



Measure what is measurable and make measurable that which is not.

Galileo Galilei (1564-1642)

Instruction Manual and Safety Information

**DSA 5000 M** 

Density and Sound Velocity Meter

instrument software version: from 2.98 (Original Instructions)

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See the Reference Guide for a comprehensive description of the instrument.

See the General Software Functions Manual for a comprehensive description of the instrument software and instructions for its use.

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# 1 Safety Instructions

- Read the documentation before using the instrument.
- Follow all hints and instructions in the documentation to ensure the correct use and safe functioning of the instrument.
- The documentation is a part of the product.
  Keep it for the complete working life of the product and make it easily accessible for all persons involved with the product. If you receive any additions or revisions to the documentation from Anton Paar GmbH, these must be treated as part of the documentation.

## 1.1 Liability

- This document does not claim to address all safety issues associated with the use of the instrument and samples. It is your responsibility to establish health and safety practices and to determine the applicability of regulatory limitations.
- Anton Paar GmbH only warrants the proper functioning of the instrument if no modifications are made to mechanics, electronics, or software
- Use the instrument only for the purpose described in the documentation. Anton Paar GmbH is not liable for damages caused by incorrect use of the instrument.
- The results delivered by the instrument depend not only on the correct functioning of the instrument, but also on various other factors. We therefore recommend that you have the results checked (e.g. plausibility tested) by skilled persons before consequential actions are taken based on the results.

### 1.2 Installation and Use

 The installation procedure shall be carried out only by authorized persons who are familiar with the installation instructions.

- Use only accessories, consumables, or spare parts supplied or approved by Anton Paar GmbH.
- Ensure that all operators have been trained beforehand to use the instrument safely and correctly.
- The instrument must not be operated in an industrial environment.
- Ensure that the instrument is sufficiently supervised during operation.
- In the temperature range 0–50 °C (32–122 °F), operate the instrument only in the pressure range 0–8 bar (0–116 psi) absolute pressure.
- In the temperature range 50–100 °C (122–212 °F), operate the instrument only in the pressure range 0–5 bar (0–72.5 psi) absolute pressure.
- When you use a syringe for filling, do not apply a force above 35 N. If you feel a noticeable resistance, a measuring cell or a hose may be blocked.
- In case of damage or malfunction, do not continue operating the instrument. Do not operate
  the instrument under conditions which could
  result in damage to goods or injuries or loss of
  life
- If liquid has been spilled over the instrument, disconnect the instrument from the AC power supply. Clean and dry the housing of the instrument. If you have a suspicion that liquid got into the instrument, have the instrument cleaned and checked for electrical safety by a service technician authorized by Anton Paar GmbH.

### Operation in areas with risk of explosion

 The instrument is **not** explosion-proof and therefore must not be operated in areas with risk of explosion.

### Operation with explosive samples

 The instrument must not be used for the measurement of samples of explosion group IIC (such as carbon disulfide or acetylene).

### General precautions

- Observe and adhere to your national safety regulations regarding the handling of all substances associated with your measurements (e.g. use safety goggles, gloves, respiratory protection, etc.).
- Before a measurement check the wetted parts of the instrument for chemical resistance to the samples and cleaning agents used.
- Take care that the liquids (samples and cleaning agents) or gases that you use are chemically compatible when they come into contact with each other. They must not react exothermally or produce solid particles, which might stick to the inner walls of the measuring cells.
- Before you start a measurement or cleaning procedure, take care that all parts, in particular the measuring cells, the injection adapters, the hoses, and the waste vessel, are properly connected and in good condition.
- Before you start a measurement or cleaning procedure, check the injection adapters for leak tightness.
- Take measures that spilled liquids cannot get into plug connections or venting slots of electrical appliances.
- Connect the measuring system to the AC power supply via a safety switch located at a safe distance from the instruments. In an emergency, turn off the power using this switch instead of the power switch on the instruments.

# Precautions for flammable samples and cleaning agents

- Keep potential sources of ignition, like sparks or open flames, at a safe distance from the instrument.
- Place the instrument on a laboratory bench made of fireproof material, preferably bricks, ceramics, or stoneware.
- Store only the minimum required amount of sample, cleaning agents, and other flammable materials near the instrument.
- Do not spill sample/cleaning agents or leave their containers uncovered. Immediately remove spilled sample/cleaning agents.
- Ensure that the setup location is sufficiently ventilated. The environment of the instrument must be kept free from flammable gases and vapors.
- Provide fire-extinguishing equipment.

### **Transportation**

- Empty the measuring cell and all hoses before you move or lift the instrument.
- To move or lift the instrument, grasp the ledge on top of the instrument at the back with one hand. Place the other hand under the display at the front. There is a hollow for your fingers.
- Carry the instrument in front of you and keep it close to your body.

## 1.3 Service and Repairs

 Service and repair procedures may be carried out only by authorized persons or by Anton Paar GmbH.

## 1.4 Disposal

 Concerning the disposal of the instrument, observe the legal requirements in your country.

## 1.5 Conventions for Safety Messages

The following conventions for safety messages are used in this document:



### **WARNING**

#### Description of risk

Warning indicates a hazardous situation which, if not avoided, **could** result in death or serious injury.



### **CAUTION**

### Description of risk

Caution indicates a hazardous situation which, if not avoided, could result in minor or moderate injury.

### NOTICE

### Description of risk

Notice indicates a situation which, if not avoided, could result in damage to property.

# 2 DSA 5000 M – An Overview

DSA 5000 M features a revolutionary new design of the sound velocity cell and the new U-tube core technology with its patented evaluation of the oscillation period. It provides highly reliable and precise, but also highly robust density and sound velocity results for the same sample under the same measurement conditions.

Your instrument is equipped with the world's most advanced digital density measurement technology. This "Pulsed Excitation Method" and various features supporting your density and sound velocity measurements are described below.

### Inside DSA 5000 M

- State-of-the-art density measurement based on the patented "Pulsed Excitation Method" (AT 516420 (B1)).
- With the additional sound velocity cell, two physical properties of the filled sample can be determined simultaneously with highest precision.
- The oscillation periods of the U-tube and the reference oscillator are measured by optical pickups.
- Two integrated Pt 100 platinum thermometers together with Peltier elements provide an extremely precise thermostatting of the sample.
- ThermoBalance™: The additional reference oscillator provides long-term stability and enables precise measurements over the whole temperature range of the instrument, with only one adjustment at 20 °C.
- Viscosity-related errors are automatically corrected over the full viscosity range by measuring the damping effect caused by the viscous property of a sample. The result is subsequently used for the calculation of the viscosity corrected density value.

### Condition monitoring

 The built-in sensor for the atmospheric pressure enables the automatic calculation of the current air density required for adjustments and checks of the instrument, as the air density is dependent on the atmospheric pressure.

- FillingCheck™: A major source of measuring errors with density and sound velocity meters are gas bubbles in the measuring cell. The instrument automatically detects inhomogeneities and gas bubbles in the whole measuring cell by an advanced analysis of its oscillation pattern. Where necessary, a warning message is generated in real time for every single measurement.
- U-View™: Real-time images by a camera with zoom function enable you to visually inspect the measuring cell.
- The patented "Pulsed Excitation Method" (AT 516420 (B1)) even improves operational safety as the condition of the measuring cell can be monitored in detail.

### User interface

The touchscreen user interface facilitates easy and intuitive operation in routine applications as well as in demanding scientific research work:

- For the most common applications, 5 measuring methods have been predefined. Just select the method suiting your application, or create your own methods.
- Density and sound velocity values are automatically converted into concentration values for a large number of factory-programmed substances. You can add further substances as required by yourself.
- Optionally operate DSA 5000 M via external keyboard, mouse, gesture control, or barcode reader.
- Optionally connect an external monitor or touchscreen (VGA interface).

### Compact and robust design

The instrument is ready for reliable measurements also in demanding environments thanks to the following strengths:

- Compact design
- Sealed housing that withstands shocks, dirt, and spillages
- Robust housing materials: coated aluminum (top and sides), aluminum (base and back), and polystyrene/butadiene (front)

# 2.1 Functional Components

### Front and right side

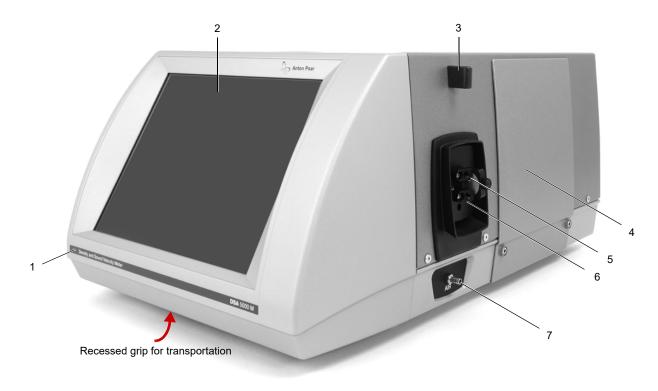


Fig. 1: View of the front and right side of DSA 5000 M

- 1 Power-on LED
- 2 Color PCAP touchscreen
- 3 Syringe holder

- 4 Xsample slot cover plate
- 5 Sample outlet
- 6 Sample inlet
- 7 Air pump outlet

