AL2021SC

H_2O_2-Monitor

Instruction Manual

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1 Safety

1.1 Safety Conventions

Warning:
A warning indicates information or instructions that, if not met can damage the instrument or even cause serious personal injury or death!

Note:
A note indicates important information or instructions that should be considered for operating the instrument!

This symbol indicates the need for protective eyewear!

This symbol indicates the need for protective hand wear!

1.2 Health and Safety

Warning:
Read the manual before installing and operating the instrument!
Observe warning labels!

1.3 Product Disposal

In conformity with European local and national regulations, electrical equipment users must now return old or end-of-life equipment to the manufacturer for disposal.

1.4 Electrical Safety

There are lethal voltages inside the instrument. Always disconnect AC line power before servicing the system.

1.5 Service and Repairs

Only personnel from Aero-Laser, its approved representatives and trained users are authorized to repair or maintain the instrument. Any attempt of unqualified persons to repair or maintain the instrument, can cause damage to the instrument and severe injuries.

Note:
Any attempt of unauthorized personnel to repair or maintain the instrument will cause the loss of warranty!

1.6 Chemicals

Some of the chemicals are hazardous. Read the material safety data sheets carefully and always wear your personal protective equipment.

Warning:
Store chemicals away from heat and protect them from temperature extremes!
Always review the material safety data sheet prior to handling chemicals!

2 Introduction

The AL2021SC is a completely automatic and continuously working H_2O_2 analyzer for gaseous and liquid samples. The detection technique is based on an enzymatic peroxidise reaction. It is sensitive to lowest concentrations of H_2O_2, less than 1ppb and linear up to 1ppm. All functions of the instrument are controlled by user-friendly software from a remote computer. The AL2021SC is easy to use, reliable and has a low need for maintenance.

***Fig. 2-1**: AL2021SC H_2O_2-analyzer. The inlet tubes in the front are used to supply the device with the necessary solutions.*

2.1 Necessary Items

Before starting the instrument make sure that you have the following items prepared:

- Laptop or PC with software installed (see chapter 3.2)
- Chemicals (see chapter 3.3.1)
- Laboratory equipment (see chapter 3.3.2)
- Cooling box or refrigerator to cool the solutions

2.2 Principle of Operation

The detection of peroxides is based on the liquid phase reaction of peroxides with p-hydroxyphenylacetic acid catalyzed by peroxidase. This reaction produces a fluorescent dimer that is excited at 325 nm by a UV-LED. The fluorescent light is detected between 400 and 420 nm. The detection of peroxides is based on the liquid phase reaction of peroxides with p-hydroxyphenylacetic acid catalyzed by peroxidase.

The above reaction is carried out in aqueous solution of H_2O_2 and other reagents. For the measurement of gaseous H_2O_2, this has to be trapped in aqueous solution first. This is achieved in a stripping coil by pumping air and a stripping solution (pH-buffered water free of H_2O_2) continuously at known flow rates. The air and liquid streams are afterwards separated in a glass separator and the solution is then analyzed for H_2O_2. The coil size and the flow rates of air and stripping solution are optimized for quantitative stripping of H_2O_2. During measurements the gas flow rate

is continuously monitored by a high precision flow controller. The concentration of H_2O_2 in air is finally calculated from the concentration in the aqueous solution and flow rates of sample gas and stripping solution.
H_2O_2 in liquid solutions can be measured directly. In this mode, stripping is not necessary and, consequently, either zero air has to be applied to the sample gas inlet or the internal zero trap has to be switched on. Automatic routine measurements require regular calibrations of the instrument. As liquid phase standards of H_2O_2 are unstable and have to be prepared freshly directly before use, the instruments can be equipped with an optional gas phase calibration unit. All the H_2O_2 molecules from this permeation gas source are transferred to the stripping system, thus giving known liquid phase calibration standards.

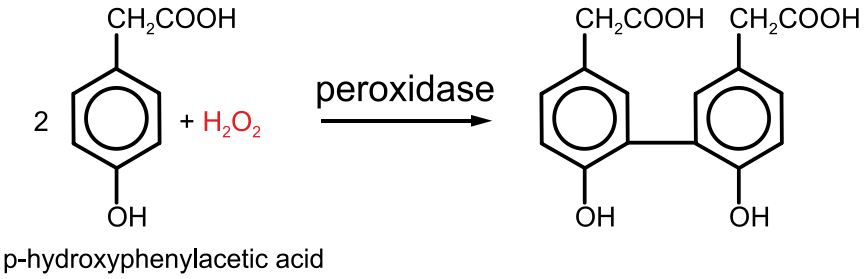


***Fig. 2-2**: Enzymatic reaction*

The technique is described in detail by Lazrus et al., Anal. Chem. 57 (1985) 917 and Lazrus et al., Anal. Chem. 58 (1986) 594.

2.3 Chemical Compartment

The instrument consists of a chemical compartment and an electronic compartment. The chemical compartment contains the reagent flow and reactor system, gas and liquid pumps, a stripping unit and an optional gas phase calibration source.
The components are described in the following.

2.3.1 Stripper

Gaseous samples and stripping solution are pumped through a stripping coil with flow rates of 1l/min and 0.5ml/min, respectively. The gaseous H_2O_2 is absorbed in the stripping solution. The stripping efficiency for H_2O_2 at temperatures below 40°C is better than 99%.
Then air and stripping solution are separated in a separator. For the measurement of external liquid samples, the stripping solution line can be switched to the external sample inlet by an internal solenoid valve, while zero air is used. The zero air is supplied internally by a very effective catalyst.

2.3.2 Reactor

In the reactor, the stripping solution, enriched with H_2O_2, is mixed with the fluorescent reagent. Additional NaOH solution increases the fluorescent intensity. To reduce the longitudinal mixing of the solution along the line of flow, the solution flow is segmented by air bubbles. After leaving the reaction coil and a glass debubbler, the sample passes the fluorimeter.

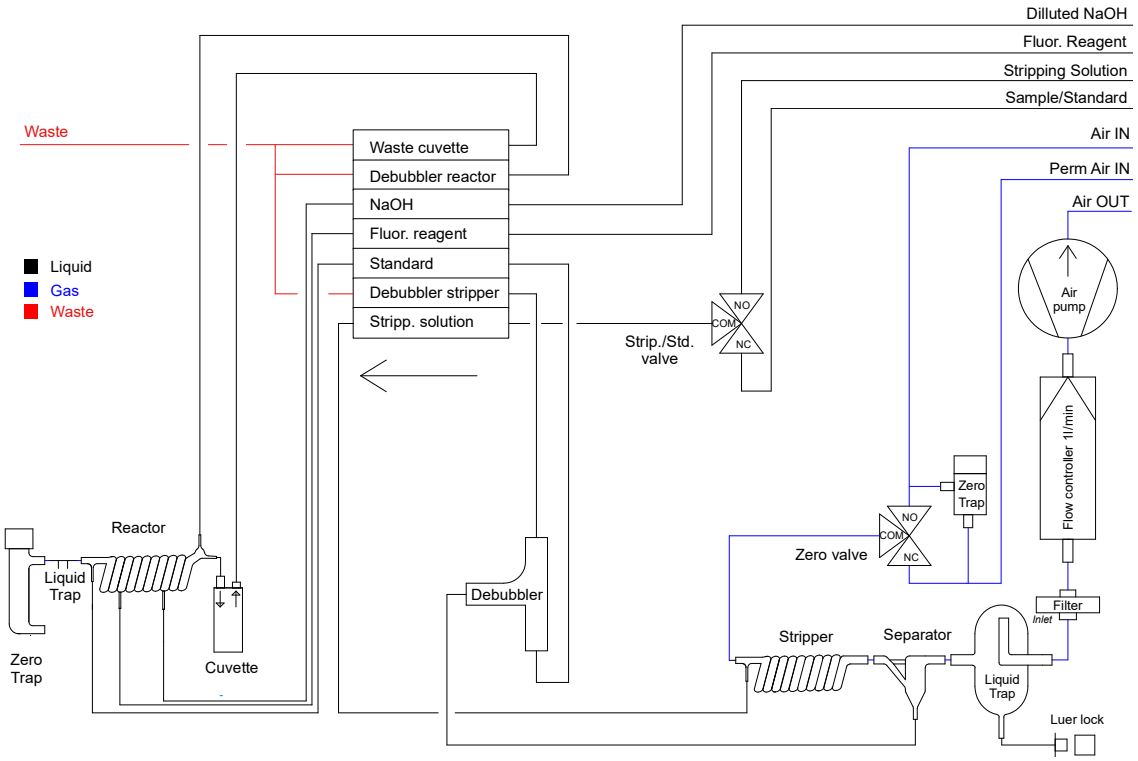


***Fig. 2-3**: Principle of detecting H_2O_2.*

2.3.3 Peristaltic Pump

The peristaltic pump contains 8 pumping cassettes, 7 are in use. The pump is electronically controlled for stable pump speed. The inner diameter of the tubing and the number of revolutions per minute determine the flow rate.

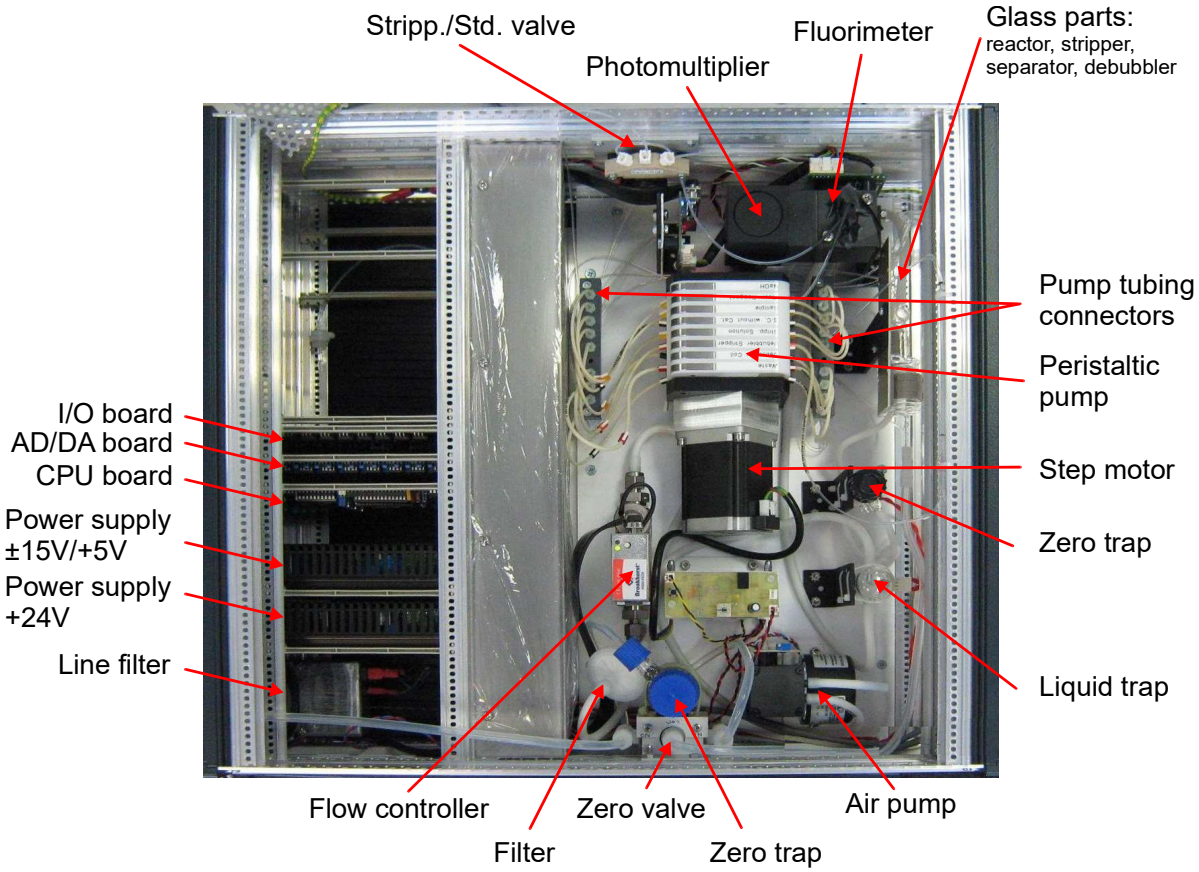


***Fig. 2-4**: Top view of the AL2021SC*

2.3.4 Mass Flow Controller

Gas phase measurement requires constant flow of air through the stripping coil. This is provided by using an air pump and a calibrated flow controller at 1L/min. The flow can be set in the Setup/System menu (see Chap. 5.6.5).

2.3.5 Permeation Source (optional)

The internal permeation source is used to provide a H_2O_2 gas standard to perform a gas calibration. It consists of a semi-permeable tubing immersed in an aqueous solution of 30% H_2O_2 (perhydrol) in a small flask. The temperature of the device is stabilized to a user programmable value between 14 - 30°C (see Chapter 5.6.6). The default value is 25 °C. After starting the instrument the first time or after refilling the permeation source, it takes approximately 24 hours to achieve stable permeation rates. To prevent damage, the AL2021SC is always shipped with an empty permeation source. Please fill it completely with approx. 60mL 30% perhydrol. For very low levels (~1ppb) use diluted perhydrol (1:20).

Warning:
Empty the permeation source with a Luer-syringe before shipment!

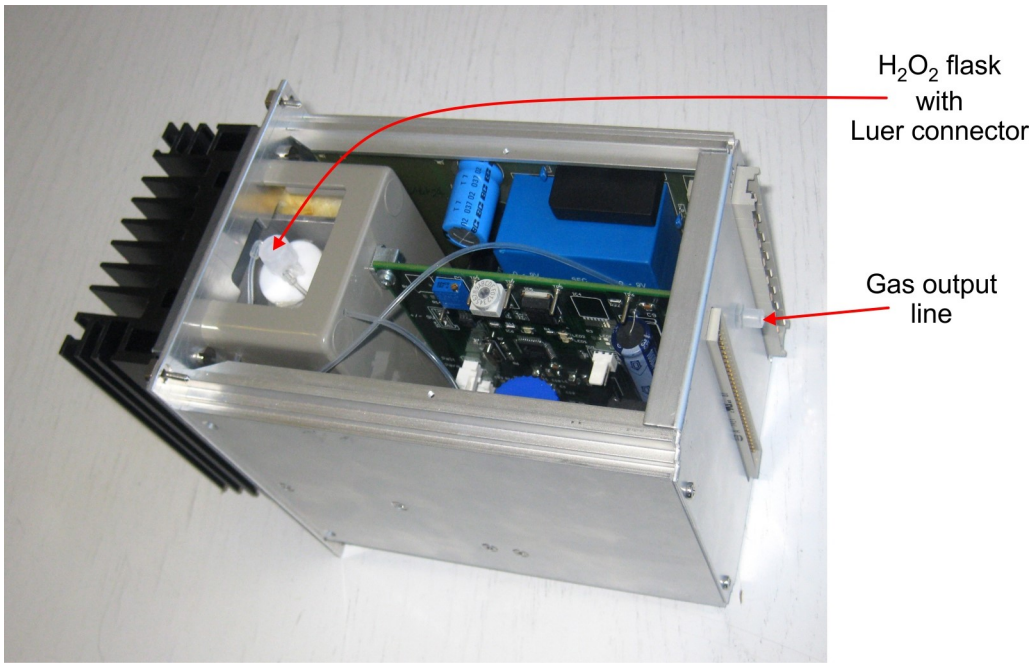


***Fig. 2-5**: Permeation source. The ports for filling the permeation source with perhydrol and the gas output line are labeled.*

2.4 Electronics Compartment

The electronics compartment contains power supplies, boards for signal conditioning and digitization and a micro computer, which is used to control the system and calculate the H_2O_2 concentration.

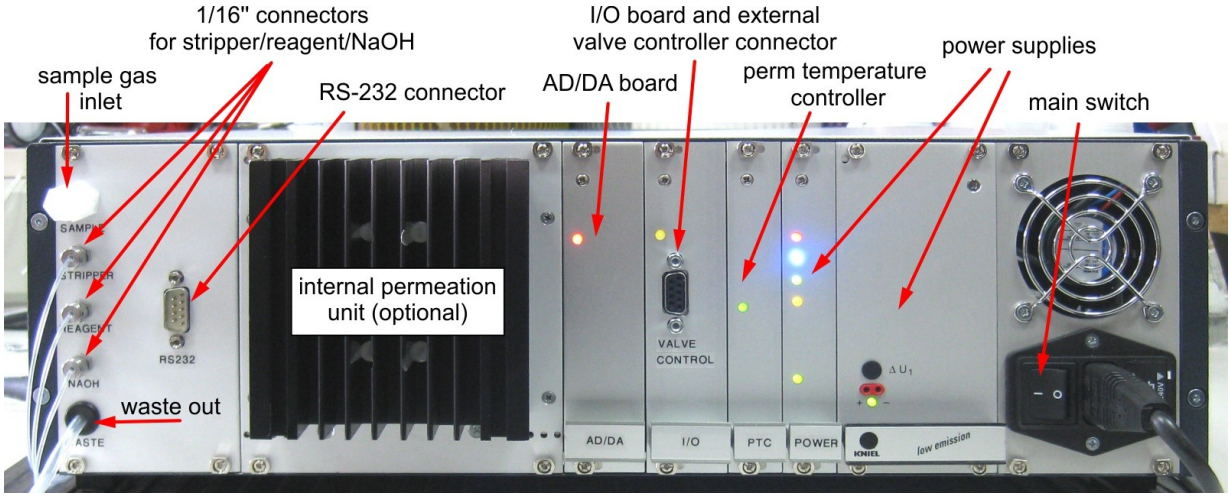


***Fig. 2-6**: Backview of the AL2021SC*

The correct operation of the electronic boards is indicated by blinking LEDs.

- AD/DA: red
- I/O: yellow
- PTC: green

LEDs indicate also that the voltage supplies are working correctly (constantly lit).

- +15V: red
- -15V: blue
- +9V: green
- +24V: yellow

2.4.1 Fluorimeter

After debubbling, the solution containing the fluorescent dimer is transferred to a cuvette inside the fluorimeter. The excitation takes place at 325nm and the sample fluorescence is detected at 400nm by a photomultiplier. The excitation light is provided by an UV-LED, which is stabilized by a reference photo diode.

2.4.2 UV-LED

The UV-LED produces the excitation radiation at 325nm. It is combined with a regulator for stabilization of the lamp intensity.

2.5 Specifications

- Provides absolute concentrations for H_2O_2 in gas- and liquid phase
- Semi automated calibration by liquid H_2O_2-standard or automatic calibration using internal H_2O_2 permeation source (optional)
- Automatic zeroing by internal zero trap

Linear detection range:	100ppt - 1ppm (gaseous) 300ng/l - 3mg/l (liquid)
Detection limit:	100ppt (gaseous) 300ng/l (liquid)
Noise:	2% full scale
Time resolution:	~90sec (10% - 90%), delay time ~300sec
Interferences from:	other Peroxides NO 100ppb / 30ppt H_2O_2 O_3 100ppb / 12ppt H_2O_2
No detectable interference:	acetone, methylamine, dimethylamine, n-butane, cis-2-butene, trans-2-butene, SO_2, PAN, NO_2, glyoxal, isobutane, isobutylene, 1-butane, HCHO, benzene, toluene, methanol, ions such as iodide, chloride, nitrate, bromide, phosphate, benzoate
Dimensions:	19” rack mount chassis 45cm x 19cm x 56cm
Weight:	20kg
Power requirement:	110/220VAC, 50/60Hz, 110W
Signal Output:	RS232 interface analog output (0V - 5V)

2.6 Consumptions

The consumption of chemicals depends on the speed of the liquid pump.
The below mentioned consumptions are estimated for 7 days (24/7 operation) at pump speed “C” (factory setting)

Consumption of solutions:

- ~ 5L stripping solution
- ~ 1.7L reagent
- ~ 1.7L diluted NaOH

Consumption of chemicals:

- 60ml 1 molar NaOH
- 5g potassium-hydrogenphtalate
- 10mg ethylene-diamine-tetraacetic acid
- 425mg p-hydroxyphenylacetic acid
- 30mg peroxidase (6kU/L)
- 0.1ml HCHO solution
- 1ml 30% H_2O_2 solution (for calibration)
- 8.4L H_2O_2 free water

2.7 Integrated data storage

The instrument can store the current parameters (concentration, signal, ..) on an USB-stick that is inserted into the USB-slot on the front panel. As default, data is logged every second. The data logging rate can be set to the user's requirement. The file naming and the parameters that are stored can be selected in the Setup/File Options menu (see Chap. 5.6.3).

Warning:
Always stop data logging before the USB-stick is pulled out from the front panel! Removing the USB-stick while data is logged could result in a loss of data and/or a damage of the USB device.

3 Getting Started

3.1 Unpacking the Instrument

Open the metal box carefully and lift the contents out. Check if the content of the box is undamaged and conforms to the packing list. After unpacking the instrument, keep the metal box for safe shipping.

Note:
Always ship the instrument in its original packaging to prevent damage!

3.2 Installing Software

To log data to a data base, the software SqeezeDat needs to be installed on a remote computer. SqeezeDat and other necessary installation packages are provided on the USB stick that accompanies the AL2021SC.

Note:
To install the software you need an administrator account!

3.2.1 SqeezeDat

Insert the DVD in your PC or laptop and follow the installation instructions.

Note:
You have to install .NET Framework 1.1 and .NET Framework Update 1.1!

You need a registration key to start the program. Send a screenshot with the customer name and base key to info@aero-laser.de.

There is a help file which describes how to set and use SqeezeDat and SqeezeDatExport.

Before installing Sqeezedat, the MS SQL Server has to be installed from the DVD.

3.2.2 Driver

As most of the new laptops have no RS232 port, an USB adapter is enclosed.
You have to install the driver from the CD which is enclosed.

3.3 Preparation of Solutions

Reagent solutions can be kept for several weeks if properly stored. The fluorescent reagent solution has to be stored cool at temperatures between 4°C and 10°C. The stripping solution must be stored dark.
The AL2021SC is designed to measure very low concentrations of H_2O_2 but it is also capable of higher concentrations. For concentration above 100ppb (~300µg/L liq.) use stripping solution instead of NaOH.

Warning:
Read the material safety data sheets carefully and always wear your personal protective equipment!

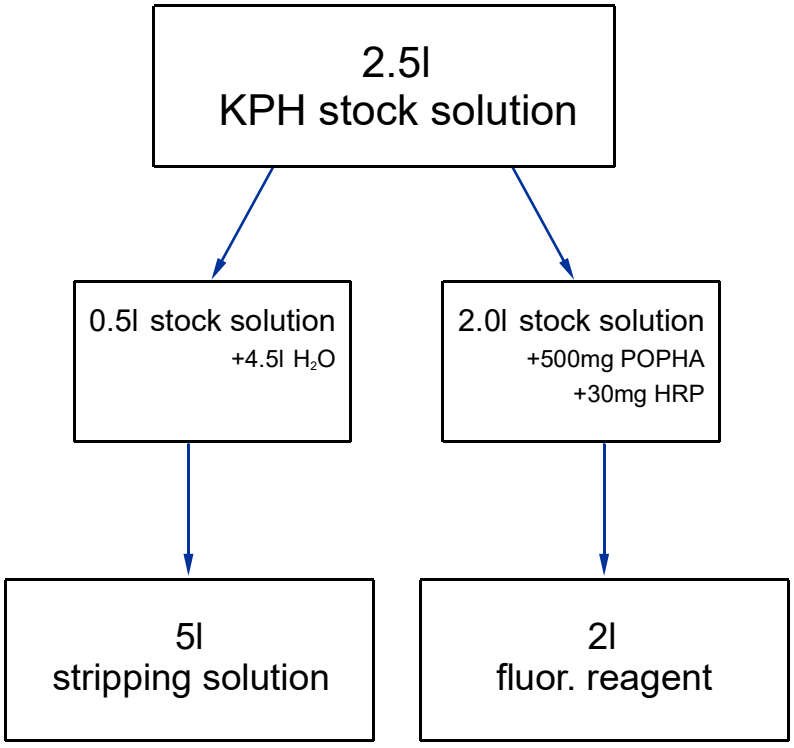


***Fig. 3-1**: Preparation of reagents for about 1 week of continuous operation. 5l KHP stock solution is prepared. This stock solution is used to prepare the fluorescent reagents.*

3.3.1 Necessary Chemicals

- 1 molar NaOH (MERCK, Order No. 1.09137.1000)
- potassium-hydrogenphtalate (MERCK, Order No. 1.04874.1000)
- ethylene-diamine-tetraacetic acid (MERCK, Order No. 1.08417.0100)
- p-hydroxyphenylacetic acid (MERCK, Order No. 8.20690.0025)
- peroxidase P8375 (SIGMA, Order No. P8375-5KU)
- H_2O_2 solution (30%) (MERCK, Order No. 1.07209.0250)
- HCHO solution (37%) (MERCK, Order No. 1.04003.1000)
- H_2O_2 free water (MERCK, Order No. 1.16754.9010)

Note:
Only use the highest purity grade of chemicals to prepare the solutions!

