

AUTOMATIC COULOMETRIC MOISTURE TITRATOR
PE-9210

Data Sheet
Operation Manual
БКРЕ.414315.002ПС

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1. General Information

This data sheet combined with the operation manual has been designed to provide general information on the construction and operation rules of automatic coulometric moisture titrator PE-9210 (hereafter referred to as “the titrator”, “the instrument”, and “the product”).

As works on the instrument modernisation are still going on, small changes which do not make technical characteristics of the product worse can be made in the design.

2. Application

Automatic coulometric moisture titrator PE-9210 is used to measure moisture content in substances which do not interact with Karl Fischer reagent in laboratories of industrial installations, research institutions, control authorities, educational institutions, and etc.

The following functions are performed in the instrument:

- Measurement of moisture content;
- Numerical indication of moisture content and concentration;
- Calculation of moisture concentration in the sample and statistical analysis of the results of the parallel measurement series;
- Determination of the bromine number (index) as per ASTM D1492-08¹;
- Creation and retention of the user measurement methods;
- Retention of measurement results and results of the measurement statistical analysis;
- Transfer of measurement results to the printer or computer;
- Control of auxiliary external devices.

3. Basic Information and Technical Data

3.1. Information on the Manufacturer

Automatic coulometric moisture titrator PE-9210 has been manufactured by ECROSKHIM Co. Ltd.

Legal address: 21 Koltsov st., lit. A. office 17H, 194214, Saint-Petersburg

Post address: 22 17 liniya V.O, build. И, office 406, 199178, Saint-Petersburg

Telephone/Fax: +7 (812) 322-9600, 449-3122, 449-3123

E-mail: info@ecohim.ru, URL: <http://www.ecohim.ru>

3.2. Certification information

Titration PE-9210 has a certificate of conformity for measuring equipment type RU.C.31.001.A No. 52607 issued by the Federal Agency on Technical Regulation and Metrology on October 4, 2013 and is registered with the State Register of measuring equipment under No. 55135-13. The certificate is valid till October 4, 2018.



¹ A special titration cell is required.

3.3. Technical Data

Table 1

Name of Parameter	Parameter Value
Titration method	Karl Fischer coulometric titration
Iodine generation	Pulsing, direct current
Endpoint detection	Alternating current polarised potential
Drift compensation	Automatic and manual modes
Stirring method	Magnetic rotor, 100÷1000 rpm
Net volume of a standard titration cell, ml	100÷150
Sensitivity, µg H ₂ O	0.1
Water weight measurement range in the sample, mg	from 0.01 to 200
Relative tolerance, %	±3.0
Relative RMSD limit of uncertainty, %	1.5
Power supply voltage, V	220
Supply voltage frequency, Hz	50
Electrical capacity, W, max.	170
Dimensions of the main unit: <ul style="list-style-type: none">• Height, mm, max.• Length, mm, max.• Width, mm, max.	<div>200</div> <div>350</div> <div>275</div>
Titration weight, kg, max.	6.5
Service lifetime, years	10
Mean time to failure, hours	10000
Signaling functions	Electrolysis current, titration result, re-titration, anolyte life time, catholyte life time, closing or break of the indicator electrode circuit, parameter input, real time clock problem.
Interfaces	<ul style="list-style-type: none">• 2 x USB A for peripheral devices connecting• 1 x RS-232C for connecting of balance• RS-485 for connecting of special external devices• 1 x Ethernet for connecting to PC

4. Operation conditions

The device has been designed for operation in laboratory conditions. It must be installed on the firm horizontal base in the place where there is no vibration, direct sun beams, and strong electrical and magnetic fields.

Main operation parameters:

- Ambient temperature, °C..... 15÷30;

- Relative humidity, % 20÷85;
- Power supply voltage, V..... 220±22;
- Supply voltage frequency, Hz 50±1.

5. Content of Precious Metals

Table 2

Material	Content
Gold	no
Silver	no
Platinum	4.6 g
Iridium	no
Rhodium	no
Palladium	no
Ruthenium	no
Osmium	no
Diamond	no

6. Scope of Supply

- Titrator PE-9210 (main unit).....1 pc.
- Titration cell1 pc.
- Power cable1 pc.
- SPTA set (formed upon request)1 pc.
- Data Sheet and Operation Manual with Annex “МП-242-1493-2013 Verification Procedure” and a primary verification note with the verification officer’s stamp impression1 pc.