### Left side



Fig. 2: Views of the left side of DSA 5000 M | DSA 5000 M CK

- 1 Blind covers | Inlet and outlet connectors for the cooling kit
- 2 USB 2.0 sockets (type A), 3x
- 3 Protection cover for the USB sockets

### Rear

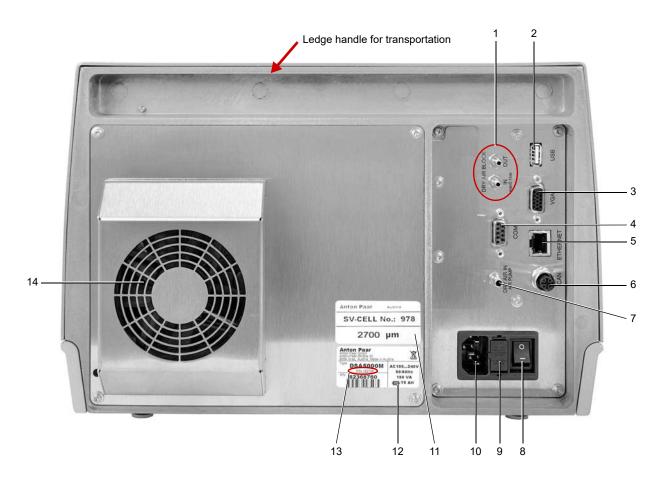


Fig. 3: View of the rear of DSA 5000 M

- 1 "DRY AIR BLOCK" connectors
- 2 USB 2.0 socket (type A)
- 3 VGA connector (DE-15F connector)
- 4 COM / RS-232 serial port (DE-9F connector)
- 5 Ethernet terminal (RJ45 connector)
- 6 CAN interface (for the connection of further measuring modules)
- 7 "DRY AIR IN AIR PUMP" connector

- 8 Power on/off switch
- 9 Fuse holder
- 10 AC power jack
- 11 Factory set path length for the sound measurement
- 12 Type plate with serial number
- 13 Product number (P/N = mat. no.)
- 14 Fan

# 3 Installing the Instrument

To install the instrument, put it on a bench, mount injection adapters and hoses, and connect the instrument to the AC power supply. Define general instrument settings and perform an air/water check to check the validity of the factory adjustment.

For the installation of an Xsample filling module, see the manual of the Xsample.

## 3.1 Installation Requirements

The instrument is designed for operation under typical laboratory bench top conditions.



### WARNING

Using hazardous or flammable chemicals as samples or cleaning liquids could lead to damage of the instrument and cause serious injuries unless special precautions are taken.

• Observe the safety instructions in section 1.

Place the instrument on a stable, flat bench that is free from vibrations and away from vibrating equipment.

To ensure temperature stability and trouble-free measurement, do **not** position your instrument:

- next to a heating facility,
- in a drafty place (e.g. near an air conditioning, ventilation system, or an open window),
- in direct sunlight.

### NOTICE

- Ensure that the power plug and the power switch are always easily accessible so that the instrument can be easily disconnected from the AC power supply at any time.
- A strong built-in cooling fan dissipates heat through the bottom and the rear of the instrument. Ensure that the air flow is not blocked, and provide for a minimum distance of 10 cm (4 in) to walls behind and beside the instrument.
- High humidity or a measuring temperature that is significantly below the ambient temperature may lead to condensation in the measuring cell. Install a drying cartridge to avoid condensation, see the Reference Guide.

# 3.2 Mounting the Injection Adapters

The injection adapter DSA UNF ½" leads to the sound velocity measuring cell and serves as the sample inlet adapter. The pre-assembled connection tube leads the sample from the sound velocity measuring cell to the density measuring cell. The injection adapter UNF ½" leads the sample from the density measuring cell to the waste, see fig. 4.

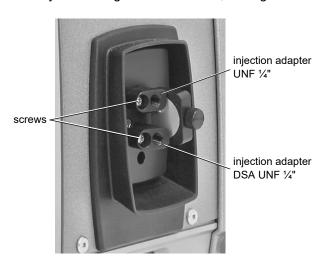


Fig. 4: Injection adapters mounted

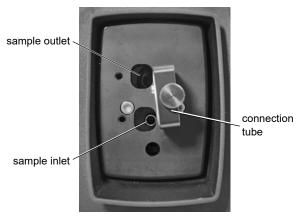


Fig. 5: Filling device on DSA 5000 M

- Take the injection adapter DSA UNF ¼" and the injection adapter UNF ¼" from the accessory kit.
- 2. Pull the plastic transport plugs out of the injection adapters' tips.

**TIP:** Keep the transport plugs for later use. In case of leaks you can widen the tips of the adapters with the transport plugs.

- Carefully insert the injection adapter DSA UNF 1/4" into the sample inlet opening of the adapter holding plate, see fig. 5.
- 4. Push the adapter towards the adapter holding plate with moderate force.
- Insert the screw through the bore hole of the adapter and screw it cautiously into the adapter holding plate until some resistance against further turning can be felt.



### **CAUTION**

If the screw for fastening the adapter is overtightened, the density measuring cell may get damaged. Dangerous liquids leaking from the instrument may cause injuries.

- Tighten the screw until some resistance against further turning can be felt, and then stop to tighten the screw.
  - The gap left between the holding plate and the adapter, where the thread of the screw can be seen, is approx. 3–8 mm (approx. 0.12–0.31 in).
- 6. Insert the injection adapter UNF ½" into the sample outlet opening and fasten it with the screw in the same way, see fig. 4.

## 3.3 Leak Tightness Test

- 1. Insert the adapters Luer ¼" UNF into the openings of the injection adapters.
- 2. Close one adapter tightly with a male Luer plug.
- Use a plastic syringe from the accessory kit to inject, with moderate pressure, air through the other adapter.
- 4. Wait a few seconds, then release the plunger of the syringe.
  - If the connections are tight, the plunger of the syringe will be slowly pushed back by the pressure in the measuring cell.
  - If the connections are leaky, the plunger of the syringe will not move.
     In this case, remount the adapters.

**TIP:** In case of leaks you can widen the tips of the adapters with the transport plugs of the adapters.

## 3.4 Mounting the Hoses



### WARNING

Liquids leaking from the instrument may cause injuries or risk of fire.

- Only use the supplied hose and waste vessel if their materials are resistant to the samples and cleaning liquids that you are going to inject.
- If the supplied parts are not suitable, use other parts made of an appropriate material.

### 3.4.1 Hoses for Standard Measurements

### Waste hose / waste vessel

- Screw one end of the hose 300x3x2 PTFE, mat. no. 3443 (from the accessory kit), into the threaded hole in the cap of the waste vessel, see fig. 6.
- 2. Screw the other end of the hose into the upper injection adapter.

### Filling hose

- Screw an adapter Luer ¼" UNF, mat. no. 64792 (from the accessory kit), into the upper screw hole of the syringe holder.
- 2. Screw one end of the hose 140x3x2 PTFE, mat. no. 187223 (from the accessory kit), into the lower screw hole of the syringe holder.
- 3. Screw the other end of the hose into the lower injection adapter, see fig. 6.



Fig. 6: PTFE hoses mounted

# 3.4.2 Hoses for Measuring Sulfuric Acid and Oleum

### Viton waste hose / waste vessel

- 1. Screw an adapter UNF ¼" Luer male, mat. no. 64793 (from the accessory kit), into the threaded hole in the cap of the waste vessel.
- 2. Screw another adapter UNF ½" Luer male into the sample outlet adapter of the instrument.
- 3. Cut a piece of approx. 25 cm (10 in) length from the supplied Viton hose 3x5, mat. no. 54629.
- 4. Connect the ends of the Viton hose to the adapters UNF 1/4" Luer male and fix them with hose clamps (from the accessory kit).

### Viton filling hose

- Screw an adapter UNF ¼" Luer male, mat. no. 64793 (from the accessory kit), into the sample inlet adapter of the instrument.
- 2. Cut a piece of approx. 25 cm (10 in) length from the supplied Viton hose 3x5, mat. no. 54629.
- 3. Connect one end of the Viton hose to the adapter UNF 1/4" Luer and fix the connection with a hose clamp (from the accessory kit), see fig. 7.



Fig. 7: Viton hoses mounted

## 3.4.3 Air Pump Hose

- 1. Cut a piece of approx. 35 cm (14 in) length from the supplied silicone hose 4x6, mat. no. 57024.
- 2. Connect one end of the hose piece to the air pump outlet.

3. Attach an adapter Luer cone, mat. no. 63863 (from the accessory kit), to the open end of the hose piece.



Fig. 8: Air pump hose mounted

## 3.5 Connecting the Cooling

(only for DSA 5000 M CK.)

When you perform measurements at temperatures lower than 20 °C (36 °F) below ambient temperature, connect the cooling to an external thermostat. If your tap water is cool enough, also connecting to a tap water supply will help. Operate the cooling with a moderate flow of water (1 to 3 liters per minute).

temperature range of the cooling unit	5–30 °C (41–86 °F)
maximum pressure	1 bar (14.5 psi) relative
connector	self-locking coupling 8 mm, type Rectus 21KBTS08MVN, mat. no. 75090

### Example

Your ambient temperature is 25 °C (77 °F), and you want to perform measurements at 0 °C (32 °F).

