radiation from a commercial frequency multiplier with a polarization attenuator (right side), reflected from bottled liquid (center)

amplified, shown on the oscilloscope, digitized by a PCMCIA acquisition card, stored and processed in the PC notebook.

The calibration of the reflectance in the set-up was carried out with a metal plate, installed on the place of a bottle. A corresponding polychromatic spectrum is shown in Fig. 9 (lower curve). The spectrum of the polychromatic source consisted of three lines at 93, 186 and 279 GHz with varying amplitudes. When the metal plate was replaced by a bottle with water, the intensities of all three peaks went down. The 279 GHz-component decreased much more compared with the 93 GHz- and 186 GHz-components (Fig. 9, middle curve). When a bottle with acetone was installed, the modification of the spectrum of reflected radiation was even more pronounced (Fig. 9, upper curve), and the components at 186 and 279 GHz are now more suppressed than the lower 93 GHz component.

The difference in the reflection coefficients at three frequencies was demonstrated for two liquids by this experiment. It is easier to choose between liquids starting from multifrequency measurements rather than from single-frequency measurements. In future we shall spread the frequency range of reflection measurements to the gigahertz range as low as 5 GHz and to the terahertz range as high as 1 THz to increase the accuracy of liquid identification. Also the scanning time will be decreased to values of around 10 ms.

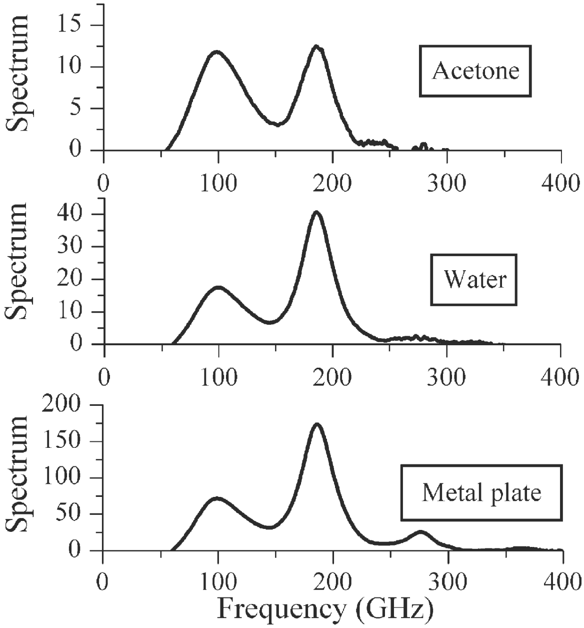

Fig. 9 Spectra, measured by the Hilbert spectrometer, from a metal plate at the place of a bottle (below), water in a plastic bottle (center) and acetone in a plastic bottle (above). Scanning times were equal to 1 s for each spectrum

6 Conclusions

We have considered the main features of the dielectric functions of various liquids and found the necessity of spectral measurements in the intermediate range from gigahertz to terahertz frequencies for reliable liquid identification.

We have presented the principles of Hilbert spectroscopy based on high-*Tc* Josephson junctions and the results of the characterization of Hilbert spectrometers in the gigahertz and terahertz ranges. Spectroscopic measurements are possible at the spectral range from 5 GHz to 5 THz, with 'power dynamic ranges up to 53 dB and NEP ~10^{-14} W/Hz ½ at LN temperatures.

Hilbert spectroscopy at the gigahertz and terahertz frequency ranges is suggested for liquid identification and first proof-of-principle experiments with scanning time of 1 s are demonstrated. We are planning to develop a demonstrator of a liquid identifier, based on Hilbert spectroscopy, operating at the frequency range from 5 GHz to 1 THz with a scanning time of around 10 ms.

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Liquid Explosives – The Threat to Civil Aviation and the European Response

C.J. De Ruiter and O.M.E.J. Lemmens

Abstract This paper deals with the specific group of homemade liquid high explosives in relation to aviation security. The sudden and irrefutable focus on homemade explosives and liquid explosives in particular after the 2006 defeated attacks in London, made the aviation security community realize that the security system and equipment were not optimized for this particular kind of substances. It is now generally accepted that hydrogen peroxide based liquid explosives (HPOM) pose a terrorist threat to civil aviation. It is easy, cheap and relatively safe to make effective HPOM IEDs. It is a high explosive of increasing and serious interest to terrorists and has been used recently.

In comparison to the well-know ‘old fashioned’ military and commercial explosives, liquid explosives and in particular HPOM are more difficult to detect because they are less well-defined by their chemical properties (poorly controlled manufacturing), they contain no nitrogen and their physical appearance (like shape, color) is poorly defined an hardly unique, in part because they need a container like a bottle or can or plastic bag etc. Both Europe and USA introduced late 2006 the so-called liquids-regulations at checkpoints to counter this threat, but this may become harder to sustain in the future.

Several countries contribute to the search for and development of existing and new technological solutions with the aim to relieve the measures and increase the effectiveness of the overall security process. The final solution would be to be able to detect liquid explosives inside a carry-on bag at operational speed. This is not yet possible with current technology. A second best option for the time being would be to be able to detect liquid explosives inside the 1 l plastic bag.

Even now existing technologies (like X-ray transmission, trace and chemical) can help in detection, sometimes after tuning to the specific problem. Inspection of single bottles or the 1 l plastic bag offers the best options today, but detection inside the carry-on bag in operation (speed requirements, false alarm behavior) offers prospects too. Further development in combination with ongoing research into the characteristics of homemade explosives, specifically liquid explosives, will probably make the balance shift towards technological solutions in the near future.

TNO Defence, Security and Safety, The Netherlands

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Keywords IED/aviation, security/liquid, explosives/detection

1 Introduction

A terrorist attack can happen everywhere in civil society. Crowded places are easy targets and there are many examples of such attacks where explosives have been used and many victims were made among the civilian population. Another example is civil aviation where in the past bomb attacks on aircraft in flight have caused many deaths and disruption of trust in this means of mass transport.

The bombs used by terrorists are so-called IED's (improvised explosive devices). They contain an explosive charge, typically a military, commercial or so-called homemade explosive or explosive mixture, and a professional or homemade detonator. Usually some electronic initiation system like a timer, barometer or radio control is added. Each individual part is not sufficient to bring out the explosive power of the IED, but the combination of these items makes it a deadly threat. This paper deals with the specific group of homemade liquid high explosives in relation to aviation security.

In order to prevent an attack with explosives to happen, one has to interfere in the process of the actor(s). Earliest disruption is of course preferred but difficult. However, in the case of a planned attack against an aircraft, a bomb has to be carried on board and there are only limited possibilities to do so. Detection of IEDs is a good option in airports. The last option, which is protection of the aircraft against explosion effects, is not preferred, if possible at all. Thus, in airports there are security checks in place on hold luggage, carry-on luggage and persons, and the process of technology and product development and implementation of regulations has a long history. Catastrophic incidents like 'Lockerbie' and '9/11', as well as the alleged plot against multiple airliners from Heathrow to the US only last year underline the necessity of effective security measures. Research on explosives, aircraft vulnerability and detection technologies go hand in hand with evolving regulations. Because this has been considered the area where a threat is well defined, the regulations for detection equipment in aviation security are leading for explosive detection equipment in general.

