

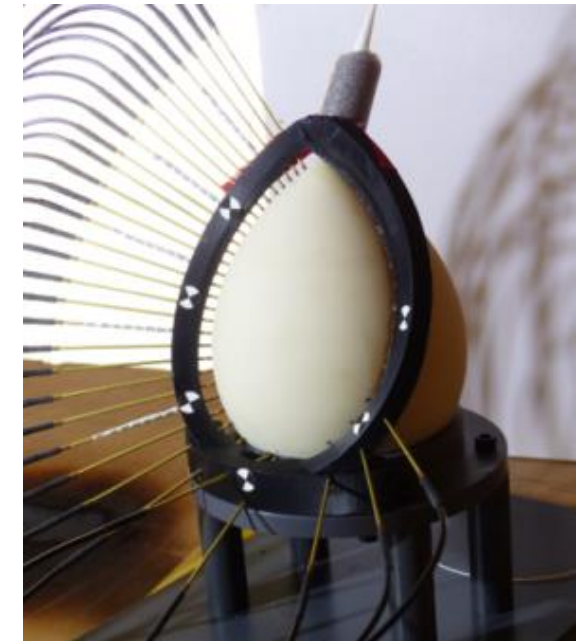
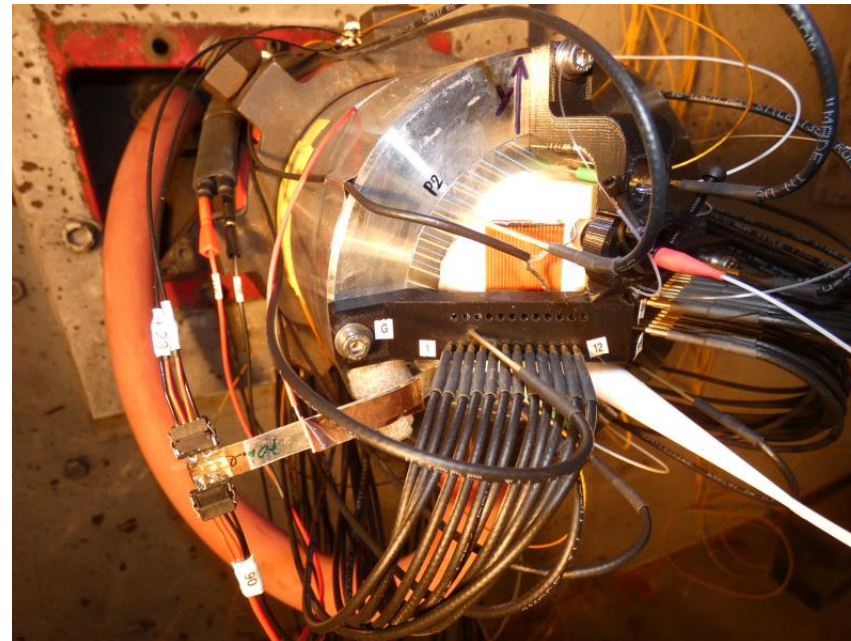
Compatibilty issues at CEA Gramat

A. Osmont

Activity at CEA Gramat

HE characterization

- Experiments
 - Detonics (SDT, rate stick)
 - Effects (Cylex, blast)
- Samples
 - melt-cast
 - cast cured
 - pressed
- Preparation
 - Mixing/pressing
 - Machining
 - Assembling (gluing)



Compatibility tests at CEA Gramat until 2017

- Apparatus available
 - DSC
 - TG
- Procedure
 - Mainly based on DSC + TG
 - Reserved compatibility solved with DSC isothermes 72 h @ 120 C

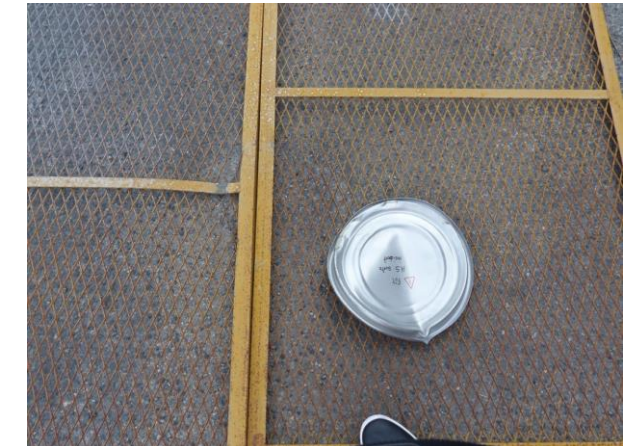
2017 events

HE characterization

- TNT + wax
 - No incompatibility warning during tests (DSC-TG only)
 - Color change at larger scale



- UN + Al
 - No incompatibility warning during tests
 - Pneumatic explosion of wet wastes in an hooped metal barrel



2017 events : consequences

- Is it possible to
 - Mix products ?
 - Glue products ?
 - Machine products under water ?
- Need for a compability procedure

Chemical compatibility : texts and norms

- **STANAG 4147** : NATO handling procedure
- **GEMO** :
 - CMP-100-00 : General principles (definitions, advice)
 - CMP-101-00 : Preliminary tests (contact at mg scale)
 - CMP-102-00 : Sample preparation (physical shape, order of introduction...)
 - CMP-200-00 : Vaccum test (mass, temperature, duration conditions)
 - CMP-207-00 : Compatibility flowchart (for vaccum test)
 - STC-500 : Compatibility with thermogravimetric method (1g HE, 95°C, 400h, → $\Delta m < 10\%$ compatible $10 \leq \Delta m \leq 20\%$ restricted, $\Delta m > 20\%$ incompatible)
- **NF Norms (GEMO ⇔ AFNOR)**
 - NF T70-516 et T70-517: vaccum test : 2 g mix, 100°C, 200h, 60h min → curve shape + réactivité : $R < 1$ cm³/g faible, $1 \leq R \leq 2$ modérée, $R > 2$ excessive
 - NF T70-535 : Compatibility by DSC 5mg HE, 5°C/min → si $\Delta T < 5^\circ\text{C}$ compatible
 - NF T70-538 : Compatibility by TGA 5mg HE, 5°C/min, → $\Delta m < 4\%$ compatible, $4 \leq \Delta m \leq 20\%$ restricted, $\Delta m > 20\%$ incompatible
-

Procedures used by different GEMO members



- Necessity to obtain results from 2 different tests

- **Different practices**

- STANAG
- GEMO rules (⇒NFT70)
- Following the customer requirements
- Following their own procedure (CEA LR)

GEMO members

DGA TT, KNDS Bourges & Tarbes,
Thales, Lacroix, Eurenco, Ariane
Group, CEA LR & Gramat

- Possible conclusion for each test

Favorable < Restricted < Unfavorable
(F) (R) (U)

- If restricted clue, need for an other analysis technique

- Difficulties shared by all members

Procedure scope

Fabrication including HE

- Contact with non pyrotechnic products
 - Glue
 - Liner
 - Painting, pigment
 - Oil, grease
 - Solvant
- Mixture with non pyrotechnic products
 - Binder
 - Dye
 - Additive
 - Other substance