Connect the instrument to an external thermostat or a tap water line delivering water at a constant temperature between 5 °C and 15 °C (41 °F...59 °F), and set the measuring temperature to 0 °C (32 °F).

### 3.6 Power Connection



### **WARNING**

# High voltage at parts of the instrument can cause serious injuries or death

- Connect the instrument only to an electrical outlet with protective earthing.
- Never connect the instrument to the AC power supply with protective separation or protective insulation.
- Ensure that the non-fused earth conductor of the power cable is connected to earth.
- Ensure that the current rating of the power cable is at least 10 A.

### NOTICE

### Possible damage due to wrong voltage

Before you switch on the instrument, make sure that the correct line voltage and line frequency are available (AC 100–240 V, 50/60 Hz). If large voltage fluctuations are to be expected, we recommend using a constant voltage source (UPS).

 Connect the AC power jack of the instrument (10, fig. 3) to a suitable electrical outlet with the supplied power cable.

## 3.7 Switching the Instrument On/Off

Use the power switch on the rear of the instrument (8, fig. 3) to switch the instrument on or off.

The green LED on the front of the instrument (1, fig. 1) indicates that the power is on.

 After power-on wait at least 15 minutes for the internal temperature to stabilize. **TIP:** Do not turn off the instrument during the night. This allows the measuring cell to achieve long term temperature stability.

**IMPORTANT:** If you have to **restart** the instrument, switch it off and wait until the green LED on the front has gone out before switching the instrument on again.

# 3.8 Basic Instrument Settings / First Checks

After hardware installation set the date and time, see General Software Functions Manual, section 6.1.1.

To check the validity of the factory adjustment, perform an air check and a water check.

The instrument has been factory adjusted over the whole temperature and viscosity range, but during transport the density adjustment may have been compromised.

### To perform first checks

**IMPORTANT:** Wait at least 15 minutes after a restart for the internal temperature to stabilize.

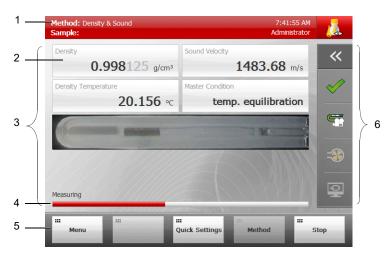
- To perform an air check, tap <Menu> and select Checks/Adjustments > Checks. Proceed as described in section 7.1.
- 2. To perform a water check, tap <Menu> and select *Checks/Adjustments* > *Checks*. Proceed as described in section 7.1.

If both checks succeed, your instrument is ready for routine measurements.

- If a check fails, clean the measuring cells thoroughly and repeat the check.
- If the check still fails, perform an air/water adjustment as described in section 7.2.1.

# 4 Operating the Instrument

## 4.1 Operating Elements on the Main Screen



**IMPORTANT:** Corresponding with your user group rights (see the General Software Functions Manual, section 6.5.1), operating elements may be locked.

Fig. 9: Example main screen

- 1 Header
- 2 Output field
- 3 Contents area
- 4 Status bar
- 5 Buttons area
- 6 Quick access area

### Header

On the left of the header you find the name of the currently active method and the sample number.

On the right of the header you find a clock and the user indicator. The user indicator indicates the type of user that is currently logged in.

### Contents area

In the contents area the measuring values are displayed in small, medium, or large output fields. The layout of the contents area can be defined in the settings of the current method according to your needs.

The status bar at the bottom of the contents area shows the status of the instrument or a measurement. If applicable, a progress bar shows the progress of activities.

### Monitor mode

If you have not started a measurement yet, or if you have terminated a measurement by tapping <Stop>, the instrument is in the monitor mode and shows a continuous reading of the current measuring values.

### Measuring mode

If you have started a measurement, a continuous reading of the current measuring values is shown until the measurement is finished. The final values stay frozen on the screen until the next measurement is started. To unfreeze the screen and change to monitor mode, tap in the quick access area.

### **Buttons** area

The buttons on the main screen have the following functions:

Button	Function
<menu></menu>	Opens the main menu.
<quick settings=""></quick>	Opens the quick settings list (only available in the "No Sample List" mode instead of the <sample list=""> button).</sample>
<sample list=""></sample>	Opens the current sample list.
<method></method>	Opens the method list (to select a method).
<start></start>	Starts a measurement.
<stop></stop>	Stops and aborts a measurement.

### Quick access area



Fig. 10: Expanded quick access area

- To collapse the quick access area, tap >> in the upper right corner of the expanded quick access area.
- To browse items, use the page navigation in the header of the expanded quick access area.
- To rearrange the items in the quick access area, tap (settings) in the upper left corner of the expanded quick access area. For details see the General Software Functions Manual, section 4.8.

#### **Function**



Opens the message list.

The general instrument status as well as all measuring errors that have occurred during the measurements of the currently active sample list are described in this list. The button changes its appearance depending on the current error status:

• Green OK sign:

The general instrument status and the error status of all measured samples of the current sample list are OK.



- Yellow warning sign:
  - The instrument (or system) has a minor problem (e.g. an air or custom check is overdue, there is a printer problem etc.).
  - There has been a filling error with one or more samples of the currently active sample list.



- Red error sign:
  - The instrument (or system) has a major problem that needs to be fixed before you continue with measurements (e.g. the sample changer is blocked).
  - One or more samples of the current sample list could not be measured (e.g. the measuring cell is partly empty so that it cannot oscillate).

To reset the message list button to the green OK sign, confirm all error messages by tapping on the <X> button on the right of the message. To confirm all messages in the list in one step, tap "Delete all" at the end of the list.

The message list button will also be reset to the green OK sign if you delete the currently active sample list, see General Software Functions Manual, section 7.6.



U-View™: Opens the live camera view of the measuring cell.



Starts/stops the air pump. The air pump is off.



The air pump is on.



Unfreezes the screen after a finished measurement.

The screen is frozen.



The screen is unfrozen. A continuous reading of the current measuring values is shown.



Displays information on using favorites.

# 4.2 Operating Elements on the Menu Screen

To access the menu, tap <Menu> on the main screen.

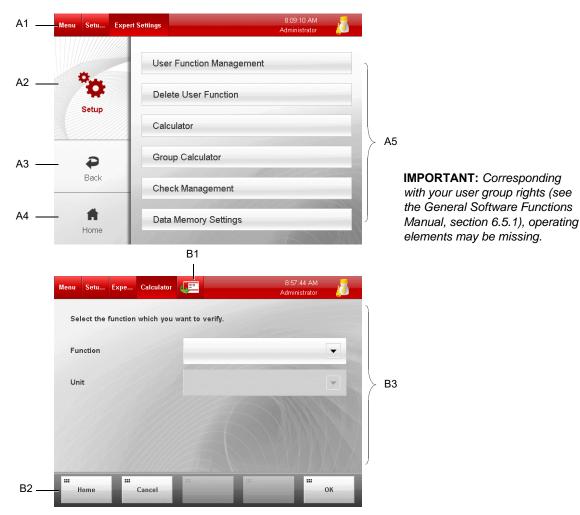


Fig. 11: Example menu screens

A1 Header

A2 Menu level 1

A3 <Back> button

A4 <Home> button

A5 Contents area

B1 <Add to Favorites> button

B2 Buttons area

B3 Contents area

### Header

On the left of the header you find the navigation path to your current position in the menu. You can go back to any menu position in your current path by directly tapping on the respective box of the navigation path. For details on using the <Add to Favorites> button, see the General Software Functions Manual, section 4.8.

### Contents area

In the contents area you find the menu options of the current menu level and the menu dialogs.

### **Buttons** area

The buttons on menu screens have the following functions:

Button	Function
<back></back>	Moves to the next higher menu level.
<home></home>	Returns to the main screen.
Buttons at the bottom of screens	Functions depending on the current menu or dialog

# 5 Defining and Using Methods

Each method contains the following kind of information:

- Instrument settings
- Xsample settings and measuring module settings (if any module is installed)
- Layout of measuring data on the main screen
- Measuring units
- Parameter list for printout and data export

You can use the factory preset methods as they are or change them to suit your needs. You can also create new methods. For details on the settings of the measuring methods, see the Reference Guide.

### Factory preset methods

The instrument comes with a set of 5 predefined methods covering the most common applications.

The measuring temperature for these methods is set to 20 °C.

Density & Sound	<ul> <li>General purpose method</li> <li>Output fields: Density, Sound Velocity, Density Temperature,</li> </ul>
Donony a count	Master Condition, U-View™
Density (not visccorr.) & Sound	<ul> <li>General purpose method, for comparison with old instruments without viscosity correction</li> <li>Output fields: Density (not visccorr.), Sound Velocity, Density Temperature, Master Condition, U-View™</li> </ul>
Sound	<ul> <li>General purpose method</li> <li>Output fields: Sound Velocity, s, Density Temperature, Master Condition, U-View™</li> </ul>
Sulfuric Acid & Oleum	<ul> <li>For measurement of concentrated sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) and oleum in the range from 0–100 % w/w H<sub>2</sub>SO<sub>4</sub> and 0–65 % w/w SO<sub>3</sub></li> <li>Output fields: Sulfuric Acid (H2SO4) (% w/w), Oleum, Density Temperature, Master Condition, U-View™</li> </ul>
Density only	<ul> <li>General purpose method</li> <li>Output fields: Density, Specific Gravity, Density Temperature, Master Condition, U-View™</li> </ul>

# 6 Performing a Measurement



### **WARNING**

# Risk of injuries or fire caused by hot liquids spurting out of the measuring cell

In the unlikely case of a malfunction or damage of the Peltier elements, the heating control of the measuring cell is affected, and the temperature of the measuring cell might rise up to 120 °C (248 °F) (for DSA 5000 M with mat. no. 153037, up to 150 °C/302 °F).

