Anton Paar

Testing of cosmetic and pharmaceutical materials

1. **Measuring the consistency of semi-solid materials** - Penetration testing
2. **Determination of flow behavior –** Viscosity measurement
3. **Check fill volumes & incoming materials** - Density measurement
4. **Predict the effect on shelf life of different ingredients** – Oxidation stability

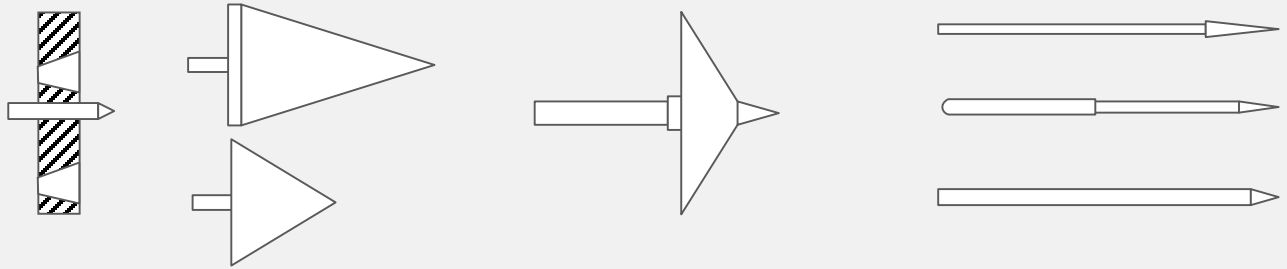
MEASURING THE CONSISTENCY OF SEMI-SOLID MATERIALS

PNR 12 Automated Penetrometer

PNR 12: Test Principle

Place the probe on top of the sample surface → Let it drop for a defined time (most standards: 5 seconds) → Measure the distance the probe penetrated into the sample.

Viscometer	PNR 12	
Liquid	Viscoplastic, semi-solid	Solid



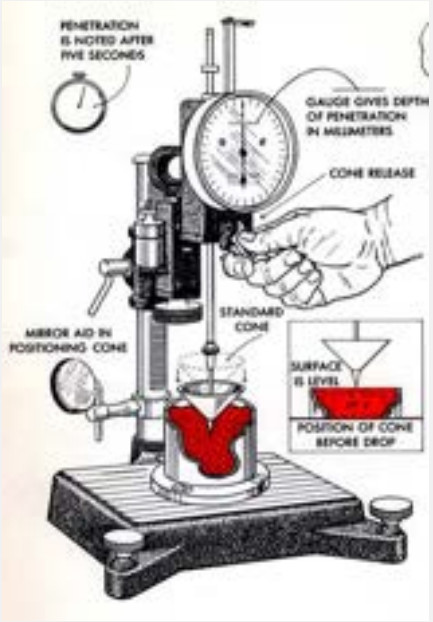
Oils	Gels	Ointment	Greases, cream	Bitumen, wax, soap

The harder the material, the smaller the cone angle.

The penetration depth provides information on the consistency of the sample; the penetration depth increases with increasing softness of the sample

User Benefits Compared to Manual Devices

Conventional manual device



Microprocessor controlled penetrometer PNR 12

- Does not require highly skilled operator
- Automatic surface detection (force
- sensing or conductive sample)
- Measuring range: 0 mm to 80 mm
- Time range: 0.1 sec to 999,999 sec
- Unit can accept external Pt100 for integrated temperature recording
- Digital read-out reduces error
- 200 results in memory
- Automatic calculations
- USB & Ethernet connection

PNR 12: Force Sensor Plunger & Micro Cone

Special force sensor plunger that automatically detects the surface of samples even underneath a layer of water.

- Patented piezo electric sensor
- Works for samples up to 160 PU (1 PU = 0.1 mm)
- For penetration probes with a mass ≤ 2.5 g
- Typically used in combination with bitumen or wax needles

Advantages of micro cone:

- Small sample volume
- Easy air bubble-free filling
- Shorter tempering time (because of small volume)
- Easy cleaning
- Wide consistency range

PNR 12: Typical Applications

Cones

- Fats
- Solid emulsions
- Creams

Perforated disks

- Semi-liquid fats
- Classification of dangerous goods

Needles

- Waxes (ASTM D1321, DIN 51579, IP 376)
- Cement & Gypsum (Vicat, ISO6873, EN26873)
- Lipstick

Rods & Rams

- Silicone
- Rubber
- Soap

PNR 12: Advantages of Penetrometers

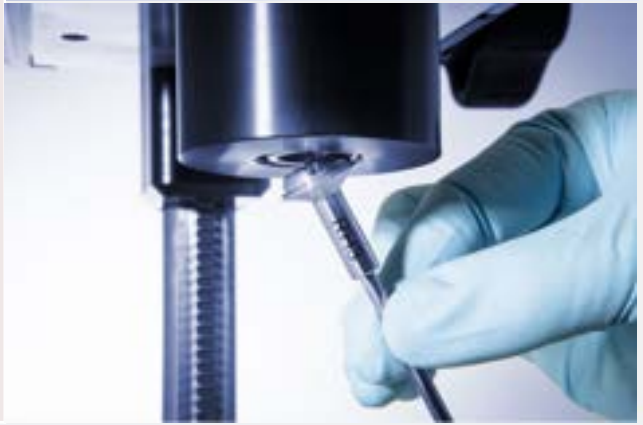
- Ease of use
- Traceable calibration using gauge blocks
- Wide range of industry accepted test methods ASTM, ISO & Pharmacopeia
- Robust and reliable technique

