AUTOMATED DEVICE FOR RAPID PREPARATION OF CHEMICALLY CONTAMINATED SAMPLES

Nicoleta PETREA[1], Vasile SOMOGHI[2], Neculai OANCEA[3], Dana CIUTAC[4], Gabriel EPURE[5], Răzvan PETRE[6], Daniel BORTOI[7], Alexandru MARIN[8]

Rezumat. *Această lucrare prezintă realizarea prototipului industrial al unui dispozitiv automat de prelucrare rapidă a probelor contaminate chimic pentru detecția și identificarea substanțelor toxice - DIPROT, prin transferul tehnologiei de fabricație a produsului de la CCSACBRNE la operatorul economic SC ATLAS CORP SRL. DIPROT este destinat cercetării CBRN, putând fi utilizat atât în condiții de laborator cât și în condiții de teren. Funcția sa este aceea de prelucrare rapidă a probelor din diferite matrice din zonele de interes, venind în sprijinul aparaturii de detecție și identificare, permițând echipamentelor de detecție să detecteze și substanțele toxice persistente, cu volatilitate scăzută, și scurtând durata prelucrării probelor, la laboratorul de analiză ajungând doar proba gata prelucrată ce va fi introdusă în echipamentul analitic.*

Abstract. *This paper presents the development of the industrial prototype for an automatic device for the rapid preparation of chemically contaminated samples for the detection and identification of the toxic compounds - DIPROT, by transferring the technology of manufacturing the product from CCSACBRNE to the economic operator SC ATLAS CORP SRL. DIPROT is designed for CBRN research, being able to be used both in laboratory conditions and field conditions. Its function is to rapidly process samples from different matrices in the areas of interest in support of detection and identification of equipment, enabling detection equipments to detect persistent toxic substances with low volatility and shortening the duration for preparation of the samples, at the analysis laboratory only sending the already processed sample to be introduced into the analytical equipment.*

Keywords: chemical warfare agents, dangerous toxic substances, detection and identification in situ, rapid preparation, chemical incidents.

[1]Senior researcher, PhD, Scientific Research Centre for CBRN Defence and Ecology, Bucharest, Romania (e-mail: nicoleta.petrea@yahoo.com).
[2]Senior Researcher, PhD, SC ATLAS CORP SRL, Bucharest, Romania (e-mail: v_somoghi@yahoo.com).
[3]Eng., SC ATLAS CORP SRL, Bucharest, Romania (e-mail: nicu.oancea@atlascorp.ro).
[4]Eng., SC ATLAS CORP SRL, Bucharest, Romania (e-mail: dana.ciutac@gmail.com; dana.ciutac@atlascorp.ro).
[5]Senior researcher, PhD, Scientific Research Centre for CBRN Defence and Ecology, Bucharest, Romania (e-mail: gabriel.epure@yahoo.com).
[6]Senior Researcher, PhD, Scientific Research Centre for CBRN Defence and Ecology, Bucharest, Romania (e-mail: petre.razvan@gmail.com). se
[7]Eng., SC ATLAS CORP SRL, Bucharest, Romania (e-mail: daniel.bortoi@atlascorp.ro).
[8]Senior researcher, PhD, SC ATLAS CORP SRL, Bucharest, Romania (e-mail: alex.marin2007@yahoo.com).

1. Introduction

At the occurrence or imminence of a chemical incident (military conflicts, technological accidents, or terrorist acts), the most difficult issues faced by decision-makers are to determine the nature of the incident and the type of toxic compounds used. DIPROT will support decision-makers in situations requiring the protection of the forces involved, the population and the environment, and avoiding false alarms, by facilitating access to analytical techniques for all military and civilian institutions that have detection and identification equipment and ensuring the development of new skills for institutions that do not have their own analytical equipment.

The detection and identification of chemical warfare agents (CWAs) and dangerous toxic substances are the first steps in the process of neutralizing them. It is necessary to know exactly which chemical agent was used and its physicochemical properties, in order to increase the protection of the personnel, find better ways of intervening, improve medical help and eliminate the consequences of chemical contamination [1, 2].

NATO member countries devote many efforts to develop the most efficient, fastest and safest detection and identification devices. At the same time, efforts are directed to minimize identification errors and increase in-situ evidence processing, to extend the limits of detection and/or reduce the time required to detect and identify dangerous substances (CWAs, industrial toxic substances, explosives, drugs, etc.) [3, 4, 5, 6].

