

## 1st European Sales Meeting for Thermo Temperature Control

The meeting was held in Besenfeld in the Black Forest from the 12th to the 14th of June. These three days provided an excellent opportunity for colleagues from the Thermo Temperature Control locations in Great Britain, France, Benelux and Germany to get to know each other better and intensively discuss future joint strategies to further extend their support and customer focus.



European Thermo Temperature Control Sales Team

## June – the month for Training at Thermo Haake

During June, Thermo Haake ran a series of training programs for their worldwide distributors across service and application areas.

The training commenced with a full week of service training for Laboratory Instruments, Processing Instruments and Temperature Control. Following this, there was a week of application training for Processing Instruments and then Laboratory Instruments.

Over 13 countries were represented at these training sessions over a two week timeframe.

It is great to know that both the companies representing the Thermo Haake product range and Thermo Haake themselves place so much value and importance on service and application knowledge.

We trust that all our customers continue to benefit from the commitment and investment our representatives continue to make to training across service and applications areas.



Service Training Participants

## Australia to Host International Exhibitions & Conferences

Over the coming months there will be two major international meetings in Melbourne, Australia - one in October 2002 and the other in July 2003.

This forward advice will hopefully allow you to plan your visit to Melbourne and the exhibition and conferences.

### EXPOPLAS 2002

15 – 18 October 2002, Melbourne, Australia

This exhibition was previously known as Ausplas and is said to be the largest trade exhibition of the Plastics Industry in the Southern Hemisphere. The last exhibition was in 1999 and over 12,000 visitors attended.

EXPOPLAS is the international trade exhibition for the plastics processing industry in Australasia Pacific and the Oceania region.

The Australian distributor of the Thermo Haake & Thermo PRISM product range, Rheology Solutions will be exhibiting along with 150 or so other exhibitors from around the globe. The exhibition will offer a series of industry endorsed workshops, seminars and technical conferences.

If you do attend, make sure you drop by the Rheology Solutions stand to catch up on the latest products and information available from Thermo Haake & Thermo PRISM.

EXPOPLAS is held every three years so don't miss the opportunity to visit this major plastics processing industry trade exhibition.

### PPS – 19 POLYMER PROCESSING SOCIETY – Nineteenth Annual Meeting

17 – 10 July, 2003, Melbourne, Australia

The first circular covering this annual meeting has been distributed. There is a call for papers to scientists and engineers working in Polymer Processing inviting them to submit papers on the selected symposia topics.

Abstracts of 300 words should be submitted electronically by October 15, 2002. Authors will be notified by December 15, 2002. The deadline for the final accepted papers is March 1, 2003. The conference will also host an exhibition of scientific instruments and processing equipment.

*Further information on this exhibition is available by visiting their website on [www.pps19.nww.com.au](http://www.pps19.nww.com.au)*

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## RheoFuture 2002

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## Looking for Application Information?

Thermo Haake provides application support worldwide. If you would like to discuss your particular application and find out where Thermo Haake can offer technical product support contact your local distributor or Email [newsletter@thermohaake.com](mailto:newsletter@thermohaake.com)

## More Info?

Send an Email to [newsletter@thermohaake.com](mailto:newsletter@thermohaake.com) and quote the Item No for the section of interest.

## Analytica 2002 – Trade Show Review

The Analytica 2002 held in Munich, Germany provided Thermo with the first opportunity to demonstrate its new united "One Thermo" presence at a trade show outside of the United States. Fourteen Thermo companies participated on 2 booths (one in the Laboratory Equipment Hall and one in the Analytical Instrumentation Hall) covering a total floor space area of 550 sqm. Both customers and Thermo businesses alike profited from the concentration of a wide range of products and solutions designed for the analytical instrument market on two dedicated Thermo booths.



The Lab Instruments Booth

The outcome of Analytica 2002 in general, which closed its gates on April 26, 2002, was outstanding: 34,000 visitors attended the four-day fair (compared to 28,300 at Analytica 2000). According to Dr. Joachim Ensslin, Managing Director of Munich International Trade Fairs, "Given the difficult state of the economy and the drop in attendance at comparable industry events, these are outstanding results." With 1,100 exhibitors and other participating companies, 38 percent of them from abroad, Analytica 2002 set new records in the number of visitors and exhibitors alike.

