Compatibility at CEA Le Ripault

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GTPS
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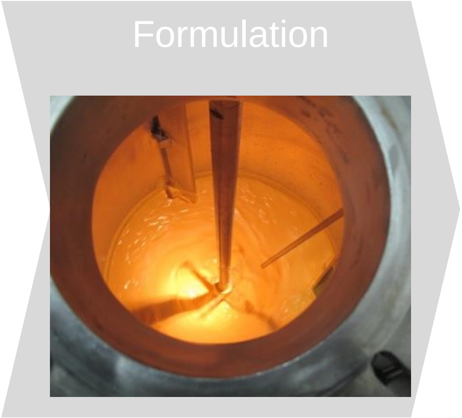
Conclusion

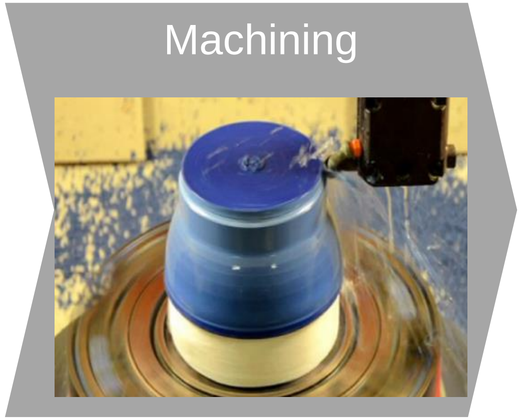
1 - Context

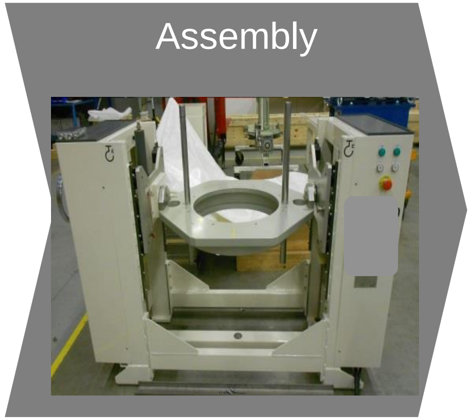
1. Secondary explosives (almost exclusively)

2. Life cycle
   a. Formulation
      → formulation ingredients (thermal stability)
   b. Pressing
      → casting pockets, oil, felt, mold release agent, ...
   c. Machining
      → cutting fluid, fingerprinting, supports, ...
   d. Assembling
      → glues, joints, diagnostics, adhesives, ...
   e. Handling/storage
      → packing materials, packaging materials, ...
   f. Operational life
      → glues and joints

