

A study of chemical micromechanism governing detonation initiation of condensed explosive mixtures by means of differential thermal analysis[☆]

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Abstract

Thermal reactivities of 11 commercial explosives with different nitric ester contents, oxidizing systems of 10 classical emulsion explosives (water in oil (W/O) type), four of these fortified by 1,3,5-trinitro-1,3,5-triazacyclohexane (RDX) or pentaerythritol tetranitrate (PETN), and three samples of W/O emulsion explosives were examined by means of differential thermal analysis (DTA) and data were analyzed according to the Kissinger method. The reactivities, expressed as the $E_a R^{-1}$ slopes of the Kissinger relationship, correlate with the squares of the detonation rates of the corresponding explosive mixtures in the form of the modified Evans–Polanyi–Semenov (E–P–S) equation. In explosive mixtures, the thermal reactivity of the oxidizing system and/or its mixture with a high explosive replaces the primary thermal reactivity of explosophore groups in the individual energetic materials. Addition of any inorganic nitrate to ammonium nitrate in a classical W/O, emulsion explosive was found to result in a decrease in the detonation parameters of the resulting mixture.

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1. Introduction

From present-day knowledge, there is a distinct similarity of the primary fragmentations in both the detonation and the thermal decomposition of explosive molecules. The similarity or identity, respectively, of the primary mechanism of low-temperature and detonation reactions is a topic of numerous papers [1–17]. The identity is also confirmed by the most striking

pieces of experimental evidence. First of all they include the evidence (obtained with the help of Raman spectroscopy and XPS) of primary fission of the N–NO₂ bond in 1,3,5-trinitro-1,3,5-triazacyclohexane (RDX) exposed to a shock wave [18,19]. On the basis of the deuterium kinetic isotope effect (DKIE), it was proved [20,21] that the rate-limiting step of the thermal decomposition of 2,4,6-trinitrotoluene (TNT) in the condensed state and that of initiation of its detonation are identical. It is also possible to add the evidence provided by furoxanes and furazanes in the XPS spectrum of 1,3,5-triamino-2,4,6-trinitrobenzene (TATB) exposed to shock [22–24]—the pyrolysis of *ortho*-nitroanilines is a method of synthesis of benzofurazane [25] and, in the case of

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1,3-diamino-2,4,6-trinitrobenzene (DATB), this reaction leads to 4-amino-5,7-dinitrobenzofurazane [26]. The said identity is reflected in a relationship between the kinetics of the low-temperature thermal decomposition of the energetic materials and the reaction rates in the reaction zone of their detonation [27,28].

The homolytic character and the identity of the primary fission in both the detonation and the low-temperature thermal decomposition of energetic materials were a motive for Zeman et al. [3] to use the Evans–Polanyi–Semenov (E–P–S) equation [29,30] in the study of the chemical micromechanism governing the initiation of detonation of energetic materials [3,4,7–9,11–13,16,17,31]. The original E–P–S equation describes a relationship between the activation energies, E , of most substitution reactions of free radicals and the corresponding heats of reaction, ΔH , [29,30]:

$$E = B \pm \alpha' \Delta H \quad (1)$$

where B is the constant for the given homological series, α' being a non-dimensional gradient [29,30]. It is valid for narrow sets of substance structures and

documents that the strength of bond being split is a decisive factor in the given reaction [29,30]. Substitution of ΔH by heat of explosion Q and E by activation energy (E_a) of the low-temperature thermal decomposition has led to the first version of the modified E–P–S equation [3,4,7,16,17] in the general form:

$$E_a = C \pm \alpha Q \quad (2)$$

where E_a and C are in kJ mol^{-1} and α (the difference between Eq. (1)) is in g mol^{-1} [3,4,7].

Eq. (2) is applicable for the detonation transformation of energetic materials [3,4,7,16,17]. The heat of explosion in this relationship can be substituted by the square of the detonation rate (D^2) because definendum exists [32,33]:

$$Q = D^2 \{2(\gamma^2 + 1)\}^{-1} \quad (3)$$

where γ is the polytropy coefficient whose value for high explosives ranges from 2.79 to 3.48 [32].