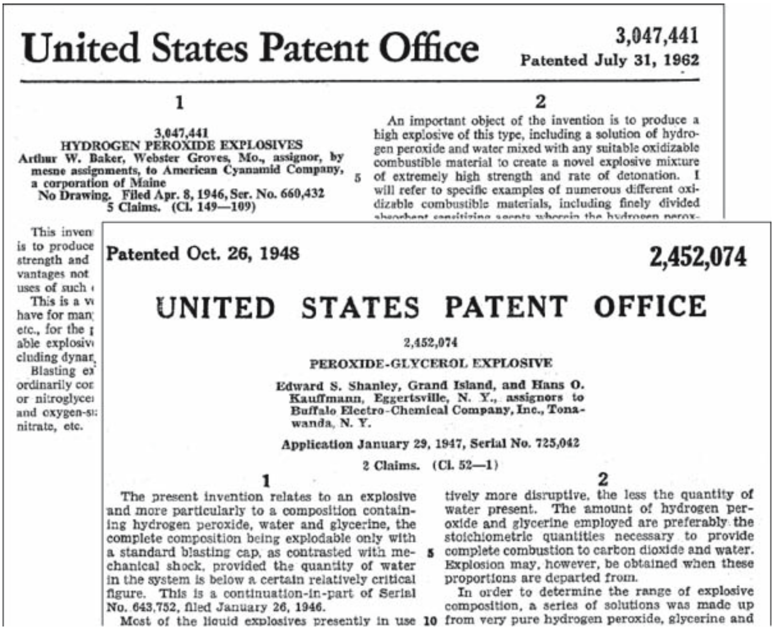
2 The Threat from Liquid Explosives

Most high explosives used in attacks against civil aviation have been commercial or military explosives. The development of technologies and regulations has been focused on these well defined substances. Analysis of trends in terrorism and recent attacks shows that another category of high explosives, the so-called homemade explosives, deserves serious attention. The term homemade is used, because the manufacturing process is not as sophisticated and well controlled as it is for military and commercial substances. As a result, these substances are generally poorly defined, impure, usually unstable, possibly very sensitive and relatively unreliable in functioning. They appear in the solid and liquid phase and in between as gels, pastes etc. Nevertheless, they have become increasingly popular among (specifically suicide) terrorists for several reasons.

- The access to successful (but also unsuccessful!) recipes is very easy over the internet.
- Most ingredients, so-called precursors, are cheap and easy to buy in grocery stores, garden warehouses or pharmacies.
- The synthesizing process is straightforward and does not require high tech laboratory equipment (hence the term homemade).
- The output can be very powerful and even comparable with military and commercial explosives.
- The initiation sensitivity is often high enough so it can be initiated without a detonator or with a homemade detonator.

Those homemade high explosives that appear in the liquid state are merely a special case. There is a considerable range of improvised liquid explosives; some of them can be homemade.

After the London 2005 metro attacks and the 2006 defeated attack against aircraft the category of liquid high explosives deserves massive attention. A specific example is Hydrogen peroxide based liquid explosives (hydrogen peroxide organic material or HPOM). This ‘new’ threat is not as new as many may think. Even before and during WWII, much research was done on HPOM (Fig. 1).



United States Patent Office 3,047,441
Patented July 31, 1962

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3,047,441
HYDROGEN PEROXIDE EXPLOSIVES
Arthur W. Baker, Webster Groves, Mo., assignor, by mesne assignments, to American Cyanamid Company, a corporation of Maine
No Drawing. Filed Apr. 8, 1946, Ser. No. 660,432
5 Claims. (Cl. 149—109)

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An important object of the invention is to produce a high explosive of this type, including a solution of hydrogen peroxide and water mixed with any suitable oxidizable combustible material to create a novel explosive mixture of extremely high strength and rate of detonation. I will refer to specific examples of numerous different oxidizable combustible materials, including finely divided

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Patented Oct. 26, 1948 **2,452,074**

UNITED STATES PATENT OFFICE

2,452,074
PEROXIDE-GLYCEROL EXPLOSIVE
Edward S. Shanley, Grand Island, and Hans O. Kauffmann, Eggertsville, N. Y., assignors to Buffalo Electro-Chemical Company, Inc., Tonawanda, N. Y.
Application January 29, 1947, Serial No. 725,042
2 Claims. (Cl. 52—1)

1

The present invention relates to an explosive and more particularly to a composition containing hydrogen peroxide, water and glycerine, the complete composition being explodable only with a standard blasting cap, as contrasted with mechanical shock, provided the quantity of water in the system is below a certain relatively critical figure. This is a continuation-in-part of Serial No. 643,752, filed January 26, 1946.

Most of the liquid explosives presently in use

2

tively more disruptive, the less the quantity of water present. The amount of hydrogen peroxide and glycerine employed are preferably the stoichiometric quantities necessary to provide complete combustion to carbon dioxide and water. Explosion may, however, be obtained when these proportions are departed from.

In order to determine the range of explosive composition, a series of solutions was made up from very pure hydrogen peroxide, glycerine and

Fig. 1 Some United States patents of peroxide based explosives dating back a long time (obtained USPTO.gov)

It is generally accepted that HPOM liquid explosives pose a terrorist threat to civil aviation. It is easy, cheap and relatively safe to make effective HPOM IEDs and it is a high explosive of increasing and serious interest to terrorists and has been used recently.

In comparison to the well-know 'old fashioned' military and commercial explosives, liquid explosives and in particular HPOM are more difficult to detect because they are less well-defined by their chemical properties (poorly controlled manufacturing), they contain no nitrogen and their physical appearance (like shape, color) is poorly defined an hardly unique, in part because they need a container like a bottle or can or plastic bag etc.

Effective means of detection, therefore, are required but the very nature of HPOM introduces specific difficulties to detection. For these reasons further understanding and characterization of HPOM by physical chemical properties is necessary and countermeasures (specifically detection) deserve urgent attention.

3 European and USA Response

As stated above, the development of detection technology in combination with regulations was based on the 'standardized' threat and has reached a high level of performance. The sudden and irrefutable focus on homemade explosives and liquid explosives in particular after the 2006 defeated attacks in London, made the aviation security community realize that the system and equipment were not optimized for this particular kind of substances. In this sense, the recent incidents were wake-up calls and it became clear that we now deal with a threat in terms of a range of liquid explosives of which HPOM is a case of special interest, precursors of high explosives and indeed flammable liquids, corrosive liquids, and other non-conventional means of violence.

The regulatory bodies in both USA and the EU responded by a 'low-technology' but effective measure in order to counter this threat. On September 2006 the USA responded and 2 months later Europe did so with more stringent regulations regarding liquids in carry-on baggage.

The USA introduced the so-called 3-1-1 regulation (see e.g. Prepare for Takeoff, Permitted and Prohibited items, Transport Security Administration, September 2006). The core being that a passenger is only allowed to carry liquids, gels and aerosols in containers smaller or equal to 3 oz in a 1 quart, plastic zip-top bag and only one bag per passenger. The regulation in Europe (see EU regulation for hand baggage, November 6, 2006) was formulated in a similar way: liquid, gels and aerosols in containers smaller or equal to 100 ml in a 1 l, plastic zip-top bag and only one bag per passenger (Fig. 2). No liquids are allowed anymore in the carry-on luggage or worn on the body.

The passenger has to present this bag separately to the security officer at the checkpoint. Exceptions in reasonable amounts over 3 oz or 100 ml are: baby food, medicines and liquids or gels for diabetic or other medical needs, also called essential liquids which must be declared to the security officer at the checkpoint. Liquids,

Fig. 2 Example of a 1 l plastic bag with smaller than 100 ml containers of liquids, gels and aerosols

gels and aerosol items, purchased at duty and tax free shops at the airport are only allowed in carry on baggage if packed in sealed and tamper-free transparent bags. They also have to be presented separately to the security officer.

Although effective, the measures taken are considered problematic for the operational process. On the EU regulatory level, a review takes place every 6 months, where threat and consequences are considered in balance. At the same time, further assessment of the threat from liquid explosives is ongoing in several countries and a search for and development of existing and new technological solutions takes place, which focuses on application at checkpoints. The aim is both to relieve the measures and increase the effectiveness of the overall security process.

4 Detection of Liquid Explosives at Checkpoints

In order to avoid liquid explosives being smuggled into the cabin of an aircraft, effective detection should be applied to persons as well as cabin baggage for now, in addition to the liquids measure. The currently allowed system in the EU of metal detection ad hand search could be made more effective by increasing the hand search fraction, but more so if so-called body-worn threat detection systems would be introduced, that can be very effective in detecting liquid explosives on the body.

