Real-time and accelerated aging tests and beyond

RSC – FST-Personal Care 2022 – 17 March, online

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1. Introduction & Motivation
2. Real-time testing using an analytical instrument
3. Physical acceleration using another analytical instrument
4. Summary & Outlook

1. Introduction of LUM GmbH

1994 | 1998 Company foundation | 1st Instrument sold to Pharma company

2022

51 People

Subsidiaries:

LUM Corp. (USA)

LUM China

LUM Japan

LUM France

Lab, at-line, inline instruments to characterize particles, suspensions, emulsions, coatings, composites, materials.

1. Motivation for (Accelerated) Stability Testing

- Market demands: short development cycles + products stable for a long time + *shelf life* of cosmetic, home and personal care products: months till years.
- But, the formulations are complex, have many ingredients, various influences and interactions (internal + external) during product lifetime.

Face Make up 6-12 months

(Liquid: liquid foundation, concealing liquids, liquid/cream blush, eyeshadow, etc.) http://www.buzzle.com/articles/makeup-expiration-dates-shelf-life-of-makeup.html (15.2.2012)

What to do? & How to do?

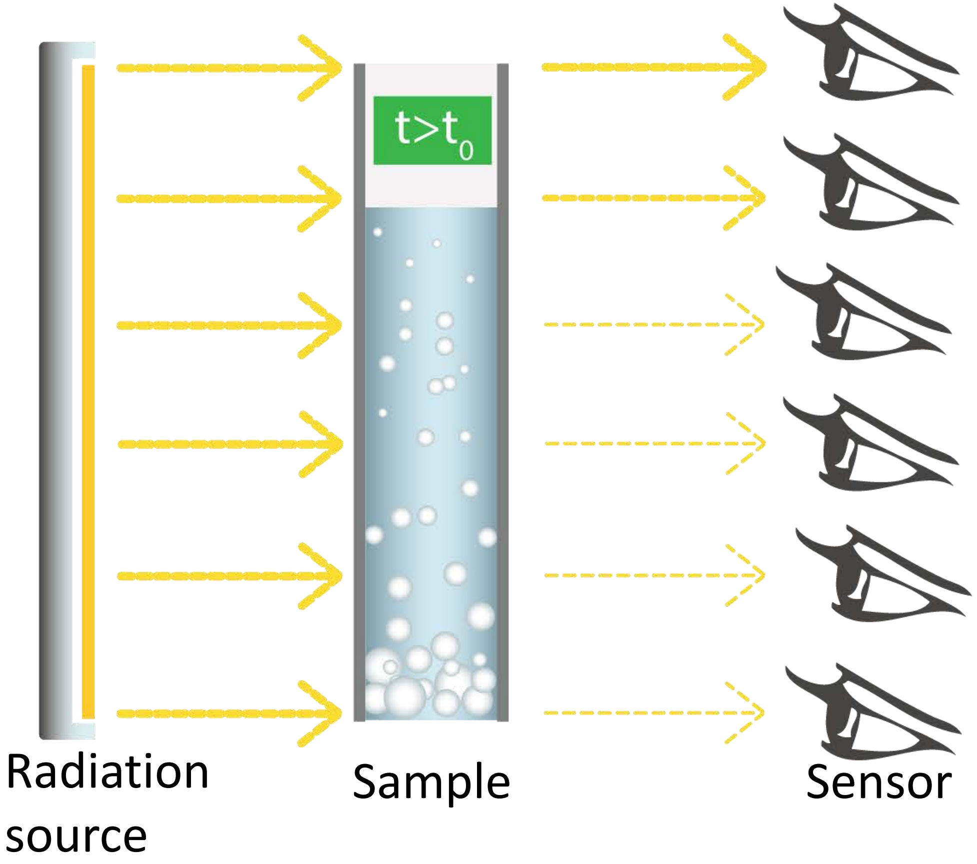
Instrumental methods for accelerated stability testing are suggested by ISO/TR 13097:2013 (Guidelines for the characterization of dispersion stability)

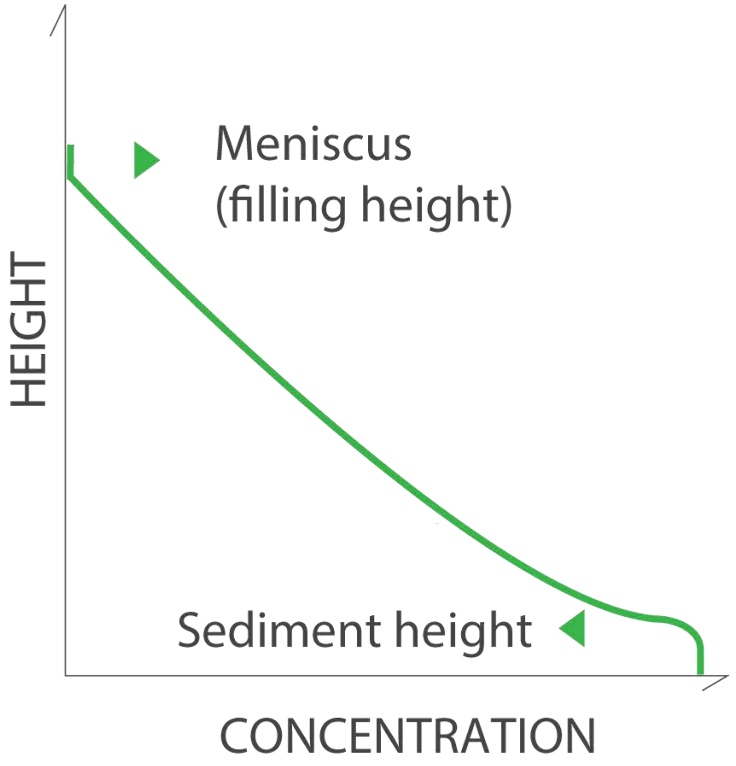
https://www.iso.org/standard/52802.html, 28.9.2020, 09:58

and ISO/TR 18811:2018 (Cosmetics -Guidelines on the stability testing of cosmetic products).