Out of the scope

- Mix of inert materials (pyrotechnic mixture)
 - → Formulation procedure

Procedures architecture

- #1 : general principles
 - Formal request
 - Preliminary analysis (a priori compatibility ?)
 - Test protocol (polymerized glue ? Proportions ?)
 - Tests → procedure #2
 - Assessment to conclude on compatibility

- #2 : operating documents
 - Description of each test
 - Compatibility clue

- #3 : scale up of mixes
 - Increase of produced quantities
 - To adapt for each process
 - Acceptation criterias

Request description

- Study context
- Informations on components
 - Nature (glue, dye, ...)
 - Physical shape (powder, granulometry, ...)
 - Safety datasheet
- Informations on handling conditions
 - Ambient conditions
 - Temperature and pressure for pressing
 - Contact duration
- Every other useful information

Request analysis

Bibliographic check

- From safety datasheets and bibliographic data
 - Known compatibilities or incompatibilities
 - Reactivity of mixtures (reducing or oxidizing agents, acids, bases, solubility)
- Analysis of constraints linked to the use or the fabrication conditions
 - Glue with exothermic polymerization
 - Pressing in temperature
 - Thermal environment during experimentation

A priori compatibility ?

- Yes : stable materials often used
 - Natural fibers (cotton, wool, wood, ...)
 - Polymeric materials (PE, PVC, PTFE, cushioning foam, ...)
 - Construction materials (concrete, plaster)
 - Metal blocks, glass
 - Honey
- No : Conducting tests
 - Other component

Tests to be carried out

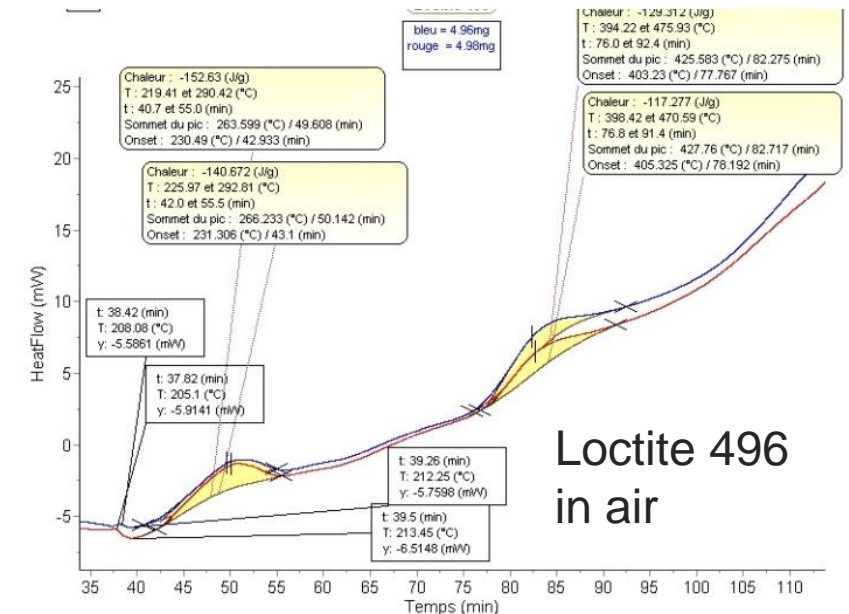
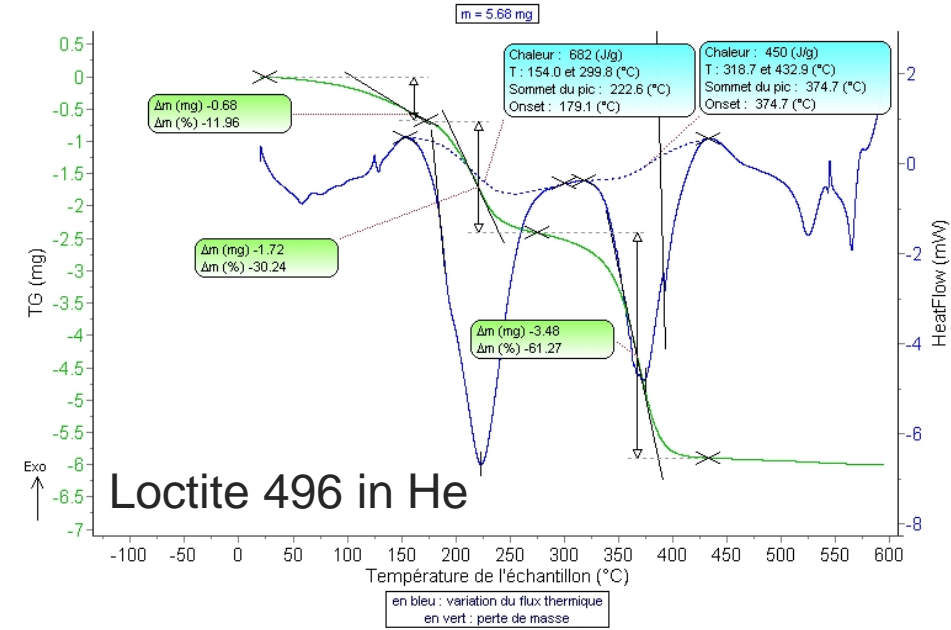
Sample preparation

- Glue : polymerized or not ?
- Solvant evaporation for liquids (dilutants, paintings, oil)
- Tests under inert atmosphere ?



Effect of oxygen in the crucible

- Reaction of decomposition
 - HE : exothermic
 - Other organic compound : endothermic
- Organic compound / oxygen reaction
 - Exothermic
 - 1 μmol oxygen in the crucible
 - Exemple of benzene : $3\text{MJ/mol} \Rightarrow 0,4 \text{ J}$
 - Loctite $\Rightarrow 0,7 \text{ J}$
 - Consistent order of magnitude
 - \Rightarrow Initiation of HE possible by the combustion of the organic compound
- Point to address
 - Cheminal incompatibility between the HE and the organic compound
 - Not the possibility to ignite the HE with the burning compound
 - Necessity to work under inert atmosphere

