

Study of Inhibition and Extinguishment of Diffusion Flames by Organophosphorus and Organofluoric Compounds

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Abstract

Inhibition and extinguishing effectiveness of diffusion flame by new organophosphorus compounds (OPCs), organofluoric compounds (OFCs) and some metal-containing compound (MCCs) as halons replacements was studied using cup-burner technique. Some of the most promising compounds were tested with the help of Transient Application, Recirculating Pool Fire (TARPF) apparatus which simulates the fire of a jet engine inside the nacelle. The fire extinguishing effectiveness determined using by TARPF apparatus was compared with that measured using cup-burner technique. Halon 1301 (CF₃Br) was used as a comparison standard in all tests. The experiments demonstrated a possibility of practical application of organophosphorus fire suppressants in form of fine-dispersed aerosol.

Introduction

Search and study of novel effective and ecologically safe fire suppressants is one of foreground goal of combustion science. In this connection organophosphorus (OPCs), organofluoric (OFCs) and metal-containing compounds (MCCs) are of interest. In spite of at present the results of comparative testing of various flame inhibitors are available [1-10] there is a deficit in experimental data on minimal extinguishing concentration (MEC) for many compounds and especially composite fire suppressants. In the first turn it relates to non-volatile compounds. This parameter (MEC) makes possible to evaluate the perspectives of practical application of a compound properly. We believe that search and testing of novel fire suppressants - halon alternatives among OPCs, OFCs and MCCs still presents a perspective direction of investigation in area of fire extinguishing. High boiling point (low volatility) of studied OPCs brought us to synthesis of more volatile fluorinated derivatives of OPCs. However a high reactivity of some of fluorinated OPCs, which were recently synthesized [1-5], hinders their practical use. Nevertheless, the application of moderately volatile OPCs 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 extinguishing by various OPCs in

laboratory conditions were published in [6-10]. 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 extinguishing effectiveness. Thus, in spite of a appreciable theoretical and experimental knowledge accumulated on organophosphorus fire suppressants no bench tests on apparatus like [11] 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 OPCs, OFCs and their mixtures at inhibition and extinguishment of different types of flames in laboratory and bench tests.

Experimental

In present paper we studied novel OPCs and OFCs. Their formulas and boiling points are presented in Table 1. In addition we studied the extinguishment of n-heptane flame by fine dispersed aerosol of inorganic and organic salts (K₃PO₄, KOOCH₃, KOCCOOK and K₄[Fe(CN)₆]). The salts were introduced into the air flow in form of aqueous solutions.

The fire extinguishing effectiveness and minimal extinguishing concentrations of the compounds of diffusive heptane/air flame were measured using the cup-burner technique (Fig. 1). It represents the reduced variant of the standard cup burner with small constructive changes. n-Heptane was used as fuel. The cup burner consists of a pyrex tube, which is used as a chimney, of

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Table 1. Novel OPCs and OFCs tested and their boiling points

Formula	B.p. at pressure [Torr], experimental data	B.p. at pressure 760 Torr
$(\text{CF}_3\text{CH}_2\text{O})_2\text{P}(\text{O})\text{CF}_3$	147-148/760	147-148
$(\text{CF}_3\text{CH}_2\text{O})_2\text{P}(\text{O})\text{OCH}(\text{CF}_3)_2$	83/10	210 ^a
$(\text{CF}_3)_3\text{P}$	45/760	47
$\text{CF}_3\text{CH}_2\text{OP}(\text{O})[\text{OCH}(\text{CF}_3)_2]_2$	77/15	190 ^a
$[(\text{CF}_3)_2\text{CHO}]_3\text{P}$	60/55	140 ^a
$[(\text{CF}_3)_2\text{CHO}]_2\text{P}(\text{O})\text{C}_2\text{H}_5$	65-67/65	135 ^a
$(\text{CF}_3\text{CH}_2\text{O})_2\text{P}(\text{O})\text{C}_2\text{H}_5$	70-72/70	135 ^a
$(\text{C}_4\text{F}_9\text{O})_3\text{PF}_2$	180/760	180
$(\text{CF}_3\text{CH}_2\text{O})_3\text{P}$	131/760	131
$[(\text{CF}_3)_2\text{CHO}]_2\text{P}(\text{O})\text{OCH}_3$	93/50	180 ^a
$[(\text{CF}_3)_2\text{CHO}]_2\text{P}(\text{O})\text{CF}_3$	135/760	135
$\text{HCF}_2\text{CF}_2\text{CH}_2\text{OCF}_2\text{CHFCF}_3$	80/760	80
$\text{HCF}_2\text{CF}_2\text{OCH}_2\text{CH}_3$	82/760	82
$\text{CF}_3\text{CF}_2\text{CF}_2\text{OCHFCF}_3$	70/760	70
$(\text{CF}_3)_2\text{C}=\text{CFCF}_2\text{CF}_3$	42/760	42