With regard to detection of liquid explosives either in the plastic bag or in the cabin baggage, the following approach is taken in searching for technology solutions in a coordinated effort in several countries. All explosives detection technologies available now are considered, both selective and less selective, when they are applicable to the aviation threat in particular liquid explosives. Operational viability is a prerequisite, as they have to be implemented in checkpoint operation, either with or without the liquid regulation in place.

The final solution would be to be able to detect liquid explosives inside a carry-on bag at operational speed. This is not yet possible with current technology. A second best option for the time being would be to be able to detect liquid explosives inside the plastic bag which is presented to security separately, as it is now. Another (additional) option would be to detect the presence of liquids (not necessarily explosives) in 100 ml and more containers in the carry-on baggage. In that case a violation of the measure would be detectable rather than a direct detection of liquid explosives. Alarm resolution should take place on the liquids in the plastic bag or taken out of the carry-on luggage.

For inspection of liquid containers out of the carry-on bag the following technologies and products were considered and evaluated so-far with regard to operational viability (speed), false alarm behavior and alarm resolution.

- Visual inspection is not reliable because it is impossible to discriminate between a threat and an innocent liquid due to the many ways in which a liquid explosive can appear (clear, intransparent, different colors).
- Chemical testing by enzyme based strips (Fig. 3). The principle is that the strip only decolorizes if the HP molecule is detected in the liquid. Thus, it requires opening of the container and dipping of the test strip into the liquid under test. The method is cheap and relatively slow (1 min per container demonstrated in operational tests), but very reliable. Such test strips are available for hydrogen

Fig. 3 Enzyme test strips

peroxide, nitric acid, etc. For some others (nitro-glycerin, etc.) they are more expensive and analysis takes about 3 min.

- Trace analysis of air surrounding the closure of bottles. It has been shown that vapor (i.e. HP, nitro methane) will leak out of threads of closed containers, and thus it concentrates inside the plastic bag, leaks out and can be collected either inside or outside the plastic bag. Specifically the Smiths Sabre 4000 and Sabre FR were evaluated in operational trials. The software was adjusted and optimized for the detection of HP/HPOM. The process time was in the order of 30 s per plastic bag. This method offers detection capability in plastic bags, by opening the bag but not opening individual containers. In addition to HP, several types of liquid explosives can be detected. In the US extensive tests have been done using the ICx Fido.
- Bottle scanners, based on Raman spectroscopy (e.g. Ahura First Defender and Smiths First Responder), microwave interaction (e.g. CEIA EMA2+) and an X-ray CT based single bottle scanner (Nuctech Th-Scan) were evaluated (Fig. 4). Specifically Raman based (depending on bottle material for transmission) and CT based technologies offer good possibilities for single bottle inspection.

For inspection of whole carry-on bags in order to detect the presence of liquid (containers) or even detection of hazardous materials, trace detection is not a viable option, as sampling is very inefficient and unreliable. The use of X-ray transmission (single view, multi-view, CT) was evaluated to an extent (Fig. 5). From explosives detection in hold baggage this is a well-known process and the same principles can be applied to detect liquids in carry-on bags. The main characteristics in this case are density (and possibly Z_{eff} value) calculated from absorption spectra.

In general, liquid explosives are substantially more dense (1.2 g/cm^3+) than most innocuous liquids. It turns out that the image representation of transmission X-rays is very difficult to scrutinize for a screener with regard to liquid (containers) due to

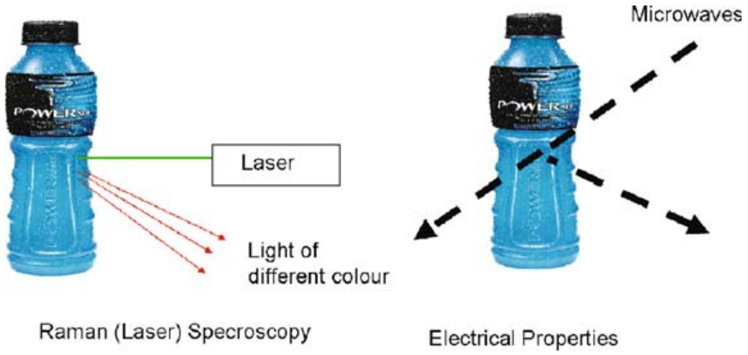

Fig. 4 Bottle scanner principles

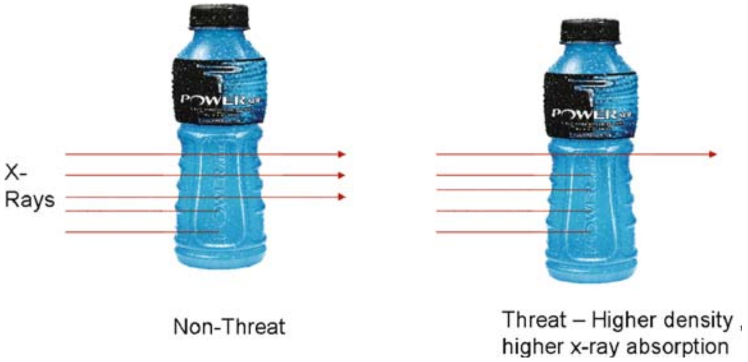

Fig. 5 X-ray interrogation principles

very subtle changes in color as displayed on screen. Automated equipment has been and continues to be developed to do this job and can be quite successful. However, due to the fact that the density range also occurs among innocent liquids (e.g. drinks and toiletries $<1.1\,\mathrm{g/cm^3}$, alcohols $<1\,\mathrm{g/cm^3}$, medicines $1.15 - 1.3\,\mathrm{g/cm^3}$, honey, syrup $\sim 1.4\,\mathrm{g/cm^3}$), a certain false alarm rate is inherent and cannot be avoided.

5 Future

The liquid measures (3-1-1 bag and 1l plastic bag) that have been implemented, though effective, have some serious by-effects. Some technologies under development can certainly play a positive role in reducing these negative effects while not compromising or even increasing security. Further development in combination with evaluation of tailor made trace detection equipment, bottle scanners and liquid detection options on X-ray cabin baggage screening equipment in combination with ongoing research into the characteristics of homemade explosives, specifically liquid explosives, will probably make the balance shift towards technological solutions.

6 Conclusion

The existence of liquid high explosives has been known for a long time, but the threat appreciation has changed dramatically in the recent year. Some liquid high explosives pose a realistic threat to civil aviation, especially but not exclusively hydrogen peroxide based (HPOM) based homemade explosives including their precursors. Experimental research on this group of materials is necessary in order to determine the specific threat and the characteristics on which detection by technologies is based.

Today Europe and USA have implemented the 1 l bag regulations, which will become harder to sustain in the future. Technology can help to reduce the burden and sustain the level of security or even increase that. Even now existing technologies (like X-ray transmission, trace and chemical) can help in detection, sometimes after tuning to the specific problem. Inspection of single bottles, the 1 l plastic bag offers the best options today, but detection inside the carry-on bag in operation (speed requirements, false alarm behavior) offers prospects too. This technology development should go hand in hand with evaluation of equipment and ongoing research into the characteristics of homemade explosives, specifically liquid explosives.

Miniaturized Photonic Sensor Devices for Real Time Explosive Detection

W. Schade[1], C. Bauer[1], R. Orghici[1], S. Waldvogel[2] and S. Börner[1]

Abstract New possibilites of diagnostic in the field of explosive detection are described applying noval photonic sensor devices using evanescent field spectroscopy and nano-dimensioned waveguides in combination with miniaturized lasers such as microchiplasers or quantum cascade lasers. A new concept is developed which allows the identification of TNT by using a fiber optic evanescent-field sensor.