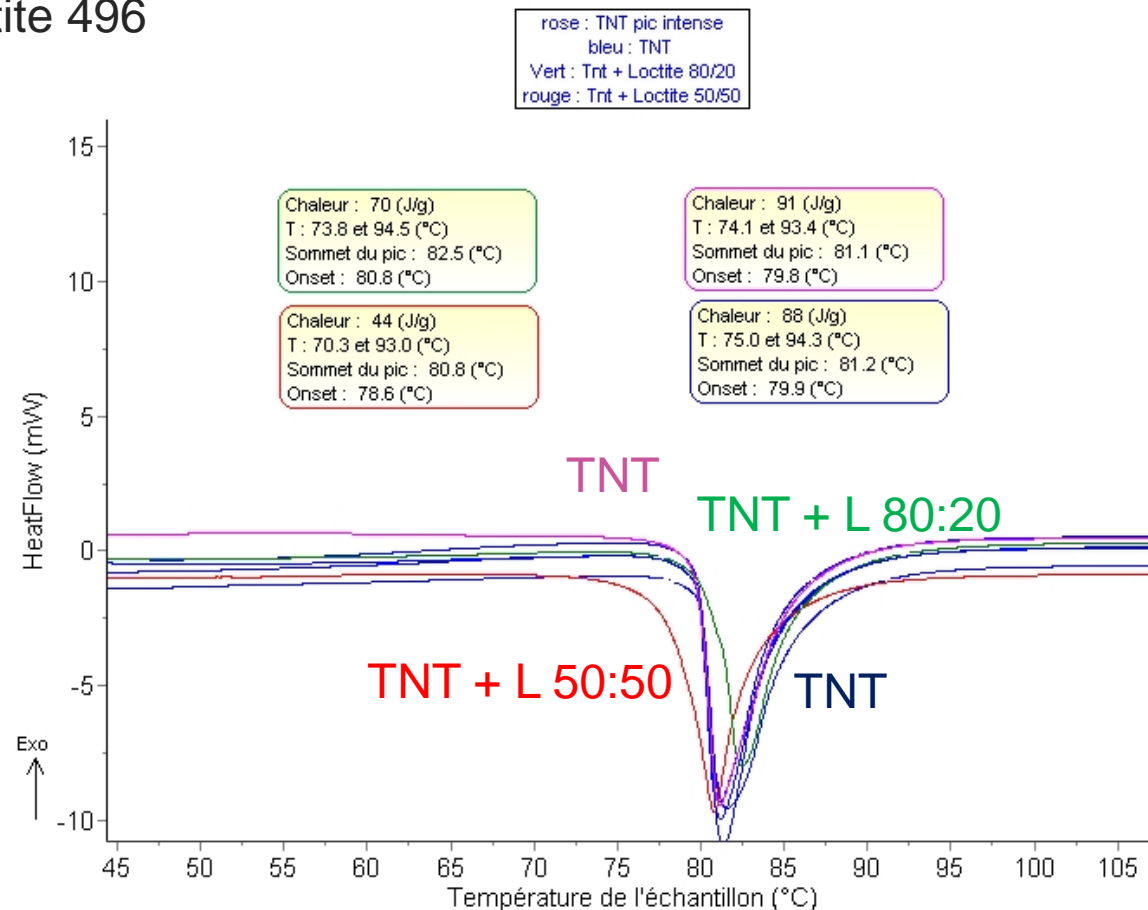
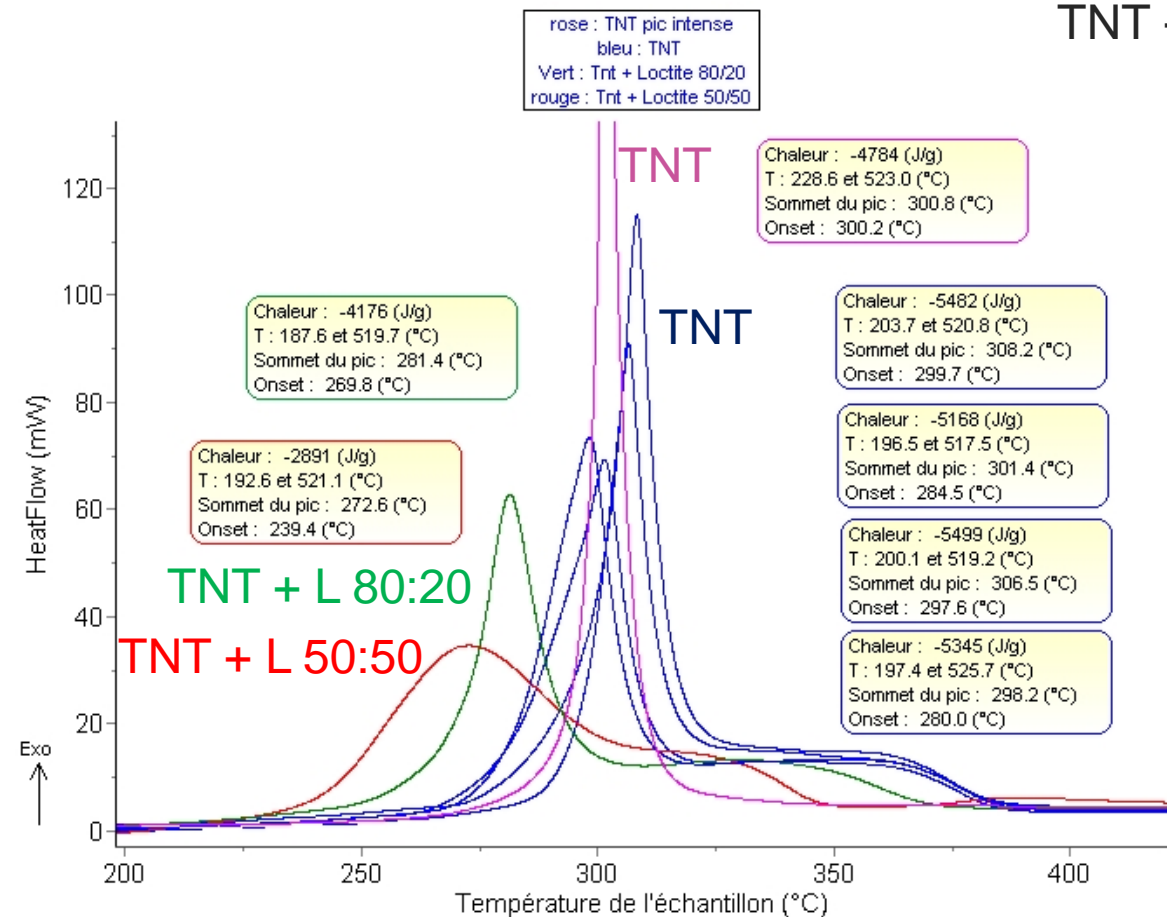