For exploring relationships of the type of Eq. (2), we can use the results of differential thermal analysis (DTA) [3,31]. In the case of plastic explosives, the Kissinger DTA method [31,34] proved useful. The

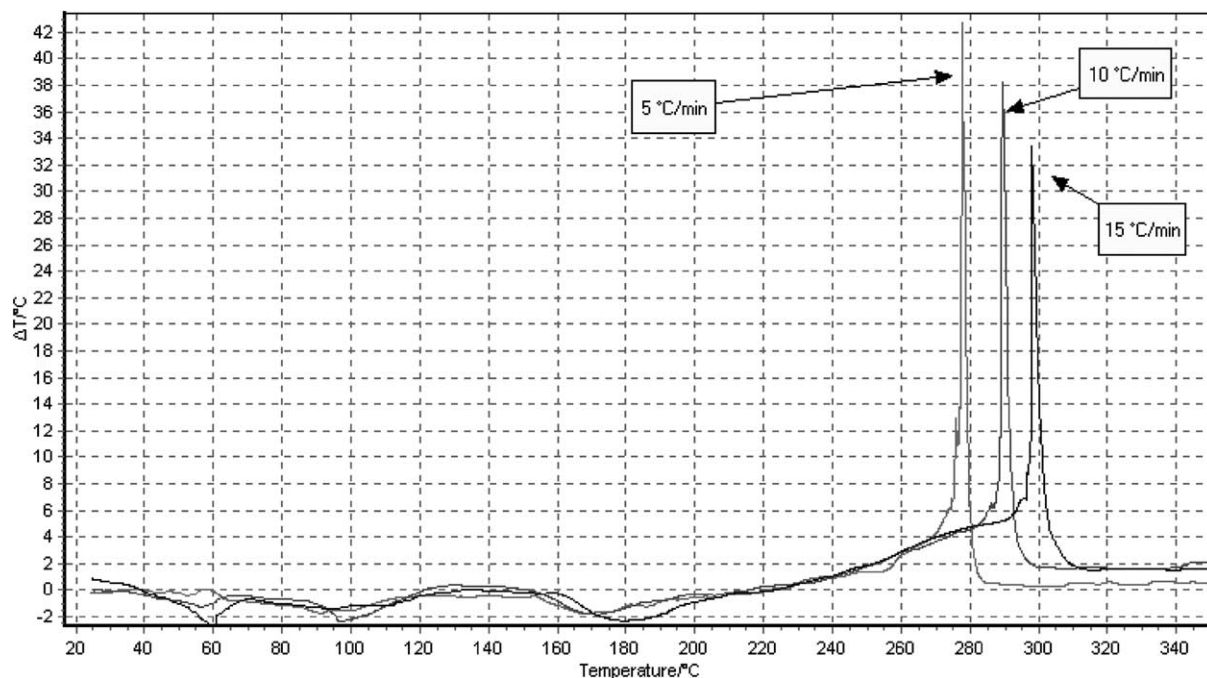


Fig. 1. DTA records of the oxidizing system “ammonium nitrate–cobalt(II) nitrate” (i.e. mixture AN–CoN according to the Table 2) leaching rates: 5, 10 and 15 K min^{-1} and sample mass 0.05 g.