^a Estimated

an internal diameter 5.5 cm and length about 65 cm. The lower part of the chimney was heated electrically. The rate of airflow was about 10 slpm. OPCs was introduced into the air flow using a nebulizer. OPCs feeding in to the nebulizer was performed by a syringe pump that made it possible to vary the OPCs concentration in the wide range.

The volume rate of air through nebulizer at pressure of 0.6 MPa was 1.0-0.95 slpm. The air flow with OPCs particles went upwards and was heated. The cup has conic expansion upwards with an angle 30°. The airflow velocity near the cup was about 10 cm/s. The diameter of the cup at the top edge - 13 mm. The feeding of OPCs through nebulizer was begun after ignition of a flame and establishment of its stationary height 4.5-5 cm in 2 min. The CO₂ flow was introduced in the airflow in discrete steps so long as the flame was not extinguished. Such method of testing described in Ref [9], allows to compare suppression efficiency of various fire suppressants in a wide range of loadings and to determine extinguishing concentrations of fire suppressants more precisely.

The solutions of the salts were introduced into the heated air flow 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. The deposition

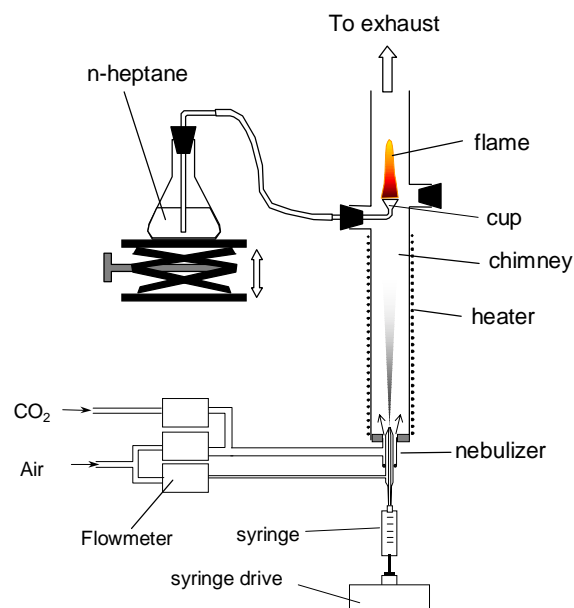


Fig. 1. Cup-burner setup.

of the salts inside the chimney were taken into account. The contribution of water from the solutions into the inhibition effect was also taken into account.

