Instruction Manual

HAAKE CaBER 1

"Translation of the original instruction manual"   version 1.8

Thermo
S C I E N T I F I C

Konformitätserklärung /
Declaration of Conformity

| 075-5019 |

Produktbezeichnung / Product name

HAAKE CaBER 1

Identifikation / Identification

398-0001, 398-0002

Hersteller / Manufacturer

Thermo Electron (Karlsruhe) GmbH
Dieselstraße 4
D – 76227 Karlsruhe
Germany

$C\epsilon$

Dokumentationsbevollmächtigte Person
Authrised person for technical file

Henry Eisenlohr
Thermo Electron (Karlsruhe) GmbH

Richtlinie / Directive

| 2006/42/EG | Maschinenrichtlinie / Machinery directive |

Konform zu weiteren Richtlinien / Conform to other directives

| 2004/108/EG | Richtlinie für elektromagnetische Verträglichkeit
Electromagnetic Compatibility Directive |
| 2006/95/EG | Niederspannungsrichtlinie
Low voltage directive |

Folgende harmonisierte Normen wurden angewandt:
Following harmonized standards are used:

EN ISO 12100-1:2003 + A1:2009 12100-2:2003 + A1:2009	Sicherheit von Maschinen - Grundbegriffe, allgemeine Gestaltungsleitsätze- Safety of machinery – basic concepts, general principles for design
EN 61326-1: 2006	Elektrische Mess-, Steuer-, Regel- und Laborgeräte - EMV-Anforderungen, Teil 1 Allgemeine Anforderungen Electrical equipment for measurement, control and laboratory use EMC-requirements, Part 1 general requirements
EN 61010-1: 2001	Sicherheitsbestimmungen für elektrische Mess-, Steuer-, Regel-, und Laborgeräte - allgemeine Anforderungen Safety requirements for electrical equipment for measurement, control and laboratory use - general requirements
EN 61010-2-010: 2003	Besondere Anforderungen an Laborgeräte für das Erhitzen von Stoffen Particular requirements for laboratory equipment for the heating of materials

Wir erklären in unserer ausschließlichen Verantwortung, dass das Produkt, auf das sich diese Erklärung bezieht, mit den oben genannten Normen, normativen Dokumenten und den Bestimmungen der genannten Richtlinien übereinstimmt. Die Prüfprotokolle werden bei Thermo Electron (Karlsruhe) 10 Jahre aufbewahrt.

We declare under our sole responsibility, that this product to which this declaration relates is in conformity with the a.m. standards or other normative documents and is following the provisions of the a.m. directives.
All test certificates will be kept by Thermo Electron (Karlsruhe) for 10 years.

| Unterschrift / Signature
Geschäftsleitung/Business Management | 23.03.2010
Datum/Date | Thermo Electron (Karlsruhe) GmbH
Dieselstr. 4 * 76227 Karlsruhe
Tel. + 49-721-4094-444, Fax + 49-721-4094-418
Hersteller/Manufacturer |

Table of Contents

Table of Contents

Table of Contents

3

1. Key to symbols

1.1 Symbols used in this manual

! Warns the user of possible damage to the unit, draws attention to the risk of injury or contains safety notes and warnings.

☞ Denotes an important remark.

1 Indicates the next operating step to be carried out and …

⇒ … what happens as a result thereof.

1.2 Symbols used on the unit

Caution: laser beam

Caution: danger of injury your hands

Caution: hot surfaces

Caution: general warning, risk of danger

2.   Quality Assurance

Dear customer,
Thermo Fisher Scientific implements a **Q**uality **M**anagement **S**ystem certified according to ISO 9001:2008.
This guarantees the presence of organizational structures which are necessary to ensure that our products are developed, manufactured and managed according to our customers expectations. Internal and external audits are carried out on a regular basis to ensure that our **QMS** is fully functional.
We also check our products during the manufacturing process to certify that they are produced according to the specifications as well as to monitor correct functioning and to confirm that they are safe. The results are recorded for future reference.
The "Final Test" label on the product is a sign that this unit has fulfilled all requirements at the time of final manufacturing. Please inform us if, despite our precautionary measures, you should find any product defects. You can thus help us to avoid such faults in future.

3.   Your Contacts at Thermo Fisher Scientific

Please get in contact with us or the authorized agent who supplied you with the unit if you have any further questions.

International / Germany

Thermo Fisher Scientific
Dieselstraße 4
D-76227 Karlsruhe, Germany
Tel.   +49(0)721 4094–444
Fax   +49(0)721 4094–300

support.mc.de@thermofisher.com
www.thermo.com/mc

The following specifications should be given when product enquiries are made:

Unit name printed on the front of the unit and specified on the name plate.
Typ: Order No. (e.g.: 557–3001)
Ser.:Nr.:

Thermo Electron (Karlsruhe) GmbH	Made in Germany	Dieselstraße 4 D-76227 Karlsruhe	CE
Unit name			IP20
Typ	Ser. Nr.		
V / Hz			

_   _ _ _ _ _ _ _ _ _ _ _

_   Manufacturing order no.: (1–9)

_ _   Manufacturing year (e.g. 09)

                                                     Production order no.:
_ _ _ _ _ _   (000001 –99999)

          _ _ _   Serial–number

Mains voltage in V / power input:
e.g. 115 V/ 50 Hz/ 2 A

4. Warranty

For the warranty and any potential additional warranty, the user shall have to ensure that the devices are serviced by an expert at the following intervals:

The maintenance is required after approx. 2000 operating hours, at the latest, however, twelve months after the initial operation or the last maintenance, respectively.

Two thousand operating hours are achieved:

> at an operating period of eight hours daily (five days a week)
> about once a year

> at an operating period of more than eight to sixteen hours daily
> about every six months

> at an operating period of more than sixteen hours daily
> about every three months

We recommend to have the maintenance carried out by Thermo Fisher Scientific or by staff authorised by Thermo Fisher Scientific as special knowledge and tools are required.

The maintenance and calibration work carried out has to be recorded by certificates in conformity with ISO 9000 ff.

5.   Safety notes and warnings

The rheometer corresponds to the relevant safety regulations. However you are solely responsible for the correct handling and proper usage of the instrument.

This instrument is designed for the determination of rheological behavior of fluid and semi-solid materials. These materials may not be tested if people can be hurt or devices be damaged.

! The heart of the instrument is a laser micrometer that emits light at 780 nm (invisible light) with a beam power of 1.4 mW. As a consequence this product is a "Class 1 laser product" according to the degree of hazard specified in the classification system of the FDA, standards 21 CFR 1040.10 and 1040.11.

• The use of optical instruments with this product will increase eye hazard.

• Do not stare into the beam directly in succession.

• Use of controls or adjustments or performance of procedures other than those specified byt Thermo Fisher Scientific may result in hazardous radiation exposure.

! The device may not be operated if there are any doubts regarding a safe operation due to the outer appearance (e.g. damages).

! A safe operation of the instrument cannot be guaranteed if the user does not comply with this instruction manual.

! Ensure that this instruction manual is made readily available to every operator.

! This unit should only be used for the applications it was designed for.

! Make sure that the unit has been switched off before you connect or disconnect the cables. This is to avoid electrostatic charging resulting in a defect of the electronic circuit boards.

! Do not operate the unit with wet or oily hands.

! Do not immerse the unit in water or expose it to spray water.

! Do not clean the unit using solvents (fire danger!) – a damp cloth applied with a household cleaning substance is often sufficient.

Safety notes and warnings

! **Repairs, alterations or any work involving opening up the unit should only be carried out by specialized personnel. Considerable damage can be caused by incorrect repair work. The Thermo Fisher Scientific service department is at your disposal for any repairs you may require.**

! **Have the unit serviced by specialists at regular intervals.**

! **We do not know which substances you intend to test using this unit. Many substances are**…

- inflammable, easily ignited, explosive

- hazardous to health

- environmentally unsafe

i.e.: **dangerous**

You alone are responsible for your handling of these substances!

Our advice:

- If in doubt, consult a safety specialist

- Read the product manufacturer's or supplier's "EU SAFETY DATA SHEET"

- Read the REGULATIONS CONCERNING DANGEROUS MATERIALS

- Observe the "Guidelines for Laboratories"

6. Unit description

6.1 Introduction

The HAAKE CaBER 1 (**Ca**pillary **B**reakup **E**xtensional **R**heometer) is a compact and affordable desktop extensional rheometer. Currently there are few other commercially available methods for obtaining data on the extensional behavior of complex fluids (e.g. colloids, adhesives, paints, foods, consumer products, melts).
This is despite increasing academic and industrial interest in measuring the extensional viscosity of a material. Existing instrument designs are bulky, complex and expensive. Two examples of commercial extensional rheometers are the RFX^{TM} opposed jet rheometer (no longer manufactured) and the RME^{TM} melts rheometer (Rheometrics Scientific). These devices are targeted at very specific ranges of fluid viscosities as depicted in Figure 1.

The HAAKE CaBER 1 addresses the wide spectrum of fluid viscosities not accessible with existing instrumentation. In fact, the HAAKE CaBER 1 operational range overlaps with the ranges accessible to both these instruments. No other instrument in use today can provide unambiguous information on the response of fluids and melts to an extensional flow field.
This type of flow is of primary interest to industry because almost all processing conditions in manufacturing involve a component of extensional flow. For instance, pumping, fiber spinning, extrusion, molding and filling processes all involve stretching kinematics. However, the behavior of all but the simplest materials in such a flow is markedly different from that predicted from knowledge of the shear rheology. Consequently, for process improvement, manufacturing control and the development and design of materials, knowledge of the extensional properties is critical. The HAAKE CaBER 1 will be useful over a broad range of materials, covering such fluids as paints and inks through to foods, shampoos, gels and pastes (see Fig. 1).
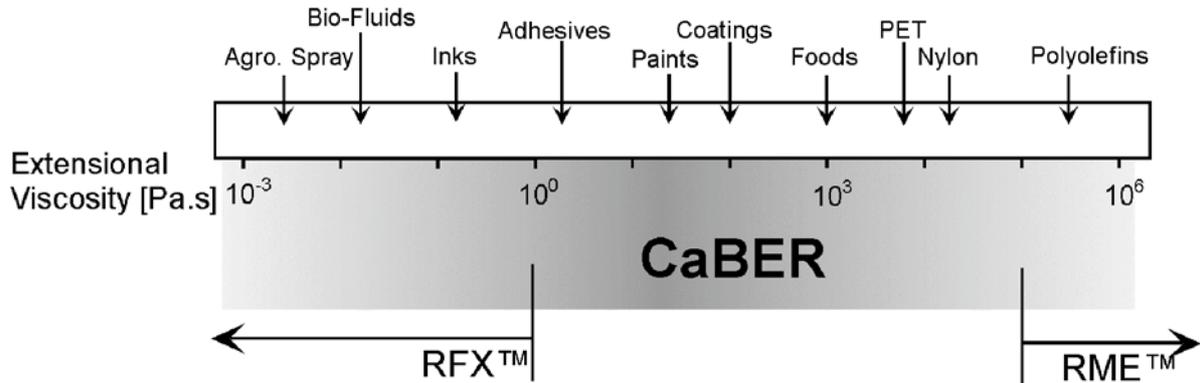
Fig. 1: Schematic of the range of applicability of the HAAKE Ca-BER 1 RFX^{TM} and RME^{TM} are registered trademarks of Rheometric Scientific

6.2 Extensional flow

Extensional flows are common in most industrial processes but the fluids involved are often poorly understood, or poorly characterized, in extension. The concept of extensional rheometry is analogous to that of shear rheometry. Instead of obtaining the shear viscosity (and other related parameters) by applying a known force (stress) in shear and measuring the resulting displacement (or strain), a similar procedure is carried out in extension (tension). Hence the extensional rheological properties bear the same relationship to their shear counterparts as the Young's modulus does to the shear modulus in elastic solids. From the filling of shampoo bottles to the manufacture of artificial fibers and the coating of rollers in the printing industry, there is invariably an extensional kinematic component in industrial processes. Extensional kinematics always arise in free surface flows (e.g. in jets, fibers and sheet drawing processes), or if there is a squeezing mechanism or streamline acceleration. However, most viscometric methods available today for rigorously analyzing fluid properties rely on shear rheometry. Since polymer solutions, melts and suspensions can have markedly different shear and extensional behaviors, this approach can lead to identification of highly misleading parameter values.

There are currently methods that give some indication of the extensional behavior of materials, such as capillary rheometers or falling ball viscometers. However these approaches yield data that index, or rank materials, rather than provide absolute quantitative parameters, and it is very difficult to obtain results that are independent of the experimental configuration. In addition, the thermophysical behavior of the fluid in a stretching flow field exposed to ambient conditions may in itself be of interest. The conditions under which these stretching flow fields occur industrially, such as fiber spinning applications, often include curing, gelation or mass or heat transfer mechanisms which can only be studied successfully in analogous flow fields. Curing, vitrification, and crystallization all are strongly influenced by the flow field, and can be greatly enhanced in the presence of extensional flows. A technique that can measure relevant material properties for such processes would therefore be invaluable.

6.3   The HAAKE CaBER 1 rheometer

6.3.1 Operation principle

The **Ca**pillary **B**reakup **E**xtensional **R**heometer (**CaBER 1**) is conceptually based on the designs of Bazilevsky et al. (Bazilevskii et al., 1990, Bazilevskii et al., 1997). The instrument uses a laser micrometer to monitor the diameter of a thinning filament.
Fig. 2 shows a sequence of video images depicting the breakup of two fluids. The oil is a simple Newtonian fluid that is dominated by viscous forces. The lower set of images is for a dilute polymer solution comprised of a high molecular weight polystyrene molecule dissolved in the Newtonian oil utilized in the upper images. This solution is a model elastic fluid known as a "Boger Fluid" that strongly strain hardens in extension. Visually there is a clear difference between the samples even though their shear viscosity is virtually identical. These plots highlight the utility of the HAAKE CaBER 1. The evolution in the mid-point diameter is plotted versus time in Fig. 3.

The difference between the two fluids of Fig. 2 in extension is clearly shown. As a comparison a third set of data for a pressure sensitive adhesive is also plotted where the solvent in the solution is evaporating throughout the experiment. The thread then "sets", yielding a constant final diameter. Although these data do not describe the rheology of the fluid quantitatively (much like force and displacement are not rheological parameters for shear rheometers) they do give an indication of the fluids behavior and do not depend on any

constitutive model for interpretation.
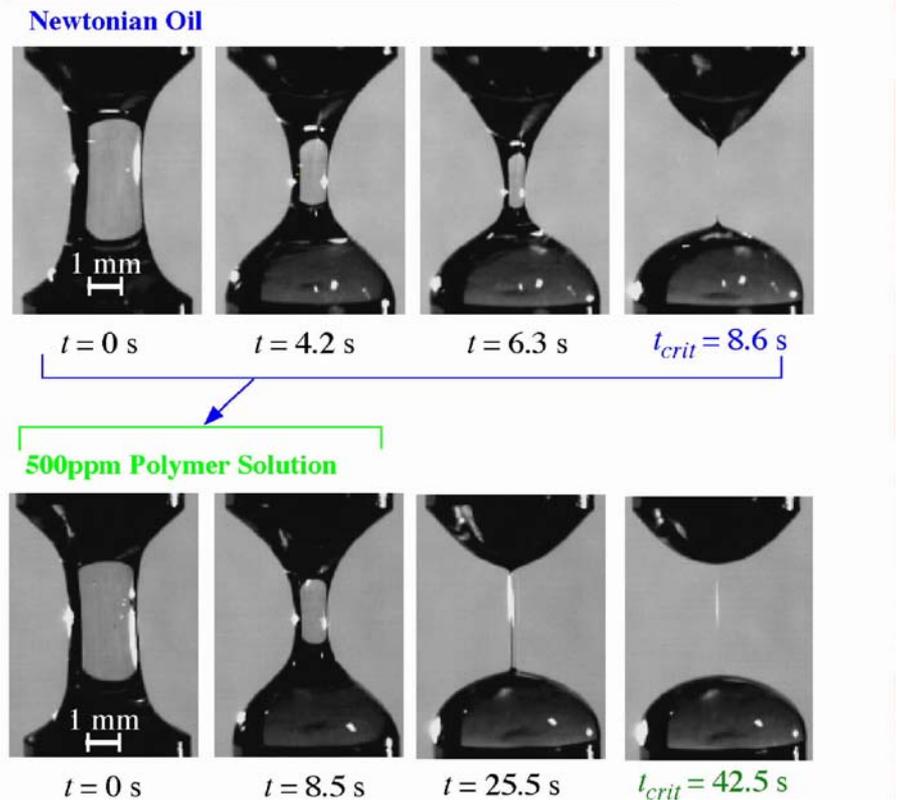
Fig. 2: Two sequences of images showing the breakup of a styrene oligomer (Newtonian) and the same oligomer with a dilute (500 ppm) high molecular weight polystyrene polymer added. This results in an extremely elastic solution that has a distinctly different behavior (from McKinley and Tripathi, Journal of Rheology).

Qualitative information that can be obtained from these data are time-to-breakup, and, from the shape of the curve, the "stringiness" of the fluid. It is clear from the data in Fig. 3 that the Newtonian fluid rapidly pinches off in a short time and the Boger fluid takes much longer, but does eventually reach zero diameter. Note that the Y-axis here is logarithmic and therefore an exponentially slowing drainage (as seen in the Boger fluid) is seen as a linear decrease on this plot. The adhesive, with an evaporating component "sets" and never falls below a certain diameter.
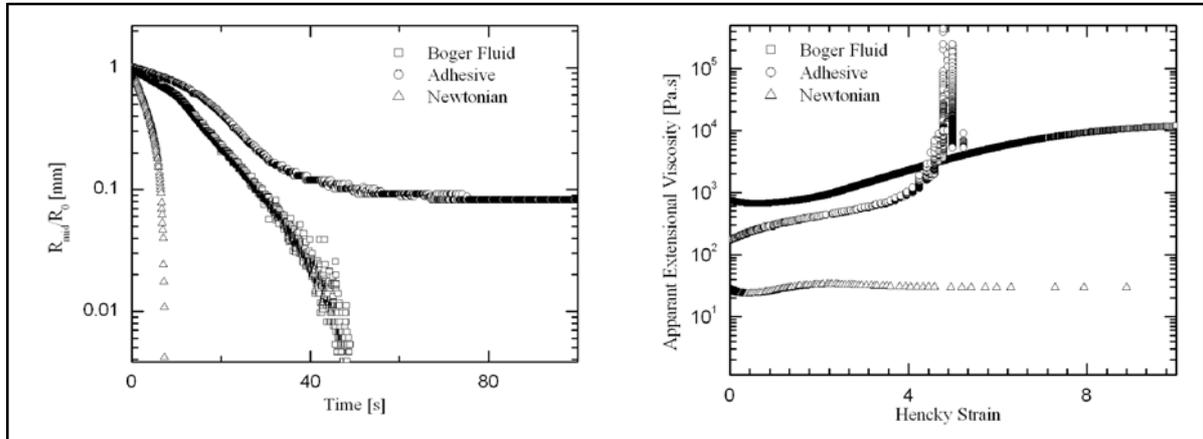
Fig. 3: Three sets of data showing normalized filament radius versus time. The data are for three fluids, the simple Newtonian and model elastic fluids of Fig. 2 and a Pressure Sensitive Adhesive. Left hand plot shows diameter versus time. Right hand plot shows an apparent extensional viscosity versus strain. The analysis and software required to compute extensional viscosity from the measuremed evolution in D_{mid} (t) is described in chapter 6.

The diameter versus time data that is the raw output of the HAAKE CaBER 1 is then used by the proprietary software to determine rheological parameters. More detailed discussion of this process will be given in Chapter 11, but in general two methods of extracting quantitative information from the instrument are available: 1) the available models can be fitted to extract rheological parameters; 2) the diameter data can be converted to an apparent extensional viscosity where the strain is defined by the diameter of the filament and hence varies with time. Note that both of these approaches assume a constitutive model, much like the analysis that would be traditionally performed on shear rheometer data.

6.3.2 Instrument

The HAAKE CaBER 1 instrument is shown in Fig. 4. The central column contains the linear motor and the rheometer plates. These rheometer plates are mounted axially in the unit and will be referred to as the upper and lower measurement plates in this document. Both plates are usually stainless steel and are supplied with a 6 mm diameter (the user may choose other options available from Thermo Fisher Scientific). The lower plate is mounted on a manual micrometer that allows vertical adjustment of the plate position. It is this adjustment that defines the rheometer geometry for the tests.

Closed   Opened   Plates detail

Fig. 4: HAAKE CaBER 1 instrument

Note the groove around the lower plate is intended to catch any waste material or act as a solvent reservoir if necessary. As can be seen the "wings" of the instrument can be moved backward for ease of access. This part of the housing contains the laser micrometer.

☞ The upper plate is removable and has a hole through its center that allows injection of fluid during loading.

6.3.3 Laser micrometer

The HAAKE CaBER 1 uses a high precision laser micrometer to accurately track the filament diameter as it thins. This laser micrometer gives the HAAKE CaBER 1 marked advantages over alternative techniques in the literature. Aside from its resolution (around 10μm) the micrometer is also immune to large ambient light fluctuations and can resolve small filaments easily (a different issue from the resolution).

This instrument is a class 1 laser, operating in the infrared. Although this type of laser is intrinsically safe due to its low power, as with all lasers, care must be taken when handling the unit since using any kind of laser could potentially cause ocular damage.

> **! Do not look into the beam and be aware that light scattered from the HAAKE CaBER 1 plates may be coherent enough to cause harm to the eye.**

6.3.4 Linear motor

The plate motion is controlled by a linear drive motor. This system allows fast response and reasonable control over the stretch profiles used. In the configuration supplied the user is free to choose between linear, exponential and "cushioned" stretches. These options will be discussed in more detail in sections 10.6.3. Note that the fastest stretch time is of the order of 20 ms (depending on stretch distance) and the motor has a positional resolution of 20 μm. There are no user serviceable parts in the linear motor and the user should be aware that high voltages and currents are present in this mechanism. Do not disassemble.

6.3.5 Temperature control

The measurement cell of the HAAKE CaBER 1 is equipped with a double cover. The outer cover (or door) is manually operated. The inner cover slides up and down automatically with the movement of the outer cover. The temperature inside the measurement cell of the HAAKE CaBER 1 can be controlled by means of an external circulator, the circulator hoses are connected to the back of the HAAKE CaBER 1 instrument.
The circulator fluid flows through the two blocks that guide the axial movement of the upper and lower measurement plates, thereby controlling the temperature of the two plates.

