

# Research on the Application of Controlled Pyrotechnic Reaction with the Use of Ammonium Nitrate for Transport from Seabed

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**ABSTRACT:** The increasing interest in the exploitation of underwater mineral deposits is due to the increase in demand for some mineral resources because of the depletion of their deposits on land. Therefore worldwide research is aimed at developing efficient technologies exploitation of minerals from the bottom of the seas and oceans.

The biggest problem in the development of underwater systems operating at great depths is transport from the seabed to the surface. Previous solutions represent various assumptions and technical concepts. Unfortunately, their biggest drawback is high energy consumption and thus high costs.

The authors proposed the use of a new method which involves the use of pyrotechnics as the source of energy in transport from the seabed at great depths (Filipek & Broda, 2016, 2017) presenting the results of theoretical and experimental research of pyrotechnic reaction involving potassium nitrate.

This paper explains the concept of the use of pyrotechnic materials involving ammonium nitrate transport from great depths in the aquatic environment. It presents the results of experimental verification of possibility of ammonium nitrate use in pyrotechnic reactions as a source of energy needed to raise the excavated material from the seabed to the surface. The authors used previously developed method of control for the effects of pyrotechnic reaction, i.e. mechanical damping of the shock wave in order to limit its harmful effects on the housing of the transport device performed also by controlling the time of pyrotechnic reaction. In theoretical research, particular attention was paid to determine the depth to which one can apply this method and determine the energy needed for transport depending on the density of the transported material (spoil).

We compared the results of the reaction using potassium nitrate in the reaction with the use of ammonium nitrate. The results confirm the possibility of the use of pyrotechnic materials for the transportation of large depths.

## 1 INTRODUCTION

One of the toughest problems researchers and engineers are facing while dealing with the exploitation of mineral deposits located on the bottom of the seas and oceans at great depths is the transport of excavated material to the surface. This problem has been addressed by academic institutions and industrial consortia (Depowski et al. 1998, Sobota et

al. 2005). This is due to huge interest in the exploitation of underwater deposits due to an increase in demand for some mineral resources and the depletion of their deposits on land. Wide operation areas of shelves in the sea mining (Karlic 1984; Depowski et al. 1998) and interest in polymetallic nodules and massive sulphide polymetallic (SMS) (SPC 2013) occurring in huge areas led to the creation of many concepts and

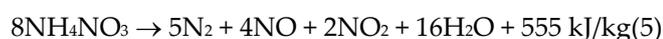
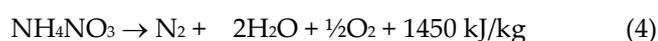
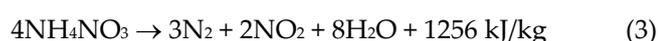
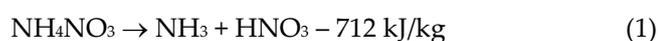
solutions. They have their advantages and disadvantages, the largest of which is energy consumption.

In previous publications (Filipek & Broda 2016, 2017) the authors proposed the use of a new method which involves the use of pyrotechnics as a source of energy in transport from the seabed with depths presenting the theoretical results (Filipek & Broda 2016) and the experimental pyrotechnic reactions with the participation of black powder (potassium nitrate) (Filipek & Broda 2017). This publication presents the results of the continuation of research – this time with the use of ammonium nitrate in the controlled pyrotechnic reaction.

## 2 COURSE OF THERMAL DECOMPOSITION OF AMMONIUM NITRATE

NH<sub>4</sub>NO<sub>3</sub> ammonium nitrate has a molecular weight of 80.05 g/mol and its natural occurrence in a natural environment is rare. For the first time it was received in 1659 by Glauber (Błasiak et al. 1956). Ammonium nitrate under normal conditions is a solid, crystalline body which is readily soluble in water. The solid NH<sub>4</sub>NO<sub>3</sub> absorbs H<sub>2</sub>O from the air. At 169.5°C (442.5 K) pure ammonium nitrate melts. The heat of fusion of NH<sub>4</sub>NO<sub>3</sub> equals 68 kJ/kg. In contrast, at 210°C (483 K) pure ammonium nitrate boils.

Ammonium nitrate is an unstable substance on heating and its decomposition may take place according to different patterns depending on the temperature (Cagnina et al. 2013, Chaturvedi & Dave 2013):



Reaction (1) occurs at a temperature below 160°C (433 K) (Urbański, 1985), leading to decomposition of ammonium nitrate into ammonia NH<sub>3</sub> and HNO<sub>3</sub> nitric acid. This process starts in ambient temperature but is very slow.

