

Original article

## Application of thermal analysis and calorimetry for assessment of safety and quality of nitrogen fertilizers

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### ABSTRACT

*The paper presents the results of studies carried out using differential thermal analysis (DTA) and differential scanning calorimetry (DSC), conjugated with thermogravimetry (TG). Measurements were made for samples with differentiated compositions, mainly consisting of ammonium nitrate with fertilizer purity and compounds that may be present in nitrogen fertilizers as potential additives or contaminants. The possibilities of applied techniques and recommendations concerning proper selection of measurement conditions are described. Furthermore, the method of interpretation of the obtained results is presented, which allows evaluating the thermal stability of the tested mixtures for the safety and quality of nitrogen fertilizers.*

### KEYWORDS

*ammonium nitrate, fertilizers, safety, quality, calorimetry, thermal analysis*



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## Introduction

Ammonium nitrate is the main and most important ingredient in commonly used agricultural nitrogen fertilizers. This compound contains 35% of nitrogen, as per elemental nitrogen. This makes it a rich source of essential macronutrients in plants in two different forms: ammonium and nitrate. In order to calm the needs of the fertilizer market, ammonium nitrate is produced in huge quantities; in Poland it amounted over 1.3 million tons in 2015 [Produkcja... 2016]. It is used both for the production of solid fertilizers, such as ammonium nitrate or nitrate, and liquid fertilizers. The production, storage and transport of these preparations are, however, accompanied by a number of safety hazards arising from the risk of uncontrolled ammonium nitrate decomposition [Biskupski et al. 2006; Kolaczowski et al. 2000]. According to the commonly accepted theory, the first step in the breakdown of ammonium nitrate is its endothermic dissociation into ammonia and nitric acid. The second stage consists of highly exothermic

complex reactions with products of dissociation. Depending on the conditions in which the breakdown occurs, its course may be varied. This may consequently lead to the formation of slightly different products as shown in Table 1 containing the summary equations of decomposition reactions of ammonium nitrate [Oxley et al. 2002; Chaturvedi and Dave 2013].

**Table 1.** Equations of ammonium nitrate decomposition reactions and their standard enthalpy values

Decomposition reaction	$\Delta H^{\circ}_{r,298}$ [kJ/mol]
$\text{NH}_4\text{NO}_3 \rightarrow \text{N}_2\text{O} + 2\text{H}_2\text{O}$	-124.5
$\text{NH}_4\text{NO}_3 \rightarrow \text{N}_2 + 1/2 \text{O}_2 + 2\text{H}_2\text{O}$	-206.0
$\text{NH}_4\text{NO}_3 \rightarrow 1/2 \text{N}_2 + \text{NO} + 2\text{H}_2\text{O}$	-115.8
$\text{NH}_4\text{NO}_3 \rightarrow 3/4 \text{N}_2 + 1/2 \text{NO}_2 + 2\text{H}_2\text{O}$	-189.1
$5\text{NH}_4\text{NO}_3 \rightarrow 4\text{N}_2 + 2\text{HNO}_3 + 9\text{H}_2\text{O}$	-218.2

Source: [own elaboration].