7. Information concerning the CE sign

Thermo Scientific electrical equipment for measurement, control and laboratory use bears the CE marking.

The CE marking attests the compliance of the product with the EC-Directives which are necessary to apply and confirms that the apparatus meets all relevant essential requirements of the directive, the defined relevant protection requirements.

The conformity assessment procedures were performed following a defined methodology according to each applicable directive.

The council decision 93/465/EEC shall be authoritative concerning the modules of the various phases of the conformity assessment procedures and the rules for the affixing and use of the CE marking, which are intended to be used in the technical harmonization directives.

To confirm compliance with the EC-Directive 2004/108/EC Electromagnetic Compatibility (EMC) our product was tested according to the EMC requirements for emission and immunity for electrical equipment for measurement, control and laboratory use.

This is a class A product. Compliance with the protection requirements in industrial areas is ensured. In a residential area this product may cause radio interference in which case the user may be required to take adequate measures.

Our strict standards regarding operating quality and resulting considerable amount of time and money spent on development and testing reflect our commitment to guarantee the high level of quality of our products even under extreme electromagnetic conditions.

Practice however also shows that even electrical equipment which bears the CE marking such as monitors or analytical instruments can be affected if their manufactures accept an interference (e.g. the flickering of a monitor) as the minimum operating quality under electromagnetic compatibility phenomena. For this reason we recommend you to observe a minimum distance of approx. 1 m from such equipment.

7.1 WEEE Compliance

This product is required to comply with the European Union's Waste Electrical & Electronic Equipment (WEEE) Directive 2012/19/EU. It is marked with the following symbol:

Thermo Fisher Scientific has contracted with one or more recycling/disposal companies in each EU Member State, and this product should be disposed of or recycled through them. Further information on Thermo Fisher Scientific compliance with these Directives, the recyclers in your country, and information on Thermo Fisher Scientific products which may assist the detection of substances subject to the RoHS Directive are available at www.thermo.com/WEEERoHS

8. Unpacking / Ambient conditions

8.1 Transportation damage

- Notify carrier (forwarding merchant, railroad, post office) etc,

- Compile a damage report.

Before return delivery:

- Inform dealer or manufacturer (Small problems can often be dealt with on the spot).

8.2 Contents of delivery

8.2.1 Standard delivery rheometer

The Rheometer is delivered in a recyclable package with the following content:

006-0072 HAAKE CaBER 1 instrument (includes linear motorcable to control box)
006-0033 HAAKE CaBER 1 control box
222-1643 Measuring system 6 mm (upper & lower plate)
222-0563 Data cable, instrument to control box, 25 pole
222-1322 RS232 cable, control box to PC, 25 to 9 pole

Connection cable conntry specific
087-0532 Fuse, 1.6 A for 230 V
087-1191 Fuse, 2.5 A for 115 V

222-1646 Data acquisiton card PCI for desktop PC + software + Data cable control box to data acquistion card
or
222-1645 Data acquisiton card PC-Card for notebook PC + software + Data cable control box to data acquistion card

098-5030 HAAKE CaBER 1 software on a CD
006-0075 Instruction manual HAAKE CaBER 1

8.2.2 Measuring system

Three different measuring systems, consisting of a lower and upper plate each, are available. These measuring systems have plates with diameters of 4, 6 and 8 mm respectively.

222-1642 Measuring system D = 4 mm
(upper and lower plate)
222-1643 Measuring system D = 6 mm
(upper and lower plate)
222-1644 Measuring system D = 8 mm
(upper and lower plate)

One set of 6 mm diameter plates is part of the standard delivery of the instrument.

8.2.3 Application software

The instrument is delivered with the National Instruments NI-DAQ^{TM} software (for Windows 95 / 98 / ME / NT4 / 2000 / XP) which is needed for communciation with data acquisition card and running the HAAKE CaBER 1 measurement software.

The HAAKE CaBER evaluation software does not need the NI-DAQ software.
The instrument is controlled with the HAAKE CaBER software for Windows 95 / 98 / ME / NT4 / 2000 / XP.

8.3 Space requirements

Good working conditions for a complete installation require an area of about 2 x 0.6 meters. The bench should be rigid with a level surface and easy to clean. The circulator used for the temperature control of the rheometer should be located on a separate bench or on the floor to avoid possible mechanical oscillation when the highest accuracy is set on the instrument.

8.4 Ambient conditions according to EN 61010

It is recommended to run tests in an air-conditioned room, (T = approx. 23 °C):

- indoors, max. 2000 meters above sea level,
- ambient temperature 15 ... 40° C,
- relative humidity max. 80% / 31°C ($\rightarrow$ 50% / 40°C)
- excess voltage category II, contamination level 2

19

9. Functional elements

9.1 Measuring instrument
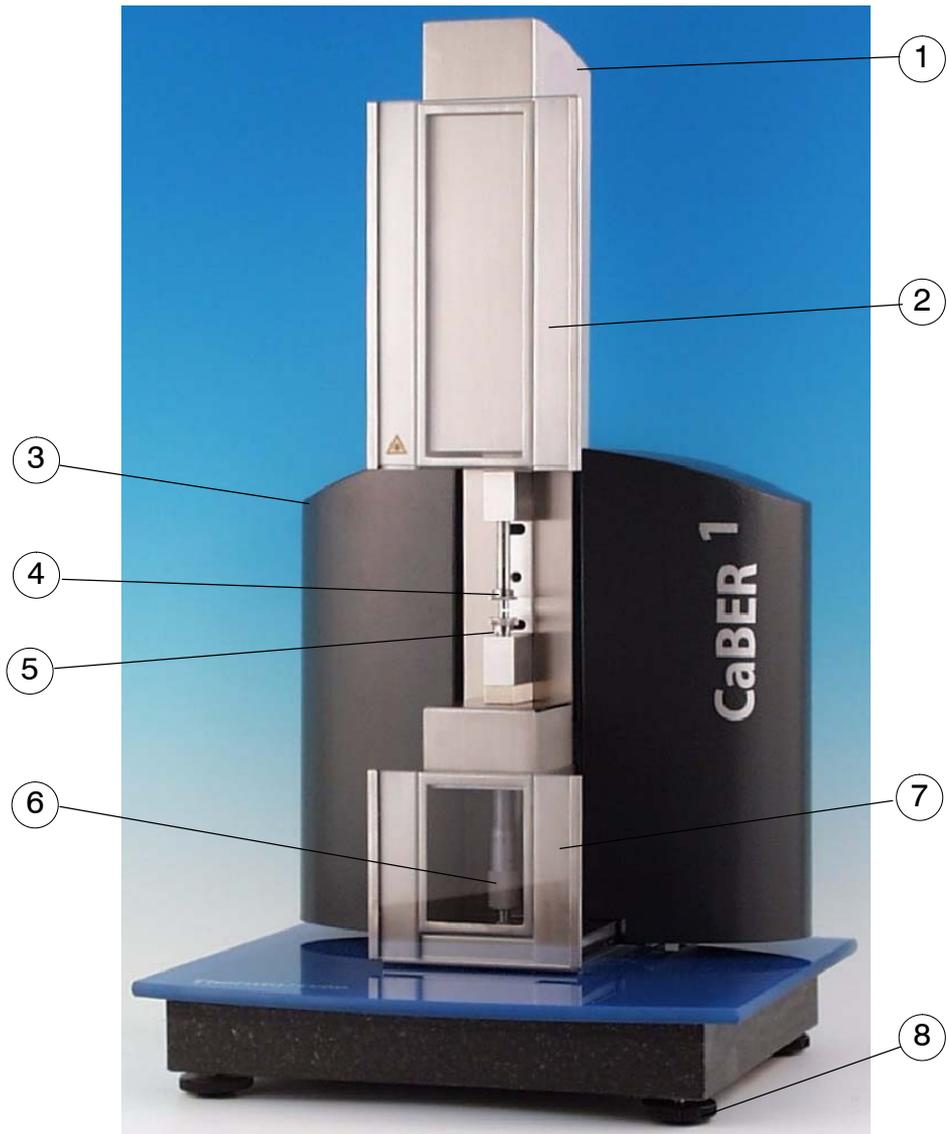
Fig. 5: Functional elements

1	Cover (fixed) for linear-motor
2	Sliding door (up-down) for measurement cell
3	Sliding covers (front-rear) for laser-micrometer
4	Measuring system, upper plate
5	Measuring system, lower plate
6	Micrometer screw
7	Sliding door (up-down) for micrometer screw
8	Adjustable feet

Functional elements

9.2   Measuring instrument - rear
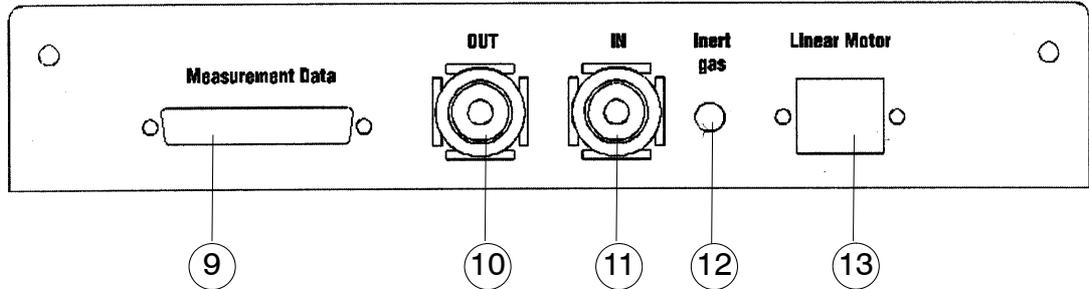
Fig. 6: Rear panel of the CaBER 1 instrument

9	Connection for measurement data	**12**	Connection for Inert gas
10	Water outlet	**13**	Connection for linear motor
11	Water inlet		

9.3   Control box - rear
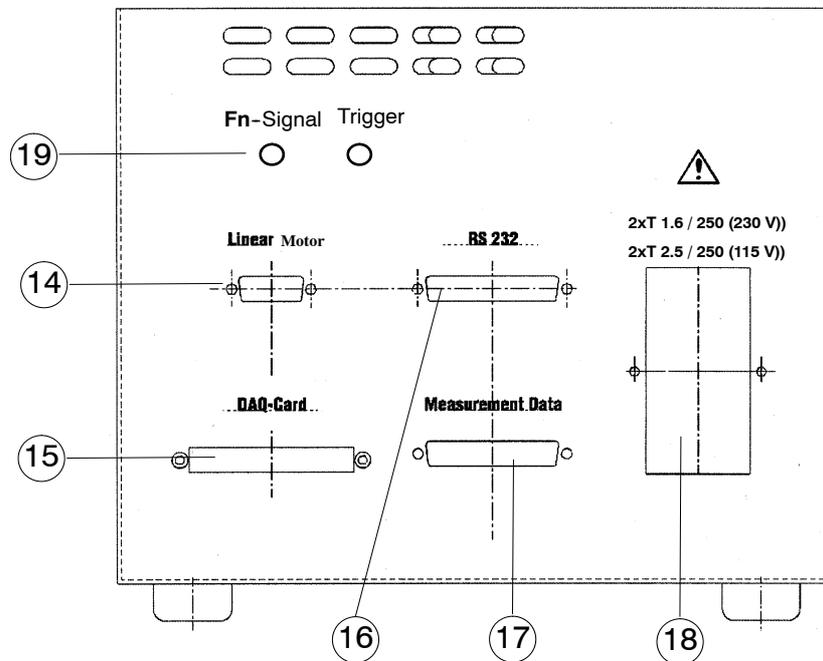
Fig. 7: Rear panel of the CaBER 1 control box

14	Connection for linear motor	**17**	Connection for measurement data
15	Connection for DAQ-Card	**18**	Mains switch with socket and fuses
16	RS232 interface connection	**19**	Fn–Signal and Trigger for Fn setup

10. Installation

10.1 Install the software and hardware

The Haake CaBER 1 device is delivered with two separate CD's which provide the NI–DAQ card driver and the Haake CaBER software. Please install all software elements on your PC before running the instrument. It is strongly recommended to perform the following steps in the order as listed below.

10.1.1 Install the National Instruments NI-DAQ^{TM} driver software

The CaBER 1 is supplied as standard with a National Instruments Data Acquisition card (NI–DAQ) in either PCI or PC–Card format for use in a standard desktop PC or notebook respectively.
After installing the NI–DAQ card start the PC again. The NI–DAQ installer software now asks if you want to install the documentation on your PC, there is no real need to do so. You can now check if the NI–DAQ card is installed properly by starting the Measurement & Automation Explorer software. In this software open the Devices and Interfaces folder, check that your device appears under Devices and Interfaces.

10.1.2 Install the NI-DAQ card

Insert the NI–DAQ driver CD in your PC's CD-ROM drive. The installation program should start automatically. If not, run the setup.exe program from the CD root directory.
Please make sure that you install the correct software which is related to your computer system:

- NI–DAQ 7.4.4 (Traditional legacy) for Windows XP

- NI–DAQ 7.5.0 (Traditional legacy) for Windows Vista and Windows 7

Additionally you need the driver for the RS 232 Com Ports: NI–VISA 5.0.2

To install the NI–DAQ software on a Windows XP computer please perform the following steps:

1. Insert the NI–DAQ software CD, open the folder "NI–DAQ 7.4.4" and start the setup program.

2. Chose the directory in which you want to install the program

22

To install the NI–DAQ software on a Windows Vista or Windows 7 PC please perform the following steps:

1. Insert the NI–DAQ software CD, open the folder "NI–DAQ 7.5.0", start the setup program and follow the instructions in Fig. 8
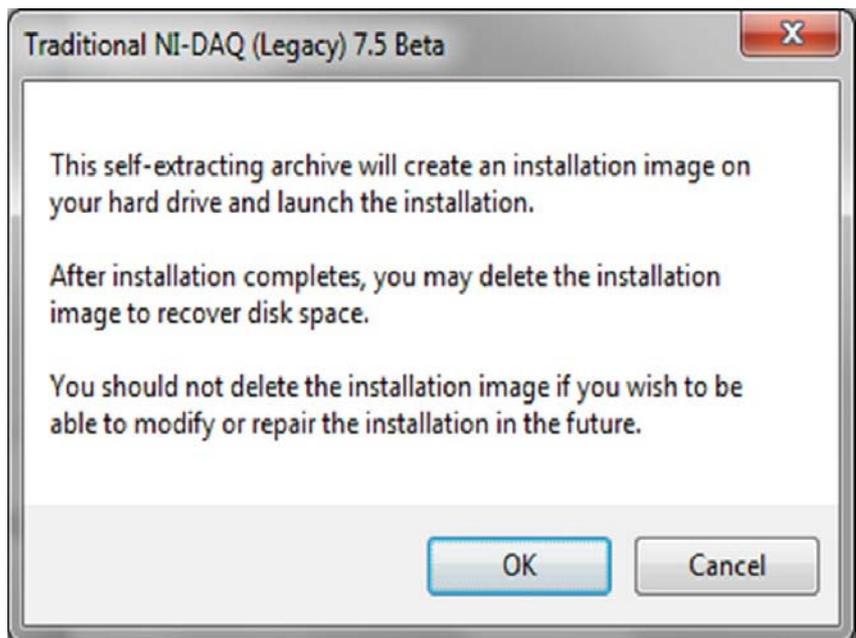
Fig. 8: Installation – instructions

Press ok and the Fig. 8 will appear:
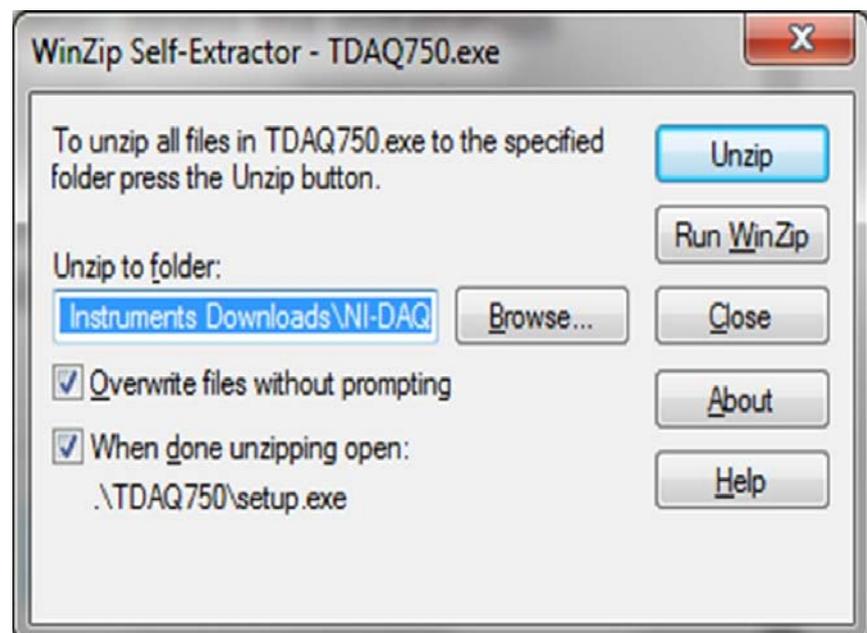
Fig. 9: Installation – instructions

2 Chose a folder in which you want to install the image data and press unzip. You can delete this image after the installation is complete.
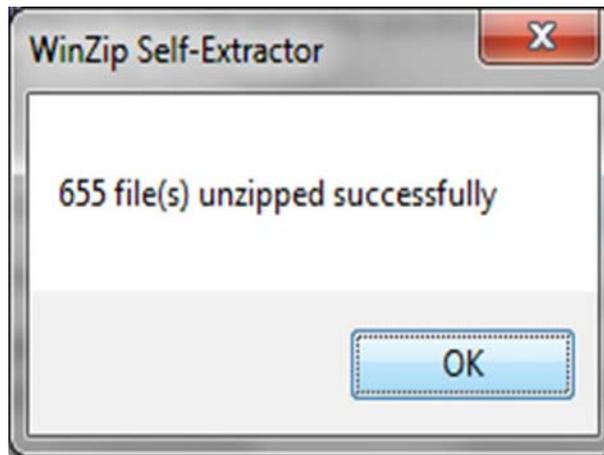
Fig. 10: Image installation successfully completed

After the image is built proceed with the installation by pressing ok and the installation program of the NI–DAQ software will start. Then press next to continue:
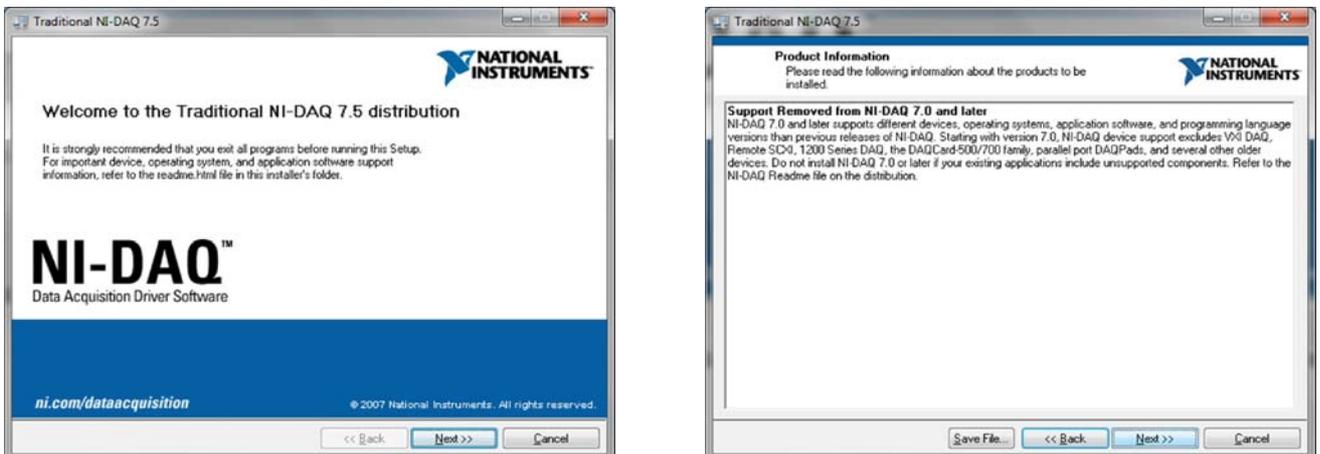
Fig. 11: Start of NI–DAQ – software installation

3 Select the directory in which you want to install the NI–DAQ software and continue the installation by pressing next:
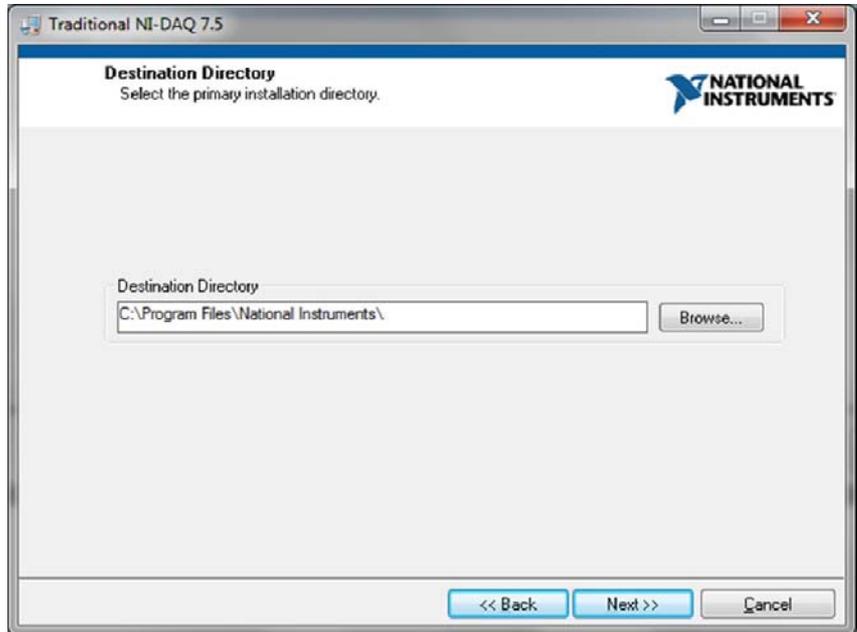
Fig. 12: Directory selection for NI–DAQ – software