3. Materials
   - in production (large feedback)
   - in development (low level of maturity)







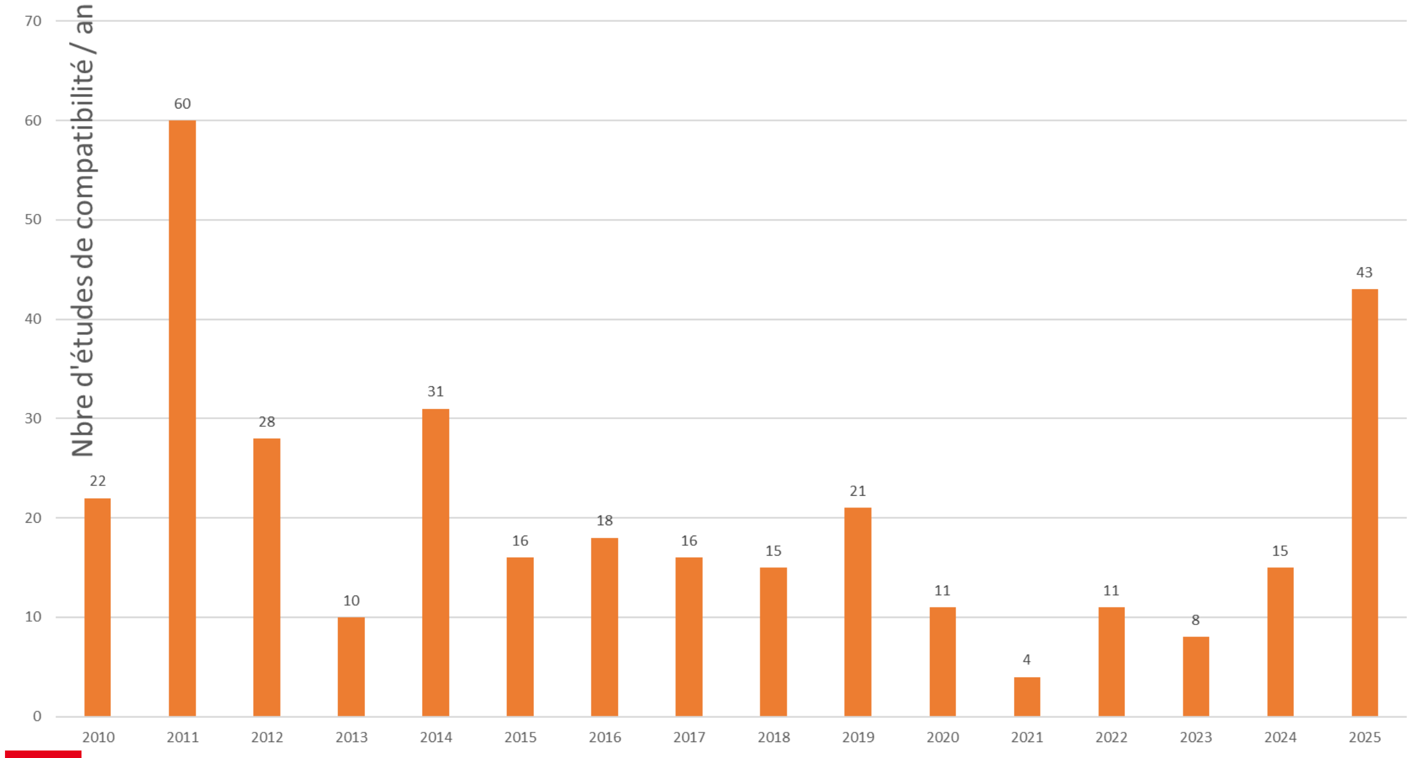
1.1 - Origin of needs

Materials in production

- Process evolution, regulatory evolution, supplier disappearance/change, qualification of alternative supplies, etc

Materials in development

- For each new pyrotechnic material, the compatibility of all materials encountered at the different stages of the life cycle must be retested



Number of compatibility studies per year

1.2 - Normative bases

- **STANAG 4147** (Edition 2) : NATO standardization agreement
  → compatibility of munition components with explosive substances

  The STANAG applies to all categories of energetic materials and lists the main tests used by member countries :
  - Measurement of gas release under vacuum (V-test)
  - Microcalorimetry at flow
  - Thermogravimetry (TGA)
  - Differential scanning calorimetry (DSC)
  - Chemical analysis after aging

- Operational modes **GEMO** (managed by DGA = French procurement agency)

  → compatibility of munition components with explosive substance

  The methods proposed by the GEMO are not all listed in the STANAG 4147

Remark: Nuclear armament is not within the scope of either the STANAG or the GEMO modes

1.3 - Definitions

Reactivity :

Tendency of a substance to participate in chemical reactions

Stability :

Ability of a material not to change. It is always evaluated in relation to a domain of use

A material is stable if it retains its form, its state, its properties in the intended domain of use (time, temperature, pressure, atmosphere, etc.)

Compatibility :

1) Evaluate any modification of the stability domain of substances as a result of their contact
2) Take a position on the acceptability (or not) of these modifications in relation to the intended domain of use :

It is the pronouncement of compatibility that is established in relation to a domain of use specified by the requestor.

The expression of need (domain of use: intended use, time, temperature, etc.) is determining.

