

STUDY OF NITROCELLULOSE SELF-IGNITION TEMPERATURE

Petra Loudová, Jiří Schejbal, Jaroslav Štěpán, Oldřich Večerek

SYNTHESIA, a. s.
Czech Republic



Synthesis, a.s.

We are the major European producer of qualified chemistry located in the “Hearth of Europe” in Pardubice, Czechia



Synthesis & Nitrocellulose

- Nitrocellulose production since 1923
- Current portfolio of “NC related products”:
 - ✓ Energetic nitrocellulose (propellants, dynamites)
 - ✓ Industrial nitrocellulose
 - ✓ Nitric acid, sulphuric acid, mixed acids
 - ✓ Diethyl ether
 - ✓ Propellant stabilizers



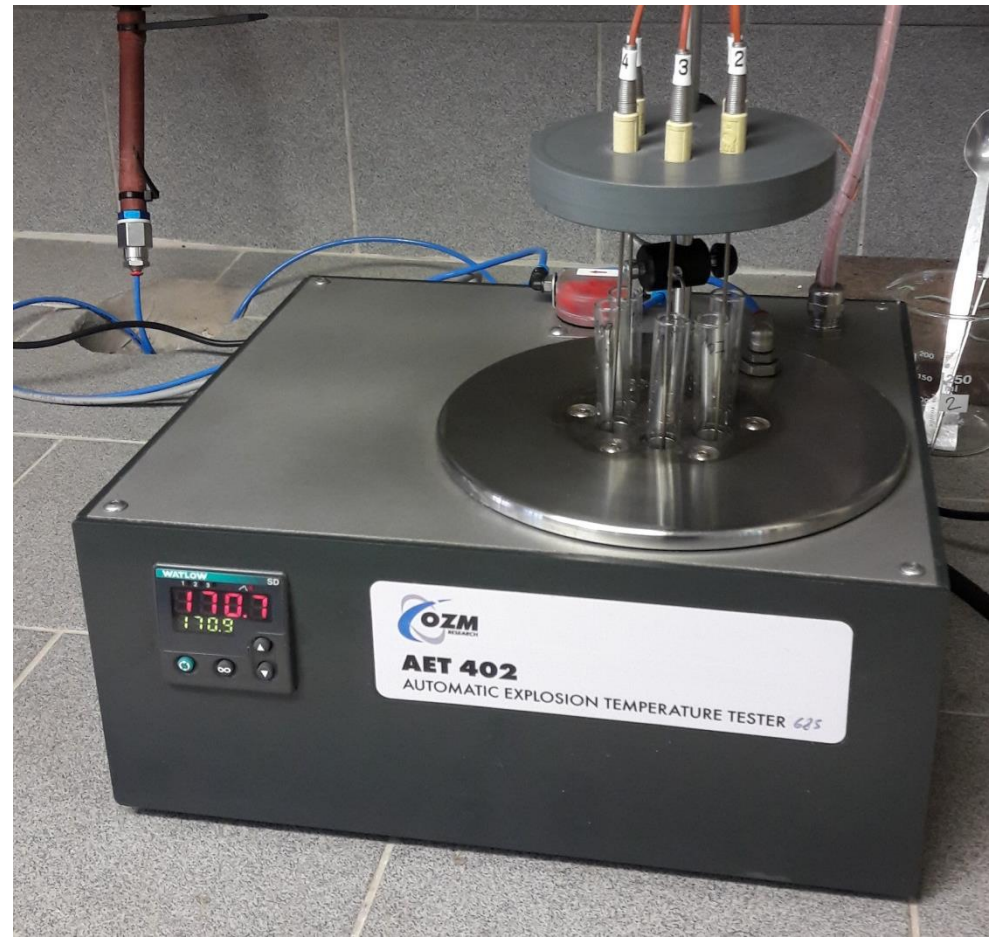
NC Self-Ignition Temperature

- Measuring method is described in STANAG 4491 [1] or in ADR [2]
- Principle: NC sample is heated from 100°C, at a rate of 5°C/min, until self-ignition occurs. The corresponding temperature is recorded as NC deflagration (self-ignition) temperature
- Typical results of properly stabilized NC are above 180°C
- For particular NC type, the self-ignition typically occurs within $\pm 1^\circ\text{C}$, even for different stability results. This is not sufficient for more detailed study
- Slower temperature gradients or even constant temperature levels were used for more detailed study