https://www.iso.org/standard/63465.html , 28.9.2020 09:59

2. STEP: Space and Time resolved Extinction Profiles

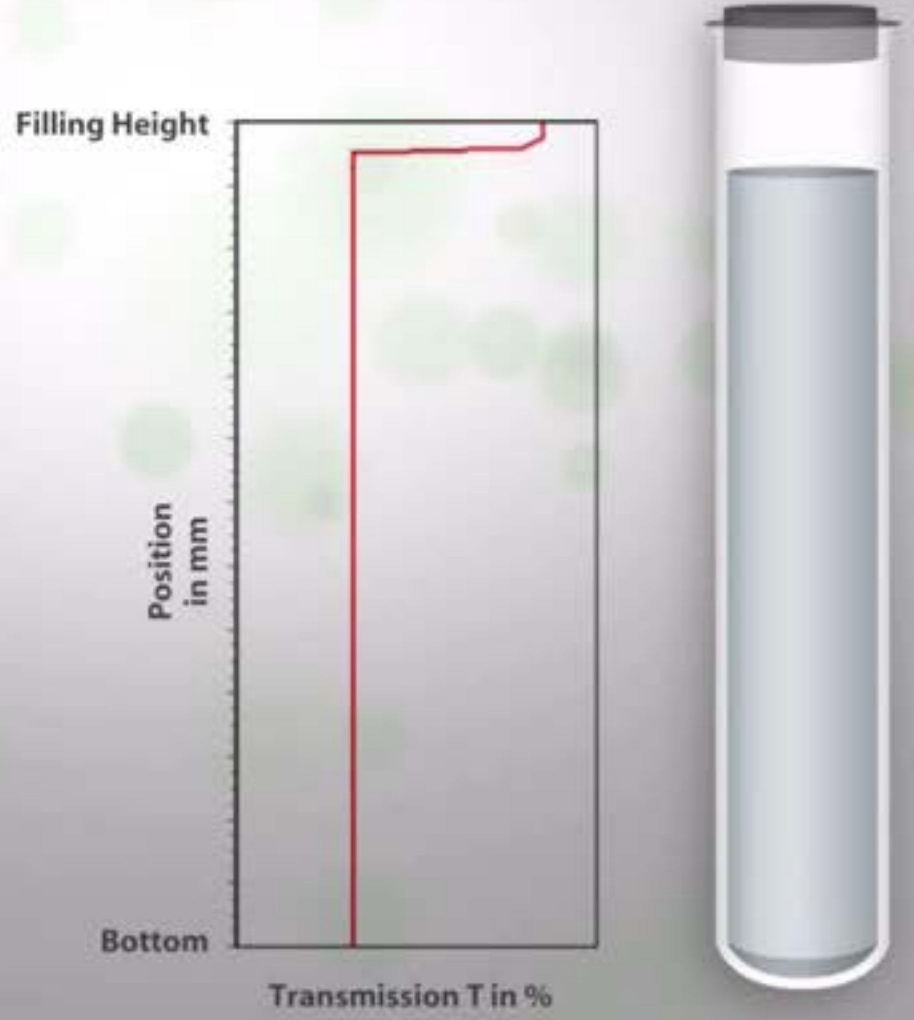




NIR light for almost all samples
Blue light for nanoparticles, transparent formulations
X-ray for high concentrated sediments and inorganic particles

Concentration at any position
at a selected time
or at any time at a selected position

Polydisperse Creaming



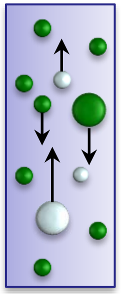
2.Basics of STEP-Technology® at gravity

Particle migration

Low particle concentration

Stokes law*:

$$v_{Stokes} = \frac{h}{t} = \frac{(\rho_p - \rho_l) \cdot x^2 \cdot g}{18 \cdot \eta} = K \cdot x^2 \cdot g$$



High particle concentration :

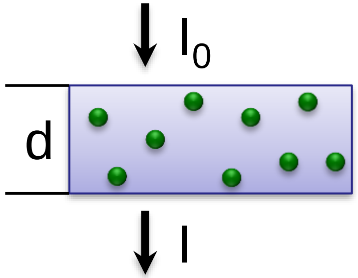
$$v = v_{Stokes} \cdot H(\phi)$$

v	particle velocity
h	settling distance
t	settling time
ρ	density
η	dynamic viscosity of continuous phase
x	particle size (diameter)
g	gravity
$H(\phi)$	hindrance function
ϕ	volume concentration

*Comprehensive characterization of nano- and microparticles by in-situ visualization of particle movement using advanced sedimentation techniques, D. Lerche, KONA Powder and Particle Journal 34 (2017), https://www.jstage.jst.go.jp/article/kona/advpub/0/advpub_2019012/_article 21.1.19

2. Basics of STEP-Technology®

Optical detection of particle concentration
Lambert-Beer law



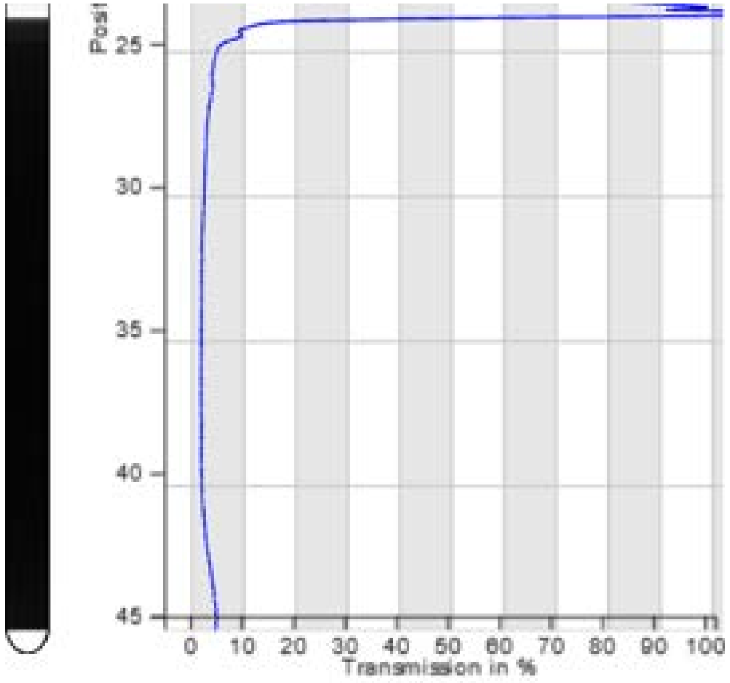
$$- \ln \frac{I}{I_O} = E = \varepsilon \cdot \phi \cdot d$$