A part of cup burner experiments were performed at constant airflow temperature of 75°C that provided complete evaporation of the OPCs droplets. For the experiments, which were carried out at reduced temperature 25 – 65°C 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 of OPCs, the air with aerosol was sampled from the flame region. 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

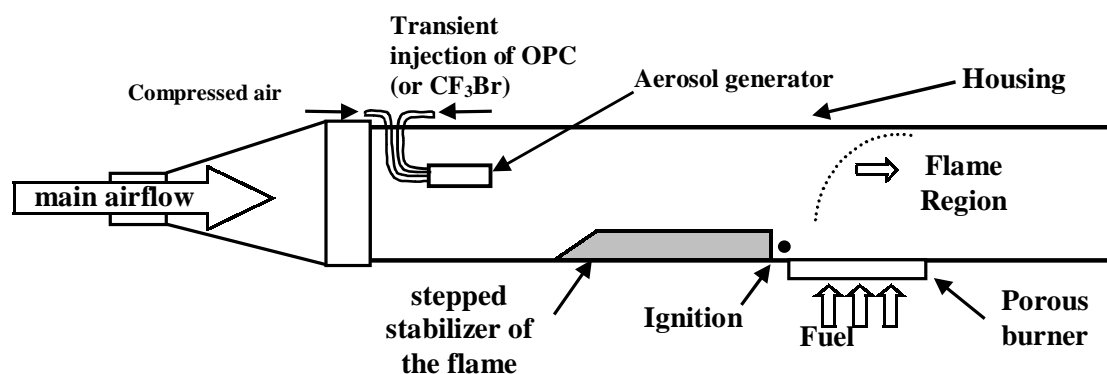


Fig. 2. A scheme of Transient Application, Recirculating Pool Fire (TARPF) Apparatus.

designed by Grosshandler et al. [11], which simulates the fire of a jet engine inside the nacelle was fabricated. The apparatus of the same size as in Ref.[11] is shown schematically in Fig. 2. The apparatus includes a wind tunnel of square cross section, 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 OPCs the generator of superfine aerosol of original design, which provides very high concentration up to 10% (by volume) of OPCs, 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 $Re \geq 10000$ 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 $40 \text{ cm}^3/\text{s}$), which was ignited by a spark-plug. The authors [11] 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 $40 \text{ cm}^3/\text{s}$. The process of flame extinguishing was observed visually and recorded by video camera. The extinguishing concentration of an agent was shown in Ref. [11] 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

A number of novel OPCs, OFCs and MCCs were tested using the cup-burner technique. Figure 3 shows the extinguishing concentration of CO_2 as a function of the dopant loading. The measured extinguishing concentrations for the tested compounds are tabulated below. 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 OPCs and OFCs a distinctive dependence of CO_2 extinguishing concentration on suppressants loading is observed (see Fig. 3). For example, when suppressants 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 suppressants 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 suppressants combustion that counteracts flame extinguishing.

Table 2. Extinguishing concentrations of novel OPCs and OFCs at 75°C .

Compound	Extinguishing concentration, % by volume	Extinguishing concentration, g/m^3	Air stability
$(\text{C}_4\text{F}_9\text{O})_3\text{PF}_2$	1.8 ± 0.2	621	Some fumes
$[(\text{CF}_3)_2\text{CHO}]_2\text{P}(\text{O})\text{C}_2\text{H}_5$	2.0 ± 0.2	366	No fumes
$[(\text{CF}_3)_2\text{CHO}]_3\text{P}$	2.2 ± 0.2	523	No fumes
$(\text{CF}_3\text{CH}_2\text{O})_2\text{P}(\text{O})\text{CF}_3$	2.3 ± 0.2	322	No fumes
$(\text{CF}_3\text{CH}_2\text{O})_3\text{P}$	2.6 ± 0.2	381	No fumes
$(\text{CF}_3)_3\text{P}$	flammable		Some fumes
$(\text{CF}_3\text{CH}_2\text{O})_2\text{P}(\text{O})\text{OCH}(\text{CF}_3)_2$	No extinguishing at 3%		No fumes
$\text{CF}_3\text{CH}_2\text{OP}(\text{O})[\text{OCH}(\text{CF}_3)_2]_2$	flammable		No fumes
$(\text{CF}_3\text{CH}_2\text{O})_2\text{P}(\text{O})\text{C}_2\text{H}_5$	flammable		No fumes
$[(\text{CF}_3)_2\text{CHO}]_2\text{P}(\text{O})\text{CH}_3$	3.0 ± 0.2	530	No fumes
$[(\text{CF}_3)_2\text{CHO}]_2\text{P}(\text{O})\text{CF}_3$	2.0 ± 0.2	366	No fumes
$\text{HCF}_2\text{CF}_2\text{CH}_2\text{OCF}_2\text{CHF}_2\text{CF}_3$	6.1 ± 0.3	768	No fumes
$\text{HCF}_2\text{CF}_2\text{OCH}_2\text{CH}_3$	flammable		No fumes
$\text{CF}_3\text{CF}_2\text{CF}_2\text{OCH}_2\text{CF}_3$	6.6 ± 0.3	843	No fumes
$(\text{CF}_3)_2\text{C}=\text{CFCF}_2\text{CF}_3$	4.8 ± 0.3	642	No fumes
CF_3Br	4.6	306	

