

**Installation Instructions** 

### Sartorius WDS 400

Coulometer for Detection of Water Content

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### **Intended Use**

The WDS 400 Water Detection System is a coulometric analyzer designed for detection of water in solids, using the combined techniques of thermal analysis and coulometry. The detection limit of the analyzer is in the parts-per-million (ppm) range. The sample quantity required for successful detection depends on the expected water content in the material, and may be anywhere from 2 mg to 2000 mg.

The WDS 400 analyzer offers the following special features:

- Drying technique similar to that of a drying oven. Unlike Karl Fischer titration, this technique uses no toxic chemicals (which are subject to strict disposal regulations). This precludes problems with the acquisition and disposal of special tracing fluids, as well as any need for wet-chemical fusion of the sample material.
- High sensitivity of measurement (detection of water content from 15% down to the ppm range).
- Fast and even heating of the sample from room temperature to a maximum of 400°C in the electrically heated oven.
- Programmable heating curve for separate detection of different water components (surface water, capillary water and water of crystallization) through fractionated expulsion of water from the sample.
- Storage of temperature curves for multiple analysis routines performed on a single sample type (for example, routine analysis for quality control in incoming and final inspection).
- Functions for storing and loading temperature curves ensure flexibility when analyzing different types of samples. Development of optimized temperature curves by analyzing series of a single sample type.
- Storage and loading of measurements for later evaluation.
- Printout of measurements for ISO/GLPcompliant documentation. Sensor current and temperature curve are included in the documentation.

- Compensation of measurement results to take ambient humidity into account ("tare measurement").
- Calibration standard (Na₂WO₄) for calibrating the analyzer included in delivery.
- Optional password protection for analysis and evaluation software.
- Easy-to-clean sample scoop made of nickel.

The WDS 400 analyzer is ideal for use as an inspection, measuring and test instrument in development, quality control, in-process control and incoming inspection.

### Symbols

The following symbols are used in these instructions:

- indicates required steps
- indicates steps required only under certain conditions
- > describes what happens after you have performed a certain step
- indicates an item in a list
- $\underline{\wedge}$  indicates a hazard

### Note:

The program window illustrations in this manual were made using the latest program version at the time of printing. Sartorius reserves the right to make changes to the program without notice.

For technical advice on applications: Phone: +49.(0)551.308.3160 Fax: +49.(0)551.308.3495

### Contents

### Warning and Safety Information

### 3 Intended Use

### 4 Contents

### 4 Warning and Safety Information

### **Getting Started**

- 6 Storage and Shipping Conditions
- 6 Unpacking the Analyzer
- 6 Recycling the Packaging
- 6 Equipment Supplied
- 7 Installation Instructions
- 7 Conditioning the Analyzer
- 7 Setting up the Analyzer
- 8 General View of the Equipment
- 9 Operating Elements and Display
- 10 Connecting the Analyzer to the PC
- 10 Connecting the Analyzer to AC Power
- 10 Connecting the Gas Supply
- 11 Installing the Control and Evaluation Software

### Operating the Analyzer

- 14 Analyzer Functions
- 16 Preparation
- 24 Flushing the System
- 24 Continuous Operation
- 25 Tare Measurement
- 26 Calibration Measurement
- 29 Sample Analysis
- 34 Loading a Measurement File
- 36 Evaluating a Measurement File
- 37 Printing a Measurement File
- 39 Troubleshooting Guide

### Care and Maintenance

- 40 Service
- 40 Repairs
- 40 Cleaning
- 40 Safety Inspection
- 40 Recycling
- 41 Cleaning and Replacing the Teflon Filter
- 41 Cleaning and Replacing the Moisture Sensor

### Overview

- 44 Specifications
- 44 CE Conformity
- 45 Index

The WDS 400 analyzer complies with the European Council Directives as well as international regulations and standards for electrical equipment, electromagnetic compatibility, and the stipulated safety requirements. Improper use or handling, however, can result in damage and/or injury.

To prevent damage to the equipment, read these operating instructions thoroughly before using your analyzer. Keep these instructions in a safe place.

Please observe the following to ensure safe and reliable operation of your analyzer:

▲ Use the analyzer only for determining the water content of samples. Any improper use of the analyzer can endanger persons and may result in damage to the analyzer or other material assets.

▲ If you use electrical equipment in installations and under ambient conditions requiring higher safety standards, you must comply with the provisions as specified in the applicable regulations for installation in your country.

▲ Do not use this analyzer in a hazardous area/location; operate it only under the ambient conditions specified in these instructions.

- Personnel operating the analyzer must have certified training in handling chemicals. Furthermore, the operator must have thorough knowledge of the properties of the sample material being analyzed.
- ▲ The analyzer must be connected to a power supply (mains) that is equipped with a protective grounding conductor. A power supply of 230 V, 50 Hz is required (see "Connecting the Weighing Platform to AC Power" in the chapter entitled "Getting Started").
- The analyzer comes with a power supply that has a grounding conductor.
- Use only extension cords that meet the applicable standards and have a protective grounding conductor.
- Disconnecting the ground conductor is prohibited.

- To avoid danger of overload, do not connect the analyzer to a multiple socket.
- Connect only Sartorius accessories and options, as these are optimally designed for use with your analyzer.
- Note on installation: The operator shall be responsible for any modifications to Sartorius equipment and must check and, if necessary, correct these modifications. On request, Sartorius will provide information on the minimum operating specifications (in accordance with the Standards listed on page 44 for defined immunity to interference).
- Protect the analyzer from contact with liquid
- If there is visible damage to the equipment or power cord, turn off the power and disconnect the equipment from AC power immediately. Lock the equipment in a secure place to ensure that it cannot be used for the time being.
- ▲ Do not insert anything into the ventilation slots or other openings in the housing.
- ▲ If any liquid enters the equipment housing, disconnect the analyzer from power immediately and notify the customer service center.
- The paint and the powder coating on the housing are chemical-resistant to a limited extent only. Avoid contact with aggressive chemicals such as acids or solvents.
- ▲ Clean your analyzer according to the cleaning instructions only (see "Care and Maintenance").
- $\triangle$  Do not open the equipment housing.
- This device uses nitrogen or dried compressed air as carrier gas.
- ▲ Nitrogen can supplant the oxygen in the air. This is especially hazardous when working with nitrogen in closed areas. If the oxygen concentration in a given area drops below 15%, this will reduce the ability of persons in that area to work. Oxygen concentrations below 11% will quickly result in loss of consciousness.
- ▲ If a gas cylinder is used for the carrier gas supply, make sure the cylinder cannot tilt or fall (see "Getting Started").

In case you have any problems with your analyzer:

 contact your local Sartorius office, dealer or service center.



Warning: Severe Burns!

- This analyzer is approved for use in continuous operation.
- To prevent build-up of heat, install the analyzer in a well-ventilated location with at least 40 cm (approx. 16 in.) space on all sides. The surface on which the analyzer rests must be heat resistant up to at least 60°C (140°F) (see "Installation Instructions" in the chapter entitled "Getting Started").
- Do not place flammable materials on, \_ under or near the analyzer.
- The tube oven inside the analyzer can reach temperatures of 400°C (752°F) and higher. For this reason, use extreme care when inserting and removing samples. Use only the forceps supplied to handle the sample scoop. The sample temperature is displayed by the operating program.

Hazards for persons or equipment posed by using specific samples:





- Explosion
- Do not heat samples beyond the melting point. Evaporation of uncontrollable chemical compounds or denaturation of the sample due to excessively high temperatures must be avoided at all costs. Otherwise, there is a risk of contamination of the gas flow system and damage to the electrolytic cell. Overheated sample material can adhere to the sample scoop permanently, in which case the scoop must be replaced.

It is the responsibility of the user to have or acquire specific knowledge of any alterations that can occur in the chemical composition of the sample material, and of which chemical substances can evaporate or be emitted from the sample material, as a result of exposure to heat.





Poisoning

**Caustic burns** 

- ∧ Gases which are toxic and hazardous to the health can be released when working with this analyzer. For this reason, the exhaust tubing must be connected to a discharge conduit, or must conduct the exhaust into the open air.
- ▲ Never place the following types of substance inside the analyzer:
- Flammable or explosive substances
- Substances that contain solvents
- Substances that release flammable or explosive gases or vapors during the heating process

When in doubt, perform a risk analysis. The user shall be liable and responsible for any damage that arises in connection with this analyzer.

The emergence of any of the gases listed below will restrict the functioning of the WDS 400. In this case, the phosphorous pentoxide electrolytic cell may have to be regenerated or replaced.

Gases that react with phosphorous pentoxide:

alkaline gases, such as ammoniacal gas chlorous gases

Gases that are dehydrated by phosphorous pentoxide:

alcohols, for example

Gases that can form solid or liquid polymerization products:

- for example, unsaturated carbon hydrides such as acetylenes, alkadienes, and alkenes (higher than propylene)
- Gases that attack platinum: hydrogen fluoride

### **Getting Started**

The WDS 400 Water Detection System consists of the analyzer, a control and evaluation program, and accessories.

The analyzer contains the heating and operating units, the phosphorous pentoxide electrolytic cell and the serial data interface for connection to the PC on which the control and evaluation program is installed. The accessories supplied with the analyzer include, among other things, the power cord, cable for PC connection, connectors for the inlet and outlet of the carrier gas (nitrogen or compressed air) and a case containing additional equipment for performing analysis (sample scoop, forceps, etc.).

### **Storage and Shipping Conditions**

Allowable storage temperature: -10°C to +50°C (+14°F to +122°F).

Before putting the equipment into operation, allow it to acclimate to the ambient temperature inside the dust cover to prevent condensation within the equipment.

Do not expose the analyzer to extreme temperatures, moisture, shocks, blows or vibration.

### Unpacking the Analyzer

- After unpacking the equipment, please check it immediately for any visible damage.
- If any sign of damage is visible, proceed as directed under "Safety Inspection" in the chapter entitled "Care and Maintenance."

It is a good idea to save the box and all parts of the packaging until you have successfully installed your analyzer. Only the original packaging provides the best protection for shipment.

Before packing your analyzer, unplug all connected cables to prevent damage. Use the plug supplied to close the gas inlet.