$$\varepsilon = f\,(\lambda, x, n, \ldots)$$

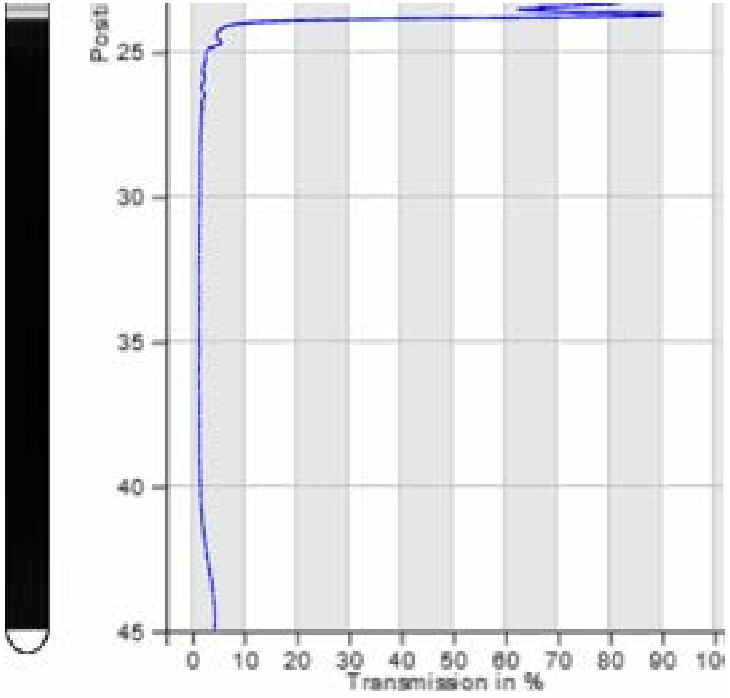
I_0 light factor| light intensity
ε extinction coefficient
d optical pathlenght (sample cell thickness)
λ wavelength
n refractive index