Keywords Detection of explosives, evanescent-field spectroscopy, photonic sensor devices

1 Introduction

The clearance of mine fields and the detection of improvised explosive devices (IEDs) in conflict areas, as well as the threat posed on common welfare by terrorist attempts have increasingly become focal points of interest in recent years. In spite of technical progress anti-personnel mines are retrieved conventionally with metal detectors and mine prodders, and the search for explosives during inspection of baggage and persons is done in most cases still randomly. Novel photonic sensor devices applying evanescent field spectroscopy and nano-dimensioned waveguides in combination with miniaturized lasers such as microchiplasers or quantum cascade lasers reveal entirely new possibilities for diagnostics [1].

One application of evanescent-field-spectroscopy is the detection of explosives, e.g. TNT (Trinitrotoluene) which is commonly used by the military and is a major component of most landmines. It is also highly toxic and carcinogenic. The detection

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of this kind of explosive is very difficult due to its low vapour pressure. A new concept is developed which allows the identification of TNT by using a fiber optic evanescent-field-sensor that has been coated with a receptor film (triphenylene-based ketals). The receptor molecules bind selectively with the TNT molecules in a kind of key lock system which causes a change in optical properties of the coating and thus a reduction of signal intensity when light is guided through the fiber. In this way the selective and sensitive identification of TNT molecules is possible. The evanescent field effect can significantly be enhanced when nanowires with a diameter of hundreds of nanometers are used for light guiding. This opens new possibilities for miniaturized photonic sensor devices.

2 Basics of Absorption and Evanescent-Field Spectroscopy

The operation principle of an evanescent-field-sensor is based on the total internal reflection at the interface between two media with different index of refraction. The light that has undergone total internal reflection penetrates into the less refractive medium. An evanescent wave is built parallel to the interface and its electric field amplitude within the less refractive medium decreases exponentially with distance normal to the surface. The established electric field is called the evanescent field. However, if the optically less refractive medium is non-absorbing, no energy loss along the surface is measured. If this medium is an absorbing one, the intensity of the evanescent wave is attenuated and the transmitted power is reduced. These losses are caused by changes in refractive index (frustrated total reflection) and in absorption coefficient (attenuated total reflection) in the vicinity of a molecular resonance and can be used as signature for detecting specific species.

In standard direct absorption spectroscopy the Beer-Lambert law is applicable around a given absorption line. It describes the relationship between the intensities of light before and after interaction with the absorbing medium:

$$I(\lambda) = I_0(\lambda) \cdot e^{-a(\lambda) \cdot CL}$$

where I_0 is the incident intensity, I is the transmitted intensity, $a(\lambda)$ is the attenuation coefficient, λ is the wavelength, C is the concentration of the absorbing species present in the medium and L is the length of the sensing region.

The extinction which can be used to determine the concentration of the species is given by:

$$-\ln \frac{I(\lambda)}{I_0(\lambda)} = a(\lambda) \cdot C \cdot L \tag{1}$$

If absorption is the only loss mechanism, the attenuation coefficient can be replaced by the absorption coefficient $\alpha(\lambda)$ and the concentration can be determined by line shape analysis. However, for evanescent-field-spectroscopy the decrease in intensity can not be attributed to absorption alone since effects of attenuated total internal reflection and frustrated total reflection are superimposed in the vicinity of the critical angle and the line shape is consequently distorted.

3 Mid-infrared Open Path Sensor for TATP Detection

A handheld sensor device needs to be all-fiber coupled in order to avoid misalignment of optical elements. The interaction with the species under investigation is restricted to a short open optical path that can be positioned at places of interest. For the detection of TATP (tri-acetone tri-peroxide) in ambient air a mid-infrared (MIR) continuous wave (cw) quantum cascade laser (QCL) (Alpes Laser sbcw888) operating around 8 is used. The output beam of the QCL is coupled into a silver-halide fiber (artphotonics, PIR 400/500) which guides the light to an open path sensor. Here, the light is coupled out of the fiber, collimated and directed to a CaF_2 prism which acts as retro-reflector. The light is then coupled with a lens back into a second silver-halide fiber and guided back to a detector. The optical path length for interaction is $l = 15$ cm. The sensor system setup is shown in Fig. 1a and a photograph of the handheld open path sensor is given in Fig. 1b. Gaseous TATP evaporating from a sample of a few milligrams of TATP was measured under ambient air conditions. Figure 1c shows the tuning characteristic of the QCL and the absorp-

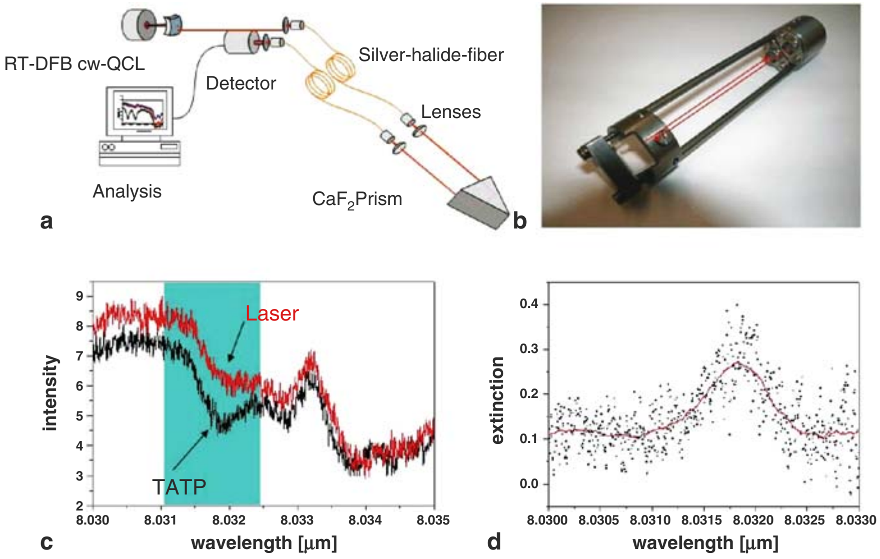

Fig. 1 Mid-infrared open path absorption sensor device: (a) Setup, (b) photograph of the sensor head, (c) tuning characteristics of the laser and raw data for absorption of TATP, (d) evaluated extinction of the TATP signal around $\lambda = 8\,\mu m$ using the raw data from (c)

tion due to TATP at $\lambda = 8.03\,\mu\text{m}$. A maximum extinction of 0.3 was calculated as shown in Fig. 1d. For this measurement the TATP sample was located on a laboratory table and the sensor was about 1 cm above the TATP. The measurements were repeated with and without sample for 20 times, demonstrating the reproducibility of the method.

It has to be emphasized that no sample preparation or pre-concentration is needed. The evaporated TATP has directly been measured under ambient conditions without use of a cell.

Not only is the detection of harmful substances required for such a sensor system but also the false positive alarm rate has to be extremely low for practical applications. If an alert system wrongly places an alarm, considerable costs for counter measures might apply and a lost in confidence will decrease the acceptance of the system. Therefore, possible interferences with other gases present have to be investigated carefully and must be eliminated. As an example, TATP, acetone and H_2O_2 have to be considered. Our experiments indicate, that the wavelength of the QCL can be chosen such that only overlap with TATP absorption lines is obtained. In further investigations absorption spectra of other species that might have potential interferences with TATP will be analyzed systematically.

4 Evanescent Field-Sensor for TNT Detection

For ultra-sensitive and selective detection of TNT, the evanescent-field-sensor [2] was coated with a specific receptor film [1, 3]. A charge-transfer-complex is generated in TNT ambience, which shows color change from transparent to red [3]. The maximum of its absorption spectra is at 532 nm. In order to measure the build-up of this complex, a frequency doubled passively Q-switched Cr^{4+}:Nd^{3+}:YAG microchip laser is used as a light source.

