STUDY OF EFFECTIVENESS OF FLAME SUPPRESSION BY ORGANOPHOSPHORUS COMPOUNDS IN LABORATORY AND SCALED-UP TESTS

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ABSTRACT

Inhibition and suppression effectiveness of recently prepared volatile organophosphorus compounds (OPC) was studied. For this purpose the influence of OPC on concentration limits of combustion of cup-burner flame and on inhibition of premixed propane/air flame using Mache-Hebra burner was studied. The most promising compounds were tested with the help of Transient Application, Recirculating Pool Fire (TARPF) apparatus. Propane was used as a fuel. The fire suppressants were injected into the airflow with the help of originally designed aerosol generator, which produces micron and submicron droplets. The fire suppression effectiveness determined on TARPF apparatus was compared with that measured using cup-burner technique. Halon 1301 (CF_3Br) was used as a comparison standard in all tests. The experiments demonstrated a possibility of practical application of non-volatile organophosphorus fire suppressants in form fine-dispersed aerosol. The toxicity of OPC on laboratory mice was assessed. General parameters of toxicity of OPC under various route of administration including lethality (LD_{50}) were determined and compared with those of parent substances.

INTRODUCTION

Search and testing of novel fire suppressants - halon alternatives among organophosphorus compounds (OPC) still presents a perspective direction of investigation in area of fire extinguishing. High boiling point (low volatility) of studied OPC brought us to synthesis of more volatile fluorinated derivatives of OPC. However a high reactivity of some of fluorinated OPC, which were recently synthesized [1-3], hinders their practical use. Nevertheless, the application of moderately volatile OPC as fire suppressants is quite possible using an aerosol technology for delivering the agent to a fire source. As we demonstrated earlier [3] the effectiveness of organophosphorus suppressants is not affected by the form (vapors or aerosol), in which the fire suppressants reach the flame because the droplets evaporate in the flame front.

At present some data on effectiveness of flame inhibition and suppression by various OPC in laboratory conditions were published in [4-6]. The major part of these data does not give a possibility to evaluate the extinguishing concentrations in real conditions but provides only ranging the compounds according their fire suppression effectiveness. Thus, in spite of a appreciable theoretical and experimental knowledge accumulated on organophosphorus fire suppressants no bench tests on apparatus like [7] with turbulent pool fire were performed until now. That is why now there is an urgent need for screening tests of fire suppressants, which are an indicator of full-scale tests. The goal of present work is to determine the effectiveness of OPC at inhibiting and suppressing different types of flames in laboratory and bench tests with turbulent flame. The toxicity of some OPC was also assessed.

EXPERIMENTAL

In our previous paper [3] we studied the effect of OPC on different types of flames – premixed and diffusive. In present paper we continued studying novel OPC, which were synthesized during the last year. Their formulas and boiling points are presented in Table 1.

Table 1. Novel OPC tested and their boiling points

Formula	B.p. at pressure [Torr], experimental data	B.p. at pressure 760 Torr
$(CF_3CH_2O)_2P(O)CF_3$	147-148/760	147-148
$(CF_3CH_2O)_2P(O)OCH(CF_3)_2$	83/10	210[a]
$(CF_3)_3P$	45/760	47
$CF_3CH_2OP(O)[OCH(CF_3)_2]_2$	77/15	190[a]
$[(CF_3)_2CHO]_3P$	60/55	140[a]
$[(CF_3)_2CHO]_2P(O)C_2H_5$	65-67/65	135[a]
$(CF_3CH_2O)_2P(O)C_2H_5$	70-72/70	135[a]
$(C_4F_9O)_3PF_2$	180/760	180
$(CF_3CH_2O)_3P$	131/760	131

[a] Estimated

In addition we studied the influence of fine dispersed aerosol of inorganic salts on speed of a premixed stoichiometric C_3H_8/air flame. The salts were introduced into the flame in form of aqueous solutions. Table 2 shows the studied salts and the concentration of aqueous solutions.