7. Design and Operation Procedure

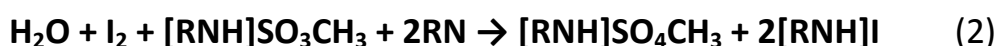
7.1. Measurement Principles

7.1.1. Moisture Titration

Automatic coulometric moisture titrator PE-9210 is an instrument which measures the moisture content in samples of substances based on Karl Fischer’s method. The core of the method is that the sulfur dioxide reacts with the alcohol to form an ester that is subsequently neutralized by the base:



The titration of water constitutes the oxidation of the alkyl-sulfite anion to alkyl sulfate by the iodine:



In the instrument iodine is generated electrochemically from anolyte solution which contains iodide-ions:



In the result of the chemical reaction (2) iodine combines with moisture contained in the sample. A small excess amount of unused iodine is registered by the indicator electrode; when this happens electrochemical desorbing of iodine stops.

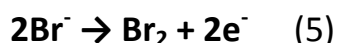
Electricity charge coming through the anolyte solution from Faraday's law is in proportion to quantity of used iodine. According to equation (2) iodine and water interact in ratio one to one. 1 mole of water is equal to 18.01448 grams and equivalent to 193000 coulombs; it is in compliance with the fact that 10.713604 coulombs are spent for 10 mg of water. The instrument determines moisture content in the substance while calculating charge of electricity passing through during electrolysis.

7.1.2. Bromine number determination

While coulometric determination of a bromine number electrochemically generated bromine joints to olefinic links of organic compounds:



Free bromine is formed on the anode of a generating pair of electrodes:



A bromine number [**1 g of bromine/100 g of a sample**] indicates the quantity of bromine used for the reaction in compliance with equation (4) and is calculated on the basis of charge of electricity consumed for bromine generation, analogical to calculation for Karl Fischer method.

7.2. Titration Cell

For titration procedure the instrument includes a titration cell which in general can be of two types: a cell without diaphragm (Figure 1) and a cell with diaphragm (Figure 2). Accordingly hereafter referred to as type 1 cell and type 2 cell.

Type 1 cell has 1 chamber. Anodic and cathodic reactions take place in the same electrolyte; that is why when using it you should apply a suitable Fischer reagent, so that a cathodic reduction reaction would not generate products which can oxidize at the anode which results in overestimation of the results.

Type 2 cell consists of an anode compartment where Karl Fischer reaction takes place and a cathode compartment where a complementary cathodic reduction reaction takes place. The anode compartment is filled with anolyte solution; the cathode compartment is filled with catholyte solution. The compartments are divided with a diaphragm which prevents two solutions from being mixed.

Simple iodine is generated in the anode compartment of the cell and reacts with other components of anolyte solution and binds water contained in the sample. So the anolyte solution is a reagent which provides anodic oxidation of iodide-anion into simple iodine with current output 100%.

In the cathode compartment a complementary cathodic reduction reaction takes place which is provided by catholyte solution. As a diaphragm which divides two compartments

cannot prevent active components from diffusion completely, the catholyte components should be compatible with anolyte solution.

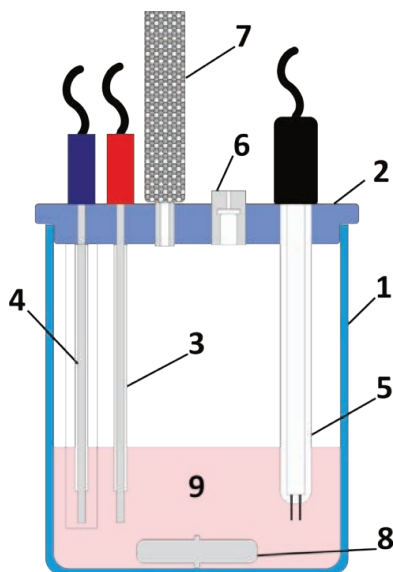


Figure 1

1 – cell; 2 – cover; 3 – anode; 4 – cathode; 5 – indicator electrode; 6 – sample inlet port; 7 – desiccant tube; 8 – magnetic rotor; 9 – anolyte.

In both types of cells while a reduction reaction is taking place on the cathode, hydrogen gas evolves which should be carried off from the cell in such a way to prevent moisture from coming back from atmosphere. For that purpose a desiccant tube filled as a rule with mole sieves is used. Sometimes a layer of indicating silica gel is also used to determine when it will be necessary to regenerate the desiccant.