3.3.2 Necessary Items

- magnetic stirrer
- balance and microbalance (0g – 100g)
- clean 2l and 5l glass bottles with caps
- 100ml glass flasks
- 25ml pipette
- 50ml pipette
- 100ml pipette
- 10µl - 100µl pipette with tips
- 100µl - 1000µl pipette with tips

3.3.3 KHP Stock Solution

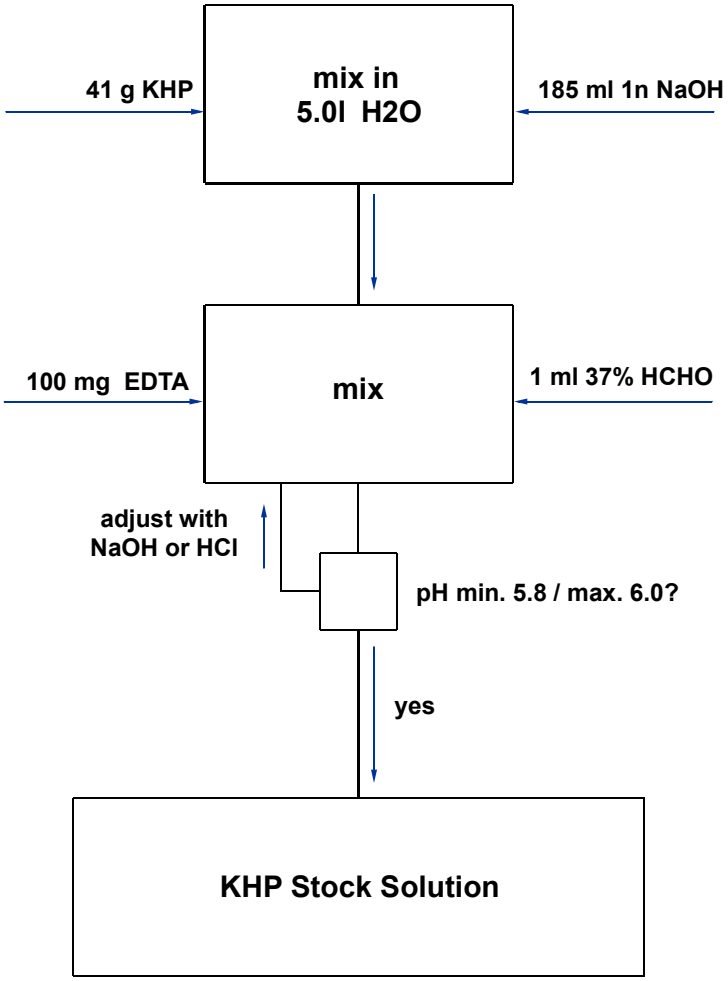
5 litres are prepared from:

- 5l cold, H_2O_2 free water
- 41g potassium-hydrogenphtalate
- 185ml 1 molar NaOH
- 100mg ethylene-diamine-tetraacetic acid (EDTA)
- 1ml 37% HCHO solution

Note:
Use cold water for preparing the solutions!
Store the water overnight in a refrigerator before preparing the solutions!

41g potassium-hydrogenphthalate (KHP) and 185ml 1n NaOH-solution are diluted in a 5l volumetric flask with deionized water. 100mg ethylene-diamine-tetraacetic acid (EDTA) and 1ml 37% HCHO solution are added to this solution. The pH value of this

solution should be between 5.8 and 6.0. The pH-value can be adjusted by addition of NaOH or HCl solutions.



3.3.4 Fluorescent Reagent Solution (mix 2 days before measurement)

2 litres are prepared from:

- 2l KHP stock solution
- 500mg p-hydroxyphenylacetic acid
- 30mg peroxidase P8375

Dissolve 500mg p-hydroxyphenylacetic acid (POPHA) and 30mg peroxidase in 2l of the KHP buffer solution (4350 units/l of peroxidase Sigma 8375).

3.3.5 Stripping Solution

5 litres are prepared from:

- 500ml KHP stock solution
- 4.5l deionized water

Dilute 500ml of the KHP stock solution with 4.5l of deionized water.

3.3.6 NaOH

2 litres are prepared from:

- 50ml 1n NaOH
- 1.95l deionized water

50 ml 1n NaOH-solution are diluted with deionized water to 2l.

3.3.7 Standard Stock Solution

1 litre is prepared from:

- 1ml 30% H_2O_2 solution
- 1l cold, deionized water

A 10^{-2} molar standard stock solution is prepared by diluting 1ml 30% H_2O_2 solution to 1l with cold water. The H_2O_2 concentration of the stock solution is determined by titration. This solution has to be kept in a refrigerator and should be titrated on a regular time basis. Under these conditions the solution can be stored for several months.

3.3.8 H_2O_2 Standard Solution

The 10^{-2} molar H_2O_2 standard stock solution has to be diluted with cold stripping solution. Highly diluted solutions of H_2O_2 (10^{-6} - 10^{-8} molar solutions) in water are stable only for short time intervals even if stored in a refrigerator. They always have to be prepared freshly before use.

Note:

The AL2021SC is designed to measure very low concentrations of H_2O_2 but it is also capable of higher concentrations.

For concentrations above ~100ppb (eq. to ~300µg/L liq.) use stripping solution instead of NaOH.

4 Installation

4.1 Mounting the Instrument

As the instrument contains liquid reagents, which have the potential to leak, never install it directly over other rack-mounted electronic equipment. Never tilt the instrument at an angle exceeding 45°.

Make sure there is enough room to reach the inside and the back of the instrument.
If a permeation source is used, make sure there is enough space for air circulation at the cooler of the permeation source.

Locate the cooling box below the instrument. The length of the inlet tubes should not exceed 1.5m.

Warning:
Do not locate the liquid containers above the device!

For concentrations below ~100ppb (eq. to ~300µg/L liq.) connect the stripper input line with the stripping solution using the tygon tube and the NaOH input line with the diluted NaOH.

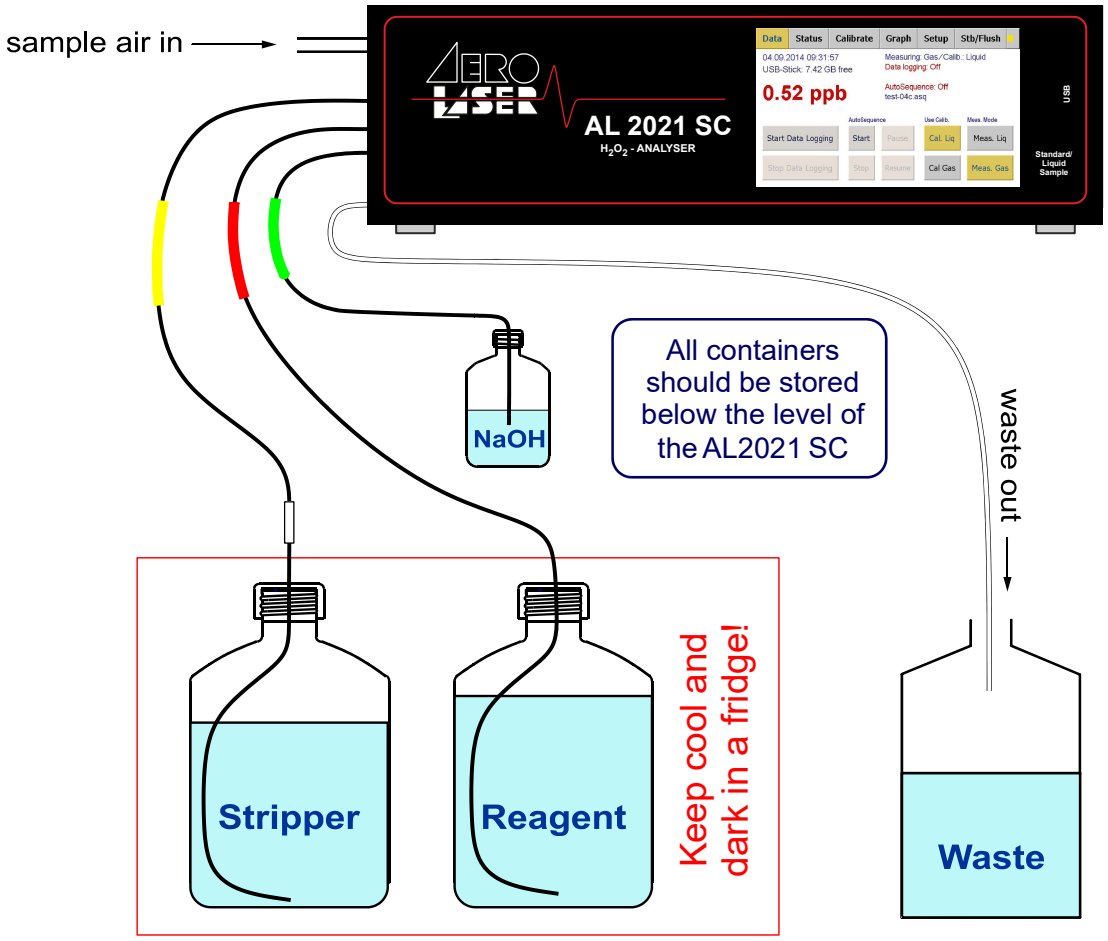


***Fig. 4-1**: The containers which contain the solutions should be stored below the AL2021SC. For easy connection the tubes are marked with colors.*

For concentrations above ~100ppb (eq. to ~300µg/L liq.) use the stripping solution instead of the NaOH. Connect the NaOH input line with the stripping solution using the Y-piece.

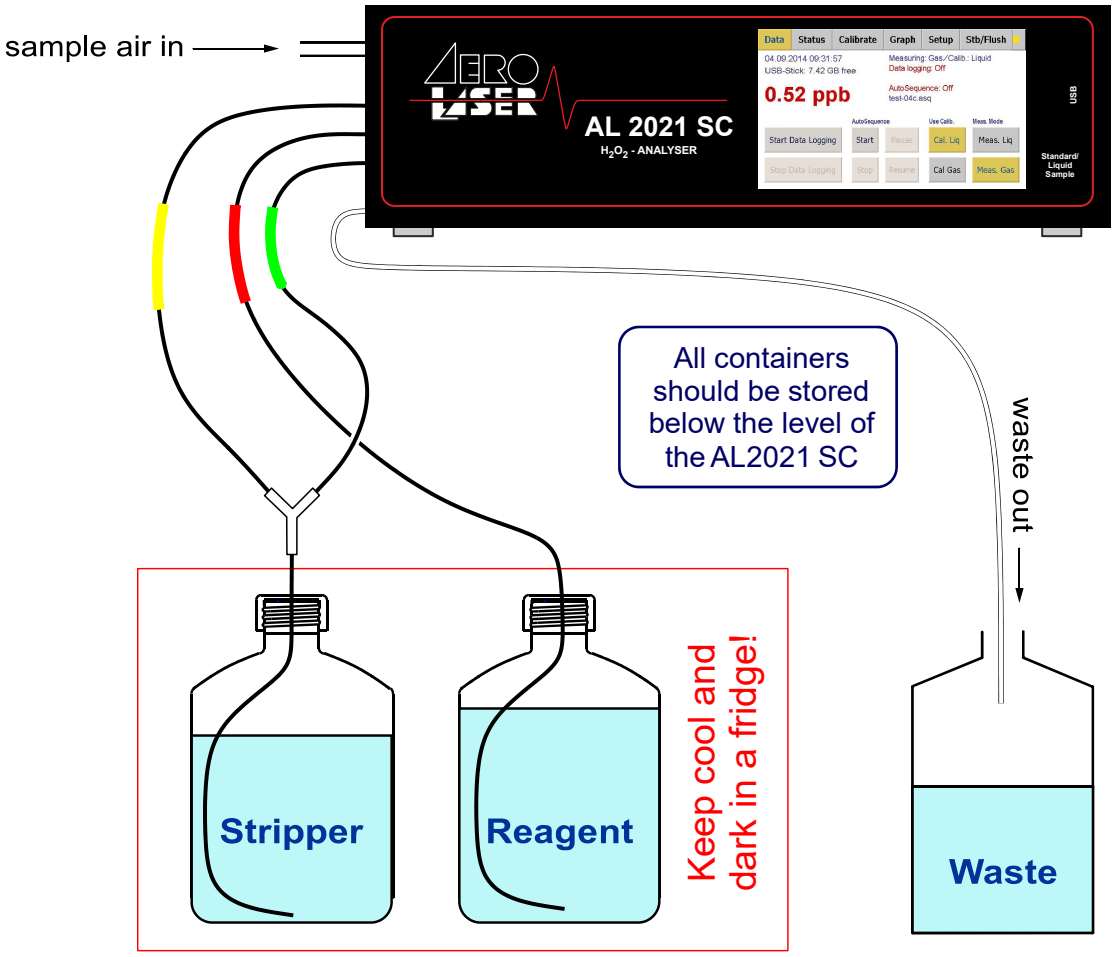


***Fig. 4-2**: The NaOH input line and Stripper input line connected with the stripping solution via a Y-piece. This configuration is used for the measurement of high H_2O_2 concetrations.*

4.2 Electrical Connections

Make sure you are using the correct voltage (110V or 220V) marked on the rear side of the instrument.
Connect the instrument to an AC power supply.

Warning:
Always run the instrument with the correct voltage!
A wrong supply voltage may destroy the instrument!

There are two RS232 serial ports. The first one is on the front of the device labeled “REMOTE” and the other one is on the back panel of the CPU.
Use the null modem cable to connect the remote output to your PC or laptop.

4.3 Testing

Warning:
For testing the instrument do not connect it to the solutions!

- Open the cover of the instrument
- Switch on the main power
- Check if the LED's of the 24 Volt power supply and 15 Volt power supply glow
- Check if the lamp voltage reaches 3 Volt
- Check if the pumps work

5 Operation

The AL2021SC is operated by the touch screen on the front panel. All operation can also be done remotely via the RS232 interface. See Chapter **Fehler! Verweisquelle konnte nicht gefunden werden.** for a detailed description of the commands. The touch screen is optimized for the use with fingers directly, but it can also be used with any pen or similar.

Note:
The values shown in the screenshots are only examples!

5.1 Touch Screen Controls: General layout

The layout of the touch screen is divided into a menu bar (grey, on top) and an info window/dialog with white background. The menu has six main options (grey buttons on top). The option currently active is highlighted in orange. By pressing one of the menu buttons it can be switched easily between the menu options.

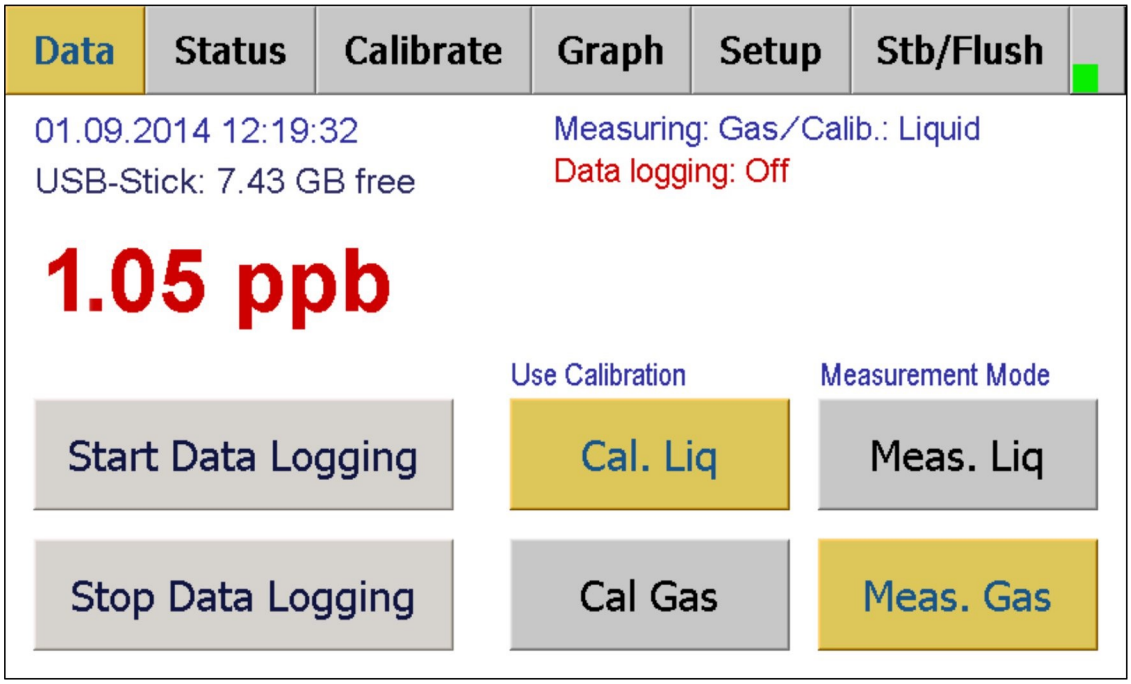
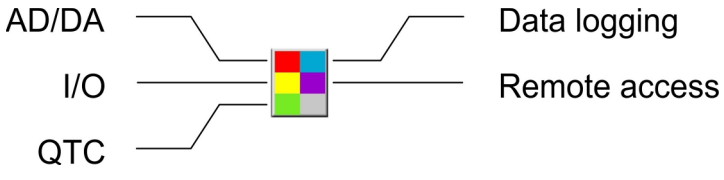


Fig. 5-1: *Layout of the AL2021SC touch screen. The screen has six main menu options (grey buttons on top). The option currently active is highlighted in orange.*

On the upper right corner of the touch screen, there is a LED display indicating the operation of the instrument. Every time there is an internal or external data transmission between the components of the instrument or a remote PC, one of the LED segments is lit. The meaning of colors is shown below:



5.2 Menu: Data

The instruments main reading is displayed on the Data screen in large red numbers. For liquid samples the concentration is given in µg/L, for gaseous samples in ppb. The unit for gaseous measurements can be changed by the user, see Chapter 5.6.5. If the instrument is not calibrated the raw signal is given in Volt. Above the concentration, additional information is displayed. Left: The current time and the free space on the USB stick, if any. Right: Measurement and calibration status and status of the data logging.

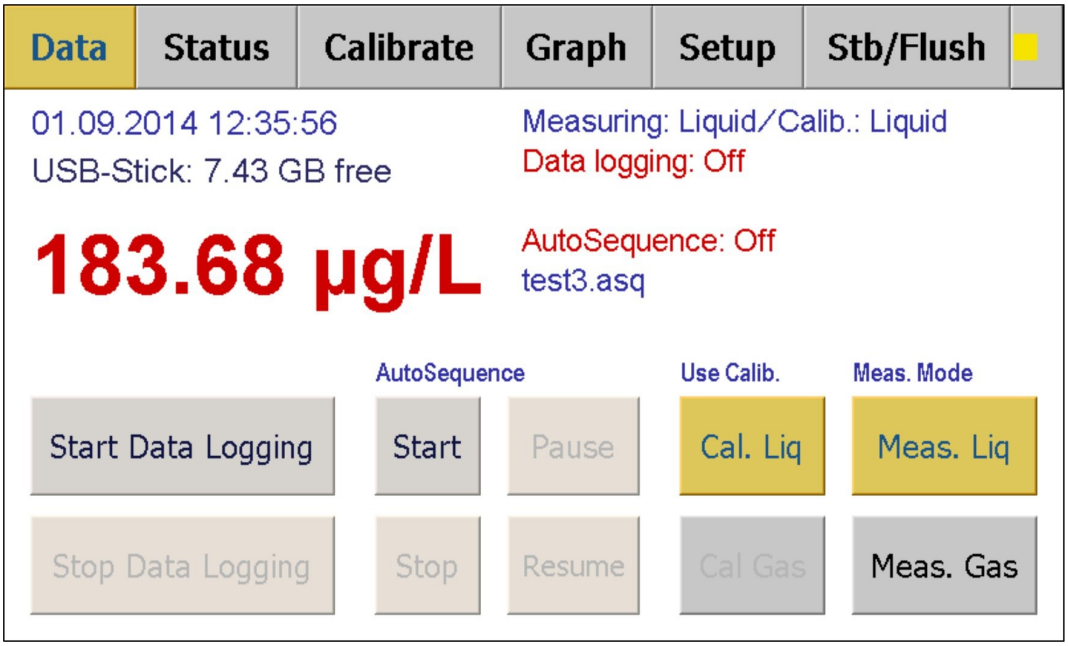


Fig. 5-2: *The Data screen shows the Signal in Volt, if the system is not calibrated, or the concentration (ppb for gaseous samples and µg/L for liquid samples). Buttons for changing the measurement mode, and the calibration mode (only if a permeation unit is installed) are located here. If AutoSequences are activated, four additional buttons are shown.*

The data menu looks different depending on the features of the instrument. If a permeation module is present, two buttons are present to switch between the calibration modes. If AutoSequences are activated four additional buttons are shown to control the AutoSequence (see Chapter 5.6.2).

Data Logging

If an USB-stick is inserted into the USB slot, data logging can be switched on and off by the buttons "Start Data Logging" and "Stop Data Logging". The file name is generated automatically from the time stamp, when the data logging is started with a name prefix that can be set in the Setup menu (see Chap. 5.6.3).
If data logging is activated, the data logging status text appears in green and the file name is displayed (see Fig. 5-3). Otherwise the reading is "Data logging: Off" in red.

Warning:
Always stop data logging before removing the USB-storage device!
Otherwise a loss of data could occur.

Measurement Mode
The AL2021SC can be set to measure liquid or gaseous samples by the "Meas. Liq" or "Meas. Gas" button. The currently pressed button is highlighted in orange and the current mode is given in the status line as "Measuring: Liquid/..." or "Measuring Gas", respectively. The unit of the concentration reading is set accordingly.

Note:
After switching between liquid or gaseous measurement modes, it needs several minutes until the reading is reasonable!

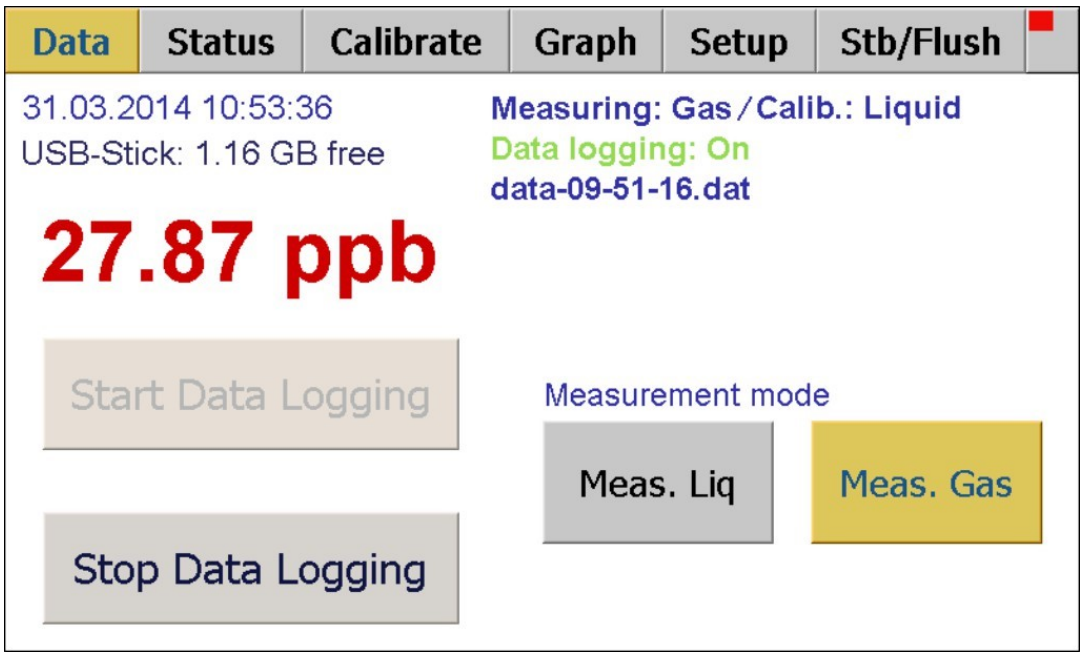


Fig. 5-3: *The data screen for instruments without permeation unit.*

Calibration Mode
The appearance of the Data screen depends on whether a permeation unit is installed in the instrument or not. If a permeation unit is present and a valid gas calibration exists, the calibration can be selected by the Calibration mode buttons. The active calibration is indicated by a highlighted button and in the status text ".../Calib.: Liquid" or "Calib. Gas". Calibrations can also be stored to and loaded from files. For details see Chapter 5.6.1.

