

# Universal Decontaminant for Neutralization of Nerve and Vesicant Chemical Warfare Agents

Liubov Vakhitova

L.M. Litvinenko Institute of Physical-Organic  
Chemistry and Coal Chemistry of National  
Academy of Sciences of Ukraine  
Kyiv, Ukraine

Varvara Drizhd

L.M. Litvinenko Institute of Physical-Organic  
Chemistry and Coal Chemistry of National  
Academy of Sciences of Ukraine  
Kyiv, Ukraine

Ramil Vakhitov

Department of Applied Ecology  
Kyiv National University of Technologies and Design  
Kyiv, Ukraine

Volodymyr Bessarabov

Faculty of Chemical and Biopharmaceutic Technologies  
Kyiv National University of Technologies and Design  
Kyiv, Ukraine

Nadiya Taran

L.M. Litvinenko Institute of Physical-Organic  
Chemistry and Coal Chemistry of National  
Academy of Sciences of Ukraine  
Kyiv, Ukraine

Mykola Korotkikh

L.M. Litvinenko Institute of Physical-Organic  
Chemistry and Coal Chemistry of National  
Academy of Sciences of Ukraine  
Kyiv, Ukraine

**Abstract:-** The invention describes a method of obtaining a composition with universal neutralizing action, intended for urgent decontamination of human skin, animal fur, clothes, equipment and premises, contaminated with organophosphorus nerve agents (VX, GB and GD) or sulfuric vesicants (HD). The mentioned toxicants can be used as part of chemical warfare (CW), industrially used pesticides or pharmacological components. Decontamination composition according to the invention is produced and supplied as a dry mixture of active ingredients that should be mixed with water before use. The mixture consists of a peroxosolvate (hydrogen peroxide – urea, a nucleophilic oxidizing agent), an activator (either ammonium bicarbonate or boric acid), a cationic surfactant (for micelle formation) and sodium hydroxide (for pH regulation). The proposed decontamination composition provides 99% degradation of diethyl 4-nitrophenyl phosphate (paraoxon, used as a nerve agent simulant) by nucleophilic mechanism in 30 seconds. The time it takes for the same composition to provide 99% oxidation of methyl phenyl sulfide (used as a vesicant CW agent simulant) is up to 30 minutes. Mentioned chemical processes can be accelerated significantly by adjusting decontamination system pH levels: at  $\text{pH} > 10$  the rate of nucleophilic substitution increases, while at  $\text{pH} < 10$  oxidation reactions accelerate.

**Keywords:-** Chemical Warfare Agent; Detoxification; Hydrogen Peroxide; Paraoxon; Methylphenyl Sulfide; Oxidation; Nucleophilic Substitution.

## I. INTRODUCTION

The universal action of studied decontamination system is based on the dual chemical essence of hydrogen peroxide, which is one of the well-known compounds used for neutralization of various CW agents [1, 2]. It is highly effective in both oxidative and nucleophilic reactions involving VX, GB, GD and HD agents, while simultaneously being non-corrosive and environmentally safe [3, 4, 5]. Hydrogen peroxide is commonly found in industrially produced reactive decontamination kits, like Decon Green, M291, DS2, DF-200, used by NATO military and rescue teams [6]. Over the years of their use in field, these systems have proven themselves to be effective decontaminants for a variety of toxic agents. However effective and versatile an agent  $\text{H}_2\text{O}_2$  is, it does have certain limitations of use. First and foremost, most decontamination systems containing hydrogen peroxide are water-based, as it is required to maintain them safe for health and environment, while certain toxicants display hydrophobic behavior and, therefore, low solubility in aqueous media. Additionally, when it comes to simultaneous neutralization of mixed toxicants, certain inconsistency exists in the levels of acidity required to reach maximum rates of oxidation vs nucleophilic substitution. Among the factors that limit the possibilities of decontamination, there is also the unsatisfactory reactivity of hydrogen peroxide when it comes to sulfoxidation of yperite analogues [7]. Therefore, to ensure the chemical compatibility of the neutralizing agent with the toxicants, a decontamination system is needed, which simultaneously effectively solubilizes the substrates, and ensures a sufficiently high rate of their decomposition [1, 8, 9].

