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Development of a Marking Composition Based on Nanoaluminum and Iron Ore Concentrate for Mixed Explosives and Their Subsequent Identification

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Abstract

In this article, an experimental study of the methods for forming a marking composition based on nanoaluminum and iron ore concentrate for use as marking additives in mixed explosives was considered. The prepared marking composition consists of nanoaluminium - 95%, iron ore concentrate - 5%, and according to X-ray spectral analysis the intense peaks of aluminum (97.62%), iron (0.89%), calcium (0.64%), manganese (0.05%), chromium (0.009%), titanium (0.01%), sulfur (0.78%) are visible. Two methods of introducing a marking composition based on nanoaluminium and iron ore concentrate into the composition of the explosive "Granulite M" were studied. The marking of mixed explosives with a powdered fine marking composition by the first method, in amounts sufficient for uniform distribution in the composition of the explosive (at least 2%), leads to a change in the formulation composition. When using the second method, instantaneous precipitation of particles of nanoaluminum and iron ore concentrate was observed. In industrial conditions, this will lead to the need for a device for mechanical mixing of containers with liquid petroleum products. Thus when using finely dispersed metals and their alloys for marking of mixed explosives, it is fundamentally possible, provided they are introduced into explosive compositions through a liquid combustible component with constant stirring for uniform distribution. It is expedient to identify explosives marked in this way by X-ray spectral analysis by the presence of certain metal markers, and the remains of fragments at the site of the explosion - by the presence of oxides of marker metals.

Keywords: *Granulite M, Iron ore concentrate, Markers, Mixed explosives, Nanoaluminium, X-ray spectral analysis.*

1. INRODUCTION

In modern conditions, blasting operations require constant improvement of the used explosives and gas-generating compositions, both their formulations and technologies for the production and use of explosives [1-6]. This is largely due to the need to take into account the cost of explosives, the mobility and mechanization of explosives preparation points, the safety of preparation technologies, as well as the explosives themselves and gas generating compositions (including retarders). A large number of methods have been studied in the world to improve the power characteristics of both individual chemicals and various mixtures capable of an explosive transformation reaction that has a destructive effect due to the instantaneous expansion of the explosion products [2-4]. This phenomenon is effectively used both for military purposes and in the national economy. However, even with the use of explosives in the national economy, there is also a downside to the energy of the explosion, which is expressed in human mortality, injury, destruction of property as a result of periodic accidents caused by unauthorized initiation of explosives, as well as a result of the illegal use of explosive materials for criminal and terrorist purposes [7-11].

The most popular requirements for the control of the turnover of industrial explosives, which include licensing, authorization or other similar requirements for storage, use, transportation, purchase and sale, and other types of commercial activities in relation to explosives do not fully provide full control. “Criminal elements”, as a rule, acquire explosives from the most easily accessible and least risky sources. Usually, identifiers include those substances that have oil-fat solubility, chemical resistance in media with different pH, resistance to free radicals, chemical inertness to explosive components, chemical inertness to explosion products, and the absence of toxic properties, for example: ethylene glycol dinitrate (EGDN), 2,3-dimethyl-2,3 dinitrobutane (DMNB) [12-16]. In each individual case, the marking chemical is introduced into the composition of the explosive composition in such a way that the marking agent is evenly mixed in the finished product [14-18].