### Note:

Always keep the door to the sample chamber closed; to insert a sample, open it only briefly. This prevents moisture from entering the system.

### **Recycling the Packaging**

Sartorius products are packaged to ensure safe shipment using environmentally friendly materials. After successful installation of the analyzer, you should return this packaging for recycling because it is a valuable source of secondary raw material.

### **Equipment Supplied**

The equipment supplied includes the components listed below:

- WDS 400 coulometric analyzer
- Power cord
- Interface cable for connection to the PC (9-pin D-Sub male connector to 9-contact D-Sub female connector)
- CD with control and evaluation program
- Exhaust tubing for the WDS 400, plastic, 3 m
- Adapter for gas cylinder, 1/8 inch
- Gas connection tubing, extendable, approx. 2 m
- Regeneration kit
- Accessory Set containing:
- 5 sample scoops
- 1 forceps
- 2 Teflon filters
   1 sample spatula
- 1 fine-bristle brush
- 1 calibration standard, 20 g in a 50 ml bottle Substance:  $Na_2WO_4$  (sodium tungstate)

The following additional equipment, not included in delivery, is also required for operation of the WDS 400 analyzer:

- Weighing instrument for precise determination of sample weight.

The following table shows the optimum sample size and required weighing instrument resolution (accuracy) based on the expected water content of sample substances:

Water content	Optimum sample qty.	Accuracy of balance
%	mg	mg
10	2	0.01
1	20	0.1
0.1	200	1
0.01	2000	10

In general, the relative error of the sample quantity should be lower than 0.2 %. For more information on this topic, please refer to "Sample Analysis" the chapter entitled "Operating the Analyzer."

 Computer system for control of the analyzer and evaluation of results: Windows<sup>®</sup> NT/2000/XP with at least one RS-232 interface available.

Windows® NT, Windows® 2000 and Windows® XP are registered trademarks of Microsoft Inc.

- Carrier gas:

Nitrogen or argon of quality class 4.0 / 5.0, with adjustable pressure reducer to at least 1 bar and fine pressure gauge, or dried air with a dew point of < -70°C. If connected to an in-house supply, add a dust filter ( $\leq$  5 µm).

<u>∧ Note:</u>

When working with compressed nitrogen in gas cylinders, make sure to observe the applicable regulations and safety instructions (see the chapter entitled "Warning and Safety Information").

 Recommended tools for putting the analyzer into operation: Open-ended wrenches: 30/32, 12/13, 10/11 Ring wrench: 20/22

### Installation Instructions

The analyzer is designed to provide reliable results under normal ambient conditions in the laboratory and in industry. The following minimum requirements on the place of installation must be met:

- Choose a place of installation with sufficient space to position the weighing instrument for weighing samples next to the analyzer. Thus the place chosen for installation of the analyzer must also meet the conditions required for installation of the weighing instrument (for details, see the installation and operating instructions for the particular weighing instrument).
- Chose a place of installation with sufficient space for positioning the PC, required for control and evaluation of analysis, near the analyzer.
- The place of installation must permit the supply of carrier gas (nitrogen, argon or compressed air) required for operation.
- The analyzer must be installed at a location that permits safe discharge of the carrier gas from the analyzer outlet (for example, near a discharge conduit).
- Set up the analyzer on a stable, even surface that is not exposed to excessive vibrations.
- The analyzer produces heat during operation. For this reason, the surface on which the analyzer rests must be heat-resistant up to 60°C (140°F). Make sure to choose a place where excessive heat cannot build up (allow at least 40 cm space on each side). Leave sufficient space between the analyzer and materials that are affected by heat.
- Avoid placing the analyzer in close proximity to a heater or otherwise exposing it to heat or direct sunlight.
- Protect the analyzer from direct exposure drafts that come from open windows or doors.
- Make sure the place of installation is dry and free of dust.
- Protect the analyzer from aggressive chemical vapors.
- ▲ If a gas cylinder is used for the carrier gas supply, make sure the cylinder cannot tilt or fall.

### **Conditioning the Analyzer**

Moisture in the air can condense on the surface of a cold analyzer whenever it is brought to a substantially warmer place. If you transfer the analyzer to a warmer area, make sure to condition it for about 2 hours at room temperature, leaving it unplugged from AC power. Afterwards, if you keep the analyzer connected to AC power, the constant positive difference in temperature between the inside of the analyzer and the outside will practically rule out the effects of moisture condensation.

### Setting up the Analyzer

- Unpack the analyzer and set it up at the place of installation.
- Connect the analyzer to the carrier gas supply (gas cylinder or in-house supply) and provide for the safe discharge of the carrier gas that exits the analyzer (for example, by connecting the exhaust tubing to a discharge conduit).
- Connect the serial interface of the analyzer to the PC.
- Connect the analyzer to power.

Further details on these topics are provided in the corresponding sections in the following pages.

### Note:

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to Part 15 of the FCC rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense. Changes or modifications not expressly approved by Sartorius AG could void the user's authority to operate the equipment.

### **General View of the Equipment**



### Pos. Designation

- Sample chamber (oven) 1
- Housing fan for cooling electronic elements 2
- RS-232 interface port for connecting the PC 3
- (9-contact D-Sub female connector) 4
- Gas outlet for connection to the exhaust tubing 5 Gas inlet for connection to the extendable connection
- tubing (sealed with cap in this illustration) 6 Power switch
- Power socket/inlet connector with built-in fuse 7
- 8 Left-hand side of analyzer:
- Fine adjustment valve for gas flow when oven door is open 9 Left-hand side of analyzer:
- Fine adjustment valve for gas flow during analysis

### Designation Pos.

- 10 Electrolytic cell with  $P_2O_5$  moisture sensor
- 11 Oven fan
- Teflon filter 12
- 13 Gas flow indicator
- Sensor current indicator (10-stage LED band) 14
- 15 Power supply indicator



### Accessory Set

- Pos. Designation
- 16 Sample scoop
- 17 Forceps
- 18 Brush 19 Teflon f
- 19 Teflon filters (2 pcs)20 Sample scoops (5 pcs)
- 21 Calibration standard: sodium tungstate, 20 g

### **Operating Elements and Display** Front view:

- Sample chamber (1).
   The sample chamber (oven) is designed for insertion of the sample scoop.
   The door of the sample chamber has a locking device for gas-tight closure.
   Always keep the door closed to prevent moisture from entering the sample chamber, and open it only briefly when inserting a sample.
- Power supply indicator (15).
   The green LED is lit when the power supply to the analyzer is active.
- Sensor current indicator (14). The electrical current flowing through the  $P_2O_5$  moisture sensor is indicated (in mA) by the 10-stage LED band. Each diode represents 10 mA.
- Gas flow indicator (13).
   A movable ball indicates the currently active gas flow in ml/min. This provides a reference for adjustment of the gas flow using the fine adjustment valves (8) and (9) on the left-hand side of the analyzer. Multiply the displayed value by 100 to arrive at the actual flow rate.

### Left-hand side:

- Fine adjustment valves for gas flow control (8) and (9).
- Use these valves to set the flow rate of the carrier gas. The valve labeled "load" controls the flow of gas when the oven door is open (counter-flow). This almost entirely prevents moisture from entering the sample chamber. The valve labeled "measure" controls the flow of gas when the oven door is closed (in standby mode and during analysis). The gas flow is switched from

"measure" to "load" automatically when the oven door is opened and closed. The active flow rate is shown on the gas flow indicator (13).

Rear view:

- Power switch (6). The power switch is used to turn the analyzer power supply on and off.
- Power socket (7).
   This socket is for connection of the power cable to the power supply (mains). The fine-wire fuse is located directly above this socket.
- ▲ Operate the analyzer only with a supply of 230 V, 50 Hz.
- Gas inlet (5). The extendable gas connection tubing for the carrier gas supply (included in the Accessory Set supplied) is connected here. The gas inlet is closed with a special cap to keep the inside of the analyzer clean. Keep this cap in a safe place and always use it to seal the gas inlet when no gas supply is connected (for example, for storage and shipping).
   Gas outlet (4).
- Connect the exhaust tubing (included in Accessory Set supplied) to this outlet to conduct the gas out of the analyzer. To avoid the hazard of poisoning, the carrier gas should be conducted either into the open or to a discharge conduit.

- Serial RS-232 interface (3).
   9-contact D-Sub female connector for the cable that connects the PC to the analyzer.
- Electrolytic cell with moisture sensor (10).
  - If the sensitivity of the sensor decreases, the sensor must cleaned, re-coated or replaced. Any of these tasks can be performed by the user (see "Cleaning and Replacing the Moisture Sensor" in the chapter entitled "Care and Maintenance").
- Teflon filter (12).
  - Protects the electrolytic cell from contamination by particles larger than 3 µm. Check the filter regularly and clean it if necessary (see "Cleaning and Replacing the Teflon Filter" in the chapter entitled "Care and Maintenance").

### Connecting the Analyzer to the PC

Use the cable supplied to connect the analyzer to the PC. The corresponding 9-pin D-Sub female connector (3) is located on the back of the analyzer. Connect the cable to an RS-232 interface on the PC; for example, on COM1. If the desired port on the PC has a 25-pin rather than a 9-pin connector, use an adapter (not included in delivery) to connect the cable from the analyzer.

### Connecting Electronic Peripheral Devices

Disconnect the analyzer from AC power before connecting or disconnecting peripheral devices to or from the PC (RS-232 interface).

Connecting the Analyzer to AC Power

- Make sure the power supply specifications match the requirements (230 V, 50 Hz).
- $\underline{\wedge}$  If the specifications do not match, do not put the analyzer into operation.

Use only

- Power cords supplied with the analyzer
- Power cords approved by a certified electrician
- If you need to connect an extension cord, use only a cable that has a protective grounding conductor.
- Powering a device of protection class 1: Plug the power cord into an electrical outlet (mains supply) that is properly installed with a protective grounding conductor (protective earth = PE).

### **Safety Precautions**

If you use an electrical outlet that does not have a protective grounding conductor (protective earth), make sure to have a certified electrician install equivalent protection according to the installation requirements valid in your country. Make sure the protective grounding effect is not neutralized by use of an extension cord that lacks a protective grounding conductor.



