

Coulometer WTD – User Manual

Water Determination

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1 Coulometer WTD

The **Coulometer WTD** has been developed, by Diram s.r.o., to perform routine analysis of moisture. Utilizing Karl Fischer titration, the coulometric generation of an iodine titrant allows determination of even trace amounts of water within a sample.

1.1 Description

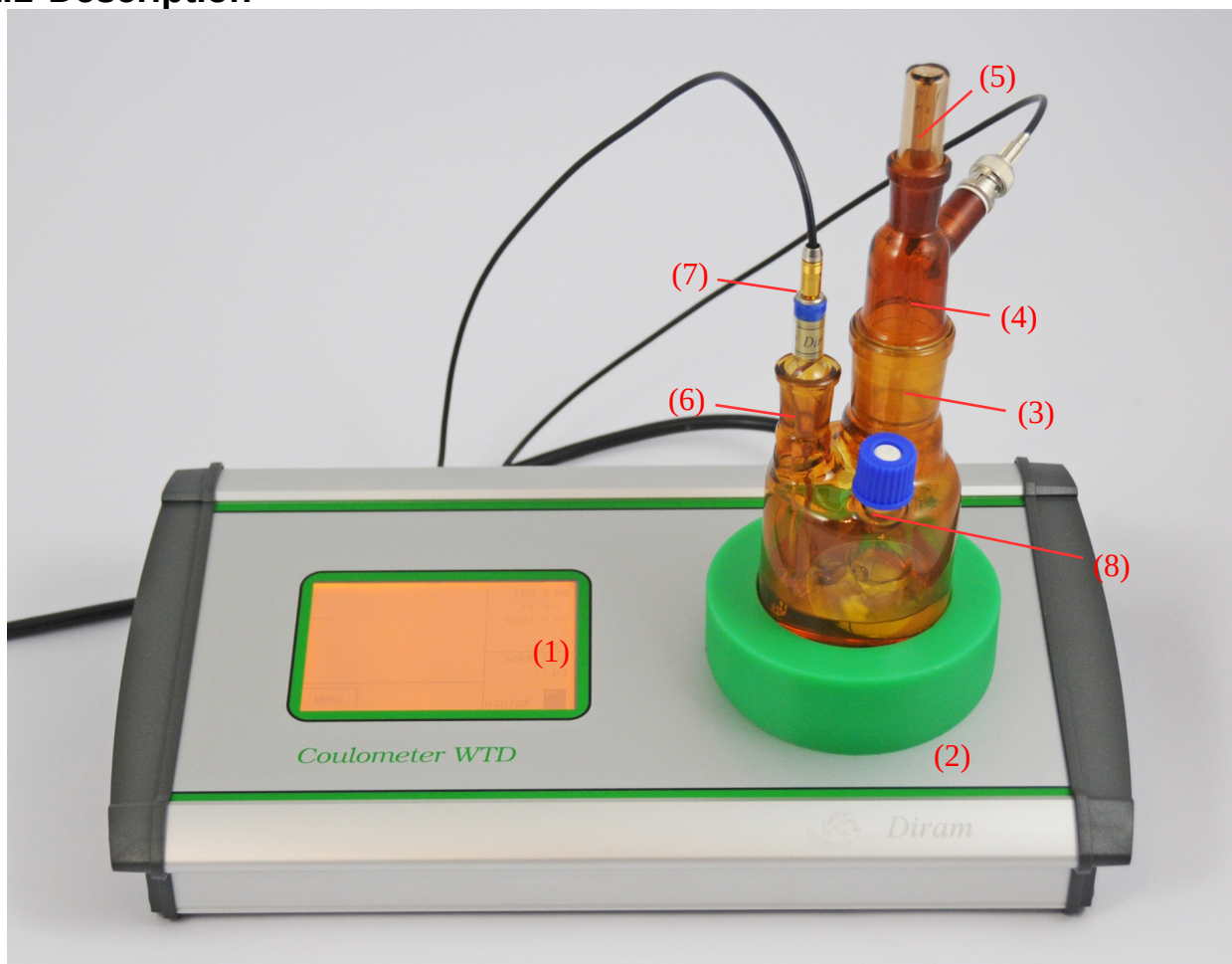


Figure 1: Coulometer WTD

The automatic titrator Coulometer WTD is shown in Fig. 1. It is composed of an aluminum housing, touchscreen (1) and a nest (2) with built-in magnetic stirrer accommodating the titration vessel. The titration vessel consists of a glass bottle with three necks. The large neck (3) with ground joint no 29 serves for a working electrode (4) fitting. On the top it has a glass cap equipped with a capillary (5). A small neck (6) with ground joint no 14 serves for indication electrode (7) fitting and a small screw cap with septum (8) serves for sample insertion. The electrodes are connected by cables to the rear panel of the device.



Figure 2: Rear panel

The rear panel (Figure 2) consists of following components: main switch (9), IEC power socket (10), connector of working electrode (11), connector of indication electrode (12), extension connector for additional modules (13), USB connector (14).

1.2 Delivery content

The set contains: Coulometer WTD device, complete titration vessel with indication and working electrode, two glass caps, one plastic cap with a septum, 2 sets of interconnection cables for electrodes, power supply cable, interconnecting USB cable. Service program **Diram Measure** for Windows is provided on a flash drive.

1.3 Technical specification

Power supply:	230V AC, 35W, fuse T630mA
Display:	monochromatic touchscreen with backlight, 240 x 160 pixels, 4,2"
Indication electrode connector:	SMA
Working electrode connector:	BNC
Connection to PC:	connector USB B
Connection of modules:	connector D-SUB 9 male
Dimensions:	340 mm x 190 mm x 75 mm
Weight:	1.6 kg (3.5 lb)

1.4 Modules

It is possible to connect **Drying oven** (see 2.8 Drying oven) for indirect water determination or **KOH module** for acid number determination as additional modules to Coulometer WTD by D-SUB 9 connector.

2 Water determination

2.1 K. Fischer water determination

Analytical determination of water is usually performed by K. Fischer titration. The original method, published in 1935, defined an iodometric *determination of water* using a solution containing pyridine and methanol with sulfur dioxide. As water reacts stoichiometrically with iodine and other components, it is possible to convert the classical volumetric *determination* to the highly precise coulometric method, with the electrochemical generation of titrant. As thus, coulometry has shown its convenience for *determination* of small and trace amounts of water in organic acids, alcohols, esters, ethers, hydrocarbons and other organic solvents.

A sample to be analyzed is transferred into a titration vessel containing iodide, sulfur dioxide, an amine, alcohol and other solvents and eventually other additives. The iodide is converted to iodine by anodic oxidation on a platinum electrode, and the iodine enables the sequential reaction of water with other components of the solution. The solution's composition can be varied according to the chemical composition of the material to be analyzed or adjusted according to the construction of the coulometric cell. (Steve: This refers to the different composition using a diaphragm or not.)

The iodine concentration in the vessel is monitored by indication electrodes. After the sample insertion the iodine is consumed by the reaction with water, the indication signal changes and the iodine is generated on the anode until the indication signal reaches its original value. The time integral of the current needed for iodine generation is proportional to the water content in the sample - where 1 mol of iodine reacts with 1 mol of water so that 1 mg of water is equivalent to charge 10.71 As.

