

**CAMMAG**<sup>®</sup>



# INSTRUCTION MANUAL ADC 2



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## 1. Introduction

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### 1.1. CAMAG ADC2

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The Automatic Development Chamber (ADC2) permits reproducible chromatogram development without the need for constant monitoring by the user. The ADC2 is shipped with a RS232 cable and built-in interface for connecting to a computer with vision-CATS/winCATS from which the instrument can be run but it can be used in Stand-Alone mode too.

By means of ADC2, developing of HPTLC chromatograms will be reproducible. The user is free from any process monitoring.

The user decides whether and how to precondition the plate, pre-selects the distance to be migrated by the solvent system and the drying parameters.

Development proceeds automatically and under controlled conditions. A CCD-sensor monitors the progress of the solvent front; the distance covered and the time elapsed are continuously displayed. On completion of development, the HPTLC plate is dried with air and finally stays safely protected in the chamber until removed by the user.

If used in Stand-Alone mode all method parameters are entered via the keypad. Ten complete development programs can be saved in nonvolatile memory and started at will without PC-control.

HPTLC plates with a thickness of up to 2.5mm can be used in the ADC2. If using aluminum foils it is mandatory to utilize a special foil holder # 115.8363.

The ADC2 controlled by the software permits full control of the development process by monitoring and reporting all parameters including all TLC steps of an analysis, i.e. definition of plate material, sample application, derivatization, development and evaluation. All data are handled in a cGMP/cGLP compliant environment. With the appropriate software option, the ADC2 can be used in a 21 CFR Part 11 compliant environment.

### 1.2. Precautions

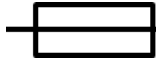
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- Please read this operating manual before starting the installation! This manual contains information and warnings the user has to follow to ensure reliable operation of the instrument
- If the instrument is used in a manner not specified in this manual, the protection provided by the equipment may be impaired



- Some interior parts of the instrument are under AC power. Careless and improper use can cause injury. Unauthorized manipulations can cause damage
- This sign indicates (on instrument and in this manual) that failure to take note of the accompanying information may result in damage of the instrument
- The instrument is manufactured and tested in accordance with the respective European safety publications shown on the Declaration of Conformity (DoC). The instrument complies with safety class 1 and has been designed for indoor use only (IP 20). Further, this device has passed the CAMAG Quality Assurance tests and has been delivered in safe operation condition. For detailed instrument data see chapter technical data
- Attention: For safety reasons the instrument may only be used for the purposes described in the operating manual
- To avoid injury use adequate safety equipment (protective goggles, gloves etc. if applicable) when working with the instrument
- Before first operation, check whether the voltage shown on the instrument matches your local mains voltage. The power cord may only be connected to a grounded, fused (not higher than 16A) outlet. Do not use extension cords without ground contact
- When working with the fluids of the instrument, be sure to take the appropriate caution (protect your eyes from direct contact with liquid)
- Risk of finger squeeze between moving parts; be careful closing the door
- The instrument may be used only by properly trained laboratory staff
- The instrument may not be used in rooms with danger of explosions
- The instrument contains highly sophisticated electronics and optical parts. It may be operated only in a non-condensing atmosphere in the temperature range outlined in the chapter "Technical Data". Before installation and use, the instrument should be acclimated properly
- Use a damp lint free cloth for cleaning the instrument surface. Do not employ aggressive detergents
- Protect yourself and the instrument from electrostatic shock which can cause damage to the electronic parts

- Only authorized personnel may open the instrument. Service and repair is only to be performed by trained specialists. Use spare parts and consumables supplied by CAMAG only. The warranty is voided if parts from other sources are used. Check the service manual before you start service to reduce product-specific risks
- The power cord has to be removed before the instrument is opened. It is not permitted to work on an instrument that has been opened and is connected to the power supply



- Spare fuses must be of the type specified by the instrument manufacturer. It is forbidden to short-circuit or manipulate fuses
- Use only the original, with the instrument delivered power cord type
- If the instrument is found to be defective, it must be switched off and steps must be taken to ensure that it cannot be switched on by mistake
- If liquids penetrate the inside of the instrument, the power has to be disconnected immediately. Small amounts of liquid can be wiped off and/or dried by means of a hairdryer, with larger amounts of liquid a service technician has to be called. A test of functionality has to be performed in all cases
- Carry out all safety checks and the preventive maintenance as recommended by the manufacturer in order to assure your personal safety and the full functionality of the instrument. Have an authorized service specialist perform any service not described by this manual
- See original manufacturers' manuals for further safety data on third party equipment supplied with the system
- Lift/move/transport the system with the necessary care and with sufficient manpower (install the transport security devices if applicable, transport it only in the original packaging)
- The safety of any system incorporate with the equipment is the responsibility of the assembler of the system



- This symbol indicates that this equipment must not be disposed of as unsorted municipal waste but is to be collected separately as electrical and electronic equipment (WEEE-Directive 2002/96/EC). To properly recycle the instrument or parts of it you are requested to send the equipment back to the distributor, producer or an adequate collection system at the end of its life. This will have potential effects on the environment and human health

- The use of the instrument without adequate ventilation to outside air may constitute a health hazard depending on the substances in use

### 1.3. Parts supplied

Part no	Description
140.8350	Tool kit, comprising:
672.0059	Exhaust tube
125.1021	RS232 connecting cable
362.0006	Fuses 1AT (2 pc)
705.0012	Allen wrench 3mm
960.0200	Cap for solvent inlet (2 pc)
700.1011	CCD-test plate ADC2/AMD2
115.8357	Plate holder
022.5261	Twin Trough Chamber
941.0006	Saturation pads (pack of 25)
	Respective power cord
B.022.8350E	Instruction manual (English)

### 1.4. Options

Part no	Description
022.8360	Humidity Control Module
022.8373	Foil holder

### 1.5. Spare parts

Part no	Description
022.5261	Twin Trough chamber
022.8370	Saturation pads (pack of 100)
022.8372	Plate holder
022.8373	Foil holder
672.0059	Exhaust tube
960.0200	Cap for solvent inlet
022.8375	Filters for Humidity Control Module (pack of 10)
105.8352	Humidity control tank complete
956.0094	Humidity control tank (glass only)



## 2. Installation

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### 2.1. Instrument

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- ☞ *Observe the environmental requirements (2.2 Installation environment) when setting up the instrument.*
- ✓ Carefully unpack all components and accessories listed on the shipping list from the upper part of the packing. Make sure the shipment is complete.
- ✓ Remove the instrument carefully from the package and place it on a table.  
**Attention:** Do not pull out the instrument by holding it at the blue top-cover, hold the instrument at the bottom when taking it out of the package and carrying it around!
- ✓ Remove carefully the fixation under the Drying module.
- ✓ For shipment, the Twin Trough Chamber has been securely positioned below the bottom of the instrument.



Fig. 1: The ADC2 instrument



Fig. 2: The instrument ADC2 (opening the front door)

## 2.2. Installation environment

The place for installation must meet the following requirements:

Bench space	Width 320mm Depth 320mm without Humidity Option Depth 460mm with Humidity Option (additional for tubes 250mm) Height 520mm Weight 18.5kg
Operating temperature	The temperature should be within a range of 18 to 30 degrees centigrade and free from significant variations.
Humidity	Humidity and temperature conditions must not cause condensation.
Atmospheric conditions	Adequate ventilation free from acidic, alkaline or other gas that may corrode metal or painted surfaces must be secured.



**Further requirements:**

- Do not place the instrument in a location where the temperature significantly changes (e.g. under an air conditioning duct or by a window). Significant changes in temperature will affect the performance of the unit
- Do not place the instrument in direct sunlight. Direct sunlight will disturb the measurement of the CCD sensor
- Do not use the instrument in an environment with moving ambient air (draft)
- Do not expose the instrument to any strong vibration or shock
- Avoid placing the instrument near equipment that radiates heat. Do not place the instrument near gas burners, electric heaters or ovens
- Do not place the instrument near equipment that generates intense magnetic fields such as electric welding equipment, high frequency furnaces, pole transformers, etc.
- Protect the instrument from excessive dust
- Connect the instrument to power lines that are free from sudden changes or voltage fluctuations
- If you must use power motor driven equipment (such as a stirrer or shaker) in the same line as your instrument, ensure that a noise reduction unit is in the same power line

### 2.3. Conditions for the installation

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Confirm that the following requirements exist before installing the instrument:

**Power supply and ground**

Line voltage: 100 – 240V (see rating plate on instrument).

Frequency: 50 / 60 Hz

Power capacity: 20 W

Ground terminal: A grounded outlet should be located within 2 meters of the instrument.

Connection for Exhaust tube

## 2.4. Mains connection

Use the supplied power cord for connecting the instrument to the mains outlet.



Fig. 3, Connections: 1 = RS232 Interface, 2 = Humidity module, 3 = Power switch and mains connection with fuse box.

## 2.5. Connection to the PC

The CAMMAG ADC2 is shipped with a 9-pin RS 232 interface cable for connection to the PC.

If you wish to use the instrument controlled by a software:

- ✓ Connect the cable to the instrument and to a free COM-port of the PC to enable data transfer.

