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Haake PolyLab OS

Material Characterization

Modular Polymer Processing System

PolyLab OS

PolyLab OS – Open Solution

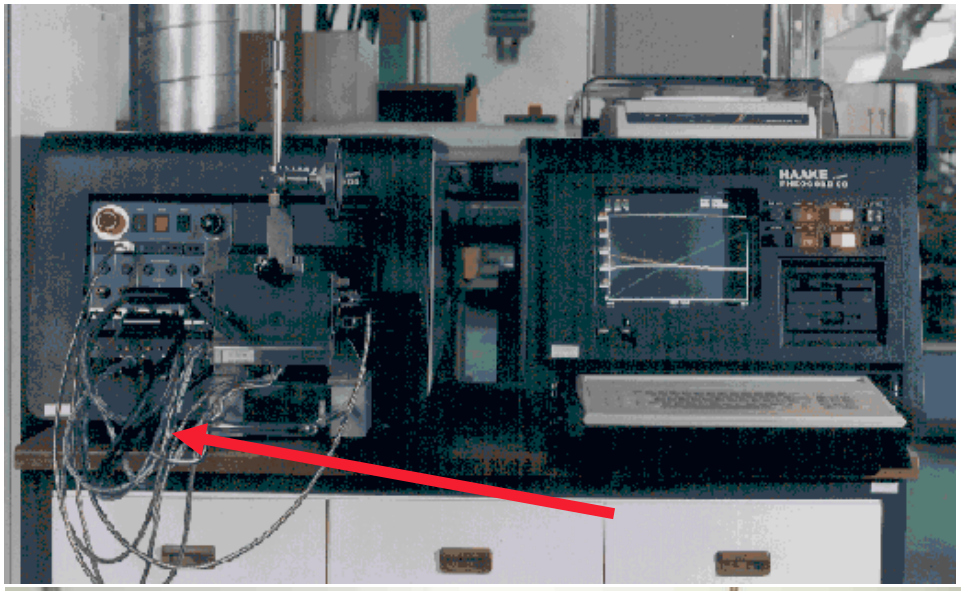
PolyLab OS is a polymer processing instrument platform with a modular design to allow the connection of newly updated components (extruders, mixers, ancillaries), and support legacy system components, as well as future developments using an open architecture control system based on industry standard components

Torque Rheometer System

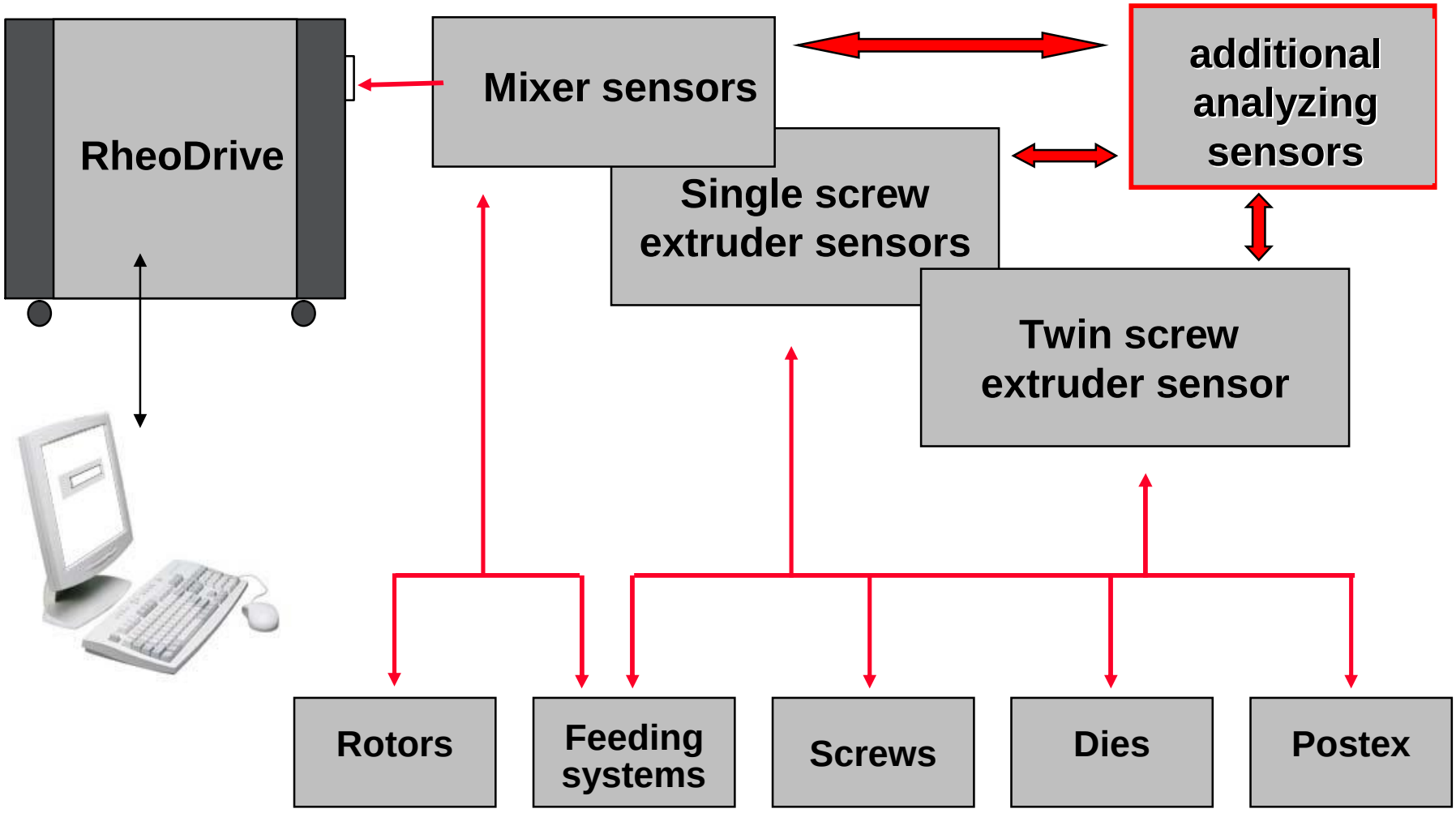
Why use Torque Rheometers ?

- Polymers are processed in a liquid state - Extrusion, injection moulding
- The flowabillity (viscosity) will influence:
  - Processabillity
  - Quality of the final product
- Torque Rheometer and its sensors are Down–scaled production machines (mixers, extruders, dies, postex)
- Test procedures similar to production
- Sample Preparation under evaluative conditions

Legacy of Predecessors – 40 year history



PolyLab OS = RheoDrive+Mixer/Extruder+Sensor



PolyLab System Components

- RheoDrive 4 – 16 kW
- PolySoft Control & Application Software
- Mixer
- Extruder
- PTW Extruder
- CTW Extruder
- Dies
- Postex
- Ancillaries
- Analyzing Sensors

PolyLab Customer Values

Ø Open Solutions

- integration of new sensors and systems with CAN bus (viscosity, spectroscopy, optical)
- open solutions for all the applications (expand system with additional sensors)
- open for the future (standardized interfaces, serviceability)
- new sensors for combined measurements or methods (viscosity, optic, analytical tests)
- needs only one instrument for polymer testing

Ø Plug and Measure

- system with user compatibility of components, reliability, ease of use
- connect all our offerings at site, no factory installation required
- connect to PC, network or PDA with TCP/IP (cable-wireless) or USB

Ø Time Saving Software

- one software suite for the complete PolyLab OS platform with run mode and data evaluation
- time saving software (automation of tests, ease of use, unsupervised running)

Ø Serviceability

- modular in design for service using industry standard components where possible
- remote diagnostics, exchange of components, minimum downtime
- floating data storage of 96 hrs for traceability and data security
- remote supervision from the office with browser/PDA monitoring alarms/events/data

Ø Traceability of Experimental Results

- traceability of experimental results and FDA compliance (21CFR11)
- prepared for FDA regulations in design and materials used

Features of PolyLab OS

Ø System

> Modular system for mixer, single screw extruder or twin screw extruder to be connected (one at a time) to a single drive unit
> Open System to connect and integrate different components

Ø RheoDrive

> new drive concept with intelligent 2-4-16 kW motors
> very smooth and silent running (RD2 is a synchronous motor) drive system specifically selected for improved mixer test results

Ø Docking station

> new mechanical mechanism to look the docked systems in place; easy to use and safe
> only one central socket/connector to eliminate cables and wires hanging all over
> Rigid and stable chassis on rolls to travel to different locations in a pilot plant

Ø CAN Open Bus

> Intelligent CAN bus integrates all peripherals with self-identification and transfer of characteristic data like calibration data table
> Adaptation of all standard CAN open components (thermocouple, pressure sensor, additional sensors) without software modification
> integration of downstream equipment with standardized interface

Ø Connectivity

> Simple plug & play computer connection with USB or TCP/IP (option)
> Remote controller (cable based) to operate the drive manually
> Remote diagnose and status info with Web browser or PDA

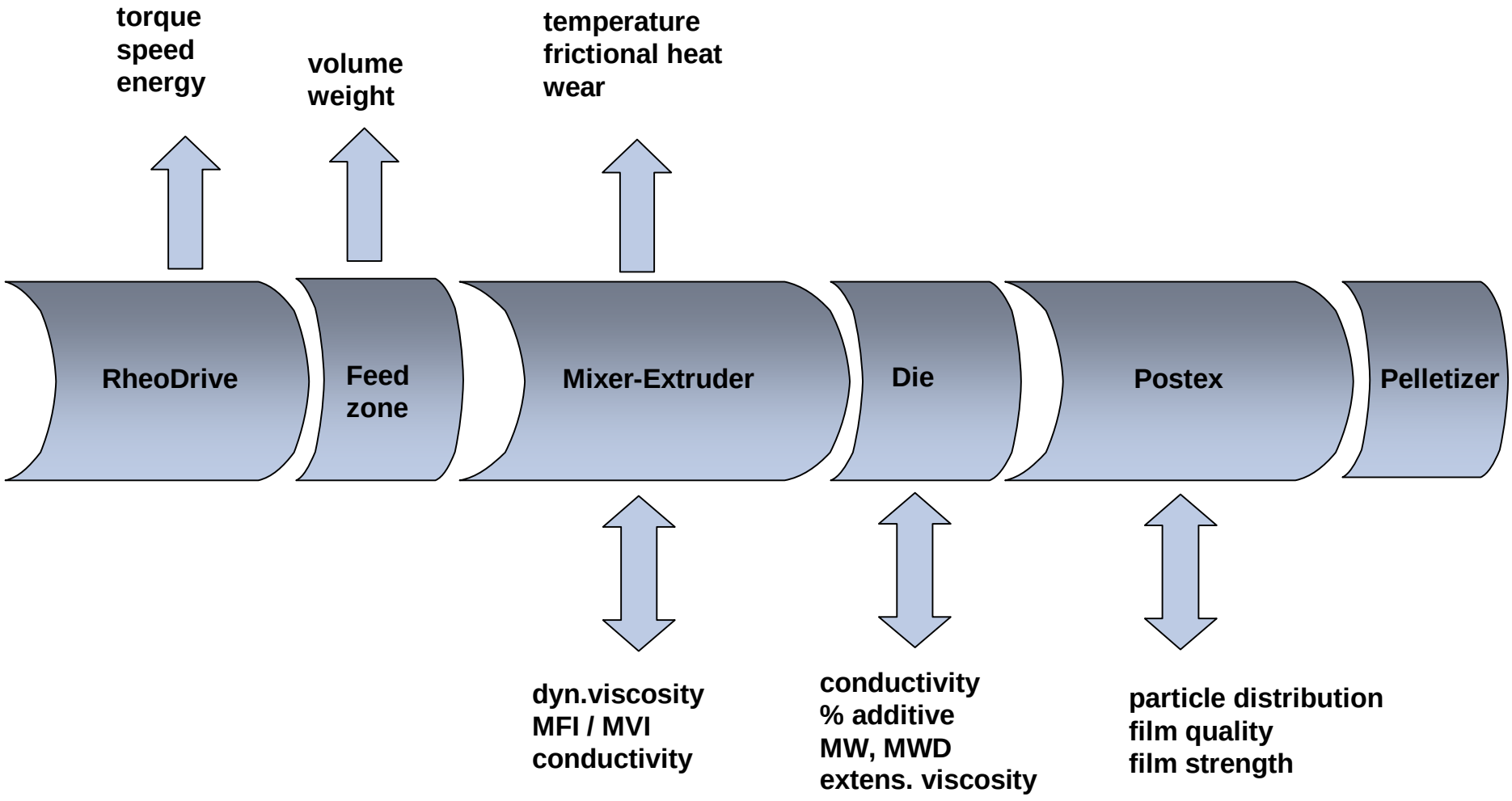
What CAN sensors are available on the market?

CAN Open Sensors	
Instrument values	Pressure Temperature Torque Speed (rpm) Velocity Force
Material values	Thickness Weight Humidity Conductivity (thermal) Conductivity (electric) Spectroscopy Adsorption Fluorescence Particles (Camera)

PolyLab OS Applications

n Standard O Option	PVC mixer test	Batch com-pounding	Filter test	Film blowing	Flat film extrusion	Capillary rheology	Rubber extrusion	Color screening test	Powder coating	Tablet production
Customer segment	QC	Prod/R&D	QC	R&D/QC	R&D/QC	R&D	QC	R&D/QC	Prod	Prod.
RheoDrive 2 kW^{CAN}	n			n						
RheoDrive 4 kW^{CAN}			n			n	n			
RheoDrive 16 kW^{CAN}		n			n			n	n	n
Gear box	n	----	n	n	n	n	n	----	----	n
Electric (heat zones)^{CAN}	n	n	n	n	n	n	n	n	n	n
Application Software	n		n			n				n
Display^{CAN}		n		n	n		n	n		
PLC^{CAN}		n						n		
PC/Computer	n	O	n			n				n
Mixer^{CAN}	n									
Feeder^{CAN}		n		O	O	n	n	n	O	n
Single screw extruder^{CAN}			n	n		n	n			
Parallel twin screw^{CAN}		n			n			n	n	n
Die^{CAN}		n	n	n	n	n	n	n	n	n
Postex^{CAN}		Take off	Melt pump	Film Tower	Take off	Melt pump		n	n	n
Sensor^{CAN}	Torque		Pressure	Camera	Camera	Rheo die				
Postex^{CAN}				Take off					Nibbler rolls	

PolyLab OS System



RheoDrive – the heart of the PolyLab system

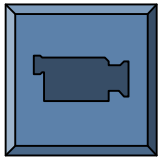
2 Models:

§ RheoDrive 4
- 4 kW
- 250 rpm output speed
- Standard applications:
  - R252, PTW16, RX600

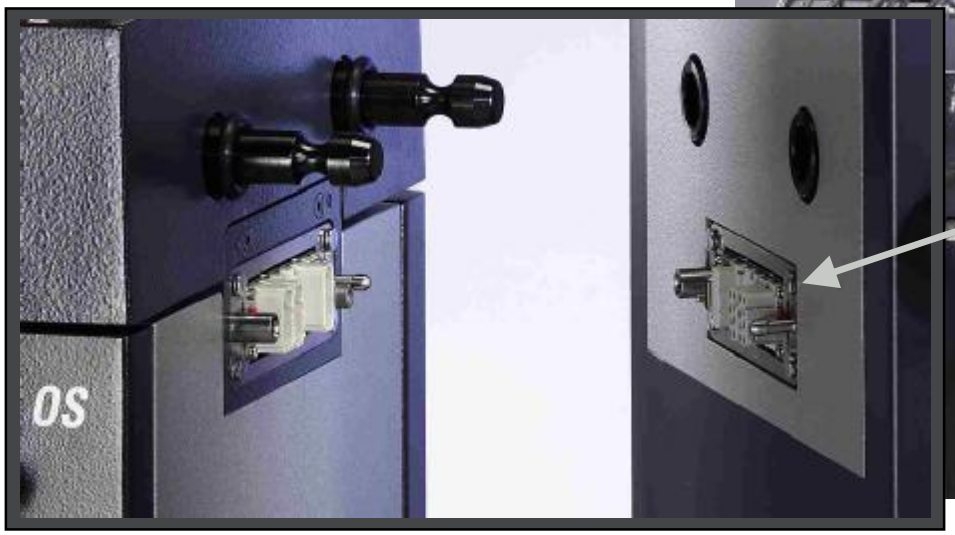
§ RheoDrive 16
- 16kW
- 550 rpm output speed
- High torque applications:
  - PTW24 or RX3000

PolyLab OS – Central Coupling

- Central coupling for docking Mixers and Extruders
- Connects the communication and power lines

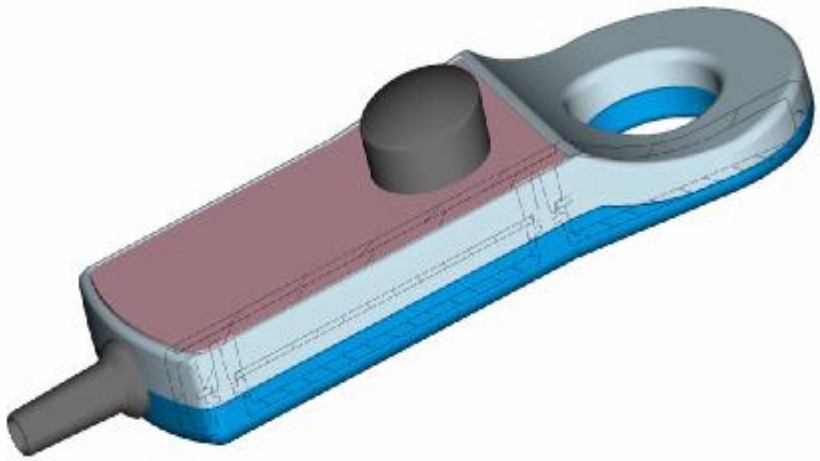


Changing Accessories



PolyLab OS – Remote Control

Control PolyLab OS from where ever it is suitable

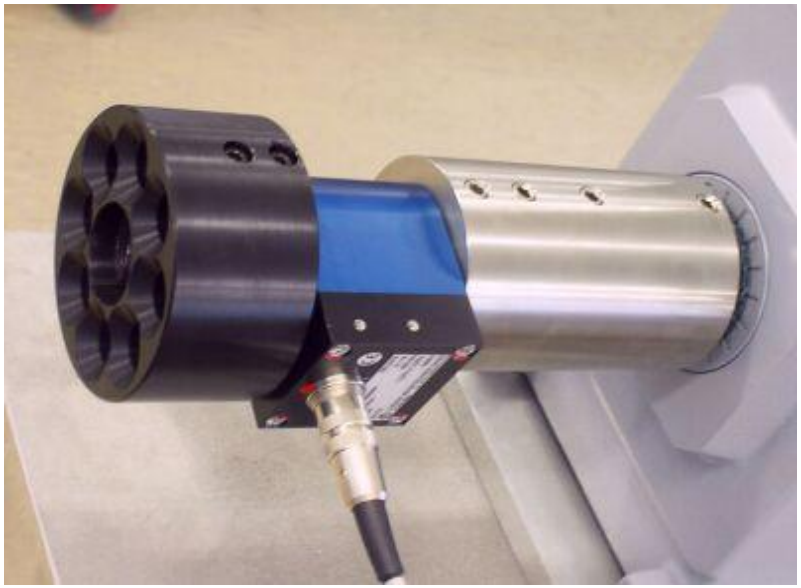


Optimise the process at any location by not losing visual control on your process

CANOpen Pressure Sensor

Pressure sensor with CanOpen Bus which are securely stored for protection from damage

PolyLab OS – Torque Measurement



Located just behind coupling to sense only torque from accessory (no gear box or motor influence)

3 torque measuring options:

1) Via motor current
2) Standard torque sensor
3) Multi range torque sensor

Torque Specifications

Type	Torque range	Resolution	Accuracy *
Motor current	400 Nm	1.0 Nm	5 % FSD
Torque transducer	400 Nm	0.1 Nm	0.15 % FSD
Dual range torque transducer	60 / 400 Nm	0.01 / 0.1 Nm	0.045 / 0.15 % FSD

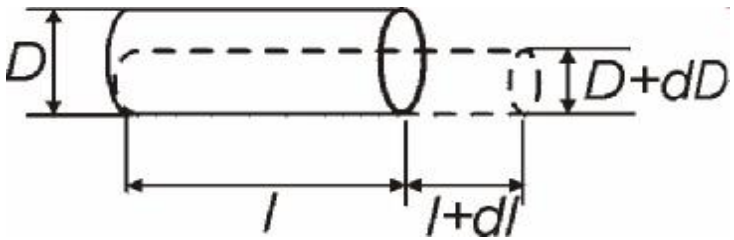
Multi Range Torque Sensor 400Nm / 120Nm

- Strain gauge principle
- Dynamical Resolution Enhancement
  ≤ 60 Nm : 0.01 Nm
  > 60 Nm : 0.1 Nm
- Dual torque Sensors
- The PolyLab Torque Sensor has non contact digital data transmission from shaft to casing (no brushes, no collector ring). This means no failure of transmission and it is maintenance free.
- Lower Range useful for low torque applications like low viscosity resins, powder blending and absorption testing

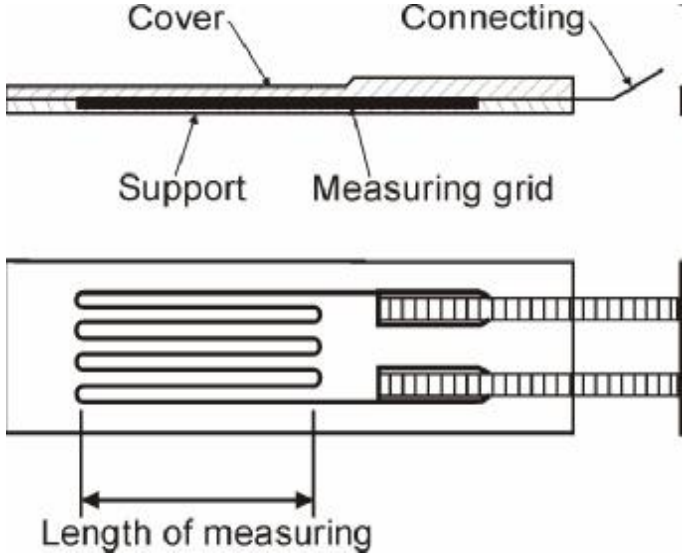
high accuracy: 0.15 % FSD and high reproducibility:0.02 % abs.

PolyLab Torque Measurement – operating principle

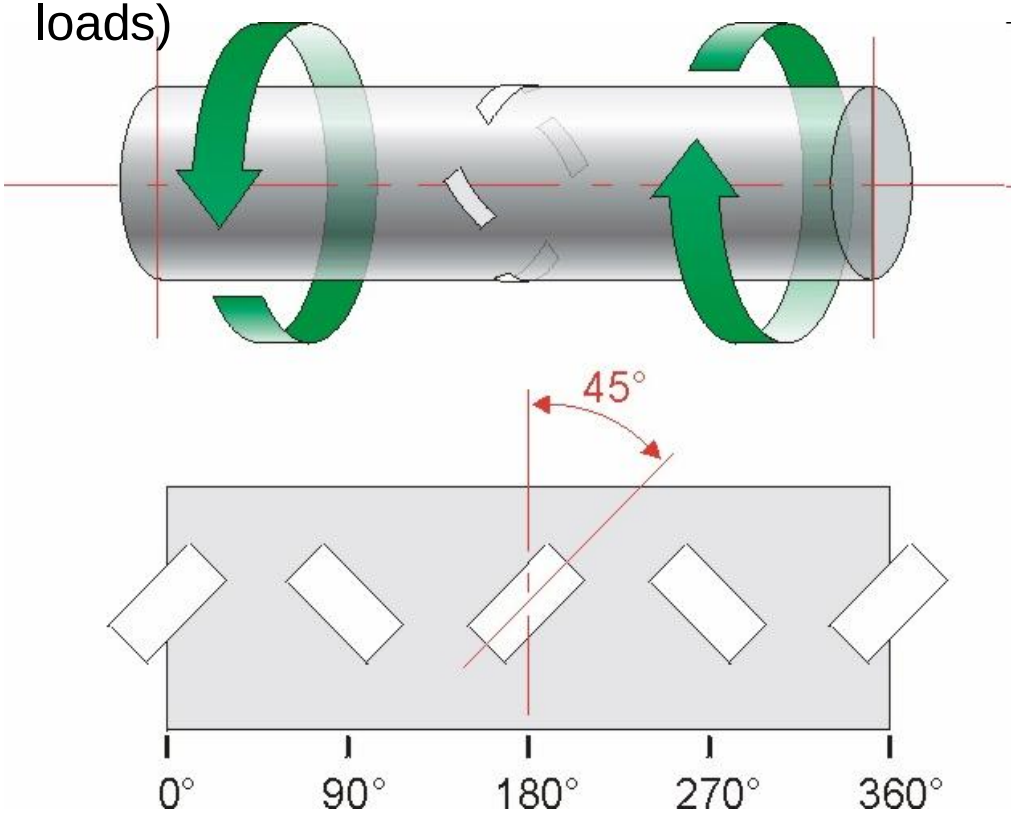
a) Change of electrical resistance



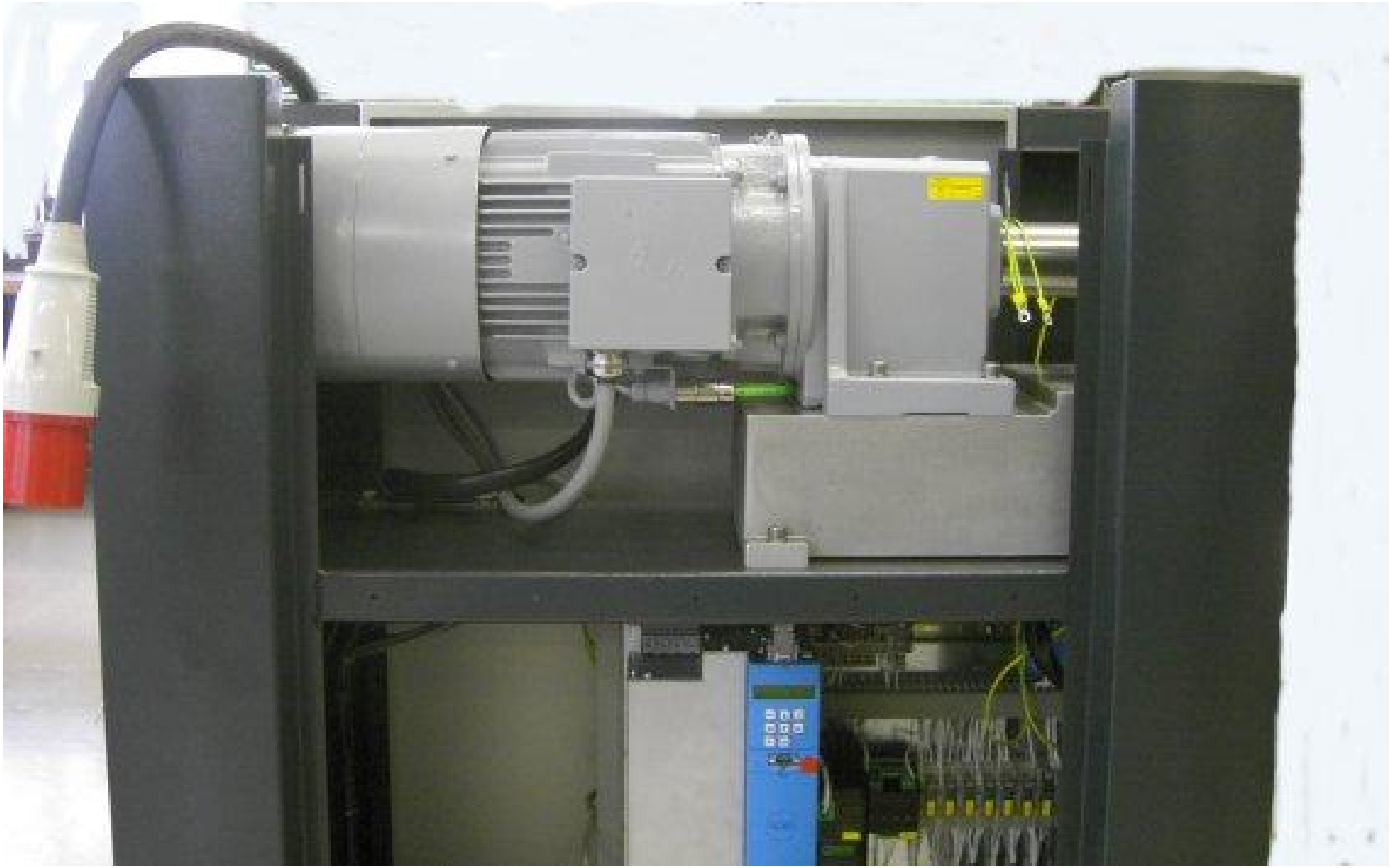
b) Principle of strain gauge (horizontal stress)



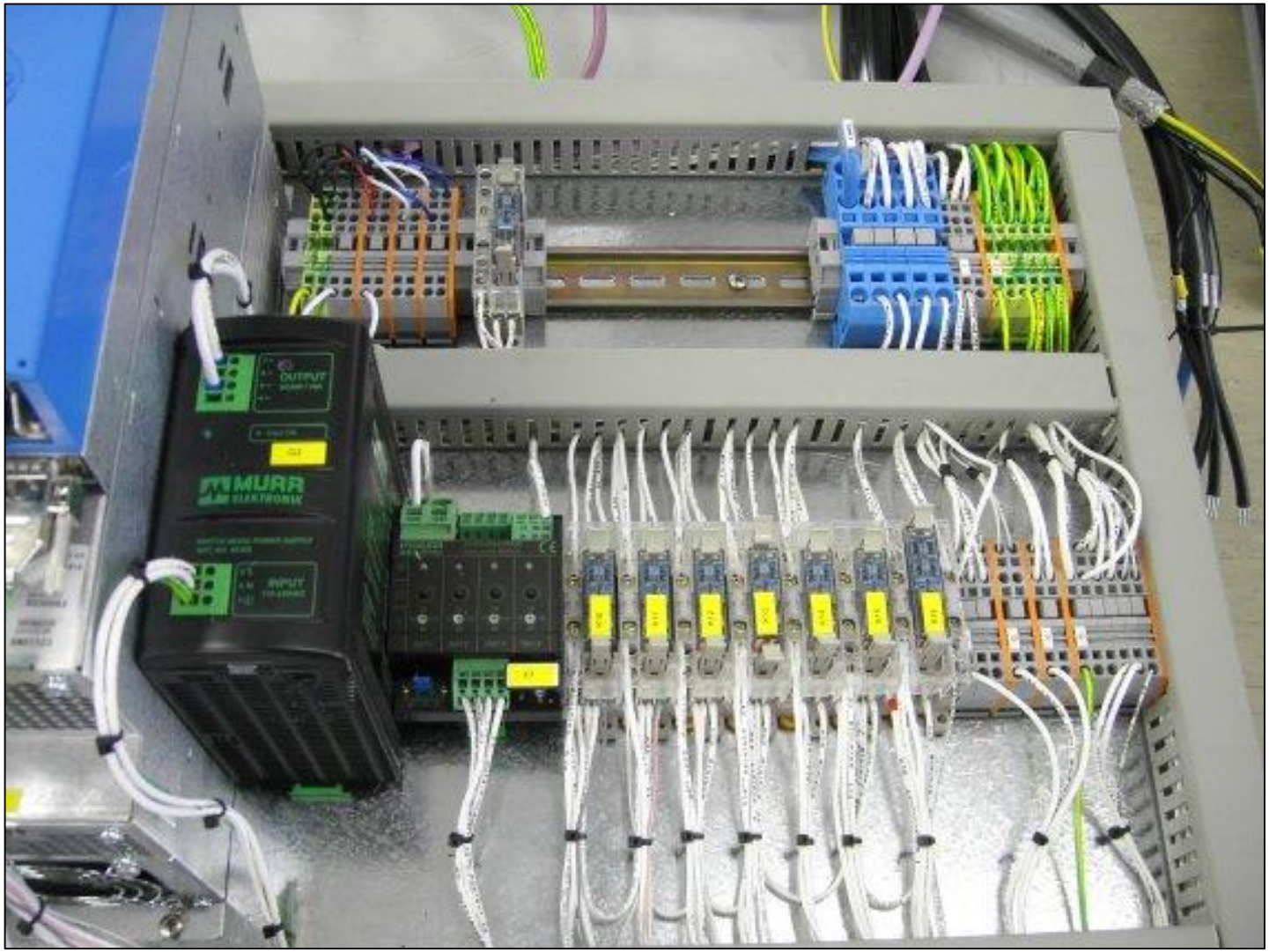
c) Array of 4 single strain gauges affixed with 90° misalignment (Wheatstone Bridge: The changes of strain resulting from the resistance change are detected. These are proportional to the applied loads)



Serviceability – easy to access to motor and motor control



Serviceability – standard components for motor control



PolyLab OS – Software Concept

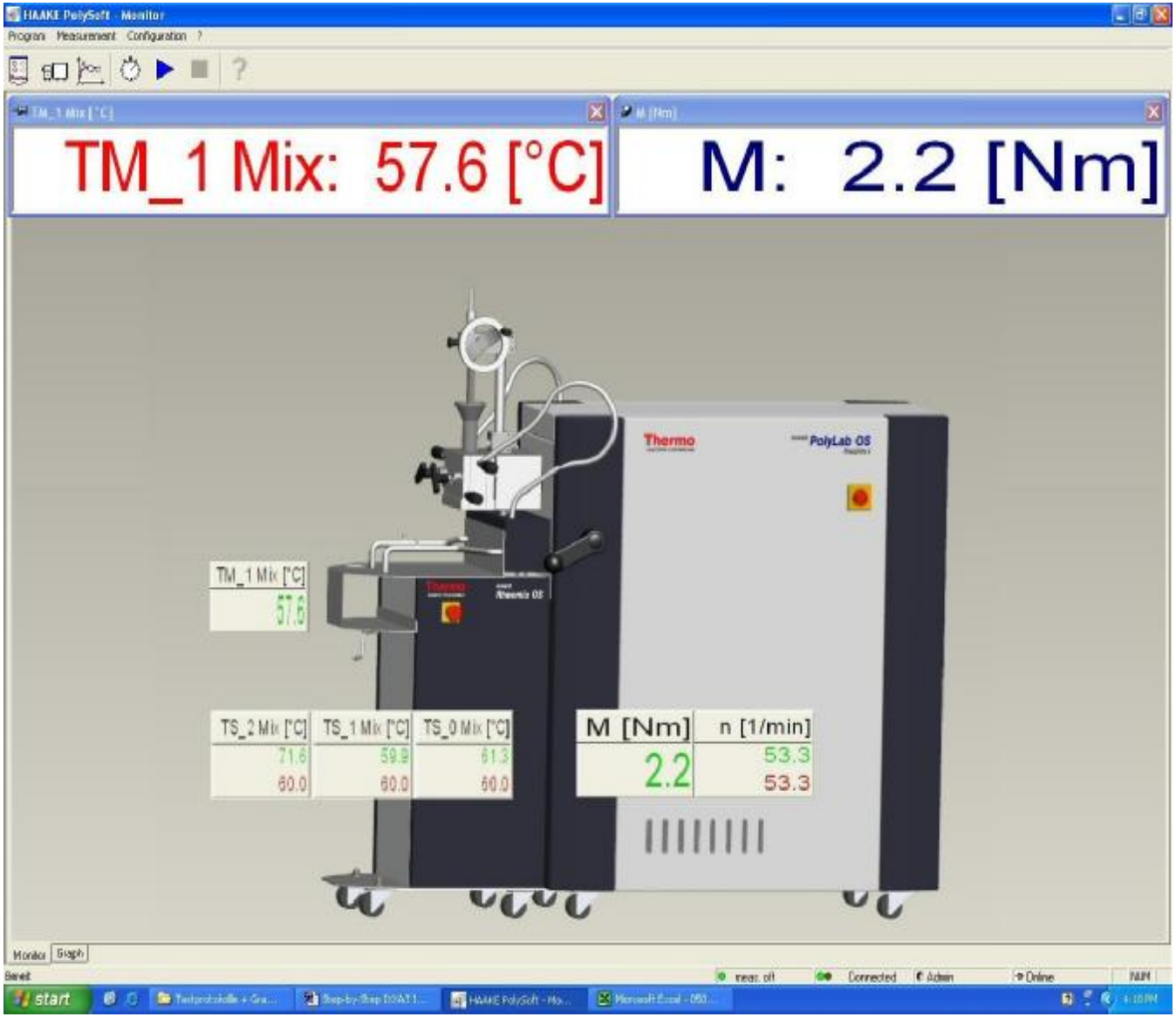
Two software layers:

1) **PolyLab Monitor OS:**
   - Basic software to control and monitor the status of PolyLab OS
   - Visualization of the attached equipment and sensors
   - Online data export in Excel (XP)

2) **PolySoft Modules:**
   - Application dedicated software modules
     (Mixer Test / Capillary-Rheology / Filter Test / Advanced Extrusion)
   - Job stream structure, incl. data evaluation (similar to Haake RheoWin)
   - The user is guided through the measurement
   - Different user levels as preparation for 21 CFR part 11

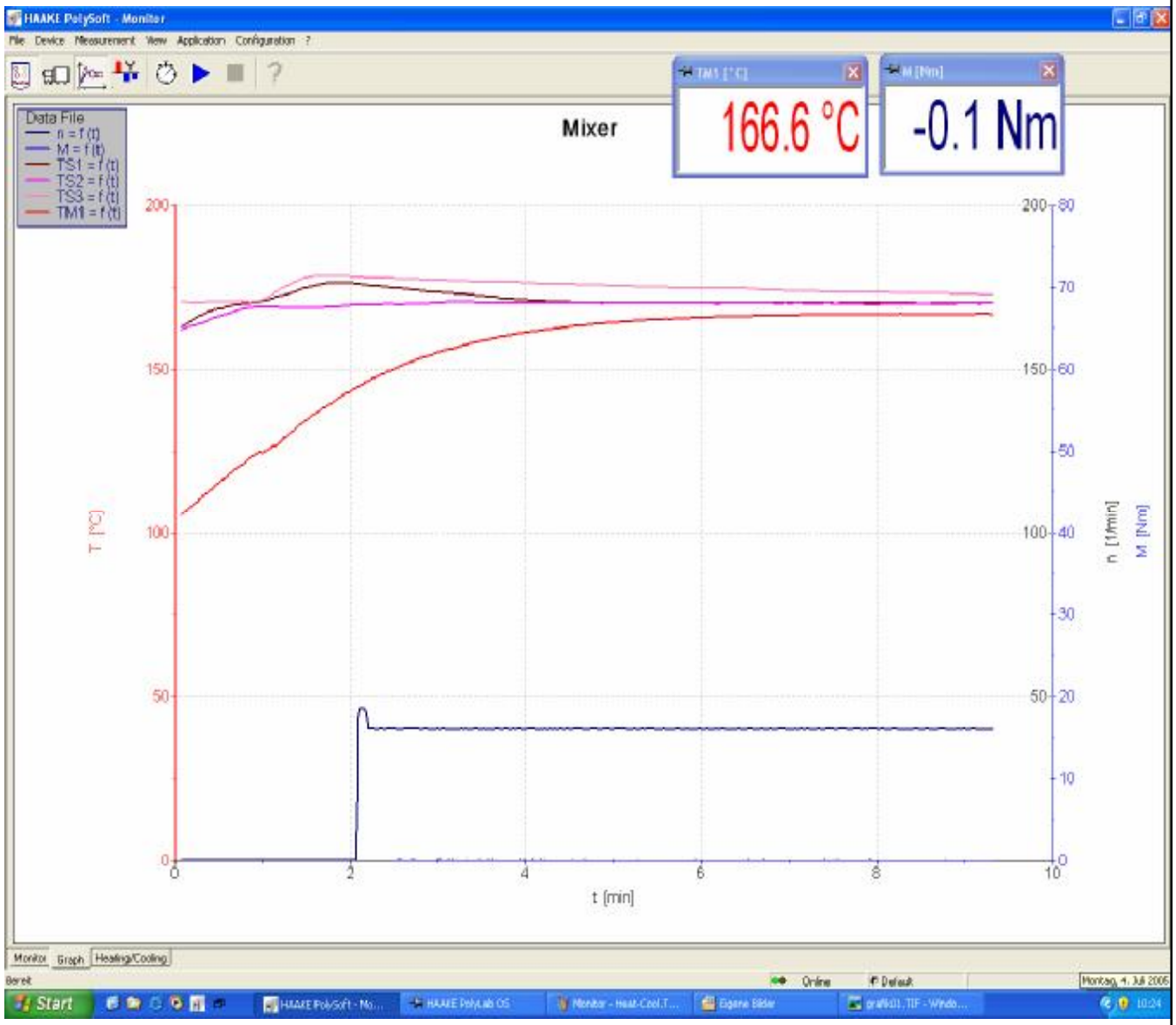
PolyLab Monitor OS

- Plug & Measure
- Easy Data Monitoring
- Scaleable windows for measured values
- Visualized Process Values
- Online export data to MS Excel (XP)
- Online Sensor recognition and “hot plugging”



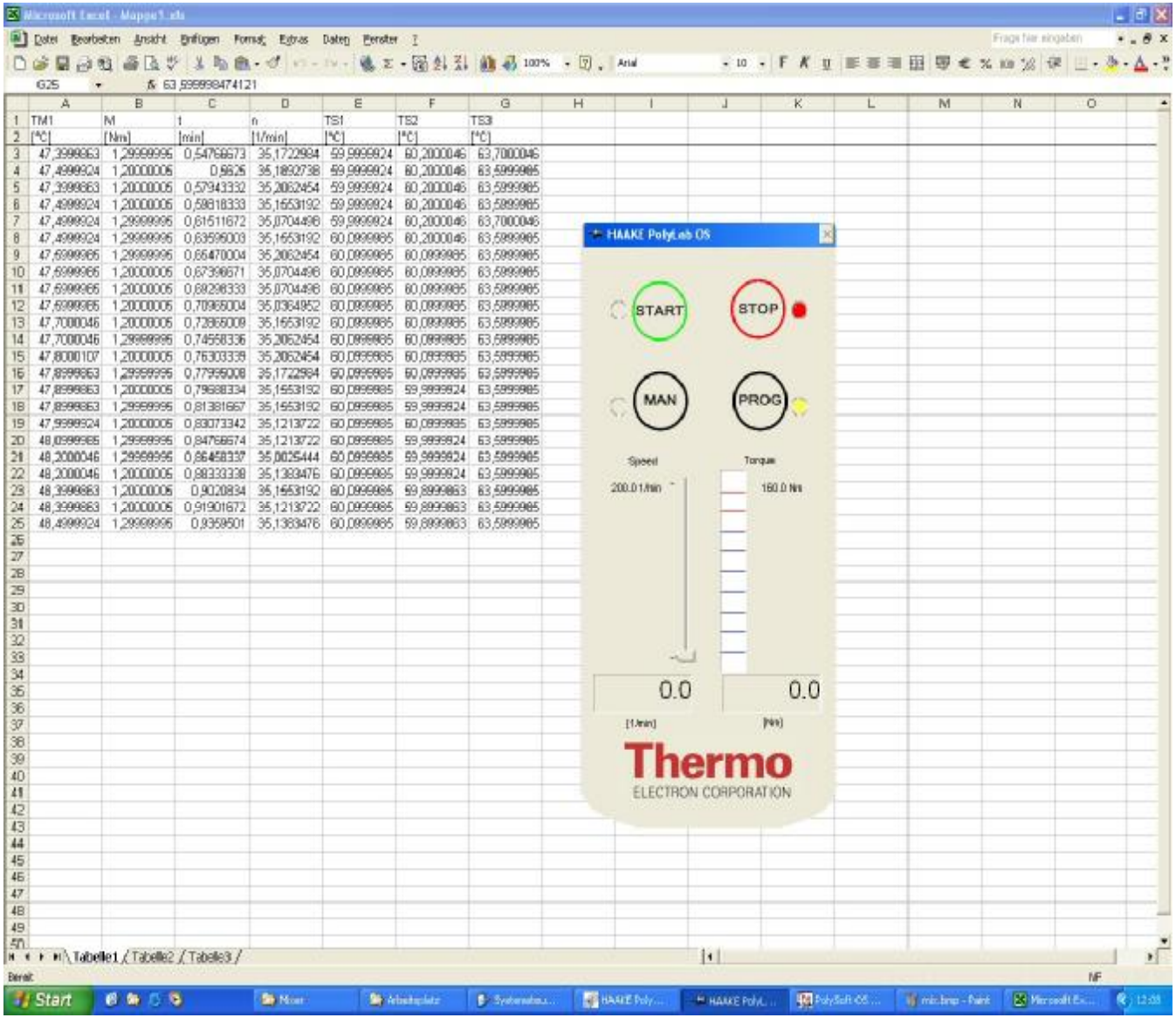
PolyLab Monitor OS

- Graphical monitoring of instrument and measured values
- Scaleable windows for test data