While this technique is simple and accurate, testers need to apply standard care and thought about the materials to be analyzed. For example, when determining water in ketones and aldehydes, it is necessary to exclude methanol. Alternatively, when determining water in gasses, or using the distillation method where a carrier gas goes through the reaction vessel, it is recommended to use less volatile solvents i.e. propylene glycol to avoid excessive evaporation.

In situations when the titration cell is constructed without a diaphragm separating the cathode and anode compartments, electrochemically reducible substances are added, to eliminate unfavorable reduction of the iodine by the cathode, that can distort the results.

Addition attention is also required when involving oxides of some metals, such as HgO and ZnO as they form iodides or boric acid, carbonates, sulfides, thiols, etc. As such the basic method is not suitable for *determination* of materials that react with the titrant, however, many of the compounds can be determined indirectly.

The indirect variation of the K. Fischer titration technique involves heating the sample separately, in a special oven which then directs the water released from the sample to the titration vessel using an inert carrier gas (i.e. nitrogen or pre-dried air). With this method, it is possible to determine water in solid materials or oils, creating a simple but commercially valuable, technique to perform analysis of water in oils, drugs, sugars, powder extracts, cereals, etc.

Thou in cases when the amount of water in a sample is especially large, it is more convenient to use a volumetric titration.

2.2 Titration vessel

The titration vessel is silica glass chamber with a working volume of 250mL. The working and indication electrodes (cathode and anode electrodes) are mounted in the vessel using ground glass joints no 14 and 29, and are often not separated by a diaphragm; which promotes a more rapid stabilization and a greater stability of the compensation current during the measurement recording. Samples can be injected into the vessel through a septum in a polypropylene cap.

Careful attention is required create an air tight titration assembly. It should be remembered the volume within the vessel will change when injecting fluid samples and because of possible gas production at the cathode. To allow for reasonable equilibrium of internal pressure, the electrode system includes a small capillary vent. The diffusion of water vapor through the capillary vent from the environment to the titration vessel is negligible.

When it is required to use the drying/distillation oven, an additional ground glass joint no 14 is used as part of the assembly; this also allows possible easier insertion of solid samples. Solid samples are weighted in a special small glass bulb equipped with ground no 14 and are inserted into the titration vessel after a rotation of the small bulb in the ground joint.

In situations when a carrier gas is used as a transport method from the drying chamber, it is optionally possible to incorporate a small water condenser to diminish the evaporation of solvent from the titration vessel.

2.3 Preparation of the titration vessel

A new vessel does not need to be specially cleaned. When the vessel is used continuously for multiple analyses of the same sort it normally does not require any cleaning. If necessary the vessel can be rinsed by common solvents (ethanol, acetone) and carefully dried. To remove the remains of oils wash the glass vessel with warm concentrated lye, and then thoroughly wash with water. Dry the glass vessel in the oven; not exceeding 60 °C. When the glass diaphragm is clogged, consider carefully the use of concentrated lye, sulfuric acid or chromosulfuric acid (be sure to take all precautions to avoid potential health and environment risks).

After some time, the *indication electrodes* can become dirty. It is recommended to wipe the electrodes carefully with a cleansing pad every couple of days. The ground glass joints should be coated with an appropriate grease to avoid their locking.

IMPORTANT: minimize the time the solutions are exposed to air to reduce unnecessary moisture absorption. Stock solutions should always be kept in air-tight containers.

It is possible to use 2 variations of electrodes – with a diaphragm, or without. When there is no specific reason for using a diaphragm, it is more convenient to use the electrode system without it.

With either arrangement make sure to select the correct setting in the software before starting measurement.

IMPORTANT: samples containing lithium ions damage the cathode. Lithium containing samples can be analyzed exclusively in the arrangement with a diaphragm. The electrolyte should be exchanged immediately after analysis in order to avoid diffusion of lithium ions into the cathode space.

2.3.1 Vessel with electrodes separated by a diaphragm

The vessel represents, from the viewpoint of electrolysis, an anodic compartment where the titration takes place and the titration agent – iodine – is formed. The anodic compartment is filled to half of the vessel volume, by anolyte AQUATITRON PF, or AQUATITRON BD. The anodic compartment is separated from the cathode by a diaphragm (a porous glass barrier).

The cathodic compartment is filled by a catholyte AQUATITRON K, keeping its level slightly lower than the anodic compartment solution surface. Otherwise, the humidity from catholyte contaminates the anodic space and prolongs the stabilization of the compensation current.

The use of a diaphragm is therefore not suitable for the distillation method or for water determination in gasses where evaporation of the electrolyte occurs. The decrease of the surface causes the contamination of the anodic compartment by moisture from the cathodic part and resulting in an increase in measurement error.

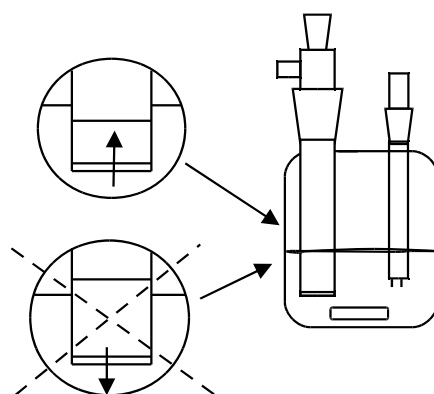


Figure 3: Filling of the vessel with a diaphragm

2.3.2 Vessel with electrodes without a diaphragm

The method is suitable for *determination* of water in materials that can cause clogging of the diaphragm, e.g. some oils. Usually, the titration vessel is filled by AQUATITRON BD solution to a half of its volume.

The advantage of this configuration is a faster stabilization of the compensation current. The disadvantage is possible side reactions of the analyzed material with the cathode; that may lead to a bias of measurement. In some cases, it can negatively affect the measurement precision.

IMPORTANT: Use the relevant standards to establish the required accuracy for the measurements.

2.4 Exhausting of the titration solution

Normally with a single filling of the titrant vessel, it is possible to perform analyses up to approximately 0.2 g of water. The exact amount depends on many factors such as pre-drying of the titration vessel, the manner of filling, etc. When measuring samples with very low water content, the dilution of the electrolyte might limit the use. The electrolyte should be replaced when it is diluted to a double of its original volume.

The reduction of titration capacity is exhibited by an increase of the compensation current from one analysis to the next. Also, the sensitivity of the feedback of the indication signal decreases and the compensation current is unstable. It furthermore leads to a slowing down of the reaction rate due to the dilution of the solution and a prolongation of the measurement at the end of titration by a small current which increases the measurement error.

TIP: Practical experience has shown that in the case of transformer oils, the amount of oil that can be determined by a single filling of the titrant is approximately only 120 ml (50 – 150 determinations for one filling), even though the chemical capacity of the titration solution is theoretically higher. This is due to inevitable extraction of active components to the oil layer separated on the electrolyte surface. Despite these circumstances, KF coulometry is the method of choice for determination of water in transformer oils.

TIP: When determining water in oils, it is possible to remove the oil phase from the titration vessel by using a syringe.

IMPORTANT: Use the relevant standards to establish the required accuracy for the measurements.