DETERMINATION OF FLOW BEHAVIOUR

ViscoQC 100/300
Rotational Viscometers

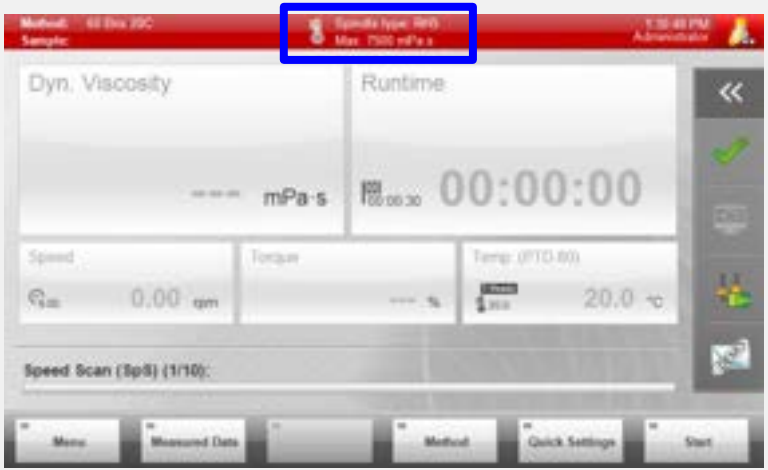
Introducing the ViscoQC from Anton Paar

ViscoQC 100	ViscoQC 300
	
• Stand-alone with 3.5” display • Single point rotational viscometer • Relative spindles (L/RH, Vanes), DIN systems (CC, DG), SSA systems (SC4) • TruMode™ & TruSine™ • Magnetic coupling • Multi language (10 installed) • Optional Peltier temperature control (+15 °C to +80 °C) and sensing • Heli-Plus helical movement and T-bar spindle option • Full traceability with: Toolmaster™, TruGuard™ Digital leveling documentation • Fulfills GMP pharma regulations • On-site replaceable sensor, arrives pre-adjusted	• Stand-alone with 7” touch display • Single and multi point rotational viscometer • Relative spindles (L/RH, Vanes), DIN systems (CC, DG), SSA systems (SC4) • TruMode™ & TruSine™ • Magnetic coupling • Multi language (13 installed) • Optional Peltier temperature control (-45 °C to +175 °C) and sensing • Heli-Plus helical movement and T-bar spindle option • Full traceability with: Toolmaster™, TruGuard™ Digital leveling documentation Adjustment data record • Optional V-Comply pharma qualification Package (PQP) for 21 CFR Part 11, GMP, GAMP 5 • Data memory (up to 999 measurements) • On-site replaceable sensor, arrives pre-adjusted



Real-world useful features of the ViscoQC range

Wish to recognise your spindle and guard automatically?



Toolmaster™

- Manual selection from a spindle list often leads to incorrect viscosity results.
- Solution: Automatic spindle recognition ensures that correct spindle factors are used for viscosity calculation. Every spindle is documented error-free by its unique ID.

TruGuard™

- Measuring with or without guard influences your viscosity result up to 6.5 %.
- Solution: Automatic detection and documentation whether a spindle guard is attached or not guarantees traceability.

Magnetic Coupling

- Easy, single handed removal/attachent of spindles
- Solution: Protects spindles against accidental damage when placing sample

TruMode™

How to test a sample, if optimum spindle/speed combination is unknown?

- Solution: Use TruMode™ – Search for speed automatically
- How?
  - ViscoQC searches the best speed for measurement
  - If spindle/speed combination not OK: ViscoQC tells you to use smaller or bigger spindle
- Use the determined speed for further measurements (e.g. in Stop at Time)

TruSine™ - Automatic bearing health check obsoletes manual wind up

Typically, a manual wind up (oscillation bearing check) is needed to check the bearing friction of a rotational B-type viscometer

ViscoQC automatically performs this health check by TruSine™ after tapping “Start”

SPINDLES / MEASURING SYSTEMS

Relative spindle sets:

Standard spindle sets
–Comparative measurements require spindle size and speed to be specified
–2 common set types
  –Low Viscosity set (L)
  –Mid/High Viscosity set (RH)

T-bar sets:

–Measure non-flowing, highly viscous
–Helical spindle movement ensures contact to the sample during viscosity measurement and avoids air channeling problem

DIN, UL & SSA spindles:

–For "absolute" viscosity values
–For measurement according to ISO 3219, DIN 53019-1 and DIN 54453 (DIN only)
–Suitable for shear rate control and data analysis using mathematical models
–Lower sample volumes, better temperature control is possible

General considerations

In rotational viscosity testing

1. Avoid air bubbles (sample preparation)
2. Particle size:
   i. < 0.1 mm for Concentric Cylinder systems
   ii. < 0.05 mm for Double Gap systems
3. Choose correct instrument torque model
   L – low viscosity samples
   R – medium viscosity samples
   H – high viscosity samples
4. Ensure that instrument is leveled properly
5. Choose correct spindle for measurement
   a. Viscosity range vs. surface area
   b. Manual or automatic selection from list

$$\text{Viscosity} = \frac{TK * SMC * 10\,000}{\text{Speed}} \text{ x } T\%$$

6. Consider the torque range while measuring

7. Turbulences might occur at high speeds

8. Viscosity can be extremely temperature dependant
   a. Is the sample at the correct temperature?
   b. Is it stable for the duration of the test?

ViscoQC 300 – Software Packages

Upgrade your stand-alone instrument: V-Curve and/or V-Comply

Required to fulfill regulations of 21 CFR Part 11

V-Curve includes:

- Online graph to see live measurement data
- Programmability: Up to 50 steps
  (Sample preparation as separate step possible)
- Analysis (Graph, Compare)
- Mathematical models*
  (IPC Paste, Power Law, Bingham, Herschel-Bulkley,
- Casson, NCA/CMA Casson, Gelation Time, Shear
- Thinning Index, Thixotropy Breakdown Coefficient,
- Unique Best Fit Feature Functionality, Statistics)
- Further Methods
  Yield method to determine the yield stress – needs vane spindles

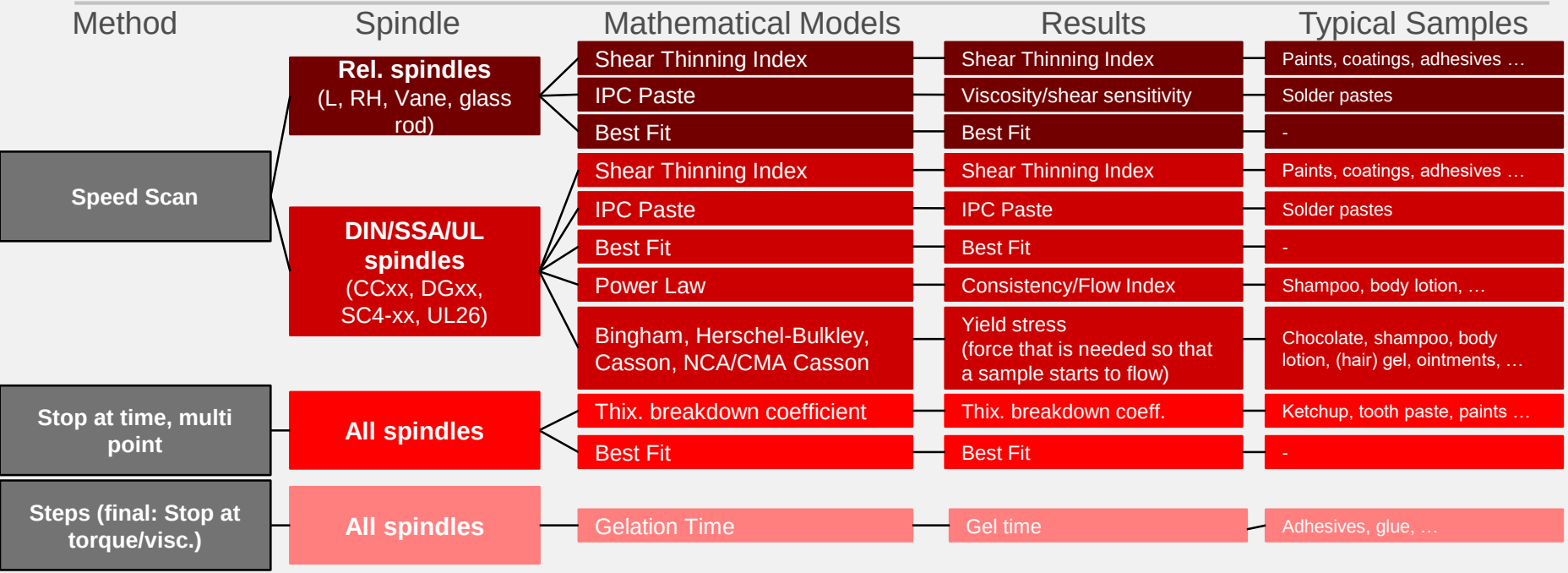
V-Comply includes:

- Audit trail (log book)
- Electronic signature
  (Submit, Review, Approve)
- Compliance modes
  (ADI, Non-storage, VNC, Value visibility)
- Increased security functions
  (date and time stamp, password access, password complexity, restrict use of defined spindles through Toolmaster™, disable USB memory device and more...)
- Delivery includes PQP document

* DIN/SSA/UL spindles are required for mathematic models like Bingham, Herschel-Bulkley, Casson and NCA/CMA Casson.