An elevated temperature or a mechanical stimulus can be identified as the direct cause of decomposition and even detonation of ammonium nitrate. The susceptibility of ammonium nitrate to these agents may, however, be increased by the presence of some destructive impurities. The circumstances in which this was first perceived were recorded in the history of chemistry and of humanity as a particularly tragic event, due to the number of victims and the value of material losses caused by uncontrolled self-accelerating explosive decomposition of ammonium nitrate [Pittman et al. 2014]. Since then, the influence of selected salts, metals and organic compounds on the transformation of ammonium nitrate has been intensively studied. Despite several decades of experience, in many cases, the nature of these interactions has not been fully understood and described. However, many researchers have confirmed that both the presence of chloride ions and the acidic environment strongly accelerate the decomposition reactions of ammonium nitrate, and their collective action may have a synergic character. In case of chlorides, even their small amount is able to significantly affect the stability of ammonium nitrate, reducing its decomposition temperature by up to 70°C. This action is attributed, among others, to the reaction of chloride ions with nitronium ions. So far, however, there has been no coherent theory on the mechanism of these phenomena [Oxley et al. 2002; Chaturvedi and Dave 2013; Sun et al. 2005; Li and Koseki 2005]. Other compounds, such as certain carbonates or phosphates, when in appropriate amounts may thermally stabilize the ammonium nitrate by increasing the temperature of its decomposition or by reducing the exothermic effect associated with the process [Oxley et al. 2002; Rubtsov et al. 2005]. The production method of fertilizers can also significantly affect the ammonium nitrate's susceptibility to detonation. In this case, the porosity of granules is the most important parameter, which should be as small as possible from a safety point of view.

Due the above reasons, the composition of nitrogen fertilizers and the conditions of their production must be properly selected and strictly controlled. For this purpose, it is necessary to assess the impact of selected additives and potential pollutants on the

thermal stability of mixtures with ammonium nitrate under varying temperature conditions. Such tests can be performed using thermal analysis techniques [Poplawski et al. 2016].

## 1. Research methodology

The study was carried out using two techniques: differential thermal analysis (DTA) and differential scanning calorimetry (DSC), both conjugated with thermogravimetry (TG). The STA 449 F3 Jupiter thermal imager, made by Netzsch Company, was used for measurements in both modes. The employed measuring crucibles were made of alumina. Open crucibles with the capacity of 300  $\mu\text{L}$  were adopted in the case of DTA, while for the DSC measurements – crucibles with the capacity of 85  $\mu\text{L}$ , both open and with lids made of alumina. In all cases, the samples analyzed were heated to 450°C at the speed of 5 K/min in the synthetic air atmosphere with the total flow of 120 mL/min. Each measurement was preceded by heating an empty crucible to 600°C in order to remove possible impurities.

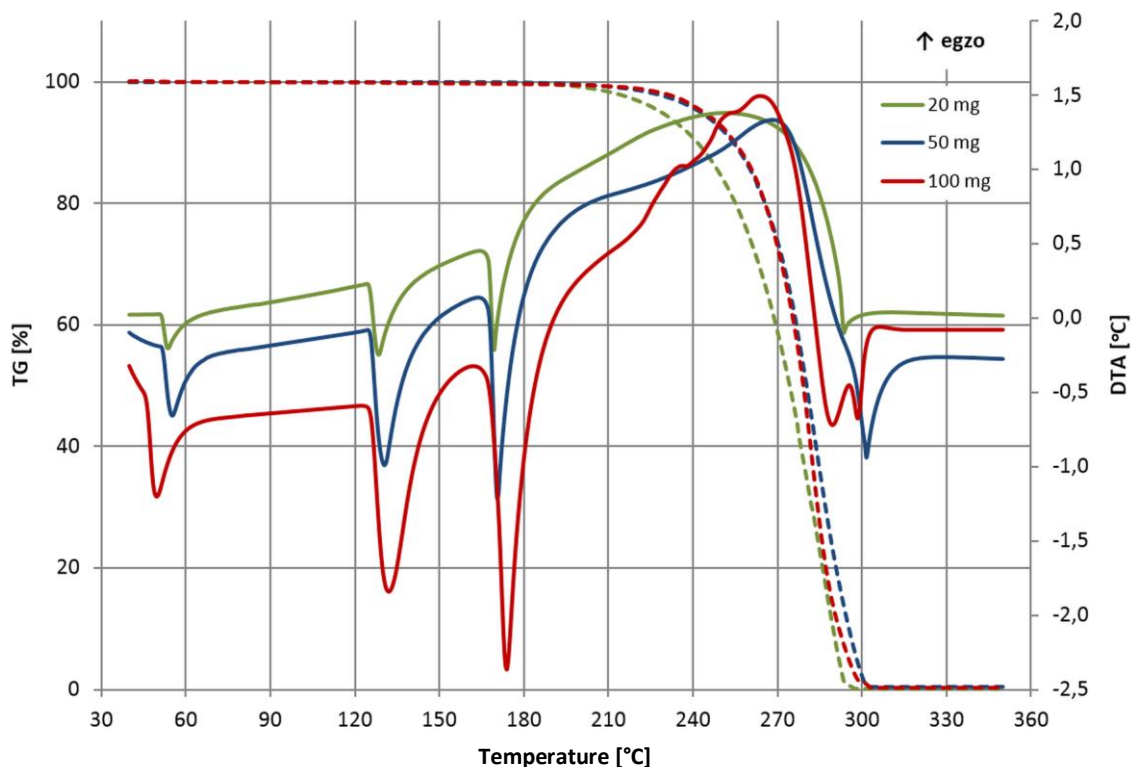
The study used pure ammonium nitrate fertilizer, taken directly from the neutralization node in one of the national plants producing nitrogen fertilizers. In addition, potassium chloride (AR, POCh) and two types of dolomites with a grain size of <125  $\mu\text{m}$  were adopted as the source of lime and magnesium carbonates. The composition of minerals was as follows: Dolomite D1 (29.3% CaO, 18.5% MgO), Dolomite D2 (39.6% CaO, 13.4% MgO). The calcium and magnesium content was determined by dissolving the raw materials in hydrochloric acid and then titrating the samples with a solution of disodium edetate at pH = 10, against eriochrome black T and then by calcination.