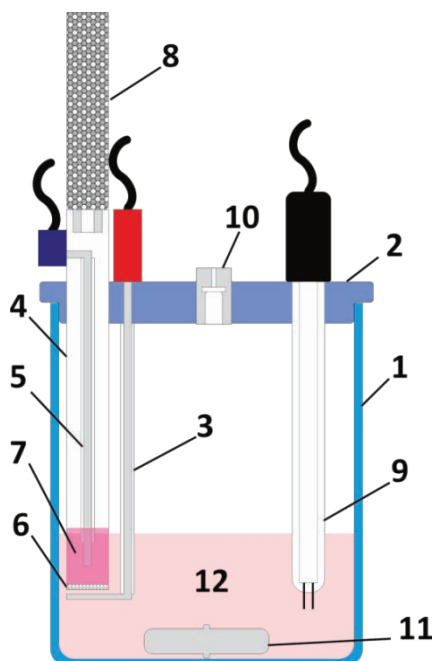


Figure 2

1 – cell; 2 – cover; 3 – anode; 4 – cathode chamber; 5 – cathode; 6 – diaphragm; 7 – catholyte; 8 – desiccant tube; 9 – indicator electrode; 10 – sample inlet port; 11 – magnetic rotor; 12 – anolyte.

7.3. Operation Algorithm of the Instrument

7.3.1. Working Procedure

The main element of the operation algorithm of the titrator is automatic control system (ACS) which has been built on the adaptive digital controller implemented in software and control modules of the indicator system and the generator system of the instrument hardware (Figure 3).

The ACS is aimed to maintain constantly a certain small excess quantity of free iodine in anolyte solution with a help of electrochemical generation of iodine to bind moisture coming to the cell.

The control module of the indication system (**D**) provides current necessary for polarization of the double-pin platinum indication system (detection electrode) and measures polarised potential (U_i), which reflects simple iodine content in anolyte solution.

The measured value is compared with the polarised potential value U_0 set in the instrument software (base line) which corresponds to a certain excess quantity of free iodine.

The received discrepancy (ΔU) is processed by the adaptive regulator (**AR**) which forms a control action for the input of the control module of the generator system (**G**); in the result it lets current impulses (I_g) necessary for electrochemical generation of a required quantity of iodine in anolyte through generator electrodes.

If there is an excess amount of iodine a control action is not formed, iodine is not generated, and its excess amount is bound with water coming from the atmosphere due to imperfect sealing capacity of the cell (cell drift).

