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Review

Review of Methods for Testing the Compatibility of High Energy Mixed Components

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Abstract: A review of methods for testing the compatibility of high energy mixed components is presented. The advantages, deficiencies as well as the limitations of particular research methods are described based on selected applications reported in the literature. The most frequently used techniques for testing compatibility are thermal methods, such as DSC, TG, VST, and HFC, in which the processes of decomposition of samples conditioned at elevated temperatures are analyzed. Examples of non-thermal methods for testing compatibility, such as DFT, FTIR or XRD are reported in the literature as well. Incompatibility may lead to thermal detonation, which can occur even at low degrees of conversion. For this reason, the authors focused specifically on the limitations of methods for determining compatibility at high degrees of conversion. The methods allowing

testing of compatibility based on an analysis for the initial decomposition stage are recommended.

Keywords: STANAG 4147, VST, HFC, TG, DSC, FTIR, XRD, compatibility, high energy materials

1 Introduction

Explosives are high energy mixed components utilized in civil, space and military applications. Although a number of contemporary high energy mixtures were developed as early as in the second half of the 20th century, they continue to be used despite having some disadvantages. These include the high sensitivity to external stimuli of composition B [1, 2], and the presence of hydrogen chloride in the combustion products of heterogeneous rocket fuels containing ammonium perchlorate (NH₄ClO₄, AP) [3]. Ongoing research is being conducted on new, explosive containing mixtures that can be used as blasting explosives [4-19], powder [20], rocket fuels [3, 21-25] and pyrotechnic mixtures [26-28]. The introduction of new explosive mixtures contributes to improved properties of these products, such as safety and operational performance. Testing the compatibility of a mixture of components constitutes an extremely important step in developing new formulations containing high energy materials [29]. The incompatibility of the components may lead to the uncontrolled exothermic decomposition of the mixture and consequently to a thermal detonation [30]. New high energy mixtures should be characterized for stability whilst being resistant to decomposition, even during long-term storage. The determination of the compatibility of components under real conditions requires many years of study. As such testing would be time-consuming, some methods have been developed to assess the compatibility in a significantly shorter time.

A considerable number of methods for testing compatibility is based on the analysis of the decomposition of samples stored at elevated temperature. The thermal effects resulting from a decomposition process are analysed predominantly with:

- differential scanning calorimetry (DSC) [5, 6, 10, 11, 23, 31-35],
 - heat flow calorimetry (HFC) [36-38],
 - adiabatic reaction calorimetry (ARC) [15, 39].
- The compatibility of components may also be determined by means of:
- mass loss analysis (thermogravimetry (TG)) [6, 11, 14, 15, 17, 40],

- analysis of changes in the composition,
- volume of released gases in the vacuum stability testing (VST) method [10, 11, 14, 16, 41-43], as well as in
- chemical reactivity test [44, 45].

Examples of these methods can be found in the STANAG 4147 standard [46]. Non-thermal methods for testing compatibility can also be encountered in the literature. Such methods are primarily used to test the compatibility of drug components, polymers [47-49], however these are also applicable in testing the compatibility of explosives [8, 10, 25, 50-53]. The most frequently used non-thermal methods for testing compatibility include:

- Fourier transform infrared spectroscopy (FTIR) [8, 50, 53], and
- X-ray diffraction (XRD) [10, 50, 52].

The purpose of the present paper is to present the most popular methods for testing the compatibility of high energy mixed components, which are accompanied by a short description, recommended applications, as well as the presentation of the principal advantages and disadvantages.

2 Compatibility Testing Methods

2.1 Vacuum Stability Test (VST)

The vacuum stability method, used for testing compatibility, involves placing the individual samples and their mixtures in measuring pans, followed by conditioning under reduced pressure and increased temperature conditions. The conditioning temperature is chosen to adequately match the thermal performance of the compound. The conditioning time depends on the conditioning temperature. According to STANAG 4147 standard [46], a reduction of the conditioning temperature by 10 °C increases the measurement time by 2.5 times:

- 40 h at 100 °C,
- 100 h at 90 °C,
- 250 h at 80 °C, *etc.*

The released gas volume is determined in the test. The difference in the amount of gas generated by the pure substances and their mixture allows for the determination of their compatibility. Samples of less than 0.2 mm granulation and in a form as close as possible to that in their real formulation should be used in the test. 2.5 g of pure substances and 5 g of the mixture are used, respectively. When the volume of gas produced by decomposition of the mixture is 5 cm³ higher than the sum of the amounts of gas released from the

pure substances, it is necessary to repeat the measurement. If the repeated result also exceeds 5 cm³, the system is considered as incompatible. This method is quite straightforward and hence commonly used. Its most popular application includes testing the compatibility of explosives with polymers and metals. The VST method was used, for example, to determine the compatibility of 1,3,5-trinitro-1,3,5-triazacyclohexane (RDX) and 1,3,5,7-tetranitro-1,3,5,7-tetrazacyclo-octane (HMX) with Viton and aluminium [54]. The compatibility of these materials with the polymer and aluminum was determined in the tests. The volume of gas released as a result of mixing did not exceed 1 cm³ in all of the tested systems. The VST technique also allows compatibility tests to be performed in relation to different explosives. An example is the compatibility study of CL-20 with selected non-sensitive explosives, such as nitroguanidine (NQ) or TNT [52]. The analysis involved DSC analysis and the results obtained demonstrated the so-called “uncertain compatibility” (changes in temperature of the maximum decomposition peak amounting to 4.9 and 5.5 °C, respectively). A VST analysis provided results which confirmed the compatibility of the systems, as the amount of gas released in the test did not exceed 1 cm³. Both NQ and TNT are compatible with CL-20 and such mixtures can be used. An interesting application of the VST method was a study of the compatibility of RDX/HMX with lead [55]. The purpose of these tests was to verify the stability of detonating cord made from lead tubes filled with RHA (RDX/HMX 94/6). The routine measurement performed according to STANAG 4147 standard [46] attested to the compatibility of the components. The researchers introduced a number of modifications to the method: they changed the composition from equilibrium to the one similar to that of the formulation, they also applied increased pressure and a conditioning temperature up to 135 °C. The increased pressing force served to increase the incompatibility of the resulting system. In the authors’ opinion, this may have been caused by the greater contact surface between the substances through dense packing.

In the VST method, samples of relatively large mass are used, which contributes to the better reproducibility of the measurements. The use of high energy materials in such large quantities, however, is associated with increased hazard in the case of uncontrolled decomposition of the sample. An indisputable disadvantage is related to performing measurements under reduced pressure. The tested substances must have a low vapour pressure at the conditioning temperature and avoid low molecular weight components, such as water and solvents used in processing.

2.2 Heat Flow Calorimetry (HFC)

This method involves placing a 1-5 g sample in a flow calorimeter which is then exposed to accelerated ageing at elevated temperature. The measurement is performed at 85 °C for 168 h [46]. The measurements should be taken for the pure components and their mixture in a 1:1 mass ratio, the total mass of the sample not exceeding 5 g. The resulting heat flow curves as a function of time for the pure components are summed and compared with the curve obtained for the mixture using Equation 1:

$$D = \frac{2M}{E+S} \quad (1)$$

where M is the heat released by the mixture of the explosive and the tested material (in J/g); E is the heat released by the explosive alone (in J/g), and S is the heat released by the tested material alone (in J/g).

The value obtained for parameter D allows the determination of the compatibility of the mixed components:

- if $D > 3$ then the components are incompatible,
- when $2 < D < 3$, additional tests should be carried out to test the compatibility of the system.

Before testing, the sample should be dried and sieved to eliminate particles smaller than 0.2 mm. The heat flow analysis method cannot be used in relation to substances characterized by a low temperature of decomposition. An example of the application of this method involves testing the compatibility of long-term (over 10 y) stored rocket fuels containing polymers such as [56]:

- cellulose acetate (CA),
- polyacetate (Delrin),
- polyamide (Nylon),
- polymethylmethacrylate (PMMA),
- polystyrene (PS),
- polyvinyl alcohol (PVA),
- polyvinyl chloride (PVC).

The tests were carried out at 85 °C for 168 h, an increased sample mass of 5 g was used for each component and 10 g for the mixture. VST analysis was also performed, however the results were inconsistent with the calorimetric measurements in most cases. The authors observed that the VST method was capable of measuring the volume of only the released gas, which does not always need to be directly proportional to thermal decomposition. It was also noted that the measurement often affected the colour of the sample,

which was consistent with the results obtained by heat flow calorimetric measurements, and demonstrated their incompatibility. STANAG 4147 [46] contains the information that the heat flow method should be used solely for nitrate ester-based propellants.