Proper distribution of ammonium nitrate begins at 185°C (458 K) according to the reaction (2) (Urbański 1985, Błasiak 1956). In its course ammonium nitrate decomposes into nitrous oxide, N<sub>2</sub>O and water H<sub>2</sub>O. Above 280 °C (553 K) decomposition proceeds more rapidly according to reaction (3) (Urbański 1955, Błasiak 1956), wherein the breakdown products of NH<sub>4</sub>NO<sub>3</sub> are nitrogen N<sub>2</sub>, nitrogen dioxide NO<sub>2</sub>, and water H<sub>2</sub>O. While at higher temperatures than 400 °C (673 K) decomposition proceeds with a strong burst of the distribution of proceeds according to reaction (4) (Urbański 1955, Błasiak 1956). Temperature of

explosion amounts to about 1500 °C (1773 K) (Urbański 1955, Błasiak 1956), the pulse of pressure – approximately 200 MPa for 0.5 · 10<sup>-5</sup> s and approximately 0.98 m<sup>3</sup> of gas is emitted from one kg of NH<sub>4</sub>NO<sub>3</sub>. According to recent data, ammonium nitrate may also be decomposed during the explosion in accordance with equation (5) (Saunders 1922).

It should be emphasized that the indicated temperatures, at which the initiation of the process occurs refers to normal pressure and pure ammonium nitrate. In the case of impurities, the decomposition of NH<sub>4</sub>NO<sub>3</sub> may occur at much cooler temperature. According to the authors of the equation, (1) is an unfavourable reaction from the viewpoint of use in the transport of seabed due to the fact that both reaction products (ammonia and nitric acid) are liquid and therefore have a relatively high density. The problem of the appearance of these decomposition products of ammonium nitrate according to equation (1) can be disregarded since the rate of this reaction at a temperature of approx. 169 °C (442 K) is very small (according to d) and during the day only 6% of total weight will decompose).

Another unfavourable option of decomposition of ammonium nitrate is (3). One of the products of the decomposition of nitrogen dioxide NO<sub>2</sub> which is a highly toxic gas and as a result of the reaction with water, a nitric acid and nitrous acid emerge, a compound which is chemically unstable and nitric acid is one of its breakdown products.



Consequently, we found that the decomposition of the ammonium nitrate is to be carried by the option (2) and (4) or (5).

## 3 EXPERIMENTAL EXAMINATION OF PERFORMANCE OF PYROTECHNIC REACTION OF AMMONIUM NITRATE DECOMPOSITION USING "GEOMETRIC" SUPPRESSION

In order to investigate the suitability of the use of pyrotechnics to transport in the aquatic environment ready-made special test stand was used (Fig. 1) which was constructed by the authors (Filipek & Broda, 2017). It consists of the combustion chamber (1) with a pyrotechnic charge, which is connected to the radio-controlled ignition. The chamber is connected to the tank (2) which is the expansion space of the gas, i.e. pyrotechnic reaction products. The volume of the reservoir (2) was approximately 3 · 10<sup>-4</sup> m<sup>3</sup> ± 10<sup>-5</sup> m<sup>3</sup>. The volume was not determined exactly because the experiments were aimed at determining the quality and not the quantity of pressure distribution over time. A pressure gauge (3), thermometer (4) and a check valve (5) for installation filling and leak testing are connected to the reservoir (2), at the end of which there is a valve (6) closed during the experiment or the leak test position. In order to empty the position of the gas being the reaction product after the completion of the experiment, the valve (6) is turned open.

Start of the experiment was made by loading the load chamber (1). Chamber (1) was then connected with the tank (2), the connection being sealed. Next step was to conduct a leak test. It consisted of raising pressure to 0.59 MPa using pump connected to the valve (5) and then after adjusting the temperature of compressed air (4) the ambient temperature for 1 hour, it was being checked whether the pressure was not falling. After confirming the leak tightness, the pressure was being lowered to 0.1 MPa. This pressure was the baseline for the experiments. Then the radio initiated pyrotechnic reaction followed. Pressure changes indicated by the pressure gauge (3) were recorded using a camera with a frequency of 25 frames per second. After the experiment gas products of the reaction were removed from the inside of the stand by opening the valve (6). Experiments and removal of the gas from the installation were performed in the open air. Because – as already mentioned – experiments aimed at determination of qualitative rather than quantitative distribution of pressure in time, the works on the quantitative analysis of the resulting products of combustion are continued.