Note:
Automatic gas calibrations are only available, if a permeation source is installed!

AutoSequence
An automatic sequence of commands is coded into a so-called AutoSequence. For instance, an AutoSequence can automatically schedule calibrations (if a permeation unit is installed) and zeroings during field campaigns. Another example is the automatic switching between gas samples, e.g. from test chambers according to the appropriate standards (requires a valve controller).
Each AutoSequence consists of a series of commands that are executed after each other. Each command can be either started at a fixed time or consecutive after the last command. To each command a wait time can be assigned. The AL2021SC has a simple and intuitive editor for those sequences. For a list of the available commands and options and on how to edit AutoSequences see Chapter 5.6.2.
The pressing on of the four buttons the following will happen:

Start: Resets the internal counter of the AutoSequence. If the AutoSequence has no fixed starting time, it will start immediately. Otherwise it will be scheduled to start with the command having the next fixed starting time after the time of starting. If there is no such command, it will schedule the AutoSequence until the first command of the next day.

Stop: Resets the internal counter of the AutoSequence and terminates it. The instrument remains in a state according to the last command.

Pause: Interrupts the AutoSequence without resetting its internal counter.

Resume: Resumes a pause AutoSequence at its current position.

5.3 Menu: Status

The Status screen displays several parameters of the instrument in addition to the main parameters that are also displayed on the Data screen:

Pump Speed: Speed of the peristaltic pump from 0 to F. This parameter codes the pump speed. For the definition see the Appendix 9.3.
The peristaltic pump speed can be set in the Setup/System menu (see Chap. 5.6.5).

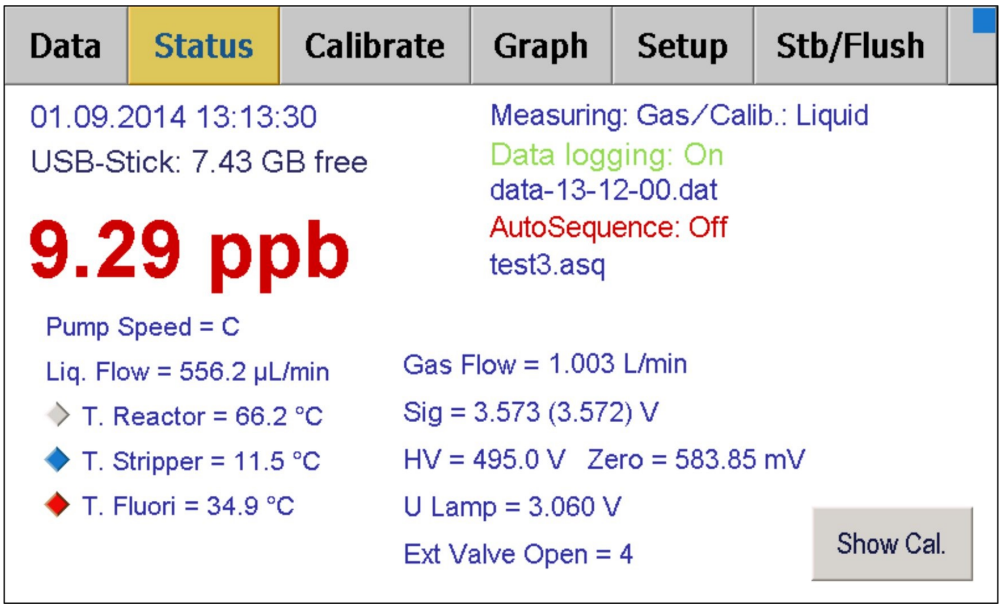


Fig. 5-4: *The status screen displays several parameters of the instrument.*

Gas Flow: shows the current gas flow in L/min measured by the in-built flow controller. The set-point can be set in Setup menu (see Chap. 5.6.5):

Liq. Flow: shows the current flow of the stripping solution or liquid sample in µL/min measured by the in-built liquid flow meter. The liquid flow changes when the peristaltic pump speed is changed. Please note that the stripper flow is not measured correctly if the peristaltic pump is set to a speed of "E" or above.

Sig: shows the current signal in Volt measured by the fluorimeter
. The value in brackets shows the average calculated by the internal ring buffer, This value is taken for the calculation of the concentration. The ring buffer size (in seconds) can be set in the Setup menu (see Chap.5.6.6).

Internal Temperatures: Here the temperatures of the Reactor, the Stripper, the Fluorimeter and, if installed, the Permeation source are given in centigrade. The signs on the left show, whether the unit is heating (red), cooling (blue) or inactive (grey).

HV/Zero: shows the current high voltage (HV) of the photomultiplier in the fluorimeter and the zero value. These values are determined by the calibration procedure. For testing purposes the HV can also be set manually (see Chap. 5.6.1).

U Lamp: shows the reference voltage of the UV-lamp in the fluorimeter in V. This value should be around 3 V. A value significantly below 3 V indicates a broken lamp.

Ext Valve Open: Shows the external valve currently open. Requires a valve controller.

Show Cal. Params: The complete set of current calibration parameters is shown in an info window. See a description of those parameters below. By pressing on the info window, it disappears.

Status:	Liquid Calibration	Setpoint HV:	634.3 V
Last Cal:	01.04.2014 14:57:12	Sens:	14.83 mV/(µL/min)
Last Zero:	---	Zero:	1042.09 mV
Standard Liq:	200.0 µg/L	Standard 4V:	4.008 V
Stripp Flow:	554.5 µL/min	Perm Rate:	10.30 ng/min
Stripp Eff:	1.000		

Fig. 5-5: *The Calibration Info Window shows all calibration parameters.*

Status: Can be "Not Calibrated", "Liquid Calibration", or "Gas Calibration"(*).

Last Cal: Date and Time of the last Calibration.

Last Zero: Date and Time of the last stand-alone Zeroing. This value is reset every time a new calibration is made.

Standard Liq: Concentration of the liquid standard in µg/L.

Stripp Flow: Flow of the stripping solution, determined during calibration (average).

Stripp Eff: Stripping efficiency. To be set in the Setup/Calibration menu.

Setpoint HV: High voltage of the photomultiplier in the fluorimeter. Set by the calibration routine so that the output voltage is 4.0V when measuring the standard.

Zero: Zero level of the system: The output signal of the fluorimeter if no formaldehyde is present in the sample.

Standard 4V: The real 4.0V output signal, when measuring the standard. Should be very close to 4.0V.

Perm rate: Permeation rate: output of the permeation unit. This value is determined in the Liquid Calibration routine, and used as gas standard in the Gas Calibration routine (*).

(*) Only displayed if a permeation unit is installed.

5.4 Menu: Calibrate

The AL2021SC is calibrated by a two-point calibration procedure, consisting of the determination of a well known standard and the zero level. The stripper speed is also measured during calibration.
Calibrations and zeroings are started in the Calibrate menu. An isolated determination of the stripper flow is also possible. The "Start Gas Calibration" option is only available, if a permeation unit is installed. All processes are automatic and can be terminated by the "Terminate" button. The liquid calibration requires at least 50 mL of liquid standard connected to the standard input line on the front panel of the

instrument. The concentration of the standard, the start value for the high voltage of the photomultiplier and the stripping efficiency are set in the Setup menu (see Chap. 5.6.1). For the calculation necessary for preparing the standard see Chap. 5.6.7.

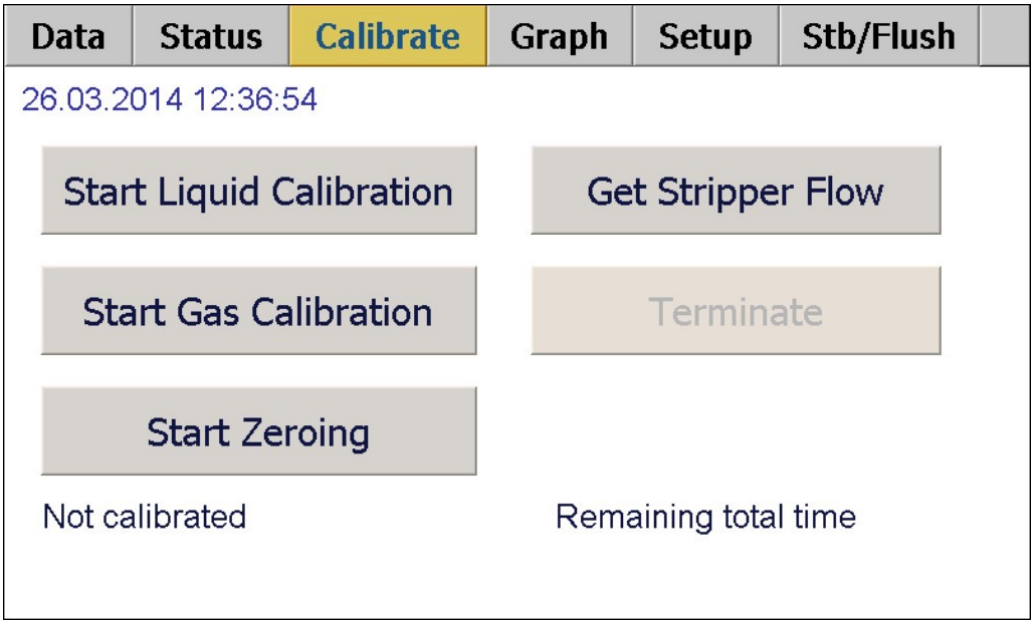


Fig. 5-6: *The Calibrate screen is used to start calibrations and zeroing.*

The state of the calibration procedure and the remaining time for the current step, are given on the bottom of the screen.
The calibration procedure consists of three parts: Measuring the concentration of the standard, measuring the zero voltage and getting the stripper speed. If a permeation unit is installed, the permeation rate is also determined.
When the standard signal is stable, the high voltage of the photomultiplier is adjusted in a way that the output signal is exactly 4.0V. This is called regulation.
The figure below shows the calibration sequence in principle. During the "check for stabilisation" phases, the instrument determines the signal to noise ration and the curve gradient continuously. The next step is only performed when those values are below a certain limit, which can be set in the Setup menu (see Chap. 5.6.5).
The meaning of the time constants in the figure below is:

t_{std}:	wait time for standard signal
t_{stabi}	time-out for stabilisation (applies for std, zero, perm)
t_{reg}	time-out for regulation
t_{av}:	averaging signal (applies for std, zero, perm)
t_{lqf}	averaging the liquid flow meter, determining stripper speed
t_{zero}:	wait time for zero signal
t_{perm}:	wait time for permeation source

The above wait times and time-outs are set in the Setup/Wait Times section of the menu (see Chap. 5.6.2)

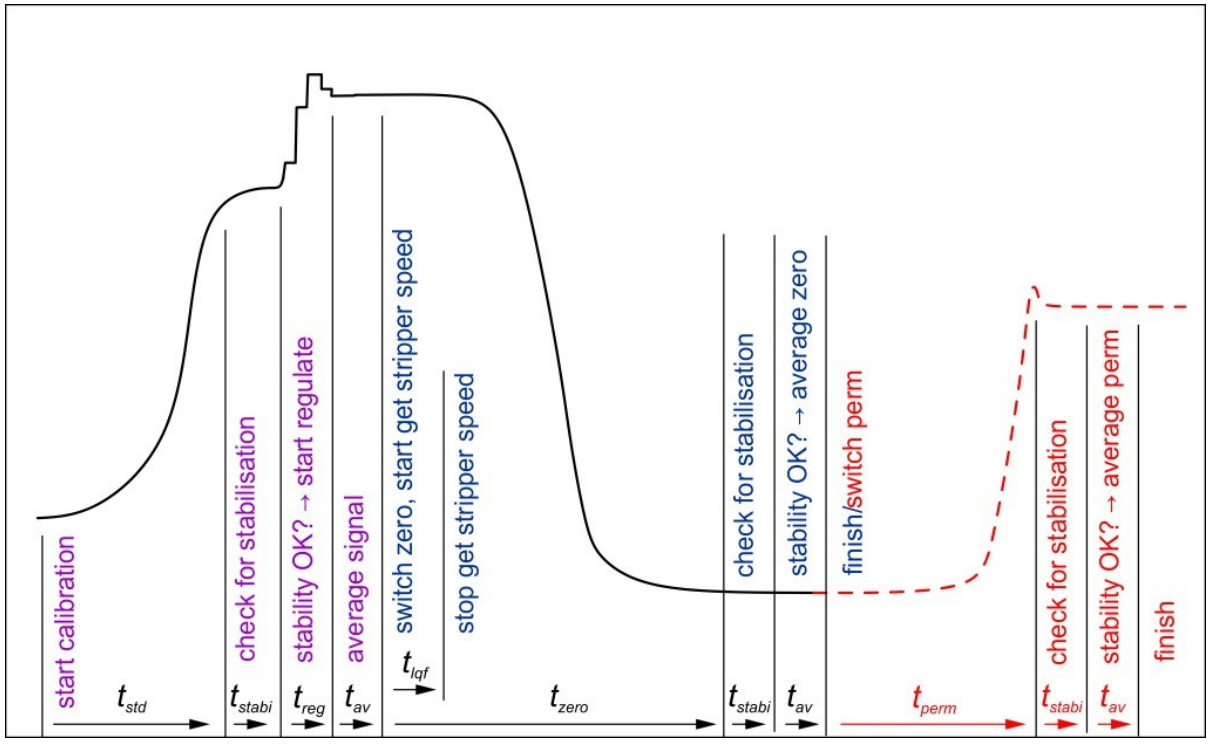


Fig. 5-7: *Typical calibration curve. The violet and blue sections show the complete calibration procedures. The red section only applies if a permeation unit is installed. The blue section shows the procedure, which is performed during zeroing.*

When performing a zeroing, only the section marked in blue is performed. The stripper speed is also measured during zeroing.

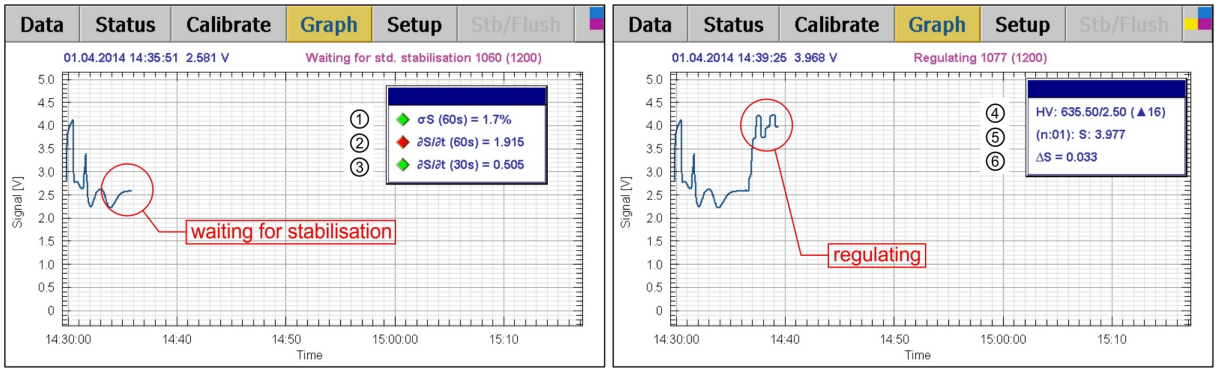


Fig. 5-8: *Info windows displayed during calibration. Left: stability criteria. Right: regulation parameters.*

During calibration two special windows appear on the Graph, displaying the following values:

Waiting for Stabilisation	Regulation
① standard deviation within t_{eval}	④ current HV/ ΔHV (regulation time)
② long time gradient (t_{eval})	⑤ (n: regulation time step): Signal [V]
③ short time gradient (t_{eval} /2)	⑥ ΔS = 4.0 - Signal [V]

The stability limits, evaluation time (t_{eval}) and regulation time, can be set in the Setup menu (see Chap. 0). The info windows can be turned on/off in the Setup menu (Calibration: option "verbose mode" (see Chap. 5.6.1).

5.5 Menu: Graph

The on-display graph is intended as a helpful tool to easily view the development of a parameter with time. It is not a replacement for an external data logging and displaying software. Some important parameters can be visualized as a plot vs. time. New data is written to the plot every second. There are four parameters available for plotting:

- Concentration
- Signal
- Gas Flow
- Stripper Flow

The parameter to be displayed can be set in the Setup/Graph menu. If the option "Automatic" (default) is selected, the signal in volt is plotted during calibration and the concentration is shown after the calibration is finished. Information on the progress of the calibration is displayed in a text line on the right above the plot. The parameters "Gas Flow" and "Stripper Flow" can be displayed for tracing the stability of the system. Please note that the stripper flow is not measured correctly, if the peristaltic pump is set to a speed of "E" or above.
During calibration an extra window inside the plot is shown, displaying details of the current process: Stabilization of the signal, zero, or perm signal, and during regulation. These windows can be dragged using the blue bar on top if they are overlaying the data plot (see Fig. 5-8).

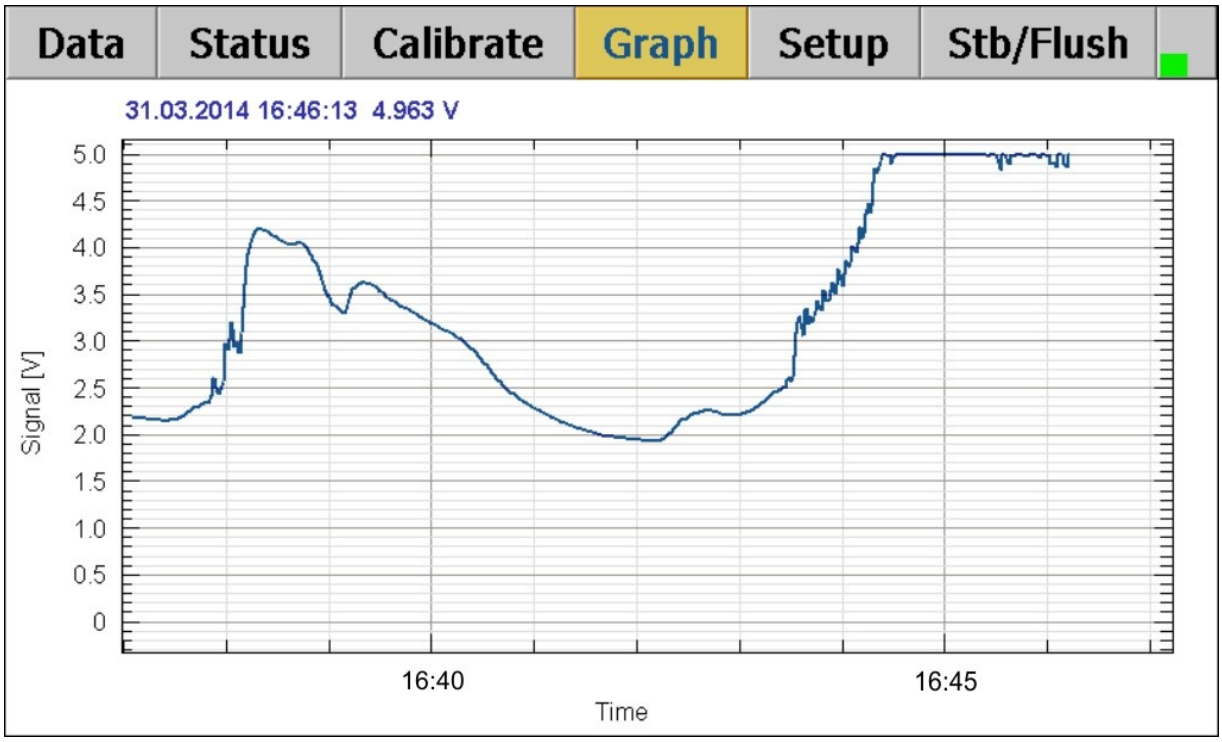


Fig. 5-9: *The Graph screen displays parameter vs. time, here: the signal in V.*

The data range (y-axis) and the time range (x-axis) can be set almost free in the Setup/Graph menu (see Chap. 5.6.4). Due to the limited computing power of the CPU, the maximum time range is restricted to 24 hours.

5.6 Menu: Setup

The Setup Menu is divided into 8 sections. Some of the sections have sub-sections. The Admin section is only accessible with a password. The last section, "Calculate Standard", contains a convenient calculator for converting gas concentrations into the corresponding liquid concentrations and vice versa. This is necessary for the preparation of a liquid standard for calibration (see Chap. 5.6.7).

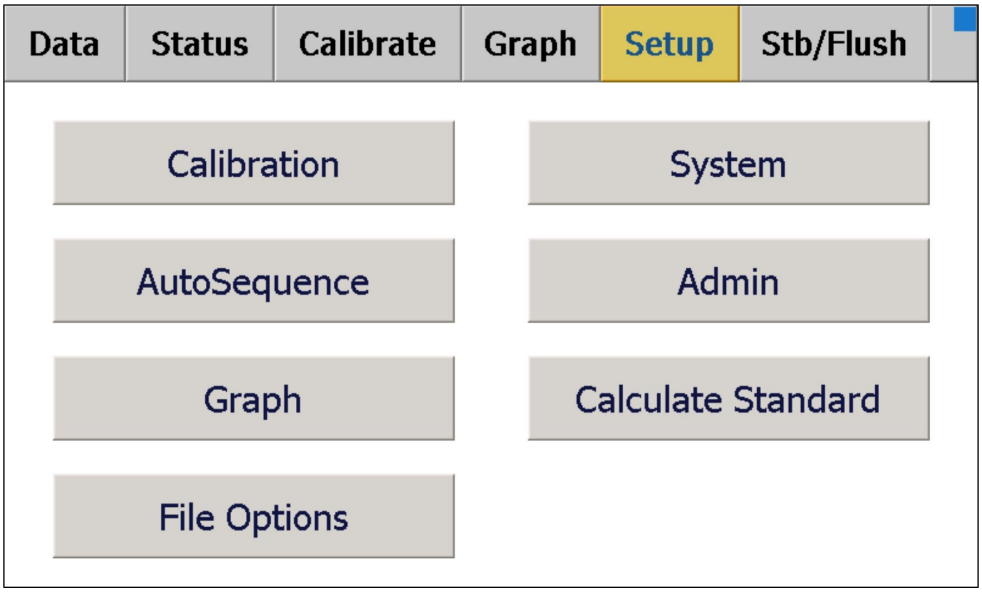


Fig. 5-10: *The Setup Menu. The "Admin"-section requires a password.*

5.6.1 Setup: Calibration

This menu is organised in three parts. The first part "Parameters" contains the parameters, which must be set before starting a liquid calibration. In the second part "Wait times" the wait time for each step of the calibration is set. All time constants are in seconds. In the third part "Calibration: More..." advanced settings functions for storing and loading calibrations are found.

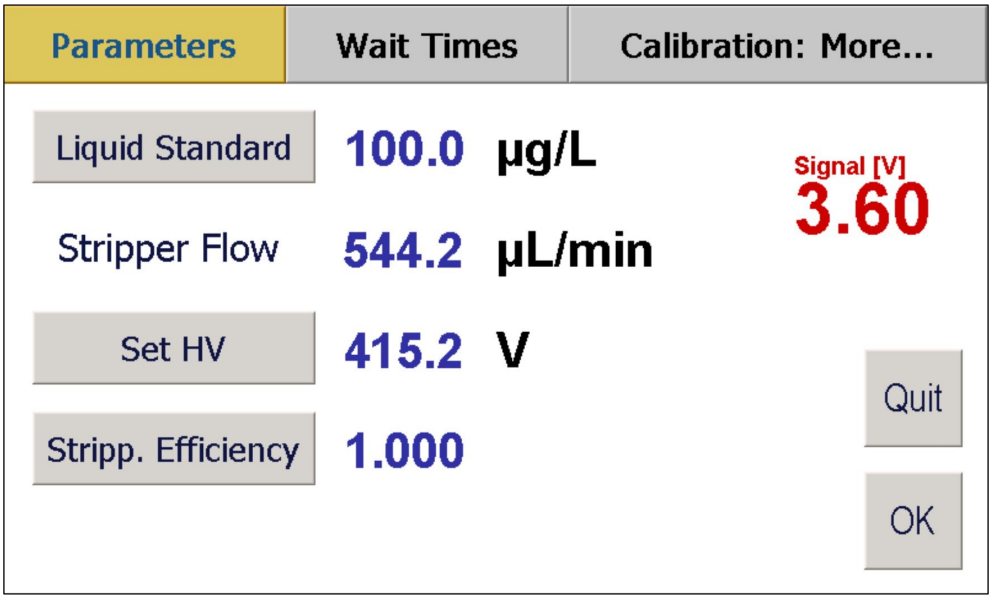


Fig. 5-11: *Part 1 of the Setup/Calibration menu contains the general settings necessary for calibration.*

The calibration is a two-point calibration, using a well known standard and the zero. The standard has to be provided by the user, the zero signal is obtained by using the internal zero-trap. During each zeroing, the stripper flow is determined.