A method of marking explosives is known, when a fine powder of a metal alloy – “a marker” is introduced into the composition of an explosive substance at the manufacturing stage. The component composition of the metal alloy corresponds to a specific manufacturer of products, and the mass ratio of the alloy metals determines the date of manufacture [19]. It is recommended to use aluminum or its alloy with magnesium as the basis of the alloy, and rare earth metals as marking additives. The identification of explosives in this case is reduced to a qualitative and quantitative analysis of the components of the marker [20]. Significant disadvantages of this marking method include: due to the difference in the densities of the marker (more than 2.7 g/cm^3) and molten trinitrotoluene (1.6 g/cm^3), marker particles in the powdered state can actively settle in the TNT melt, which makes it difficult its marking during the manufacturing process and will simplify the removal of the marker by repeated melting and the spontaneous process of deposition of the marker powder [21]. In [22-24], a marking composition based on Balkhash iron ore concentrate and nanoaluminium was prepared. Such marking compositions were used for marking industrial mixed explosives and gas-generating compounds [25]. Ultimately, the main possible dangers arising from the use of mixed explosives are: the insufficiency of existing methods of controlling their turnover, the lack of effective methods of detection and visual identification, which increases the likelihood of illegal use of this type of explosives.

It is known that rare earth metals and alloys based on them are extremely expensive and the raw material base of rare earth metals and their industrial production are severely limited [26-28]. It is advisable to use rare-earth metals and alloys based on them in small quantities for marking gas-generating compositions (including retarding ones), since the industrial consumption of these products has a rather narrow focus - the destruction of construction sites, small mountain ranges, the opening of productive strata, the cleaning of perforation channels and treatment of the bottomhole formation zone with combustion products of gas-generating charges [29-34]. However, it should be taken into account that rare earth metal oxides, entering into a combustion reaction, emit a glow. And this phenomenon must be remembered when using rare earth metals, for example, for pyrotechnic compositions from non-ferrous metals containing

$Sr(NO_3)_2$, Mg and other substances that produce a certain color effect when burning.

In accordance with the above written, the problem of ensuring the possibility of marking of industrial explosives with hidden marking additives at the stage of their production is urgent. This can make it possible to identify by technical means both the products themselves, that is, explosives, and to establish the brand of the detected explosive, the manufacturer and other necessary information.

In this study, as an alternative to rare earth metals, it is proposed to use fine aluminum for the alloy base, and iron ore concentrate as marking additives. Identification of explosives marked in this way is not difficult because traces of metal oxides that make up the marking composition remain at the site of their explosion.

The purpose of this work is an experimental study of methods for forming a marking composition based on nanoaluminium and iron ore concentrate for use as marking additives of mixed explosives. We propose to use nanoaluminum 95% and iron ore concentrate 5% as the marking composition used, the initial components of which were studied by X-ray fluorescence analysis.

2. MATERIALS AND METHODS

In this study, the initial materials and conditions for the study of chemicals were selected to determine their special marking properties, as well as to identify industrial mixed explosives containing marking chemical additives in their composition [26, 27].

The following components were used to perform the experiments:

- aluminum nanopowder brand Alex (90-100 nm);
- iron ore concentrate as a marking agent;
- granules of ammonium nitrate grade B (NH_4NO_3 according to the State Standard 2-2013);
- “Granulite M” according to the State Standard 21987-76.

The components were weighed on a Pioneer PA214 electronic balance and thoroughly mixed using the MS300 Hotplate Magnetic Stirrer and the AM110W-T mechanical paddle stirrer. Grinding of particles of metal alloys was carried out in an agate mortar.

The sample preparation procedure was as follows: weighing → grinding of metal alloy particles → mechanical mixing of marking substances with each other, with a solvent and components of explosive compositions → mechanical mixing of explosive components.

The calculation of the oxygen balance of explosives was carried out according to the formula:

$$K_b = \frac{d-(2a+\frac{b}{2})\cdot 16}{M_E}\cdot 100 \qquad (1)$$

where: *a* is the number of carbon atoms in a substance molecule; *b* is the number of hydrogen atoms; *d* is the number of oxygen atoms; M_E is the molecular weight of the explosive; *16* is the atomic mass of oxygen.

Zero is called such an oxygen balance, in which oxygen in the composition of the explosive is sufficient for the complete oxidation of all the combustible elements that make up the explosive, i.e. the amount of oxygen fully corresponds to the amount of combustible components.