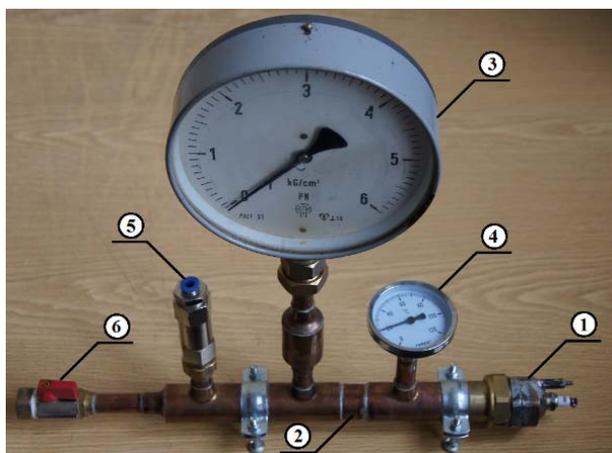


Figure 1. Laboratory test stand for the examination of the performance of pyrotechnic reaction with "geometric" suppression. (Filipek & Broda 2017)

As a result of experiments conducted without the use of geometric suppression we obtain waveforms in the form of a harmonic oscillator which is a non-preferred from the viewpoint of use in the transport of seabed and future operation of the tool based on this method due to the local sudden changes in stresses of the structure within a short time of a few dozen milliseconds.

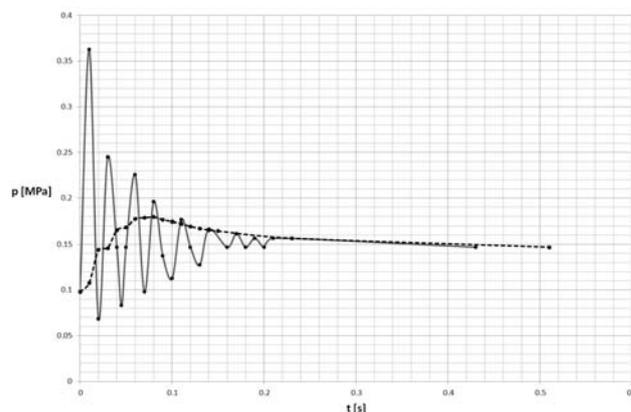


Figure 2. Comparison of pressure distribution during the pyrotechnic reaction with and without suppression.

After applying suppression we get the desired effect of suppression of oscillations. A sample selected from the many results we present in Figure 2. The Solid line shows the waveform's recorded without suppression, and the broken line shows the recorded waveform with the use of the insulation material.

#### 4 EXPERIMENTAL VERIFICATION OF THE SUITABILITY OF CONTROLLED DECOMPOSITION OF AMMONIUM NITRATE FOR TRANSPORT FROM THE SEABED

In the reaction according to the option (2) to two products: nitrous oxide and water. Wherein under normal conditions of nitrous oxide is a colourless gas with a sweet odour. It is used, among others in anaesthesia. From our point of view, more interesting is the use of gas in the car tuning and as an oxidizer in rocket engines (Herdy 2016). However, this gas is not indifferent to the environment because it is one of the main greenhouse gases. Therefore, in industry the reactions of thermal decomposition of nitrous oxide is used wherever it arises in technological processing. The reaction looks as follows:



Unfortunately, the process of natural decomposition takes place at a very high temperature of about 1600 °C (1873 K). However, with the use of catalysts, we are able to lower the temperature to about 200 °C (473 K) (Bryan & Pederson. 1995, Lohner et al. 2008). Since this gas has strong oxidizing properties, it can be used in the combustion reaction. An exemplary process includes the coal combustion process:



The starting point for carrying out the above (7.8) combustion of the decomposition reactions is to obtain the nitrous oxide decomposition of the ammonium nitrate according to reaction (2). This process is one of the fundamental processes producing nitrous oxide for technical purposes.

Experiment with nitrous oxide from the thermal decomposition of ammonium nitrate was carried out

using a laboratory stand constructed by the authors Figure 3 (Filipek & Broda, 2017). The stand comprises a combustion chamber (reactor) (1) connected to a pressure gauge (3) through a tube (2) in which the gas generated during the reaction is cooled in order to prevent damage to the pressure gauge (3). Tube (2) also serves as an expansion chamber. We use valve (4) for leak testing position and to remove gases after the experiment.