What's new in DIPROT? i) It is the only equipment in the world able to prepare potentially contaminated samples in situ, in a very short time (a few minutes) and with low amount of reagents consumption, allowing intervention teams to make rapid and informed decisions, by being aware of the nature and severity of the contamination. The rapidity of sample processing and the fact that this is done automatically ensure the detection of compounds of interest before decomposing them under the influence of environmental factors. ii) It is able to ensure the efficient processing of samples from different matrices (water, solvents, soil, vegetation, etc.). iii) It can also be used successfully in chemical analysis laboratories, replacing the "classical" processing of samples, which is laborious and lasting and involves a high consumption of chemical reagents and the use of laboratory glassware that will need to be decontaminated and washed. So, add extra solvents, water, effort, etc.!

The research prototype developed within CCSACBRNE represents a unique technology of this type existing at national level, and, according to our information, even internationally, the idea of its realization arising from the need

to shorten the intervention time for CBRN and ecological events for the EMI-CBRN mobile intervention teams of the Romanian Army.

The device will enable intervention teams to prepare the samples in the area of interest in a standardized manner in a short time, reducing the risks associated with the manipulation of contaminated chemical samples [7]. The device will provide a viable alternative to the current sampling mode, which involves the transport of large quantities of chemical agents together with the samples taken. By using the device, the chemical extraction of the environmental samples will be made in a short time, avoiding the decomposition of the substances of interest under the influence of environmental factors, in line with the current demands of the armed forces that have responsibilities in the field of CBRN defence [8].

The Scientific Research Centre for CBRN Defence and Ecology (CCSACBRNE), the research organization that designed and developed the DIPROT device up to the prototype research stage, ensuring its functional testing in laboratory conditions, has as main field of activity scientific research applied in the development of CBRN specific equipment and defence techniques, being an advisory and expertise body within the MoD in the fields of: synthesis and analysis of hazardous chemicals (CWAs, industrial toxic substances) within the limits allowed by the Chemical Weapons Convention (OPCW); detection and decontamination; interventions to CBRN and environmental emergencies, etc. The centre has RENAR accredited testing laboratories, being the only Romanian centre accredited to carry out the functional testing of the DIPROT device with real CWAs.

SC ATLAS CORP SRL, the economic operator that transferred the manufacturing technology of the DIPROT device, is an industrial company that has constantly invested in the continuous renewal and diversification of production, the company's activity consisting in the implementation of innovative production technologies, in particular automation and control systems for industrial and residential applications.

The company's development strategy in recent years has been to diversify its areas of activity, especially the niche ones, with a significant involvement in the defence industry. In this respect, after the development stage of the product transferred to the industrial prototype level, the operational testing and its approval in the test fields of the MoD, SC ATLAS CORP SRL intends to start its production and sale.

SC ATLAS CORP SRL **will not have direct competition** on this product market, as it will be **the only manufacturer and supplier**, DIPROT representing an absolute novelty in the world, none of the famous manufacturers of chemical detection and identification equipment having such technology in their portfolios.

2. Destination

The DIPROT research prototype proposed to be transferred is a portable system designed for the preparation of solid and/or liquid samples in order to facilitate the process of detecting dangerous substances when using IMS (ion mobility spectrometry) [9], flamephotometry and colorimetric tubes, and the identification process for these substances when using GC-MS (Gas-Chromatography coupled with mass spectrometry) equipment [10].

DIPROT is used independently of other systems, and is equipped with a battery power supply to provide work autonomy even in the least accessible places where the specialized intervention vehicle cannot enter. The equipment can also be connected to the public power distribution network and to the electrical system of the transport vehicle. It is fully automated with a command and control unit, the operator can choose from the display the programme he wants to run. The device alerts acoustically and visually when it starts working or when it does not operate properly. The system configuration greatly relieves transport and operating activities [11].

DIPROT will be serviced by a single operator, equipped with individual protection equipment appropriate to the nature of the event being investigated and complying with occupational safety and health standards. **In the case of DIPROT use, the staff running it undergoes considerably lower risks than by using the classical preparation of chemically contaminated samples.**

The specific **national** market for the DIPROT product to be developed by SC ATLAS CORP SRL will be formed mainly by mobile intervention teams at CBRN and ecological events within the National Defence System (Ministry of National Defence, Ministry of Internal Affairs, The Protection and Guard Service, the Romanian Intelligence Service) and the Environmental Guard. These teams are equipped with special intervention vehicles, most of them packed with equipment for detecting and identifying chemical agents. It also addresses the analytical laboratories of the Ministry of Environment, Waters and Forests and private-sector analysis laboratories that process and analyse environmental samples.