Testing Instrument

- Automatic Explosion Temperature Tester with five measuring cells and one reference cell was used
- Automatic temperature control with possibility to select
 - Constant temperature
 - Temp. gradient
- Recording of time and temperature data



Influence of Temperature Gradient

Temperature Gradient [°C / min]	Initial Temperature [°C]	NC Self-Ignition Temperature [°C]	Time to Self-Ignition [s]
5 *	100	184 – 187	1000 - 1050
1	165	177 – 181	700 - 1000
0,5	165	175 – 177	1200 - 1500
0,2	165	171 – 173	2000 - 2500

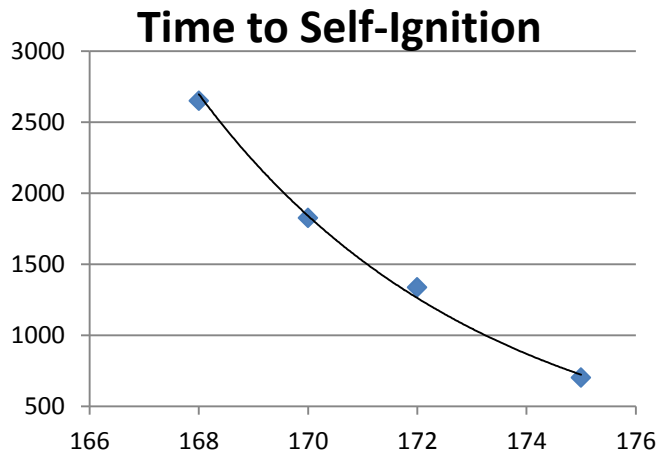
* Standard method according to STANAG or ADR

- Smaller temperature gradient results in wider time interval
- Temperature gradient has no significant influence to the width of temperature interval



Temperature Influence - Isotherm

Constant temperature [°C]	Average time to self-ignition [s]	Time to self-ignition [s]
168	2649	2500 – 3000
170	1825	1400 – 2400
172	1336	1100 – 1500
175	701	500 – 900