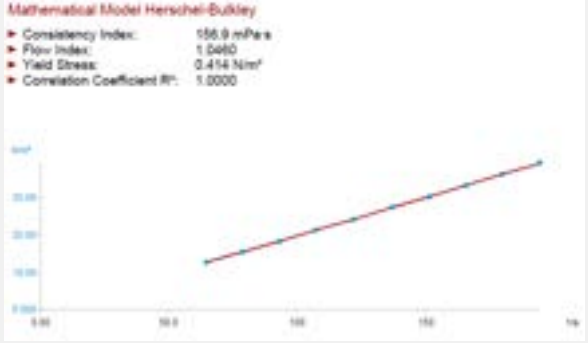
Mathematical models I

For easy analysis of multi-point test data

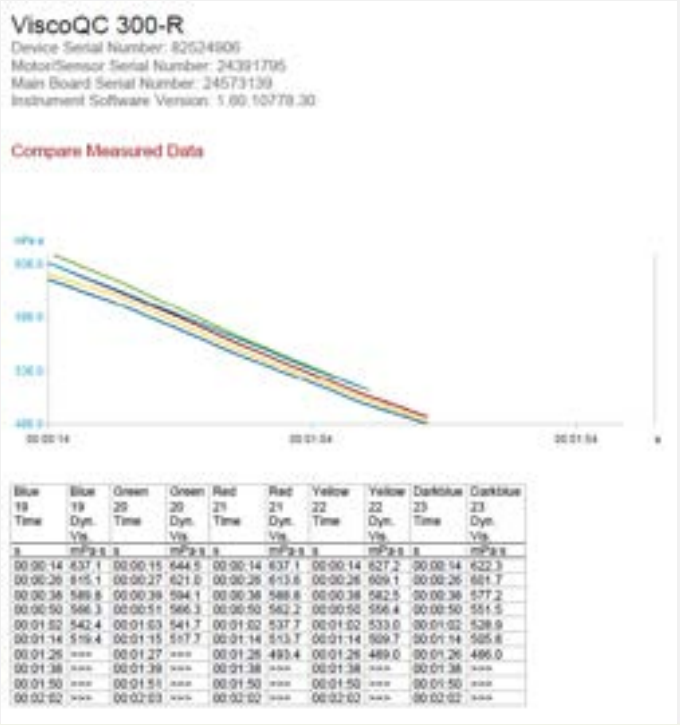


Mathematical models II

For easy analysis of multi-point test data

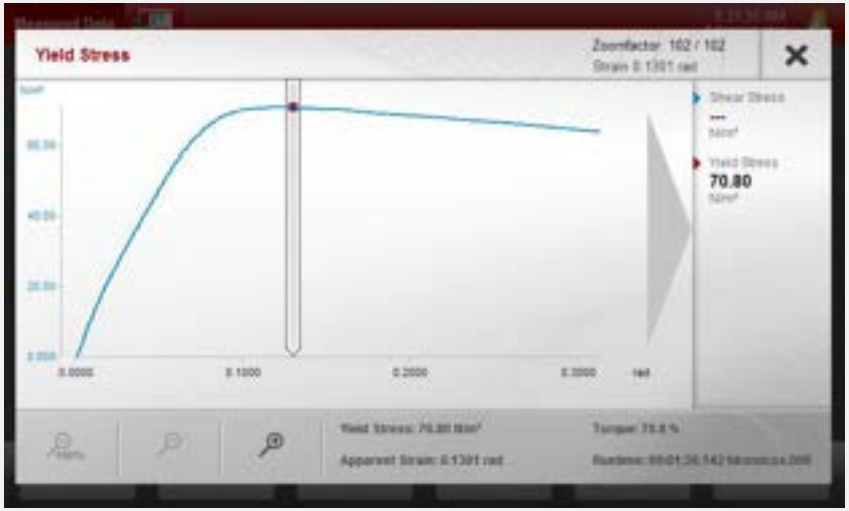


- A range of standard models are available
- Simplify and standardise data for comparision and quality control
- Determine flowability of sample for process control and optimisation
- Compare between batches of material easily



Yield Stress Method (YiS)

Determine a sample‘s static yield stress with vane spindles



- Yield stress= force that is needed so that a sample starts to flow
- Static yield stress determination
  - Measures the start of flow at zero speed/shear rate
  - Only possible with vane spindles V71 to V75
    - V71 to V73 feature 2 immersion marks (Full/Half)
  - Ideally at low rotational speed: 0.01 rpm to 0.5 rpm

Typical samples: Gel & ointments

EXAMPLE APPLICATIONS

ViscoQC 100/300
Rotational Viscometers

Hair Care Products

Confirmation of shampoo consistancy in the QC Lab

Customer Requirements

- QC of incoming raw materials (responsible for cleansing efficiency, users' perception, foaming properties)
- Maintain the correct viscosity in final product, in line with target customer expectations
- Avoid occurrences of poor processability (difficult to fill or pump)
- Assess structural changes during storage (= poor long-term stability?)