Tests to be carried out

Mixture proportion

- General rule : 50:50 : safety margins

TNT + Loctite 496

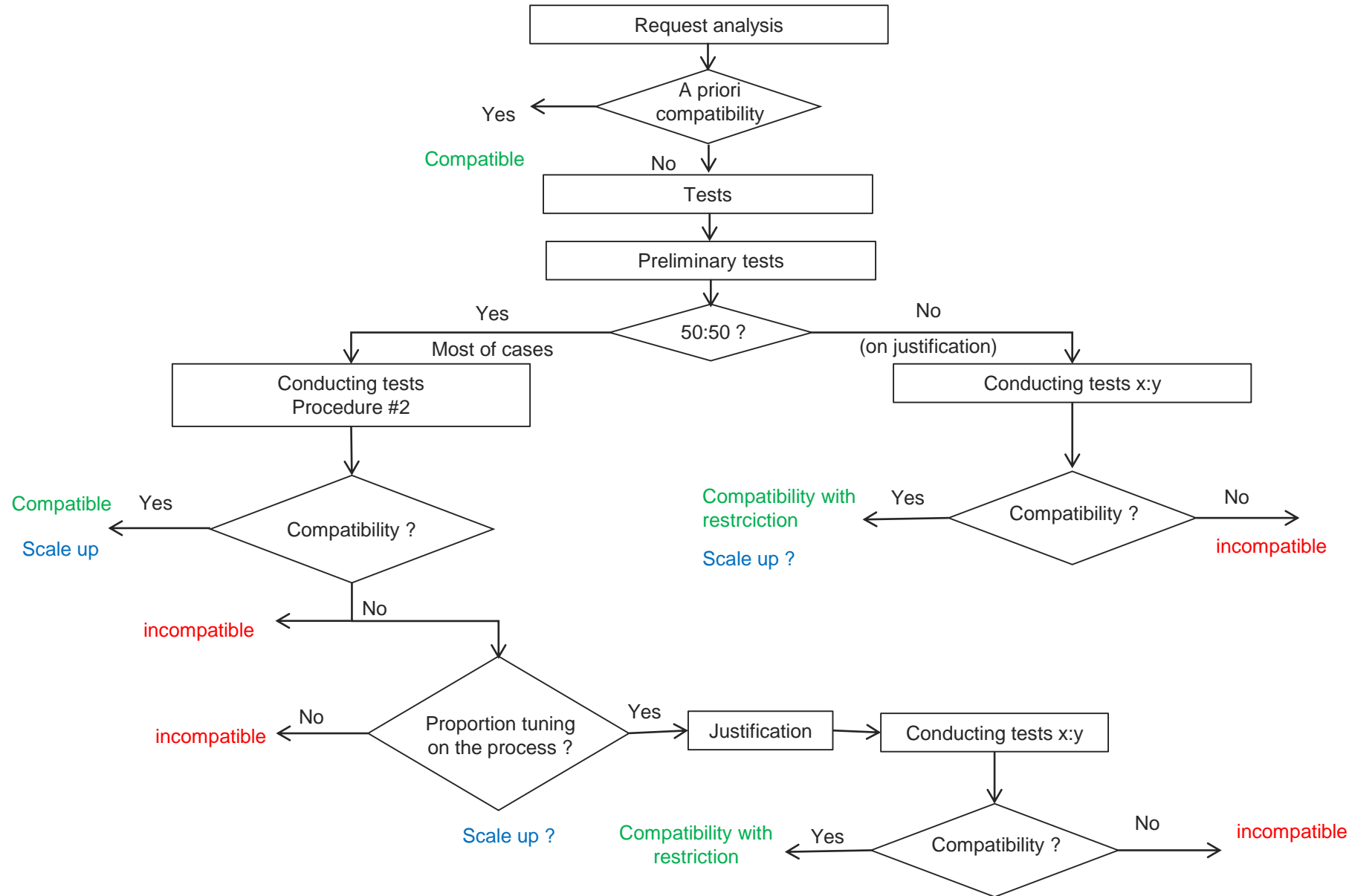


Tests to be carried out

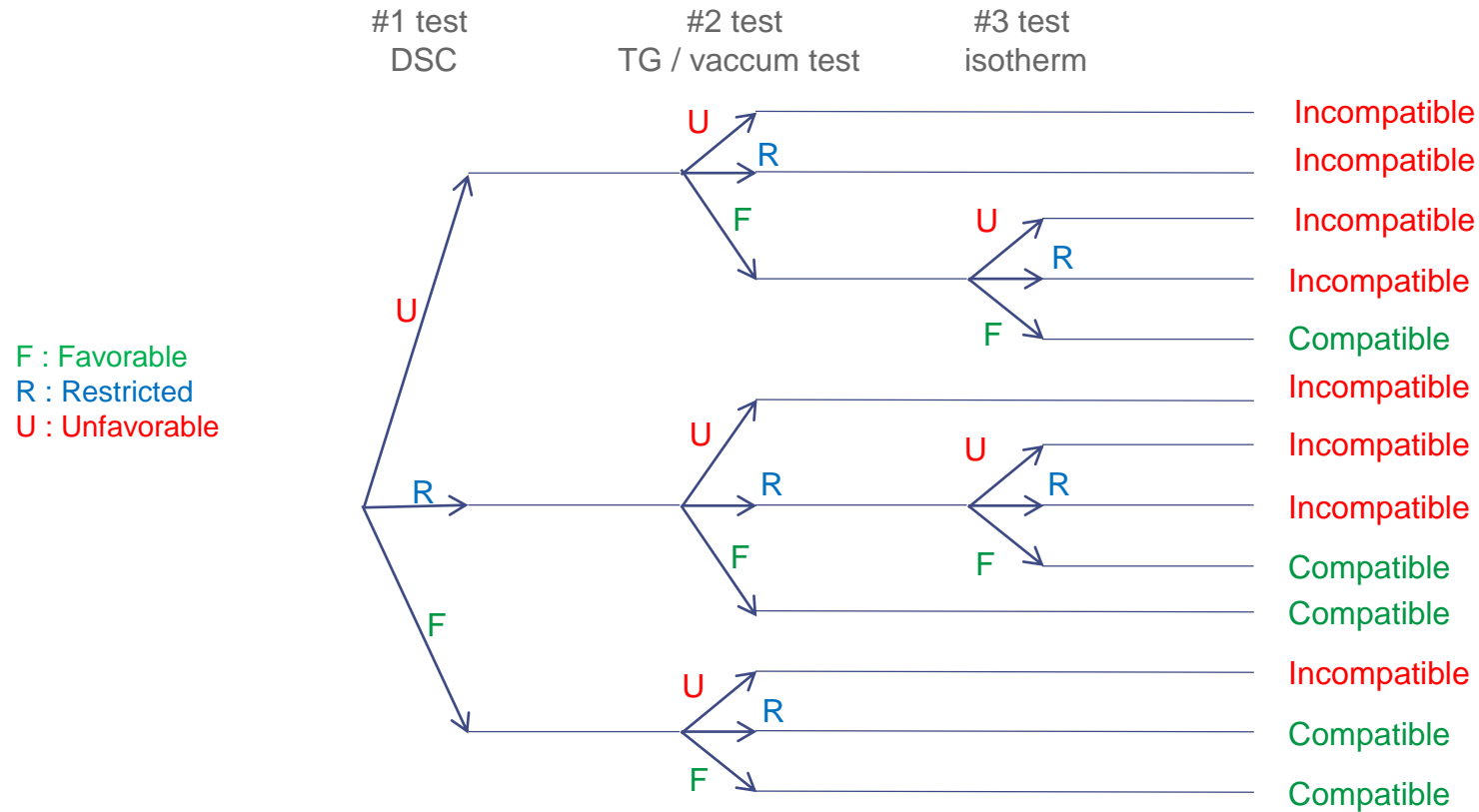
Mixture proportion

- General rule : 50:50 : safety margins
- Possibility of adaptation of the ratio due to handling conditions
 - Contact gluing : small quantity of glue
 - Use of painting : large quantity of solvent, risk of dissolution of the HE
- Mixture with 3 components A-B-C (a:b:c)
 - Binary tests A-B, B-C, A-C 50:50, then A-B-C real proportions
 - A-B 50:50, then AB (a:b) – C 50:50
 - For some laboratories : A-B-C a:b:c