The determination of the elemental composition of the studied compositions was performed by X-ray phrase-core analysis on the spectrometer of the X-ray “FOCUS-M2”. Detection limits of elements according to the 3σ criterion (depending on the element, sample matrix and analysis technique), in %: for elements with atomic number from 15 to 20 - $1\text{-}10^{-2}$; for elements with atomic number from 21 to 92 - 10-^{2} - $3\cdot 10^{-3}$. Using an NtegraTherma Scanning Electron Microscope, the morphology of the sample, changes in structure and surface were determined. All experimental data were checked for reproducibility, conducting each experiment at least three times. The relative measurement error is within 1.9-5.2%. The temperature measurement error on the setup was ±0.5%. The error in conducting a quantitative analysis of the surface composition was 2-9% depending on the element and its concentration. In the X-ray fluorescence analysis, the photoelectron peaks were calibrated with an error of ±0.1 eV.

3. RESULTS AND DISCUSSIONS

3.1. Experimental Study of the Properties of Nanoaluminum and Iron Ore Concentrate to Determine the Possibility of Using It as a Marking Additive for Mixed Explosives

As a marking composition, it is proposed to use nanoaluminum 95% and iron ore concentrate 5%, the initial components of which were studied by X-ray fluorescence analysis. According to the results of the study, the X-ray diffraction pattern of nanoaluminum (Figure 1, Table 1) shows intense identification peaks for the compound of aluminum (99.52%) and iron (0.48%).

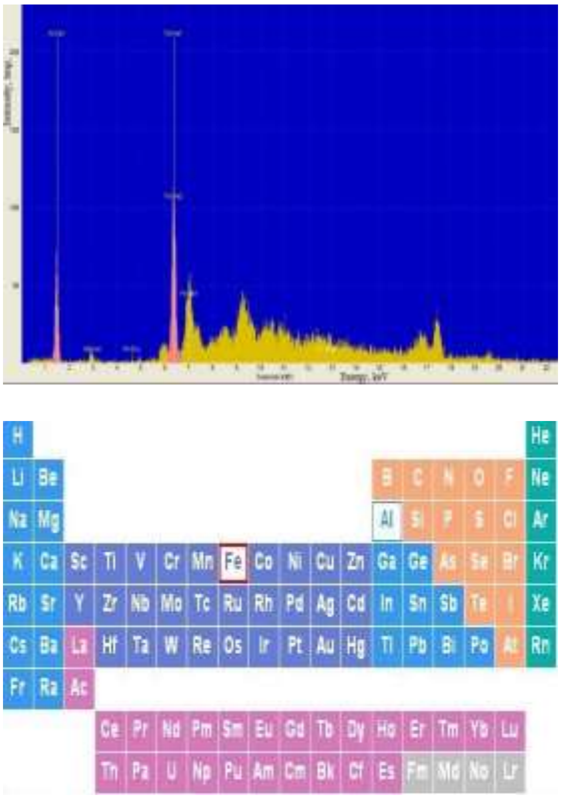

Figure 1. *X-ray spectral analysis of nanoaluminum.*

Table 1. *Results of X-ray spectral analysis of nanoaluminum.*

No	Element	Concentration, %	Interval, cps
1	Iron	0.481	12.2
2	Aluminum	99.52	6.0

According to the results of the study of iron ore concentrate on the X-ray (Table 2, Figure 2), intense peaks of identification of iron compounds (54.84%), calcium (31.28%), manganese (1.48%), chromium (1.10%), titanium (1.20%) and sulfur (10.10%) were determined.

