STUDY OF EFFECT OF ORGANOPHOSPHORUS COMPOUNDS ON PROPAGATION LIMITS AND EXTINCTION CONDITIONS OF HYDROCARBON FLAMES

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ABSTRACT

Minimum extinguishing concentration for various mixtures based on organophosphorus compounds with inert agents and iodine-containing compounds for suppression of *n*-heptane flame was evaluated using the cup burner and "cylinder" techniques. The effective mixtures demonstrating synergism were developed. The lower temperature limit of the mixtures application was estimated.

By using symmetrical counterflow configuration, the effect of $(CH_3O)_3PO$ and $(CF_3CH_2O)_3P$ additives at various concentrations on lean propagation limit of a premixed flame CH_4/Air was studied. The lean propagation limit of the flame is determined by extrapolating the dependence of extinction strain rate on methane concentration in the mixture to zero value of extinction strain rate. The obtained experimental data are compared with those for CF_3Br and CF_3Br data available from the literature as well as with modeling results. The peculiarities of effect of the inhibitors on lean propagation limit of a CH_4/Air flame are discussed.

Ozone depletion potential and global warming potential for $(CF_3CH_2O)_3P$ are estimated using mathematical model of middle atmosphere and other estimation methods for potentials of ozone depletion. The calculations showed that this compound does not affect the ozone layer of the atmosphere and climate warming.

INTRODUCTION

At present time there are data available on comparative testing of organophosphorus compounds (OPCs) as flame inhibitors and fire suppressants [1-10] but there is a lack of results on the effectiveness of OPCs-based blends. The perspectives of application of such compositions consist in a possibility to overcome problems connected with OPCs physical properties. The limitation of the most OPCs tested is their relatively high boiling point that impedes their application as individual substances because at normal conditions OPCs vapor concentration is less than their minimal extinguishing concentration (MEC). A possible way of solving this problem may be the application of aerosol technology for delivering the fire suppressants. Aerosol particles mainly evaporate in the flame front and fire suppressant reaches combustion zone as vapor. But aerosol technology has some limitations. Usually nebulizers and nozzles provide formation of aerosol with mass-averaged particle size of 100-500 microns. However, these particles gravitate rapidly that results in losses and over-expenditure of the fire suppressants. Producing an aerosol with smaller particles requires application of more complex atomizers or aerosol generators that seems to be not reasonable from practical point of view because of low reliability and high cost of these devices. An alternative for aerosol technology can be the application of blends of fire suppressants, which components provide superatmospheric pressure for extruding and delivering of the active component, reducing oxygen concentration near the fire source, decreasing the flame temperature. The application of these blends does not require modification of currently used fire extinguishers and fire-fighting systems. The concentration of the active component in such blends is sufficiently low that improves their characteristics in comparison with those of individual fire suppressants as lower toxicity, high reactivity, resistance to air and water and higher effectiveness of fire suppression. The most of OPCs were shown to dissolve in CO_2 [8] and in halon $C_2F_4H_2$, which can be used as a medium for delivering of chemically active components of fire-suppressing blend.

Blends of OPCs with HFC (e.g. CF_3H) were shown to demonstrate the effectiveness equal to sum of the effectiveness of each component [11], i.e. the components provide additive effect. OPCs with salts of alkaline metals (e.g. $K_2C_2O_4 \bullet H_2O$) decrease the effectiveness of one another. Thus, both of these types of blends were shown to be ineffective.

Iodine-containing compounds (CF_3I, CH_3I, C_2F_5I, $C_2F_4I_2$) are also known to be effective inhibitors and fire suppressants [12,13]. The mechanism of their action as similar to that of OPCs: iodine atom formed in a flame reacts with H producing HI and thus terminating the radical chains that results in flame extinguishing. The interaction of fire suppressants like OPCs and iodine-containing compounds was not studied that is testing of such blends is of interest.

The goal of the present study consists in a search for blends of fire suppressants based on OPCs, inert agents and iodine-containing compounds, determination of optimal concentration of each component by measuring of MEC using cup-burner and "cylinder" techniques. Besides, OPCs effect on limits of flame propagation was studied and ODP and GWP for $(CF_3CH_2O)_3P$ were estimated.