The increase in the number of visitors was due to a significant increase in Analytica's influence both abroad and in northern and eastern portions of Germany. 26 percent, or more than one-fourth of all visitors were from countries other than Germany (104 nations).

The share of international trade representatives at the last Analytica was 15 percent. That corresponds to a 108-percent increase in the percentage of foreign visitors among all trade visitors – or twice as many visitors from abroad.



The Analytical Instrumentation Booth

The share of trade visitors from northern, western and eastern Germany increased from 15 percent in 2000 to 20 percent in 2002. The share of visitors from southern Germany decreased accordingly from 70 percent in 2000 to 53 percent in 2002.

## News about RheoFuture, and the "Young Scientists Award"

### Conference.

Thermo Haake is in the process of inviting specialist speakers for the RheoFuture conference from both academic and industrial fields. The latest addition to the program is Mr. Norbert Willenbacher from BASF, who will be giving a plenary lecture about combined methods. The event will be held in the auspicious surroundings of the University of Karlsruhe, which is also acting as the preferred location partner for RheoFuture 2002.

### Supporting companies

Since June Thermo Haake has invited several international companies to make their contribution. The idea is to bring companies and young scientists together for discussing international career opportunities.

### Young Scientists Award.

With six weeks to go until the new extended paper deadline of September 20, papers have been already been submitted covering a wide range of topics from food rheology to polymer processing. Participation has been truly worldwide with entries from Korea, Australia, Germany, Japan, France, Malaysia and Hungary to name just a few.

### Press

Press releases have been sent out to a wide range of leading magazines and specialist journals already resulting in extensive press coverage for the Young Scientists Award and RheoFuture Conference. The official press sponsorship has been taken over by "Applied Rheology", the leading European journal for rheology.

[More Info?](#)

[Visit www.rheofuture.de](http://www.rheofuture.de) > [Young Scientists Award 2002](#)

## Free Sample Testing

Do you know what is going on in your product?

Thermo Haake offers to measure two of your samples with the RheoScope for free.

The RheoScope 1 is a high-end rheometer with a built-in microscope and video camera.



RheoScope 1

The possibilities that result from this unique combination open new horizons for research and practical applications. Images of shear-induced structural changes can be recorded and correlated with the rheological properties of the sample under shear.

[Interested? To arrange to have your samples tested free register on-line \[www.thermoaake.com\]\(http://www.thermoaake.com\) and follow the prompts to RheoScope 1 and complete the Free Sample Testing Form.](#)



## Temperature Control Update & News

### Facts behind the Phoenix-Line: Energy Management of Cooling for Phoenix-Circulators

Wasting energy when using refrigerated circulators and cryostats is no longer in line with the times. In the past, the desired temperature was often reached by allowing controlled heating to work against permanent cooling. This resulted in a high degree of temperature accuracy as the cooling characteristics rarely changed during the working process. The system then works in the optimal range only for low cooling capacities.

This procedure however becomes questionable when it is applied to units with a high cooling capacity or when using the units without a considerable supply of heat from external objects. The latter case can be often observed in practical applications. This results in an expensive and unnecessary waste of energy.

### The Challenge

The control technologies available only allow the user the decision between operating at full cooling capacity or completely without it.

A cooling capacity control system for the whole application range should fulfill the following requirements:

- Automatic adaptation of the cooling capacity to the requirements of the respective application
- The attainment of optimum temperature accuracy at every working temperature, despite the higher installed cooling capacity
- The greatest possible saving of energy and therefore of regular operating costs
- Optimum control safety and robustness even for continuous operation

### The Solution

The cooling energy management system developed by Thermo Haake is based on the FuzzyStar® control with neural adaptation. This control system does not only recognize the heating capacity as a correcting variable

### Editors Note:

There is no guarantee that comments or feedback received will be published. However, we will answer all communications direct.

but is also capable of varying the cooling capacity according to the preset behavioral rules.

This necessitates the ability to feed the refrigerant supply in pulses to the expansion valve via a solenoid valve or to be able to cut it off completely. A bypass capillary allows some of the refrigerant to pass by the solenoid valve in any case in order to guarantee a basic cooling capacity level. This basic capacity level is determined according to the unit used at approx. 30% of maximum. Maximum power is available when the solenoid valve is continuously open. The FuzzyStar® control system ensures a stepped capacity adaptation between 30% and 100% proportional to the energy requirement at the respective working temperature.