In Fig. 2a the experimental setup is shown. The laser radiation is divided into two parts by a beam splitter, serving as signal and reference beam. To distinguish between effects that are due to the formation of the charge-transfer-complex and other attenuations of the transmitted power that might occur, the data are analysed at two different wavelengths. This is done by use of a beam splitter and adequate filters to enable the transmission of the green light (532 nm) and the infrared light (1,064 nm) to the respective detector only. The same is done in the reference beam. In addition to the color-change of the receptor molecules (Fig. 3), a change of the refractive index in the receptor film takes place. This leads to an additional decrease of intensity. Resultant losses of the transmitted intensity are measured and this can be utilized for the determination of the concentration of TNT. In Fig. 2b the transmitted laser intensity at 532 nm is shown. The receptor film has the ability to bind TNT molecules reversible. This can practically be observed during the measurements by the decrease of signal intensity when TNT molecules are present and signal increase when the cell is evacuated or flushed by ambient air [4].

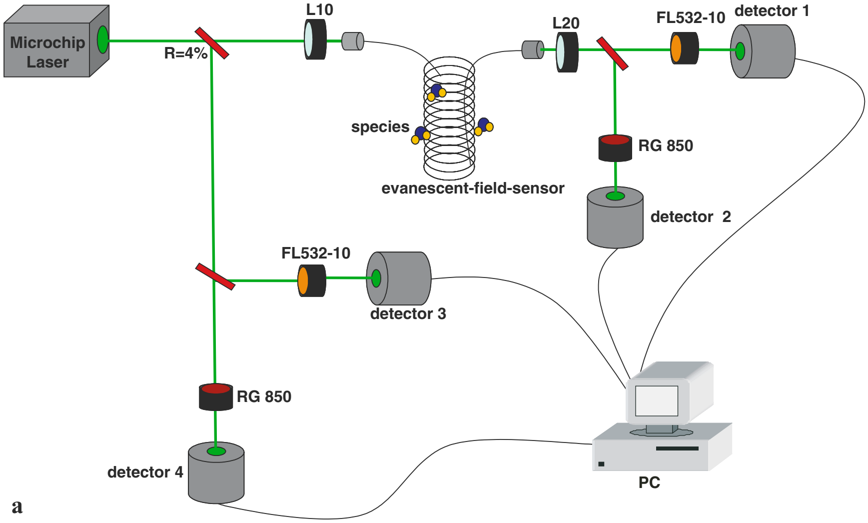

Fig. 2 (a) Experimental setup of the evanescent field sensor device applying receptor films for TNT detection

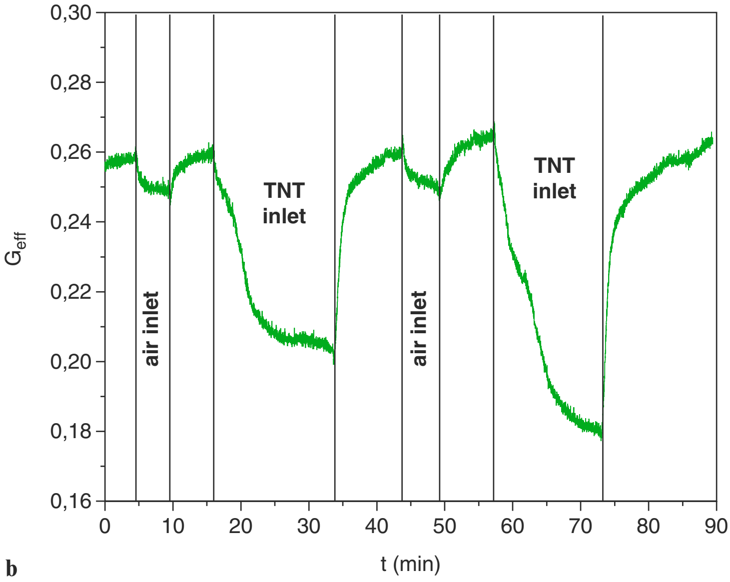

Fig. 2 (b) Sensor signal for TNT contact and recovering of the receptor film when flushing the sensor device by air

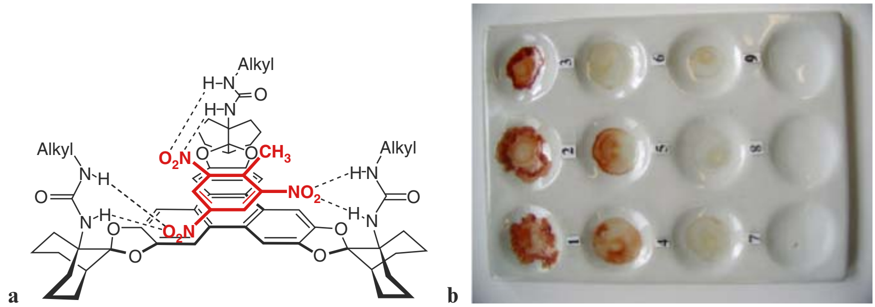

Fig. 3 (a) Chemical composition of the receptor film and (b) color change of the complex after contact with TNT

5 Evanescent Field Spectroscopy Applying ZnO Nanowires

Optical waveguides with diameters in the range of ten to hundreds of micrometers have been well studied for many decades and are successfully applied in optical communication and sensing. Commercial optical fibers, where a bare core acts as sensing element, are used for evanescent detection. The sensitivity can be increased by the use of long fibers with a large number of internal reflections or by adequate chemical sensitive layers.

Nanotechnology is providing miniaturized low-loss waveguides in the nanometer region and thus offers subwavelength-diameter waveguiding structures. Nanorods or nanowires offer a number of advantages in contrast to optical micrometer-fibers. A high percentage of light coupled into a nanorod can be guided outside as an evanescent field (Figs. 4 and 5). That offers some advantages in contrast to conventional systems, e.g. high sensitivity by an enhanced evanescent field, a high surface area

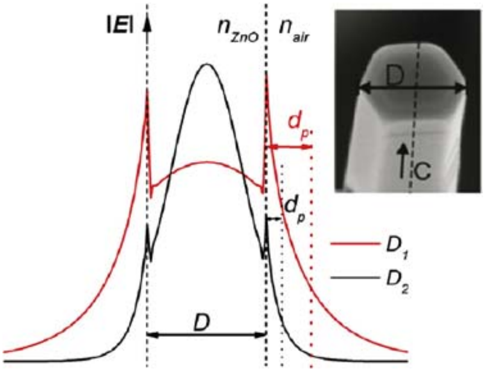

Fig. 4 Scheme of horizontal cut across the electric field distribution |*E*| inside and outside a nanorod at diameters $D_1 < D_2$. Penetration depth d_p is indicated. Inset: SEM image of ZnO nanorod with indication of diameter *D* and propagation direction of light ***c***

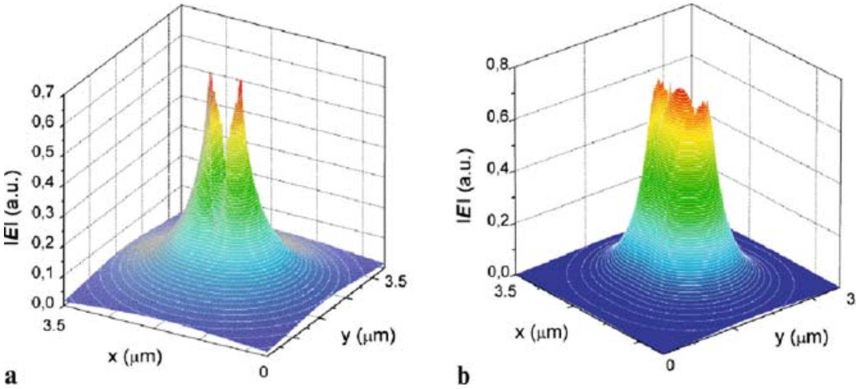

Fig. 5 Three-dimensional model of the absolute value of the electric field |E| (propagating and evanescent) of nanorods with diameters (a) D = 500 nm, (b) D = 1,000 nm at λ = 1,570 nm [6]

and miniaturization. Zincoxide (ZnO) nanorods are perfectly suited for sensor arrays or single nanorod sensors based on evanescent field interaction with surrounding molecules. The material properties of ZnO allow the application at near-infrared (NIR) wavelengths due to their high refractive index. Furthermore, ZnO nanorods can easily be produced in high crystalline quality, defined size, orientation and arrangement by a simple vapor-liquid-solid (VLS) process [5].