2. Real-time at gravity @constant temperature

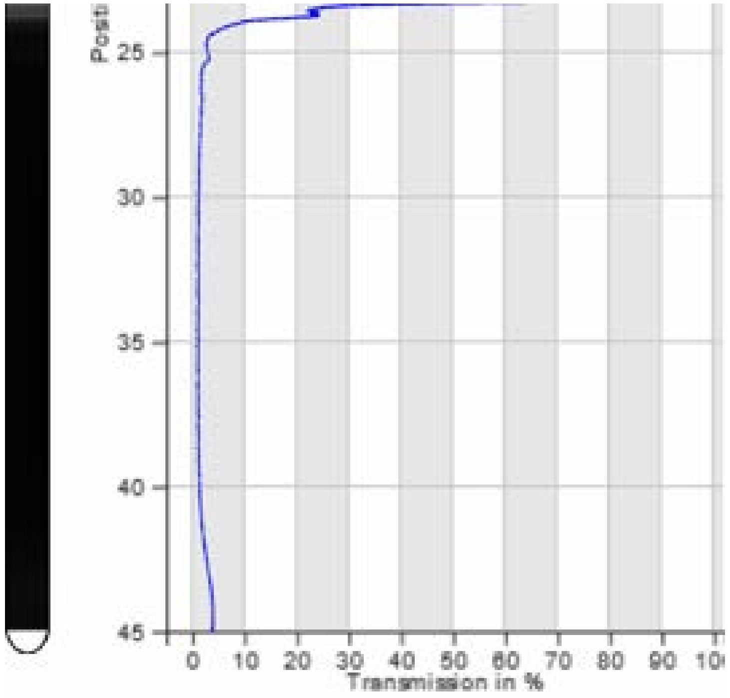
NIR 870 nm



Red 630 nm

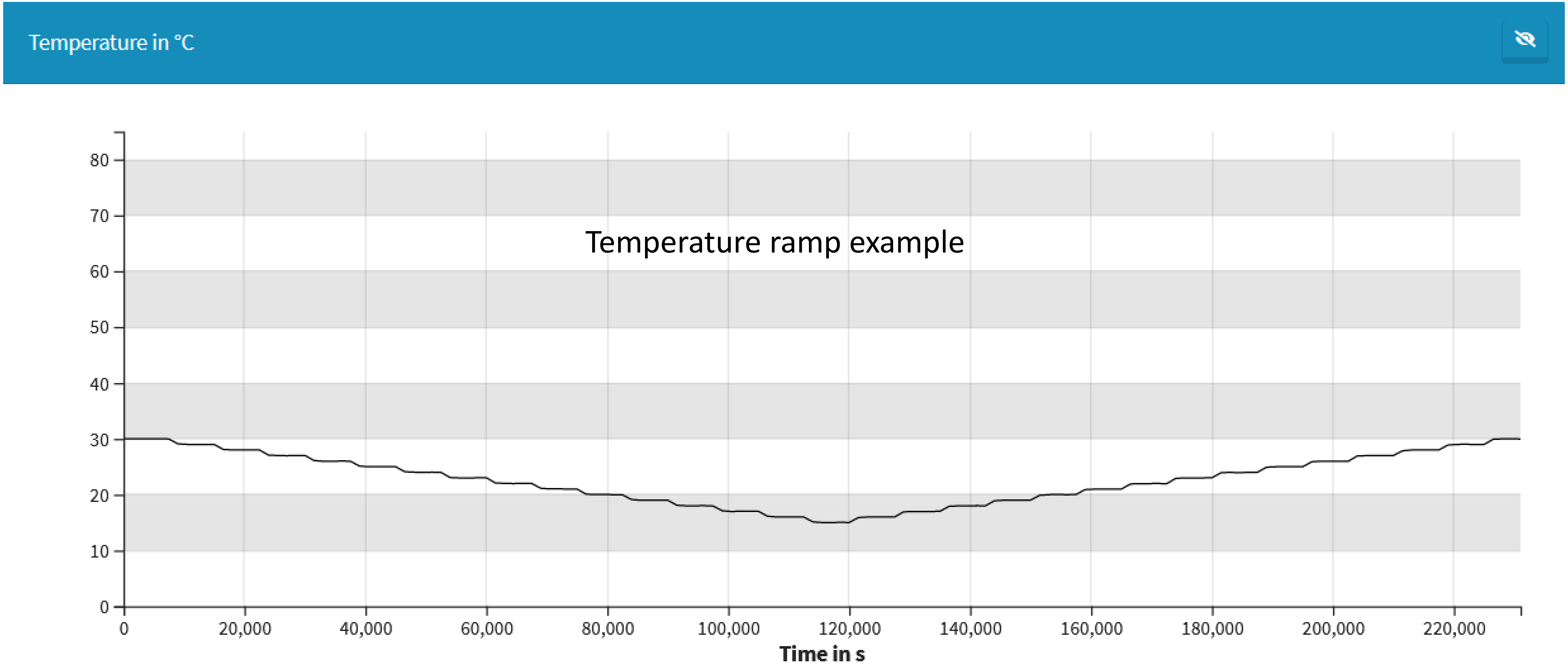


Blue 410 nm

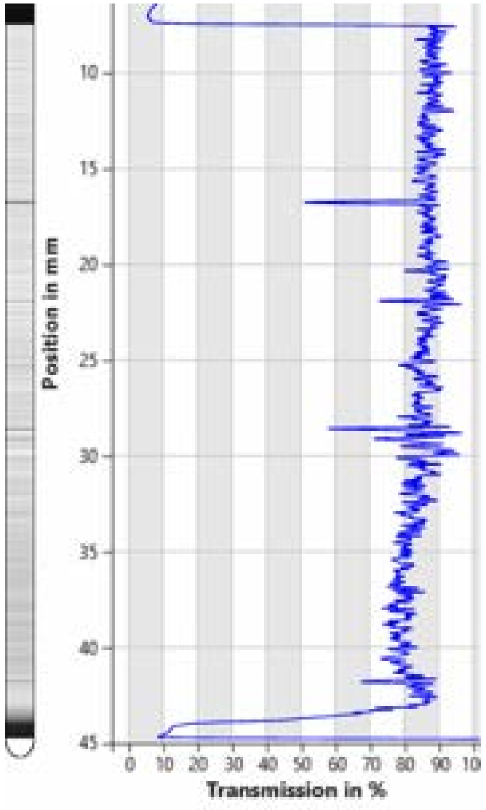


O/W-emulsion real-time separation at gravity, detected by LUMiReader PSA, three wavelengths simultaneously, 2 mm cell, 25 °C, first changes identified by NIR.

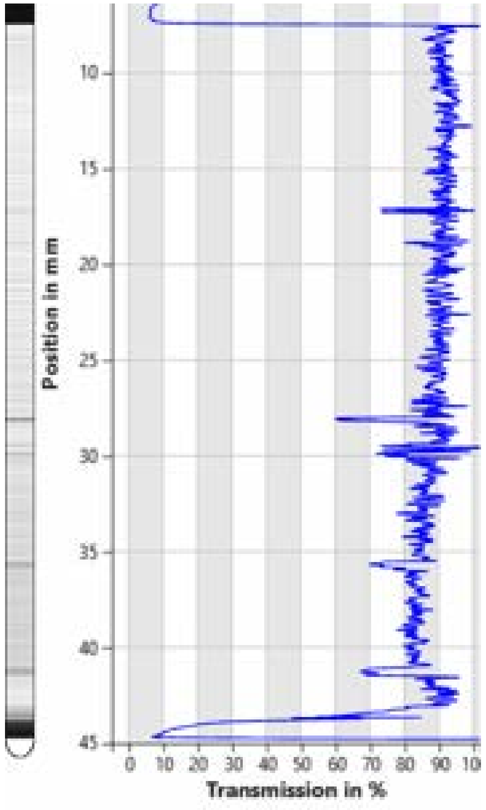
2. Quality of ingredients, real-time @gravity



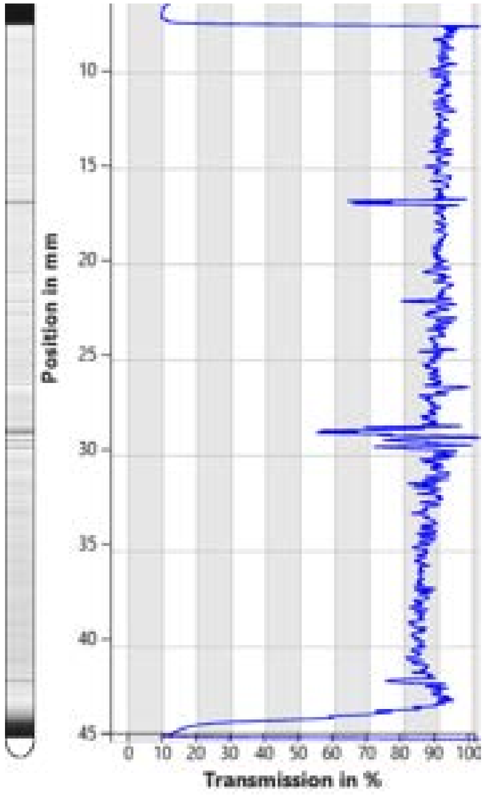
2. Three parallel views on a pure coconut oil