The study of ammonium nitrate alone without additives was carried out both with the use of thermal analysis and calorimetry. In the first case three analogous measurements of 20, 50 and 100 mg samples were made. For DSC, two measurements of 25 mg ammonium nitrate samples were performed using an open and then closed crucible. All other measurements were also made using DSC, a crucible with a lid and samples of approximately 25 mg. A mixture of ammonium nitrate with potassium chloride was prepared by blending and thorough trituration in the mortar of both ingredients. Their quantities were adjusted so as to produce a potassium chloride content of 1.00% in the sample, which is 0.46% of chloride ions by mass. In the same manner, two mixtures of ammonium nitrate with D1 and D2 carbonate fillers were prepared. The ratio of the components was adjusted so that the total nitrate content of the both mixtures was typical for calcium ammonium nitrate, that is 27.5% of N mass.

## 2. The analysis and interpretation of results

Contemporary thermal analysis and calorimetry devices allow measurements made using very small amounts of the test substance, ranging from several to several hundred milligrams. This reduces the consumption of samples, which are often very laborious and expensive to produce. First of all, however, the accuracy of the measurement increases with the reduction of the sample size. This is due to the lower inertia of heat

and mass transfer, so that a researcher is provided with more certainty that the phenomena recorded by the measuring device take place in the entire volume of the analyzed sample, not just on its surface or at the sides of the crucible. In most cases, the weight loss of the sample will therefore have a positive effect on the quality of results. Due to the specific phenomena occurring during the decomposition of some compounds, this is not always desirable.



**Fig. 1.** Results of the DTA-TG analysis of three samples of ammonium nitrate with differentiated mass  
Source: [own elaboration].

Figure 1 shows the results of the thermal analysis of three samples of ammonium nitrate with varying masses. The course of endothermic phase transitions characteristic for this compound is very similar in all three cases. It differs only by the intensity of the signal, which results from the different sample mass. The exothermic effect occurring above 200°C is associated with the decomposition of ammonium nitrate, which is becoming less and less apparent as the sample's mass is reduced. In the case of 20 mg ammonium nitrate, it cannot be clearly stated whether the increase in the DTA curve is related to an exothermic process or results only from the very course of the baseline itself. By interpreting the result of this measurement, it can be mistakenly concluded that the ammonium nitrate breakdown is not accompanied by an exothermic effect. The cause of the above-described differences is the two-stage course of decomposition of this compound. In the first place ammonium nitrate undergoes endothermic dissociation. In the second stage the formed nitric acid and ammonia are subjected to a series of secondary, strongly exothermic reactions. If dissociation products previously