4. For the CaBER instrument only the NI–DAQ device driver and the NI–DAQ OPC Server need to be installed. These two components are selected by default in the component–tree dialog, no manual selection of components is necessary (see Fig. 13). For that reason you can directly proceed with the installation by pressing next.
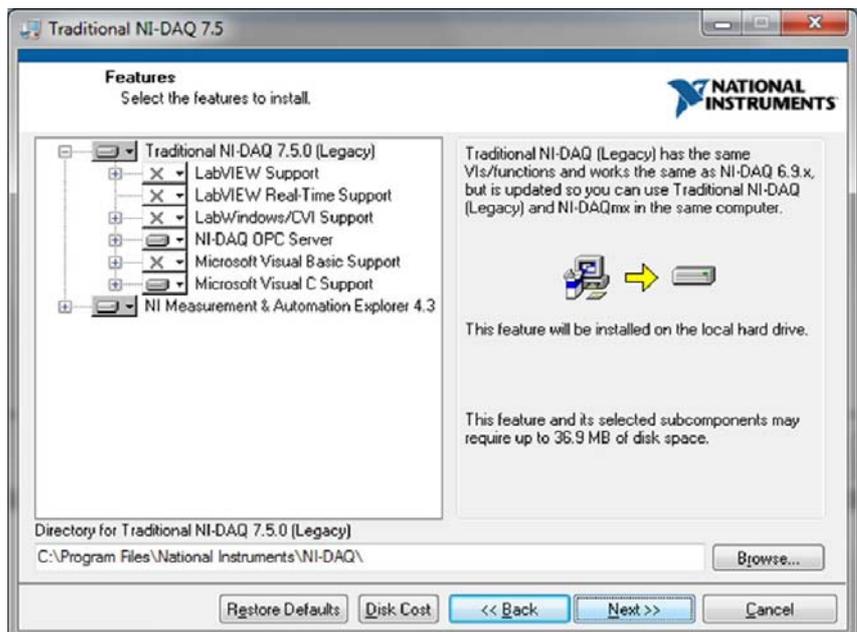
Fig. 13: Software components selection

5 Accept the license agreement and press next (see Fig. 14).
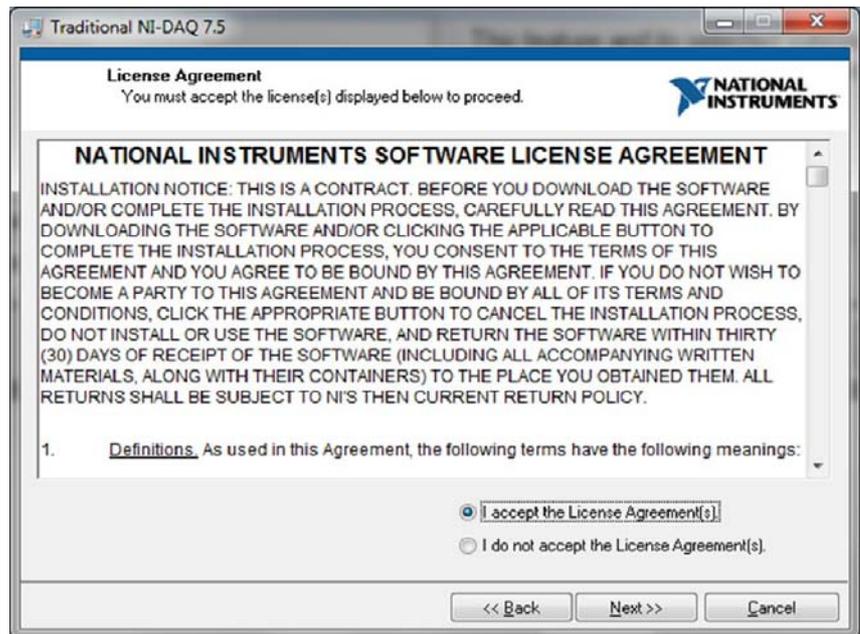
Fig. 14: Licence agreement

6 To avoid the breakup of the software installation by Windows please confirm that you trust the software of National Instruments Coorporation and continue the installation procedure.
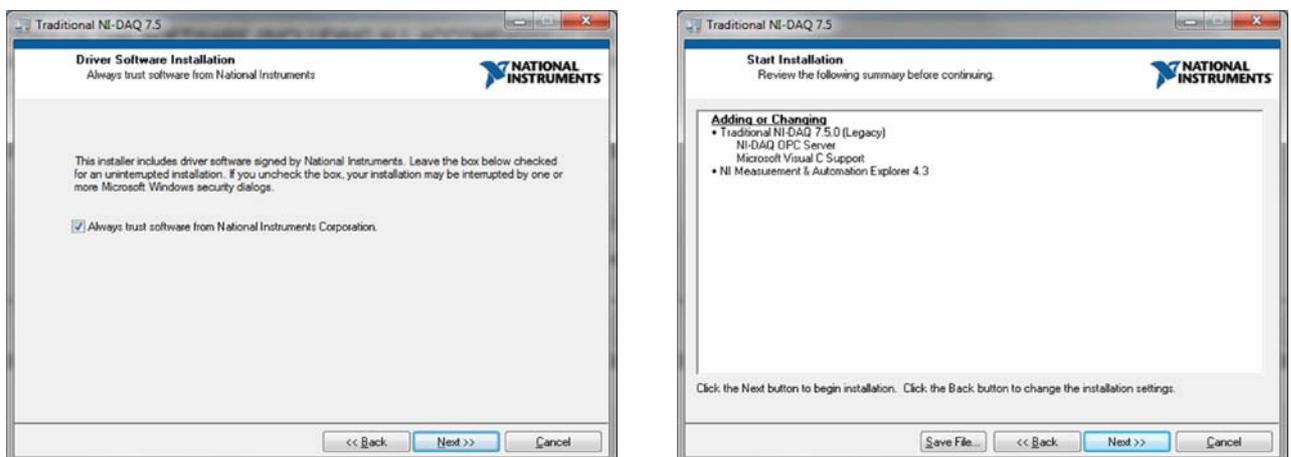
Fig. 15: Last steps of software installation

7 Please restart the PC when the installation of the software is finished.

10.1.3 Install the HAAKE CaBER 1 Software

The CaBER1 software is shipped on a single CD. Double click the setup icon and follow the prompts. The install software will also save an installer for the linear motor that is the core of the CaBER1. This item will be saved below the chosen CaBER directory but should not be needed unless the user encounters problems with the motor. Once all of the installation procedures has been completed, proceed to the next section.

10.2 Setting up the instrument

1. Lift the rheometer out of the package and place it on a stable, level table.

2. In the base of the measuring instrument, there are four feet which can be screwed in or out for levelling the unit.

3. Upon completion of the preliminary visual levelling, exact precision levelling can be carried out using a spirit level placed on the glass plate.

10.3 Connecting up

10.3.1 Cable connections

The HAAKE CaBER 1 instrument and control box are connected by two cables. The cable for the linear motor is attached to the instrument and can not be detached (for technical reasons), connect the plug at the other end of the cable to the linear drive socket on the control box. The measurement data sockets on the instrument and control box are connected using the cable with 25 pole plugs at both end.

The RS232 socket on the control box should be connected with the RS232 socket on your computer. The DAQ-Card socket on the control box should be connected with the socket on the NI-DAQ card in your PC using the 68 pin shielded cable.
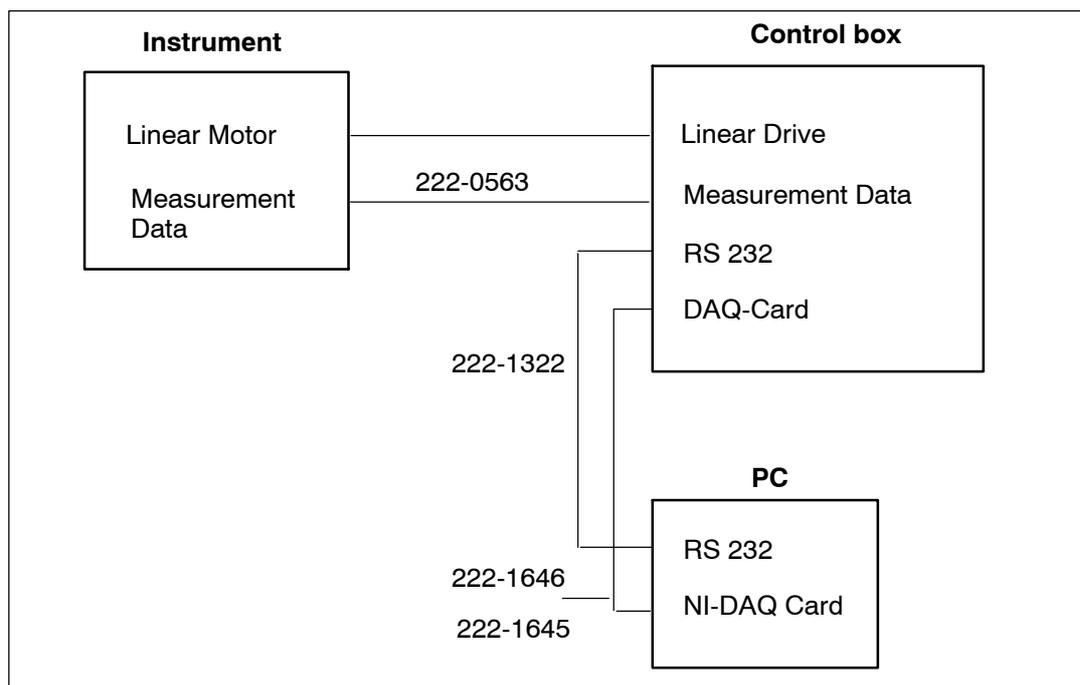
Fig. 16: Connection scheme

10.3.2 Hose connection

The HAAKE CaBER 1 measuring cell can be temperature controlled by connecting a suitable circulator to the nozzles labelled **In** and **Out** at the back of the HAAKE CaBER instrument.

The nozzle labelled **Inert gas** at the back of the HAAKE CaBER instrument can be used to flush the measurement cell with an inert gas.

10.4 Mains supply

! **Only attach the unit to a main socket with a grounded earth. Compare the local mains voltage with the specifications written on the name plates of the measuring instrument and the control unit. Voltage deviations of ± 10% are permissible.**

Mains cable and unit fuses:

Pull out the fuse holder from the mains socket and insert the fuses according to your local mains voltage.

230V	2xT1.6A
100/115V	2xT2.5A

Reinsert the fuse holder and make sure that the arrow on the mains socket is opposite to the arrow on the fuse socket that corresponds to your local mains voltage.

Use the mains cable according to your mains voltage.
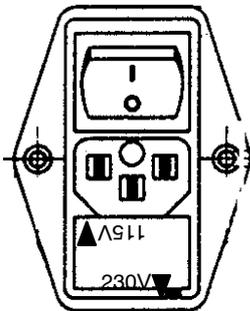
Fig. 17: Mains socket

10.5 Installing the normal force option

10.5.1 The HAAKE CaBER 1 normal force option

The CaBER 1 normal force option (order no. 222‑2021 for‑merly 603‑0469) consists of a Kistler 5015A charge meter (Fig. 18a), a Kistler type 9215 force sensor (Fig. 18b) plus cable, an adapter shaft (603‑0466) (Fig. 18c) for mounting the Kistler force sensor in the CaBER 1 and two cables, a normal force signal cable and a trigger signal cable.

Fig. 18a: Kistler 5015A charge meter   Fig. 18b: Kistler 9218 force sensor   Fig. 18c: adapter shaft

Fig. 18: Option normal force for HAAKE CaBER 1

In order to be able to use the CaBER 1 normal force option the CaBER control box must be equipped with two extra con‑nectors (one for the normal force signal and one for the trig‑ger cable) which are shown in Fig 17.

In case a new CaBER 1 is ordered together with the normal force option the CaBER control box will of course be equipped with these two connectors.

In case the normal force option is ordered for a CaBER 1 which has already been delivered (without this option) the CaBER 1 control will have to be modified. For this it is neces‑sary to ship the control box to the factory in Karlsruhe, Ger‑many.

The CaBER 1 software version 4.511 or newer is needed for operating the instrument.

Installation (Hardware)

The adapter shaft parts A, B, C and D (only of one the parts D is needed) and the Kistler force sensor (see Fig. 19a) have to be assembled into one unit as shown in Fig. 19b.
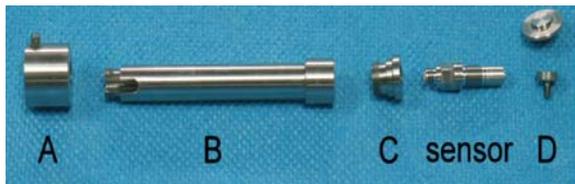
Fig. 19a: Adapter shaft parts + force sensor

Fig. 19b: Sensor mounted in adapter shaft

Fig. 19: The adapter shaft

After assembling the parts, the complete unit (as shown in fig. 19b) can NOT be mounted in the CaBER 1 because the BNC connector at the end of green cable will not fit through the mounting hole for the adapter shaft in the CaBER 1, therefore the assembly must be performed as described below.

1. The main hood consisting of two parts, which covers the laser micrometer, is easily removed by lifting the two hood parts from the frame after removing the two 3 mm hex bolts at the front (see Fig. 20a) and the two 3 mm hex bolts at the rear (see Fig. 20b). For removing the two rear bolts, the use of a 3 mm hex or Allen key tool with a length of at least 20 cm is recommended, see fig 20a. In order to gain access to the two rear bolts the hood has to be in the rear position.

! The two hood parts must both be removed at the same time. The two hood parts can only be separated after they are removed!

Fig. 20

Fig. 20a: Two 3 mm hex bolts on the front.

Fig. 20b: Two 3 mm hex bolts on the rear.

2. The lower sliding door is easily removed by spreading the rear part (see the arrows in Fig. 20c) and pulling it out of the brass slide guides.

Fig. 20c: Remove the lower sliding door.

Fig. 20d: Lower sliding door removed.

3. In order to remove the lower cover plate two screws at the side (see Fig. 21a) and one screw at the top (see Fig. 21b) must be removed. After the screws are removed the cover plate can be removed with some slight bending and wiggling.

Fig. 21:
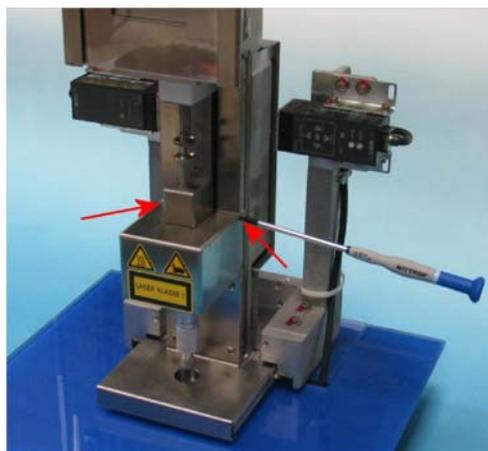
Fig. 21a: Remove screws at the side.
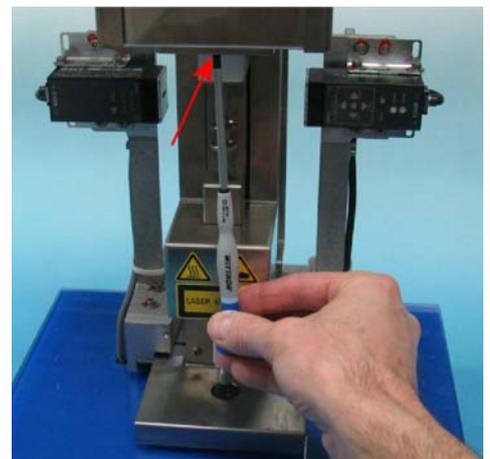
Fig. 21b: Remove top screw.

4. In order to remove the lower shaft, first remove the upper and lower measuring plates (see arrow A in Fig. 22a), then loosen the two 2 mm hex screws which hold the lower shaft on the micrometer screw shaft (see arrow B in Fig. 22a), then loosen the 4 mm hex bolt which clamps the micrometer screw flange (see arrow C in Fig. 22a). The lower shaft can now be removed by sliding it downwards out of the hole.
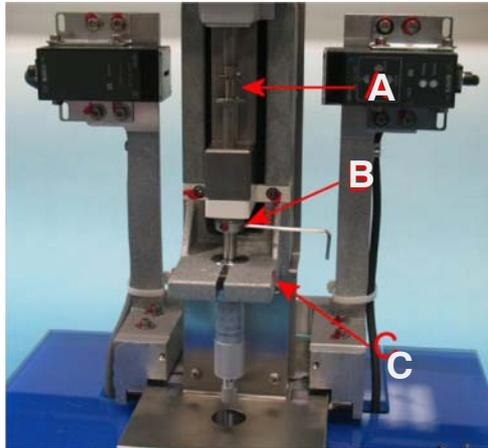
Fig. 22:

Fig. 22a: Remove original lower shaft.

Fig. 22b: The original lower shaft + measuring plate

5 Insert the adapter shaft (part B in Fig. 19a) in the hole (see Fig 23a), then slide the small connector of the green normal force signal cable through the adapter shaft (see Fig 23b). It is recommended to guide the cable through the hole in the CaBER 1 base plate (see Fig. 23c, Fig. 23d) in order to prevent problems with the lower sliding door.

Fig. 23:
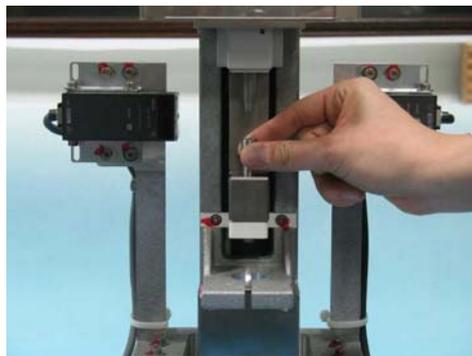
Fig. 23a: Insert the adapter shaft.
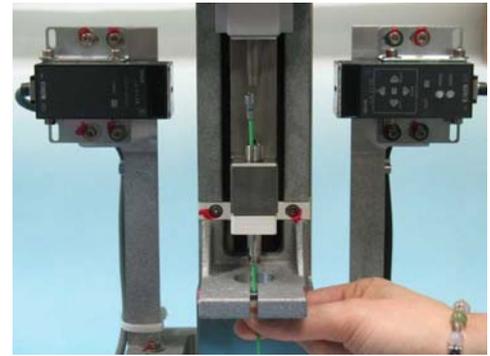
Fig. 23b: Insert the sensor cable.
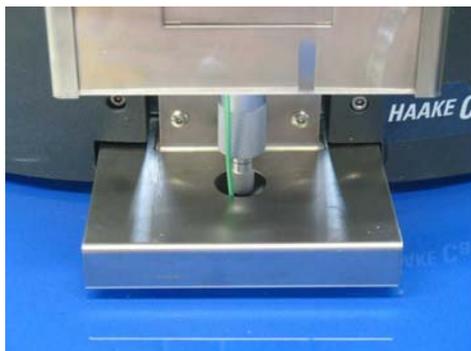
Fig. 23c: Guide cable through base plate hole.
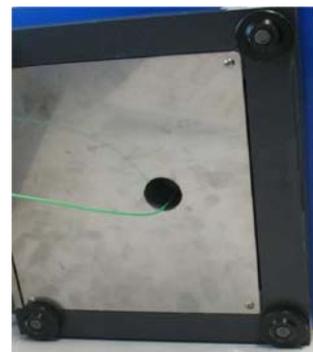
Fig. 23d: Guide cable through base plate hole.

Connect (screw) the normal force sensor to the green normal force cable and screw the adapter ring (part C in Fig. 19a) on the normal force sensor (see Fig. 23e).

Then mount (screw) the normal force sensor plus adapter ring into the adapter shaft (see Fig. 23f). Please note that due to long sensor cable it is easier to hold the sensor and adapter ring assembly and screw (rotate) the adapter shaft!
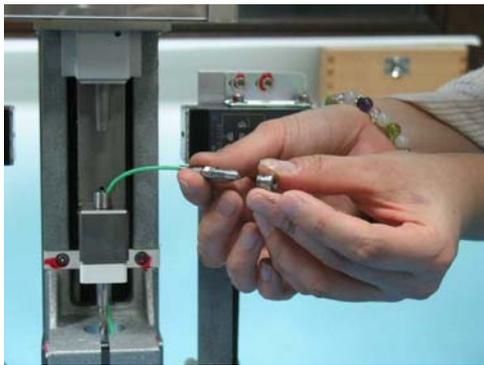
Fig. 23e: Mount the upper adpter ring.
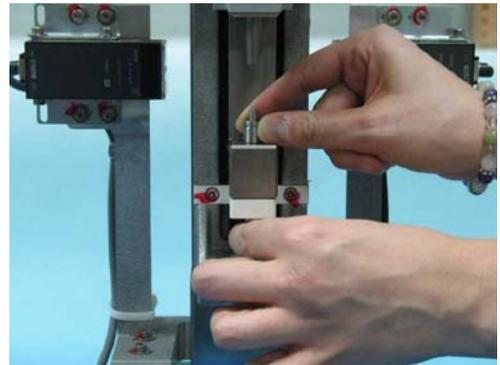
Fig. 23f: Mount the sensor inthe shaft.

Guide the green cable through the slit in the shaft and mount (screw) the lower adapter ring (part A in Fig. 19a) onto the lower end of the adapter shaft (see Fig. 23g) make sure not to damage the green normal force sensor signal cable.

Put the micrometer screw back in place and tighten the clamping bolt (see arrow A in Fig. 23h). Then tighten the screws which clamp the adapter shaft on the micrometer using a small screwdriver (see arrow B in Fig. 23h).
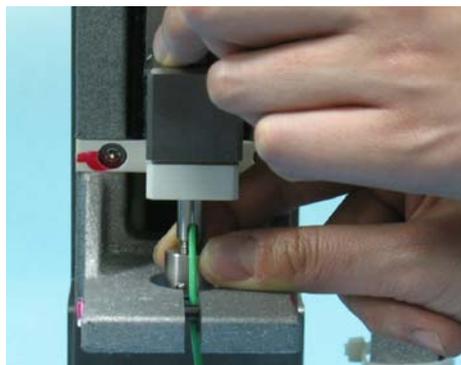
Fig. 23g: Mount the lower adapter ring.
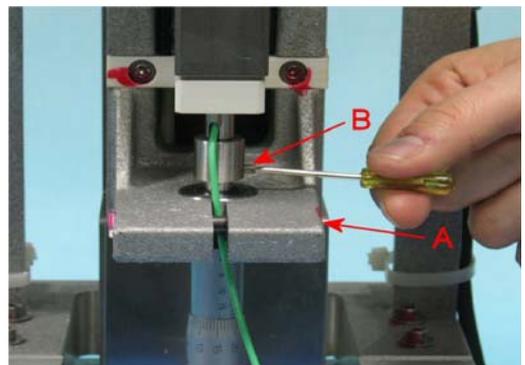
Fig. 23h: Tighten the bolts.