Part 1 (Parameters):
Liquid Standard: The concentration of the liquid standard in µg/L. This standard must be prepared from a stock solution of formaldehyde in water by diluting it with stripping solution. The concentration of the stock solution should be 1.0×10^{-2} mol/L. The exact value should be prepared by another independent method, like titration. In Chapter 5.6.7 the necessary calculation for preparation of the standard is described in details.

Stripper Flow: The velocity of stripping solution. This value is determined automatically during calibration or zeroing. When opening the Setup/Calibration menu, the value from the last valid calibration is shown.

Set HV: The high voltage set-point of the photomultiplier. Also here the value of the last valid calibration is shown. The HV can be set manually between 100V and 900V. When changing the HV, the corresponding signal is shown on the right immediately (in red). Before starting a new calibration the signal should be above 1.0V and below 5.0V when measuring standard solution.

Stripp. Efficiency: The efficiency of the stripper: A value slightly below or equal to 1.0 (100%).

Part 2 (Wait Times):
The wait times define time constants used in the calibration procedure: All time constants are in seconds.

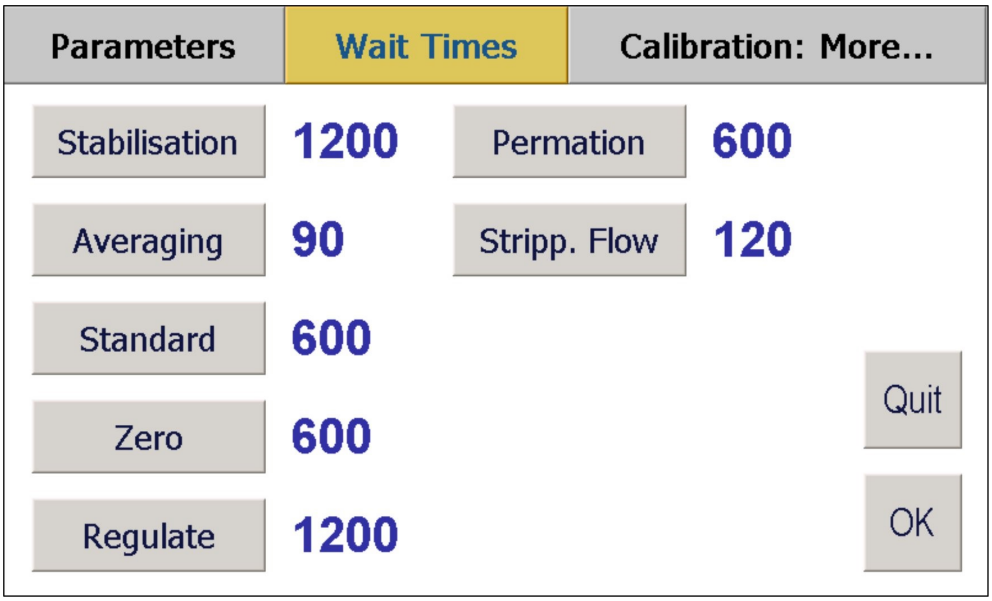


Fig. 5-12: *The wait times, averaging times and timeouts.*

Stabilisation: Timeout for stabilisation of the signal. If the stabilisation criteria are not fulfilled within the timeout, the signal is assumed to be unstable and the calibration fails. During stabilisation the signal is analysed by a moving linear fitting model on a subset of the signal data. The length N of this subset (default: 60sec) as well as the

stability limits, are set in the Setup/System menu (Chap. 5.6.5). The stabilisation criteria are:

- Standard deviation of the signal (σS) over N seconds.
- Long time gradient (δS/δt) over N seconds.
- Short time gradient (δS/δt) over N/2 seconds.

The stabilisation timeout applies for the determination of the standard, the zero and the permeation signal (*). When all three stabilisation criteria are fulfilled the routine measures the signal, by averaging over the time set by the Averaging button.

Averaging: Time for averaging the signal after the signal has stabilised. The same time applies for standard, zero, and permeation signal (*).

Standard: Wait time after starting the calibration, before the signal is analysed for stabilisation.

Zero: Wait time after switching the zero-valve, before the signal is analysed for stabilisation.

Regulate: Timeout for the regulation routine. If the routine can't regulate the signal to 4 Volt within this time, the calibration fails.

Permeation: Wait time after switching to the permeation unit, before the signal is analysed for stabilisation (*).

Stripp. Flow: Averaging time for the determination of the stripper flow.
(*) Applies only if a permeation unit is installed.

Part 3 (Calibration More...):
Calibrations can be stored on and loaded from the virtual hard disk of the instrument. The calibrations are stored as xml-files and the extension of all files is "cal". The file location is always "FlashDisk\AeroLaser\Cal" on the virtual hard disk of the instrument. By using the Calibration List Editor (see below) calibrations can be exported to and imported from an USB stick connected to the USB port. The file directory on the USB device is "Data\Cal". This path cannot be changed.

Set Perm Rate: When an internal permeation unit is installed, the permeation rate of this unit is determined during a liquid calibration. The system can also be calibrated using an external gas generator or similar equipment. In this case the (external) permeation rate in ng/L can be set here (Not supported yet!).

Verbose mode: If the verbose mode is switched on, info windows displaying details are shown during calibration on the Graph section. Switching this option off does not affect calibration.

After calibration go to: The measurement mode to that the system will go to after calibration automatically. The current option is indicated by the green button.

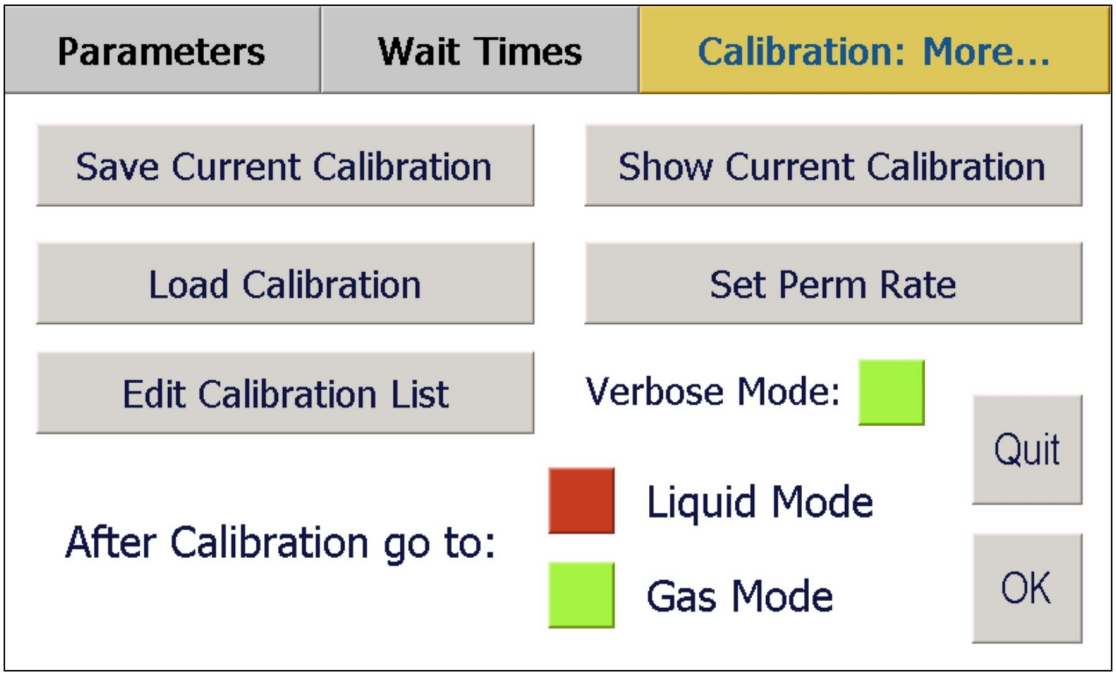


Fig. 5-13: *Part 3 of the Setup/Calibration menu contains additional features.*

Save current calibration: By pressing this button, a dialog to enter a file name for the calibration, opens up (See Fig. 5-14). The current calibration is stored on the internal virtual hard disk of the AL2021SC. The path is always: "FlashDisk\AeroLaser\Cal".

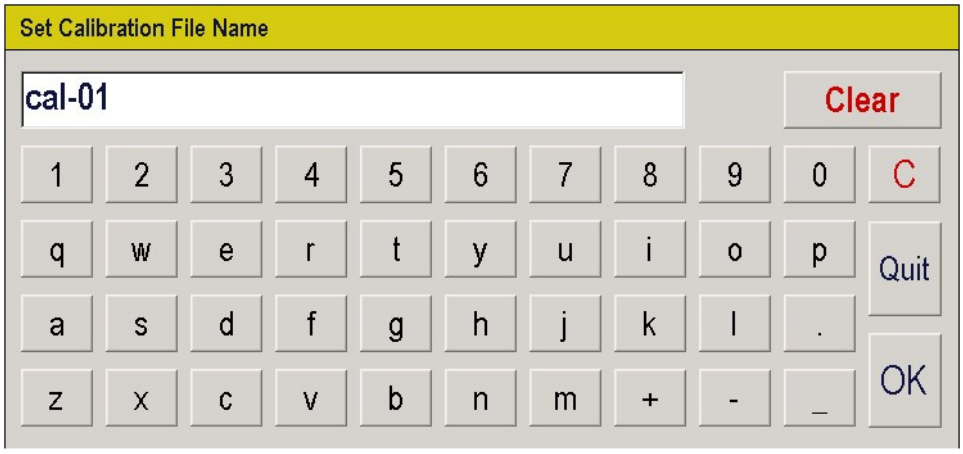


Fig. 5-14: *The Set File Name dialog. The calibration is saved as an xml-file with the extension "cal" in the folder "FlashDisk\AeroLaser\Cal" on the virtual hard disk of the instrument.*

Load calibration: By pressing this button, a menu to load a calibration, opens up, where one of the previously saved calibrations can be selected. It is loaded into the current system setting and overwrites the current calibration. This cannot be undone.

Show current calibration: For details see Fig. 5-5 and explanation below.

Edit Calibration List: All calibrations previously saved, are shown in a list. The highlighted calibration can be deleted, renamed or exported as xml-file to an USB device, connected to the USB port of the instrument. The export directory on the USB device is always "Data\Cal". If this directory is not available, it is created by the

instrument. Calibrations can also be imported from an USB-device. The import dialog lists all calibration files available in the "Data\Cal" folder. Importing a calibration simply adds it to the internally stored files. It does not load the calibration.

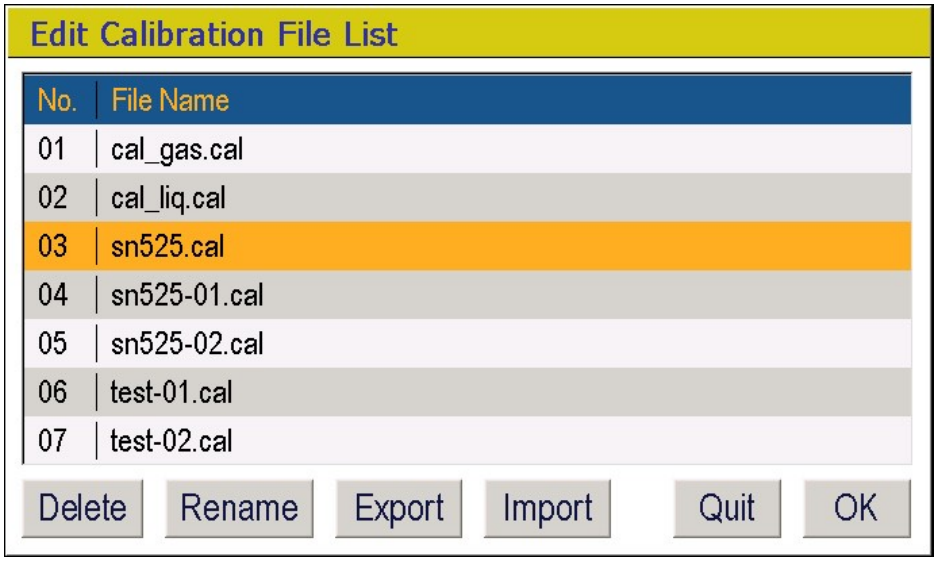


Fig. 5-15: *Calibration list editor. The list shows all calibrations previously stored on the instrument. The highlighted calibration can be renamed, deleted or exported to USB. The Import button allows importing a calibration from an USB device.*

5.6.2 Setup: AutoSequence

The instrument is equipped with a powerful command sequencer that can automatically execute a series of commands, like gas calibration, zeroing, switching external valves or changing the measurement mode. Technically, this is done by running a previously programmed sequence of commands, the AutoSequence. AutoSequences are programmed on the instrument and can be stored to and loaded from files on the internal virtual hard disk of the AL2021SC. The path is always: "FlashDisk\AeroLaser\Asq". The file extension is ".asq". Like Calibrations, AutoSequences can be deleted, renamed or exported as xml-file to and imported from an USB device connected to the USB port of the instrument.

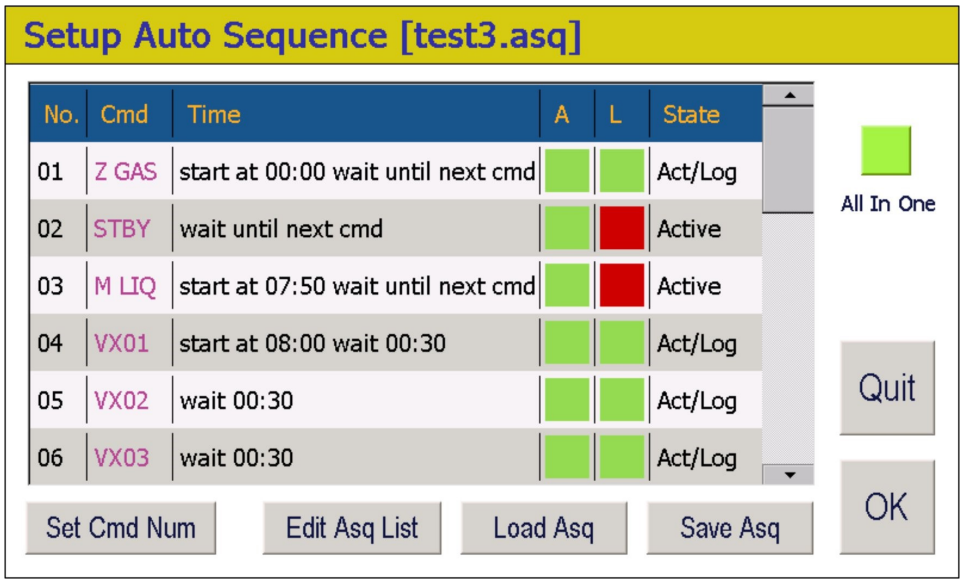


Fig. 5-16: *The AutoSequence Editor shows a list of commands, which are executed according to the numeric order.*

Set Cmd Num: Sets the total number of commands.

Save Asq: By pressing this button, a dialog to enter a file name for the AutoSequence opens up (Similar to Fig. 5-14). The current AutoSequence is stored on the instrument's internal virtual hard disk. The path is always: "FlasDisk\AeroLaser\Asq".

Load Asq: By pressing this button, a menu to load an AutoSequence opens up, in witch one of the previously saved AutoSequences can be selected. It is loaded into the current system setting and overwrites the current AutoSequence. This cannot be undone.

Edit Asq List: All AutoSequences previously saved are shown in a list. The highlighted AutoSequence can be deleted, renamed or exported as xml-file to an USB device, connected to the USB port of the instrument. The export directory on the USB device is always "Data\Asq". If this directory is not available, it is created by the instrument. AutoSequences can also be imported from an USB-device. The import dialog lists all calibration files available in the "Data\Asq" folder. Importing an AutoSequence adds it to the internally stored files. It does not load the AutoSequence.

All In One: If this option is activated all data logged during the execution of the AutoSequence are logged into one file (if the "New File Interval" option is set to "Never" in the Setup\File dialog). Otherwise a new file is created for each command.
Fig. 5-15 shows the AutoSequence editor. All commands within an AutoSequence are listed in numeric order. Each command line consists of six fields (from left to right):

1.) The order number of the command. By pressing on the number a dialog appears, which allows to Insert or Insert and Edit a command before the line. By pressing Delete the command is deleted without prompt.

2.) The command: By pressing on the command the command editor appears (see Fig. 5-18). The following commands are available:

   1.) **Standby (STBY)**: Sets the instrument in Standby mode. All other non-empty commands return to normal mode automatically.

   2.) **Zeroing (ZERO)**: Starts a zeroing of the instrument to determine the zero-level automatically. No wait time can be set, zeroing continues with the next command, when it has finished. Zeroing will not change the measurement or calibration mode.

   3.) **Gas Calibration (CAL G)**: Starts a gas calibration (requires a permeation unit). No wait time can be set, the gas calibration continues with the next command, when it has finished. After gas calibration the instrument is in the "gas cal" mode. The measurement mode will not be affected.

   4.) **Meas Liq (M LIQ)**: Liquid Measurement Mode.

5.) **Meas Gas (M GAS)**: Gas Measurement Mode.

6.) **Meas Gas Zero (Z GAS)**: Zero Gas Measurement.

7.) **Switch Valve N** (VXN): Switches External Valve N, the number of available valves corresponds to the number of valves in the external valve controller. If no valve controller is installed, these commands are not available.

8.) **None (NONE)**: Empty command.

3.) Start Time and Wait Time of a command. Both can be zero. Pressing on this field also activates the command editors (See Fig. 5-18).

4.) Activates (green) or deactivates (red) the command. A deactivated command is ignored by the AutoSequence.

5.) Activates (green) or deactivates (red) data logging.

6.) Info Field shows the state of the command.

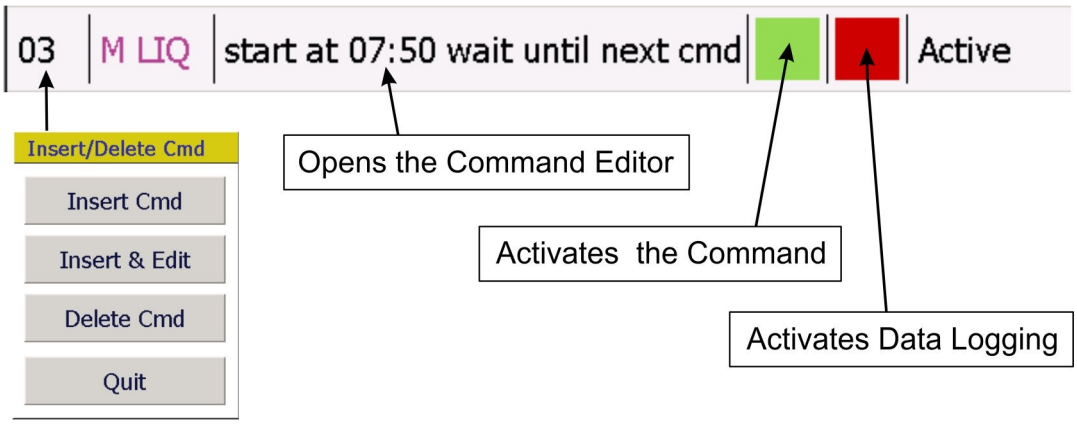


Fig. 5-17: *Details of a command line in the AutoSequence editor. By pressing on a section different dialogues appear (see explanation above) .*

Generally, there are two different types of AutoSequences:

1.) AutoSequences **without fixed Starting Times**. These AutoSequences are started immediately, when pressing the Start button in the Data menu. Each command has a defined wait time after it is executed. When the wait time is over, the next command is executed. After the last command is executed, the AutoSequence continues with the first command. This is repeated until the AutoSequence is stopped by the user. The Wait Time can also be zero. In this case, after execution of the command, the next command is executed immediately. For all commands the option "Start at absolute time" must be deactivated (red, see Fig. 5-18).

2.) AutoSequences **with fixed Starting Times**. When starting the AutoSequence, it is scheduled until the next command after the current time. If there is no

such command, the AutoSequence is scheduled for the next day. All commands are executed at their Start Time, while the Wait Time is ignored.

Even if it is theoretically possible to mix both types, it is not recommended.

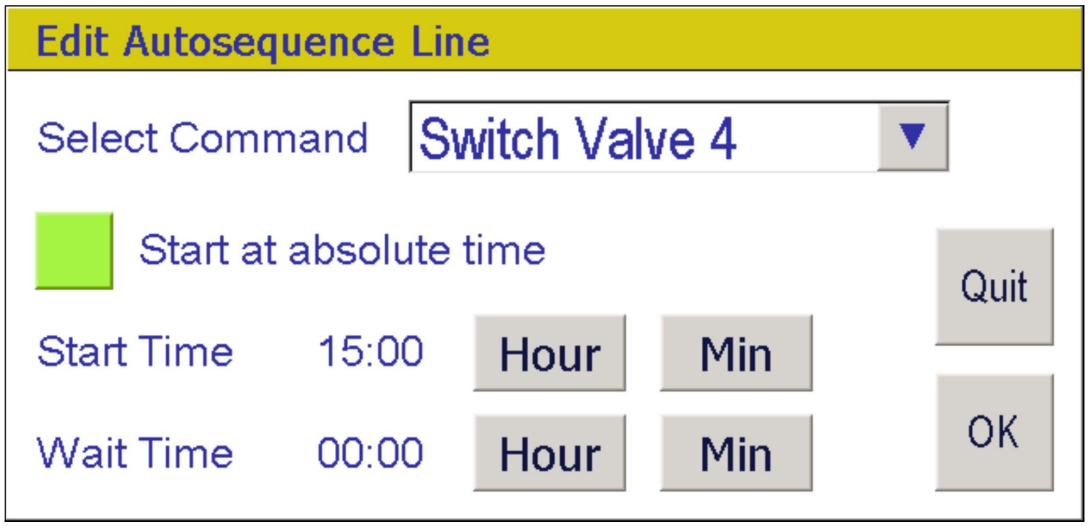


Fig. 5-18: *The command editor: All details of a command are set here.*

5.6.3 Setup: File Options

This menu is organised in two parts. In the first part "Data Storage: Parameters" the parameters to be saved are selected.
Every time data is saved, a new line is added to an existing file or written into a new file.

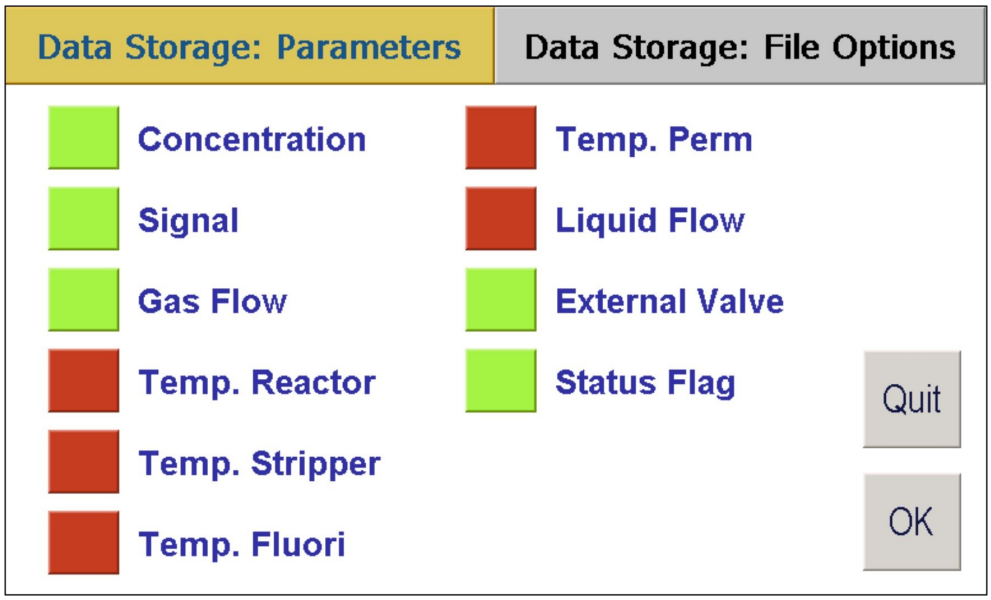


Fig. 5-19: *Setup File Menu, part 1: Select the parameters to save.*

In part 1, the parameter to be saved can be selected. The dialog shows all available parameters. A green button means, that the parameter is selected, a red button means the opposite. The Status Flag combines detailed system information in a 32bit unsigned integer. For the definition of the Status Flag see Appendix 9.4.