### **Connecting the Gas Supply**

- Connect the carrier gas supply (e.g., nitrogen cylinder, quality class 4.0 / 5.0) to the analyzer.
- $\triangle$  If a gas cylinder is used for the carrier gas supply, make sure the cylinder cannot tilt or fall.
- Remove the protective cap from the gas inlet on the back of the analyzer and keep it in a safe place for future use.
- Connect the extendable, flexible stainless steel gas connector tubing to the gas inlet and use an open-ended wrench to tighten the connection.
   To connect a gas cylinder, use
- the 1/8" adapter pipe thread supplied.

### Note:

Do not use fittings or tubing made of plastic; otherwise, moisture could penetrate the gas lines through diffusion.

 Hand-tighten the union nut on the stainless steel tubing, and then tighten it an additional 1/4 turn with an open-ended wrench.

### Note:

- Use the built-in fine pressure gauge and fine adjustment valve to maintain constant pressure in the gas lines.
- For gas cylinders, use the pressure reduction valve (up to 1 bar) in conjunction with a fine pressure gauge.
- If connected to an in-house supply, add a dust filter (< 5 µm).</li>
- Connect one end of the exhaust tubing to the exhaust connection supports on the analyzer, and the other end to a discharge conduit or open air outlet.
- ▲ Always provide for safe discharge of the exhaust gas from the analyzer, as the products of disintegration from certain sample substances may be toxic.



Next >

< Back

Cancel

### Installing the Control and Evaluation Software

- Shut down all active programs make sure no memory-resident programs are running in the background during installation.
- Re-start Windows<sup>®</sup> to make sure none of the operating system components required for installation are being used by other programs.
- Temporarily deactivate any anti-virus software.
- Insert the CD in the CD drive.
- Open the "Start" menu and click on "Run."
- Enter "d:\setup.exe" (for "d:" enter the letter used by the drive in which the installation CD is inserted) on the command line and click on "OK."
- > The installation routine starts.

• Continue installation: click on "Next." To stop the installation, click on "Cancel."

### Note:

The program checks whether it has already been installed. If it has, the user is prompted to choose one of three options: "Modify," to make changes in an existing installation of the program; "Repair," to correct errors in the earlier installation, or "Remove," to de-install the program. Before selecting "Modify" or "Repair," it is advisable to make a backup copy of the program.

 $\bigcirc$  Select the desired option and click on "Next" to continue.

• Continue in accordance with the information and prompts given by the installation program.



. . .

- Enter the target drive and directory for installation.
- $\bigcirc$  To change the path name, click on "Change." This opens a window where you can select the desired folder (see next illustration).

Otherwise, click on "Next" to use the default path suggested by the program.

 $\odot\,$  To install the program on a different drive or in a different directory than the defaults, enter the desired path under "Folder name: ".

Ide Next to install to this folder, or dick Chan Install Sartorius - WD5400 - Water D E:\Programme\Sartorius\WD5400\	ge to install to a	a different folo	der.
Instal Sartorius - WDS400 - Water D E:\Programme\Sartorius\WDS400\	etection System	n to:	C Shange.

• Confirm the selected installation path: click on "Next."

Look in:

CF WD5400

Eolder name:

E: (Programme)(Sartorius)(WDS400)





- To start the installation procedure, click on "Install." To stop installation, click on "Cancel."
- > The installation routine copies the required data into the target directory, installs the program, and then reports the successful installation.

- Confirm the end of installation: click on "Finish."
- Create a shortcut to the "WDS 400" program as follows: Right-click on an empty area of the Desktop and select "New > Shortcut" from the shortcut menu.
- > This opens the "Create Shortcut" window.

- Click on the "Browse" button and select the installation path and file name of the WDS 400 program. Click on "Next" to confirm the selected path.
- Enter a name for the shortcut, such as "WDS 400," and click on "Finish" to confirm.
- > The shortcut to the "WDS 400" program is created. The Desktop icon for the program is state, representing a water molecule.
- Re-start the PC and reactivate the anti-virus software. To start the "WDS 400" program, double-click on the *set* icon.

### **Operating the Analyzer**

### **Analyzer Functions**

The WDS 400 coulometric analyzer combines two field-proven methods, thermal analysis and coulometry, to create a powerful technique for detection of trace amounts of water in solids.

The thermoanalytical procedure involves heating a sample at a defined temperature curve to distinguish between the different forms of water bond. Because water is bound to the sample material through different physical forces, such as van der Waals forces, hydrogen bonds, dipole forces and electrostatic interactions, it takes different forms of thermal energy to release the water from the sample. Thus the controlled application of heat at different temperatures makes it possible to distinguish between surface water, capillary water and water of crystallization.

The electrochemical sensor in the WDS 400 operates on the principle of coulometry, which enables selective and quantitative detection of the water expelled from the sample. Coulometry involves measuring the amount of electricity, or the charge, required to disintegrate (dissociate) a material through electrolysis. Faraday's Law is applied to compute the initial mass of the disintegrated component from the electrical charge (i.e., the product of current intensity and time) produced during disintegration.

With the WDS 400 analyzer, the electrolytic dissolution of the water molecules released during heating is caused by the chemical reaction with phosphorous pentoxide, which takes place in the electrolytic cell (sensor).

The diagram on the left illustrates the operating principle of the electrolytic cell in the WDS 400. A thin coating of phosphorous pentoxide ( $P_2O_s$ ) is applied between two parallel electrodes using a special coating process.

The entire detection technique is illustrated on the next page and described briefly in the following:

The water expelled from the sample by the application of heat is taken up by an inert (chemically inactive) carrier gas that is fed into the oven, and is conducted from the oven to the electrochemical sensor through a system of tubing. In the sensor, the gas carrying the water from the sample flows over a layer of phosphorous pentoxide. Phosphorous pentoxide is extremely hygroscopic, which means it has a high affinity to water. Thus the water is completely absorbed in the phosphorous pentoxide coating.







The chemical reaction with the phosphorous pentoxide causes the water molecules to disintegrate; in other words, they are dissociated into their components parts, hydrogen and oxygen, which are then conducted out of the system by the carrier gas. From each electrolyzed water molecule, two electrons contribute to an electrical charge. Applying Faraday's Law, the electrolytic current is linked directly to the number of water molecules detected and thus to the mass of the water evaporated from the sample.

The WDS 400 analyzer contains all of the components required for this analytical process. The analyzer performs the analytical measurements, calculations and evaluations automatically, and subsequently provides the user with a quantitative and graphic representation of the water content in the sample material. The measuring range of the WDS 400 extends from approx. 15% water content to amounts in the ppm range.

A basic measurement sequence is displayed in the program window shown on the left (for details, see the section entitled "Sample Analysis"). An example of a measurement is shown on the basis of a defined temperature curve (actual heating curve, indicated by the red line). The area below the analysis current (i.e., the timeintegral of the analysis current) indicates the total water content of the sample. If the analysis current curve has multiple peaks, this indicates water bonds of varying strengths. Fractionated drying of the sample can be implemented by selecting certain temperature levels for the different stages of the heating process, to enable separate measurement of the different forms of water.



# WDS 400 start up

≝∠ 🔚 WDS 400 is ready

### Preparation

The following requirements must be met to perform successful analysis with the WDS 400:

- The equipment must be put into operation and connected to the PC in accordance with the instructions.
- The control and evaluation program supplied with the analyzer must be installed.

These procedures are described in the chapter entitled "Getting Started."

### Starting the Control and Evaluation Program

- Start the PC and double-click on the  $\frac{1}{1000}$  icon (H<sub>2</sub>O molecule).
- > The "WDS 400" program starts.
- Enter the password, if one has been assigned, and click on "OK." The first time you start the program, no password is assigned. (If the program was started unintentionally, click on "Cancel" to close it down.)

> The main program window opens, showing the "New Analysis" window.

The menus, buttons, information and results blocks in the main window are described briefly in the following:

The toolbar is located below the menu bar. The "View" menu contains a command for showing and hiding the toolbar. A checkmark in the menu ( $\sqrt{}$ ) indicates the current setting.

Charts mapping the analysis current ("Current/Time") and oven temperature ("Temperature/Time") are shown below the menu bar and toolbar.

The upper half of this window shows the sensor current over time. The area below this curve corresponds to the charge used in the sensor for dissolution of the water, and thus provides a measure for the water content of the sample. When the program is not in use (illustration on the left), this chart is blank.

The lower half of the window shows the temperature in the drying oven over time (target and actual curves). The target curve is defined by the temperature curve file (for example, "Standard.pfl"). When the program is not in use (illustration on the left), no actual temperature is displayed.



Description Number Origin Comment Date Analysis time 0:00 mm:ss Weight 0.000 mg Rel 100 2 Abs. 0.000 µg Current act mΑ Tare act mΑ Calculated act mΑ Temp act °C Switch off current 1.000 mA Min analysis time 60 min

### Open... Strg+0 Save Strg+S Save gs... Strg+P Print... Strg+P Print preview... Printer Properties...

Exit

Below these two windows is a status bar. The "View" menu contains a command for showing and hiding the status bar. A checkmark next to the menu item ( $\checkmark$ ) indicates the current setting. The symbol on the left shows the analyzer status; i.e., indicates whether it is off, in start-up phase, or ready for use. The symbol on the right shows whether the carrier gas is passing through the oven (during analysis) or through the bypass line. In the example shown here, either the analyzer is not connected to the PC, or the analyzer has not been switched on. The message "No connection to WDS 400" is displayed.

The "Sample," "Humidity" (moisture) and "Analysis Data" information blocks are shown to the right of the analysis charts in the main window. These blocks show the target and actual values for the analysis. The "Sample" block contains the initial sample weight, which must be entered before analysis begins. The "Humidity" block shows the analysis results, and the "Analysis Data" block shows data on the current analysis.

The charts and information blocks are described in detail in the section entitled "Sample Analysis."

### Menu Bar and Toolbar Buttons

The menu bar contains four menus: "File," Edit," "View" and "?" (Help).