Thus, a different effectiveness of various suppressants can be explained by influence of several reasons: the heat of formation of suppressants, heat capacity of their vapors, their destruction rate and destruction products in a flame. The results presented in Table 2 and Figure 3 indicate that under the same conditions the effectiveness of some OPCs 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 OPCs depend on the airflow temperature

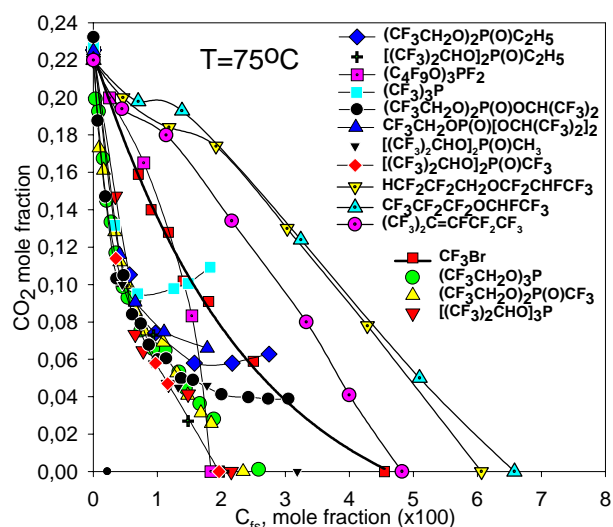


Fig. 3. Cup burner tests: extinguishing concentration of CO_2 as a function of loading of fire suppressants. significantly. At decreasing the temperature from 75 to

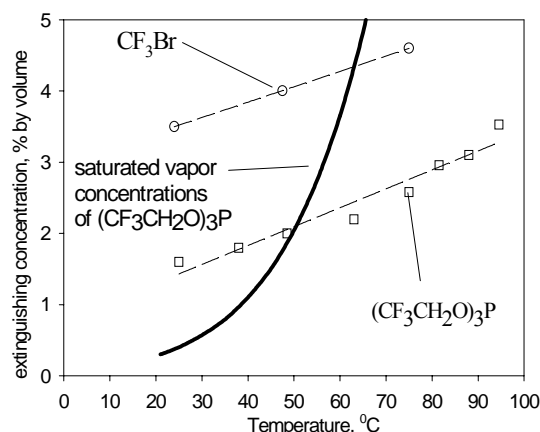


Fig. 4. The dependencies of extinguishing concentration of CF_3Br and $(\text{CF}_3\text{CH}_2\text{O})_3\text{P}$ obtained using cup burner on temperature of extinguishing mixtures (air + fire suppressant).

25 °C the extinguishing concentration of CF_3Br decreased from 4.6 to 3.5 %, and for $(\text{CF}_3\text{CH}_2\text{O})_3\text{P}$ from 2.5 to 1.6 %. The nature of the dependence indicates that $(\text{CF}_3\text{CH}_2\text{O})_3\text{P}$ is more effective than CF_3Br not only at studied temperature but in the wider range.

