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Thermal Stability of Explosives

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Abstract: Thermal decomposition rates and mechanisms of explosives are of obvious importance to efforts designed to enhance safety in manufacturing, storage and handling of these energetic materials. To assess the thermal stability of energetic materials a series of analyses have to be performed. This paper gives an overview on the classical tests used to establish the safety parameters. The modern analytical methods applied today are also presented and illustrated. The development of technology and the use of advanced numerical techniques such as AKTS-TA-Software® enables prediction of the reaction progress of materials in broad temperature ranges. In fact, the goal for numerical simulations is to replace experiments in situations when direct accessibility to the test item or set-up is not possible for timing or safety reasons. The introduction of such new methodology will lead to approaches as close as possible to the reality but will never be able to reach it.

Keywords: Advanced numerical techniques · Explosives · Finite element analysis · Ignition temperature · Thermal decomposition · Thermoanalytical techniques

1. Introduction

What is thermal stability of explosives? Explosives, also called energetic materials, are compounds that may deliver a huge amount of energy in a very short time when they are submitted to specific conditions. The most popular of these conditions is thermal load. Every energetic material submitted to high temperature will react more or less strongly, according to a thermal decomposition process. This thermal decomposition and its mechanism are very important to characterize the behavior of the energetic material when submitted to thermal loads. The knowledge of the behavior of the respective energetic material will enhance safety in manufacturing, storage, and handling of these energetic materials.

The objective of studying the thermal stability of explosives (energetic materials) is to determine the details of explosives thermal chemistry at a molecular level, so that the macroscopic behavior (combustion, explosion, and detonation) can be better understood.

To assess the thermal stability of energetic material, the use of different thermal analyses is necessary. The thermal analysis techniques used for explosives are the same well-established methods used for the characterization of a wide range of materials and have seen a rapid development over the last decade. These techniques may be described as the measurement of physical properties of energetic material as a function of temperature or time, using a controlled temperature program. This definition of thermal analysis was recently subjected to discussion and a new definition of thermal analysis was given [1][2]: *Thermal analysis means the analysis of a change in a property of a sample, which is related to an imposed temperature alteration.*

Hemminger and Sarge [1][2] give the following explanations on the last definition:

- 'Analysis' means much more than 'monitoring'.
- In most experiments it is a *change* in a property, rather than the property itself which is monitored.
- It is the temperature of the sample's environment (*e.g.* a furnace), rather than the actual sample temperature, which is programmed.

When studying energetic material it is important to distinguish two reaction processes: the reaction *without ignition* and the reaction *with ignition, i.e.* when the energetic material undergoes through combustion.

The first reaction mechanism, *without ignition,* occurs at low and room temperature. Such a thermal decomposition will occur during the chemical ageing of energetic materials. This type of reaction will not be treated in this paper.

This paper will focus the interest on the second mechanism, the thermal decomposition with ignition of the energetic material and will describe the most commonly used methods.

2. Thermal Analysis Techniques

Testing energetic material happens very often in the solid state, at least at the start of the investigation. The concept of a *perfect solid* is useful as the reference point for *real solids*. A perfect solid has a ***completely ordered*** arrangement of its constituents, while real solids have ***imperfections*** of many kinds. The degree of arrangement in real solids is marginally greater than for liquids, but considerably less than in a perfect crystal; the substance is sometimes referred to as a 'non-crystalline solid'. The reactions of *real solids* can be defined by the following changes:

- *Phase transition:* a new arrangement of constituents may become more stable than the original.
- *Melting:* when sufficient energy becomes available, the forces of attraction between constituents become insufficient to maintain the ordered arrangement of the solid and the system relaxes to the more disordered arrangement of constituents in a liquid.
- *Sublimation:* when the kinetic energy of the constituents increases very rapidly, direct transition to the disordered arrangement of a gas may occur, with-

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Table 1. Thermal events on heating a single solid, A, in an inert atmosphere (s = solid, l = liquid, g = gaseous)

			ΔH	sign	ΔMass
A (s, structure 1)	→ A (s, structure 2)	Phase transition	yes	+ or -	no
A (s)	→ A (l)	Melting	yes	+	no
A (glass)	→ A (rubber)	Glass transition	no	no	no
A (s)	→ A (g)	Sublimation	yes	+	yes
A (s)	→ B (s) + gas(es)	Thermal decomposition	yes	+ or -	yes

Table 2. Measured ignition temperature of well investigated explosives

Explosive	Measured ignition temperature [°C]
Illumination composition ($Mg/Sr(NO_3)_2$)	260
Smoke composition (NH_4ClO_4/ZnO)	300
Lead azide	315–360
Hexogen	230
Octogen	287
Nitroglycerine	223–225
Nitrocellulose	154–160

out the intermediate formation of a liquid phase.

- *Thermal decomposition:* when the bonding forces within constituent molecules or ions are weaker than those between the atoms constituting these units, increasing the temperature may result in bond redistribution and the formation of products chemically different from the reactant.

The thermal processes that can occur are represented symbolically in Table 1.

In this paper we will concentrate only on the last event, thermal decomposition, where energetic material decomposes into gases and oxidized residue. Of course, the presence of a melting point near the decomposition temperature would have an great influence on the thermal behavior of the energetic material.