Acceptance flowchart



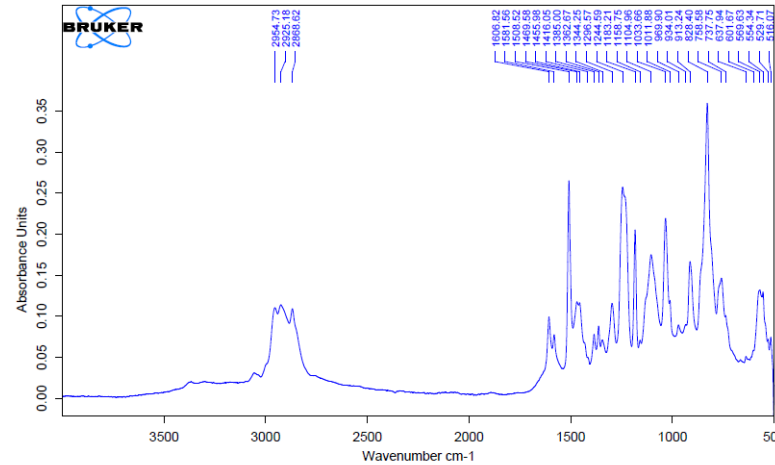
Compatibility acceptance



Procedure #2 : preliminary tests

IR spectrometry

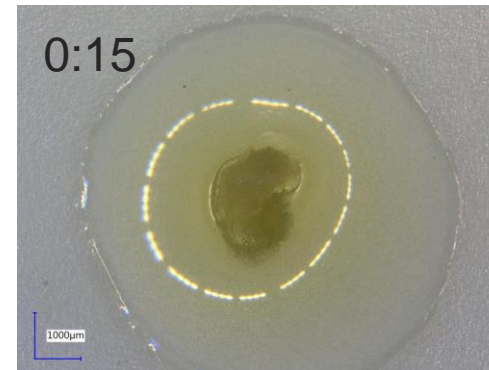
- Spectrum of each component
- Use of spectra comparison to validate the compatibility for new lots of components



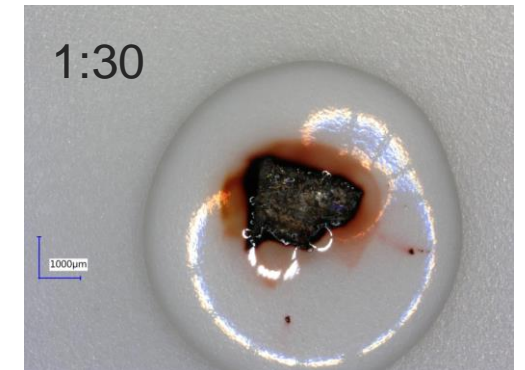
Contact test

- Test under microscope
- 2 scales : 10 mg / 50 mg
- Temperature evolution with IR gauge
- Incompatibility criteria
 - Fumes or gas release (bubbling, effervescence)
 - Anormal change of texture (viscosity, physical state)
 - Abnormal color change

TNT + Loctite 496



TNT + epoxy



28/10/2022

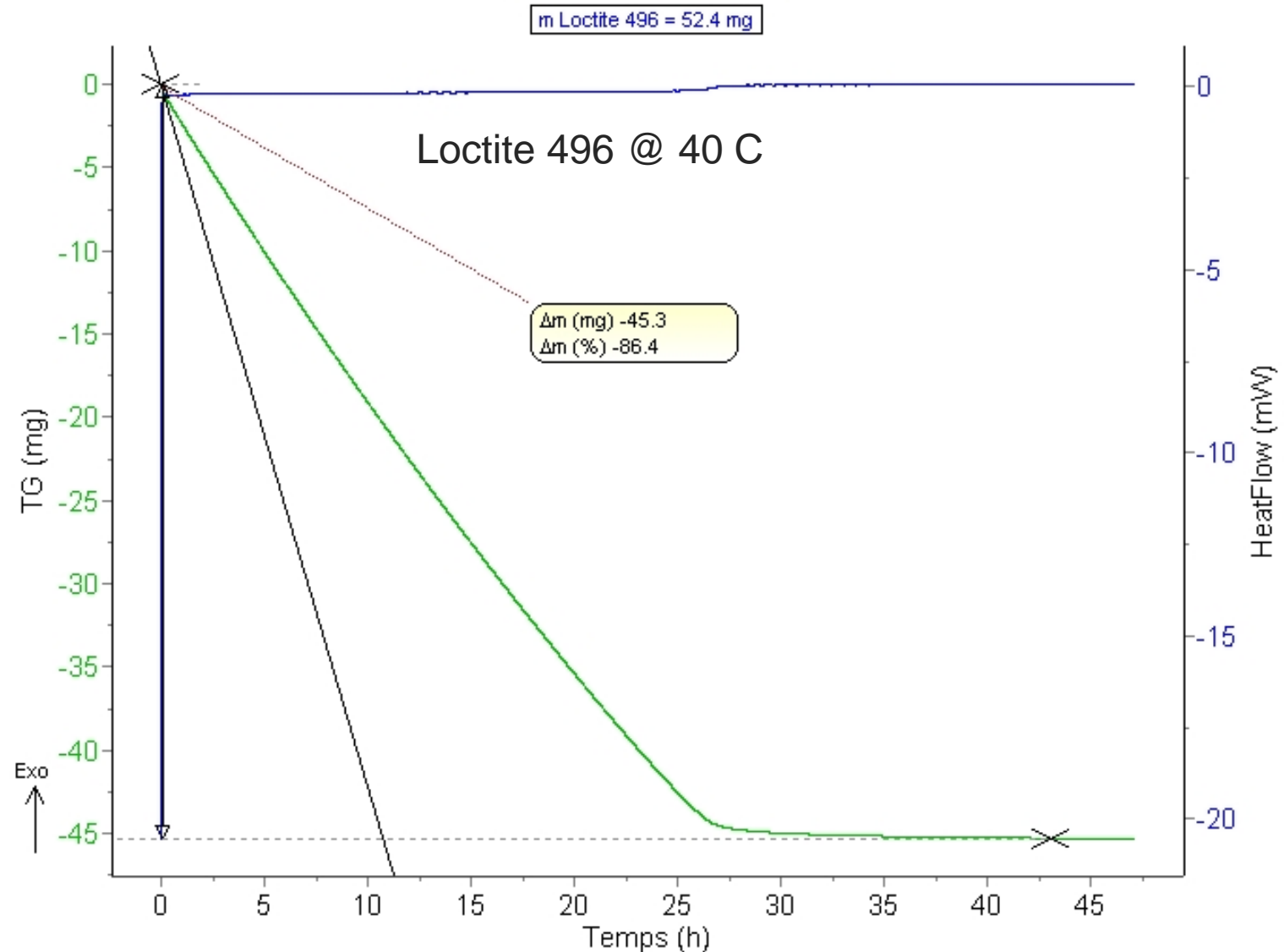
Procedure #2 : compatibility with a glue

Polymerization study

- Isotherm 40 C or $T_{pol} + 10$ C
- If exothermic : admissible ?