To avoid injuries and fire:

- Ensure that the waste vessel is properly installed.
- Use method settings that constantly display the temperature of the measuring cell.
- Check the temperature of the measuring cell before you fill a sample or start a measurement.
- If you measure aggressive, toxic, or flammable samples, use only the smallest possible sample amount.
- If your sample is toxic or highly flammable, always handle it in an appropriate environment, e.g. under a fume hood, and ensure that the location is sufficiently ventilated.
- Measure only samples with an ignition point over 120 °C (248 °F) (for DSA 5000 M with mat. no. 153037, over 150 °C/302 °F).
- Wear protective clothing, and safety goggles.

### Preparatory steps

Before you start a measurement, check that:

- the measuring cell is clean and dry,
- hoses are connected correctly,
- hose connections are tight,
- the waste hose leads into the waste container,
- the volume of the waste container is large enough for the number of samples,
- the correct sample names are entered,
- the method settings are set correctly,
- suitable cleaning liquids are at hand.

### To speed up measurements

- Use automatic sample naming (see General Software Functions Manual, section 6.3).
- Bring the sample to measuring temperature in advance to reduce the time necessary for the temperature equilibration.
- Using precision class "Ultrafast" reduces the measuring time significantly.

## 6.1 Sample Name

If you have defined automatic sample name parts (see General Software Functions Manual, section 6.3), they will be added to each sample name after the measurement has finished.

The complete sample name, including automatic sample name parts, can be up to 50 characters long.

## 6.2 Filling Samples

To achieve highly accurate measuring results, fill the sample into the measuring cell steadily and without bubbles.

### IMPORTANT: For high accuracy measurements:

- Be sure to apply exactly the same filling procedure for checks, adjustments, and measurements.
- If you use a syringe for filling, use the syringe holder to fully utilize the instrument's accuracy.
   Inject the entire sample volume. The syringe can stay connected to the filling adapter during the measurement.



### **WARNING**

Filling samples or cleaning liquids that the wetted parts are not resistant to will corrode the wetted parts. Sample leaking from corroded parts may cause serious injuries.

Before you fill any sample or cleaning liquid into the instrument:

- Make sure that all safety instructions concerning the use of chemicals and the use of flammable chemicals are met, see section 1.
   Borosilicate glass is not resistant to samples containing hydrofluoric acid, even in traces.
- Make sure that all wetted parts are resistant to the filled-in liquid, see appendix A.3.
   Consider also the wetted parts of an optionally installed Xsample filling module (refer to the manual of the Xsample).
- Make sure that suitable cleaning liquids for cleaning the measuring cells are at hand, see section 8.

### NOTICE

### Corrosive samples require special care

Samples with a moderate tendency to corrode borosilicate glass, such as strong alkali solutions (e.g. caustic soda), can be measured with the instrument. However:

- Remove corrosive samples immediately after measurement and rinse the measuring cell thoroughly.
- Check the validity of the adjustment more frequently than generally recommended.
   Perform a new adjustment if necessary.
- The measuring temperature for strong alkali solutions should not be higher than 20 °C (68 °F). Higher temperatures dramatically increase the speed of corrosion.

### Sample amount

If the measuring cell is clean and dry, you need approx. 3.5 mL of sample.

If you measure without cleaning and drying between samples, you will need a higher amount of sample because you have to flush residues of the previous sample out of the measuring cell to avoid crosscontamination.

# Bubble detection using the camera – U-View™

- Tap in the quick access area to open the live camera view of the density measuring cell.
  - Tap <Zoom In> to get a magnified view.
  - Attach a USB memory device and tap <Save Picture> to save the current picture.
- Tap <X> to exit the measuring cell view.



Fig. 12: Camera window

# To fill with a glass syringe (Luer tip) – for measuring sulfuric acid and oleum

#### NOTICE

- Always place the waste container below the level of the filling adapter of the instrument in order to prevent a sample backflow from the waste container into the instrument.
- Acid drops will destroy the surface of the instrument. Never put any sulfuric acid containers on top of the instrument.
- It cannot be avoided that acid drops emerge from the measuring cells when filling with a syringe. Place an acid-proof catch basin underneath the filling equipment.
- Inserting the glass tip of the syringe directly into the top connector of the syringe holder (as shown with the plastic syringe in fig. 14) may result in the fracture of the tip. Always use a Viton hose to connect the glass syringe to the inlet adapter (see fig. 13).
- 1. For measuring sulfuric acid or oleum, mount Viton hoses as described in section 3.4.2.
- 2. Connect the glass syringe to the free end of the Viton filling hose.



Fig. 13: Filling with a glass syringe

### To fill with a plastic syringe (Luer tip)

**IMPORTANT:** Do not use syringes that contain lubricants. The lubricants can dissolve into your sample and lead to a systematic measuring error.

- 1. Connect the syringe to the adapter Luer 1/4" UNF, mat. no. 64792, in the top connector of the syringe holder, see fig. 14.
- 2. Push the plunger of the syringe slowly and steadily until a drop emerges from the sample outlet adapter.
- 3. Leave the syringe in the filling position during the measurement.



Fig. 14: Filling with a plastic syringe

### To fill with a peristaltic pump

### NOTICE

- The liquid levels in the sample container and the waste vessel must be below the filling level of the instrument. Never put the peristaltic pump or the waste vessel on top of the instrument.
- · Check the hoses of the peristaltic pump daily.
- Check the life time of the hoses specified by the supplier, and change the hose frequently.
- Mount an adapter Luer cone onto the sample inlet adapter.
- 2. Connect the adapter Luer cone and the sample container with a silicone hose 3x5.
- 3. Mount an adapter Luer cone onto the sample outlet adapter.
- 4. Connect another silicone hose 3x5 to the outlet adapter Luer cone and lead it through the peristaltic pump into a waste vessel.
- 5. Set the flow rate of the pump to 10–25 mL/min.
- 6. Start the pump.
- 7. Turn off the pump after filling a sufficient amount of sample.

### To fill automatically with an Xsample

For details see the manual of the Xsample.

### 6.3 Measurement Procedure

Proceed by the following steps:

- Select a method.
- Fill the sample.
- Perform the measurement.

### To perform a measurement

- 1. Enter a sample name if required (see also section 6.1).
- Tap <Start> and wait until the measurement is finished.

The progress bar shows the progress of the measurement by a growing red bar and the message "Measuring" or "Measuring (delayed)" if a measurement delay time has been set.

During measurements, the last 1, 2, or 3 **digits** may be **grayed** signifying that the set temperature has not been reached yet. In this case, only the solid black digits are valid. The number of valid (black) digits will increase during measurement progress until all digits are black (depending on the set precision class).

When you perform measurements of gases/air, the **sound velocity** values are **grayed** because the values are outside the instrument's specifications.

When the measurement is finished, the progress bar turns green, the message "Finished" is displayed, and an acoustic signal is given. The output field "Master Condition" shows the status "valid". The result values are saved in the data memory and can be viewed, printed, exported or deleted.

The result values on the screen are frozen. Tap in the quick access area to unfreeze the screen.

# 6.4 Measuring Sulfuric Acid and Oleum

### 6.4.1 General Description

DSA 5000 M is designed to measure the concentration of pure sulfuric acid and oleum (fuming sulfuric acid) in the range of 0–100 % w/w  $\rm H_2SO_4$  and 0–65 % w/w  $\rm SO_3$ .

The concentrations are automatically derived from density or sound velocity measured by the instrument.

The samples are filled into the measuring cells using a glass syringe or a peristaltic pump and Viton hoses, which are resistant to concentrated sulfuric acid and oleum.

### 6.4.2 Additional Safety Instructions

Beside the safety instructions in section 1, the additional safety instructions below have to be followed when measuring sulfuric acid and/or oleum:



### **WARNING**

Oleum (fuming sulfuric acid) and sulfuric acid are highly caustic substances which may cause irritations and serious injuries to skin, eyes and mucous membranes.

- Follow all the precautions for the handling and measurement of samples and cleaning materials written below. The list does not cover all regulations and safety procedures. Safety standards are available from government agencies and various chemical associations.
- Working with oleum and sulfuric acid requires special training of employees. Ensure that all employees handling these hazardous substances are periodically instructed in all handling, safety and emergency procedures.
- Ensure that all operators are fully trained in the correct use of the instrument and its safe operation.
- Always wear protective clothing and eye protection (protective goggles or face shield) when handling oleum or sulfuric acid.
- Ensure there are fire extinguishing equipment, first aid kits, overhead drench showers, and eye baths within reach.



### **WARNING**

Using highly corrosive strong mineral acids (e.g. oleum, sulfuric acid) as samples could lead to damage of the instrument and cause serious injuries unless special precautions are taken.