2.1. *Small Scale Testing*

Due to the huge energy delivered from energetic material, it is crucial to perform thermal analysis on a very small scale. In the small scale testing of explosives, the evolution of the technology also required evolution of the apparatus and the techniques used to measure the thermal stability. This paper will first illustrate old classical techniques, but then will concentrate on the modern and more accurate techniques.

2.1.1. *Classical Tests*

A search on the Internet about thermal stability tests of explosives leads to many different methods. All are based on ignition temperature or ignition time. The most famous and classical methods are mentioned briefly.

Fisher-Johns Auto-Ignition Test

The Fisher-Johns auto-ignition test determines the thermal/time conditions under which an explosive of a specified sample will ignite. Auto-ignition is investigated in two ways: (a) Heat the sample at a constant rate (normally 15 °C/min.). (b) Time the sample maintained at a constant, elevated temperature until ignition occurs (normally the test material is held at 120 °C for up to 1h).

During heating, any physical changes of the sample such as melting, fuming, or smoking, and so on, is recorded noting the temperature at which these changes occur. During a normal auto-ignition study, three tests are conducted using method (a) and an average auto-ignition temperature is reported. For a screening study, one test is sufficient.

Henkin Bath Test

The Henkin bath test is used to determine the thermal stability of explosives by isothermally testing samples in a heated metal bath. A sample of explosive is weighed (50 mg) and placed into the cap shell. The cap is then sealed (aluminum shell). Testing is done on the sealed test samples at various temperatures. The test measures the time to explosion. A graph is made from final data showing time *vs*. temperature to determine the thermal stability of the explosive sample being tested (cook off time and reaction rates). This differential thermal analysis combined with other tests (impact) may be used to determine proper explosive handling techniques.

Determination of Ignition Temperature

This method to determine the temperature of ignition has been performed for many years. The ignition temperature of an explosive can be defined as the temperature by which a small sample of explosive, placed in an externally heated glass-tube, ignites to a flame, reacts to a deflagration or a violent explosion. Normally the test consists of placing 0.5 g of explosives in a glass-tube and to introduce 2 cm of the bottom of the tube into an oven heated at 100 °C. Afterwards the temperature is raised until the thermal event appears with a heating rate of 20 °C/min. For nitrocellulose-based explosives, the test is performed in the same manner, but the heating rate is set to 5 °C/min. Table 2 gives an overview of the results obtained by applying this old method to well investigated explosives.

2.1.2. *Differential Scanning Calorimetry*

Differential scanning calorimetry (DSC) is a modern differential thermal analysis in which the sample compartment of the apparatus is a differential calorimeter, allowing an exact measure of the heat of transition independent of the specific heat, thermal conductivity and other variables of the sample. DSC is the technique *most widely applied* to study any physical or chemical change that is accompanied by a change in energy. The DSC measurement takes place in an insulated furnace with two separate compartments: a sample compartment and a reference compartment, which are schematically illustrated in Fig. 1. The instrument acts somewhat like a thermostat in that two compartments are separately maintained at a given temperature to within ±0.01 °C of each other. The temperature setting is then swept through a range and the instrument records the difference in heat flow to the two compartments as a function of temperature. The furnace is typically purged with dry nitrogen, helium or argon, although under some circumstances it is useful to flow air or pure oxygen.

On the laboratory DSC instruments (assuming one is scanning up), a positive deflection on the plot corresponds to an exothermic process and a negative deflection corresponds to an endothermic one. The use of DSC for the characterization of the thermal stability of energetic materials has a big advantage and brings a lot of information:

- *Glassy solids,* including many polymer samples, do not exhibit the same high degree of ordering that is found in crystalline solids. Therefore, they do not exhibit the same type of sharp melting point as crystals: the transition from disordered solid to liquid occurs more gradually over a broad temperature range. This is called the *glass transition.*
- *Crystallization* is a first-order transition that appears as a relatively sharp peak in the DSC. For ordinary solids, crystal-