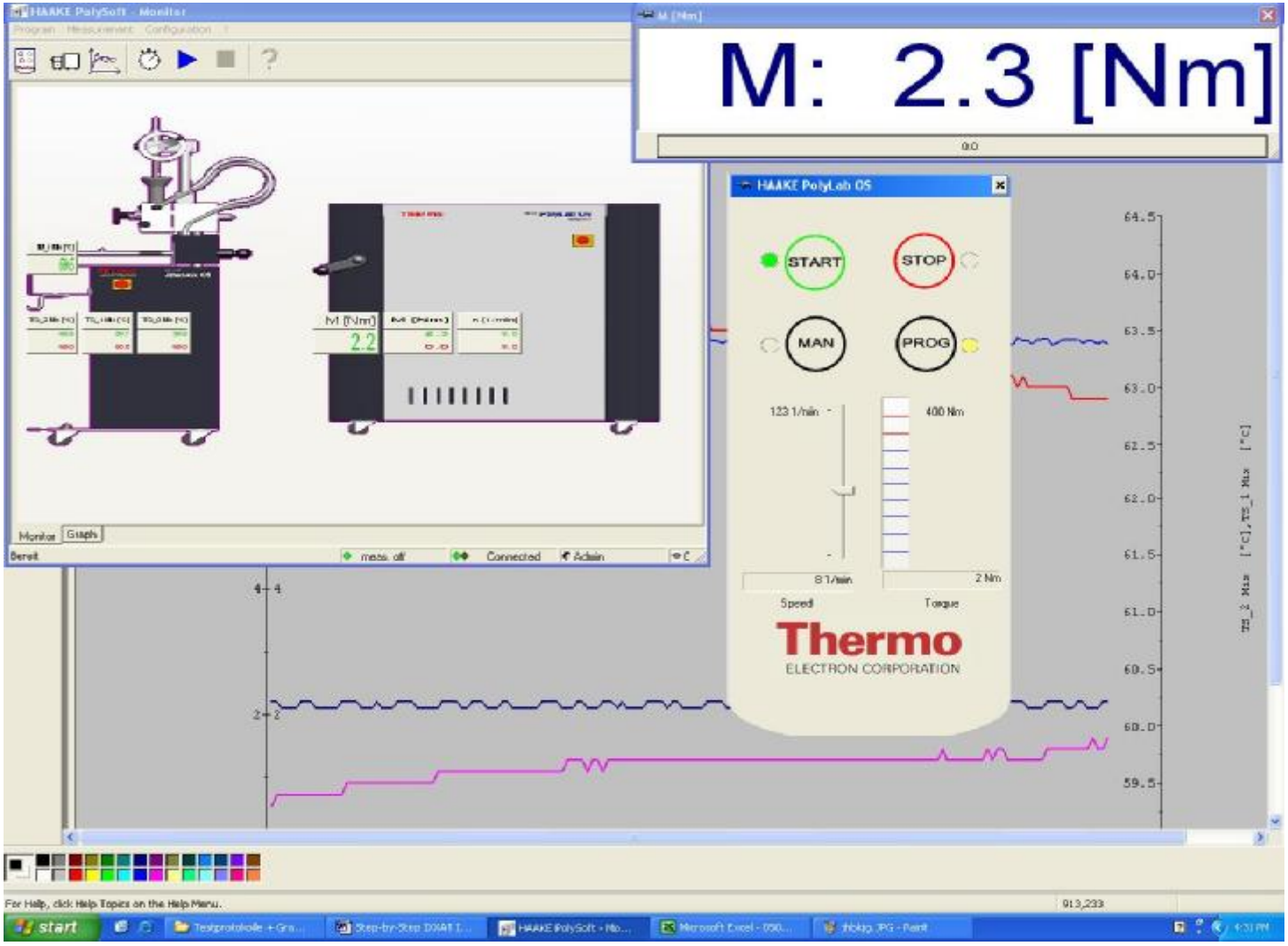
PolyLab Monitor OS

On-Screen remote control object to supervise the PolyLab even when using any other Windows based software



PolyLab Monitor Software

Flexible Window arrangement



Supporting a 30 year Legacy of Customer's Equipment

§ PolyLab OS Docking Station

- 4 Temperature Control Zones
- Excellent Upgrade Capability of existing equipment

PolyLab OS Docking Station

1990 Era RX600 Mixer

1980 Era TW100 TSE & Continuous Mixer

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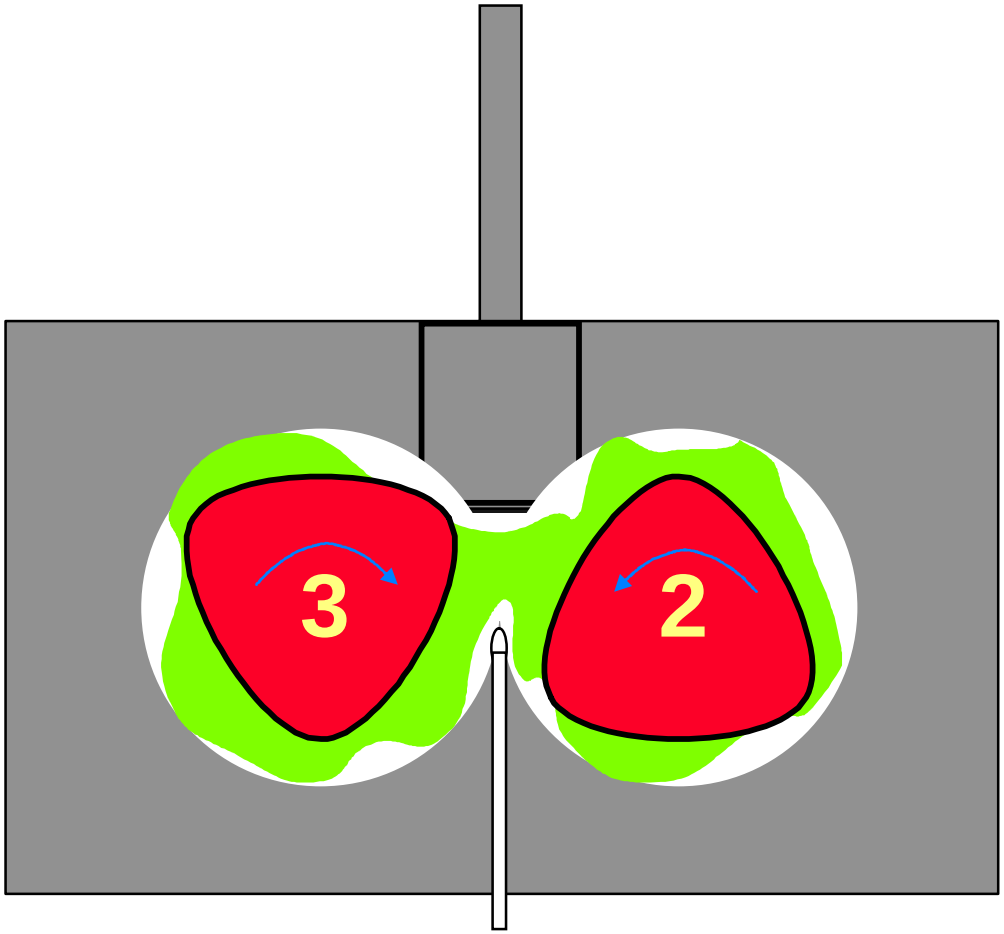
Mixer Testing

PolyLab OS with Mixer

Mixer Test

Mixer Cleaning

PolyLab Mixers- measuring principle



Shearing of a test sample

in a heated mixing chamber

with counter rotating rotors

3:2 speed ratio creates a

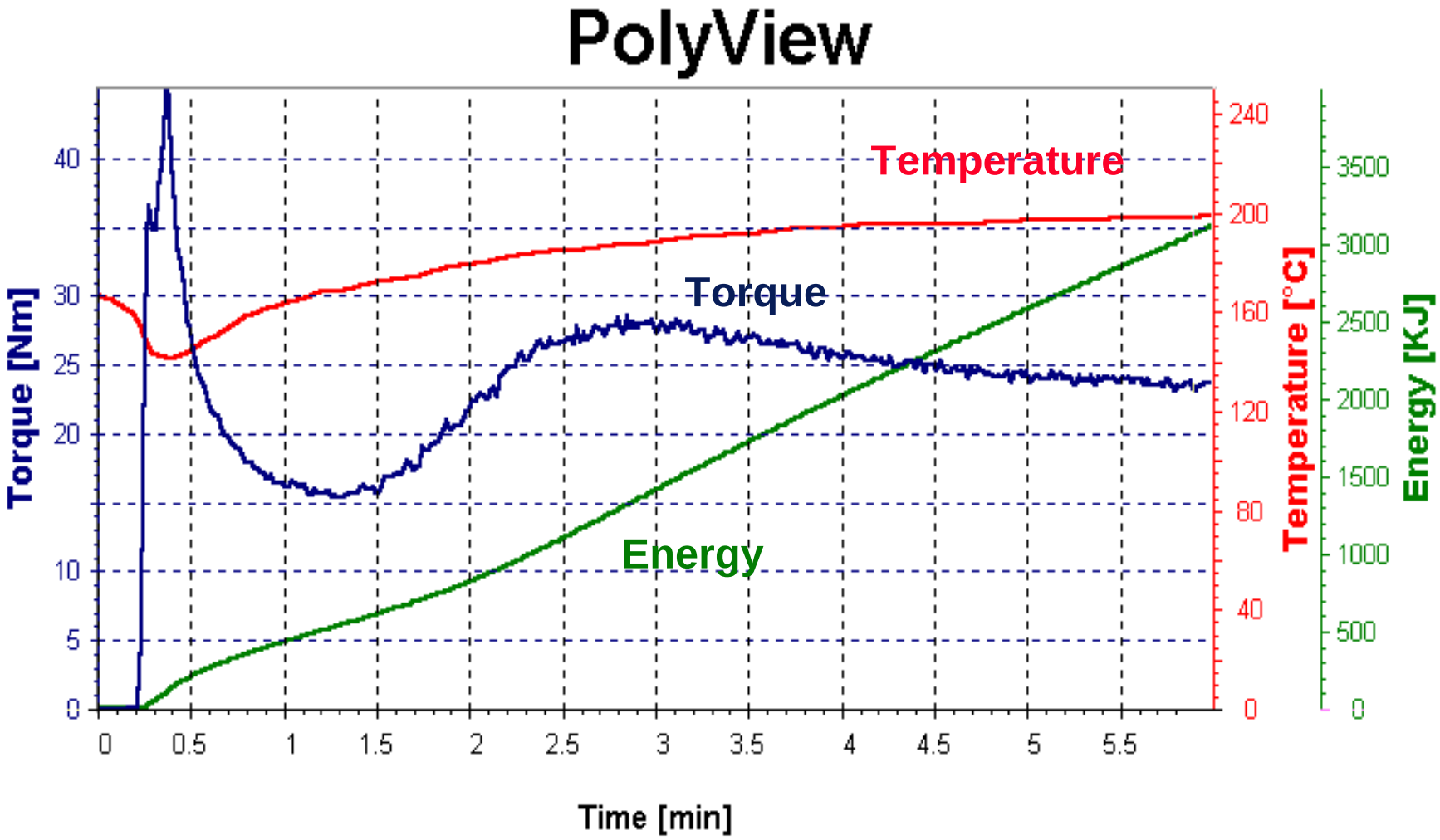
“figure 8” flow/shear field

<u>Test results:</u>

è Torque

è Melt temperature

Typical Mixer Rheogram



Mixer Models for various applications

Technical Specifications

Item	Rheomix 600	Rheomix 610	Rheomix 540	Rheomix 3000	Rheomix 3010	Planetmix OS
Chamber volume	120 cm^3	120 cm^3	130.6 cm^3	625 cm^3	625 cm^3	2500 cm^3
– with rotors	69-90 cm^3	69-90 cm^3	58-100 cm^3	310-541 cm^3	310-541 cm^3	2460 cm^3
Material	Stainless steel DIN 1.4301					Stainless steel
Gear ratio	3:2 (optional 2:3)					1:1
Max. speed	250 min^{-1}	250 min^{-1}	250 min^{-1}	250 min^{-1}	250 min^{-1}	200 min^{-1}
Max. torque	160 Nm	160 Nm	160 Nm	300 Nm	300 Nm	50 Nm
Max. temperature	400 °C (opt. 500 °C)	350 °C	400 °C (opt. 500 °C)	400 °C (opt. 500 °C)	350 °C	150 °C
Temperature control	3 zones electric heating and air cooling	Heated with thermal liquid, circulator required	3 zones electric heating and air cooling	3 zones electric heating and air cooling	Heated with thermal liquid, circulator required	Heated with thermal liquid, circulator required

- RheoMix 540 designed for ThermoSet (conical chamber for de-molding
- PlanetMix designed for dry-blend absorption testing

Rotors for RheoMix Mixers

Mixer Rotors - Applications

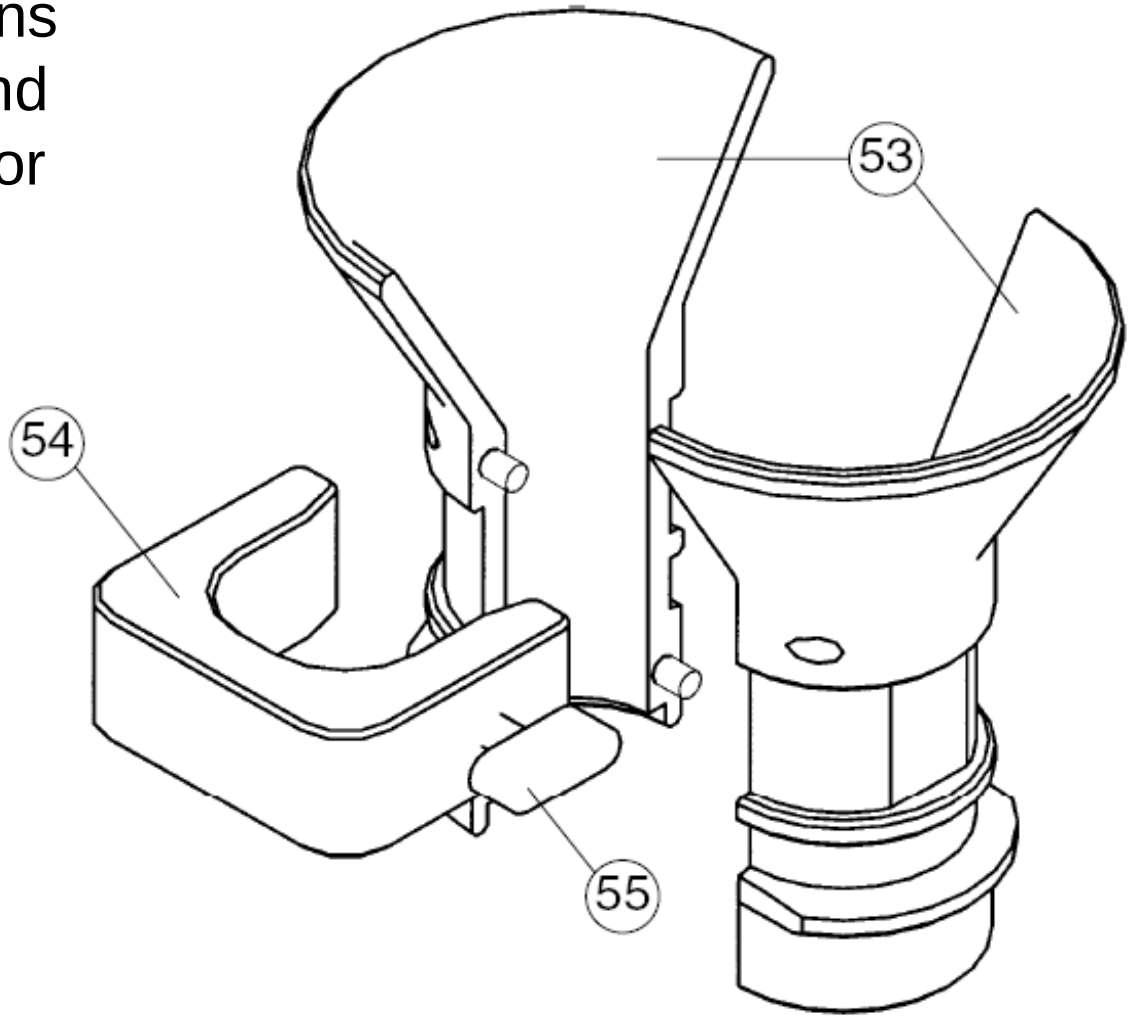
Roller Rotors	Highest Shear – simulates extrusion Most common for Thermoplastics, PVC
Banbury Rotors	Medium Shear – simulates Banbury type mixing specific to Rubber
Cam Rotors	Medium Shear – less axial distribution Elastomers and Food (sticky, high torque)
Sigma Rotors	Low Shear – highest free volume Adsorption testing, Food applications

Simple Mixer Cleaning and Sample Collection

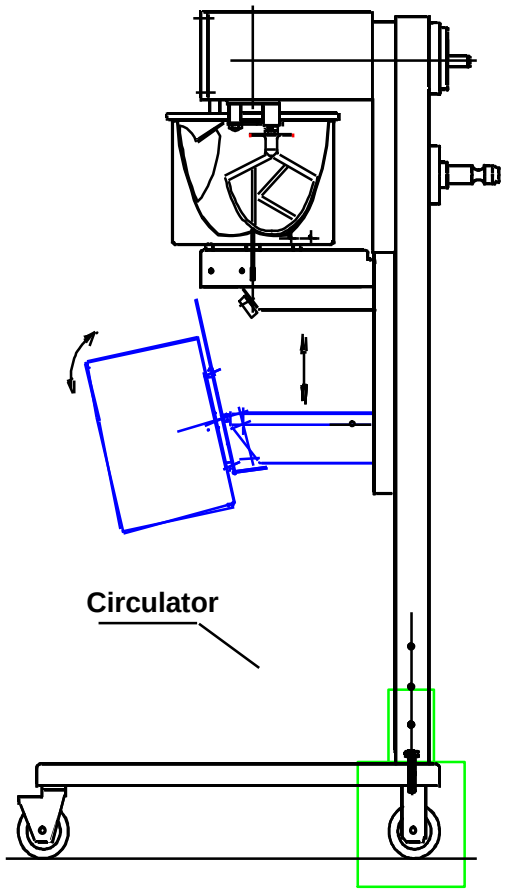
- Mixer Bowl in 3 sections for simplified cleaning
- Storage locations for all parts and sample collection pan

Removable Loading Funnel

- Removable Funnel lessens temperature variations and prevents funnel heating for subsequent tests



Planetary Mixer for PolyLab system



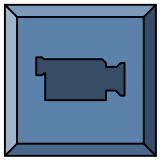
§ Applications:

- Determination of plasticizer absorption in vinyl chloride polymers (PVC-Dryblends)
- Mixing of solid powders with liquids
- Preparation of a PVC paste for testing purposes under controlled conditions

§ Standards:

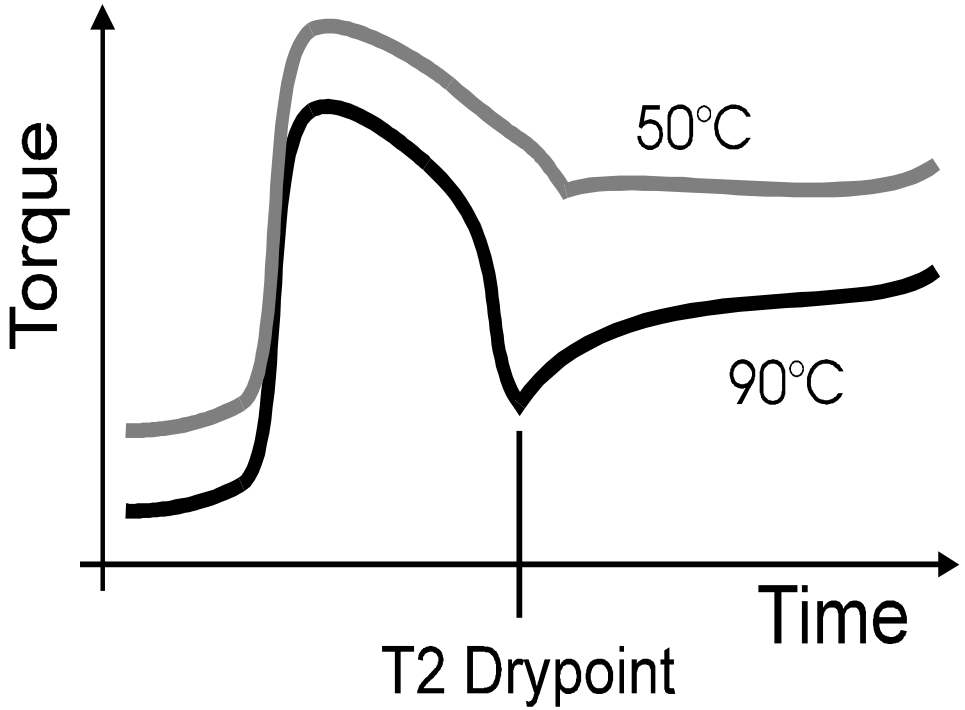
DIN 54 800 / DIN 54802 / ISO 4612

PlanetMix OS Video



Planetary Mixer curve - Plasticizer absorption

- Speed 100 rpm
- 400g PVC Powder
- 200g DOP Plasticizer
- Temperature variation



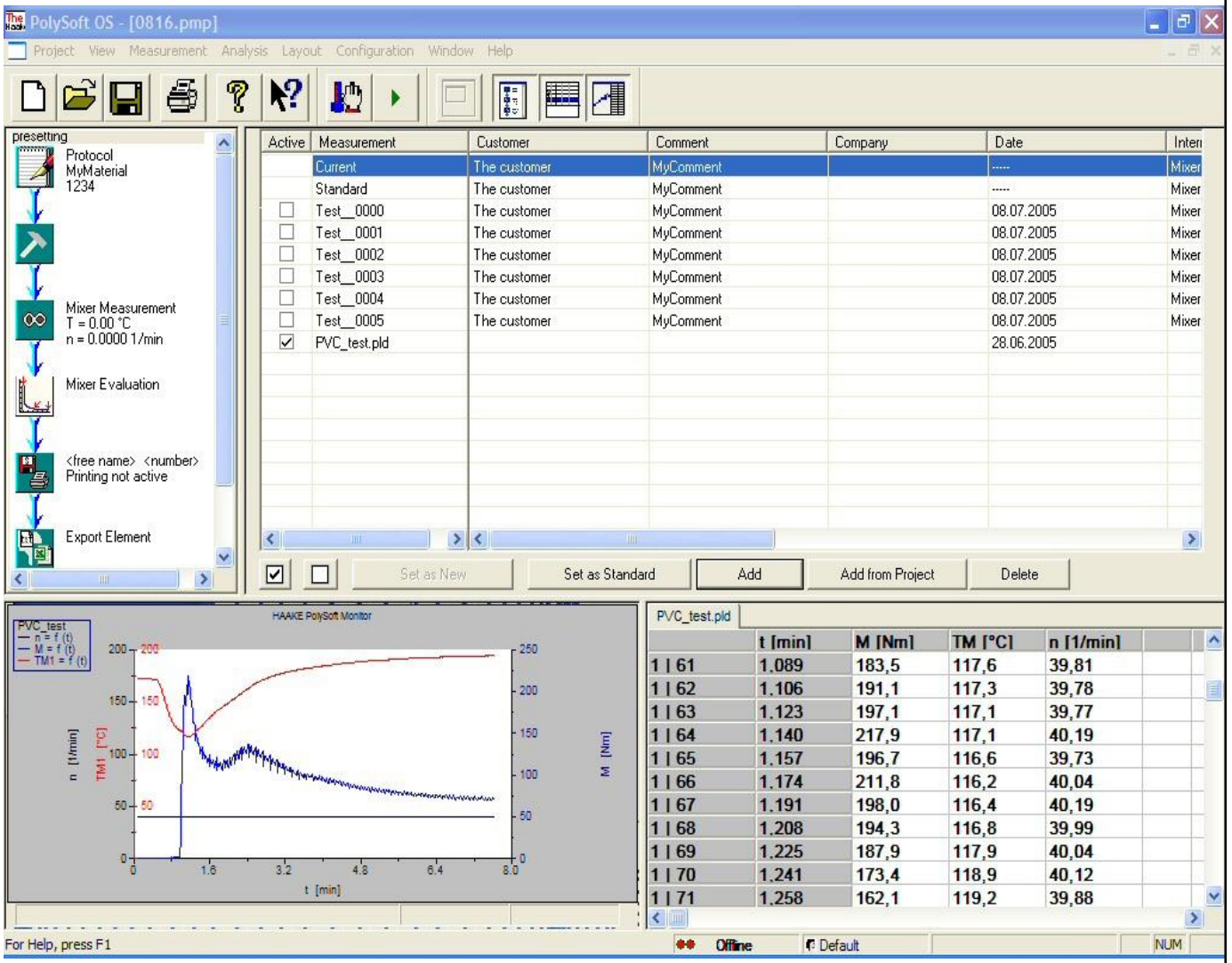
PolyLab OS – PolySoft Mixer

PolySoft Mixer SW:

- Job stream structure like Haake RheoWin

- The user is guided through the test

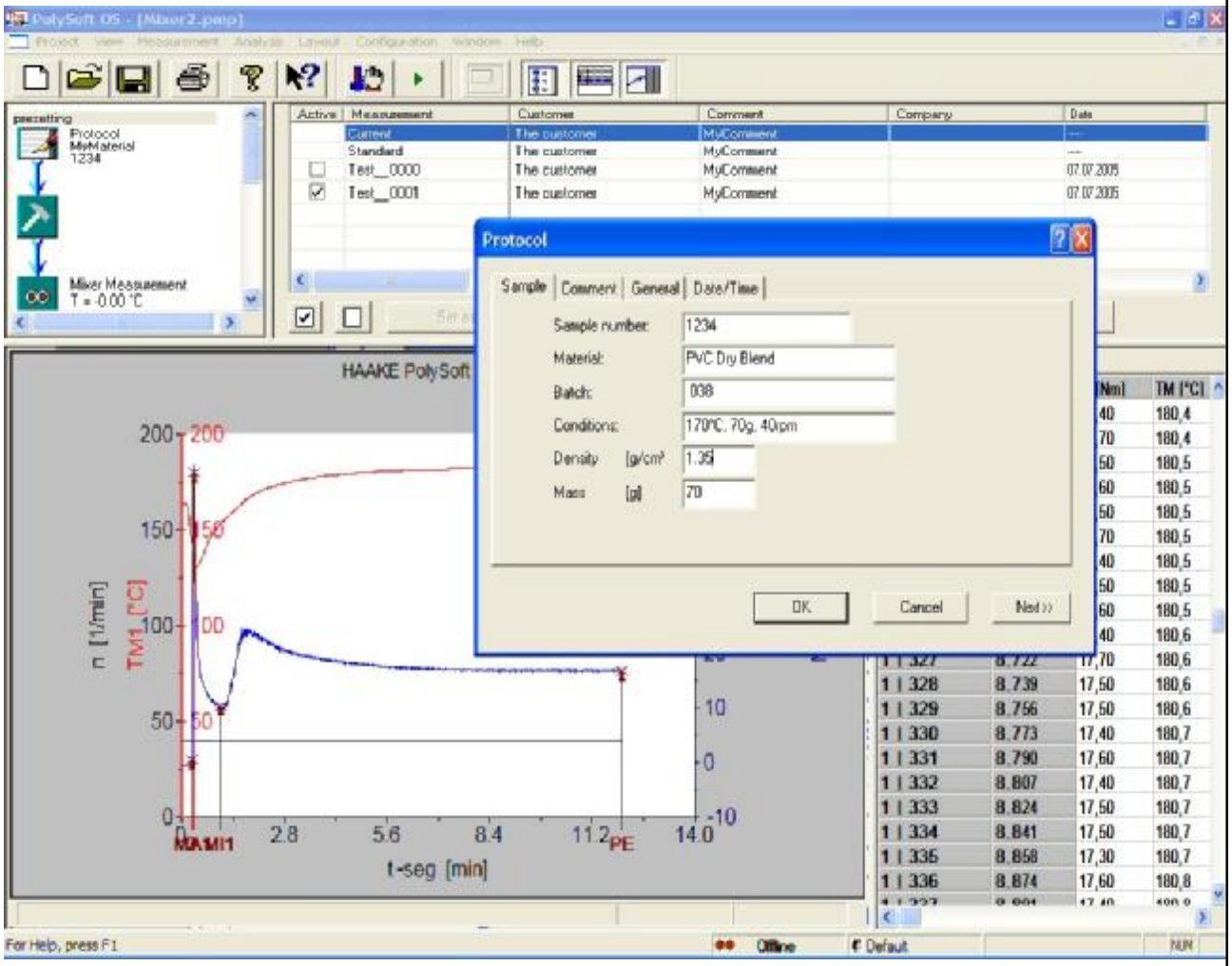
- Project handling as several tests can be saved in a project



PolyLab OS – PolySoft Mixer

Protocol Element:

- Documentation of sample information

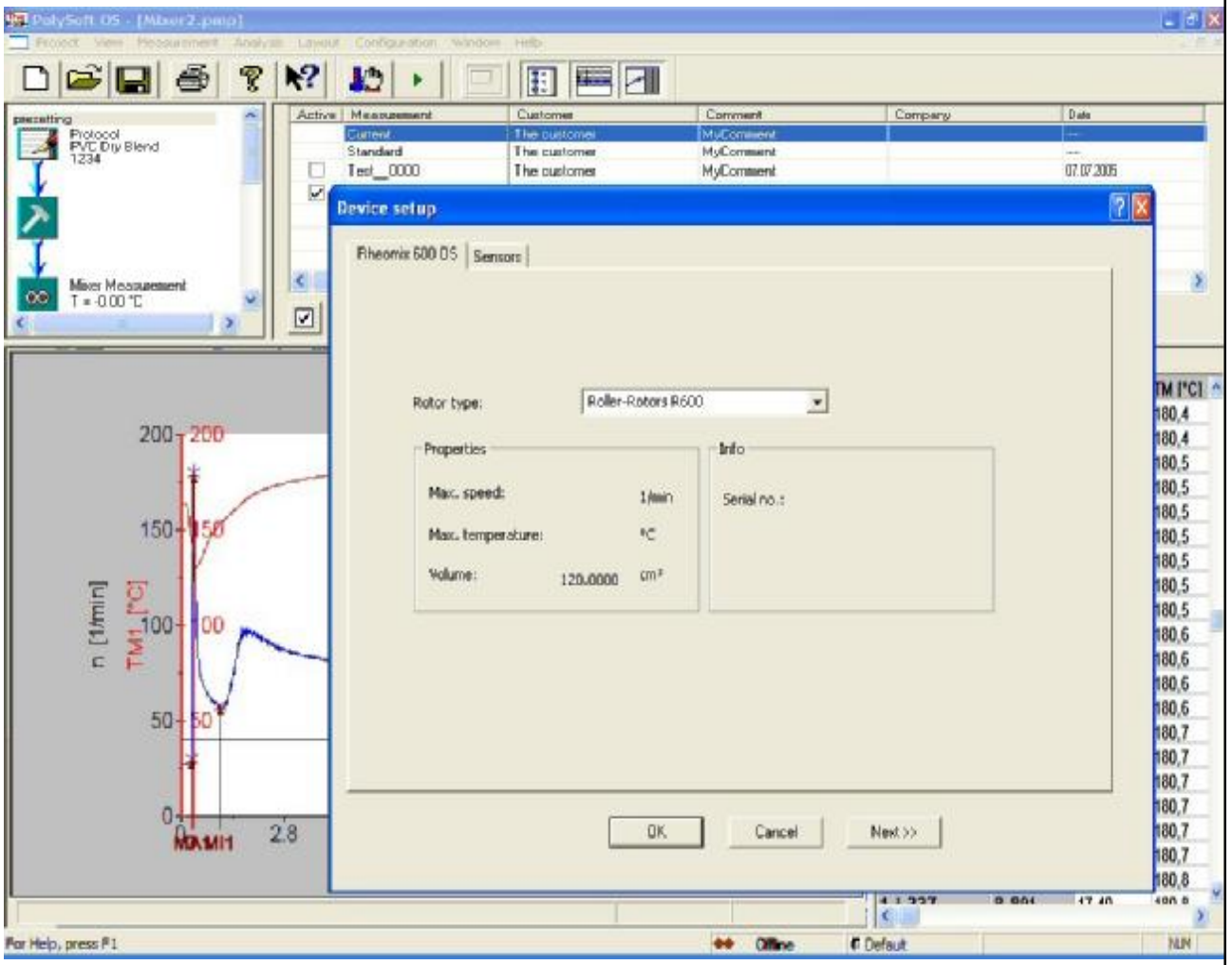


PolyLab OS – PolySoft Mixer

Device Setup:

- Automatic recognition of mixer setup

- Rotor selection

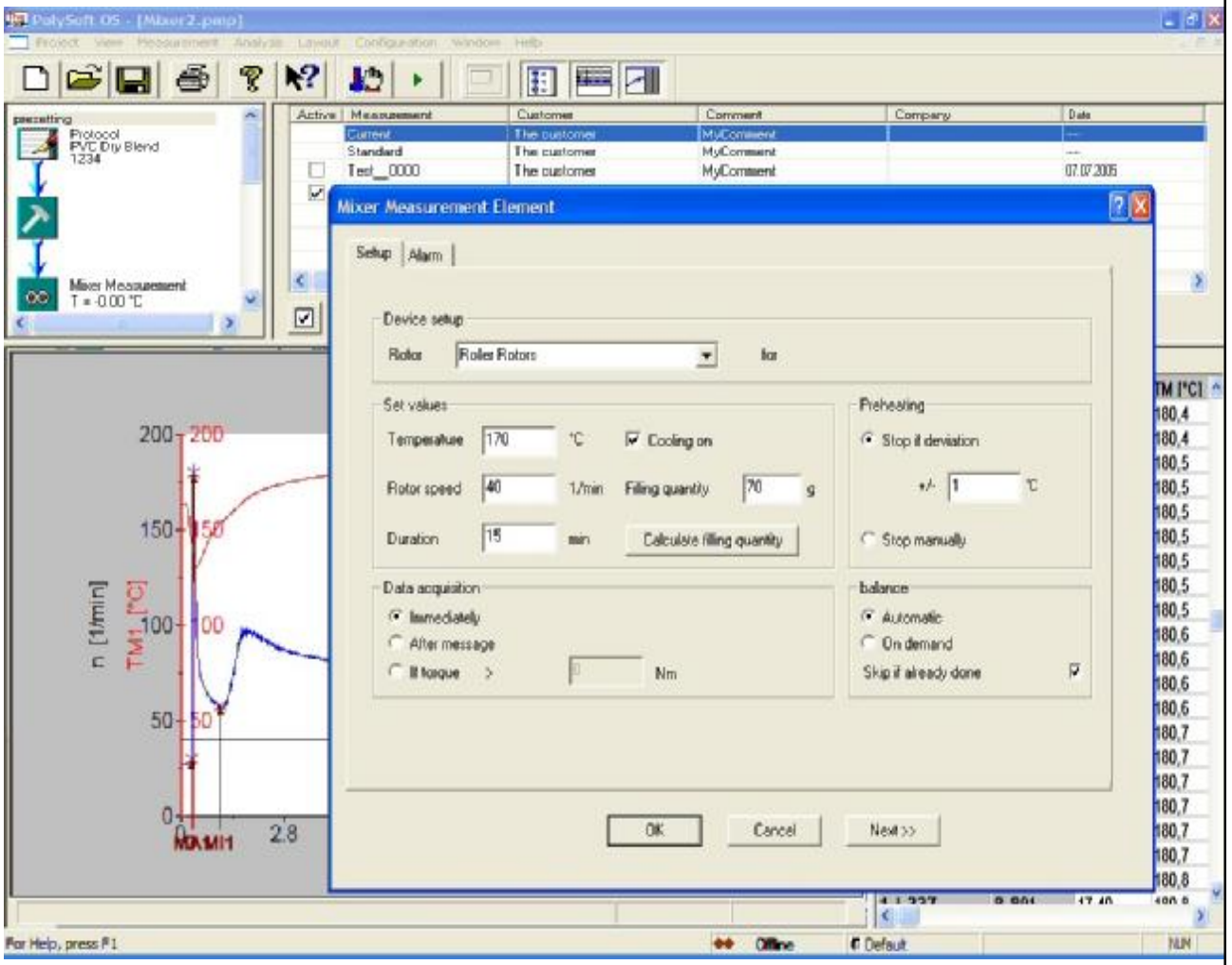


PolyLab OS – PolySoft Mixer

Measurement Element:

- Setting of the testing parameters

- Calculation of the sample-weight

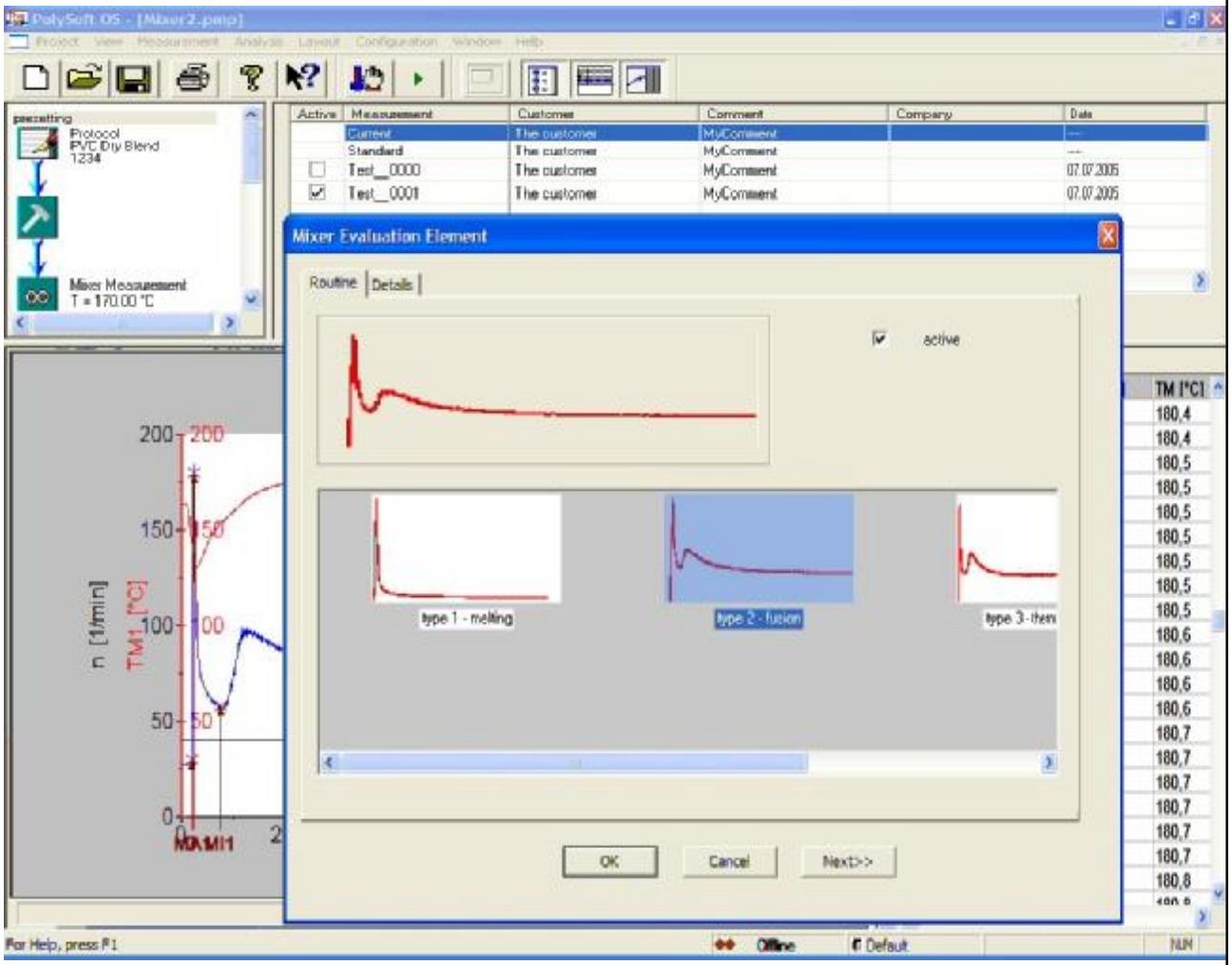


PolyLab OS – PolySoft Mixer

Evaluation Element :

- Selection of evaluation routine for the determination of the curve maxima, minima and gradients

- Modification of the evaluation routine

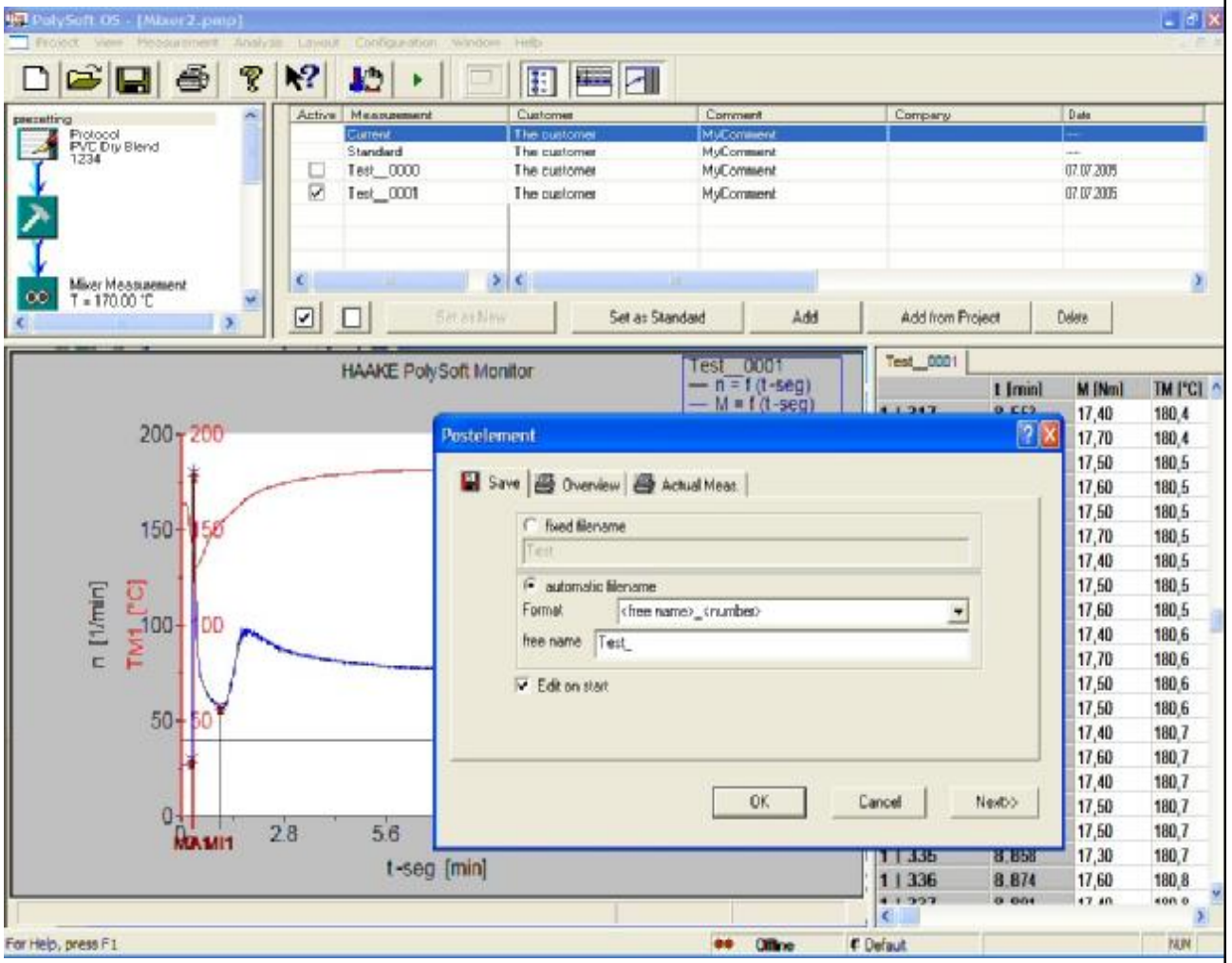


PolyLab OS – PolySoft Mixer

Post Element :