- Ensure the chemical resistance of the instrument and the materials used during the measurement
- Do not exceed the maximum temperature of 40 °C (104 °F) when measuring sulfuric acid to ensure chemical compatibility with the wetted parts of DSA 5000 M.

For the wetted parts, see appendix A.3.



### **WARNING**

Mixing oleum or concentrated sulfuric acid with water or organic solvent will cause a very strong exothermic reaction with the danger of serious injuries. An exothermic reaction inside the measuring cells may destroy them.

- Never mix oleum or concentrated sulfuric acid with water nor diluted acid in the measuring cells
- Always dilute oleum by adding it drop by drop to 98 % w/w H<sub>2</sub>SO<sub>4</sub> while stirring and cooling.
- Dilute concentrated H<sub>2</sub>SO<sub>4</sub> by adding it drop by drop to water while stirring and cooling.
- Before filling any kind of sulfuric acid or oleum into the instrument, ensure that the measuring cells have been cleaned and dried or that they already contain concentrated sulfuric acid or oleum.
- Do not fill concentrated sulfuric acid or oleum into the measuring cells if there is still water, organic solvent, or diluted acid in the cells.



### WARNING

Oleum and concentrated sulfuric acid react with oxidizable organic materials and solvents, reducing agents, chlorates, permanganates, ammonia, oxides and hydroxides of alkali and alkaline earth metals. The chemical reaction may lead to serious injuries or may destroy objects containing those substances.

- Never bring oleum or concentrated sulfuric acid into contact with those materials and substances
- Always use separate waste containers for sulfuric acid waste and ethanol (or other solvent) waste.
- Label the waste containers properly so that no mix-ups are possible.
- Ensure that the material of the sulfuric acid waste container is resistant to sulfuric acid and oleum.
- · Clean all spillages immediately.
- Do not leave sample containers uncovered.
- Do not leave the instrument unattended while in use.
- Operate the instrument under a fume hood or in a sufficiently ventilated area free of flammable gases and vapors.

### 6.4.3 Setting the Correct Temperature

After having selected the correct measuring method "Sulfuric Acid & Oleum" (see the General Software Functions Manual, section 7.4), the measuring temperature has to be set to either 20 °C or 40 °C. The temperature is set to 20 °C by default.

If the concentration of free  $SO_3$  in all your samples is 27 % w/w or below, then a measuring temperature of 20 °C should be selected. All samples are liquid at this temperature.

If you regularly have to measure samples with concentrations of free  ${\rm SO_3}$  higher than 27 % w/w, then 40 °C has to be selected as the measuring temperature. This switches the instrument's software to full range evaluation from 0 % w/w  ${\rm H_2SO_4}$  to 65 % w/w  ${\rm SO_3}$ .

Note that measurements at 20 °C take considerably less time than measurements at 40 °C.

# 6.4.4 Filling Sulfuric Acid and Oleum Samples

You can fill sulfuric acid and oleum either with a glass syringe or semiautomatically by using an external peristaltic pump. For details see section 6.2. Observe the additional instructions when filling sulfuric acid/oleum.

### NOTICE

Mixing concentrated sulfuric acid or oleum with water, organic solvent, or dilute acid causes an exothermic reaction. An exothermic reaction in the measuring cells may destroy them.

- Before filling any kind of sulfuric acid or oleum into the instrument, make sure that the measuring cells have been cleaned and dried, or that they already contain concentrated sulfuric acid or oleum.
- Do not fill concentrated sulfuric acid or oleum into the measuring cells if there is still water, organic solvent, or dilute acid in the cells.

### NOTICE

Oleum samples with 28 % w/w to 58 % w/w of free  ${\rm SO_3}$  may freeze inside sample containing parts, such as hoses, sample containers, etc., at temperatures below 35 °C.

 Keep the sample containing parts at a temperature above 35 °C to prevent freezing of sample.

### NOTICE

If the hoses are not properly fixed or not resistant to the sample, sample could leak onto the bench and cause corrosion.

- Place the peristaltic pump in an acid-proof catch basin for safety reasons.
- Never use hoses that are not resistant to sulfuric acid and oleum.

**TIP:** If you measure a series of samples of very similar concentrations, you can displace the previous sample by the new sample. The minimum amount necessary to fully replace the sample is

- approx. 10 mL of new sample when filling by syringe,
- approx. 25 mL of new sample when filling by peristaltic pump.

### 6.4.5 Calculations

For basic information about the measuring principle see the Reference Guide.

The concentration of sulfuric acid in % w/w is calculated using a function of the density or the sound velocity, depending on the concentration range.

The following table shows for the measuring temperatures 20 °C and 40 °C, in which concentration ranges the density or sound velocity is used for the calculation.

Temp.	Density	Sound velocity
20 °C	0–87 % H <sub>2</sub> SO <sub>4</sub> 0–27 % free SO <sub>3</sub>	87-100 % H <sub>2</sub> SO <sub>4</sub>
40 °C	0-90 % H <sub>2</sub> SO <sub>4</sub> 0-47 % free SO <sub>3</sub>	90–100 % H <sub>2</sub> SO <sub>4</sub> 47–65 % free SO <sub>3</sub>

The density and sound velocity graphs of sulfuric acid and oleum for the whole concentration range at a measuring temperature of 40 °C are shown in fig. 15.

The concentration (% w/w) of free  $SO_3$  is calculated from the concentration of sulfuric acid according to:

% w/w free 
$$SO_3 = \frac{MW_{SO_3} \cdot (\% \text{ w/wH}_2SO_4 - 100)}{MW_{H_2O}}$$
  
= 4.444 \cdot (% \text{ w/wH}\_2SO\_4 - 100)

MW ..... molecular weight

### Sound Sound velocity velocity Density Density 1600 Density [g/cm<sup>3</sup>] 1.75 1400 1 25 1.00 114% H<sub>2</sub>SO<sub>4</sub> 100 65% SO<sub>3</sub> 47 Concentration [%] Density Sound velocity

Measurement of:

### Fig. 15: Density and sound velocity of sulfuric acid and oleum at 40 °C

# 6.4.6 Corrections for Calculated Analysis Results

If you see deviations between the analysis results of DSA 5000 M and a reference method, corrections can be stored as a special function. The corrections will be applied to the results automatically.

- 1. Create a new user function. (For details see the General Software Functions Manual, section 9.2.)
- 2. Specify if you want to apply your correction with a formula, a polynomial, or a table.
- 3. Define either "Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>)" or "Oleum" as the input quantity.
- 4. Specify
  - a correction formula in case of "user formula",
  - correction coefficients in case of "polynomials"
  - table values in case of "user tables".
- 5. Tap <OK>.

## 6.5 Degassing Samples

There are various methods to degas liquid samples. The preferable method for your application depends on the kind of sample and the amount of gas that is dissolved in the sample. Pay attention to always treat all samples in the same way in order to get reproducible measuring results.

**IMPORTANT:** Be aware of the fact that the composition of some samples may slightly change during the pretreatment due to the evaporation of volatile components.

### Boiling the sample



### **WARNING**

Samples containing toxic volatile compounds can cause irritation and serious injuries to your eyes, skin and mucous membranes, as well as toxication.

 If your sample contains volatile compounds that are toxic, always handle it in an appropriate environment, like under a fume hood, especially when you boil your sample.



### **WARNING**

When you boil flammable liquids, there is a risk of fire. Serious injuries are possible.

• Do not boil flammable liquids.

- 1. Boil the liquid for several minutes to remove dissolved air.
- 2. Fill the boiled liquid into a clean glass flask and cover it.
- 3. Wait until the liquid has cooled down approximately to measuring temperature.

### Stirring the sample

- Stir your sample vigorously for 2–15 minutes (depending on the stirring equipment) until bubbling ceases.
- You can pour the sample through a paper filter after stirring to degas it even more efficiently.

### Using an ultrasonic bath

 Put your sample for approx. 5–10 minutes into an ultrasonic bath until bubbling ceases.

# 6.6 Special Filling Techniques

For details on special filling techniques for samples as listed below, see the Reference Guide.

- bubbling samples
- suspensions and emulsions
- highly viscous samples
- pastes
- liquids in aerosols
- gases

## 6.7 Measuring at High Temperatures



### WARNING

### Risk of leakage

At high sample temperatures in combination with high pressure the injection adapter Luer may leak.

 At measuring temperatures of 50 °C (122 °F) and higher, the applied pressure must be limited to 5 bar (72.5 psi) absolute pressure.

### To prevent bubble formation

If you measure samples at temperatures significantly higher than ambient temperature, the tendency to form gas bubbles in the measuring cell will dramatically increase. To ensure precise results, you can:

 Degas your samples thoroughly directly before measuring, see section 6.5.  Heat your samples up to a temperature significantly higher than the measuring temperature with stirring, directly before measuring.

## 6.8 Measuring at High Pressures

The supplied silicone hose and injection adapters can only be used at atmospheric pressure. Before you apply high pressures, exchange the supplied filling components with pressure-resistant components.



### WARNING

### Risk of explosion

The sound velocity measuring cell of instruments with mat. no. 153037 (assembled until January 2018) cannot withstand pressures above 3 bar (43 psi) relative pressure.

• Operate these instruments only in the pressure range of 0–3 bar (0–43 psi) relative pressure.