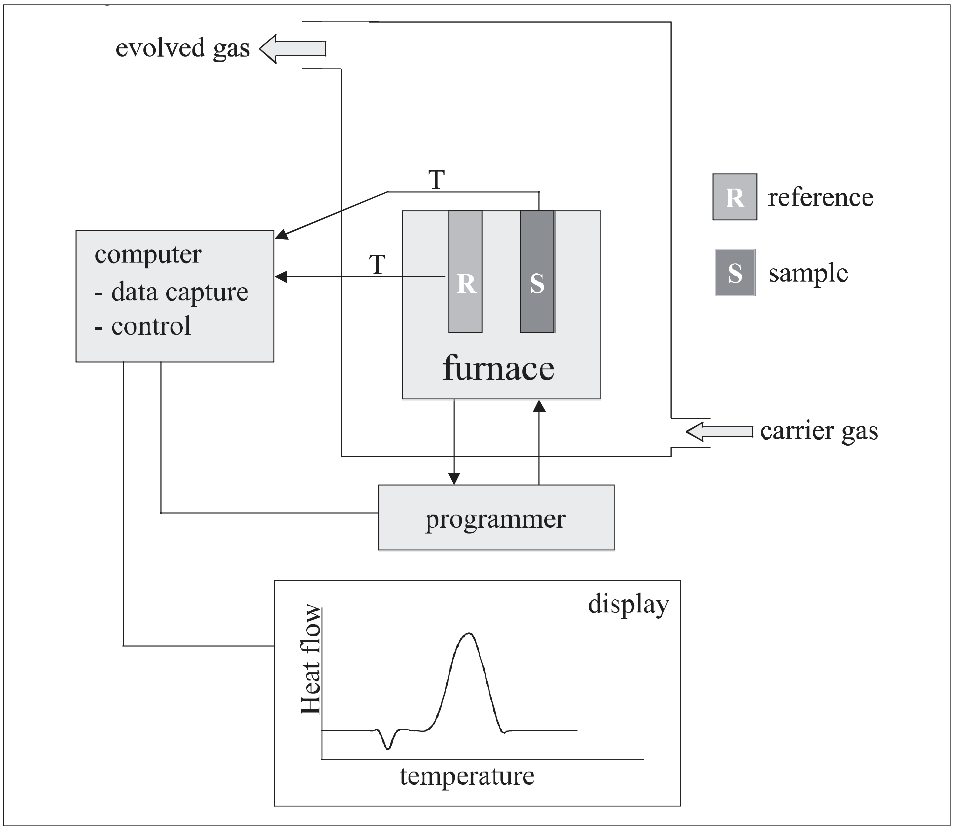

Fig. 1. Schematic description of a calorimeter used for differential scanning calorimetry (DSC)

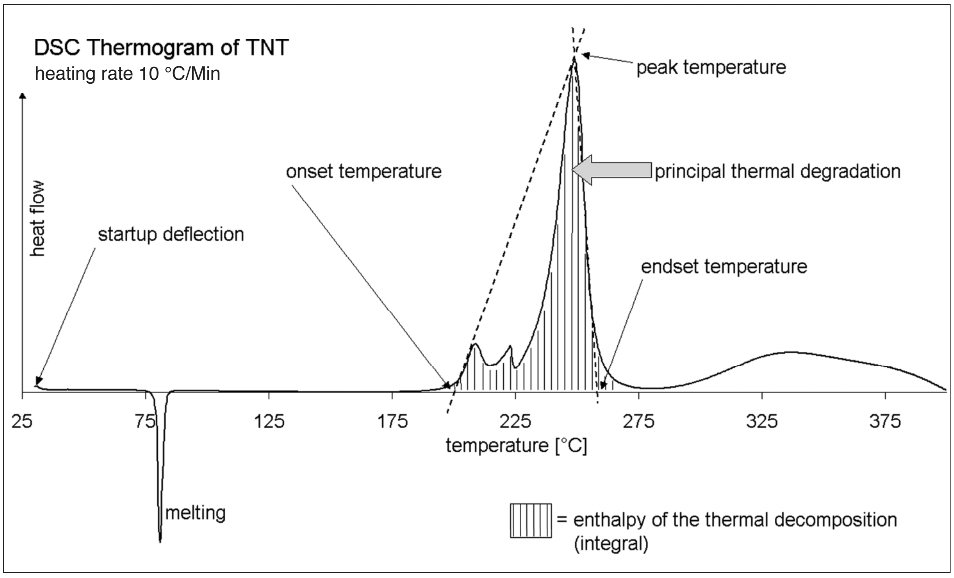

Fig. 2. Typical DSC curve for an organic high explosive. This thermogram of 2,4,6-trinitrotoluene (TNT) delivers important thermal information such as melting point, onset- and endset-temperature, enthalpy of the multistep thermal decomposition.

lization generally appears only when sweeping from a high temperature to a low temperature; however, crystallization after melting through a glass transition can be observed in certain types of polymers.

- *Melting* of a crystalline solid is an endothermic first-order transition that appears as a negative peak on the DSC scan.
- *Characterization of the high exothermic thermal decomposition*. The important features of thermal decomposition include the decomposition temperature, or peak position, the width of the thermal reaction, or the onset and endset temperatures of the peak and the enthalpy of the thermal decomposition, or area under the peak. These quantities are shown on the thermogram of TNT in Fig. 2. The intersection between the baseline and the tangent line at the point of inflection on the edge of a peak defines where the peak starts. The first peak edge is called the peak 'onset' temperature, and the trailing edge is called the 'endset' temperature. The area under the peak will give the enthalpy change ΔH. An accurate value of ΔH requires that the baseline as well as the starting and ending points for the integration be properly defined.

Today DSC is the most adequate method to perform the thermal characterization of an explosive. This technology allows a lot of information to be obtained in a short time concerning the thermal stability of an explosive and not only the ignition temperature.

2.1.3. *Thermogravimetric Analysis*

In thermogravimetry (TG) a sample is heated at a linear rate while it is being weighed. Such an apparatus is described in Fig. 3. Since it is difficult to embed a thermocouple in a sample while it is being weighed, there must be a compromise between weighing accuracy and sensitivity and accuracy of temperature measurement.