Table 2. The studied salts and concentration of their solutions

Salt	Mass of salt [g] in 100 cm^3 of solution
KBr	15
$NH_4H_2PO_4$	18
K_2CO_3	15
Na_2CO_3	15
$FeCl_3*6H_2O$	15
KH_2PO_4	15

The influence of an inhibitor on speed of a premixed flame was determined with the help of Mache-Hebra nozzle burner, which was described in details earlier [3]. The solutions of the salts were introduced into the hot unburnt gases using a nebulizer. The mass-median diameter of the aerosol droplets was about 10-20 microns whereas after evaporation of water the size of solid particles became 2.5 - 5 microns. To evaluate the dependence of burning velocity on loading of directly potassium, sodium, iron and phosphorus their losses due to the deposition inside the burner were taken into account. The contribution of water from the solutions into the inhibition effect was also taken into account. The fire suppression effectiveness and extinguishing concentrations of the compounds of diffusive heptane/air flame were measured using the cup burner technique described earlier [3]. It is noteworthy that application of the technique, which includes the addition of CO_2 to the flame, proposed by Linteris et al. [8], facilitates a more precise measurement of the extinguishing concentration. It makes also possible to understand whether an inhibitor is flammable in airflow.

A part of cup burner experiments were performed at constant airflow temperature of 75 ^oC that provided complete evaporation of the droplets. For the experiments, which were carried out at reduced temperature 25 – 65 ^oC the generator of superfine aerosol was used. The generator produces the aerosol with droplet's size <1 micron. The temperature of the extinguishing mixture was increased if necessary by heating the chimney electrically. At low temperature the losses of suppressants due to deposition inside the chimney were taken into account. For this purpose simultaneously with feeding OPC, the air was sampled from the flame regon. The sampled gas was passed through aerosol filter and liquid nitrogen trap. Comparing the amounts of a substance, which were fed and trapped, the dopant losses were evaluated and corresponding corrections were made.

To carry out experiments on a turbulent pool fire extinguishing by transient application of fire suppressants, the apparatus (TARPF) similar to that designed by Grosshandler et al. [7], which simulates the fire of a jet engine inside the nacelle was fabricated. The apparatus of the same size as [7] is shown schematically in Fig. 1. The apparatus includes a wind tunnel of square section,

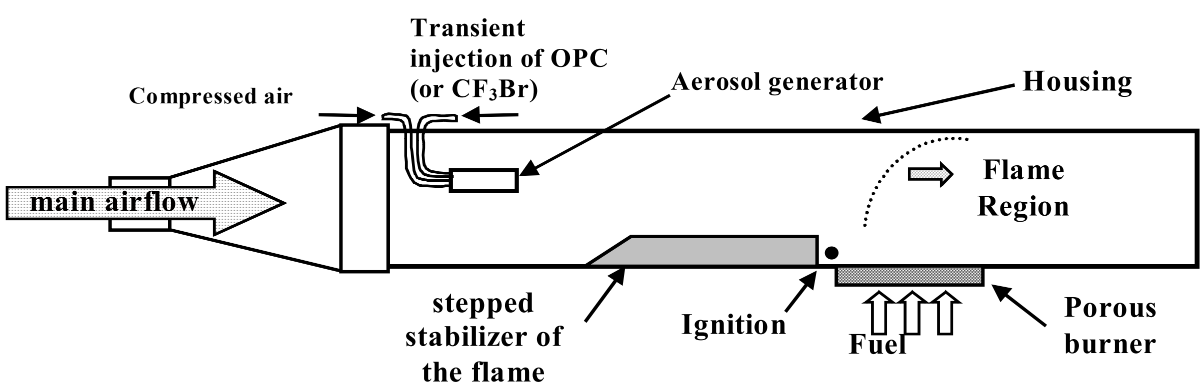

Fig. 1. A scheme of Transient Application, Recirculating Pool Fire (TARPF) Apparatus.