The second part "Data Storage: File Options" contains advanced settings:

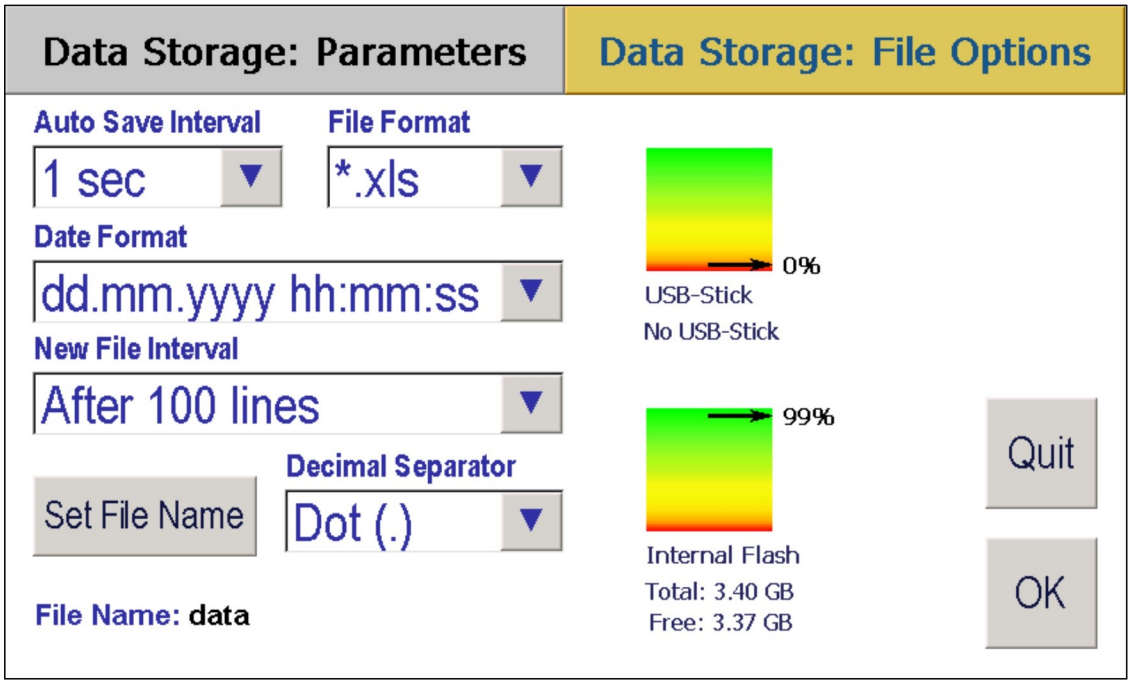


Fig. 5-20: *Setup File Menu, part 1: Advanced options.*

USB-Stick: Info about the free space available on the USB device.

Auto Save Interval: Drop down menu. Available options:

- 1 sec
- 5 sec
- 10 sec
- 30 sec
- 1 min
- 5 min
- 10 min

File Format: Defines the output data format Available options:.

- *.dat
- *.csv
- *.xls

In the csv-format all data are enclosed in quotation marks and separated with a semicolon.
In the dat-format, plain data is saved in ASCII format, separated by tabulators.
The xls-format stores data as a spread sheet, together with calibration data and an event log.

New File Interval: To prevent huge files, a new file can be created after a certain time. Available options:

- Once per hour
- Once a day
- After 10000 lines
- After 1000 lines

- After 100 lines
- Never

Date Format: Several common date/time formats can be selected. Available options:

- dd.mm.yyyy hh:mm:ss
- dd-mm-yyyy hh:mm:ss
- yyyy.mm.dd hh:mm:ss
- yyyy-mm-dd hh:mm:ss
- yyyy.dd.mm hh:mm:ss
- yyyy-dd-mm hh:mm:ss

Set File Name: Files are named automatically using a user-defined prefix, which is set here. The file names are generated in the following way:

prefix-hh-mm-ss.dat or
prefix-hh-mm-ss.csv

On the USB medium inserted into the USB slot of the AL2021SC, a folder named "Data" is created. The data is stored into subfolders, which are named by the date of the day in the following way: "Data-yyyy-mm-dd".

5.6.4 Setup: Graph

The graphical data display of the AL2021SC can be configured by the user.

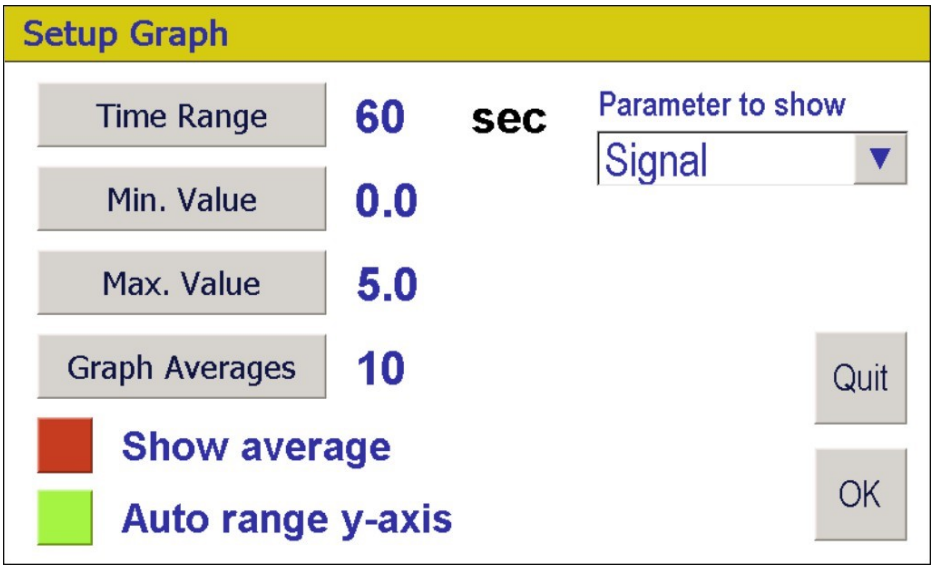


Fig. 5-21: *Setup File Menu, part 1: Advanced options.*

Time range: Sets the range of the x-axis in seconds. Due to limited calculation power, the maximum value is one day (86400 secondes).

Min. Value: Minimum value of the y-axis.

Max. Value: Start Minimum value of the y-axis. In the example above the range of the y-axis is set from 0.0 – 5.0, which is the range of the analog signal.

Graph Averages: If the "Show Average" option is selected, the graphic display can show a moving average of the data. Here the number of data points (seconds) to average is set.

Show Averages: Turn the display of an moving average on/off.

Auto range y-axis: The graphic display can resize the y-axis if the data go beyond the limits set above. Here this option is turned on/off.

Parameters to show: Selects the parameter to display. Available options:

- Automatic (recommended)
- Concentration
- Signal
- Gas Flow
- Liquid Flow

If the option "Automatic" is selected, the display shows the signal during calibrations and the concentration otherwise.

5.6.5 Setup: System

This menu is organised in three parts. Part 1: General system settings. Part 2: Valves allows manual setting of internal and external valves (if available). Part 3: Internal Values.

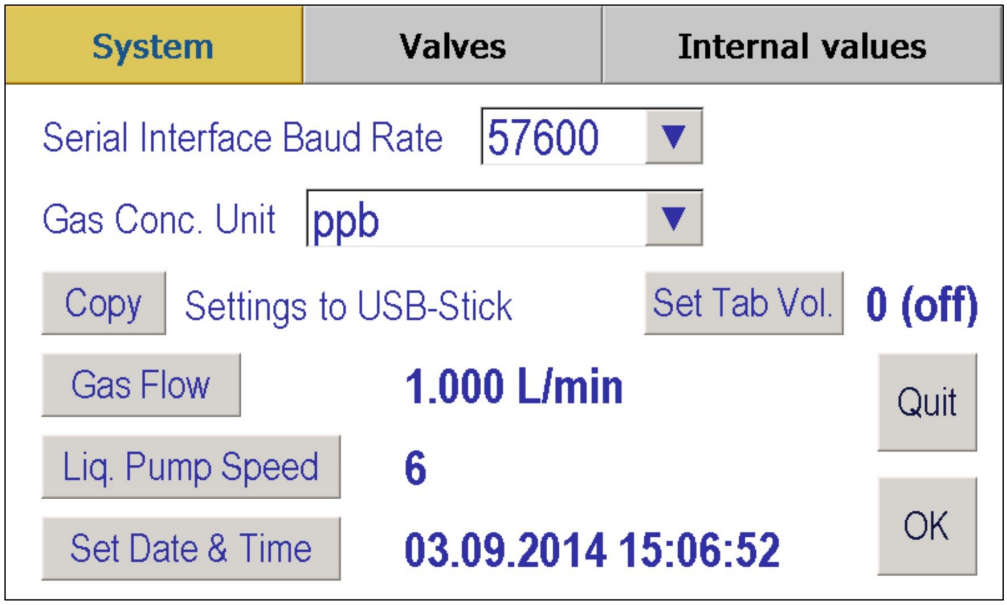


Fig. 5-22: *Setup Sytem Menu, part 1: General System Settings.*

Serial Interface Baud Rate: Baud rate of the external serial interface. The following baud rates are available:

- 9600
- 14400
- 19200
- 38400
- 57600
- 115200

For the other parameters of the interface see Chapter **Fehler! Verweisquelle konnte nicht gefunden werden.**. Please note, that the parameters of the serial interface must be equal to the setting on the remote PC.

Gas Conc. Unit: The gas concentration can be displayed and saved in another unit than the standard unit ppb. In case another unit is selected, the concentration is internally recalculated using a constant factor. The following table shows the available options and the corresponding factors f that converts between the units:

$$c_{gas}\left[\frac{\mu g}{m^3}\right] = f \cdot c_{gas}[ppb]$$

Option	Pressure	Temperature	f
ppb			1.000
SATP	1013.00 mbar	25°C	1.227
NORM	1013.25 mbar	0°C	1.340
IUPAC82	1000.00 mbar	0°C	1.322

Copy Settings to USB-Stick: Copies the current setting to an xml-file named "settings.xml" to an USB-stick.

Gas Flow: Sets the gas flow of the system.

Liq. Pump Speed: Sets the speed of the peristaltic pump. The hex values between 0 and F are possible. A setting resulting in a speed between 450 and 500 µL/min is recommended (A – C). For details on the peristaltic pump speed see Chapter 0.

Set Date & Time: Sets the current date and time. Change this setting, if the local time at the location of the instrument differs from GMT.

Set Tab Vol.: Sets the tab sound volume from 0 – 9, 0 means no sound (default). The tab sound is played always, when the user touches the screen.

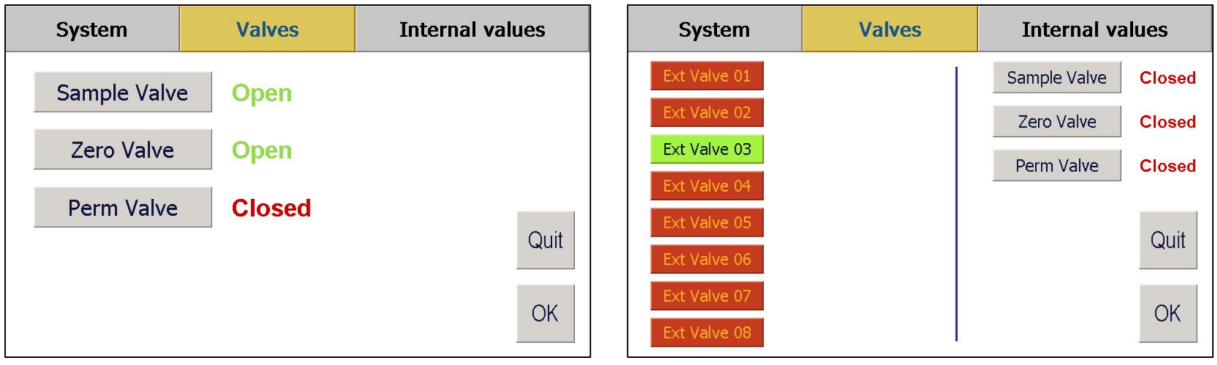


Fig. 5-23: *Setup System Menu, part 2: Valves can be switched manually. This menu appears differently, if an external valve controller is installed (right).*

The second part of this menu is intended to switch valves manually. It appears differently if an external valve controller is installed (Fig. 5-23 right) or not (Fig. 5-23 left). The external valve currently open is indicated by the green button.

The third part of this menu shows internal parameters of the system and allows to set up the parameters of the stabilisation detection implemented in the regulation routine.

Upper Left - AD/DA: Current analog voltages at the AD/DA converter card. Left: AD (analog readings), right DA (digital settings). The available channels are shown in blue, the others in grey.

Upper centre – Temperature set-points: This column shows the set points of the quad temperature controller (QTC) inside the system:

- SP TR: Set-point of the reactor (68°C)
- SP TS: Set-point of the stripper (10°C)
- SP TF: Set-point of the fluorimeter (35°C)
- SP TP: Set-point of the permeation oven (45°C)

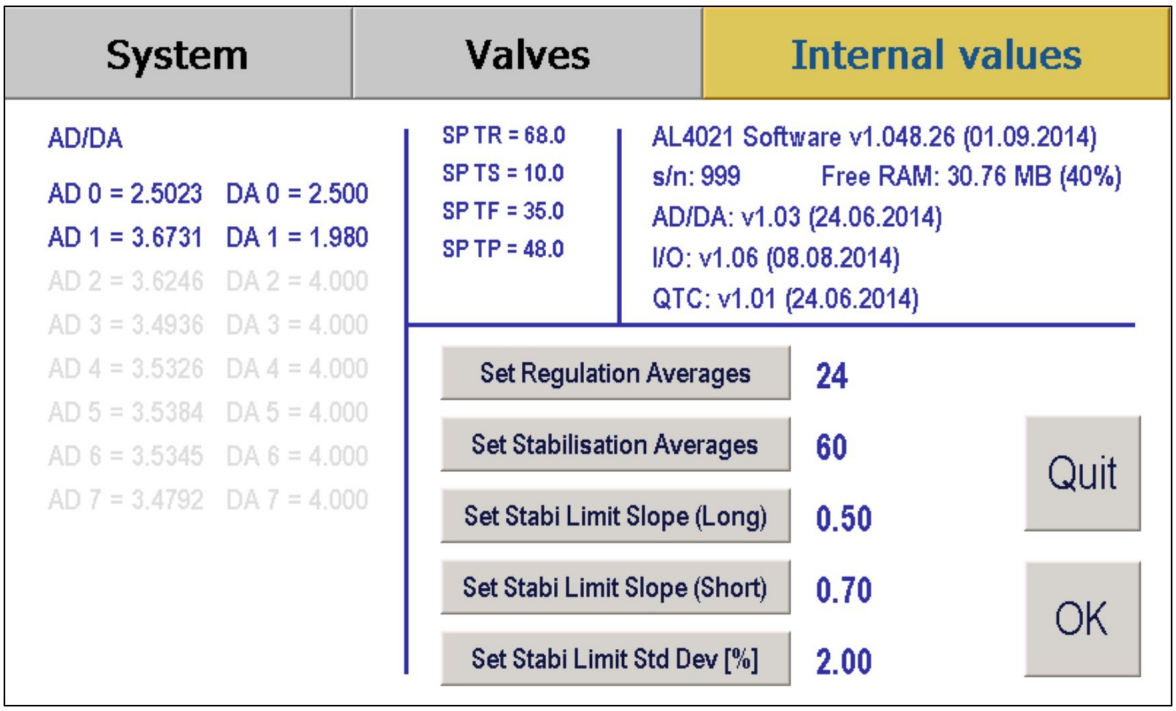


Fig. 5-24: *Setup System Menu, part 3: Internal values are displayed and advanced parameters of the automatic stabilisation detection can be set.*

Upper centre – Serial/Version:
1st line: Instrument's type (AL2021SC) and software version.
2nd line: left: serial number, right: memory in use.
3rd line: version of the AD/DA board firmware.
4th line: version of the I/O board firmware.
5th line: version of the QTC board firmware.

Set Regulation Averages: Number of seconds the signal is averaged for each fine regulation step (Default = 24).

Set Stabilisation Averages: Size *N* of a subset (in seconds) of the data that is continuously analysed for stability (Default = 60).

Set Stabi Limit Slope (Long): Upper limit for a gradient on a data subset of length *N* that is recognised as stable (Default = 0.5).

Set Stabi Limit Slope (Short): Upper limit for a gradient on a data subset of length *N/2* that is recognised as stable (Default = 0.7).

Set Stabi Limit Std. Deviation [%]: Upper limit for the standard deviation on a data subset of length *N* in percent (Default = 2.0).

Note:
The values above affect the system's ability to calibrate.
Be very careful with changes!

5.6.6 Setup: Admin

This section is intended for maintenance. Only administrators are allowed to access. In order to enter the admin, a password is required (82467). This menu is organised in two parts. Part 1: Set-points and device settings. In Part 2 the DAC of the AD/DA board can be set manually for maintenance.

Flow Controller Calib.: Sets the flow controller's calibration flow. Only needs to be set if the flow controller is replaced by another type.

Gas Pump Voltage: Operation voltage of the gas pump. Can be increased, if the gas pump is not able to generate the adjusted gas flow. This could be necessary, if the gas pump is fatigued by age. The voltage supply can be increased up to 4.8V. If this occurs, a replacement pump should be ordered immediately.

Meas. Averages: Size of an additional ring buffer smoothing the raw data. The default value is 7. A too high value will result in unnatural smooth data.

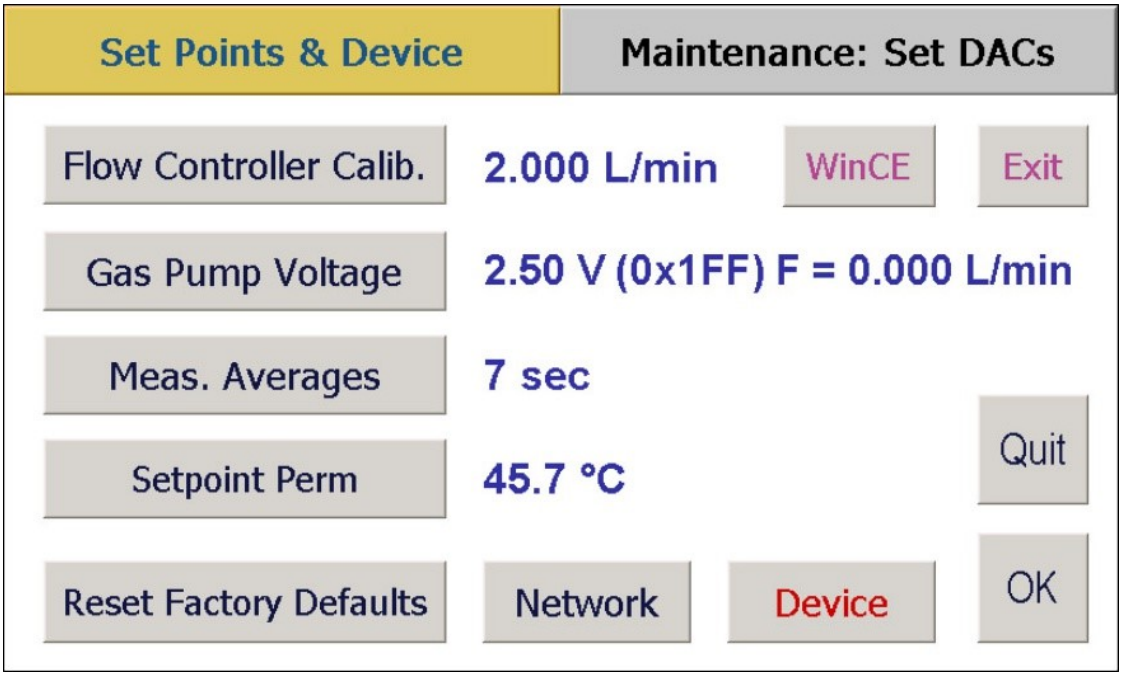


Fig. 5-25: *Setup System Menu, part 1: Internal values are displayed and advanced parameters of the automatic stabilisation detection can be set.*

Setpoint Perm.: Sets the temperature set-point of the permeation oven. The permeation rate can be fine-tuned by changing this value. Please note that after

changing the set-point, the permeation unit has to stabilize for 24 hours, before it can be used (only available, if a permeation unit is installed).

Reset factory defaults: Resets all settings to the factory defaults. Be careful, this action cannot be undone.

WinCE: Starts the Windows CE shell of the CPU. For maintenance only. Before using the AL2021SC it needs to be switched off and on again.

Exit: Shuts down the program.

Network: Pressing the Network button opens the Setup Network dialog. If the instrument is connected to a local area network (LAN), the local IP address is displayed. The address depends on the router/network configuration on site. The address shown in the figure below is only an example. There are three different services available:

1. HTTP (web-server). The HTTP-server provides the current parameters (concentration, temperatures, ...) on a simple web-site, which is updated automatically every second. To access this site type the following line into any web browser.

http://xxx.xxx.xxx.xxx/index.html

(Please replace xxx.xxx.xxx.xxx with the instrument's current IP address.)

This service can be switched on and off by pressing the button. If it is switched on, the button is displayed in green, otherwise it is red.

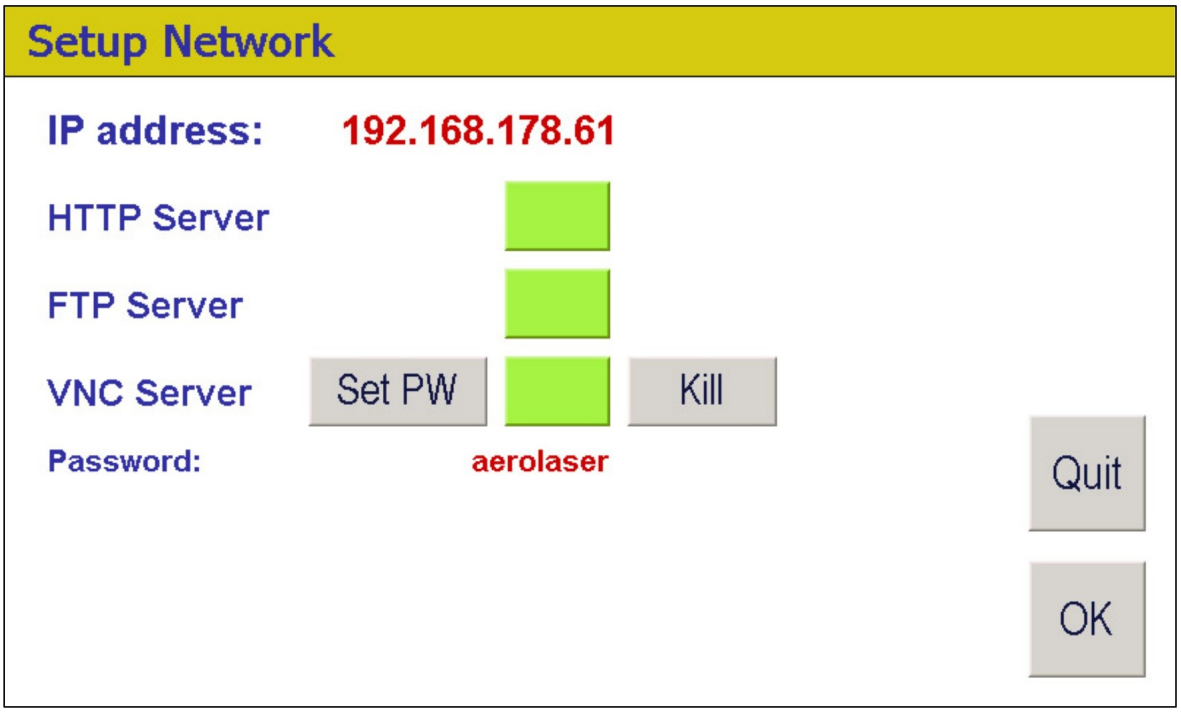


Fig. 5-26: *Setup Network The current IP address is given. The three available services can be switched on and off and a VNC password can be set.*

2. FTP (File Transfer Protocol). The instrument is equipped with a non-volatile internal memory of 4Gb. Measurement data are back-upped to this memory automatically. If the memory run full, the oldest datasets are deleted to prevent a memory overrun. These data can be accessed via the FTP-protocol. This can be done by typing the following line into a web browser able to handle ftp-transfers.

fttp://xxx.xxx.xxx.xxx

(Please replace xxx.xxx.xxx.xxx with the instrument's current IP address.)