2.5 Parameters of water determination by Coulometer WTD

Range:	1 ppm to 5 % H ₂ O
Measurement error:	0.5 % at 1 mg H ₂ O
Maximum titration current:	300 mA
Indication current:	1 to 25 µA
Sample weight:	0.02 to 2 g
Results:	µg H ₂ O / g
Titration vessel:	200 mL with or without a diaphragm

2.6 Performing the measurement

2.6.1 Preparation of the measurement

Insert magnetic stir bar into the vessel. Fill the vessel with solution as described in section 2.3 Preparation of the titration vessel. Add electrodes and a closure with capillary as shown in Fig. 4. Settle the titration vessel to the built-in magnetic stirrer and connect the indication electrode and working electrode to the rear panel.

In the following paragraphs, it is described how to control the device using the touchscreen. When the Coulometer WTD is connected to a computer it can equally be driven using Diram Measure software to which the last Chapter of this User Guide has been dedicated.

The main screen displays important information during both compensation and measurement (Figure 5). The graph (a) displays a course of indication voltage (b). Under the graph there are displayed buttons in dependence on the state of the device (c). In the right part, there is an information on current passing through the generation electrode (f), potential of the generation electrode (g), voltage of the indication electrode (h), oven temperature, when connected (i), timer (j), intermediate amount of H_2O (k) and last determined amount of H_2O (l). When the oven is connected, its temperature is shown (i). In front of temperature value might be sign X when the heating is in the course. In front of indication potential (h) there is displayed A for absolute value of indication potential or R for relative value of indication potential. In the case of R value there is displayed a difference between the indication potential and the target value – in compensated state this fluctuates near zero value. When touching the display next to the state icon (e), the stirring can be switched on and off.



Figure 4: Titration vessel prepared for measurement

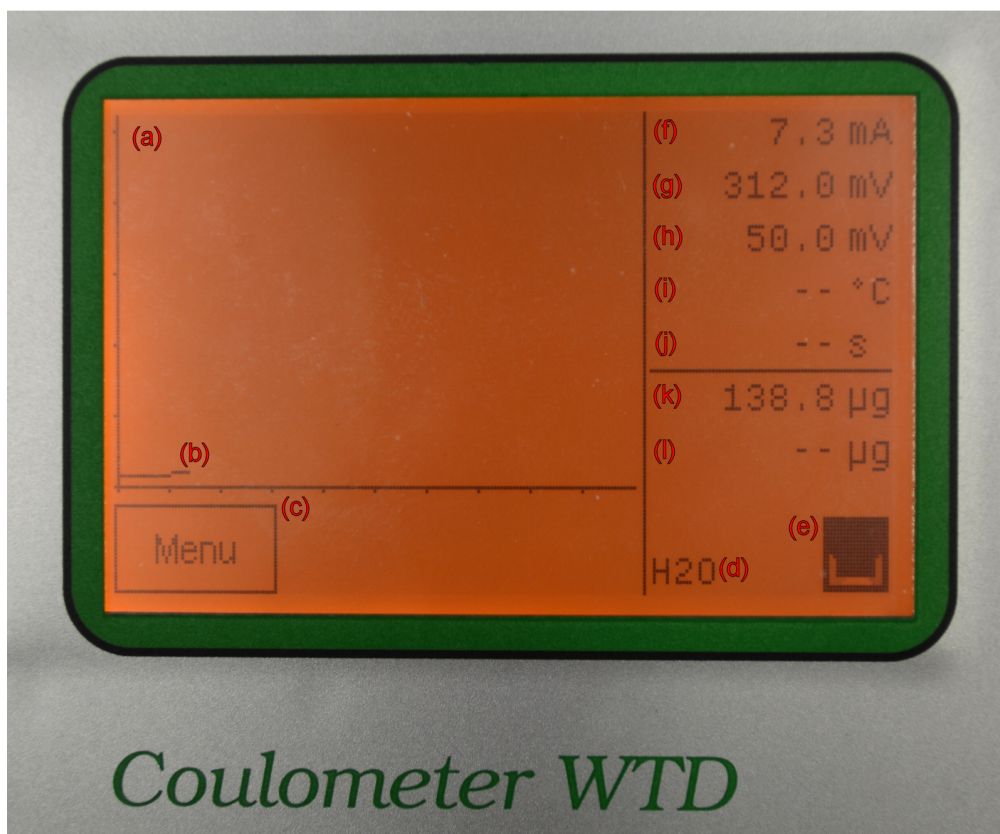


Figure 5: Main screen.

Icon	State description	Buttons available
	Titration. The device titrates toward the target level. The measurement cannot be launched. It is necessary to wait until stabilization occurs.	<i>Menu</i> : The main menu without any restriction.
	Compensation. The device continuously compensates the vessel. The measurement cannot be launched. The stability of the indication signal is evaluated.	<i>Menu</i> : The main menu without any restriction.
	Ready for measurement. The indication signal is stable and the device is prepared for measurement. The measurement can be launched by buttons <i>Start</i> or <i>Timer</i> or directly by a sample insertion.	<p><i>Menu</i>: The main menu without any restriction.</p> <p><i>Start</i>: Manual start of the measurement. The device waits until the sample insertion occurs. When the insertion does not occur the measurement is canceled – the device returns no result.</p> <p><i>Timer</i>: Manual start of the measurement in fixed time interval. The device starts measurement and measures for the given time interval or until the vessel</p>



Icon	State description	Buttons available
		compensation is achieved.
	Countdown. Used in connection with regimes <i>Timer</i> and <i>Start</i> .	<p><i>Menu</i>: the button is visible but inactive. The main menu is not accessible.</p> <p><i>Cancel</i>: The measurement is canceled – no result is issued.</p> <p><i>Finish</i>: If the measurement has been launched by <i>Start</i> button the countdown is stopped and the measurement is launched immediately.</p>
	Measurement. Titration in course.	<p><i>Menu</i>: the button is visible but inactive. The main menu is not accessible.</p> <p><i>Cancel</i>: The measurement is canceled – no result is issued.</p>

Table 1: Device states and buttons.

Connect to power source. Switch on the instrument by the main switch. After switching on, it is first important to select the type of determination. Using *Setup / Device* select a current device. It is possible to switch between: determination of water in a vessel with a diaphragm (H₂O/DF), without a diaphragm (H₂O/BD), or acid number determination (KOH).

When in operation it is always necessary to enable the stirring otherwise an overtitation by iodine occurs. Using *Setup/ Setup/ Stirring speed* adapt the stirring speed. The stirring speed should be high but not too much to avoid mixing of air bubbles into the volume of the electrolyte. During measurement do not modify the stirring rate as it influences the indication current. Use exclusively a Teflon magnetic stir bar.

After switching on, there is generated iodine in the vessel until a small excess, then the titration is stopped and the instrument switches to the compensation regime. If a fresh electrolyte is used, after longer stay or when the filling is partially exhausted, this preparation stage can take a longer time, even a few minutes. On the display there is shown a value of the compensation current. In the beginning the compensation current shows increased values and sometimes a titration is automatically restarted before the full stabilization occurs.

The instrument is ready to measure when in the right down corner there is “ready“ icon displayed. The compensation current may not be in the beginning really constant so it is better to wait for a few minutes. The constant value of the compensation current is particularly important for the distillation method and for the determination in gases. It is convenient to leave the instrument switched on during all the working day. It leads to a better stability of the compensation current. Negative compensation current can be caused by a photochemical reaction. Do not expose the titration vessel to a direct sunlight. **To launch the measurement three different ways can be used: automatic start of the measurement, manual start with the time delay and start of the**

measurement in a predefined time delay. The sample has to be injected into the solution through the septum.