### The Technology

The refrigerated circulators and cryostats of the Phoenix-Line which are designed to cover an entire temperature range from -90°C to +200°C are equipped with the cooling energy management system. Once the units are switched on, they attempt to reach the desired set temperature as quick as possible starting from the ambient temperature.

- For temperature increases >80°C, i.e. set temperatures above 100°C, without the cooler being switched on
- For temperature increases <80°C, i.e. set temperatures below 100°C, using the basic cooling capacity
- Full cooling capacity is used in any case for a drop in temperature



Once the desired set temperature has been reached, the energy management system checks whether the basic cooling capacity is sufficient to maintain the temperature with a slight cooling capacity excess. If this is not possible, the energy management system selects the optimum pulse ratio in steps according to the behavioral rules of fuzzy logic.

The user normally does not notice this as all processes are carried out automatically.

For reasons of safety, only the basic cooling capacity is available at temperatures >70°C.

The control status of the unit is visible for the user. Maximum cooling is represented by a large star and the basic cooling by a small star on the large LCD display. The star flashes intermittently large and small at every intermediate capacity level. The user is therefore kept continually informed on the level of the energy requirement.



LCD display screen

The extremely robust fuzzy logic controller with neural adaptation is designed to ensure a very stable control process and to guarantee the respective best possible temperature accuracy for the intended application.

*More Info? Send an Email to [newsletter@thermohaake.com](mailto:newsletter@thermohaake.com) and Quote No: 98 for Phoenix-Line Quote No: 99 for a copy of the General Temperature Control Catalogue Quote No: 112 for a reprint of this article*

### Customer Training

Thermo Haake offer a number of training programs throughout the year to cover application areas within Laboratory Products and Processing Instruments.

Interested? Further information on future customer training programs is available. Send an email to [newsletter@thermohaake.com](mailto:newsletter@thermohaake.com) and request details for the specific area you are interested in.

### The RheoStress 600 Normal Force Option – Rheometry not only in one direction!

#### Introduction

The measurement of normal forces plays an important role in advanced rheometry, especially in the analysis of visco-elastic materials. Normal force is relevant for the following applications; measuring the non-linear viscoelastic properties of materials, controlled sample loading, gap-correction for shrinking or expanding samples using AutoTension and squeeze flow. All of the above is possible with the RheoStress 600 and the latest version of RheoWin.

#### Normal force sensor

Thermo Haake engineers have developed a truly unique optical normal force sensor for the RheoStress 600, which is directly integrated in the air-bearing of the rheometer drive motor. This sensor, which consists of a light emitter and receiver, detects the axial displacement of the motor shaft by measuring the variation of the light intensity. Due to the high axial stiffness of the air-bearing it is necessary to detect axial displacements in the order of nanometers! Together with the new MFC (Micro Force Correction) this very high displacement resolution makes it possible to extend the measurable force range down to 0.01 N.

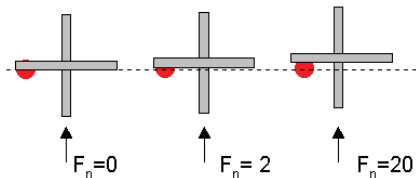


Fig. 1: Principle of operation of optical  $F_n$  sensor

Because the normal force sensor is an integral part of the drive motor, the normal force signal can be used with all measuring geometries and temperature control units.

The normal force signal is also available in all measurement modes (CR, CR and CD) with a maximal acquisition rate of 400 data points per second.

#### Customer Profiles

Users of any Thermo Haake or Thermo PRISM products are invited to submit a story on their specific application and or use of the Thermo Haake or Thermo PRISM product.

Interested? Send an email to [newsletter@thermohaake.com](mailto:newsletter@thermohaake.com) with a brief overview.

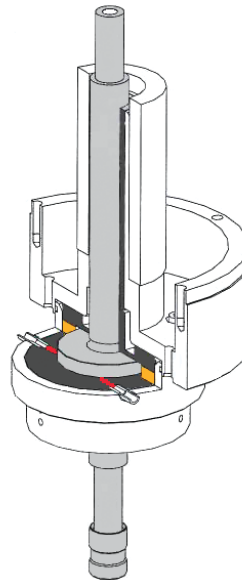


Fig. 2:  $F_n$  sensor integrated in air bearing

#### Applications

##### Non-linear visco-elasticity

The first normal stress difference  $N_1$ , which is calculated from the measured normal force, is a measure of the non-linear visco-elastic properties of a material, this in contrast to the elastic moduli  $G'$  and  $G''$  which are measures of the linear visco-elastic properties. Therefore  $N_1$  is an important additional parameter for the complete characterization of the rheological properties of materials.