A promising direction in the design of environmentally friendly decontamination systems is the use of organized aqueous nanoscale systems - micellar solutions or microemulsions [10, 11] where hydrogen peroxide is activated to form peroxy anions [12, 13]. In the presence of surfactants, reagents are concentrated at the interface between the micellar phase and water, and favorable conditions are created for nucleophilic attack on the electrophilic centers in the toxicants.

Market reviews of commercially produced decontamination compositions designed for neutralization of CW agents are not published in open sources. One can assume that this is due to the data being classified, as such materials are mainly used by military personnel. However, it is safe to assume that the main reason for this lack of information lies in the overall absence of interest in chemical warfare, which was not deemed a real modern threat until year 2022. The leading producers of decontamination materials and kits for neutralization of CW agents registered in the US are Sandia National Laboratories, FFRDC (developed and commercialized DF-200 decontamination foam) and First Line Technology, LLC (produces Dahlgren decontamination solutions). When it comes to European market of chemical protection, local manufacturers are mostly represented by Cristanini SpA (Italy), Kärcher Futuretech GmbH and OWR GmbH (Germany). All these companies produce a wide range of means for decontamination and neutralization of chemical, biological, radioactive and nuclear warfare [14, 15].

Military conflicts in Ukraine and the Middle East, which evoked a real threat of CW use, create a constant challenge for both the defense contractors and civil research institutions. Recently, in accordance with NATO's 2022 Chemical, Biological, Radiological and Nuclear (CBRN) Defence Policy [16] a new task was frameworked for researchers and engineers involved in the design and production of protective means. It is to modernize or develop new technologies of personal decontamination kits, which would simultaneously be effective, cost-efficient and easy-to-use. The report on developments in science and technology to the fifth special session of OPCW states convention [15] also emphasizes the urgency of developing and introducing new environmentally safe reactive means of personal chemical protection, as well as other portable solutions for neutralization of CW agents.

## II. RESEARCH AIM AND OBJECTIVES

The aim of this research was to develop method for obtaining a decontamination composition for urgent detoxification of personnel, clothes, equipment and premises that have been exposed to direct contact with organophosphorus nerve agents (VX, GB, GD) and vesicant agents (HD or analogues).

The G-type agents (tabun, sarin, soman), while originally developed and produced as insecticides, later were also adopted for military use due to their ability to inhibit esterase enzymes in body, when the maximum permissible concentrations of those agents are exceeded. Predominantly affected are acetylcholinesterase in synapses and nerve cell membranes, as well as butyrylcholinesterase in blood plasma, the adverse

effects can also extend to reproductive functions. Organosulfur vesicant agents (yperite, lewisite and their analogues) display severe cytotoxicity forming blisters on exposed skin by disrupting the integrity of cell membranes. This leads to the destruction of epidermal barrier and if left untreated, can be accompanied by a critical dehydration in the body. In order to prevent serious damage to nerves, integumentary system, or other systemic effects detrimental to human health, it is crucial to perform urgent (up to 30 minutes from the time of exposure) detoxification, which can be effectively done using individual decontamination kits. Such kits can be produced industrially and should be part of the standard equipment for CBRN units and hazmat personnel.

Within the limits of their capabilities in terms of decomposition of toxicants, individual decontamination kits must satisfy the following conditions:

- commercial availability and affordability, high reactivity of the composition and maximum ability to neutralize a variety of toxicants;
- versatility of chemical action of decontamination system towards various types of toxic compounds;
- practicality, no restrictions or special conditions required for use of the decontamination composition;
- environmental safety of the material, its components, and the products of their reactions with target toxicants;
- low corrosiveness, safety of use for decontamination of human skin surface;
- chemical stability of decontamination system during prolonged storage and transportation in various climatic conditions.

## III. FORMULATION, MANUFACTURING, AND STABILITY OF THE DECONTAMINATION COMPOSITION

The following chemicals can be used to produce the decontamination composition.

Urea peroxide (hydrogen peroxide-urea,  $\text{CO}(\text{NH}_2)_2 \cdot \text{H}_2\text{O}_2$ ) was used as a neutralizing agent with nucleophilic and oxidizing action. It is a commercially available non-toxic, shelf-stable crystalline peroxide product.