With explosives and propellants, rapid decomposition reactions produce a thrust on the sample pan, causing erroneous mass measurements: the mass appears to increase as reactant decomposes. Catastrophic decompositions or detonations tend to destroy expensive equipment.

TG is very useful to elucidate the mechanism of the thermal decomposition of an explosive. It would allow the observation of multistep decomposition as for example the degradation of ammonium dinitramide ($NH_4N(NO_2)_2$) shown in Fig. 4. It can be observed that the thermal decomposition of ammonium dinitramide is a two-step mechanism, where during the first step about 30% of the mass is lost and about 70% of the mass is lost during the second step [3]. Without thermogravimetric analysis, it would not have been so easy to identify such a multistep mechanism.

Using the DSC method alone does not allow the certain assessment of whether the transition observed is a crystal change or melting or a reaction, therefore coupling differential scanning calorimetry with thermogravimetry would identify if the thermal event is coupled with a loss of mass. These two modern thermal analyses are the most suited methods to assess the thermal stability of energetic material.

2.2. *High-scale Testing*

Self-heating accidents have often occurred when small-scale procedures have been scaled up without considering the effects of size. Safe amounts can be estimated in very straightforward ways. A large number of tests have been used to observe relative thermal stabilities at small scale. But is it allowed to apply directly the results

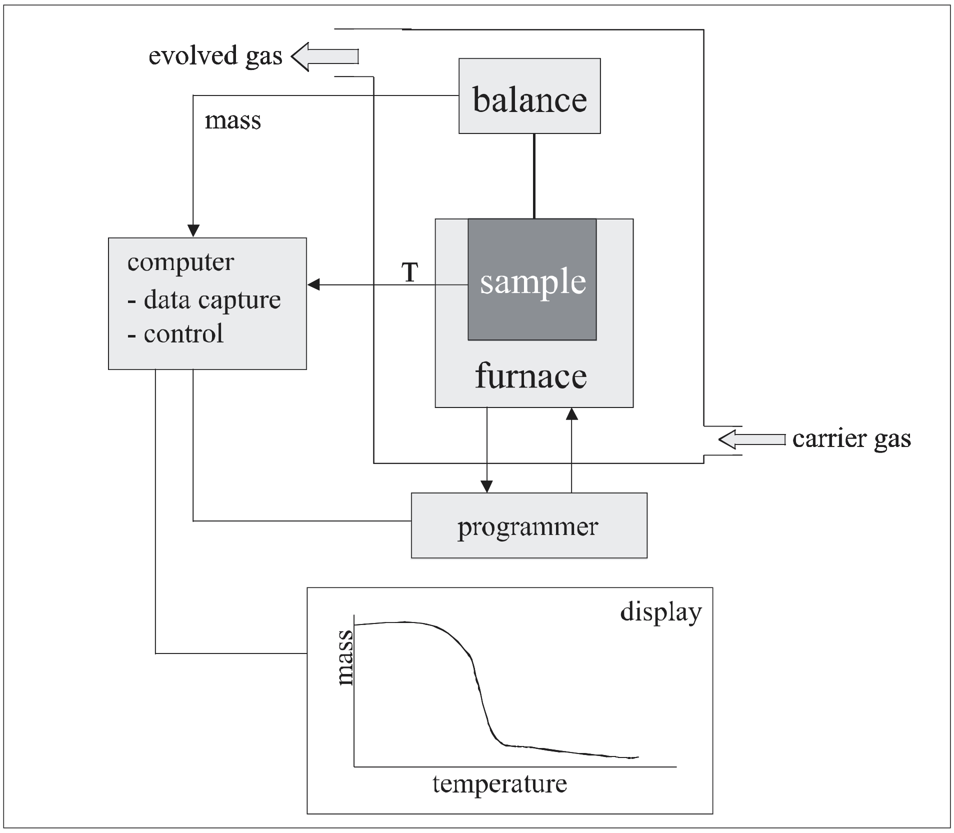

Fig. 3. Schematic description of a thermobalance used for thermogravimetry (TG)