NIR 870 nm



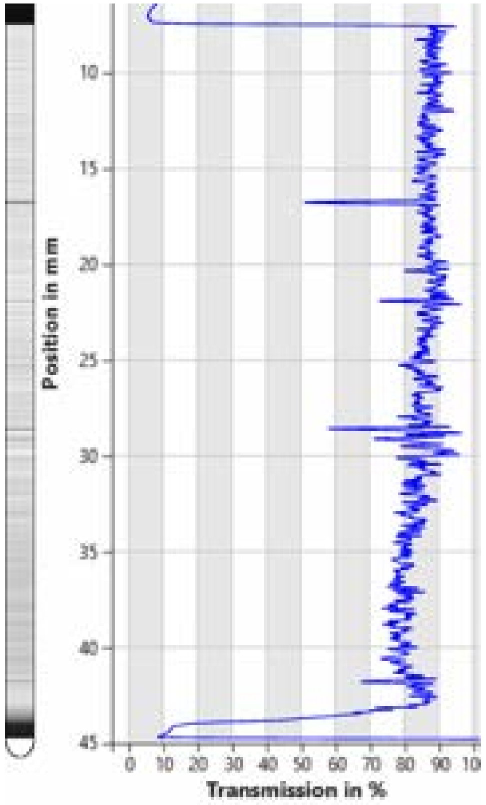
Red 630 nm



Blue 410 nm

Clear @30°C -> turbid @15°C -> again clear @30 °C, fully reversible

2. Comparing a pure with a contaminated coconut oil



NIR 870 nm

Clear @30°C

-> turbid @15°C

-> again clear @30 °C

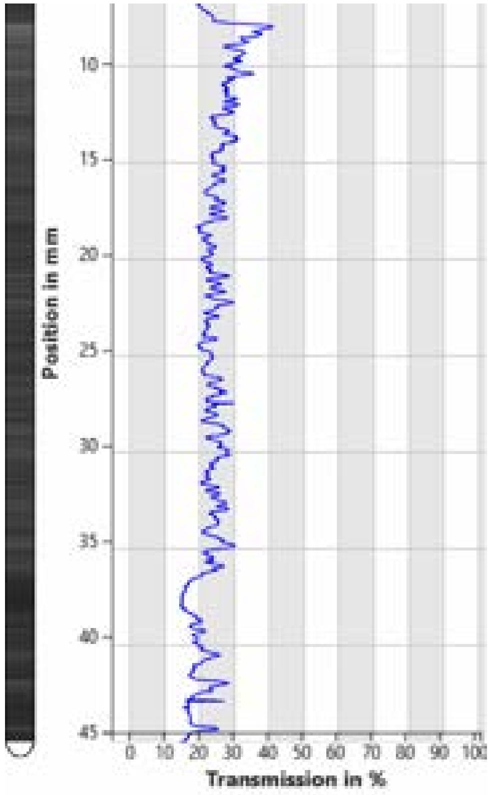
fully reversible

Less transparent @30°C,

Sedimentation of some contaminants

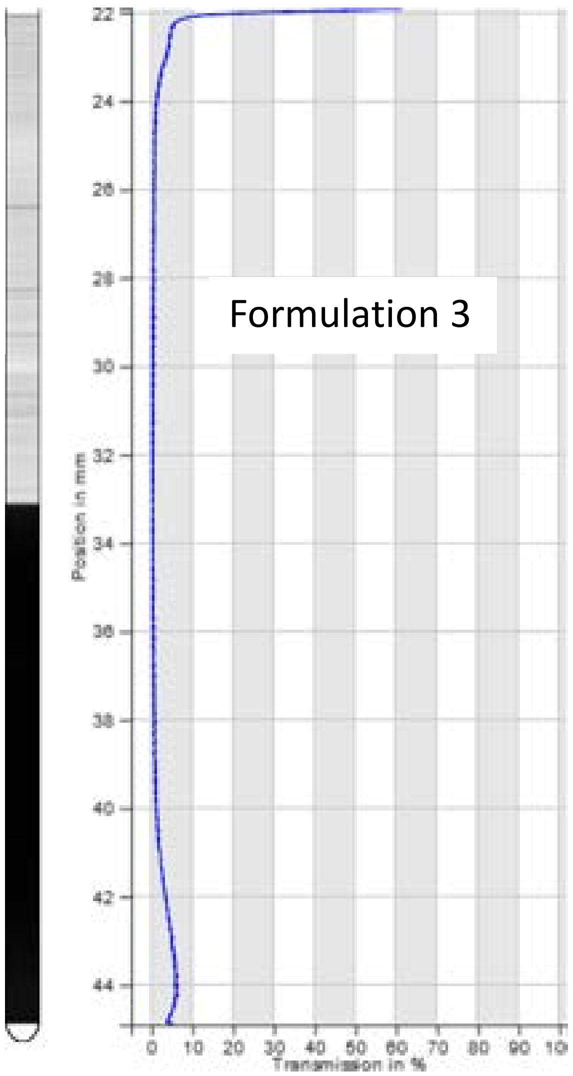
-> turbid @15°C

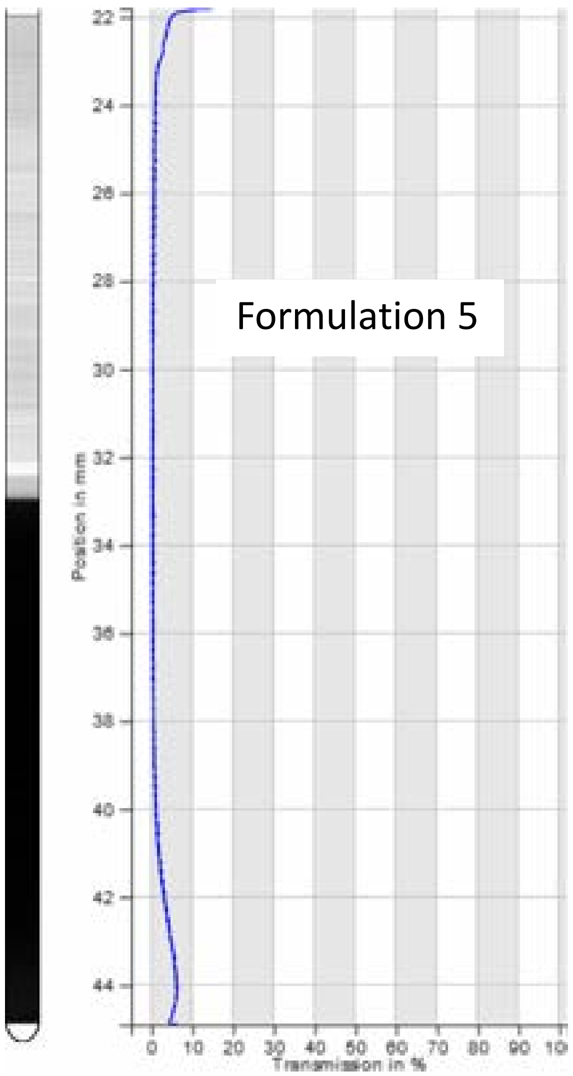
-> more transparent @30 °C, non-settled particles scatter NIR = remains contaminated



