# **Technology Development for the Production** of ABCE Fire Extinguishing Dry Powders

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**Abstract**—A classification of fires depending on combustible material and fire extinguishing means was considered. Advantages offered by fire extinguishing dry powders were described. It was shown that multifunctional ABCE fire extinguishing dry powders mainly consist of ammonium phosphates (mono- and diammonium phosphate and ammophos). Based on the literature and patent review, the technology development for the production of ABCE fire extinguishing dry powders was analyzed and summarized. Possible future directions in the technology development for the production of fire extinguishing dry powders based on ammonium phosphates were explored.

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Selection of fire extinguishing means should be based on the fire class, with the view of obtaining a maximum extinguishing effect with minimum cost. Depending on the combustible material, fires are classified as follows [1–4]:

**class A**: fires in combustible solids;

**class B**: fires in liquids;

class C: gas fires; and

**class D**: metal fires; and class E: fires in electrical installations under tension [3, 4].

In accordance with the substance employed, modern fire extinguishing means are subdivided into water, foam, dry powder, gas, and combined extinguishers; each of them is effective in extinguishing a certain class or group of fires [3–5]. Dry powders are the most effective in extinguishing all fire classes, i.e., represent a multifunctional means for preventing flame propagation in the bulk and on the combustible surface [3, 4].

Foreign national and international standards [6–9] define a fire extinguishing dry powder (FEDP) as a fine mixture of solid salts, comprising one or several principal (active) ingredients with certain additives which prevent the extinguishing agent from clogging and clumping and render it free-flowing.

The principal ingredients of FEDPs are alkali metal carbonates and bicarbonates (KHCO<sub>3</sub>, NaHCO<sub>3</sub>, K<sub>2</sub>CO<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub>), ammonium phosphates (NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>, (NH<sub>4</sub>) <sub>2</sub>HPO<sub>4</sub>), alkali metal oxalates and halides, urea, alkali metal citrates, magnesium hydroxide, and other [9–14]. Additives include inert (talc, graphite, zeolite, silica) and water-proofing (organosilicon liquids) agents improving the water repellency and flowing capability of FEDPs. The content of the principal ingredient should exceed 80 wt %, and that of additives should not exceed 20 wt % [15].

Russian and foreign experts emphasize the following advantages offered by FEDPs [10, 15, 16]:

In the Soviet Union and abroad, intensive development of fire fighting activities involving the use of dry powders was started in the 1960s [17]. At that time, general-purpose fire extinguishing powders

bicarbonate, potassium sulfate, potassium chloride, urea-fused metal carbonates, and other; and - B, C, E, or D (special-purpose) powders whose

containing ammonium salts; - BCE powders (general-purpose powders), whose principal (active) ingredients are sodium or potassium

principal (active) ingredient is potassium chloride,

extinguishing agents into BC and ABC powders [15].

Most of the foreign standards classify dry powder

Multifunctional ABCE fire extinguishing dry

powders mainly consist of ammonium phosphates

(monoammonium phosphate, diammonium phosphate,

they can fight, are subdivided into: ABCE powders (general-purpose powders) whose principal (active) ingredients are phosphorus-

According to regulatory documents [3-5] and relevant publications [10, 12, 14], all fire extinguishing dry powders, depending on the purpose and fire class

- diversity of fire fighting ways, including those

temperatures, from -50 to +60°C; and

- applicability over a wide range of operating

intended for explosion prevention (phlegmatization)

agents used in water and foam-making installations;

- minimized indirect damage inflicted by fire to a room and property it accommodates, by contrast to

chemical inertness);

- extremely high extinguishing ability of agents

- ability to quickly knockdown the flames from the

- effective shielding of structures against the heat

when used both individually and in conjunction with

water/foam sprays which are especially effective in

extinguishing flammable liquids in open spaces;

surface of the burning material;

flux emanated from the flames;

and suppression.

graphite, and other.

- suitability for multipurpose usage; environmental friendliness (lack of toxic components, ozone-depleting substances, low corrosivity,

based on bicarbonates and phosphorus-containing ammonium salts, as well as means to fight metal fires were produced [18].