It conditions the test parameters and the pronouncement of the test
because **there is no absolute compatibility.**

1.3 - Generalities

Two products are said to be **compatible** if their interactions do not harm the required properties

Mechanisms involved and the parameters to be monitored are different depending on the explosive materials :

- Secondary explosives (gas production)
- Nitrocellulose, nitroglycerin, liquid nitric ester propellants (stabilizer consumption)
- Pyrotechnic compositions (sensitive to humidity)
- Primary explosives (sensitive to humidity)
- Composite propellants (sensitive to humidity, modification of the binder)

To make the effects appreciable, it is necessary to accelerate the reactions by raising the temperature. "Standardized" tests exist for the different modes of degradation. However, gas release measurement is the most frequently used parameter.

Remark: it may be interesting to define an approach combining several tests in order to produce a more substantiated compatibility.

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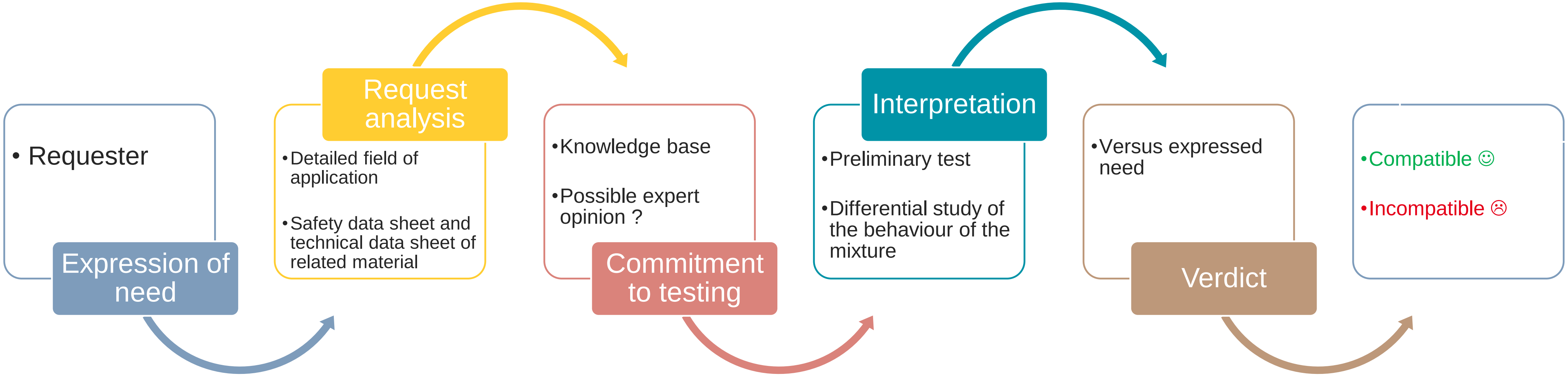
2.1 - General approach



2.2 - Characteristics of available methods

Activation devices

- Oven
- Calorimeter

Characterisation methods (GEMO – STANAG*)

- Loss of mass
  - gravimetry (pycnometer)
  - TGA
- Gases production
  - Vacuum test
- Heat production
  - DSC
  - µ-calorimetry

Poorly adapted to products with high vapor pressure (liquids or volatile solids)

Characteristics of some characterisation methods

Gravimetry (pycnometer)	TGA	Vacuum test (V-test)	DSC	µ-calorimetry
Isothermal 400h (17 days)	Isothermal (16h) Dynamic (3h)	Isothermal 40 h (STANAG) 200h (GEMO) 70h (CEA)	Dynamic (1/2h)	Isothermal (145h) (STANAG)
2 g	2 at 5 mg	**2 g**	2 - 10 mg	**2 g**
Differential loss of mass (> 20%)	Differential loss of mass (> 20%)	Differential gases production (> 1 cm^3/g)	Shift of decomposition temperature (> 4°C)	Differential heat production (> 30J/g)