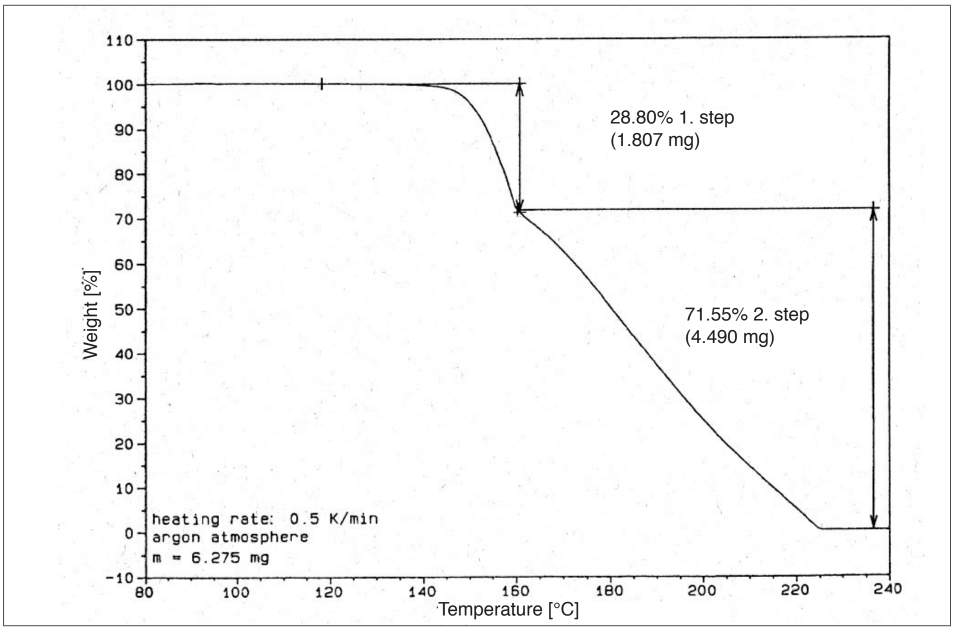

Fig. 4. Thermogravimetric analysis (TGA) of ammonium dinitramide (heating rate: 0.5 °C/min)

obtained for small scale testing or is a large scale test obligatory? In order to answer this question slow cook-off experiments have been developed.

2.2.1. *Slow Cook-Off Test*

This test determines the reactivity of an explosive sample to a gradually increasing thermal environment. In addition, the temperature at which a reaction occurs may be measured. The test sample is subjected to a gradually increasing temperature at a rate of 3.3 °C *per hour* until a reaction occurs. The test may begin with the test item preconditioned at 40 °C for 6 h. Measurements of elapsed time and temperature are recorded. Fig. 5 represents a typical temperature profile of a slow cook-off test. The violence of the thermal event has also to be documented with pictures. This test can be performed with all types of explosives. For example, 3 mg of nitrocellulose propellant will ignite at 190 °C, measured by DSC. The slow cook-off test, performed with about 350 g of the same propellant confined in a steel tube already ignites at 130 °C [4]. Such results indicate that the behavior of explosives depends strongly on the scale and confinement. Therefore results obtained by small scale analysis (*e.g.* DSC) have to be carefully interpreted for the use at a larger scale. The use of thermal stability results for safety analysis must be compared with large scale testing. Today, with the development of computer technology, an interesting solution for correct safety analysis originating from small-scale testing is the use of new thermal analysis software. The next chapter will illustrate how the use of a thermal analysis software like the modern AKTS-TA-Software® [5] may be useful for the safety analysis of a rocket motor propellant, compared to the results obtained by small-scale thermal analysis.

3. Safety Analysis

Implicit or explicit thermokinetic model parameters can be extracted in principle from experimental data gathered by any of the following dynamic thermo-analytical small-scale methods:

- DSC
  (Differential Scanning Calorimetry)
- TGA (Thermogravimetry Analysis)

The use of advanced kinetic analysis [5] enables prediction of the reaction progress of materials in wide temperature ranges that may be difficult to explore for time, sensitivity or safety reasons.

The analysis process requires determination of the kinetic characteristics of the reaction. The application of these techniques is widely recognized for the characterization of the degradation of solids [6].

The correct kinetic analysis of a decomposition reaction has at least three major stages: (1) collection of experimental data; (2) computation of kinetic parameters, and (3) prediction of the reaction progress for required temperature profiles applying determined kinetic parameters. Kinetic evaluation of the reactions should be carried out with thermoanalytical data obtained at several heating rates (non-isothermal) or temperatures (isothermal) to ensure reliable results. Kinetic methods that use single heatingrate experimental results should be avoided because they tend to produce highly ambiguous kinetic descriptions [7][8].

Applying the results obtained by thermoanalytical techniques (DSC, TGA), the kinetic analysis presented in this section enables accurate prediction of the reaction progress of materials in a wide temperature range under various temperature modes such as:

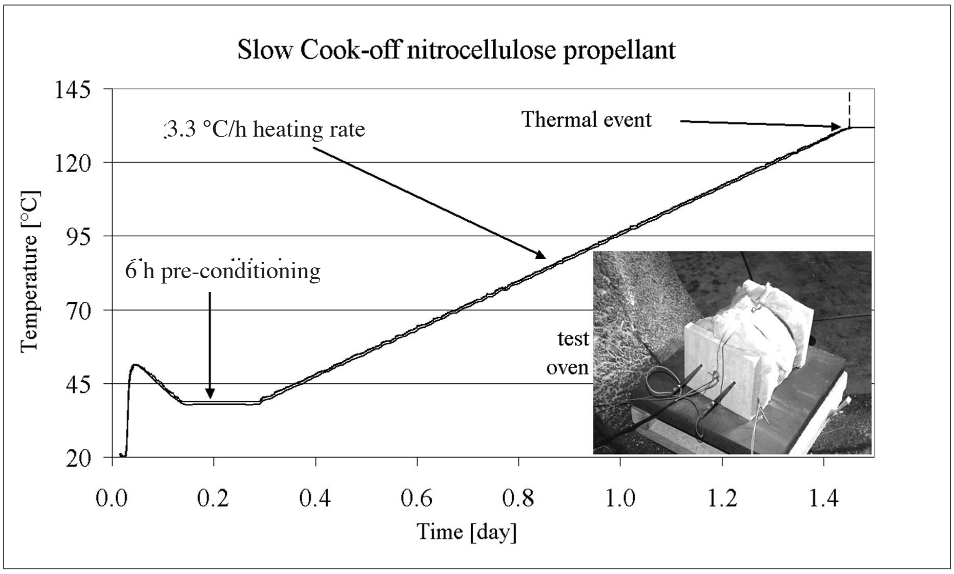

Fig. 5. Typical temperature profile for a slow cook off test of nitrocellulose propellant