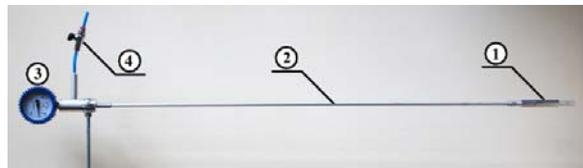


Figure 3. Stand for laboratory research with control of reaction. (Filipek & Broda, 2017)

The experiment was started by filling the load chamber (1). After checking leak tightness tool at 5 Bar the valve (4) was opened to align the pressure of the chamber atmospheric pressure and after it had reached the level, the valve (4) was closed and the reaction was initiated.

As a result of experiments we received a monotonic increase in pressure over time which is presented in the graph (Fig. 4).

The experiment was performed in two stages. In the first stage, decomposition of ammonium nitrate was carried out at 200 °C (473 K) (curve 1 in Fig. 4). The process lasted until it reached the values marked with P point on the graph (Fig. 4). Then decomposition process of ammonium nitrate was interrupted, which automatically led to a decrease in temperature (up to the ambient temperature), and hence the pressure (curve 2 on Fig 4). The decomposition process of ammonium nitrate was resumed after about 43200s (12 hours), wherein the decomposition temperature was adjusted to 250 °C (523 K). It showed an increase in pressure up to the point P (curve 3). The process of decomposition carried on according to curve 4. In the diagram in order to avoid excessive elongation of the time axis, and thereby reduce the transparency (readability) of figure waveforms obtained before and after cooling were combined in point P.

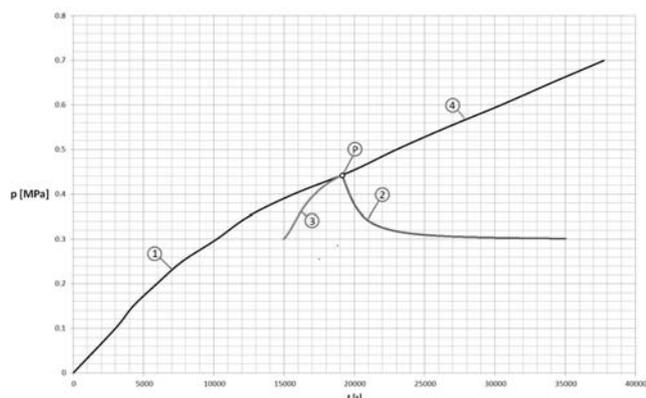


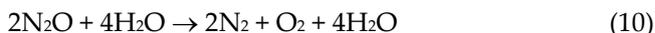
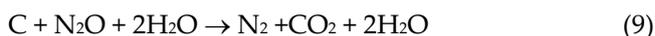
Figure 4. Pressure distribution during the reaction with performance control

There are methods to reduce the sudden increase in pressure over time in the event of the use of

decomposition of ammonium nitrate in accordance with (4) and (5), but then additional compounds in the form of the so called inhibitors must be employed. This method can reduce the rate of decomposition of ammonium nitrate (4.5). However, after the initiation of this reaction it is still a self-sustaining reaction with no possibility of stopping or changing the speed of its course.

##### 5 IDENTIFICATION OF DEPTH DOWN TO WHICH THE METHOD OF OUTPUT TRANSPORT EXCAVATED FROM THE SEABED BASED ON PYROTECHNIC REACTION WITH AMMONIUM NITRATE CAN BE USED

For the analysis we chose three options of chemical reactions, two for ammonium nitrate (distribution-distribution, distribution-combustion) and one for potassium nitrate (combustion) reaction which is a comparative reaction. In the case of thermal decomposition of ammonium nitrate reactions occur in two stages. The first step is the thermal decomposition of ammonium nitrate in accordance with equation (2). Next, the nitrous oxide is burned in accordance with equation (9) or decomposed as in equation (10).



However, in the case of potassium nitrate we chose reaction (Filipek & Broda 2016, 2017):



Reason for the choice and the reaction course is described in detail in the above cited publication.