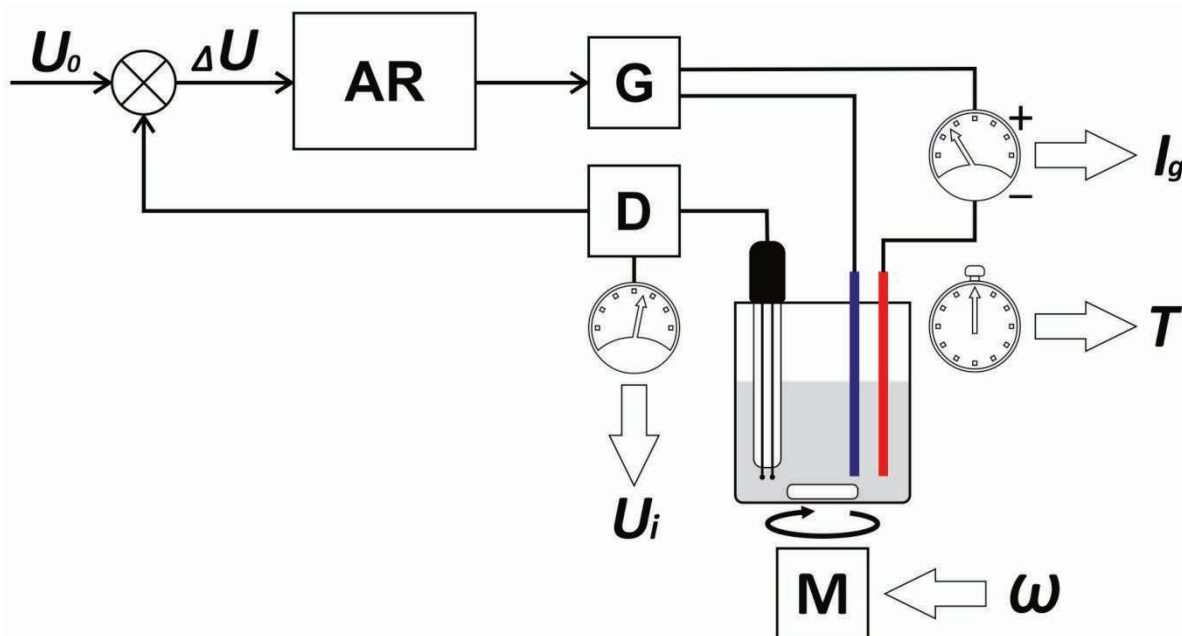


Figure 3

7.3.2. Preliminary Titration

When the titrator is switched on the above-mentioned ACS does not work, only the indicator system is on and the polarized potential (U_i) is measured. In the initial state there is

moisture excess in the cell, that is why the indicator system is in the state of saturation, and the greatest possible value U_i is displayed.

Moisture excess must be bound before measurement procedure. For that purpose the preliminary titration mode (Pre-titration) is switched on. In that mode a control system is switched on, and the iodine generation procedure starts; when it happens, current I_g passing through generator electrodes per a time unit is continuously measured. This value stated in micrograms of water per second is usually called a baseline drift or just drift and displayed in the instrument as D_0 ($\mu\text{g/s}$).

As far as water is bound the indicator system potential U_i is coming back step by step to the baseline (U_0), and the drift value D_0 is decreasing quickly.

It is considered that when the drift value D_0 is about $20 \mu\text{g/min}$, it is already possible to take evaluating measurements which do not require high accuracy. In this state the message "READY" is displayed in the instrument.

The value equal to D_0 about $10 \mu\text{g/min}$ is considered to be a natural drift for a standard cell. In this state the message "STABLE" is displayed in the instrument, and when it is reached, measurement accuracy specified in metrological characteristics of the instrument is provided.

7.3.3. Titration

When the "START" button is pressed just before injection of the sample to the titration cell the instrument memorizes the current value D_0 . It is suggested that it reflects intensity of moisture coming to the cell from outside which remains unchanged during measurement time.

So, if you multiply this value by the measurement time, you will receive amount of moisture coming not from the sample injected and can subtract it from the total titration result.

At this moment the ACS is also switched off, iodine generation is stopped, and titration time starts to be recorded.

Immediately after the sample is injected and the "ENTER" button is pressed, as a rule a certain time is given to let the sample be mixed with solution. When this time is over the ACS is on again, and iodine generation procedure, titration current measurement I_g and consumed electricity calculation begin.

The system works in the same way as when water excess is bound in the pre-titration mode, but in this mode titration time is measured and electricity consumed for iodine generation procedure is calculated.

The following events can serve as a criterion of titration end (adjusted parameter):

- When the titration system reaches the drift level D set as an absolute value D_{abs} , or in relation to the initial value D_0 ($D_0 + \Delta_{abs}$);
- When the maximal set titration time T_{max} is reached.

A classical criterion for reset of the indicator system potential U_i to the initial value U_0 is not applied, as it does not provide necessary accuracy at the high titration speed. It happens because of response of electrochemical processes which results in the following: when necessary iodine excess is generated, and the indicator system potential U_0 is reached, mois-

ture can still exude from the sample and enter to Karl Fischer reaction, but because of quick compensation of consumed free iodine by the control system there will be no changes of the indicator system potential, and the drift level will remain high.

Titration procedure takes place till the criterion of titration end is reached. When the criterion is reached, titration time and charge of electricity consumed for titration are fixed for the result calculation.

Then the system continues working in the pre-titration mode, and soon the drift level **D** is reached again which enables the next titration cycle.

7.4. Design

Titration PE-9210 (Figure 4) is a stationary table laboratory instrument which consists of electronic and electromechanical units installed in single housing 1 and titration cell 2 placed on the special area on the top of the housing. Colored touch screen 3 which provides functions of input and output of necessary information is located on the front sloping panel of the titrator. On the back panel of the instrument there are ports for connecting of a power cord 4 with a built-in fuse holder 5, mains switch 6 and connectors for connecting the titration cell electrodes 7 and external devices 8.