flat porous burner protected by a backward step (ramp) and feed systems for gaseous and liquid fire suppressants. The setup provides transient injection of fire suppressants. For supply of liquid OPC the generator of superfine aerosol of original design, which provides very high concentration up to 10% (by volume) of OPC, was used. The generator produces the aerosol with droplet's size <1 micron that is why the deposition of the droplets inside the wind tunnel does not occur. Though all aerosol droplets reach the flame loss-free. The main stream of air providing the turbulent flow with Re≥10000 mixes with flow of the fire suppressant forming the extinguishing mixture. The velocity of gas above the burner varies from 2 to 6 m/s. The apparatus was supplied with system of heating of the extinguishing mixture that makes possible to study the influence of temperature on extinguishing concentration. Propane was used as a fuel (volumetric flow rate 40 cm^3/s), which was ignited by a spark-plug. The authors [7] demonstrated that propane flow rate varied in known range does not influence on extinguishing process. The same authors recommended the value of flow rate equal 40 cm^3/s. The process of flame suppression was observed visually and recorded by video camera. The extinguishing concentration of an agent was shown in Ref. [7] to depend on its time of application and to reach its minimum at the time of about 1 s. That is why in spite of having the opportunity to vary the time of application in present work we kept it constant and equal to 1 s. The loading of fire suppressant was varied by changing the flow rate of main air stream, i.e. by diluting the flow of fire suppressant.

RESULTS AND DISCUSSION

Flame Suppression

Figure 2 presents the dependence of speed of a premixed stoichiometric C_3H_8/air flame on loading of aerosol of inorganic salts counting on an atom of the metal (for $FeCl_3$, Na_2CO_3, K_2CO_3, KBr) and phosphorus ((for KH_2PO_4, $NH_4H_2PO_4$). The burning velocity in Fig. 2 is normalized for the burning velocity of the undoped flame. The same figure shows the dependencies for orthophosphoric acid and TMP. The results obtained for potassium and sodium salts are in agreement with data reported in Ref. [9]. The effectiveness of TMP, orthophosphoric acid and $NH_4H_2PO_4$ are very close that is explained by the same molar content of phosphorus in molecules of the compounds. $NH_4H_2PO_4$ is the typical ammonia salts, which readily dissociates in a flame. Its destruction products as for OPC are evidently phosphorus oxides and oxyacids providing inhibition effect. The suppression of diffusive flames by solutions of orthophosphoric acid was also studied by Fisher et al. [10]. These authors taking orthophosphoric and methylphosphonic acids and OPC as an example demonstrated that aqueous solutions of various phosphorus-containing species provide approximately the same inhibition effect.

A number of novel synthesized OPC were tested using the cup-burner technique. Figure 3 shows the extinguishing concentration of CO_2 as a function OPC loading to a n-heptane/air flame. The measured extinguishing concentrations for the tested compounds are tabulated below. The more precise extinguishing concentration for $(CF_3CH_2O)_3P$ is given in Table 3 as well.