Quaternary ammonium salts (QAS) in the composition are usually represented by substances with a general structure  $\text{R}_3\text{R}'\text{NBr}(\text{Cl})$ , wherein  $\text{R} = \text{CH}_3, \text{C}_2\text{H}_5, \text{R}' = \text{C}_n\text{H}_{2n+2} (n \geq 10)$ . They should be able to form stable micellar solutions and to solubilize hydrophobic substrates. Cetyltrimethylammonium bromide (CTAB) and its close structural analogues with  $n=16-24$  were found to be the best choice of QAS for obtaining the decontamination composition according to the invention. Alternatively, benzyltriethylammonium chloride, benzyltrimethylammonium chloride or mixture thereof, as well as cetylpyridinium chloride can be used as equally effective detergents.

Hydrogen peroxide activators, such as ammonium bicarbonate  $\text{NH}_4\text{HCO}_3$  or boric acid  $\text{B}(\text{OH})_3$  are added to the composition.

pH level of the micellar solution is regulated by introducing either sodium (NaOH) or potassium (KOH) hydroxides into the composition.

The decontamination composition is manufactured in the form of a powdered mixture of the following components: urea peroxide, quaternary ammonium salt, activator (either ammonium bicarbonate or boric acid), sodium hydroxide. The mixture is prepared by blending the components using a ball mill at 20–35 °C for 5–10 minutes. The target size of particles

in final product is in the range of 50–80  $\mu\text{m}$ . MILLCHOC-500 (KARYEV-TEC sp.zo o., Poland) grinder was used for preparing the test batch of the composition, however any similar available equipment can be applied. If the components used in the production are supplied as finely grinded powders (particle size up to 80  $\mu\text{m}$ ), it is expedient to use a dry powder mixer (for example, EQ-DRY-PL, MTI Corporation, USA) rather than a grinder. Mixing time in this case should also be approximately 5–10 minutes.

After either mixing or grinding stage is complete, the final mixture is separated into 200 g portions and placed into string sealed stand-up pouches (Fig. 1). Packaged product can be stored in dry storage premises at 5–35 °C, away from direct sunlight.



Fig. 1. Sample of the Decontamination Composition Packaging.

The composition is applied to contaminated surfaces in the form of a solution, which should be prepared just before use, as it is not subject to storage. The solution is prepared by mixing the contents of the kit package (200 g) and 800 g of water (preferably warmed up to ~ 40 °C) in a glass or plastic vessel. The mixture should be stirred continuously until the dry components are completely dissolved, after which the solution is ready for use.

Prepared solution of the decontamination composition can be applied in various ways – mechanized spraying using air foam gun or spray gun; manual application onto the contaminated objects, materials and surfaces using brushes, napkins, rags, sponges or any other appropriate equipment; immersion of contaminated objects or clothes directly into the solution. The composition quickly neutralizes toxicants to harmless substances. However, it is recommended to further remove decontamination products from the surfaces using any available multi-purpose detergents.

It is known that a solution of hydrogen peroxide in water is thermodynamically unstable and decomposes with the formation of water and oxygen. Decomposition, predictably, accelerates with the increase in temperature, concentration and pH level of the solution. In alkaline media (pH>10) the process is accelerated 3–5 times, which significantly reduces the concentrations of active hydrogen peroxide in the composition. Therefore, the composition should be supplied as a dry mixture and the solution is prepared immediately before use.

Dry decontamination composition produced as described in this study is stable for 1–3 years if stored in a sealed container with no exposure to air and direct sunlight. It was confirmed by the results of permanganometric titration, the results of which are provided in table 1.

As is evident from table 1, the maximum loss of hydrogen peroxide content in the proposed decontamination composition does not exceed 7 % in 1 year and 12% in 3 years. Thus, the use of exclusively dry ingredients does solve the

problem of prolonging the shelf life of decontamination compositions with no loss in the rate of toxicants decomposition.

TABLE I. AMOUNT OF H<sub>2</sub>O<sub>2</sub> IN THE DECONTAMINATION COMPOSITIONS DURING THEIR STORAGE<sup>A</sup>

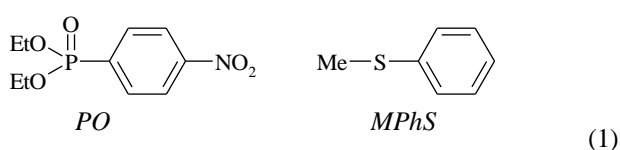
Sample №	1	2	3	4
[H <sub>2</sub> O <sub>2</sub> ], mol/l, initial	2.06	1.02	1.05	0.59
[H <sub>2</sub> O <sub>2</sub> ], mol/l, stored for 1 year	1.99	0.98	0.99	0.55
[H <sub>2</sub> O <sub>2</sub> ], mol/l, stored for 3 years	1.95	0.93	0.96	0.52

<sup>A</sup>Composition of samples 1–4 provided in Table 2.