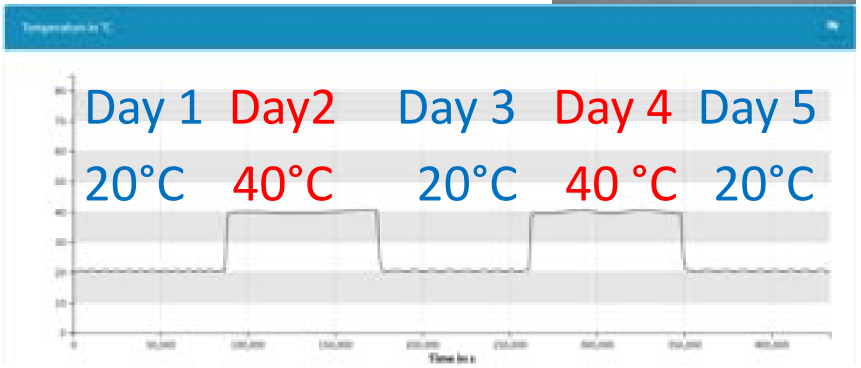
NIR 870 nm

2. Challenges of Real-time Testing (5 days, gravity, temp. ramp)





Replay of all NIR measurement data of 5 days – animated gifs.

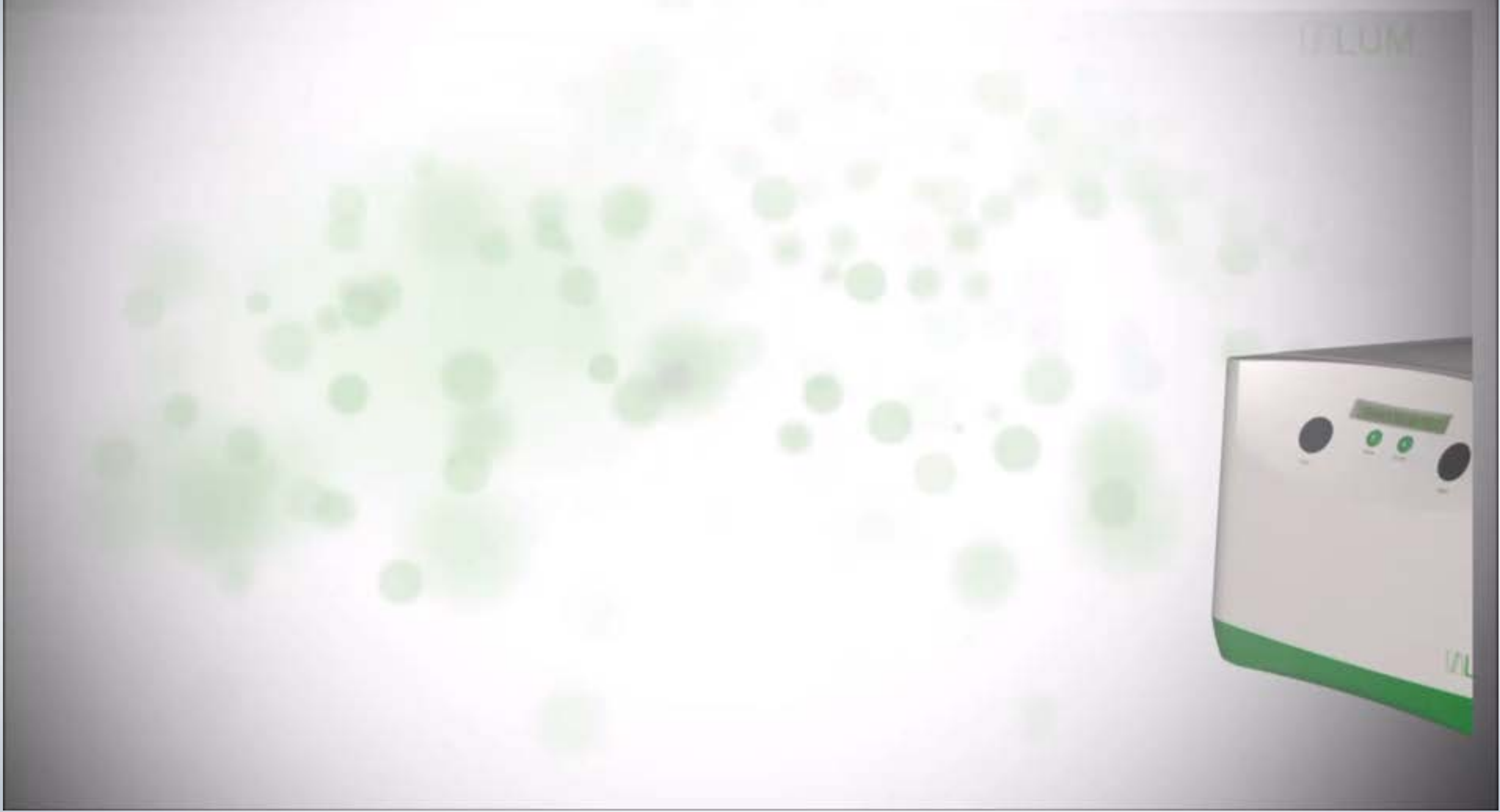


-> NO SIGNIFICANT CHANGE detected by naked eye or by the more sensitive instrument LRPSA 452 using near-infrared (NIR) wavelength.

Do we need much longer than 5 days?

Is there another chance for quick results?

https://www.youtube.com/watch?v=ThRgva2Qczc

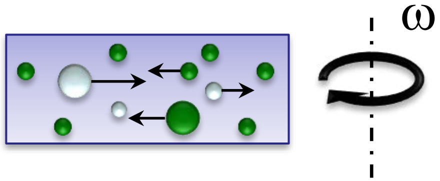


3. Basics of STEP-Technology® at higher gravity

Particle migration

Low particle concentration

Stokes law:



$$v_{Stokes} = \frac{h}{t} = \frac{(\rho_p - \rho_l) \cdot x^2 \cdot g \cdot RCA}{18 \cdot \eta} = K \cdot x^2 \cdot g \cdot RCA$$