Two possible setups of an evanescent field sensor are using either a single nanorod for smallest possible setup or an array of aligned nanorods. The waveguiding conditions of a single nanorod are simulated to describe and quantify the optical processes in theory. A dispersion relation diagram gives information about the single mode operation range depending on the guided wavelength and rod diameter. Additionally the penetration depth and the magnitude of the evanescent field are analyzed for the fundamental mode to estimate the interaction zone and strength [6].

This is also done to obtain the optimum diameter for both, efficient wave guiding and maximum evanescent field. By examining the influence and interaction of neighbouring nanostructures the applicability of nanorod arrays will be proved. An array of closely packed nanorods again has the advantage of small size and in addition a high interaction volume due to the higher surface area.

Evanescent field interaction of nanoscale systems is certainly a promising method to adopt for miniaturized optical sensors. A nanorod-based evanescent wave sensor is an attractive tool for monitoring gases or other media, especially for explosive detection.

6 Stand-off Detection of Explosives

The reliable detection of explosives is of great importance but also the safety of personnel operating the devices. Therefore a stand-off remote system where operator and detection device are located at a safe distance from the object to be investigated is preferred for most applications. In stand-off geometry hazardous materials can be detected in a contact free mode which is realized in the present investigation by recording absorption features of back-reflected and/or back-scattered light intensities. In case of detection of nitro based explosives this is done in two steps as described in detail in earlier publications [7, 8]. First, pulsed laser fragmentation (PLF) is applied and then MIR-absorption spectroscopy with a synchronized probe QCL is used for sensing of constituents of the generated fragments of the surface layer of the sample to be investigated in a second step. Back-scattered and back-reflected light of the probe laser is collected by a telescope system consisting of two gold coated mirrors. The signals are recorded by a LN_2 cooled detector and analyzed via labview^R standard software. The stand-off setup in our laboratory is shown in Fig. 6. The emission of the fragmentation laser (wavelength typically 1,550 nm) is aimed through a hole within the collection mirror onto the sample. There, molecules from the surface layer are fragmented forming a plume very close to the surface. The synchronized probe beam of the QCL is adjusted collinear to the PLF beam and part of it is back-reflected and back-scattered. The plume generated by PLF contains NO_x molecules and our results indicate that information about the surface contamination can be gained from measuring the amount of generated NO_x in the vicinity of the sample surface. Spectroscopic analysis of the NO_x in the plume shows that explosive and non-explosive surface contamination can be distinguished simply by measurement of the NO/NO_2 ratio (Fig. 7).

This effect is investigated in detail for different materials and surface contaminations by application of infrared PLF at 1.5 μm and detection of the photofragments by FTIR spectroscopy in transmission geometry. Results are shown in Figs. 8 and 9, respectively. Only for contamination by energetic materials an NO signal is obtained (Fig. 9).

The results of these experiments indicate that it is possible to discriminate between energetic and non-energetic materials by detection of NO/NO_2 ratios. Similar experiments are performed with PMMA samples contaminated by TNT

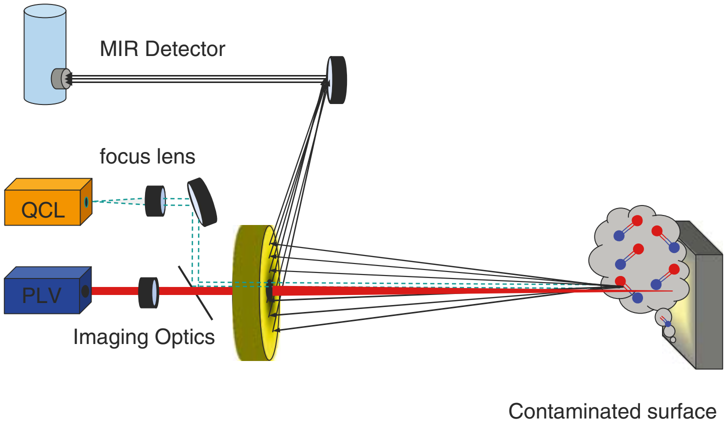

Fig. 6 Schematic scheme for the stand-off detection using PLF and mid-infrared laser spectroscopy

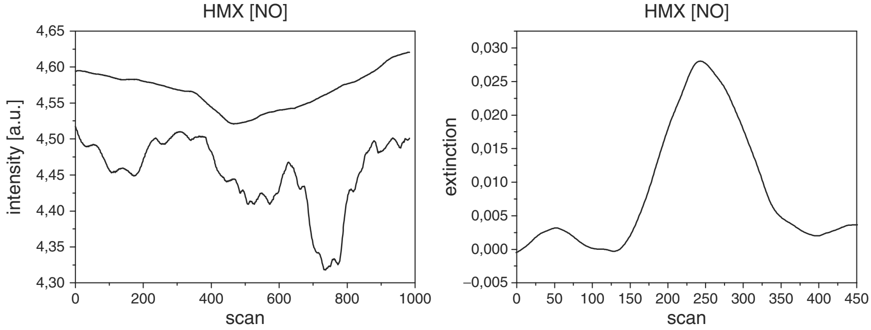

Fig. 7 NO absorption signal detected via MIR laser absorption after PLF above a HMX contaminated metal surface. (a) Raw data. (b) Evaluated extinction for a NO concentration of 75 ppm

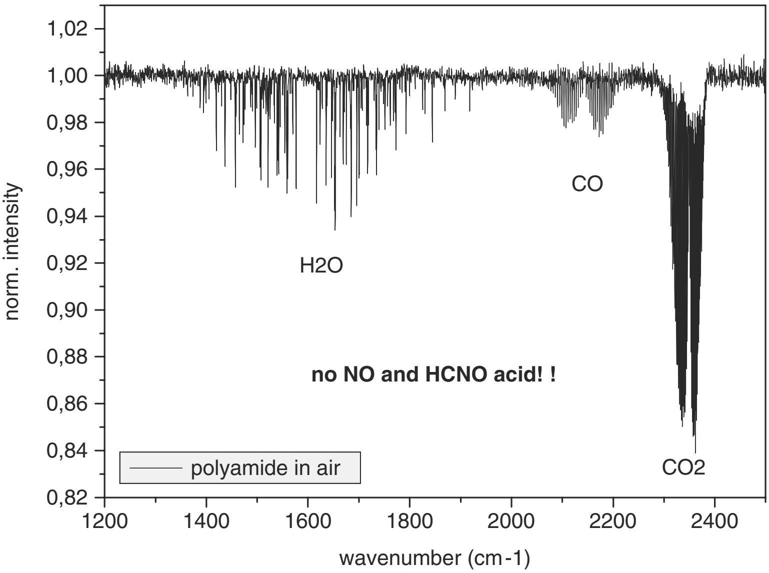

Fig. 8 FTIR spectrum of polyamide after PLF by 1.5 μm laser radiation

which are provided by the Wehrwissenschaftliches Institut für Werk-, Explosiv- und Betriebsstoffe (WIWEB). PMMA samples are loaded with 20 μg TNT per cm^2 surface. It can clearly be seen that the amount of NO is much higher for the TNT contaminated PMMA sample than without contamination [9].

In the present investigation the distance for stand-off detection with this laser setup was d = 5 m. Here the telescope optics restrict the distance for stand-off detection to this range; however, with modified optics distances up to 10 m and even more seem to be a realistic goal.