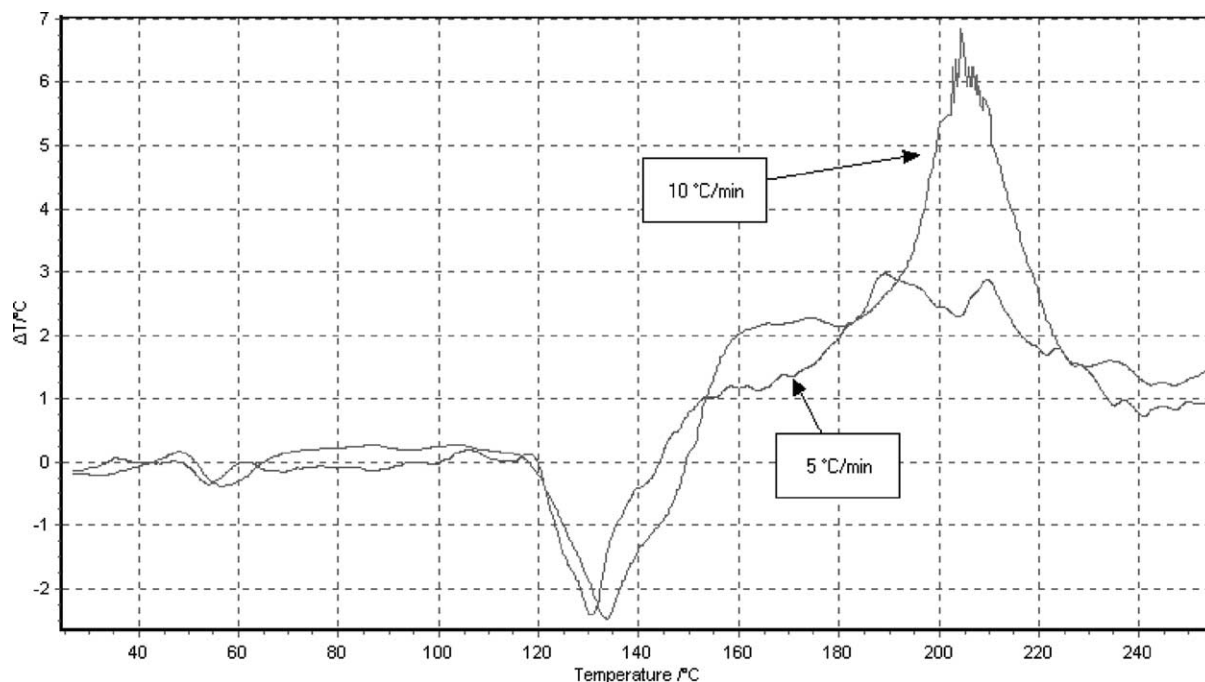


Fig. 2. Two DTA records of the gelatinous explosive Perunit 20 leaching rates: 5 and 10 K min⁻¹; temperature of the first peak of exothermic decomposition at the rate of 5 K min⁻¹ was taken for $E_a R^{-1}$ value calculation.

method plots $\ln(\phi/T_{\max}^2)$ against reciprocal peak temperature (T_{\max}) for a series of experiments at different heating rates, ϕ (see also Figs. 1 and 2 in the present paper). Using the slope in the Kissinger relationship, i.e. $E_a R^{-1}$, and taking into account Eqs. (3) and (2) were modified into the following form [31]:

$$E_a R^{-1} = b \pm aD^2 \quad (4)$$

where the D 's are the experimentally determined values of the rate of detonation. In the present paper, the earlier mentioned method and Eq. (4) have been used to study the detonation reactivity of explosive mixtures of types of emulsion and gelatinous industrial explosives.

2. Experimental

2.1. Materials

Some commercial explosives produced by Istrochem Bratislava (Slovak Republic) and Synthesia Pardubice (Czech Republic) were used in this study. The

explosives contained nitric esters as sensitizing agents. A survey of the explosives used, their rates of detonation and characteristics of their thermal reactivities are given in Table 1.

Samples of the water in oil (W/O) emulsion explosives were prepared using a simple apparatus consisting of a thermostat and container equipped with stirrer. The methods of preparation and the compositions of most of the explosives are described in [37]. The solution of oxidisers was heated to 120 °C and then slowly added to the container in which a preheated mixture of the fuels (oil and polybutadiene) with the emulsifier (95 °C) was agitated with the stirrer at 900 rpm. After adding the whole amount of the solution, the agitation was continued for about 2 min in order to obtain fine particles of the emulsion. The basic emulsion matrix was prepared from a solution of ammonium nitrate, polymer, oil and emulsifier. The addition of other nitrates, sodium (SN), calcium (CN), lithium (LiN), cobalt (CoN), copper(II) (CuN), nickel (NiN), potassium (PN) and aluminium (AlN), to the emulsion matrix was carried out in such a ratio as to maintain the oxygen balance within 0–1% O₂. Thereafter an

Table 1

Survey of the commercial explosives studied, their nitric esters contents and rates of detonation and thermal stabilities ($E_a R^{-1}$); products of Istrochem Bratislava and Synthesia Pardubice