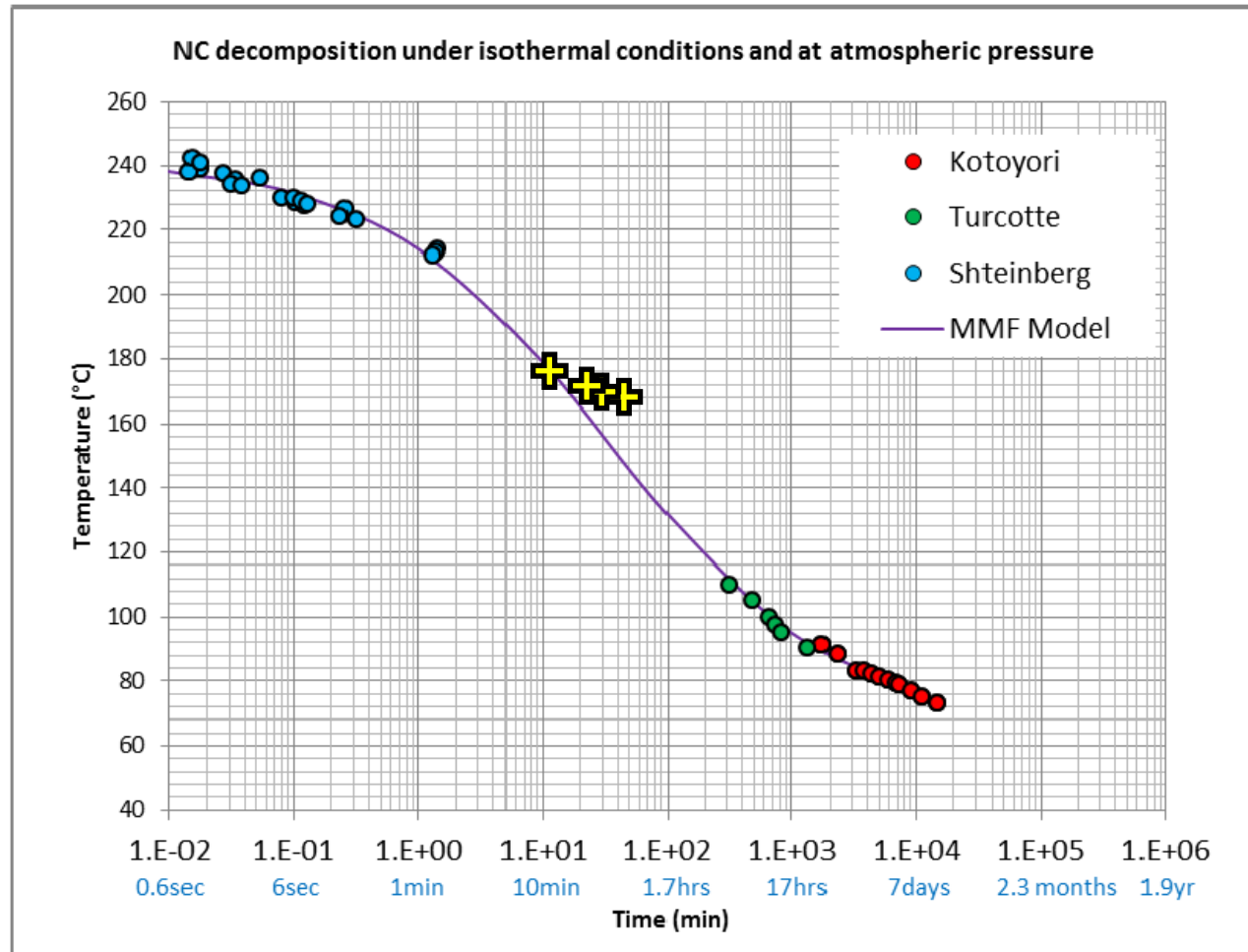
Time to self-ignition strongly depends on selected constant temperature.