2.6.2 Automatic start of measurement

When Automatic Mode has been selected, the measurement recording is started when the control unit detects a significant signal increase from the indication electrode

2.6.3 Manual start of measurement with time delay

In some situations – samples with low water content - it can be more convenient to start the measurement process manually; simply select [**START**] on the touchscreen of the control unit. Measurement recording will then start after a predefined time-delay, or when a significant change of the indication current is detected. The time delay can be adjusted in control unit's system menu.

2.6.4 Start of measurement in a predefined time delay

Choosing the [**TIMER**] button from the control unit's touchscreen, immediately starts the measurement process and is not halted/finished until a pre-set time has elapsed. This time interval can be changed via the control unit's system menu.

This function is especially useful when measuring solid materials that need time to dissolve.

During the measurement process, the time to finish the analysis is displayed on the screen of the control unit.

2.6.5 Finishing the measurement

The control unit will automatically stop measurement recording once the reaching the initial value of the indication current.

The measurement recording can be stopped at any time by pressing the [**STOP**] button on the touchscreen or via the PC interface software.

NOTE: The current measurement is discarded if the system is manually halted via [**STOP**].

2.6.6 Sample number and weight input

At the end of the analysis, the measured water content is displayed, on the control unit's touchscreen, in the units of $\mu\text{g H}_2\text{O}$.

Depending on the particular setup for the analysis, it may be necessary to insert or confirm the Sample Number and the Sample Weight. When required they can be entered directly into the control unit via the touchscreen; note the sample weight is expected as a value of *grams*. After insertion of Sample Weight the concentration of water in sample is immediately displayed in a pre-defined form ($\mu\text{g H}_2\text{O}$ / g of sample, ppm, etc.).

After the measurement analysis is finished it is necessary to press [**CANCEL**] or [**SAVE**].

IMPORTANT: [**CANCEL**] will clear and erase all results from the current analysis

When [**SAVE**] is chosen the results are stored in the internal memory of the control unit and, if connected, transmitted to a computer and saved in a result data base. .

2.6.7 Displaying the previous measurement result

The [**LAST RESULT**] button will display the last valid recorded measurement.

2.7 Touchscreen options

The main parameters of the device can be controlled from the **Menu** in the main screen.

- **Sample number**
Sample number can be inserted both before and after the measurement.
- **Sample weight**
Sample weight can be inserted both before and after the measurement.
- **Last result**
Displays the result of a last successfully finished measurement. Button *Erase* removes the last result from the memory.
- **Setup / Setup / Stirring speed**
Adapt the stirring speed.
- **Setup / Setup / Back-light**
Adapt the back-light of the display.
- **Setup / Setup / Result unit**
Select a unit used for the result.
- **Setup / Setup / Language**
Select a language. The restart is demanded.
- **Setup / Setup / Indication signal**
The indication signal can be displayed either in its absolute (Abs) value or relatively to the target level (Rel).
- **Setup / Constants / Waiting for sample insertion**
Insert a number of seconds for which the device awaits after pressing button *Start* before launching the measurement.
- **Setup / Constants / Measurement in constant time**
Insert a number of seconds during which a measurement will take place. The measurement is finished after the preset time elapses or later when the signal reaches the zero level. The measurement in a fixed time interval is launched by pressing *Timer* button. The default is 600 s.
- **Setup / Constants / Indication signal offset**
It is possible to set a shift of the indication signal. The change is made in the electrical circuit.

- ***Setup / Constants / Set-point***

Here an absolute value of indication signal at which the compensation occurs can be set. The change is made in the software.

- ***Setup / Device***

Selection of the device. It is possible to switch between: determination of water in a vessel with a diaphragm (H₂O/DF), without a diaphragm (H₂O/BD), or acid number determination (KOH).

- ***Setup / CpCAN / Oven / Function enabled***

CpCAN menu appears only when drying oven is connected. Option “Yes” is required to perform measurements with the drying oven.

- ***Setup / CpCAN / Oven / Heating mode***

“Auto” is predefined mode for performing measurement with the drying oven. “On” switches on the heating (the red light on the front of the oven lights up) and “Off” switches off the heating.

- ***Setup / CpCAN / Oven / Heating mode / Temperature T1***

Allows for setting the preheating temperature. Default is 220°C.

- ***Setup / CpCAN / Oven / Heating mode / Temperature T2***

Allows for setting the desired temperature of the oven. Default is 150°C.

- ***Setup / CpCAN / Oven / Heating mode / T1 time***

Defines preheating time. Default preheating time is 200 s.

2.8 Drying oven

2.8.1 Use of the drying oven

When analysing substances that are not soluble or are reactive with the iodine titrant (i.e. additive motor oils), the direct water titration is generally not suitable, but can sometimes still be conducted in combination with a drying oven. The sample for analysis is heated in the drying oven to release all water present within the sample. The resultant vapour is transferred via a dry carrier gas into the titration vessel.

2.8.2 Description of the drying oven

The drying oven is shown in Fig. 6. Dry carrier gas flows by a blue inlet (1) to a flowmeter with a flow regulator (2), inlet PTFE hose (3) to a glass inlet (4) of a glass tube (5) embedded in a heating chamber (6). The glass tube is equipped with a small screw cap with a septum for sample insertion (7). The carrier gas with released moisture then flows through outlet (8) via PTFE outlet hose (9) to a special gas inlet (10) of the titration vessel. On the top of the generation electrode there is a PTFE exhaust hose (11) leading the gases outgoing from the titration vessel. In the front of the oven there is a green power connection indicator (12) and a red heating indicator (13). On rear panel there is a main switch, a power socket and a connector for connection with the Coulometer WTD instrument.