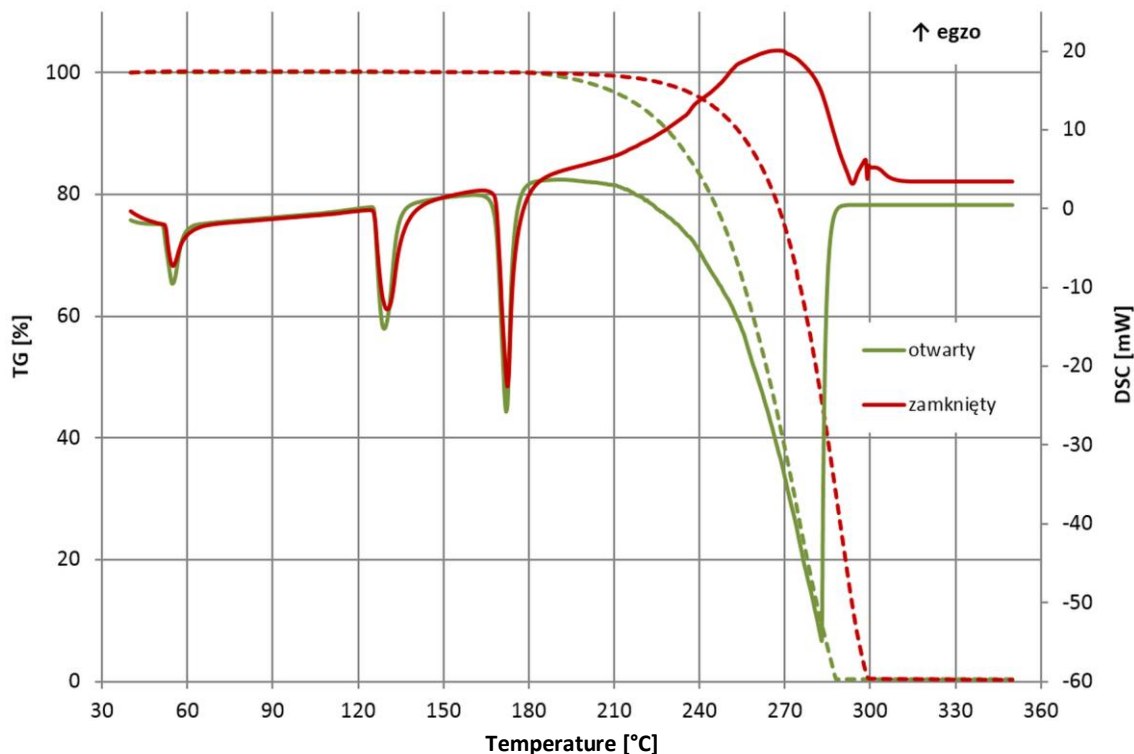
leave the reaction system and are dispersed, secondary reactions will not be able to occur, or they will go beyond the measuring system and consequently their thermal effect will not be recorded. The endothermic evaporation of ammonium nitrate and products of its dissociation is a surface process. Consequently, while maintaining the same dimensions of the measuring crucible, the smaller the mass of the sample, the greater the ratio of the surface of the sample to its volume, and thus the greater the share of endothermic surface processes in the total recorded thermal effect. Under certain circumstances, the dominant part of the sample may evaporate, and only its small amount undergoes secondary exothermic reactions and consequently thermal effects of the two processes can be compensated as shown in Figure 1 for a sample of 20 mg ammonium nitrate. The results of the thermogravimetric analysis seem to confirm the above observations. In the range of temperatures of about 210-280°C, a significantly greater mass loss is observed for the smallest sample compared to the other two samples. In the absence of a noticeable exothermic effect, this may mean the intensified evaporation of ammonium nitrate and products of its dissociation.