Contact test

- If exothermic reaction
 - HE + glue TG analysis
 - Same isotherm : reaction of the HE ?



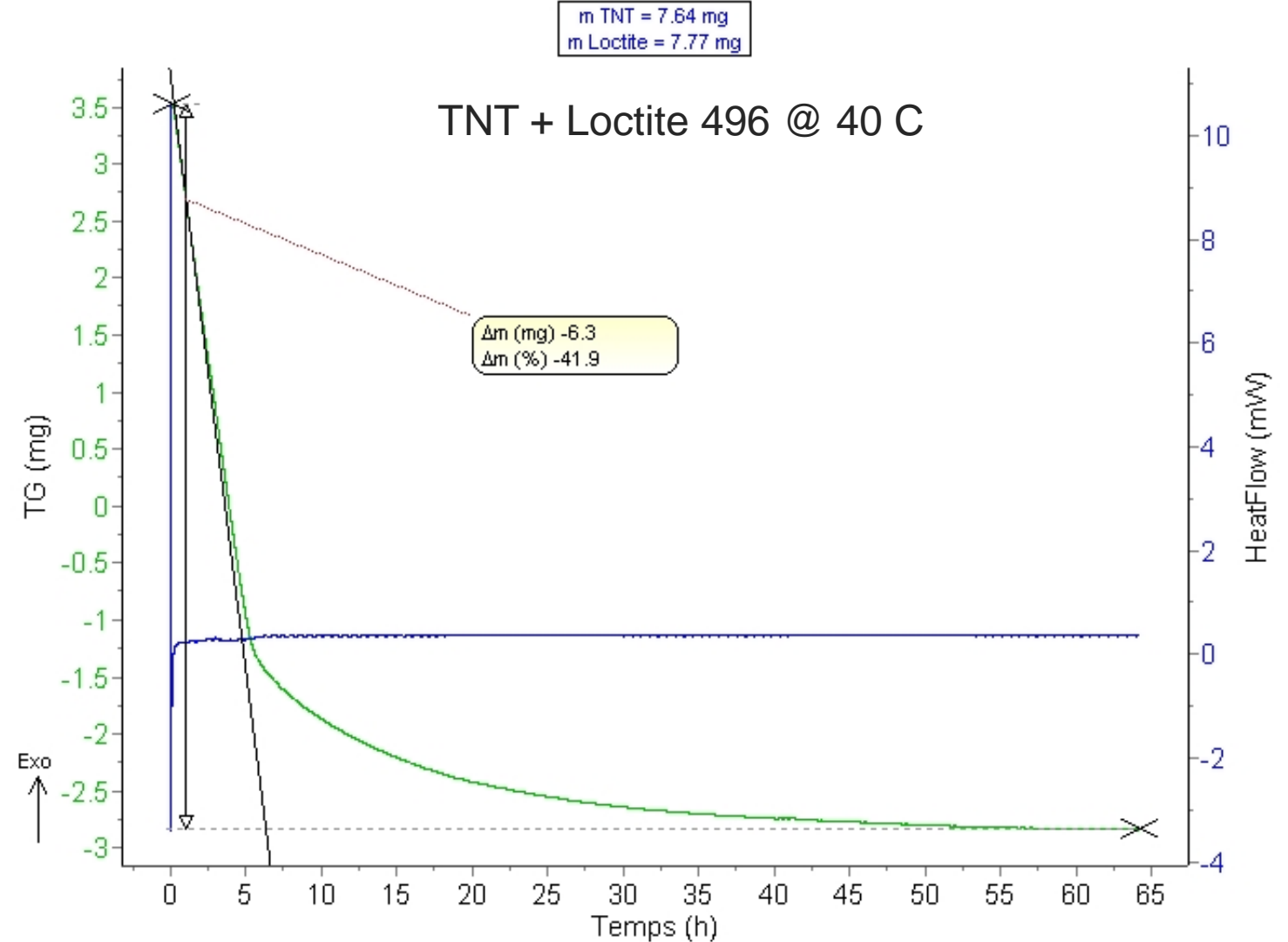
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Contact test

