

# DETERMINATION OF NITROCELLULOSE PROPERTIES BY NIR SPECTROSCOPY: NITROGEN CONTENT, VISCOSITY, SOLUBILITY, TOTAL VOLATILE CONTENT AND RESIDUAL WATER

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# SYNTHESIA, Czech Republic

**Synthesia, a.s.** is the major European producer of qualified chemistry

**Synthesia** consists of four Strategic Business Units:

- SBU **Energetics** (steam, electricity, water, compressed air, gases...)
- SBU **Pigments and Dyes** (organic pigments, dyes, optical brighteners)
- SBU **Organic Chemistry** (pesticides, intermediates, custom synthesis)
  - ✓ Propellant stabilizers
- SBU **Nitrocellulose**
  - ✓ Nitric acid, sulphuric acid and their salts
  - ✓ Nitrating acid mixtures
  - ✓ Diethyl ether
  - ✓ Nitrocellulose (Energetic, Dynamite, Industrial grades)
  - ✓ Oxidized cellulose (hemostatic substance)



# NC analyses by NIR in Synthesia

## **2012** First instrument 'InfraLab'

- Measurement of NC water content
- High sensitivity to sample preparation (not pressed samples)
- Is used for fast indicative measurement during dewatering process

## **2014** Research of NIR possibilities

- In cooperation with equipment provider
- First calibrations and results for Nitrogen, Viscosity and Water content

## **2015** Instalment of our NIR spectrophotometer

- Extensive calibrations of N, Viscosity, E:A solubility, Content of Ethanol, Propanol, Butanol and Water

## **2016** Transfer of routine internal NC analyses to NIR

- Ongoing calibration and validation process



# Sample preparation

- Slurry samples are centrifuged to have roughly 30% of water. No other samples adjustment (not dried)
- Samples are filled to the compression cuvette for solid samples, closed by a spring loaded lid and measured by NIR



# Nitrogen Content

- More than 6000 standards used for calibration and analyzed by both NIR and conventional FS titration methods
- One calibration model can cover all NC grades and types (including dynamite and industrial NC grades)

## Accuracy

- Max.  $\pm 0,03\%$  abs. required by STANAG 4178, Ed. 2, for accepting alternate method
- $\pm 0,010 - 0,025\%$  abs. achieved for different NC types