The "File" menu contains commands for the following functions:

- Open a file for a new analysis ("New")
- Open an existing file ("Open")
- Store a file ("Save")
- Store a file under a different name ("Save as...")
- Print a file ("Print...")
- View the file on screen as it will appear in printed form ("Print preview...")
- Configure printer settings ("Printer Properties...")
- Close the program ("Exit")

<u>E</u> dit <u>V</u> iew <u>?</u>	
<u>A</u> nalysis	A
Calibration	
<u>T</u> are	
Eractioning	
<u>S</u> etup	
S <u>w</u> itch gas flow	F4
✓ <u>U</u> se tare offset values	
<b>F</b>	го

<u>⊻</u>iew <u></u>?

✓ <u>T</u>oolbar
✓ <u>S</u>tatusbar





The "Edit" menu contains commands for the following functions:

- Begin performing moisture content detection on a sample ("Analysis...")
- Calibrate the analyzer ("Calibration...")
- Begin what is referred to as a "Tare measurement," to determine any effects of moisture not contained in the sample ("Tare...")
- Perform fractionated evaluation of an existing analysis ("Fractioning")
- Configure program settings; for example, minimum analysis time, minimum analysis current for shutoff, serial interface settings for the connection between analyzer and PC, and passwords ("Setup...")
- Switch the gas flow from the "measure" mode to "bypass" and vice versa ("Switch gas flow")
- Define whether compensation values (results of tare measurement) are considered in analysis ("Use tare offset values"). When this function is activated (" $\sqrt{$ "}), the "background" moisture (i.e., residual moisture detected with no sample in the oven) is subtracted from each point of measurement. To activate or deactivate this function (i.e., toggle it on and off), click on it in the menu.
- Confirm error messages to continue working with the program ("Error confirmation")

The "View" menu contains commands for the following functions:

- Show or hide the toolbar ("Toolbar")
- Show or hide the status bar ("Status bar")

To show or hide the toolbar or status bar, click on the corresponding menu item. A checkmark (" $\checkmark$ ") in front of the menu item indicates that the bar in question is shown.

The "?" (Help) menu contains commands for the following functions:

- Open the Help program ("Help")
- View program information ("About WDS 400...")

Which functions can be activated from the toolbar depends on whether the analyzer is ready to start an analysis (as shown in the illustration on the left) or a measurement is in progress. The "View" menu contains a command for showing and hiding the toolbar. It is advisable, however, to leave the toolbar displayed at all times, as certain functions can be activated only using toolbar buttons. The buttons shown during analysis are described in the section entitled "Sample Analysis."

When the program is started, the toolbar contains the following buttons:

- Open a file for a new analysis (corresponds to "New" in the "File" menu)
- Open an existing file (corresponds to "Open" in the "File" menu)
- Store a file (corresponds to "Save" in the "File" menu)
- Begin an analysis (corresponds to "Analysis..." in the "Edit" menu)
- Change the temperature curve. This function cannot be activated from any menu.
- Define whether compensation values are considered in analysis (toggle function; corresponds to "Use tare offset values" in the "Edit" menu). The graphic display of the button shows whether the function is active or not.
- Open the Setup program (corresponds to "Setup..." in the "Edit" menu)
- Open the Help program (corresponds to "Help" in the Help ("?") menu)
  - View information about the program (corresponds to "About WDS 400..." in the Help ("?") menu)

The buttons shown in the upper right-hand corner of the

"Current/Time" graph let the user change the display of the time axis. Click on "+" or "-" to expand or collapse the time axis. Click on "<" or ">" to move the axis to the left or right by five minutes.



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The button shown in the upper right-hand corner of the "Temperature/Time" graph permits configuration of the maximum oven temperature that can be set.

To change the maximum temperature, click on 📉.

> The "Temperature" window opens.

Select the maximum temperature that can be set: 250°C or 400°C. Factory setting: 250°C.

○ To change the setting, click on the ▼ button and select the desired temperature. Confirm your selection by clicking on the √ (checkmark) button.

Temperatur	е	×
Temp. max	250 -	$\checkmark \times$

l emperature	Э	×
Temp. max	250 💌	$\checkmark \times$
	250 400	



	10 8 6 4	
	2	

🖿 WDS 400 start up

### Preparing the Analyzer for Performing Measurements

- Activate the power supply to the WDS 400 (i.e., switch the "On/Off" switch to the "On" position).
- The first time the analyzer is put into operation, enter the serial number of the analyzer and the port number of the serial interface on the "Device" page of the "Setup" dialog in the control and evaluation program (see "Program Configuration (Setup)" in this chapter).
- Before activating the supply of carrier gas to the analyzer (i.e., before opening the gas supply valve), start the control and evaluation program.

### Note:

As long as there is no communication between the analyzer and the PC, all valves are kept closed to prevent moisture from entering the gas lines.

- > As soon as communication is established between analyzer and PC, the program begins its start-up phase. This is indicated in the status bar.
- Open the supply valve (pressure reducer valve) on the carrier gas supply carefully and use a pressure gauge to set the pre-pressure to approx. 1 bar.
- Set the gas flow rates for "oven door open" ("load") and "performing analysis" ("measure") modes. The "load" and "measure" fine adjustment valves are located on the left-hand side of the analyzer. The active flow rate is shown by the indicator on the front of the analyzer. (Refer to the diagram of the analyzer under "Overview.")

Set the flow rates as follows:

- Close the oven door.
- Open the two valves ("load" and "measure") all the way (turn counterclockwise).
- Regulate the "load" valve until the gas flow rate is 600 to 800 ml/min. This is the counter-flow, activated automatically when the oven door is open to prevent moisture from entering the chamber.
- Regulate the "load" valve until the gas flow rate is 100 to 200 ml/min. This is the gas flow rate used to conduct water vapor from the oven to the moisture sensor. This flow rate should be maintained at a constant level over an entire analysis series, including tare measurement, calibration and sample analysis.

During operation of the analyzer, the PC program switches automatically between the "load" and "measure" rates when the oven door is opened and closed.

## Setup × Analysis Data Device Correction User Oven Min. analysis time Image: min Switch off current 1.000 mA Image: Display time </td

### Program Configuration (Setup)

- To run the Setup program, select "Setup..." from the "Edit" menu, or click on the *P* button.
- > The "Enter Setup Password" window opens.
- Enter the password, if one has been assigned, and click on "OK." The first time you start the program, no password is assigned.
- > The "Setup" dialog opens. There are five dialog pages: "Analysis," "Data," "Device," "Connection" and "User." The "Analysis" page is active when you first open the Setup dialog. To select a different page, click on the desired tab.
- Enter or change the data in the input fields as needed (see below).
- To close the Setup dialog and save any changes in the configuration, click on "OK."
- To close the Setup dialog without saving changes, click on "Cancel."
- 1. "Analysis" Page The minimum analysis time and minimum analysis current are configured here.
- Minimum analysis time: This is the shortest period of time during which the analysis must run before the minimum analysis current is applied as shutoff parameter.

Recommended setting: 15 minutes, depending on the sample to be analyzed. The longer the analysis time, the greater the accuracy of measurement. Perform a series of tests to determine the optimum time for standard analysis of a given sample.

### Note:

The temperature curve defined for a particular analysis specifies a length of time for that analysis. If the time specified is shorter than the minimum analysis time configured in the Setup program, analysis is interrupted when the time specified by the temperature curve has elapsed, even if the minimum analysis current has not been exceeded.

 Switch-off current: After the configured minimum analysis time has elapsed, the analysis is stopped when the current drops below this limit. The lower this limit, the greater the accuracy of measurement.

Recommended setting: 0.500 mA.

Setup		n]]]	×
PID Amplification Intergration Derivation	25 sec 100 sec	Days to log	1
<u> </u>			



• 2. "Data"	Page
-------------	------

- The parameters of the PID regulator for the oven heater are configured in the "PID" column. The values for amplification factor, integration range and differential range ("Derivation") are configured for optimum regulation. These parameters may be changed only by a service technician.
- The "Days to log" parameter is shown in the "Settings" column. This defines the interval, in number of days, at which a new log file is created. These log files are intended for use by service technicians in error diagnosis.
- 3. "Device" Page

This page contains information on the analyzer connected and the parameters for communication between analyzer and PC. Enter the serial number of the analyzer and the port number of the serial interface on the PC the first time the analyzer is put into operation. The baud rate ("Speed") is set automatically.

The "Type" field shows the analyzer model; this is also entered automatically.

- $\odot\,$  Enter the serial number of your WDS 400 in the "Serial no." field. This number can be found on the manufacturer's ID label on the back of the analyzer.
- Enter the data required for connection to the PC in the "Port" and "Speed" fields. Click on "OK" to store the data; the interface is automatically configured for communication with the analyzer.

Setup	x
Analysis Data Device Correction	User
Calibration	Tare
Standard	Standard
Date 31.03.2003	
Cell value 10711 As/g	
Water content 1.070 %	
Cell factor 1.000	
	· · · · · · · · · · · · · · · · · · ·
<u>0</u> K <u>C</u> a	ncel <u>A</u> pply <u>H</u> elp

• 4. "Correction" Page

The "Standard" fields show the names of the files used for compensation of "background" moisture (see "Tare Measurement") and for calibration. The results of the most recent calibration measurement are also shown here, including:

- Date: Date of the most recent calibration
- Cell value: The amount of charge required for the electrolysis of 1 g of water (i.e., the Faraday constant in relation to 1/18 Mol). The value is 10711 As/g; this is a constant value.
- Water content: This value is obtained from the calibration standard (listed on the sample container, for example), and is entered at the start of calibration.

### Example:

The water content of the calibration standard supplied with the analyzer, sodium tungstate ( $Na_2WO_4$ ) is 1.07 %.

Analysis   Data   Device   Correction   User		
Passwords		
Login	Setup	Master
Old		
New		
Confirm		
Change	Change	Change
<u>0</u> K	<u>Cancel</u>	ply <u>H</u> elp

Cell factor: The percentage of water measured is multiplied by this factor to calculate the water content of the sample.
 This factor is calculated as follows in calibration: Cell factor = Nominal value of water content / actual water content as measured.

Example: A calibration measurement is performed with sodium tungstate ( $Na_2WO_4$ ) which has a water content of 1.07 %. The water content measured is 1.20 %. This yields a cell factor of 1.07 / 1.20 = 0.89.