We made our analysis assuming that products in the gas phase include nitrogen and oxygen in the liquid phase include water and carbon dioxide and calcium carbonate present in the solid phase. This assumption entailed the necessity of analysis at temperatures below condensation temperature (CO<sub>2</sub>, H<sub>2</sub>O) for a given pressure. Therefore we assumed that we would conduct the analysis of the pressure values above 5 MPa for two temperatures of 5 °C (278 K) and 15 °C (288 K). Under these conditions, both CO<sub>2</sub> and H<sub>2</sub>O are in liquid state. In addition, we assumed that in the case of nitrogen and oxygen being in the gas phase Amagat's law is satisfied (Szewczyk 2009), which states that the volume occupied by the gas mixture is equal to the sum of the volumes occupied by individual ingredients in the solution, assuming that each of the components has the same temperature and pressure. We used it also for the mixture of CO<sub>2</sub> and H<sub>2</sub>O due to the fact that we did not find any indication in the literature that this law is not fulfilled for this mixture. In our analysis, there are two-phase mixtures (gas-liquid), and obviously the dissolution of part of the vapour phase in liquid phase takes place, the mechanism of which is quantitatively described as a function of pressure (Henry's law) in a wide variety of literature (Carroll

1991,1999). In contrast, there is no clear information on the dependence of the volume of the two-phase mixture on the amount of the soluble component in the gas phase. It forced us to apply the Amagat's law also for this case. Let us schematically introduce the symbols as shown below (Fig. 5) where  $\rho_\alpha$  is density of the mixture,  $V_\alpha$  volume of the mixture,  $\rho_i$  is i-density of the i-component in the mixture, the volume  $V_i$  of the i-component in the mixture

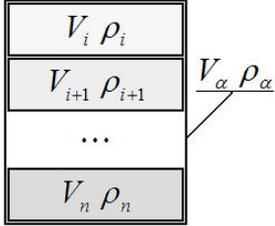


Figure 5. Concept diagram for  $\rho_\alpha$  determination

If we consider the three-component mixture with a mass  $m_\alpha$ , the density of suspension matter can be determined from the equation:

$$\rho_\alpha = \frac{m_\alpha}{V_\alpha} = \frac{\rho_i V_i + \rho_{i+1} V_{i+1} + \rho_{i+2} V_{i+2}}{V_i + V_{i+1} + V_{i+2}} \quad (12)$$

Introducing another variable

$$\phi_i = \frac{V_i}{V_{i+2}} \quad \text{and} \quad \phi_{i+1} = \frac{V_{i+1}}{V_{i+2}} \quad (13)$$

equation (12) takes a new form (14).

$$\rho_\alpha = \frac{\rho_i \phi_i + \rho_{i+1} \phi_{i+1} + \rho_{i+2} \phi_{i+2}}{\phi_i + \phi_{i+1} + 1} \quad (14)$$

Due to the possibility of replacing  $m_i$  component mass of mixture with the product of the number of moles of the  $n_i$  component and its  $M_i$  molar mass we obtain:

$$\phi_i = \frac{m_i \rho_{i+2}}{\rho_i m_{i+2}} = \frac{n_i M_i \rho_{i+2}}{n_{i+2} M_{i+2} \rho_i} \quad (15)$$

Substituting the above equation in (14) formula we obtain:

$$\rho_\alpha = \frac{\rho_{i+2} \left( \frac{n_i M_i}{n_{i+2} M_{i+2}} + \frac{n_{i+1} M_{i+1}}{n_{i+2} M_{i+2}} + 1 \right)}{\rho_{i+2} \left( \frac{n_i M_i}{n_{i+2} M_{i+2} \rho_i} + \frac{n_{i+1} M_{i+1}}{n_{i+2} M_{i+2} \rho_{i+1}} \right) + 1} \quad (16)$$

When considering a mixture of i-components, the above formula can be represented as:

$$\rho_\alpha = \frac{\sum_{i=1}^{n-1} \frac{n_i M_i}{n_n M_n} \rho_n + \rho_n}{\sum_{i=1}^{n-1} \frac{n_i M_i}{n_n M_n} \frac{\rho_n}{\rho_i} + 1} = \frac{\sum_{i=1}^n \frac{n_i M_i}{n_n M_n} \rho_n}{\sum_{i=1}^n \frac{n_i M_i}{n_n M_n} \frac{\rho_n}{\rho_i}} \quad (17)$$