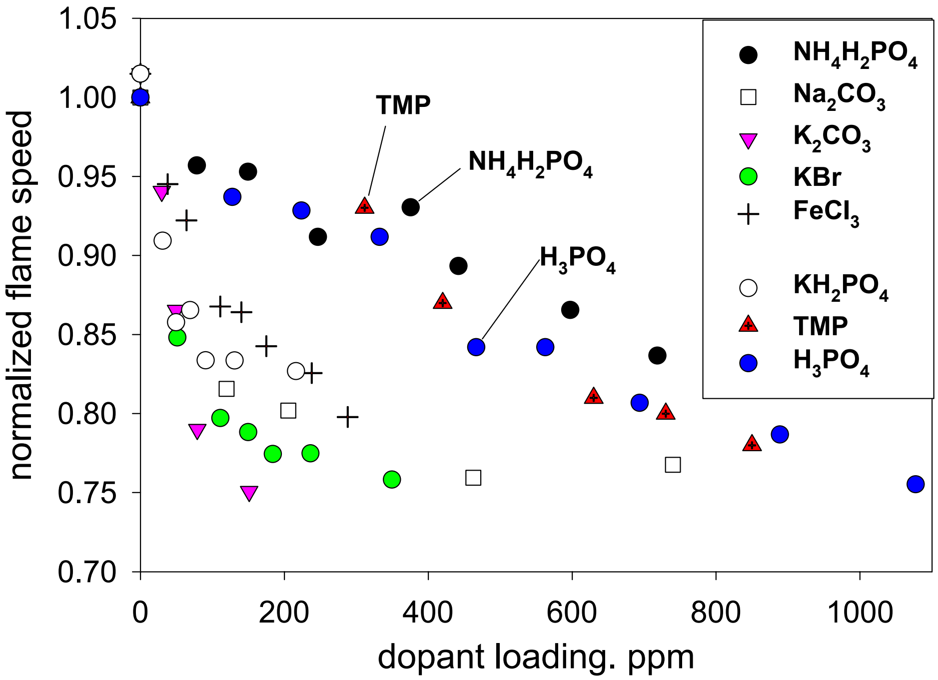

Fig. 2. Normalized speed of a stoichiometric premixed C_3H_8/air flame as a function of the dopants loading.

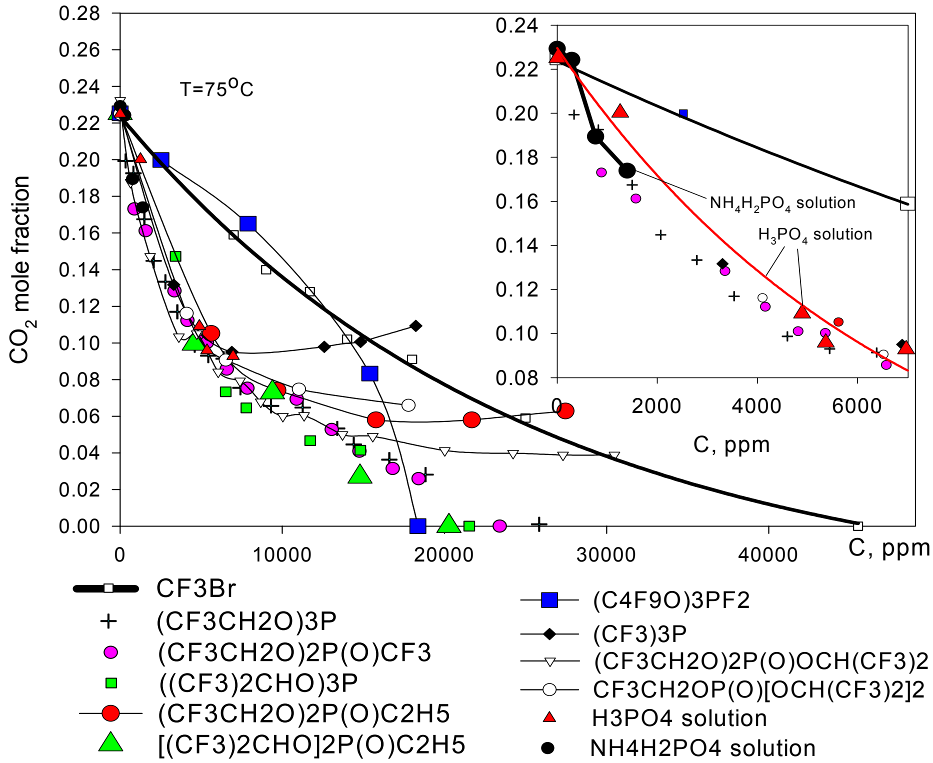

Fig. 3. Cup burner tests: extinguishing concentration of CO_2 as a function of loading of fire suppressants.

Table 3. Extinguishing concentrations of novel OPC at 75 ^{0}C.