- ▲ The first time the program is run, the input fields on this dialog page contain factory default values and the program cannot be used for performing measurement on samples. Tare measurement and calibration measurement must be performed before the analyzer is ready to use on samples. The factory default setting for the cell factor is 1.0. This is the value used for the first tare measurement.
- 5. "User" Page

X

Passwords for program access are configured on this page:

- Login password: Enables access to the general user level.
- Setup password: Enables access to the Setup program
- Master password: Enables access to both the user level and the Setup program

When the program is first installed, all input fields on this page are blank.

Because the master password enables access to both the user level and the Setup program, it must be configured if either of the other two passwords are configured.

Recommendation: Assign all three passwords immediately after installing the program.

### Note:

Passwords are case-sensitive; for example, "xyz" is not the same as "Xyz".

- Entering or changing a password:
- In the desired column ("Login," "Setup" or "Master"), enter the currently valid password under "Old." If no password is currently assigned, leave this field blank.
- Enter the new password under "New," and again under "Confirm."
  Click on the "Change" button. The password is changed, and the
- Click on the "Change" button. The password is changed, and the text on the button for closing the Setup program changes from "OK" to "Close."
- Change one or both of the other two passwords, if desired.
- Click on "Close" to exit the Setup program, or click on a tab to open a different dialog page.





### **Flushing the System**

- The analyzer must be flushed thoroughly with dry gas before it is used for the first time, and after a long period of disuse, to remove all traces of moisture or unwanted gases from the system.
- To do this, the program must be in "ready-to-operate" mode (the status bar shows "WDS 400 is ready").

### Note:

If the status message "WDS 400 is ready" is not displayed, the inlet and outlet valves are closed to prevent moisture from entering the analyzer.

### Note:

When the analyzer is in "ready-to-operate" mode, the flow of carrier gas can be switched from normal operation (gas flows through the oven and the Teflon filter) to bypass mode (oven and filter are bypassed) and back again. To do this, select "Switch gas flow" from the "Edit" menu or press the "F4" function key. The switching of the valves should be audible. The active mode (normal or bypass) is indicated in the status bar of the "WDS 400" program.

- Normal operation: The wide line at the top, representing the oven, is colored blue.
- Bypass mode: The "oven" symbol is black, and the narrow line passing below this symbol is blue.
- Set the gas flow rate at the "measure" valve to approx. 100 ml/min.
- Observe the level of analysis current.
   If the current exceeds 50 mA, reduce the gas flow until the current is no higher than 50 mA.
- After a brief pause, reset the gas flow rate to 100 ml/min.

### **Continuous Operation**

The analyzer is approved for use in continuous operation.

- If the analyzer will not be used for an extended period of time (e.g., overnight or on weekends), reduce the gas flow rate. To prevent moisture from entering the analyzer when the next measurement is started, however, and to ensure consistently accurate measurements, do not set a flow rate lower than 50 ml/min.
- If the analyzer will not be used for a longer period, it can be shut off. In this case, it must be flushed thoroughly (see "Flushing the System") before the next analysis, which is relatively time-consuming.

### **Tare Measurement**

To attain the highest possible accuracy it is essential to detect the presence of any moisture that does not come from the calibration standard (nor, during analysis, from the sample) before performing the calibration measurement. The procedure for this detection is referred to as "tare measurement." This procedure detects "back-ground" moisture; i.e., residual moisture in the flow of carrier gas, moisture that enters the system when opening and closing the oven door, and moisture on the sample scoop. Always perform tare measurement using the same temperature curve definition (for example, "Standard.pfl") and under the same conditions as the subsequent sample analyses, with the single exception that no sample is present on the sample scoop.

### Note:

The tare measurement routine automatically uses the "Tara.pfl" file for temperature curve definition. For this reason, once you configure your temperature curve you need to save your definition under the file name "Tara.pfl" (by selecting "Save as..." from the "File" menu). For details, see "Setting the Temperature Curve" in the section entitled "Sample Analysis."

### Note:

The heating time configured in the Setup program is not overwritten when performing tare measurement and calibration measurement.

- Select "Tare..." from the "Edit" menu.
- > The "Tare Assistant" window opens.
- Click on "Finish" (to cancel tare measurement, click on "Cancel").
- > The "Tare" window opens.
- Open the oven door, use the forceps supplied to insert the empty sample scoop (as far as it will go) and close the oven door until you hear the fastener click into place. Click on "OK" to begin the tare measurement.
- > The tare measurement is shown in the program window. The blue line in the upper graph represents the analysis current. In the lower graph, the brown line represents the target temperature curve and the red line shows the actual temperature curve. The absolute value for the moisture detected is continuously updated and displayed in the "Humidity" block, while the actual values for temperature and analysis current are shown in the "Analysis Data" block.

### Note:

The minimum analysis time configured in the Setup program is not overwritten when performing tare measurement and calibration measurement.

This assistant will belo you to determi	ne a tare offset value
of the given air moisture.	
	Coursel Links
<u>Hexc</u>	Lancel <u>H</u> elp





WDS400		$\times$
?	New tare offs	set found.
	Do you want	to save the new value?
	<u>y</u> es	No
Setup		$\boxtimes$
Analysis Data	Device Correction	User
Calibration		Tare
Standard		Standard Tare20030505-095626
Date	05.04.2003	
Water content	1 070 %	

Cancel

1.068

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- > The following toolbar buttons are available during measurement:
- Stop measurement
- View information about the program (corresponds to "About WDS 400..." in the Help ("?") menu)
- Open the Help program (corresponds to "Help" in the Help ("?") menu)
- Change the temperature curve while the current measurement is running
- > After the minimum analysis time configured in the Setup program has elapsed, the tare measurement is ended when the current drops below the limit configured under "Switch-off current."
- If you wish to save the tare measurement results in a tare file, click on "Yes;" otherwise, click on "No."
- > The result of the tare measurement and the name of the tare file are shown in the Setup program, on the "Correction" dialog page. The data stored in this file is applied to all subsequent calibration measurements and sample analyses for which the tare function is active, until a new tare file is created.

### **Calibration Measurement**

Calibrate the analyzer by performing measurement on a sample with a known water content. Calibration measurement is required in the following instances:

- The first time the analyzer is put into operation
- Following a period of disuse (for example, at the beginning of work following a weekend or, in some cases, every morning)
- Before performing high-precision analysis of sample material
- After cleaning the filter or moisture sensor
- After re-coating or replacing the moisture sensor
- Any time a sample is evaporated by heating at an excessively high temperature

### Notes:

- Three measurement cycles are recommended for the calibration procedure. For the highest demands on accuracy of measurement, perform five measurement cycles.
- Any time more than one cycle is performed for calibration, all cycles are included in the evaluation.

Cell factor

 Calibration Assistant

 Calibration

 How many cycles do you want to perform ?

 Cycles
 3 No. of cycles

 < Back</td>
 Next.>

 Einish
 Cancel

Calibration		$\mathbf{X}$
Sample data		
Please	enter weight and sample data	
Weight	23,9 mg	
Content	1.070 %	
	ОК	

- Perform all calibration measurements carefully, using a smooth, even technique. Use the same technique to be employed during sample analysis.
- The program uses the "Calib.pfl" file as temperature curve definition for calibration measurements. This file is configured for use with the calibration standard supplied with the analyzer (sodium tungstate ( $Na_2WO_4$ ), water content: 1.07 %). If you use a different calibration standard, make sure you modify the "Calib.pfl" file accordingly.
- The following routine is recommended for use the first time the analyzer is put into operation: Flush the system, perform tare measurement, perform calibration (3 cycles).
- For high accuracy, the current should reach a maximum of approx.
   85 mA; i.e., a long, narrow curve is preferable. Bear in mind that the analyzer will automatically switch to bypass mode if the analysis current exceeds 95 mA, to prevent sensor overload. If this happens, the measurement results cannot be used for evaluation.
- To use the calibration standard supplied (sodium tungstate), analyze an initial sample quantity of 20 to 25 mg to obtain a maximum analysis current of 70 to 90 mA.
- Check the calibration of the analyzer regularly by performing analysis on the calibration standard.
- Select "Calibration..." from the "Edit" menu.
- > The "Calibration Assistant" window opens.
- Enter the number of calibration cycles (for example, 3) and click on "Finish" (to stop calibration, click on "Cancel").
- > The window for entering the sample quantity and water content opens.
- Place the required sample quantity on an empty sample scoop. When using the calibration standard supplied with the analyzer, Na<sub>2</sub>WO<sub>4</sub> with 1.07 % relative moisture, use approx. 20 to 25 mg.
- Use the forceps supplied to insert the sample scoop in the oven as far as it will go. To do this, grasp the tab on the end of the sample scoop using the forceps. Close the oven door until you hear the closure mechanism click into place.
- Enter the initial sample weight and water content in the respective input fields. The water content is indicated on the sample container; for the calibration standard supplied with the analyzer (Na<sub>2</sub>WO<sub>4</sub>), this is 1.07 %. Click on "OK" to confirm your input.





Calibration finished					
Calibra	Calibration has been finished				
Data determination sucessf	ul:				
Run G (mg) Abs(µg)	Rel(%)	G Exp(µg)	Corr		
1 23.90 288.155	3 1.070	255.7	0.9		
Cell Factor 0.887					
Please, choose an option					
Save Save calibration and exit					
Cancel Exit calibration without saving					

Setup 🛛					
Analysis Data Device Correction User					
Calibration Tare					
Standard Calib20030505-101537	Standard Tare20030505-095626				
Date 05.05.2003					
Cell value 10711 As/g					
Water content 1.070 %					
Cell factor 0.887					
	ncel <u>Appy</u> <u>H</u> elp				

- To start the calibration measurement, click on "OK."
- > The calibration routine runs automatically until all measurements have been completed. The analysis display is continuously updated during calibration. The upper graph shows the analysis current over time, and the lower graph shows the target and actual curves for the temperature over time.

### Note:

If the measurement begins at a temperature higher than that specified by the target curve, a warning is displayed on the PC screen. In this case, measurement begins only after you click on "Yes" to confirm the warning message. To stop the measurement, click on "No."