In the 1970s, the scientific work at All-Union Research Institute for Fire Protection and its Kiev branch, established in 1977, was undertaken along several lines, specifically: study of the mechanism of the extinguishing action of powders, search for new FEDP formulations, and development of their preparation technologies. Also, large-scale comprehensive tests were conducted. In particular, the mechanism of heterogeneous inhibition of flame by fire extinguishing dry powders and metal halides was explored [19, 20]. The results of M. E. Krasnyanskii and coworkers' studies on thermal decomposition of ammophos [21] made possible development of two types of fine powders P-2AP [TU (Technical Specifications) 12-100-155-77] and P-3AT (TU 12-100-196-78), intended for remote fire extinguishing in closed spaces and horizontal channels. Also, new techniques were proposed for determining the extinguishing concentration of FEDPs [22]. Aerosil AM-1-300 was extensively used for hydrophobization of the powders [23, 24].

Since the late 1970s, the focus in the technology development for the production of FEDPs has been placed on decreasing the number of process stages [25. 26] and on endowing the powders with additional components, mainly of mineral origin (e.g., nepheline concentrate [27], chamotte-kaolin powder [28], tricalcium phosphate, mica, talc, graphite, pyrophyllite [29]) reducing not only the moisture absorption and caking, but also the fire fighting capacity. At the same time, there had been a tendency to increase the fire fighting efficiency of powders by introducing additional chemical compounds, e.g., lithium 12hydroxystearate improving the adhesion of the polyphosphate smelt to burning metals [30] and urea allowing the use of powders based on alkali metal carbonates for extinguishing all fire classes [25, 26]. In the mid-1980s, A. V. Antonov and V. M. Zhartovskii developed and implemented at Fosforit, Joint-Stock Company (Kingisepp, Leningrad oblast) a flowsheet for production of Pirant-A, a fire extinguishing powder based on phosphorus-containing ammonium salts [17].

In the early 1990s, PSB-3, OPU-5, and other fire extinguishing powders were produced. At that time, domestic producers satisfied only 20% of the overall Russia's demand for FEDPs. In response to this situation the expansion of the production of fire extinguishing dry powders, relying on Russia's own raw materials base was begun in 1993. The Ekokhimmash, Joint-Stock Company (Bui, Kostroma oblast), developed a technology and organized commercial production of PSB-3M powder, in batch quantities since 1995. The new modification of PSB-3 powder contained fumed silica treated with an organosilicon liquid instead of Aerosil AM-1-300. Since 1997, a fire extinguishing powder based on phosphorus-containing ammonium salts has been produced by "Ekokhimmash" [31, 32]. In 1996, the manufacture of ammophos-based P2-ASh fire extinguishing powder was started in Russia.

The technologies of preparation of FEDPs based on both phosphorus-containing ammonium salts and other chemical compounds (alkali metal chlorides. bicarbonates, etc.) are underlain by mechanical grinding of the ingredients [33]. Some of the existing FEDP production methods involve pretreatment of the raw material (drving ammonium phosphates and sulfate [34, 35] and hydrophobization of fumed silica [31, 36]), and some do not [37]. However, along with the above-mentioned methods, there exist more sophisticated technological solutions. For example, patents [38, 39] describe methods for producing a multifunctional fire extinguishing dry powder by neutralization of phosphoric and sulfuric acids with an ammonia solution, followed by mixing the resulting product together with an organosilicon liquid and silicate additives improving the performance characteristics of the product.

To expand the range of fire extinguishing agents, manufacturers vary the ratio and type of the ingredients used. A method for preparation of a multifunctional FEDP by reacting solutions of Group II–IV metal salts with aqueous LiOH, NaOH, KOH, RbOH, CsOH, or NH<sub>4</sub>OH solutions was proposed in patent [40]. Also, the use of ammonium polyphosphates as an ingredient of dry powder extinguishing agents was reported [41].

A common feature shared by most of the dry powder extinguishing agents known by 2000 is the use of modified Aerosil AM-1-300 as a highly dispersed anticaking additive [31, 33, 42–44]. However, since the beginning of the XXI century the tendency has been toward replacement of this additive by highly dispersed fumed silica due to scarcity, high cost [44], and lack of production in Russia of Aerosil AM-1-300 [34, 45]. In particular, patent [46] describes a dry powder extinguishing agent based on ammonium phosphate (55–100 wt %) and ammonium sulfate, which contains, instead of Aerosil, a highly dispersed additive prepared from domestic raw materials, hydrophobic fumed silica, in the amount of 2–4 wt %. Also, corundum powder (3–11 wt %) was introduced as flow improver.