## 2.6. Mounting the exhaust tube

The shipment contains an exhaust tube, which can be slipped on to the "black" air outlet fig.4, permitting exhaust solvent vapor to be directed into a fume hood or other vent.



*Fig. 4: Mounting the exhaust tube to the air outlet*

### 3. Getting started

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#### 3.1. Automatic instrument monitoring

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A sensor system controls and monitors the proper handling and guarantees a monitored developing process. In case of failure a signal will sound and an error message displayed. For error messages see chapter 6.8.

#### 3.2. Checking system functions

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Switch on the instrument. The ADC2 will now show the installed firmware version and perform the following checks and tasks:

1. Initializing the drying module.
2. Initializing the lift drive.

If startup was successful the display will show:

**\* \* ADC2 \* \***  
**SYSTEM READY**

In this phase, only physical blocking of a drive can cause an error. If necessary, check whether there are obstacles in the instrument.

## 4. Manual operation

### 4.1. HPTLC plate / plate holder / foil holder

The plate-holder is designed to attach and handle the HPTLC plates in the ADC2. When inserting a plate into the plate holder the layer side must be facing the user ('LAYER' side).

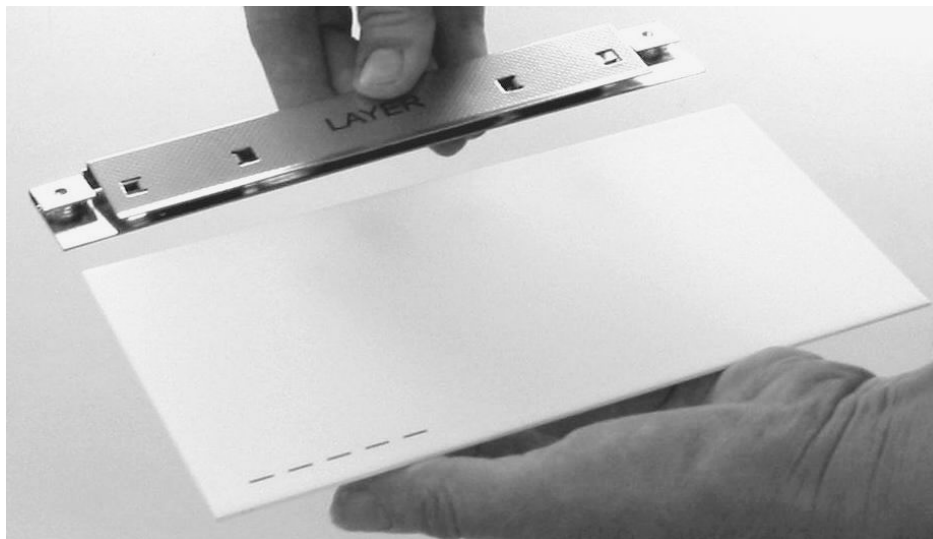


Fig. 5: Insert TLC plate in plate-holder

**Important:** While clamping tight the TLC plate the user should be aware the plate fits the inside stop position and the edges of holder and plate do not overlap.

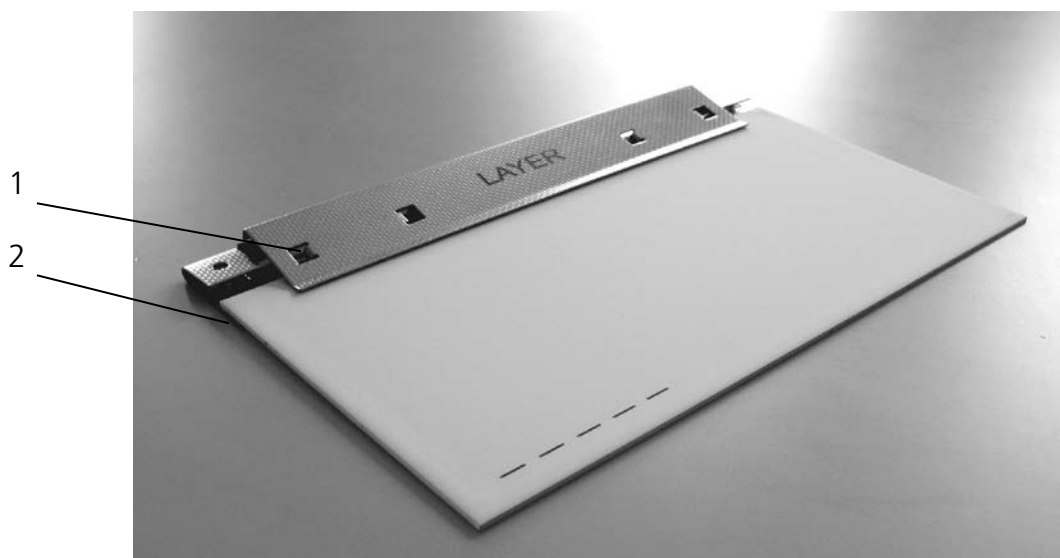
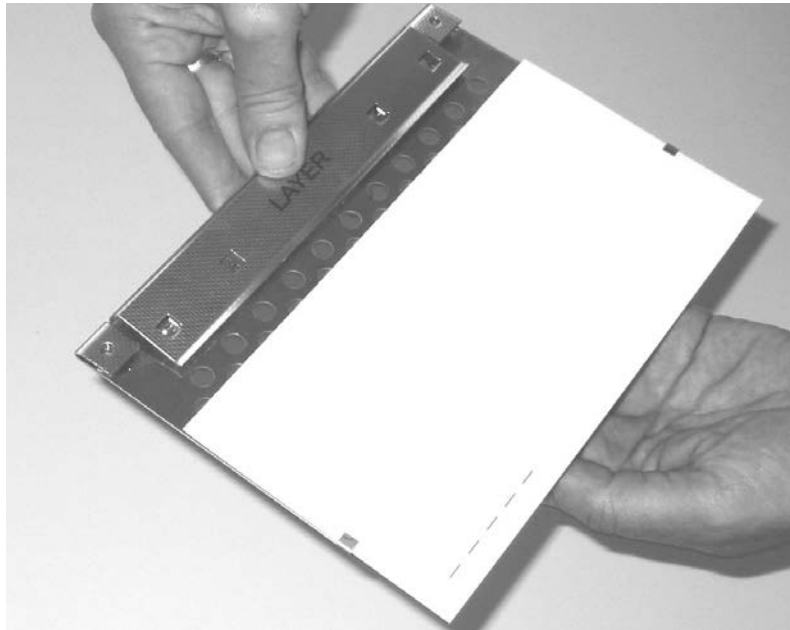


Fig. 6: Plate clamped in Plate-holder

**Important:** When clamping the HPTLC plate make sure the plate has hit the stop position (1) and the edges of the holder (2) do not overlap the glass plate.

**Note:** Clamping foils works similar to that of HPTLC plates; the two edges of the foil, left/right, must be inserted underneath the respective down holders, fig.7.



*Fig. 7: Mounting a foil in a foil-holder*

#### 4.2. Drying module and plate lift

**To attach the plate or foil holder into the drying-module make sure "LAYER" and substances are facing towards the inside of the drying module, fig.8.**



*Fig. 8: Fixation of plate holder in drying module*



### 4.3. Positioning of Twin Trough Chamber

To insert or take out the Twin Trough Chamber the lever (2) must be in the upper position, see fig. 9.

When the Twin Trough Chamber has been positioned inside the ADC2 make sure the chamber is touching the two stoppers at the back, otherwise chromatography cannot be started. The 'CAMAG' label of the Chamber should normally face the user; strongly inhomogeneous glass walls may thus require turning the chamber around.

After a correct positioning of the Twin Trough Chamber, turn the lever down in order to lower the drying module onto the edge of the Chamber (1).



Fig. 9: 1 = Twin Trough Chamber, 2 = lever

When a saturation pad is needed, make sure it has been inserted in the right position, i.e. the fold of the pad is contacting the bot-

tom of the Twin Trough Chamber while the shorter side is lying on the bulge of the chamber, fig. 10.



*Fig.10: Positioning of saturation pad*

#### 4.4. Developing and preconditioning solvent

The inlet reservoirs for developing (mobile phase) and preconditioning solvents are located on top of the ADC2. The left inlet is

used for developing solvent and the right inlet for preconditioning solvent. The volume of developing solvent should be 10ml, the preconditioning volume 25ml.

Developing  
solvent (10ml)

Preconditioning  
solvent (25ml)



Fig.11: Inlet reservoirs

## 4.5. Display and function of keys

### Control lamps

The control lamps in the display indicate the following status:

- POWER ON: this lamp is lit when the instrument is switched on.
- ON LINE: this lamp is lit when the instrument is ON-LINE with PC and software.

**The following keys are available:****DIALOG**

Activates the manual parameter input mode (Method stack and manual functions).

**ARROW KEYS** ▲ and ▼

Select the next / previous parameter (or method) in the dialog.

- ☞ If the "SYSTEM READY" display is active, pressing these keys will display the actual temperature and humidity. By pressing another key, these measurements will be cleared.

**ENTER**

Confirms the current parameter, switches to the selected dialog or (re-)starts the (last) selected method in manual function.