Compound	Extinguishing concentration, % by volume	Extinguishing concentration, g/m^3	Reference data	Air stability
$(C_4F_9O)_3PF_2$	1.8±0.2	621		Some fumes
$[(CF_3)_2CHO]_2P(O)C_2H_5$	2.0±0.2	366		No fumes
$[(CF_3)_2CHO]_3P$	2.2±0.2	523		No fumes
$(CF_3CH_2O)_2P(O)CF_3$	2.3±0.2	322		No fumes
$(CF_3CH_2O)_3P$	2.6±0.2	381	1.78-3.1 [2]	No fumes
$(CF_3)_3P$	No extinguishing, flammable			Some fumes
$(CF_3CH_2O)_2P(O)OCH(CF_3)_2$	No extinguishing at 3%			No fumes
$CF_3CH_2OP(O)[OCH(CF_3)_2]_2$	No extinguishing, flammable			No fumes
$(CF_3CH_2O)_2P(O)C_2H_5$	No extinguishing, flammable			No fumes
CF_3Br	4.6	306		

For some compounds a secondary flame in upper part of the chimney was observed because of combustion of their vapors in heated airflow. For these OPC a distinctive dependence of CO_2 extinguishing concentration on OPC loading is observed (see Fig. 3). For example, when OPC loading increases a higher concentration of CO_2 is necessary for flame extinguishing. This effect as it was reported by us earlier [3] is connected with 2 competitive processes: (1) the flame inhibition by OPC and temperature decrease due to an increase of heat capacity of the combustion products; (2) an increase of flame temperature due to additional heat release from OPC combustion that counteracts flame suppression. Thus, a different effectiveness of various OPC can be explained by influence of several reasons: the heat of formation of OPC, heat capacity of their vapors, their destruction rate and destruction products in a flame. The results presented in Table 3 and Figure 3 indicate that under the same conditions the effectiveness of some OPC is higher than that of CF_3Br in 1.8 - 2.5 times. In this case the relative effectiveness is the ratio of minimal extinguishing concentrations. To find out how the temperature of the extinguishing mixture (fire suppressant + air) influences the extinguishing concentration, the experiments were carried out where temperature of airflow was decreased up to actually room one. The results obtained are presented in Fig. 4. It was demonstrated that extinguishing concentrations of CF_3Br and OPC depend on the airflow temperature significantly. At decreasing the temperature from 75 to 25 ^oC the extinguishing concentration of CF_3Br decreased from 4.6 to 3.5 %, and for $(CF_3CH_2O)_3P$ from 2.5 to 1.6 %. The nature of the dependence indicates that $(CF_3CH_2O)_3P$ is more effective than CF_3Br not only at studied temperature but in the wider range.

It is noteworthy that extinguishing concentration of $(CF_3CH_2O)_3P$ at 25 °C is 1.6% by volume (Fig. 4) at that 75% of the compound reaches the flame in form of aerosol. So, the results obtained demonstrate a benefit of practical application of fire suppressants with comparatively low volatility in form superfine aerosol. Such compounds seem to have even an advantage over more volatile suppressants, which consists in their slow evaporation from the surfaces of combustible materials, that in some cases can prevent a fire propagation. Besides, these compounds can effectively suppress smoldering.