The manufacturing process for the decontamination composition described in this study is technologically facile, does not require the use of any harmful or corrosive substances, and does not involve complex processes and energy-consuming equipment.

#### IV. EXPERIMENTAL CONFIRMATION OF THE DECONTAMINATION ACTION

Compounds with chemical structures and reactivity similar to those of the target toxicants were used as model substrates (simulators of the toxic agents). The substances used (1) are considered equivalent to real CW agents. Commercially available uncontrolled insecticide paraoxon (PO, diethyl 4-nitrophenyl phosphate) emulates nerve CW agents, and is a direct analogue of other hazardous organophosphorus pesticides. Whereas, methyl phenyl sulfide (MPhS) can be considered as an analogue of mustard gas in terms of its reactivity and hydrophobic properties.



In neutral and acidic environments (pH < 9), hydrogen peroxide, produced from hydrogen peroxide-urea present in the decontamination composition, rather slowly oxidizes mustard gas and V-series agents. It is well-known that stronger alkaline media is required for nucleophilic substitution in organophosphorus esters involving HOO<sup>-</sup> ions. At the same time the acid dissociation constant for hydrogen peroxide pK<sub>a</sub> equals 11.6, therefore, at pH ≤ 9.0 only 0.03–0.3 % of hydrogen peroxide is present in its reactive HOO<sup>-</sup> ionic form.

This problem with carrying out simultaneous oxidation and nucleophilic processes at “compromise” pH levels can be solved by adding activators (NH<sub>4</sub>HCO<sub>3</sub> or B(OH)<sub>3</sub>) to the decontamination composition containing hydrogen peroxide-urea.

In the presence of NH<sub>4</sub>HCO<sub>3</sub> at pH 7-10, the activation of hydrogen peroxide by hydrogen carbonate ion HCO<sub>3</sub><sup>-</sup> in the process of sulfides oxidation in H<sub>2</sub>O<sub>2</sub>/NH<sub>4</sub>HCO<sub>3</sub><sup>-</sup> system occurs through the formation of peroxydicarbonate anion HCO<sub>4</sub><sup>-</sup>. This anion is characterized by stronger oxidizing ability than that of H<sub>2</sub>O<sub>2</sub>.

The addition of B(OH)<sub>3</sub> into the decontamination composition leads to the formation of peroxy anions: peroxyborates B(OH)<sub>3</sub>(OOH)<sup>-</sup> and B(OH)<sub>2</sub>(OOH)<sub>2</sub><sup>-</sup>, which display stronger oxidizing power compared to hydrogen peroxide.

In the presence of QAS an additional acceleration is observed for the decomposition processes of both substrates (PO and MPhS) due to the reagents intensified concentrating and solubilization. This is characteristic of micellar catalysis. At the same time, the maximum increase in the rate of micellar nucleophilic processes with the participation of PO corresponds to pH values of 9-10. The reaction rate constants K<sub>s</sub> measured for the binding reactions between the studied substrates and the decontamination composition are quite high. They were determined to be 500-700 l/mol for PO and 300-400 l/mol for MPhS depending on the pH of the composition.

The degree of conversion for PO and MPhS in aqueous solutions of decontamination compositions prepared according to recipes 1–4 (Table 2) was determined as follows.

TABLE II. THE INFLUENCE OF THE COMPOSITION OF STUDIED SAMPLES ON THE DEGREE OF CONVERSION OF PO AND MFS

№ Components, wt. %	1	2	3	4
Hydrogen peroxide-urea	97.09	76.8	71.2	60
Boric acid	-	-	8.8	-
Ammonium bicarbonate	-	18.5	-	-
Cetyltrimethylammonium bromide	2.90	4.6	5.7	10
Sodium hydroxide	0.01	0.01	0.05	0.1
PO conversion after				
30 seconds, %	65	98	99	99.9
30 minutes, %	82	99	99.9	99.9
MPhS conversion after				
30 seconds, %	25	47	60	13
30 minutes, %	36	99	99	25