- isothermal, non-isothermal and stepwise
- modulated temperature or periodic temperature variations and rapid temperature increase (temperature shock)
- real atmospheric temperature profiles for investigating properties of low-temperature decomposed solids under different climates (yearly temperature profiles with daily minimal and maximal fluctuations)
- adiabatic (safety analysis)

3.1. *Use of Thermal Analysis Software and DSC Measurements for Determination of Thermal Stability of Rocket Propellants*

The precise prediction of reaction progresses in adiabatic conditions is necessary for the safety analysis of many technological processes. Calculations of an adiabatic temperature–time curve for the reaction progress can also be used to determine the decrease of the thermal stability of materials during storage at temperatures near the threshold temperature for triggering the reaction. Due to insufficient thermal convection and limited thermal conductivity, a progressive temperature increase in the sample can easily take place, resulting in an explosion.

Several methods have been presented for predicting the reaction progress of exothermic reactions under heat accumulation conditions [9–11]. However, because decomposition reactions usually have a multi-step nature, the accurate determination of the kinetic characteristics strongly influences the ability to correctly describe the progress of the reaction. The use of simplified kinetic models for the assessment of runaway reactions leads to economic drawbacks, since they result in exaggerated safety margins. For adiabatic self-heating reactions, incorrect kinetic description of the process is usually the main source of prediction errors.

The decomposition of the boost propellant in a solid rocket motor is a highly exothermal process. Using the reaction heat (ΔH_r) and the heat capacity (c_p), one can calculate the reaction progress due to self-heating for different values of ΔT_{ad} (with $\Delta T_{ad} = \Delta H_r/c_p$). In Fig. 6A, the simulated T-time relationships with a starting temperature of 92 °C are presented for $\Delta T_{ad} = 2651 \pm 233$ °C (boost propellant, isochoric conditions). Fig. 6B presents the starting temperature and corresponding adiabatic induction time TMR_{ad} relationship. The confidence interval was determined for 95% probability.

The adiabatic induction time is defined as the time which is needed for self-heating from the start temperature to the time of maximum rate (TMR_{ad}) under adiabatic conditions. Depending on the decomposition kinetics and ΔT_{ad}, the choice of the starting temperature strongly influences the adiabatic induction time and, therefore, the boundary conditions valid for achieving necessary safety (*e.g.* storage or transport of self-reactive substances). It can be observed that the reaction is more exothermal (higher ΔT_{ad}) and less stable (lower starting temperatures for the same TMR_{ad}) for the boost-phase compound than for the sustain-phase propellant. The same observation is also valid for the isochoric conditions (closed crucibles) compared to the isobaric conditions (open crucibles).

The second field of application for numerical simulation techniques in process safety is the solution of partial differential equations as they are encountered in heat conduction problems. These problems arise when heat accumulation situations are to be analyzed. One very important criterion for thermal safety is for example the 'critical radius', defined as the largest radius above which a specific material of a specific size, shape and environment will self-heat catastrophically. Depending on the radius self-reactive chemicals can either: (i) decompose quietly, (ii) ignite and burn, or (iii) self-heat to explosion or detonation [12][13].

More generally, applications of Finite Element Analysis (FEA) and accurate kinetic description enable the determination of the effect of scale, geometry, heat transfer (isolation), thermal conductivity and ambient temperature on the heat accumulation conditions. In fact, the assumption that it is safe to handle an energetic material at any temperature below the first appearance of an exothermic signal on the DSC curve can be false and even dangerous. The highest safe temperature for handling any energetic material depends on several factors such as its size, shape, and prior thermal history. Safe storage or transport conditions with tailored safety margins can be defined using numerical simulation. Since the rocket has a small radius, we can examine the effect of isolation material for preventing explosion in case of a sudden heat source (*e.g.* fire) for a short period of time.

As an example one may consider the rocket motor with cylindrical geometries. It is composed by four serial layers. The boost component is at the center bordered by the sustain propellant. It is assumed that the propellants form a cylinder of two cylindrical layers of 4 cm each. The thickness of the wall containing the two propellants is 3 mm. The rocket is then embedded in an isolation material of 5 cm thickness. With a rocket radius of 8.3 cm, an initial temperature of 20 °C in each layer and an environment temperature following a stepwise temperature profile, the temperature evolution inside the self-reactive propellants can be simulated.

If the isolation is well designed the temperature may increase rapidly and then decrease as presented in Fig. 7A. The emergency procedure can thus operate safely. However, if the rocket is not sufficiently isolated the process suddenly deteriorates catastrophically. Fig. 7B presents the simulated explosion after about 332 min for the rocket with weak isolation. The temperature evolution and reaction progress during the early (induction), intermediate (acceleratory), and late (decay) periods of the self-heated reaction are now considerably different.