When accessing the ftp-server a list of directories containing the data is displayed. By clicking on the directories name a list of files to download opens up.

Similar to the HTTP-service, the FTP-service can be switched on and off by pressing the related button. If it is switched on, the button is displayed in green, otherwise it is red.

3. VNC (Virtual Network Computing). The VNC-protocol is used to forward the instruments display to an external application, which runs on a computer, smart phone or tablet. The display is updated several times a second and shows the same than the touch panel of the instrument. The external application works as a remote control: Pressing or klicking a button on the remote application has the same effect than doing so physically on the touch-panel.

Since remote commands could be set by VNC, the VNC-server is protected by a password (Use the Set PW button to set the password). The VNC service allows only one connection from one remote device at a time. The VNC service can be started and stopped using the Start/Kill button. If the related button is green, the service will run automatically when starting the instrument.

There are applications available from Aero-Laser for Windows and Android operating systems.

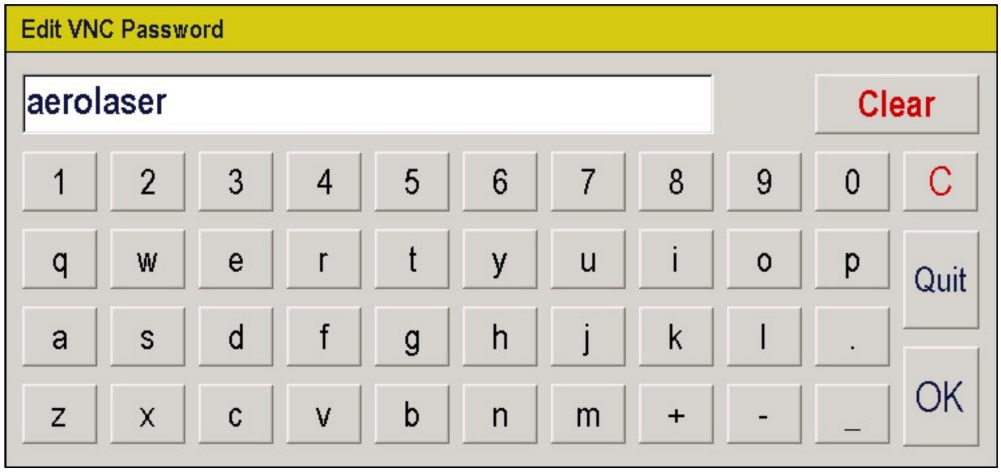


Fig. 5-27: *Dialog for setting the VNC password.*

Note:
After changing any of the network settings, the instrument must be restarted.

Device settings: Settings that need to be changed if the system is significantly changed, e.g. by installing or removing components of the system, like a permeation unit. **For the use by Aero-Laser personnel only**.

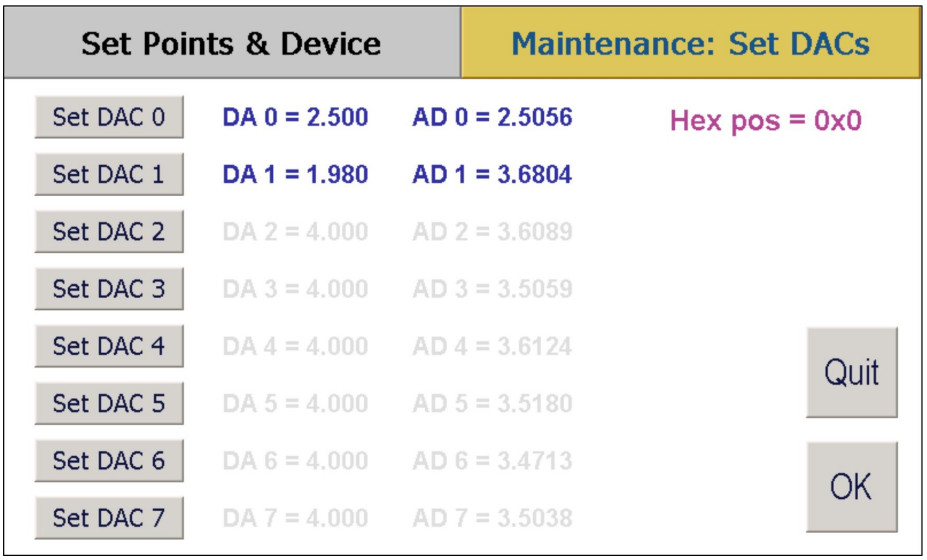


Fig. 5-28: *Setup System Menu, part 3: Internal values are displayed and advanced parameters of the automatic stabilisation detection can be set.*

In the second part of this menu the DACs on the AD/DA board can be set manually for maintenance purposes. The available DAC channels are displayed in blue the others in grey. "Hex pos" shows the position of the Hex switch on the AD/DA board. This switch is only to be used by Aero-Laser personnel to calibrate the board.

5.6.7 Setup: Calc. Standard

This section contains a convenient calculator for the liquid standard needed for calibration. It also calculates the volume of a standard stock solution (also called Ur-Standard) necessary to prepare the standard solution.

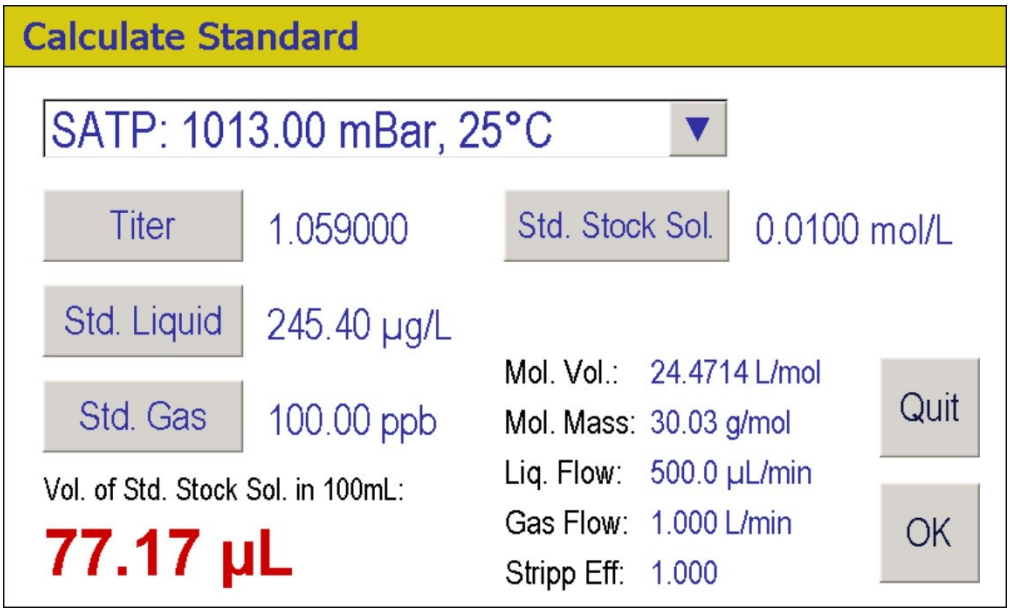


Fig. 5-29: *Calculator for standard concentrations. The volume of standard stock solution to be diluted in 100 mL is shown in red.*

This calculation assumes an standard stock solution of 0.01 mol/L, but other values are possible The exact concentration of the standard stock solution should be determined e.g. by titration. The calculation employs the formula which is used for getting the gas concentration from the measured liquid concentration:

$$c_{gas} = \frac{c_{liq}}{Eff_{Stripp}} \frac{F_{liq} \cdot v_{mol}}{F_{gas} \cdot m_{mol}}$$

c_{gas}	Gas concentration [ppb].
c_{liq}	Liquid concentration [µg/L].
F_{liq}	Liquid flow = flow of the stripping solution [mL/min].
F_{gas}	Gas flow [L/min]
v_{mol}	Molar volume [L/mol]
m_{mol}	Molar mass[g/mol] (formaldehyde: 30.03 g/mol)

The molar volume depends on temperature and pressure. For SATP conditions (1013mbar, 25°C) it is 24.4714 L/mol.

Fig. 5-29 shows the dialog for the calculation of the standard solution. It can be either calculated for a desired gas measurement range, as well as for a liquid measurement range. In the drop-down menu on top, the standard conditions can be set. For details on the options see Chapter 5.6.5. On the right side of the dialog other parameters for the above calculation are shown.

Titer: The titer of the Ur-standard, determined by titration.

Std. Stock Sol.: Concentration of the standard stock solution in mol/L. 0.01 mol/L is recommended.

Std. Liquid: Concentration range for liquid measurements. When setting this value, the corresponding gas concentration range is set automatically.

Std. Gas: Concentration range for gas measurements. When setting this value, the corresponding liquid concentration range is set automatically.

Vol of Std. Stock Sol. in 100mL: The volume of the standard in µL to be filled up to 100mL with stripping solution (for gas measurements) or with water (for liquid measurements). This value is displayed in red.

Note:

The inputs in the Calculate Standard menu **do not influence** the measurement or the calibration! This menu is intended as a helper for standard preparation.

5.7 Menu: Standby

Instead of a complete shut-down the instrument can be set in Standby mode. The peristaltic pump is set to the lowest possible speed (1 rpm). This prevents plastic deformation of the tubing and saves reagent and stripper. The temperatures of all components are still controlled. After returning from Standby, the instrument is ready for measurement in some minutes.
When shutting down the instrument (see Chapter **Fehler! Verweisquelle konnte nicht gefunden werden.**) it should run dry completely.

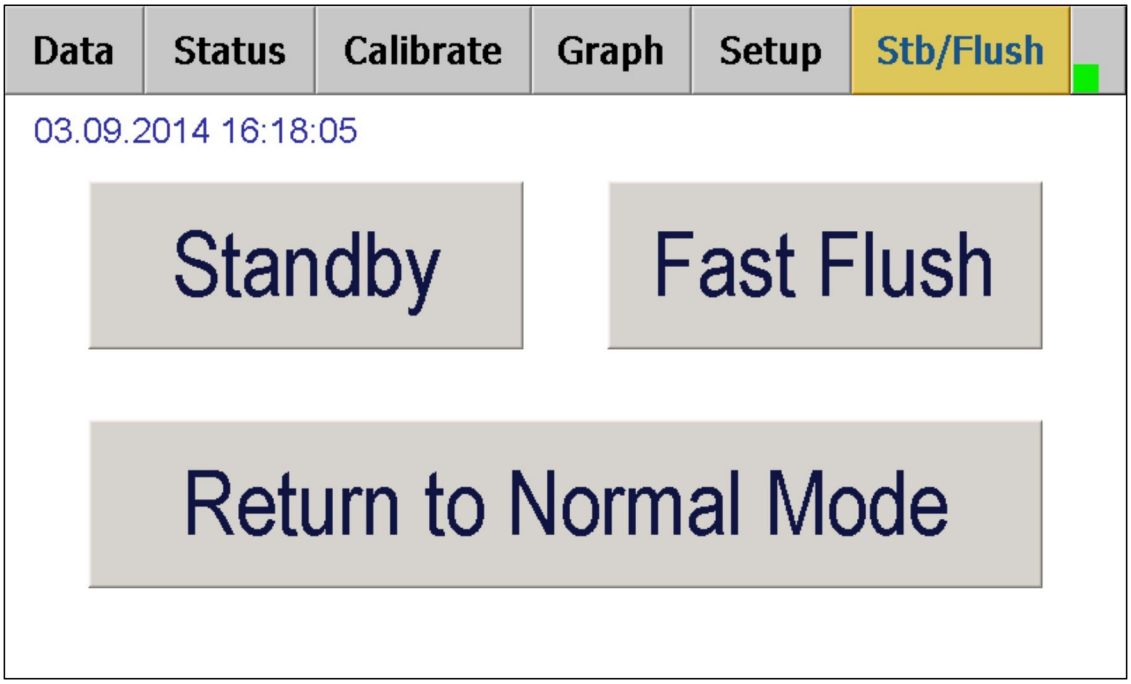


Fig. 5-30: *Standby/Fast Flush dialog.*

The Fast Flush mode increases the pump speed to a maximum level of 40 rpm, acceleration the shut down procedure by a factor of ~5. When returning to normal mode, the instrument returns to the same parameters that were valid before Standby/Fast Flush.

6 SqeezeDat

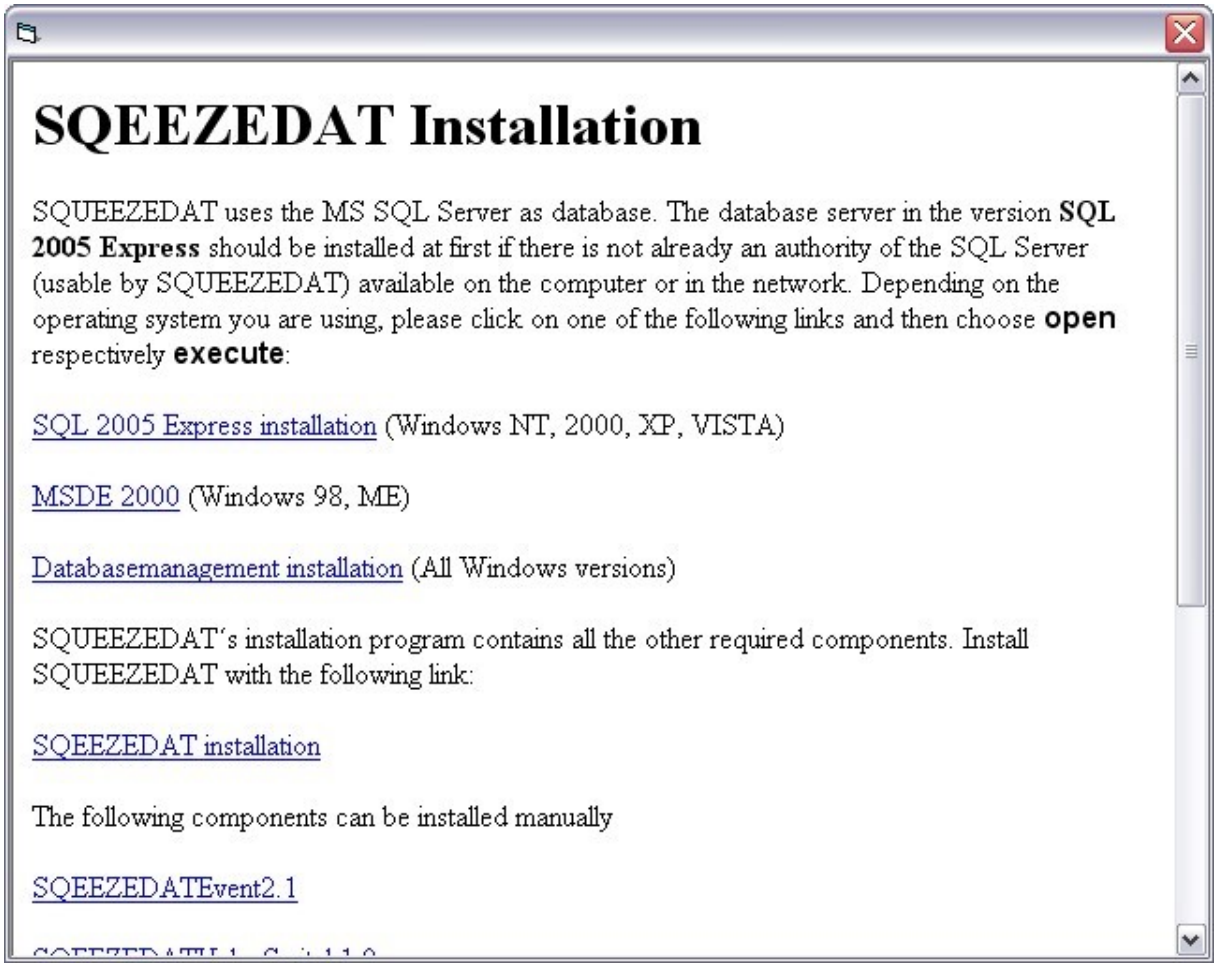
SqeezeDat is a data base software based on the widely used SQL standard. It was designed to record the data of up to 8 individual Aero-Laser instruments via the RS232 interface. The data can be exported from the SQL data base to ASCII or XML format for further calculations or analysis. SqeezeDat works with all Aero-Laser instruments. Furthermore it can record data from other non Aero-Laser devices, if they are equipped with a RS232 interface and use a compatible command structure. While recording, SqeezeDat can display graphically up to 8 parameters in separate windows. For an enhanced clarity, more than one parameter can be displayed in a single window

6.1.1 Installation (Windows XP and lower)

After inserting the CD into the computer the following window is shown:

If this window is not shown, open the file “InstallStart.exe”. Select the language you want to install.

The following window gives instructions how to install SqeezeDat:



For running SqeezeDat it is necessary to install the following components:

- .NET Framework 1.1
- .NET Framework Update 1.1
- .NET Framework 2.0 or higher (if not already installed!)
- SQL 2005 Express
- SqeezeDat

To install them click on the link and follow the installation instructions.

If you run SqeezeDat for the first time you have to enter a license key. In this window a customer name and a base key are shown. Make a screenshot of this window and send it to info@aero-laser.de. You will get the license key by e-mail.

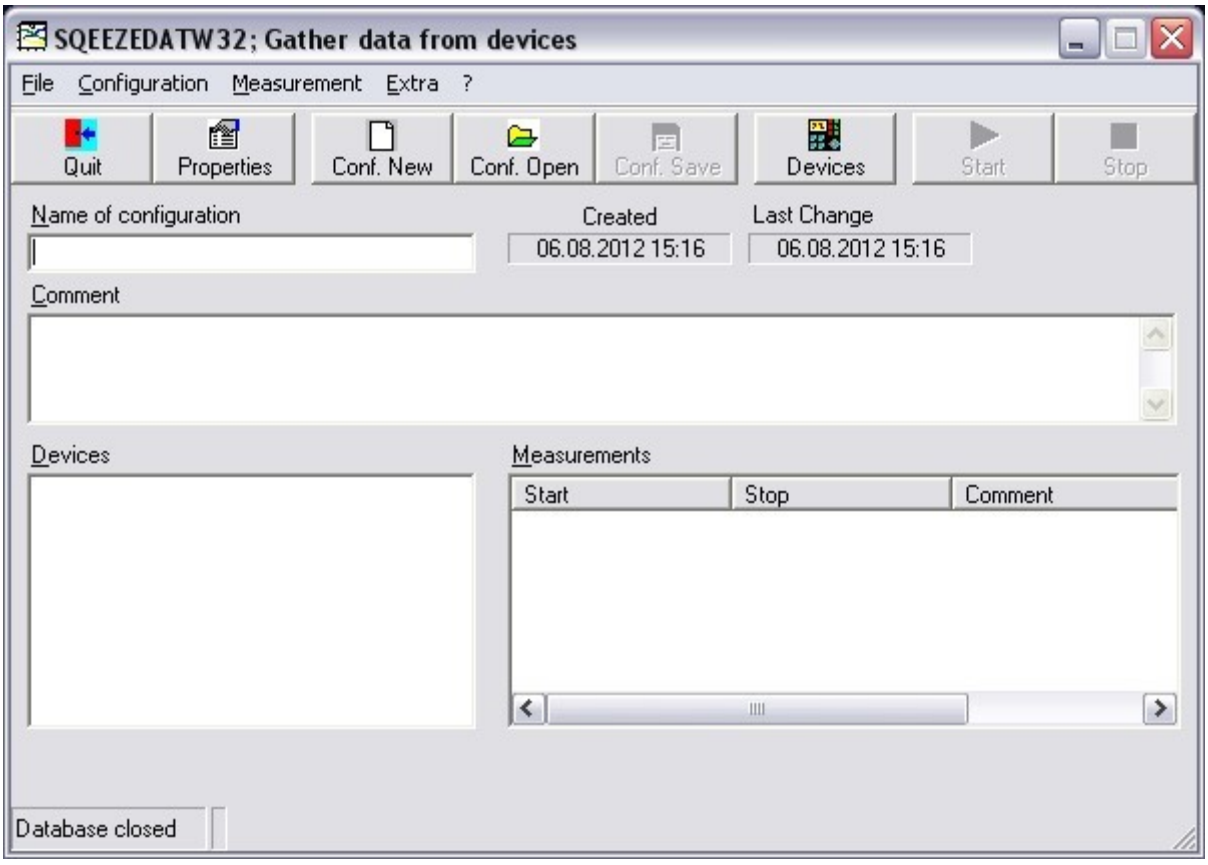
Warning:
If SqeezeDat shall be installed on a Win7/8 64bit system, install SQL 2005 Express service pack 3 after installing SQL 2005 Express.
Under no circumstances install service pack 4!

6.1.2 Setup

This manual only gives an overview on how to set up and use SqeezeDat and SqeezeDatExport. For detailed information please have a look at SqeezeDat helpfile.

If SqeezeDat is started for the first time the following window is shown:

Confirm with “Yes”. The database is created automatically and the SqeezeDat window opens:

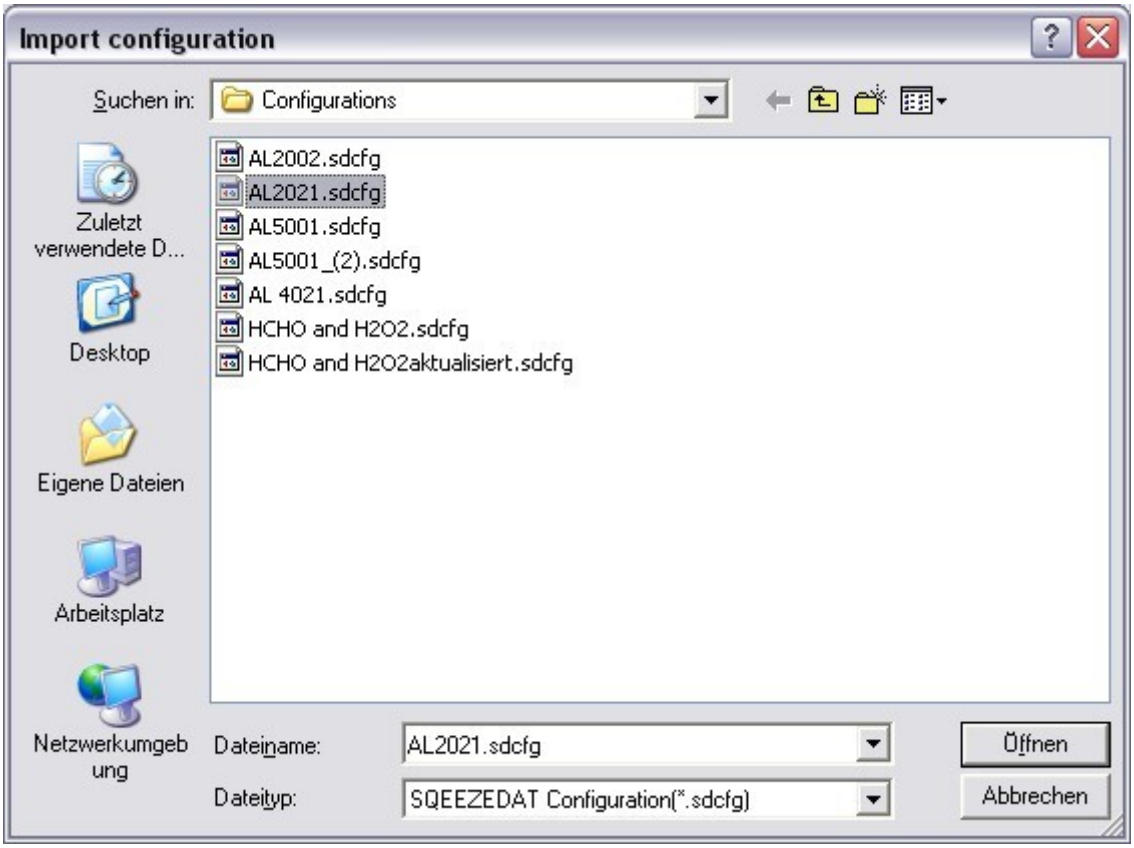


This table shows the possible options and commands:

File	Properties	Program settings of Sqeezedat
	Show Protocol	Show current application-flow protocol of Sqeezedat
	Database Info	Indicates information about the current database
	Edit Database	Edit databse
	Shrink Database	Shrink database to save storage space
	Backup Database	Save database into a file
	Restore Database	Restore database from a file
	AddIn Management	Install Add Ins
	Quit	Quit Sqeezedat
Configuration	New	Create new Configuration
	Open	Open existing configuration
	Save	Save data of the active configuration
	Delete	Delete active configuration
	Import	Imports configuration from extern file
	Export	Save configuration into extern file
Measurement	Start	Start measurement
	Stop	Stop measurement
Extra	Define Devices	Define predefined devices, which can be inserted into configurations
?	Content	Opens the help file
	Info	Shows the version

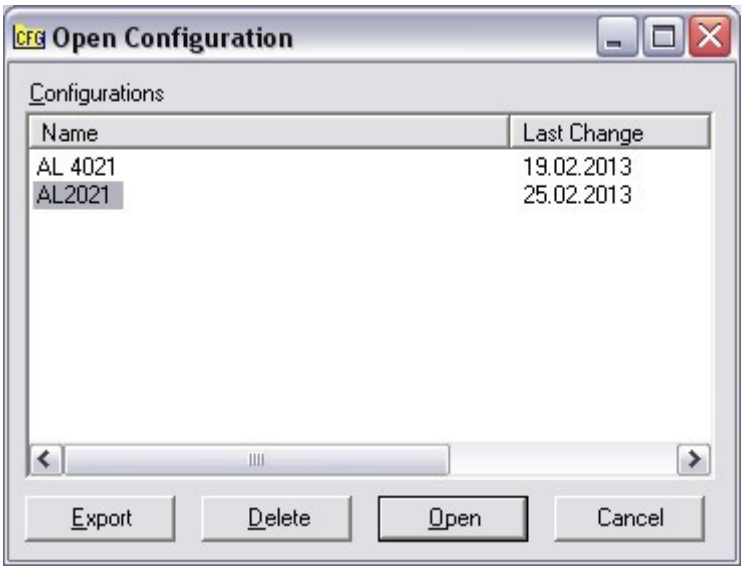
SqeezeDat is able to record data from all devices which work with RS232 commands. For recording the data a configuration is needed which contains the commands. There are configurations on the enclosed DVD. For using these configurations you have to import a configuration.