**END**

Interrupts the current application or switches back to the previous dialog.

- ☞ If the "SYSTEM READY" display is active, you can enter the manual function stack.
- ✓ Select any manual function by arrow keys ▲ or ▼.
  - ✓ Press **ENTER**, to start a manual function or **DIALOG**, if you want to change the manual parameters.
    - IQ-TEST (performs instrument's functionality tests)
    - HUMIDITY CTRL (performs a timed humidity process)
    - DRYING (performs a drying step)
    - RINSING (performs a rinsing step)
    - LAST RESULTS (displays the results of the last run)

**RESET**

Resets and re-initializes the instrument. The current method is interrupted but all parameters remain in memory.

**RUN**


Shows the selection of stored methods.

- ✓ Use the arrow keys ▲ or ▼ to select a method to start.
- ✓ Press **ENTER** to start the selected method or **DIALOG** to change the parameters of the actual method.

## 5. Stand-Alone operation

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The instrument can be used in Stand-Alone mode. Up to 10 methods can be stored and used locally. Further you can start 4 permanent programmed methods (S1 to S4).

 *Note that locally started methods cannot be used in an analysis created in **visionCATS/winCATS**, and that local parameter changes are not documented in the software!*

### **Procedure for Stand-Alone start:**

- ✓ Prepare the instrument for development.
- ✓ Press the **RUN** key.

The most recently used method is displayed. With the **▲** or **▼** keys you can select another locally stored method.
- ✓ Press the **ENTER** key to start the displayed method. The last method is re-started if **ENTER** is pressed in the "SYSTEM READY" mode.

### **Procedure for local input of parameters:**

- ✓ Press the **RUN** key.

The most recently used method is displayed. With the **▲** or **▼** key you can select another locally stored method.
- ✓ Press the **DIALOG** key to locally change parameters of the selected method.

Press the **▲** or **▼** key to select a parameter group (global parameters, parameters or save method) for local input and press **ENTER** to confirm.
- ✓ Now you can step through the parameters of the group by means of the **ENTER** key or use the **▲** or **▼** key to change the current parameter. To accelerate the change speed of a parameter you have to press the other arrow key a little bit later. Press **ENTER** to confirm the new value and move on to the next parameter.

### **Procedure for saving a changed method:**

- ✓ Select the parameter group **Save method** or press the **END** key after having changed the parameters.
- ✓ Select the memory position to save the new method by means of the **▲** or **▼** key. Press **ENTER** to confirm position and save the parameters.

**Procedure for local delete of a saved method:**

- ✓ Press the **RUN** key.  
The most recently used method is displayed. With the ▲ or ▼ key you can select the locally stored method to delete.
- ✓ Press the key combination **RUN-END** (press and hold **RUN**, then press **END**) and confirm deleting with **ENTER**.

 *Flow diagrams for these dialogs are depicted in chapter 6.*

## 5.1. Aborting an application in progress

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**Procedure:**

- ✓ Press the **END** key  
The development in progress is aborted and if the plate was immersed in solvent, a drying process will follow. An aborted development cannot be continued.

## 5.2. Resetting the instrument

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**Procedure:**

- ✓ Press the **RESET** key  
This will abort all activities in progress and re-initialize the instrument.

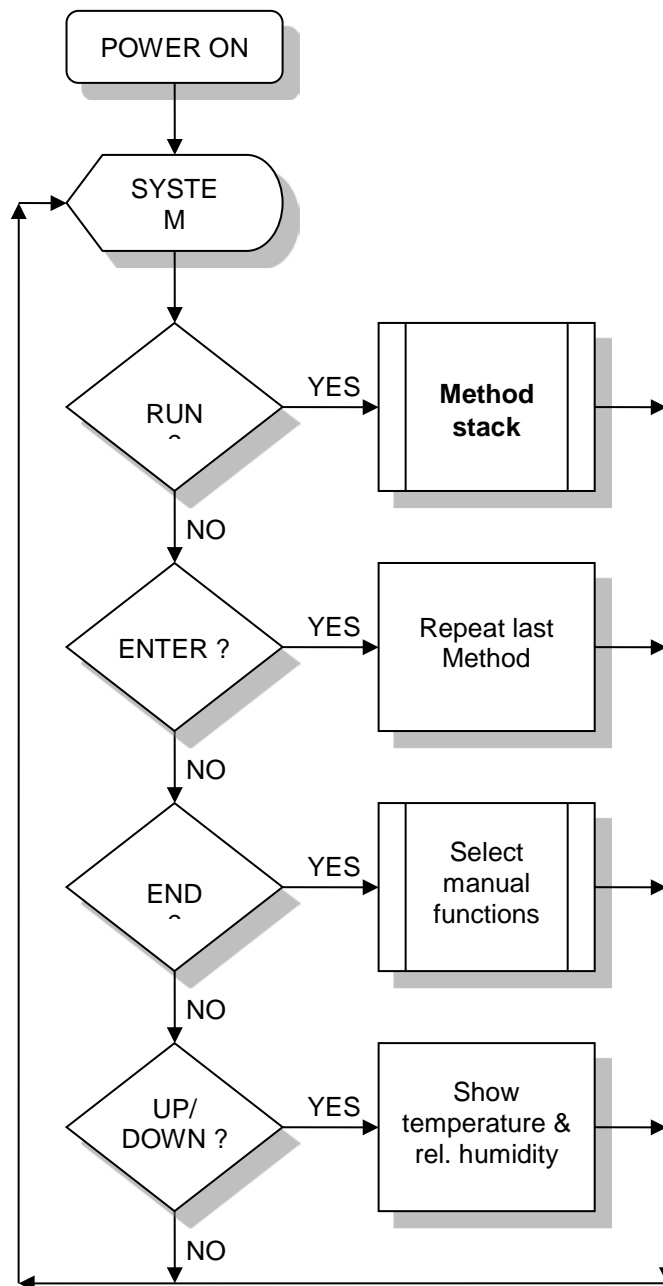


## 6. Display texts and diagrams

### 6.1. Texts and their meaning

Text	Description
<b>FW V: 1.00.01</b> <b>*****</b>	Shows the actual firmware version at startup.
<b>** CAMAG **</b> <b>* ADC2 SER. NO. *</b>	Display during initialization of ADC2 at power up.
<b>** ADC2 **</b> <b>SYSTEM READY</b>	The system was properly initialized and is waiting for a development.
<b>THIS METHOD ?</b> <b>METHOD 0-10</b>	Selection of a stored method.
<b>THIS FUNCTION ?</b> <b>MAN. FUNCTION</b>	Selection of a manual function.
<b>WAITING FOR</b> <b>SOMETHING</b>	Device is waiting for closing the door, moving the lever down or proper positioning of the Twin Trough Chamber.
<b>INIT</b> <b>DRIVE</b>	Initialization of specified drive.
<b>GO TO</b> <b>POSITION</b>	Lift or spindle drive are moved to specified position
<b>TEMP. HUMIDITY</b> <b>+24.9°C 40.1%RH</b>	The current temperature and relative humidity are shown.
<b>'ACTION'</b> <b>'REST TIME'</b>	An action is running (for example drying) and the rest time is shown.
<b>DEVELOPMENT</b> <b>45.3mm 22:45</b>	Development in process since 22 min and 45s. The migration distance is currently 45.3mm.
<b>OPERATION</b> <b>COMPLETED</b>	Development has been properly completed.
<b>** ERROR 38 **</b> <b>ERROR MESSAGE</b>	See chapter 6.8, page 26 for error messages.