Based on the above example, it can be seen that uncritical seeking for maximum sample weight reduction is not always justified. By having one type of crucibles of defined dimensions, it would be advisable to carry out preliminary measurements using samples with various masses to select the optimum sample size allowing certain processes to be exposed. As explained above, the parameter determining the evaporation of a sample is not so much its mass but rather the surface/volume ratio. For the fixed dimensions of the crucible, this parameter can be influenced by the amount of the sample. When deciding on a measuring vessel, a crucible with the smallest diameter or even a capillary can be chosen. In the latter case, the surface of the sample remaining in contact with the environment is so negligible that the evaporation is virtually eliminated. Thus, using a capillary it is possible to record the exothermic effect of ammonium nitrate decomposition even for samples of a few milligrams [Oxley et al. 2002].

Another way to limit the sample's evaporation is to use a crucible with a lid. Figure 2 shows the results of two ammonium nitrate samples with the identical mass measured using differential scanning calorimetry. An open crucible was used for one measurement, while in the second one – the same crucible, but with a lid with a small hole in the middle. When analyzing the DSC curves for both measurements, it can be seen that when an open crucible is used, the loss of sample mass is accompanied by an endothermic effect only. The application of a small sample weight of 25 mg and an open crucible for the DSC analysis, the diameter of which is greater than that of the DTA crucible, made the surface endothermic phenomena dominate the entire decomposition process of ammonium nitrate. The vast majority or even the whole sample evaporated in the form of ammonia, nitric acid and ammonium nitrate.

The use of a crucible with a lid prevented the removal of ammonium nitrate dissociation products from the measuring system, allowing secondary reactions with their participation to occur. This effect is clearly visible in the form of a highly exothermic effect at the temperature of above 200°C. The course of the TG curve confirms that when an open crucible is used, the mass loss begins at a significantly lower temperature,

whereby the sample evaporates before it is exothermically transformed. The mass exchange conditions between the measuring system and its surroundings can therefore have a significant impact on the processes taking place during the measurement, and hence on the nature of the recorded phenomena.