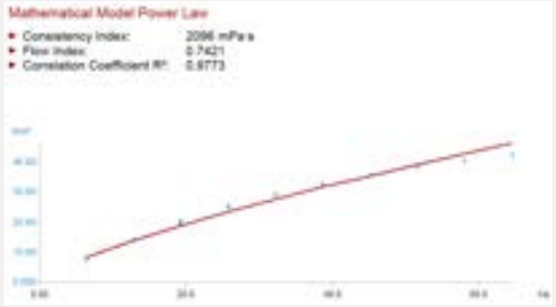
Test

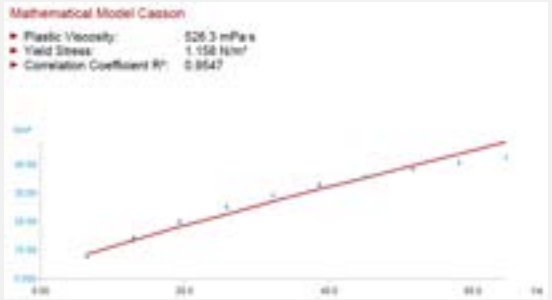
- Flow behavior - Power Law: Flow Index, Consistency Index (η at 1 s^{-1})
- Yield point (mathematical model e.g. Casson)

Solution*

- **ViscoQC 300 – R**
- **Spindle: CC18**
- **DIN Adapter**

CC12 measuring system only requires 6.4ml of sample





* This is an example configuration. Instrument model + spindle have to be selected according to the viscosity of the sample.

Body Care 1

Will the formulation be easily be rubbed onto your skin?

Customer Requirements

- QC of incoming raw materials (responsible for e.g. emulsifying, hydration effect on skin)
- Formulate to the correct viscosity to please different target customers (men, women or children)
- Assess structural changes during storage (= poor long-term stability?)

Test

- Flow behavior: Shear Thinning Index
- Yield point (mathematical model e.g. Casson)

Solution*

- **ViscoQC 300 – R**
- **Spindle: RH5**
- **Guard R**

Standard 600ml beaker with 500ml of sample recommended

* This is an example configuration. Instrument model + spindle have to be selected according to the viscosity of the sample.

Body Care 2

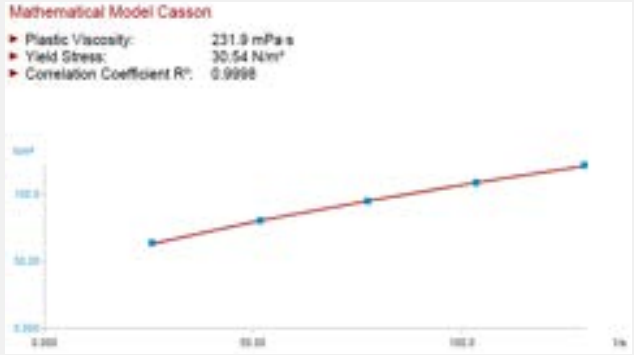
Will the formulation be easily be rubbed onto your skin?

Customer Requirements

- Equivalent results as Brookfield/current viscometer
- Avoid poor processability (difficult to fill or pump)
- Avoid difficult applicability (body lotion is not easily absorbed by the skin)
- Right viscosity to please the target customer (men, women or children)

Test

- Flow behavior (Shear Thinning Index >1 = shear thinning)
- Yield point (mathematical model e.g. Casson)



Solution*

- **ViscoQC 300 – H**
- **Spindle: CC18**
- **PTD 80 (T-control) or DIN adapter with Pt100 (T-sensing)**

Sample volume required is 6.4ml. PTD ensures precise temperature control for repeatable results

* This is an example configuration. Instrument model + spindle have to be selected according to the viscosity of the sample.

Shower Gel

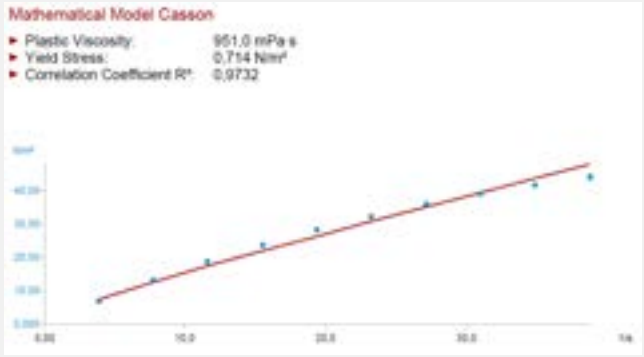
Ensure the body wash only flows from the tube when squeezed