170°C was chosen for future isothermal experiments

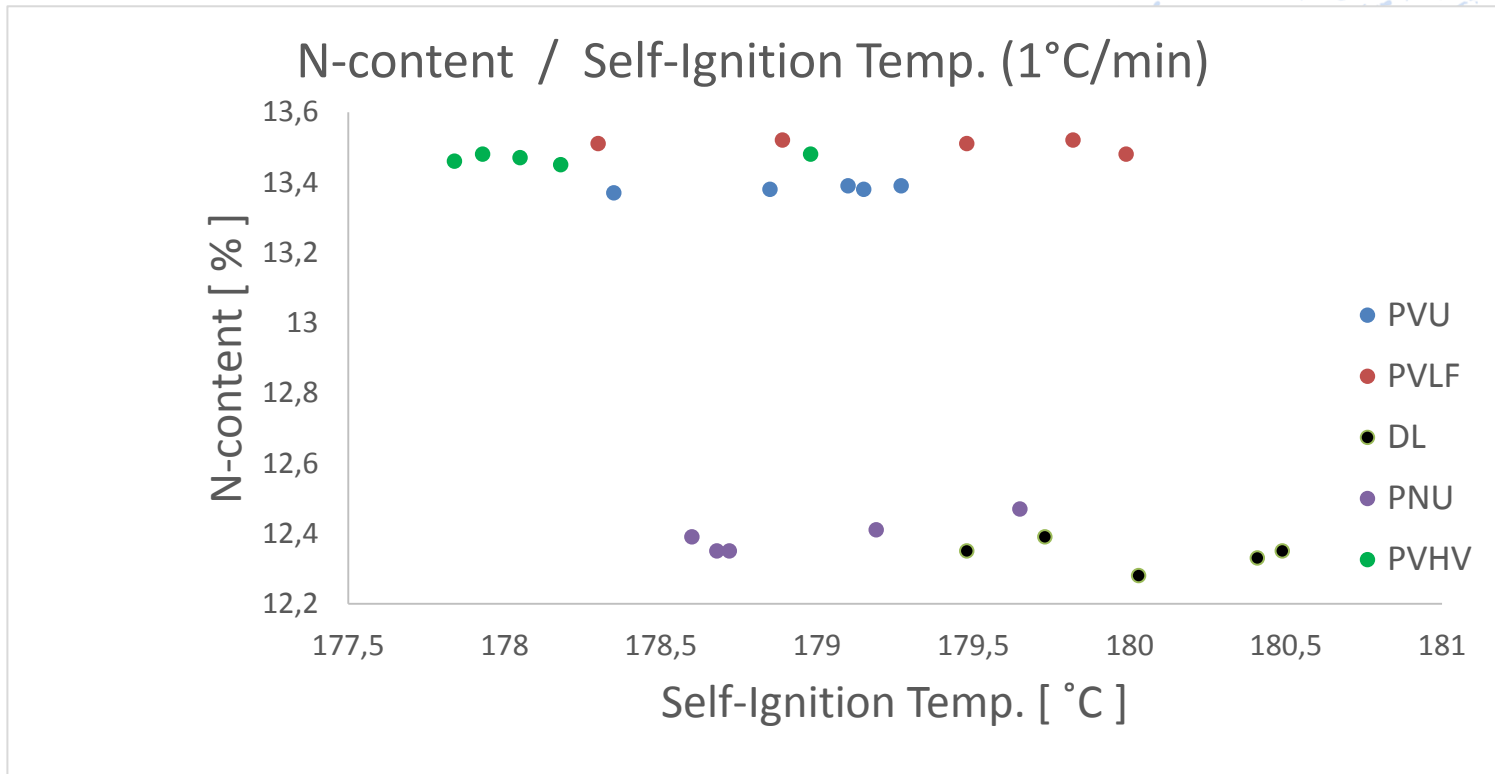


NC Decomposition Time

- + Our isothermal experiments shows good conformity with results from work [3]