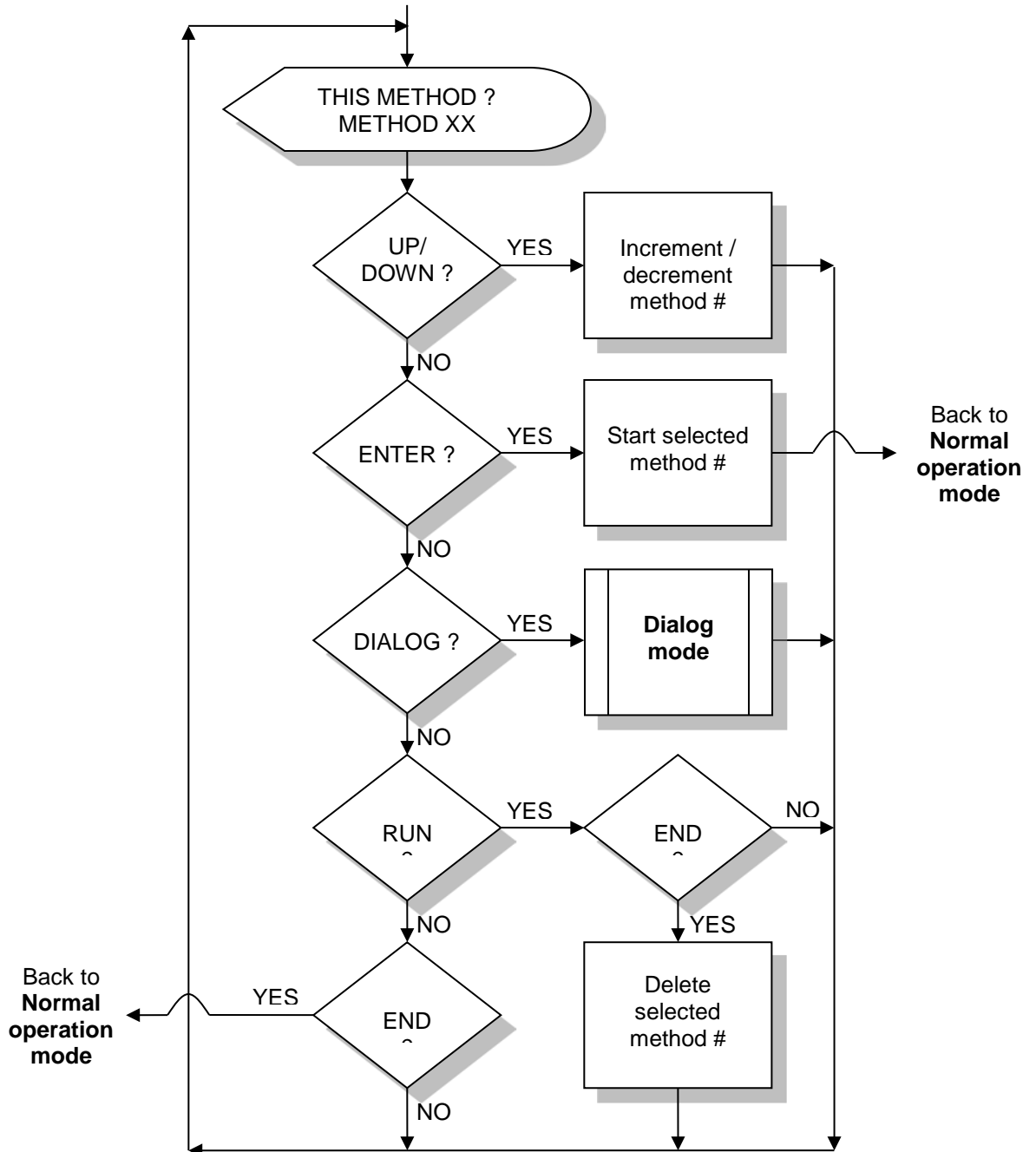
6.2. The Normal operation mode dialog



Boxes with two lines on both sides include another diagram, shown in the next pages.

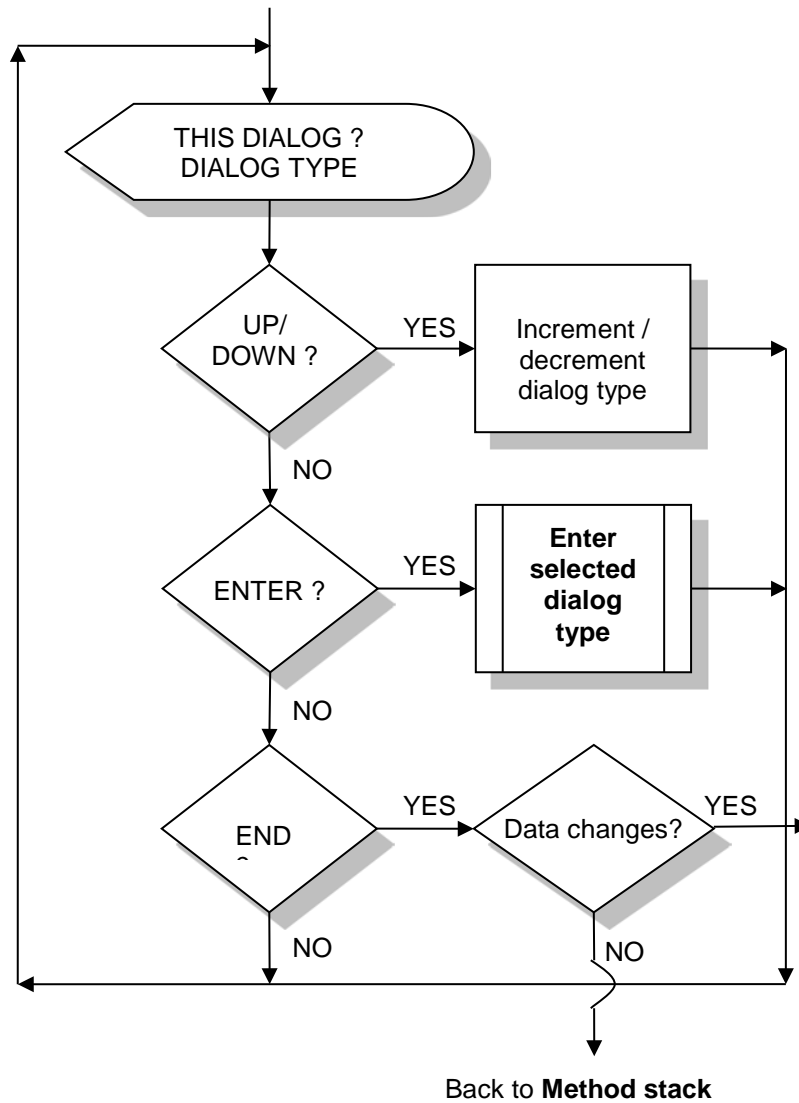
### 6.3. The method stack dialog

To enter the method selection dialog (Method stack), press the **RUN** key when the instrument display shows **"System ready"**.



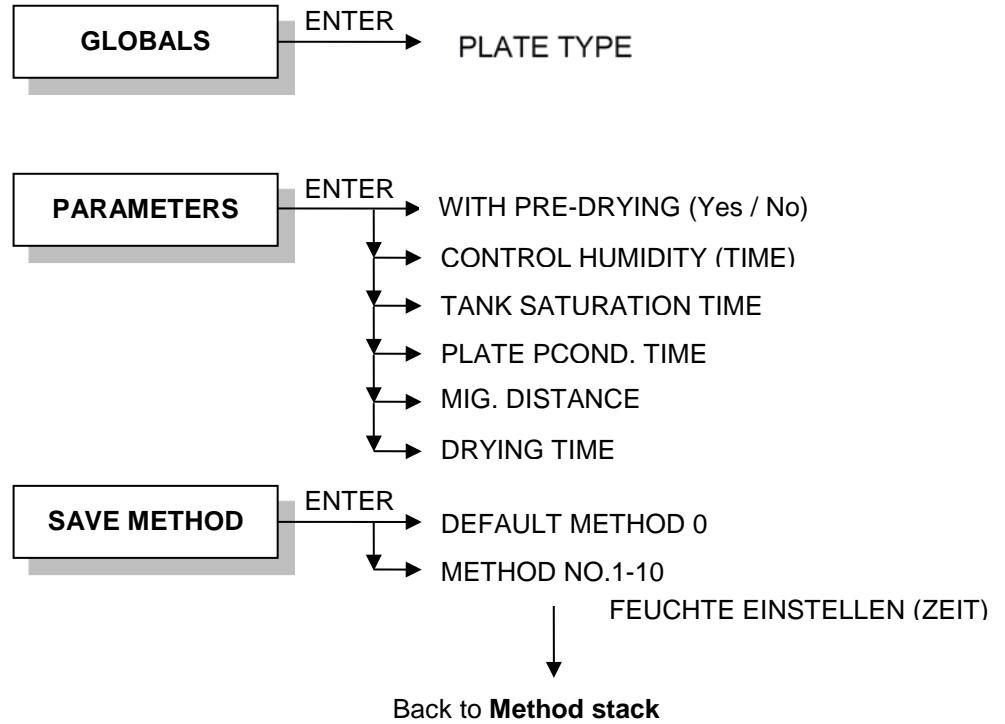
### 6.4. The Dialog mode

To enter this parameter type selection dialog, press the **DIALOG** key when the instrument display shows the name of the method to be edited (in the method stack dialog).