Table 2*. Data of X-ray spectral analysis of iron ore concentrate.*

No	Element	Concentration, %	Interval, cps
1	Iron	54.84	79.00
2	Aluminum	00.00	00.00
3	Calcium	31.28	22.80
4	Manganese	1.48	2.40
5	Chromium	1.10	1.70
6	Titanium	1.20	1.10
7	Lead	10.10	0.56

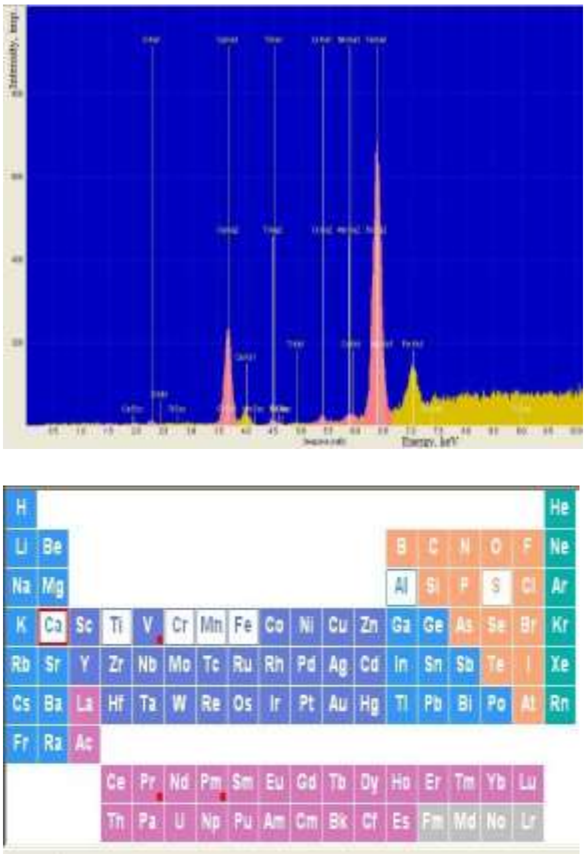

Figure 2. *X-ray spectral analysis of iron ore concentrate.*

Thus, the content of the main elements in the composition of iron ore concentrate and nanoaluminum was experimentally established, this information is necessary for encoding information and subsequent identification of explosives marked with the materials under study.

3.2. Development of a Marking Composition Based on Nanoaluminum and Iron Ore Concentrate

Taking into account the experimentally obtained data of X-ray spectral analysis of nanoaluminum and iron ore concentrate, the detected individual elements were conditionally taken as possible identification technological parameters of the explosive (Table 3).

Table 3. *Possible identification technological parameters of the explosive, encrypted in the marking composition based on nanoaluminum and iron ore concentrate.*

Element	Concentration, %	Identification data
Aluminum	99.52	The name of the explosive is “Granulite M” according to the State Standard 21987-76
Iron	55.35	Manufacturer’s name - Alfa LLP
Calcium	31.28	Name of the end user - LLP “Omega”
Manganese	1.48	Each specific element can conditionally designate a certain month of manufacture and year, and subject to constant concentration, theoretically, it is possible to derive the digital value of the batch number of the product.

Thus, a marking composition based on nanoaluminum - 95% and iron ore concentrate - 5% was prepared, which was studied by X-ray spectral analysis (Figure 3, Table 4).