### Note:

When performing multiple measurement cycles, proceed as follows: Use a second sample scoop. While the first measurement is running, place the second sample scoop on the weighing instrument and fill it shortly before the first measurement is completed. At the end of the first analysis, open the oven door, remove the first sample scoop and place the second sample scoop in the oven. Close the oven door, enter the initial weight in the PC program and start the next measurement. While this measurement is running, remove the sample residue from the first sample scoop (if necessary, use the brush supplied to clean the scoop), place the sample scoop on the weighing instrument and fill is shortly before the second measurement is completed. At the conclusion of the second measurement, remove the second sample from the oven and insert the third the sample. Enter the initial weight in the PC program and start the third measurement. To perform more than three cycles, continue as described above.

> At the end of the calibration procedure, the result is displayed in the program window.

The most important value obtained from the calibration measurement is the cell factor. In all subsequent sample analyses, the water content measured is multiplied by this factor to calculate the actual water content. In the example shown here, the calibration measurement yielded a value of 1.2 % for the relative water content. The nominal value for the calibration standard used is 1.07 %. This yields a cell factor (correction factor) of 1.07 / 1.2 = 0.89.

- To store the calibration measurement, click on "Save." (To cancel the calibration routine without saving the result, click on "Cancel").
- > Once calibration has been completed successfully, enter the name of the calibration file, the target value for the water content of the calibration standard used, and the cell factor on the "Correction" page of the Setup program. This calibration data is valid until it is overwritten by a subsequent calibration measurement.

### **Sample Analysis**

In order to choose an appropriate sample quantity for testing, it is important to know approximately which temperatures are required for expelling water from the particular sample material. The temperature curve developed for the actual analysis defines both the starting temperature and the various levels of temperature to be attained during analysis, as well as the length of time each temperature is maintained to expel the different types of water.

The peak level(s) of current produced during analysis depend(s) on the quantity of water released at a given temperature, which in turn is dependent on the quantity of sample material tested. The higher the peak, the greater the accuracy of measurement. At the same time, the analysis current must not exceed 95 mA, as this would cause the bypass valve to be activated to protect the electrolytic cell from overload, and the measurement would have to be discarded. The optimum peak value for the analysis current is 75 to 90 mA.

- The analysis time defined by the temperature curve supercedes the minimum analysis time configured in the Setup program.
- Leave the gas flow rate unchanged, if possible. Perform tare measurement, calibration measurement and sample analysis using a single gas flow rate.
- When analyzing new or unknown substances, perform a series of test measurements to determine the optimum temperature curve.
- The following is generally applicable: For powdered substances, use a flatter temperature curve; for granulates, use a steeper curve. For inorganic substances, try temperatures from 250 to 400 degrees, while making sure the substance does not disintegrate (examine the color and smell after drying). Use > 20 mg as sample quantity; do not exceed the maximum initial weight of 25 to 250 mg.



### Setting the Temperature Curve

- Click on the *is* button ("Load temperature curve"). There is no menu command for accessing the temperature curve. If the toolbar is hidden, select "Toolbar" from the "View" menu to re-activate the toolbar display.
- > The "Temperature profile" window opens.

Temperature curves are stored as files on the PC with the file name extension ".pfl". When you open the temperature curve window, the most recently used temperature curve file is displayed. If the PC has been restarted since the program was last used, the default file ("Standard.pfl") is opened. The file name is shown at the top of the window, under "File" (in the example shown here, "Standard.pfl").

Temperature profile				
File				
Gypsum.pfl 🔄 🖃				
Points				
ti + =				
Time (min) Temp. (°C) 🔺				
0:00 45				
2:00 45 🗏				
4:00 110				
10:00 110				
12:00 250 💌				
<				
OK				



Temperature profile	;			
Gypsum	99			
Points				
16 + -				
Time (min)	Temp. (°C) 🔺			
2:00	45			
4:00	110			
10:00	110			
12:00	250			
15:00	250 💌			
•				
OK				

The corresponding temperature curve is shown under "Points" and is also mapped out graphically in the "Temperature/Time" graph in the lower portion of the main window.

Selecting a different temperature curve file:
 Click on the "Browse" button (folder symbol) next to the file name field and select the desired file.

If the time/temperature table of the temperature curve has more than six time points, only part of the table is displayed; use the scroll bars at the bottom and on the right-hand side of the table to view all points.

○ Modifying the temperature curve:

Click on the red "plus" sign in the upper left-hand corner of the window. The gray background changes to white. In the "Temperature/Time" graph, the mouse cursor is displayed as a cross, and a marker below the graph indicates its position along the X-axis (time). The coordinates of the mouse cursor are displayed on a gray background in a separate field.

- Entering a new temperature level:

Position the mouse at the desired coordinates in the "Temperature/ Time" graph and click. The coordinates are automatically added to the table. Click again to remove the new point (see the illustration on the left).

Editing the time/temperature table:

Click on the desired row to select it, and then click on the "+" button. The selected row is copied, with a time 30 seconds later (see the illustration on the left). Double-click on the new row to edit time and temperature. The "Time" field accepts only full minutes. Click on "OK" (to the left of the edited row) to confirm.

Deleting a temperature level: Position the mouse on the desired point in the "Temperature/Time" graph and click.

Deleting a row from the time/temperature table: Click on the row to select it, and then click on the "-" button.

- Toggling between "Edit" and "List" mode: Click on the button with the red "plus" sign. In "List" mode, the table is displayed on a gray background; in "Edit" mode, the background is white. The mode changes each time you click on the "+" button (i.e., the table is toggled between modes).
- Saving the temperature curve under a different name: Click on the "Drive" button (symbol of a computer drive) next to the file name field, select the desired path and enter a file name.
- Confirming changes in the temperature curve: Click on "OK" at the bottom of the window. The window closes and this file is used for subsequent measurements.



Analysis Assistant 1/2 Temperature	×
Date of analysis	04.06.03 10:13:00
Description	Gypeum
Number	Lot #45
Origin	Production line #2
Comment	
< Back Newt > Em	하 Qancel Help



### Performing the Analysis

Note:

Store the sample material in close proximity to the place of measurement (i.e., the place of installation of the WDS 400 and the weighing instrument). Short distances prevent errors in measurement that result from evaporation of volatile water components, or from absorption of humidity in hygroscopic samples.

- Click on ("New file") or select "New" from the "File" menu. This creates a new file for saving measured values.
- Select a suitable temperature curve (see the previous section). In the example shown here, a temperature curve with 4 heating stages, saved as "Gypsum.pfl," is used to analyze gypsum (see the illustration on the left). The analysis time derived from this temperature curve is transferred to the "Analysis time" field in the "Sample" block in the main window.
- Specify whether analysis will be performed with or without background moisture compensation (i.e., whether the value determined in tare measurement is taken into account; see the "Note" at the end of this section for details). If the **I** button is graphically displayed as "pressed," this means tare compensation is activated. Click on this button to turn the tare compensation function on or off.
- Click on ("Start analysis") or select "Analysis..." from the "Edit" menu.
- > The first window of the "Analysis Assistant" opens.
- Enter the data specifying the sample here (i.e., designation, batch number, source, comment). The date of analysis is entered automatically. The data configured here is transferred to the "Sample" block in the main window.

Click on "Next" to confirm your input (to cancel, click on "Cancel").

- > The second "Analysis Assistant" window opens.
- Place the sample scoop on the weighing pan, tare the weighing platform and weigh in the sample material.

See "Equipment Supplied" in the chapter entitled "Getting Started" for a table of estimated sample quantities, listed by water content. When analyzing samples with unknown water content, conduct a series of test measurements first to determine the optimum parameters for initial weight and temperature curve.

• Enter the initial weight in the "Sample weight" field. The value is transferred to the "Weight" field in the "Sample" block in the main window.

Click on "OK" to confirm your input (to cancel, click on "Cancel").

> A window opens with a prompt to insert the sample into the oven.

Use the forceps supplied to insert the sample scoop in the oven.







### Note:

Close the oven door.

To start the measurement, click on "OK."

If the measurement begins at a temperature higher than that specified by the target curve, a warning is displayed on the PC screen. In this case, measurement begins only after you click on "Yes" to confirm the warning message. To stop the measurement, click on "No."

During analysis, the toolbar in the main window is changed (see the section entitled "Tare Measurement"). In addition to the buttons for starting the Help program and viewing program information, the following two buttons are available:

- Stop measurement
- Change the temperature curve while the current measurement is running

The analysis current is displayed and continuously updated both in the "Analysis Data" block in the main window and on the front of the analyzer. The indicator on the front of the analyzer is a 10-stage LED band. Each LED represents a current of 10 mA (see "General View of the Equipment" in the chapter entitled "Getting Started").

If the sample releases too much water per unit of time, there is a hazard that the analysis current might exceed the tolerance limits of 95 mA. In this case, the gas flow should be reduced. This reduces the amount of water carried to the moisture sensor per time unit and causes the analysis current to drop; the measurement does not have to be discarded.

If the tolerance limit for the analysis current is exceeded, the system switches the valves to bypass mode (indicated in the status bar in the main window). The measurement is stopped and can no longer be used for evaluation, because the carrier gas is not flowing through the oven.

In this case a warning message is displayed. The valves remain in the bypass position until you click on the "OK" button to return the system to "normal operation" mode.



WDS 4	00-Error 🛛	
0	Analysis current too high!	
w	Gasflow has been switched to bypass.	
	For more information please take a look at the manual, chapter 'Error handling'.	
	Please press <ok> button to enable gasflow through sample container.</ok>	
	OK	



Sa	ample	
Description		
Gypsum		
Number		
Lot #45		
Origin		
Production line #	2	
Comment		
Date (	)5.05.2003	
Analysis time	25:00	mm:ss
Weight	21.90	mg
_		_
Н	umidity	
Rel	6.171	%
Abs.	1351	μg
Analy	sis Data	
Current act	0.544	mΑ

	Humidity	
Rel	61712	ppm
Abs.	1351	μg

Rel		6.171	%
Abs.		1351	μg
Ar	nalysi	s Data	
Current act		0.544	mΑ
Tare act		0.492	mΑ
Calculated ac	:t	0.070	mΑ
Temp act		370	°C
Switch off cu	rent	0.550	mΑ
Min analysis t	ime	15	min

Generally, the measurement continues automatically until the analysis time has elapsed or until the analysis current goes below the minimum limit configured.

Click on the A button to change the temperature manually. This can be useful to determine optimum settings when testing unknown sample materials. Once the manual temperature modification function is deactivated, the analysis procedure continues with the temperature curve originally configured.