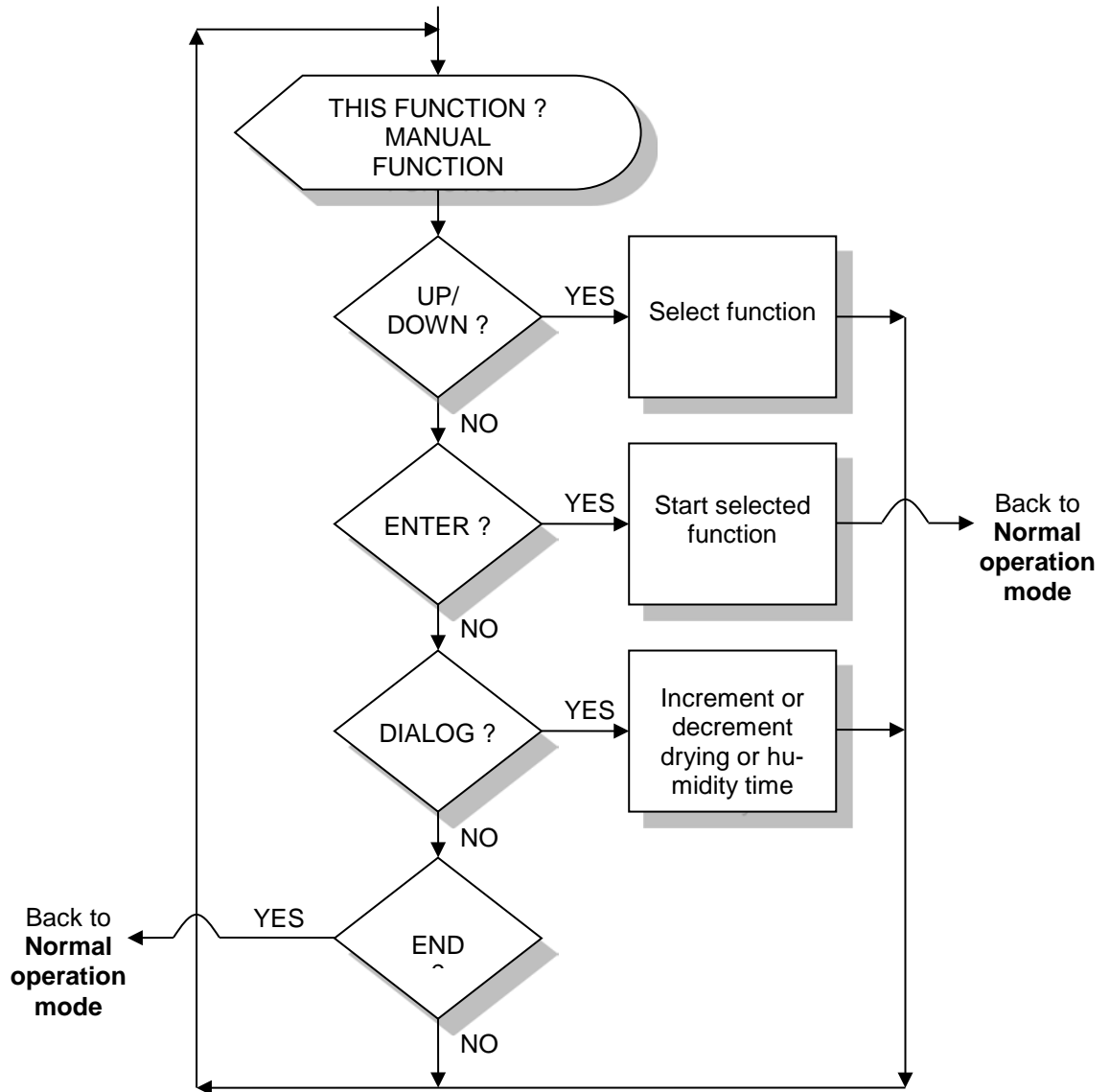
### 6.5. The Parameters Input dialog

To enter this parameter input dialog, press the **ENTER** key when the display shows the name of the parameter type to be edited (in the Dialog mode).



### 6.6. The Manual functions dialog

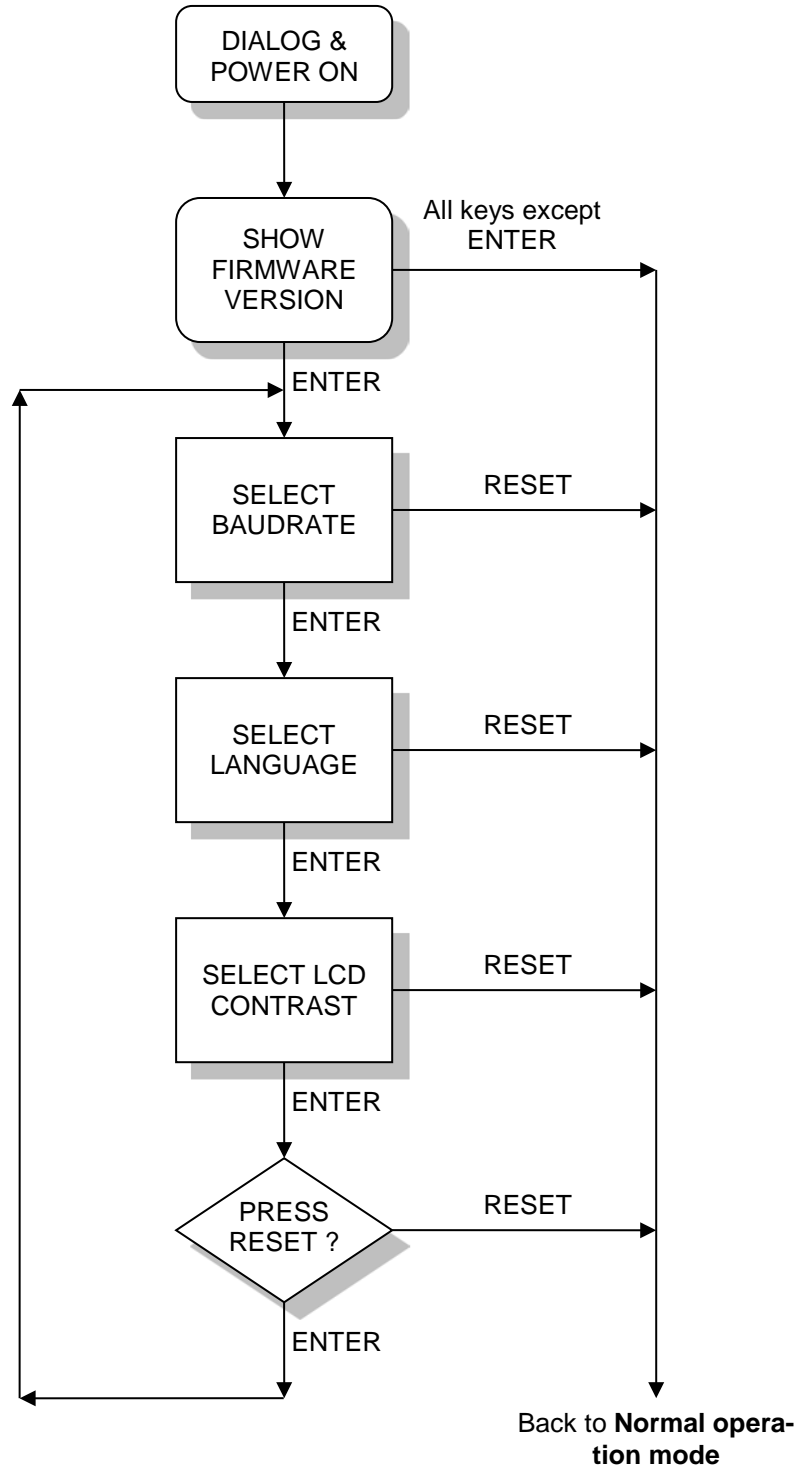
To enter the Manual function selection dialog, press the **END** key when the instrument display shows **"System ready"**.





### 6.7. The Setup dialog

To enter the Setup dialog, hold down the **DIALOG** key while switching on the instrument.



## 6.8. Error messages

Error #	Description
30	There is no glass chamber or the chamber is not in the correct position.
31	The lever is in the up position.
32	The drying compartment door is open.
33	The position of the humidity module valve was not detectable.
34	The changing of positions of the humidity module valve was lasting too long, i.e. valve is locked or dirty.
35	There is no solvent (mobile phase) in the chamber.
36	There is no TLC plate in the ADC2.
37	Humidity / temperature sensor damaged, bad connection or failed.
38	Migration front is moving backwards. There is too little solvent available or there are temperature problems, e.g. due to sun light.
39	Tolerances of TLC plate or TT-Chamber too big.
40	The TT-Chamber has been moved during development.
41	The drying compartment lid has been opened during development.
42	The lever has been moved to the up position during development.
45	The humidity module is disconnected or there is no module present.
47	The edge of the plate holder could not be detected while lowering the plate into the TT-Chamber.
59	Interruption of chromatography by mains power failure
100	Spindle drive (drying comp.): ref-switch (LSX) active
101	Lift drive (plate lift): ref-switch (LSX) active
104	Spindle drive (drying comp.): ref-switch was expected
105	Lift drive (plate lift): ref-switch was expected:
120	Internal device voltage 24V too low

## 7. Maintenance and trouble shooting

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Regular check/maintenance of the instrument is strongly recommended.

Make sure that the instrument is tested for proper functionality after maintenance or trouble shooting with an appropriate procedure.

### 7.1. Cleaning

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Cleaning the instrument on a regular basis in an appropriate way assures problem-free functionality over a long period.

A good way to perform a cleaning would be to follow the decontamination instruction outlined below.

### 7.2. Decontamination

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Before transportation or a longer term of not using your system, decontaminate it in an appropriate manner. The decontamination procedure below reflects the minimal requirements, so keep in mind that the procedure for your instrument has to be adapted according to the substances used.

Decontamination procedure:

- Fill the inlet reservoirs for developing (mobile phase) and pre-conditioning solvents with 25ml of a solvent suitable to dissolve possible impurities
- (most likely Ethanol or Methanol)
- Mount a glass chamber
- Press "end"
- Chose "Rinsing"
- Press "Enter"
- Wait until the end of the process (30sec)
- Repeat the process with the same solvent 3 times
- Repeat the processes 2 times with a cleaning solvent (most likely Ethanol or Methanol)
- For further use: repeat the process with the mobile phase to be used 3 times
- Clean the glass tank respectively

### 7.3. General maintenance

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The instrument may be serviced only by authorized technicians familiar with its technical/functional properties. In general,

CAMAG strongly recommends performing a preventive maintenance depending on the usage, but latest after 12 Month. The chapter Maintenance Data Sheet informs about the parts and their frequency to be changed.