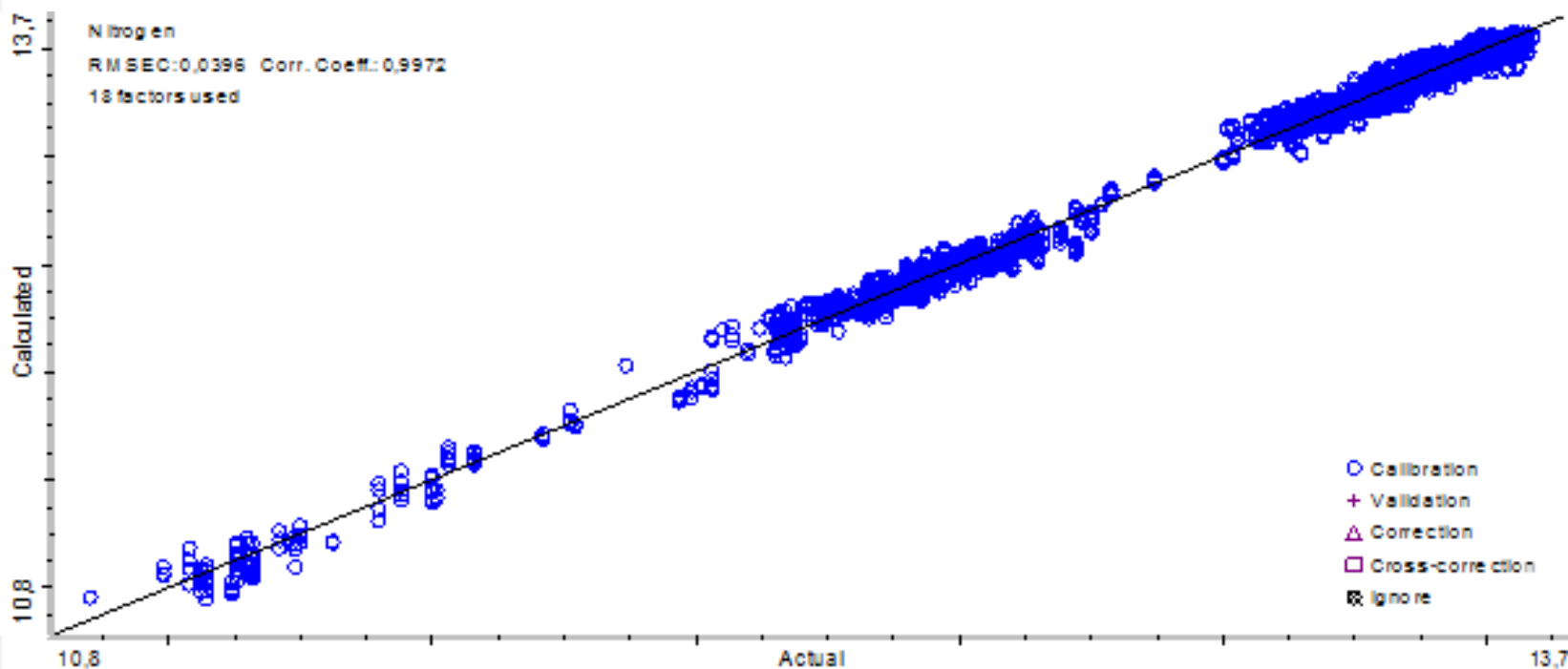
## Repeatability

- Achieved standard deviation  $s_r$ ;  $n=12$ :  $0,008\%$  abs. is in range “Excellent” according to STANAG 4178, Ed. 2





# Nitrogen Content – Calibration Curve



# Viscosity

- Divided to several calibration models according to
  - ✓ Concentration, solvent (acetone 1%, 2%, butyl acetate 2%, 5%)
  - ✓ Expected viscosity (high, medium, low)
- 150 – 7000 standards analyzed by both NIR and conventional method in each category
- Influence of sample origin – NC process step, homogeneity of sample
- Ongoing calibration and validation