$$RCA = a/g^{*}$$

$$a = (2 \cdot \pi \cdot n)^2 \cdot r = \omega^2 \cdot r$$

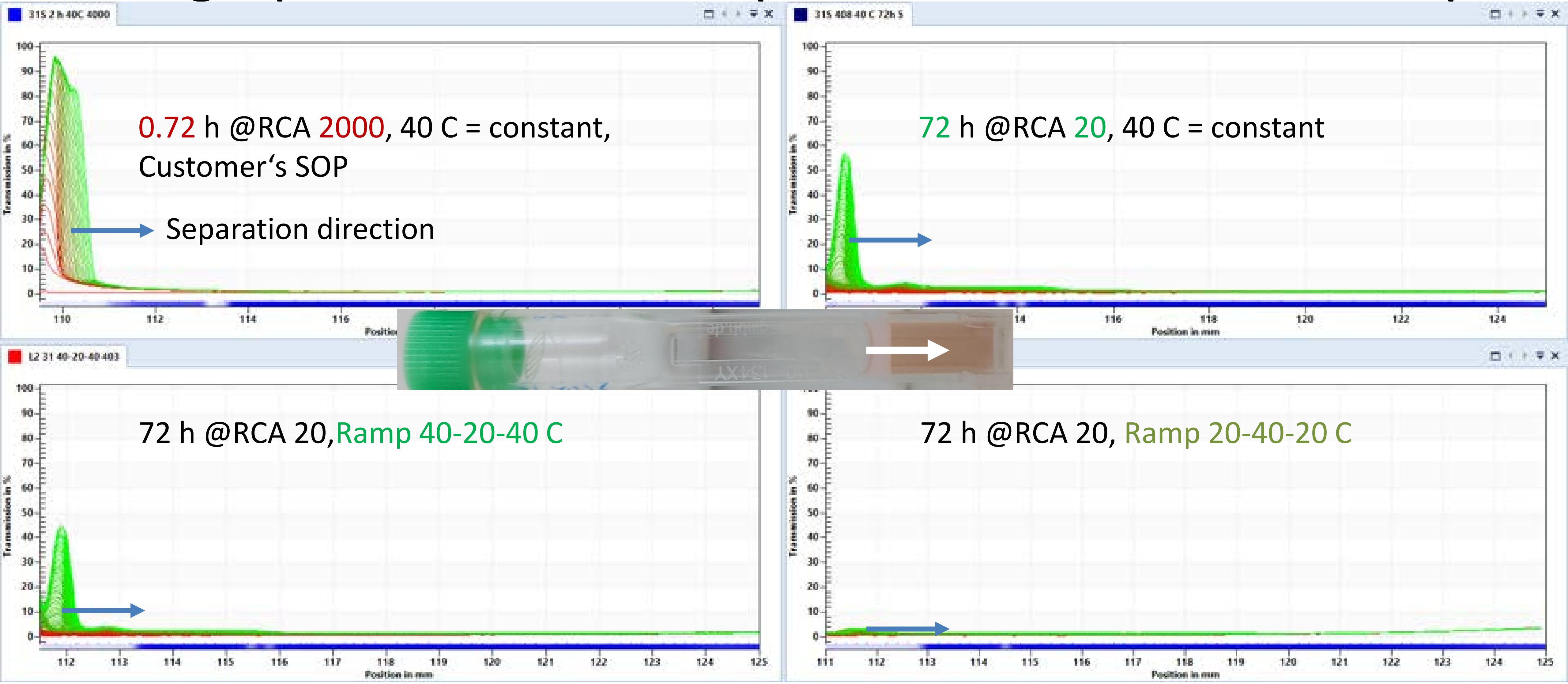
High particle concentration:

$$v = v_{Stokes} \cdot H(\phi)$$

a applied local gravity
n frequency of revolution
r distance from the axis of rotation
ω angular speed
RCA relative centrifugal acceleration

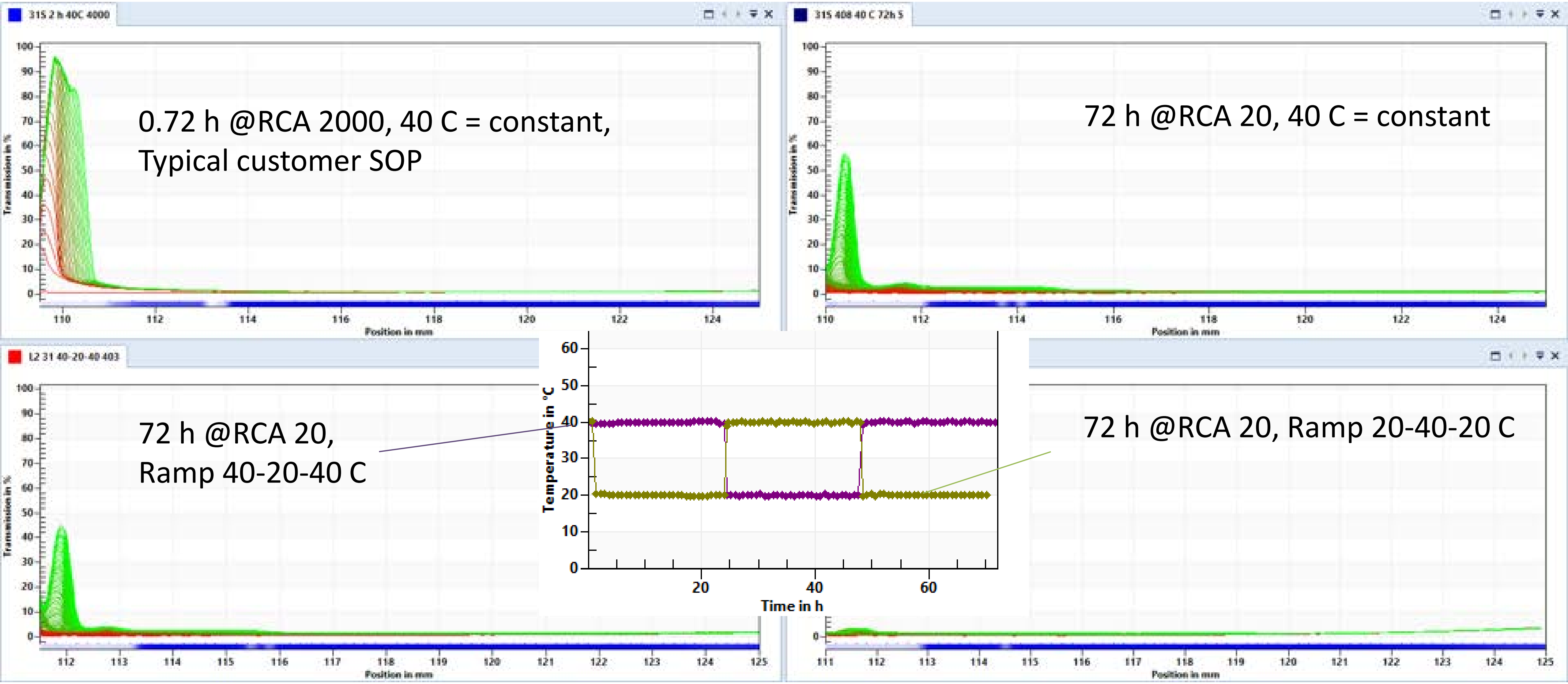
*Comprehensive characterization of nano- and microparticles by in-situ visualization of particle movement using advanced sedimentation techniques, D. Lerche, KONA Powder and Particle Journal 34 (2017), https://www.jstage.jst.go.jp/article/kona/advpub/0/advpub_2019012/_article 21.1.19