In the 2000s, the trend toward reducing the weight content of ammonium phosphates in dry powder extinguishing agents persisted, with ammonium phosphate being replaced by significant amounts (up to 25– 70 wt %) of various minerals, clay, talc [34], quartz [47], muscovite, nepheline concentrate [31], silica, aluminum silicate [48, 49], which improve the flow and increase the bulk density of the powder. Inventions [50, 51] describe new fire extinguishing dry powders based on natural zeolite. The above-considered technical solutions allow reducing the cost of the powder via using cheaper raw materials and decreasing the number of stages in the FEDP preparation flowsheets but fail to preserve high extinguishing ability of the finished product.

With the view of increasing the fire extinguishing ability of FEDPs, combined agents were developed [52, 53] on the basis of two principal ingredients, ammonium phosphate and ammonium chloride, which provide synergistic enhancement of the fire extinguishing ability of the mixture. Invention [54] employs potassium chloride and diammonium phosphate as the basis for extinguishing powder. This allows obtaining an FEDP intended for fighting all class (A, B, C, E, and D) fires. Patent [55] describes an improved FEDP preparation technology compared to the existing Pirant-A [56]. In particular, through the use of a grinding-vacuum unit this technology eliminates the stage of dissolution of the raw material, as well as wear of the crushing bodies and contamination of the end product.

The currently available extinguishing powders use ammonium phosphate as the principal ingredient both individually [34, 45, 47, 57–61] and in a mixture with a second principal ingredient, ammonium sulfate [32, 46, 49, 62, 63]. The ammonium phosphate to ammonium sulfate ratio is varied depending on the powder application purpose and conditions. For example, CENTRIMAX and ISOCOMP (United Kingdom) ABC-extinguishing powders contain 20 to 90 wt % monoammonium phosphate, the rest being ammonium

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substance (ammonium phosphates). In particular, invention [65] describes an FEDP production method in which the particle size of the ammonium phosphates fraction is 35-60 µm, and that of ammonium sulfate, 60–140 µm, which provides high extinguishing performance and hydrophobicity parameters for the FEDP. The review of the scientific provision of fire safety [66] noted the research on the use of nanosubstances for preparation of fire extinguishing powders that is under way now. The dispersity of the agents prepared with the use of nanoparticles is by three orders of magnitude higher than that of the conventional extinguishing powdered agents. Adding cesium salts allows increasing the extinguishing ability of the nanopowder approximately 15-fold and reducing the extinguishing concentration from 0.25 to 0.015 kg  $m^{-3}$ 

agents, and ammonium sulfate acts as the carrier of the

extinguishing fraction. The carrier particle size in

FEDPs typically exceeds that of the fire extinguishing

flame [67]. Excellent explosion- and flamesuppressing properties with respect to methane combustion reactions were revealed for zirconia nanoparticles by Chinese scientists [68]. The main extinguishing powders producers in

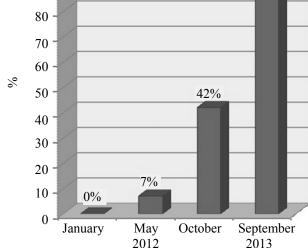
Russia are Ekokhimmash, Scientific and Technical Center, Closed Joint-Stock Company (Bui, Kostroma oblast), Kalancha, Limited Liability Company (Sergiev Posad, Moscow oblast), VVP, Joint-Stock Company (Nizhny Novgorod) (Volgalit), Istochnik Plyus, Closed Joint-Stock Company (Biisk), and some other companies. The III All-Russia fire tests of primary fire fighting means showed that it was not until the end of 2013 that a trend emerged toward improvement of the quality in the segments of OP-2 "automobile" fire extinguishers, as well as of OP-4 and OP-5 "office" fire extinguishers in comparison with the results of the field trials conduced in 2012 (Fig. 1) [68].

The expert commission revealed low quality of heavier portable OP-8 fire extinguishers. Only a portion of the specimens tested was suitable for suppressing class A fires at the seat, and none of them, for suppressing class B fires. As shown by fire tests of OP-35 wheeled powder extinguishers, none of the

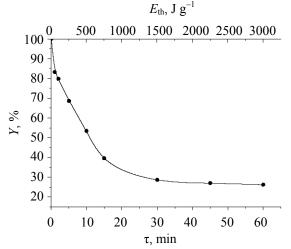
Fig. 1. Fire tests of the primary fire extinguishing equipment (OP-2, OP-4, and OP-5 fire extinguishers) based on "Soyuz 01" Association activities [68]. (■) % of fire extinguisher specimens whose fire extinguishing capacity is compliant with GOST (State Standard) R 51017-2009 [4].

specimens could suppress fires at all the model seats included in the appropriate State standard. However, this type of fire extinguishers is most common in places crowded with people, cinema-concert halls, shopping and entertainment centers, railway stations, and gas stations. Therefore, the development of new extinguishing powders remains topical and requires close attention from both leading manufacturers and research scientists.