In heat flow calorimetry, as well as the VST method, samples of relatively large mass are used, which entails better reproducibility of the measurements and, at the same time, an increased hazard of damaging the equipment when dealing with uncontrolled sample decomposition. In order to prevent such damage from taking place, initial stability tests of samples at the scheduled conditioning temperature should be performed. The heat flow technique allows analysis of the thermal effect of the initial decomposition stage, hence for determining the basic parameter related to the thermal detonation hazard.

2.3 Thermogravimetry (TG)

Thermogravimetric analysis is the method in which the loss in mass of a sample is measured at increased temperature. According to the STANAG 4147 standard [46] measurements are carried out for pure substances and their mixtures in a 1:1 mass ratio. 1 mg of the pure substances and 2 mg of the mixtures should be used. The measurement is performed in open measuring pans. The criterion for compatibility testing is the percentage of mass loss observed in the mixture versus the pure substances, measured at the lowest temperature of the occurrence of the maximum peak of exothermic decomposition determined by DSC measurement. The theoretical mass loss is determined according to Equation 2.

$$TML = \frac{(A \cdot U_a + B \cdot U_b)}{A + B} \quad (2)$$

where TML is the theoretical mass loss (in %), A is the mass of substance A in the mixed sample (in g), B is the mass of substance B in the mixed sample (in g), U_a is the mass loss in substance A at a specified temperature (in %), and U_b is the mass loss in substance B at the same temperature (in %). The TML value is compared with the measured mass loss of the mixture:

- If the mass of mixed sample has decreased by no more than 4% in relation to TML , the tested system is regarded as compatible.
- Differences in the range of 4-20% indicate “uncertain compatibility” and further testing is required.

– Differences exceeding 20% point to the incompatibility of the tested system.

In STANAG 4147 standard [46], two feasible temperature programs are listed, *i.e.* isothermal programs, and the one with a linear increase in temperature of 2 °C/min is recommended. In non-isothermal measurements, when the result is within the range of 4-20% of mass loss of the mixed sample in relation to *TML*, the isothermal method is recommended. This method involves measurements taken at the temperature corresponding to the one of the *TML* calculations, or at a temperature 20 °C lower than the temperature corresponding to the maximum rate of mass loss. The isothermal measurement takes 1000 min.

The determination of compatibility using thermogravimetric analysis can also be conducted via analysis of changes in the kinetic parameters of the decomposition reaction. For this purpose, measurements using the non-isothermal method should be taken at three rates of temperature increase, ranging from 0.2 to 2.0 °C/min. The kinetic parameters are determined using the Kissinger method [57]. If the tested material causes a decrease in the activation energy, increased collision frequency or increased rate of decomposition of the mixture, then the tested materials are incompatible. According to the recommendations of the International Confederation of Thermal Analysis and Calorimetry (ICTAC), the Kissinger method should be avoided when seeking to determine kinetic parameters [58].

Examples of TG-based compatibility testing are the studies of FOX-7 and FOX-7 with HMX phlegmatized with Viton A [11]. The authors took measurements for 5 and 15 mg samples, whilst not noting any significant changes in the mass loss they concluded that the components were compatible. One of the most interesting modifications of this method involves the use of large samples, *i.e.* 2 g [59]. The authors tested the compatibility of mixtures containing RDX as well as 10 other components. The sample mass was increased due to the number of the mixed components. The results obtained point to uncertain compatibility in the tested system, bearing in mind however, that this is a significant departure from the conditions specified in STANAG 4147, as stated by the authors themselves in their conclusions. A CL-20 study is also an example of the interesting application of TG [60], in which the empirically obtained measurements were used to create a model of the decomposition kinetics using numerical methods. The author postulated that the behaviour of the pure components in the mixture can be predicted through elaborate processing of measured data.

Compatibility testing using the thermogravimetric method is conventionally based on low mass samples and therefore this method is safer than the heat

flow calorimetry and VST techniques. Low sample mass, however, can cause a wide range of findings. The thermogravimetric method, as is the case in the VST technique, determines the compatibility based on processes unrelated to the thermal effect of decomposition. TG-based compatibility testing should not be used for substances characterized by a high vapour pressure below the decomposition temperature. The processes of evaporation and sublimation taking place during the measurement may be misleading in the determination of compatibility [29, 61].

2.4 Differential Scanning Calorimetry (DSC)

The DSC technique involves recording of changes in heat flow as a function of temperature. According to STANAG 4147 standard, measurements are taken at a rate of temperature increase of 2 °C/min. The sample mass for pure substances and the mixture, in a mass ratio of 1:1, should be 1 and 2 mg, respectively. Hermetic pans or pans with a lid with a hole are used in the analysis.