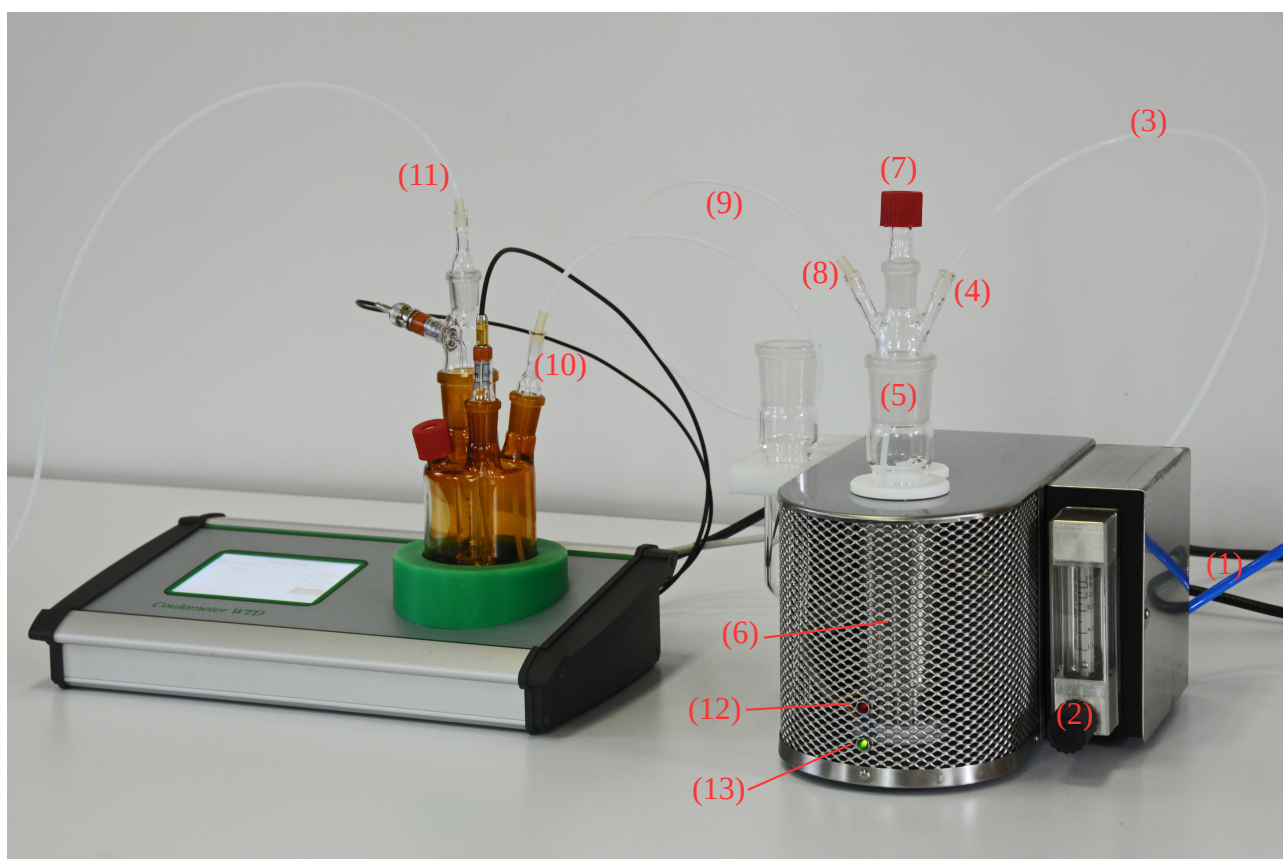


Figure 6: Drying oven

2.8.3 Assembly

Open the metal cover on the right side of the oven. The blue inlet hose is intended to bring the dry gas from the gas cylinder. Attach one end of the blue hose to the gas source and the other to the flowmeter as shown in Figure 7. (For disconnection press the yellow ring and drag the hose out.)

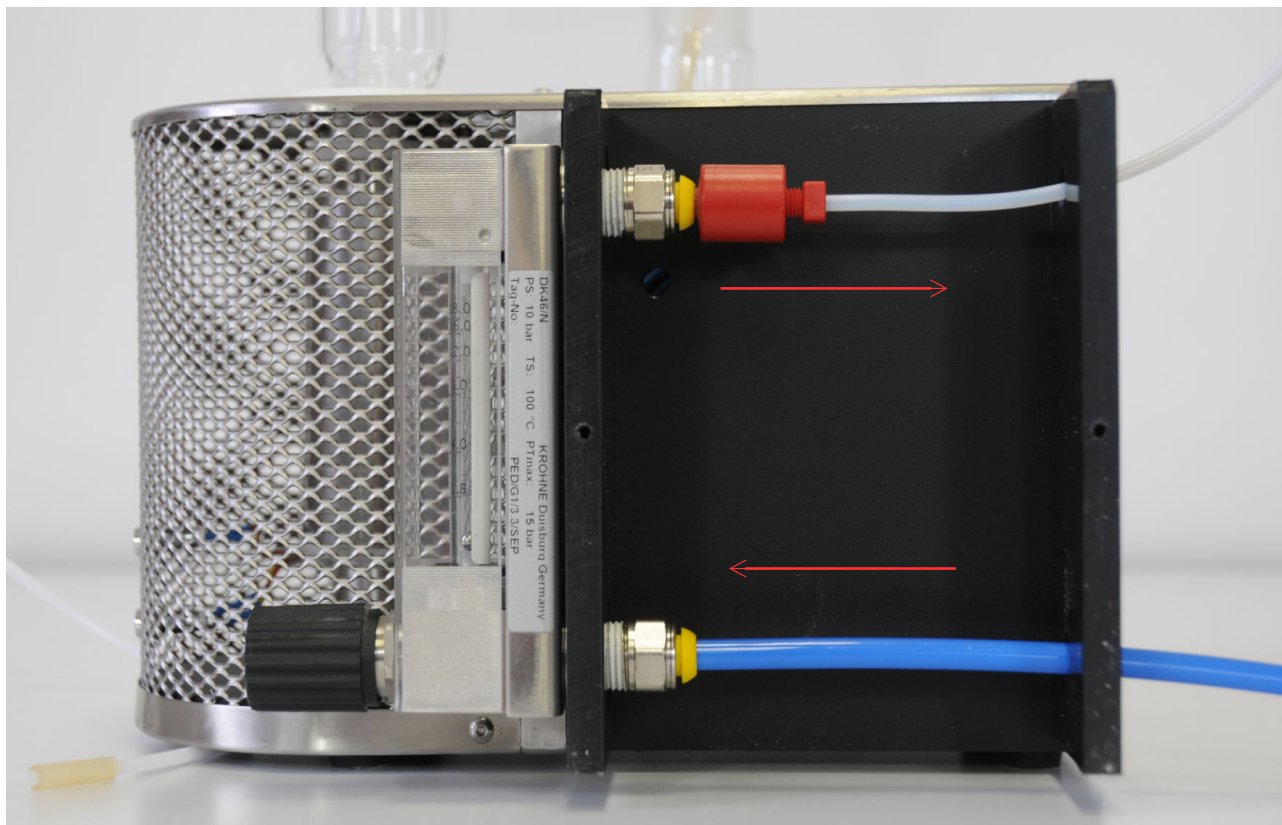


Figure 7: Connection of the hoses to the flowmeter. The red arrows show the direction of the gas flow.

Attach a PTFE hose to the red holder at the flowmeter outlet as in Figure 7. The detail of the PTFE hose attachment is depicted in Figure 8. It is important to include a piece of a soft hose in front of the screw fitting to ensure a seal before screwing.