It is noteworthy that extinguishing concentration of $(\text{CF}_3\text{CH}_2\text{O})_3\text{P}$ 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.

The results on extinguishing of diffusive cup-burner flame (n-heptane + air) by aqueous solutions of various salts are presented in Table 3. The values of MMC obviously demonstrate potassium salts to be on order of magnitude more effective fire suppressants than OPCs and halons [9]. The data obtained indicate that the suppression effectiveness counting on one molecule of organic salts is actually proportional to number of potassium atoms the effect of water being taking into account. This rule is not correct for $\text{K}_4[\text{Fe}(\text{CN})_6]$ containing iron atoms except potassium ones. Iron-containing compounds are known to be one of the most effective inhibitors of combustion [9]. In the case of $\text{K}_4[\text{Fe}(\text{CN})_6]$ iron and potassium act reciprocally suppressing the flame. The results obtained do not give a possibility to reveal synergy of iron and potassium but this will be the main goal of future research.

It is noteworthy that a combined application of phosphorus and potassium (as potassium phosphates – KH_2PO_4 and KOOCH_3 solution with OPCs additive) for flame suppression is as effective as inert agent. The suppression effectiveness of the composition is very low, so we failed to extinguish the cup-burner flame. Nevertheless earlier [5] we demonstrated that

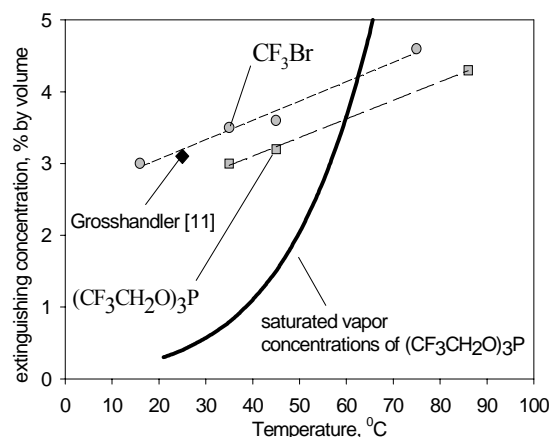


Fig. 5. The dependencies of extinguishing concentration of CF_3Br and $(\text{CF}_3\text{CH}_2\text{O})_3\text{P}$ obtained using TARP apparatus on temperature of extinguishing mixtures (air + fire suppressant).

$\text{NH}_4\text{H}_2\text{PO}_4$ and OPCs at loading of 0.2% by volume demonstrate nearly the same effectiveness of suppression counting on one atom of phosphorus. Thus the expected effectiveness of K_3PO_4 must be much higher than that for OPCs but it is not confirmed experimentally. Thermally stable potassium phosphate (K_3PO_4) do not dissociate effectively producing chemically active species of potassium and phosphorus oxy-acids. To understand the detailed mechanism for mutual decrease of effectiveness K- and P-containing inhibitors additional research is required. Thus the practical application of such combined fire suppressants has no perspectives.

Table 3. The studied salts and their minimal extinguishing concentration

Salt	minimal extinguishing concentration	
	mole fraction ($\times 100$)	g/m^3
K_3PO_4	No extinguishing at 1%	
KOOCH_3	0.25	10.9
KOOCCOOK	0.13	9.6
$\text{K}_4[\text{Fe}(\text{CN})_6]$	0.035	6.6

Following fire suppressants: $(\text{CH}_3\text{O})_3\text{PO}$ (TMP), *tris*-2,2,2-(trifluoroethyl)phosphite $((\text{CF}_3\text{CH}_2\text{O})_3\text{P})$ and CF_3Br were tested using TARP 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 extinguishing 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.