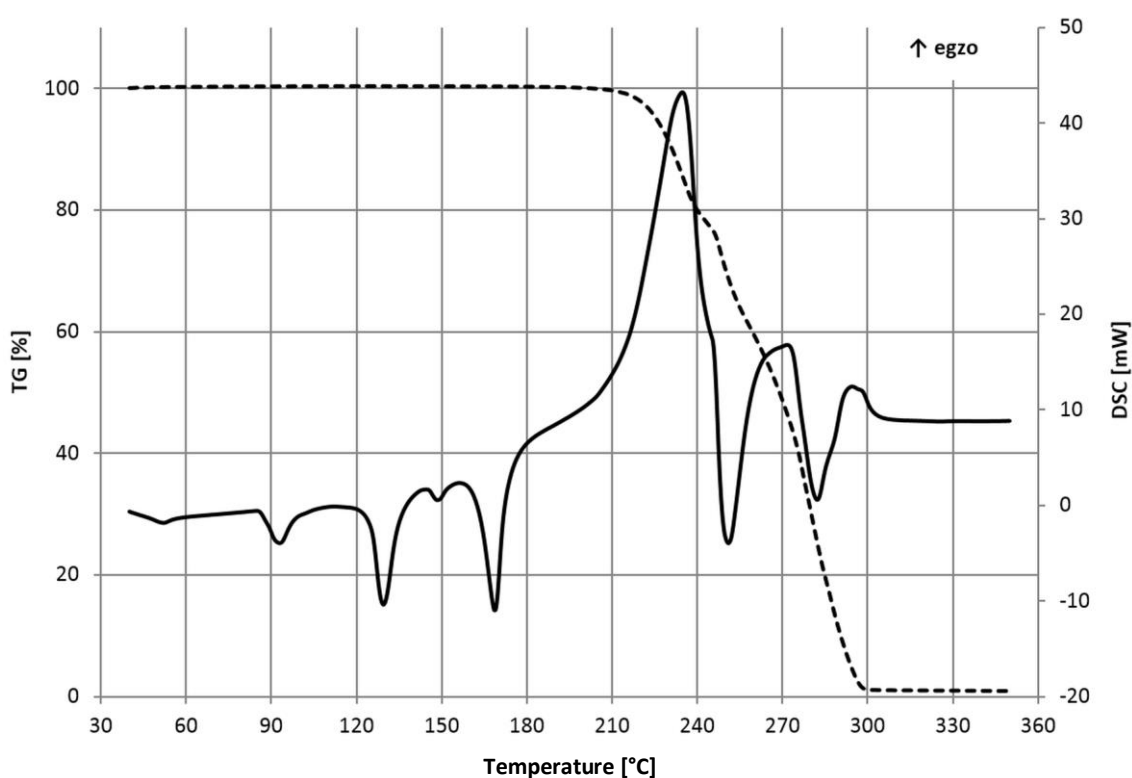


**Fig. 2.** Results of the DSC-TG analysis of ammonium nitrate in open and closed crucibles  
Source: [Own elaboration].

The use of an open large diameter crucible containing a small amount of ammonium nitrate may lead to the conclusion that ammonium nitrate's breakdown is endothermic. The identification of the recorded phenomena as decomposition of the ammonium nitrate in general does not seem to be a mistake since, as a result of endothermic dissociation the compound actually decomposes to ammonia and nitric acid. However, using such measurements to assess the technical safety of ammonium nitrate processes or products seems to be a critical omission and may present catastrophic consequences of decisions made resulting in the apparent sense of safety. By examining, for example, the effect of certain additives on the thermal stability of ammonium nitrate and only the endothermic effects on the DTA or DSC curve it can be deduced that the test additive is effective, whereas the observed phenomena can only result from poorly selected measurement conditions.

The negative impact of chloride ions on the thermal stability of ammonium nitrate was often described in the literature. Figure 3 shows the result of the DSC-TG measurement of the mixture containing both components. The maximum stream of generated heat, associated with the breakdown of ammonium nitrate catalyzed by chlorines, is more than double and appears at the temperature lower by 33°C compared to the