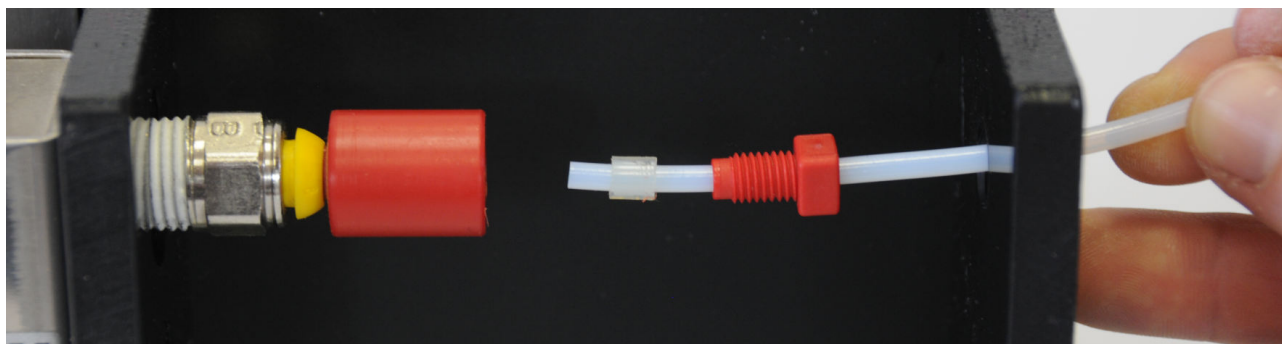


Figure 8: Detail of a connection between PTFE hose and the red holder



Figure 9: Detail of glass parts

Assemble the glass parts and the hoses as in Fig.9. The PTFE hoses are accommodated in the glass necks by pieces of soft hoses in order to make a seal. The inlet hose leading the carrier gas from the flowmeter to the glass tube should lead to the bottom of the glass tube so that after sample insertion, the gas can bubble through the sample. The outlet hose is above the sample level. On the other side, the outlet hose is coming to the titration vessel of Coulometer WTD through the special glass part with the ground joint and is immersed in the titration electrolyte (as in Fig.6). Adjust the PTFE hose in the glass part by a piece of a soft hose. Add the exhaust hose of the titration vessel to the top of the generation electrode using the special glass part with ground joint and a piece of soft hose to seal. Lead the exhaust hose to a hood.

2.8.4 Preparation of the measurement with the drying oven

Prepare a four-neck titration vessel of Coulometer WTD with the special gas inlet (as in Fig. 6), equipped with the indication electrode, generation electrode without a diaphragm, screw cap with a septum and a teflon magnetic stirring bar inside.

IMPORTANT: Always use a titration vessel without a diaphragm when measuring with drying oven and select **Menu / Setup / Device** H2O/BD (w/o diaphragm) mode on the touchscreen. (Measurement with a diaphragm is inaccurate due to evaporation of the titration medium and changes in titration medium level with respect to the cathodic part of the titration vessel.)

Fill the titration vessel with a suitable electrolyte as described in section 2.3.1. Use electrolyte without volatile components intended for use with oven. Connect the titration vessel with the drying oven by the outlet hose of the drying oven (Fig. 6, (9)). The outlet hose should be immersed in the electrolyte of the titration vessel. Connect cables of indication and generation electrodes of

the Coulometer WTD and switch on the Coulometer WTD. Leave the titration vessel to equilibrate as described in the section 2.6.1.

In the meantime connect the drying oven by a connecting cable to the Coulometer WTD, switch on the Coulometer WTD - on the touchscreen of the Coulometer WTD appears a letter H. Connect the drying oven to the power supply – the green control on the front of the oven lights up.

After the equilibration of the titration vessel without the gas flow, turn on the carrier gas and adjust the flow. Allow the titration vessel to equilibrate again until the generation current is low enough and the signal “ready for measurement” appears. This can take several minutes.

Run one heating cycle by pressing [**TIMER**] button on the touchscreen in order to remove water adsorbed in the system. The red control lights up and temperature is indicated on the touchscreen. The default heating cycle duration is 600 s.

2.8.5 Setting of the heating cycle

To enable a measurement with the pre-defined heating cycle it is necessary to switch to:

Setup / CpCAN / Oven / Heating mode “Auto”.

The duration of the heating cycle can be adjusted from the touchscreen in the Setup menu:

Setup / Constants / Measurement in constant time The default value is 600 s.

When the measurement is started by pressing the [**TIMER**] button, the oven is heated by maximum power until temperature **T1** is reached at the heating element. **T1** is then maintained for a time period **T1 time**. Temperature **T1** can be higher than the required sample temperature as the onset of temperature at the sample is slower. **T1** and **T1 time** has to be selected depending on the sample amount in order to quickly reach the required sample temperature. When **T1 time** elapses, temperature **T2** is maintained until the end of measurement.

Temperatures **T1**, **T2** and **T1 time** can be adjusted from the touchscreen:

Setup / CpCAN / Oven / Heating mode / Temperature T1 Default is 220°C.

Setup / CpCAN / Oven / Heating mode / Temperature T2 Default is 150°C.

Setup / CpCAN / Oven / Heating mode / T1 time Default is 200 s.

Temperatures **T1** and **T2** are measured and maintained at the heating element not at the sample. For a 10 mL oil sample the default parameters: **Constant time** = 600 s, **T1**=220°C, **T2**=150°C and **T1 time**= 200 s lead to a satisfactory sample temperature 130-140°C during the measurement and ensured complete water transfer to the titration vessel. When different measurement parameters are required it is recommended to check the temperature profile in sample by an independent thermometer.

2.8.6 Measurement with the drying oven

Prepare measurement according to 2.8.4. Preparation of the measurement with the drying oven.

Allow the titration vessel to equilibrate.

Press [**TIMER**] button.

Immediately insert sample to the glass tube through the septum. The measurement is finished after preset time (default 600 s) or later after the indication signal reaches its original level.

Insert sample weight.

Read result.

2.8.7 Notes

Measurement can be finished before the end by pressing [**FINISH**] button. This option might be advantageous when indication signal returns to the original (zero) value before the preset time elapses.

Autostart option is not allowed when the oven is connected.

When **Menu / Setup / Device** H₂O/DF (with diaphragm) is erroneously selected, the oven does not start heating.

3 Diram Measure Software

Diram Measure software is a complex system developed to control all devices produced by Diram company. The application consists of several modules where each device is driven by a specific module independent of other modules. After connection by USB connector, the device is automatically recognized and a the program is prepared for device control, parameters settings and for measurement.

The **Coulometer WTD** has been developed as independent of PC it can however be connected to computer and controlled by **Diram Measure** software. In this mode, a broader range of setting options can be used.

3.1 System requirements

Diram measure software can work in any computer under operation system Microsoft Windows XP and higher in both 32 and 64 version. Requirements for operation memory are not important. The installation requires 30 MB space on hard-disk.

3.2 Installation and uninstalling

The installation is done by running the installation file. For proper installation administrator rights are required. During the installation also a USB driver is installed common for all Diram devices.

Uninstalling is done either by option Uninstall in the menu Start or by a standard way by the operation system. All program files are deleted. Data files and settings and the USB driver are kept and can be erased by user.

3.3 Main screen

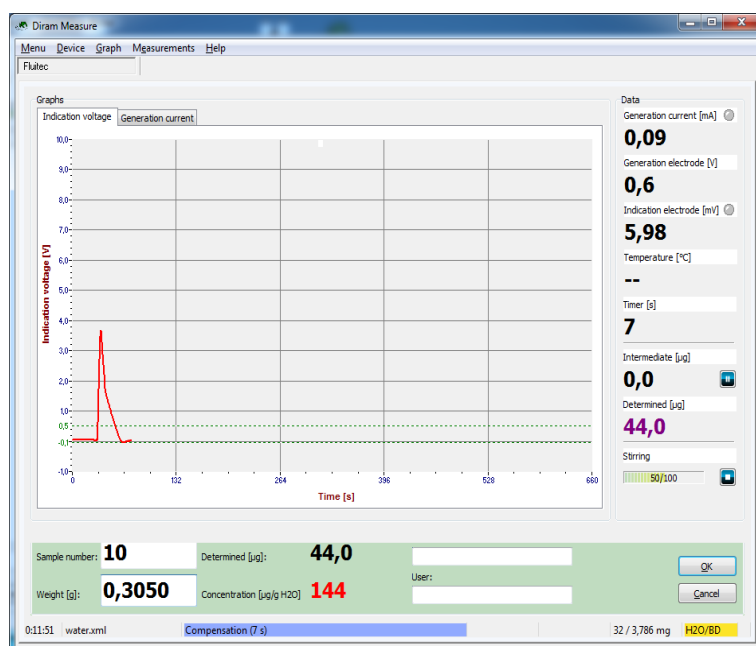


Figure 10: Main screen

The main screen displays information equivalent to the main screen of the Coulometer WTD touchscreen. The graph displays indication potential or generation current time dependency. The couple of green lines defines a ready interval – a range of indication voltage in which the indication signal should be to allow the start of measurement.