During and after analysis, the analysis current is represented by the blue line in the upper graph ("Current/Time"), and the target (brown line) and actual (red line) oven temperatures are shown in the lower graph ("Temperature/Time"). A marker under each graph indicates the current point in time.

The following data concerning the measurement in progress is displayed and continuously updated in the three information blocks to the right of the diagrams:

- "Sample" block:

Shows data describing the sample. The data is entered by the user at the start of measurement, in the two "Analysis Assistant" windows.

- "Humidity" (moisture) block:

Shows the relative ("Rel.") and absolute ("Abs.") water content of the sample. These are the actual results of the coulometric analysis. The values shown here are calculated and continuously updated during measurement. Click on the "Rel." button to toggle that field between "ppm" and "%" values.

### - "Analysis Data" block:

"Current act:" Active\* analysis current. "Tare act:" Offset (tare) value for the active analysis current (data from tare measurement). "Calculated act:" Calculated net analysis current (i.e. present analysis current minus tare value for analysis current). "Temp act:" Active oven temperature.

\* "active" in this context means "active at the time displayed"

Switch-off current: Level of analysis current below which the measurement will be stopped automatically (value configured in Setup program).

"Minimum analysis time:" Time limit after which the switch-off current (see above) is applied as shutoff parameter (value configured in Setup program).

### Note:

Each analysis can be performed with or without a tare offset, or "background" moisture compensation (see "Tare Measurement" in this chapter). This parameter must be defined before the beginning of analysis and cannot be changed later. When background compensation is active, the offset value is subtracted from each measuring point. The "Analysis Data" block shows the current actually measured ("Current act"), the "tare" current ("Tare act") and the difference calculated between the two ("Calculated act"). When the background compensation function is not active, the "Current" and "Calculated" values are identical, and the "Tare" field shows zero. Negative calculated values are displayed and stored numerically, but are not displayed in the graphs. They are, however, included in the "Integration" calculation. At the end of measurement, both corrected and uncorrected values are stored.

Arabia	has been finished, please select an option
Sale	Tave analysis clara to file and exit
Canol	Est eithest seving

- > When analysis is concluded, whether automatically when the analysis time runs out or the minimum current is reached, or manually interrupted by clicking on , the "Analysis finished" window opens.
- To store the measurement, click on "Save." To quit without storing, click on "Cancel."

When you click on "Save," two files are stored: one containing uncorrected data (i.e., without background compensation), with the file name extension ".ods", and one containing corrected data, with the extension ".wds". Both files are handled in the same manner when opened at a later point (see "Loading a Measurement File" below). If the measurement was performed without background compensation, both files have the same contents.

### Loading a Measurement File

• Click on e or select "Open" from the "File" menu; browse as needed for the required path and select the desired file.

### Note:

The default setting for "File type" is for files with corrected data. To open a file with uncorrected data, change the "File type" setting from "WDS 400 files (\*.WDS)" to "WDS 400 Realdata files (.ODS)."

> The selected file is opened in the main program window.

In the example shown here, the file created for the analysis of gypsum (see "Sample Analysis" above) is opened again for further processing, such as fractionated evaluation, for example.

### Note:

Loading and evaluating existing files can be performed "off line;" i.e., the PC does not have to be connected to the analyzer.

Öffnen				? ×
<u>S</u> uchen in:	🔄 Wds400	- 6	t d	
log Neuer Ord WDS400 WDS400, Tare2003	dner Data _Data_1 0408-101625.WDS			
Datei <u>n</u> ame:	*.WDS			Ö <u>f</u> fnen
Datei <u>t</u> yp:	WDS 400 files (*.WDS)			Abbrechen
	WDS 400 files (*.WDS) WDS 400 Realdata files (*.0DS)			





In addition to the commands for opening a new or existing file, saving a file, viewing program information, and starting the Help program, the following buttons are available in the toolbar:

- Blue line: Display the analysis current in relation to time ("Current/Time") in the upper graph (default setting).
- Green line: Display the weight of the water contained in the sample in relation to time ("Weight/Time") in the upper graph.
- Fractioning function: Manually define the integration limits for detection of different forms of water bond.

When the file is loaded, the upper portion of the program window shows the "Current/Time" graph (see the illustration above). The  $\checkmark$  button (blue line) is displayed as "pressed."

○ If desired, click on the button (green line) to change the display from the "Current/Time" graph to the "Weight/Time" graph (see the illustration on the left). This button is now activated (graphically displayed as pressed), while the blue button is displayed as inactive. Click on each of these buttons in turn to switch the display back and forth between the two diagrams.





The buttons in the upper right-hand corner of the upper graph are used to change the "Time" axis (see also "Menu Bar and Toolbar Buttons" under "Preparation" in this chapter).



Fraction 🗾					
secor • 1 3	nds 0 t2	<ul> <li></li> <li>10 45</li> </ul>			
Water content					
Integration		4.920 %			
Value (µg)	tt	t2			
1077	3:0	10:45			
0.000	0:0	0:0			
0.000	0:0	0:0			
0.000	0:0	0:0			

### **Evaluating a Measurement File**

The measuring principle of the WDS 400 coulometric analyzer makes it possible to distinguish the individual water components that are bound to the sample material through different physical forces. The water content of the sample is determined through integration of the analysis current curve over time. By selecting explicit integration limits, the content of a particular peak can be determined. This is referred to as "fractionated evaluation," and is performed as follows:

- Open the file that contains the measurement values from the sample to be tested (see "Loading a Measurement File").
- > The file is shown in the program window. The upper graph shows the analysis current, and the lower graph shows the temperature (target and actual values) in relation to time. Click on the (green line) and (blue line) buttons to switch the display from the "Current/Time" graph to the "Weight/Time" graph and back again. For fractionated evaluation, the default display ("Current/Time") is the most practical alternative.

- > The "Fraction" window opens.

The "Water content" window shows a table with four rows for integration of up to 4 user-definable time periods. To integrate additional time periods, overwrite the values in an existing row in this table.

- Click on a row in the table (for example, the first line, as shown in the illustration on the left).
- > The line selected is then displayed on a gray background.
- Define the integration range.

To do this, set the lower ("t1") and upper ("t2") integration limits in the "Movement" (integration) block using the "<" and ">" buttons. If necessary, click on the  $\checkmark$  button to change the time unit for one or both integration limits (units available: seconds and minutes). For each integration limit, minutes are shown on the left and seconds on the right.



> The result of the integration is displayed immediately in the "Water content" block.

The water content is shown as a percentage under "Integration." Click on the "%" button to toggle the value displayed between "percentage" and "ppm." The row selected in the table shows the absolute value for the water released within the integration range defined by t1 and t2. The selected integration range is marked by gray shading on the "Current/Time" or "Weight/Time" graph and on the "Temperature/Time" graph.

 $\bigcirc$  Integrating additional time periods:

Click on a row in the table and proceed as described above. If there are no empty rows, simply overwrite existing data with the desired values to configure additional integration ranges. If necessary, write down the existing data first.

 $\bigcirc$  Ending the fractionated evaluation: Click on  $\exists$  again, or click on the "x" in the upper right-hand corner of the "Fraction" window. When the "Fraction" window is opened again with the same measurement file, the most recently entered data is loaded automatically and the fractionated evaluation can be continued as described above.

### Printing a Measurement File

Measured data can be printed out on the default printer installed under Windows.<sup>®</sup> The printout generated by the "WDS 400" program includes all data required for compliance with GLP standards, together with the "Current/Time" or "Weight/Time" graph and the "Temperature/Time graph. If one or more fractionated evaluations have been calculated for a particular sample, the corresponding results (i.e., from the selected row in the table) are printed on a separate page.

Windows ® is a registered trademarks of Microsoft Inc.

### Note:

Measured data can be printed at any time. Simply open the measurement file containing the desired data (see "Loading a Measurement File" above).

• Open the "File" menu.

The following options are available here:

- "Print..."
- "Print preview..."
- "Printer Properties..."





○ Select "File > Print" to generate a printout on the default Windows<sup>®</sup> printer.

 $\bigcirc$  Select "File > Print preview..." to view the file as it will be printed.

- This window has a toolbar with buttons for increasing the on-screen size of the file ("Zoom In," "Zoom Out") and activating the printout. To close the print preview and return to the main program window, click on "Close."
- $\bigcirc$  Select "File > Printer Properties..." to open the standard printer configuration window.

### **Troubleshooting Guide**

Error	Possible cause	Solution
Software error 7	Data transmission error between WDS 400 and PC	Check PC < - > WDS connecting cable
Software error 9	Data transmission error between WDS 400 and PC	Software installation of second device (e.g., mouse) must be configured to use the same interface
WDS analyzer not connected (Status: "no connection")	Incorrect COM port setting	Check the COM port settings
	Defective connecting cable Analyzer not switched on	Check the connecting cable (null modem cable) Switch on analyzer
Current overload after power-up	Analyzer switched off too long	Reduce gas flow until the current reaches a level below 90 mA
	Oven door left open	Flush analyzer continuously
Current too high during measurement	lnitial weight too heavy	Reduce gas flow until all water has been expelled Choose a smaller initial quantity for testing
	Temperature increased too quickly	Lessen the increase gradient of the target curve (e.g., from 30°C to 200°C in 3 minutes rather than in 1 minute)
Excessive fluctuation in oven temperature (> 5 °C)	Unfavorable PID parameters	Check the PID parameters; increase if necessary (contact service center)
Water content much lower than expected (e.g., during routine measurements)	Defective sensor coating System contaminated	Re-coat sensor In the event of excessive accumulation of impurities in analyzer and sensor, contact the service center
Strip conductors working loose from sensor	Sensor too old, or chemically corroded Large number of samples with high water content (> 5 %) analyzed	Install new sensor Avoid frequent overloading of the sensor (current > 100 mA)
When switching from oven to bypass, the gas flow changes significantly (> 50 ml / min)	Oven is leaking	Replace the seal on the oven door Check closure mechanism
Cell factor > 1.3 or < 0.8	Sensor coating damaged Leak in gas system	Re-coat the sensor Check for leakage; if necessary, send analyzer to service center
	System contaminated	In the event of excessive accumulation of impurities, contact the service center

• In case of current overload, reduce gas flow immediately.