## Accuracy

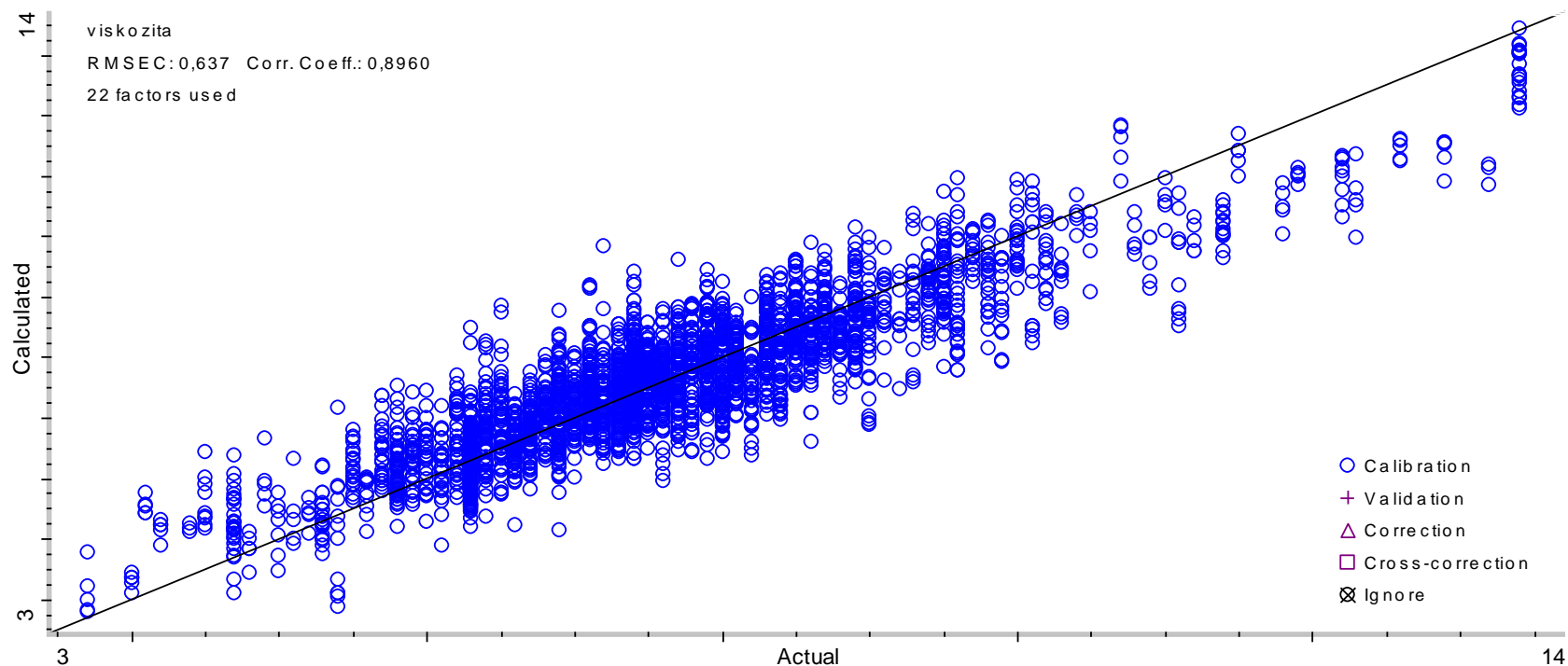
- $\pm 2 - 20\%$  relative for different NC types, solvents and concentration

## Repeatability

- Achieved standard deviation  $s_r$ ;  $n=12$ : around 2,5% relative



# Viscosity – Calibration Curve Example





# Ether Alcohol Solubility

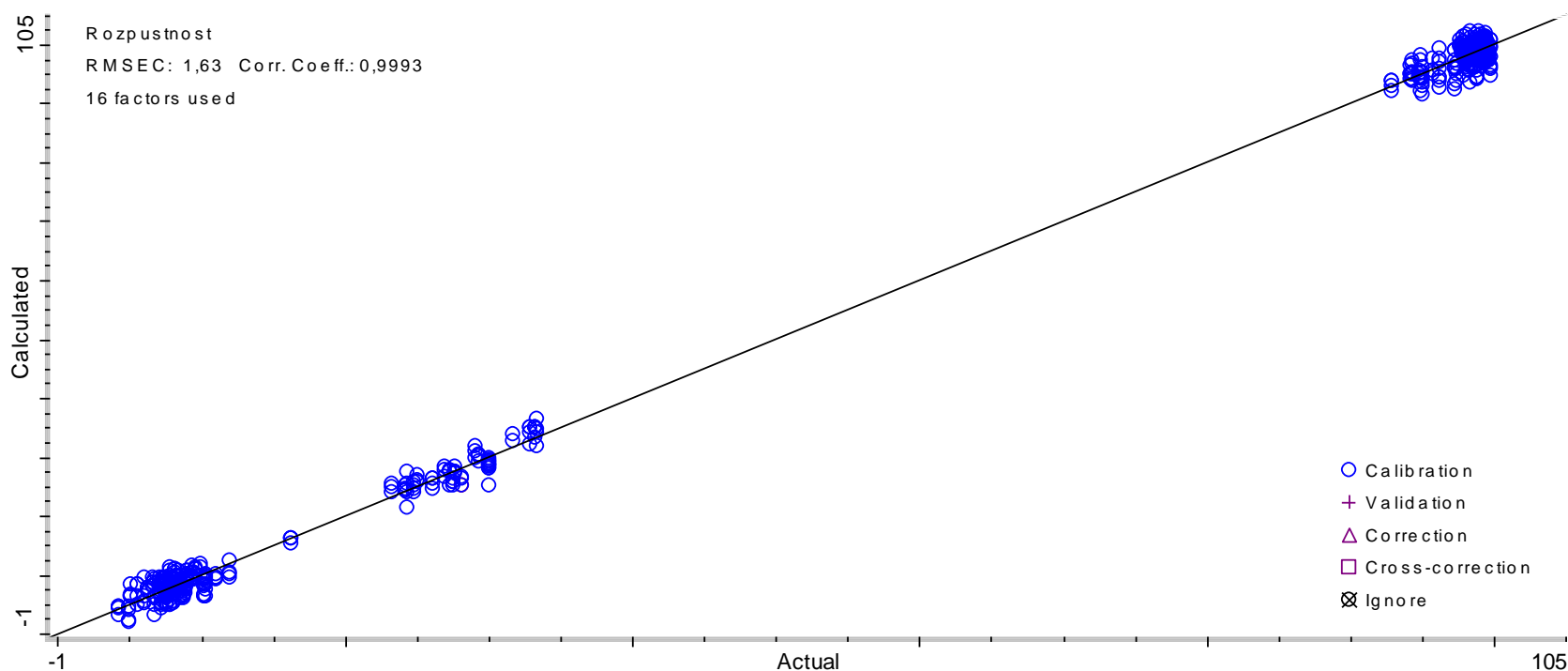
- Initial positive results obtained by analyzing over 500 standards by both NIR and conventional methods
- Currently one calibration model covering all NC grades and types
- Future samples and calibrations may lead to splitting the model according to NC grades