- Generation of file name

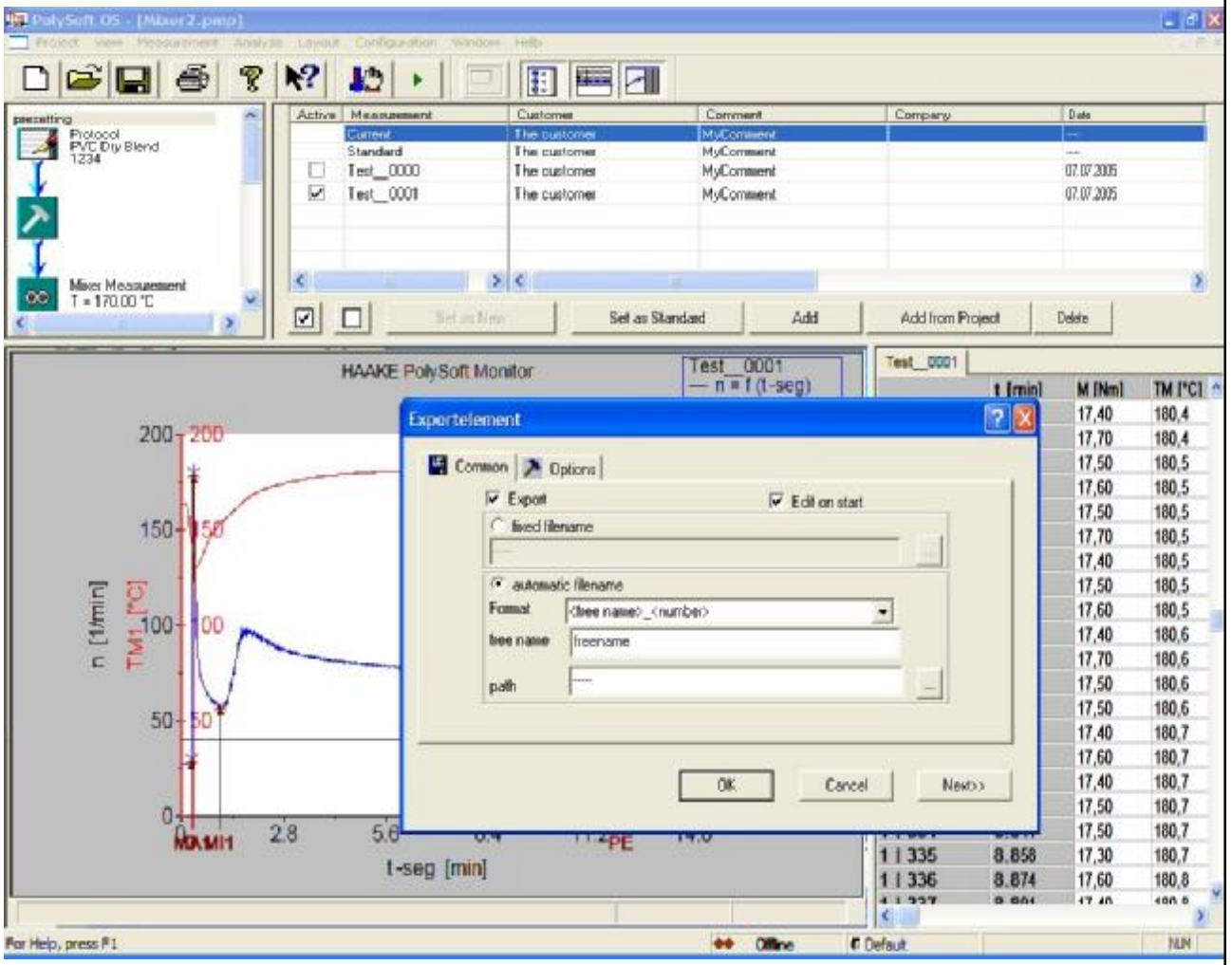
- Definition of printouts



PolyLab OS – PolySoft Mixer

Export Element :

- Definition of data export

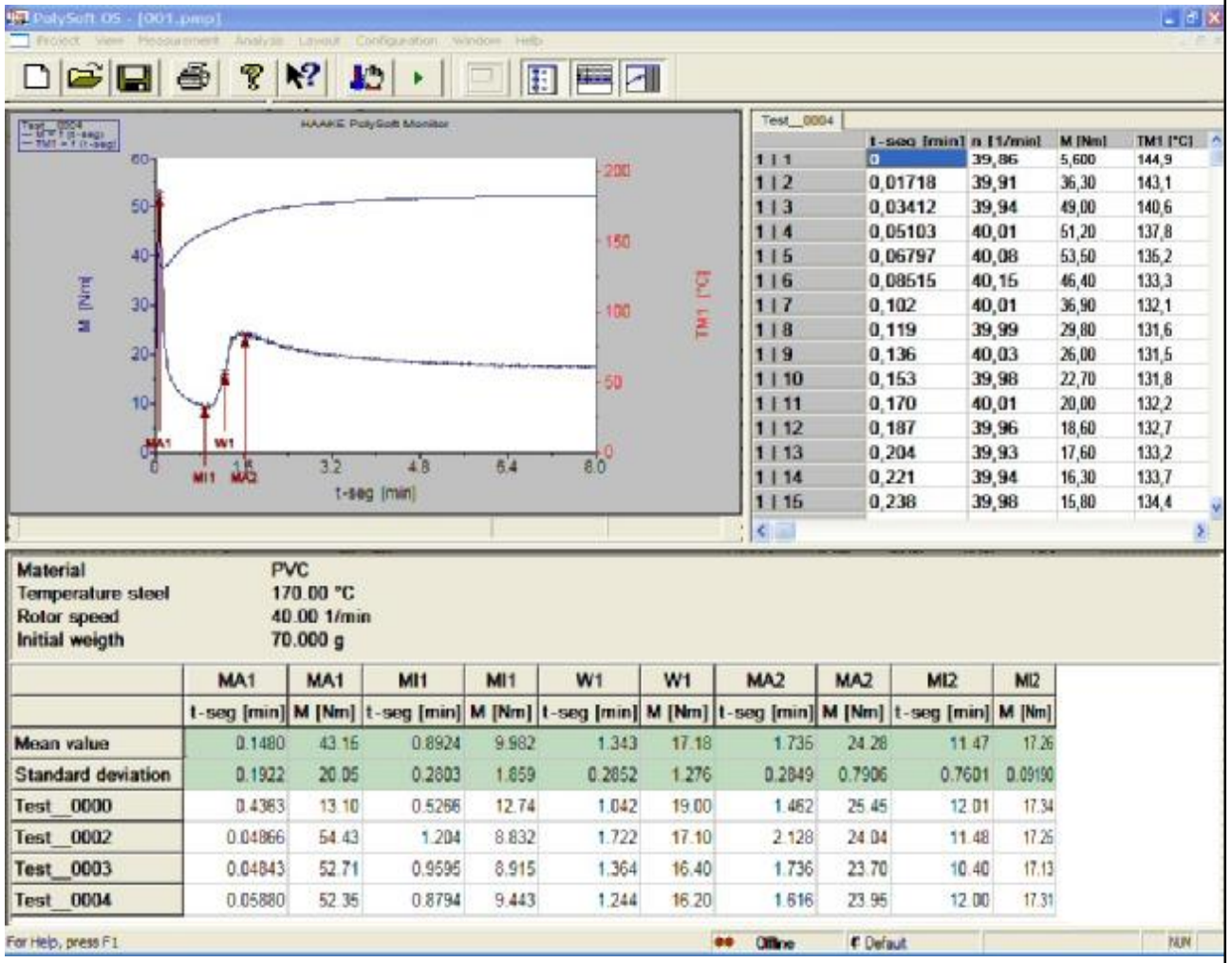


PolyLab OS – PolySoft Mixer

Statistic function :

- Statistical evaluation of measurement data

- Comparison of sample behavior over a longer time period



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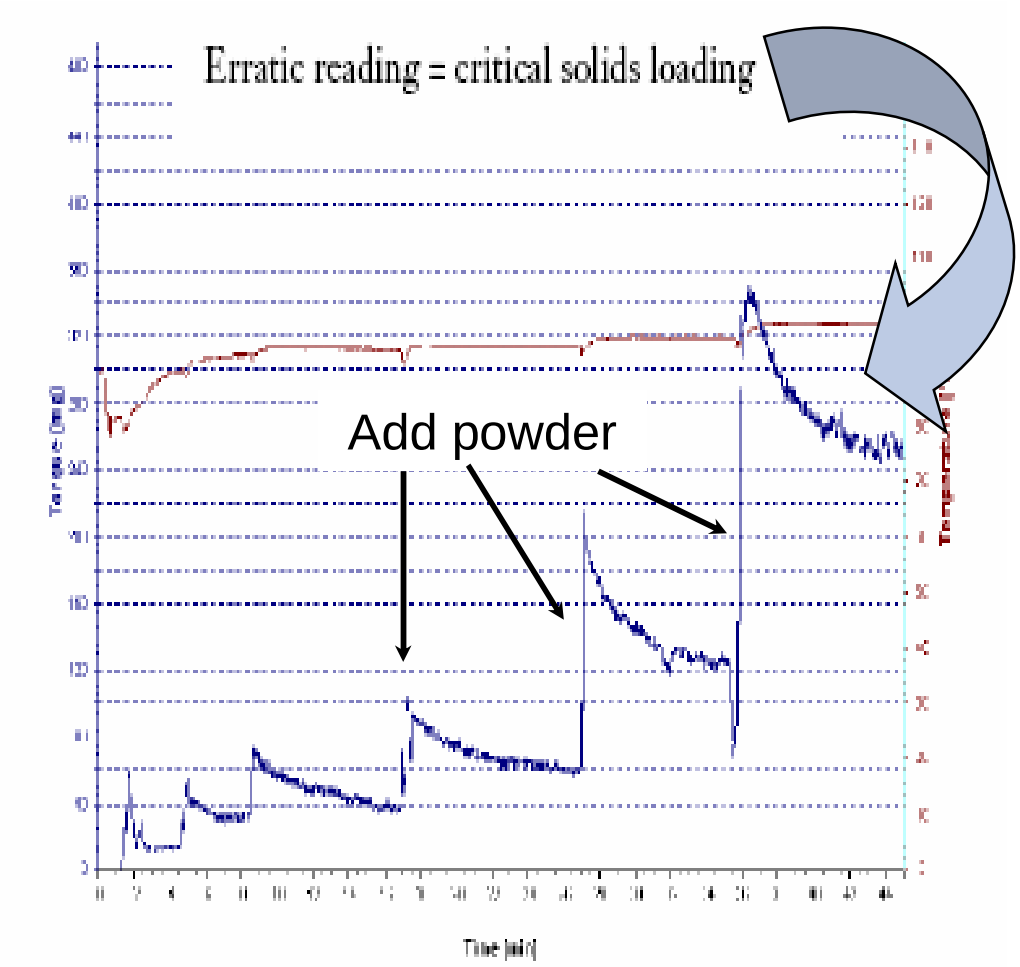
Mixer Application Examples

High resolution of new OS Torque Sensors



Ref. LR45-e, Joseph A. Krudys, ThermoHaake 2002

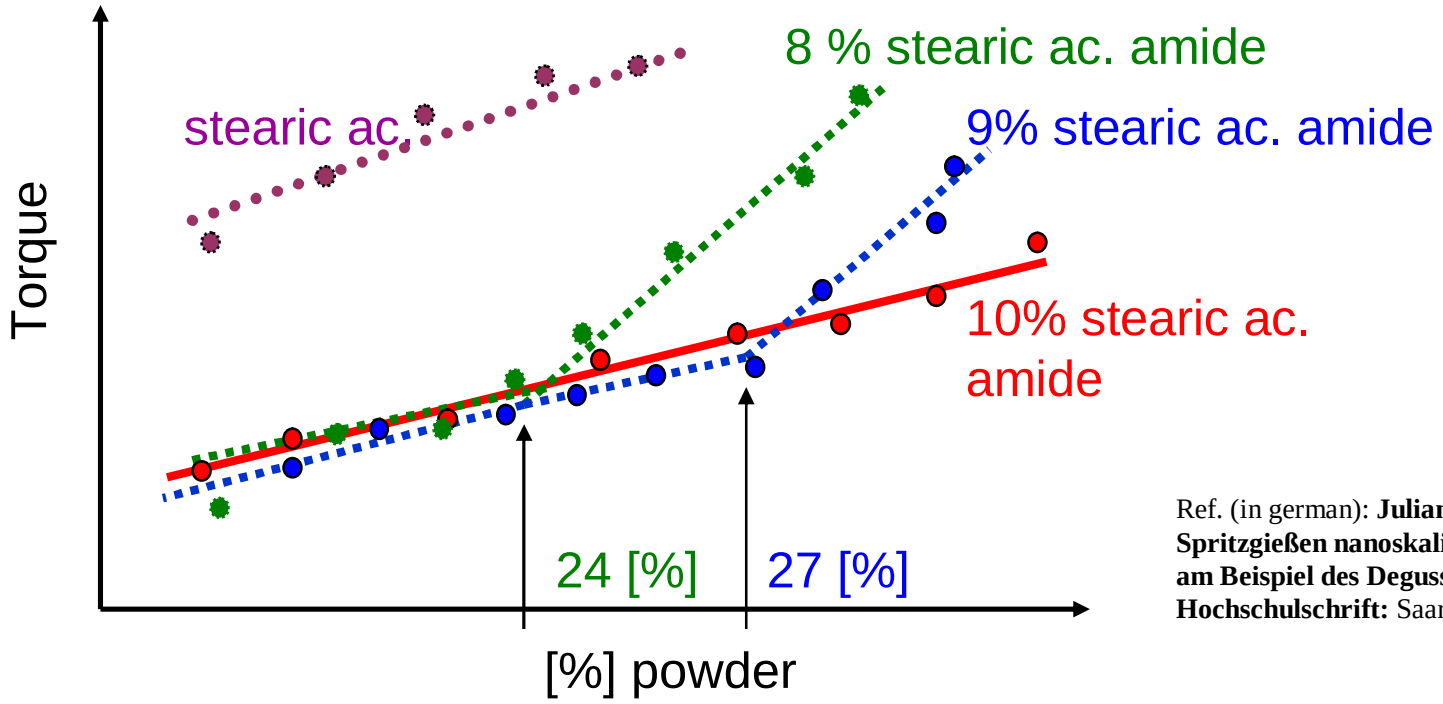
The erratic reading in the steps for increased powder load show the limiting powder [%] in the mixture.

Contact free and fast, high resolution torque sensors have a big advantage over old style “dynamometer” where the reaction of a heavy motor is measured, the erratic reading, here key feature is damped or smoothed

Nanoscale Ceramic Al_3O_2 Powders for PIM

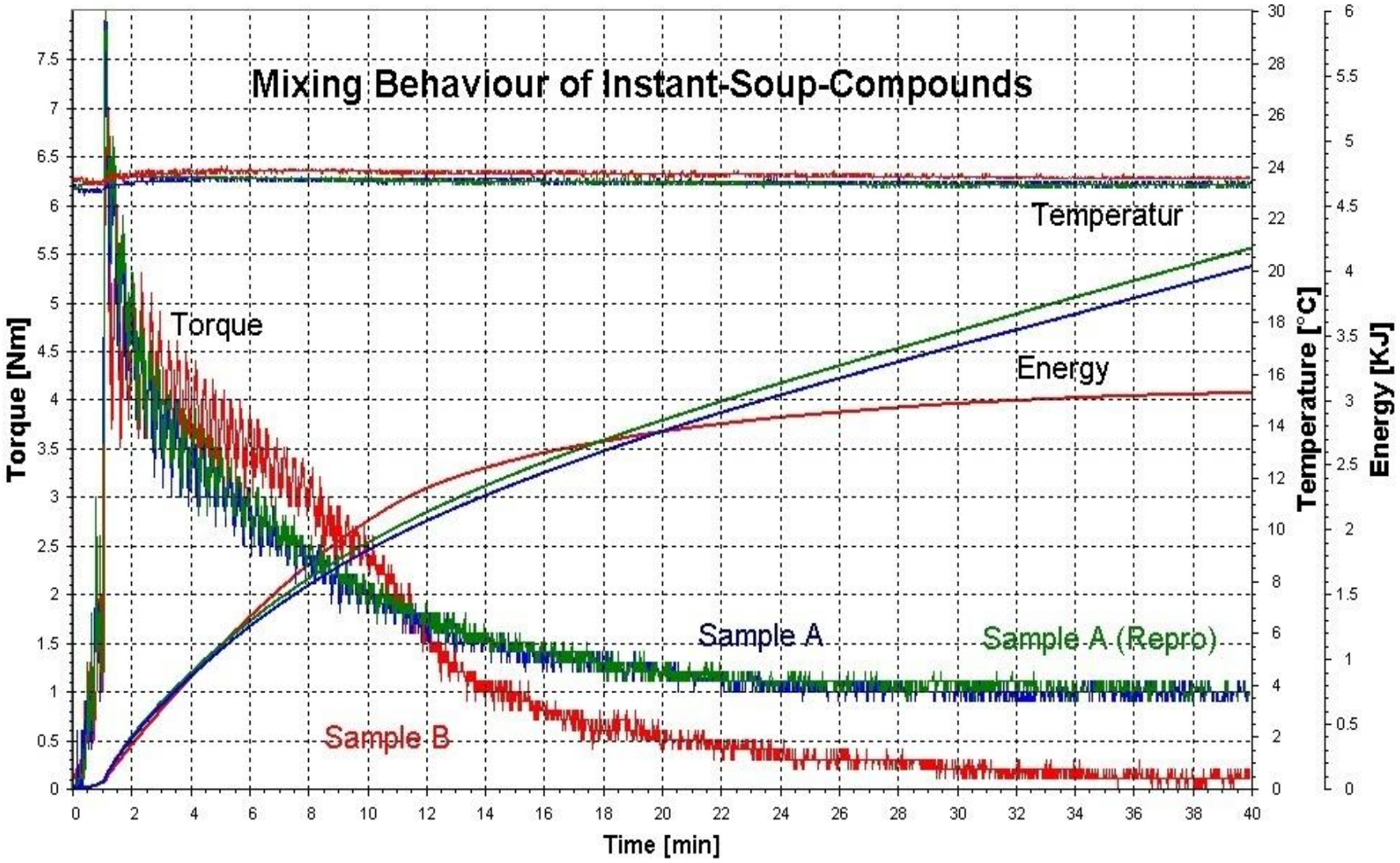
Juliane Kraus (Ref.) describes in detail how to optimize a binder system in a Torque Rheometer system (RX600, roller rotors).

- Type modifier (better: lower torque with stearic acid amide)
- Minimum content of this modifier (Torque depends only on [%]powder)
- batches in a larger scale are produced for the complete PIM process.

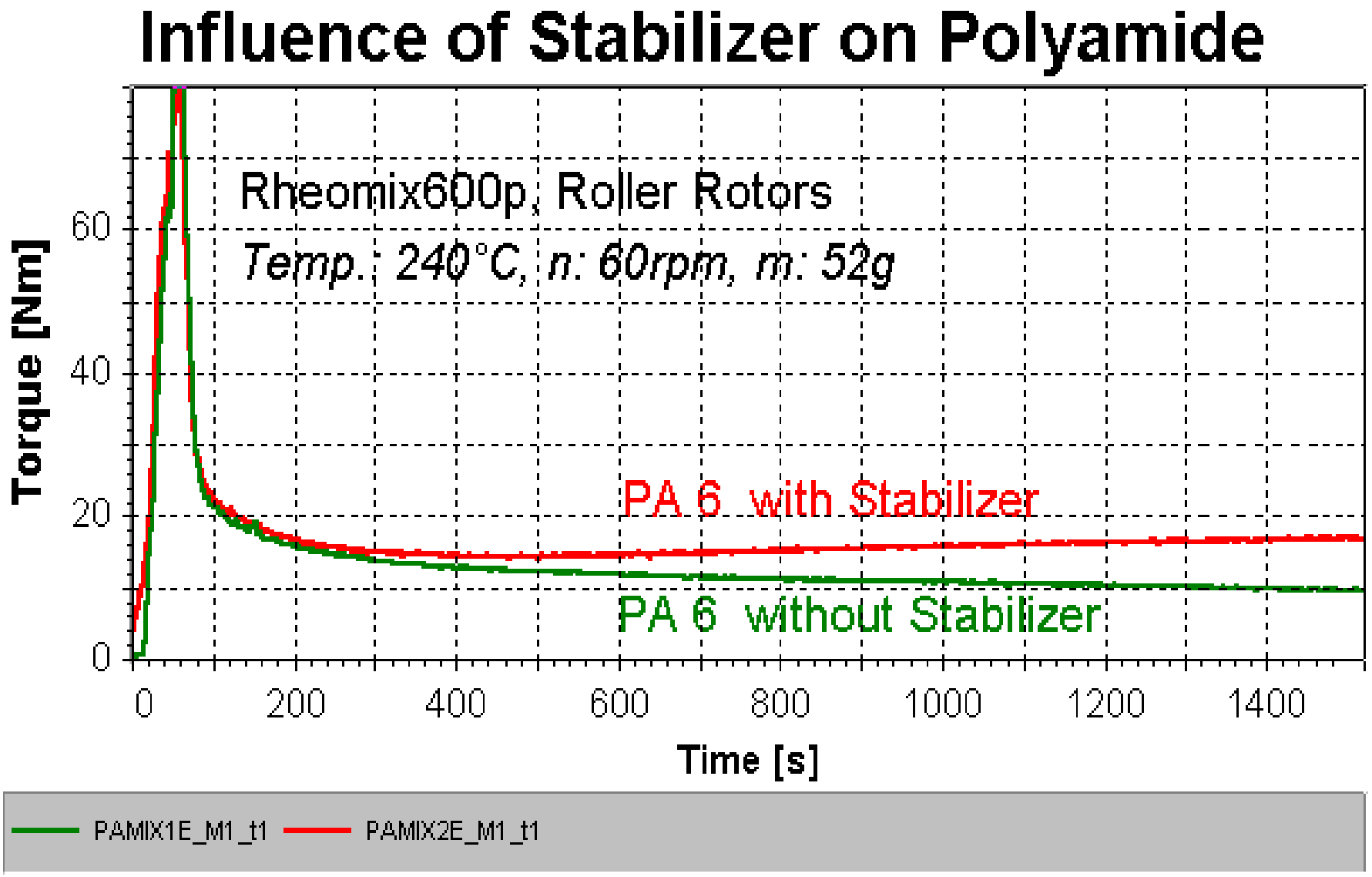


Ref. (in german): **Juliane Kraus**
Spritzgießen nanoskaliger keramischer Pulver am Beispiel des Degussa Aluminiumoxid C
Hochschulschrift: Saarbrücken, Univ., Diss., 1999

Application – Food (RheoMix 3010)