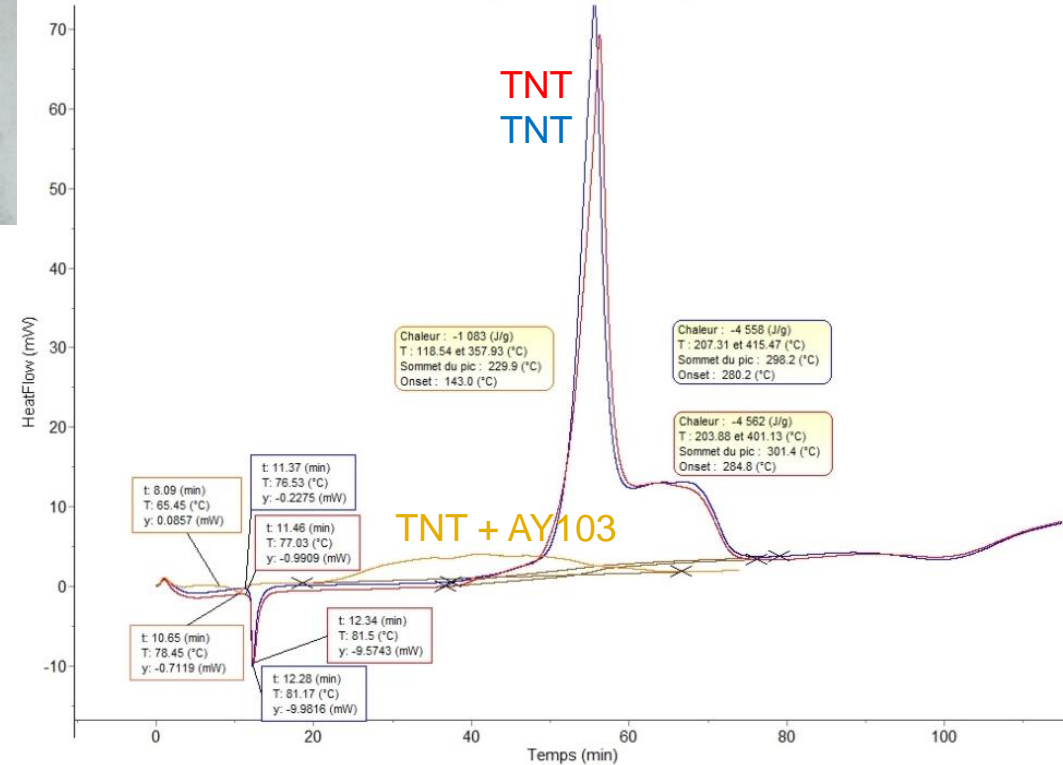
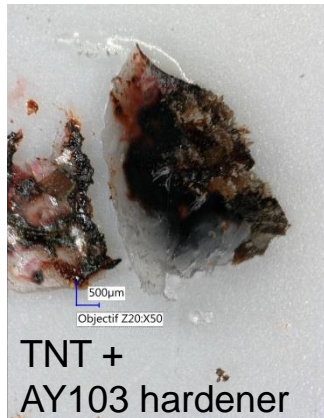
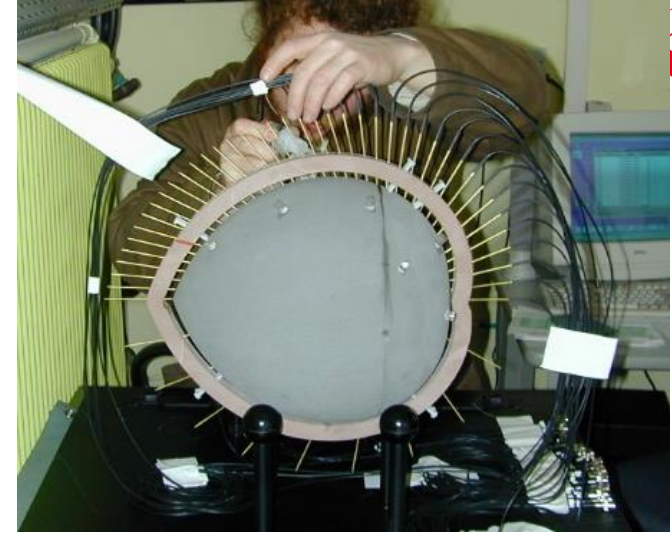
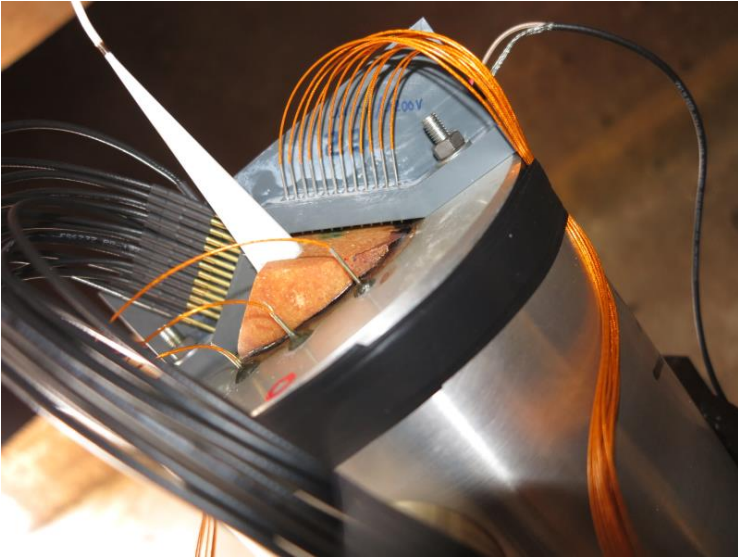
- If exothermic reaction
 - HE + glue TG analysis
 - Same isotherm : reaction of the HE ?



Procedure #2 : Color change when gluing

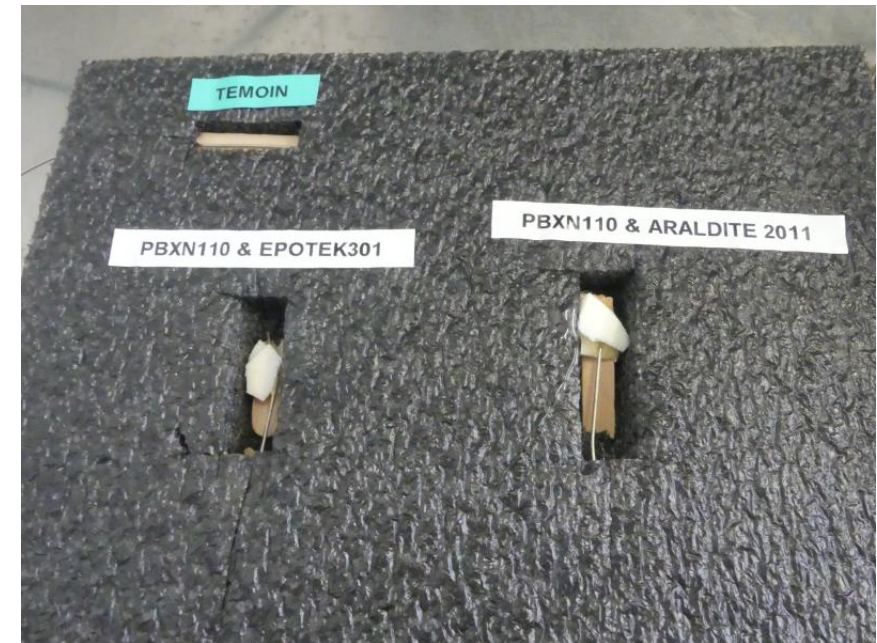
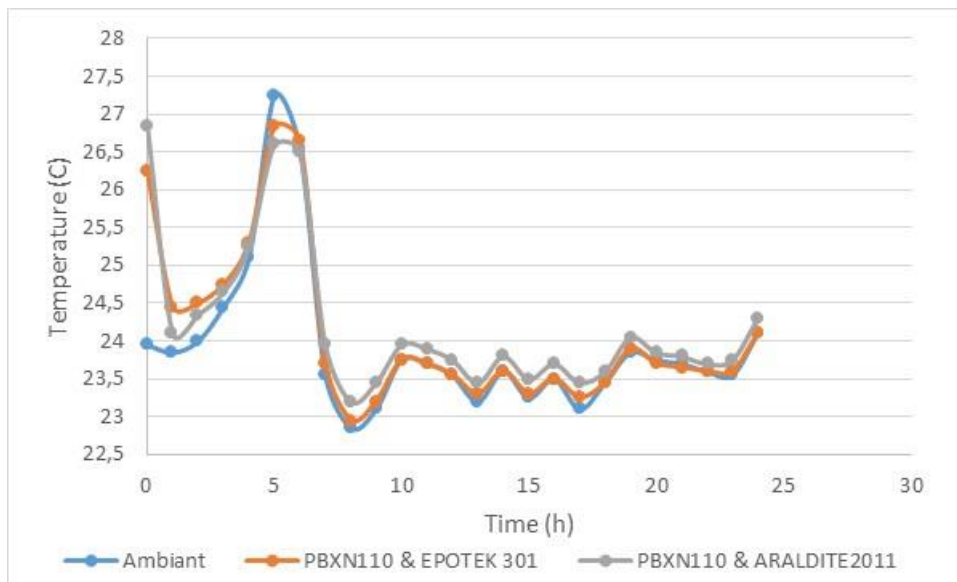
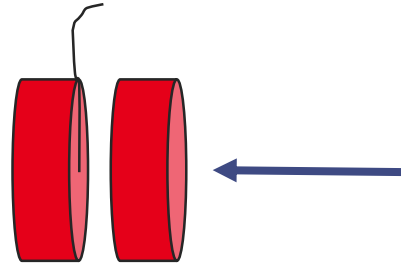
Surface change

- Is it relevant ?