As a matter of principle all visco-elastic materials show a non-zero first normal stress difference under shear flow. A well known example is the die swell effect during extrusion and injection moulding of polymers melts which can be attributed to normal forces. In coating processes, excessive normal forces at high shear rates cause phenomena like roller splatter.

The graph shows measurement results of the RheoStress 600 on the visco-elastic standard reference material 2490 from the NIST. The curves with the open blue and red rectangles show the RheoStress 600 results for  $N_1$  as a function of the shear rate  $\dot{\gamma}$ , the curve with the black closed rectangles shows the NIST reference data. Measured data and reference data compare very well.

The two dotted grey curves show the measured normal force values, which start well under 0.01 N.

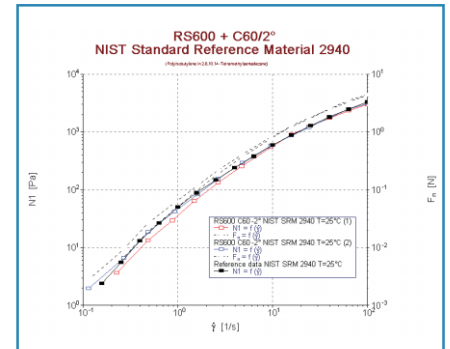


Fig. 3: Comparison of RS600 measurement results with reference data

##### Normal force controlled gap loading

Normal force measurement is also a very useful tool for loading sensitive and difficult samples. In order to enhance the reproducibility of the measurement results of such samples, it is often helpful to close the gap of the measurement geometry while applying a constant normal force instead of a constant speed. On the RheoStress 600 this is easily realized by selecting the corresponding option in the RheoWin Lift-Element.

##### AutoTension

The new AutoTension functionality increases the quality of oscillation experiments of materials that shrink or expand as a function of time or temperature (curing experiment) by keeping the sample under a constant normal force during the experiment. This is done automatically by monitoring the normal force and adjusting the measuring gap in such a way that the applied normal force stays constant.

##### Squeeze flow

With the new axial ramp element in RheoWin the RheoStress 600 can also perform squeeze flow measurements. During such an experiment the gap of a parallel plate geometry is decreased linearly or logarithmically as a function of time, thereby squeezing the sample. During this bi-axial (semi) extensional flow the measured normal force gives information on the extensional properties of the material.

*More Info? Send an Email to [newsletter@thermohaake.com](mailto:newsletter@thermohaake.com) and Quote No: 100 for reprint of this article. Quote No: 101 for RheoStress 600 product brochure*

#### Reader Comments

Contributions to this newsletter are welcomed. Please send an Email to [newsletter@thermohaake.com](mailto:newsletter@thermohaake.com)

### NEW PRODUCT

The CaBER 1 ...  
for quantifying the extensional  
properties of fluids.

The Capillary Breakup Extensional  
Rheometer (CaBER) is the only  
commercially available rheometer for  
measuring extensional properties.



**It is suitable  
for examining:**

- Adhesives
  - Measurement of tackiness
  - Solvent loss or gain
- Associating polymers
- Biomaterials
- Surfactants
- Consumer good
  - Filling of bottles
  - Time to breakup
  - Solvent loss
  - Processability

The CaBER 1 was developed by the Cambridge Polymer Group and built by Thermo Haake

- Food products
  - Stringiness/strand formation
  - Time to breakup
  - Relaxation of doughs
  - Elastic instabilities
- Industrial resins
  - Relaxation time spectrum
  - Constitutive modeling
  - Spinnability

**The rheometer features:**

- Computer control
- Class 1 laser micrometer
- Easy operation with sample loading and cleaning easy
- Linear motor drive with variable speed
- Closed temperature-controlled sample chamber
- Automatic repeated testing
- Exchangeable geometries
- User defined strains
- Sample volume <1ml

*More Info? Send an Email to [newsletter@thermohaake.com](mailto:newsletter@thermohaake.com) and Quote No: 102 for the CaBER 1 product brochure*