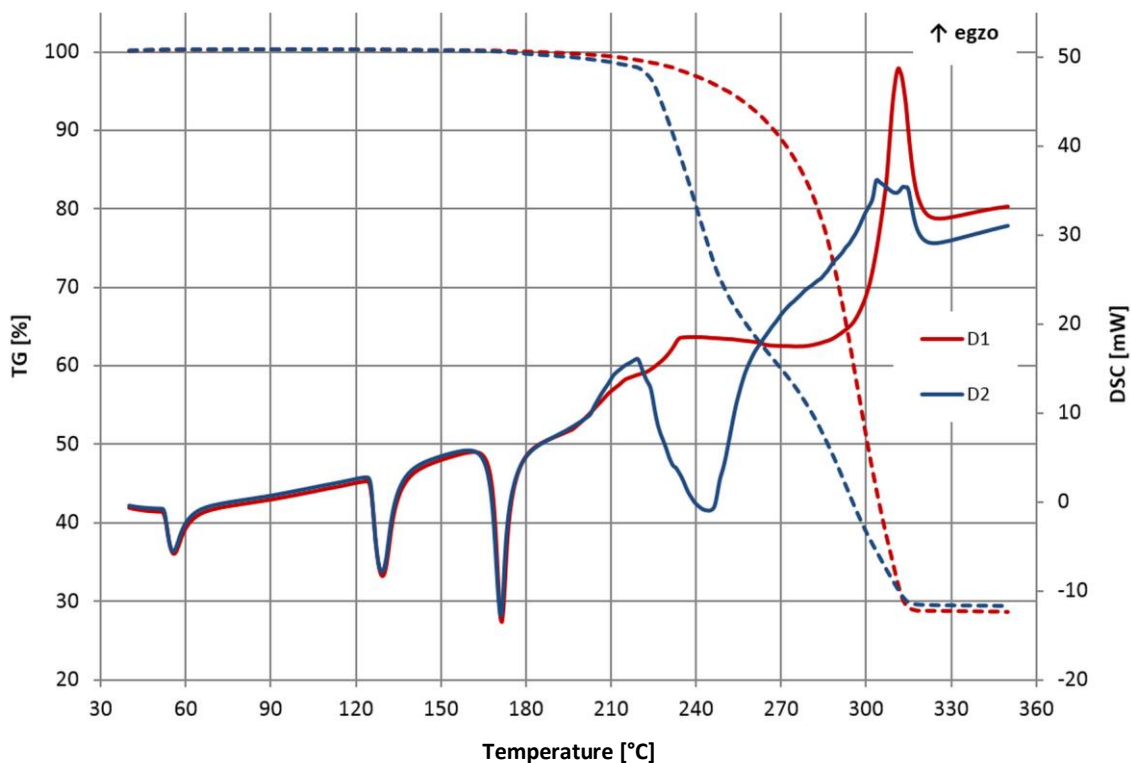
pure substance (Figure 2, a closed crucible). This significant difference was due to the addition of very small amounts of chloride ions below 0.5% of mass, which strongly suggests their destabilizing effect on ammonium nitrate. Thus, calorimetric measurements performed under appropriately chosen conditions can identify significant safety risks coming from the thermal stability of fertilizer mixtures. However, attention should be paid to the occurrence of the chlorides' period of induction, broadly described in the literature, followed by the destabilizing effect of these ions on ammonium nitrate [Colvin et al. 1965; Keenan and Dimitriadis 1962; Saunders 1922; Sinditskii et al. 2005]. As a result of this phenomenon, the heating rate in dynamic measurements should be chosen to prevent overheating of a sample during the induction period, which could result in an apparent overestimated temperature of the inception of chlorides' adverse effect. It seems worth comparing the results of this type of analysis with those of measurements performed under isothermal conditions.



**Fig. 3.** Results of the DSC-TG analysis of ammonium nitrate with addition of 1% potassium chloride  
*Source: [own elaboration].*

Calcium and magnesium carbonates belong to the additives used to improve the safety and physicochemical properties of nitrogen fertilizers. Their source is usually dolomites and limestone. This type of fertilizer, containing 20-28% of nitrogen by mass and at least 20% of filler by mass is called calcium ammonium nitrate. Under the threat of the uncontrolled temperature rise, carbonates may react with ammonium nitrate leading to the appearance of more thermally stable calcium and magnesium nitrates and the strongly inhibiting process of ammonia decomposition. In addition, the reactions with

carbonates are of endothermic nature, so that the ammonium nitrate decomposition reaction can be extinguished. The choice of suitable carbonate filler requires the assessment of its reactivity to ammonium nitrate, and this can be carried out using techniques of thermal analysis.



**Fig. 4.** Results of the DSC-TG analysis of mixtures of ammonium nitrate with dolomites D1 and D2  
*Source: [own elaboration].*

Figure 4 shows the results of the DSC analysis of the two mixtures containing ammonium nitrate and the selected dolomite in proportions corresponding to the commercial fertilizer. The composition of the dolomites was varied. In both cases, however, the reduced exothermic effect of pure ammonium nitrate in the temperatures ranging 200-280°C is observed. Possible phenomena of heat generation can be seen in the case of both calcium ammonium nitrates at a much higher temperature of over 300°C. The analysis of the TG curve evidences that Dolomite D2 is much more reactive than ammonium nitrate. At the temperature of up to 270°C, the sample containing this mineral was decomposed in over 40%, whereas in the case of Dolomite D1 it was only about 11%. The greater amount of the reacted filler results in a significant amount of ammonia being released, which positively influences the thermal stability of the sample. Moreover, the endothermic effect associated with these changes makes it possible to significantly reduce the amount of heat generated by the decomposition of ammonium nitrate. With low reactive fillers, inhibitive effects of ammonia loss their significance if the pressure of ammonia produced in the reaction with ammonium nitrate is less than the equilibrium ammonia pressure in the dissociation reaction of ammonium