| Commercial explosive | | | | Nitric esters content (mass%) | Rate of detonation (km s^{-1}) | Kissinger's slope [51] ($E_a R^{-1}$) ($\times 10^{-3}$ K) |
|---------------------------|-----------------|-----------|---------------|-------------------------------|---|---|
| Trade mark | Type | Producer | Utility | | | |
| Danubit 1 | Gelatinous | Istrochem | Rock blast | 20.7 | 5.929 | -11.864 |
| Danubit 2 | Gelatinous | Istrochem | Rock blast | 22.5 | 5.750 | -11.746 |
| Carbodanubit ^a | Gelatinous | Istrochem | Antidust | 23.0 | 5.632 | -14.656 |
| Danubit Geofex | Gelatinous | Istrochem | Special | 40.0 | 6.285 | -16.633 |
| Perunit 20 | Gelatinous | Synthesia | Rock blast | 20.0 | 5.500 | -14.154 |
| Perunit 22 | Gelatinous | Synthesia | Rock blast | 22.0 | 5.600 | -15.606 |
| Perunit 28 | Gelatinous | Synthesia | Rock blast | 28.0 | 6.100 | -22.894 |
| Perunit HG | Water gel | Synthesia | Rock blast | 45.0 ^b | 6.102 | -18.205 |
| Ostravit C ^c | Semi-gelatinous | Synthesia | Permissible | 11.0 | 2.000 | -15.789 |
| Semtinit 50 ^a | Gelatinous | Synthesia | Permissible | 25.0 | 2.100 | -17.661 |
| Obrysit ^d | Gelatinous | Synthesia | Pre-splitting | 40.0 | 2.038 | -16.718 |

^a Sodium chloride is used as a cooling agent.

^b PETN is applied as the nitric ester.

^c An oxidizing and, at the same time, cooling system is made by mixing of sodium nitrate and ammonium chloride.

^d The oxidizing system is sodium nitrate only and the charge density is 500 kg m^{-3} .

emulsion matrix with SN and sodium chloride (permissible explosive of the first class) was prepared. The final explosive mixtures were prepared by mixing the emulsion matrices with 3% by mass of glass microspheres (3M made, grade K1). Some samples were fortified by addition of 30% by mass of pentaerythritol tetranitrate (PETN) or 1,3,5-trinitro-1,3,5-triazacyclohexane. Explosive mixtures with RDX were further sensitized with 2% by mass of glass microspheres [48].

Three samples of classical W/O emulsion explosives from the research activity of Military University of Technology (MUT) were also measured. Their characteristics are as follows (their detailed composition is secret).

- Emulgit 42P (code designation 1E) contains 85% mass of ammonium and sodium nitrates mixture, 8.5% mass of water and 6.5% mass of fuel and emulsifier mixture. Its detonation rate was 4660 m s^{-1} and the $E_a R^{-1}$ value of Kissinger's slope was $-21.327 \times 10^3 \text{ K}$ ([51]).
- Emulgit LWC-ALAN1 (code designation 2E) contains 88.5% mass of ammonium and sodium nitrates mixture, 4% mass of aluminium, 4% mass of water and 3.5% mass of fuel and emulsifier mixture. A partially solidified mixture had a detonation rate of 3950 m s^{-1} and the $E_a R^{-1}$ value of Kissinger's slope was $-27.799 \times 10^3 \text{ K}$ ([51]).

- Emulgit LWC (code designation 4E) contains 89.3% mass of ammonium and sodium nitrates mixture, 4.7% mass of water and 6% mass of fuel and emulsifier mixture. Its detonation rate was 4200 m s^{-1} and the $E_a R^{-1}$ value of Kissinger's slope was $-16.175 \times 10^3 \text{ K}$ ([51]).

2.2. Non-isothermal differential thermal analysis

We have used a DTA 550 Ex apparatus [35,36] specially developed at the Department of Theory and Technology of Explosives for thermal analysis of explosives. The measurements were done at atmospheric pressure, the tested sample being in direct contact with the air atmosphere. The sample tested (0.05 g) was placed in a test tube made of Simax glass, 5 mm in diameter and 50 mm in length. The reference standard was 0.05 g aluminium oxide. We used linear rates of temperature increase, like 5, 10, and 15 K min^{-1} . The results of these measurements were treated by means of the software delivered with the DTA apparatus [35].

2.3. Strategy of DTA measurements

Except for the commercial explosives from Table 1 and the Emulgits from MUT, only the most reactive components or their mixtures of the other ex-

Table 2

Survey of constitutions of oxidizing systems measured, their code designation, characteristics of their thermal reactivities ($E_a R^{-1}$) and detonation velocities (D) of emulsion explosives, derived from these systems