The measured value shows the shift in the temperature of the maximum peak of decomposition versus the peak for decomposition of the pure substances. If the shift towards lower temperatures is less than 4 °C, the substances are regarded as compatible.

If the shift towards lower temperatures lies in the range between 4 and 20 °C, the resulting “uncertainty” requires additional testing. The system is incompatible if the shift towards lower temperatures exceeds 20 °C. It should be noted that all decomposition peaks in the mixture are studied. According to the standard, the compatibility of components is determined independently, so component A can be incompatible with component B, whereas component B could be compatible with component A [46].

There are few reports in the literature on the study of the compatibility of high energy materials based on DSC measurements according to STANAG 4147 standard [29, 36, 54]. On the other hand, studies of compatibility of high energy materials based on the DSC technique performed with a higher rate of temperature increase of 10 °C/min are quite frequent [50, 52, 60, 62-65]. Compatibility studies based on DSC measurements under increased pressure, *e.g.*:

- 1,3,3-trinitroazetidine (TNAZ),
- *trans*-1,4,5,8-tetranitro-1,4,5,8-tetraazadecaline (TNAD),
- poly(3-nitromethyl-1,3-methyloxetane (PNIMMO),

with other high-energy compounds can be found in the literature [62-64]. The use of increased pressure (10 atm.) is aimed at eliminating the effects of evaporation and sublimation of the tested compounds, and also the effects of the by-products of decomposition on the course of the decomposition reaction.

The determination of the compatibility of components using the DSC technique is a commonly used testing method. The advantage of this method lies in its simplicity in terms of sample preparation and interpretation of the results. As in the thermogravimetric technique for testing compatibility, a small sample mass ensures increased safety in performing the measurements, but may cause considerable discrepancies in the results obtained. According to the STANAG 4147 standard [46], hermetic pans or pans with a hole in the lid can be used in the analysis. The application of the latter type of pan to measure volatile substances will generate errors resulting from uncontrolled mass loss. On the other hand, the use of a hermetically sealed pan in the measurement of high energy mixtures which may be completely converted into gas may damage the instrument. Performing measurements in line with the STANAG 4147 recommendations, i.e. in the two types of pans, can entail changes in testing the compatibility, which has been reported in the literature [29]. Measurements performed at the rate of 2 °C/min are time-consuming and therefore a considerable section of compatibility testing reported in the literature has been performed at the increased temperature rate of 10 °C/min. Regretfully, changes in the rate of temperature increase can entail significant changes in the decomposition reaction [29, 61]. Thermal detonation can occur even at low degrees of conversion [66]. For this reason, compatibility testing should focus on analysis of the initial stage of decomposition. Testing of compatibility with the DSC technique of complete decomposition can be substituted with DSC analysis of the partial decomposition. Such a solution allows for the analysis of thermal decomposition at low degrees of conversion using samples of several milligrams [67]. During the analysis, complete decomposition of the sample in the measuring pan should be absolutely avoided. The measurement is terminated when the increase in the calorimetric signal exceeds the experimentally determined critical heating power.

2.5 Chemical analysis

This method involves conducting an ageing process for the samples, followed by analysis of the resulting chemical changes. In the STANAG 4147 standard, two procedures that are applicable for testing the compatibility of ammunition components with nitrate ester-based propellants, as well as ammunition components with lead azide and silver, are described. The measured values include the level of stabilizer residue in the nitrate ester sample after 14 days of heating at 80 °C using gas chromatography (GC) or high-performance liquid chromatography (HPLC), or titration of the azide remaining in the sample.

2.6 Non-thermal methods

The results of compatibility studies using non-thermal methods such as:

- rheological change analysis,
- density functional theory (DFT),
- Fourier-transform infrared spectroscopy (FTIR),
- X-ray diffraction (XRD)