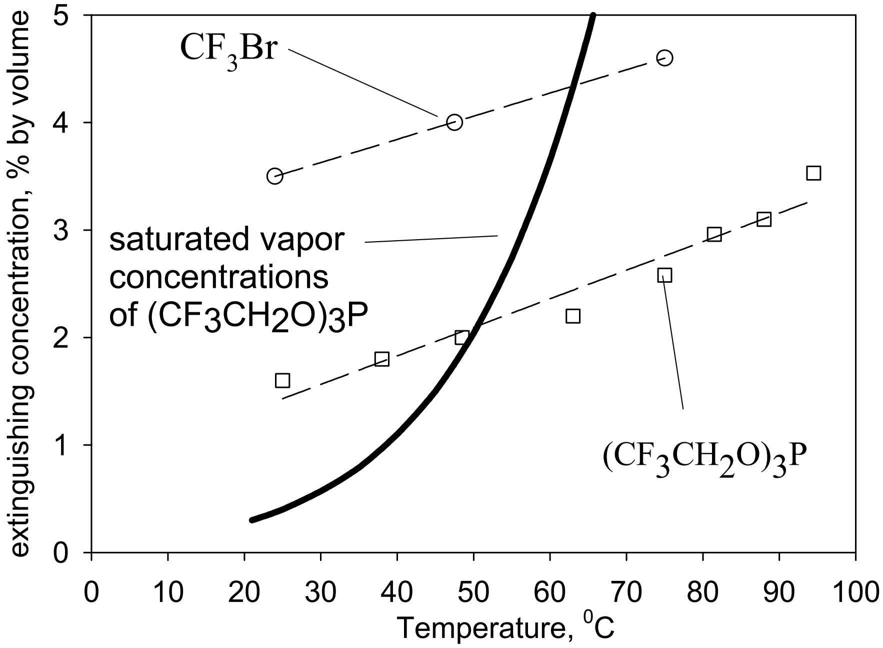

Fig. 4. The dependencies of extinguishing concentration of CF_3Br and $(CF_3CH_2O)_3P$ obtained using cup burner on temperature of extinguishing mixtures (air + fire suppressant).

Following fire suppressants: TMP, *tris-2,2,2-*(trifluoroethyl)phosphite ($(CF_3CH_2O)_3P$) and CF_3Br were tested using TARPF apparatus. We failed to determine minimal extinguishing concentration of TMP reliable. After extinguishment, TMP vapors self-ignited down stream from the burner and reignited propane. The combustibility of TMP with air at elevated temperature was reported earlier [3]. Practically the minimal concentration of TMP, at which reignition did not occur, was of 8.5% by volume at temperature of 90 °C.
The flame suppression by Halon 1301 was performed at temperatures of 16, 32, 39 and 75 °C. The values of minimal extinguishing concentrations of both suppressants are given in Table 4 and Fig. 5.

Table 4. Minimal extinguishing concentrations of CF_3Br and $(CF_3CH_2O)_3P$ at various temperature of suppressing mixtures.

t, ^0C	extinguishing concentration, % by volume	
	CF_3Br	$(CF_3CH_2O)_3P$
16	3.0	–
32	3.5	–
35	–	3.1
39	3.7	–
45	–	3.3
75	4,6	–
86	–	4.3