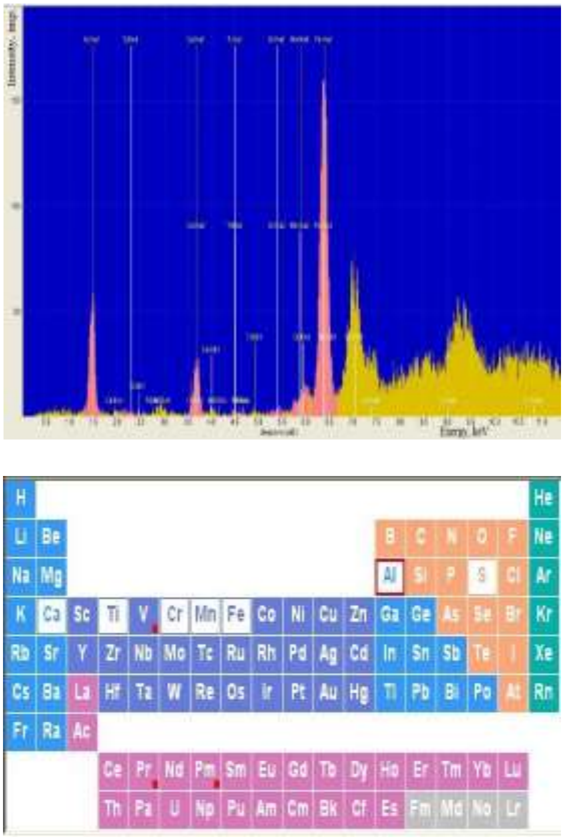

Figure 3. *X-ray spectral analysis of the marking composition based on nanoaluminum and iron ore concentrate.*

Table 4. *Results of X-ray spectral analysis of the marking composition based on nanoaluminum and iron ore concentrate.*

No	Element	Concentration, %	Interval, cps
1	Iron	0.888	19.0
2	Aluminum	97.62	4.8
3	Calcium	0.646	2.5
4	Manganese	0.0520	0.96
5	Chromium	0.00929	0.14
6	Titanium	0.0115	0.100
7	Sulfur	0.778	0.14

According to the results of the study, the X-ray diffraction pattern of the marking composition based on nanoaluminum and iron ore concentrate shows intense peaks in the identification of the following compounds: aluminum (97.62%), iron (0.888%), calcium (0.646%), manganese (0.05%), chromium (0.009%), titanium (0.01%) and sulfur (0.78%). It is also possible to observe a sharp decrease in the concentrations of the constituent elements of the iron ore concentrate, which may be due to the difference in the dispersity of the particles of the iron ore concentrate and nanoaluminum, the finer particles of which can be concentrated (sprayed) in a thin layer on larger particles of the iron ore concentrate alloy.

To confirm or refute these conclusions, studies of the morphology and microstructure of the marking composition based on nanoaluminum and iron ore concentrate were carried out (Figure 4).

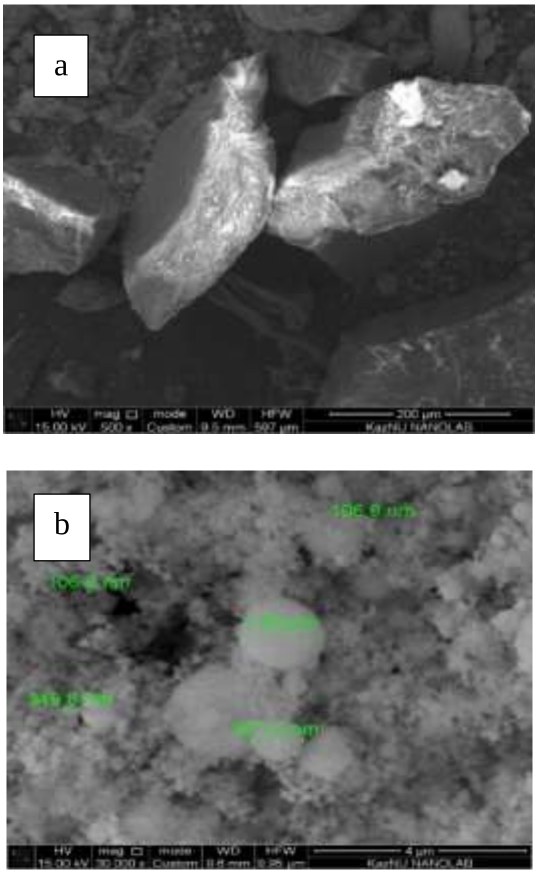

Figure 4. *SEM images of the marking composition based on nanoaluminum and iron ore concentrate: a - 200-fold increase; b - 4-fold increase.*

It can be seen from the microscopic images that the ammonium nitrate granule has nanopores, a nanodepression, and a nanocrack, in which nanoaluminum and nanocarbon compounds tenaciously hold.