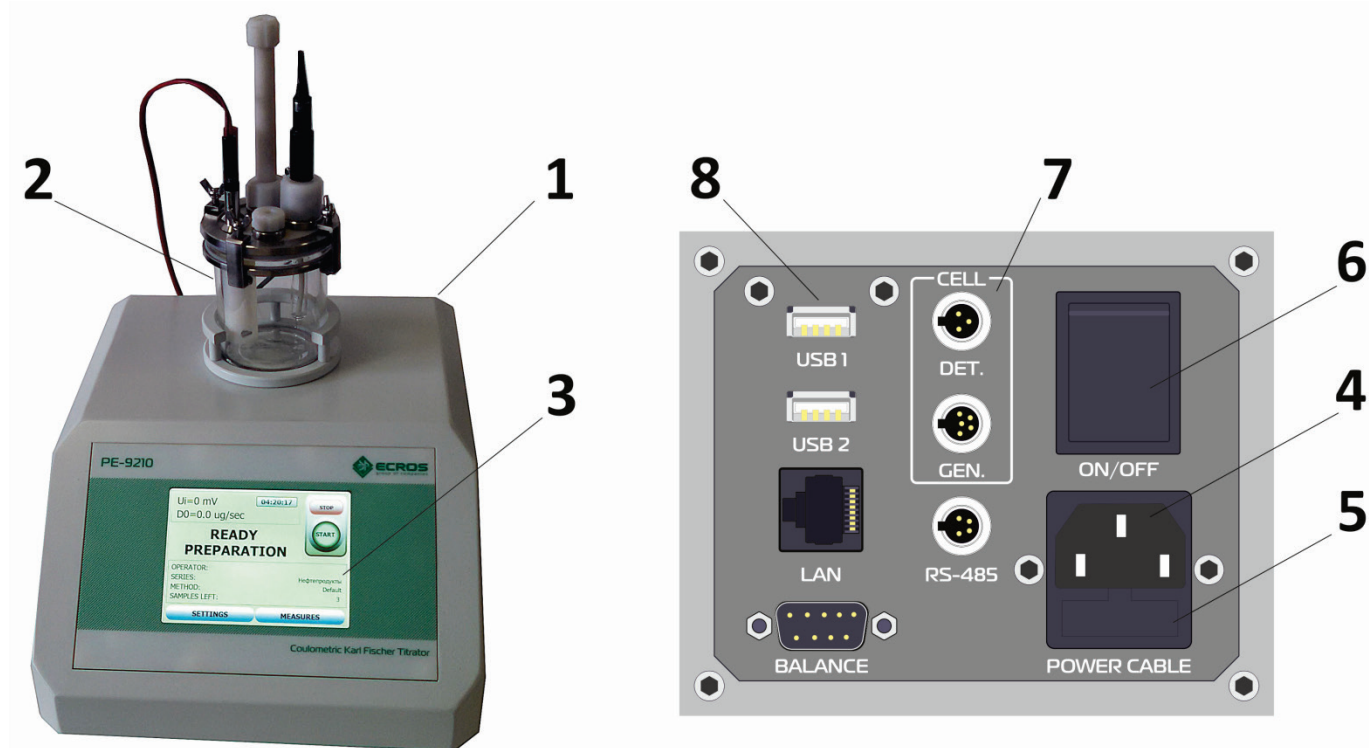


Figure 4

8. Operation Safety Precautions

8.1. Individual Protective Measures

- To avoid a risk of electrical shock, use an electrical socket to plug the instrument which is equipped with a grounding terminal. In case there is no grounding, a technical failure can result in fatality.

- Disconnect a power cord from the instrument when replacing the fuse. Otherwise, there is risk of electrical shock.
- When replacing the fuse install fuses of specified type and nominal only.
- When working with chemical agents and solvents follow the manufacturer recommendations and general safety rules during laboratory operations. All Fischer reagents are highly inflammable and toxic.
- In case of contact of the reagent with skin, wash it off with a large amount of water.
- In case of contact of the reagent with the conjunctiva, wash the eye intensively with a large amount of water and call the doctor.