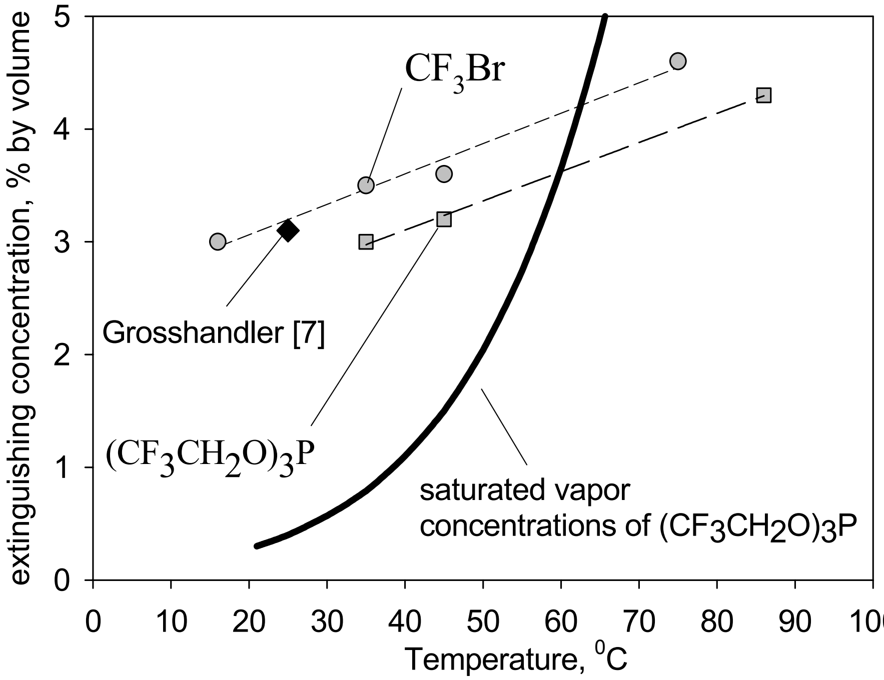

Fig. 5. The dependencies of extinguishing concentration of CF_3Br and $(CF_3CH_2O)_3P$ obtained using TARPF apparatus on temperature of extinguishing mixtures (air + fire suppressant).

Thus, the data obtained for CF_3Br are in a good agreement with data reported in Ref. [7], where extinguishing concentration of CF_3Br was found to be 3.1% by volume at room temperature.
The experiments using TARPF apparatus were performed at 35, 45, and 86 ^oC. The results are shown in Table 4.

The data presented in Fig. 3 indicate the organophosphorus fire suppressant to be more effective than Halon 1301. The temperature dependence of extinguishing concentration is presented in Fig. 4. and Fig. 5. The results for the halon obtained using both techniques are quite close, whereas the data for $(CF_3CH_2O)_3P$ are appreciably different. The extinguishing concentration obtained by cup burner technique is lower.

So, we have demonstrated that a number of fluorinated OPC are more effective even at suppressing a turbulent, baffle-stabilized pool fire at their transient injection. As it is not likely that novel volatile OPC (b.p. <80-100 ^{o}C) can be synthesized, the application of organophosphorus fire suppressants as a streaming agent will be practised using an aerosol technology. An alternative of the aerosol technology may be the application of mixtures where OPC are dissolved in HFC, CO_2, etc.

Later on we are going to extend a number of tested compounds containing phosphorus in order to search for novel effective fire suppressants and to improve techniques for their application.

Assessment of Toxicity

We assessed toxicity of following 6 compounds: $(CH_3O)_2(CH_3)PO$ – dimethyl methylphosphonate (DMMP), $(C_2H_5O)_2(CH_3)PO$ – diethyl methylphosphonate (DEMP), $(CF_3CH_2O)_3PO$ – *tris-2,2,2-*(trifluoroethyl)phosphate, $(CH_3O)_3P$ – trimethylphosphite and $(CF_3CH_2O)_3P$ – *tris-2,2,2-*(trifluoroethyl)phosphite. Toxicological parameters were determined on laboratory mice by intra-stomach administration of OPC in various doses. DMMP and DEMP were dissolved in water just before administration whereas $(CF_3CH_2O)_3PO$, which is poorly dissoluble in water, was administered in form of emulsion with olive oil. Each test series included 4-8 mice. The symptoms of poisoning are similar to those for TMP, which were reported earlier [3]. We have not observed any toxicological action of the compounds on skin and mucous membranes.

The necropsy of dead animals revealed an elevated blood filling of all internal organs. The reason of dying was found to be a cardiac-pulmonary syndrome that resulted in breakdown of circulation of the blood and in all internal organs. The data obtained are summarized in Table 5.

Table 5. Toxicity of organophosphorus compounds.

Compound	Lethal dose (LD), ml/kg			
	LD_{16}	LD_{50}	LD_{84}	LD_{100}
TMP	1.4	2.3±0.35	3.1	3.5
DMMP	–	10.5±0.09	–	–
$(C_2H_5O)_2(CH_3)PO$	–	2.0± 0.22	–	–
$(CF_3CH_2O)_3PO$	–	0.45±0.031	–	–
$(CH_3O)_3P$	2.9	3.5±0.20	4.1	4.3
$(CF_3CH_2O)_3P$	0.19	0.27	0.35	0.39