## 7.4. The Setup dialog

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By means of the Setup dialog you can select the communication baud rate between the instrument and the PC, the language and LCD contrast.

### To enter the Setup dialog:

- ✓ Hold down the **DIALOG** key and press the **RESET** key.
- ✓ Release the **RESET** key, then release the **DIALOG** key.

The display will now show **ADC2 Vx.xx.xx Setup Mode**, where x.xx.xx is the actual firmware version of the ADC2.

- ✓ Press the **ENTER** key to start the Setup dialog.

### 7.2.1. Set baud rate

---

Start the Setup dialog according to the procedure above. The display now shows the current baud rate (default is 57'600).

- ✓ Press the arrow keys **▲** or **▼** key to change the setting.
- ✓ Press the **ENTER** key to accept the current selection (and move onto the language selection).
- ✓ Press the **RESET** key to quit the user dialog.

### 7.2.2. Set display language

---

Start the setup dialog according to the procedure above. The display now shows the current baud rate.

- ✓ Press the **ENTER** key to move on to the language selection display.

The current language setting is displayed.

- ✓ Press the ▲ or ▼ key to change the setting.
- ✓ Press the **ENTER** key to accept the current selection (and move on to the LCD contrast selection).
- ✓ Press the **RESET** key to quit the setup dialog.

### 7.2.3. Set the LCD contrast

---

Start the user dialog according to the procedure above. The display now shows the current baud rate.

- ✓ Press the **ENTER** key twice to move on to the LCD contrast selection display.

The current contrast setting is displayed.

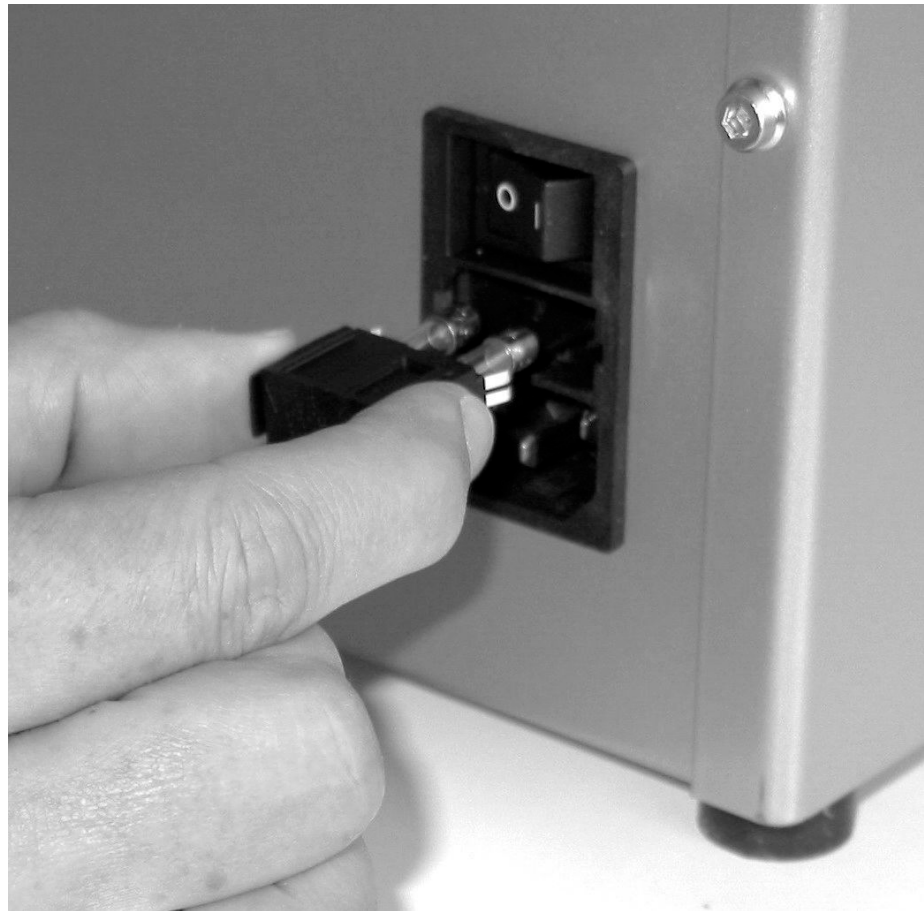
- ✓ Press the ▲ or ▼ key to change the setting.
- ✓ Press the **ENTER** key to accept the current selection.
- ✓ Press the **RESET** key to restart the instrument.

## 7.5. Replacing fuses

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### Procedure:

- ✓ First disconnect the power cord from the mains connection of the instrument.
- ✓ Unlock the fuse holder over the main socket with help of a small screwdriver. Press it on both sides of the holder into the recess. Now you can pull out the fuse holder.
- ✓ Replace the fuse(s). The label 250 V 1.0 AT (slow blow) is valid over the full voltage range.
- ✓ Push back the fuse box.
- ✓ Reconnect the power cord.



*Fig. 12: Mains connection, switch and fuse holder*



## 7.6. Maintenance Data Sheet

<b>CAMAG Maintenance data sheet</b>	
ADC 2	
April 2015/UB	

Purpose	The maintenance data sheet informs about maintenance interval of the respective instrument as well as the proposal for IQ/OQ interval if applicable. In addition, it identifies consumable parts with the respective replacement cycle.
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<b>Maintenance interval</b>	
Maintenance	12 Months
IQ/OQ	12 Months

<b>Consumable parts</b>		
Part No.	Description	Replacement cycle
237.0013	Temperature and humidity sensor	36 Month
	Salt/Solution in humidity control tank	12 Month
022.8370	Saturation pad (100pcs)	Upon need
022.8375	Filter for humidity control tank (10 pcs)	12 Month

## 8. Technical data

Main power connections	100 – 240 V; 50 / 60 Hz; 20 W
Drying module drive	Stepper motor 1600 steps/rev., 160 steps = 0.1mm Positioning with acceleration ramp
Lift drive	Stepper motor 1600 steps/rev, 5 steps = 0.1mm Positioning with acceleration ramp
In- output connection	RS232
Dimensions	Width 330mm, depth 330mm, height 520mm
Weight	18.5 kg

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## Appendix A      Option Humidity Control (022.8360)

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The chromatographic separation on silica gel can be influenced by the activity of the stationary phase and is thus depending on the relative humidity (rH) in the laboratory. The rH is varying significantly for different climatic regions or different seasons which results in varying chromatographic results.

With the option "Humidity Control" the activity of the stationary phase is adjusted with air of a defined relative humidity. This facilitates the possibility to standardize all developments either to a fixed relative humidity or to select a specific humidity for a specific task.

With the ADC 2 and the option "Humidity Control" favorable chromatogram comparisons can be ensured at all times and all places. As a result, chromatography performed in humid summers or coastal climates can be compared to those performed in dry winters or in highland climates.

### 1.      General / saturated salt solution

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The desired humidity is adjusted in the ADC2 by means of a saturated salt solution (not included in shipment). Alternatively it is possible to achieve very dry atmospheric conditions if a molecular sieve (not included in shipment) is used instead.

For nearly every relative humidity a corresponding salt solution can be found, which produces the desired climatic condition (relative humidity). Good experimental results have been achieved with salts generating moderate humidity (40 – 50%) and with a molecular sieve generating high plate activities (below 20%). Relative humidity above 70% is not recommended because of problems with the binder.

Extreme humidity or humidity which differs a lot from the ambient air of the lab takes longer to equilibrate in the ADC2.

As a guideline for possible salt solutions the following table can be used as a start off.

### Relative humidity above saturated salt solutions:

Salt	relative humidity [%] at a temperature of ...°C							Solubility at 20°C <sub>2)</sub> [g / 100 g H <sub>2</sub> O]
	10	15	20	25	30	35	40	
Magnesium chloride	33	33	33	33	33	33	33	54,4
Potassium thiocyanate <sup>1)</sup>			47					222,5
Sodium chloride	76	75	75	75	75	75	75	36,0

Table 1: If not stated differently the values are from: CRC Handbook of Chemistry and Physics 1995 by CRC Press.

1) Values from Römpp Lexicon Chemistry (9. Edition).

2) The solubility values are from: Küster, Friedrich W.: Rechentafeln für die chemische Analytik. Berlin, New York: de Gruyter, 103. bearb. Aufl., 1985.

### Remarks:

It should be added that heating air of 75% relative humidity from 20 to 21°C reduces the relative humidity by 5% down to 70%. A constant temperature is thus necessary for reaching optimal humidity constancy. The salt solution should in any case have the same temperature as the ADC2, which, especially with recently manufactured salt solutions is generally not immediately the case.