1 ml of a 0.1 M solution of PO in 1,4-dioxane was added to 20 ml of the aqueous solution of decontamination composition and mixed until PO is equally distributed. After set time intervals (first measurement after 30 s), the amount of 4-nitrophenolate ions in the studied solution was evaluated using UV spectroscopy (Optizen POP UV spectrophotometer, Mecasys, South Korea) at 405 nm. In order to do this, test samples were prepared by dissolving 1 ml of decontamination system, taken at the end of each set time interval, in 99 ml of water. The amount of 4-nitrophenolate anion in each sample corresponds to the amount of decomposed PO, while the remaining quantity of the substance was determined according to the following equation:

$$[PO] = [PO]_0 - D \times 100/\varepsilon \quad (2)$$

where  $\varepsilon = 18600$  l/mol when the length of the cuvette is 1 cm, D - optical density.

Similarly, the amount of MPhS in the decontamination system was determined. 1 ml of 0.04 M solution of MPhS in dioxane was added to 20 ml of the decontamination solution and mixed until all MPhS was dissolved. After 30 seconds, the amount of MPhS in the reaction solution was determined by UV spectroscopy at a wavelength of 260 nm. A 3 ml sample of the decontamination composition was prepared, followed by the evaluation of its optical density. The amount of residual MPhS was calculated using the following equation:

$$[MPhS] = D/\varepsilon \quad (3)$$

where  $\varepsilon = 850$  l/mol when the length of the cuvette is 1 cm.

An aqueous solution of the corresponding decontamination composition (with no MPhS added) was used as a reference sample in each case.

The data presented in Table 2 show that the optimal decontamination compositions are those listed under № 2 and 3, containing  $\text{NH}_4\text{HCO}_3$  (№2, table 2) and  $\text{B}(\text{OH})_3$  (№3, table 2) as activators. Those compositions demonstrate universal reactivity in both, oxidative and nucleophilic, processes, thus providing effective neutralization of both studied substrates with high reactions rates. Both toxicants are almost fully converted in 30 minutes time.

Studies on the kinetics of the decontamination reactions in the invented systems demonstrate that the formation of peroxyanions accelerates oxidation up to 200 times in the presence of  $\text{NH}_4\text{HCO}_3$  (pH = 9, №2, table 1) and 300 times when  $\text{B}(\text{OH})_3$  is present (pH = 10, №3, table 2).

The studied decontamination composition can be used for neutralization of organophosphorus and organosulfur toxicants, which are commonly found in pesticides, chemical warfare materials, pharmacological ingredients and subproducts.

As mentioned above, simultaneous neutralization of organophosphorus and organosulfur toxicants (mixtures of GB, GD and HD type, or VX type compounds) can be effectively carried out when pH of the decontamination composition is maintained at 9–10, which is also neutral enough to make decontamination of human skin surface safe. The concentration of  $\text{H}_2\text{O}_2$  in such case should not exceed 1 M, which is equal to that of a common drug store available 3% hydrogen peroxide solution.

For individual neutralization of organophosphorus compounds, which occur via nucleophilic reactions, it is recommended to increase pH of the decontamination composition to 12–13 by adding sodium hydroxide. In such case, the required concentrations of hydrogen peroxide also increase to 3–5 M. However, such conditions are only suitable for decontamination of inanimate objects, premises and equipment. Similarly, it is recommended to use higher concentrations of hydrogen peroxide to oxidize sulfides, that contaminate non-living objects, provided that pH is maintained in 8–9 range.

## V. CONCLUSIONS

The results of this study can have a significant applied importance, as they are related to a significant aspect of environmental and public safety – minimization of health risks and environmental threats from toxicants released due to the use of chemical warfare (CW) agents or certain agricultural chemicals. Chemical protection of the population is especially relevant in countries and regions with active military conflicts, threats of terrorist acts, when the use of CW is a valid concern. Proposed decontamination system can be used by military CBRN units, emergency and rescue services, disaster medicine teams for urgent decontamination of premises, human skin and clothes, animals.

The offered simple method of obtaining the decontamination composition for neutralizing blistering and nerve agents can be useful for chemical engineers involved in designing and manufacturing of products aimed at combating the consequences of chemical contamination for population and environment. The invention is recommended for use in the production of individual decontamination kits for the simultaneous decontamination of organophosphorus (GB and GD) and sulfur-containing (HD) toxicants.

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