Besides we assessed the parameters of cumulative toxicity for DMMP, DEMP, trimethylphosphite and $(CF_3CH_2O)_3PO$. For this purpose mice were administered intra-stomachally the substances 5 times a week. During the first week the dose was 1/10 LD_{50}, during the second one 3/10 LD_{50}, during the third one 6/10 LD_{50}. During subsequent weeks the dose was increased in 1.5 times/week. The cumulative dose of the compounds and the loss of the mice were recorded. Basing on the data obtained we calculated the toxicity coefficients (LD_{50}, LD_{16} and LD_{84}) using least-squares procedure and probit analysis. The cumulative coefficient K, which characterizes the cumulative properties of compounds, was estimated. The more the value of K (>1) the higher is cumulative toxicity of a compound. If K of a compound is less than 1 it means that a compound causes adaptation. Cumulative coefficients for trimethylphosphite was found to be 0.20, for $(CF_3CH_2O)_3PO$ - 0.28, for diethyl methylphosphonate - 0.07. The calculated coefficients indicate that these compounds have a very low cumulative toxicity and moreover, their reiterated administration in increasing doses causes tolerance. At assessment of cumulative toxicity of DMMP the cumulative dose of $5 \times LD_{50}$ was administered and here no symptoms of poisoning were observed. All mice remained alive during 2 next weeks of observation after administration of the compound was stopped. Thus assessment of cumulative coefficient (K) of DMMP have not been finished, as it demanded significant amounts of the compound. Nevertheless the toxicological study of the compound made it possible to conclude that DMMP also has very low cumulative toxicity.

We also studied the toxicity of $(CF_3CH_2O)_3P$ at inhalation of its vapors by laboratory mice. After inhalation exposure in the test chamber during 15 minutes at concentration of 0.4% by volume (59 mg/L) at temperature of 25 ^{0}C the mice were flabby but all 5 animals survived and were alive during 5 weeks of observations. However when the temperature inside the chamber was increased up to 30-35 ^{0}C that corresponds to concentration of the compound of 0.68% by volume (100 mg/L) one mouse of 4 mice perished within 15 minutes. The rest mice died in 2 days. The reason of mice's loss was a lesion of lungs and an abnormality of breathing.

Although the toxicity of vapors of *tris-2,2,2*-(trifluroethyl)phosphite at inhalation is not assessed thoroughly it is clear that they are appreciably more harmful than the compound at intra-stomach administration.

CONCLUSIONS AND FUTURE PLANS

Nine novel fluorinated organophosphorus compounds have been synthesized and their fire suppression effectiveness have been determined using laboratory cup-burner technique and scaled-up bench test (only for $(CF_3CH_2O)_3P$), which consists in extinguishment of the turbulent pool fire by transient injection of superfine aerosol (<1 micron) of tested compound. Obtained results demonstrated that following fluorinated OPC - $(C_4F_9O)_3PF_2$, $[(CF_3)_2CHO]_2P(O)C_2H_5$, $[(CF_3)_2CHO]_3P$, $(CF_3CH_2O)_2P(O)CF_3$, $(CF_3CH_2O)_3P$ - are more effective fire suppressants than CF_3Br and have acceptable toxicity, air and water vapor stability. Reasoning about the practical application of OPC we came to conclusion that it is difficult to expect the preparation of highly volatile OPC. Though their application as streaming agents may require the use of advanced aerosol technology, which provide fine aerosol.

The toxicity (lethal doses and cumulative toxicity) for 4 compounds was assessed. The results obtained revealed that they have acceptable toxicity.

Further efforts should be directed to synthesize fluorinated organophosphorus nonflammable compounds and investigate their effectiveness using cup-burner technique. Besides study of fire suppression by mixtures of fluorinated OPC with ozone-friendly halons is of a great interest. Further tests of promising fluorinated OPC using TARPF apparatus and fine aerosol delivering system will be continued. To evaluate the perspectives of OPC practical application the large-scaled tests of efficiency of fluorinated OPC using large test chamber about 1 m^3 in volume will be carried out. OPC incorporated in propellant gas generator compositions will be tested.

The toxicity of novel and the most promising fluorinated OPC including investigation of inhibition activity of some compounds *in vitro* of choline esterase will be assessed.

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