| Component of the oxidizing system | Constitution of the oxidizing system (mass% of the final emulsion composition) | | | | | | | | | | | | | | |
|--|--|--------------------|--------------------|---------------------|--------------------|---------------------|---------------------|---------------------|---------------------|-----------------------------|-----------------------------|------------------------|---|-------------------------------|--------|
| | AN ^a | AN/CN ^a | AN/SN ^a | AN/LiN ^a | AN/PN ^a | AN/AlN ^a | AN/CoN ^a | AN/NiN ^a | AN/CuN ^a | AN/SN/ NaCl ^a | AN/CN/ PETN ^b | AN/CN/RDX ^b | AN/SN/CN/PETN ^b | AN/SN/ CN/RDX ^b | |
| NH ₄ NO ₃ | 84.0 | 65.6 | 65.7 | 68.5 | 53.8 | 67.1 | 67.3 | 65.7 | 62.3 | 57.3 | 43.3 | 43.3 | 45.4 | 45.4 | |
| Ca(NO ₃) ₂ | – | 15.0 | – | – | – | – | – | – | – | – | 15.0 | 15.0 | 9.6 | 9.6 | |
| NaNO ₃ | – | – | 15.0 | – | – | – | – | – | – | 15.0 | – | – | 5.1 | 5.1 | |
| LiNO ₃ | – | – | – | 12.0 | – | – | – | – | – | – | – | – | – | – | |
| KNO ₃ | – | – | – | – | 25.0 | – | – | – | – | – | – | – | – | – | |
| Al(NO ₃) ₃ | – | – | – | – | – | 15.0 | – | – | – | – | – | – | – | – | |
| Co(NO ₃) ₂ | – | – | – | – | – | – | 15.0 | – | – | – | – | – | – | – | |
| Ni(NO ₃) ₂ | – | – | – | – | – | – | – | 15.0 | – | – | – | – | – | – | |
| Cu(NO ₃) ₂ | – | – | – | – | – | – | – | – | 20.0 | – | – | – | – | – | |
| NaCl | – | – | – | – | – | – | – | – | – | 9.0 | – | – | – | – | |
| PETN | – | – | – | – | – | – | – | – | – | – | 30.0 | – | 30.0 | – | |
| RDX | – | – | – | – | – | – | – | – | – | – | – | 30.0 | – | 30.0 | |
| Kissinger's slope in ($E_a R^{-1}$) ($\times 10^{-3}$ K) | –64.07 | –45.76 | –39.59 | –35.53 | –27.71 | –27.61 | –19.56 | –18.83 | –17.79 | –8.11 | –37.58 | –32.45 | –20.20 ^a | –22.85 ^b | –25.55 |
| Rate of detonation in km s ⁻¹ | 5.100 | 4.800 | 4.740 | 4.730 | 4.320 | 4.201 | 4.560 | 4.410 | 4.677 | 4.463 | 5.573 | 5.660 | 5.161 ^a , 5.240 ^b | 5.962 | |

^a Data taken from [50].

^b Data taken from [49].