Thus, from the images shown in Figure 4, it can be seen how fine particles of nanoaluminum with a size of 106.8 nm are distributed in a thin layer on the surface of the particles of the iron ore concentrate alloy, the size of which exceeds 5 µm. As a result of the study, it was possible to confirm the assumption that the decrease in the concentrations of the constituent elements of the iron ore concentrate, revealed as a result of X-ray analysis, is associated with a difference in the dispersity of the particles of the iron ore concentrate and nanoaluminum.

Taking into account the obtained data of X-ray spectral analysis of the marking composition based on nanoaluminum and iron ore concentrate (Figure 3), it was decided to reduce the amount of conditionally encrypted information about the explosive composition in the marking composition (Table 5).

Table 5*. Identification technological parameters of the explosive encoded in the finished marking composition based on nanoaluminum and iron ore concentrate.*

Element	Concentration, %	Identification data
Aluminum	97.62	The name of the explosive is “Granulite M” according to the State Standard 21987-76.
Iron	0.89	Manufacturer’s name is Alfa LLP
Calcium	0.64	Name of the end user is LLP “Omega”
Manganese	0.05	Month of manufacture

3.3. Experimental Introduction of a Marking Composition Based on Nanoaluminum and Iron Ore Concentrate into the Composition of the Explosive “Granulit M” According to the State Standard 21987-76 in Laboratory Conditions and Introduction into its Composition

Taking into account the peculiarities of the industrial production of mixed explosives, two methods were considered for introducing a marking composition based on nanoaluminum and iron ore concentrate into their composition:

1) the method of “dry injection” - when the marking composition is introduced into the composition of the explosive mixture

directly during the manufacturing process; 2) the method of introducing the marking composition into the composition of explosives through a liquid combustible component.

3.3.1. Dry Injection Method

On the example of nanoaluminum, by an empirical method, its minimum amount was established, which is necessary for uniform distribution in the composition of the explosive “Granulit M” (94.5% of the free-flowing phase of the oxidizer with 5.5% of the liquid oil product).

In order to maintain the oxygen balance of the explosive close to zero, the content of the liquid oil product was proportionally replaced by nanoaluminum.

Nanoaluminum was introduced into explosive compositions in the following order: ammonium nitrate granules were mixed with liquid petroleum products using a mechanical laboratory paddle mixer, and then the required amount of nanoaluminum was added in batches with continuous stirring. Further, the resulting mixture was settled for 24 hours, after which the uniformity of the distribution of nanoaluminum particles in the composition of the explosive was visually assessed. Obtained observational data were entered in Table 6.

As a result of the experiment, it was found that the marking of mixed explosives with a powdered finely dispersed marking composition by the “dry injection” method, in quantities sufficient for uniform distribution in the composition of the explosive (at least 2%), leads to a change in the prescription composition.

According to the calculation below, it is clearly seen how the total number of gram-atoms of elements in the gross formula changed when nanoaluminum was added:

- unmarked explosive composition:

$$945/80\times NH_4NO_3+55/170\times C_{12}H_{26}$$

$$11.81\times NH_4NO_3+0.32\times C_{12}H_{26}$$

- marked explosive composition:

$$945/80\times NH_4NO_3+35/170\times C_{12}H_{26}+20/27\times Al$$

$$11.81\times NH_4NO_3+0.2\times C_{12}H_{26}+0.74\times Al$$

Table 6. *Data on the empirical determination of the minimum amount of marking composition in the composition of the explosive “Granulite M”, added by “the dry injection” method (NA- nanoaluminium, AN - ammonium nitrate, OP - oil product).*

No	Amount of NA, %	Amount of AN, %	Amount of OP, %	Observation results
1	0.1%	94.5%	5.4%	AN granules are uniformly oiled, 5% of the granules contain traces of NA particles
2	0.5%	94.5%	5.0%	AN granules are uniformly oiled, 10-15% of the granules contain traces of NA particles
3	1.0%	94.5%	4.5%	AN granules are uniformly oiled, 30-40% of the granules contain traces of NA particles
4	1.5%	94.5%	4.0%	AN granules are not evenly oiled, 70% of the granules contain traces of NA particles, part of which settled at the bottom of the container
5	2.0%	94.5%	3.5%	AN granules are not uniformly oiled, 100% of the granules contain traces of NA particles, more than 50% of which settled at the bottom of the container

On the basis of the above reactions, the material balance was compiled element by element with the number of gram-atoms of elements in the gross formula of the explosive “Granulit M” before and after the addition of the marking composition by the “dry injection” method (Table 7).