6 Replace the lower cover plate, then the sliding door and then the main hood. For this please see steps 3 to 1 above.

☞ Please make sure not to damage the cable when moving the lower sliding door downwards when the cable is not guided through the hole in the instruments base plate.

7 Connect the BNC connector of the green force sensor signal cable to the "Sensor charge" BNC connector on the back of the Kistler charge meter (this cable is not shown in Fig. 24). Then connect the short black BNC cable to the "Output" connector on the back of the Kistler charge meter and the "Fn–Signal" connector on the back of the CaBER 1 control box.
Connect the trigger cable (green connector and black connector) to the "Remote Control" connector on the back of the Kistler charge meter and to the "Trigger" connector on the back of the CaBER 1 control box.

Fig. 24: Signal and trigger cables

10.6 Switching on

Operating the single switch at the back of the control box should illuminate the green power indicator on the front panel . The linear motor will now more the upper plate down and up again and then stop moving. Some noise may be audible from the linear motor (a slight "hiss" or "hum"). This is normal. At this point the laser micrometer is switched on.

☞ For good laboratory practice, do not look into the square apeture of the micrometer at any time-even if you think it is off.

☞ After switching on the instrument **always** wait 15 minutes before starting any measurement. This time is needed for the laser micrometer to "warm-up" and reach a stable diameter measurement value.

11. The HAAKE CaBER 1 control software

11.1 Introduction

The operation of the HAAKE CaBER 1 software is intended to be simple and intuitive. This chapter will lead the user through the various functions of the HAAKE CaBER 1 control software and give some guidance in terms of run-times, options, and the applicability of the models available. Note that when a help button is visible on the panel there is run-time context sensitive help available (although at the time of press this function is somewhat limited).

11.2 Start-up

Fig. 25: Determinig system geometry
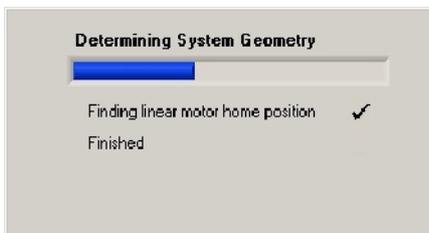
On start-up the HAAKE CaBER 1 software performs some diagnostics and initial setup routines that are invisible to the user. During this time a "splash" screen is shown, and then the instrument determines the system geometry (see Fig. 25). When this cycle is finished the HAAKE CaBER 1 front panel is opened as shown in Fig. 26.

When the DAQ-card and or the linear motor are not detected by the software a message will be displayed in the splash and the software will run in "demo" mode.

11.3 Front panel
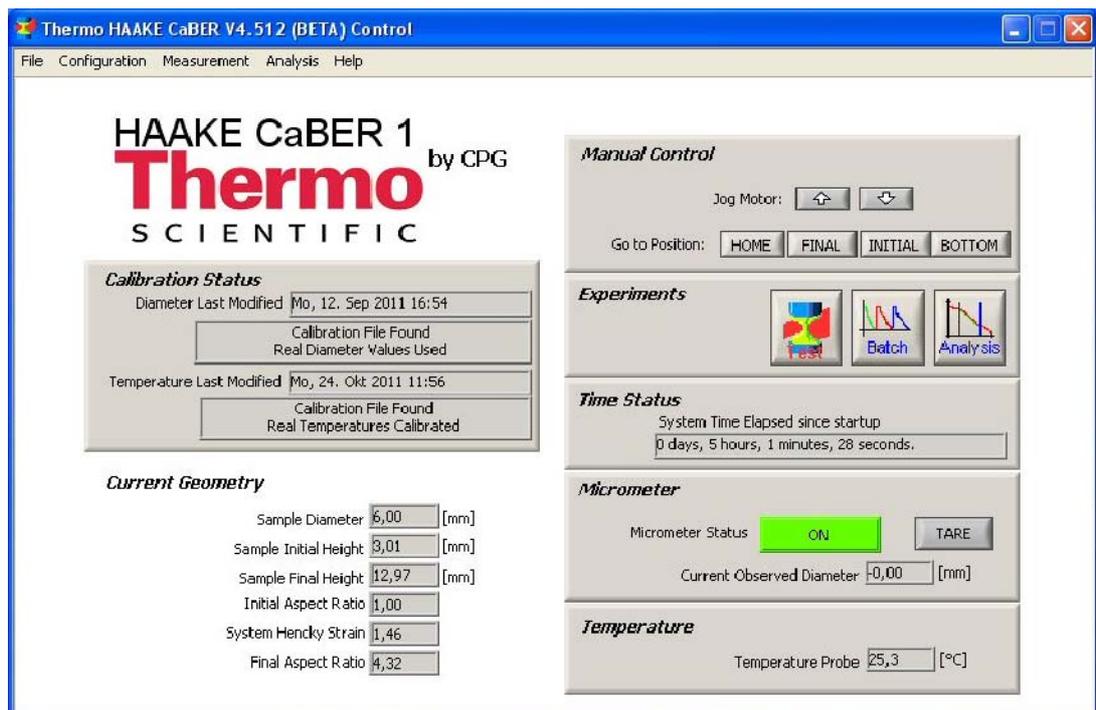
Fig. 26: The HAAKE CaBER 1 front panel

All functions of the CaBER 1 software is reached via the front panel which is displayed in Fig. 26. In the following sections the operation software will be explained in order of the front panel menus from the left to right hand side. The menus with the options available to the user are shown in Fig. 27.
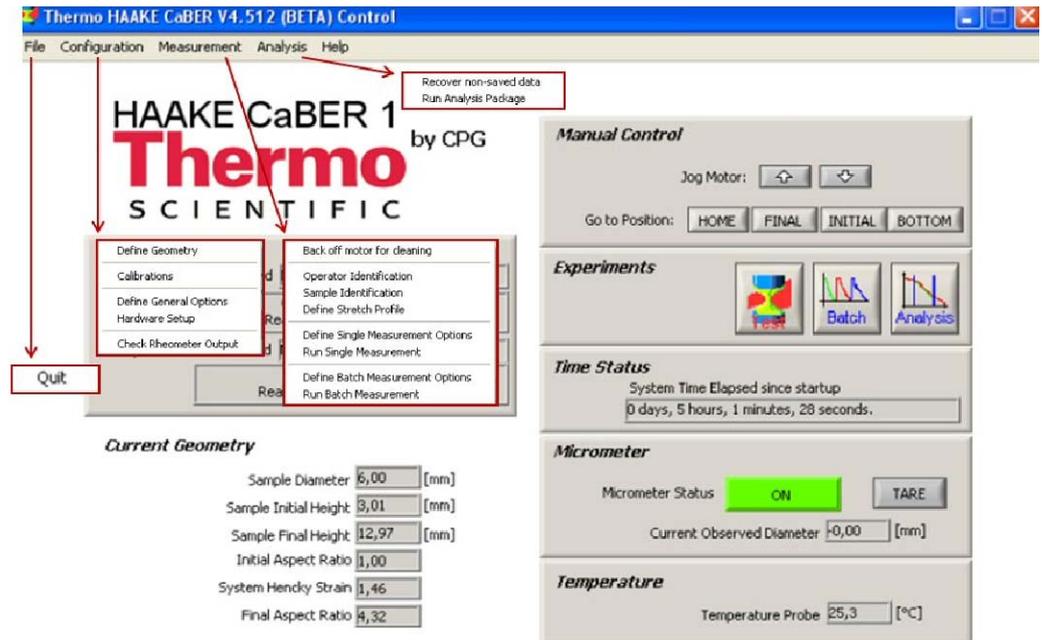
Fig. 27: The main menu

11.4 File menu

The Quit function (Fig. 27) allows the user to close the software. Note that the experimental software in general runs in one window. The analysis software is a separate window. Although all windows (apart from pop–up options) can be minimized, none of them can be closed by pressing the "X" symbol in the top right corner.

11.5 Configuration menu

Fig. 27 presents the functions available in this menu.

11.5.1 Define geometry

This window (Fig. 27) allows the user to adjust and define the configuration of the CaBER 1 measuring system. Note that the only adjustable parameter in this screen is the plate diameter.
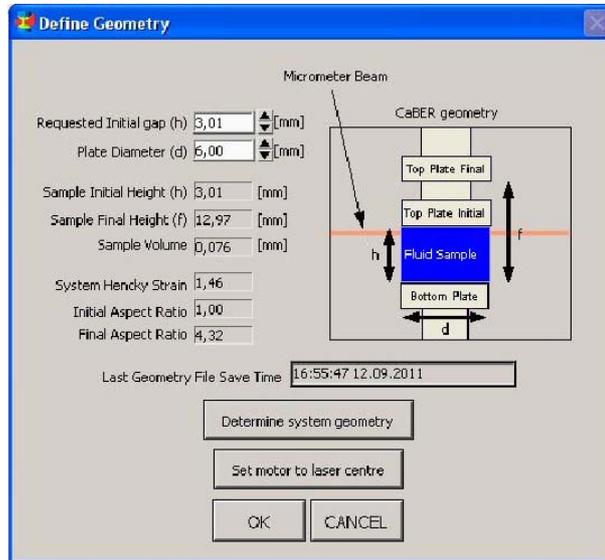
Fig. 28: Define Geometry

Click on the "Determine system geometry" button to have the software determine the plate configuration and then determine the positions of the laser beam and bottom plate. Any time the bottom plate is moved this routine should be run.

The linear motor will first determine the "home" position of the upper plate.
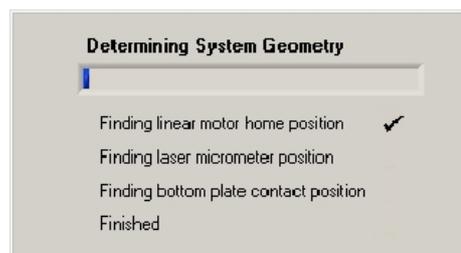
Fig. 29: Monitoring of the motor homing function

OK will save the geometry data listed in the "caber_config.cfg" file in the HAAKE CaBER 1 program directory.

Cancel will restore the old values.

11.5.2 Calibrations

The calibration status of the instrument can be viewed by accessing the calibration menu as shown in Fig. 30. This menu also allows the instrument micrometer and temperature sensors to be recalibrated independently. The Calibration status window is shown in Fig. 30. This window shows the calibration factors currently in use and when the instrument was last calibrated. If the instrument has been calibrated since the software was last started, this information is shown also. The micrometer and temperature sensors can be calibrated from this window as well.
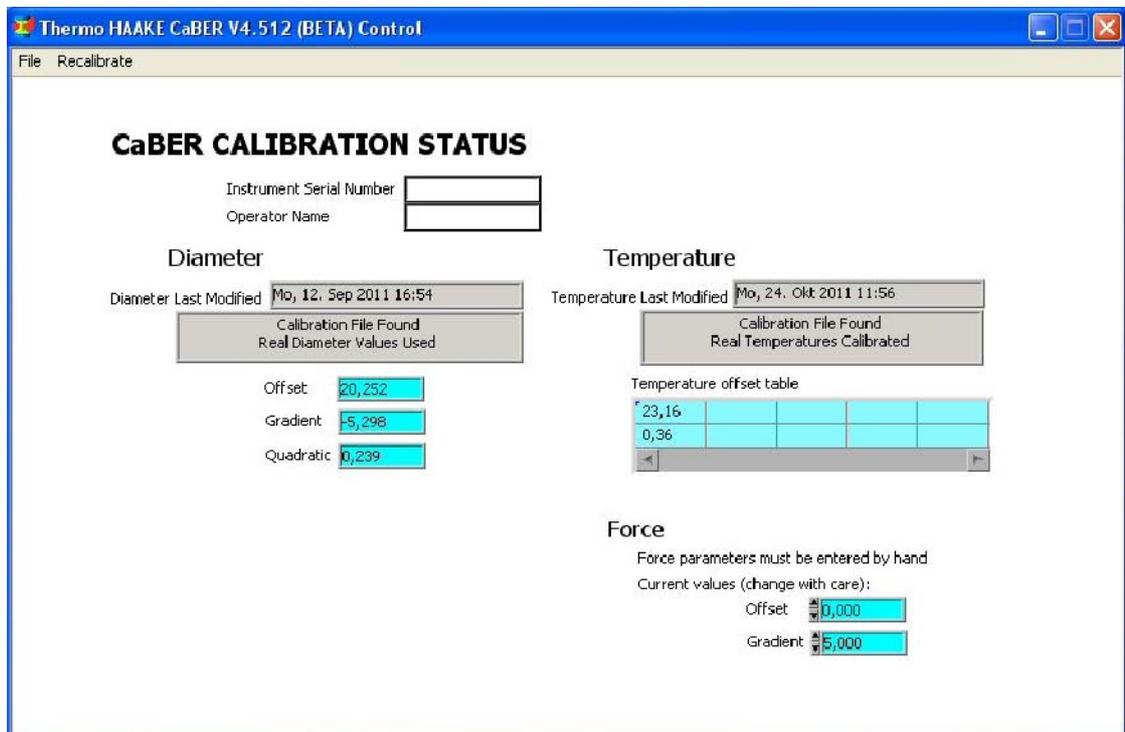
Fig. 30: Calibration status

11.5.3 Define general options

The define general options function allows the user to select a number of options. Fig. 31 presents these options as they appear by default.

Force Analysis after save: Forces the software to start the analysis package after every measurement.
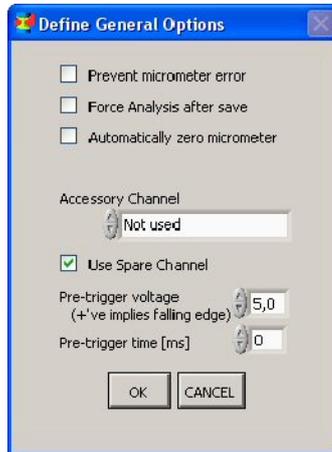
Fig. 31: General options

Automatically zero micrometer: Every time a measurement is taken the micrometer voltage will be zeroed if this option is checked.

11.5.4 Hardware setup

This window (Fig. 32) allows the setup of the instrument (specifically the data acquisition card) to be verified. Depending on which DAQ card is being used this option may show slightly different parameters that are automatically selected by the software. The only user adjustable parameter is the device number, which will normally be 1 unless there are other NI-DAQ cards present. This value should be consistent with the device number in the National Instruments "Measurement Explorer" installed with the card.
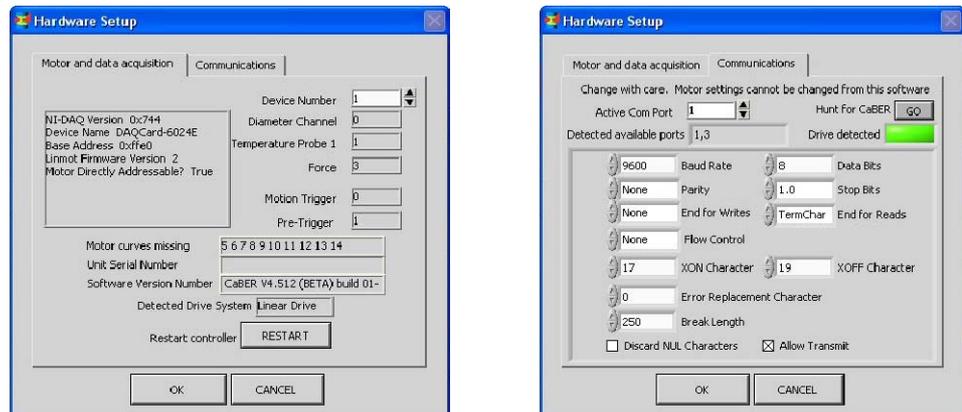
Fig. 32: Hardware setup

11.5.5 Check rheometer output

Clicking the check rheometer output command will open the window shown in Fig. 33. This window allows the raw signals from the instrument to be examined and simple linear motor motions to be performed. This section should not normally be required and is intended to be used as a diagnostics screen if you are having problems with the instrument.

In case of any problems with the micrometer output first make sure that nothing is blocking the laser beam. For that reason please turn the micrometer screw in its lowest position an check the rheometer output once again. If the rheometer output still shows bad values the laser beam has to be readjusted. To perform this first remove the small cover at the right front side of the CaBER. Then turn the screw behind that cover carefully with a small screwdriver until the micrometer output is around 4.95 V.
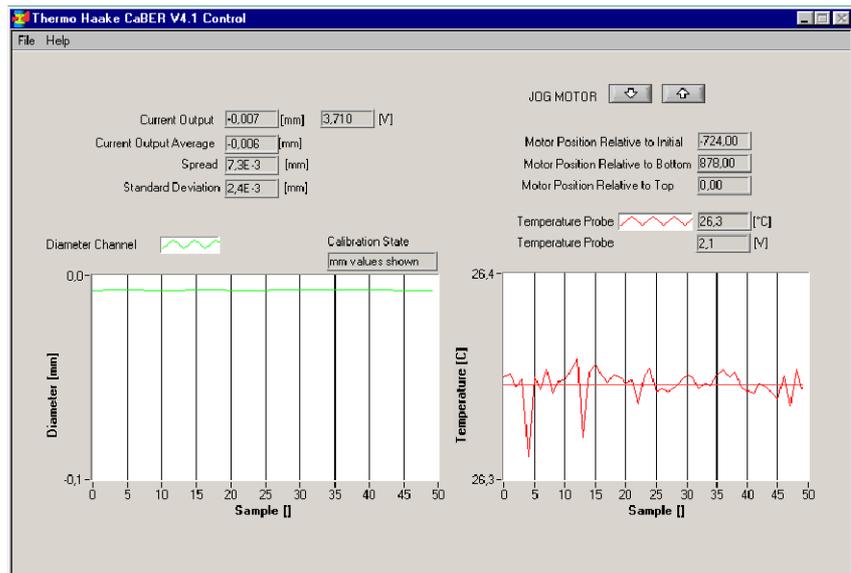
Fig. 33: Check rheometer output

11.6 Measurement menu

Operational details of the measurement options will be given in the next chapter.

11.6.1 Back off motor for cleaning

This command allows the user to back off the upper plate to the home position. No other functions are active while the upper plate is backed off.

11.6.2 Operator identification and Sample identification

Clicking one of these two commands will open the Identification dialog (see Fig. 34) with the corresponding tab. The information on the Operator tab is saved and will be restored when restarting the software. All of the information entered in this dialog is saved in the HAAKE CaBER 1 data files.

41

By pressing the sample clip all information about the sample can be entered. As shown in the introduction, the surface tension is used to calculate the extensional viscosity. Additionally, for Newtonian materials, one of the fitting models yields the capillary velocity which is the ratio of the surface tension to the viscosity. If the surface tension is entered into the software, the viscosity can be reported.
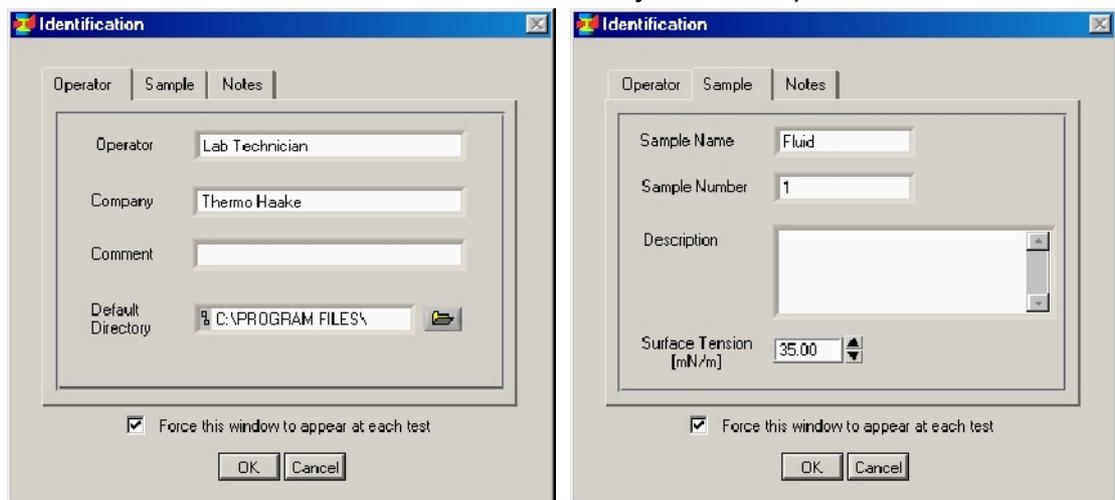
Fig. 34: Sample and Operator identification

11.6.3 Define stretch profile

The linear motor of the HAAKE CaBER 1 can be set to use three different "Stretch" profiles currently. In all cases the distance moved (d) relative to a reference distance (d_0) is defined by the geometry of the HAAKE CaBER 1 (i.e. the position of the lower plate) and is not an adjustable parameter in this menu. The time requested is scaled against a reference stretch time of 20 ms (t_0). The stretch options are "Linear", "Cushioned Strike" and "Exponential" (See Fig. 35):
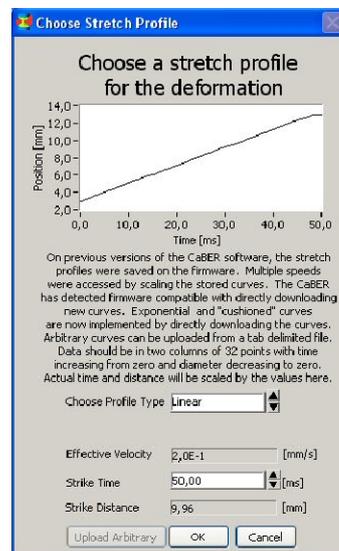
Fig. 35: Stretch Profile Options

Linear:
A simple linear motion of the form

$$d_{(t)} = (d / d_o) (t / t_o)$$

The strike time, or step strain deformation rate, can be adjusted for two reasons. First and foremost, the stretch rate has to be fast enough to finish stretching the fluid before capillary forces cause it to breakup, in order to get some measurements of the breakup. For a lower viscosity material, a fast strike time should be used. Secondly, the stretch rate can influence the breakup kinetics of complex non–Newtonian materials, much like non–Newtonian materials have a shear rate dependence. In order to explore material responses to different deformation histories, a researcher will often adjust the strike time.

Please note that the strike time does not influence the strain or strain rate in the sample. The actual extensional flow that is measured by the HAAKE CaBER 1 is driven by the sample properties only. This is in contrast to rotational rheometry, where the flow of the fluid is directly influenced by the mechanical deformation force that is applied to the fluid.