3. Fingerprints of 1 Make up without and with T-Ramp

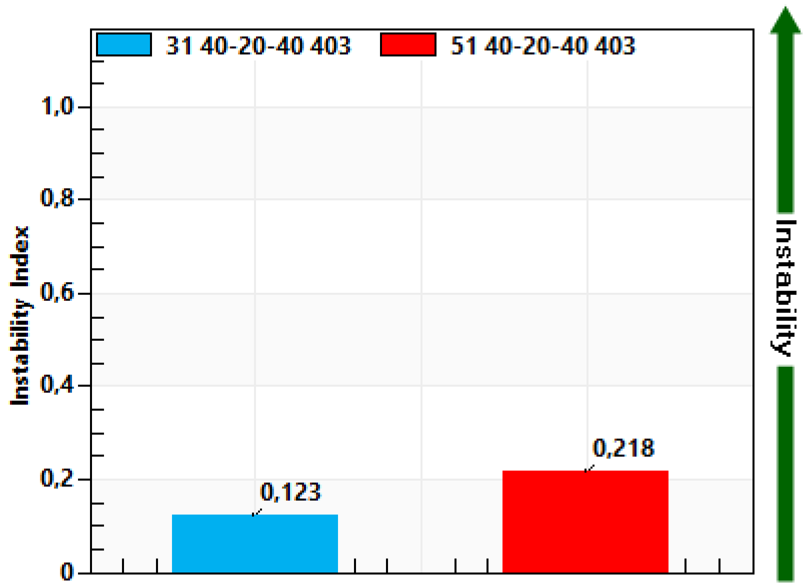


Measured as purchased, in original concentration. Photo after 2.25 h @RCA 2000, 40 C.

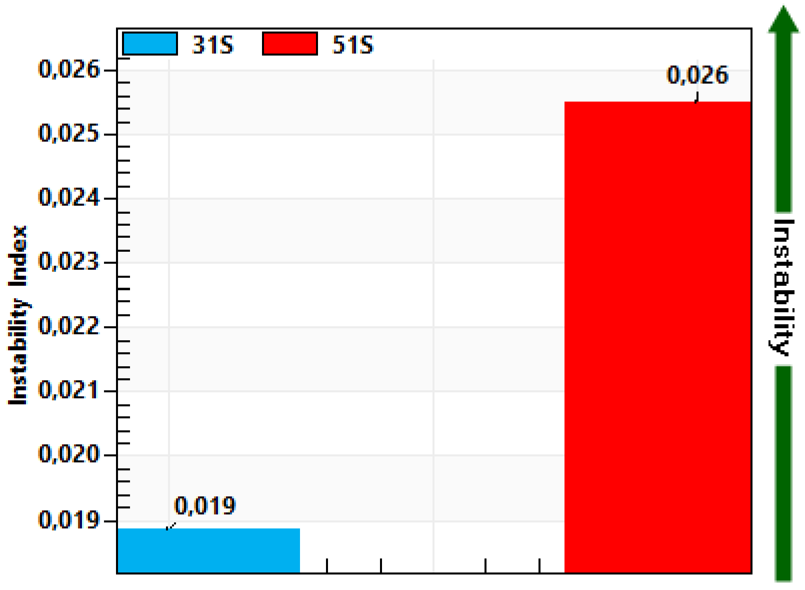
3. Fingerprints of 1 Make up without and with T-Ramp



3. Comparison of two commercial Make up formulations



After 70 h @RCA 20, Ramp 40-20-40 C.



After 70 h @RCA 20, Ramp 20-40-20 C.

Same ranking at all conditions. **Temperature ramp at low RCA allows for more detailed investigation of diffusion driven destabilization processes.**

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4. Real-time *view* on cosmetic products and ingredients

Multiwavelength-Separation Analyser LUMiReader® PSA 452

1. Real-time testing at constant temperature or with a temperature ramp.
2. Faster detection of changes than by naked eye.
3. Samples are measured in their original concentration.
4. ISO/TR 13097, ISO/TR 18811 applied.

RSC
17 March 2022

Particle Size Distribution

Droplet Size Distribution

Multiwave-length PSA

Hansen Solubility/ Dispersib. Parameters

Magnetic Properties

Hydrodyna-mic Density Distribution

Inclined settling/crea-ming (Boycott)

Unsteady particle systems (Agglom., flakes, fibres)

Real-time separation and stability in original conc.

Aging/Real-time Particle Changes

Further opportunities in the same Multi-wavelength LUMiReader PSA 452.

Get more details by keyword search on: https://www.lum-gmbh.com/Literature_Database.html

4. Accelerated *view* on cosmetic products & ingredients

Multiwavelength-Dispersion Analyser LUMiSizer® 651

1. Direct stability testing of up to 12 samples in original concentration under identical conditions.
2. Direct physically accelerated testing with RCA 6-2300.
3. NOW with temperature ramping as additional option, besides constant temperature between +4 and +60 C.
4. Rapid formulation screening due to accelerated testing.
5. **SAVE TIME & MONEY & STORAGE SPACE**.
   Reduce the no. of formulations for subsequent real-time tests.
6. For further opportunities of the instrument see next slide, please.

Your further opportunities with the same Multi-wavelength-LUMiSizer.

Get more details by keyword search on: https://www.lum-gmbh.com/Literature_Database.html

Thank you!

Your questions and enquiries are welcome at info@lum-gmbh.de.