An important parameter affecting the extinguishing capacity of powders is their particle size. The standard size of the extinguishing fraction is 10–75 µm. In [15], the minimum and maximum FEDP particle sizes prescribed by the standards adopted by different countries are presented. According to ISO and NFPA standards, the maximum particle size ( $D_{max}$ ) of ammonium phosphate is 350 µm. The relevant Chinese standard prescribes  $D_{max} = 250$  µm and the minimum particle size  $D_{min} = 40$  µm with  $\geq 45\%$  total  $\leq 40$  µm fraction. In the United Kingdom, the minimum particle size of the FEDP fraction is 37 µm, and this fraction should account for 56–60% of the total powder weight. In FRG,  $D_{min} = 40$  µm is prescribed at a 52–67% content of this fraction.



88%



**Fig. 2.** Variation of the yield of the fine powder fraction of ammophos *Y*, %, with the grinding time  $\tau$ , min, and the energy supplied  $E_{\text{th}}$ , J g<sup>-1</sup>.

In the mid-1950s it was revealed that the inhibitory effectiveness of finely dispersed solid phase is a function of the surface area of all the particles in the flame [69].

Along with the size, there is a need to consider the density, momentum, and convective characteristics of the particles of the powder passing through the flames. It was found that, in terms of all these characteristics, finely dispersed powder particles offer an undoubted advantage over coarse fire extinguishing powders of the same composition. On the other hand, salts with the particle size of >100  $\mu$ m are not entrained with upward spreading flames.

The performance characteristics of FEDPs depend not only on the size but also on the morphology of the particles [15]. Flame retardant powders typically consist of variously shaped individual particles. The spherical shape and high surface area are preferred, since they provide high flow and high rate of ejection of powder from extinguisher, prolonged time during which the particles remain in a suspended state, and acceleration of their decomposition in the combustion zone. In [70, 71] it was demonstrated that the extinguishing ability of highly dispersed spherically hollow monoammonium phosphate particles exceeds 2.5–3 times that of existing extinguishing agents. Due to their low fall speed spherical particles are characterized by higher probability and duration of contact with the flame. Also, spherical shape ensures low agglomeration and high flow of powder [70, 72].

The material properties are strongly dependent on the particle size and morphology, so the preparation of nanoparticles and particles of desired shape is topical now and will remain topical in the future [15]. To this end, a variety of technological solutions are being developed and implemented.

Extensive application in FEDP production is enjoyed by differently designed mills. Grinding implies exertion a mechanical action by the grinding bodies on the particles and mechanochemical activation or modification of ingredients, so that the desired product with the performance characteristics satisfying relevant specifications can be obtained. In Russia, the use of ammophos (a complex fertilizer produced in significant amounts) as the raw material for the production of muntifunctional FEDPs has become a frequent practice.

During grinding of ammonium phosphates the product attachment to the mill walls occurs, this effect being the more pronounced the larger the energy supplied to the material (Fig. 2).

This renders impossible obtaining a finely dispersed powder product with the required performance characteristics without additional treatment [73–77].

To improve the performance of fire extinguishing compositions, a number of additives were proposed and implemented in the FEDP production. From our viewpoint, they can be classified as shown in Table 1. Thus, selection and substantiation of the type and amount of additives to be introduced into fire extinguishing composition for improving their properties represent an important task in the development of new FEDPs.