8.2. Operational Safety Precautions

- According to the method of human protection from electrical shock the titrator corresponds to class 1 of GOST 12.2.007.0. When working with the instrument Rules for Operation of Customers' Electrical Installations and Safety Rules for Operation of Customers' Electrical Installations approved by state energy supervision (Gosenergonadzor) and requirements of GOST 12.2.007.0 must be followed.
- Only the personnel who have the necessary qualification, who have learned the safety rules and have read this document must operate the instrument.
- Connection of the instrument to the ground network is performed with a help of a two-pole socket and a plug with a grounding terminal. Electrical resistance of the ground network must not exceed 4 Ohm. Operation of a non-grounding instrument is strictly prohibited!
- Before connecting the titrator to the mains ensure that the power cord is not mechanically damaged.
- Remove immediately liquid drops from the instrument surface, as the instrument housing is not waterproof.
- Operation of the instrument in the explosion hazardous zones is not allowed. The instrument housing is not leak tight, and gases penetrating inside can flame out from an electrical spark or as a result of corrosive processes.
- Maintenance of the instrument must be performed only by the service authorised by the manufacturer.

9. Operation Procedure

9.1. Switching On the Instrument

- Before switching on the instrument make sure that the power cord is not damaged and connected to the instrument correctly.
- Place the titration cell prepared for work into the cavity of the top part of the instrument (see section 12 "Preparation of the Titration Cell").
- Connect the plugs of the generator and indicator electrodes to the relevant ports of the instrument (Figure 5).

- Switch on the power of the titrator with a help of the switch “POWER”. A self-diagnostics process of the instrument will begin. The self-diagnostics procedure is shown on the display (Figure 6).
- After self-diagnostics the main working window appears on the display, preparation and measurement processes are controlled in this window. The initial mode of the instrument after switching on the power and performing the self-diagnostics is “WAITING” mode. In this mode iodine is not generated, the instrument is waiting for the pre-titration mode switching on in order to prepare the titration cell for work.

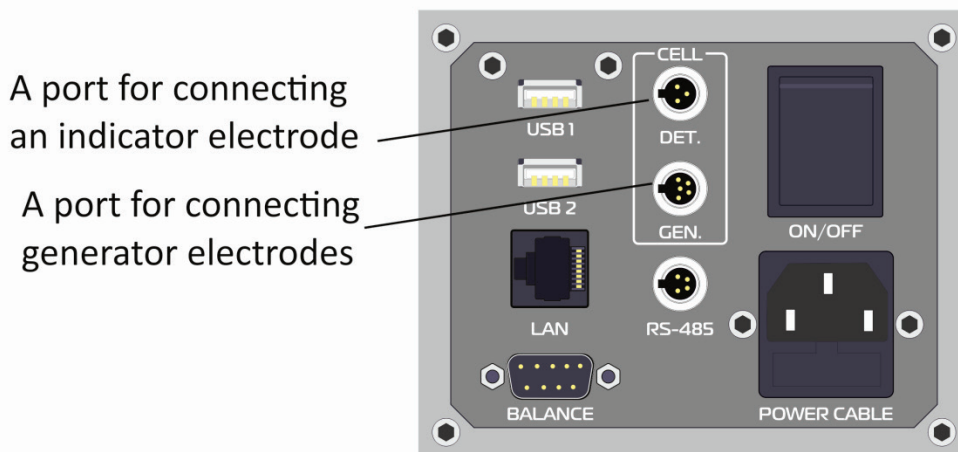


Figure 5

- For changing to the pre-titration mode press the “START” button. Now the iodine generation function is switched on, and the stirring device begins to work. When the work starts the titrator is in the state of non-readiness “NOT READY”. When the titration cell is ready, the mode “READY” or “STABLE” is switched on depending on the drift value of the titration cell measured in the pre-titration mode. The “START” button becomes green; the instrument is ready for measurements (Figure 7).

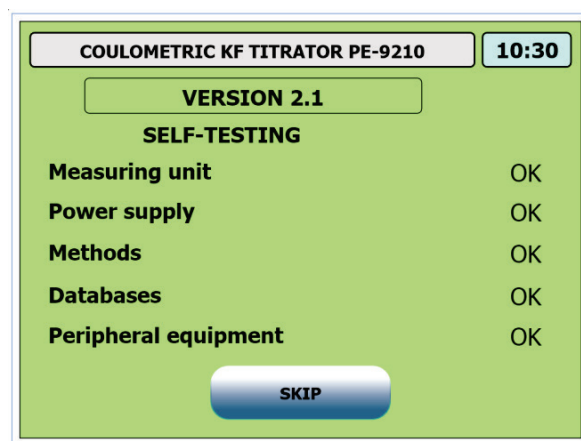


Figure 6

- When the titrator is switched on after the cell maintenance or long downtime, significant time to reach readiness to perform measurements may be required.
- To switch off the pre-titration mode, press the “STOP” button.
- When you finish working with the instrument it is necessary to stop pre-titration process by pressing the “STOP” button and disconnect the power with the power switch.