### **WARNING**

Leaky components of the measuring system can cause sample to splash out when pressure is applied to the measuring system. Injuries and risk of fire possible.

To avoid injuries, do the following:

- At pressures higher than ambient pressure, only use one injection adapter DMA/CarboQC (mat. no. 159537), one injection adapter DSA/CarboQC (mat. no. 159578), and polyurethane hoses 2x4 mm (mat. no. 135259).
- Do not exceed the maximum operating pressure of any single component.
- Before you start a measurement at high pressure, check the pressure tightness of the system with air.
- If you measure any aggressive, poisonous, or flammable sample at high pressure, use only the smallest possible sample amount.

### Adjustment with air and water

To reach the highest possible accuracy for high pressure measurements, perform the air adjustment as usual at ambient pressure, and the water adjustment or an adjustment with any other reference liquid at the pressure at which you are going to perform the measurements.

# 7 Checks, Adjustments, Calibrations

### 7.1 Checks

- 1. Tap <Menu> and select *Checks/Adjustments* > *Checks* to open the checks list.
- 2. Highlight a water check, an air check, or a custom check in the list.
- Tap <Start> and follow the instructions on the screen.
  - For a water check, use freshly degassed ultra-pure water.
  - For an *air check*, clean and dry the measuring cell thoroughly.
  - Use the camera image to check that the measuring cell is clean or that water has been filled free from bubbles.

When the check is finished, the following information is displayed:

- Check name/type and check result ("Passed" or "Not passed")
- Date and time
- Method used
- Check result
- User name
- For water checks:
  - Module Density
    - Reference density value calculated for the set temperature
    - Lower deviation
    - Upper deviation
    - Measured density value
    - Check result
  - Module Sound Velocity (only water check)
    - Sound velocity of water at the check temperature (reference value)
    - Lower deviation
    - Upper deviation
    - Measured sound velocity value
    - Check result
  - Set temperature for the measuring cell
- For air checks:
  - Reference density value calculated for the set temperature
  - Lower deviation
  - Upper deviation
  - Measured density value
  - Check result
  - Air pressure
  - Set temperature for the measuring cell

- For custom checks:
  - Lower limit
  - Upper limit
  - Measured value
  - Check result
  - Set temperature for the measuring cell
- 4. Tap <Print or Export> if you want to print or export the results of the check.
- 5. Tap <OK> or <Home> to exit the check routine.

### If the water check has failed

We recommend taking corrective actions until the check is valid again:

- Examine the camera image included in the results to check that the water has been filled bubble-free.
- · Check the quality of the water.
- · Clean the measuring cells thoroughly.
- If above actions do not help, perform an air/water adjustment.

## 7.2 Adjustments

If the instrument is operated in the non-storage mode, the adjustment data are not viewable, see also the General Software Functions Manual, section 9.1.2.

**IMPORTANT:** When operating the instrument in the non-storage mode, print or export the adjustment results immediately after the adjustment.

For detailed information on all available adjustments, adjustment data management, resetting to factory adjustment, see the Reference Guide.

### 7.2.1 Air/Water Adjustment

An air/water adjustment has to be performed if the water check has failed ("not passed" as the result), and using freshly degassed ultra-pure water and cleaning the measuring cell did not help.

The adjustment media are dry air and freshly degassed ultra-pure water.

The ThermoBalance™ technology allows for precise measurements over the whole temperature range with only one adjustment at 20 °C. To achieve the highest possible precision of measurements at different temperatures, you can additionally perform a temperature range adjustment, see the Reference Guide.

The air/water adjustment takes 5–10 minutes if the instrument is already clean and dry and equilibrated to 20 °C.

The adjustment procedure can be aborted by tapping <Cancel>.

# To set the reference values for the air/water density adjustment

The reference densities for air can be selected according to your needs. They are either based on the formula of

- Spieweck and Bettin<sup>1</sup> or
- CIPM<sup>2</sup>.

The reference densities for water can be selected according to your needs. They are either based on the formula of

- Spieweck and Bettin<sup>3</sup> or
- CIPM<sup>4</sup> / IAPWS<sup>5</sup>.
   (The CIPM formula is limited to 0–40 °C only, so for temperatures above 40 °C, the IAPWS formula is used.)

The factory adjustment has been set using reference values for the density of air (humidity 50 %) and water based on the formula of Spieweck and Bettin<sup>1,3</sup>. This formula covers the whole measuring range (0–100 °C) of the instrument.

**IMPORTANT:** You have to readjust the instrument after changing the selected air and/or water table.

- 1. Tap <Menu> and select Setup > Measuring System Settings > Density Module.
- 2. Use the drop-down box "Air Table" to select the preferred reference table.
- 3. Use the drop-down box "Water Table" to select the preferred reference table.
- 4. Tap <OK> to save your settings.

**TIP:** For use in metrology, the CIPM density formula is the preferred standard over its recommended range (0–40 °C) at pressures near atmospheric. The formula should not be extrapolated outside this range.

Densities from the IAPWS formula are consistent with the CIPM standard within the range of validity of the CIPM formula. Outside the CIPM range of validity, the IAPWS formula is the preferred method for obtaining accurate densities of water.

**IMPORTANT:** The choice of reference table also affects derived quantities (e.g. Apparent Density, SG, API values, etc.).

All factory-set concentration tables consist of calculated data based on the reference values according to Spieweck and Bettin.

### To perform an air/water adjustment

- 1. Tap <Menu> and select Checks/Adjustments > Air/Water at 20 °C.
- 2. Choose whether you want to adjust the density module, the sound module, or both, then tap <OK>.
- 3. Rinse and dry the measuring cell.

**TIP:** If you use undenatured ethanol as the final rinsing liquid, only 3–4 minutes drying time are required.

Tap <Air Pump on> to dry the measuring cell.

- 4. Tap <OK>.
- 5. Enter the air humidity and the atmospheric pressure.

The air humidity is set to 50 % per default. The atmospheric pressure displayed is measured automatically by a built-in sensor.

- 6. Tap <OK>.
  - The air adjustment routine is carried out.
- 7. Fill freshly degassed ultra-pure water into the measuring cell and tap <OK>.

Be careful to fill the water without air bubbles.

**TIP:** If the water has been filled without air bubbles, you can ignore a possible warning message "Master Condition: filling warning" during the adjustment routine. A proper adjustment is required for the FillingCheck<sup>TM</sup> function.

<sup>1</sup> F. Spieweck, H. Bettin: Review: Solid and liquid density determination. tm – Technisches Messen 59 (1992) 7–8, pp. 285–292.

<sup>2</sup> A. Picard, R.S. Davis, M. Gläser, K. Fujii. Metrologia 45 (2008), pp. 149-155.

<sup>3</sup> Cf. Spieweck/Bettin, p. 291.

<sup>4</sup> M. Tanaka, G. Girard, R. Davis, A. Peuto, N. Bignell. Metrologia 38 (2001), pp. 301-309.

<sup>5</sup> International Association for the properties of water and Steam: Release on the IAPWS formulation 1995 for the thermodynamic properties of ordinary water substance for general and scientific use [1996]. Available at www.iapws.org.

The water adjustment routine is carried out.

When the adjustment is finished, the following information is displayed:

- Density air/water adjustment
  - Reference density of water
  - Deviation to reference value using old adjustment constants
  - Deviation to reference value using new adjustment constants
- Sound velocity water adjustment:
  - Reference sound velocity of water
  - Deviation to reference value using old adjustment constants
  - Deviation to reference value using new adjustment constants
- 8. Check the recommendation on the screen for each adjustment and select one of the options <Reject>, <Print>, or <Apply>.

## 7.3 Calibration

See the Reference Guide.

# 8 Upkeep and Cleaning

To ensure a constant and high accuracy of your measurements, employ a regular and effective cleaning routine, and store the instrument under the recommended conditions.

# 8.1 Cleaning and Drying the Measuring Cells



### **WARNING**

**Risk of injuries and fire by liquids leaking**Before you fill any sample or cleaning liquid into the instrument:

- Strictly follow all safety instructions concerning the use of chemicals and the use of flammable chemicals, see section 1;
- Make sure that all wetted parts are resistant, see appendix A.3.



### WARNING

Cleaning the measuring cells after the measurement of **sulfuric acid** or **oleum** samples requires special precautions. If these are not taken, serious injuries and damage of goods are possible.

· For details see section 8.2.

### NOTICE

Do not use any mechanical action for cleaning the measuring cells.

### Cleaning frequency

Clean and dry the measuring cells at least after each working day or working shift.

Cleaning more frequently can be necessary ...

- before you perform adjustments,
- before you want to measure using a minimum sample amount,
- before you measure a sample that is not miscible with the previous sample (e.g. water after a petrochemical sample),
- before you measure a sample that could chemically react with the previous sample.

### Cleaning liquids

For cleaning and drying after standard measurements with harmless samples, employ two cleaning liquids in a row:

- Cleaning liquid 1 dissolves and removes sample residues in the measuring cells. It has to be a good solvent for all sample components.
- Cleaning liquid 2 removes cleaning liquid 1 and is easily evaporated by a stream of dry air so that drying of the cells is accelerated. Cleaning liquid 2 has to be a good solvent for cleaning liquid 1.

Recommended for aqueous samples / beverages: water (cleaning liquid 1) and non-denatured ethanol (cleaning liquid 2).