By grinding ammonium phosphates in the presence of inert additives (talc-magnesite, MT-Sh-M microtalc, halite, mica, sylvinite, dolomite, and BS-120 silica) it is possible to reduce the amount of the product attached to the walls of the mill. However, along with selection of an appropriate additive it is necessary to optimize the energy supplied to the material being ground. For example, co-grinding of ammonium phosphates with 10 wt % BS-120 fumed silica or with 5 wt % talc-magnesite at ~105 J g<sup>-1</sup> energy supplied allows reducing the amount of the product attached to the walls of the mill to 3–7%. Since inert additives affect the particle size during grinding, though leave their hydrophobic properties almost unaffected, grind-

### TECHNOLOGY DEVELOPMENT FOR THE PRODUCTION

Type of additive	Substance	Effect produced		
Water-proofing:		Reduced moisture absorption, partially improved flow		
(a) solid	Modified aerosils, alkaline-earth metal stearates			
(b) liquid	Organosilicon water-proofing liquids, organosilicon compounds, fatty acid amines (in a solvent)			
Dusting solid finely dis- persed	Microtalc, fumed silica, phlogopite, vermiculite, etc., as well as a number of natural zeolites	Improved flow and vibration resistance		
Anticaking	Highly dispersed silica (silica, fumed silica, aerosil)	Improved flow and water repellency		
Inert (hydrophobic)	Aluminosilicates phosphogypsum, micas, natural zeolites, dolomite, talc, talc- magnesite, apatite, magnesite, chamotte kaolin powder, halite, sylvinite, etc.	Increased bulk density and water repellency and partially reduced propensity for moisture absorption due to the increased amount of the hydrophobic constituent of the powder		
Abrasive (abrading)	Corundum, quartz sand	Reduced particle size of the principal ingredient via grinding and prevented attachment to the mill walls		
Thermoplastic	Paraffin, stearin, etc.	Effective clinging of the extinguishing powder to the vertical surface (for extinguishing class D fires)		
Blowing	Urea, dicyandiamide, allophanate, pentaerythritol	Increased extinguishing capacity due to release of noncombustible gases upon thermal decomposition of additive		

Table 1. Additives improving the properties of fire extinguishing powders

ing should be followed by hydrophobization of the composition, which procedure is carried out commercially most often in mixers.

Grinding in the presence of inert anticaking additives can be advantageously combined with mechanochemical hydrophobization of ammonium phosphates to obtain particles of a predetermined size with a hydrophobic surface. Therefore, the mill with specific energy input of 0.878 kWh kg<sup>-1</sup> was loaded with ammophos and organosilicon water-proofing liquid GKZh 136-41 from poly(methyl hydrogen)siloxanes class. Table 2 shows the ammophos grinding results. It is seen that the bulk density of the uncompacted powder initially increases (to 0.74 g cm<sup>-3</sup>) with increasing grinding time (to 30 min) and then decreases (to 0.69 g cm<sup>-3</sup>). In the initial period of grinding the resultant fine particles occupy the free spaces between large particles, so that the bulk density increases. With increasing content of the fine powder fraction the bulk density begins to decrease.

An increase in the GKZh 136-41 content from 1 to 3 wt % led to deterioration of the properties of the finished product: An upward trend was observed (Table 3) both in the moisture absorption rate (from 2.8 to 3.8%) and in the particle size (the amount of the  $>140 \mu m$  fraction decreased from 97 to 60%). This is due to the oiling effect consisting in that the waterproofing agent molecules interact with one another and form salt bridges. Specifically, a water-proofing agent excess leads to overlayering of the primary chemisorbed layer by subsequent layers with disordered orientation, already weakly bound to the surface. As a result, the water repellency of the samples decreases. A 15-min and further grinding of ammophos in the presence of 3 wt % GKZh 136-41 leads to its zeroth water repellency (Table 3). With increasing grinding time the apparent

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Grinding time, min	Particle size fraction, µm					Bulk density, g cm <sup>-3</sup> , of indicated		
	>1.25	1.25-0.63	0.63-0.315	0.315-0.25	0.25-0.14	<0.14	powder	
	Proportion of indicated particle-size fraction, wt%						uncompacted	compacted
5	0.0	0.0	11.3	2.5	68.3	17.9	0.67	0.83
15	0.0	0.0	7.4	5.2	31.1	56.3	0.70	0.85
30	0.5	5.9	18.6	6.4	25.0	43.6	0.74	0.99
45	0.0	2.9	16.2	13.8	13.8	53.3	0.73	0.93
60	0.0	1.6	13.0	5.5	36.4	43.2	0.69	0.83

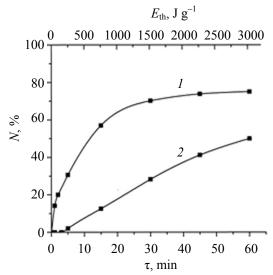
**Table 2.** Fractional composition and bulk density of ammophos in relation to the time of grinding in the presence of 1 wt %GKZh 136-41