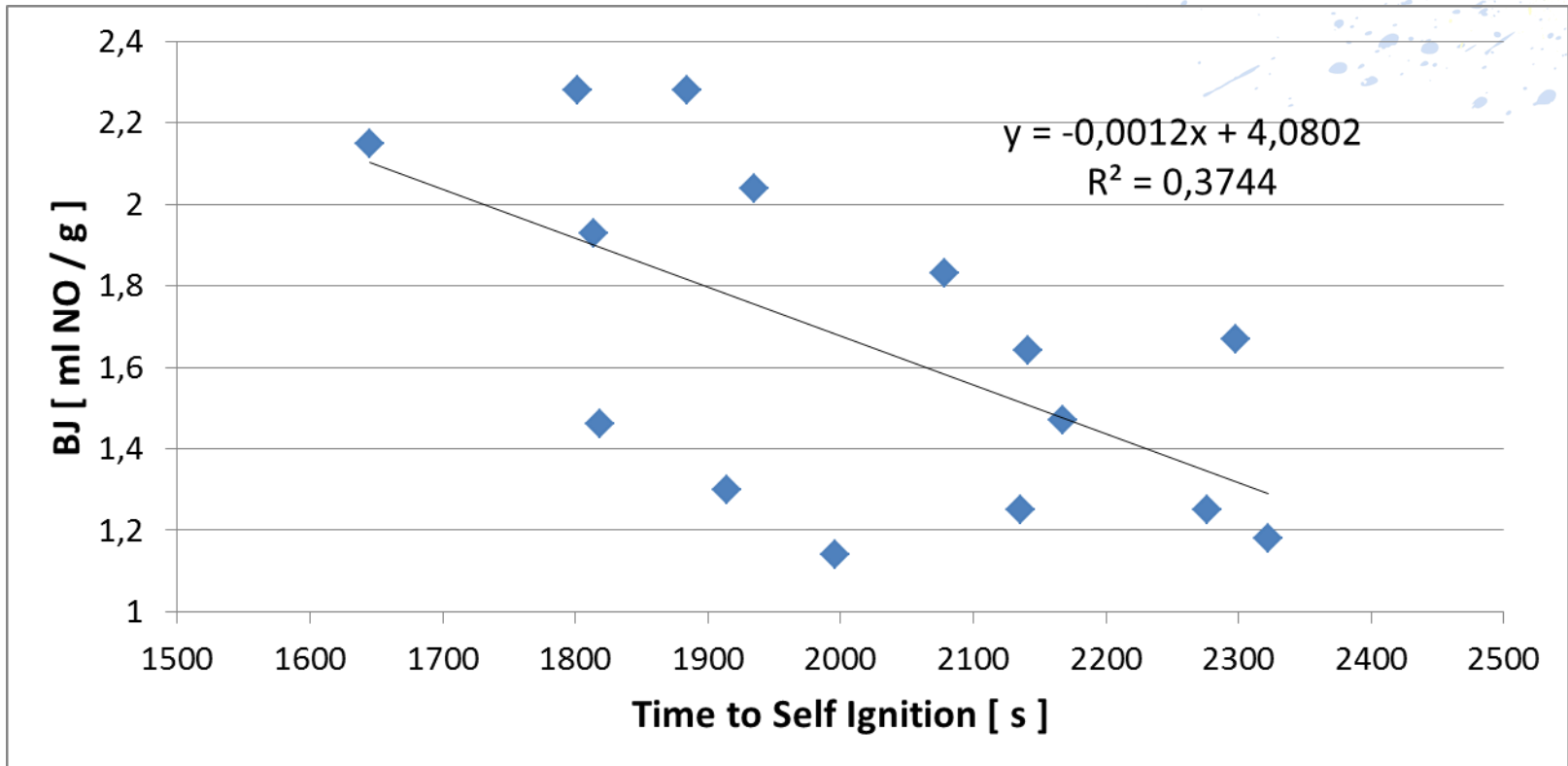
Nitrogen Content Influence



Different results for various NC types, even within the same nitrogen content level.