 If the analyzer has already switched to "bypass" mode, leave the gas flow at the lower rate for several minutes before confirming the error message to switch back to normal mode. Reduce the gas flow first.

### **Care and Maintenance**

### Service

Regular servicing by a Sartorius technician will extend the service life of your analyzer and ensure its continued accuracy of measurement. Sartorius can offer you a service contract with your choice of service interval.

The optimum length of the service interval depends on the operating conditions at the place of installation and on your requirements.

### Repairs

Repair work must be performed by trained service technicians. Any attempt by untrained persons to perform repairs may lead to hazards for the user.

In the event that repairs are to be performed, whether at the place of installation or upon shipment of the analyzer to the manufacturer, the user must provide a certificate of safety. In this certificate, the user declares that there are no toxic substances accumulated in or contaminating the analyzer. This applies in particular when the gas tubing must be replaced due to sample evaporation.

### Cleaning

- ▲ Make sure that no dust or liquid enters the analyzer housing.
- ▲ Do not use any aggressive cleaning agents (solvents, abrasive cleaning agents, etc.); clean the analyzer using a piece of cloth which has been wet with a mild detergent (soap) only.
- Disconnecting the power supply: Unplug the power cord connector from the socket on the back of the analyzer and disconnect the data cable connecting the analyzer to the PC.
- Check the sample chamber (oven) at regular intervals for deposits or spills of sample material. Carefully remove any sample residue/spilled powder using a brush or a hand-held vacuum cleaner. Use acetone to remove stubborn residues.
- ▲ When cleaning the sample chamber, make sure the thermoelement at the rear of the chamber is not damaged or shifted.

- Check the inner seal of the closure mechanism on the door; if necessary, clean the seal by wiping it with a dry cloth.
- If more thorough cleaning is necessary, contact the Sartorius service center.
- Check the Teflon filter at regular intervals. For details, see section "Cleaning and Replacing the Teflon Filter" at the end of this chapter.
- If a correction factor of < 0.8 or > 1.3 is obtained from a calibration measurement that was performed with all due care, the moisture sensor must be cleaned or replaced. For details, see "Cleaning and Replacing the Moisture Sensor" at the end of this chapter.
- Clean the nickel sample scoop included with the analyzer (in the Accessory Set) as follows:

Pure nickel is especially resistant to corrosion by alkaline and neutral solutions. Due to its high melting point (1453 °C), organic substances that adhere to a nickel vessel (such as resins or plastic fusions) can be removed through annealing. The resulting discoloration of the vessel does not affect its utility for subsequent analyses. Because nickel will dissolve in acids over time, do not use concentrated acids for cleaning.

- Use a brush to remove powdered and granulated sample material.
- Use a solvent (acetone, ethanol or methanol) to remove organic material that adheres to the sample scoop. Residues that cannot be removed using solvent can be burned off by annealing.
- Use water or an alkaline solution to remove inorganic residues; do not soak the scoop in an acidic solution for more than a very brief period.

### Safety Inspection

If there is any indication that safe operation of the device is no longer warranted:

- Disconnect the power supply: Unplug the power cord from the wall socket (mains).
- > Lock the equipment in a secure place to ensure that it cannot be used for the time being.

Safe operation of the equipment is no longer ensured when:

- there is visible damage to the device or power cord
- the analyzer no longer functions properly
- the equipment has been stored for a relatively long period under unfavorable conditions
- the equipment has been subjected to rough handling during shipment.

In this case, notify your nearest Sartorius Service Center. Maintenance and repair work may be performed only by service technicians who are authorized by Sartorius and who:

- have access to the required service and maintenance manuals, and
- have attended the relevant service training courses.

### Recycling

Sartorius products are packaged to ensure safe shipment using environmentally friendly materials. After successful installation of the analyzer, you should return this packaging for recycling because it is a valuable source of secondary raw material.

When submitting the analyzer for disposal, the user must provide the disposal technician with a certificate of safety.





### **Cleaning and Replacing the Teflon Filter**

The Teflon filter protects the moisture sensor from particles > 3 µm. Check the filter regularly for impurities and clean as needed.

Proceed as follows to check and, if necessary, clean the filter:

• Open the filter retainer on the back of the analyzer.

- Remove the Teflon filter.
- Check the filter (visual check).
- $\bigcirc$  If the filter is contaminated (indicated by discoloration or noticeable odor), it must be replaced.
- $\bigcirc\,$  If the filter was contaminated, clean the filter housing with acetone or naphtha.
- $\bigcirc$  When replacing the filter, make sure the new filter is pressed firmly into both O-rings for proper sealing.
- Close the filter retainer carefully.
- When closing the filter housing, make sure to clean the outer O-ring thoroughly and apply a lubricant.

### **Cleaning and Replacing the Moisture Sensor**

The moisture sensor steadily loses sensitivity through sustained use. This is caused, for example, by impurities accumulated in the sensor which deactivate the coating, as a result of which only part of the water in the carrier gas is electrolyzed. In this case, a lower proportion of water is detected than that actually contained in the sample. The correction factor (cell factor) is initially > 1.0. If a cell factor of > 1.3 is measured, check the sensor and replace it if necessary. The same applies for a cell factor of < 0.8. In this case, a greater proportion of water is detected than that actually contained in the sample. If the calibration measurement was performed with all due care (i.e., the proportion of water not originating from the sample is equal to the tare measurement value), the apparent detection of excessively high water content may be caused by leakage current in the moisture sensor.





Proceed as follows to check and, if necessary, replace the sensor:

- Loosen the fastening screws in the holding device on the back of the analyzer until the holding device can be pulled out of the electrolytic cell.
- △ Use extreme caution when handling the sensor holding device, as the ceramic sensor is fragile and easily damaged or broken.
- Removing the ceramic sensor from the holding device: Remove both Phillips head screws. The two springs between the ceramic sensor and holding device cushion the contact between the electrodes on the small ceramic plate and the connections inside the analyzer.
- Cleaning the moisture sensor (ceramic plate): Rinse the sensor under running water; use a soft-bristled toothbrush to remove stubborn deposits, if necessary.
- $\triangle$  Make sure the strip conductors are not loosened during cleaning. Otherwise, the sensor must be replaced.
- Dry the sensor.
- Re-coat the sensor:
- Make a solution of 1 part 85% orthophosphoric acid ( $H_3PO_4$ ) and 9 parts acetone.



▲Warning:

Orthophosphoric acid is caustic. Acetone is highly volatile. For this reason, always wear protective gloves, glasses and lab coat when mixing this solution.







- Soak a lint-free paper towel in this solution and apply an even coating to the ceramic plate. Coat both the front and back of the sensor.
- Perform the steps described above in the opposite order to re-install the moisture sensor as follows: Position the springs in the holding device and affix the ceramic plate, allowing for spring suspension, using the Phillips head screws. Install the holding device in the analyzer, aligning the pin on the back of the analyzer with the bore hole on the holding device. Install the screws to affix the holding device to the analyzer. The electrodes on the ceramic plate are pressed against the contacts in the device by the springs; this closes the electrical circuit.
- Allow at least 60 minutes drying time, under constant gas flow, after re-coating the sensor. The power supply of the sensor is automatically cut off in the event of current overload.

### **Overview**

### **Specifications**

Power supply: Frequency: Power consumption:

Fuse: Operating temperature:

Detection limit: Repeatability: Display range:

Analysis time: Allowable ambient operating temperature: Dimensions:

Weight: Max. sample volume: Carrier gas: Gas throughput during operation: Gas pre-pressure:

230 V  $\pm$  10 % 50 to 60 Hz Standby mode: 100 W Full capacity: 400 W 4 A flink From room temperature to 400°C (752°F) 1 µg water ≤ 2 % ppm - 99.99% (can be toggled between % and ppm) 2 min. ... 10 h +10° C to + 35° C (+50° F to 95° F) Depth: 500 mm Width: 500 mm Height: 180 mm 20 kg Approx.. 3.5 cm<sup>3</sup> Nitrogen, argon, air 100-200 ml / min Max. 1 bar

### **CE Conformity**

The WDS 400 coulometric analyzer meets the tolerance limits set forth in the following standards:

EN 50081 (Limitation of emissions) EN 50082 (Defined immunity to interference) EN 61010 (Safety)

This equipment is suitable for installation in residential and commercial areas and in small businesses. If installed in an industrial area, the equipment may be subject to interference.

### Index

	Page		Page		Page
Accessories	6, 9	Installation (analyzer)	7	Recycling	6, 40
Analyzer functions	14	Installation instructions Installing the control and	7	Repairs	40
Background compensation	18, 19, 25,	evaluation program	11	Safety inspection	40
	33, 34	Intended use	4	Safety precautions	10
Bypass mode	18, 24, 32	Interface	8, 10	Sample analysis Sample quantity	29 6, 31
Calibration measurement	26	Loading a measurement file	34	Service	40
Care and maintenance	40	Login password	16, 23	Setting the gas flow rate	20
<b>CE</b> conformity	44			Setup password	23
Cleaning the analyzer	40	Maintenance	40	Setup	21
Cleaning the moisture sensor	41	Master password	23	Shutoff parameter	21, 29
Conditioning the equipment	7	Menu bar	17	Specifications	44
Connection to PC	10	Moisture sensor	8,41	Storage and shipping	
Contents	4			conditions	6
Continuous use	24	<b>O</b> perating elements	9		
Correction factor (cell factor)	22, 23, 28	Operating the analyzer	11	<b>T</b> are measurement Technical advice	25
Displays	9	Passwords	23	on applications	3
		Performing sample analysis	31	Teflon filter	8,41
Equipment supplied	6	Power supply	8,10	Teflon filter, cleaning	41
Evaluating a measurement	36	Preparation for measurement	16	Temperature curve	29
Exhaust tubing	8, 10	Preparing the analyzer for		Toolbar buttons	17
		performing measurements	20	Troubleshooting guide	39
Flushing the system	24	Printing a measurement file	37		
Fractionated evaluation	36	Program configuration (Setup)	21	Unpacking the equipment	6
		Program start	16		
Gas connection	8, 10	Program window	16	Warning and safety	
General view of the equipment				information	4
(overview)	8				
Getting started	6				

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