It can be seen from the above reactions that when nanoaluminum is added to the composition of the explosive in an amount of 2%, the latter will actively participate in the explosive transformation reaction. In addition, given the fire and explosion hazard of nanoaluminum and other finely dispersed metals, their use in large quantities represents an increased danger, and because of the high cost, it also becomes economically unfeasible.

Table 7. *The total number of gram-atoms of elements in the gross formula of the explosive “Granulit M” before and after adding the marking composition by the “dry injection” method.*

Element name	The total number of gram-atoms of elements	
	before marking	after marking
$\sum_N$	11.81·2 = 21.98	11.81·2 = 21.98
$\sum_C$	0.35·12 = 4.2	0.2·12 = 2.4
$\sum_H$	11.81·4 + 0.32·26 = 49.9	11.81·4 + 0.2·26 = 52.44
$\sum_O$	11.81·3 = 35.43	11.81·3 = 35.43
$\sum_{Al}$	0.004	0.74

3.3.2. Method for Introducing a Marking Composition into the Composition of Explosives through a Liquid Combustible Component

In our previous works [35-39], the method of introducing marking compositions into the compositions of mixed explosives was effectively used precisely through the liquid combustible component. According to this method, with the introduction of a marking composition into a liquid oil product in an amount of 10 g / ton of explosive, it was possible to observe the instantaneous deposition of particles of nanoaluminum and iron ore concentrate (Figure 5), which in industrial conditions will lead to the need to equip a container with a liquid oil product mechanically in a mixing device.

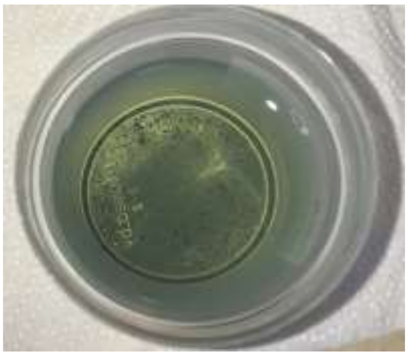

Figure 5. *The process of deposition of the marking composition in instrument oil.*

After mechanical mixing of 94.5% of the free-flowing phase of the oxidizer (ammonium nitrate) with 5.5% instrument oil, it was possible to visually observe the presence of particles of the marking composition on the surface of the ammonium nitrate granules moistened with instrument oil (Figure 6).

Instrument oil is a petroleum lubricating oil. In composition, it is a petroleum or synthetic product and its mixtures with animal or vegetable fats. Instrument oil is used for devices and precision mechanisms. It is used to minimize the percentage of wear of parts and friction units in precision, control and measuring, geodetic, medical and similar devices.

The photographs clearly show the presence of particles of nanoaluminum and iron ore concentrate on each individual granule of ammonium nitrate, which, at the same time, are sufficiently oiled, which actively contributes to the retention of particles of the marking composition.

After carrying out X-ray spectral analysis of the explosive “Granulit M” with a marking composition based on nano-aluminum and iron ore concentrate and comparing the obtained data with the data of X-ray spectral analysis with a pure marking composition, from the data in Table 8 one can clearly see the values of the errors in the concentrations of substances that make up the marking composition.