## Accuracy and Repeatability

- Not yet established, only initial testing performed



# Solubility – Calibration Curve



# Alcohol and / or Water Content

- Divided to several calibration models according to wetting agents: water, ethanol, isopropanol, n-butanol
- 150 – 7000 standards analyzed by both NIR and conventional methods (mass analyzer, Karl Fischer titration) in each category

## Accuracy

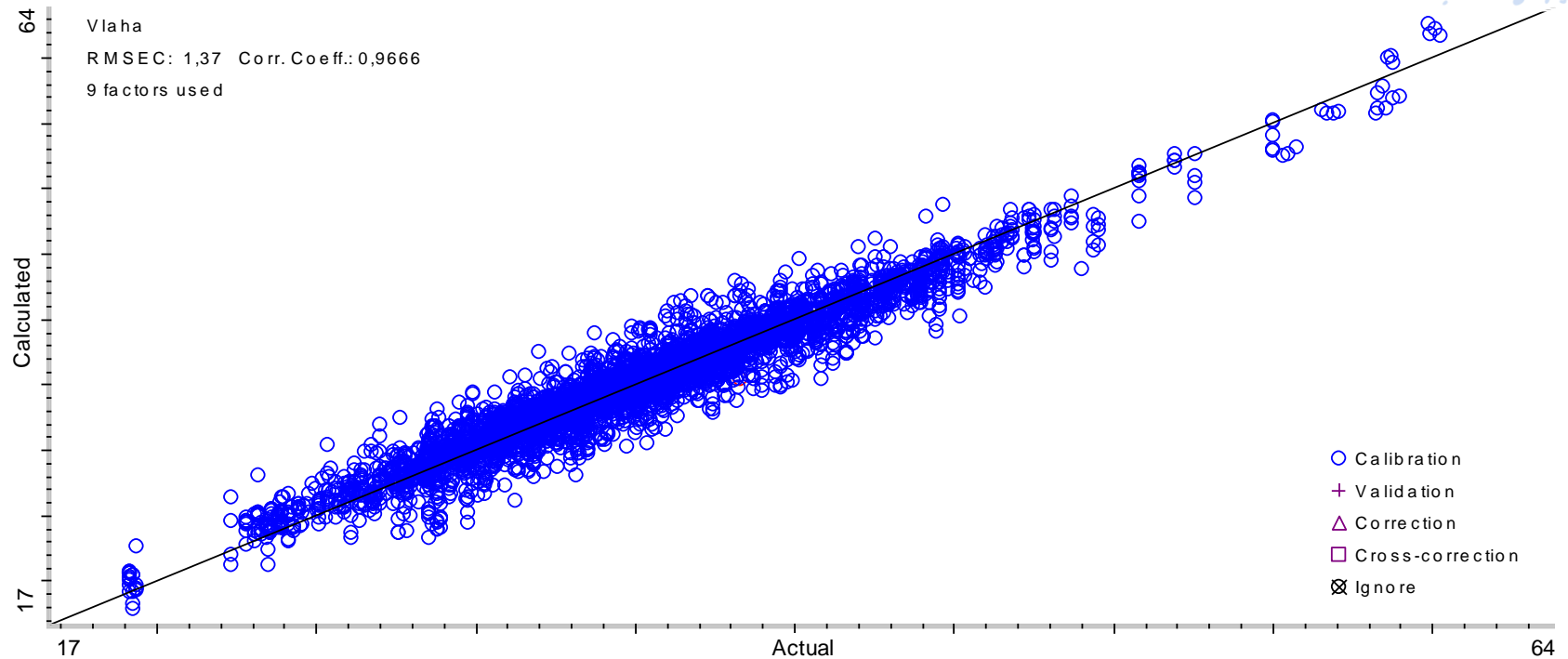
- $\pm 0,08\% - 0,45\%$  absolute for different NC types and wetting agents
- Max.  $\pm 0,5\%$  abs. required by STANAG 4178, Ed. 2