### Application Articles & Technical Notes

#### Rheological Behavior During Phase Separation Induced by UV Curing

Su-Yong Nam<sup>1</sup>, Mikihiro Saki<sup>2</sup>  
and Yasufumi Otsubo<sup>3</sup>

<sup>1</sup> - Division of Image & Information, College of Engineering, Pukyong National University, Korea

<sup>2</sup> - EKO Instruments Trading Co, Japan

<sup>3</sup> - Department of Urban Environment Systems, Chiba University, Japan

The rheological changes during UV curing are examined for solutions of UV-reactive acrylic prepolymer and alkyd resin. To measure the dynamic viscoelasticity in the entire process of UV curing by a single run, a rheological program is developed, by which the storage modulus can be detected in the range of 100 – 108 Pa under linear conditions. The solutions containing alkyd resin at concentration of 10 – 30 wt% show phase separation in the course of curing. The storage modulus of such systems rapidly

increases at first, goes through a maximum and then decreasingly approaches the equilibrium value. The microscopic observation of cured films clearly indicates that the droplets generated by phase separation can grow and migrate in the three-dimensional network. The growth of droplets accompanies the rupture of continuous network in a narrow range. The delicate balance between the local rupture of network by phase separation and formation of cross-links by polymerization controls the appearance of peak in the time-dependent cure of the storage modulus. Reference V199-E

*More Info? Send an Email to [newsletter@thermohaake.com](mailto:newsletter@thermohaake.com) and Quote No: 103 for a copy of the article*

### Technical Notes – Mineral Slurries Series

T Kealy, Rheology Solutions Pty Ltd, Australia

Mineral processing involves the transportation and processing of large quantities of materials. Combining solid materials with a carrier fluid (usually water) to create a slurry has long been popular in the industry, principally due to the increased efficiency and alacrity of mineral processing steps through use of a slurry, since the finely ground particles afford greater surface area exposure to the extracting medium. Also, the ease with which the particulate matter can be processed in this form is important for unit processes including grinding, classification, separation processes (settling, thickening, screening, filtration) and transport processes like flow through pipes or launders, mixing, etc.

Slurry characteristics pose many engineering challenges. These characteristics include particle settling, pH, bulk/carrier fluid viscosity, particulate flocculation or dispersion, attrition, pipe/fitting/impeller wear, degradation of flocculated or friable solids and the pumpability of the slurry. The effects of these challenges can be further altered by process or environmental variables such as temperature or solids loading.

Wet comminution and classification can be effected by viscosity, particle size distribution, fines concentration etc. For example, hydrocyclone classification is effected by the presence of a yield stress (the minimum force required for a slurry to flow). Often, cut point increases and efficiency decreases with increasing yield stress. Separation processes are influenced by inter-particle attraction and viscosity (how easily the fluid flows once it is in motion). These variables may be manipulated through the addition of modifying agents to the slurry. The key effects of

these additions should be monitored through changes in the rheological parameters of the slurry, which are directly related to process performance. In-transit particle settling causes uneven and excessive wear or complete blockage in the transport system. Particle attrition may be a QC matter both for disposal and in-process and, changes in particle size distribution may affect the ability of the particles to remain suspended. Problems with the pumpability of slurries include the high cost of initiating and continuing pumping of fluids with high yield stresses or viscosities.

Rheological techniques can help assess process design and manipulation. A range of instruments is available to measure the rheological effects, through knowledge of the characteristic flow properties of slurries. These rheometers allow one to directly measure and assess the causes and effects listed above. Physical quantities such as yield stress; plastic viscosity and hysteresis (the changes in plastic viscosity and/or yield stress which are due, usually, to particle attrition or settling) can be quantified and predicted using appropriate instruments.

*More Info? Send an Email to [newsletter@thermohaake.com](mailto:newsletter@thermohaake.com) and Quote No: 104 for a copy of Rheo012 Brief report on the rheological considerations for applications with brown coal Quote No: 105 for a copy of Rheo013 Slurry rheology and pipeline transport properties, and overview Quote No: 106 for a copy of Rheo014 Overview of rheology-based process challenges for the mineral processing industries*

### Influence of the sample weight on PVC fusion test

#### Introduction

In the PVC industry mixer tests are well established for the investigation of the plastification and degradation behavior of PVC Dry Blends. For this test the laboratory mixer is set at a constant temperature and the rotors are set to a constant speed. The PVC Dry Blend powder is pushed into the hot mixer chamber using a manual or pneumatic ram. The drive torque and the melt temperature is recorded and displayed against the mixing time. This resulting mixer rheogram is then evaluated and maxima and minima of the curve are calculated. Because of the sensitivity of the measurement method, it is essential to keep the testing conditions (like mixer temperature, rotor speed and sample weight), as well as the measuring procedure (loading, cleaning) as constant as possible. The mixer test can then provide very reliable and good reproducible test results.