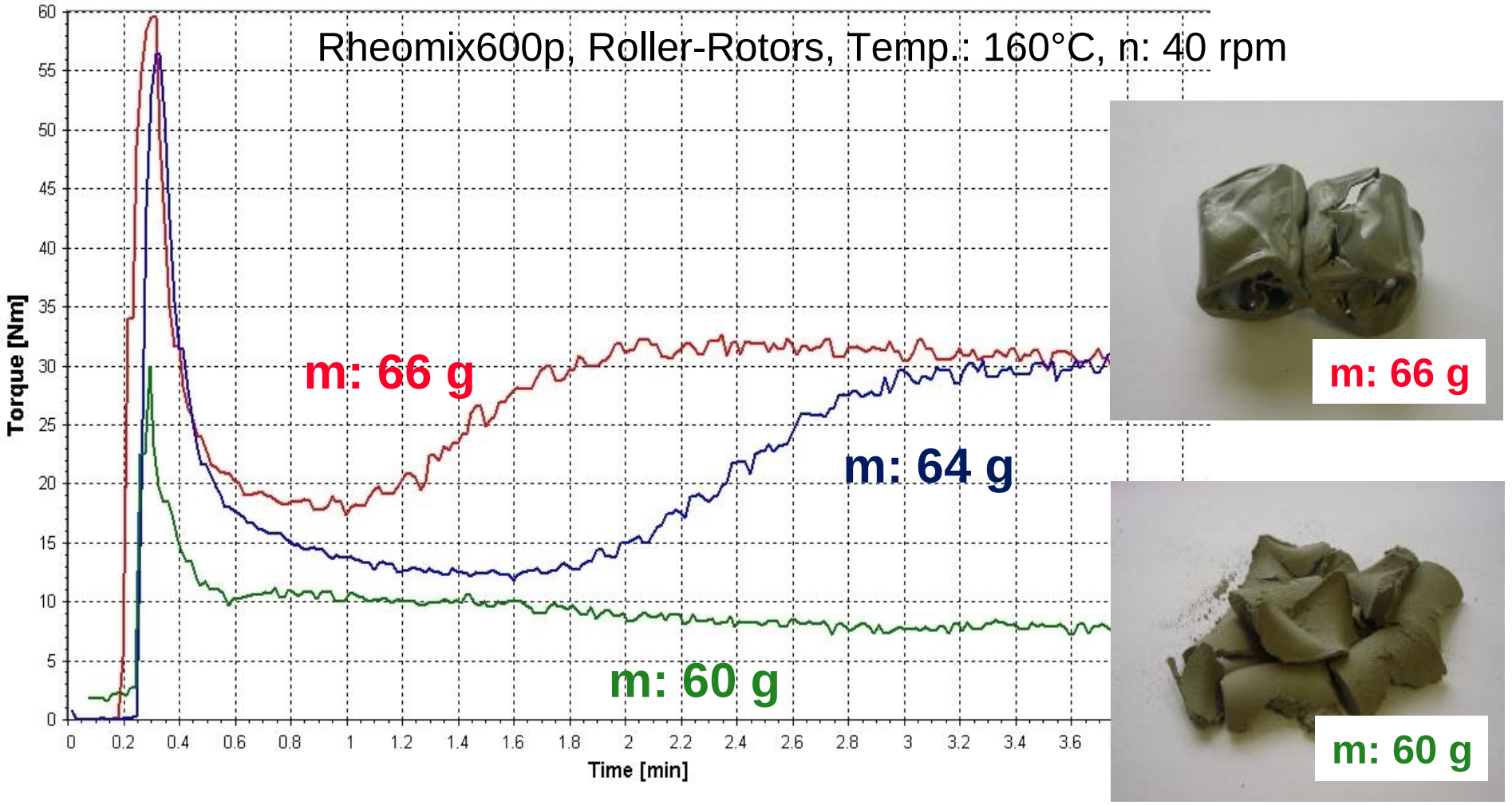


Application – Stabilizer effect in Thermoplastics

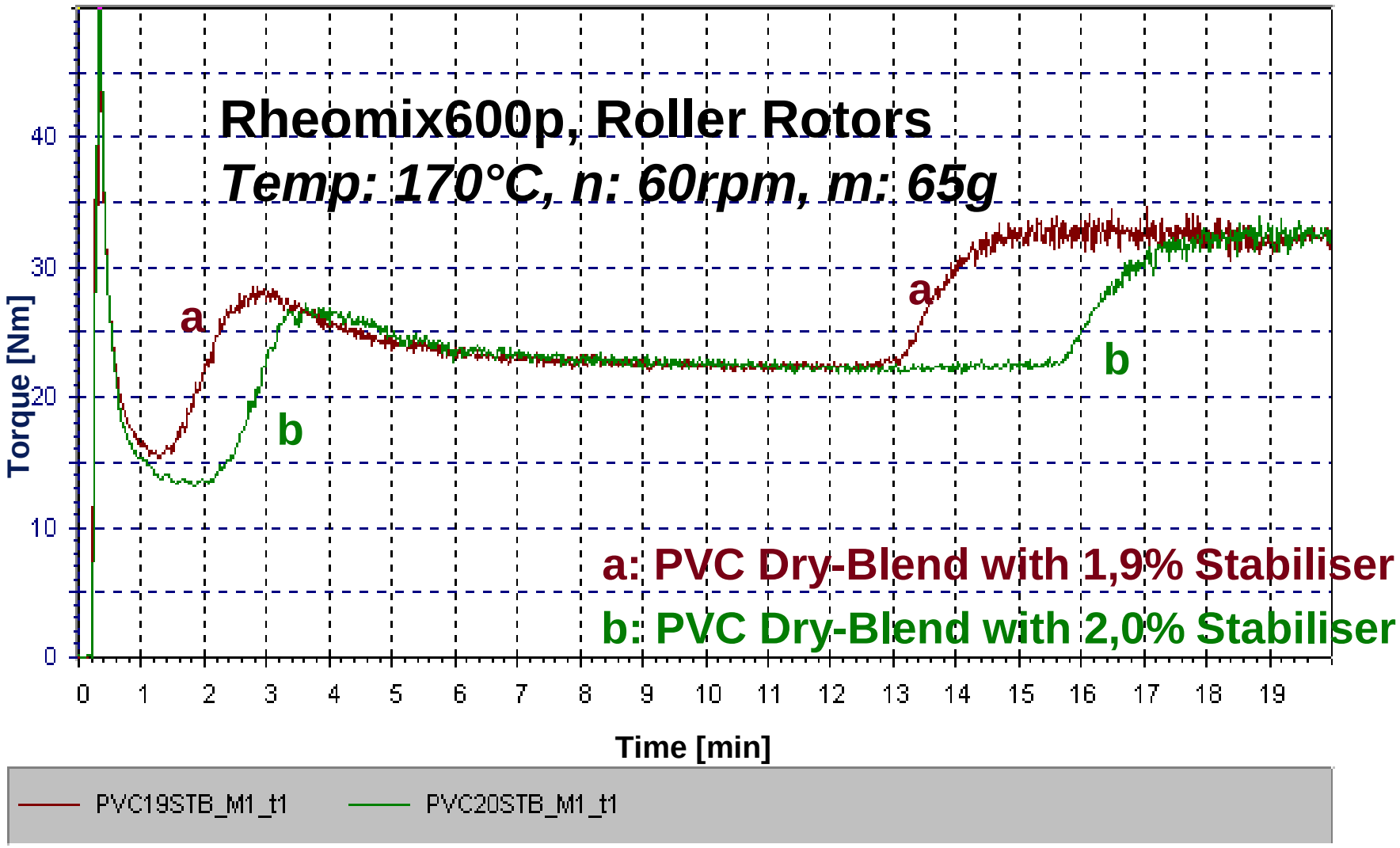


Application – PVC Fusion

Influence of sample weight on PVC fusion – adequate shear

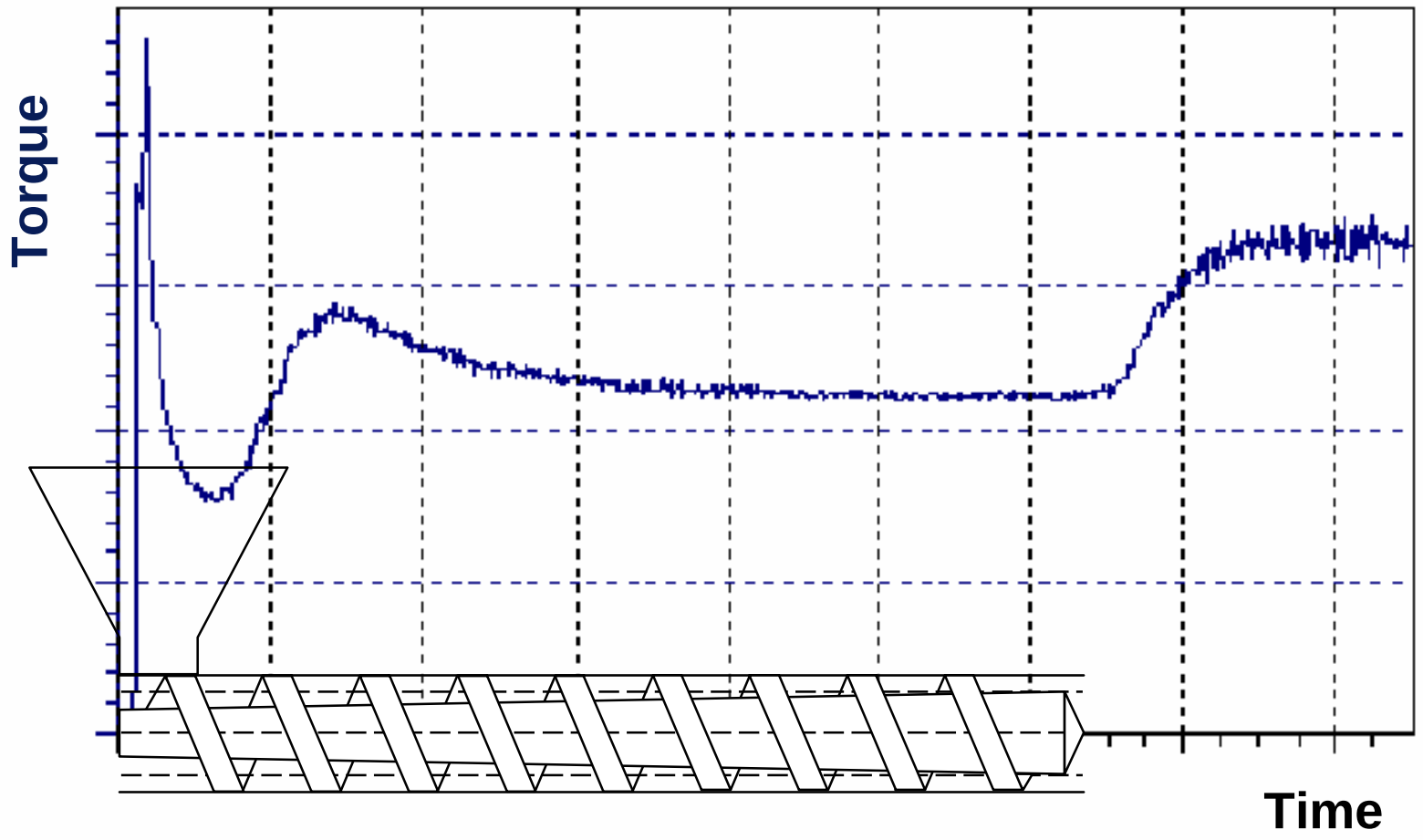


Application - PVC Stability test

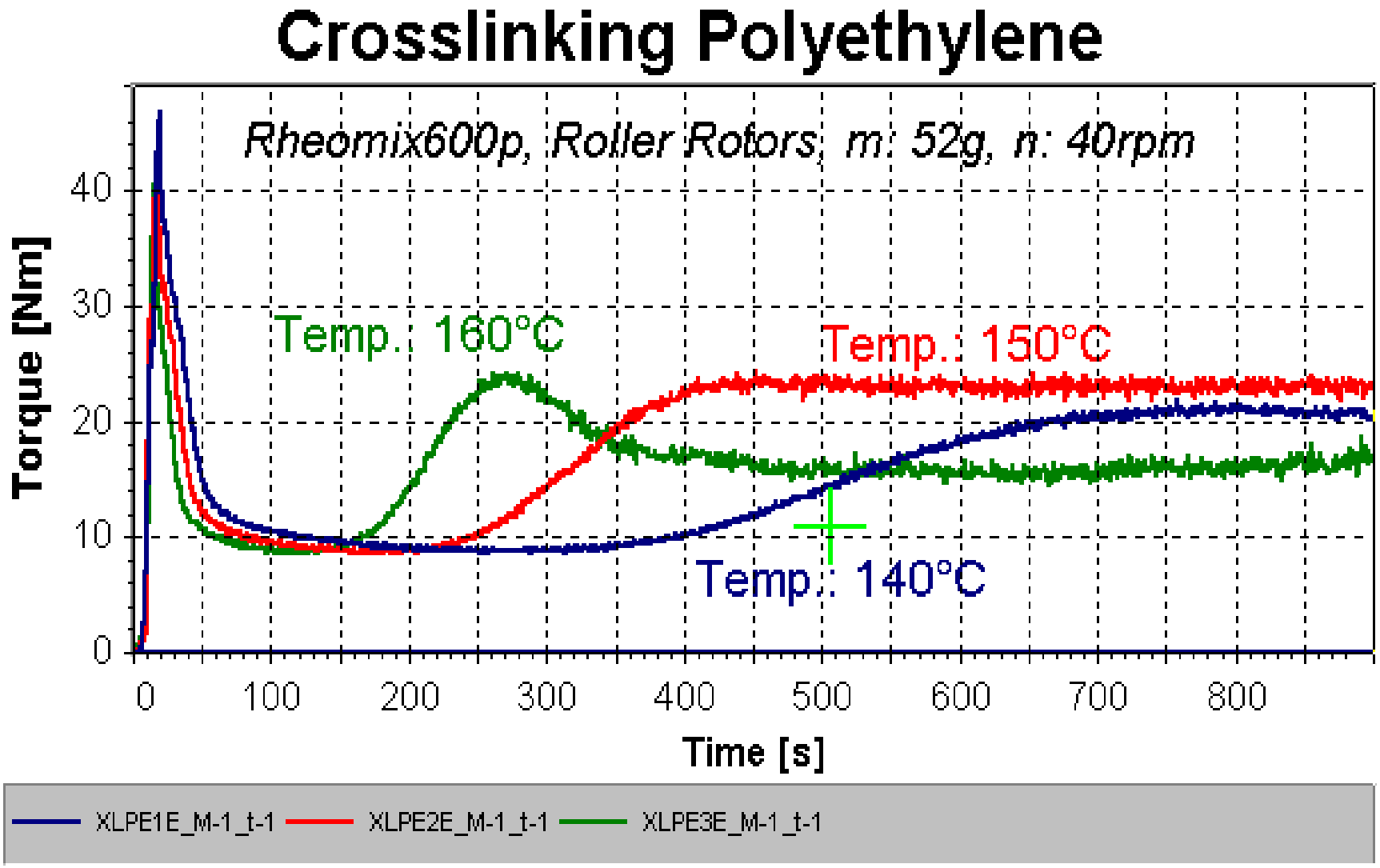


Correlation - Mixer Test with Extrusion

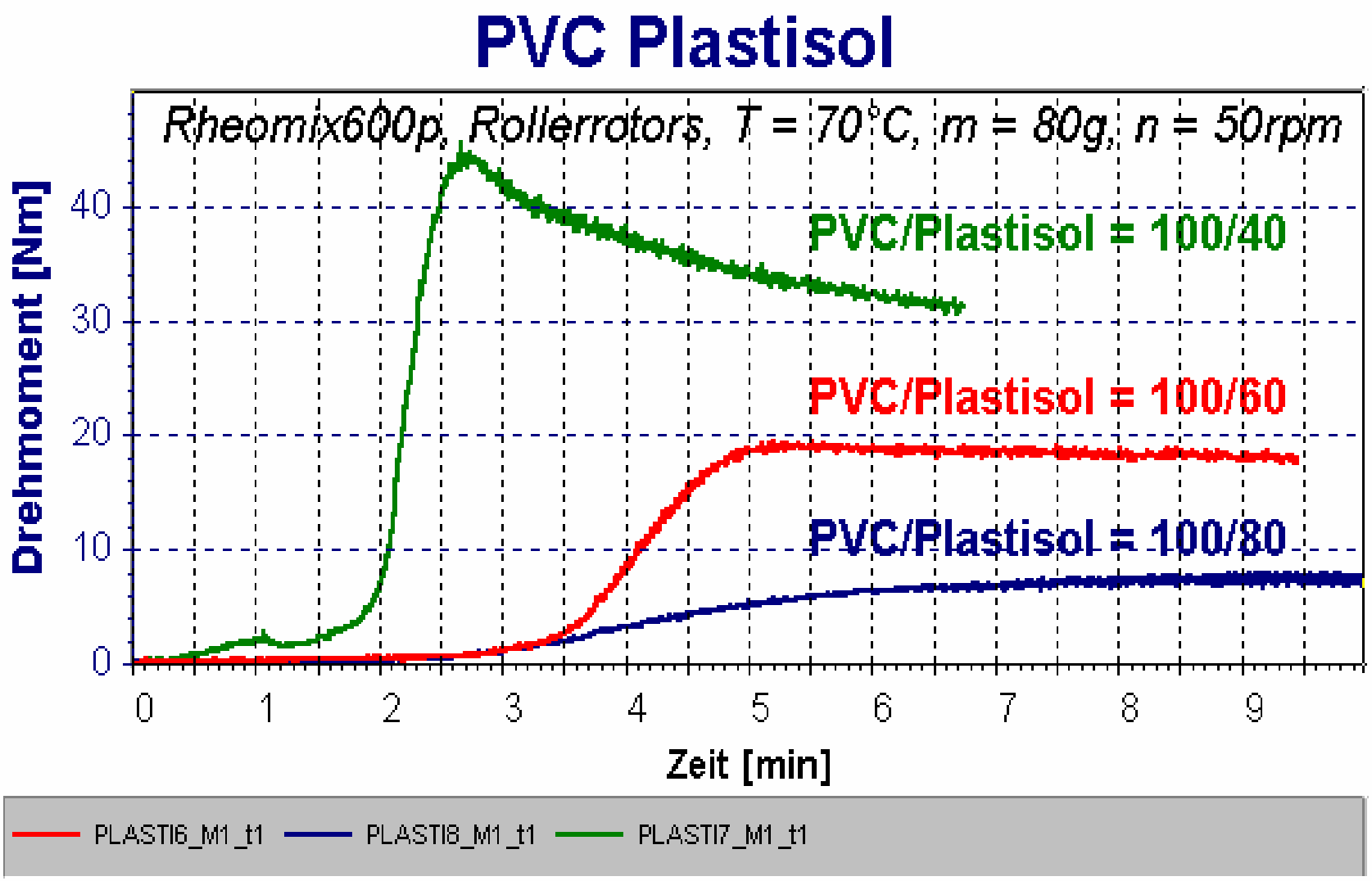
Example: PVC-Stability



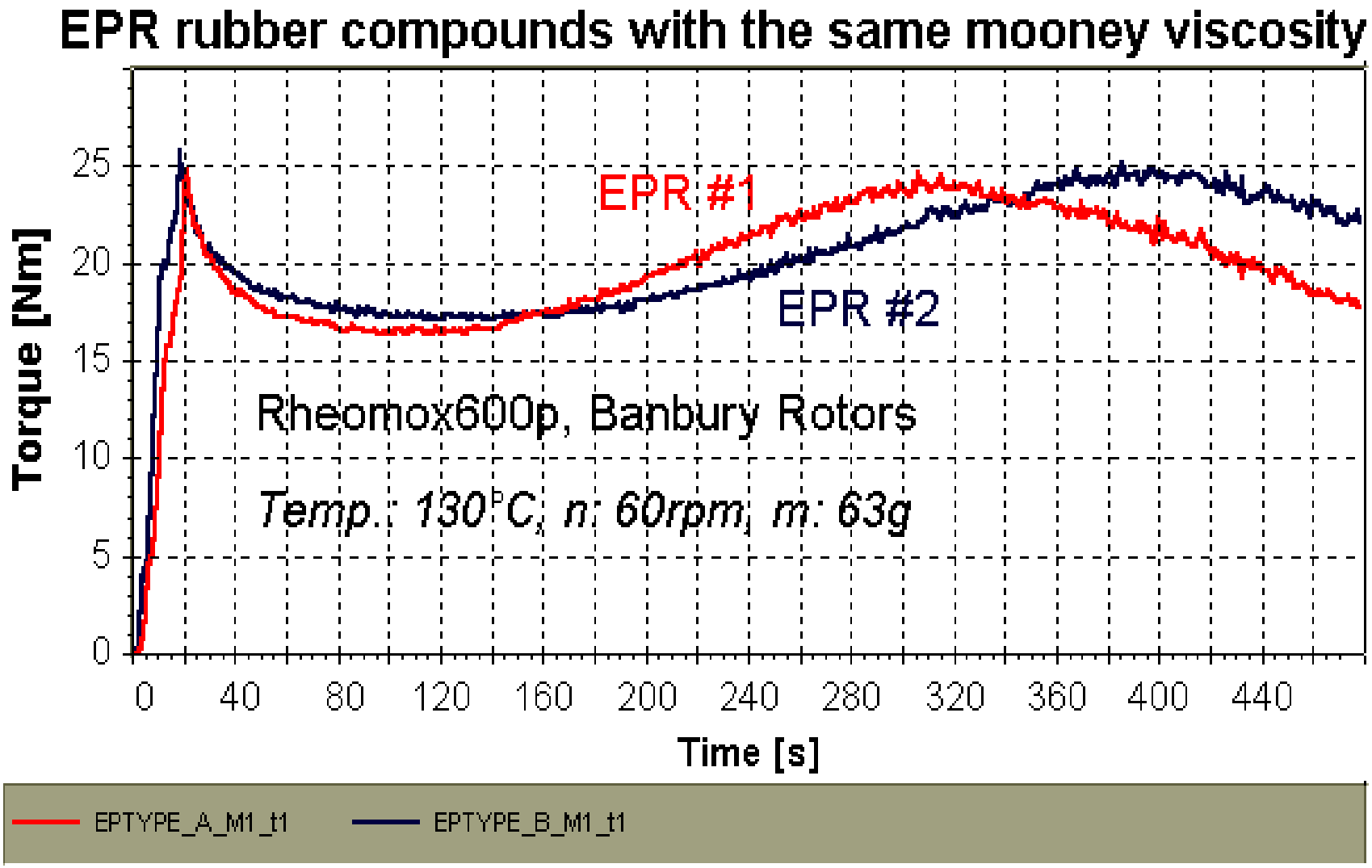
Application – Crosslinking Polymers



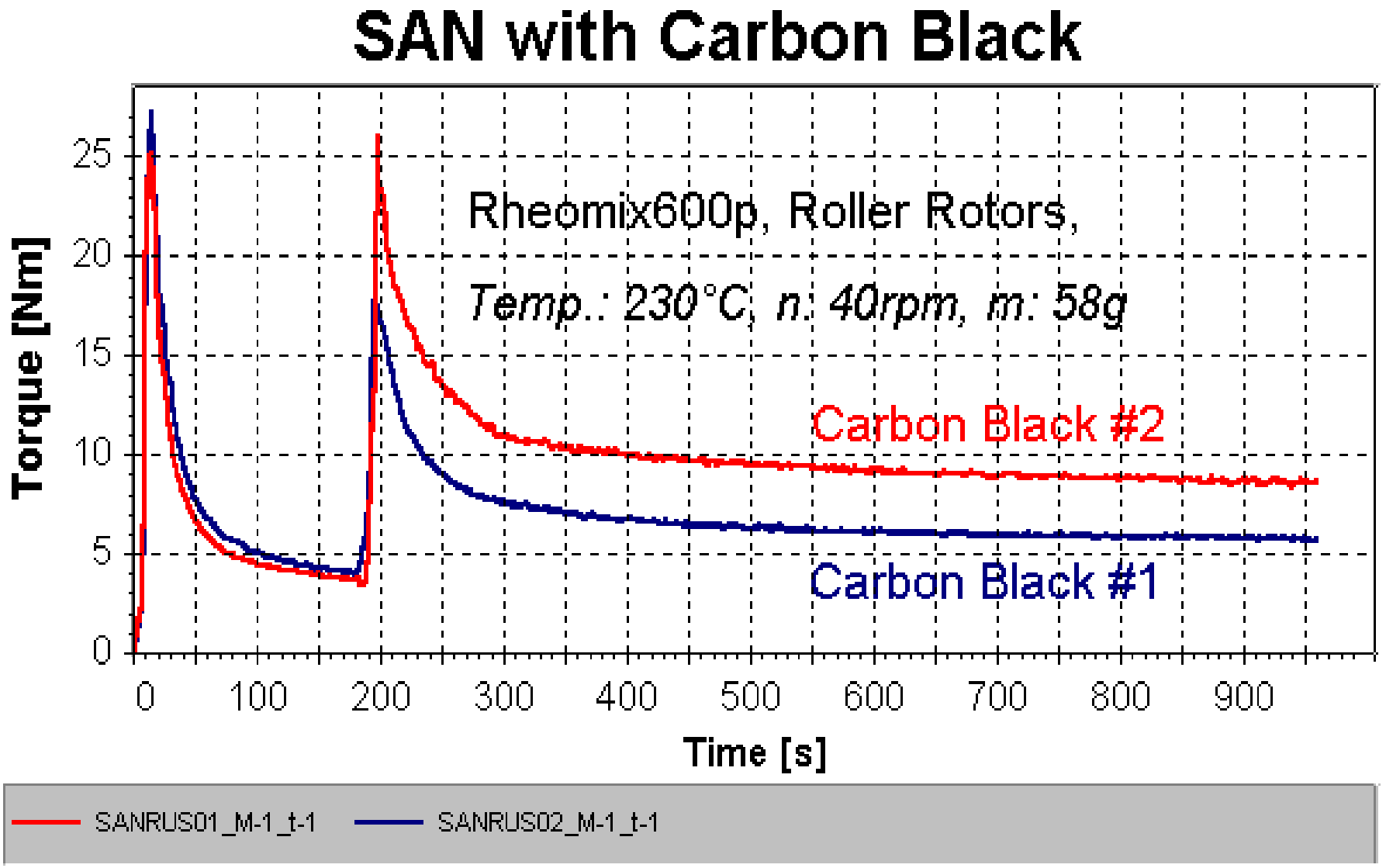
Application – PVC Plastisol gelation



Application – EPR Rubber



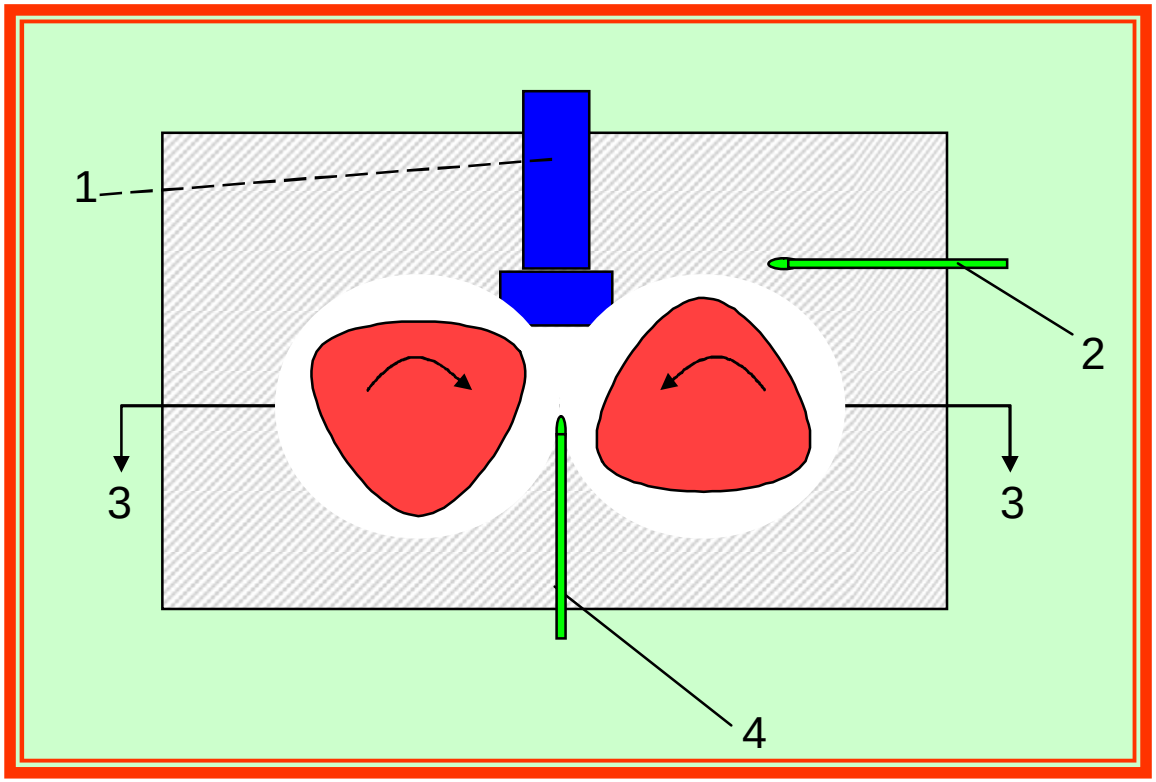
Application – effect of different Carbon Black



Analytical Test Methods for PolyLab OS

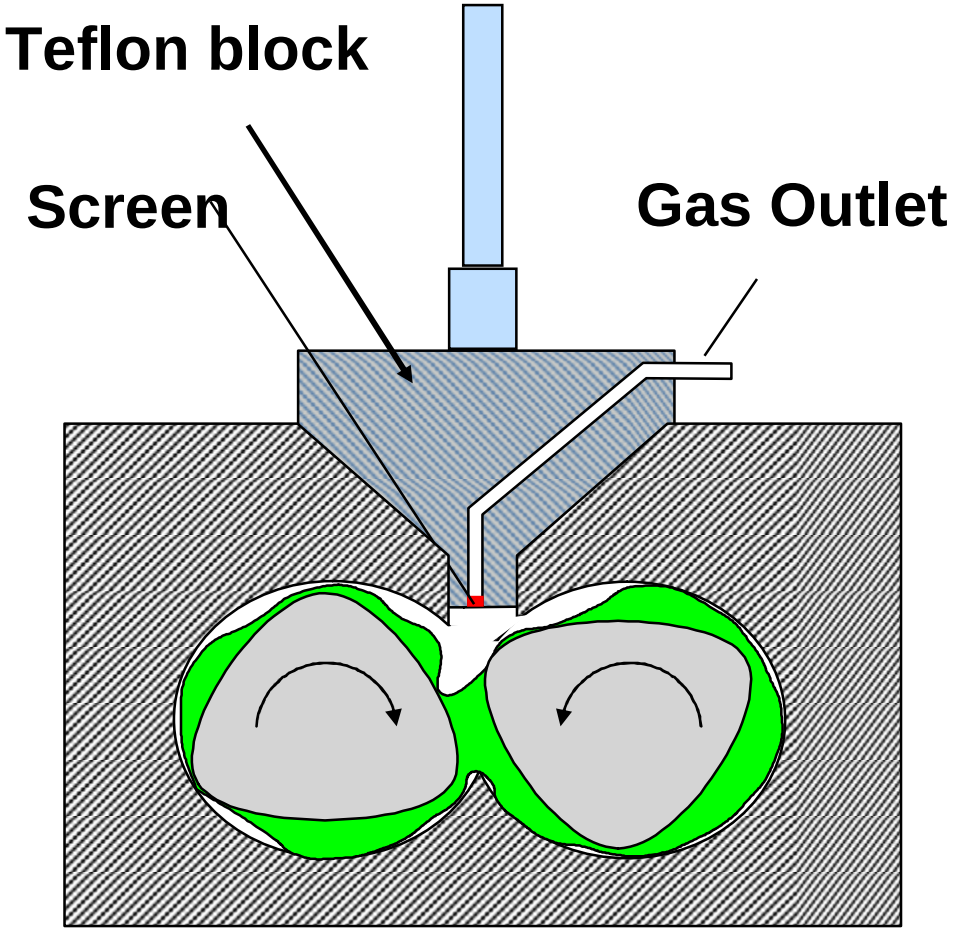
Electrical Conductivity Measurement

The Conductivity of rubber compounds as a measure for the dispersion of carbon black has been shown to be a reliable test method



1 Ram
2 Control Thermocouple
3 Rotors
4 Conductivity Sensor

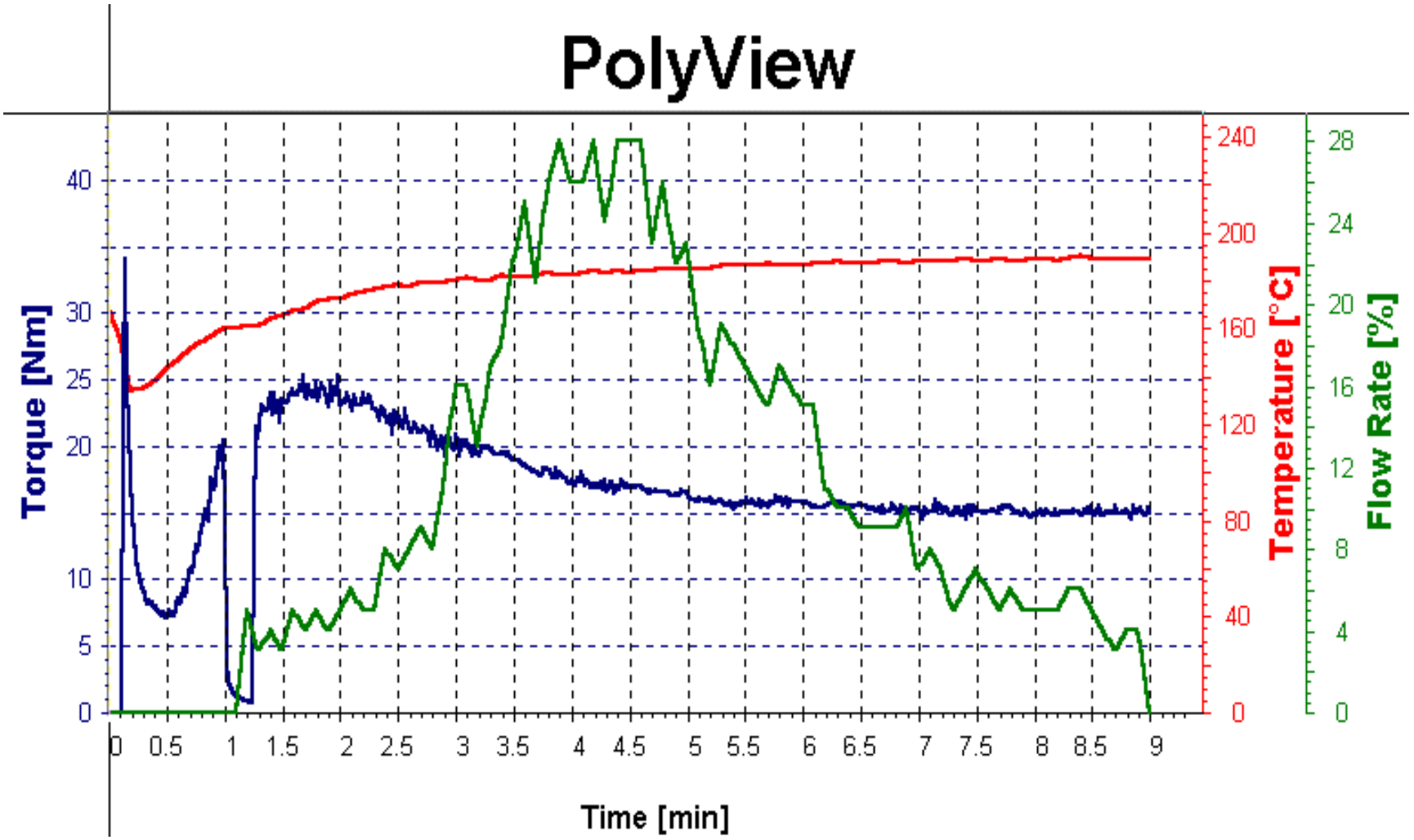
Gas Flow Sensor for RheoMix Mixers



Application:

- Foaming Compounds
- Degradation Tests

Gas Flow Sensor - Measuring results



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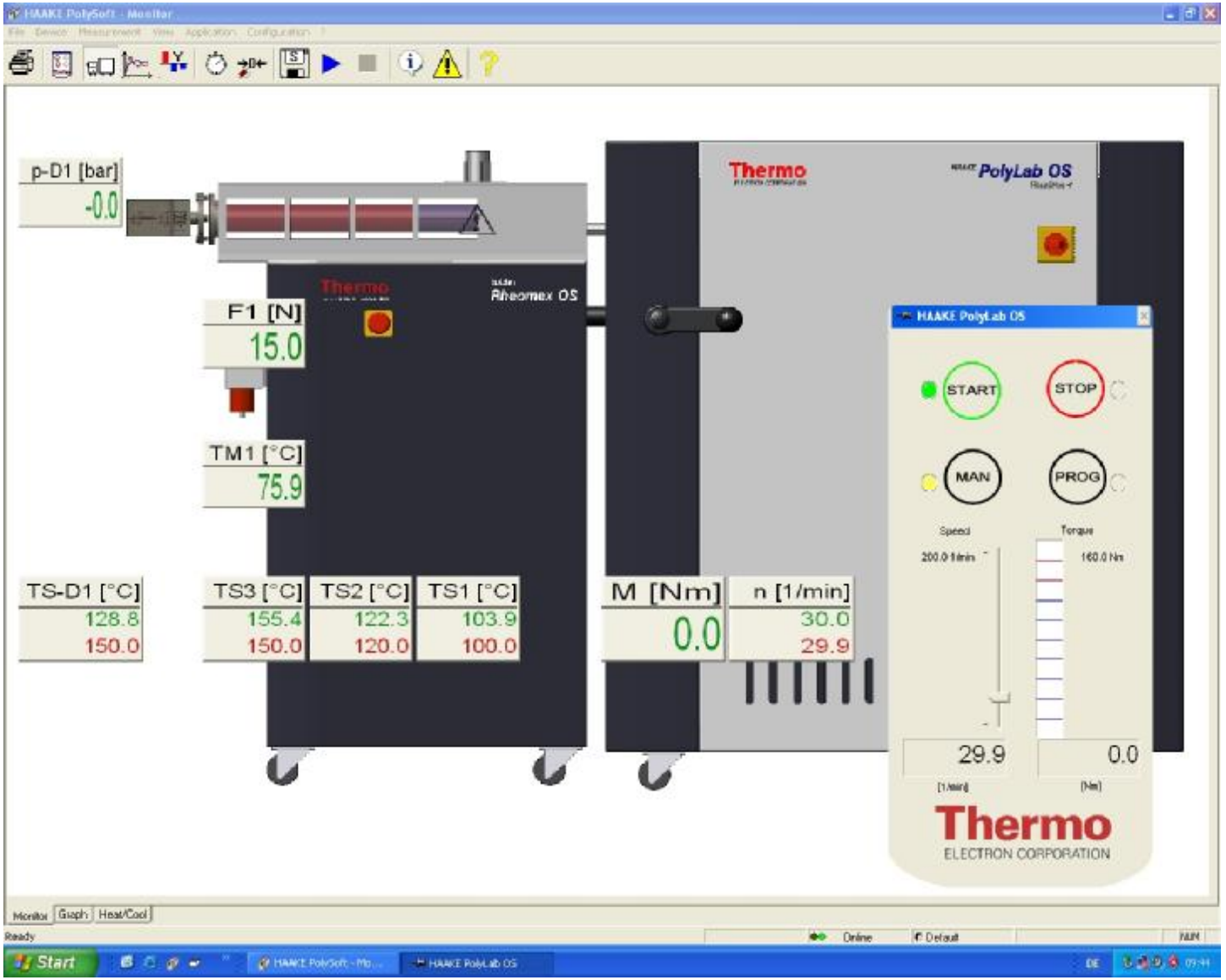
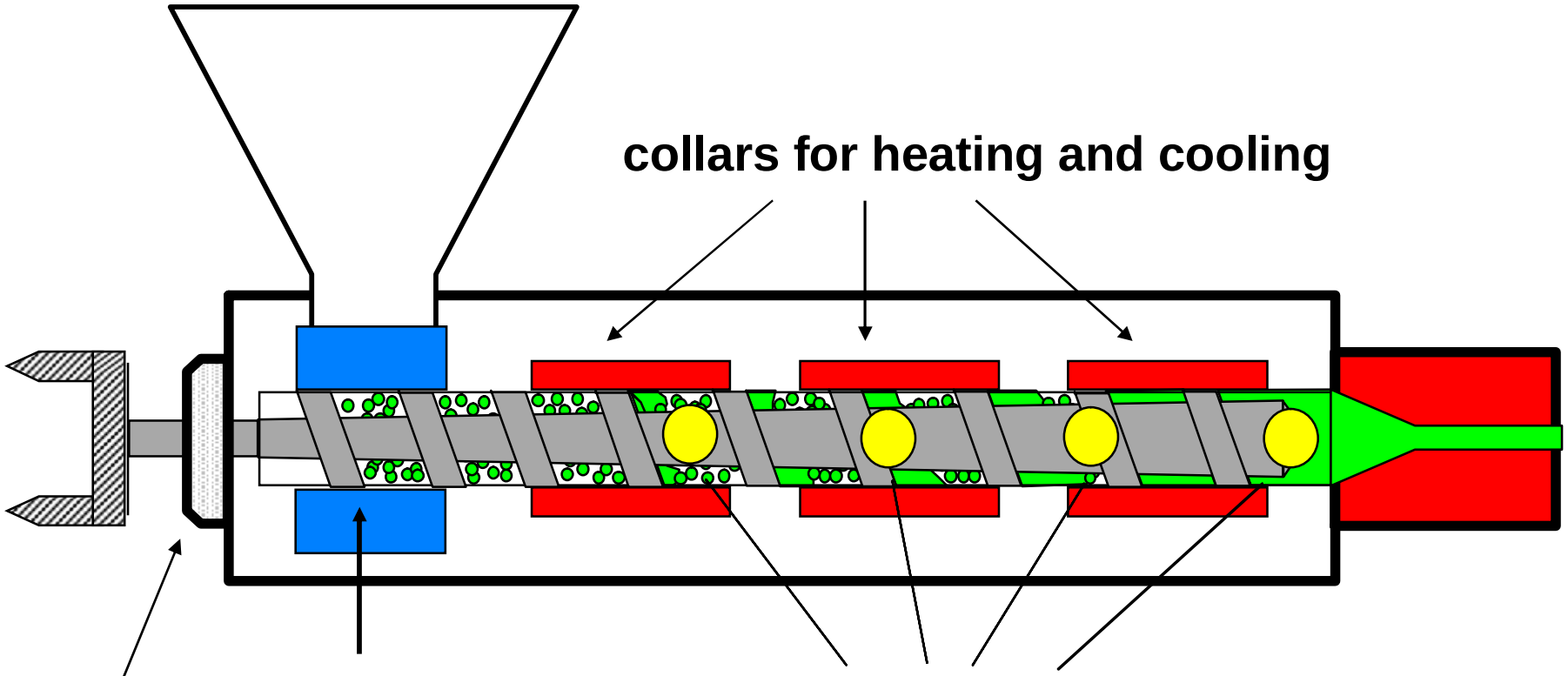
Single Screw Extrusion Testing

PolyLab OS with Single Screw Extruder

PolyLab Monitor Software – Single Screw Extruder View



Rheomex Single Screw Extruder



Rheomex Single Screw Extruder Applications

Single Screw Extruders are generally used to:

- melt and transport material
- plasticize
- compress
- vent / de-gas
- building up pressure

Measuring of process variables:

- torque
- melt temperature
- melt pressure
- mass flow

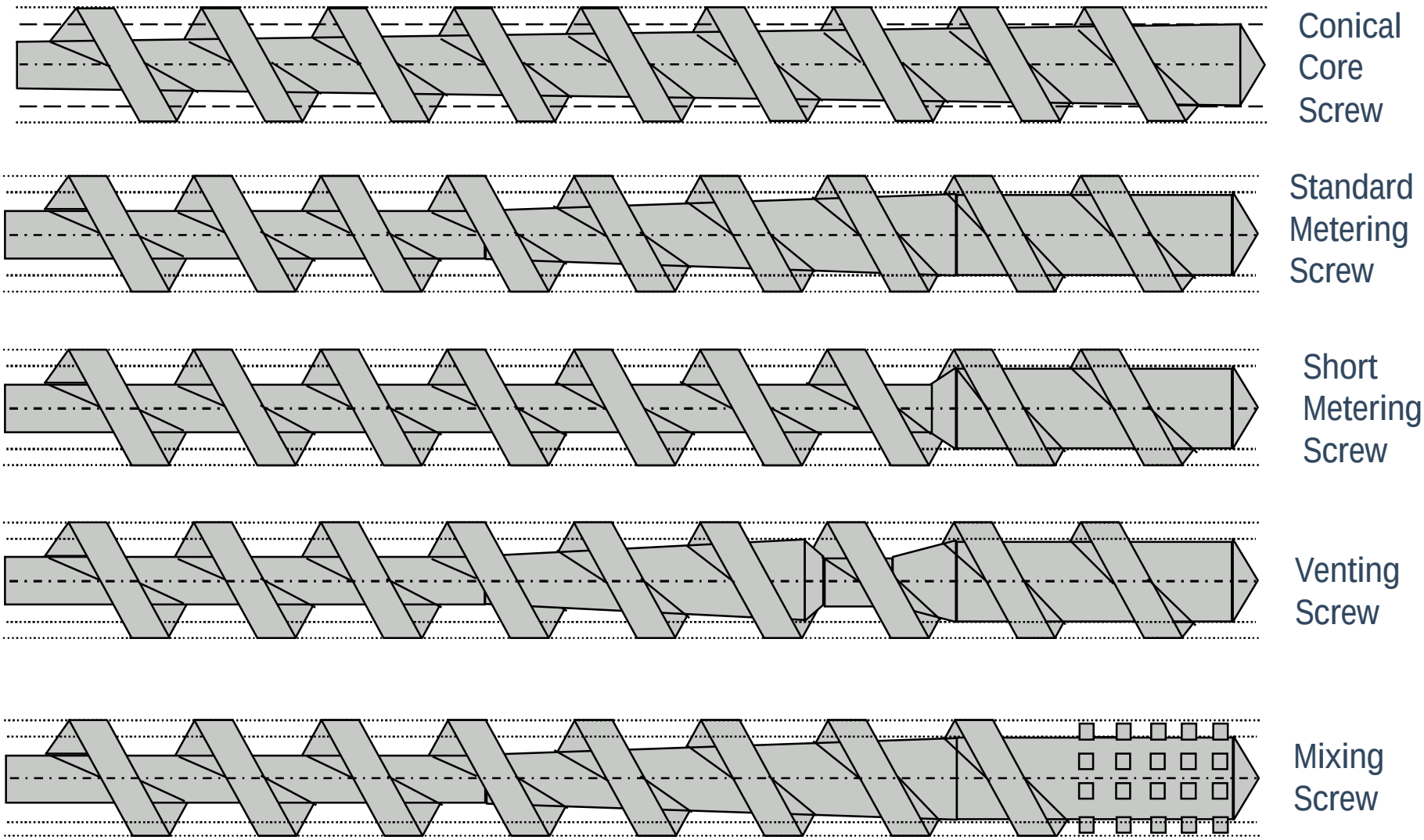
Optimizing of process parameters:

- extruder speed
- temperature profile
- pressure
- screw design

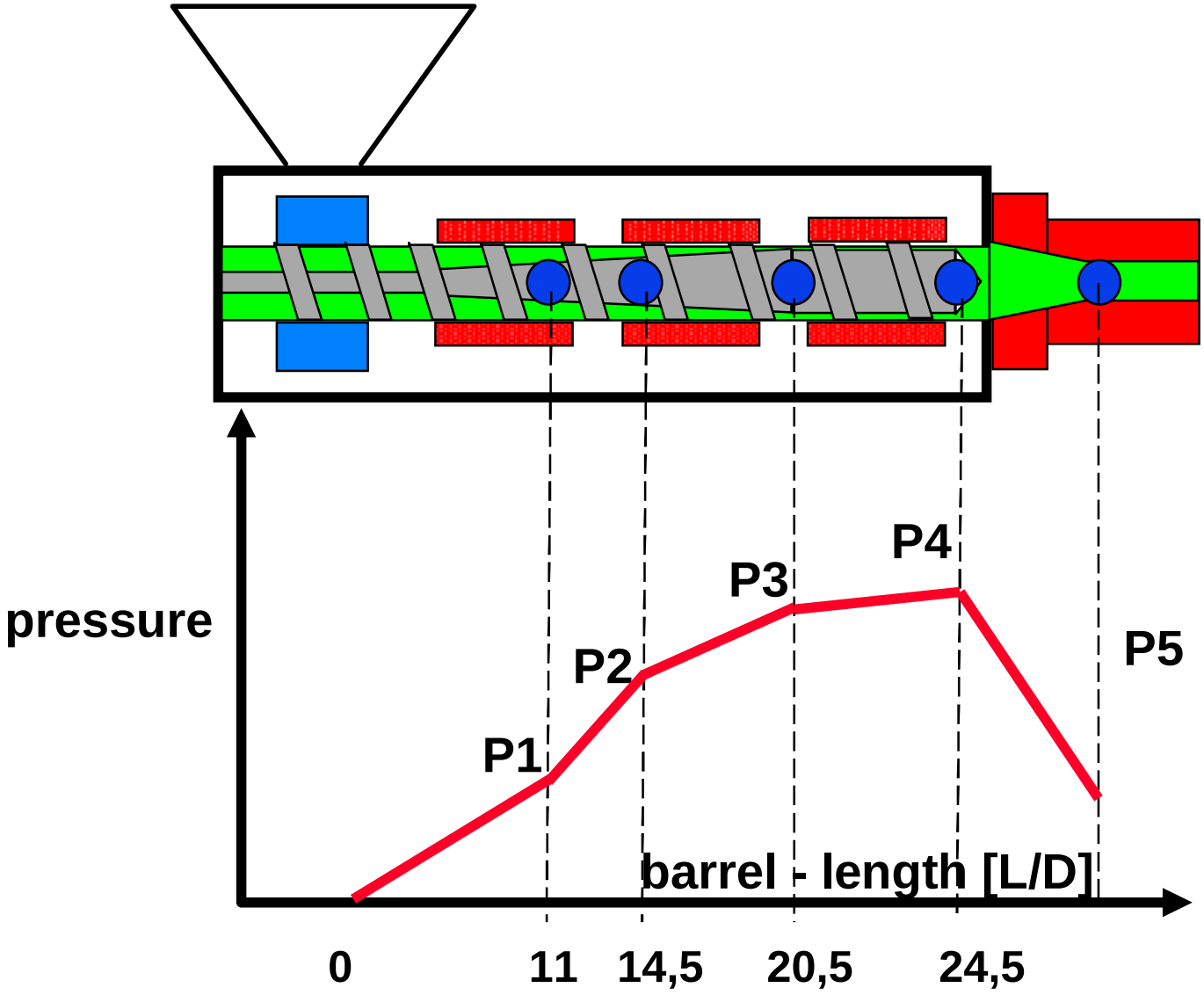
RheoMex Single Screw Extruder Models

Item	Rheomex 19/10	Rheomex 19/25	Rheomex 19/33
Screw diameter	19.05 mm (3/4″)	19.05 mm (3/4″)	19.05 mm (3/4″)
L/D	10	25	33
Material	Stainless steel nitrided DIN 1.8550		
Max. temperature	450°C	450°C	450°C
Max. pressure	700 bar	700 bar	700 bar
Max. speed	250 min^{-1}	250 min^{-1}	250 min^{-1}
Max. torque	160 Nm	160 Nm	160 Nm
Heating zones	1	3	4
Cooling	Air	Air	Air
Application	Profile extrusion and rheological tests on unvulcanized rubber or elastoplastics	Profile and sheet extrusion, rheological tests for standard thermoplast materials	Extended barrel and screws for special applications e.g. foaming.
Options	Separate controllable feed roller for rubber	Additional sensor ports, back force sensor, wear resistance material, chemical resistance material	Additional sensor ports, back force sensor, wear resistance material, chemical resistance material

Screws for Single Screw Extruders

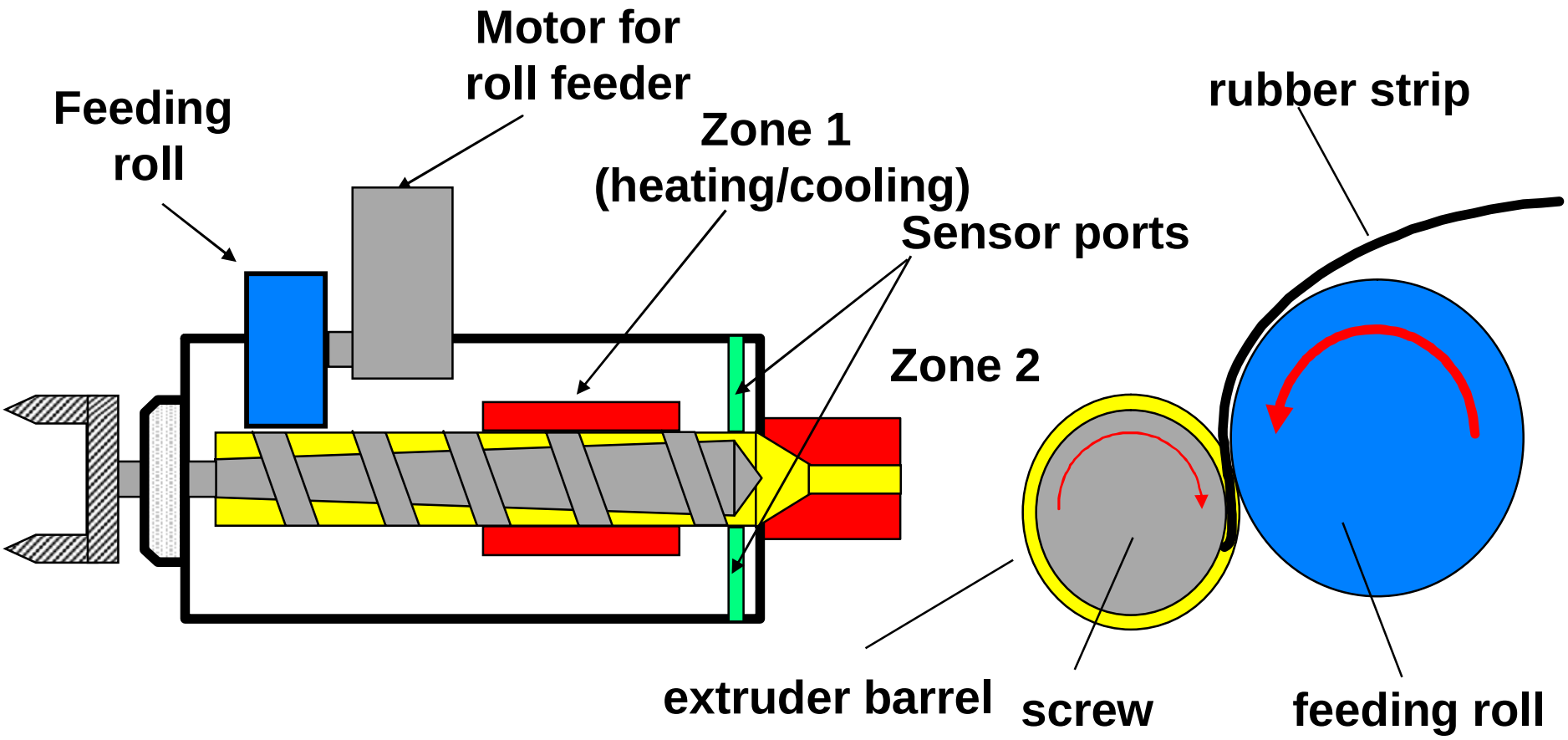


Pressure built-up in a single screw extruder



Rheomex 104 – 10:1 L/D

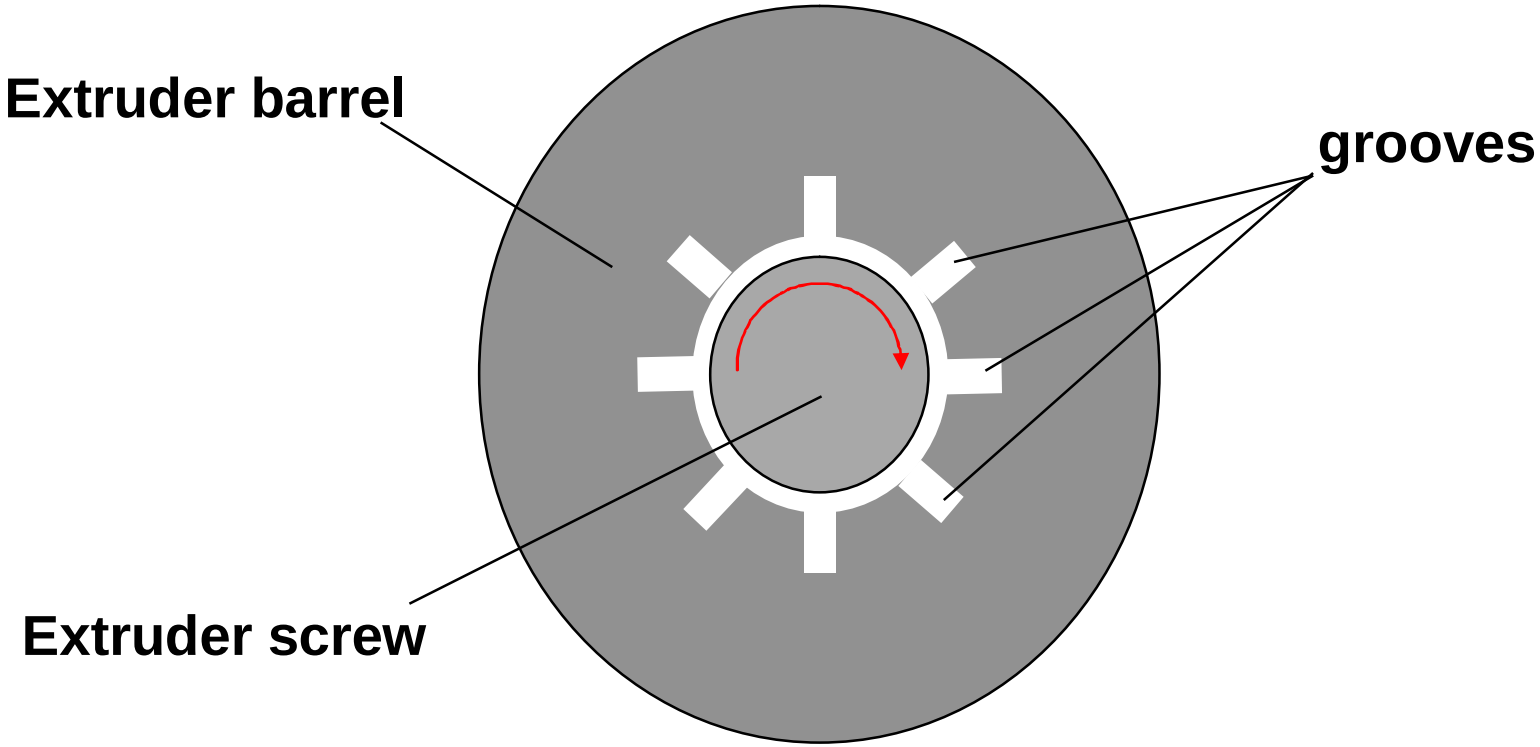
for rubber application:



Special single screw extruders for food extrusion

è **Special grooved extruder barrel:**

non-wallsticking material can be extruded



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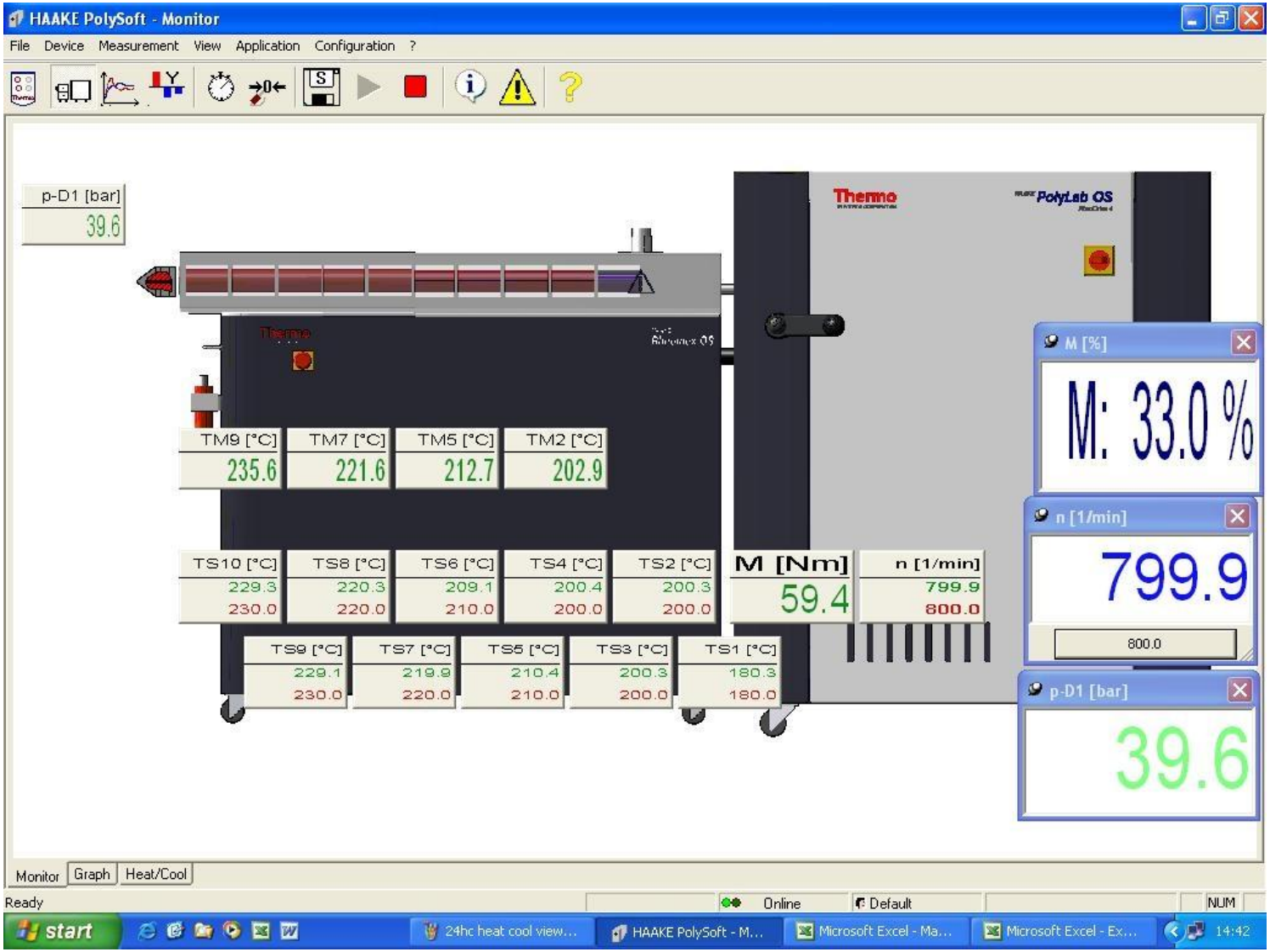
Twin Screw Extrusion Testing

TSE Technology PPT

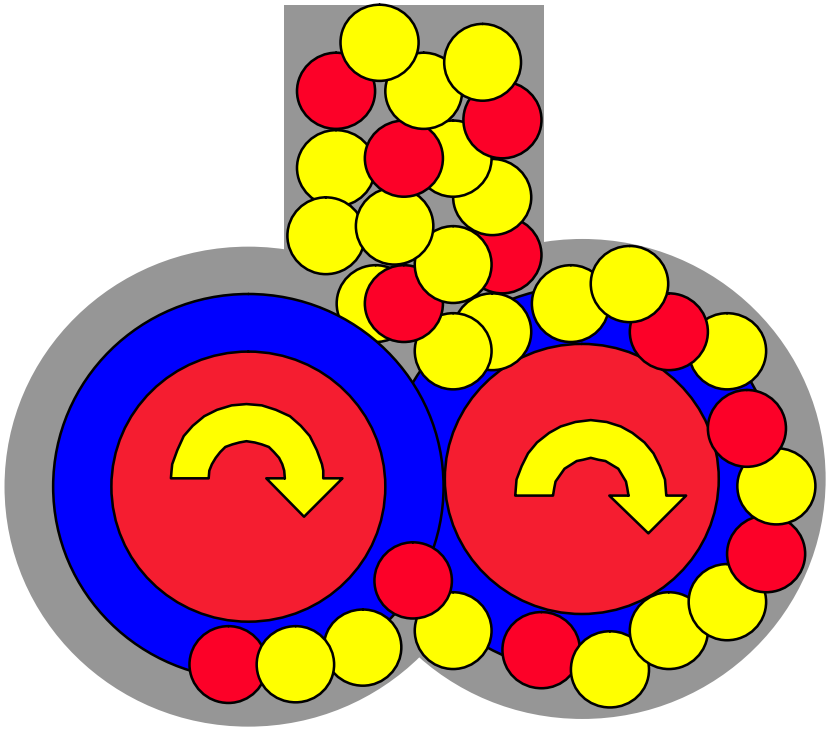
PolyLab Monitor Software – TSE View

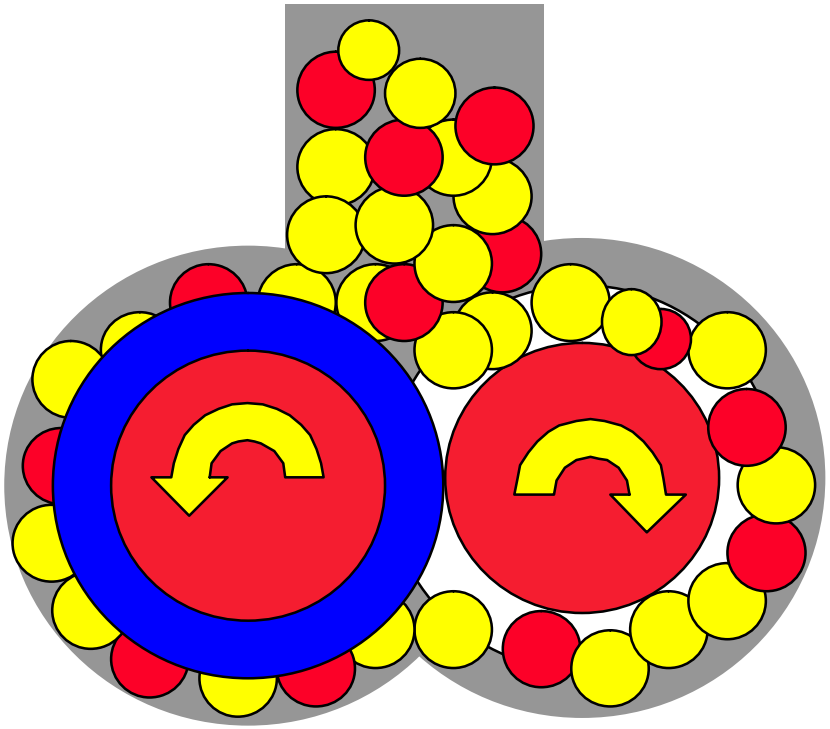


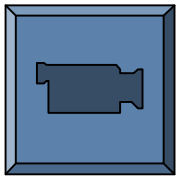
Processing with Twin Screw Extruders

- Plasticizing (PVC)
- Alloying or Grafting of co-polymers
- Mixing - Dispersion - Homogenizing of
  - Pigments
  - Additives
  - Plasticizers
  - Cross-Linking agents
  - Flame Retardants
  - UV Stabilizers
- Degassing (venting) of monomers, oligomers, solvents
- Incorporation of fillers and reinforcing agents such as:
  - Calcium Carbonate / Talc
  - Wood Flour
  - Glass / Carbon Fibers, etc
- Chemical Reactions:
  - Polymerization – Polycondensation - Polyaddition
  - Cooking - Gelling