## Repeatability

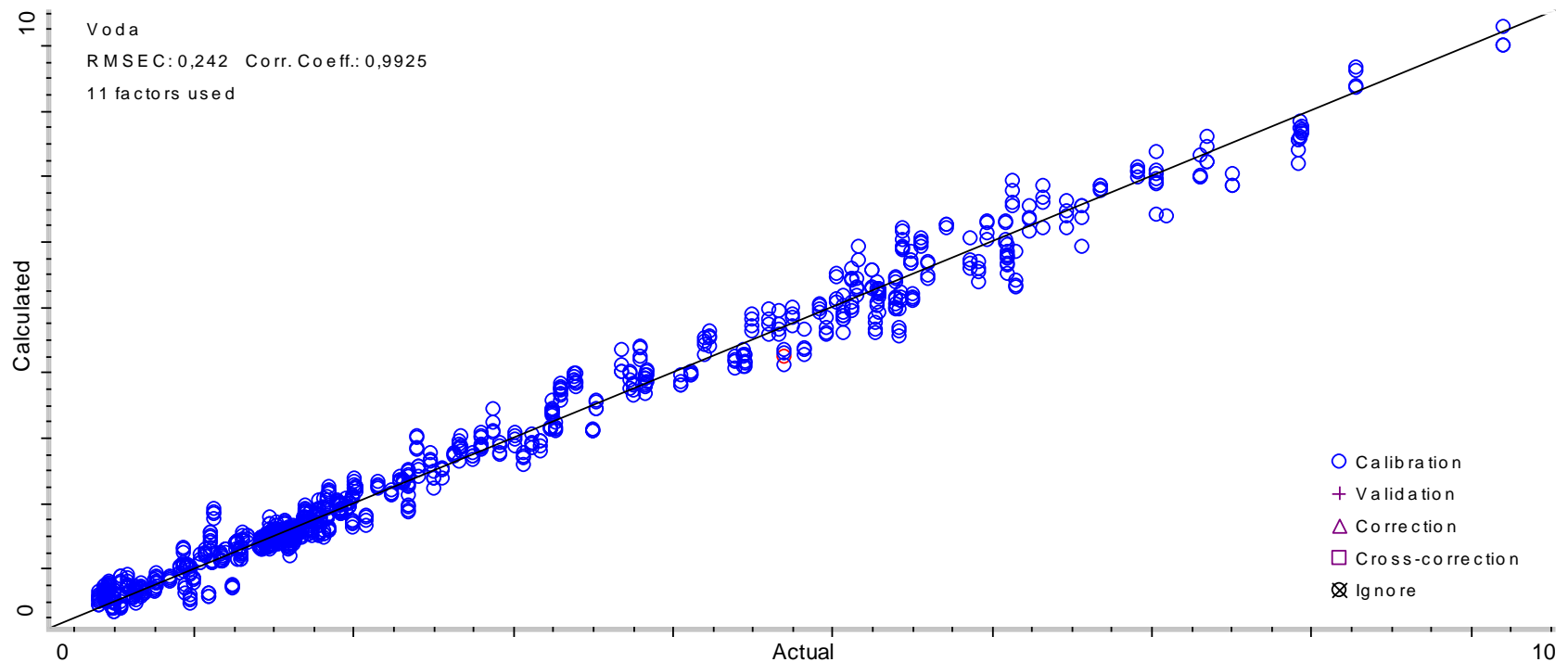
- Achieved standard deviations  $s_r$ ;  $n=12$ :  $0,1\% - 0,5\%$  abs. for different NC types and wetting agents
- Values  $>0,3\%$  are not acceptable according to STANAG 4178, Ed. 2, improvement in certain categories required