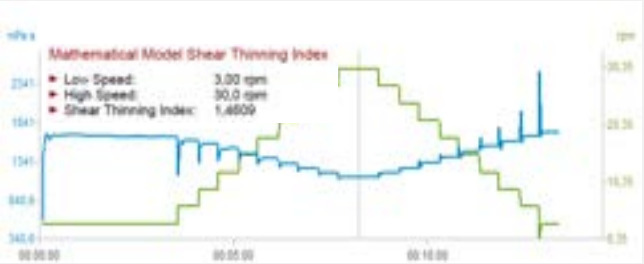
Customer Requirements

- Easy device to use, need to avoid user errors leading to repeat measurements
- Equivalent results as Brookfield/current viscometer (easy SOP conversion for operators)
- QC of incoming raw materials (responsible for cleansing efficiency, users' perception, foaming properties)
- Avoid poor processability (formulation is difficult to fill or pump)
- Difficult applicability (body wash does not come out of tube easily)

Test

- Flow behavior: Shear Thinning Index
- Yield point (mathematical model e.g. Casson)
- Plastic viscosity (viscosity after yield)





Solution*

- **ViscoQC 300 – R**
- **Spindle: CC18**
- **PTD 80 (T-control) or DIN adapter with Pt100 (T-sensing)**

Sample volume required is 6.4ml. PTD 80 ensures precise temperature control for repeatable results

* This is an example configuration. Instrument model + spindle have to be selected according to the viscosity of the sample.

Eye Make-up Remover

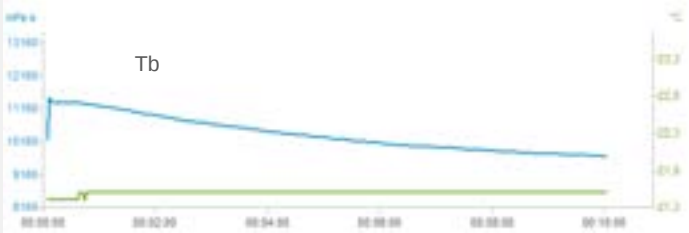
Ensure that eye make-up remover lotion is easily be applied

Customer Requirements

- Easy device, avoid user errors (do not need to repeat measurements)
- Equivalent results as Brookfield/current viscometer (operator SOP conversion)
- Avoid formulations that require excess force to move the liquid out of the tube (applicability) or during the production process (pumping or filling)
- Confirm the desired shear-thinning flow behavior, to allow user to apply the product easily and evenly on the eyelids

Test

- **Flow behavior: Shear Thinning Index**
- **Yield point**
- **Thixotropic breakdown coefficient Tb**



Solution*

- **ViscoQC 300 – R**
  - **Spindle: RH5**
  - **Guard R**

Standard 600ml beaker with 500ml of sample recommended

* This is an example configuration. Instrument model + spindle have to be selected according to the viscosity of the sample.

Face Cream

How to maintain the structure of a paste-like facial cream?

Customer Requirements

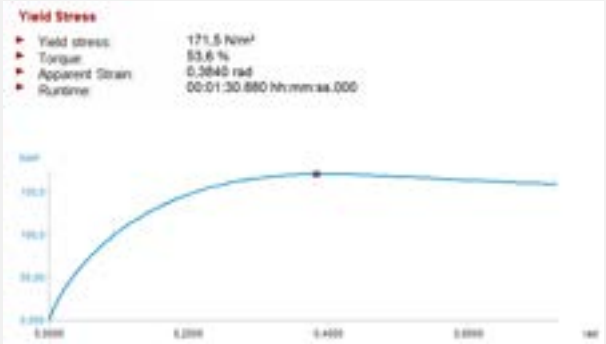
- Viscosity has a great impact across the different production stages such as pumping, filling, mixing, or squeezing the product out of the tube or applying it on the face

Test

- Flow behavior: Shear Thinning Index
- Yield point (mathematical model e.g. Yield Stress)

Solution*

- **ViscoQC 300 – H**
- **Spindle: V72 (half immersion)** Standard 600ml beaker



* This is an example configuration. Instrument model + spindle have to be selected according to the viscosity of the sample.

Nail Care

Ensure nail polish does not run off after application

Customer Requirements

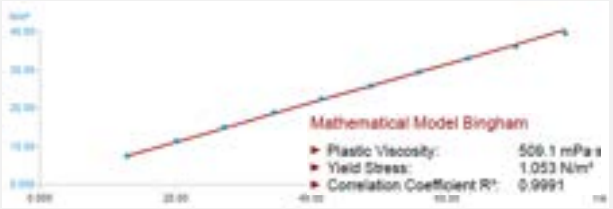
- Appropriate viscosity for easy application and drying in a reasonable time
- Correct viscosity that guarantees processability (e.g. filling)
- Producer has to ensure a proper structural recovery:
  - High viscosity so that varnish or polish stays on the brush & the nail
  - Application to the nail without leaving brush traces

Test

- **Thixotropic behavior (step test - 3ITT)**
- **Thixotropic breakdown coefficient Tb**
- **Flow behavior: Shear Thinning Index**
- **Yield point (mathematical model e.g. Bingham)**

Solution*

- **ViscoQC 300 – R**
- **Spindle: CC18/D18 (6.5 mL sample)**
- **PTD 80 (T-control) or DIN adapter with Pt100 (T-sensing)**



* This is an example configuration. Instrument model + spindle have to be selected according to the viscosity of the sample.