Exponential:
An exponentially accelerating curve, where the stretch time and the time constant are defined by the user, in the form

$$d_{(t)} = (d / d_o) (1 - e^{at}) (1 - e^{at}o).$$

Cushioned Strike:
A fundamentally linear motion at the fastest strike time available (approximately 20 ms) but with an exponential deceleration at the end of the motion to reduce problems caused by fast deceleration.

11.6.4 Define single measurement

The tests available to the user of the HAAKE CaBER 1 unit drop broadly into two categories, namely "batch" measurement and "single" measurement. The batch tests will be dealt with later in sections 10.6.6 and 10.6.7. The most common testing protocol will usually be based on the "single" measurement.
Within the single measurement option the user has two choices (Real-Time and High-Speed) that will influence the experimental parameters chosen. The Real-Time mode presents the data as it is collected. For most fluids this is perfectly adequate and allows the user to monitor the experiment as it evolves. However for some fluids that break-up

quickly (less than 0.5 s) the High-Speed mode allows a much faster sampling rate to be used. High-Speed does not refer to the plate motion, but to the sample rate. In this mode the user must choose an experiment length and sample rate. Fig. 36 shows the dialogue with which the user is presented.
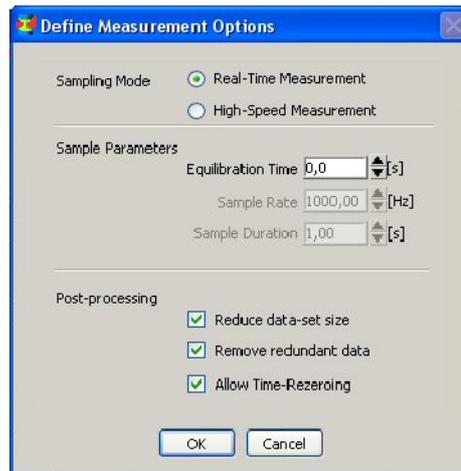
Fig. 36: Options for "single" measurement modes

In all cases the user can choose a number of post-data collection processing options:

Reduce data-set size allows the software to remove redundant data (this process is performed by knowing the start time accurately and determining the end of the data through changes based on the final data observed;

Remove redundant data "trims" the data by removing data where the rate of change of the diameter is slowest. In effect this option "resamples" the data with a varying time step, maximizing the data density for fast varying diameters.

Allow Time-Rezeroing uses the known stretch time of the motor to zero time at the point where the motor stopped moving. In principle it is only the data beyond this point that is useful in the HAAKE CaBER 1 experiment.

11.6.5 Run single measurement

The experimental parameters chosen in the previous section are used when the "run single measurement" option is chosen. An experiment is performed by following the prompts. As soon as the experiment is selected, the sample identification pop-up window appears unless it has been deselected. Next, the motor moves the plates to their initial position and then prompts the user to release the plates. At this point (and with the plates at their initial gap) the user should load their sample. For more information on this subject see the next chapter.

11.6.6 Define batch measurement

The batch measurement experiment is intended to allow the study of time-evolving samples. However, care should be taken with these materials since the air/fluid interface is often the point at which the material is changing fastest (in evaporation for example). The test assumes that the fluid does not drain away, and that the initial liquid "column" can be reliably reformed. In this setup dialogue the user must choose a period of time for both the drainage and rest steps. They must also choose a number of cycles. All of these values must be self-consistent (inconsistencies are indicated by the warning texts). The software chooses a data rate suitable to generate 1000 points during the drainage time stated by the user. Fig. 37 displays the dialogue for this measurement option.

☞ Make sure that the sample volume does not change between the single tests of the batch series. Otherwise the collected data is not reproducible.
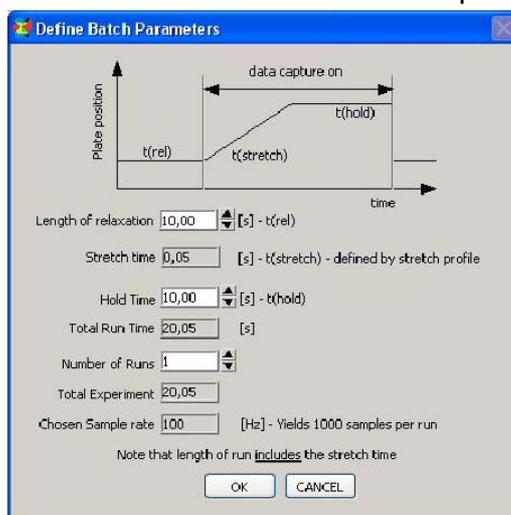
Fig. 37: Defining options for batch measurement.

11.6.7 Run batch measurement

This section runs the batch measurement test. Much like the single experiment test the user is guided by prompts to perform the experiment. As before the fluid is loaded once the plates are in their initial position.

11.7 Analysis menu

This menu runs the analysis package. This module can also be reached from the Windows start menu. More details of this software will be given in the next chapter.

11.8 Help menu

This menu accesses the About and Help options for the HAAKE CaBER 1 software. If you need to find the software version number please check here.

12. The HAAKE CaBER 1 analysis software

12.1 Introduction

The HAAKE CaBER 1 analysis program can be run either from the HAAKE CaBER 1 control program by choosing the analysis package from the analysis menu, or independently from the start menu.

In some respects the raw data obtained by the HAAKE CaBER 1 can provide much information about the fluid on its own. However, as with any rheometer, reprocessing of the data adds considerably to its value. The analysis package can be considered to operate in two ways:

- Determination of a model fit to the capillary data. As with any rheometer, the raw data can be processed by fitting it to a model, thus yielding the parameters of a constitutive equation. For many fluids this can be extremely powerful, and can clarify the behavior of the fluid quickly and easily. Naturally, care must be taken when following this approach because many models can fit data reasonably well, but yield physically irrelevant parameters. For more details of the actual theory behind these models please see the literature references at the end of this manual.

- Generate a model independent interpretation of the fluid's behavior by producing an "apparent" extensional viscosity. Although this interpretation does not return a single value parameter, and bundles all effects such as elasticity and viscosity into a single curve, it can be invaluable for studying complex fluids. The theory behind this plot is also discussed in the following section.

The HAAKE CaBER 1 analysis software screen is divided into four sections:
The graph section, the legend, the model parameter section and the program dialogs. These sections can be seen in Fig. 38 and are described in the following paragraphs.
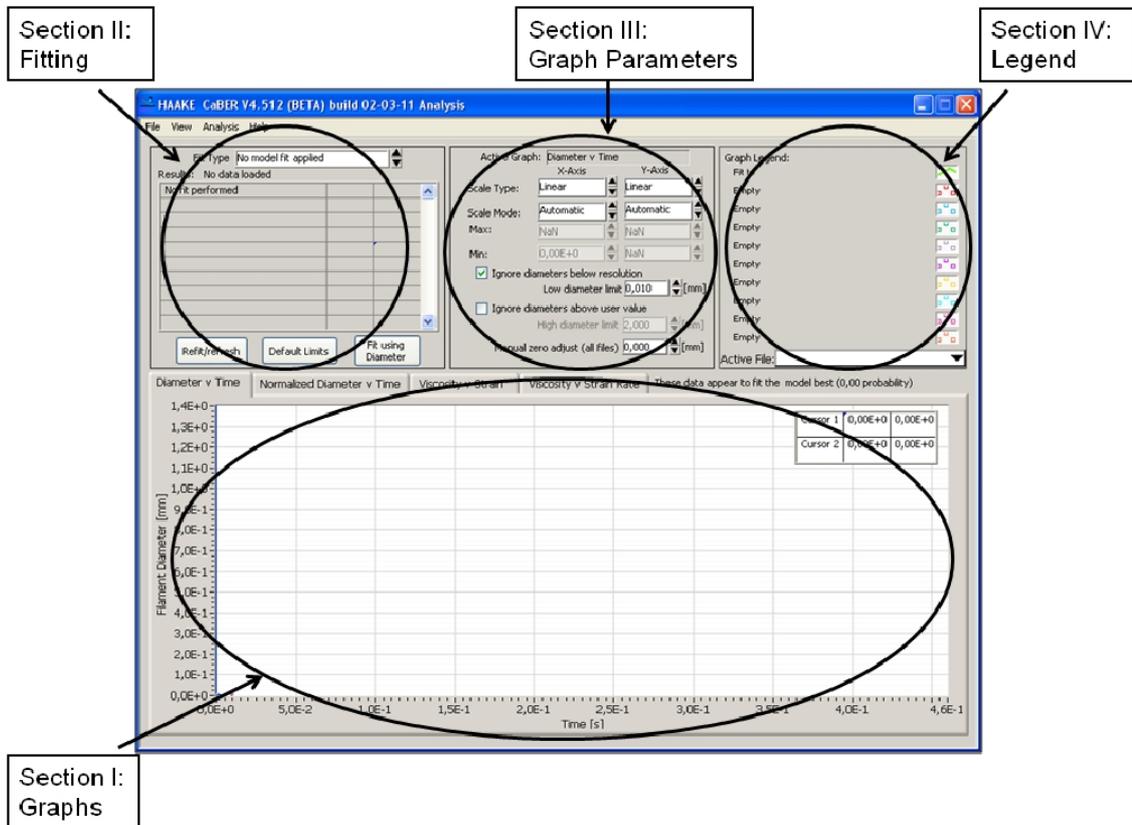
Fig. 38: Analysis package main screen

12.2 The graph section

The main section along the bottom of the screen is the data view graph. Note that there are three tabs on this section, allowing the user to access three different views of the data. The first view shows the raw data for all of the loaded files. The second tab option allows viewing of the normalized data. Note that this data is normalized to an initial diameter that depends on the model used. The final tab shows the data in the form of an apparent extensional viscosity versus strain plot. Note that the currently active file is always the first file on the right hand legend. If you wish to switch active files, you must select a new file on the menu.

12.3 The legend section

The legend for the graphs is shown in the top right of the analysis window. Below the legend there is a drop down list which is used to select the active data file.

12.4 The graph parameter section

The graph parameters are displayed in the top center section. It allows the user to change the axis scaling of each axis in each of the four possible graphs separately.

Note that the software adjusts most parameters dynamically but will only recalculate the model fit after the "Refit" button has been slected. For large data files, or complicated behaviors, this process may not be instantaneous. If the user hits a button and gets a "wait" response, please be patient while the software completes the previous request. The background model fitting resamples the data and uses a non-linear fit to obtain the best model agreement. It also recasts the data to ease the processing load.

12.5 The model parameter and fitting section

The top of this section, placed in the upper left of the display, allows the user the choice of the fitting model. The model is only fitted between the limits defined by the user in the graph. To designate this limits the cursors can be moved manually using the mouse cursor. Finally, if the use default limits button is pressed the software defines the position of these cursors automatically based on the surface tension value and the last data point. The model parameters derived by the software are displayed in the upper left section. Certain parameters will not be visible if they are not relevant for the chosen model fit, other parameters are common to all fits, including surface tension and time–to–breakup. The calculated break–up time is the break–up time as defined by the model fit. This value does not always correspond to the experimental breakup time which is defined by the last data point of the chosen data set. The parameters "shear viscosity" and surface tension can be changed in the options part of the analysis menu placed in the upper menu bar which will be explained later. The user always has to use the "Refit/refresh" button to refresh the display or to force a recalculation. In this section the user also can return to the default limits by pressing the corresponding button and either shear viscosity or filament diameter can be chosen as the relevant fitting parameter.

12.6 The menu bar

An overview of the menu bar which contains the file, the view, the analysis and the help dialogs is displayed in Fig. 39.
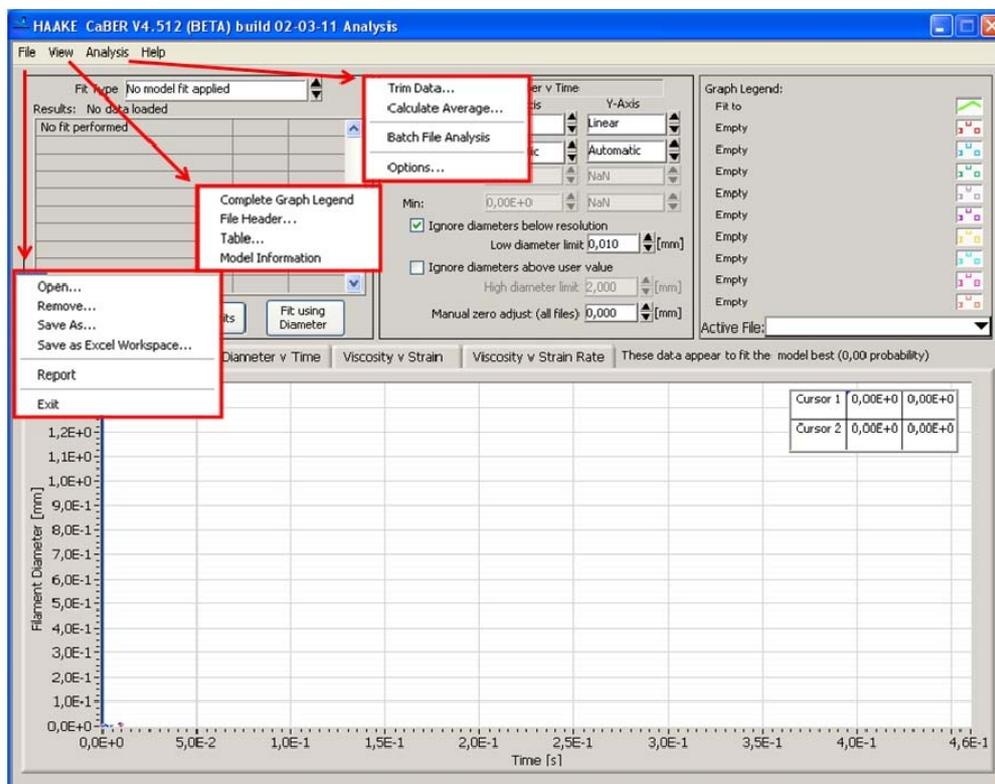
Fig. 39: The menu bar

12.6.1 Open

By choosing this menu point one or more measurement data sets can be selected and loaded from a folder. Note that the latest file is always placed at the top of the file list and therefore becomes the "active" file.

12.6.2 Remove

The data files which should be removed can be selected and the files will be removed from the graphs list and also from the graph display.

12.6.3 Save As

After finishing the work with a data set it can be saved to folder.

12.6.4 Save as Excel Workspace

The data set can also be saved as Excel file by selecting this option from the file menu.

12.6.5 Report

This button generates an HTML based report of the model fit, the returned parameters and the three graphs. The default name is the same as the HAAKE CaBER 1 text file. Note that this HTML file has a link to the HAAKE CaBER 1 text file

49

and saves the graph images in the same directory. Consequently if you wish to move or copy the report to another location you must move all of these files together.

12.6.6 Exit

Leave the program. The user will be asked if they wish to save the data file. This is only the active file. The normalized diameter values and the apparent extensional viscosity data will be saved in this file.

12.6.7 The view menu

In this menu the user can change the graph legend. Also additional about the selected data set will be provided.

12.6.8 Complete Graph Legend

After activating this submenu a complete graph list of all loaded data files will appear. Left click on the symbols of a graph will open a small window where all properties of the graph can be changed.

12.6.9 File Header

Allows the user to view the text header of the saved file. This header contains all experimental parameters (including calibrations).

12.6.10 View Table

Shows a table which includes the measurement data time and diameter and also the from the measurement data derived values for strain, strain rate and apparent elongational viscosity.

12.6.11 Model information

Provides additional information about the results derived from the applied flow model.

12.6.12 Analysis

In this menu some additional setups for analyzing the data set can be made.

12.6.13 Trim Data

In some cases the automatic file handling can leave redundant data. This option allows the user to delete redundant data. Note that this only affects the data file in memory therefore to preserve these changes the user will have to resave the data file.

12.6.14 Calculate Average

The measurement data and the derived values will be averaged over a certain set of data points which can be defined in the options submenu below.

12.6.15 Batch File Analysis

If the user has generated a file using the batch process option this section allows some simple multiple file analyses to be performed.

12.6.16 Options

This submenu provides different calculation and plotting options which can be changed.

12.6.17 Calculation options

In this menu the user can select the viscosity prefactor by selecting the model which should be used to fit the Newtonian data curve. Additional information will be provided be moving the mouse the arrows which will be used to choose the model. The user can also select the reference for normalizing the diameter. Additionally the adjustable prefactor of the selected model can be activated and the data can be reduced by adjust the parameters for averaging the data points.

Below the calculation options the fluid properties shear viscosity and density which are necessary for further calculations can be changed.

The user can change some additional plot settings by changing the plotting options which are self explaining and won't be discussed in detail in this manual.

12.6.18 Help

If this menu is selected the information about the current software version will appear.

13. Operating the instruments

13.1 Introduction

Performing an extensional rheology experiment using the HAAKE CaBER is extremely easy, not much more complex than the "thumb-and-forefinger" test one would use to determine how "stringy" a fluid is. However, to reliably and reproducibly measure the extensional rheology of a fluid takes some care. The operation of the software during a test has already been described above. Here we will discuss more the tips and tricks available to the user to optimize their experiments.

13.2 Sample loading

The very best way to load a sample is through the hole provided in the top plate. This allows consistent loading and protects the sample as much as possible from the environment. However for (high) viscous fluids this may be difficult. An alternative approach is to pipette the fluid in from the side filling from the top plate. As more fluid is pipetted in it collects as a suspended bead, wetting to the edges of the top plate until a large enough mass is present to force it to bridge the gap.

Try to ensure that the gap between the plates is fully filled, but that the plate sides are completely dry. The theory assumes that the fluid is pinned at the plate edges, with a no–slip boundary condition, and that there is no flow over the edges of the plates. Although in fact the HAAKE CaBER can be remarkably tolerant of these influences, for consistency they are best avoided. See Fig. 40.

If possible, always allow the sample to relax at least as long as the break-up time. This is particularly important for highly elastic fluids where the general rule of thumb is five times the relaxation time.

Ensure there are no bubbles in the sample and, if possible, it is totally homogeneous. Remember that the experimental length scale becomes extremely small as the diameter thins to breakup, so any particles on the order of 10 mm result in the assumed continuum fluid is no longer true. See Fig. 40.

Good sample

Overflowing sample

Bubbles in Sample

Underfilled Sample
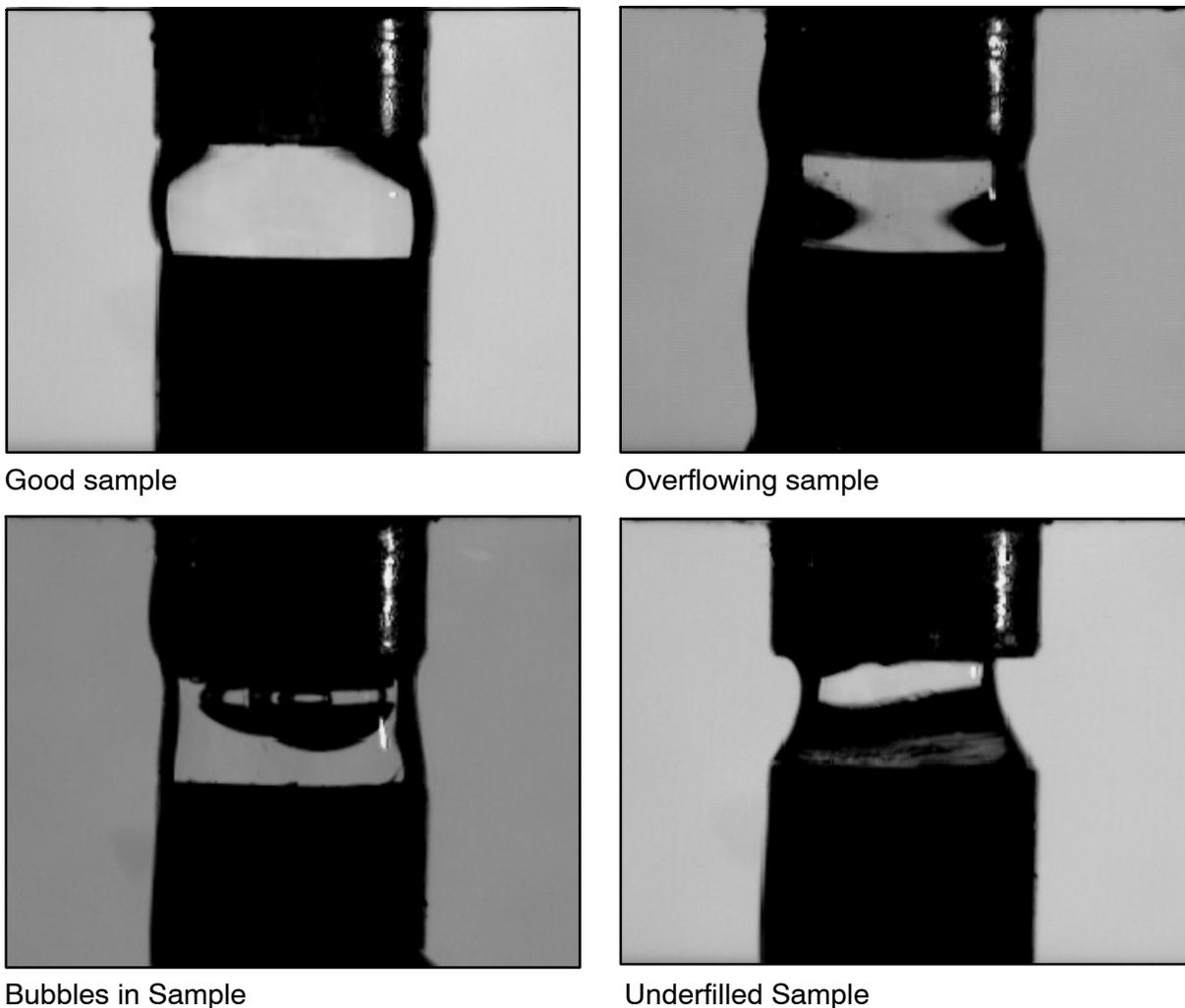
Fig. 40: Examples of sample loading

Remember that as the filament thins the surface area relative to the volume increases inversely with the radius. Therefore the filament can be very susceptible to surface evaporation.

In addition new surface is being generated very rapidly initially during testing and it is possible that surface active species do not have time to fully equilibrate. This will result in a time-varying surface tension which is difficult to account for in the HAAKE CaBER 1 experiments.

As with any rheometer it is worth taking time to refine the loading technique to maximize the value of the data generated by the HAAKE CaBER 1.