# Water Content – water wetted NC

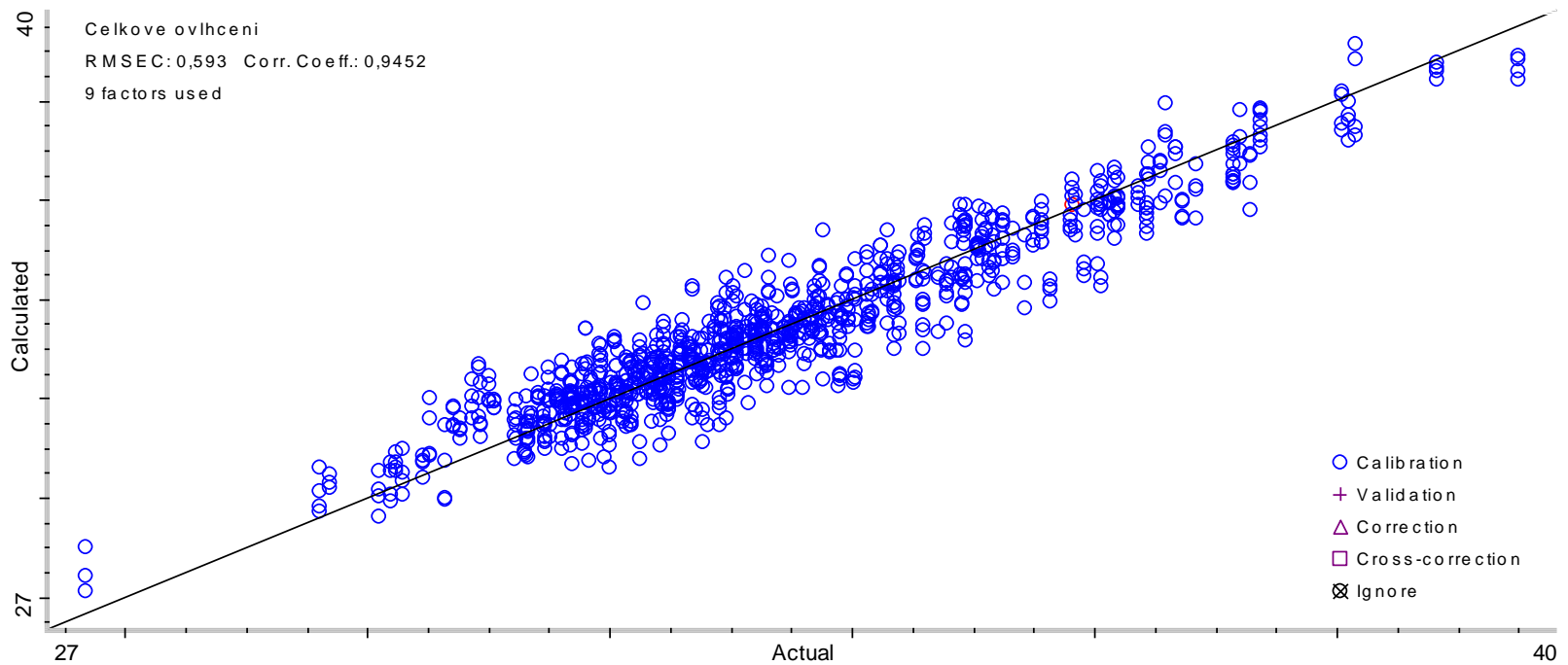


# Water Content – n-butanol wetted NC



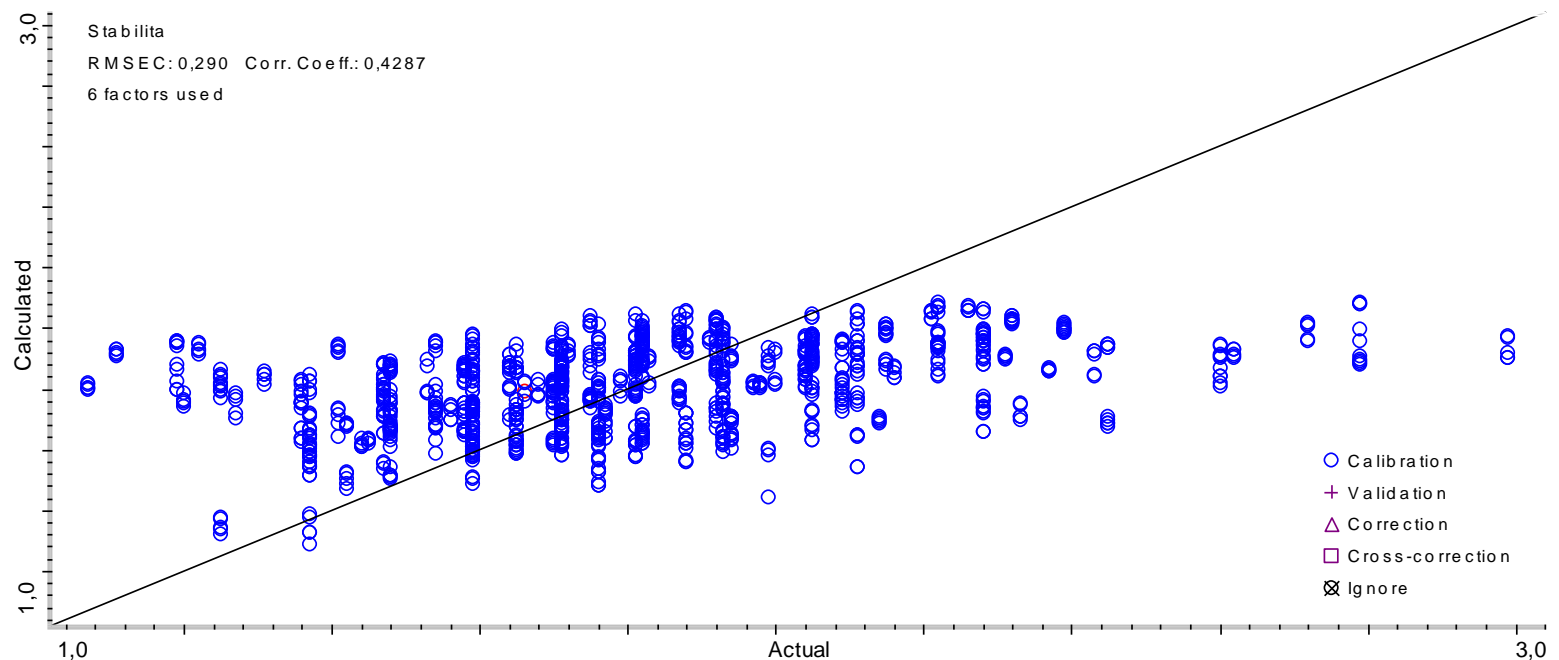


# Total Volatile Content – n-butanol wetted NC



# Stability Estimation

- Initial promising results – there may be some kind of correlation
- But finally no success in establishing reasonable calibration model