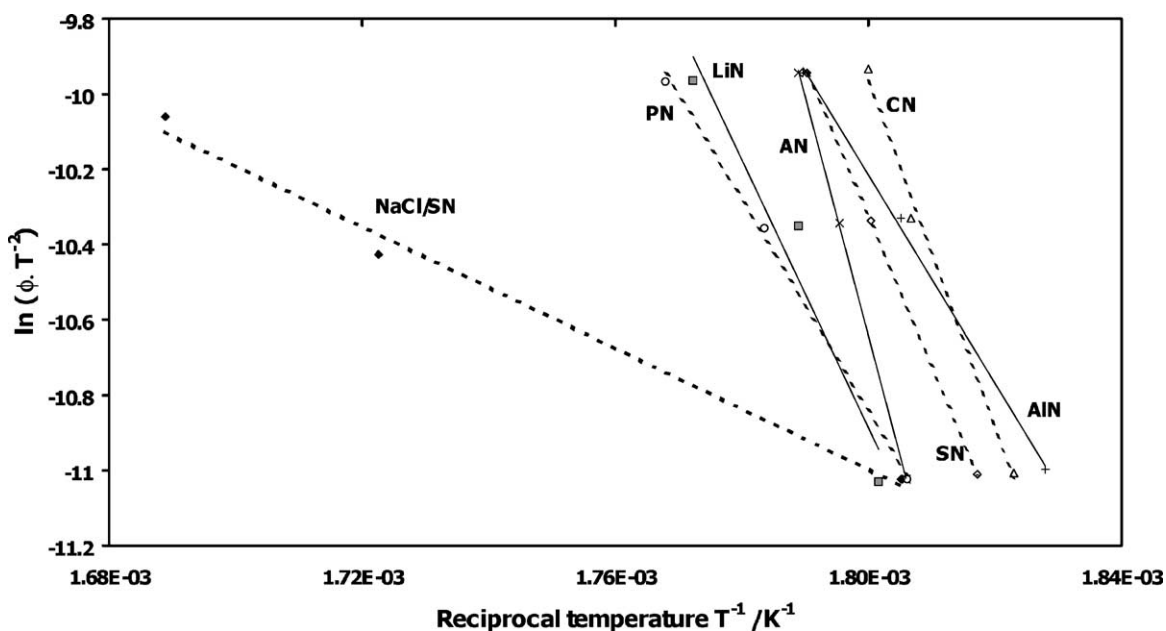


Fig. 3. Kissinger's DTA method applied to the thermal reactivity of oxidizing systems, which are based on the ammonium nitrate (AN) and its mixtures with sodium nitrate (SN), potassium nitrate (PN), calcium nitrate (CN), aluminium nitrate (AlN), lithium nitrate (LiN), and sodium chloride–sodium nitrate (NaCl/SN).

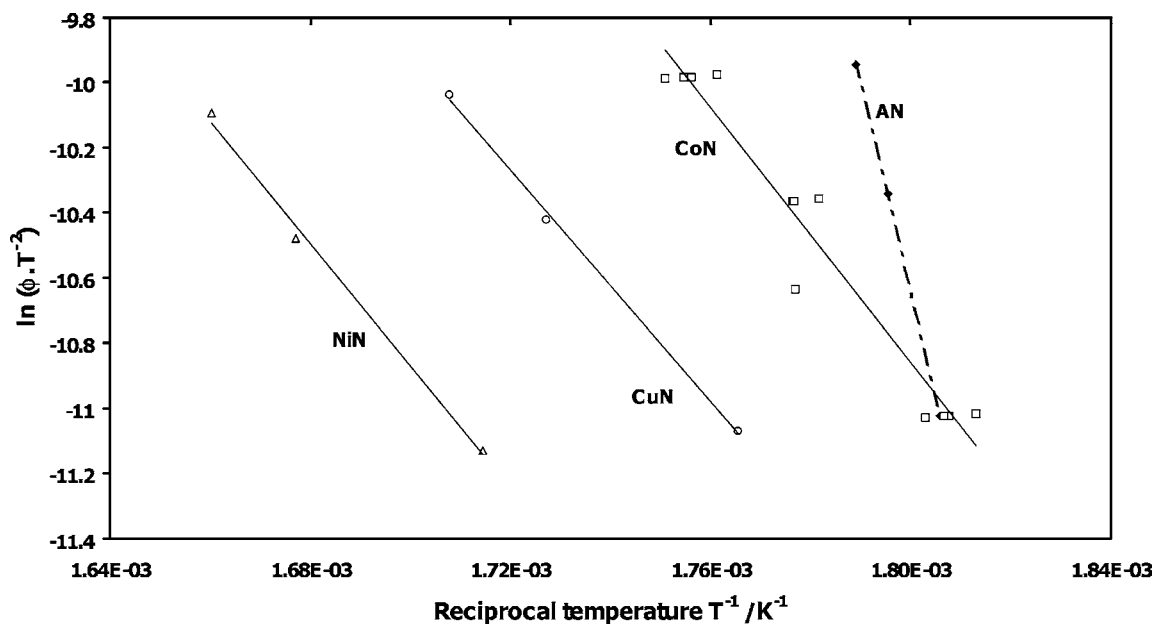


Fig. 4. Comparison according to Kissinger's DTA method of the thermal reactivities of oxidizing systems, based on ammonium nitrate (AN) and its mixtures with nickel(II) nitrate (NiN), copper(II) nitrate (CuN) and cobalt(II) nitrate (CoN).