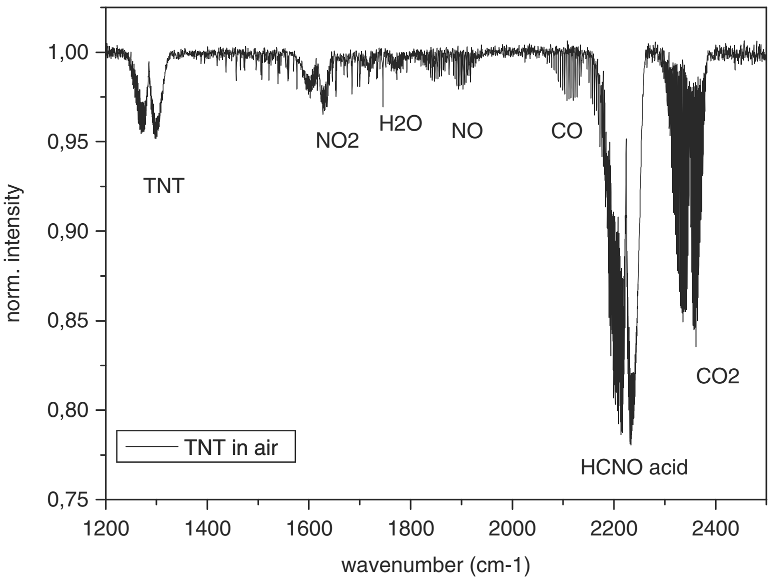

Fig. 9 FTIR spectrum of TNT after PLF by 1.5 µm laser radiation

7 Conclusion

Photonic sensor devices based on mid-infrared laser spectroscopy offer a great potential for the detection of explosives and other harmful substances. QCLs are highly suitable for compact und rugged sensor devices used for security applications. A stand-off detection scheme for low vapor pressure nitro based explosives and an evanescent field laser sensor have been developed and first results are discussed. On the other hand, explosives with high vapour pressure, e.g. TATP can directly be measured by open path absorption spectroscopy applying a QCL around 8 µm.

In conclusion, only the combination of different detection schemes and methods as well as data fusion enables a setup of a reliable sensor system that can be used for security applications. Optical sensing methods, in particular MIR absorption spectroscopy have great potential to become indispensable parts in such systems since they enable fast detection of substances without sampling or sample preparation.

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Conceptual Design of a Hand-Held Liquid Detector Based on Compton Scatter

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Abstract This paper introduces a conceptual design of a handheld liquid detector. This detector uses dual low energy γ or X-rays Compton scatter method to measure the density of liquid in a bottle, so it can judge the liquid property. This detector not only can measure the density of liquid, but also can measure the density and thickness of the bottle, so it also can judge the bottle property. If we have a database including absorbing and scattering coefficients of all kinds of bottles and liquid to the dual low energy γ or X-rays, we can decrease the complex degree of the analysis and improve the detection speed by using similar degree principle. Because this detector utilizes non-contact nuclear techniques, it can measure any kinds of bottles (nonmetal or metal, transparency or non-transparency, etc.) and liquid (colored or colorless, etc.) in principle. The scenes of practical application must be considered very much during development. In order to improve the detection speed, we must choose a good radioactive source with appropriate intensity, and a small volume radial detector with high energy resolution and high efficiency, and an optimized geometry and mechanical structure.

Keywords Compton scatter, liquid detector, density measurement, γ or X-rays

1 Introduction

Because it is very easy to get the materials of liquid explosive, and it doesn't contain metal, and it is low density, it can elude the check of common metal detectors and traditional X-ray imagine machines, liquid explosive becomes the first choice for terrorists gradually.

In order to avoid terrorists using liquid explosive to raid the airplane, nowadays international flights only allow passengers take 100 mL liquid goods (such as perfume) with them, only 1,000 mL liquid goods for domestic flights in China.

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And Passengers should open the lid of bottles for check, so it is very boring for people to travel by plane. In some other places, such as cinemas and stadiums, it also needs liquid detectors to check the liquid in bottles brought by audiences or spectators is beverage or liquid explosive or flammable agents or other dangerous chemical reagents.

For these requirements, many types of liquid detection equipments and instruments have been developed based on dual energy X-rays scanning method, X-rays Compton backscatter method, Radio frequency method, ultrasonic method and Laser Raman spectrum method according to its physical properties, such as density, electricity, acoustics, spectrum, etc. Some handheld liquid detectors based on Radio frequency method and Laser Raman spectrum method have been developed. Both of this two methods exists limitation, they can't check the liquid in metal bottles. And Laser Raman spectrum method is also sensitive to the liquid color and the transparency of the bottle wall. It is difficult for dual energy X-rays transmission method to develop handheld checker because of its inherent structure. It is easy for X-ray Compton backscatter method to develop handheld detector because of the radiation source and detector in the same side of the object checked, and it can check the liquid in any kinds of bottles.

This paper will introduce a conceptual design of a handheld liquid detector based on Compton backscatter method. Section 2 will introduce the concept of Compton scatter simply. Section 3 will describe the structure of the liquid detector and the process of its work, this section is the main and important part of this paper. Section 4 will introduce some suggestions on development this liquid detector.

2 The Principle of Detection

Compton scattering is a process that γ or X-ray photon interacts with "free" electrons which have lower binding energy in atoms of the object. The result is that the energy of photon is decreased and photon is scattered to other direction. The energy difference between incidence photon and scattered photon has been transferred to electron as its recoil energy. Fig. 1 is the sketch map of Compton scattering.

Where E_0 is the energy of incidence photon, E_θ is the energy of scattered photon, the angle θ between scattered photon and incidence photon is scattering angle, the angle ψ between recoiling electron and incidence photon is recoiling angle. Strictly speaking, the interaction between photon and out layer electron of atom is inelastic colliding. Because the binding energy of out layer electron of atom is very small (commonly several eV), it can be ignore comparing to the energy of incidence photon. So the out layer electron of atom can be seen as "free" electron. And the interaction between photon and out layer electron of atom also can be seen as elastic colliding. So the energy of the scattered photon can be gotten through formula (1):

$$E_\theta = \frac{E_0}{1 + \frac{E_0}{m_0 C^2}(1 - \cos\theta)} \tag{1}$$

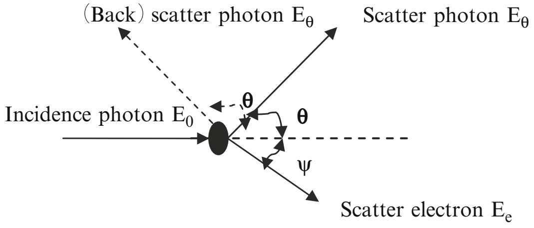

Fig. 1 Sketch map of Compton scattering

Where m_0C^2 is the rest mass energy of electron. Formula (1) shows that there are certain relationship among the energies of the incidence photon E_0, the energy of the scattered photon E_θ and the scattering angle θ. When E_0 is to a certainty, the energy of the scattered photons at different θ is different. When angle θ is more than 90°, scattering is backscatter.

In general, σ_c, the coefficient of Compton attenuation, shows the weakening situation of γ or X-rays intensity when they go through per unit distance in object by Compton interaction. σ_c is directly proportional to the atomic number Z of the object and the atom quantity in per unit volume. This means that σ_c is directly proportional to the electron density. It can be shown using formula (2):

$$\sigma_c = \sigma_e \frac{ZN_A\rho}{A} \qquad (2)$$

Where σ_c is the Compton scatter section of every electron, it can be seen as constant when the energy of the incidence γ or X-rays is during a certain range; N_A is the Avogadro constant; A is gram atom; ρ is the density of the object; $\frac{ZN_A\rho}{A}$ is the electron quantity of per unit volume, also named the density of electrons. Because the $\frac{Z}{A}$ can be seen as a constant under some stated conditions, Compton scatter method can be used to measure the density of goods.

It is important that the distribution of the scattering angle θ correlates to the energy of incidence photon. According to the study of Klein-Nishina, the probability of the scattering photons going forward and coming back is equal when the energy of incidence photon is very low.