Recommended for petrochemical samples: petroleum naphtha (cleaning liquid 1) and acetone (cleaning liquid 2).

If you are not sure whether a cleaning liquid is suitable for your sample, perform a preliminary test in a test tube to see if any phase separation, precipitate or opalescence can be observed.

### Cleaning and drying procedure without Xsample filling equipment

- 1. Rinse the measuring cells with cleaning liquid 1 (minimum 5 mL).
  - If your sample is viscous or contains particles, use more cleaning liquid.
- 2. Empty the measuring cells.
- 3. Rinse the measuring cells with cleaning liquid 2 (minimum 5 mL).
- 4. Empty the measuring cells.



Fig. 16: Drying the measuring cells

- 5. Insert the air pump hose with the adapter Luer cone into the top connector of the syringe holder, see fig. 16.
- 6. Tap 😵 in the quick access area to start the air pump.
- 7. Wait until the measuring cell is dry (stable density reading).

The time needed depends on the vapor pressure of cleaning liquid 2 and the temperature of the measuring cell (ethanol at 20 °C: approx. 5 min, acetone at 20 °C: approx. 3 min).

If the ambient humidity is > 90 % rel. humidity, use a drying cartridge (see the Reference Guide) to reduce the drying time.

- 8. Tap in the quick access area to stop the air pump, or wait for the pump timeout.
- Disconnect the air pump hose with the adapter Luer cone from the top connector of the syringe holder.



### WARNING

**Risk of injuries and fire by liquids leaking**If liquids get into the air pump system, they may destroy the pump diaphragm and leak from the instrument.

• Disconnect the air pump hose before you start filling liquids.

Cleaning and drying procedure with Xsample filling equipment

See the manual of the Xsample.

# 8.2 Cleaning and Drying the Measuring Cells after Measurements of Sulfuric Acid / Oleum



### **WARNING**

The mixture of oleum or concentrated sulfuric acid with water or solvents causes a very strong exothermic reaction, which may destroy the measuring cells and/or cause serious injuries.

- Never flush out oleum or concentrated sulfuric acid with water.
- Ensure that samples of strongly differing concentrations do not come into contact with each other. Therefore, use only 98 % w/w H<sub>2</sub>SO<sub>4</sub> to remove oleum, remove 98 % w/w H<sub>2</sub>SO<sub>4</sub> only with 70 % w/w H<sub>2</sub>SO<sub>4</sub>, and remove 70 % w/w H<sub>2</sub>SO<sub>4</sub> with 40 % w/w H<sub>2</sub>SO<sub>4</sub>. Only then water may be used to rinse the cells.
- Always use separate waste containers for sulfuric acid waste and ethanol (or other solvent) waste. Label the waste containers properly to avoid mix-ups.
- Always place the waste containers in such a way that the liquid level is below the level of the filling adapter of the instrument.
- Place the waste containers behind a safety shield and in a catch basin.
- Never flush sulfuric acid waste and ethanol (or other solvent) waste down the sink.
- Always dispose of the waste according to regional laws and regulations.

#### NOTICE

- Residues of sample on the filling adapters will cause corrosion or inaccuracy of results.
  - Leave the filling adapters on the instrument when you clean it, so that they are cleaned together with the measuring cells.
- If the air pump sucks air with high humidity into the measuring cells, or if the ambient temperature is much lower than the measuring temperature, condensation could build up in the measuring cells, bearing the risk of an exothermic reaction when sulfuric acid or oleum is filled. An exothermic reaction may destroy the measuring cells.
  - Use a drying cartridge (desiccator) to help completely dry the measuring cells. The drying cartridge has to be connected to the "DRY AIR IN AIR PUMP" connector at the rear of the instrument with a short piece of Viton hose.

# Cleaning and drying procedure using a syringe



### **WARNING**

Syringes made of polypropylene/polyethylene are not resistant to oleum and concentrated sulfuric acid, and will corrode. Sample may leak from the corroded parts and cause serious injuries.

- Do not use polypropylene/polyethylene syringes for oleum and H<sub>2</sub>SO<sub>4</sub> with concentrations higher than 95 % w/w.
- Use only glass syringes with Teflon-sealed plungers for oleum and for H<sub>2</sub>SO<sub>4</sub> in the concentration range 95 % w/w to 100 % w/w.
- 1. Fill the syringe with sulfuric acid of the proper concentration, see warning above.
- Connect the syringe to the Viton filling hose and slowly inject the liquid into the measuring cells. Leave the syringe connected to the filling hose.
- 3. Remove the liquid from the measuring cells by slowly pulling back the plunger of the syringe.
- 4. Disconnect the syringe and drain its contents into the sulfuric acid waste container.
- Repeat steps 1 to 4 with sulfuric acid of decreasing concentrations until it is safe to rinse with water (concentration of H<sub>2</sub>SO<sub>4</sub> 40 % w/w or below).
- 6. Applying the same method as above, rinse with at least 30 mL of ultra-pure water.
- 7. Exchange the sulfuric acid waste container with an ethanol waste container.
- 8. Rinse with at least 20 mL of 96 % ethanol.
- 9. Attach the air pump hose with the Luer adapter cone to the filling hose.
- Tap in the quick access area to start the air pump.
- Tap in the quick access area to stop the air pump, or wait for the pump time out.
- Disconnect the air pump hose from the filling hose.
- 13. Fill in the new sample for measuring, see section 6.3.

# Cleaning and drying procedure using a peristaltic pump

- 1. Fill the measuring cells with sulfuric acid of the proper concentration, see warning above.
- Repeat step 1 with sulfuric acid of decreasing concentrations until it is safe to rinse with water (concentration of H<sub>2</sub>SO<sub>4</sub> 40 % w/w or below).
- 3. Rinse with at least 30 mL of ultra-pure water.
- 4. Exchange the sulfuric acid waste container with an ethanol waste container.
- 5. Rinse with at least 30 mL of 96 % ethanol.
- 6. Loosen the cartridge of the peristaltic pump.
- 7. Attach the air pump hose with the Luer adapter cone to the filling hose.
- 8. Tap 😵 in the quick access area to start the air pump.
- 9. Tap in the quick access area to stop the air pump, or wait for the pump time out.
- 10. Lock the pump lever of the peristaltic pump.
- Disconnect the air pump hose from the filling hose.
- 12. Fill in the new sample for measuring, see section 6.3.

# 8.3 Cleaning the Instrument Housing and the Touchscreen



### WARNING

Before using any cleaning agents for the instrument's housing and touchscreen:

- Strictly follow all safety instructions concerning the use of chemicals and the use of flammable chemicals, see section 1.
- Make sure that all parts of the housing are resistant, see appendix A.2. In case of uncertainties contact Anton Paar GmbH.
- Decontaminate and remove aggressive sample residues on the instrument.



### **WARNING**

Ethanol is a highly flammable liquid.

• Strictly follow all safety instructions concerning the use of flammable chemicals, see section 1.

### NOTICE

Corrosion due to unsuited means of cleaning

Using substances for cleaning that are not suitable causes corrosion of the instrument housing. Never use:

- highly nonpolar solvents (e.g. toluene, hexane, solvent naphtha),
- strong acids or bases (e.g. nitric acid, sulfuric acid, hydrochloric acid, caustic soda),
- · strong mechanical action (steel brush).

To clean the instrument housing or the touchscreen, use a soft tissue, which can be wetted with ethanol or warm water, if necessary with some mild cleaning agent added (pH < 10).

## 8.4 Storing the Instrument

Clean and dry the measuring cells, see section 8.1, before you store the instrument for more than one day. Otherwise algae may grow on the glass surface, which are difficult to remove.

If you store the instrument for less than one day, the measuring cells can be filled with ultra-pure water or stay filled with the last cleaning liquid used. If you have filled by syringe, leave the syringe connected to the injection adapter to prevent spillage of the liquid.

## 8.5 Transporting the Instrument

- Empty the measuring cells and all hoses before vou move or lift the instrument.
- To move or lift the instrument, grasp the ledge on top of the instrument at the back with one hand. Place the other hand under the display at the front. There is a hollow for your fingers.
- Carry the instrument in front of you and keep it close to your body.

# 9 Maintenance and Repair

# 9.1 Maintenance Performed by an Authorized Anton Paar Service Engineer

The instrument requires a periodical maintenance, which shall be performed by an authorized Anton Paar Service Engineer. <sup>1</sup>

A missing maintenance may mean that under certain conditions your warranty is no longer valid. <sup>2</sup>

### Maintenance interval

once a year

Parts to be exchanged at every maintenance interval (wear and tear parts)

- · hoses
- adapters

Following parts are generally excluded from the warranty (wear and tear parts)

- · syringes
- · hoses
- · adapters, connectors, fittings
- · pump diaphragms
- · filters
- · O-rings, seals, gaskets

- cables
- fuses
- batteries
- · desiccants
- protection foils and covers

All parts damaged in consequence of a fall of the instrument are generally excluded from the warranty as well.

# 9.2 Repair Performed by an Authorized Anton Paar Representative

In case your instrument needs repair, contact your local Anton Paar representative, who will take care of the necessary steps. If your instrument needs to be returned, request an RMA (Return Material Authorization Number). It must not be sent without the RMA and the filled "Safety Declaration for Instrument Repairs". Please make sure it is cleaned before return.