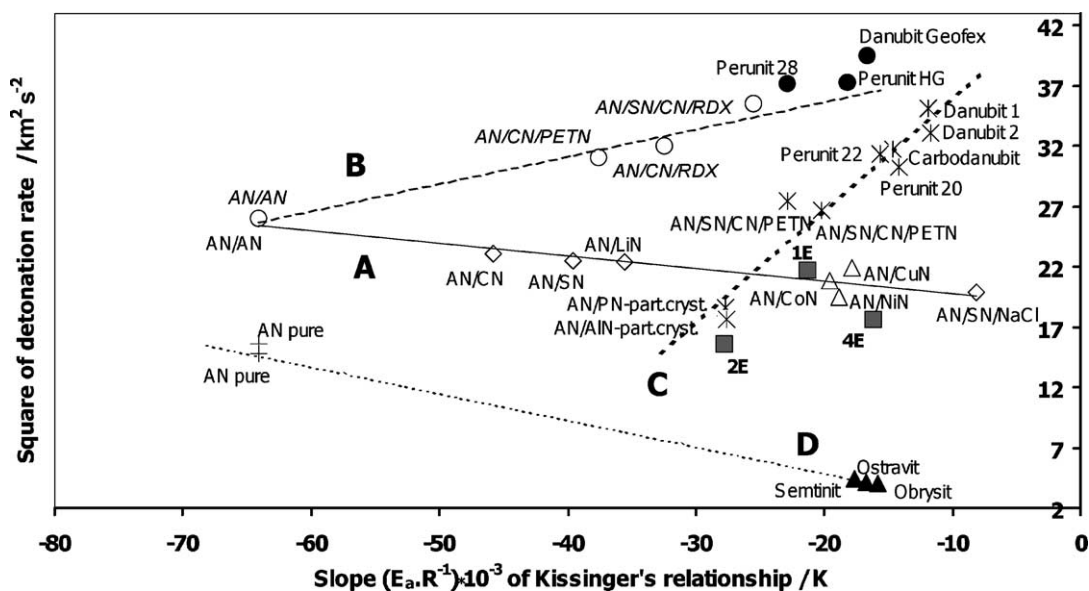


Fig. 5. Relationships between the squares of the experimental detonation rates of explosive mixtures and slopes of Kissinger's relationship, obtained from Figs. 3 and 4 (see Tables 1 and 2). Points marked as "AN pure" correspond to the maximum experimental ($D = 3.95 \text{ km s}^{-1}$ [46]) and calculated ($D = 3.85 \text{ km s}^{-1}$ [47]) values of the detonation rates of pure ammonium nitrate.

plosives were measured by means of DTA. Thus in the case of W/O emulsion explosives these were only mixtures of nitrates in such mass ratios as they are present in the final product (see Table 2 and an example of DTA record in Fig. 1). For the emulsion explosives fortified by means of a high explosive, we submitted to measurement the mixtures of this high explosive with nitrates, again in the same mass ratio as that in the final mixture (see Table 2). Commercial explosives were submitted to measurements in their total composition (an example of DTA record see in Fig. 2). The results obtained were treated using the Kissinger method [34] and some from them are presented in Figs. 3 and 4. The values of $E_a R^{-1}$ thus obtained (see Tables 1 and 2) were then plotted against the square of the experimentally determined detonation rates of the corresponding final explosive mixtures (Fig. 5).

3. Discussion

From among the components involved in emulsion explosives, ammonium nitrate is thermally the most reactive substance (see [37–41,43]). This means that the

detonation reactivity of these explosives should correspond with the thermal reactivity of their oxidizing systems. For this reason, the Kissinger DTA method [34] was used to specify the thermal reactivity of only the mixtures of ammonium nitrate with other nitrates, or as the case may be, with other additives modifying the detonation rate of the resulting explosive (RDX, PETN, sodium chloride).

It follows from Figs. 3–5 that all the additives added to ammonium nitrate increase the value of the respective slope $E_a R^{-1}$ of the Kissinger relationship of the resulting mixture. For emulsion explosives without the high explosive additive (i.e. mixtures of the blasting agent type), the earlier mentioned trend leads to a decrease in the detonation rate (see group A in Fig. 5). Slightly deviating from this trend are the mixtures with added nickel(II) nitrate, copper(II) nitrate, and cobalt(II) nitrate. These additives react with ammonium nitrate to form salts having a complex anion of general formula $\{M(\text{NO}_2)_4\}^{2-}$ [42], which must influence both the thermal and detonation reactivity of the resulting explosives. Most metal cations considerably influence the thermal reactivity of ammonium nitrate [43]. It is worth mentioning that the mixture "ammonium nitrate–cobalt(II) nitrate" caused some problems

as far as the reproducibility of DTA measurements were concerned (see Fig. 4). It must be warned that data for samples 1E and 4E of the emulsion explosives correlate well with group A (see Fig. 5), which was constituted on the basis of the thermal reactivity of only the oxidizing systems. This means that the reactivity of the oxidizing system is dominant in the initiation and development of detonation of blasting agents.