Consumption of the boost and sustain propellants with respect to temperature and reactant concentration leads to a combustion front which initial and final stage during the explosion is illustrated in Fig. 8. Using FEA, it is possible to follow very precisely the temperature distribution and propagation of the reaction front during the explosion. The latter occurs extremely fast and reaction progress increases accordingly at every point in time and space. It can be observed that the temperature distribution

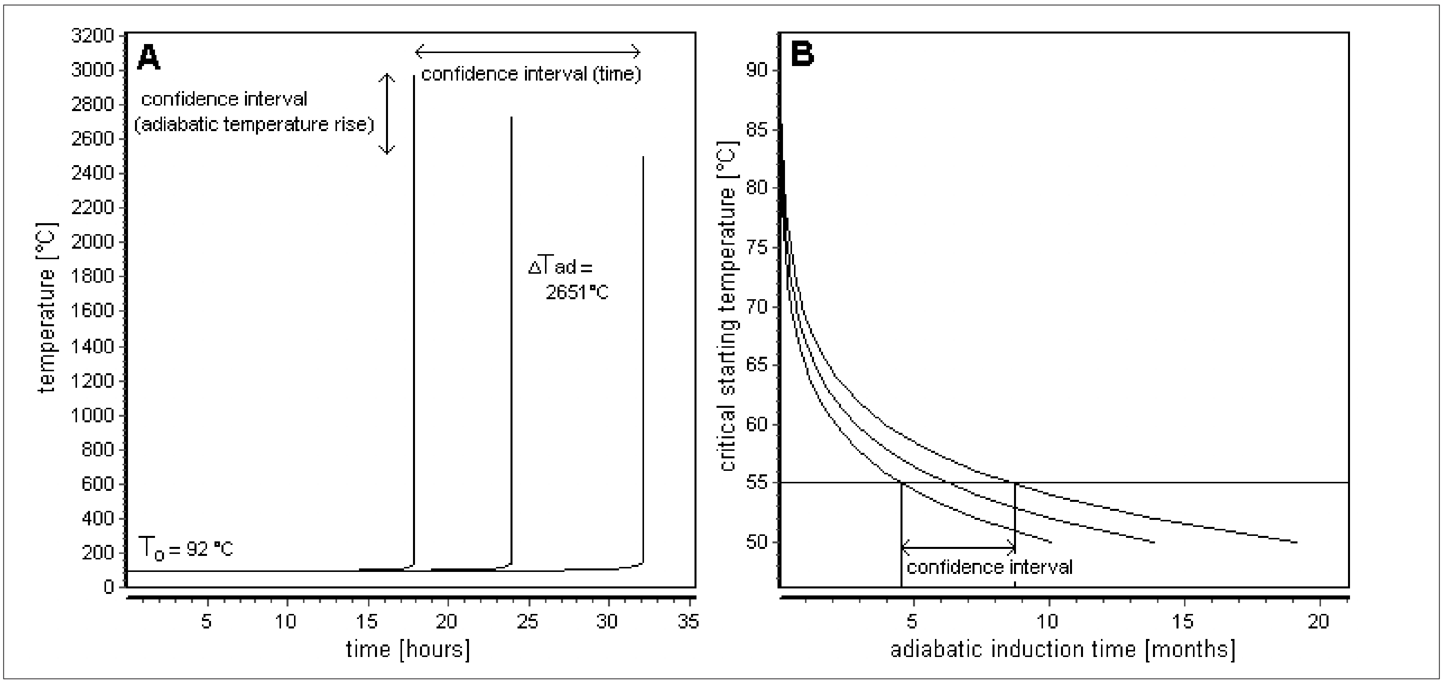

Fig. 6. (A) Adiabatic runaway curves for the boost propellant (isochoric conditions) showing the confidence interval for the prediction (T_{begin} = 92 °C and $\Delta T_{ad} = \Delta H_r/c_p$ = 2651±233 °C). The confidence interval was determined for 95% probability. (B) Starting temperature and corresponding adiabatic induction time TMR_{ad} relationship of the boost propellant under isochoric conditions. The choice of the starting temperatures strongly influences the adiabatic induction time (confidence interval: 95% probability).