13.3 How to perform a measurement:

[1] Turn on the instrument and start the HAAKE CaBER 1 Software.

[2] Check the geometrical parameters and adjust the final gap if necessary. Please note that after every change of the final gap the geometry has to be redefined (Configuration -> Define Geometry)

[3] Choose the test modus in the Experiments menu, the upper plate will drive into the initial position.

[4] The very best way to load the sample is through the hole provided in the top plate by using an inject (V = 2 ml) and a needle (d = 0.8 mm). This allows consistent flowing and protects the sample as much as possible from the environment. However for (high) viscous fluids this may be difficult.
An alternative approach is to inject the fluid by placing the needle on the top plate. As more fluid is injected in it collects as a suspended bead, wetting the edges of the top plate until a large enough mass is present to force it to bridge the gap.
Try to ensure that the gap between the plates is completly filled, but that the plate sides are completely dry. You can achieve a good sample filling by slowly turning the upper measuring system while slowly injecting the fluid onto the upper plate. Make sure that the sample is free of air bubbles and that the sample is totally homogeneous.
Always allow the sample to relax at least as long as the break–up time.
Rember that the experimental lengh scale becomes extremely ...no further changes

[5] Insert the operator– sample and additional notes into the designated text fields and start the experiment by pressing the Test – button. The plates will be separated und the time evolution of the midpoint diameter will be measured by the IR – laser.

[6] After performing the experiment clean the device for the next sample.

13.4 Adjusting the final gap

By adjusting the bottom plate vertically using the micrometer screw you can adjust the final gap that is applied by the linear motor.
The maximum distance is 40 mm. For fluid samples the used final distance is normally in the range of 10 to 20 mm.

To adjust the final gap to x mm proceed as follows:

1. In the HAAKE CaBER 1 control software go to the Check rheometer output window. The micrometer signal in the output window should be 0.0 mm.

2. Turn the micrometer screw upwards until the micrometer signal doesn't change anymore, now turn the micrometer slowly downwards until the signal starts to change significantly and proportional with the movement of the screw.

3. The lower plate is now at the upper edge of the laser–micrometer beam which is 1 mm thick.

4. Now turn the micrometer screw downwards by x/2+0.5 mm to set the final gap.

5. Run the Define geometry routine.

The exact final gap will now be determined by the software. In most cases it will not exactly be the value x because the upper edge of the laser-micrometer beam can not determined exactly using the method above.

13.5 Test philosophy

Because of hardware limitations there is a fundamental choice to be made between fast instrument response and the ability to track the evolution of the filament. Normally one would like to be able to see the evolution of the filament in real time as it thins, however this is only practical for filaments with lifetimes of greater than approximately 0.5 s (roughly relating to shear viscosities of around 1 Pa.s).
This is due to the finite "foreground" sample rate limitation of around 100 Hz. For faster breaking filaments, the user can select any sample rate up to 100 kHz for any time period (with a limit of approximately 32.000 points) to allow the capture of very fast breakup kinetics.

For this "fast" test, all of the post-collection processing steps outlined above are recommended to reduce data-set sizes.

Note of course that although the hardware can capture this quantity of data, the processing time required is usually prohibitive and unnecessary since the rate of diameter change is rarely large compared to the total breakup time.

13.6 Transparent and opaque samples

A common question asked in relation to the HAAKE CaBER 1 is how the instrument handles varying refractive index between samples. It is intuitively obvious that the amount of light reaching the detector (and thus the apparent diameter seen) must depend strongly on the transparency of the fluid used. If the HAAKE CaBER 1 were measuring large filaments this would, of course, be a serious problem.

However in practice it turns out that for the configuration used here the problem is insignificant at diameters of less than approximately 1 mm. Serendipitously it is also in this range that the filaments diameter is most governed by the fluids extensional properties. Fig. 41 supports this point. This effect occurs because although light shines through the transparent samples, refraction at the highly curved interface bends this light away from the detector. Consequently the detector perceives shadow for either transparent or opaque samples. Obviously the light that passes through the center of the filament does reach the detector but this is only a small proportion of the total.
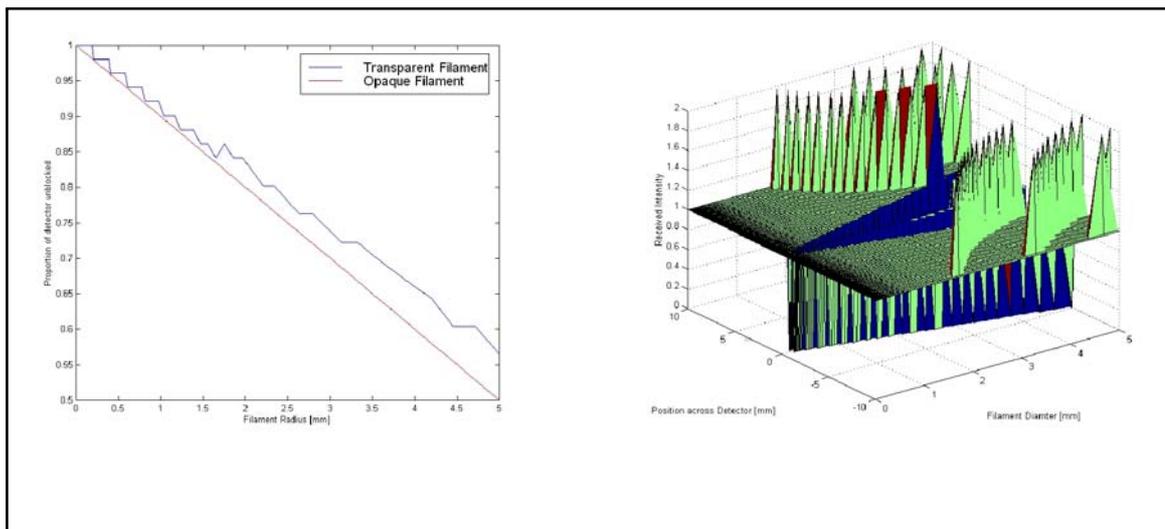
Fig. 41: Simulation comparison of transparent to opaque samples. Perceived diameter (left) and received intensity (right).

13.7 Gravity and shear flow

It is recommended to use an initial plate gap of 3 mm when using 6 mm diameter plates, thus giving an aspect ratio of 1 (L/R). This figure is chosen because of the mechanics of the stretching. If the initial gap is too large then the initial column of fluid takes on an hour-glass shape due to the influence of gravity. This shape imposes a precondition on the fluid that distorts the final filament, and thus the measured properties. On the other hand, if the initial gap is too small then there is a strong shear flow in the initial stretch (squeeze flow dominates) which also preconditions the fluid in the flow. Neither of these effects are desirable, but both can be minimized by careful choice of the initial plate height.

Gravity also plays a part in the breakup of the filament after the mechanical stretch is completed. Once the unstable bridge is formed, there is normally a significant volume of fluid at the top plate. The forces acting on the midpoint of the filament during these initial moments are surface tension driven, (which results in a flow away from the mid-point) and gravity driven (resulting in a flow from the top to the bottom plate). It is only after the filament gets thin and curved enough (and hence the capillary forces high enough) that gravity can no longer be deemed to be playing a part. In practice, for Newtonian fluids this transition point is at diameters of around 700 μm, depending on the surface tension. This is also discussed in the chapter on the Analysis software.

13.8 High speed response

Two issues are encountered when attempting to study fast breakup kinetics. The first is rheology-driven, and the second is instrument related. In the first case for low viscosities (< 100 mPa.s) the fluid can be prone to breakup into droplets. This phenomena can be reduced by careful adjustment of the initial conditions but is often the limiting condition for very low viscosity filament breakups. Thermo Fisher Scientific is currently working on developing improved experimental protocols to allow access to these fascinating materials. This effect is often seen in the HAAKE CaBER through the beam. An example of such a data set can be seen in Fig. 25. Note however that below a certain critical capillary number (*i.e.* ratio of viscosity to surface tension) these materials will always undergo non–ideal breakup due to inertial effects in the fluids. These influences are still being studied in the rheology literature.

The second issue is related to the micrometer used in the instrument. At very fast sampling rates an exponential decay will often be observed that persists for a few tens of milliseconds. This is not a rheological phenomena, and is instead an instrument artifact. An example of such an experiment is also shown in Fig. 42.
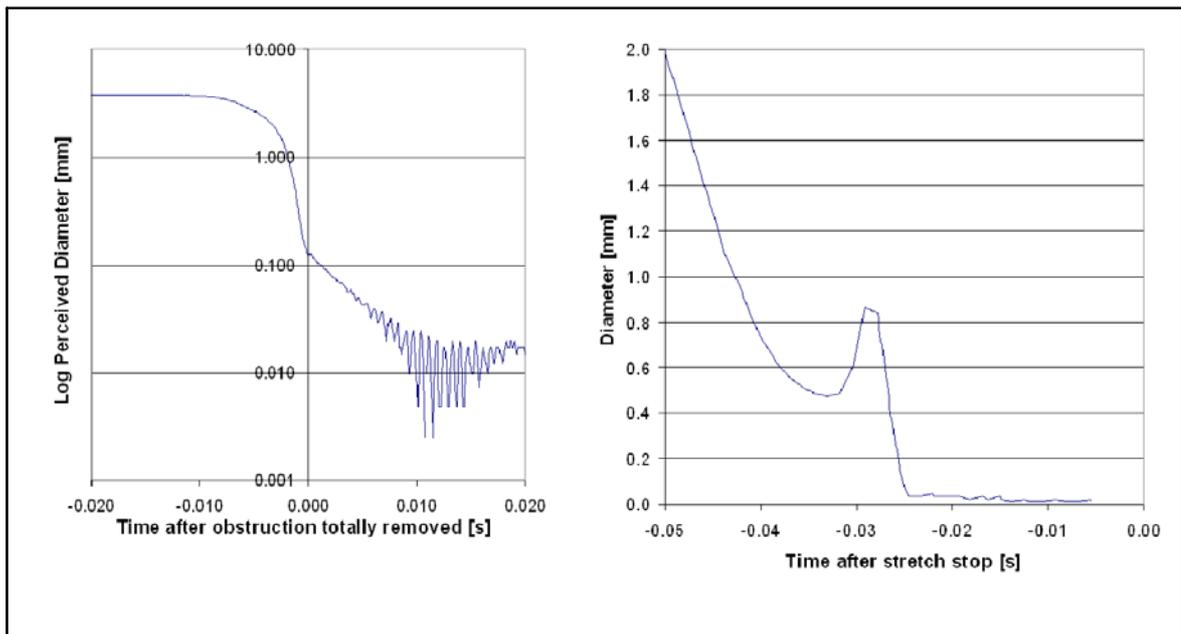
Fig. 42: Inertial droplet breakup; and b) short time response of detector.

13.9 Performing CaBER – measurements with normal force option

13.9.1 Operation

Hardware settings

It is necessary to perform some basic settings on the Kistler 5015A charge meter, which are described below. It is highly recommended to read at least the chapters 4.4, 4.5.1, 5.3, 5.4 and 5.5 of the Kistler manual in order to get familiar with the operation of the Kistler 5015A charge meter.

1. Sensor input and sensor sensitivity

– The sensor input must be set to Charge, see the Kistler manual chapter 4.5.1 on page 31.
– The sensor Sensitivity must be set to the value which is given on the plastic box in which the Kistler sensor is delivered (see Fig. 38), see the Kistler manual chapter 4.5.1 on page 32 and chapter 5.4 on page 38.
Please note that each Kistler sensor has its individual sensitivity value.

– The sensor sensitivity Unit must be set to the unit which is given on the plastic box in which the Kistler sensor is delivered (see Fig. 43), see the Kistler manual chapter 4.5.1 on page 32 and chapter 5.4 on page 38.

Fig. 43: Sensor Sensitivity value on sensor storage box

2. Voltage output scaling

The voltage output setting Output FS must be set to ±10 V, see the Kistler manual chapter 4.5.1, page 32 and chapter 5.5 on page 40.

3. Measuring range

The force Measuring range must be set according to the maximum forces occurring in a measurement. See the Kistler manual chapter 4.5.1 on page 31 and chapter 5.3 on page 36 on how to set the measuring range. As a good starting point for measurements on a unknown sample the range can be set to 0.5 N in order not to overload the normal force sensor.

4. Remote mode

On the Kistler charge meter a "measurement cycle" must be started before a measurement can be performed. This can be done manually by pressing the Measure button on the front panel or remotely controlled by another device using the Remote connection on the back of the instrument. By using the trigger cable (see above) the CaBER 1 software will start a "measurement cycle" just before the CaBER measurements starts.

In order for the remote triggering to work the Control mode of the Kistler charge meter has to be set to Remote. Chapter 5.7 (and chapter 4.4.2) of the Kistler manual describes how to do this.
For manually testing the sensor it sometimes may be usefull to switch the Control mode back to Local.

Software settings

1. Activation of the force signal measurement

In order for the CaBER 1 software to be able to measure the normal force signal the Force Sensor has to be selected for the Accessory Channel in the Define General Options dialog of the Configuration menu, see Fig. 44a and Fig. 44b below.
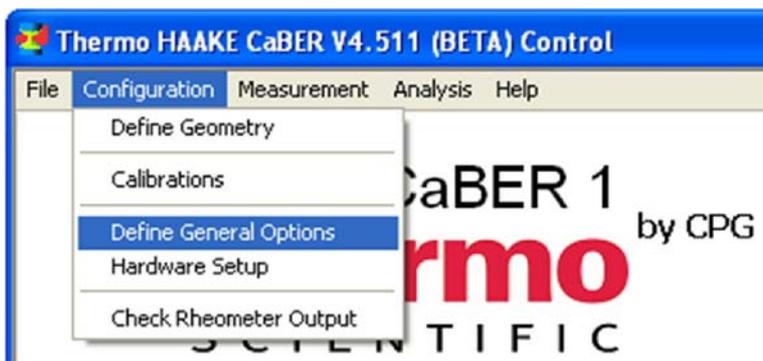
Fig. 44a: Configuration menu screenshot
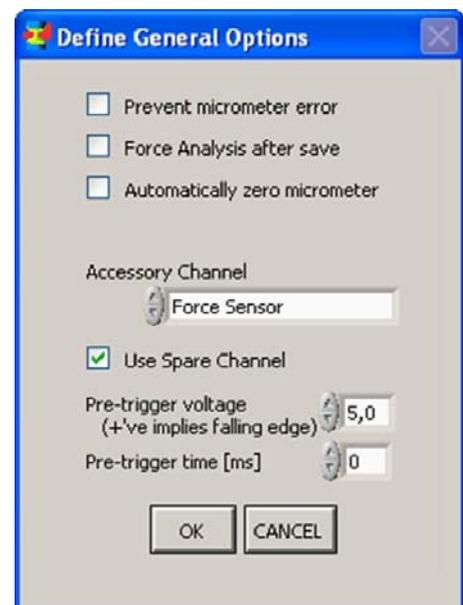
Fig. 44b: Define General Options dialog screenshot

2. Setting the sampling mode

The CaBER 1 software can only measure the normal force signal when the option High–Speed Measurement is to selected for the Sampling mode in the Define Measurement Options dialog of the Measurement menu, see Fig. 45a and Fig. 45b below.
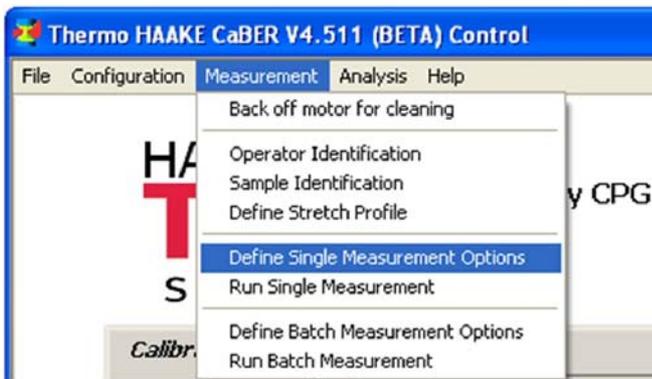
Fig. 45a: Measurement menu screenshot
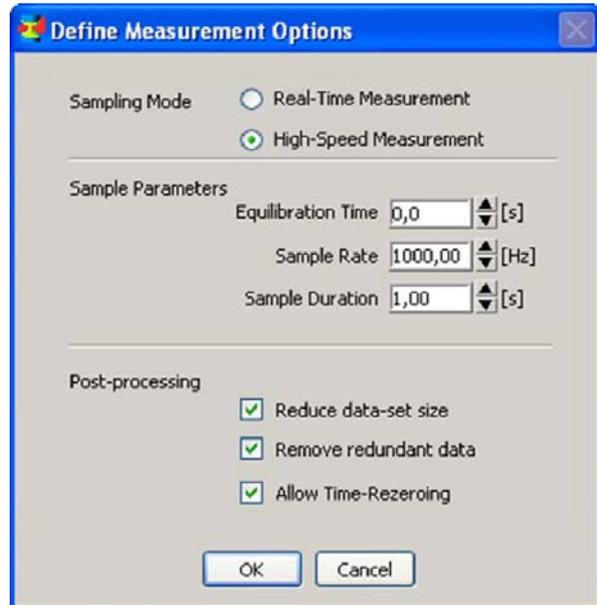
Fig. 45b: Define Measurement Options dialog screenshot

3. Force signal scaling factor

The scaling factor for the Kistler normal signal must be defined manually in the caber_config.cfg file. This file is stored in the C:\Documents and Settings\All Users\Application Data\Thermo\caber directory.
Since this file is a plain ASCII file the file can be opened and edited with a simple editor like Notepad (which is part of every Windows installation.

In this caber_config.cfg file the following lines must be added in the Caber calibration section, that is below the line [Caber calibration]:
Force grad = 1.0
Force offset = 0.0

The values entered for Force grad and Force offset are use in the following equation in the CaBER1 software to calculate the normal force value Fn in Newton from the Kistler normal force signal Voltage in Volt.

Fn = Force grad * Voltage + Force offset

The value for Force offset should be set to 0.0. The value for Force grad depends on the setting of the measurement range on the Kistler instrument. The force grad value corresponds to the overall scaling of the voltage output of the Kistler 5015A charge meter and conveniently be read from the lower line of the display. See Fig. 46, the red circled text is the scaling of the voltage output. Typical values for use with the CaBER normal force option are 5.0E–2 N/V and smaller.
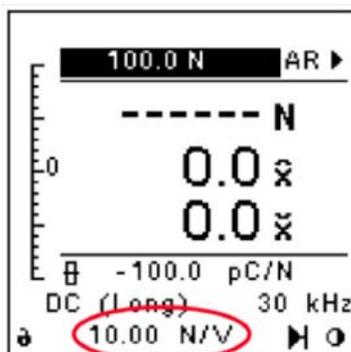
Fig. 46: Kistler 5015A display

☞ Please note: In order for changes to the Force grad value in the caber_config.cfg file to have effect, the CaBER control software has to be exited and restarted!

Running a measurement

Running a CaBER measurement with the normal force option installed and active is not different from running a normal CaBER measurement.

Please note that:

1. The High–Speed measurement sampling mode should be used only (see above).

2. The Measure LED above the LCD display on the front panel of the Kistler should light up during the actual measurement. In case it does not please check the correct mounting of the trigger cable.

3. The normal force signal data is displayed in the graph during the measurement.

4. The normal force signal data is (currently) NOT available in the CaBER 1 Analysis software at all (i.e. not in a graph, not in a table and also not in the exported Excel files).

5. The normal force signal data is (currently) only available in the CaBER *.cbr data files. Since these files are stored in plain ASCII format, these files are easily imported in Excel or any data viewing software.

When the measured normal force sensor is much lower as the value defined by the measuring range (see above), the measuring range value of the Kistler 5015A charge meter should be lowered. See the Kistler manual chapter 4.5.1 on page 31 and chapter 5.3 on page 36 on how to set the measuring range. Do not forget to edit the Force grad value in the caber_config.cfg file accordingly and to restart the CaBER 1 software after that!

13.10 Calibrations

Two components on the CaBER device that should be checked or calibrated on a monthly basis are the diameter outputs from the micrometer and the temperatures from the thermal probes. Although these have been shown to be very stable over long periods we recommend performing the basic calibration reasonably often. These calibration values are saved in the file "calc.txt" in the CaBER directory. In addition the diameter calibration used is saved in each data file generated by the instrument.

13.10.1 Diameter calibration procedure

To perform the diameter calibration procedure we recommend to use the CaBER Calibration Kit (Order-No. 222-1691) which was especially designed to calibrate the HAAKE CaBER 1 laser-micrometer. With this Calibration Kit the calibration is easily performed within 5 to 10 minutes.

13.10.2 Maintenance of the calibration tools

The pins (hardened material) of the calibration tools are manufactured high-precisely.
Advisable safety measures against rust: oil the pins with suitable means (e.g. Balistrol or machine oil).

13.10.3 Contents of the Calibration Kit

The CaBER Calibration Kit contains 7 "wires" (0.03 mm to 3 mm) mounted in special clamps. The wires of each Calibration Kit are individually calibrated, the diameters of the wires are documented on a test certificate that comes with the CaBER Calibration Kit, see for an example below.

Prüfbescheinigung/Test certificate		Thermo Electron (Karlsruhe) GmbH	

Produkt / Product	Typ /Type	Bestell-Nr./ Order-No
CaBER Kalibrier-Kit / Calibration Kit	Kalibriereinsatz Calibration insert	222-1691

Sach-Nr: Part-No.	Serien-Nr: Serial No.	Durchmesser/Diameter D Ø in mm soll / specified	Durchmesser/Diameter Ø in mm ist / actual
006-0272	04/078	0,03	0,030
006-0273	04/079	0,06	0,058
006-0274	04/080	0,12	0,119
006-0275	04/081	0,25	0,249
006-0269	04/082	0,50	0,499
006-0270	04/083	1,00	1,000
006-0271	04/084	3,00	3,000
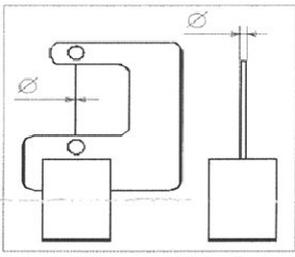
Messgerät, Rückführbarkeit / Measuring instrument, traceability:

Längenmessgerät SIP 305 m; Messunsicherheit: U95 = 0,2 µm

Length measuring instrument SIP 305 m; uncertainty of measurement: U95 = 0,2 µm

Endmaßsatz Koba, KDK Nr. 0431 [nationaler Anschluß 952 DKD-K-09001 01-02]

set of gauge blocks Koba, KDK Nr. 0431 [national traceability 952 DKD-K-09001 01-02]

Raumtemperatur: 20°C ± 1°C temperature: 20°C ± 1°C

Kraus	15.03.04	Qualitätswesen / Quality control
		Thermo Electron (Karlsruhe) GmbH * Dieselstr. 4 * 76227 Karlsruhe
Unterschrift/Signature	Datum / Date	Abteilung/Department

Formular 089-2002

13.10.4 Mechanical setup

1. Remove the lower measuring plate from the lower shaft.

2. Move the upper plate to its "home" position by using the button on the CaBER software main panel.

3. Lower the lower shaft by means of turning the (mechanical) micrometer screw until the shaft height is around 13 mm.

4. Place one of the wire-clamps of the Calibration Kit on the lower shaft so that the clamp rests on the lower part of the HAAKE CaBER 1 measurement chamber (see the photo) in order to check the height of the lower shaft.