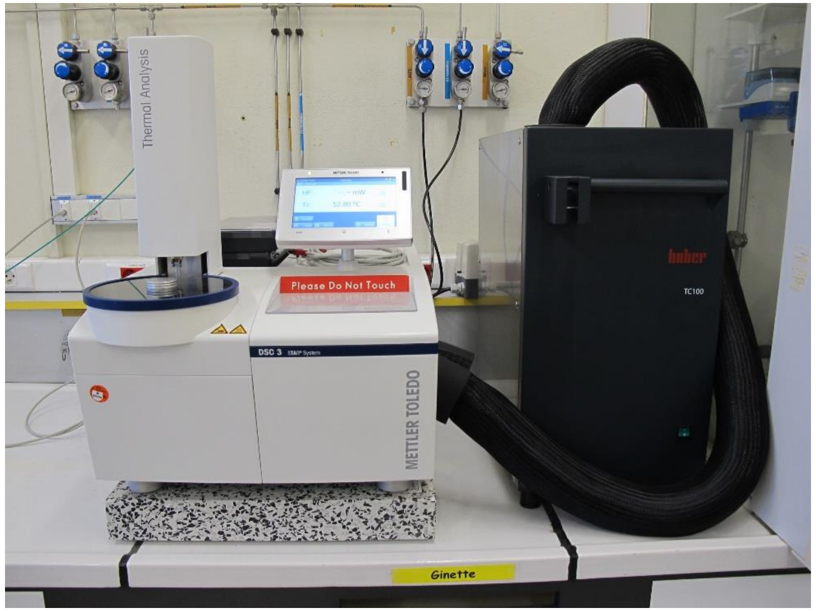
Methods used at CEA / Le Ripault (Vacuum test, DSC)

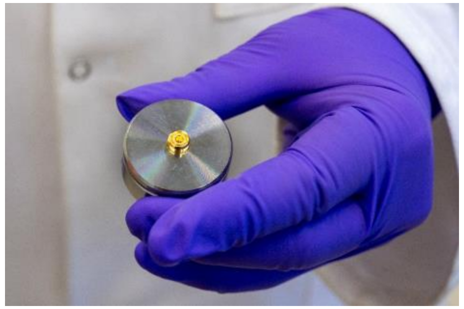
* The use of 2 complementary methods is recommended given the strengths and weaknesses of the different methods