CHECK FILL VOLUMES & INCOMING RAW MATERIALS

DMA 501/1001

Laboratory Density Meters

Why change to a laboratory digital density meter?

Forget about sample-intensive measurements. **1 mL sample** volume ensures that no product is wasted. Highly viscous samples can easily be injected.

Depending on the accuracy you need, DMA 501 (**3 digits**) and DMA 1001 (**4 digits**) deliver precise and reliable results. Density and SG results faster and more accurately than density bottles or glass hydrometers.

Anton Paar's unique one-point water adjustment makes DMA 1001 the fastest calibrated device on the market. Get your measurements done faster. UKAS certified density standards are widely available to ensure full traceability.

Ready for data printout and quick export via USB or network file sharing to a PC. Say goodbye to errors in manual data recording.

We've integrated 60+ conversion tables into our devices to free your time from manual calculations. Directly measure concentration of incoming materials and finished product. Up to 400 custom methods can be created for your common tests and samples.

DMA 501 is your entry ticket to the world of 3-digit density measurement from the market leader Anton Paar. Leave your time-intensive, breakage-prone hydrometers behind.

DMA 1001 is the world's most technologically refined 4-digit entry-level density meter, packed with features from our high-range models. It's compliant to all relevant pharma industry standards.

Both models feature fast and accurate temperature control for reliable and reproducible measurement, the robust 7" touchscreen can be operated using gloves.

PHARMA & COSMETICS

,	RAW MATERIAL CHECK	CHECKING FILLING VOLUMES	TRACEABLE QUALITY CONTROL
Challenge	When checking the quality and/or purity of raw material before use I need to get the concentration value immediately, even when the substance is highly concentrated.	The final filling of packages must meet legal requirements while still being cost-efficient with no errors	I need to document the test results for each product and all actions carried out on the instrument. The values need to be traceable.
Solution	DMA 501 has stored tables for automatically calculating concentrations. The density is measured, automatically converted into concentration, and shown within seconds. Your own tables can be imported if you need custom quantities and calculations.	The 3-digit accuracy of DMA 501 is sufficient for converting the weight and measured density into the filling volume. For each product filled you can set the lower and upper limits for acceptable volumes and see the value at a glance.	With DMA 1001 you can assign roles and responsibilities and implement audit trail to log all activities and electronically sign the final results.
Benefit	No need to look up values in tables or calculate the concentration yourself. You save up to 10 minutes per measurement with no risk of calculation errors. This means you make pass/fail decisions quickly and based on correct information.	Never overfill or underfill, while meeting all regulations and requirements.	Achieve absolute certainty in your results and data. You can certify the quality of your products for shipment and sales and have all the right information at hand for audits by regulatory authorities and in case of customer complaints.

PREDICT THE EFFECT ON SHELF LIFE OF DIFFERENT INGREDIENTS

Rapidoxy 100 Oxidation Stability Tester

Introduction

- RapidOxy 100: Determining the oxidation stability of products under accelerated conditions
  - Elevated temperature
  - Exposure to excess of pure oxygen
- Stainless steel test chamber: Sample is set under pressure
- with pure oxygen and heated
- The temperature is kept constant and the pressure is continuously traced
- Due to oxygen consumption the pressure drops
- Result: Induction period = Time until a certain pressure drop is reached

EXAMPLE APPLICATIONS

Rapidoxy 100

Investigation of Shea Butter Samples

- Determination of oxidation stability of two different shea butter samples
- Determination of the influence of the measuring temperature on the induction period at:
  - T = 120 °C
  - T = 140 °C
- Other parameters for RapidOxy 100 measurement (constant):
  - p = 700 kPa, stop at Δp = 10 %

Investigation of Shea Butter Samples

- Arrhenius dependency: Induction period about four times longer at 120 °C compared to induction period derived with measurement at 140 °C
- Oxidation stability order of shea butter samples 2 > 1
- Result is in accordance with experience and expectation of the customer

Different shea butter samples		
Sample	Result [min]	
	120 °C	140 °C
1	420.12	85.03
2	425.81	100.08

Investigation of a Fragrance Oil

- Determination of oxidation stability using the RapidOxy 100 and AOCS method Cd12b_92_13 (Rancimat/OSI instrument) of:
  - Pure fragrance oil sample
  - Fragrance oil sample with different antioxidants
- Parameters for RapidOxy 100 measurement:
  - T = 120 °C
  - P = 700 kPa, stop at Δp = 10 %
- Parameters of Cd12b_92_13:
  - T = 110 °C
  - Air flow 20 L/h

Investigation of a Fragrance Oil

- Using RapidOxy 100:
  - Time for measurement significantly shorter
  - Repeatability significantly better
- Highest influence on oxidation stability
  - Pure fragrance oil sample
  - Fragrance oil sample with different antioxidants