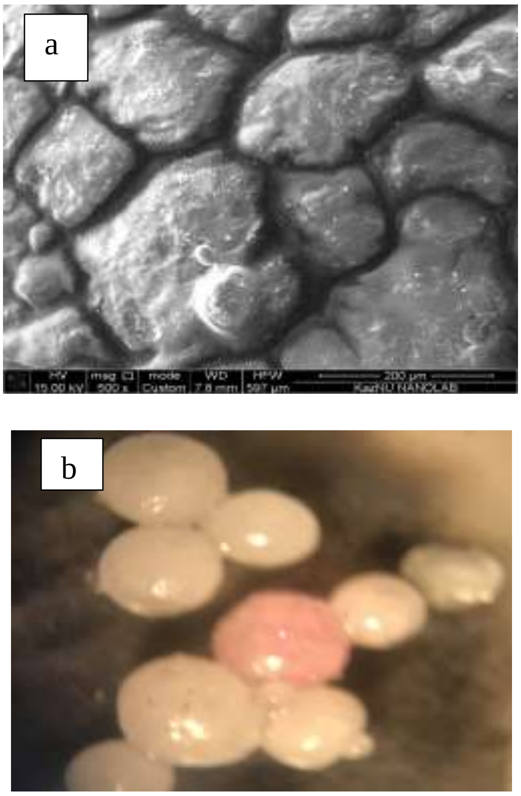

Figure 6. *SEM (a) and optical (b) images of the explosive "Granulit M" with a marking composition based on nanoaluminum and iron ore concentrate.*

It should be noted that despite the very active positions in the energy plan of nanoaluminum, according to Table 8, we can identify that the amount of aluminum is retained in the composition of "Granulite M" with an error of -0.84%. Also, presumably, when carrying out the identification by physicochemical methods of traces of an explosive after its explosive decomposition, one may encounter certain difficulties associated with not only a small amount of the marker, but also the activity of nanoaluminum entering into the oxidation reaction in the composition of the explosive.

According to the Berthelot principle [40-42], the explosive transformation reaction of "Granulite-M" with the addition of 0.01% of the marking composition based on nanoaluminum will have the form (2):

$$11.81\times NH_4NO_3+0.32\times C_{12}H_{26}+0.004\times Al = 0.00153Al_2O_3+4.2CO_2+25H_2O+10.99N_2+1.04O_2 \qquad (2)$$

According to this theory, in the process of explosive transformation in this system, aluminum is completely oxidized to Al_2O_3 oxide, and as a result, nanoaluminum in the final products should be identified by the amount of its oxides. And at this stage, we should fix the aluminum oxide as an identifier [43-46].

Table 8. *Data of X-ray spectral analysis of the marking composition and the explosive "Granulit M" with the marking composition based on nanoaluminum and iron ore concentrate, where IOC is iron ore concentrate.*

Element	Concentration, %			Identification data
	95% NA+5 % IOC	"Granulite M"	Relative Error rate, %	
Aluminum	97.62	96.78	-0.84	Explosive name is "Granulite M" according to State Standard 21987-76
Iron	0.89	0	-0.89	Manufacturer's name is Alfa LLP
Calcium	0.64	0.985	+0.345	The name of the end user is Omega LLP
Manganese	0.05	0.236	+0.186	Month of manufacture

The amount of iron disappears at its meager amount, which indicates the complexity of its further identification, and the use of iron ore concentrate as an identifier (Table 8).

The marking compositions synthesized in this work make it possible to visually identify a substance as explosive, as well as to establish information about its origin using physicochemical analysis methods.

4. CONCLUSIONS

In this paper, as an alternative to rare earth metals, it is proposed to use finely dispersed aluminum for the base of the alloy, and iron ore concentrate as marking additives. Identification of explosives marked in this way does not cause difficulties, since traces of metal oxides that are part of the marking composition remain at the site of their explosion. As a result of the experiments, a marking composition based on nanoaluminum and iron ore concentrate was developed, which has specific marking properties that allow the explosive substance to be visualized and the necessary identification information to be determined. Marking compositions can be used both as visual identifiers and as hidden markings.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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