9.2. Configuring a Measurement

The following terms are used in the instrument interface to describe a measurement:

- A Lot – it's a portion of testing substance taken from the one place and/or treated identically used for final water determination.
- A sample is a dose taken from a lot for single cycle of titration in the cell. Usually the several sample titrations are used for final water determination.

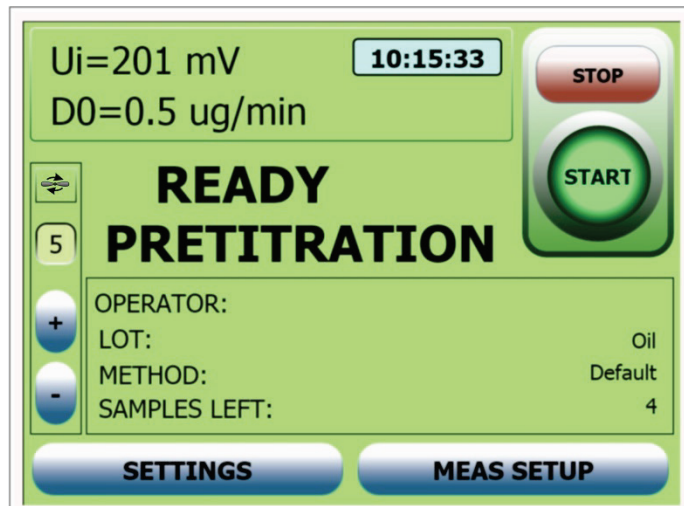


Figure 7

To configure a measurement push “MEAS SETUP” button at the main working window (Figure 7), the lot configuration window will appear (Figure 8).

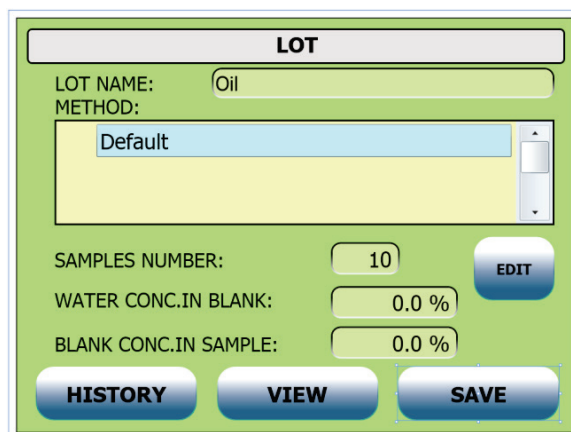


Figure 8

The following parameters are available:

- LOT NAME – the name of the current lot;
- METHOD – current method highlighted in the methods list. It is necessary to choose a suitable method from the list or create a new one using “EDIT” button (see paragraph 9.4 “Choosing a Measurement Method”);
- SAMPLES NUMBER – number of samples for titration;
- WATER CONC. IN BLANK – if a lot was prepared using a solvent containing some water, first you have to determinate water concentration in this solvent to take into account all the water in the sample;

- BLANK CONC. IN SAMPLE – also you need to know a solvent concentration in the sample.

In all other cases leave these two fields equal to zeros.

In general case water mass content in the sample is calculated based on the following formula:

$$M_x = F \times (M_T - D_0 \times T - M_0), \quad (5)$$

where

M_x – moisture mass content in the sample;

F – factor (corrective coefficient);

M_T – total titrated moisture content;

D_0 – initial drift of the cell at the moment of titration beginning;

T – total titration time;

M_0 – additional moisture content in the sample which has been calculated on the basis of the result of the blank sample titration (background).

At that dimensional values must be stated in the same weight and time units.

Additional moisture content is automatically calculated as follows:

$$M_0 = \frac{C_w^{BLANK} \times m_s \times C_{BLANK}^S}{10000}, \quad (6)$$

where

M_0 – additional moisture content in the sample;

C_w^{BLANK} – mass percentage of moisture in blank sample;

m_s – mass of the sample;

C_{BLANK}^S – mass percentage of the blank in the sample.