Thus, the data obtained for CF_3Br are in a good agreement with data reported in Ref. [11], where extinguishing concentration of CF_3Br was found to be

Table 4. Minimal extinguishing concentrations of CF₃Br and (CF₃CH₂O)₃P at various temperature of suppressing mixtures.

t, °C	extinguishing concentration, % by volume	
	CF ₃ Br	(CF ₃ CH ₂ O) ₃ P
16	3.0	–
32	3.5	–
35	–	3.1
39	3.7	–
45	–	3.3
75	4.6	–
86	–	4.3

3.1% by volume at room temperature. The experiments using TARP apparatus were performed at 35, 45, and 86 °C. 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 (CF₃CH₂O)₃P are appreciably different. The extinguishing concentration obtained by cup burner technique is lower.

So, we have demonstrated that a number of fluorinated OPCs are more effective even at suppressing a turbulent, baffle-stabilised pool fire at their transient injection. As it is not likely that novel volatile OPCs (b.p. <80-100 °C) can be synthesised, 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 OPCs are dissolved in fluorinated hydrocarbon (HFCs), organofluoric compounds, iodine-containing alkanes, CO₂, etc.

Later on we are going to extend a number of tested compounds containing phosphorus and their mixtures with fluorinated hydrocarbon (HFCs), organofluoric compounds, iodine-containing alkanes and metalloorganic compounds in order to search for novel effective fire suppressants and to improve techniques for their application.

Assessment of Toxicity

We assessed toxicity of following 7 compounds: (CH₃O)₂(CH₃)PO – dimethyl methylphosphonate (DMMP), (C₂H₅O)₂(CH₃)PO – diethyl methylphosphonate (DEMP), (CF₃CH₂O)₃P – *tris*-2,2,2-(trifluoroethyl)phosphate, (CH₃O)₃P – trimethylphosphite, (CF₃CH₂O)₃P – *tris*-2,2,2-(trifluoroethyl)phosphite and (C₄F₉O)₃PF₂. Toxicological parameters were determined on laboratory mice by intra-stomach administration of OPCs in various doses. DMMP and DEMP were dissolved in water just before administration whereas (CF₃CH₂O)₃P, 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. It was shown for the most

Table 5. Toxicity of organophosphorus compounds.

Compound	Lethal dose (LD ₅₀), ml/kg
(CH ₃ O) ₃ PO, TMP	2.3±0.35
(CH ₃ O) ₂ (CH ₃)PO, DMMP	10.5±0.09
(C ₂ H ₅ O) ₂ (CH ₃)PO	2.0±0.22
(CF ₃ CH ₂ O) ₃ P	0.45±0.031
(CH ₃ O) ₃ P	3.5±0.20
(CF ₃ CH ₂ O) ₃ P	0.27
(C ₄ F ₉ O) ₃ PF ₂	2.2

of OPCs that they non-toxic compound, which LD₅₀=2÷10 ml/kg is comparable with that of NaCl 3 g/kg. The data obtained are summarized in Table 5. We also studied the toxicity of (CF₃CH₂O)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 °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 °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-(trifluoroethyl)phosphite at inhalation is not assessed thoroughly it is clear that they are appreciably more harmful than the compound at intra-stomach administration.

Summary

The number of novel fluorinated organophosphorus and organofluoric compounds have been synthesised and their fire extinguishing effectiveness have been determined using laboratory cup-burner technique and scaled-up bench test (for (CF₃CH₂O)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 OPCs – (C₄F₉O)₃PF₂, [(CF₃)₂CHO]₂P(O)C₂H₅, [(CF₃)₂CHO]₃P, (CF₃CH₂O)₂P(O)CF₃, (CF₃CH₂O)₃P, [(CF₃)₂CHO]₂P(O)CH₃, [(CF₃)₂CHO]₂P(O)CF₃ – are more effective fire suppressants than CF₃Br and have air and water vapor stability. The toxicity (lethal doses) for 7 compounds was assessed. The results obtained revealed that they have acceptable toxicity.

Reasoning about the practical application of OPCs we came to conclusion that it is difficult to expect the preparation of highly volatile OPCs. Though their application as streaming agents may require the use of advanced aerosol technology, which provide fine aerosol.

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