Feeding Material into Twin-Screw Extruders







Co-rotating

Counter-rotating

Multiple Twin Screw Options to fit any need

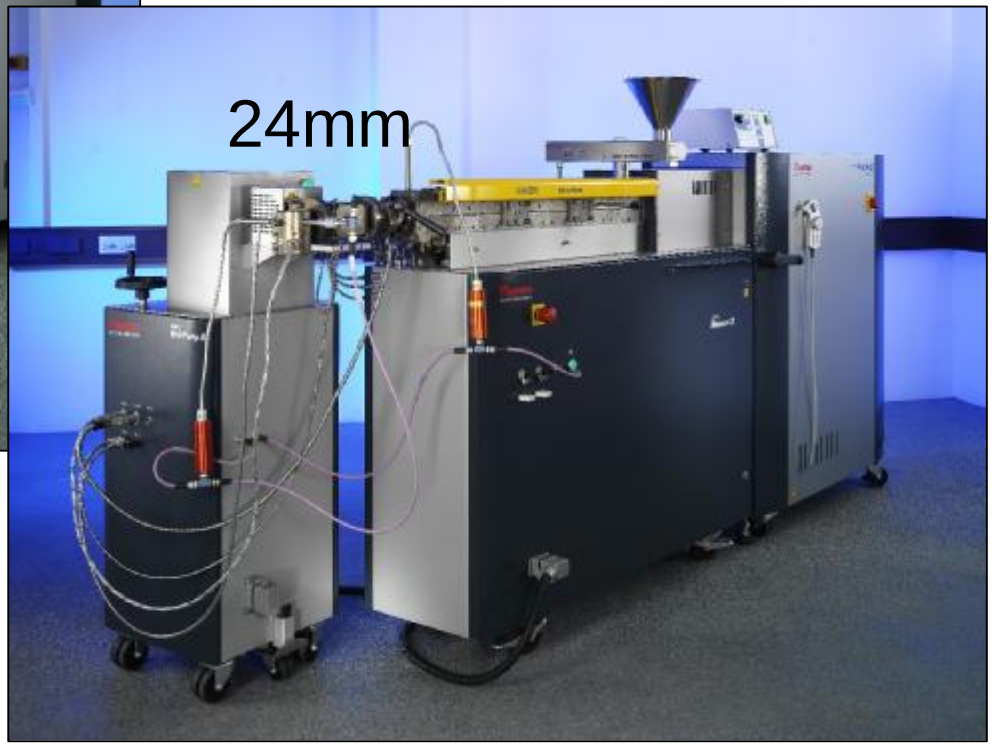
§ Co-Rotating Design (Rheomex PTW series)

- Most widely used type TSE in polymer industry
- Good for Shear Sensitive Materials
- Compounding of Masterbatch or Color Concentrates
- Best design when venting is required or multiple feed points
- Flexible Screw and Barrel design – modular design
- Controlled Feeding is required

§ Counter Rotating Design (Rheomex CTW series)

- Narrow Residence Time distribution (good materials like PVC)
- Fully Self Wiping – creates high shear & high pressure
- No controlled feeding is necessary
- Co-Rotating Design (Rheomex PTW series)

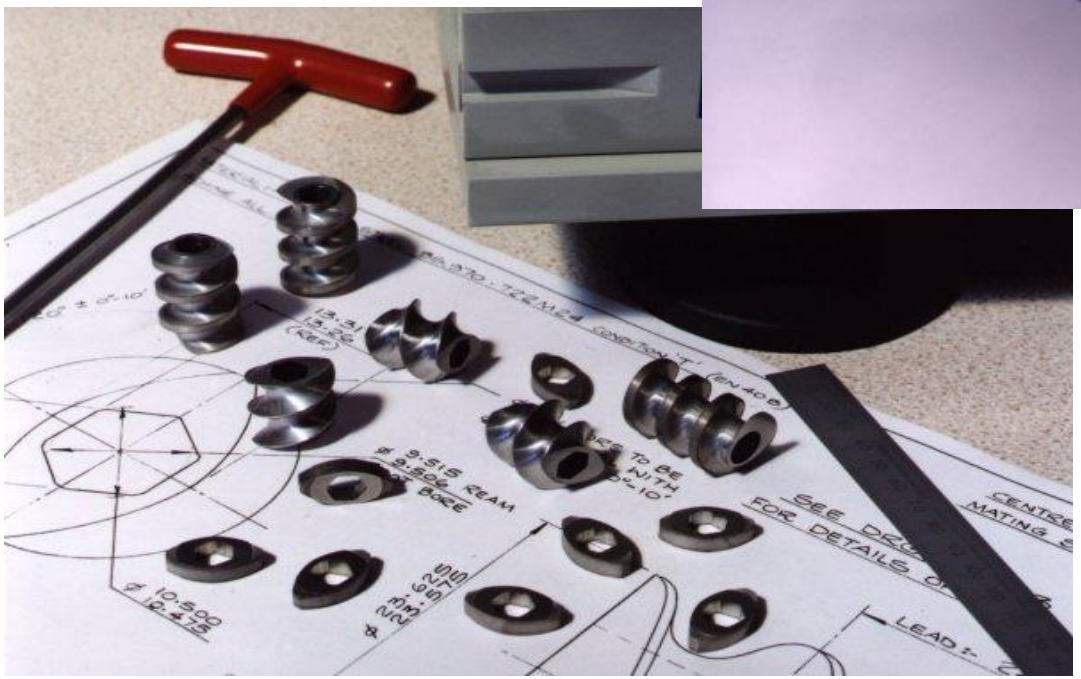
Modular Split Barrel, Segmented Screws



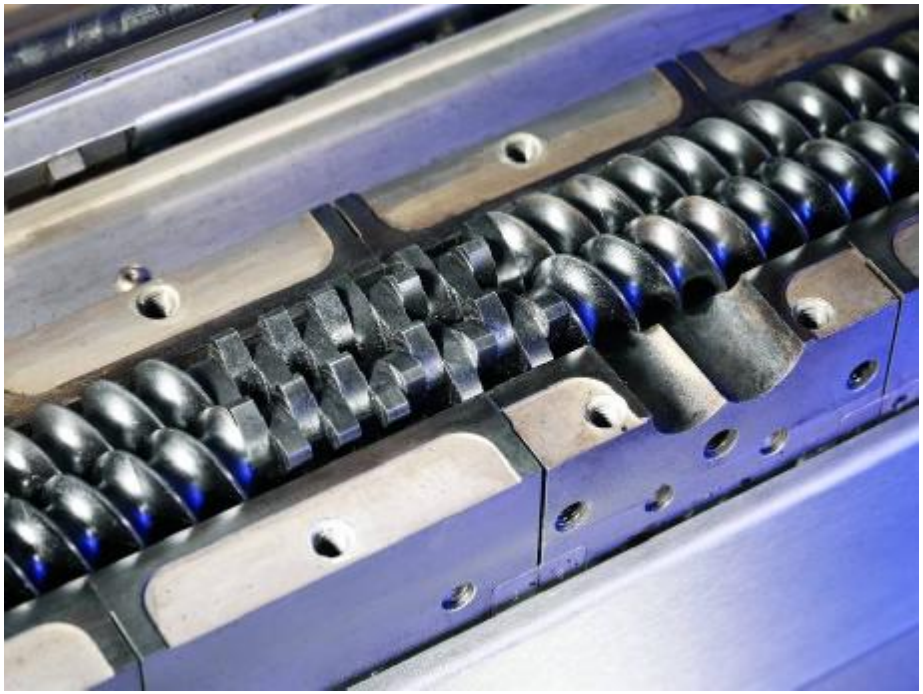
Parallel Co-Rotating
Twin Screw Extruders

Flexible Screw Profiles with Segmented Screws

Specifically designed for Lab Scale systems to maintain good free-volume to diameter ratio and channel depth



Flexibility to Meet Process Requirements



Twin Screw Agitator Elements

	CONVEYING	MIXING
Feed Screws	++++++++	+
30 deg Forward	+++++	++
60 deg Forward	++++	+++++
90 deg Alternate	zero	++++++++
60 deg Reverse	- - - -	++++++
Star / Toothed	zero	++++++++
Reverse Feed Screw	- - - - - - - -	++

Access TSE Technology PPT

PolyLab Twin Screw Extruder Models

Item	PTW 16/25	PTW 16/40	PTW 24/28	PTW 24/40	CTW 100
Screw diameter	16 mm	16 mm	24 mm	24 mm	Conical
L/D	25	40	28	40	- -
Screw setup	Variable	Variable	Variable	Variable	3 options
Gear ratio	1:5.4	1:5.4	1:2	1:2	1:1
Rotating direction	Co	Co	Co	Co	Counter
Max. screw speed	1100 min^{-1}	1100 min^{-1}	1100 min^{-1}	1100 min^{-1}	250 min^{-1}
Max. temperature	350 °C (opt. 450 °C)	350 °C (opt. 450 °C)	350 °C (opt. 450 °C)	350 °C (opt. 450 °C)	450 °C
Max. pressure	100 bar	100 bar	100 bar	100 bar	700 bar
Max. screw speed	1100 min^{-1}	1100 min^{-1}	1100 min^{-1}	1100 min^{-1}	250 min^{-1}
Max. torque	130 Nm	130 Nm	180 Nm	180 Nm	200 Nm
Heating zones	7	10	7	10	3
Cooling	Convection	Convection	Internal water circuit	Internal water circuit	Air
External heaters	2	2	2	2	2
Feeding zone	Cooled	Cooled	Cooled	Cooled	Cooled
Additional Feeding / Venting	2 Top	3 Top	2 Top, 1 Side	3 Top, 2 Side	- -
Sensor ports	(1/2" UNF)	(1/2" UNF)	(1/2" UNF)	(1/2" UNF)	2 (1/2" UNF)
Options	15 L/D extension, additional feeding ports, additional sensor ports	Additional feeding ports, additional sensor ports	Additional feeding ports, additional sensor ports	Additional feeding ports, additional sensor ports	Backforce sensor, additional sensor ports

PolyLab PTW16 XL

- Horizontally split, with a lift-off top that can be opened for ease of cleaning, simple removal of the screws and visualization of the melting process.
- The segmented top barrel part is built with 4D modules to be easily reconfigured by the user.
- Barrel segments are available for feeding solids, liquids and for venting.

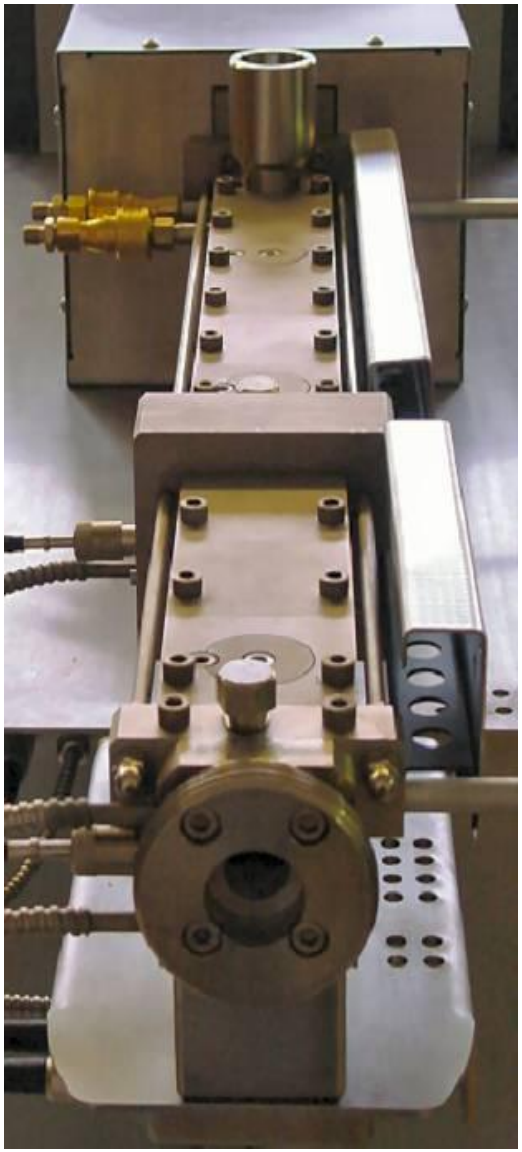
PTW16 Specifications

Max Temperature	350ºC (optional 450ºC)
Max Pressure	100-150 Bar*
Warm Up Time	10 minutes average
Minimum Sample	100 to 200 grams
Maximum Throughput	From 0.5 kg/hr - Up to 10 kg/hr
Maximum Agitator Speed	1100 rpm
Barrel Length	25:1 and 40:1 L/D ratio
Screw Center Distance	12.5mm
Screw Channel Depth	3.3mm

* Use Melt Pump for higher pressure requirements

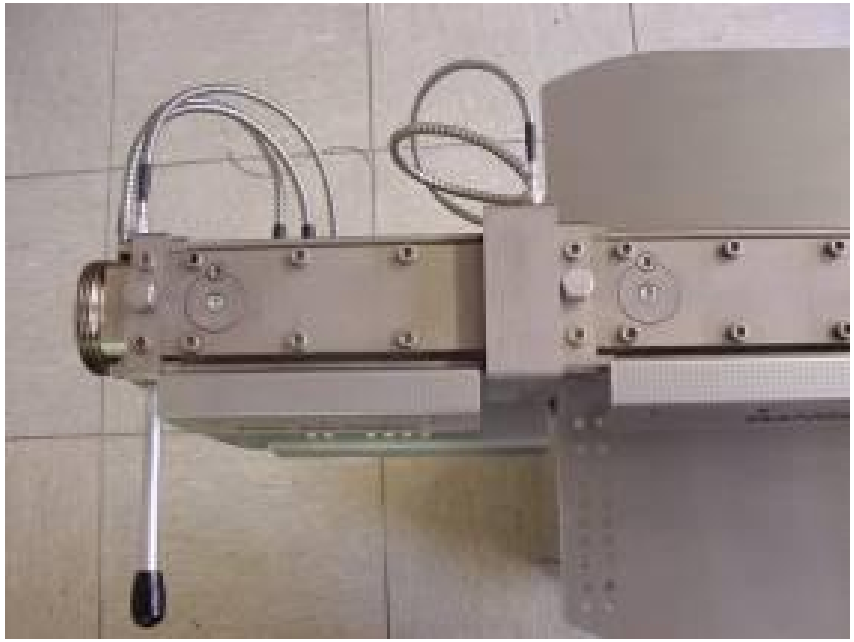
PTW16 XL 25:1 + 15D extension

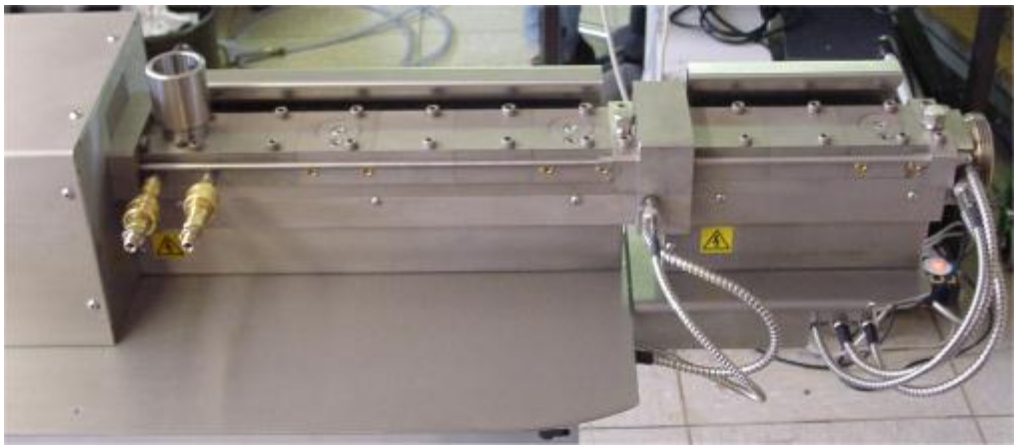
- Flexible Length: not all applications require a long extruder, such as shear sensitive materials and 25D is long enough for basic applications.
- Optional 15D barrel extension to enhance e.g. residence time (for reactive extrusion) or multiple feed ports,
- Easy to use and change - less than 15 minutes by operator!
- Software recognizes the Extension unit



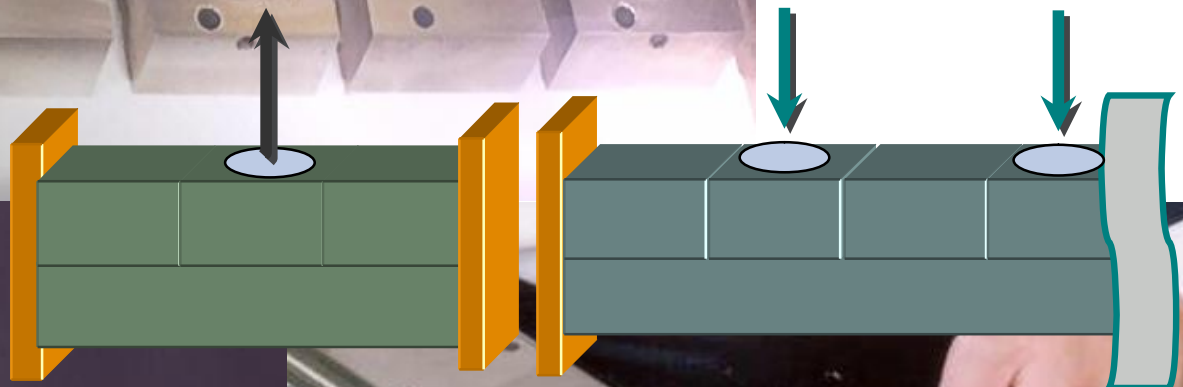
Rheomex PTW16 OS

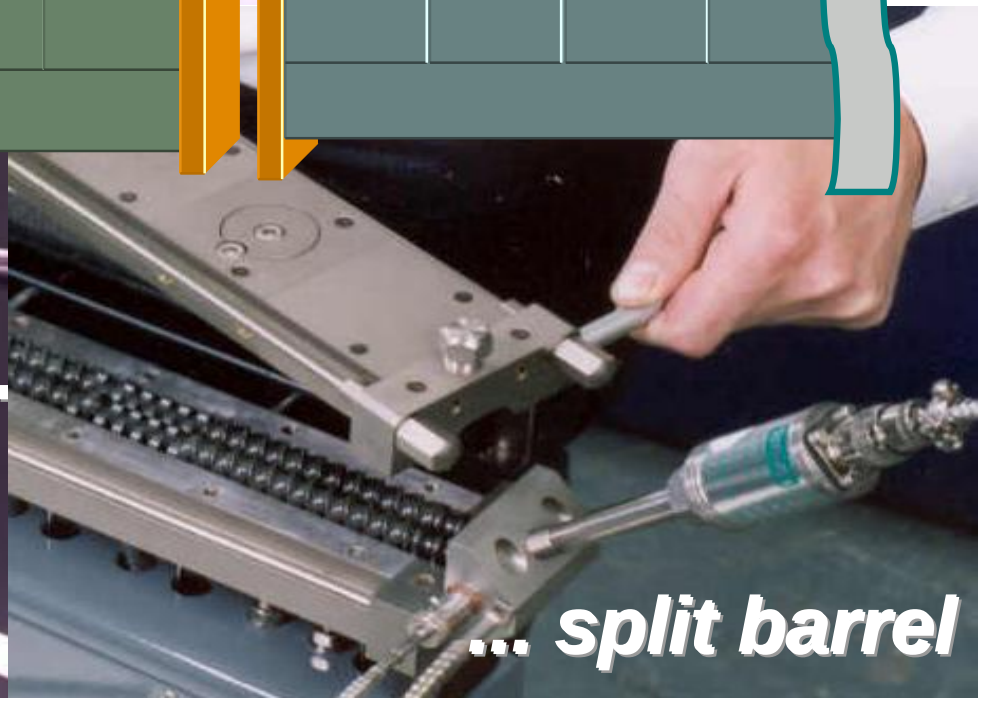
Twin Screw barrel with
25D and 15D extension





PTW16 OS - Maximum flexibility…

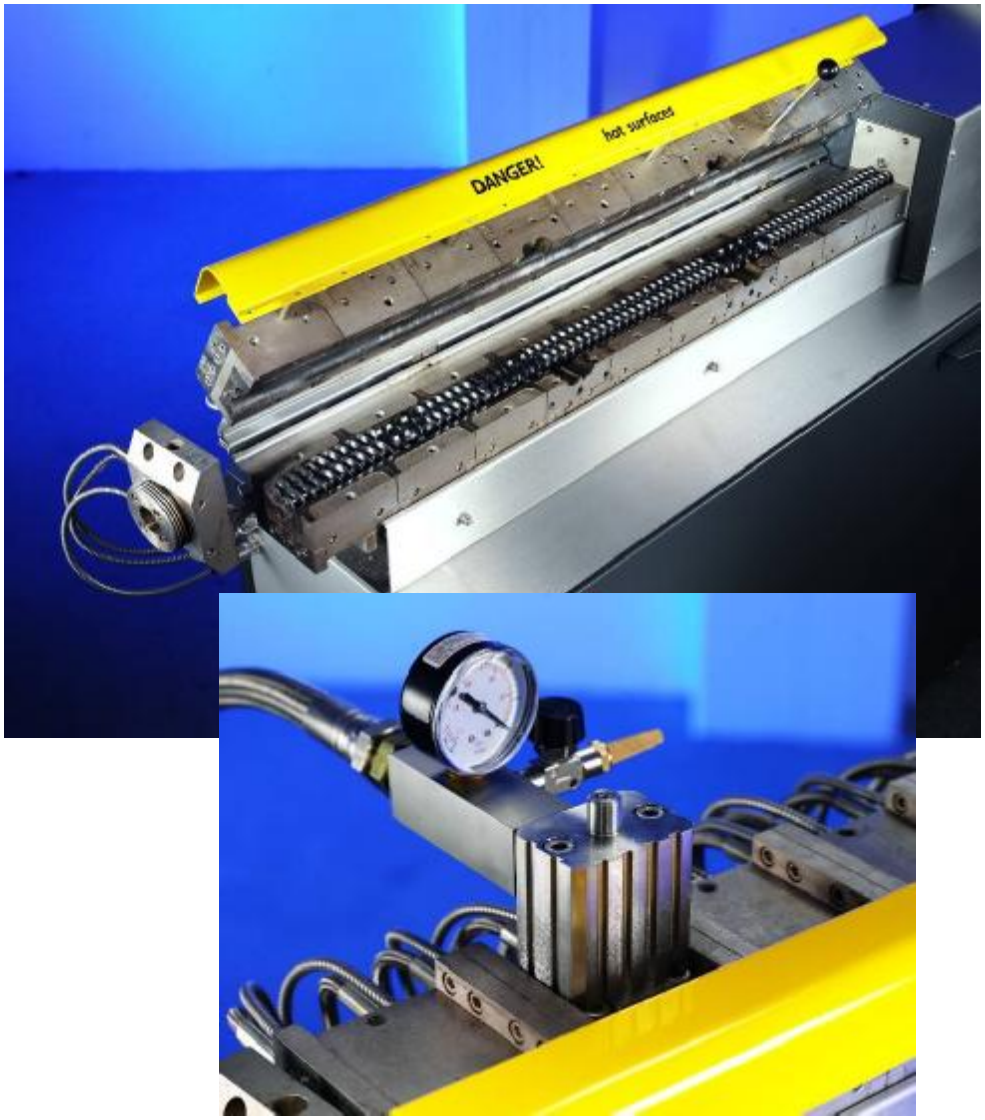




PTW16 OS - Maximum flexibility…

PolyLab PTW24

- Horizontally split, with a clam-shell barrel top that can be opened for ease of cleaning, simple removal of the screws and visualisation of the melting process.
- The segmented top & bottom barrel parts is built with 4D modules to be easily reconfigured by the user.
- Barrel segments are available for cooling feeding solids, liquids and for venting.



PTW24 with Melt Pump

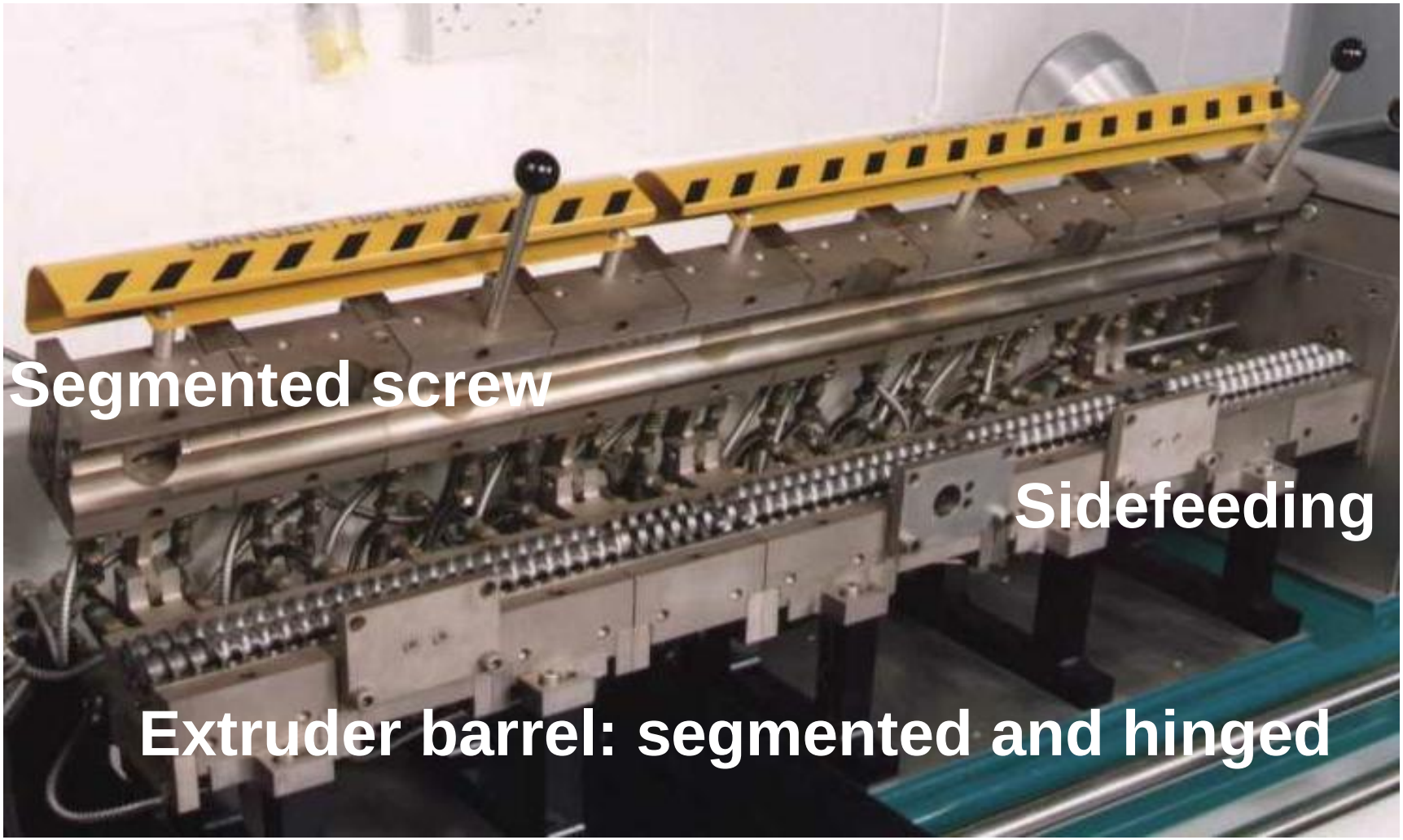
PTW24 with 3 Strand Pelletizing

PTW24 Specifications

Max Temperature	400ºC
Max Pressure	100-150 Bar*
Maximum Throughput	From 2.0 kg/hr - Up to 50 kg/hr
Maximum Agitator Speed	1000 rpm
Max Power/Torque	11 kW – 52 Nm
Barrel Length	28:1 and 40:1 L/D ratio
Screw Center Distance	18.75mm
Screw Channel Depth	5.15mm
Cooling Water Required	20 liters/min

* Use Melt Pump for higher pressure requirements

Rheomex PTW24 – Advantages



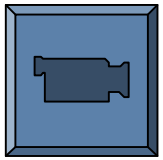
CTW100 Conical Twin Screw – counter rotating

CTW100 OS with PolyLab

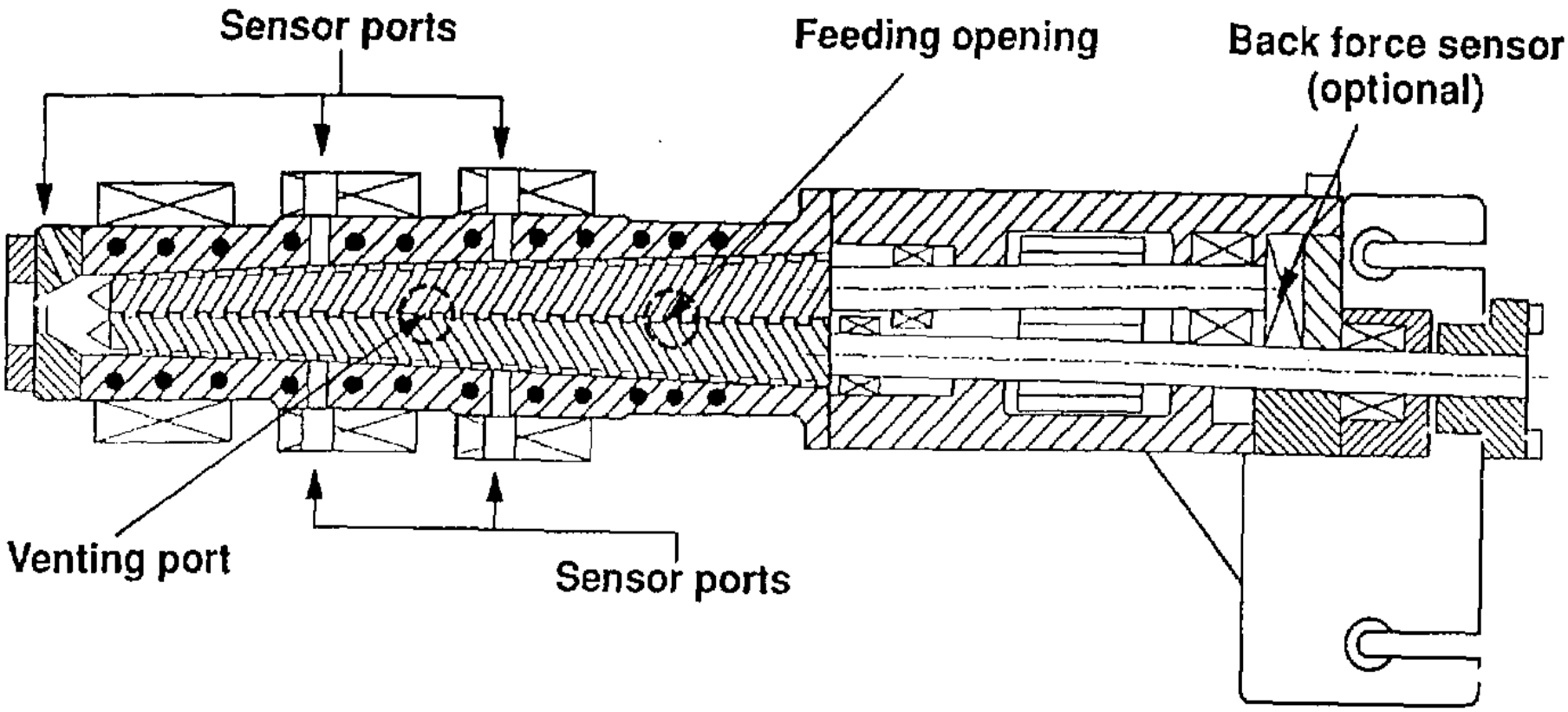
Supporting 20+ year legacy of CTW100 systems with Docking Station OS

CTW100 – Barrel Removal

Simple and Quick Barrel Removal



Rheomex CTW100p



Screw Geometries for CTW100

Venting screw

Intensive mixing screw

Standard screw

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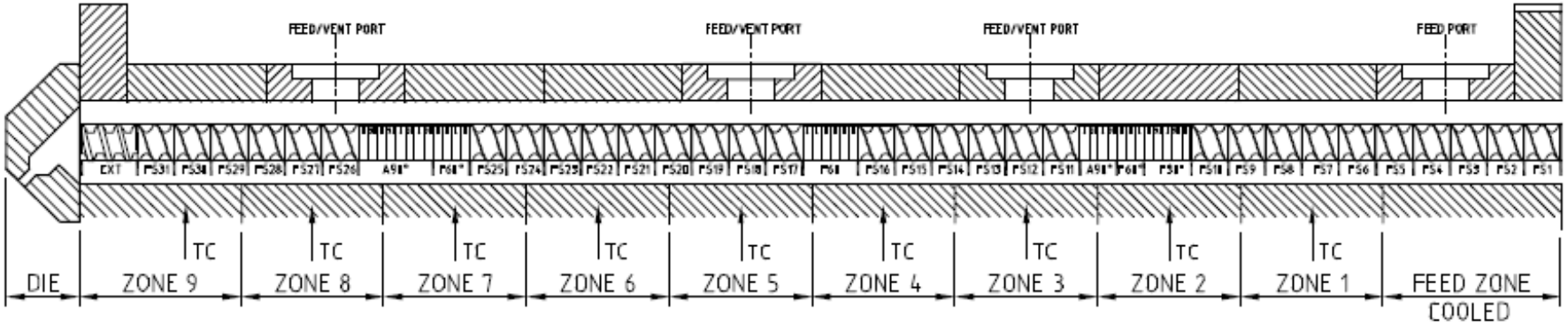
Feeders

Detailed Feeder PPT

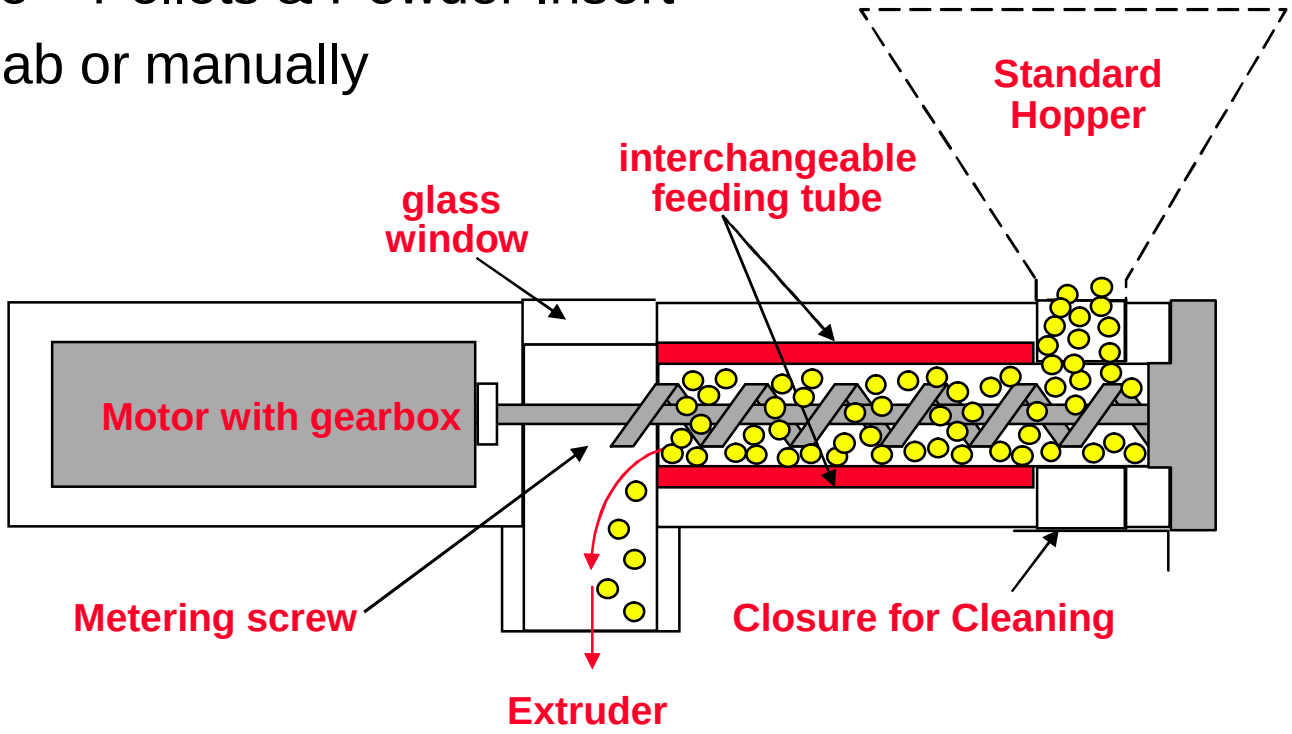
Why use Feeders for Twin Screw Extrusion?