When low energy photons transiting objects, Compton scatter absorbing is the main effect. So using Compton scatter method to measure the low density objects is more sensitive than using transition method. Compton scatter method also can measure the inner density of an object (e.g. liquid in bottle) penetrating the surface layer (e.g. wall of bottle), this can't be done by transition method.

3 The Structure and Work Process of The Liquid Detector

The structure of the liquid detector is as Fig. 2. S is γ or X-rays source, D is the detector. There are collimators on the way of photon emission and detection its aperture is small. The green parts presents the wall of bottle, its thickness is d_1. The blue part presents liquid in the bottle. The incidence photons from S transits the bottle wall and enters into liquid, only the scatter photons produced at position O and the scattering angle equals θ can be detected by the detector D, the distance from O to the bottle wall is d_2.

Let's assume the γ or X-rays at position S emit high and low energy two kinds of photons, their energy are E_{I0} and E_{J0}, their intensity of I_0 and J_0, and their linear absorbing coefficients in the bottle wall and liquid are μ_{I0p}, μ_{I0y} and μ_{J0p}, μ_{J0y}. The travel distance of scattering photons is d ($d = d_1 + d_2$), The travel distance of incidence photons is

$$\frac{d}{\cos\theta}$$

Let's also assume the energy of the photons scattering at position O and the scattering angle equaling to θ are E_{IB} and E_{JB}, and their linear absorbing coefficents in the bottle wall and liquid are μ_{IBp}, μ_{IBy} and μ_{JBp}, μ_{Jby}.

If the intensity of E_{IB} and E_{JB} at position O are I_B and J_B, then the intensity of E_{IB} and E_{JB} reaching detector D is I_b and J_b shown in formula (3) and (4):

$$I_b = I_B e^{-\mu_{IBy} d_2 - \mu_{IBp} d_1} \tag{3}$$

$$J_b = J_B e^{-\mu_{JBy} d_2 - \mu_{JBp} d_1} \tag{4}$$

Since the source intensity of E_{I0} and E_{J0} are I_0 and J_0, the intensity of E_{I0} and E_{J0} are I_{B0} and J_{B0} shown in formula (5) and (6):

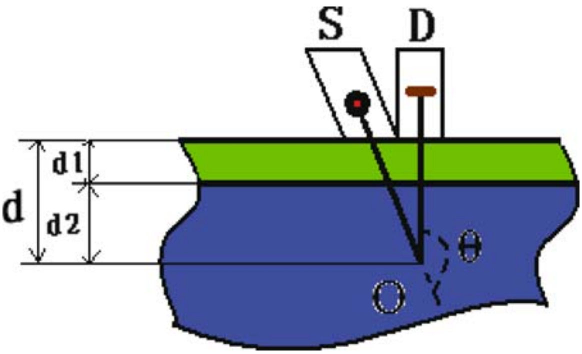

Fig. 2 The sketch map of liquid detector

$$I_{B0} = I_0 e^{-\mu_{I0y} d_2/\cos\theta - \mu_{I0p} d_1/\cos\theta} \tag{5}$$

$$J_{B0} = J_0 e^{-\mu_{J0y} d_2/\cos\theta - \mu_{J0p} d_1/\cos\theta} \tag{6}$$

Assume the converting (Compton attenuation) coefficients of photons from E_{I0} and E_{J0} to E_{IB} and E_{JB} at position O and pointing to detector D is $\sigma_{\frac{E_{j0}}{E_{IB}}}$ and $\sigma_{\frac{E_{J0}}{E_{JB}}}$ (they are proportional to the electrons density of the liquid), So below formula (7) and (8) can be got from formulas (3)–(6):

$$I_b = I_0 e^{-\mu_{I0y} d_2/\cos\theta - \mu_{I0p} d_1/\cos\theta}\, e^{-\mu_{IBy} d_2 - \mu_{IBy} d_1}\, \sigma_{\frac{E_{I0}}{E_{IB}}} \tag{7}$$

$$J_b = J_0 e^{-\mu_{J0y} d_2/\cos\theta - \mu_{J0p} d_1/\cos\theta}\, e^{-\mu_{JBy} d_2 - \mu_{JBy} d_1}\, \sigma_{\frac{E_{J0}}{E_{JB}}} \tag{8}$$

The I_0 and J_0 in formula (7) and (8) can be gotten through system calibration, and the I_b and J_b can be measured by the detector, the total thickness d ($d = d_1 + d_2$) is a constant decided by the mechanism structure.

For each measurement, these coefficients including μ_{I0p}, μ_{I0y}, μ_{J0p}, μ_{J0y}, μ_{IBp}, μ_{IBy}, μ_{JBp}, μ_{JBy}, $\sigma_{\frac{E_{I0}}{E_{IB}}}$, $\sigma_{\frac{E_{J0}}{E_{JB}}}$, and d_1, d_2 are unknown, because the material of the bottle wall and liquid are fixed, these coefficients are also fixed. Because the thickness of bottle wall d_1 plus d_2 equals the total thickness d, there are 11 unknown variables in formulas (7) and (8) in fact.

It is impossible to get 11 unknown variables through two equations. If suppose the material of the bottle wall and liquid are already known, two values of d_1 or d_2 can be gotten by formula (7) and (8). The two values of d_1 or d_2 must be the same or very close if the assuming is correct.

So, if there is a database including those coefficients in the liquid detector, after each measurement, the detector puts every possible combinations of bottle and liquid into formula (8) and (9), can get a serial d_1 or d_2 values in pairs, and then orders them according to the similar degree, after that the detector can know the possible types of the liquid, finally shows the liquid safety or danger by red or green lights and sound alarm.

4 Some Suggestions on Liquid Detector Design

4.1 Total Thickness d

Because there are so many types of bottles, especially the thickness of their wall are different, the total thickness **d** should be enough big. The intensity of photon source is proportional to the total thickness **d**, and the volume of photon interacting with liquid should be thought, so the value of total thickness **d** is suggested about 6–8 mm.

4.2 Scattering Angle θ

There is a distribution between the number of scattering photons and scattering angle θ, the number of scattering photons is proportional to the scattering angle θ, so it is good to select the biggest scattering angle θ. In fact because the limitation of to arrange the source and detector, scattering angle θ is suggested to be in the range of 120–150°.

4.3 The Photon Source ----γ or X-rays

After thought about the energy, half-life and other factors of photons source, ^{241}Am photons can be used as high energy, its energy is 60 keV. The low energy photons can be gotten by the high energy photons stimulating Ag or Sn target.

4.4 Detector

In order to distinguish the needed scattering photons from source photons and other interferential scattering photons, the energy resolution of detector must be good. Two type photon sources and their scattering photons at different scattering angles are listed in Table 1, the energy resolution of detector at 20 keV must be better than 6% in order to distinguish the needed scattering photons from source photons.

In addition, the detector should be small volume, high efficiency and firmness. CdTe or CdZnTe detector is a good choice.

Table 1 Relationship between scattering angle and energy

Source photon (keV)		
Scattering angle θ(°)	60.0	20.0
0	60.0	20.0
90	53.7	19.3
120	51.0	18.9
135	50.0	18.8
150	49.0	18.6
180	48.5	18.6

4.5 Data Acquisition and Software Design

Since the intensity of high and low two kinds of scattering photons is needed to acquire, two single channels are needed in detector. If the counter rate is not too high, multi-channels Analysis also can be used to acquire the spectrum, and the single channel function can be done by software.

The database containing absorbing and scattering coefficients of all kinds of bottles and liquid to the dual low energy γ or X-rays must be designed very well, update and maintenance must be simple and convenience.

Some standard samples must be equipped for checking the liquid checker in daily usage.

5 Summary

The handheld liquid detector based on dual low energy γ or X-rays Compton scatter has transferred the problem from measurement the electrons density of liquid to measurement the thickness of bottle wall, and gives the probability type of liquid by using the similar degrees principle. This makes the problem easier, and can improve the speed of analysis.

Paper also gives some suggestions for how to build the liquid detector.