nitrate whose intensity increases with temperature. For these reasons, the use of Dolomite D2 as a filler in nitrogen fertilizers seems to be more justified.

Comparing the composition of both examined minerals, it can be seen that the more reactive dolomite contains more calcium carbonate. It was also been observed from the earlier studies that the reactivity of fillers increases with the increase of calcium carbonate [Poplawski et al. 2016]. The grading of the dolomite or limestone used, their specific surface, humidity or the presence of other ingredients can also affect the filler's reactivity. The content of calcium carbonate, however, seems to be the most important factor. The same research has also proven that over-reactivity of the mineral may be disadvantageous from the standpoint of the finished product quality. The high content of calcium carbonate results in lowering the reaction temperature between the filler and ammonium nitrate.

If the temperature is less than 180°C, there is a risk that the intensive ammonia and carbon dioxide release may emerge during the production process, whereby the granules of the product may be more porous and brittle. In addition, the resulting calcium and magnesium nitrates change the composition of the fertilizer and can increase its hygroscopicity, which leads to the finished product's agglomeration during storage and transport. All these factors adversely affect the quality of the fertilizer, but also indirectly its safety. Increasing porosity of granules containing ammonium nitrate can lead to their increased susceptibility to the transfer of detonation. The reactivity of the effective carbonate filler should not be too low or too high. The optimal composition of the mineral can be selected using techniques of thermal analysis and interpreting properly the obtained results.

## Conclusion

Differential thermal analysis and differential scanning calorimetry are techniques widely used to evaluate the thermal stability of both pure substances and their mixtures. They allow the recording of any transformations that results in the absorption or generation of heat. Combined with thermogravimetry, they can provide a wealth of information necessary to optimize the conditions of a particular production process or to select appropriate raw materials. However, the application of these techniques to evaluate the effects of selected additives on the safety and quality of ammonium nitrate based fertilizers requires an understanding of the processes taking place during the measurement. The specific nature of the decomposition of ammonium nitrate entails the risk of incorrect selection of measurement conditions.

Conclusions drawn on the basis of such results may lead to a catastrophic decision-making outcome. When conducting the thermal analysis of samples containing ammonium nitrate, particular attention should be paid to the phenomenon of endothermic evaporation of ammonium nitrate and nitric acid and ammonia resulting from its dissociation. While this process is not intended to be the sole subject of research, it is desirable to limit its intensity in order to allow for secondary exothermic reactions that are a follow-up of ammonium nitrate's breakdown. This is especially crucial if meas-

urements are made in terms of technical safety assessment. The contribution of individual stages in the distribution can be influenced by the appropriate dimensions of a measuring crucible, a sample mass or additional items, such as a crucible lid. It has been shown in the paper that, under appropriate conditions of measurement, thermal analysis techniques can be used to identify, inter alia, the hazards associated with the destabilizing effect of chloride ions on ammonium nitrate, and the effects of selected carbonate fillers on the safety and quality of nitrogen fertilizers as well.

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### **Author contributions**

All authors contributed to the interpretation of results and writing of the paper. All authors read and approved the final manuscript.

### **Ethical statement**

The research complies with all national and international ethical requirements.

### **ORCID**

Dariusz Poplawski – The author declared that he has no ORCID ID's

Maciej Kaniewski – The author declared that he has no ORCID ID's

Jozef Hoffmann – The author declared that he has no ORCID ID's

Krystyna Hoffmann – The author declared that she has no ORCID ID's

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