5. Adjust the height of the shaft if the clamp does not rest on the lower part of the measurement chamber.

13.10.5 Performing the calibration

After the instrument has been powered-on for at least 15 minutes close the software and start it again (this is necessary for the software to find the stable zero point of the laser-micrometer).

The actual calibration is performed using the calibration routines that are built into the CaBER software.

1. From the drop-down menu Configuration (in the main menu) select Calibrations.
   The main screen of the program will change to the CaBER Calibration Status window.

2 From the drop down menu Recalibrate (in the CaBER Calibration Status window) select Diameter.
A new window Calibrate diameter will appear.

3 A message box saying Make sure that nothing is blocking the micrometer indicates that the actual calibration process can be started.
At this point make sure that there is nothing in the light path of the laser micrometer, since the first calibration point is at a diameter of D = 0.0 mm.

4 Enter 0.0 in the Next wire diameter [mm] edit field (above the graph) and click on the Find calibration button.
The software will measure the micrometer voltage signal for a few seconds and plot a data point on the graph when it is finished.

5 Place the clamp with the 0.03 mm calibration wire on top of the lower shaft of the HAAKE CaBER 1.
Enter the actual diameter of that wire (see the test certificate that came with the calibration kit) in the Next wire diameter [mm] edit field (above the graph) and click on the Find calibration button.

The software will measure the micrometer voltage signal for a few seconds and plot a new data point on the graph when it is finished.

Remove the clamp with the 0.03 mm calibration wire.

6 Repeat step 5 with the other 6 wires in order of increasing diameters. Do not forget to enter the correct wire diameter each time.
Work carefully, when you make a mistake, you have start all over again!

7 After all 7 wires have been measured the graph should look like this :
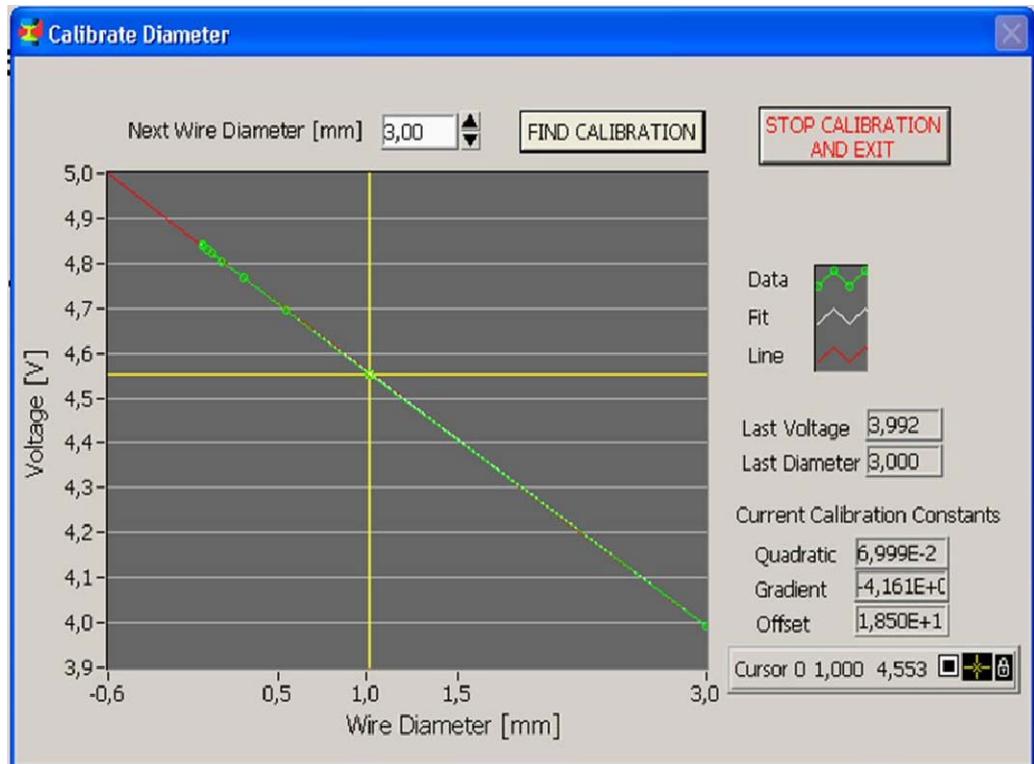
Click on the Stop calibration and exit button.
A small message box asking Accept calibration ? will popup, select Yes here.

8 We are now back on the CaBER Calibration Status window. The software displays the result of the calibration under Diameter on the main screen. These results are automatically saved in the Caber_config.cfg file.

9 The calibration results should also be saved in a separate calibration file: Select Calibration file from the File drop down menu (in the CaBER Calibration Status window). Then select the Export command to save the calibration data in a *.cal file.
The Import command in the same menu can be used to import the calibration results stored in *.cal file back into the software, i.e. into the caber_config.cfg file.

10 From the File menu select the command Return to main screen to return to the mains screen of the software. The diameter reading in the lower right corner should now read 0.0 ± 0.01 mm (with nothing in the light path of the laser micrometer!).

11 The calibration is now finished!

13.11 CaBER 1 temperature calibration

13.11.1 Preparations for the calibration

1. For the calibration the following is needed:
   – Fluid bath circulator.
   – Two hoses to connect the circulator to the CaBER 1.
   – Digital thermometer with a very thin flat probe.

2. Install the HAAKE CaBER 1 and switch the instrument and the computer on.

3. Connect the circulator to the CaBER 1 using the hoses.

4. Switch the circulator on.

13.11.2 Temperature calibration

The actual calibration is performed using the calibration routines that are built into the CaBER 1 software.

12   From the drop–down menu Configuration (in the main menu) select Calibrations.
The main screen of the program will change to the CaBER Calibration Status window.

13   From the drop down menu Recalibrate (in the CaBER Calibration Status window) select Temperature.
A new window Calibrate temperature will appear.

14   Set the the circulator to a temperature at least 5 °C lower than the lowest temperature you want to measure your samples at with the CaBER 1.

Wait for the Current Sensor Temperature value (this is the temperature measured by the temperature sensor build into the CaBER 1) to reach a constant value. This can take a while.

15   Measure the temperature between the upper and lower plate in the CaBER 1 with the digital thermometer.

Enter the measured value in the Input Real Temperature [°C] edit field (above the graph) and click on the Measure Temperature button.

16   The software will now measure the temperature using the temperature sensor build into the CaBER 1 and plot a data point on the graph when it is finished.
The software will also enter two values in the Look–up Table in the lower right corner of the Calibrate Temper-

ature window. The upper value in this table is the value measured by the temperature sensor build into the Ca-BER 1. the lower value is the offset value needed to calculate the correct temperature (as measured with the digital thermometer).

17 Now set the circulator to the next temperature you want to calibrate at.
Wait for the Current Sensor Temperature value (this is the temperature measured by the temperature sensor build into the CaBER 1) to reach a constant value. This can take a while.

18 Repeat steps 4 to 6 with increasing temperatures until you reach a temperature that is at least 5 °C higher than the highest temperature you want to measure your samples at with the CaBER 1. For normal accuracy use temperature steps of 10.0 °C.
Do not forget to enter the correct temperature as measured with the digital thermometer each time.
Work carefully, when you make a mistake, you have start all over again!

19 After you have measured at a few temperatures the graph should look like this :
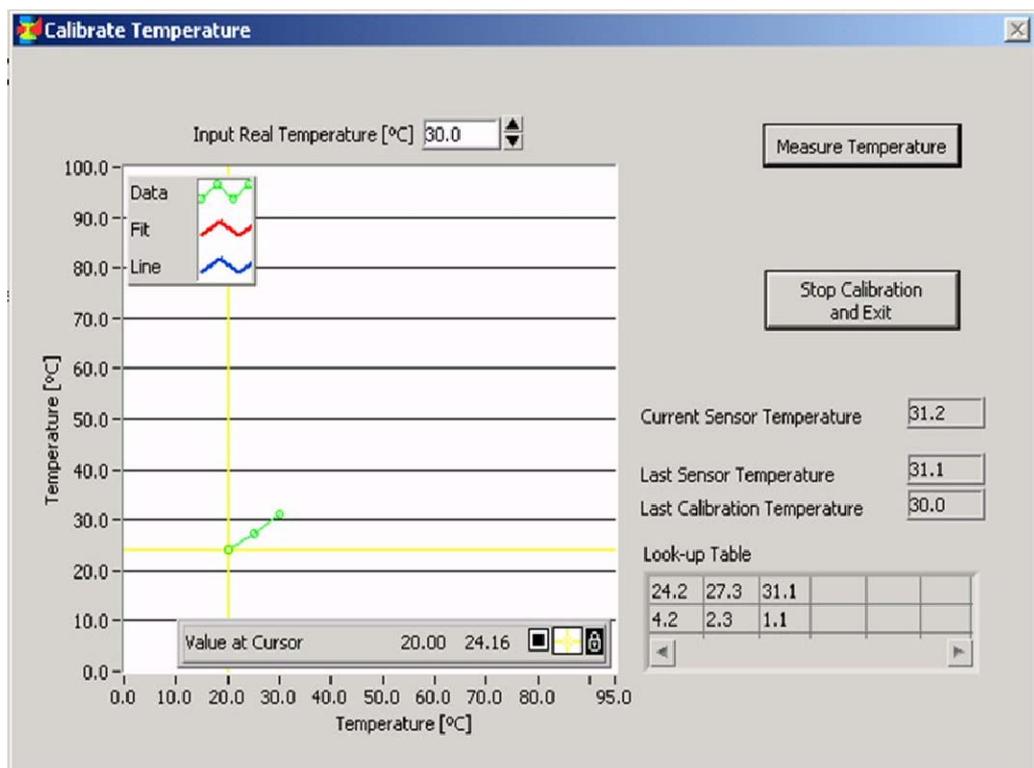
Click on the Stop Calibration and Exit button.
A small message box asking Accept calibration ? will popup, select Yes here.

20 We are now back on the CaBER Calibration Status window. The software displays the result of the calibration in the Temperature offset table in the lower right corner of the main screen. These results are automatically saved in the Caber_config.cfg file.
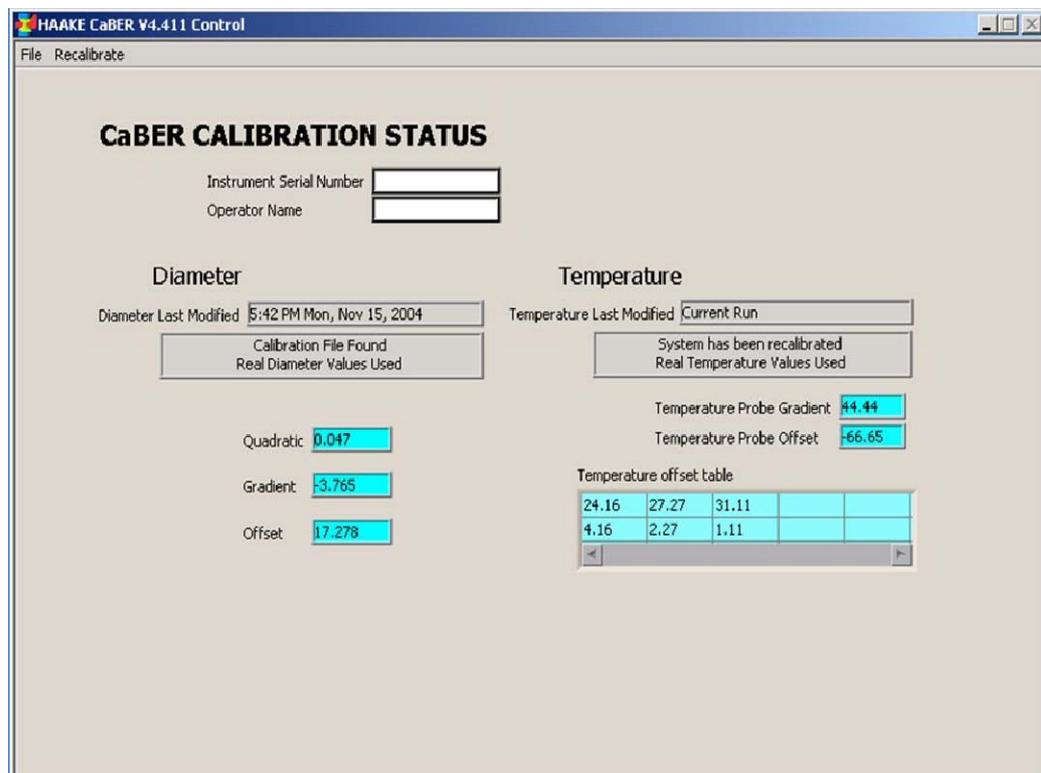
21 The calibration results should also be saved in a separate calibration file: Select Calibration file from the File drop down menu (in the CaBER Calibration Status window). Then select the Export command to save the calibration data in a *.cal file.

The Import command in the same menu can be used to import the calibration results stored in *.cal file back into the software, i.e. into the caber_config.cfg file.

22 From the File menu select the command Return to main screen to return to the main screen of the software. The temperature reading in the lower right corner should now correspond to the temperature measured with the digital thermometer.

23 The calibration is now finished!

14.   The theory of extensional rheometry

As described briefly in the introduction, a number of authors have published work using variations on the filament breakup rheometer where one monitors the breakup dynamics of a fluid thread (Bazilevskii et al., 1990, Bazilevskii et al., 1997, Stelter et al., 2000, Liang and Mackley, 1994, McKinley and Tripathi, 2000). Academic research in this area has focused principally on model fluids (e.g. constant viscosity Newtonian oils and dilute homopolymer polymer solutions) that can be described by simple constitutive models such as the Maxwell model. This restriction on the choice of test materials facilitates extraction of material properties since the dynamics of the capillary thinning are simple enough to be analyzed quantitatively; however, it clearly constrains the commercial viability of such a device. In the following sections we shall outline a little of the background to the HAAKE CaBER 1 experiment. The user is encouraged to refer to the references for more details (see Appendix V).

14.1 Newtonian fluids

Many of the details pertaining to this discussion are available in a recent paper by McKinley and Tripathi (McKinley and Tripathi, 2000). In the filament rheometer the sample is constrained axially between two smooth coaxial disks of radius R_o and forms a liquid bridge configuration that is nominally cylindrical in shape (see Fig. 26(a)). The precise shape is determined by satisfying the Young-Laplace equation and is a function of the aspect ratio $\Lambda = 2L_o / D_o$, the volume of fluid contained between the plates and the gravitational body force and surface tension (Szabo, 1997, Slobozhanin and Perales, 1993).

In the HAAKE CaBER 1, the plates separate rapidly over a short distance. In this respect the device functions as the extensional equivalent of the step–strain test in conventional rheometry. A liquid bridge is therefore generated which has a distinctly necked but axisymmetric configuration (*cf.* Fig. 47(b) and 47(c)). Once this new *unstable* necked configuration has been established (see Fig. 47(c,d and e)), the midpoint diameter $D_{mid}(t)$ is monitored as a function of time using the laser micrometer (or some other approach). If inertial effects and viscous stresses in the external fluid are negligible then this necked configuration is symmetric about the midplane; however, if these effects become significant, then more complex shapes can arise (Gaudet et al., 1996, Berg et al., 1994).
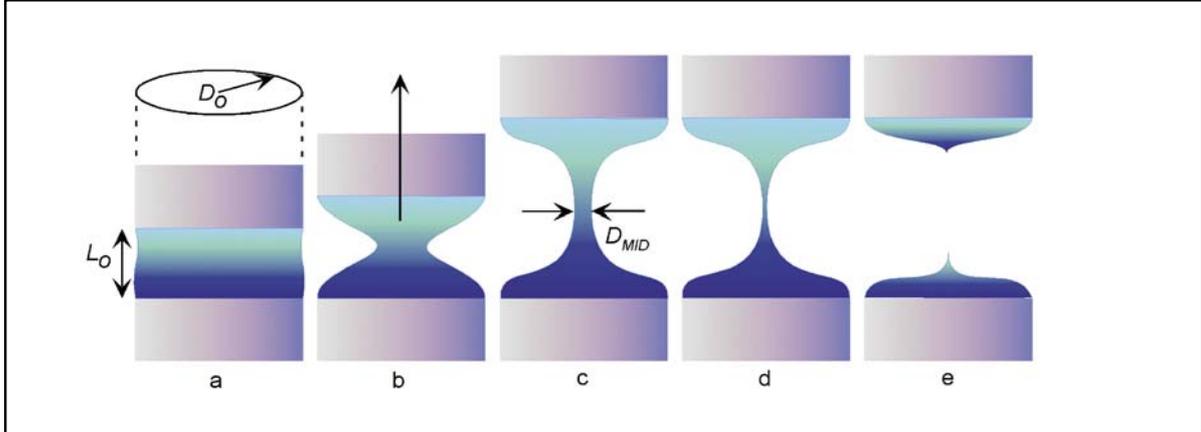
Fig. 47: Five schematic images of the axially symmetric plates in (a) closed, (b) during stretching, (c) immediately after stretching ceased, (d) during capillary drainage, (e) after breakup configurations.

The dynamics of the drainage of the thin fluid column and the ultimate rupture of the liquid bridge into two or more droplets are governed by the viscous and elastic properties of the fluid. A complete understanding of this nonlinear dynamical process has only been developed over the past decade (Eggers, 1997). Detailed theoretical analysis and numerical simulations using a slender body theory shows that the time evolution of the midpoint diameter of a Newtonian fluid can always be described by the following equation (McKinley and Tripathi, 2000):

$$D_{mid}(t) = D_1 - \frac{(2X(t)-1)}{3}\frac{\sigma}{\eta_s}t \qquad (1)$$

where the non–local effects arising from axial variations in the shape of the filament are encoded in the net tensile force *F(t)* acting on the thread. These effects appear in equation (1) through the dimensionless function

$$X(t) \equiv F(t)/\pi\sigma D_{mid}(t). \qquad (2)$$

Correct determination of the function *X(t)* allows the calculation of the ratio σ/η_s.

If an experiment is performed and the midpoint diameter $D_{mid}(t)$ is indeed found to decrease linearly in time, then the value of the ratio σ/η_s determined from regression of the data will depend critically on what value of *X* it is appropriate to use in the analysis. The experimental results analyzed elsewhere (Bazilevskii et al., 1990, Liang and Mackley, 1994, Kolte and Szabo, 1999) have all assumed implicitly that *X*= 1.

However it has recently been pointed out (McKinley and Tripathi, 2000) that in fact, for typical experimental conditions, the most appropriate value is actually $X = 0.7127$ in accordance with the self-similar solution of Papageorgiou (Papageorgiou, 1995). To date all published experimental work has assumed that $X=1$. The published estimates for the extensional viscosity have all been incorrectly scaled by a factor of approximately 2. Fig. 27 shows preliminary data on a semi–logarithmic scale fitted using $X=0.7127$. For a simple Newtonian model fluid the HAAKE CaBER allows the extraction of a characteristic ratio, σ/η_s , that fully defines the fluid.

The units for this ratio, σ/η_s , are [m/s]. We therefore identify this parameter as the characteristic "capillary velocity" of the fluid, which describes the rate of thinning in a viscous fluid.

14.2 Power-law fluids

A very similar analysis to the Newtonian case can be performed for power law fluids where the stress is proportional to some power of the strain rate. Here the characteristic ratio returned is σ/K describing the thinning of the filament where the factor K is the "viscosity index" of the model

$$\tau = K \cdot \dot{\gamma}^{\,n} \tag{3}$$

In the limit of $n=1$ the power-law solution reduces to that of the Newtonian case but the ratio σ/K has units of [m.s^{-n}] and the parameter K has units of [Pa.s^{n}] which means that they are not directly equivalent to the "capillary velocity" and viscosity of the Newtonian case. Clearly the constitutive model assumed here is a simple shear thinning model where there is no zero-shear (or high-shear) viscosity plateau as would be more usually anticipated in reality, and as is described by the Carreau model (Macosko, 1994 #19). On the face of it this is a weakness of the model assumed for this fit, but in general the low shear regions are the large diameters (where the filament is dominated by gravitational effects etc) so extensional information is dominated by the startup conditions.