EXPERIMENTAL

Following earlier investigated OPCs were used for blends preparation: $(CF_3CH_2O)_3P$ (tris(2,2,2-trifluoroethyl)phosphite - FTEP**)**, $(CH_3O)_3P$, $(CH_3O)_3PO$ (trimethylphosphate – TMP). As a iodine-containing additive methyliodide (CH_3I) was used. The choice of these OPCs is justified by their low boiling points among all phosphates and phosphites studied [4,11]. Table 1 presents boiling points and MECs of fire suppressants. CO_2 as well as N_2 were used in the most of experiments as inert agents. MECs of blends and individual components were determined using experimental setup based on cup burner. A scheme of the setup and experimental technique was described elsewhere [3]. Liquid OPCs and CH_3I were fed into a gas flow using a nebulizer. To prevent losses of OPCs on inner walls of the setup the lower part of the chimney was heated electrically. The temperature of the gas flow near the cup was 75 ^{0}C. *n*-heptane was used as a fuel.

Table 1. Boiling points and MECs of studied fire suppressants.

Compounds	T_b, ^oC	MEC, % by vol.	MEC, g/m^3
$(CH_3O)_3P$	111	- *	-
$(CH_3O)_3PO$, TMP	181	- *	-
$(CF_3CH_2O)_3P$, FTEP	131	2.6±0.2	381
CH_3I	42.5	4.1±0.2	260

* - combustible compound

OPCs-based blends were also tested using "cylinder" method and setup and technique described earlier [12]. Experiments were carried out using 2 setups differing by the volume of the test chambers. A reduced variant of setup (#1) with chamber 22.4 L (ID=0.25 m) was fabricated and was used for preliminary experiments in Institute of Chemical Kinetics and Combustion, (Novosibirsk, Russia). Another series of experiments was carried out on the setup having the volume 53 L and diameter 0.38 m (#2), which was fabricated at All-Russian Scientific Research Institute for Fire Protection. The test chamber of the setup is a cylinder and made of steel. The setup is equipped with vacuum pump providing a residual pressure of 1-2 Torr and system for preparation of gaseous mixtures. The mixtures of required composition are prepared according to partial pressure of a component. The loading of OPCs and CH_3I was determined by mass (volume) of liquid agent injected into the chamber with the help of a syringe through a vacuum seal. Then gaseous components are introduced into the chamber in order of increasing of their fraction. The test chamber of setup #1 is equipped with a fan to provide stirring of the gas mixture. A steel cup with inner diameter of 40 mm and height of 23 mm filled with *n*-heptane was used as a fire source. The flame was ignited and then was introduced into the chamber filled with prepared gas mixture. A moment of extinguishing was determined by visual observation. Experimental results are plotted as time of extinguishing versus concentration of fire suppressant (see a typical example in Fig. 3). According to the technique MEC corresponds to the time of extinguishing equal 10 s. In the chamber filled with air the fire after its introduction goes out in 2 min (for setup #1).

The cup-burner technique was used for preliminary search for the most effective blends of fire suppressants, as this method requires fewer amounts of chemicals. The "cylinder" method was applied to verify preliminary cup-burner results. Besides both fundamentally different techniques (fire suppression in co-flow and motionless atmosphere) make possible to measure MEC reliably because in practice both scenario of fire occur.

The symmetrical counterflow flame configuration was used to determine the effect of the inhibitors on lean flammability limit for CH_4/air mixtures. For this purpose, the technique proposed previously by Law and Egolfopoulos [15] was applied. Two symmetrical, planar, premixed, stretched flames were stabilized on an opposed-jet burner detailed previously [16]. The burner nozzles were straight quartz tubes (ID=16 mm) with a separation distance of 10 mm between them. The nitrogen flow was used as a sheath. The flow rates of the gases were set with an accuracy of about 1% and controlled by mass flow controllers (MKS Instruments) which were operated by PC. By continuously increasing the strain rate, the flames approach each other and extinction of the flame eventually occurs at a distinct extinction strain rate K_{ext}. In accordance with the technique proposed in [15], at first, the dependence of K_{ext} on methane concentration in the lean mixture [CH_4] was determined. Then, by plotting K_{ext} versus [CH_4], a limiting concentration was obtained through linear extrapolation to K_{ext}=0, thus, crossing point is the flammability limit. To obtain repeatable values for K_{ext} for the given methane concentration, the measurements of every point on the plot K_{ext}([CH_4]) were performed at least 3 times. The relative accuracy in lean flammability limit determination was ~ 5% in our measurements. The values of K_{ext} were evaluated using global parameters as the sum of velocities on nozzle exits divided by nozzle separation distance.