## 2. Installation

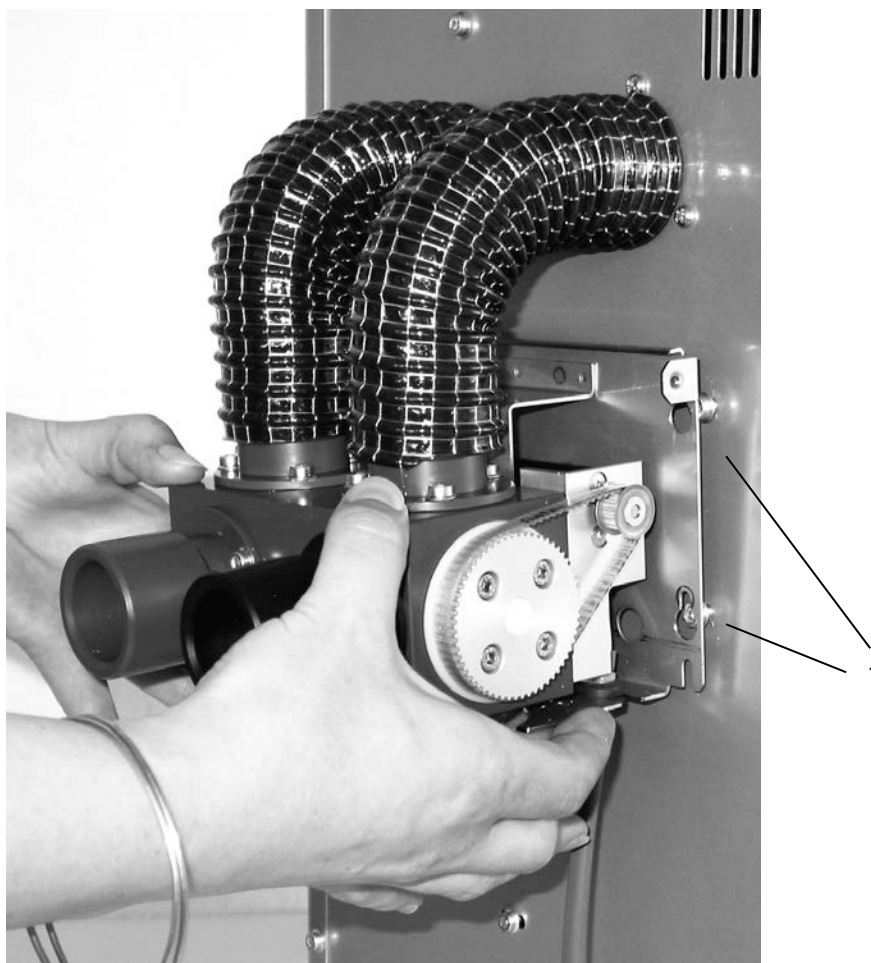


Fig.13: Ventilation valve module: 1= fixation screws.

- ✓ For adapting the Humidity Control device, four screws (1) are provided to fix it on the back of the ADC2; they are used as fixation of the ventilation valve module.
- ✓ The ventilation valve module is mounted on a holding plate that has to be placed in the position over the four screws and tightened. Next, the two hoses already fixed on the ventilation valve module must be connected to the sockets above (fig. 13).
- ✓ After this the top cover can be attached and fixed with one screw at each side of the module (see fig. 15.1).
- ✓ Connect the cable to the appropriate connector (fig. 3).

When inserting the components into the gas wash bottle, make sure the funnel is sitting on the undissolved salt layer! If properly fitted, the lid (1) touches the upper edge of the gas wash bottle (see fig. 14).

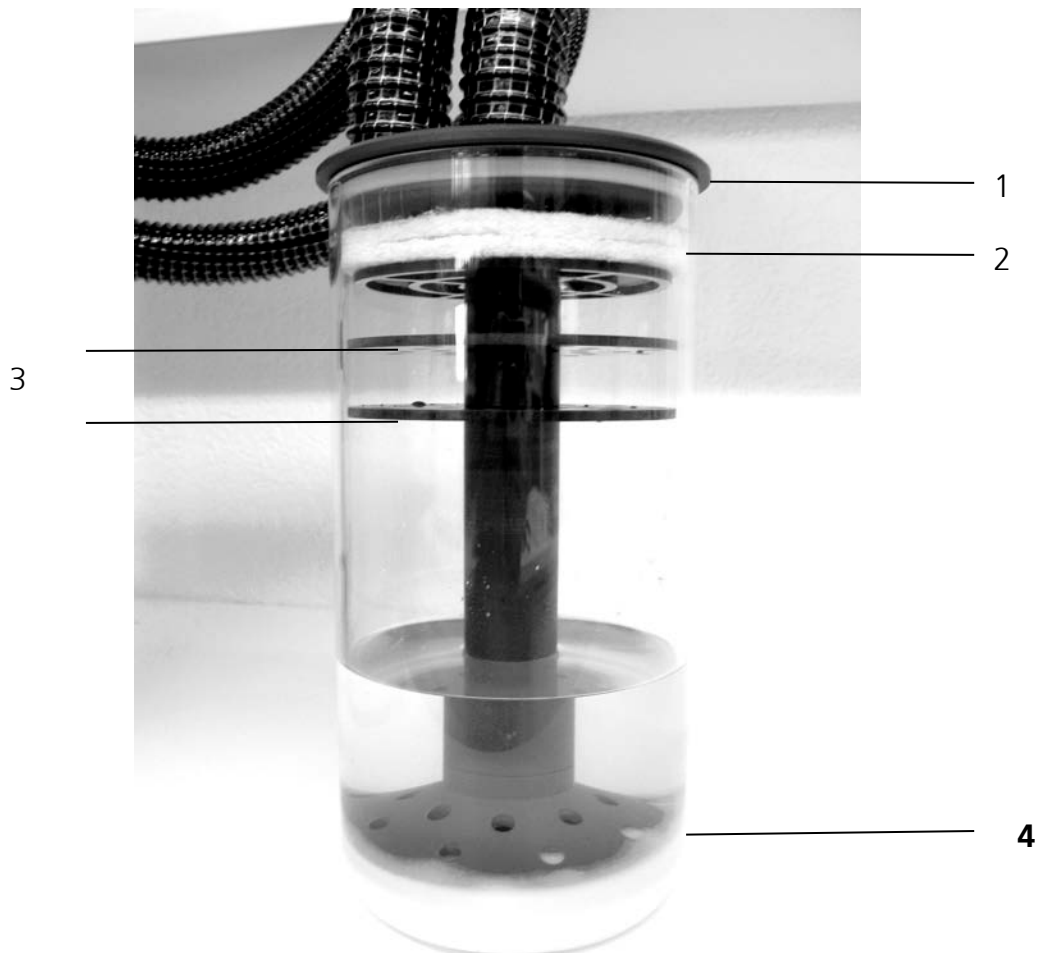


Fig. 14: 1 = top cap, 2 = filters, 3 = splash guard plates, 4 = **distribution funnel with undissolved salt underneath**

The connection of the gas wash bottle to the ventilation valve module is made with two pre-assembled hoses.

- ✓ Connect the center hose (black socket) to the "black" horizontal socket of ventilation valve and the outside hose (grey socket) to the "grey" socket of the ventilation valve!

**WARNING:** Make sure to connect these hoses correctly, because you would fill the ADC2 with salt solution in case of a wrong connection! (See fig. 14)



Fig. 15.1

- ✓ The two sockets directed down on the ventilation valve mount are for exhaust and air supply. The exhaust tube has to be slipped onto the "black" socket, fig. 15.2.

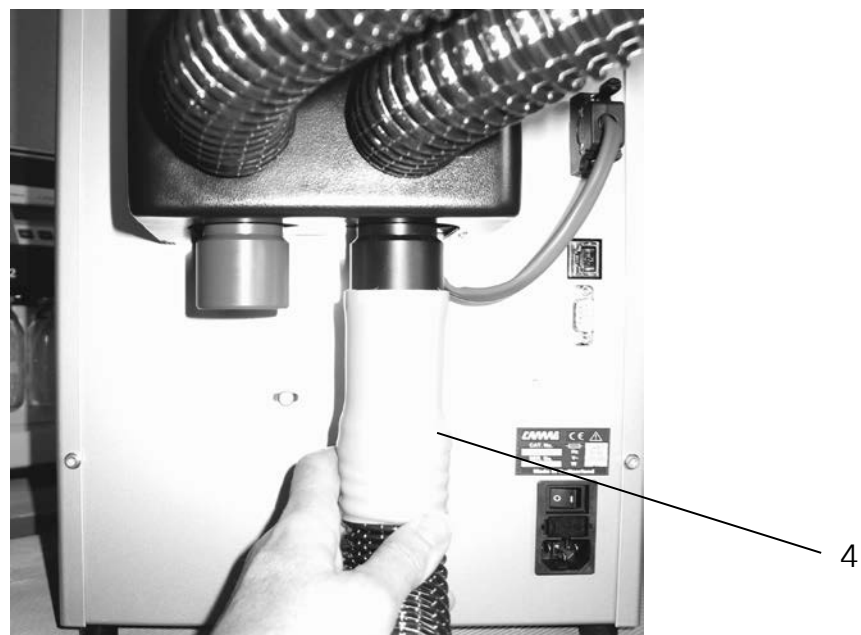


Fig. 15.2: Humidity module: 1 = center hose, 2 = top cover, 3 = outside hose, 4 = exhaust hose



**Warning! Never insert your fingers into the sockets!**

### 3. Humidity control preparations

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Before performing any of the procedures below (except for the molecular sieve), first fill the humidity control tank (large cylindrical glass tank) with 800 mL of water and mark the outside of the bottle at the water line for future reference. Pour out the water so that the bottle is empty dry.