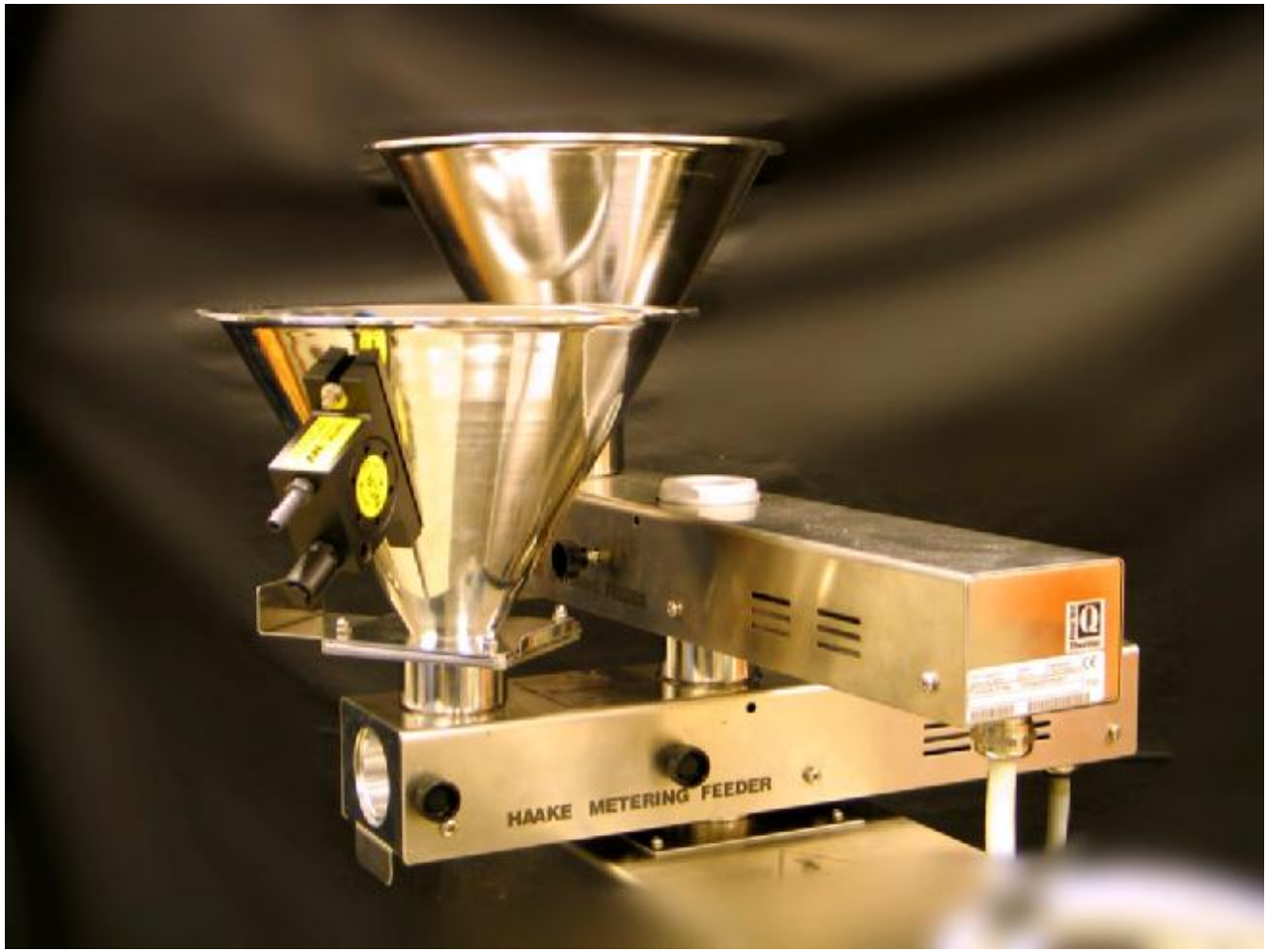
- The quality of the dispersive mixing is highly dependent on both the extruder RPM and the flow rate from the Feeders
- Starved feeding is necessary for Venting operations
- Multiple Feeders have many applications:
  - For sequential or simultaneous feeding of non-similar materials like pellets and powders that might segregate if batch fed in one hopper
  - For downstream feeding of heat or shear sensitive materials
  - To easily control the ratio of multiple ingredients by varying the feed rates

HAAKE Metering Feeder

- Designed specifically to work with PolyLab systems
- Multiple feeders can be stacked over extruder feed port
- Compact – Easy to Use
- Wide Feed Range – Pellets & Powder Insert
- Control via PolyLab or manually



Stackable HAAKE Metering Feeders



Mini Twin Screw Feeder

- Volumetric or Gravimetric version available
- Feed Rates as low as 50 grams per hour – smallest twin screw feeder on the market
- Multiple Feed Tubes & Screws available to match material requirements

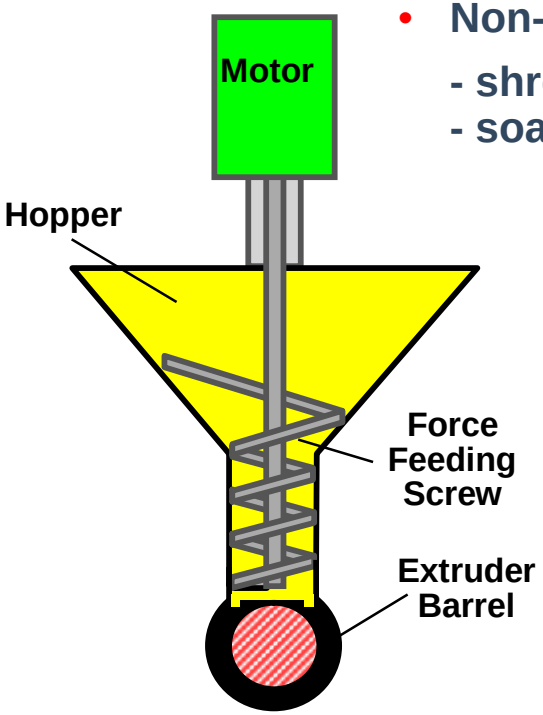
Dosing (RotoTube) Feeder

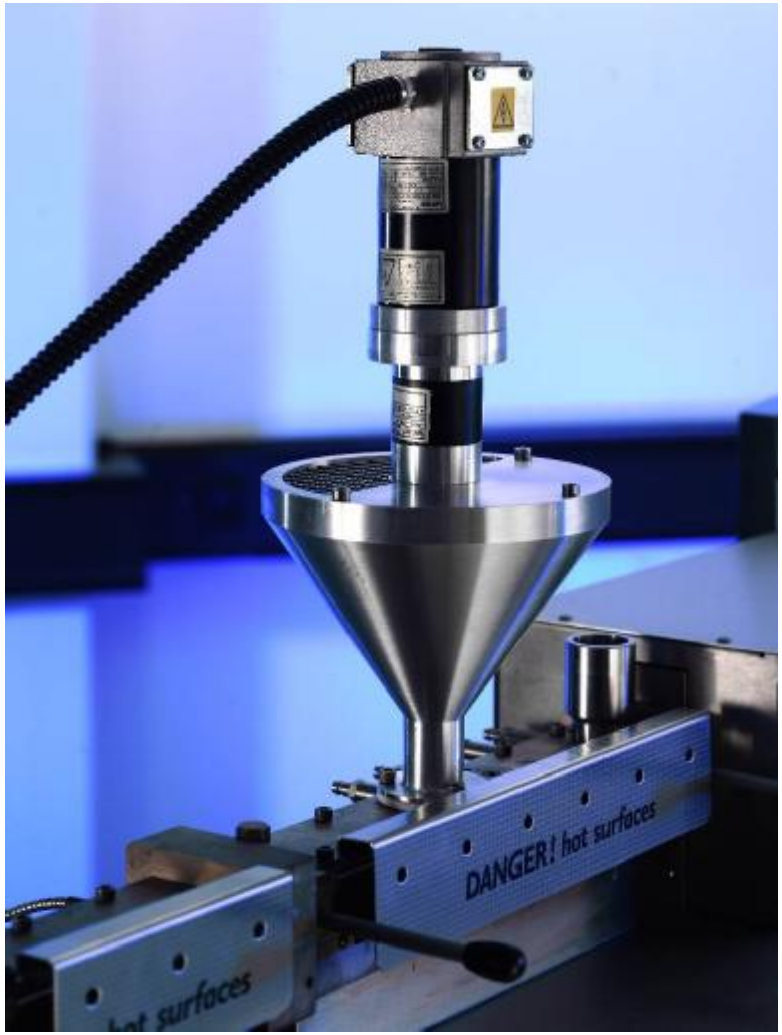
- Micro-Feeder for Fine Grain materials
- Volumetric and Gravimetric versions available
- Feed Rates as low as 30 gram per hour
- Tilting System and Anti-Vibration supports built-in

Force Feeder

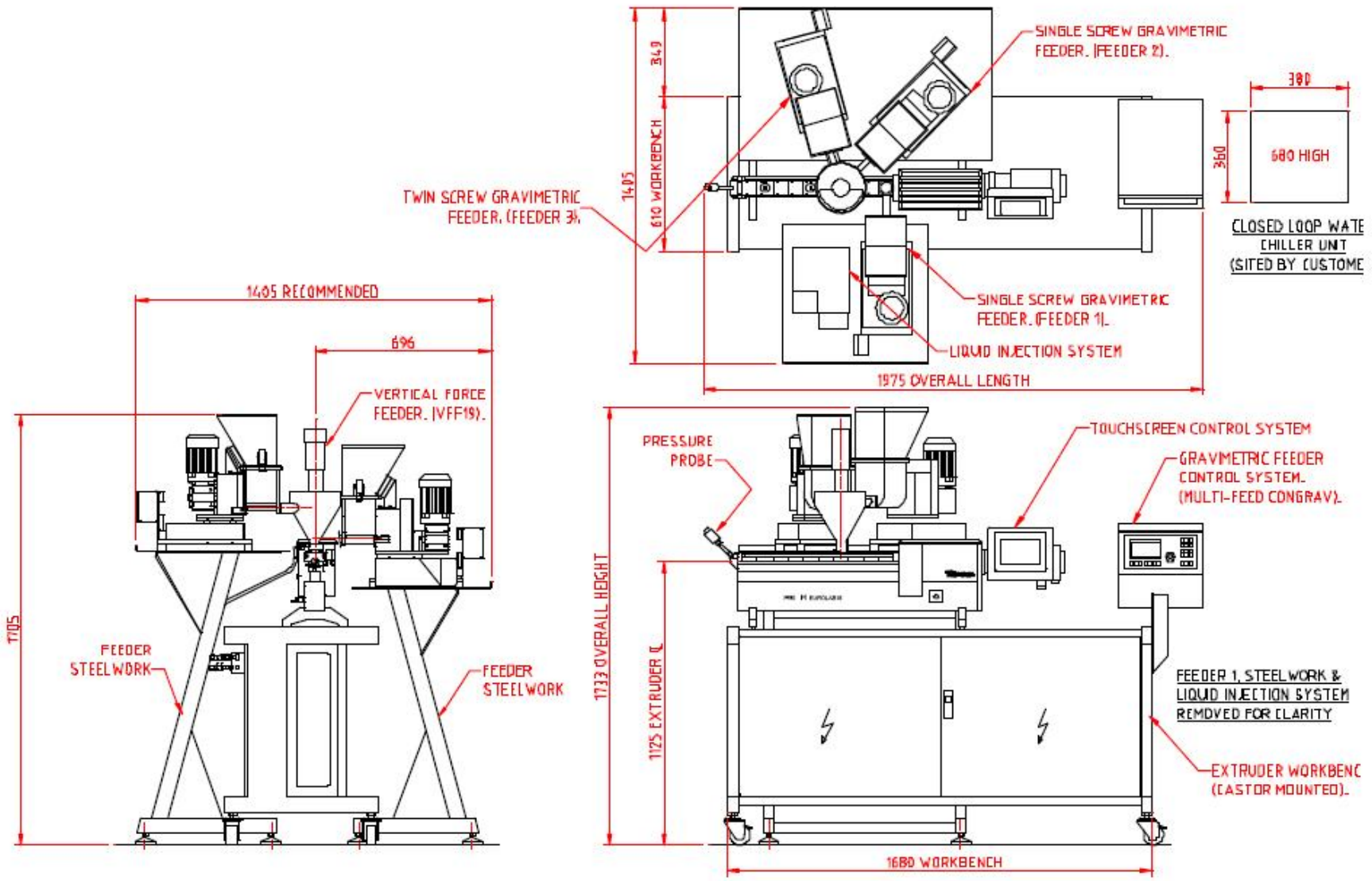
Applications:

- **Sticky Materials:**
  - food extrusion
  - dough / ceramic compounds
- **Materials that tend to "bridge"**
  - some PVC Dry Blends
- **Non-Flowing Material**
  - shredded film (fluff)
  - soap powder





Example – 2 Feed Ports, 3 Feeders & Stuffer



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Extrusion Rheology

Rheology Principles PPT

Detailed Explanations for how PolyLab is used to measure rheology data

PolyLab versus standard Piston Capillary Rheometers

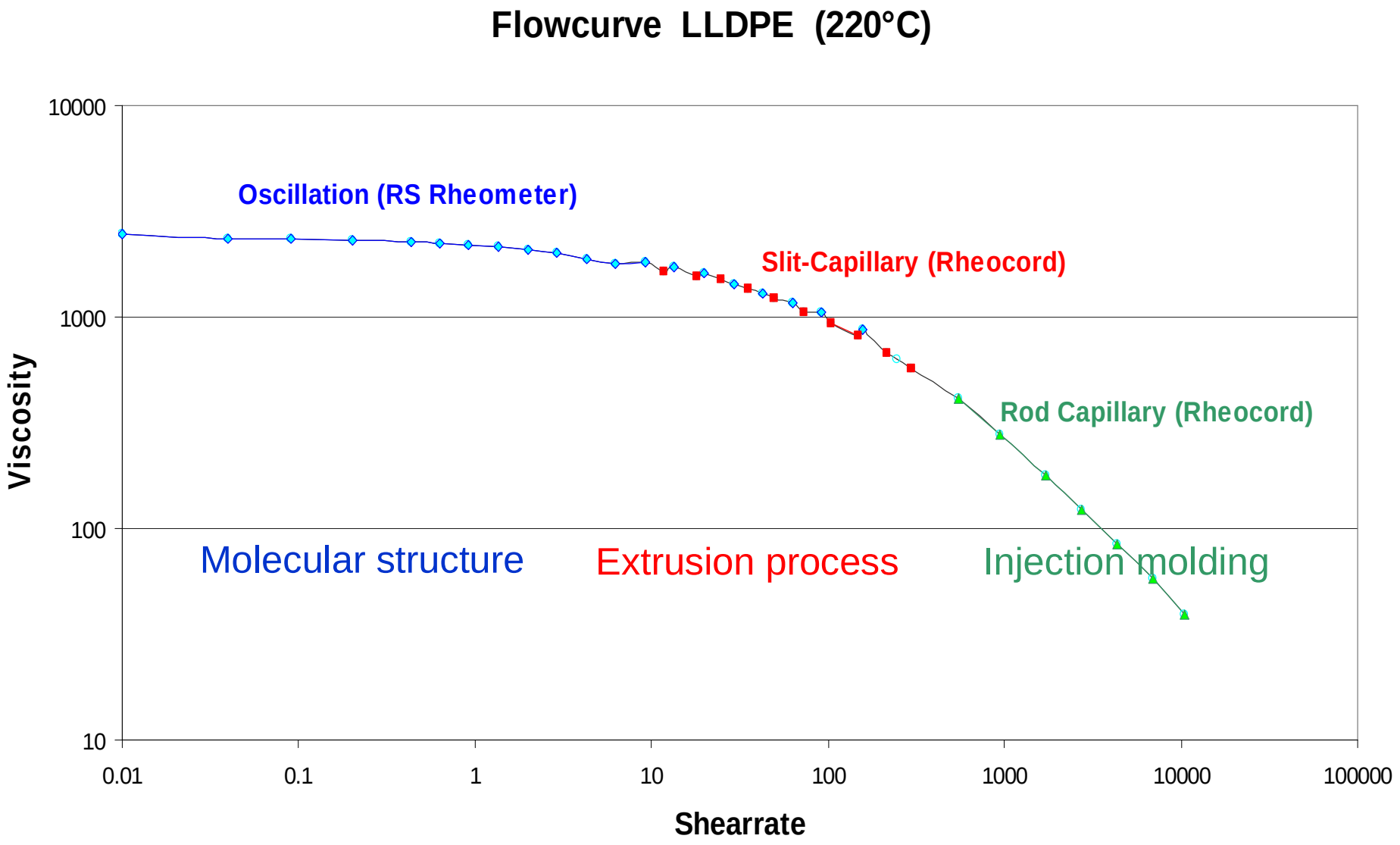
§ Advantages

- Lower “entry cost” to have capillary rheology data
- Lower overhead costs long term (space, maintenance, etc)
- Melted/Mixed multiple components directly to rheology
- Eliminate an additional melting heat history for melt mixed compounds
- Simulates “real-world” processes more closely
- Availability of Slit die for lower shear rates
- Availability of X-Die and “wedge” die for Extensional Rheology

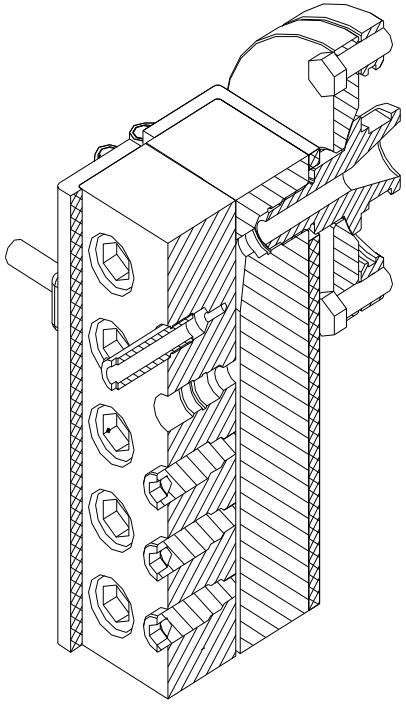
§ Disadvantage

- Uses more material than a piston type capillary rheometer
- Shear Rate range with TSE – use Melt Pump option

Complete Rheological Characterization



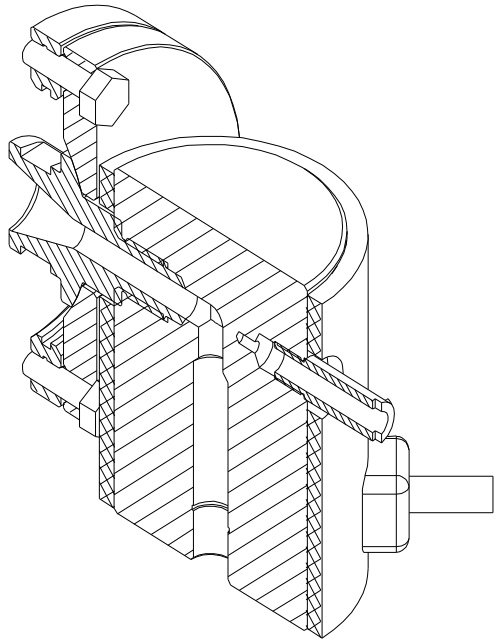
Extrusion Rheology Technique



Slit Capillary Die

(lower shear rates)

- Extrusion Rheology requires knowledge of the “shear stress” and “shear rate”.
- A well defined flow is created in the Capillary Die which allows the pressure drop to be accurately measured.
- The software uses this pressure drop and the known geometry to determine the shear stress.
- ½ of the equation is now known and next the “shear rate” portion must be determined.



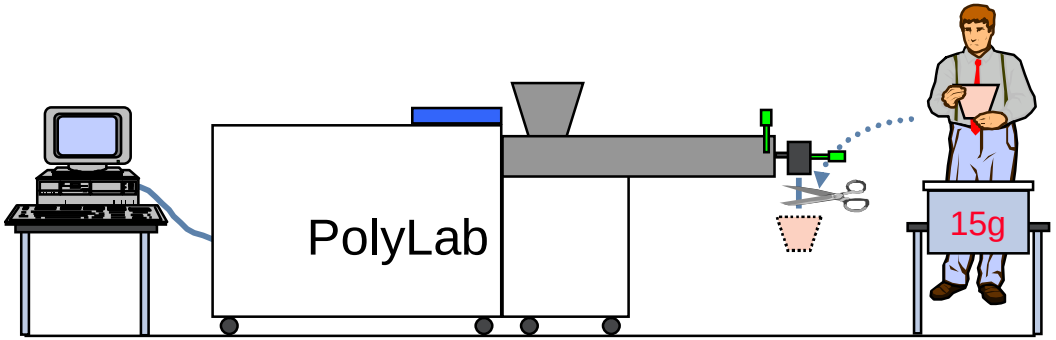
Rod Capillary Die

(higher shear rates)

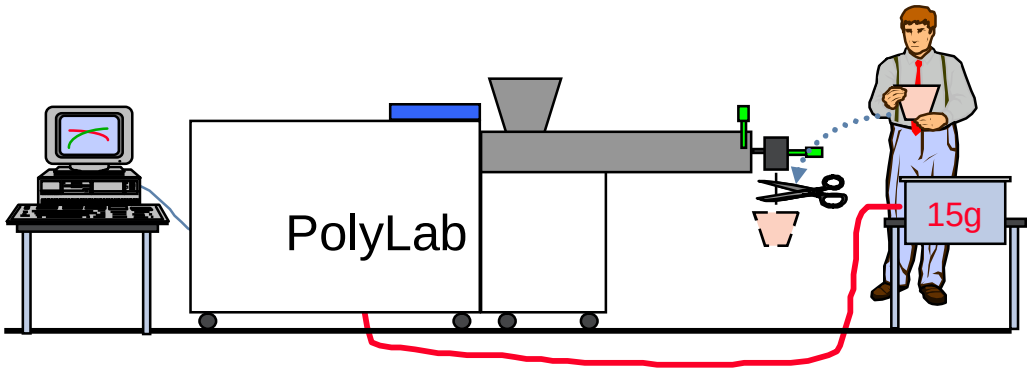
Extrusion Rheology Technique

- To determine the shear rate ½ of the equation, the volumetric flow rate must be determined during the time interval the shear stress is measured.
- This can be done by weighing a timed interval sample in a variety of methods and then allowing the PolyCap software to use an input melt density to calculate the volumetric flow rate.
- Alternately, using a calibrated melt pump, the speed of the pump determines the volumetric flow rate directly, independent of melt density. This is the optimal way to perform extrusion rheology but involves the significant cost of the melt pump.
- For occasional rheology needs, the weigh balance technique is by far the most cost effective assuming the application need will tolerate the compromises, like differing shear input due to ramping the extruder speed to create different data point pairs.
- The PolyCap software also performs or applies corrections for entrance and exit effects (Bagley) and non-newtonian flow (Rabinowitsch).

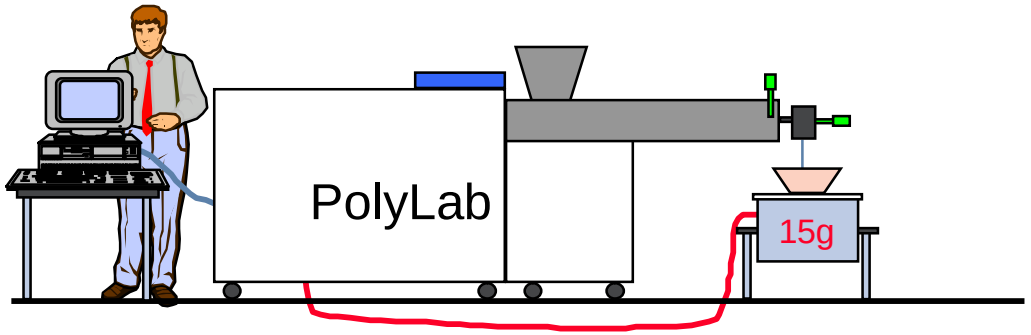
Extrusion Rheology Configurations



Using any lab balance, sample is manually Cut, Weighed, Input to PolyCap software

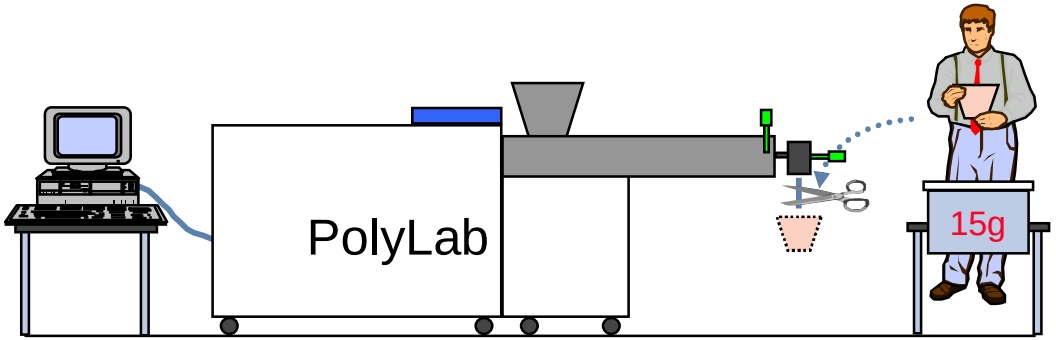


Using a balance connected to PolyLab, sample is manually Cut, weight auomatically goes into PolyCap



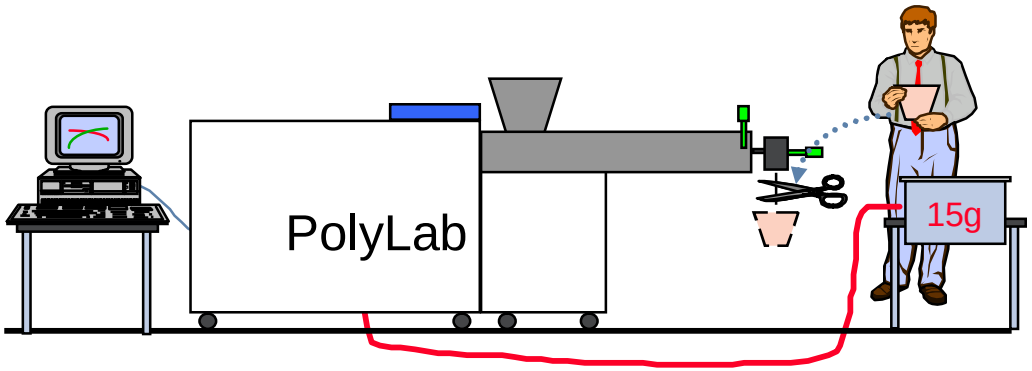
With connected balance placed beneath capillary die, extrudate causes a weight gain with time that PolyCap can detect either by: “Hot Key” or Automatically.

Extrusion Rheology Configurations



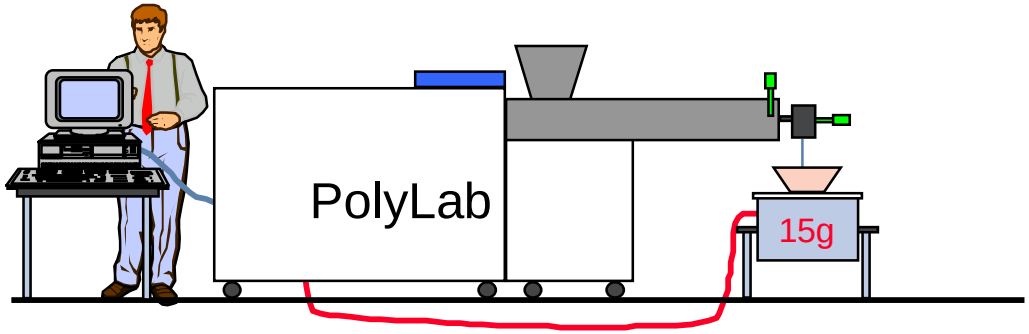
Pro: all materials, lowest cost

Con: most labor/time intensive



Pro: works with any material

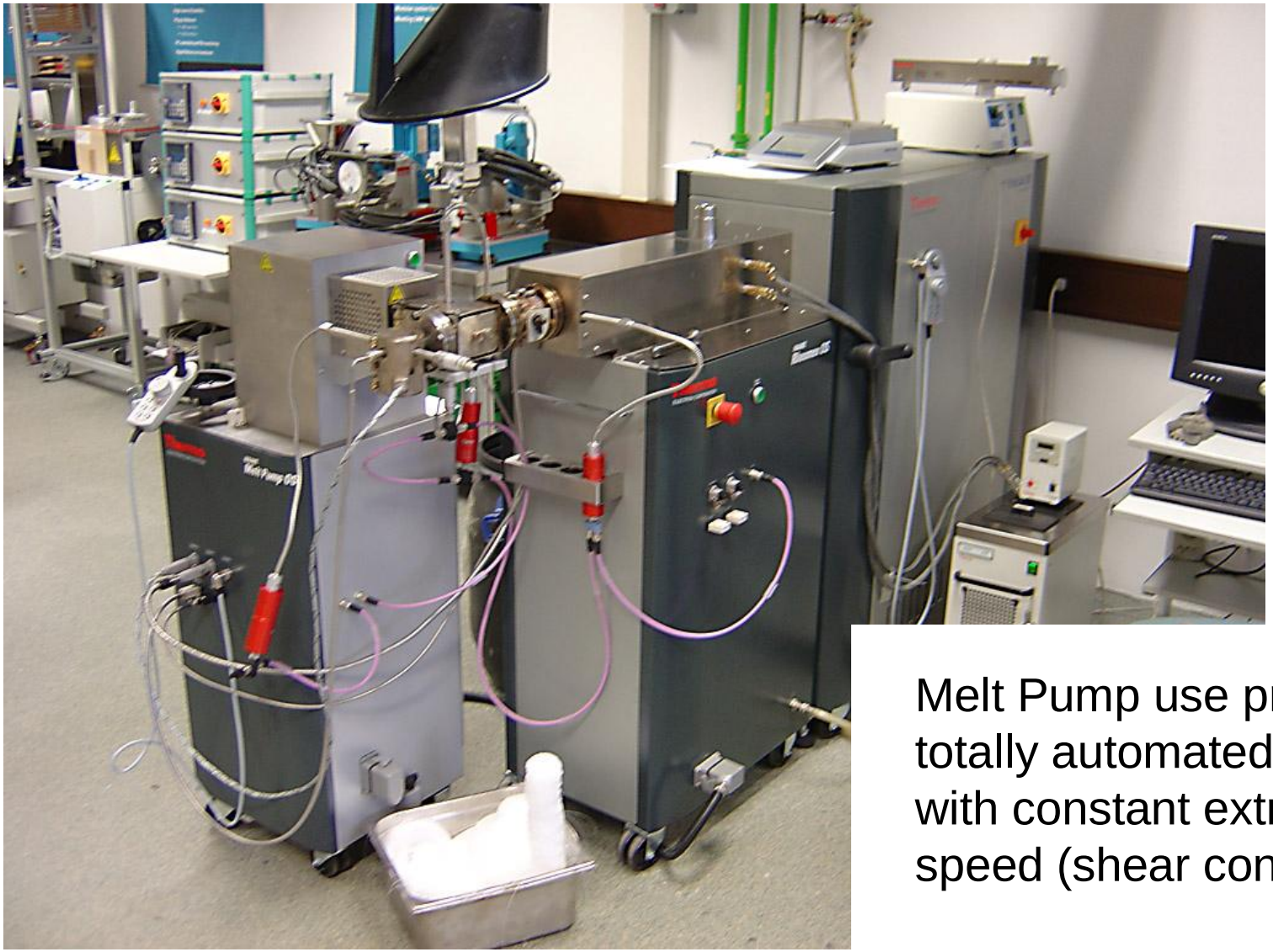
Con: labor/time intensive



Pro: automatic mode only works with materials that stay semi-molten

Con: erroneous data if extrudate “pushes” on balance

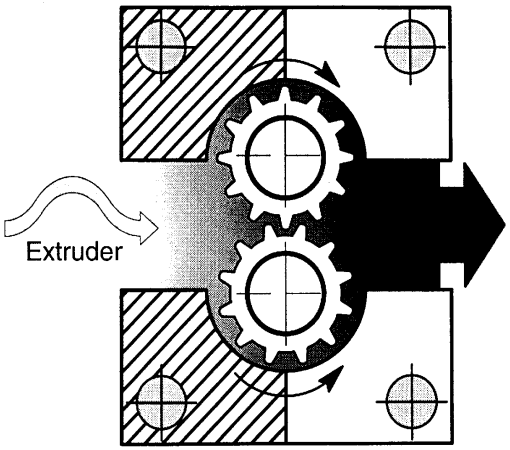
PolyLab / Melt Pump Setup for Capillary Rheology



Melt Pump use provides totally automated testing with constant extruder speed (shear conditions)

MeltPump OS

- Meters accurate flows and greatly dampens pressure pulsations and surging
- Fully CAN controlled MeltPump
- 3 integrated CAN Temperature controllers
- Digital Servo Motor for high accuracy even at low speed (i.e. for accurate rate control for viscosity tests at low shear rates)



MeltPump OS

- Hand wheel for height adjustment
- Remote control for manual speed control
- Automatic speed control via monitor software & capillary software

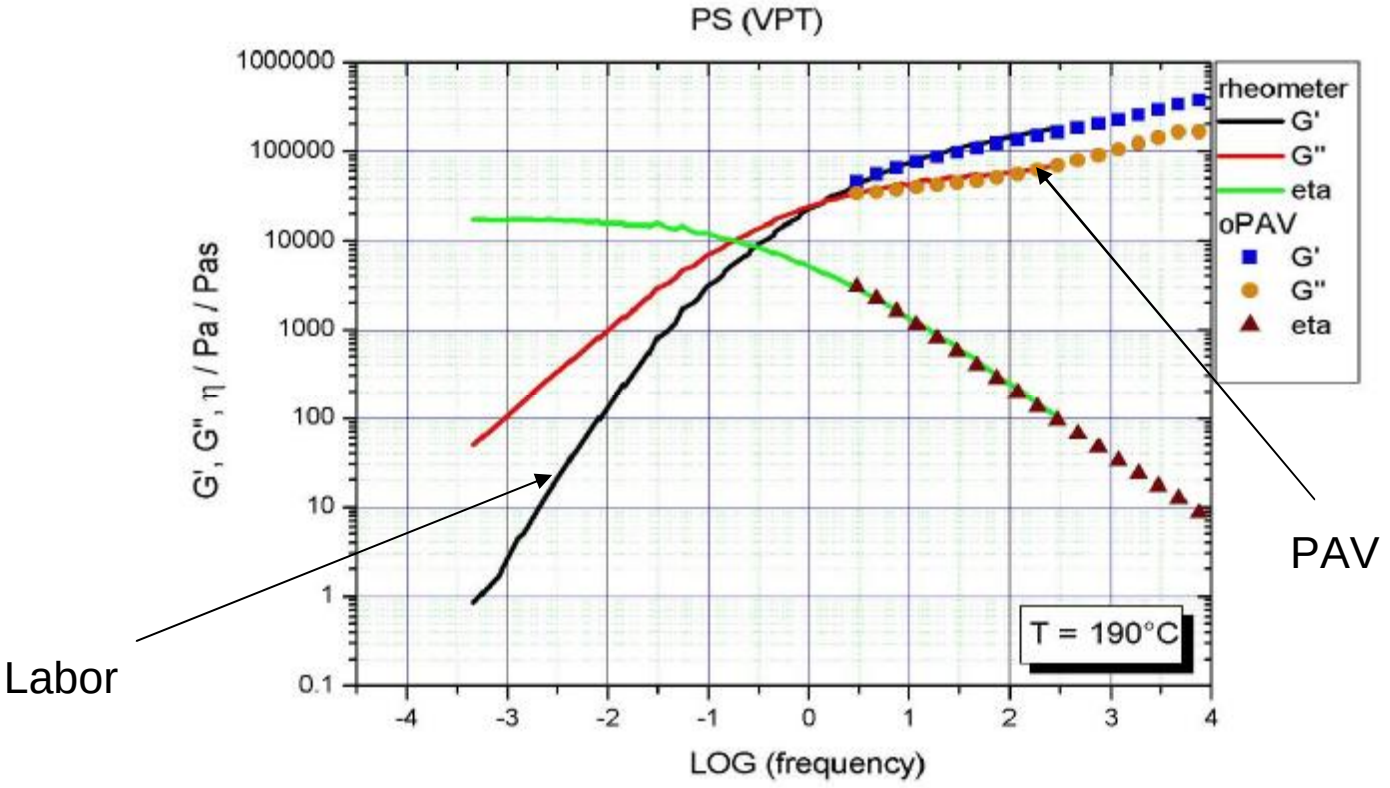
MeltPump OS

External By-Pass Valve:

- MeltPump can be used with & without a By-Pass
- By-Pass can also be used independently from the MeltPump (i.e. for rheological tests with rigid PVC)

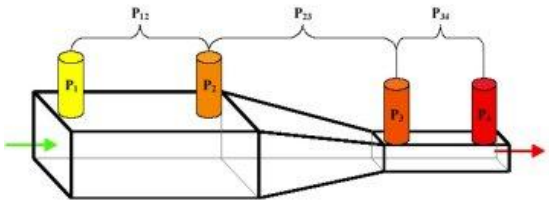
Physical test methods – G’, G” = f(ω)

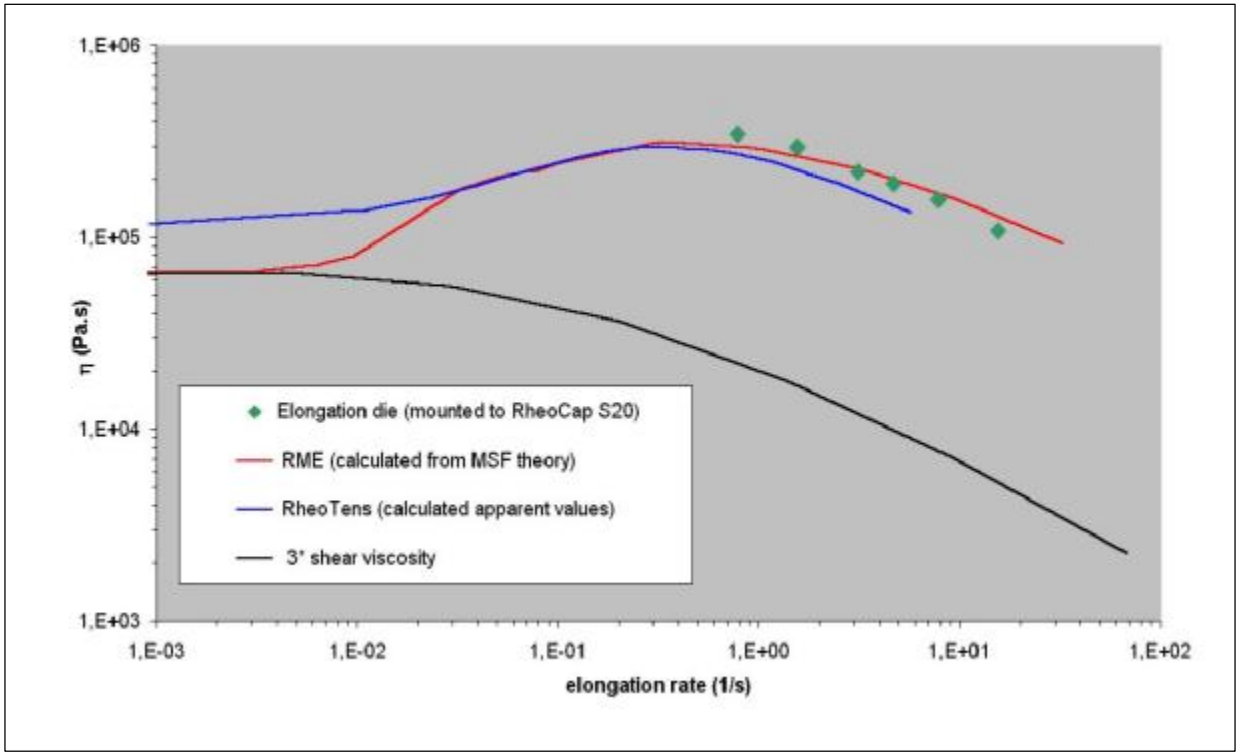
The viscosity spectra is measured with an axial oscillating rheometer in the polymer melt. The sensor is mounted in a pressure port.. (Ref.1) The measurement principle is oscillating squeeze flow



Physical test methods– Extensional Viscosity

Using a patented die geometry (US6386016) with an extruder or melt pump, the shear viscosity and the elongation viscosity can be quantified. This correlates well with die swell. Example Ref 3 LDPE at 190°C



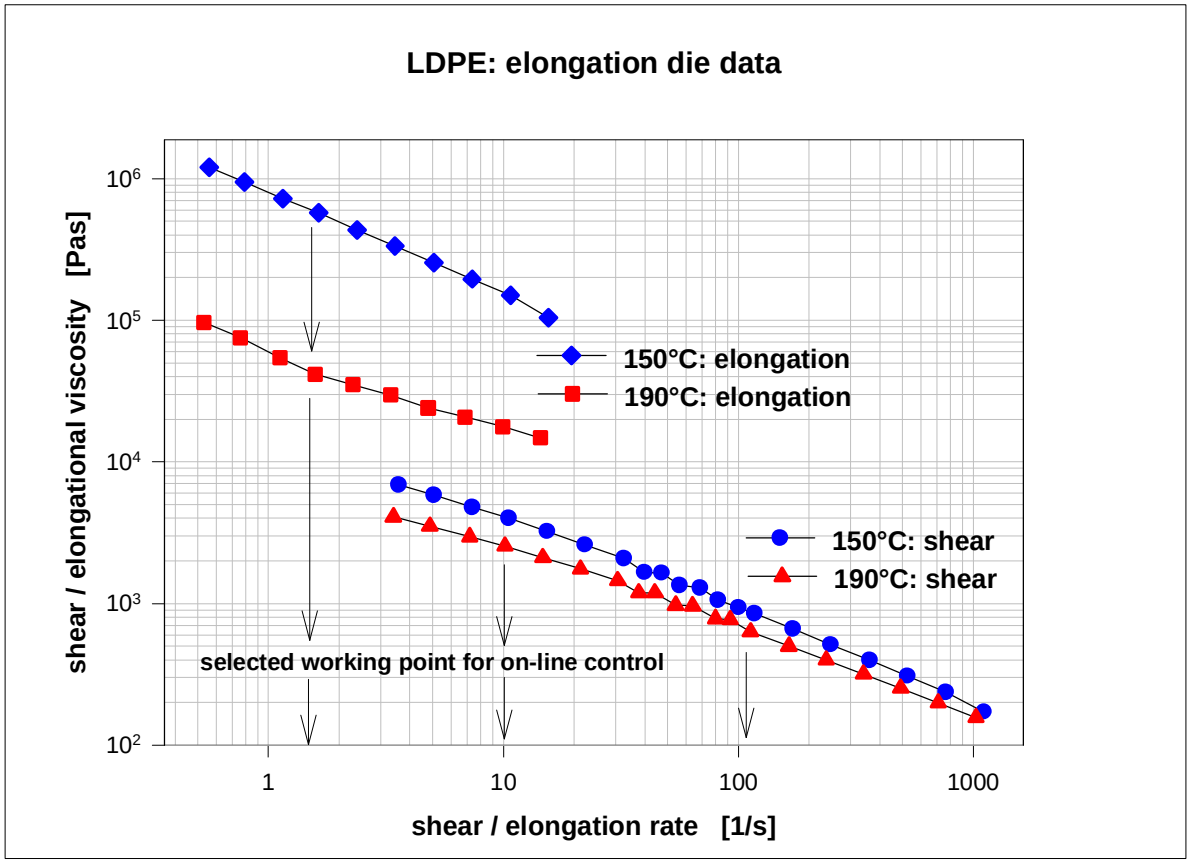


Detailed Literature Available upon request

Physical test methods– Extensional Viscosity

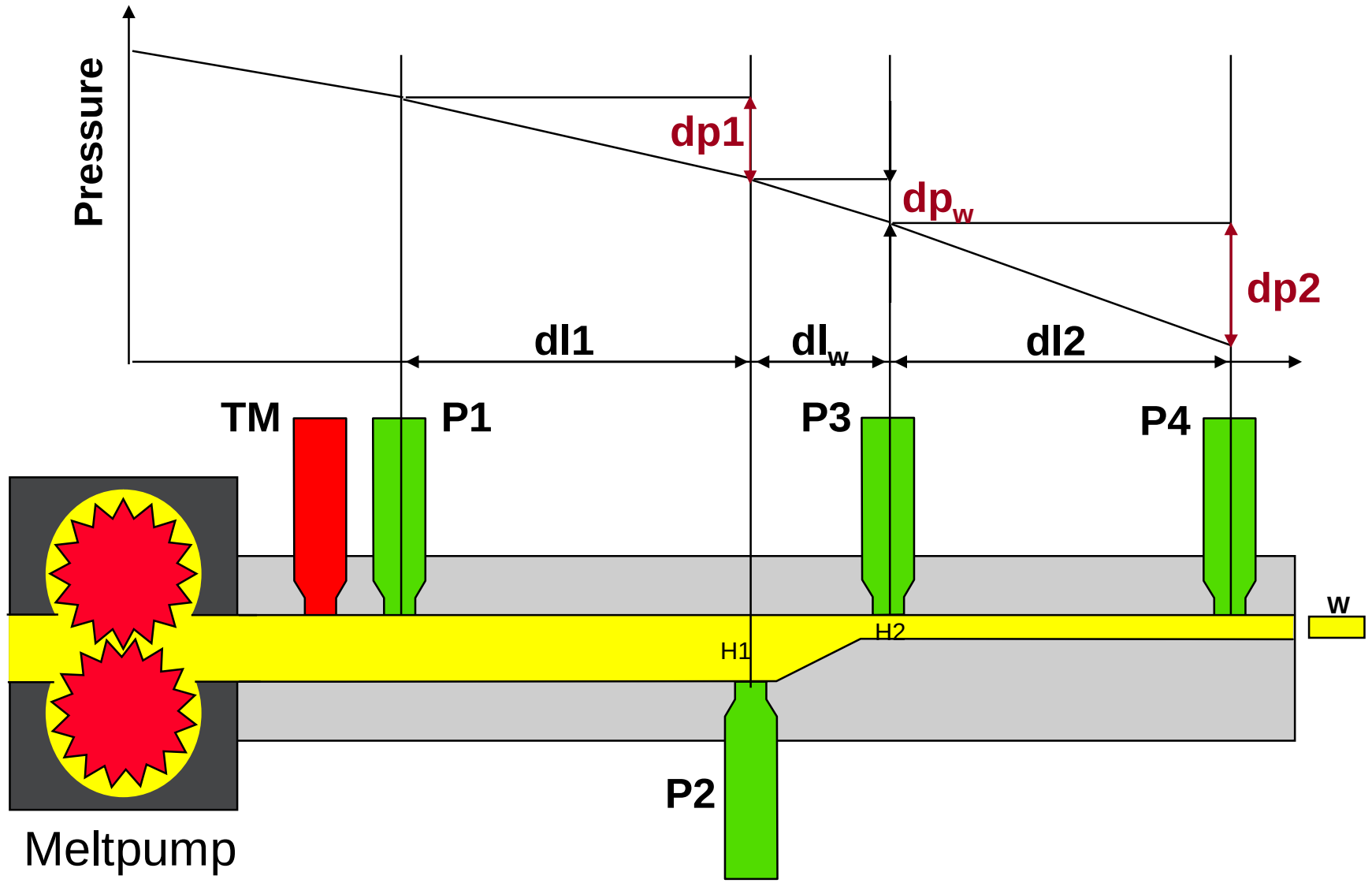
Extensional Properties of polymers are often show much greater change as an indication of morphology or molecular weight changes.

Thus, the interest in using this data and measuring technique for process development and control.



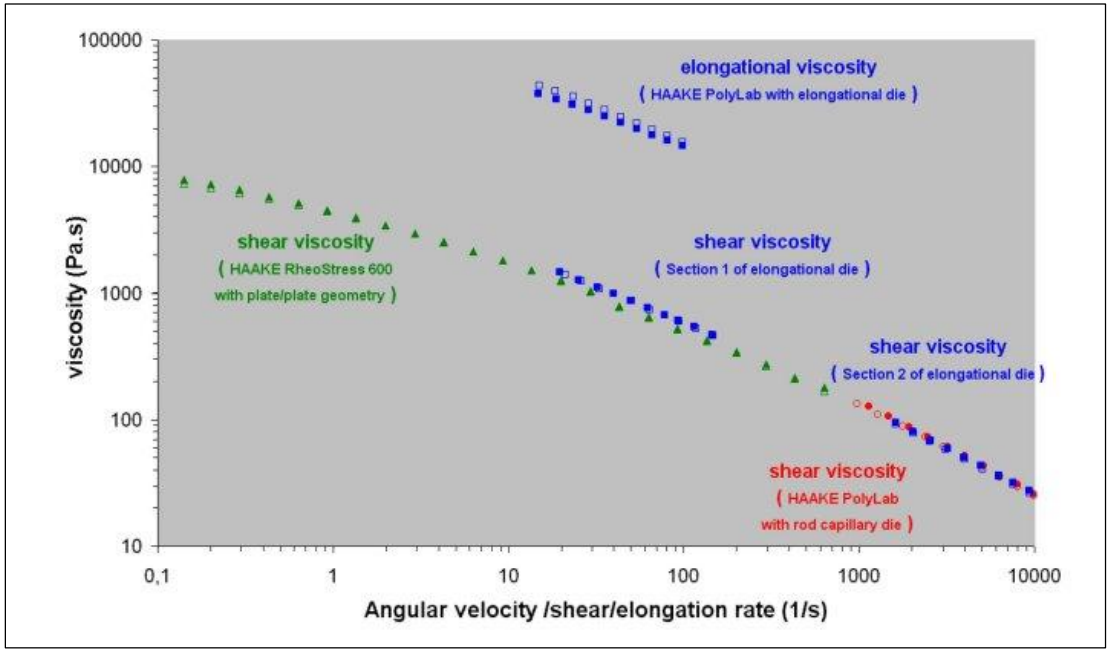
Hencky strain = 3.35 for that die

Extensional Rheology Die - Principle



Physical test methods – At-Line viscosity/MFI

At-line viscosity tests are performed with a capillary viscometer method (ΔP, ΔV). The melt is taken from the extruder and pumped in a separate rheometer (HAAKE ProFlow). Depending on the die used one can see MFR, viscosity curve or even extensional viscosity.