The measurements for the determination of lean flammability limits were performed for CH_4/air flames doped with small amounts of the inhibitors and without additives. The measurements were also carried out for the flame doped with CF_3Br to compare the inhibition effects. The dopants were added in both mixture flows using a saturator (for OPC) or by flow addition (for CF_3Br). The temperature of saturator was controlled by a thermostat to provide a desired concentration of the inhibitor vapors in the mixture. The combustible mixture streams were maintained at 90^{o}C in order to prevent the inhibitors from condensing on the walls of the burner tubes.

RESULTS AND DISCUSSION

Cup-burner tests

To determine optimal ratios of the components, which provide maximal suppressing effectiveness, binary blends of OPCs + CO_2, OPCs + N_2, OPCs + CH_3I, CO_2 + CH_3I were tested varying the fraction of the components.

In Figure 1 dependencies of MEC of a component (e.g. CO_2 or CH_3I) on loading of the second component of the blend (FTEP or CH_3I) are shown. The data obtained demonstrate that the dependencies are non-linear, i.e. a synergetic interaction between the components is observed. On the base of the data obtained the interaction index (F) was calculated according to the following formula [14]:

$$F = \frac{C^A}{C_0^A} + \frac{C^B}{C_0^B}$$

where C_0^A and C_0^B MECs of the components, C^A, C^B - fraction of the components in the blend. The dependence of index F on concentration of one of the components is shown in Fig. 2.

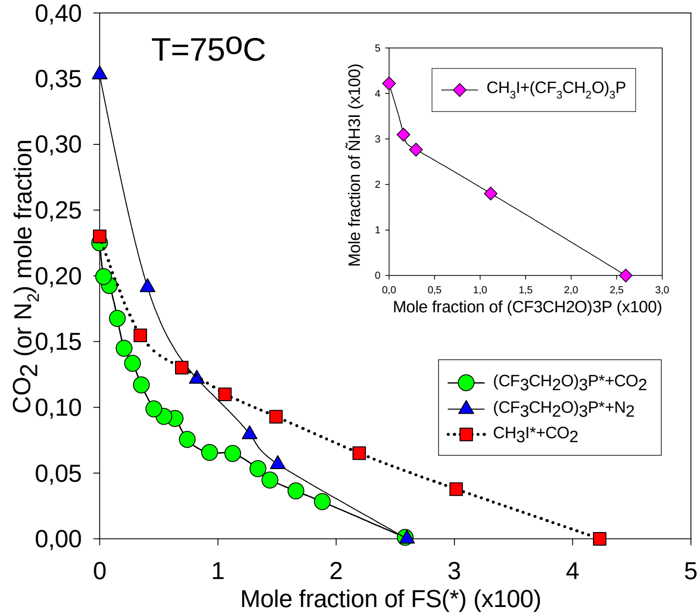

Figure 1. Cup burner tests: extinguishing concentration of CO_2 as a function of loading of fire suppressants.

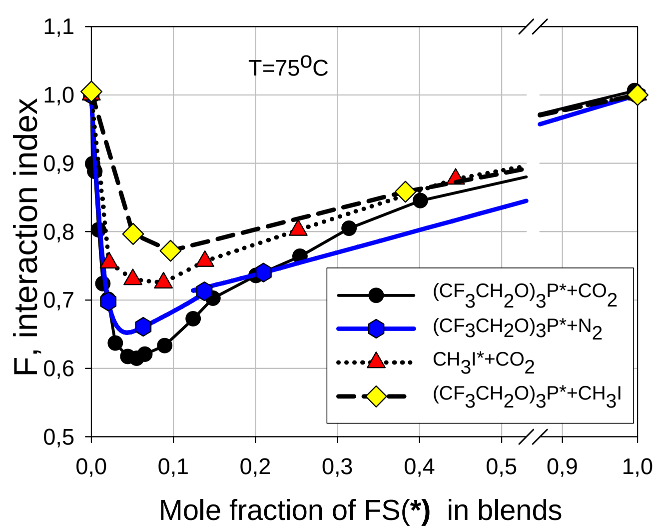

Figure 2. Interaction index for blends $(CF_3CH_2O)_3P$ with CO_2, N_2 or CH_3I.