#### 1. Magnesium chloride Hexahydrate (rel humidity at 20°C =33%)

Add 900g MgCl<sub>2</sub> hexahydrate into the humidity control tank. Add 200 mL distilled water and heat it up to 40-50 C° under stirring. Add more distilled water in order to reach the 800 mL line that was marked earlier. Cool down after 10 minutes to room temperature. The final solution should contain about 1 cm of undissolved salt on the bottom to ensure the saturation of the salt solution.

#### 2. Potassium thiocyanate (rel humidity at 20°C =47%)

Add 1000g KSCN into the humidity control tank. Add 200 mL distilled water and heat it up to 40-50 C° under stirring. Add more distilled water in order to reach the 800 mL line that was marked earlier. Cool down after 10 minutes to room temperature. The final solution should contain about 1 cm of undissolved salt on the bottom to ensure the saturation of the salt solution.

#### 3. Sodium chloride (rel humidity at 20°C =75%)

Add 360g NaCl into the humidity control tank. Add 500 mL distilled water and heat it up to 40-50 C° under stirring. Add more distilled water in order to reach the 800 mL line that was marked earlier. Cool down after 10 minutes to room temperature. The final solution should contain about 1 cm of undissolved salt on the bottom to ensure the saturation of the salt solution.

### 4. Preparation of molecular sieve

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Open the gas wash bottle. Dismantle all filters and splash guard plates and the distribution funnel. Add about 100g of the total of 500 g molecular sieve\* into the gas wash bottle. Then mount the distribution funnel, add the rest (about 400g) of molecular sieve and mount finally the splash guard plates and the filters. Close the gas wash bottle with the top cap.

\*Molecular sieve dehydrate Fluka or Sigma with indicator for drying gases, beads diameter 2-5 mm

## 5. Humidity values in practice

CAMAG has performed several tests using potassium acetate, potassium thiocyanate and sodium chloride. **Our experiments have shown that an average humidity of approx. 47% can be achieved successfully with potassium thiocyanate.** The humidity values measured by the ADC2 were generally slightly below the theoretical values: for 47% rH, 44 - 47% rH and for 75% rH, 69 - 73% rH was measured. The reason why the theoretical humidity cannot be fully reached is due to the adiabatic process and the energy balance during aggregate state changes. A good example is shown in the graphics below:

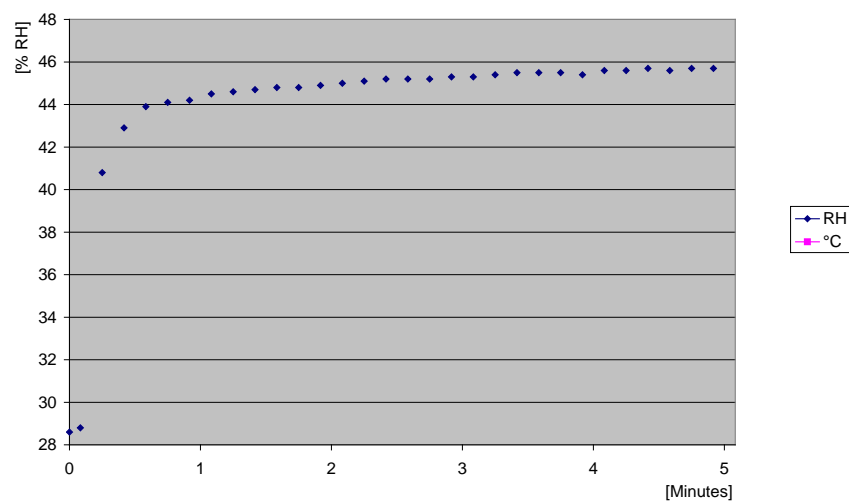


Fig.16: Measurement of the relative humidity in the ADC2 at start of the humidity control. Used salt: Potassium thiocyanate, relative humidity in the lab approx. 29%.

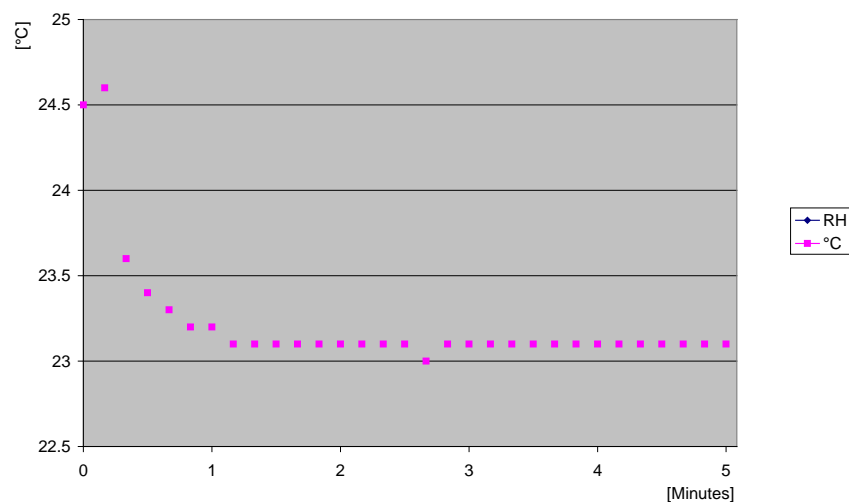


Fig.17: Measurement of the temperature in the ADC2 at start of the humidity control. Used salt: Potassium thiocyanate, temperature in the lab: approx. 23°C. The higher temperatures in the instrument at start are caused by warm up / non equilibrium effects.



## 6. Additional measurement values

Several approaches have been made to quantitatively measure the effective humidity on a HPTLC plate. It has been proven that by means of this principle for humidity control, the activity of the plate can be controlled reproducibly. The figure below shows 4 different measurements and related transfer times from dry to humid and vice versa. The HPTLC plates have been weighted with a precision balance.

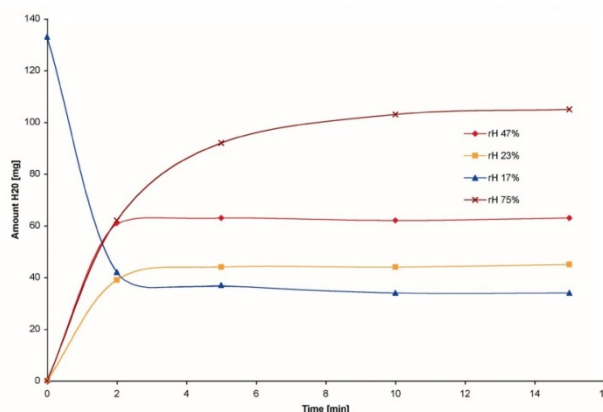


Fig.18: Measurement of the humidity of the HPTLC plate by weighting. It has been shown, that within 2 – 5 minutes a stable equilibrium is reached – for the humidifying as well as the drying process (75% rH:10 minutes). Used plate material: Merck HPTLC plates silica gel 60 F 254.

Fig. 19 shows the values for water absorption for different silica gels from Merck (courtesy Merck). Humidity larger than 70% is producing irreversible reactions in the binder and should be avoided.

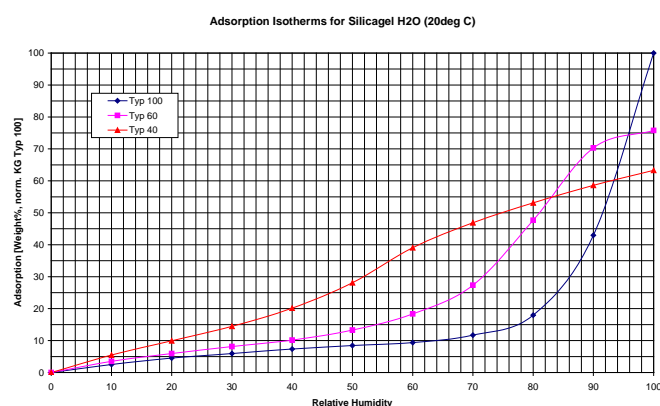


Fig.19: Humidity adsorption of different silica gel.

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## EC – Declaration of Conformity

We, CAMAG Chemie-Erzeugnisse und Adsorptionstechnik AG  
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4132 Muttenz  
Switzerland

declare under our sole responsibility that the product

### **CAMAG® ADC 2**

Product name

**022.8350**

Article number(s)

to which this declaration relates is in conformity with the following provisions of directive(s):

- 2006/95/EC
- 2004/108/EC

Following standard(s) or other normative document(s):

- EN61010-1: 2010
- EN61326-1: 2013

Year of the CE characteristic assignment: 2005

Muttenz, 26 March 2015



Walter Rahm, Head of Quality Management

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