As far as the emulsion explosives fortified with addition of RDX or PETN in the amount of 30% mass are concerned (see group B in Fig. 5), the trend of the relationship between the $E_a R^{-1}$ values and the square of the detonation rates is opposite, again the case with these mixtures of blasting agents type (a hot spots mechanism works in the initiation and growth of detonation of the agents). The reason most probably lies in the shortening of the reaction zone in the detonation wave. Higher concentrations and reactivities of the primary fragments from RDX and PETN molecules thus can increase detonation characteristics in the C – J plane (the initial rate of energy release behind the detonation front is faster, as in the case of blasting agents). This means that the hot spots mechanism should not work in detonation of the fortified mixtures. Both RDX and PETN could boost the reactivity of the oxidizing system of these explosive mixtures. Analogous behavior was proved in the case of mixtures of 1,3,5,7-tetranitro-1,3,5,7-tetraazacyclooctane (HMX) with ammonium perchlorate (AP) [44]. From the positions of the data especially for Perunit 28 and Danubit Geofex in Fig. 5, it can be deduced that the previously mentioned higher reactivity eliminates differences in the influence of the physical state of the oxidizing systems in fortified emulsion mixtures (solution) on the one hand, and in commercial explosives (crystalline) on the other. This is, of course, valid for content of high explosive in the mixture much greater than of 28% mass (i.e. much greater than the “critical content”). However, an exception exists from previous: sodium nitrate (SN) decreases the D value also of the emulsion explosive which is fortified by PETN (see positions of data AN/CN/SN/PETN in Fig. 5). Only when the PETN content exceeds 30% by mass of explosive mixture (as it is in Perunit HG) can this SN influence be eliminated. Alkali metal salts, in general, have a strong effect upon the composition of the gaseous products of detonation [45], i.e. upon the

course of reactions in the reaction zone of the detonation wave.

The commercial explosives with nitric esters content up to 25% mass form a separate group, where with the data for partially solidified emulsions AN/PN, AN/AIN and 2E and the earlier mentioned mixture AN/CN/SN/PETN correlate (see group C in Fig. 5). It shows that decomposition of a crystalline oxidizing system (containing, with the exception of AN/AIN, in all cases also SN) should play a dominant role in the initiation and development of detonation. This group thus forms a transition one between the classical W/O emulsion explosives and mixtures with “previous critical content” of RDX or nitric esters.

Commercial explosives with a reduced volume content of energy, i.e. Semtinit 50, Ostravit C and Obrysit, form a separate group (i.e. group D in Fig. 5) too. It is unusual to construct a line taking only three points, but, in this case, a relationship results with coefficient of determination $r^2 = 0.9802$. Substitution of the $E_a R^{-1}$ value for AN in this relationship (i.e. for group D) leads to a value for the detonation rate $D = 3.81 \text{ km s}^{-1}$. The highest experimental value of this rate for ammonium nitrate is 3.95 km s^{-1} [46] (in a steel tube of diameter 303 mm and for a charge density of 830 kg m^{-3}). The extrapolated detonation rate of the nitrate to an infinite diameter was calculated as $D = 3.85 \text{ km s}^{-1}$ [47]. The previously mentioned facts could mean that nitrate anions play a dominant role in the initiation and development of detonation of this group of explosives.

4. Conclusion

A modification of the Evans–Polanyi–Semenov equation, in which the activation energy is substituted by the slope $E_a R^{-1}$ of Kissinger’s relationship (an output of the Kissinger DTA method [34]) and, at the same time, the heat of reaction by the square of the detonation rate (D^2) is applicable to the study of the mechanism of detonation initiation of explosive mixtures. By means of the equation the following results were found.

- (a) The initiation of the detonation of those explosives based only on mixtures of a fuel with an oxidant (i.e. the type of blasting agents) proceeds primarily through decomposition of the oxidizing system.

- (b) A critical amount of additives of nitric esters or nitramines type exists in the mixtures (i.e. about 30% mass). Explosive decomposition is not influenced by structure of the oxidizing system (solution or crystalline) of thus fortified mixtures only in the case when the content of the additives is earlier the critical value. This effect is an analogue of the well-known influence of the shock pressure on initiation and growth of the detonation in which the critical pressure plays a decisive role [52].
- (c) The thermal reactivity of the oxidizing system and/or its mixture with the high explosive replaces the primary thermal reactivity of explosophore groups in the individual energetic materials (on example: nitrogroup in the polynitrocompounds).
- (d) From the standpoint of technology and application, it is important that an addition of any inorganic nitrate to ammonium nitrate in a classical W/O emulsion explosive results in a decrease of the detonation parameters of the resulting mixture ([48]).

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