At an international level, the specific market for our product could be represented by the First Responder Teams and the detection and identification equipment manufacturers (Bruker Daltonics, Agilent, Smith Detection, Environics, Shimadzu Scientific Instruments, Flir, PerkinElmer, Thermo Scientific, Hitachi Instruments, etc.), who will be able to offer this product as an auxiliary equipment to those produced by them. All identified customers should be interested in purchasing the DIPROT product because proper sample preparation is a primary step in detecting and identifying chemical compounds of

interest, inappropriate sample processing leading to analyser failure, and false results on samples contamination level.

3. Composition

It consists of two modules and the associated auxiliary systems (see Fig. 1) [11]:

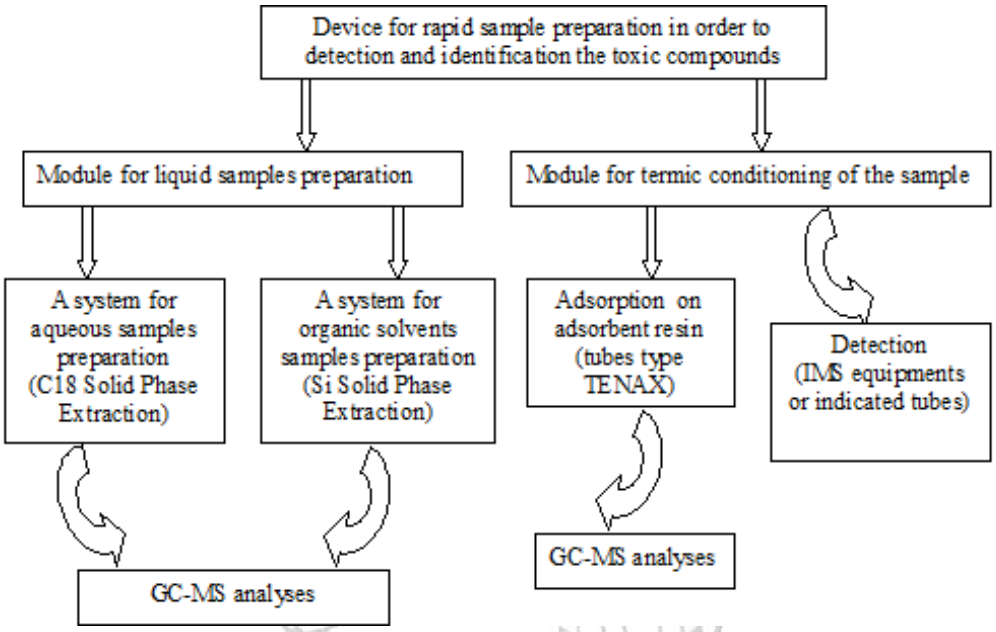

Fig. 1. The device design.

I. The Sample Conditioning Module, consisting of: heating mantle, electrical resistance for heating, temperature sensor, sample conditioning enclosure, micropump, TENAX adsorbent resin tube, connector for detector connection, hose, quick coupler, etc.

This module performs two functions: i) processing of liquid and solid samples for the detection of toxic compounds; ii) processing of solid samples in order to identify toxic compounds adsorbed in adsorbent resin tubes.

II. The liquid sample processing module, configured for the two types of samples:

II. a Aqueous sample processing system based on the extraction of toxic compounds on a C18 extraction cartridge. It consists of: 3 chemical reagent vessels (methanol, water, methylene chloride), reagents dosing system with actuators and solenoid valves controlled by microcontroller, extraction cartridge C18.

II. b Organic solvent sample processing system, based on the extraction of toxic compounds on the Si extraction cartridge. It consists of: 2 chemical reagent vessels (hexane, acetone), reagents dosing system with actuators and solenoid valves controlled by microcontroller, Si extraction cartridge.

II. c Components common to the liquid sample processing module: liquid sample system, sample trace washing system, technological air filtration system, extraction system, etc.

III. Components common to the entire system: assembly framework, support for the two modules and auxiliary components, power supply, command and control unit, etc.

The problems identified during the functional testing of the research prototype to be solved in the transition to the industrial prototype relate to:

i) reduction of the size and weight of the product;

ii) solving the tightness problems of the solvents transport routes caused by their pronounced chemical aggressiveness;

iii) increasing the robustness of the product by adopting constructive solutions to reduce the number of moving parts and eliminate elastically fixed components and connecting elements that can yield;

iv) changing the hardware and software platform by replacing the electronic components (expensive, bulky and, in some cases, hard to find) and the operating system - Windows XP (no longer supported by the manufacturer) with easy-to-purchase components anywhere in the world and open-source software (Linux);

v) optimizing product reliability by implementing an intelligent sensor system, removing from the configuration a considerable number of motors and pumps, moving parts, using the "Failure analysis" technique, by simulating the design stress under representative mechanical and thermal stimuli [12].