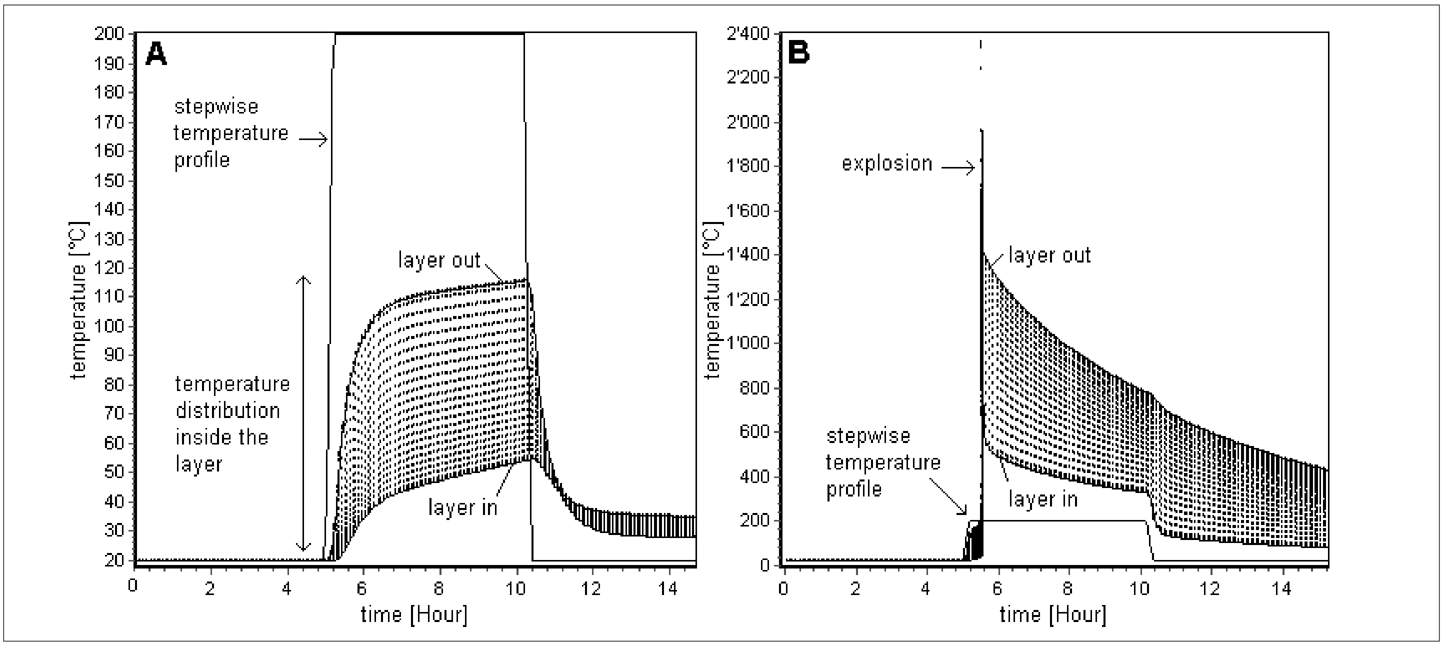

Fig. 7. The temperature evolution of the ignition by the stepwise temperature profile. Finite Element Analysis enables the computation of the isolation effect on heat accumulation conditions. Example (sustain phase layer in contact with the heated wall, isochoric conditions): (A) If the isolation is well designed the temperature increases rapidly inside the sustain phase layer and then decreases. However, if the rocket is not sufficiently isolated the process suddenly deteriorates catastrophically (B).

of the combustion front depends strongly on the decomposition kinetics and of the sustain- and boost phases. The structure of the combustion front associated with the multi-step decomposition reactions of the boost and sustain phases is, naturally, more complicated than in the case of a single step-reaction and is determined by the interaction of the various stages of the process. Knowledge of the decomposition kinetics combined with FEA can be used to determine the propagation velocity of the combustion front relative to the combustion mass flux. The examination of the isolation design is therefore very important for energetic materials. The analysis can be extended to any other critical design parameter that could lead to explosion. Note that under strictly adiabatic conditions, a thermal runaway is observed as presented in Fig. 6. Thus, FEA enables more precise description of processes that would have been declared unsafe by simplified methods because they result in exaggerated safety margins. More generally, applications of FEA and accurate kinetic description of complex reactions enable the calculation of accurate heat balances and the determination of safe storage or transportation conditions (*i.e.* determination of the best storage container size or transport temperatures). The method can also be used for shelf life prediction during transport.

4. Conclusion

The establishment of the thermal stability of an explosive is a complex question. Some years ago, this problem would have been solved by using more than one classical test and by completing the study with the existing know-how. This type of answer would have cost a lot of time and manpower.

Today, with at least three DSC experiments done under isothermal or non-isothermal conditions it is possible to compute the kinetics of decomposition of the products of interest. For these calculations the results obtained by thermoanalytical techniques recording *e.g.* change of the mass, heat generation, the rate of evolution of the gaseous products as well as the change of the pressure under isochoric conditions can be used. Application of Finite Element Analysis (FEA) enables the determination of the effect of properties of the sample and of the system on the reaction progress and on the heat accumulation conditions. This analysis can then be applied to the determination of the critical design parameters such as critical radius of a cylinder or sphere, the thickness of the isolation, influence of the surrounding temperature for safe storage or transport conditions.

The last new method proposed in this paper is still in development and would need more investigation to be sure that the extrapolation in time and quantity are allowed. In order to validate such a new technological product, the use of classical slow cook-off would be helpful. In the near future, the comparison between safety analysis obtained by treating the DSC experiments with kinetic software and real slow cook-off experiments, will allow thermal analysis to come as close as possible to reality.

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Fig. 8. The temperature distribution in the combustion front for the sustain and boost propellants (isochoric conditions). The spatial and temporal variation of the combustion front is created by ignition of the sustain propellant at the hot wall. (A) Initial stage of the explosion – Unburnt propellants. (B) Final stage.