cea

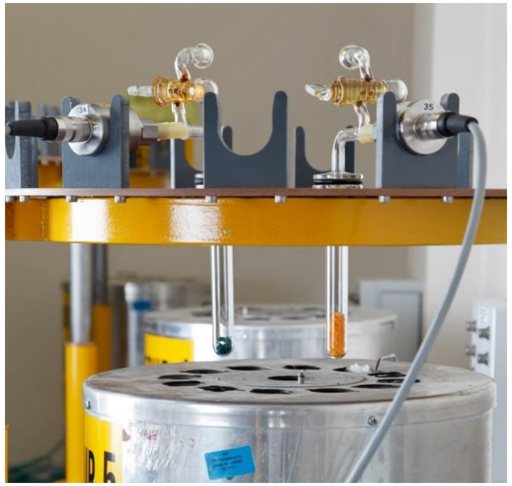
2.3 - Our equipment

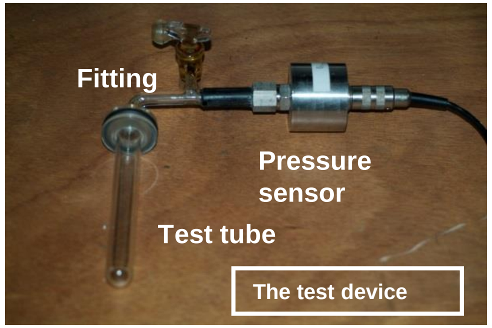
DSC





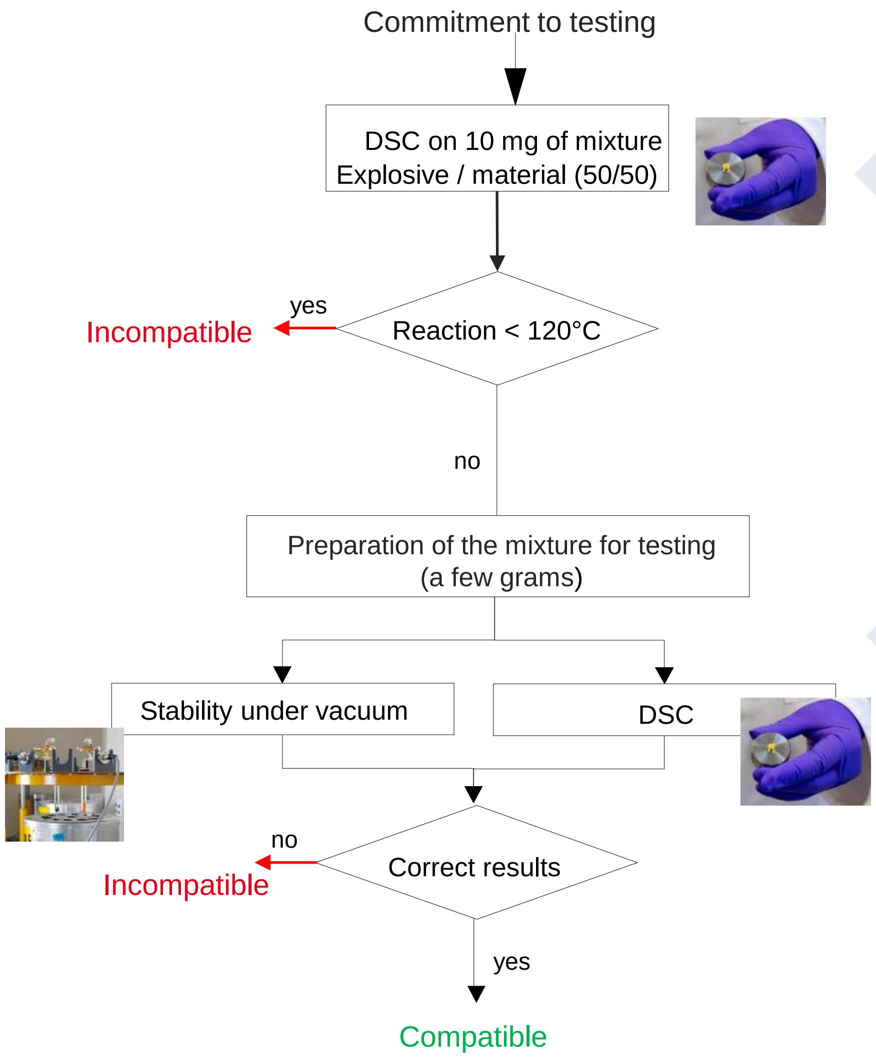
Vacuum test





2.4 - Test synoptic

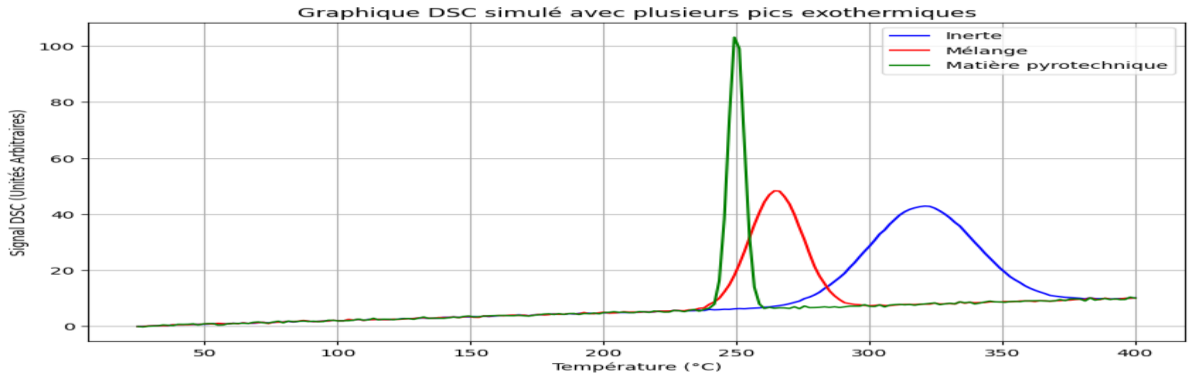
Principle : step by step, knowledge, mass and solicitation are going up together