The advantages of the changes to be introduced are: a significant reduction of the gauge, the electricity consumption, the production costs; removing moving parts; a substantial increase in product robustness.

4. Performance features

The performance characteristics that DIPROT will have to meet are:

- the ability to condition solid and/or liquid samples, ensuring the identification of compounds of interest in the processed samples within the concentration limit of 0.02 ppm;

- the ability to process aqueous samples to identify toxic substances, ensuring the identification of compounds of interest in processed samples within the concentration limit of 10^{-3} - 10^{-4} mg / l;

- the ability to process samples of organic solvents for the identification of toxic substances, ensuring the identification of the compounds of interest in the processed samples within the concentration limit of 10^{-3} - 10^{-4} mg / l;

- acoustic and visual warning when entering or failing operation;

- working time entry of 5-10 min.;

- robustness, mechanical and thermal resistance and resistance to chemical agents. Structurally, the product must exhibit the mechanical strength specific to the products of military use;

- its own power supply system (12 VDC) and power supply to the vehicle electrical system (12 VDC) or to the public electricity grid (220 VAC);

- small dimensions (case / backpack with 10-12 kg weight);

- complete automation;

- electromagnetic compatibility with the other in-service equipment provided by the special intervention vehicles;

- it requires minimal maintenance, the disposable parts being positioned in such a way as to be the easiest to access in order to be swiftly replaced;

- the possibility of being run by a single operator.

Conclusions

The DIPROT is a portable system with its own power source (12 VDC), but can also be connected to the public power distribution network (220 VAC) and to the electrical system of the transport vehicle, designed for the rapid preparation of solid and/or liquid samples even in the area where the chemical incident occurred, in order to identify dangerous toxic substances.

The device developed by CCSACBRNE and SC ATLAS CORP SRL is **the only technology of this type, nationally and internationally**, fully automated, which can efficiently process samples from different matrices (water, organic solvents, soil, vegetation, etc.) directly on the spot, in a very short time with low reagent consumption, to the analytical laboratory allowing only the ready-processed sample to be introduced into the analytical equipment at the laboratory.

The rapidity of extracting the chemical compounds of interest from environmental samples, "in situ", leads to avoiding their decomposition under environmental

factors, in line with the current demands of the armed forces that have responsibilities in the area of CBRN defence.

It is particularly useful for chemical incidents (military conflicts, technological accidents or terrorist actions), when responsible actors have to make informed and rapid decisions on the nature of the incident and the type of toxic compounds used **to protect the forces involved, the population and the environment, and avoiding false alarms**.

It can also be used successfully in chemical analysis laboratories, replacing the "classical" processing of samples, which is laborious and long lasting and involves a high consumption of chemical reagents and the use of laboratory glassware that will need to be decontaminated and washed.

DIPROT must be capable of volatilizing persistent toxic compounds from liquid and/or solid samples for the purpose of detecting them with IMS equipment, flame photometric equipment and/or colorimetric tubes.

DIPROT would be capable of processing aqueous samples, organic solvent samples and soil samples or other solid materials with a view to identifying chemical warfare agents when using analytical equipment based on mass spectrometry.

It can be used by trained personnel from:
- the mobile intervention teams at the CBRN and ecological events equipped with special intervention vehicles and chemical agents detection and identification equipment, from the National Defence System and the Environmental Guard;
- the mobile intervention teams at the CBRN and ecological events in the countries that own special vehicles for intervening in chemical and ecological incidents (first responder teams);
- the analysis laboratories from the Ministry of Environment, Waters and Forests;
- the analysis laboratories in the private field that process and analyse environmental samples.

The companies manufacturing detection and identification equipment (Bruker Daltonics, Agilent, Shimadzu Scientific Instruments, Smith Detection, Environics, Flir, PerkinElmer, Thermo Scientific, Hitachi Instruments etc.) could be particularly interested in acquiring this product, as it can be offered as an auxiliary equipment to those they already produce.

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Notations and/or **Abbreviations**

CBRN – Chemical, Biological, Radiological, Nuclear

CCSACBRNE – Scientific Research Centre for CBRN Defence and Ecology

CWA – Chemical Warfare Agents

EMI-CBRN – CBRN Mobile Intervention Teams

GC-MS – Gaz Cromatography coupled with Mass Spectrometry

IMS – Ionic Mobile Spectrometry

MoD – Ministry of Defence

OPCW – Organization for the Prohibition of Chemical Weapons

ppm – Parts per millions

R E F E R E N C E S

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