can be found in the literature. These methods look for new interactions between the mixed components and these interactions are assumed to result from incompatibility of the components. The authors of paper [65] reported a study on the compatibility of PNIMMO and plasticizers. In addition to the standard thermal methods, rheological analysis of mixtures was utilized to test their compatibility, based on variations in the viscosity. They also performed DFT simulations to model the behaviour of the mixtures, which were subsequently compared with the empirically obtained results of viscosity measurements to reach good agreement. The application of this methodology to high energy polymers may help in understanding their structure and compatibility with the plasticizers used. FTIR and XRD are other techniques that can be used to test compatibility. These methods were applied in the verification of new functional groups in the samples (FTIR) or structural changes occurring in the samples (XRD) [52]. Another application of the above methods includes the study [50] of the compatibility of 2,6-diamino-3,5-dinitropyrazine-1-oxane (LLM-105) with HMX, CL-20 and 3,4-dinitrofurazanofuroxane (DNTF). FTIR analysis served to demonstrate that the spectrum of the LLM-105/CL-20 mixture is a combination of the spectra of the individual components, while several bands disappeared in the LLM105/HMX spectrum, which may indicate a mutual interaction between these substances. In the LLM-105/DNTF spectrum, a new band was observed at 2900 cm^{-1} , attributed by the authors to the formation of a new carbon-hydrogen bond. Furthermore, the XRD results indicated mutual interactions between these components and their absence for the LLM-105/CL-20 mixture. Performing DSC and VST measurements confirmed the incompatibility of the LLM-105 systems with HMX and DNTF, as well as compatibility with CL-20.

When using the FTIR technique, the authors of paper [8] observed changes in peak intensities for a melt-cast TNT/DINA mixture. Changes were observed for the nitrate group peaks in DINA and the CH_3 - peaks in TNT. They were found to have resulted from interactions between these groups for a melt-cast mixture with eutectic composition. After DSC analysis, no destabilizing effect of the TNT/DINA mixture was observed as compared to the component of lower stability.

A study of the compatibility of FOX-7 with nine commonly used high energy materials was presented in [10], in which DSC, VST and XRD were used. A comparison of the results obtained demonstrated that the XRD and VST methods produced the same outcome. All of the investigated systems were compatible, however one mixture was found to be incompatible by the DSC method.

The authors of paper [25] tested the compatibility of 2,4-dinitro-2,4-diazapentane (DNDA-5) with energetic binders using the thermal DSC method and a non-thermal technique involving quantum-chemical calculations with DFT. The results of the thermal method came to the conclusion that the investigated compound is compatible with three out of the four tested binders. Similar results were obtained for the DFT method.

The application of thermal and non-thermal methods for testing compatibility is described in paper [51]. The compatibility of 5-amino-1*H*-tetrazole nitrate (5-ATN) with explosives and other additives such as aluminum, was investigated. The use of thermal methods (DSC and VST) demonstrated that 5-ATN was compatible with the majority of the tested substances, however only partial compatibility was exhibited with potassium perchlorate (KClO₄). FTIR analysis confirmed these findings, while XRD analysis revealed interactions for 5-ATN/Al and 5-ATN/AP mixtures. These interactions were found not to affect the safety directly, however may entail a shorter shelf life of the material containing such mixtures.

Non-thermal methods could be used as screening methods, allowing for a rapid preliminary determination of the compatibility of selected systems. For some systems however, non-thermal methods produced different results to those obtained by thermal methods [8, 25, 51], and therefore these methods should be avoided when seeking to establish unequivocal compatibility.

3 Summary

- ◆ There is a number of testing methods that can be used to determine the compatibility of high energy mixed components. The role of the experimenter is to select the method which is the most suitable in relation to the chemical and physical properties of the substance.
- ◆ Substances characterized by a high vapour pressure at the temperature of decomposition should not be tested with the VST, TG and DSC techniques using non-hermetic pans. The sublimation and evaporation processes will yield inconclusive results. The measurements should be conducted in such a manner as to ensure the safety of the operator and the equipment,

bearing in mind that increasing the mass of the sample can significantly escalate the scale of damage in the event of an uncontrolled decomposition. Notwithstanding, the increased mass of the sample allows for better reproducibility of the results obtained.

- ◆ According to the literature, non-thermal methods such as FTIR, XRD, GC-MS, NMR, and DFT can be used for the initial testing of compatibility. It should be noted however, that none of these techniques provides information on the rate of decomposition. The application of these methods should be supplemented by at least one thermal method for testing compatibility.
- ◆ The risk of a thermal explosion is associated with exothermic reactions and not the release of gaseous decomposition products. Therefore, it is more appropriate to use the methods seeking to establish the thermal effect of reactions performed by differential scanning calorimetry or heat flow calorimetry than the application of manometric or thermogravimetric methods for establishing compatibility. The compatibility of components is determined most effectively using heat flow calorimetry or partial decomposition, as these methods allow the study of changes in the initial decomposition stage. Analysis of changes taking place at the initial stage of decomposition is quite important because a thermal explosion can occur even at low degrees of conversion.

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