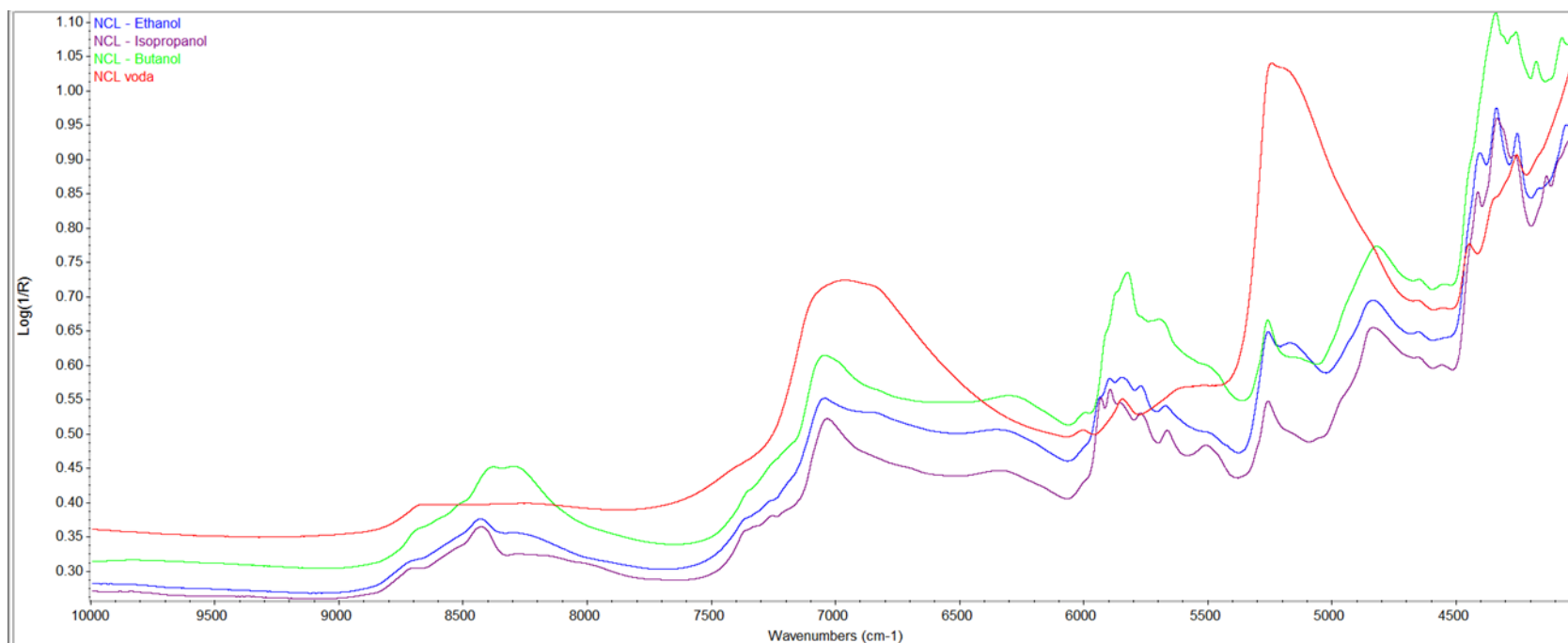
# Different NIR Spectra for Wetting Agents

Water

Isopropanol

n-butanol

Ethanol



# Other analyses / Future tasks

## Alcohol wetted nitrocellulose

- Currently total volatile content and residual water content calibrated
- Other analytics possible (nitrogen, viscosity), separate calibration will be needed for different alcohol types

## Diethyl Ether

- Possibility to measure on the same instrument with external probe
- Specific gravity, water content and ethanol content preliminary calibrated with positive result during testing in 2015

## Cellulose and other cellulose derivatives

- Planned testing of basic cellulose parameters
- Testing of oxidized cellulose parameters measurement



# Thank you for your attention!

**Synthesia, a.s.**

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