Investigation of a fragrance oil			
Sample	**Result 1**	**Result 2**	**Cd12b_92_13**
Pure fragrance oil	25.60 min	24.88 min	4.96 h
+ 0.1 % Aperoxid TLA	58.23 min	58.25 min	20.47 h
+ 0.5 % Natrox RO5	64.78 min	65.91 min	28.97 h
+ 0.05 % BHT	98.70 min	97.85 min	11.56 h 12.44 h
+ 0.05 % Tinogard® TT	100.26 min	100.00 min	47.05 h

Investigation of Massage Balm Samples

- Determination of oxidation stability of three different massage balm samples
- Parameters for RapidOxy 100 measurement:
  - T = 140 °C
  - p = 700 kPa, stop at Δp = 10 %
- Determination of repeatability by double determination

Investigation of Massage Balm Samples

- Oxidation stability order of investigated massage balm samples 3 > 2 > 1
- Massage balm 3 by far the most stable
- Very good repeatability demonstrated on all three samples
- Determined order of oxidation stabilities is in accordance with the expectation of the customer

Measurement of massage balm samples		
Samples	**Result 1 [min]**	**Result 2 [min]**
Massage balm 1	753.41	741.86
Massage balm 2	864.56	798.66
Massage balm 3	1292.06	1319.53

Investigation of Cosmetic Oils

- Cosmetic oil samples of customer serve as base for lotion
- Determination of oxidation stability of four different cosmetic oil samples
- Parameters for RapidOxy 100 measurement (constant):
  - T = 120 °C
  - p = 700 kPa, stop at Δp = 10 %

Investigation of Cosmetic Oils

- RapidOxy 100 allows for a fast determination of the most stable cosmetic oil in terms of oxidation stability
- Oxidation stability order of measured cosmetic oils: 3 > 4 > 2 > 1
- Determined order of oxidation stabilities is in accordance with the experience of the customer

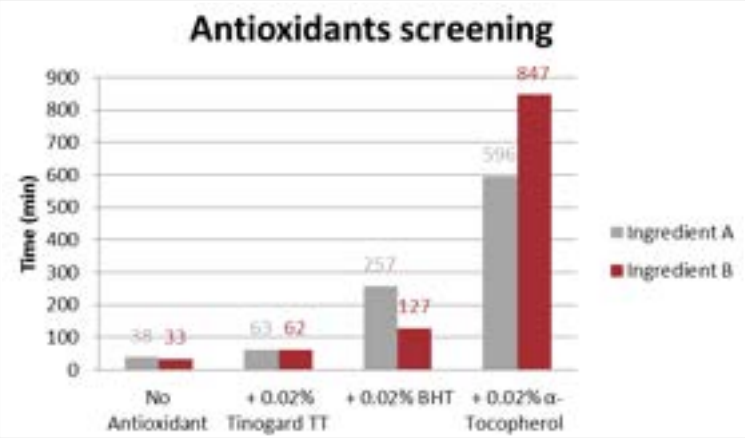
Measurment of cosmetic oils	
Sample	**Result [min]**
Cosmetic oil 1	158.53
Cosmetic oil 2	177.85
Cosmetic oil 3	206.10
Cosmetic oil 4	189.40

Investigation of Fragrance Ingredients with Different Antioxidants

- Determination of oxidation stability of different fragrance ingredients (aldehydes)
  - Fragrance A
  - Fragrance B
- Addition of 0.02 % stabilizing agent each
  - BHT (butylated hydroxytoluene)
  - α-Tocopherol
  - Tinogard® TT
- Parameters for RapidOxy 100 measurement:
  - T = 60 °C
  - p = 300 kPa (lower pressure due to volatility of samples), stop at Δp = 50 % (certain aging profile of samples desired by customer)

Investigation of Fragrance Ingredients with Different Antioxidants

- Effect of antioxidant depends on fragrance ingredient
- Most significant effect on oxidation stability of both samples through addition of α-Tocopherol
- GCMS-analyses after measurements show:
  - Decrease of aldehyde concentration
  - Formation of acids as oxidation products (formed from aldehydes)
  - Addition of antioxidants clearly represses formation of acids



Main Benefits of Measuring with the RapidOxy 100

- Time-saving, high sample throughput
  - Fast and unique measuring principle, high temperature range of up to 180 °C
  - No sample preparation necessary
  - Simple cleaning
  - Low sample volume required, only 1g of sample
- Very good precision of test results
- Fluid, solid, powder and semi-solid samples can all be measured without preparation or pre-treatment
- Easy and intuitive stand-alone operation
- Low oxygen usage

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www.anton-paar.com for further details on instruments and applications


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Rotational Viscosity Testing of Ointments with ViscoQC


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Rotational Viscosity Testing of Make-up (Remover) with ViscoQC


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Rotational Viscosity Testing of Personal Care Products with ViscoQC

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