Table 3. Powder characteristics in relation to the GKZh 136-41 content and time of grinding in the vibration mill

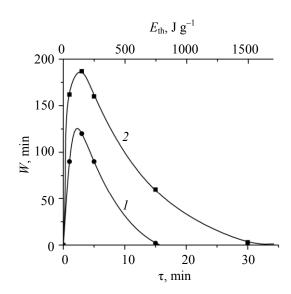
Principal ingredient, %	GKZh 136-41 content, %	Grinding time, min	Water repellency, min	Moisture absorption rate, %	<140 µm fraction proportion, %
Ammophos 100	_	5	0	2.2	33
Ammophos 99	1	5	90	2.8	97
Ammophos 99	1	15	0	2.2	90
Ammophos 97	3	5	100	3.8	60
Ammophos 97	3	15	0	3.3	54

bulk density increases from 0.632 to 0.833 g cm<sup>-3</sup>. The reason is that small particles obtained as a result of grinding occupy free spaces between large particles.

During 5-min grinding of ammophos with 4.5 wt % BS-120 fumed silica and 0.5 wt % GKZh 136-41 no product attachment to the mill walls was observed.



**Fig. 3.** Variation of the amount of the product attached to the mill walls *N*, %, with the grinding time  $\tau$ , min, and the energy supplied  $E_{\text{th}}$ , J g<sup>-1</sup>: (1) ammophos + 1.0 wt % GKZh 136-41 and (2) ammophos + 4.5 wt % BS-120 + 0.5 wt % GKZh 136-41.



**Fig. 4.** Variation of the water repellency *W*, min, with the grinding time  $\tau$ , min, and the energy supplied  $E_{\text{th}}$ , J g<sup>-1</sup>: (*1*) ammophos + 1 wt % GKZh 136-41 and (*2*) ammophos + 4.5 wt % BS-120 + 0.5 wt % GKZh 136-41

Further grinding caused the product attachment to the walls, up to 50% after 60 min of grinding (Fig. 3).

The best time of grinding in a vibratory mill for the system considered is 2 min (which corresponds to 105.32 J g<sup>-1</sup> energy supplied). In this case, the content of the <140  $\mu$ m fraction is 100%, and that of the <50  $\mu$ m fraction, 46%; the water repellency exceeds 180 min (Fig. 4). With both increasing and decreasing grinding time, the fraction particle size tends to increase, and the water repellency, to decrease (Fig. 4).

Deterioration of the hydrophobic properties of the samples (Fig. 4) is due to destruction of the powder particles coated with the organosilicon water-proofing liquid film, leading to the formation of new, untreated hydrophilic surfaces. A maximum in the dependence presented indicates the completion of the formation of a polymerization film on the particle surface within 2 min (105.32 J g<sup>-1</sup>).

Therefore, ammophos should be ground and made hydrophobic jointly with 4.5 wt % BS-120 and 0.5 wt % GKZh 136-41, and the supplied energy should be on the order of 105 J  $g^{-1}$ .

The resultant hydrophobic ammonium phosphate powder (fraction particle size 30–60  $\mu$ m) was mixed together with ammonium sulfate (particle size 70–140  $\mu$ m) and urea (a blowing additive) in the weight ratio of 52.5 : 47.5 : 5, respectively.

The FEDP samples were tested for compliance with the regulations relating to fire extinguishing powders at the accredited laboratory of the Ekokhimmash, Scientific and Technical Center, Closed Joint-Stock Company. The tests confirmed improvement of the physicochemical properties and enhancement (synergistic) of the effectiveness of the compositions in suppression of hydrocarbon flames, achieved via separate grinding of the FEDP ingredients to a certain particle size with simultaneous water-proofing treatment and subsequent mixing.

Thus, mechanochemical modification of ammonium phosphates should be carried out in the presence of inert anticaking and water-proofing additives at the optimal energy supplied to the material being ground. This will allow obtaining hydrophobic particles of a given size with a minimum amount of the product attached to the walls of the equipment. Future directions in the technology development for the production of fire extinguishing dry powders intended for extinguishing ABCE class fires can be as follows: finding new compounds effective in flame suppression and solutions to extend the service life of fire extinguishing dry powders, introducing promoting additives into the powders (e.g., cesium salts which increase the fire extinguishing capacity 15 times), using spherical particles, finely ground powders or nanopowders, etc.

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