Additional information available upon request

ThermoFisher SCIENTIFIC

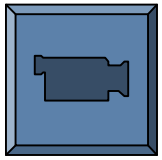
The world leader in serving science

Post Extrusion Testing Options

Cast & Blown Film applications

Accessories Needed:

- Any PolyLab Extruder
- Die
- Take-off Unit
- Pressure sensor
- (Screen package)
- (OQT 512)



Cast Film Video
(older TSR take-off)

§ Application:

- Small production of films
- Testing the processibillity
- Testing of additives
- Testing and optimization of processing conditions

§ Results / Specimens for:

- Gel / Fish-Eye testing
- Pigment distribution
- Color
- Surface quality
- Tensile / Impact testing
- Weathering

PostEx - Tape, Sheet and Ribbon Take-Off

New Sheet & Ribbon TakeOff

- New design to be used with Haake and Prism products

- Two Options:
  2-Roll Take Off
  3-Roll Take Off

- Easy access to the rollers

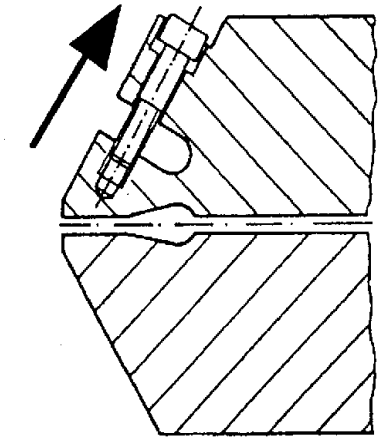
- Easy handling

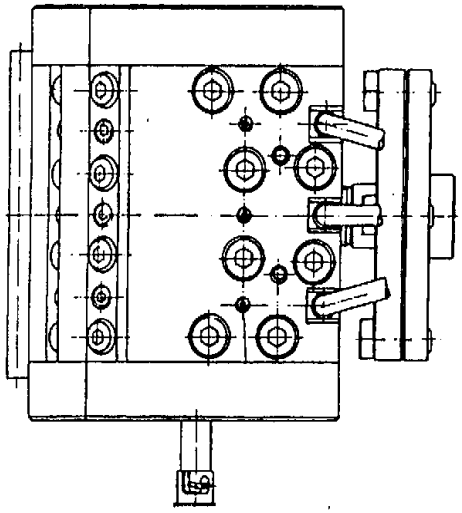
- Height adjustable

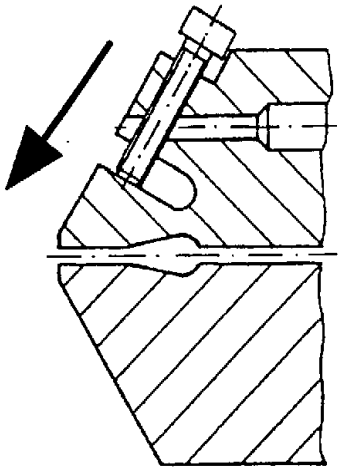
- Adjustable speed ratio between the steel rollers and the take-off rollers for slight drawing or orientation

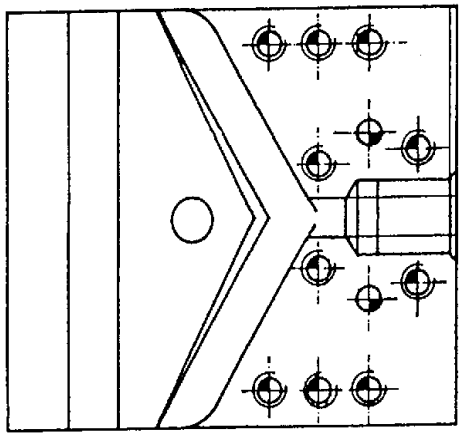
Optional Winder for TSR

Tape, Sheet and Ribbon Die









- **Die width:**

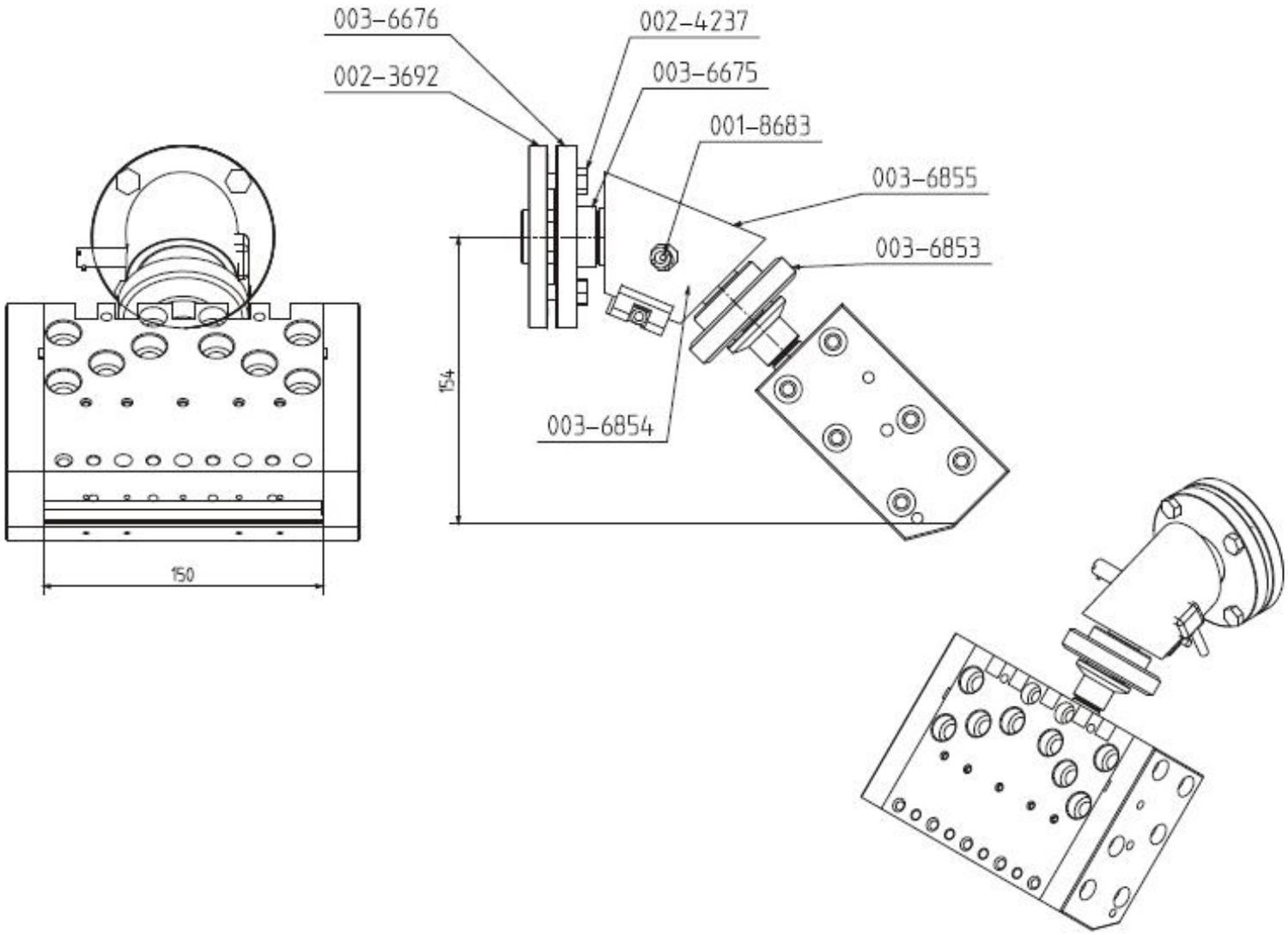
  25/50/100/150 mm

- **Die gap:**

  adjustable (standard: 0.2-1.2 mm)

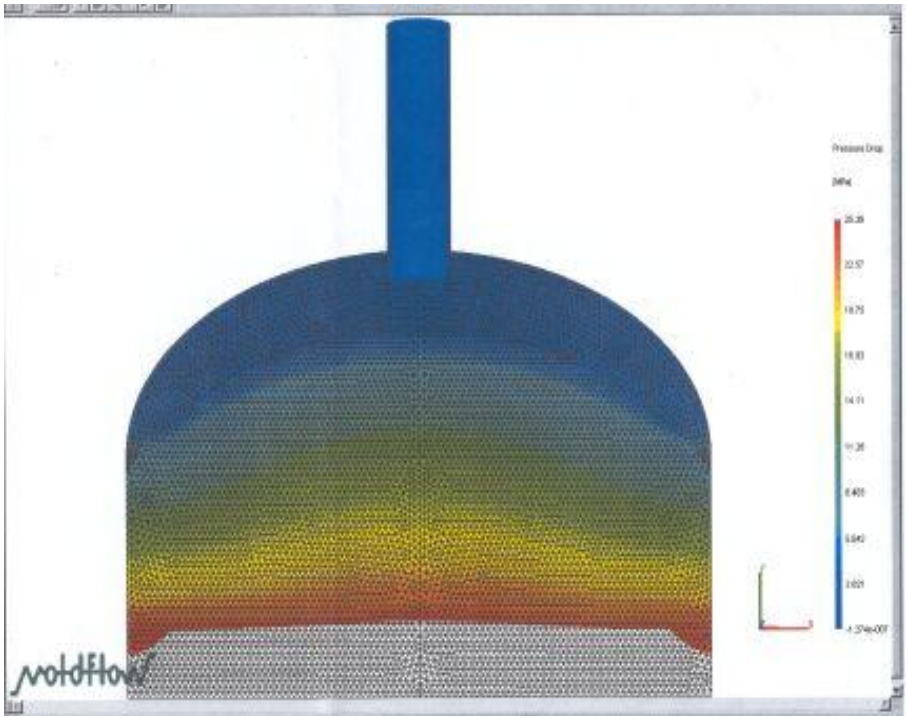
- **fixed die gap on request**
- **Max. temperature: 360°C**
- **1 measuring port**

Angled Adapter for Downwards Sheet Casting

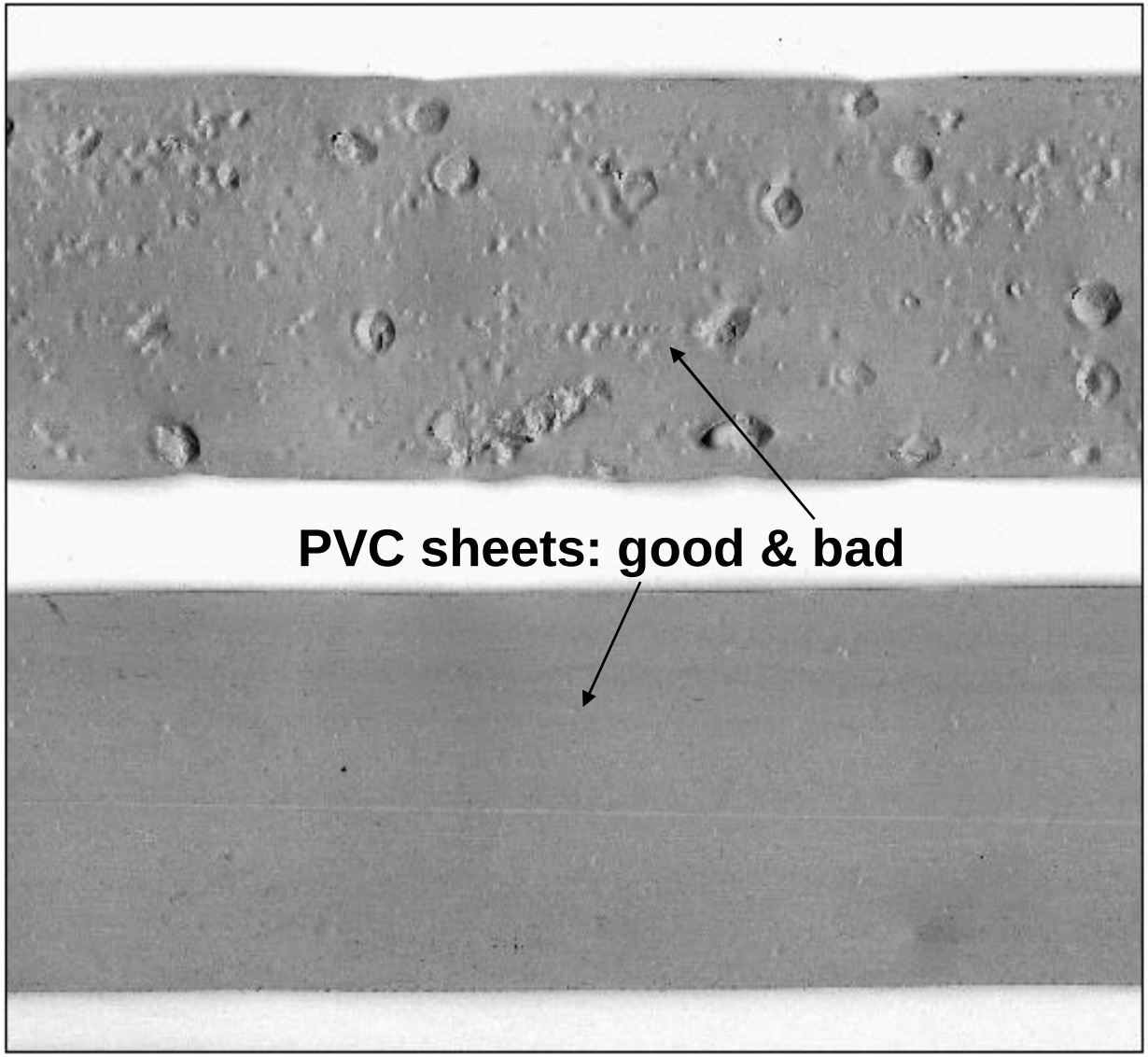


Tape, Sheet and Ribbon Die for PVC

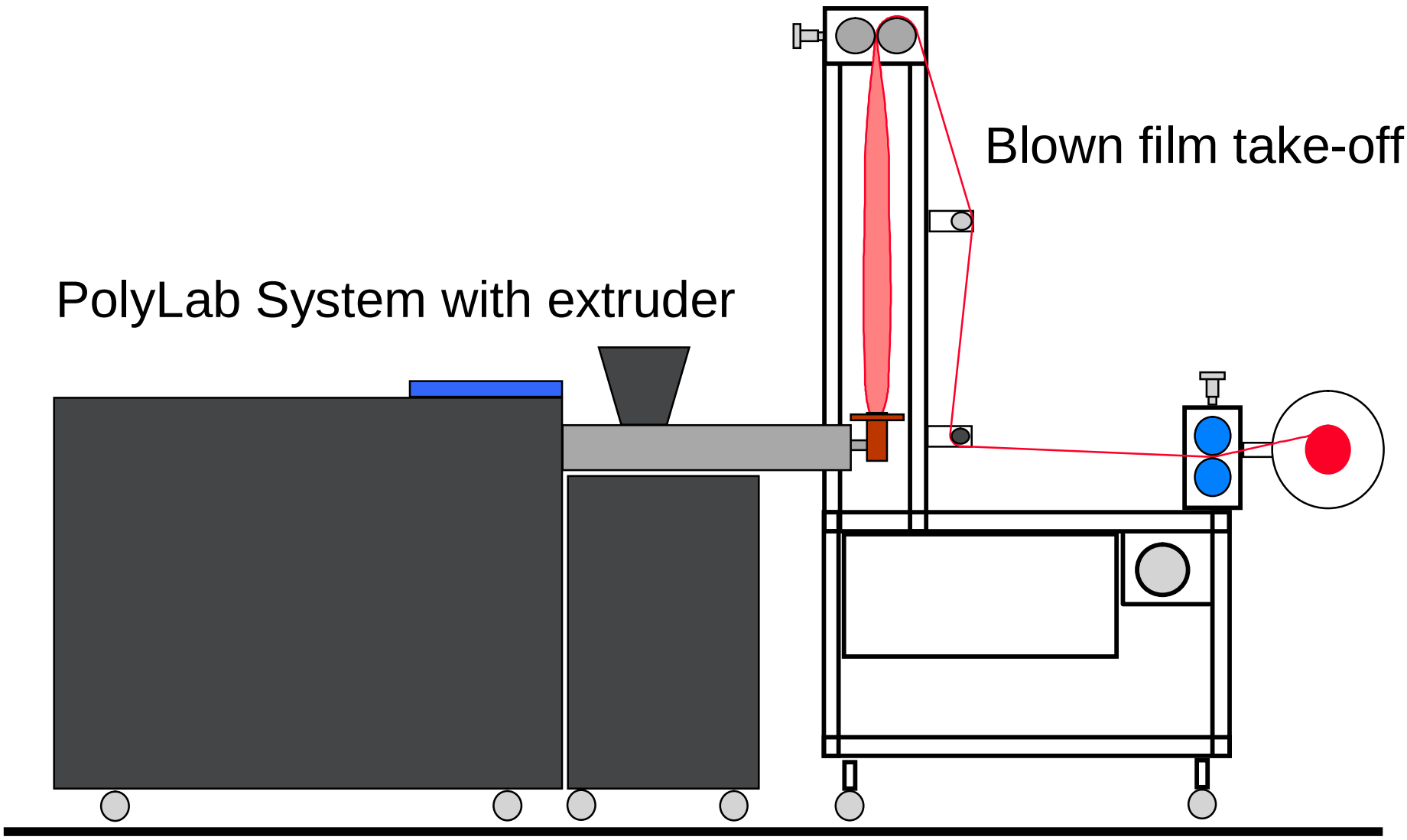
- PVC Sheet dies: 50mm, 100mm, Height 1mm
- designed using Moldflow (FEM) Simulation
- Optimizied Flow - Compact design – Fixed Die Lip



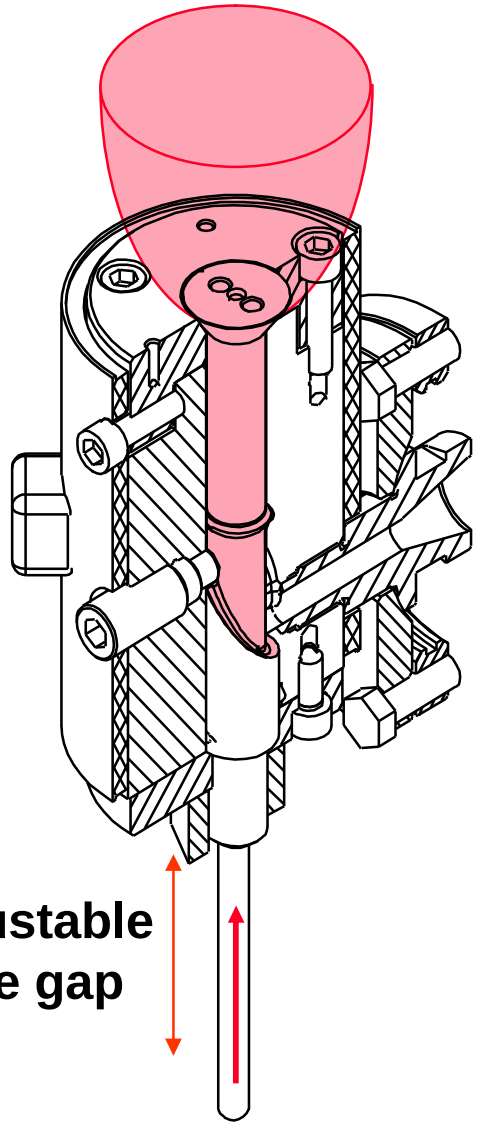
Cast film application



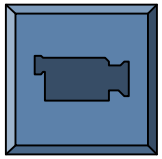
PostEx - Blown-Film Take-Off



Blown Film Die



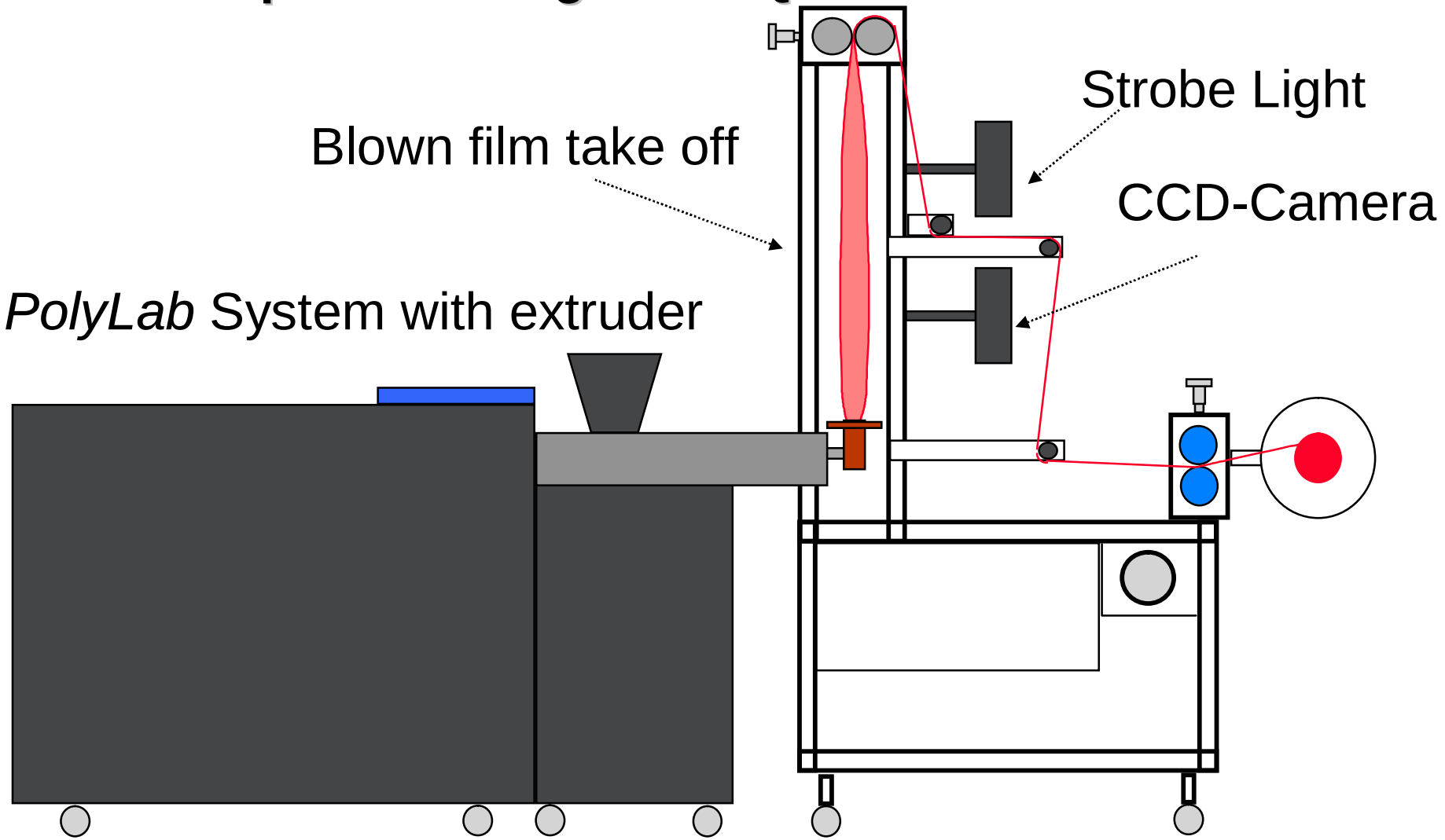
- Ring diameter 25mm (35mm)
- Pin diameter 24mm (34mm)
- Gap width 0-0.8 mm
- Measuring ports 2
- Max. temperature 480°C



Blown Film Video

PolyLab Blown Film Optical Testing

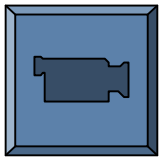
With optical testing unit OQT



Optical Test Methods – Film Quality / Gels

With a chilled take-off roll the melt is pulled from a sheet die and transported over a line camera. The spots are detected and statistically evaluated.

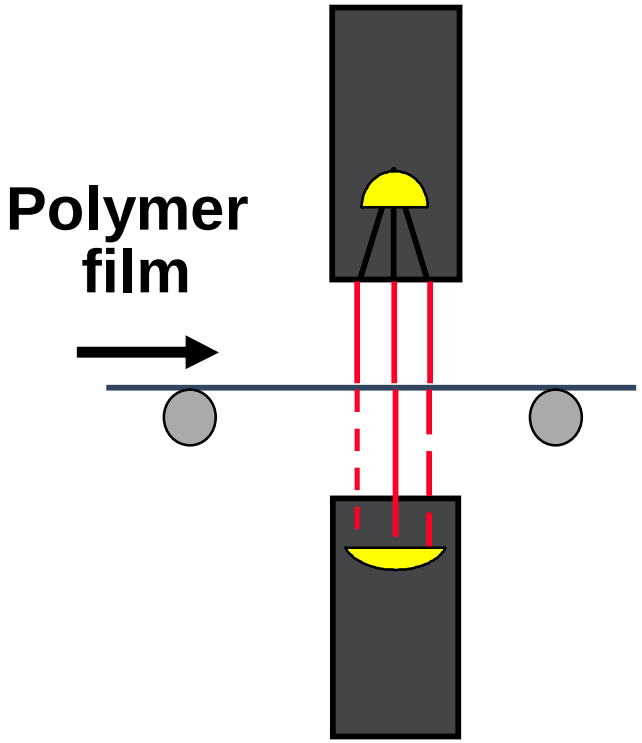
Camera system

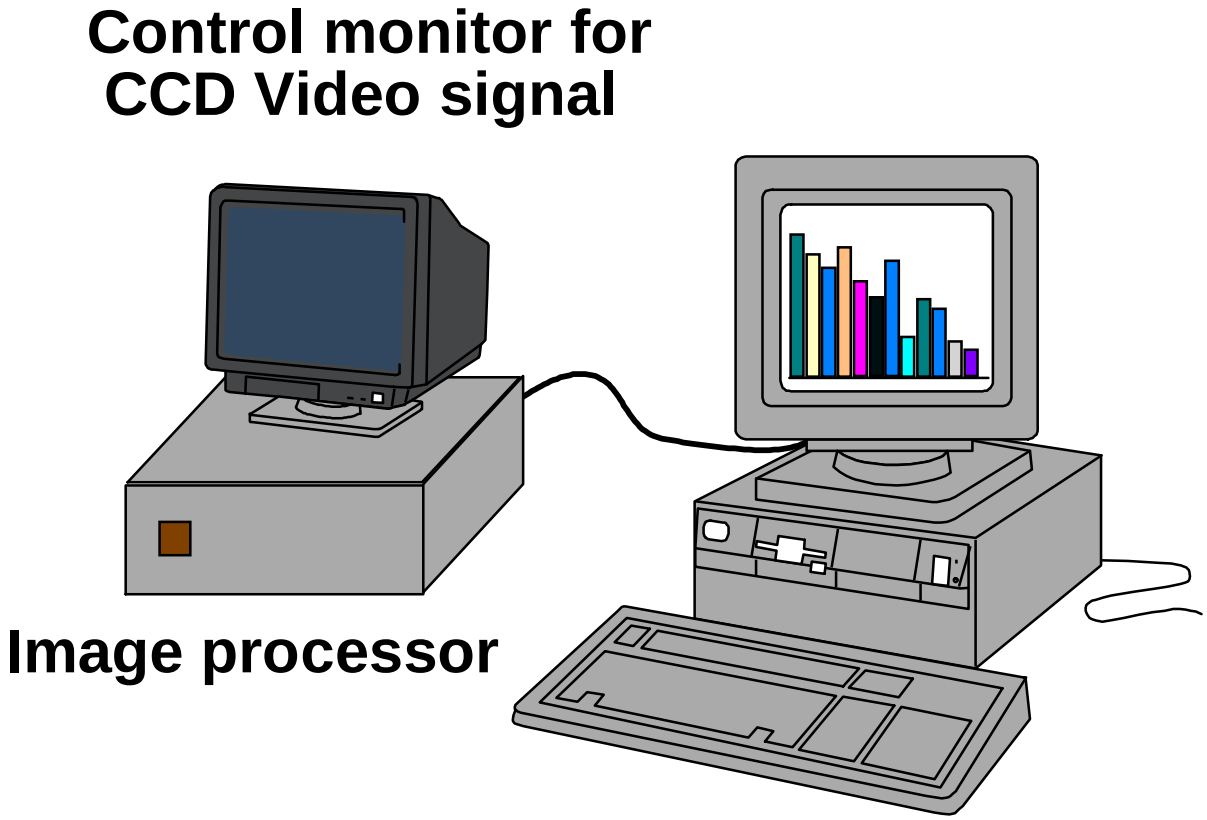


Blown Film Video

Optical Quality Testing System

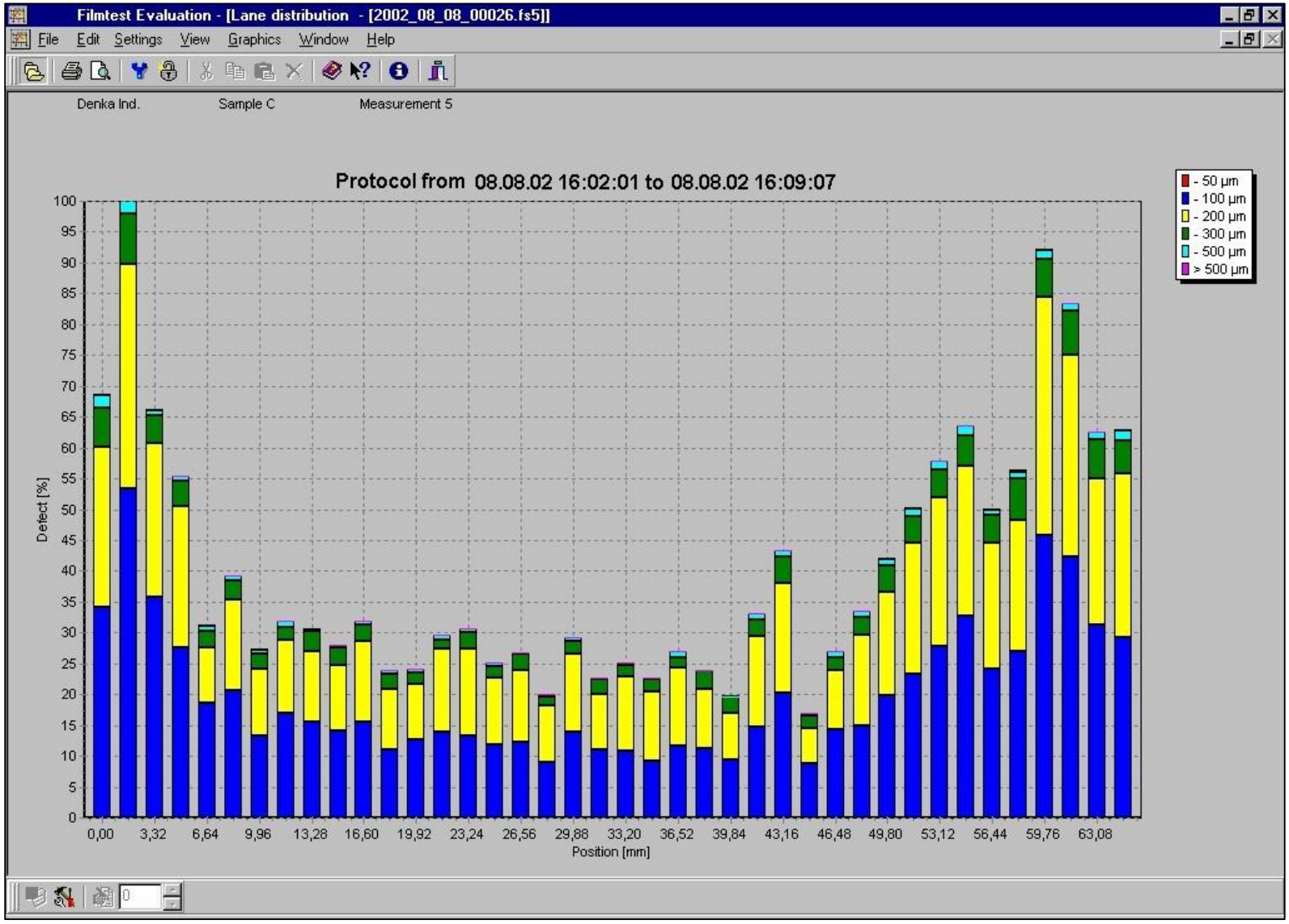
Measuring principle:





OQT- Image display of defects

OQT- Graphical display of defects



Optical Quality Testing System OQT 512

Technical data:

Camera:	**CCD Area Camera**
Frequency:	**4 pictures /second**
Test area:	**20 x 20 mm (standard)**
Resolution:	**60 µm (20 x 20 mm test area)**
Options:	**10 x 10 mm test area (30 µm resolution)**
	100 x 100 mm test area (100 µm resolution)
Number of size classes:	**20**
Detection levels:	**2**

(differentation of fisheyes and black specks)

Optical Quality Testing System OQT FS5

Technical data:

Camera:	**CCD Line Camera**
Line length:	**2048 pixels (standard)**
Inspection width:	**20 to 2000 mm**
Resolution:	**from 10 µm**
Number of size classes:	**10**
Detection levels:	**3**

(differentation of fisheyes and black specks)

Compounding / Pelletizing

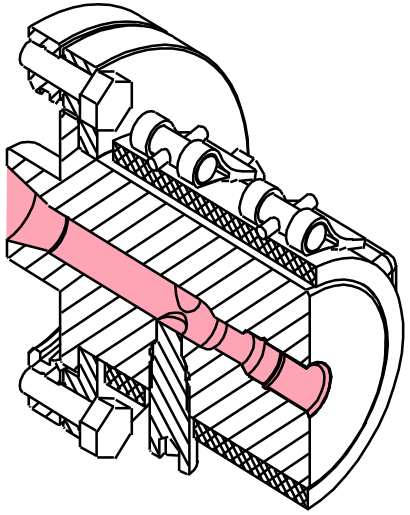
Accessories Required:

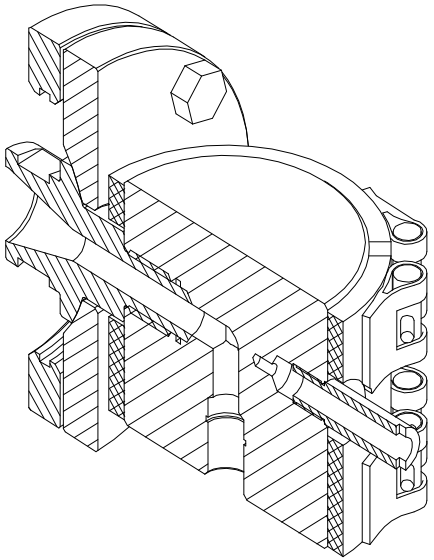
- Any PolyLab extruder
- Pressure sensors
- (Screen pack - optional)
- Rod die
  - Horizontal
  - Vertical
  - Multistrand die
- Strand Cooling
  - Waterbath
  - Air Cooled Conveyer
- Pelletizer
  - Strand type
  - Die Face

Test results:

- Production of small quantities of test materials for:
  - Injection Molding
  - Customer Trials & Samples
  - Additive trials
- Masterbatch development
- Processability of new polymers
- Optimization of processing conditions

Rod/Strand Dies – Horizontal & Vertical



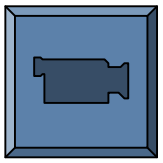


- Universal die for extrudability tests
- Strands for Pelletizing
- Nozzles available from:
  1mm - 6mm
- Two sensor ports for pressure- and temperature-measurement
- Multi-Strand horizontal die also available for higher output pelletizing

testing of Processing additives (Dynamar) at *Dyneon (NL)* which prevent build up at die exit.

EPPM info nr. 34 6,3 / p12 (2004)

Compounding / Pelletizing



Pelletizing Video

VariCut Pelletizer

Profile / Rubber Extrusion

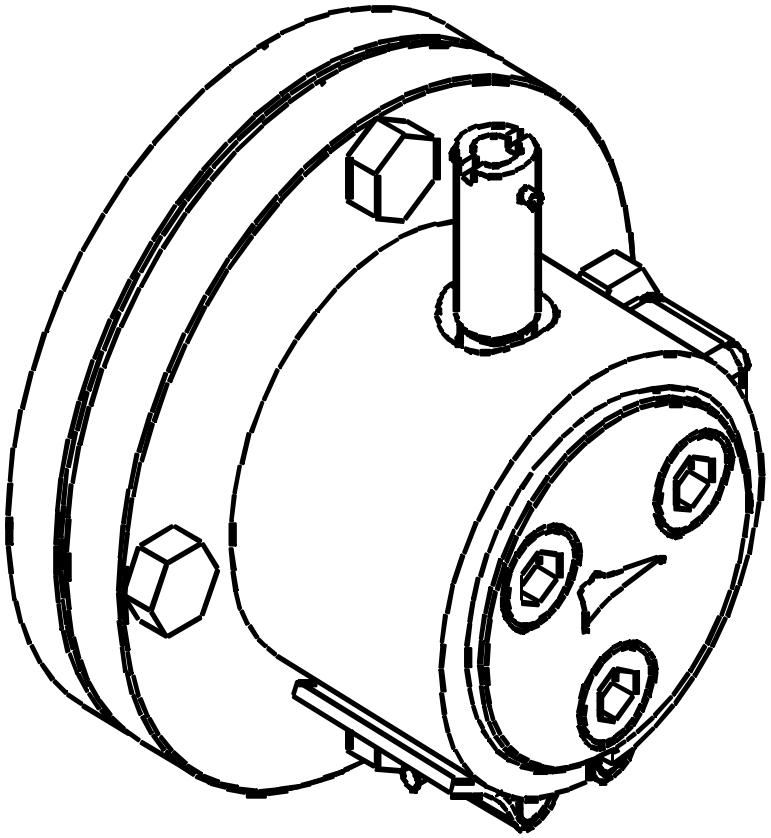
Accessories Required:

- Any PolyLab Extruder
- Pressure sensor(s)
- Profile or Garvey die
- Conveyor belt

Test results:

- Testing of the processability of rubber, elastomer, vinyl, and ceramic compounds
- Testing of the influence of additives
- Testing and optimization of the processing conditions
- Visual testing of the material flow at edges with different angle
  - Sharpness
  - Elastic “memory”

Garvey-die



Technical data:

- Die profile according to ASTM 2230-63T
- max. temperature: 360 °C
- die profile does not allow to install sensor ports

Application:

- Extrusion of elastomers and rubber for testing flowability

Additional Extrusion Dies

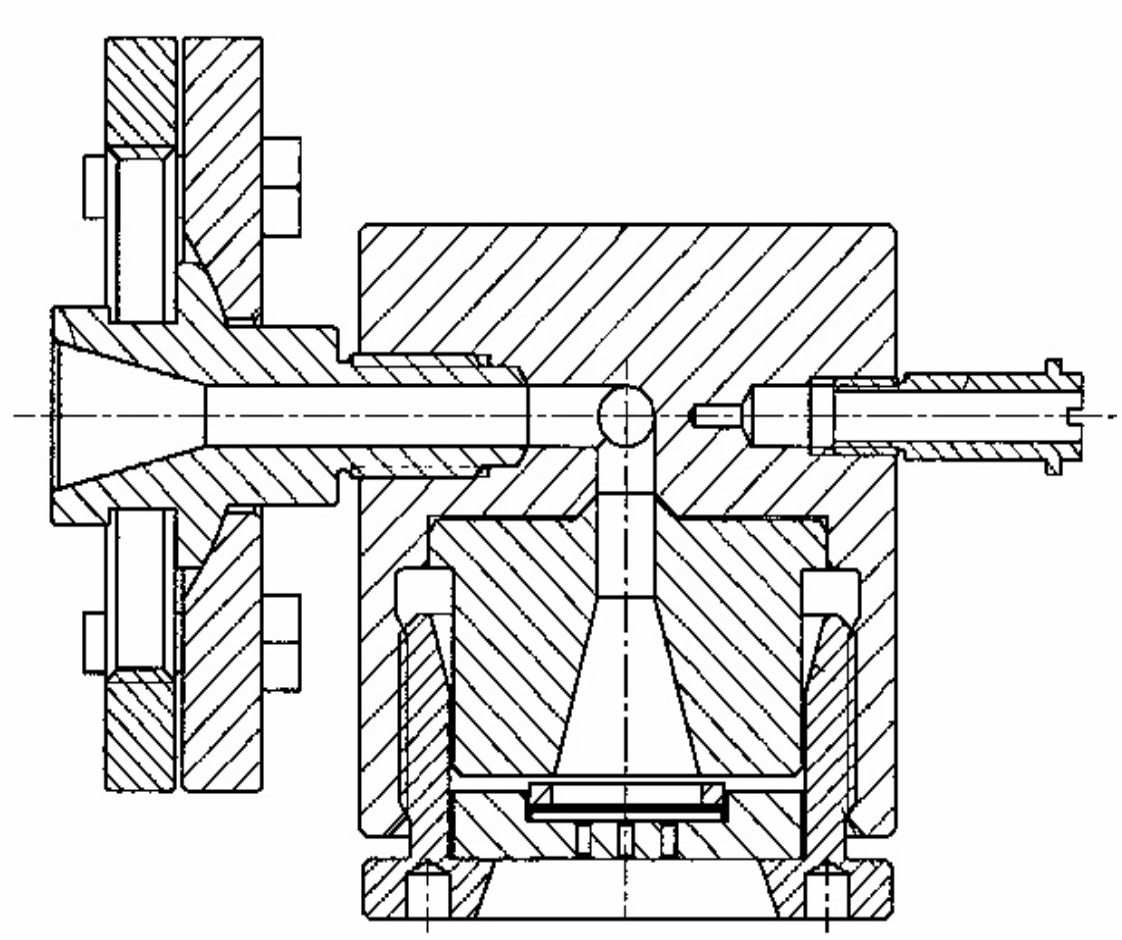
- Due to the wide variety of special processes for polymers, Thermo supplies simple dies for other processes but limits our "standard" offerings on the take-off systems as most are custom designed with special features depending on the polymer system

- Additional Extrusion Dies offered:
  - Filter Test Die
  - Wire Coating Die
  - Pipe / Tubing Die
  - Medical Tubing / Catheter Die
  - Fiber Spinning Die (multi-filament or mono-filament)

Filter Test Method Details

- Thermo MC will also assist with dimensionality of the extruder adapter required to mount custom Dies such as "Guill Tooling" and others