On the Internet at [http://www.peacesoftware.de/einigewerte\\_e.html](http://www.peacesoftware.de/einigewerte_e.html) we can find an algorithm - a tool which has been used to determine the desired density of nitrogen, oxygen, carbon dioxide and water, both for liquid phase and gas phase. In order to determine the maximum depth down to which the method can be used to transport the excavated material from the seabed based on the pyrotechnic reaction using ammonium nitrate it is assumed that the model is a multi-phase system. The gas phase includes nitrogen and oxygen, the liquid phase includes carbon dioxide and water and the solid phase - potassium carbonate (in the case of the comparative reaction). The maximum depth at which the transport will be possible is determined from the comparison of relative density of the fluid surrounding the transport system  $\rho_p$  to the density of the multiphase  $\rho_\alpha$  as a function of pressure:

$$\rho_\alpha(p) = \frac{\rho_{i+2}(p) \left( \frac{n_i M_i}{n_{i+2} M_{i+2}} + \frac{n_{i+1} M_{i+1}}{n_{i+2} M_{i+2}} + 1 \right)}{\rho_{i+2}(p) \left( \frac{n_i M_i}{n_{i+2} M_{i+2} \rho_i(p)} + \frac{n_{i+1} M_{i+1}}{n_{i+2} M_{i+2} \rho_{i+1}(p)} \right) + 1} \quad (18)$$

If the ratio is greater than 1, then the process of ascent would be theoretically possible. When  $\rho_\alpha = \rho_p$  the process of ascent will not be possible. The values of density necessary to designate the  $\rho_\alpha$  depending on the pressure were calculated using the available Internet ([http://www.peacesoftware.de/einigewerte\\_e.html](http://www.peacesoftware.de/einigewerte_e.html)) application. The resulting amounts are shown in Figure 6. The results presented were obtained at two temperatures. The dashed line shows the waveforms for the temperature of 5°C (278K), while the solid line shows the waveforms implemented at 15°C (288K).

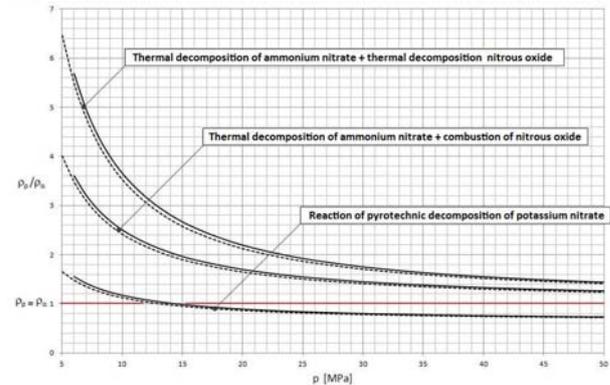


Figure 6. Comparison of the three courses of reactions as considered for the ascent.

Presented in the graph above, the pressure falls within the range of 5 MPa (liquid phase of carbon dioxide and water) to 50 MPa (the critical point for oxygen). Unfortunately, the Internet application ([http://www.peacesoftware.de/einigewerte\\_e.html](http://www.peacesoftware.de/einigewerte_e.html)) does not allow the calculation of the pressures above the critical point for oxygen. However, accepted pressure range covers the depth on Clarion-Clipperton zone in the Pacific.

## 6 CONCLUSIONS

With the ability to determine the density  $\rho_\alpha$  we are able to determine the the energy  $E$  needed to lift transport module, which amounts to (Filipek & Broda, 2016):

$$E = E_p \frac{\rho_p}{\rho_p - \rho_\alpha} \quad (19)$$

where  $E_p$  is the theoretical potential energy needed to move the weight between two points immersed in liquid  $\rho_p$  (e.g. the bottom of the sea, the surface of the sea). From the above formula we can conclude that the most significant amount responsible for the process of the ascent is  $\rho_\alpha$  parameter. The change of this parameter as a function of pressure and hence the depth was shown in Fig 6 for the three cases. It can be seen that the reactions (2) and (10) are the most effective, from the point of view of transportation from the seabed, which is the thermal decomposition of ammonium nitrate to nitrous oxide and water, and then the thermal decomposition of nitrous oxide. The reaction course in accordance with (2) and (9), which is the thermal decomposition of ammonium nitrate to nitrous oxide and water and then burning the resulting nitrous oxide is a less favourable reaction in view of equation (19) but both discussed cases can be successfully applied to the depth at which polymetallic nodules occur on the Clipperton-Clarion zone.

Unfortunately, the third method of using the potassium nitrate would not allow us to achieve a satisfactory depth. Also, at the smaller depths (up to about 1 km) it is less energetically favourable. Although the best results are achieved using (2) and (10), however, due to the need to ensure adequate temperature of thermal decomposition of nitrous oxide it may become the method technically more difficult to implement than the method based on (2) and (9) wherein the combustion process of nitrogen dioxide appears to be simpler for implementation. This aspect must be analysed in detail, which the authors will present in subsequent publications after completion.

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