A minimum of the dependence corresponds to the optimal composition from point of view of synergism. At F=1 the components act additively but at F<1 a positive synergetic effect is observed. It is noteworthy that optimal composition of the blends FTEP + CO_2 and FTEP + N_2 corresponds to FTEP fraction 4-5% by volume. The blend of FTEP with CO_2 demonstrates stronger synergetic effect than that with N_2. For the blends CH_3I+CO_2 and CH_3I+FTEP the optimal composition corresponds to fraction of 9-10% CH_3I and FTEP correspondingly by volume. Assuming that composition of the most effective triple blend corresponds to ratio of components in binary blends, we can propose some conditions for determination of optimal composition for a triple blend. For example, for CO_2 optimal fraction lies between 90 and 95% by volume, for CH_3I and FTEP the ratio is 1:10 –1:20. Taking into account that CH_3I is more volatile than FTEP and to provide the lower temperature limit of the blend application and lower MEC, the blends with maximal CH_3I concentration are preferable. Thus relatively simple criteria allow to find optimal blends composition.

As in binary blends (with CO_2) optimal loading of OPCs is 4-5% that corresponds OPCs loading 0.5-0.4% in air at fire suppression by this blend, the inflammability of some compounds (observed in laboratory experiments at loading of OPCs about 1.5% by volume) is not an obstacle for their use. To verify this suggestion 2 non-fluorinated OPCs - TMP and $(CH_3O)_3P$ were tested. Measured MECs of individual compounds and blends on their base are presented in Table 2.

Table 2. MECs of fire suppressants and blends measured using cup-burner technique at 75^{0}C.

№	Fire suppressant or composition of blend (% by vol.)	MEC of fire suppressant or blend in air, % by vol.	MEC of fire suppressant or blend in air, g/m^3 (estimated at T=25^{o}C)
1	CO_2	21-22	432
2	N_2	35,0	437
3	CF_3Br	4.6	306
4	CH_3I	4.1	260
5	FTEP	2.6	380
6	CO_2+FTEP (96:4)	10.5	267
7	CH_3I+FTEP (95:5)	3.6	**243**
8	CH_3I+TMP (95:5)	3.06	**226**
9	CO_2+CH_3I+FTEP (87:12.4:0.6)	11.5	297

"Cylinder" tests

MECs of a number of blends chosen by results of cup-burner tests and interaction index calculation were measured. The results obtained are shown in Fig. 3 and Table 3. In Table 3 the evaluated minimal temperature of application of these blends was presented also. This value indicates a minimal temperature at which condensation of even one component of the blend. The values were estimated assuming that a MEC at minimal temperature is about the same as that at 75^{0}C and 20^{0}C for cup-burner and "cylinder" methods respectively. We believe that this assumption results in a somewhat lower temperature of blend application because earlier [5] we demonstrated a decrease of MEC of FTEP with decrease of the temperature. So the temperature limits for blends application are given with a reserve. It was shown that MECs of the most effective binary blends were shown to be 210-280 g/m^3 and MECs of triple blends CO_2 + FTEP + CH_3I lie in the range 182-208 g/m^3. The results obtained demonstrate that at application of these blends their mass flow is in 1.3-2 times less than that of CO_2. Substitution of fluorinated OPC FTEP for $(CH_3O)_3P$ slightly reduces the effectiveness of the blend that is connected with combustibility of $(CH_3O)_3P$ [3].

An addition of CH_3I to CO_2 + FTEP blend not only reduces its MEC but appreciably decreases low temperature limit of it application that is very important result. An addition of 0.6 and 1.3% by volume of FTEP to a mixture of CO_2 + CH_3I increases the effectiveness of the blend (by volume) for 13 and 22% (by mass – for 9.5 and 21% respectively). Thus application of CH_3I and OPCs as an additive to CO_2 was shown to be very effective. Mutual solubility of the components allows applying these blends using fire-fighting means in which CO_2 is used. Application of N_2 instead CO_2 does not give any advantages as N_2 can be used as compressed gas only that does make possible to prepare homogeneous mixture of OPCs and CH_3I.

A comparison of data obtained using cup-burner and "cylinder" techniques revealed a good agreement especially if take into account different initial temperature of air (see Fig.4). So, both techniques provide the close results of MECs measurement.