Select “Configuration” and “Import”. The import window opens:

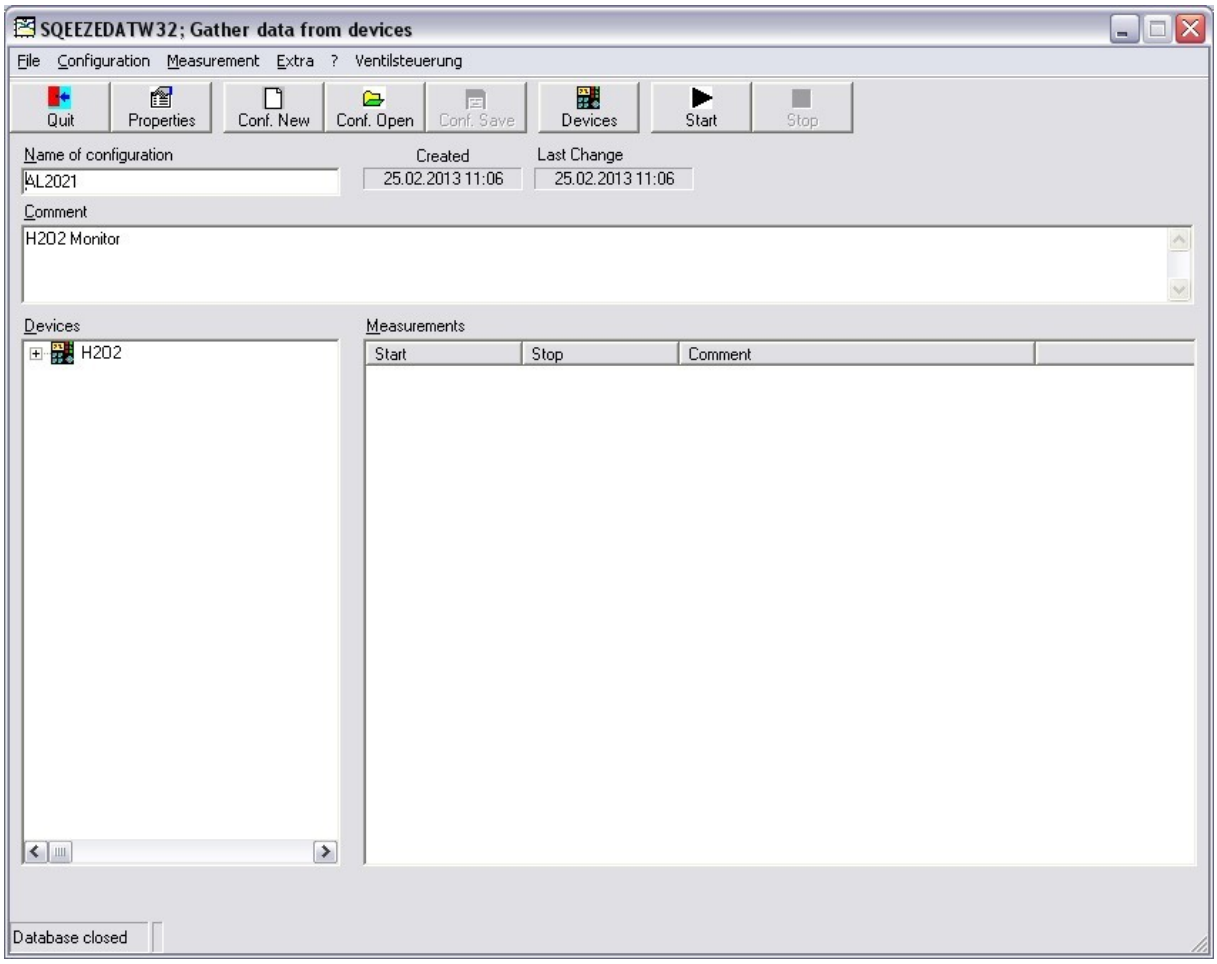


Select the configuration “AL2021SC” and “Open” it. Now the configuration is imported into SqeezeDat. Several configurations can be imported. This might be useful if several different instruments are used.

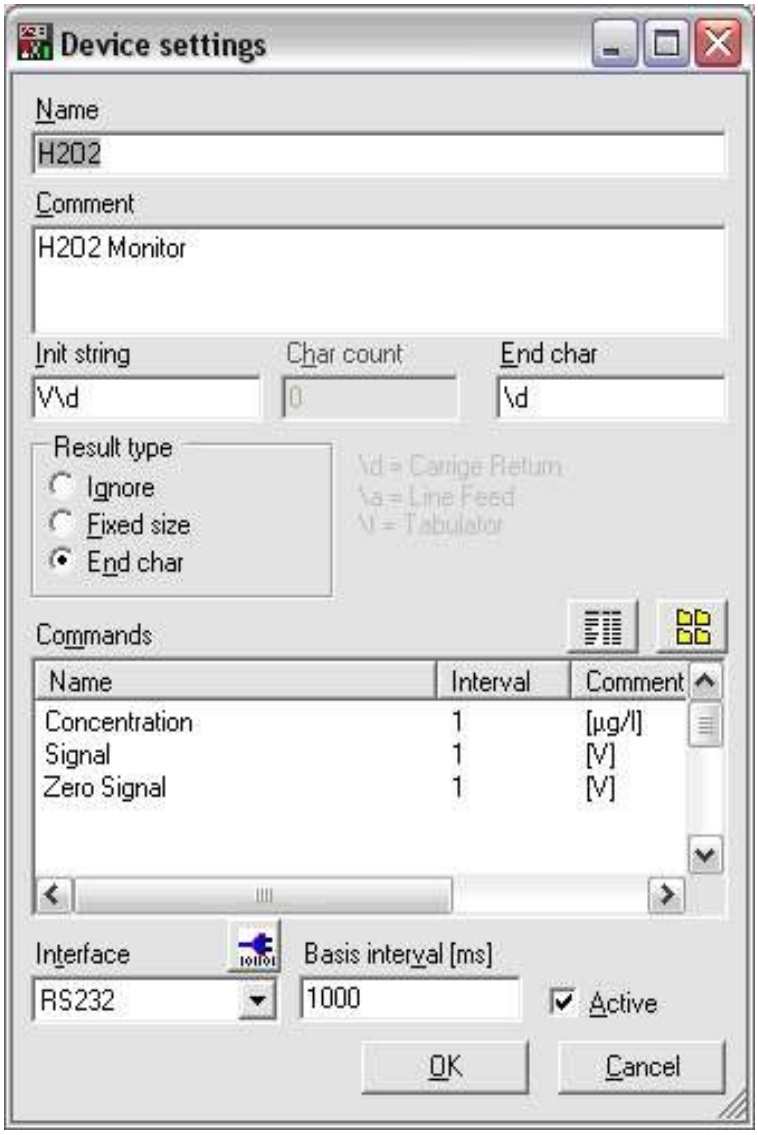
After importing, the configuration has to be opened. To open the configuration, select “Conf. Open”:



Select the imported configuration and open it. Now you can find the selected instrument in the field “Devices”:

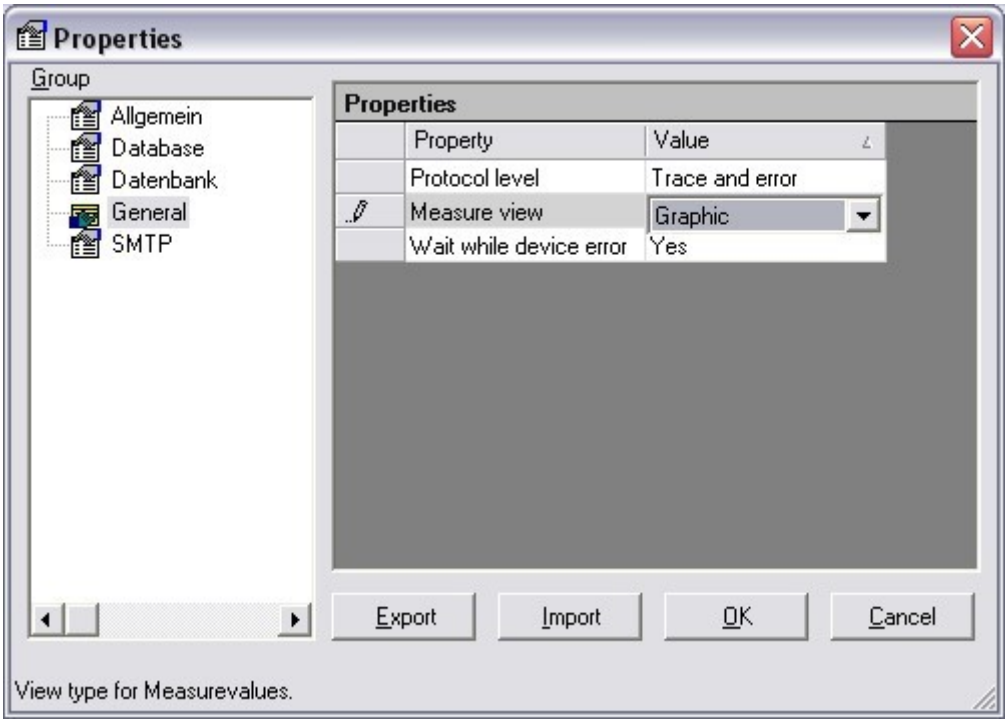


If you double click on the device (H2O2), you open the device settings:



Here you can set the parameters of the communication interface and edit commands.

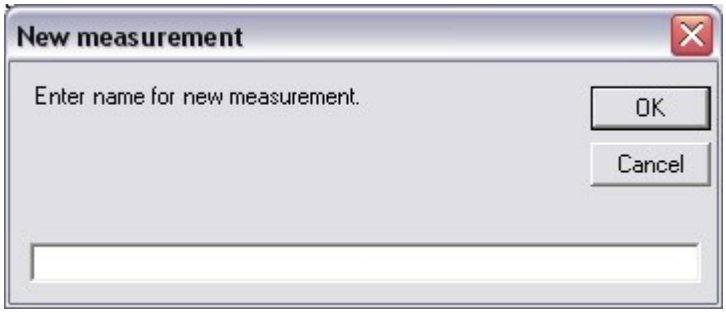
The factory setting of SqeezeDat only shows the measuring values as a text. To activate the graphic view, select “File” and “Properties”. The following window is shown:



In “General” you have to set “Measure view” from “Text” to “Graphic”.

6.1.3 Data Aquisition

In order to start a measurement, start SqeezeDat, select “Start” and enter a name for the measurement:



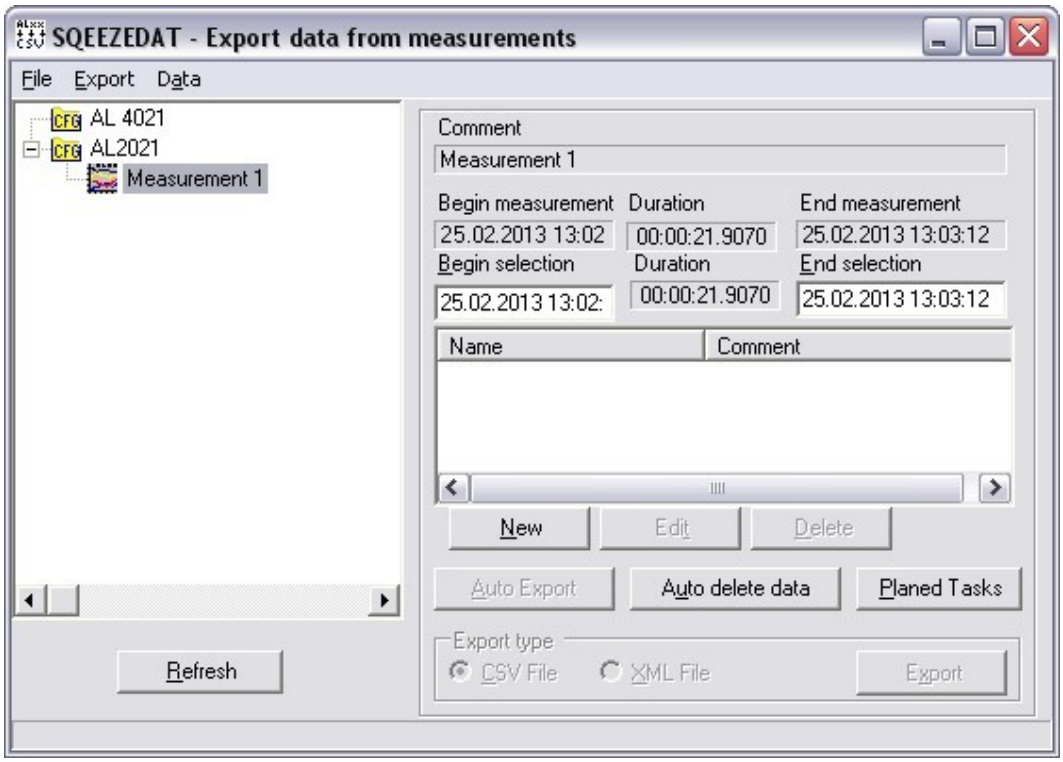
This window opens:

The left field shows all parameters which are recorded from SqeezeDat. To show the values as a text, you have to move the parameter with drag and drop into the field “Text”. To show parameters as a graphic, drag and drop the parameter into the field “Graphic”.

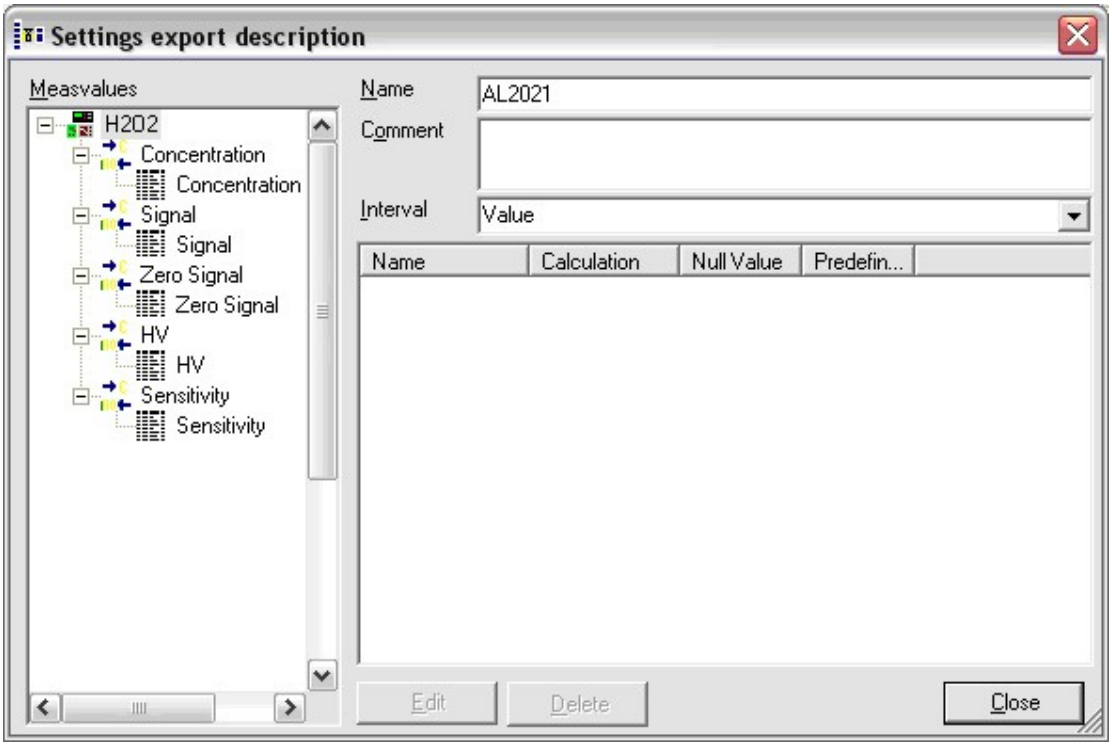
To stop the measurement select “Stop”.

6.1.4 Data Export

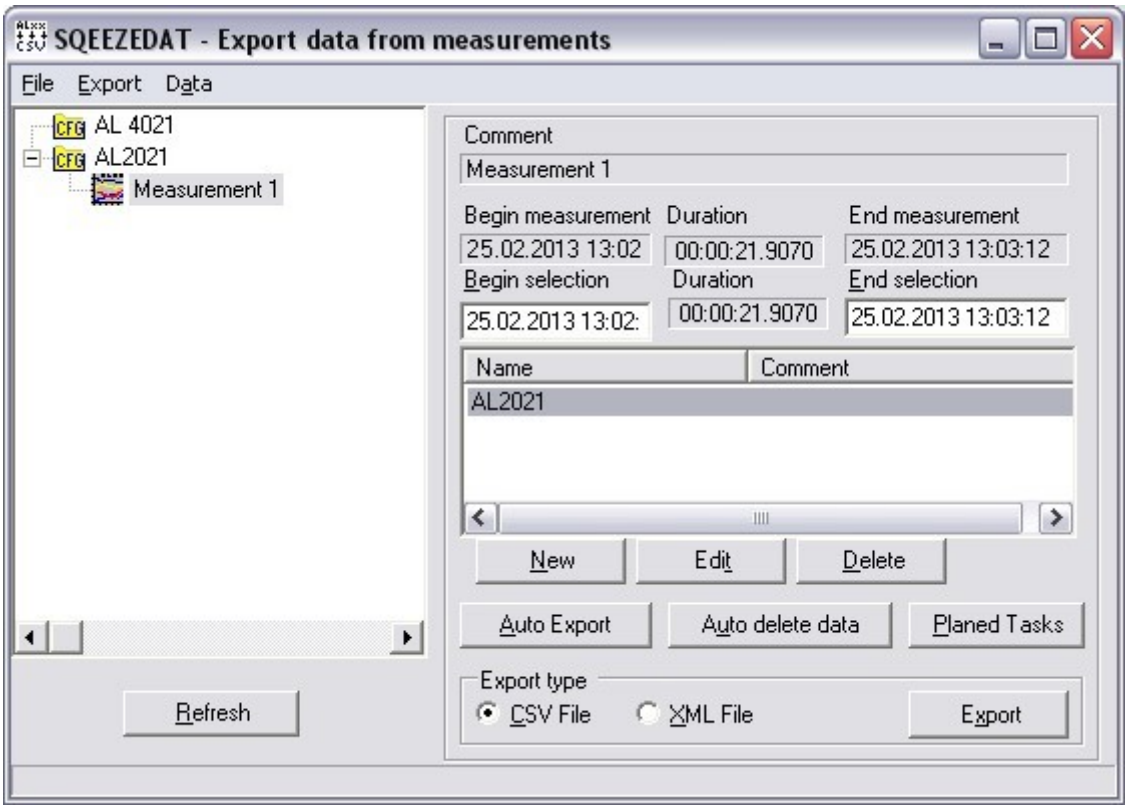
For exporting the data from the database SqeezeDatExport is used. It is possible to export old data from the database or to export data from a running measurement. After SqeezeDatExport is started, the following window is shown:



You can choose which measure values you want to export. Therefore an export configuration has to be created. Click on a measurement on the left side and click “New” to open this window:



Enter a name and a comment for the export configuration.
On the left side you find the recorded measuring values. You can drag and drop the values which you want to export into the white field on the right side. The configuration is saved with “Close”.



Now you click on the measurement you want to export and the export configuration which you want to use. Choose if you want to export the data as CSV file or XML file and start with “Export”.

To delete a measurement mark it and press “delete” on your keyboard.

6.2 Start-Up Procedure

Open the cover of the instrument. Remove the tape which fixes the white cassettes of the peristaltic pump. Switch on the device. Close the cassettes of the peristaltic pump.

Note:
Colored stoppers must fit properly to the cassettes (see chapter 7.2)!

Put the liquid inlet tubes into 1 molar NaOH and rinse for 20 minutes while changing from gas to liquid measuring mode or vice versa (e.g. 10min LL/GL – 10min LG/GG)

Connect the tubing to the solutions. Follow the colored markings.
You can check the flow of the chemicals by shortly lifting off the tubes out of their reservoirs. Slowly moving bubbles should be observed in the tubes.

6.3 Calibration/Zeroing

The AL2021SC uses a two point calibration. A gas or liquid standard with known concentration is used to adjust the photomultiplier to the measuring range. The zero signal is determined with the internal zero trap.

6.3.1 Measuring Stripper Speed (if no Liquid Flow Meter is installed)

For measuring gaseous concentrations, H_2O_2 is solved into the stripping solution. The concentration of H_2O_2 in stripping solution is measured in the liquid phase. For calculating the correct concentration in gas phase, the speed of the stripping solution has to be measured.

If the optional liquid flow meter is mounted, the stripper speed is measured automatically.

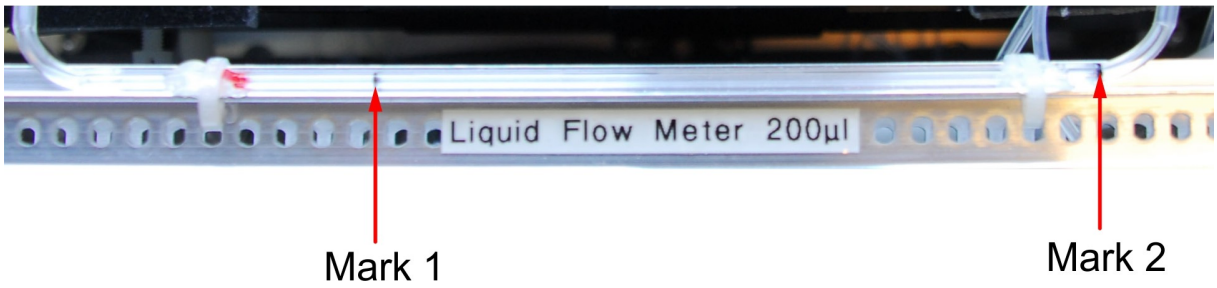


***Fig. 6-1**: Capillary for measuring the stripper speed. A bubble is produced to measure the time, which the solution needs to pass from mark 1 to mark 2. The volume between these marks is 200µl. The time which the bubble needs is used for calculating the stripper speed.*

To measure the stripper speed a bubble is produced. This is done by lifting the pump tube of the stripping solution (grey/grey) from its connector shortly. The volume between the two black markings is 200µl. Measure the time which the bubble needs to pass from the first to the second mark.

6.3.2 Liquid Calibration

Prepare a liquid standard which suits your application (e.g. 30µg/L). For your convenience use the standard concentration calculator (see Chap. 5.6.7). Set the parameters for in the Setup/Calibration and Setup/Wait Times menu (see Chap. **Fehler! Verweisquelle konnte nicht gefunden werden.** and 5.6.2). Connect the "Standard/Sample" tube (at the front of the device) to the liquid standard. Go to the Calibration menu and press "Start Liquid Calibration".