**TIP:** Contact your local Anton Paar representative from the Anton Paar website under "Contact" (https://www.anton-paar.com).

**IMPORTANT:** You must not return instruments that are contaminated by radioactive materials, infectious agents, or other harmful substances that cause health hazards.

<sup>1</sup> Please contact your Anton Paar representative to get an offer.

<sup>2</sup> For detailed information see the general terms of delivery (GTD) on the Anton Paar website (https://www.anton-paar.com).

# Appendix A: Technical Data

# A.1: Specifications

Table 1: Measuring performance

Density		
Measuring range	0 g/cm <sup>3</sup> to 3 g/cm <sup>3</sup>	
Repeatability s.d. <sup>a</sup>	0.000001 g/cm <sup>3</sup>	
Reproducibility s.d. <sup>a</sup>	0.000005 g/cm <sup>3</sup>	
Accuracy <sup>b</sup>	0.000007 g/cm <sup>3</sup>	
Sound velocity		
Measuring range	1000 m/s to 2000 m/s	
Repeatability s.d. <sup>a</sup>	0.1 m/s	
Reproducibility s.d. <sup>a</sup>	0.5 m/s	
Temperature		
Measuring range <sup>c,d</sup>	0 °C to 100 °C (32 °F to 212 °F)	
Repeatability s.d.	0.001 °C (0.002 °F)	
Accuracy	0.01 °C (0.02 °F)	
Pressure range <sup>d,e</sup>	0 bar to 8 bar (0 psi to 116 psi) absolute pressure	
Measuring time per sample <sup>f</sup>	1 to 4 minutes	
Sample volume	approx. 3.5 mL	
Accuracy and convenience features	ambient air pressure sensor   full range viscosity correction   reference oscillator   automatic bubble detection   camera image of the density measuring cell	

- a According to ISO 5725
- b Under ideal conditions and for low densities/viscosities
- c Cooling down more than 20 °C (36 °F) below ambient temperature only with DSA 5000 M CK
- d Only valid for DSA 5000 M with mat. no. 182706 or 186723 For DSA 5000 M with mat. no. 153037: 0 °C to 70 °C (32 °F to 158 °F) | 0 bar to 3 bar (0 psi to 44 psi) relative pressure
- e For temperatures above 50 °C (122 °F), the maximum allowed pressure is 5 bar (72.5 psi) absolute pressure.
- f After full temperature equilibrium

Table 2: Typical precision of concentration measurements (only valid for uncontaminated samples)

0-100 % w/w H <sub>2</sub> SO <sub>4</sub>	±0.02 % w/w H <sub>2</sub> SO <sub>4</sub>
0–28 % w/w free SO <sub>3</sub>	±0.04 % w/w H <sub>2</sub> SO <sub>4</sub>
28–65 % w/w free SO <sub>3</sub>	±0.1 % w/w free SO <sub>3</sub>
for other applications	application-dependent, typically 0.01-0.1 % w/w

Table 3: Precision classes

Precision class	Stability temperature	Stability density	Stability sound velocity
Ultrafast	< 0.1 °C for 10 s	< 0.000005 g/cm <sup>3</sup> for 15/45 s <sup>a</sup>	< 0.6 m/s for 40 s
Fast	< 0.05 °C for 10 s	< 0.000005 g/cm <sup>3</sup> for 60 s	< 0.05 m/s for 50 s
Standard	< 0.005 °C for 10 s	< 0.000005 g/cm <sup>3</sup> for 60 s	< 0.05 m/s for 60 s
Precise	< 0.002 °C for 10 s	< 0.000001 g/cm <sup>3</sup> for 60 s	< 0.01 m/s for 90 s

a 45 s for temperature scans

# A.2: Instrument Data and Operating Conditions

Environmental conditions (EN 61010)	indoor use only
Ambient temperature	15 °C to 35 °C (59 °F to 95 °F)
Air humidity	10 % to 90 % relative humidity, non-condensing
Altitude	max. 3000 m (9800 ft)
Pollution degree	2
Overvoltage category	II
Memory	1000 measuring values with / without camera pictures
Touchscreen	10.4" TFT PCAP touchscreen 640 x 480 px
Interfaces	4 x USB (2.0 full speed), 1 x Ethernet (100 Mbit), 1 x RS-232, 1 x CAN Bus, 1 x VGA
RS-232C printer settings	Baud rate: 9600; Parity: none; Stop bit: 1; Data bits: 8
Voltage	100 to 240 V~, 50/60 Hz, fluctuation ±10 %
Power consumption	190 VA <sup>a</sup>
Power inlet	according to IEC/EN 60320-1/C14, protection class I
Fuses	ceramic tube fuses 5x20 mm; IEC60127-2; AC 250 V; T 5 AH
Dimensions (L x W x H)	495 mm x 330 mm x 230 mm (19.5 in x 13 in x 9 in)
Weight	22.5 kg (49.6 lbs)
Housing materials	
Top & side cover	aluminum, coated
Back	aluminum
Front, Xsample slot cover plate	polystyrene / butadiene

a In the stand-alone configuration (no Xsample or external measuring modules) the power consumption will not exceed 85 VA.



### **WARNING**

### Risk of electric shock

Connect only devices that comply with PELV (protective extra-low voltage) according to EN 61140 or with SELV (safety extra-low voltage) according to EN 60950 to any of the electrical interface connectors of the instrument.

**IMPORTANT:** Connect only Anton Paar equipment or equipment with a maximum power consumption of 40 W to the CAN interface. Otherwise the instrument will not work.

# A.3: Wetted Parts

The following materials are in contact with the samples and cleaning agents:

### DSA 5000 M

Material	Part
Borosilicate glass	density measuring cell
HTL 5 CR	solder (sound velocity cell)
PTFE	filling adapter
Stainless Steel DIN 1.4539 / UNS N08904	sound velocity cell

### Standard accessories

Material	Part
ETFE	adapter UNF ¼" Luer male
Polyethylene	waste vessel
Polypropylene/polyethylene	syringe 5 mL Luer
PTFE	standard filling and waste hose, adapter Luer cone, male Luer plug PTFE
Silicone	hose 4x6 mm silicone
Viton	hose 3x5 mm Viton

# Appendix B: Declarations of Conformity

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### **EU Declaration of Conformity**

(original)



The Manufacturer **Anton Paar GmbH**, Anton-Paar-Str. 20, A-8054 Graz, Austria – Europe hereby declares that the product listed below

Product designation: DSA 5000 M

**DSA 5000 M CK** 

Model: DSA 5000 M

Material number: 182706, 186723

(Declaration valid for instruments with one of these numbers on the type plate)

is in conformity with the relevant European Union harmonisation legislation. This declaration of conformity is issued under the sole responsibility of the manufacturer.

#### Electromagnetic Compatibility (2014/30/EU, OJ L 96/79 of 29.3.2014)

Applied standards:

EN 61326-1:2013
 Electrical equipment for measurement, control and laboratory use - EMC

requirements - Part 1: General requirements

The product is classified as a class B equipment and is intended for the use in industrial area.

#### Low Voltage Directive (2014/35/EU, OJ L 96/357 of 29.3.2014)

Applied standards:

EN 61010-1:2010 +A1:2019
 Safety requirements for electrical equipment for measurement, control and laboratory use - Part 1: General requirements

EN 61010-2-010:2014
 Safety requirements for electrical equipment for measurement, control and

laboratory use - Part 2-010: Particular requirements for laboratory

equipment for the heating of Materials

EN 62233:2008 Measurement methods for electromagnetic fields of household appliances

and similar apparatus with regard to human exposure

RoHS Directive (2011/65/EU, OJ L 174/88 of 1.7.2011)

Place and date of issue: Graz, 2022-04-28

DI Steffen Riemer, MBA Executive Director Business Unit Measurement DI Dr. Wolfgang Baumgartner
Head of Lab Density & Concentration
Business Unit Measurement

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## **UK Declaration of Conformity**



The Manufacturer Anton Paar GmbH, Anton-Paar-Str. 20, A-8054 Graz, Austria – Europe hereby declares that the product listed below

Product designation: DSA 5000 M

**DSA 5000 M CK** 

Model: DSA 5000 M

Material number: 182706, 186723

(Declaration valid for instruments with one of these numbers on the type plate)

is in conformity with all the relevant UK legislation

Electrical Equipment (Safety) Regulations 2016, 2016 No. 1101

Electromagnetic Compatibility Regulations 2016, 2016 No. 1091

Restriction of the Use of Certain Hazardous Substances in Electrical and Electronic Equipment Regulations 2012, 2012 No. 3032

complies with the designated standards:

- EN 61010-1:2010 +A1:2019 +A1:2019/AC:2019
- EN 61010-2-010:2014
- EN 61326-1:2013
- EN 62233:2008

This declaration of conformity is issued under the sole responsibility of the manufacturer.

Importer: Anton Paar Ltd, Unit F, The Courtyard, Hatfield Rd, St Albans AL4 OLA, United Kingdom;

Tel.: 03303 / 50 40 66

Fax.: 03303 / 50 40 68

Place and date of issue: Graz, 2022-04-28

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DI Steffen Riemer, MBA Executive Director

Business Unit Measurement

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Head of Lab Density and Concentration Business Unit Measurement