In the right side there are displayed current values of generation current, indication potential, temperature of the drying oven when connected and measured values of water content. The bar serves for a magnetic stirrer control.

The stirring is switched on and off by button *Start/ Stop* on the right side of the speed bar. The stirring speed is controlled both by text and graphical way. The stirring speed can be modified by clicking on the speed bar. Left mouse button click on the speed bar +*Ctrl* enables to insert the stirring speed as a number. During measurement the speed modification is disabled by default.

In the bottom part of the main screen there are fields where sample number and sample weight can be inserted. The values are used for the next measurement. The same specification can be used for further measurements or cleared depending on the settings. When the measurement is completed there is displayed a green form with results where the sample number and weight can be modified again. The result is displayed in red color. The data are sent to the file by **OK** or canceled by **Cancel** button. Until selecting **OK** or **Cancel** the next measurement is blocked.

In the status line on the bottom of the main menu is indicated time elapsed from the start of the module, path to the data file, actual state of the device (Measurement, Compensation etc.), the total determined value from the last zeroing and indication of the device.

3.4 Common menus

Common menus for all devices are **Menu** and **Help**. **Menu** enables to select a language and start program modules independent of a device connected. **Help** displays a dialog box with program version.

3.4.1 Menu / Plug-ins / Data files management

This self-standing module can be opened either by *Menu / Plug-ins / Data files management* or *Measurements / Show data*. It enables to open any data file created by **Diram Measure** program and export it in formats: Microsoft Excel 97, Microsoft Excel 2000 and higher, Open Text Document for OpenOffice or LibreOffice.

3.5 Menu Device

3.5.1 Device / Settings...

Selecting this item opens a dialog which allows for a complete setting of the measurement parameters.

3.5.2 Device / Settings.../ Application

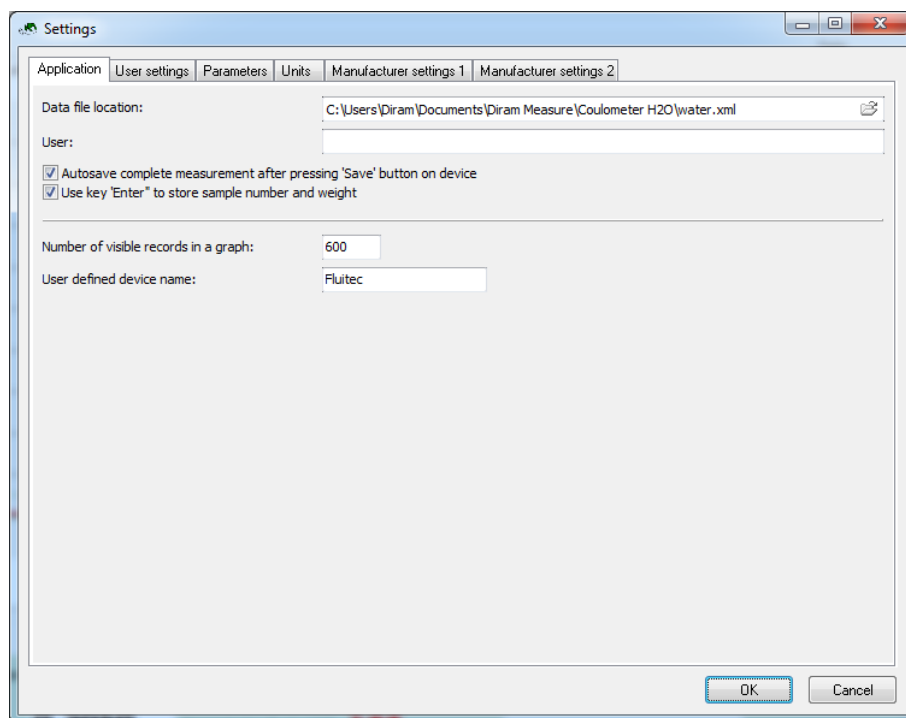


Figure 11: Application

Data file location gives a full path to the file where the measurement results are saved.

User name is automatically saved to the results.

When **Autosave complete measurement after pressing Save button** is checked the result is saved

immediately after **Save** is pressed. Otherwise a dialog opens where sample weight and number can be edited and the result can be saved or rejected.

Number of visible records in the graph gives a number of points displayed in the x-axis of the graph. The higher the number is the longer time window is displayed before it is scrolled.

User defined device name serves for user distinction of the device *i.e.* H2O lab 1.

3.5.3 Device / Settings.../ User settings

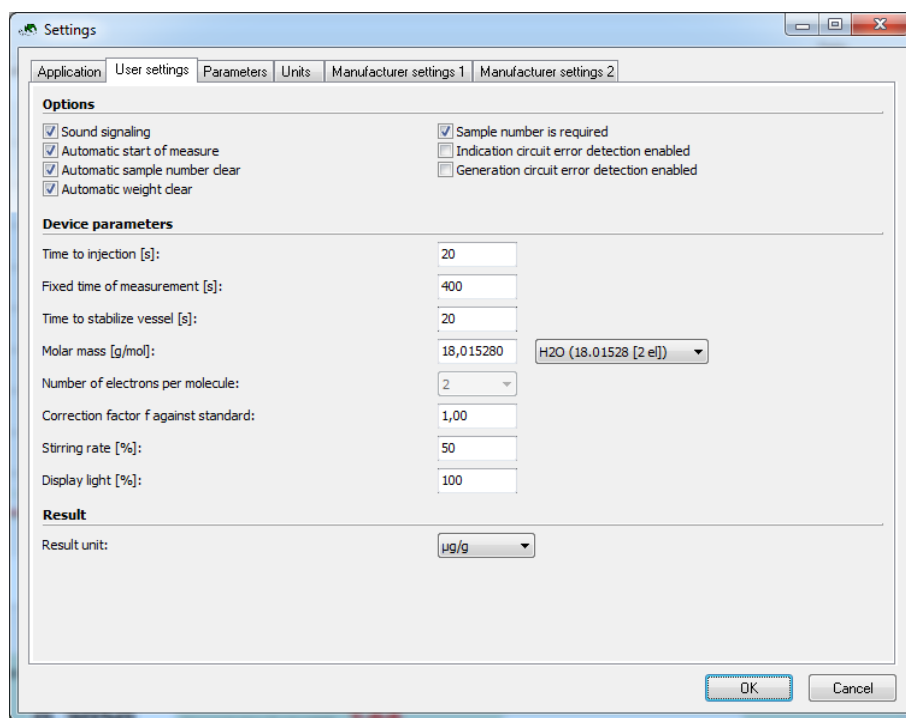


Figure 12: User settings

In the group **Options** there are several check-boxes. It is possible to switch on/off the sound signalization of the device; enable/disable automatic start of measurement; enable/ disable automatic clearing of the sample number after saving of the measurement; enable/ disable automatic clearing of the sample weight after saving of the measurement; enable/ disable requirement of sample number; enable/ disable detection of indication circuit error (usually disconnection of the circuit); enable/ disable detection of generation circuit error.