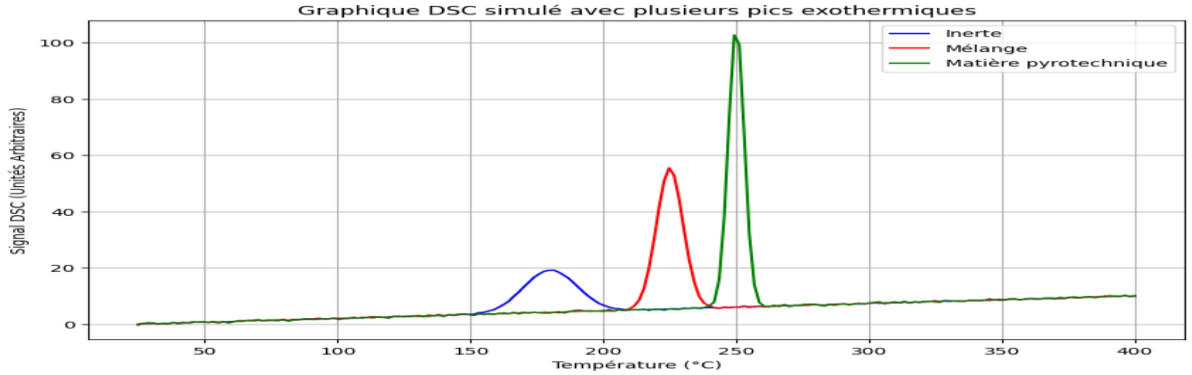


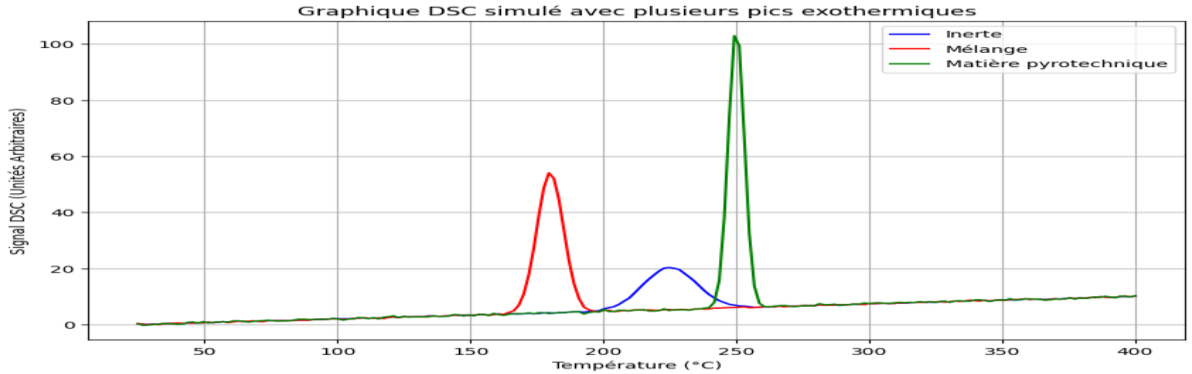
Step	What	How	Remarks	Stop event
Preliminary test	Behaviour at room temperature	Visual observation after mixing products together	Low mass (few mg) Low temperature Isothermal Low time (few minutes)	Fumes, bubbles, colour changes, obvious instability, etc
	Thermal stability discovery	DSC of the mixture up to decomposition	Low mass (few mg) High temperature Dynamic Low time (few minutes)	Thermal stability < 120°C
Differential comportment study	Knowledge of thermal behaviour of mixture compare to individual components	DSC comparison of products vs mixture up to decomposition : detection of limits and margins	Low mass (few mg) High temperature Dynamic Low time (few minutes) This is a limit test; objective : knowledge of limits	No stop excepted when limits are unacceptable for expected use
		Vacuum stability test comparison of products vs mixture at fixed temperature (1)	High mass (few g) High temperature Isothermal Long time (few days) (High sensitive test based on gas production) This is a test at fixed temperature : objective : validation of the intended use temperature	Mixture excess gases production is more than 1 cm^3/g