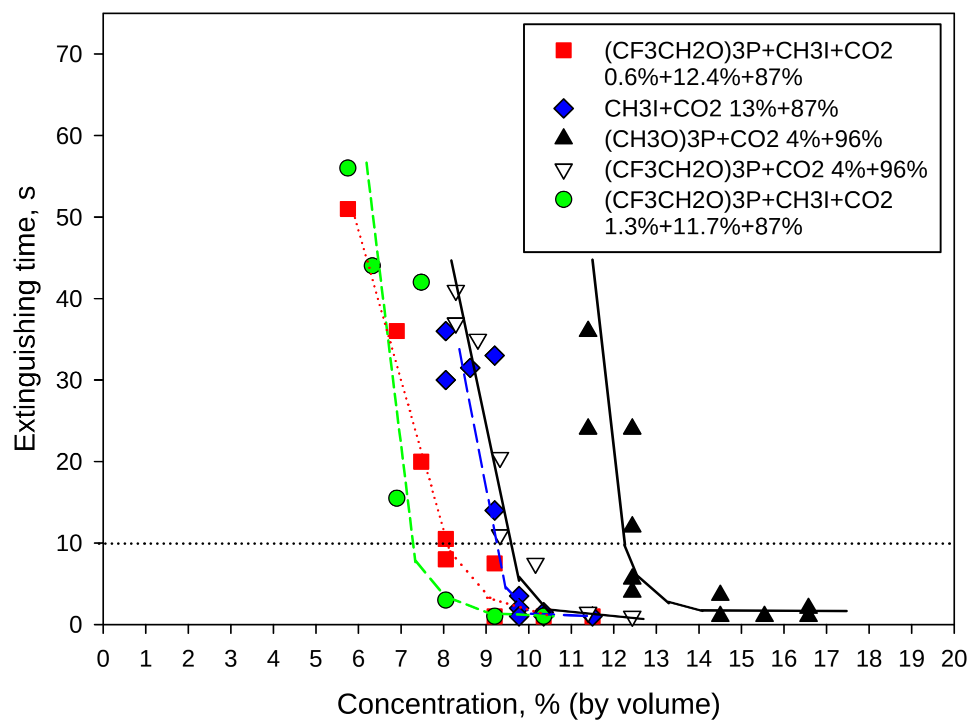

Figure 3. Typical dependencies of time of extinguishing on a volume concentration of blends.

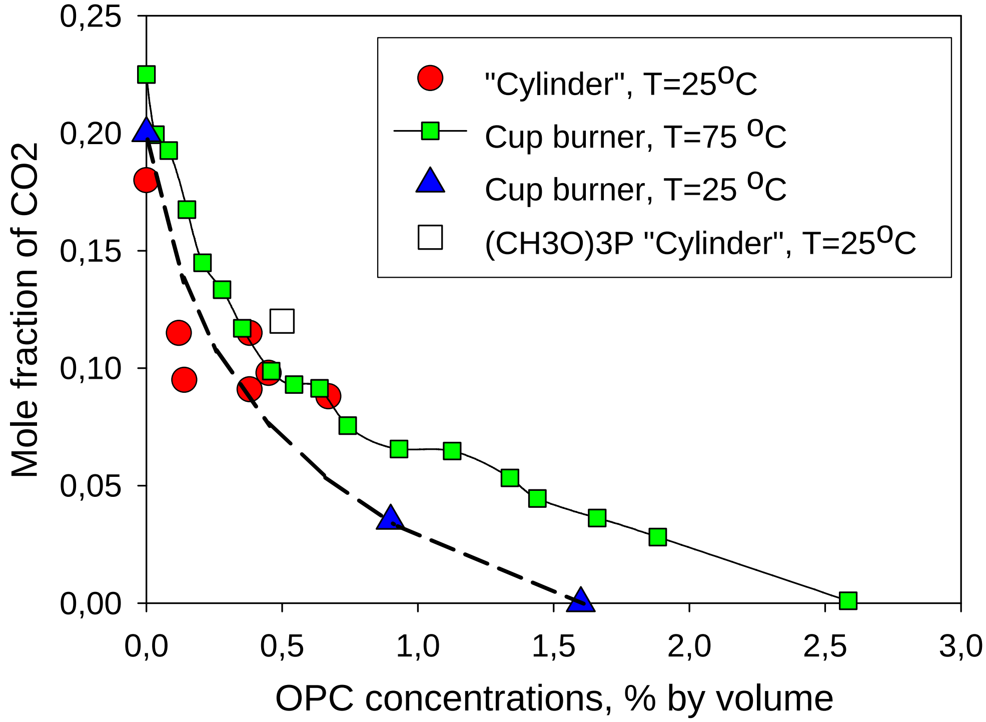

Figure 4. Comparison of extinguishing concentrations obtained by using cup-burner and "cylinder" technique for blends of CO_2 with $(CF_3CH_2O)_3P$.