Filter Die

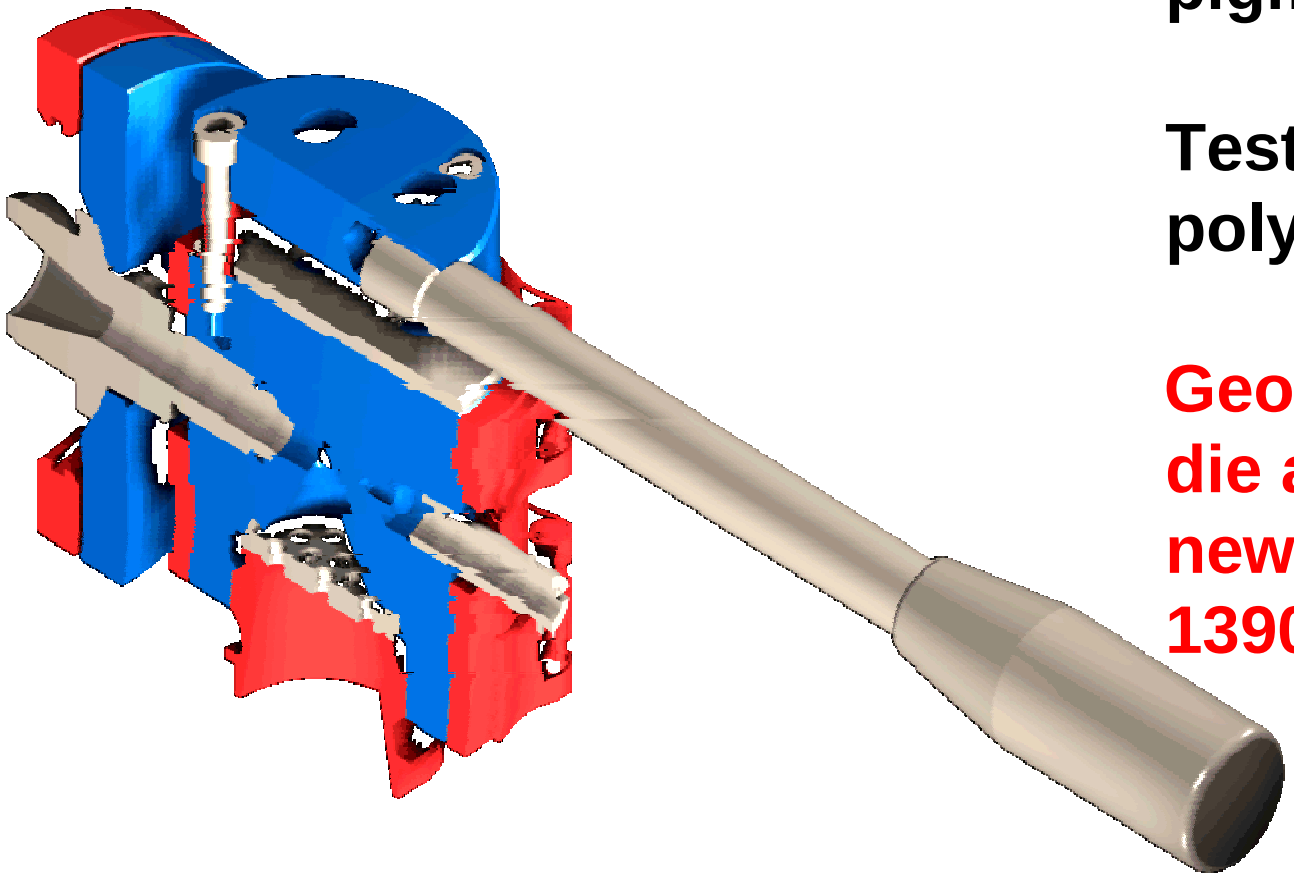


Measurement of the pigment distribution

Testing of the purity of polymers

Suitable for different filter-dimensions

Filter Die *(according EN 13900-5)*



Measurement of the pigment distribution

Testing of the purity of polymers

Geometry of the filter die according to the new standard EN 13900-5

Filter test dies with breaker plate and central quick locking screw

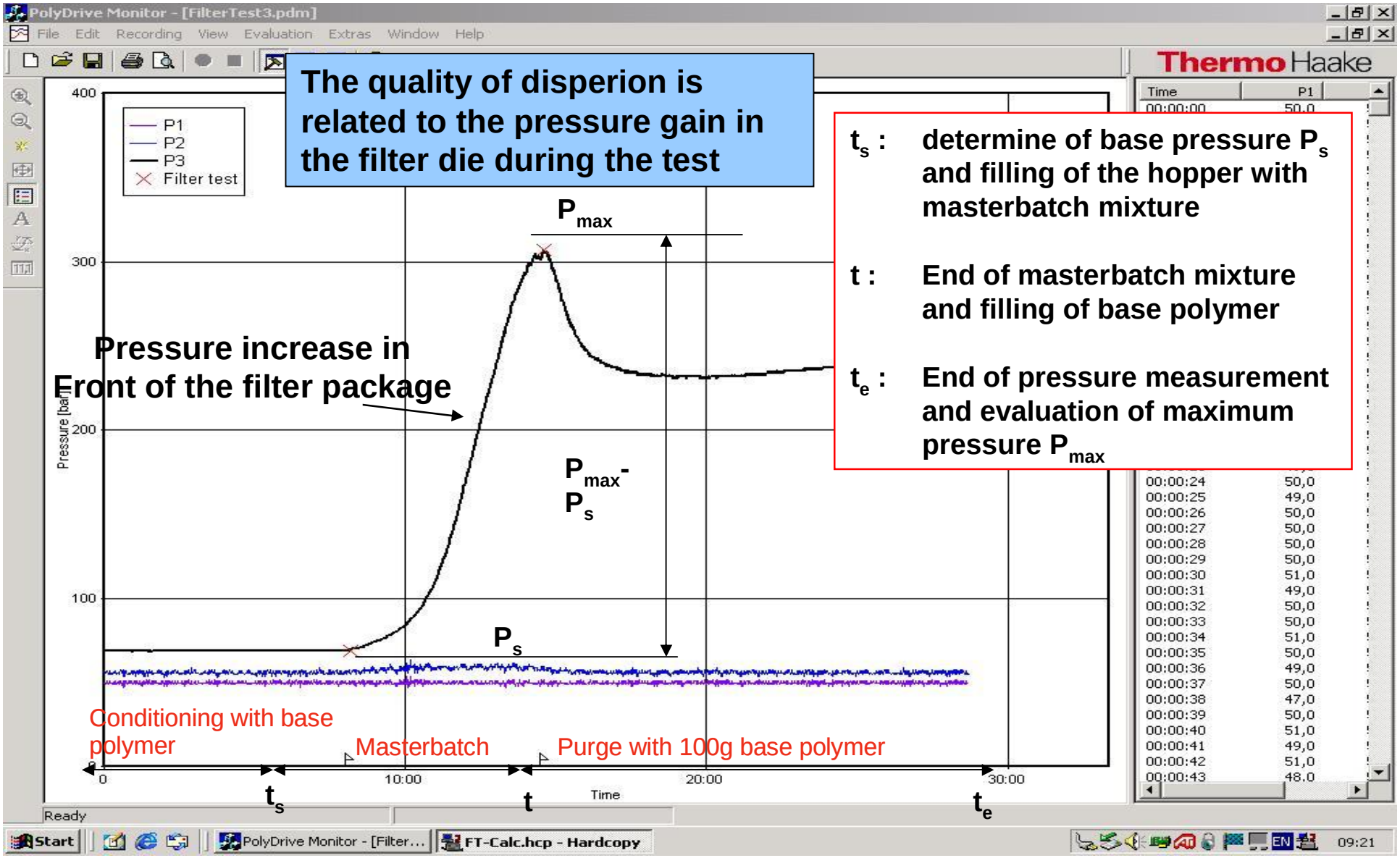
Filter Pressure Value test EN 13900-5

Test Procedure:

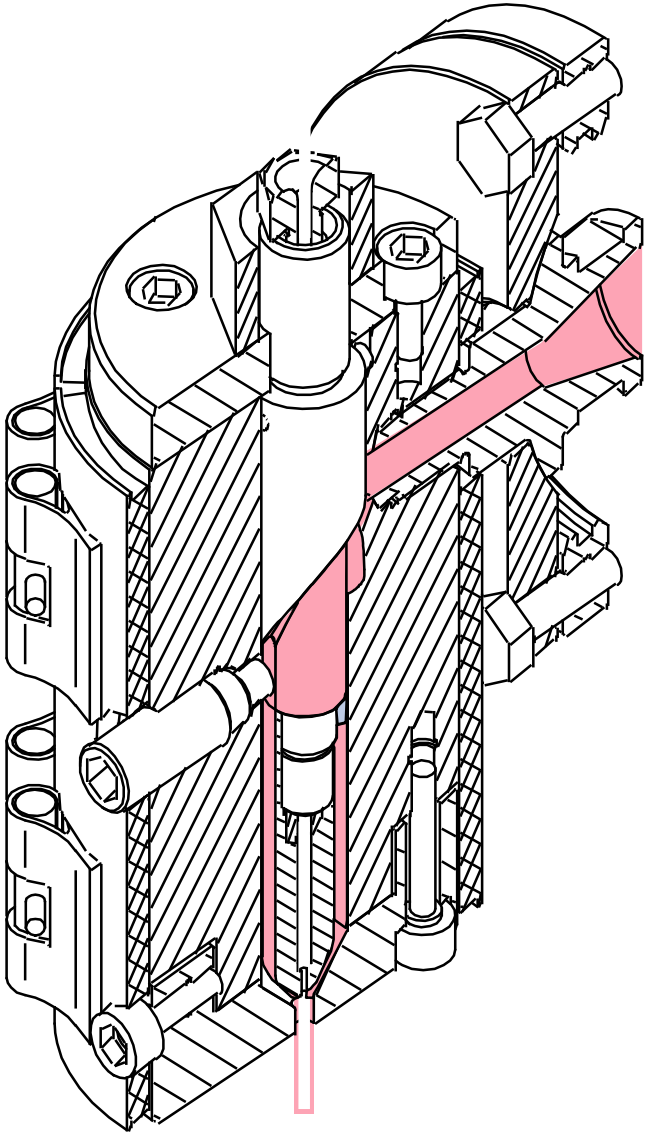
Filter Test Method Details

1) A new screen pack is placed on the breaker plate, melt flows through the finer screen first
2) "Clean" base test polymer is pumped through the screen pack with a constant volume flow (50 to 60 cm^3/min – feeding pressure of the melt pump 30 to 60 bar (400 to 900 psi)
3) Melt temperature and pressure upstream of Melt Pump has to be constant (Melt temperature ± 2°C)
4) Measure start pressure P_S at Filter Die inlet
5) TEST mixture put into the hopper once the hopper is almost empty of the base polymer
6) Once the hopper is empty and the screw is visible, purge with 100 g of base polymer
7) The screen package and breaker plate is removed under operating temperature and collected material can be assayed for content, contamination, etc.
8) Purge the system with base test polymer.

Filter Pressure Value – automated software

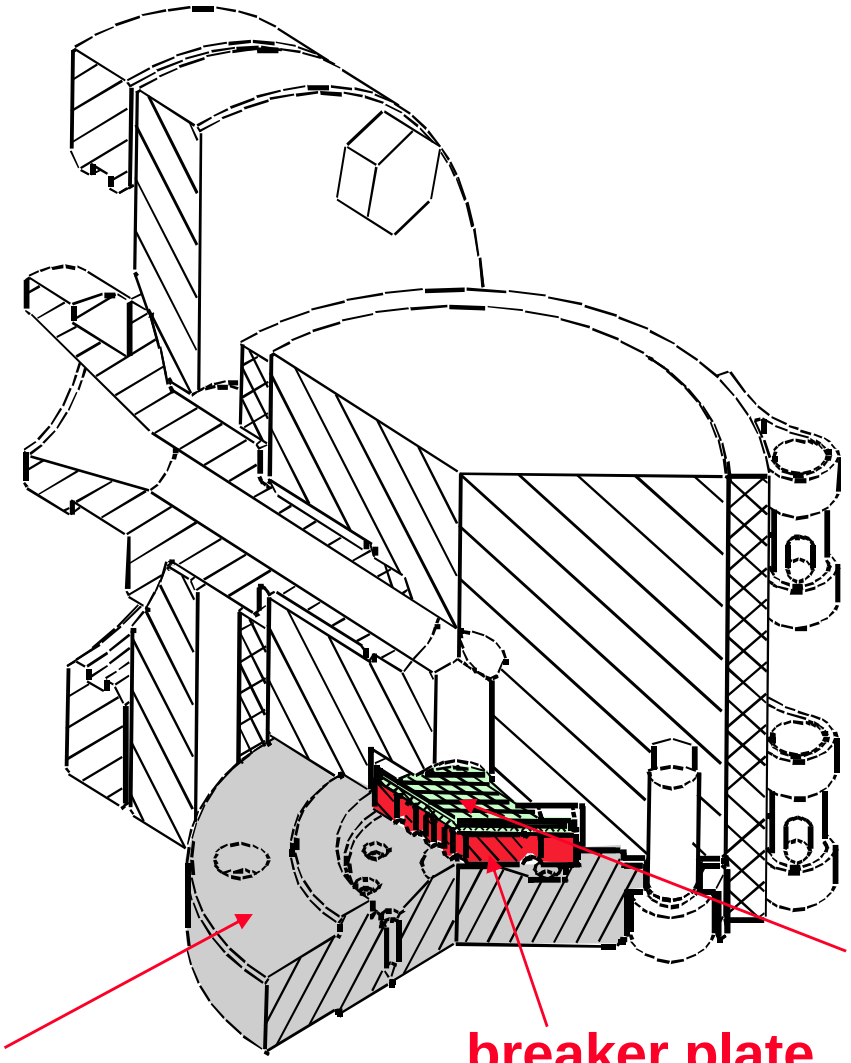


Wire Coating Die



- di: 0.7 - 2.0 mm
- da: 0.8 - 4.0 mm
- Standard: 1.2 / 2.0 mm
- Max. temperature: 480°C
- Measuring ports 2

Spinning Die



Application:

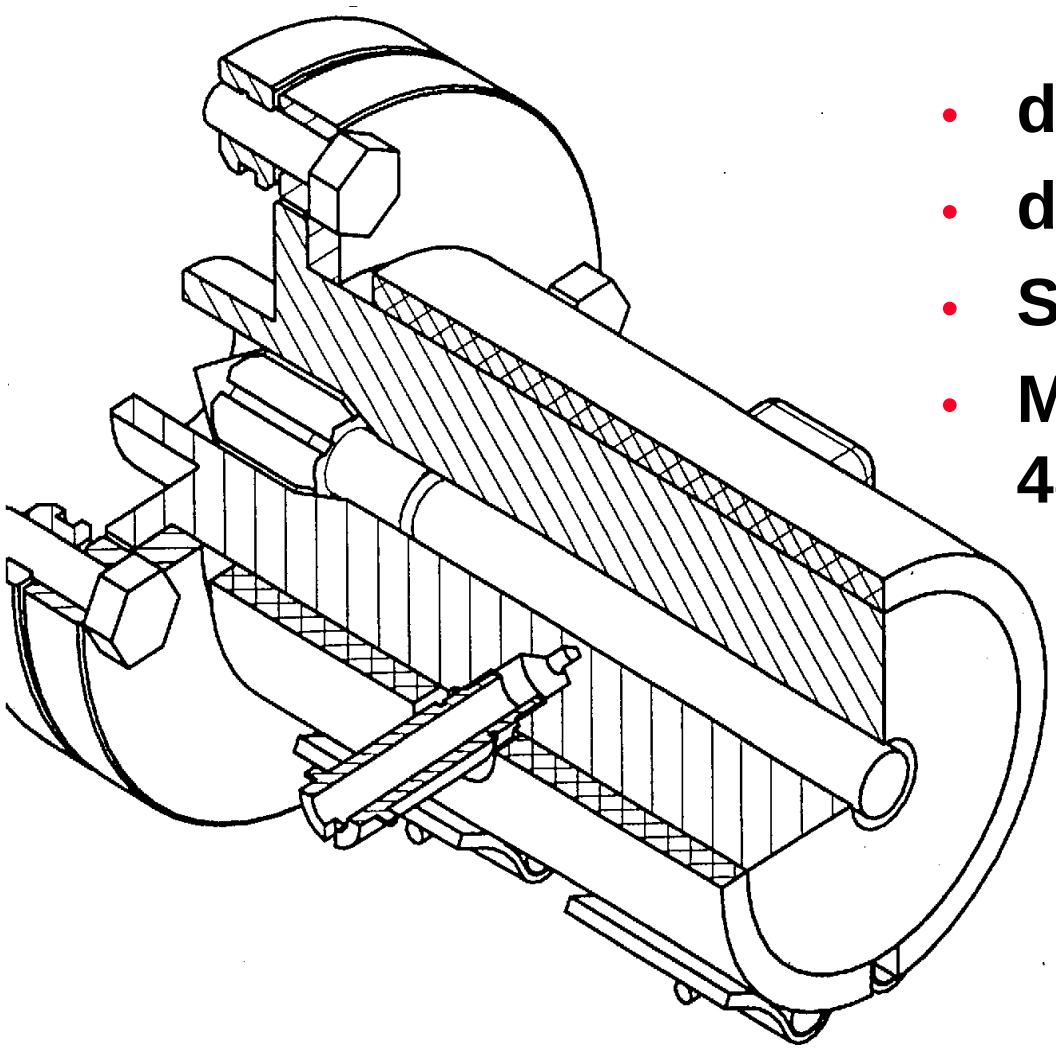
Fiber spinning

Technical data of spinning plate:

number of holes:	**10**
diameter:	**200 µm**
length:	**0.5 mm**
entrance angle:	**60°**

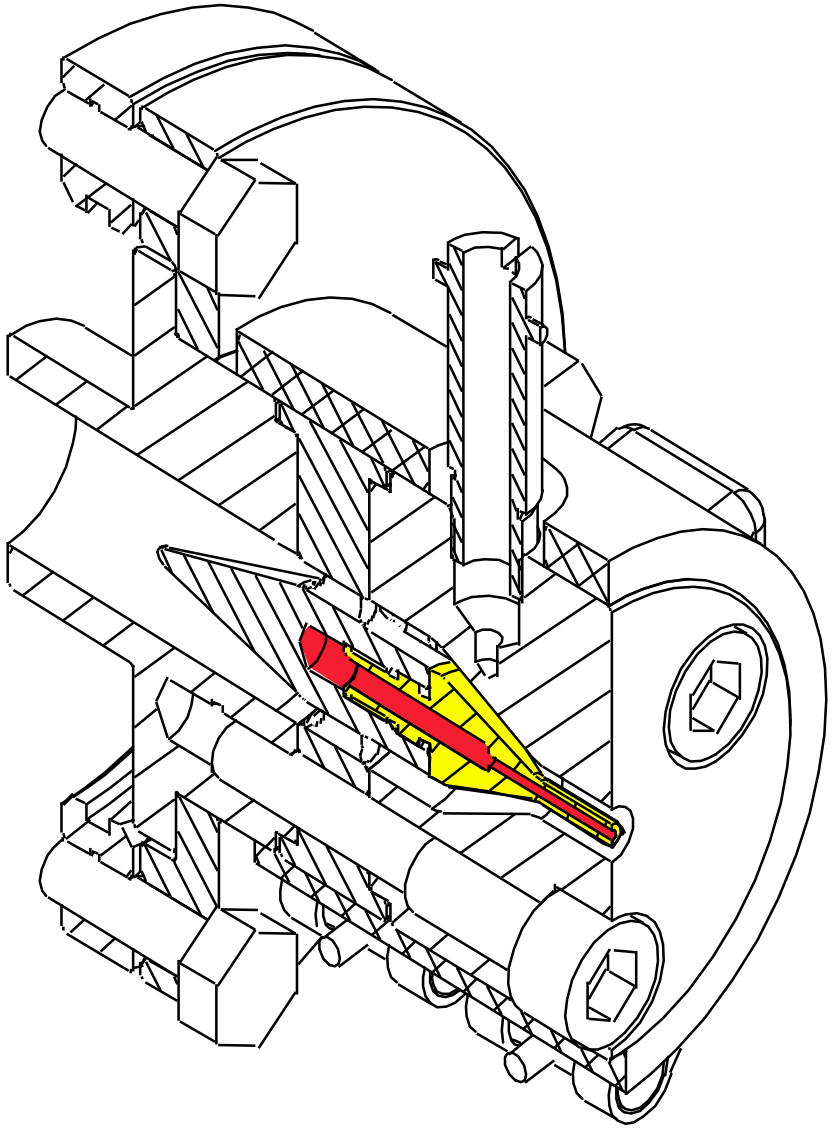
Blank Spinning plates available for customer made spinnerettes

Pipe & Tubing Die



- di: 5 - 12 mm
- da: 10 - 15 mm
- Standard: 9 / 12 mm
- Max. temperature: 480°C

Medical Tubing / Catheter Die



- di: 1.5 - 7.0 mm
- da: 2.0 - 8.0 mm
- Standard: 2.4/4.5 mm
- Max. temperature: 480°C

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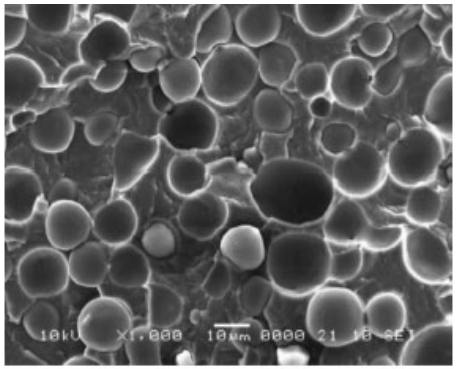
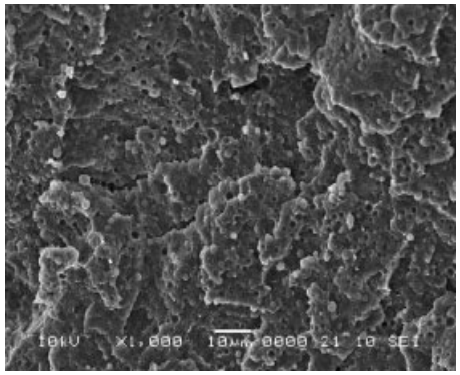
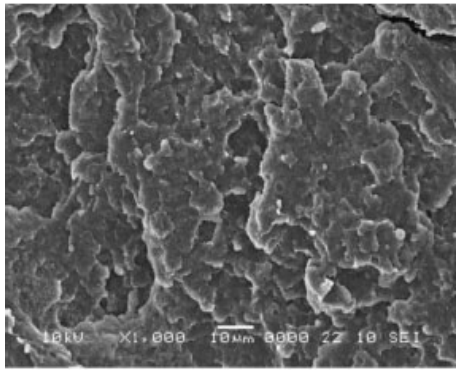
Additional Application Examples

Polymer alloying to improve mechanical properties (PTW16/25) 1/2

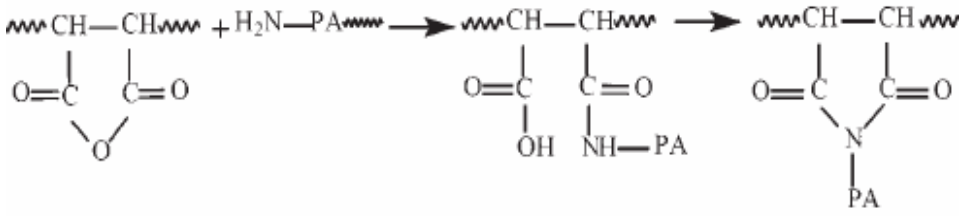






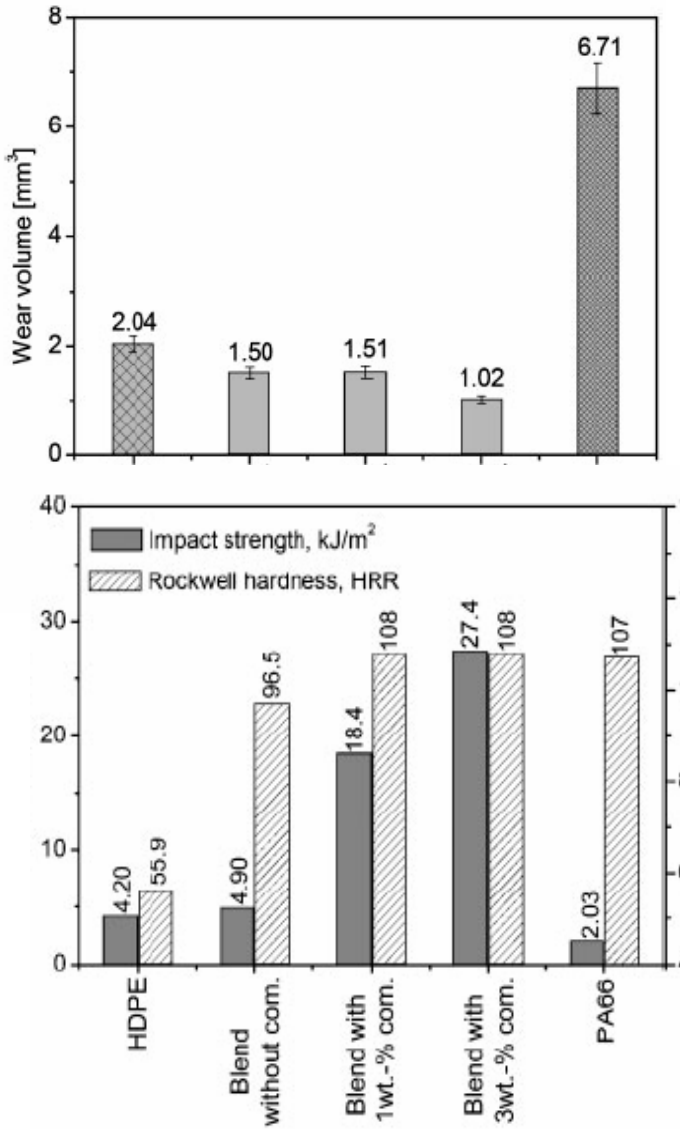
The HAAKE PTW16 is used to blend Polymers. During the extrusion the compatibilizer reacts. Result is a polymer with better triobological properties and higher impact strength.

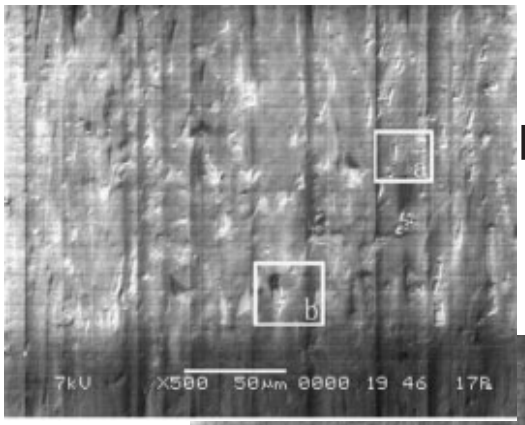
"PA66 has no thermodynamic miscibility with HDPE because of the different internal structure. The acid anhydride groups in HDPE-g-MAH, which was used as reactive compatibilizer, are expected to react readily with the amine end group(s) of the polyamide as shown below."



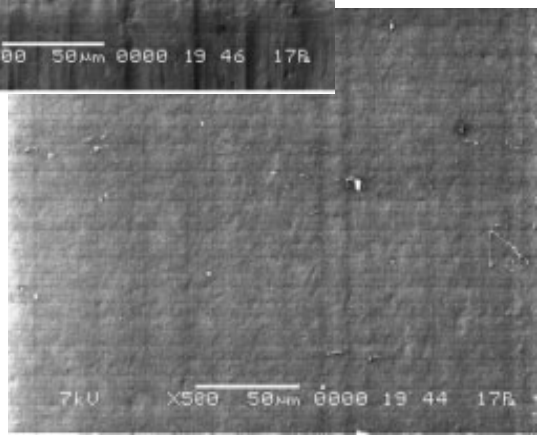
Lit: Macromol. Mater. Eng. 2004, 289, 662–671

Polymer alloying to improve mechanical properties (PTW16/25) 2/2





PA66/HDPE



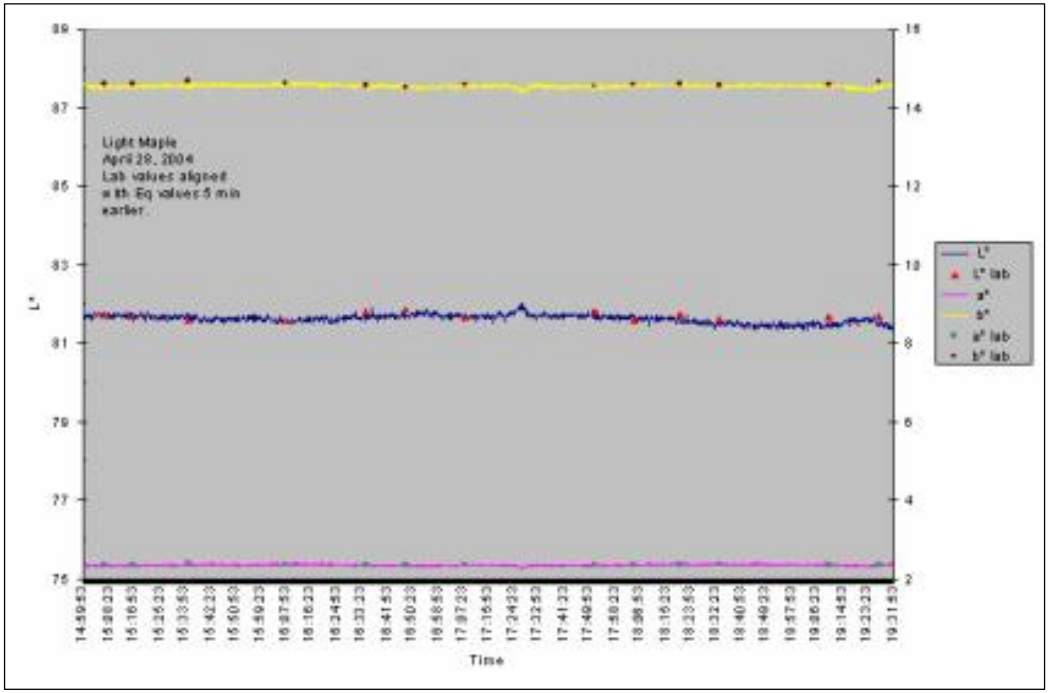
PA66/HDPE/HDPE-g-MAH

The Effect of Phase Structure on the Tribological Properties of PA66/HDPE Blends

Zhao-Bin Chen, Tong-Sheng Li, Yu-Liang Yang, Yu Zhang, Shi-Quan Lai
Department of Macromolecular Science, Key Laboratory of Molecular Engineering of Polymers, Ministry of Education, Fudan University, Shanghai, Plastics Division, Sinopec Shanghai Petrochemical Company Limited,
Macromol. Mater. Eng. 2004, 289, 662–671

Analytical test methods - Color

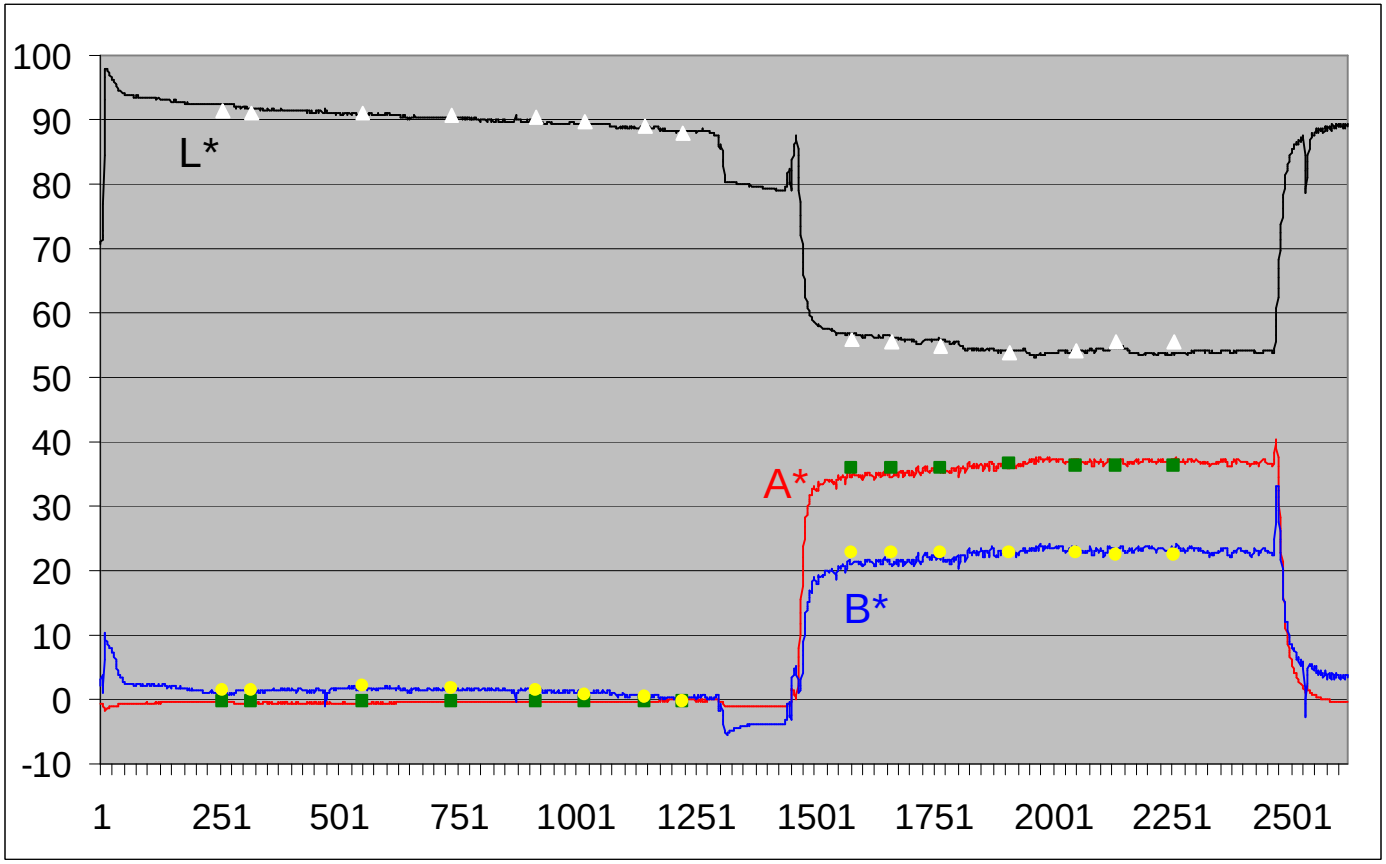
Many would like to measure color directly in the melt and control it without running sample through the laboratory. First trials indicate that this is possible by using a fiber optical probe in the melt which is connected to an UV/VIS spectrophotometer. An intelligent software allows the correlation with the laboratory color data. (Equitech Inc, USA)



Analytical test methods - Color

Offline Measurements with a GM7000A on Color Chips Manufactured from pellets at the same time reading

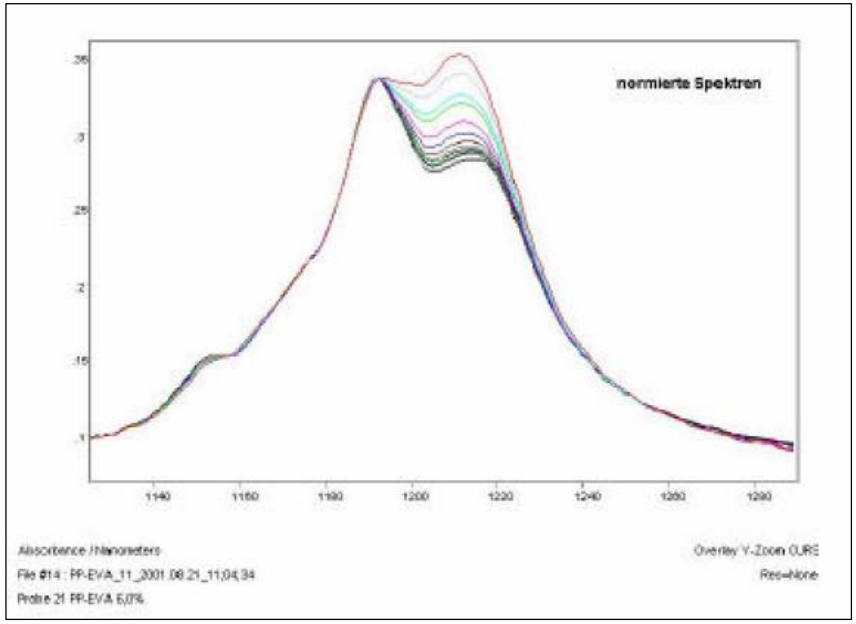
White Run
Red Run



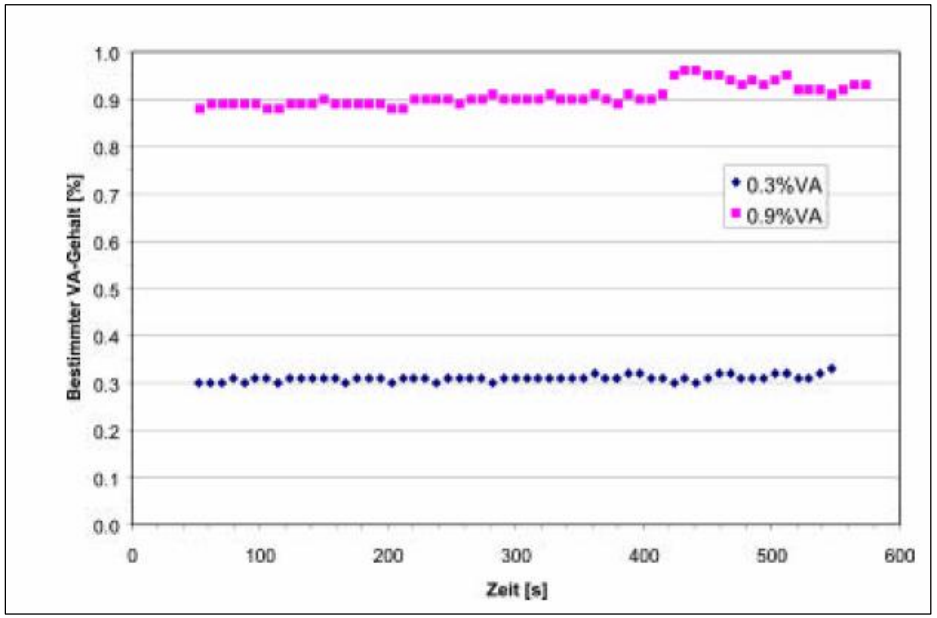
Analytical test methods - Concentration

SentroProc NIR - Quantitative In-line measurement of vinylacetats (VA) in polymer blend polypropylen (PP)/ethylenvinylacetat-copolymer (EVA) in the range of 0 to 10 % VA during extrusion. (Ref.4)

Calibration was done using the software GRAMS32 and the chemometry-application PLSplus/IQ from Galactic Ind. Corp

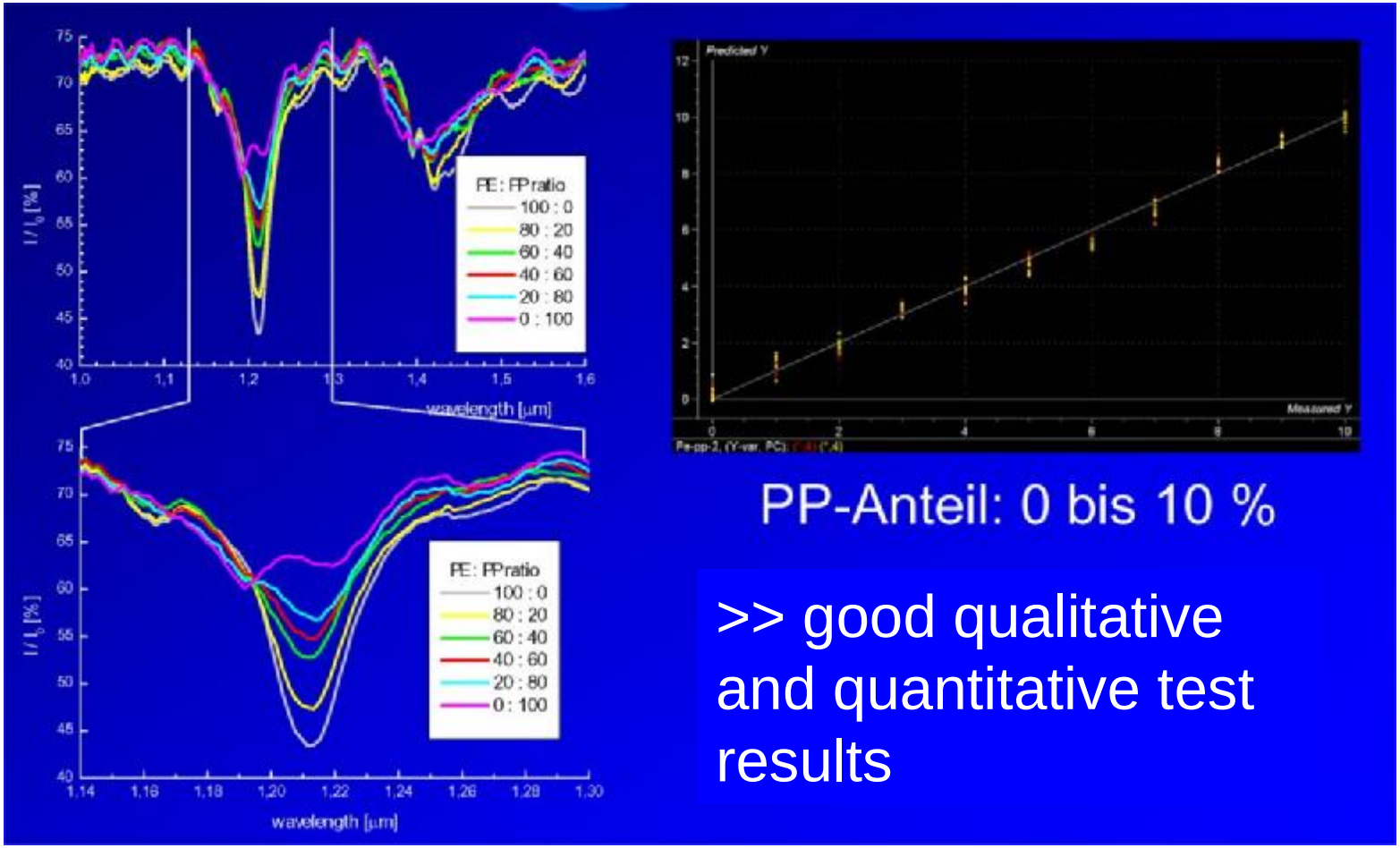


The NIR system shows a very high stability over a period of 3 days.



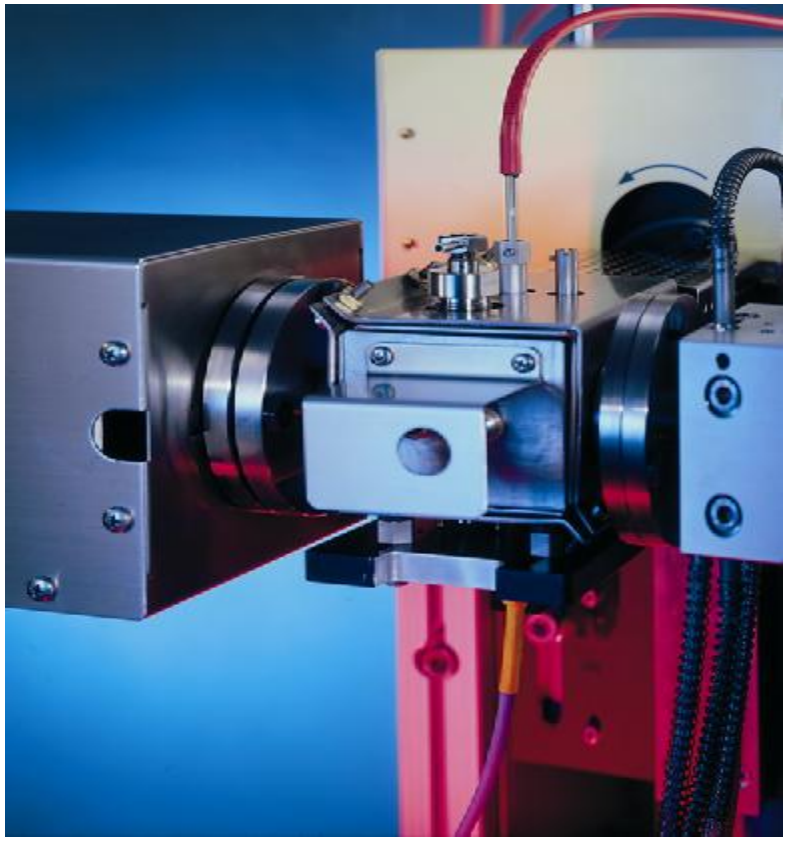
Analytical test methods - Concentration

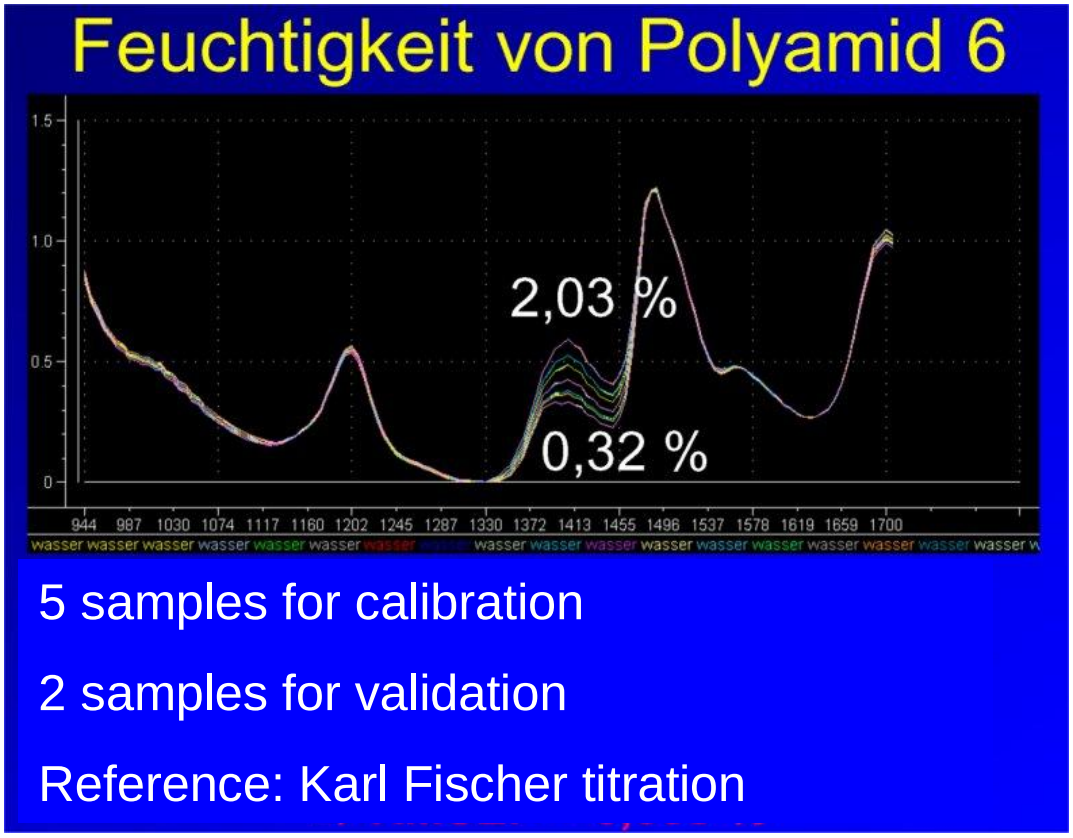
The ratio of PE-PP can be seen in the NIR spectra and with the help of an absolute calibration quantitative measures made. (Ref. 5)



Analytical test methods – Moisture Content

The moisture content in Polyamid 6 can be measured with NIR with the help of calibration; in the melt and on-line. (Ref. 5)





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