Procedure #2 : gluing test

- Heat increase risk during gluing
- Is there a risk during the polymerization of the glue ?



Procedure #2 : acceptance criteria

■ DSC

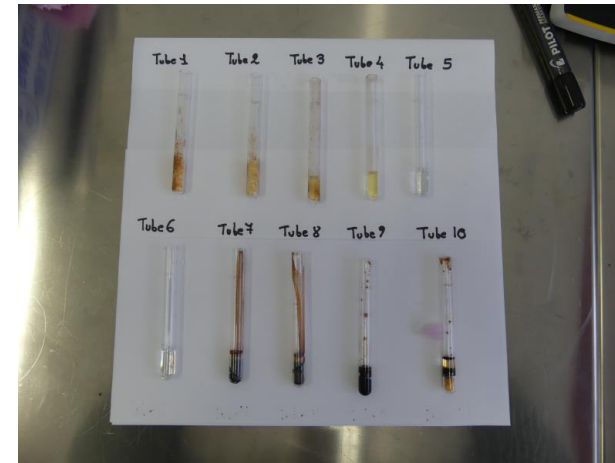
- $\Delta T_{\text{dec}} < 5 \text{ C}$: favorable
- $5 < \Delta T_{\text{dec}} < 20 \text{ C}$: restricted ([4;20] for STANAG)
- $\Delta T_{\text{dec}} > 20 \text{ C}$: unfavorable

■ Vaccum test

- $R = v_c - 2 (av_a + bv_b)$
- $R < 1,0 \text{ g/cm}^3$: favorable
- $R < 1,0 \text{ g/cm}^3$: unfavorable at this T, other test possible at a lower temperature → limitation of use
- $R > 2,0 \text{ g/cm}^3$: unfavorable

■ TG

- $\Delta m < 4 \%$: favorable
- $4 < \Delta m < 20 \%$: restricted
- $\Delta m > 20 \%$: incompatible



Procedure #3 : mix scale up

2 g scale

- Shape modification ?
- Temperature change ?
- Impact sensitivity (NF T70-500)
- Friction sensitivity (NF T70-503)

100 g scale

- Fabrication process adapted to the scale
- Shape modification ?
- Temperature change ?
- Impact and friction test
 - Test at the lowest non reaction level
 - To be confirmed on 5 to 10 tests

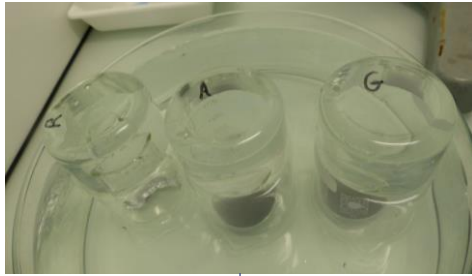
Process scale

- Shape modification ?
- Temperature change ?

Machining under water

Al containing HE

- Al + H₂O reaction
 - Very low



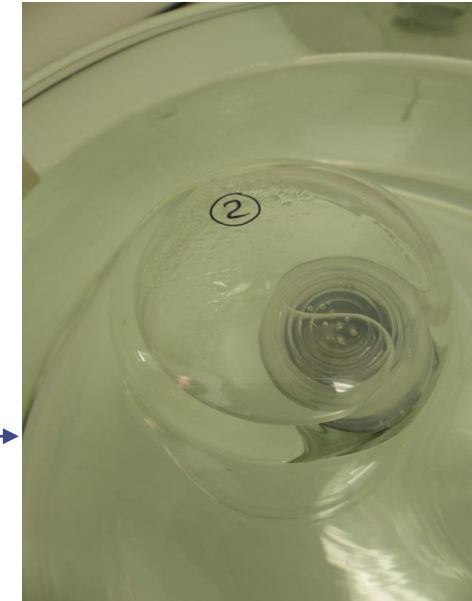
2 days



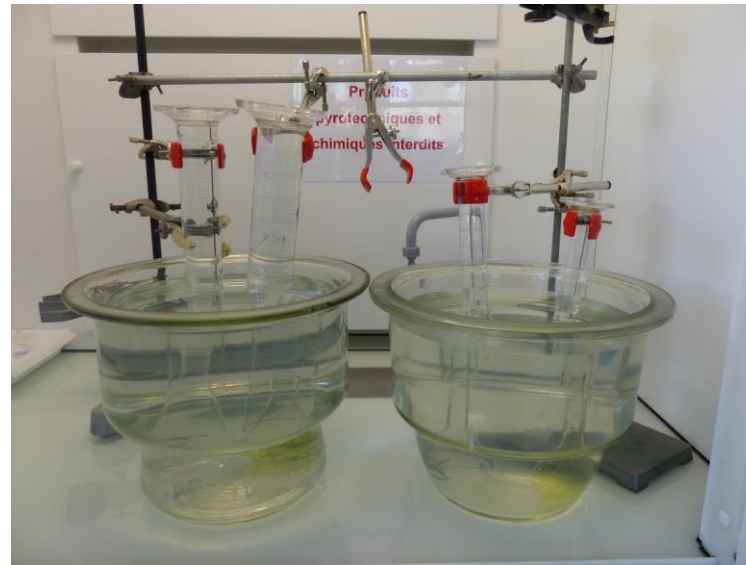
- UN + Al reaction
 - Possible



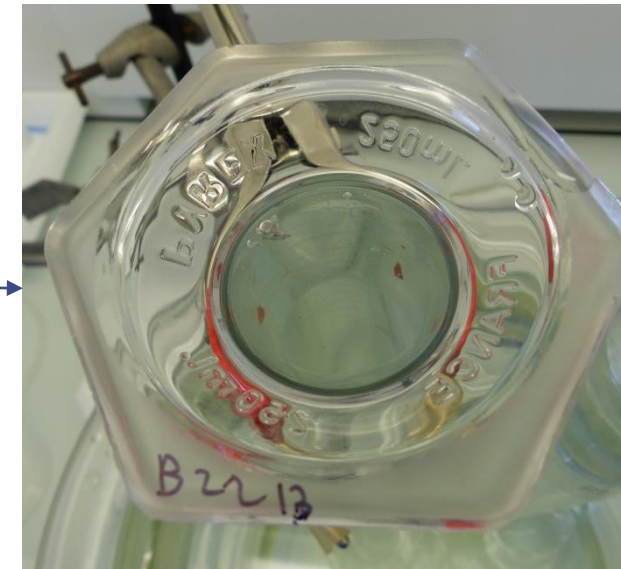
2 days



- Test of compatibility with water
 - 100-400 mg
 - H₂ releasable : ~ 100 ml
 - Duration : 3 days
 - Performed before machining



3 days



Conclusion

Proposed procedures for

- Products in contact with HE
- Products mixed with HE

- New tests proposed for gluing and machining under water

- Need for exchange with other defense companies to share on the tests and results