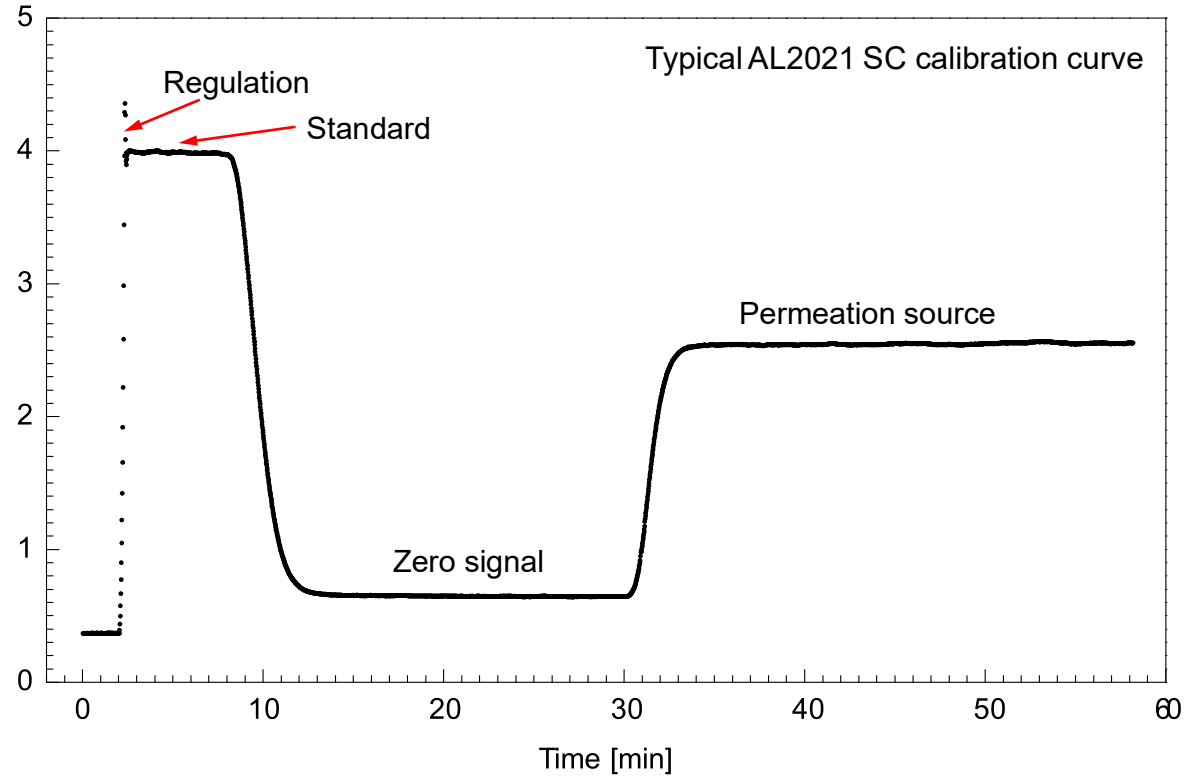


***Fig. 6-2**: Typical liquid calibration. After stabilization of the standard signal, the high voltage of the photomultiplier is regulated to 4 Volt. Then the signal of zero air is determined. After the successful calibration, the emission of the permeation source is measured.*

There is no concentration signal output. Error bits are set at the digital output and analog output during calibrations and zeroings.

6.3.3 Gas Calibration

(requires a permeation unit)

Note:
The permeation source has to run at least 24 hours for stabilization!

Before running the first gas calibration, you have to perform a liquid calibration.
This is necessary to measure the permeation rate, which will be used as standard in the gas calibration procedure.

Go to the Calibration menu and press "Start Gas Calibration". As zeroings, gas calibrations can be scheduled automatically using AutoSequences. For more details on programming AutoSequences see chapter 5.6.2.

6.4 Measurement

For measuring gaseous samples, the sample inlet at the rear of the device is used. A tube with 6 mm outer diameter can be connected.
Liquid samples are measured via the “Standard/Sample” tube at the front of the device. Either the liquid or the gas calibration (if available) can be used. Select the calibration in the Data menu.

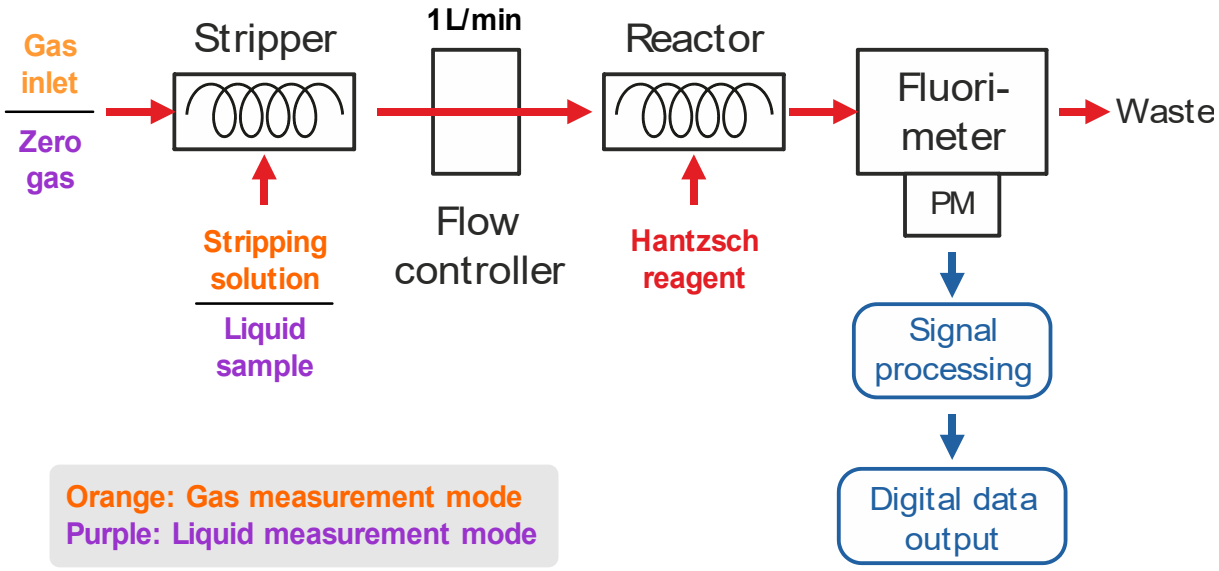


***Fig. 6-3**: The flow scheme shows the difference between measurement of liquid samples (purple) and gaseous samples (orange). Gaseous samples are provided by the inlet at the rear of the device. If a liquid sample is measured, the inlet at the front of the AL2021SC is used. Instead of Stripping solution the liquid sample is sucked in and the air is cleaned from formaldehyde by an internal formaldehyde scrubber.*

6.5 Auto Mode

The Auto Mode allows running calibration and zeroing cycles automatically. For automatic gas calibration the permeation unit is required (optional). The instrument uses the gas calibration mode for measuring in Auto Mode. This means before starting the Auto Mode you need to perform a liquid calibration. Automatic zeroing is possible without a permeation unit. Recommended gas calibration frequency is once a day. The auto calibration procedure takes approximately 30 minutes, depending on the times set in the Setup/Wait Times menu.

6.6 Switch Off Procedure

To switch off the instrument disconnect all solutions. If the device is switched off for more than 1 day, the tubes should be flushed with deionised water. Therefore connect the inlet tubes to deionised water and flush the instrument for about 30 minutes. Then disconnect the tubes from the water and let the device run empty.
To accelerate flushing switch into Fast Flush mode in the Stb./Flush menu. This will speed up the procedure by a factor of ~5.

Note:

Always release the pump cassettes when the instrument is not in use!

7 Maintenance

7.1 Cleaning

To clean the instrument put the liquid inlet tubes into 1 molar NaOH and rinse for 10 minutes while changing from gas to liquid mode or vice versa. Then rinse the tubing again with deionised water for 30 minutes.

7.2 Tubes and Replacement

The pump tubes have a lifetime of approximately 2 weeks if the device is operated under 24/7 conditions. After that time the flow will become unstable and will cause signal noise which has a negative effect to the accuracy.
If the AL2021SC is used under other conditions, the lifetime changes and can be calculated.

To replace the tubes disconnect the solutions and let the device run empty. Then open the cover, release the cassettes, remove and replace the tubes according to the scheme and color code below.

Pump tubes and color code

Waste:	Black / Black
Debubbler Coil:	Red / Red
Debubb. Str.:	Black / Black
Stripp. Solution:	Orange / Orange
Sample:	Black / Black
Fluor. R. :	Orange / Yellow
NaOH:	Orange / Yellow

Note:
Make sure that the peristaltic tube stoppers are properly installed in the peristaltic pump and that the inlet tubes are not kinked or bent!

Pump tubes have a longer life time when the outside is greased with silicon grease.

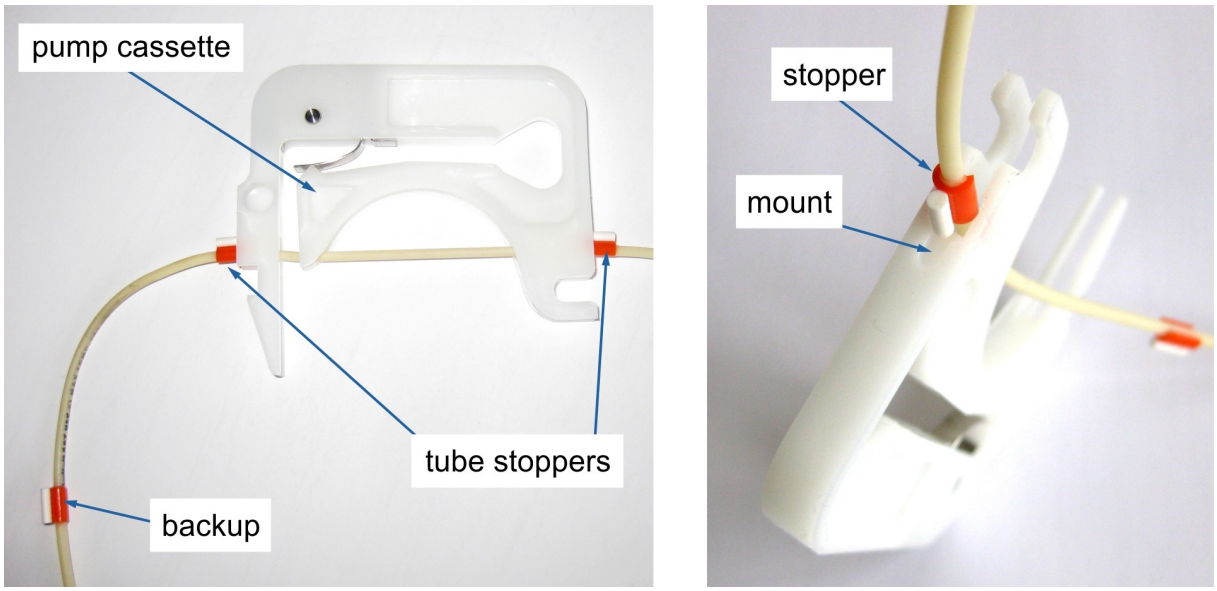


***Fig. 7-1**: Take care that the stoppers of the tube are inserted correct to the mount. There are two segments which can be used for pumping. Each segment can be used for about 2 weeks (under 24/7 conditions).*

7.3 Daily

- Check level of solutions in the bottles and waste canister
- Check the inlet tubes for damages and kinks or bents
- Check for leaks inside the instrument
- Check the lamp voltage

7.4 Weekly

- Check if the air filter is dirty
- Check the air flow rate
- Inspect the sampling tubes for dirt
- Change position of the pump tubes every 2 weeks, when operated 24/7

7.5 Monthly

- Replace the pump tubing, lubricate the outside with silicon grease
- Check the permeation rate
- Check stability of zero signal

7.6 Annual

- Replace the zero trap material
- Calibrate flow controller

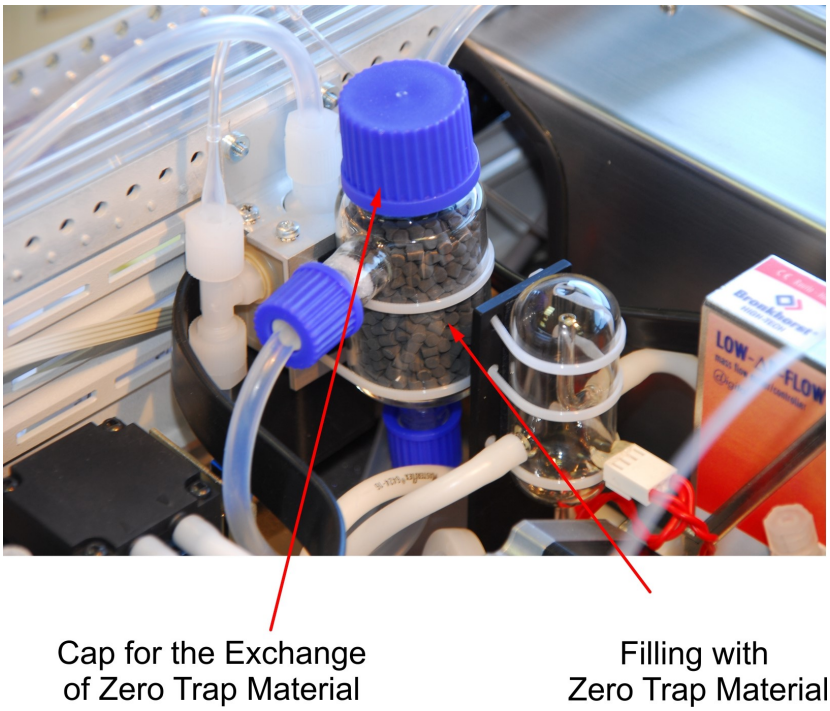


***Fig. 7-2**: Zero Trap. To replace the zero trap material, the zero trap has to be dismounted. Then open the cap and replace the filling.*

8 Troubleshooting

8.1 System is Not Booting

- Check AC power supply
- Check if the device is switched on
- Check the fuse at the switch
- Check if all LED's of the internal DC power supply glow
- Check if the LED's of the AD/DA, I/O and QTC boards flash

8.2 No Lamp Signal

- Check the cables of the UV-LED board
- Check DC power supplies

8.3 Peristaltic Pump

8.3.1 Pump does not rotate

- Check if there is a liquid trap error
- Check 24 Volt power supply
- Check if the cable of the pump is connected

8.4 Bubbles in Cell

8.4.1 If debubbler operates normally

- Check the cell connectors for leaks

8.4.2 If bubbles are too large for debubbler

- Check the inlet tubes for blocks and damages
- Check if the tubes are connected to the solutions
- Check if you are in gas or liquid measurement mode

8.4.3 If bubbles are irregular in size

- Check all connections and tubes inside the instrument for leaks

8.5 Air Flow Rate Too Low

- Check if the sample air inlet is blocked
- Check the particle filter for dirt
- Check if the tubes of the air flow path are blocked
- Check the flow controller settings in the Setup/System menu

8.6 "Liquid Trap Error"

- Check if the sample air inlet and waste out are blocked
- Check for leaks in liquid system
- Check if the pump cassettes are closed before the inlet tubes are connected
- Clean and dry liquid trap and tubing
- Check particle filter, flow controller, zero trap and liquid valve for liquids inside; dry and clean or replace them

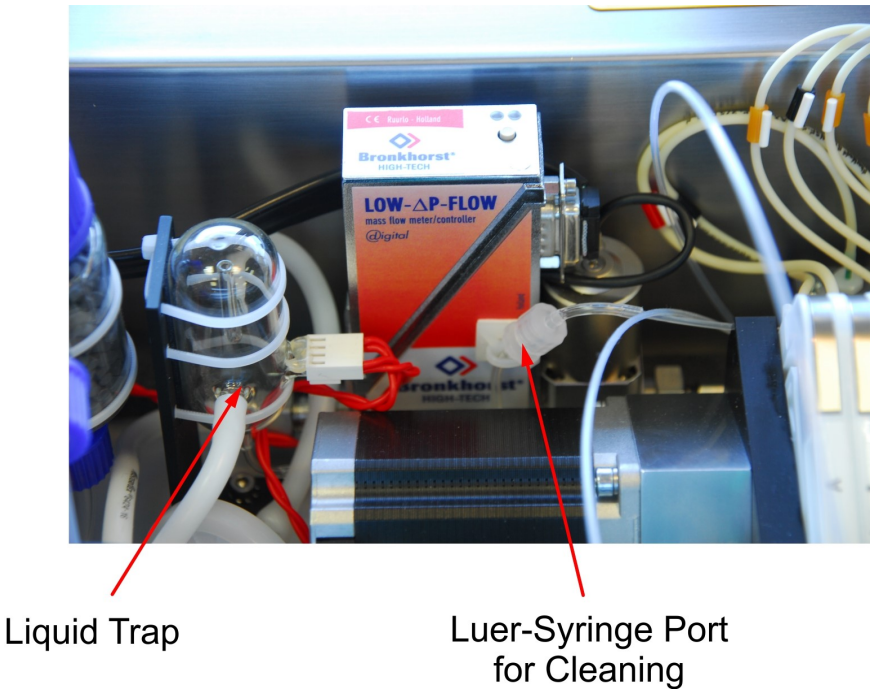


***Fig. 8-1**: Liquid Trap. In case of liquid trap error check if there is liquid in this trap. To empty this trap, switch off the device, open the Luer-Syringe port and connect a Luer-Syringe to empty the trap.*

8.7 Calibration Not Possible

8.7.1 Not possible due to unstable signal

- Rinse tubes with 1mol NaOH for at least 10 minutes
- Check inlet tubes for kinks or blocks
- Change pump tubes
- Prepare new reagents
- Check is the Stability limits are too strict (Setup/System menu)

8.7.2 Unable to regulate HV (Regulation Timeout)

- Check if concentration of liquid standard is too high
- Check if the regulation timeout (Setup/Wait Times menu) is too short
- Check if the standard signal is stable

8.8 No Connection to PC

- Check if the instrument and the PC are connected with a null modem cable (Pin 2 and Pin 3 crossed)
- Check COM port and parameters of the COM port are the same in the instrument and the remote PC
- Check if another program on the remote computer is using the COM port
- If a USB to RS232 adapter is used, check if the driver is installed properly

9 Appendix

9.1 Analog Output (optional)

For analog monitoring of the raw signals or the measured concentrations, a strip chart recorder can be connected to the instrument via the analog output connector. The output voltage is 5 Volt maximum, where 4 Volt is equivalent to the standard solution. The zero signal depends on the measuring range and the quality of the used chemicals and water: It may reach up to 1.5 Volt.

Note:
The analogue port is not a serial port!
Do not connect a serial cable to this port!

Pin assignment of the Analog Port	
Pin 2:	Signal Out [V] (raw)
Pin 5:	Ground
Pin 6:	Concentration Out [V]
Pin 7:	Error Bit (TTL high)

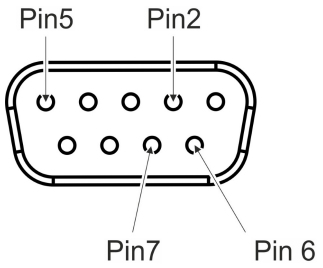


***Fig. 9-1**: Pin assignment of the ANALOG output. Pin 2: Signal Out; Pin 5: Ground; Pin 6: Concentration Out; Pin 7: Error Bit*

All signals are digitized every 989ms. The calculated concentrations are stored in a ring buffer in order to be able to average the concentration values. To display the actual concentration the instrument takes the average of a certain number of digitized signals and calculates the concentration by using the calibration factors.

9.2 Digital Output

For connecting the instrument to the PC, a null modem cable is necessary. If the remote PC is not equipped with and serial port, use the USB/serial adapter and the null modem cable. The RS232 interface parameters are:

- The baud rate is set in the Setup/System menu (See Chap. 5.6.5)
- No parity
- 8 data bits
- 1 stop bit

No hardware or software handshake is declared for the transmission.

9.2.1 Remote Commands

The commands are send out in ASCII and returned back to sender in ASCII-strings. The basic structure is as following:

<CMD>[Param]CR

The commands are arranged in three groups:

1. Set / inquire parameters
2. Inquiry of concentration and signals
3. Instrument control

The following commands are defined. The command for setting parameters takes care of the actual priority mode. In wrong mode the error message “ERR_2” occurs.

Command	P 1	P 2	P 3	Description
A				Gets Flag (see Appendix 9.4)
B				Gets sensitivity [V/(µg/L)]
C				Gets concentration [µg/L]or [pbb], if the gas concentration unit is not changed.
D				Gets date (mm.dd.yyyy)
d				Gets date (dd.mm.yyyy)
F				Gets air flow [L/min]
H				Gets high voltage [V]
K	G|L|Z|S|T			Starts calibration, zeroing, or determination of stripper speed G = gas calibration L = liquid calibration Z = zeroing S = stripper speed T = terminates a cal/zero/ stripper speed measurement, if running. Set start times in Setup/Auto Cal/Zero
M	G|L	[G|L](*)		Set measurement mode: M G = gas meas. with current calib. M L = gas meas. with current calib. M G G = gas meas. with gas calib. M G L = liquid meas. with gas calib. M L G = gas meas. with liquid calib. M L L = liquid meas. with liquid calib.
L				Gets lamp voltage [V]
N	0|1			Starts/stops data logging
p	0 – F			Sets peristaltic pump speed
R				Gets liquid flow [L/min]
S				Gets signal [V]
s				Gets averaged signal [V]
T	P(*)			Gets temperature [°C] P = permeation oven
t				Gets time stamp in the format defined in Setup/File Options
U				Gets time (hh:mm:ss)

v				Gets liquid pump speed (hex: 0 – F)
V				Gets version string
W				Gets serial number
X	0 – F			Switches external valve 1 – 16 on and closing the valve formerly open
x				Gets number of external valve currently open (1 – 16)
Y	mm/dd/yyyy	hh:mm:ss		Sets date and time
y	dd.mm.yyyy	hh:mm:ss		Sets date and time
Z				Gets zero signal [V]
%	S|Z|P	0|1		Switches internal valve S = sample valve Z = zero valve P = permeation valve
#				Standby mode on/off
+				Fast Flush mode on/off

(*) If no permeation unit is installed, the second parameter is ignored.

9.2.2 Error Messages

Errors are indicated with error numbers. To identify error messages the system puts "ERR" ahead of the number, i.e. "ERR_1" = not recognized command.

Error #	Description
ERR_1	Invalid command
ERR_2	Wrong operation mode
ERR_3	Wrong password
ERR_4	COM port malfunction
ERR_5	Remote administration malfunction
ERR_6	Not enough parameters
ERR_7	Wrong syntax in parameters
ERR_8	Malfunction while transmitting parameters
ERR_9	Too many parameters transmitted
ERR_10	Unknown parameter ID request
ERR_11	Ring puffer empty
ERR_12	Calibration / zeroing running
ERR_13	Device is defect
ERR_14	No USB-stick or disk full
ERR_15	No valid liquid calibration
ERR_16	No valid gas calibration

9.3 Peristaltic Pump Speeds

The hexadecimal number of the pump speed codes the following values:

0	1.0 rpm (Standby)	8	5.0 rpm
1	1.5 rpm	9	6.0 rpm
2	2.0 rpm	A	7.0 rpm
3	2.5 rpm	B	8.0 rpm
4	3.0 rpm	C	10.0 rpm (Factory default)
5	3.5 rpm	D	12.0 rpm
6	4.0 rpm	E	15.0 rpm
7	4.5 rpm	F	40.0 rpm (Fast Flush)

9.4 Definition of the Status Flag

The bits of the Status Flag can be read as follows:

Bit 0:	Normal Mode (*) Yes/No
Bit 1:	Calibration running Yes/No
Bit 2:	Zeroing running Yes/No
Bit 3:	Averaging stripper speed On/Off
Bit 4:	AutoSequence scheduled Yes/No
Bit 5:	AutoSequence running Yes/No
Bit 6:	Standby On/Off
Bit 7:	Fast Flush On/Off
Bit 8:	Data Logging On/Off
Bit 9:	Calibration valid Yes/No
Bit 10:	Calibration mode: 0 = Liquid, 1 = Gas
Bit 11:	Measurement mode: 0 = Liquid, 1 = Gas
Bit 12 – 15:	Reserved
Bit 16	Sample Valve On/Off
Bit 17	Zero Valve Valve On/Off
Bit 18	Perm Valve On/Off
Bit 19	HCl Valve On/Off (not used by AL2021SC)
Bit 20	Cat Valve On/Off (not used by AL2021SC)
Bit 21 – 23	Reserved
Bit 24 – 27	Currently Open External Valve (0 – F) eq. Valve 1 – Valve 16 (**)
Bit 28 – 31	Liquid Pump Speed (0 – F)

(*) Normal mode means that no process like calibration, etc. is running.
(**) Only available, if an external valve controller is installed.