✓ (1) Depending on the use : generally maximum temperature level during lifetime or process (but never less than 80°C)

2.5 - DSC criteria







Criteria	Conclusion
$T_{explosive} = T_{mixture}$ at ± 10°C	No modification of the thermal stability domain of the pyrotechnic material
$T_{inert} < T_{mixture} < T_{explosive}$	It may be an interaction or a thermal entrainment: is the new stability limit acceptable in relation to the intended use ? The modification may be prohibitive
$T_{mixture} < T_{inert} < T_{explosive}$	Manifest interaction: is the new stability limit acceptable in relation to the intended use ? The modification may be prohibitive

2.5 - Vacuum stability

R = C – 2 (aA + bB)	Conclusion
R < 1,0	COMPATIBLE
1,0 < R < 2,0	Incompatible at the intended temperature. Based on the need, it may be possible to repeat the test at lower temperature
R > 2,0	INCOMPATIBLE

With

- R: Volume of gas released as a result of a reaction between the mixture constituents
- A: Volume released by 1 g of pyrotechnic material alone, explosive or explosive composition (cm^3)
- B: Volume released by 1 g of the inert material to be tested alone (cm^3)
- C: Volume released by 2 g of pyrotechnic material/material to be tested - mixture (cm^3)
- a and b: mass fraction of A and B in the mixture (example: mixture of 90 % of A and 10 % of B → a=0.9; b=0.1)

Examples

at 110°C

Volume released by 1 g of explosive composition V1	Volume released by 1 g of the material tested V2	Volume released by the mixture consisting of 1 g of explosive composition and 1 g of the material tested V0	$V_0 - (V_1 + V_2)$ (excess of gaz release)	Degree of reactivity
0,12	0,63	2,02	**1,27**	Incompatible at the intended temperature

at 100°C

Volume released by 1 g of explosive composition V1	Volume released by 1 g of the material tested V2	Volume released by the mixture consisting of 1 g of explosive composition and 1 g of the material tested Vo	$V_0 - (V_1 + V_2)$ (Excess of gaz release)	Degree of reactivity
0,09	0,45	0,85	**0,28**	Compatible

* The volumes (cm^3) are brought back to normal temperature and pressure conditions

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3 - Special cases

Expert opinion (possible under conditions)

- The material is inert and stable (according to the Safety data sheet) and has no chemical reactivity in itself.
- The explosive material is perfectly known and is chemically stable over a wide temperature range.
- Contact is planned under natural climatic conditions only.

Development of a composition = compromise

- This case is not about compatibility but thermal stability.
  - Ingredients are added to confer or improve a property (rheology, mechanical strength, mechanical sensitivity ...).
  - Sometimes a compromise with an acceptable decrease in thermal stability is required

Sometimes formulating new energetic materials needs to mix inert + inert to get explosive

- In this case, we start with a study of the mixture (on a few mg) using DSC
  - Verification of no chemical modification at room temperature (NMR)
  - Verification of the decomposition temperature is within the reference (>181°C Pentrite)

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In conclusion

Compatibility at CEA Le Ripault

- We rely on two techniques: **DSC and Vacuum test**
- A **precise and detailed expression** of need, concerning the field of application (use, duration, temperature), is essential.
- Technical and regulatory monitoring is mainly ensured through participation in GEMO meetings.

Volume

- 700 compatibility studies since 1977
- 300 studies since 2010
- Nominal capacity: ~ 20 to 25 studies per year

Load/capacity → Point of vigilance

- **Limit the load**
  - Expert advice for simple cases
  - Slow down comfort requests
  - Avoid requests for over-security
- **Adapt the capacity**
  - Smooth the load
  - Reinforcements if necessary
- Difficult to correctly anticipate the future load: multiple requestors

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Thank you