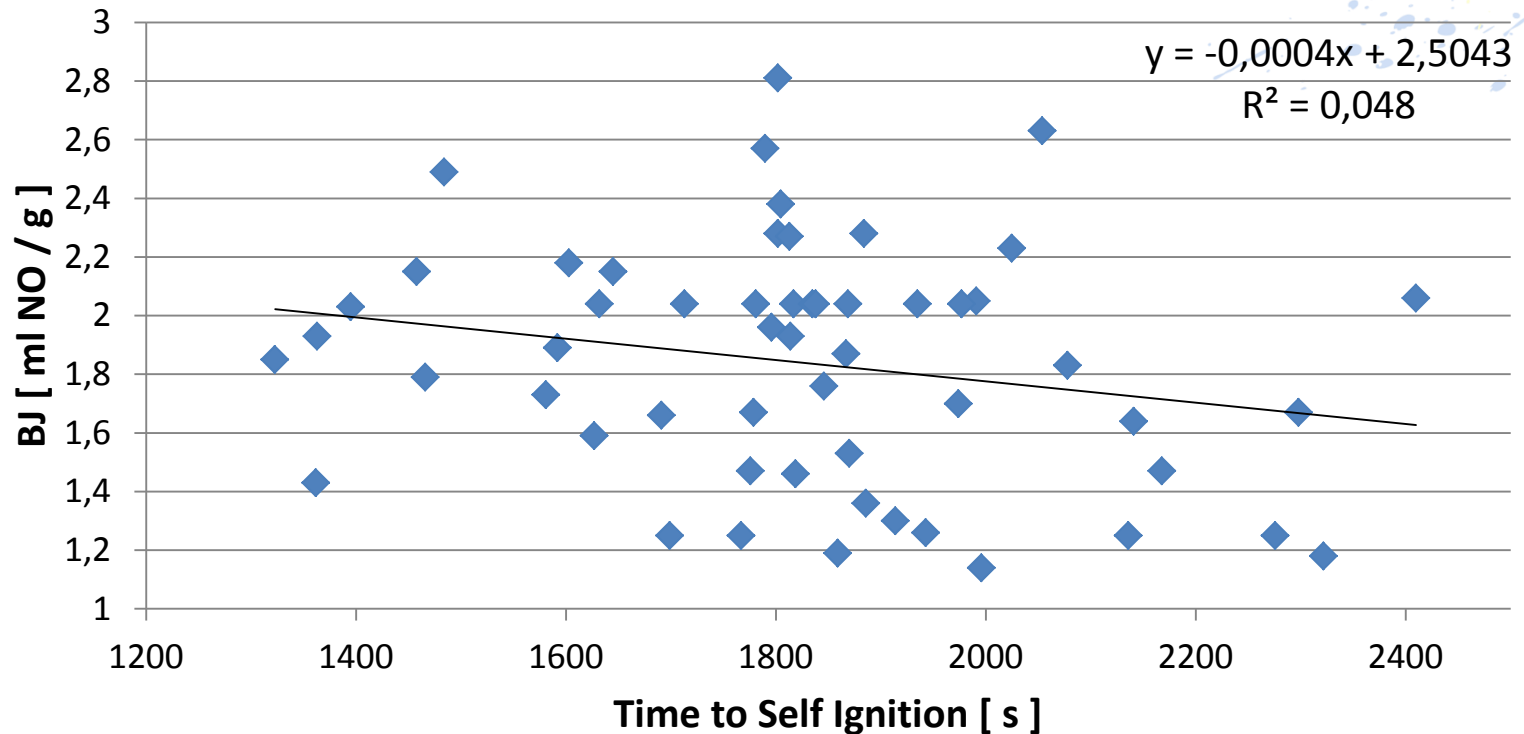
Bergmann-Junk Test Correlation



Promising results with one NC type, sample taken from the same process step of NC stabilization



Bergmann-Junk Test Correlation



Not satisfactory results for combination of NC types and different sample origin (from different stabilization process steps).