Table 3. MECs of individual and blends of fire suppressants obtained using "cylinder" method at 20^0C.

№	Fire suppressant or composition of blend (% by vol.)	MEC of fire suppressant or blend in air, % by vol.	MEC of fire suppressant or blend in air, g/m^3	Lower temperature limit of application, ^oC**
1	CO_2+FTEP (96:4)	9,6	237	~25
2	CO_2+CH_3I+FTEP (87:12.4:0.6)	8,0	208	-5
3	CO_2+CH_3I+FTEP (87:11.7:1.3)	**7,2**	**182**	**5**
4	CO_2+CH_3I (87:13)	9,2	230	-50
5	CO_2+$(CH_3O)_3P$ (96:4)	12,5	263	
6	N_2+FTEP (94:6)	12,8	266	~35
7*	CO_2+FTEP (99:1)	11.6	244	~10
8*	CO_2+FTEP (98:2)	9.6	210	~15
9*	CO_2+FTEP (96.8:3.2)	11.9	281	~25
10*	CO_2+FTEP (96:4)	10.3	258	~25
11*	CO_2+FTEP (93:7)	9.5	270	~32
12	CO_2	18,1	362	
13*	CO_2	18,0	360	

* - setup #2 (VNIIPO), **- estimated

Effect of OPC on lean flammability limit of CH_4/Air mixture

Effect of TMP and FTEP additives on lean flammability limit of CH_4/Air mixture was studied in the present work. Figures 5a and 5b display the plots of $K_{ext}([CH_4])$ for lean premixed CH_4/Air flame doped with TMP and FTEP, respectively. These figures also show the plots of $K_{ext}([CH_4])$ for undoped flame. One can see that the represented dependencies are qualitatively similar: the additive causes the reducing of extinction strain rate in comparison with those in undoped flame at the same methane concentration in the mixture, that is, the additives inhibit the mixture burning.

The dependencies of lean flammability limits of CH_4/air mixtures with the inhibitors on the inhibitors loading were derived using the data represented in Figure 5. Figure 6 shows the obtained curves, which define the flammable regions of the mixtures containing the investigated inhibitors. The value of lean flammability limit for undoped mixture obtained in the present work (4.0 % by vol.) is somewhat lower than those according to literature data [17] (4.9% by vol.) because of elevated temperature of the mixture in our measurements. As indicated by Figure 6, the dopants give rise to increasing of lean flammability limit for methane-air mixtures. It is also evident that the effectiveness of the studied inhibitors in terms of their influence on lean flammability limit is different. In particular, this is evident from Figure 6 that P-bearing inhibitors are about 4.5 times more effective than CF_3Br. The effectiveness of TMP differs slightly from that of FTEP (this is within the experimental uncertainties). This is in accordance with the results for flame suppression effectiveness obtained using other techniques (cup burner, Bunsen burner) [3, 4], which also demonstrate that the effectiveness of P-bearing inhibitors does not depend on parent compound at low inhibitor loading (up to ~10 000 ppm).