14.3 Elastic fluids

The above discussion is applicable only for constant viscosity Newtonian fluids, and in the restricted case of a power law fluid. In viscoelastic solutions and melts theoretical work (Bousfield et al., 1986, Renardy, 1994, Bazilevskii et al., 1990) and subsequent numerical analysis (Entov and Hinch, 1997) shows that following a rapid initial viscous–dominated phase, there is an intermediate time–scale in which the dynamics of the filament drainage are governed by a balance between surface tension and elasticity, rather than fluid viscosity. In this regime, the filament radius decreases exponentially as equation (4):

$$D_{mid}(t) = D_0 \left(\frac{GD_0}{\sigma} \right)^{1/3} e^{-t/3\lambda_c} \tag{4}$$

where λ_c is a characteristic relaxation time governing the capillary breakup and G is the elastic modulus of the filament. For a semi–dilute PIB/PB Boger fluid (a model elastic fluid) it has been shown (Kolte and Szabo, 1999) that λ_c is closely related to the longest relaxation time, λ_1 of the fluid. These authors also elegantly show how the effects of a radial inhomogeneity in the stretch can account for the remaining discrepancy between λ_c and λ_1. It has recently been demonstrated that by choosing an appropriate aspect ratio it is, in fact, possible to use measurements of the exponential decrease in radius with time to quantitatively determine: (i) the longest (Rouse/Zimm) relaxation time for ideal elastic fluids (consisting of dilute solutions of monodisperse polystyrene); and (ii) the approximate scaling of the steady-state elongational viscosity with molecular weight (Anna and McKinley, 2001). Fig. 28 shows such a fit. For a model elastic fluid a simple exponential fit will yield the material relaxation time.
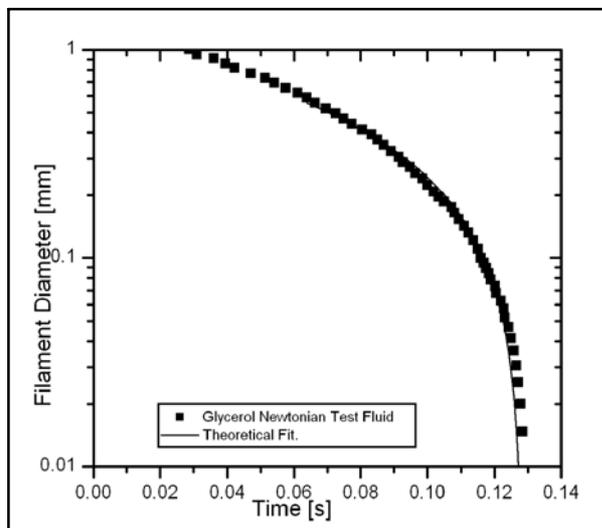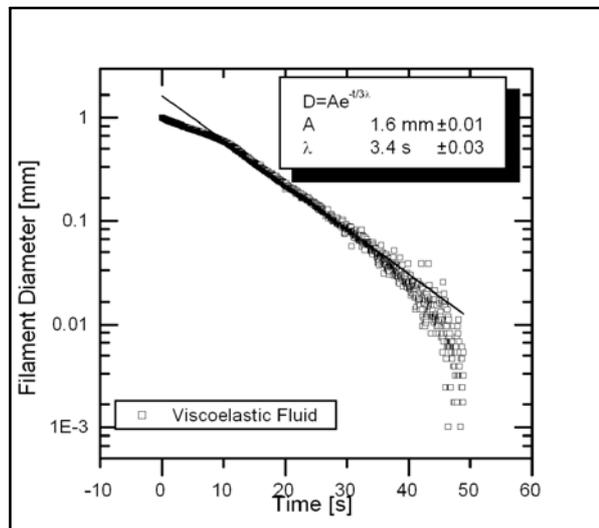
Fig. 48

Fig. 49

Fig. 48: Glycerol fluid at room temperature with Newtonian fit overlaid. The fit yields a capillary velocity of s⁄h =16.5m/s. Through the known surface tension s = 64.8 mN/m this gives a viscosity of 1.07 Pa.s (compared to an accepted literature value of η =1.03 Pa.s).

Fig. 49: Semi-logarithmic plot of necking in a viscoelastic fluid fitted using a decaying exponential. l=3.4s gives the relaxation time of the fluid.

14.4 Complex fluids

In the two previous subsections, it is clear that given the correct theoretical foundation, the proposed instrument can extract values of material properties such as the capillary velocity, σ/η_s, the relaxation time, λ_c, and modulus, G, of simple test fluids. However, such idealized fluids are rarely encountered in industrial applications. In reality, commercial material formulations can often display a number of complex responses. These complexities originate from the presence of components such as volatile solvent, phase separating materials, associative materials, chemical changes (such as curing) and yield stresses. Consequently many materials have viscometric properties that also evolve with time. The HAAKE CaBER 1 is ideal for the study/quantification of these materials because of the short experimental times required for each. A brief list of some example materials are given below:

- Absorption:
  e.g. Glycerol is a highly hygroscopic material that absorbs water from the atmosphere and hence shows a marked viscosity change with time.

- Setting:
  e.g. adhesives/wet spinning/melt spinning. Materials that set, or dry etc are common in many fields. For example see Figure 3.

- Curing and gelation:
  e.g. epoxies. Materials that are self associating or are chemically curing also change with time.

14.5 Generic model

In order to fully encompass the complex phenomena listed above, a generic model is required that encompasses time–varying Newtonian and non–Newtonian effects. A balance of forces on the fluid filament governs the evolution in the mid-point profile of the liquid bridge. For a slender fluid filament this can be written compactly in the following form (Renardy, 1995):

$$\underbrace{3\eta_s\left(-\frac{2}{D_m}\frac{dD_m}{dt}\right)}_{\text{Viscous Stress}} = 3\eta_s\dot{\varepsilon} = \underbrace{\frac{4F_z}{\pi D_m^2}}_{\text{Tensile Stress}} - \underbrace{\left[\tau_{zz}-\tau_{rr}\right]}_{\substack{\text{Elastic/Non-}\\\text{Newtonian Stress}}} - \underbrace{\frac{2\sigma}{D_m}}_{\substack{\text{Capillary}\\\text{Pressure}}} \tag{5}$$

where s is the surface tension of the fluid, F_z is the tensile force acting on the column ends, η_s is the Newtonian viscosity of the solvent, and ($\tau_{zz} - \tau_{rr}$) represents the non-Newtonian contribution to the total normal stress difference in the fluid. This last term is model-dependent and the resulting solution to the differential equation depends on how the polymeric contribution to the stress varies with the rate of deformation. Solutions to this evolution equation have been found for a number of models (Bazilevskii et al., 1997, Entov and Hinch, 1997, McKinley and Tripathi, 2000) and are summarized in Fig. 50. More complicated multi-mode models predict a spectrum of relaxation times, which is more realistic for real polymeric fluids. These models will usually capture the initial more rapid decay in radius during relaxation. This initial rapid drop is usually attributed to the relaxation of shorter time scales, after which point the longer time scales yield a more gradual radial decay. The HAAKE CaBER 1 software currently handles all of these models.

Constitutive Model	Form of Solution	Parameters found from regression to data
Newtonian, $\tau = \eta_s \dot{\gamma}$	$D_{mid}(t) = 0.142\left(\sigma/\eta_s\right)\left(t_c - t\right)$	$t_c,\ \sigma/\eta_s$
Power-Law Fluids $\tau = K\dot{\gamma}^n$	$D_{mid}(t) = 2^{1-n}\left(0.142\right)\left(\sigma/K\right)\left(t_c - t\right)^n$	$t_c,\ \sigma/K,\ n$
Upper Convected Maxwell $\tau + \lambda\tau_{(1)} = \eta_p\dot{\gamma}$	$D_{mid}(t) = D_0\left(GD_0/\sigma\right)^{1/3}\exp\left(-t/3\lambda_c\right)$	$t_c,\ \lambda_c,\ G/\sigma$
"Advanced Model" Gives correct limiting expressions for Newtonian/Oldroyd/Bingham Fluids	$D_{mid}(t) = \left[D_1\left\{1 - \left(\frac{D_1 G}{2\sigma}\right)^{1/3}\right\} + \frac{\tau_y}{2\rho g}\right]$ $-\left[\frac{0.42\sigma}{12\eta_s}t\right] + \left[D_1\left(\frac{D_1 G}{2\sigma}\right)^{1/3}e^{-t/3\lambda}\right]$	$t_c, \tau_y, G/\sigma, \sigma/\eta_s$

Fig. 50: Evolution of the midpoint diameter in a fluid thread undergoing capillary-driven breakup

Time to breakup

In each case, one of the parameters determined is the critical time to breakup, t_c. This is not strictly a material property but depends on the properties of the fluid, the flow geometry and the surrounding medium (e.g. the relative humidity, or partial pressure, of solvent). However, this parameter is of utility as a possible way of quantifying concepts such as 'stringiness', 'stranding' and the general processability of complex materials such as foodstuffs, shampoos and other consumer products. Although this is related to the capillary velocity, σ/η_s, it also holds information about the non-Newtonian behavior (and evolution) of the material.

The apparent extensional viscosity

The principal experimental results obtained from the HAAKE CaBER are the evolution of the midpoint diameter of fluid samples with time. This evolution is driven by the capillary pressure and resisted by the extensional stress in the fluid. The measurements can thus also be represented in terms of an *apparent extensional viscosity*, which we define by

$$\bar{\eta}_{app}(\varepsilon) = \left[\tau_{zz} - \tau_{rr}\right]_{total}/\dot{\varepsilon}(t), \tag{6}$$

while the Hencky strain is defined as

$$\varepsilon = \ln\left(D_{mid}(t)/D_1\right). \tag{7}$$

By rearranging equation 5 it can be shown that the apparent extensional viscosity is given by

$$\bar{\eta}_{app}(\varepsilon) = \frac{2\sigma/D_{mid}(t)}{\left\{-\dfrac{2}{D_{mid}}\dfrac{dD_{mid}}{dt}\right\}} = \frac{\sigma}{\dfrac{dD_{mid}}{dt}} \qquad (8)$$

where the instantaneous rate of stretching of the midpoint fluid element, ε (t), is given by the term in the braces. If the surface tension, s, of the test fluid is known from independent measurements, then HAAKE CaBER data can be replotted in the form of an extensional viscosity (see Fig. 51).

14.6 Association time

The theory of the capillary breakup of associative fluids is still under development but given their importance in industry it is worth mentioning these systems briefly. There is some evidence that complex self-associating materials can relax through two different mechanisms, which result in two distinct time scales. One of these time scales will be the molecular relaxation modes commonly probed using rheology where the distorted molecule takes time to recover its equilibrium conformation. However, for materials that self-associate, there will be a characteristic "association time" of the fluid that probes the time it takes the material to build an internal structure. During the stretching process, if the material is stretched slower than this time, the fluid will have time to adapt to the high strain field and will behave as expected for a non-associating material. If the fluid is stretched faster than this time, the fluid will not have time to rearrange and will hence behave differently in the HAAKE CaBER test.
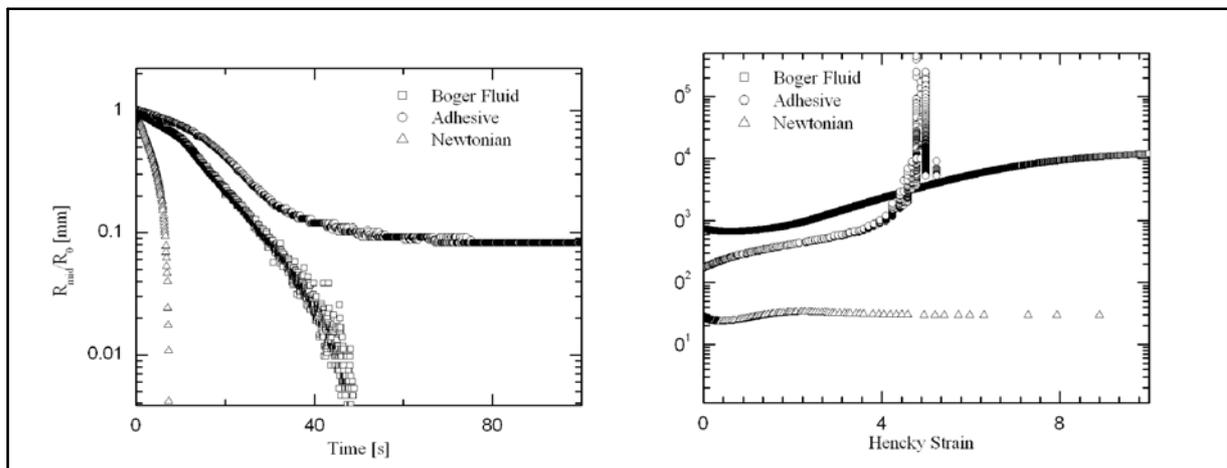

Fig. 51: Three sets of data showing normalized filament radius versus time. The data are for three fluids, simple Newtonian and a model elastic fluid, and a Pressure Sensitive Adhesive. Left hand plot shows diameter versus time. Right hand plot shows an apparent extensional viscosity versus strain.

15. Technical specifications

15.1 Instrument specifications

Hencky strain	up to $\varepsilon_0 = 10$
Strain rate range: Imposed strain rate	$0.0 < \varepsilon_0 < 300 \ \text{s}^{-1}$
Fluid strain rate:	$10^{-5} < \varepsilon_0 < 10 \ \text{s}^{-1}$
Shear Viscosity range	$10\text{--}10^6$ mPas
Plate diameter	4, 6 and 8 mm
Linear motor resolution	0.02 mm

Laser micrometer
    Type
    Resolution
    Wavelength
    Power
	
	Class 1
	0.01 mm
	780 nm
	1.7 mW

System response time	10 ms
Temperature range	0 to 80°C

Ambient conditions
    Temperature
    Relative humidity
	
	15 to 40 °C
	35 to 85%

15.2 Data file format

HAAKE CaBER 1 Thermo Fisher Scientific

Filename: example file.cbr

Experiment Start

Time and Date: 10:41:22 AM Friday, August 23, 2002

File Save
Time and Date: 10:44:07 AM Friday, August 23, 2002

Temperature Probe 1°C: 25.5
Temperature Probe 2°C: 25.3

Force Sensor: Not Connected

Drive System Used: Linear Drive

Strike Time [ms]: 50.000000E+0

Operator: Laboratory Technician

Company: Thermo Fisher Scientific

Comment: An example Newtonian fluid

Sample Name: s2000

Sample Number: 1

Notes:
Sample End height   12.070312E+0
Sample start height 3.007812E+0
Sample Diameter   6.000000E+0
Hencky Strain   1.389536E+0

Conversion to actual size:
Second coeff   381.999999E–3
grad   –6.886000E+0
offset   24.600000E+0

No Fit Performed

SURFACE TENSION   40.000000

START OF DATA

Experimental Time	Experimental Data
–5.000E–2	4.432E+0
–4.900E–2	4.432E+0
–4.700E–2	4.432E+0
–4.600E–2	4.422E+0
–4.499E–2	4.422E+0
–4.399E–2	4.422E+0
–4.299E–2	4.432E+0

15.3 Minimum computer requirements

– 500 MHz Pentium based PC

– Screen resolution 1024 x 768 pixels (256 colors)

– One free PCI slot (Desktop PC) or
– one free PCCard slot (Notebooks)

– One free serial port (RS232)

– Mouse

– 40 MB free disk space

– Operating system : Windows XP

15.4 References

Anna, S., McKinley, GH. (2001). Elasto-capillary thinning and breakup of model elastic liquids. Journal of Rheology, 45(1), 115.

Arnolds, O., Buggisch, H., Sachsenheimer, D., Willenbacher, N. (2010). Capillary breakup extensional rheometry (CaBER) on semi-dilute and concentrated polyethylene oxide (PEO) solutions. Rheologica Acta, 49, 1207-1217.

Bazilevskii, A. V., Entov, V. M. and Rozhkov, A. N. (1990) In Proceedings of the 3rd European Rheology Conference(Ed, Oliver, D. r.) Elsevier, pp. 41-43.

Bazilevskii AV., Entov VM. et al. (1997). Failure of Polymer Solution Filaments. Polym. Sci., A(39), 316-324.

Bazilevskii AV., Entov VM. et al. (2001). Failure of an Oldroyd Liquid Bridge as a Method for Testing the Rheological Properties of polymer Solutions. Polym. Sci., A 43, 1161-1172.

Berg, S., Kröger, R. and Rath, H. (1994) "Measurement of Extensional Viscosity by Stretching Large Liquid Bridges in Microgravity" Journal of Non-Newtonian Fluid Mechanics, 55, 307-319.

Bhardwaj, A., Miller, E. et al. (2007). Filament stretching and capillary breakup extensional rheometry measurements of viscoelastic wormlike micelle solutions. J. Rheol., 51, 693-719.

Bousfield, D., Keunings, R., Marrucci, G. and Denn, M. (1986) "Nonlinear Analysis of the Surface-Tension Driven Breakup of Viscoelastic Fluid Filaments" Journal of Non-Newtonian Fluid Mechanics, 21, 79-97.

Christanti Y, Walker, LM. (2001a). Effect of fluid relaxation time of dilute polymer solutions on jet breakup due to a forced disturbance. Journal of Rheology, 46, 733-748. Christanti, Y., Walker, LM. (2001b). Surface tension driven jet break up of strain-hardening polymer solutions. Science, 100, 9-26.

Clasen, C., Plog, JP, Kulicke, WM., Owens, M., Macosko, C.W, Scriven, LE., et al. (2006). How dilute are dilute solutions in extensional flows? Journal of Rheology, 50(6), 849.

Eggers, J. (1997) "Nonlinear Dynamics and Breakup of Free-Surface Flows" Review of Modern Physics, 69, 865-929.

Entov, V. M. and Hinch, E. J. (1997) "Effect of a spectrum of relaxation times on the capillary thinning of a filament of elastic liquid." J. Non-Newtonian Fluid Mech., 72, 31-53. Gaudet, S., McKinley, G. and Stone, H. (1996) "Extensional Deformation of Newtonian Liquid Bridges" Physics of Fluids, 8, 2568-2579.

Kheirandish S., Guybaidullin, I., Wohlleben, W., Willenbacher, N. (2008a). Shear and elongational flow behavior of acrylic thickener solutions. Rheologica Acta, 47(9), 999-1013.

Kheirandish, S., Gubaydullin, I., Willenbacher, N. (2008b). Shear and elongational flow behavior of acrylic thickener solutions. Part II: effect of gel content. Rheologica Acta, 48(4), 397-407.

Klein, C., Naue, I., Wilhelm, M., Brummer, R., Nijman, J., Co, A., et al. (2009). Addition of the force measurement capability to a commercially available extensional rheometer (CaBER). Soft Materials, 7(4), 242-257.

Kolte, M. and Szabo, P. (1999) "Capillary Thinning of Polymeric Filaments" Journal of Rheology, 43, 609-626.

Liang, R. F. and Mackley, M. R. (1994) "Rheological Characterization of the Time and Strain Dependence for Polyisobutylene Solutions" Journal of Non-Newtonian Fluid Mechanics, 52, 387-405.

McKinley, G H., Tripathi, A. (2000). How to extract the Newtonian viscosity from capillary breakup measurements in a filament rheometer. Journal of Rheology, 44(3), 653.

Niedzwiedz, K., Arnolds, O., Willenbacher, N, & Brummer, R. (2009). How to Characterize Yield Stress Fluids with Capillary Breakup Extensional Rheometry (CaBER)? Appl. Rheol., 19(4), 41969.

Niedzwiedz, K., Buggisch H., Willenbacher N. (2010). Extensional rheology of concentrated emulsions as probed by capillary breakup elongational rheometry (CaBER). Rheol. Acta, 49(11-12), 1103-1116.

Oliveira, MSN, McKinley, G H. (2005). Iterated stretching and multiple beads-on-a-string phenomena in dilute solutions of highly extensible flexible polymers. Physics of Fluids, 17, 71704.

Oliveira, M., Yeh, R., Mckinley, GH. (2006). Iterated stretching, extensional rheology and formation of beads-on-a-string structures in polymer solutions. Journal of Non-Newtonian Fluid Mechanics, 137(1-3), 137-148.

Papageorgiou, D. (1995) "On the Breakup of Viscous Liquid Threads" Physics of Fluids, 7, 1529–1544.

Renardy, M. (1994) "Some Comments on the Surface-Tension Driven Breakup (or the lack of it) of Viscoelastic Jets" Journal of Non–Newtonian Fluid Mechanics, 51, 97–107.

Renardy, M. (1995) "Numerical Study of the Asymptotic Evolution and Breakup of Newtonian and Viscoelastic Jets" Journal of Non–Newtonian Fluid Mechanics, 59, 267–282.

Slobozhanin, L. and Perales, J. (1993) "Stability of Liquid Bridges between Equal Disks in an Axial Gravity Field" Physics of Fluids A, 5, 1305–1314.

Sridhar T. (1990). An overview of the project M1. Journal of Non–Newtonian Fluid Mechanics, 35(2), 85–92.

Stelter, M., Brenn, G., Yerin, A., Singh, R. and Durst, F. (2000) "Validation and application of a novel elongational device for polymer solutions" Journal of Rheology, 44, 595–616.

Szabo, P. (1997) "Transient Filament Stretching Rheometer I: Force Balance Analysis" Rheologica Acta, 36, 277–284.

Tirtaatmadja, V. (1993). A filament stretching device for measurement of extensional viscosity. Journal of Rheology, 37(6), 1081.

Tirtaatmadja, V., McKinley, GH., Cooper–White, J.J. (2006). Drop formation and breakup of low viscosity elastic fluids: Effects of molecular weight and concentration. Physics of Fluids, 18(4), 043101.

Wang, M., Hsieh, A.J., Rutledge, G.C. (2005). Electrospinning of poly (MMA–co–MAA) copolymers and their layered silicate nanocomposites for improved thermal propeties. Polymer, 46(10), 3407–3418.

ThermoFisher
SCIENTIFIC

**Servicekontakte zu Thermo Fisher Scientific
Your Service Contacts at Thermo Fisher Scientific
Thermo Fisher Scientific: Contact Service**

Bitte wenden Sie sich bei Servicefragen an uns, unsere Partnerfirmen oder an die für Sie zuständige Generalvertretung, die Ihnen das Gerät geliefert hat.

Please get in contact with us or the authorized agent who supplied you with the unit if you have any services questions.

Veuillez vous adresser pour tout renseignement à votre fournisseur ou directement à :

Thermo Fisher Scientific

International / Germany	**USA**
Thermo Fisher Scientific	Thermo Fisher Scientific
Dieselstrasse 4	2 Radcliff Rd.
D-76227 Karlsruhe, Germany	Tewksbury, MA 01876
Tel. +49(0)721 4094-444	Tel. 603 436 9444
Fax +49(0)721 4094-300	Fax 603 436 8411
Hotline +49(0)18 05 04 22 53	
info.mc.de@thermofisher.com	info.mc.us@thermofisher.com

UK	**China**
Thermo Fisher Scientific	Thermo Fisher Scientific
Ion Path, Road 3	Building 6, No. 27,
Winsford	Xin Jinqiao Rd., Shanghai 201206
Cheshire, CW7 3GA	
Tel. +44(0) 1606548100	Tel. +86(21) 68654588
Fax: +44(0) 1606548101	Fax: +86(21) 64457830
info.mc.uk@thermofisher.com	info.mc.china@thermofisher.com

Japan	**India**
Thermo Fisher Scientific K.K.	Thermo Fisher Scientific
C-2F, 3-9, Moriya-cho, Kanagwa-KU	403-404, Delphi-B Wing,
Yokohama, 221-022	Hiranandani Business Park,
	Powai, Andheri (E),
	Mumbai - 400076
Tel. +81-45-453-9170	Tel. +91 22 6680 3000
Fax: +81-45-453-9082	Fax: +91 22 6680 3001
info.mc.japan@thermofisher.com	info.mc.in@thermofisher.com

www.thermoscientific.com/mc