Time to injection defines time for which runs a countdown after the manual start of measurement by button **Start**. When an important change of indication voltage occurs before the defined time elapses, the measurement is automatically started.

Fixed time measurement defines a time for which the device performs measurement after pressing **Timer** button in the main screen. The measurement is finished regardless the end point is reached or not.

Time to vessel stabilization defines a time for which the indication signal is followed after it enters

the preparation zone. If the value of the indication signal stays within the preparation zone during all the stabilization time, the measurement can be allowed.

Molar weight and **Number of electrons per molecule** are parameters used for the calculation of the resulting mass.

Stirring speed and **Display back light** define default values set after switching on the device.

Result unit defines a displayed unit.

3.5.4 Device / Settings.../ Parameters

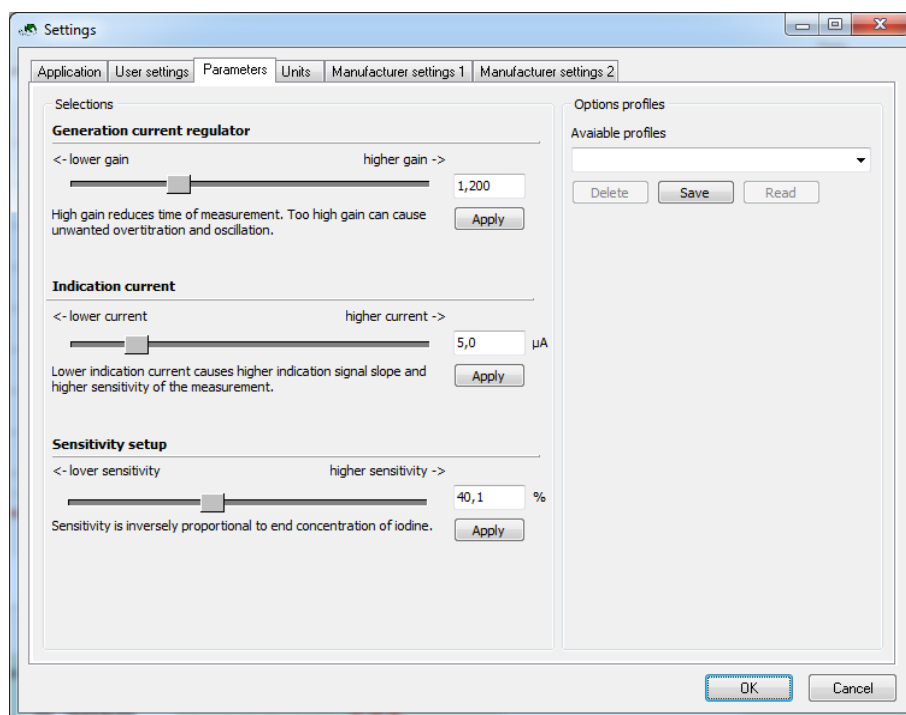


Figure 13: Parameters

Generation current regulator sets the value of proportional amplification. The generation current is given as a proportional amplification times the difference between the current value of indication signal and the end point.

Indication current determines the slope of the indication signal and influences the measurement sensitivity.

Sensitivity setup: the sensitivity is inversely proportional to the iodine end concentration.

The values of the regulation parameters should be determined empirically. The setup can be saved in a user defined profile for later use.

3.5.5 Device / Settings.../ Units

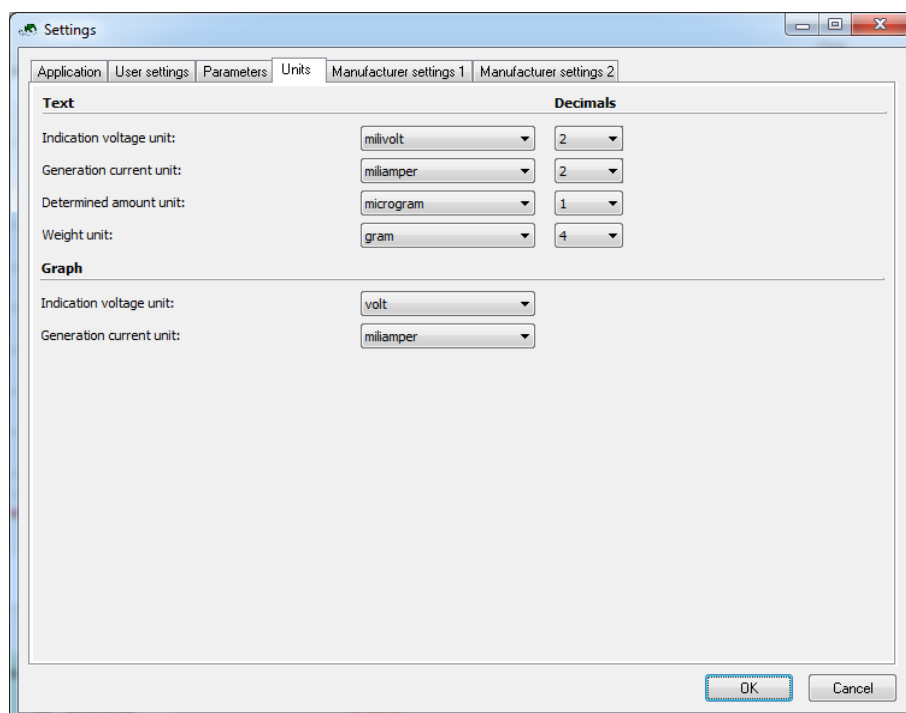


Figure 14: Units

The dialog enables for selection of units.

3.5.6 Device / Stirring speed

The stirring speed in percents of maximal value.

3.5.7 Device / Clear intermediate result

Zeroes the intermediate result of the measurement.

3.5.8 Device / Clear determined amount

Zeroes the determined amount.

3.5.9 Device / Unblock new measurement

Enables to preform a new measurement.

3.5.10 Device / End point

This dialog enables to define a titration end point as a level of indication voltage. The newly entered value is used immediately.

3.5.11 Device / Device selection

Select a current device. It is possible to switch between: determination of water in a vessel with a diaphragm (H₂O/DF), without a diaphragm (H₂O/BD), or acid number determination (KOH).

3.6 Menu Graph

Enables to manage measurement results in the graphical form.

3.6.1 Graph / Show data...

Opens a dialog with Indication voltage and Generation current history from the last switch on of the device. It enables to display even the data which are already not visible in the main graph.

3.6.2 Graph / Save to file...

The data in the graph are exported as a table in Microsoft Excel format.

3.6.3 Graph / Clear data

Deletes all data in the graph.

3.6.4 Graph / Center, Center all

Sets the y-axis of the graph to the default position for current graph or all graphs.

3.7 Menu Measurements

Starts a self-standing program module which enables a management of a current data file, printing data, export.... To return to the measurement mode select a bookmark with the device user name.