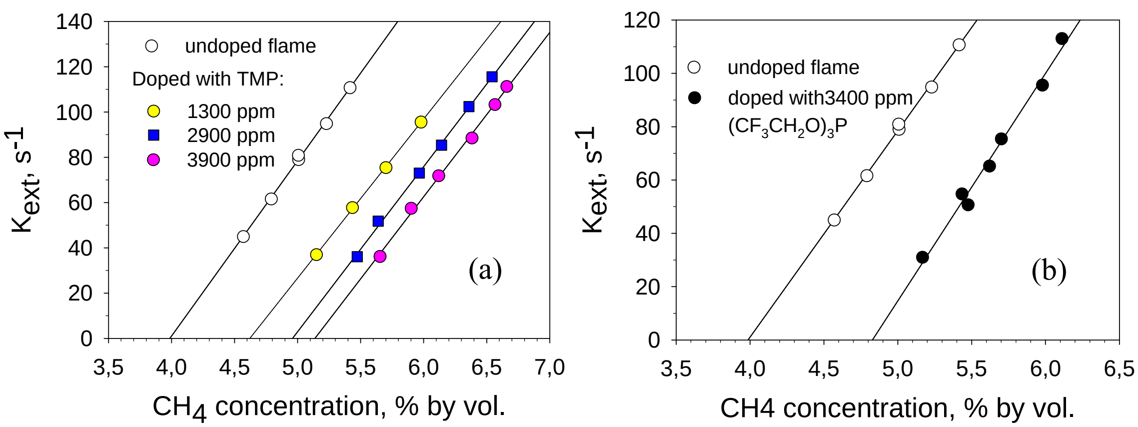

Figure 5. Global extinction strain rates K_{ext} versus CH_4 concentration [CH_4] for lean premixed CH_4/Air counterflow flames doped with TMP (a) and $(CF_3CH_2O)_3P$ (b), and without additives.

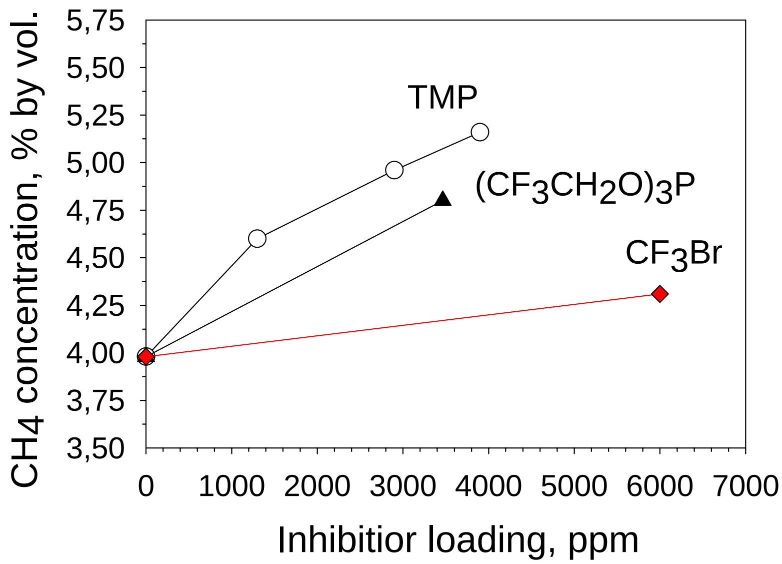

Figure 6. The dependence of lean flammability limits of CH_4/Air mixtures on inhibitors loading.

ESTIMATION OF OZONE DEPLETION POTENTIAL AND GLOBAL WARMING POTENTIAL FOR FTEP.

The analysis of the available data on physical and chemical properties of FTEP, executed with the help of the methods developed in Laboratory of Chemical physics of the atmosphere (Institute of energy problems of chemical physics of the Russian Academy of Sciences) has shown, that coming of this substance in the atmosphere practically in unlimited amounts will not put any damage to the ozone layer and climate of the Earth. Estimated Ozone Depletion Potential of FTEP is equal to zero, Halocarbon Global Warming Potential (HGWP, relative to CFC-11 for $t = \infty$) is equal $2{,}71 \cdot 10^{-4}$, and Global Warming Potential (relative to CO_2 (for $t = \infty$)) = 0,426. HGWP of FTEP for time horizons 20, 100 and 500 years are equal, accordingly, $7{,}56 \cdot 10^{-4}$; $3{,}10 \cdot 10^{-4}$ and $2{,}74 \cdot 10^{-4}$, and GWP of FTEP for the same time horizons are equal, accordingly, 4,8; 1,4; and 0,43.

CONCLUSIONS

MECs of a number of blends including CO_2, OPCs and CH_3I were determined using a cup-burner technique. It was discovered that these components provide a positive synergetic effect. Basing on interaction index estimation a composition of binary and triple blends with maximal synergetic effect was determined. The data obtained allow concluding that blends of CO_2, OPCs and CH_3I can be used for practical needs as extinguishing agent.

The influence of OPCs additives on lean limit of CH_4/air flame propagation was studied using a method of counterflow premixed flame. The additives of TMP and FTEP were found to decrease the propagation limit of CH_4/air flame 4.6 times more effectively than the same additive of CF_3Br.

The estimation of FTEP influence on ozone layer and climate of the Earth revealed that it produces no effect neither on ozone layer nor on the Earth climate.

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