#### Test aim

The attached tests should show the influence of the sample weight on the fusion behavior of a PVC sample.

#### Test equipment

- Torque Rheometer Rheocord300p
- Laboratory Mixer Rheomix600p
- Roller Rotors
- Evaluation software for mixer tests PolyView

#### Test conditions

- Temperature: 160°C
- Rotor speed: 40 rpm
- Test time: 4 minutes
- Sample weight: a) 60g, b) 64g, c) 66g

#### Test results

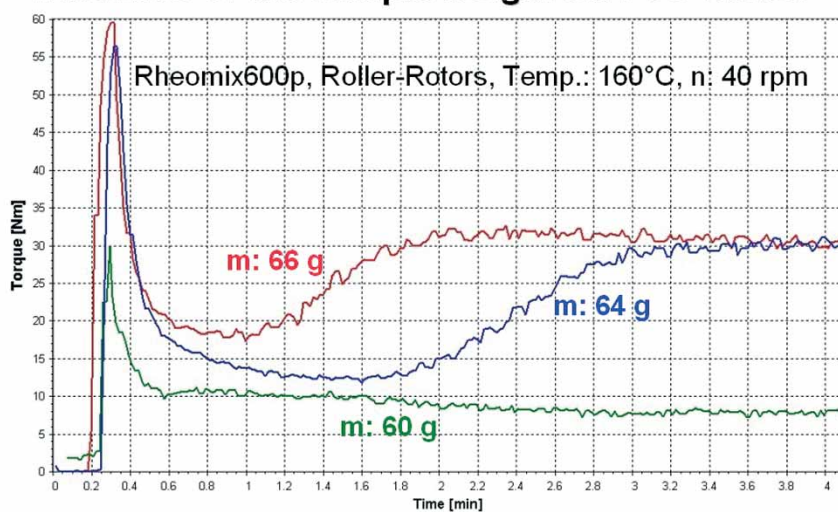
Picture 1 shows the superposed torque curves of the measurements with the three different sample weights. All curves show an increase in torque during the loading procedure. After the loading, the PVC powder distributes in the mixer chamber and the torque goes down again. After one minute the test with the highest sample weight (66g) shows an increase in torque again. This increase is caused by the plastification of the PVC Dry Blend.

The test with 64g sample weight shows this increase after 2 minutes.

The test with a sample weight of 60g shows no second increase in torque.

So the differences in sample weight have a huge effect on the plastification behavior of this PVC Dry Blend. This result can also be seen in the visual inspection of the mixed sample.

### Influence of the sample weight on PVC fusion



Picture 2 and Picture 3 show the mixed PVC sample after the mixer tests.

Picture 2 shows the test result with 60 g sample weight. The sample was still powder like and not plastified at all.



Picture 3 shows the test result with 66 g sample weight. This sample was well plastified and had a shiny appearance.



*More Info? Send an Email to [newsletter@thermo-haake.com](mailto:newsletter@thermo-haake.com) and Quote No: 107 for a reprint of this article  
Quote No: 108 for Torque Rheometer Rheocord 300p  
Quote No: 109 for Mixer – RheoMixer 600p  
Quote No: 110 for PolyView Software*

### High Pressure Capillary Rheometer ... Rheoflizer

The Rheoflizer is a high pressure capillary rheometer for the determination of the flow behaviour of polymers as a function of shear rate and temperature.

The computer controlled instrument covers a wide range of shear rates with a variety of measurement dies.

The RheoCap Software offers unique capabilities that enable the user to customise the software.

*Rheoflizer – high pressure capillary rheometer*

*More Info? Send an Email to [newsletter@thermo-haake.com](mailto:newsletter@thermo-haake.com) and Quote No: 111 for Rheoflizer brochure  
Quote No: 113 for RheoCap Software*