Conclusions

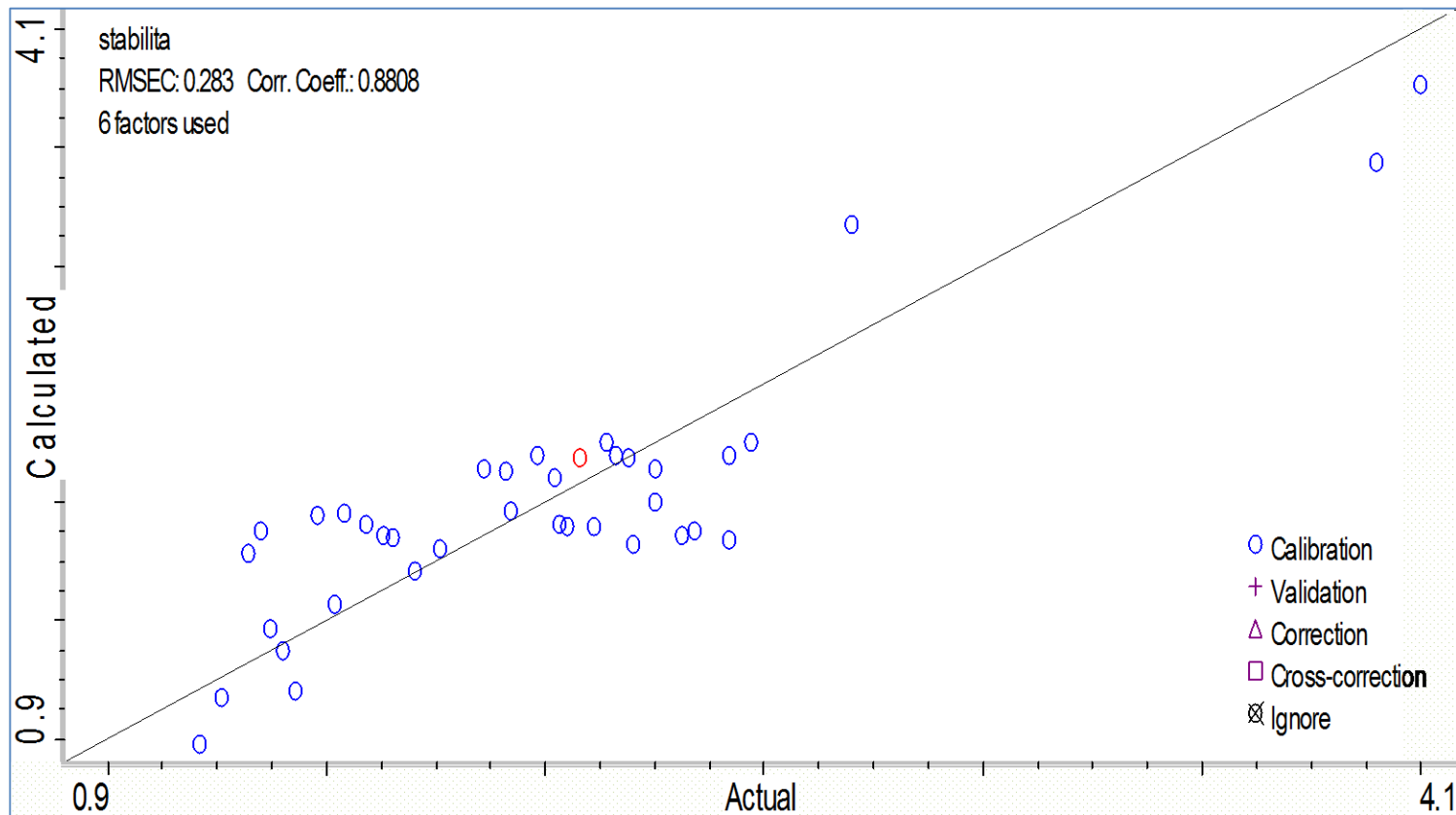
- Influence of nitrogen content of used NC sample
- High sensitivity to the temperature during experiment: small temperature deviations from selected constant temperature or temperature gradient may lead to large deviations in results
- Influence of other NC parameters, possibly physical properties of NC or raw material, may change the heat transfer and obtained results

The method does not represent a suitable alternative to commonly used Bergmann – Junk stability test



Steps Forward

Promising results were achieved using dispersive Raman spectroscopy, but only on a small group of samples.



References

- [1] NATO – STANAG 4491, ed. 2. Explosives, thermal sensitiveness and explosiveness tests. North Atlantic Treaty Organization, 2015
- [2] European Agreement concerning the International Carriage of Dangerous Goods by Road (ADR 2017), United Nations, 2016.
- [3] Mario Paquet: Review of basic concepts related to the thermal decomposition of nitrocellulose. NC Symposium Spiez, 2012.



Thank you for your attention!

Synthesia, a.s.

Semtín 103, 530 02 Pardubice

Česká republika

www.synthesia.eu

 **Synthesia**
Chemistry for the future