The formula for calculation of moisture mass concentration is the following:

$$C_w^S = \frac{M_x}{m_s} \times 100\%, \quad (7)$$

Correspondingly, for the original substance taking into account water content in a blank:

$$C_w^S = \frac{M_x}{m_s \cdot \left(1 - \frac{C_{BLANK}^S}{100}\right)} \times 100\%, \quad (8)$$

where

C_w^S – moisture mass concentration in the sample (%);

C_{BLANK}^S – mass concentration of the blank in the sample (%);

M_x – moisture mass content in the sample;

m_s – sample mass.

After making all necessary changes push the “SAVE” button to save lot parameters.

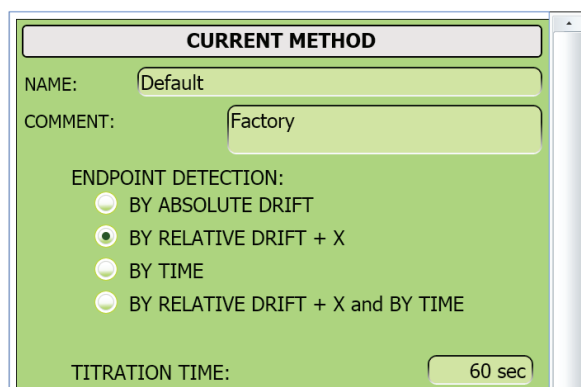
9.3. Preparation of the Measurement Method

9.3.1. Method Structure

The measurement method is a combination of parameters of titration and analysis results calculation saved in the memory of the instrument.

The following parameters are included to the method:

1. Method name and comment. "COMMENT" field is for reference only and never processed by the instrument.
2. A criterion of titration termination – it determines the order of using the titration termination cut-off criteria. The following values can be a possible stop criterion:
 - "BY ABSOLUTE DRIFT". An absolute drift value. In this stop mode the instrument stops titration when the set drift value is reached. This value is set in the edit box "ABS. DRIFT or X" (Figure 10).



CURRENT METHOD

NAME: Default

COMMENT: Factory

ENDPOINT DETECTION:

☐ BY ABSOLUTE DRIFT

☒ BY RELATIVE DRIFT + X

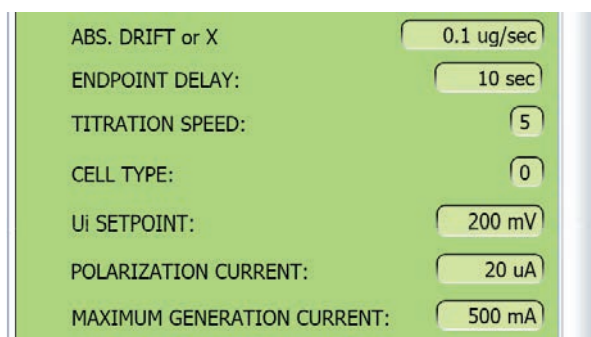
☐ BY TIME

☐ BY RELATIVE DRIFT + X and BY TIME

TITRATION TIME: 60 sec

Figure 9

- "BY RELATIVE DRIFT + X". A drift initial value of the titration cell plus the value set in the edit box "ABS. DRIFT or X" of the method. In this mode the instrument stops titration when the drift value equal to the sum of the initial drift and the set parameter is reached. The initial drift is determined by the instrument automatically while it is prepared for the titration procedure during the pre-titration process.



ABS. DRIFT or X: 0.1 ug/sec

ENDPOINT DELAY: 10 sec

TITRATION SPEED: 5

CELL TYPE: 0

Ui SETPOINT: 200 mV

POLARIZATION CURRENT: 20 uA

MAXIMUM GENERATION CURRENT: 500 mA

Figure 10

- "BY TIME". A titration time. After the expiry of the time value set by the parameter "TITRATION TIME" the instrument stops titration process and calculates the result.

- “BY RELATIVE DRIFT + X and BY TIME”. If this parameter is set, and a maximal titration time “TITRATION TIME” is set, the titration process is finished either when the set time is expired or the chosen drift criterion is reached depending on what event happens earlier. If the drift criterion is not set (there is a zero value in the field “ABS. DRIFT or X”), only maximal titration time is taken into account. If no criteria are set, an error message appears.
3. “TITRATION TIME” and “ABS. DRIFT or X”. These fields are used in combination with endpoint detection parameters as described above.
 4. “ENDPOINT DELAY” is an additional time the instrument waiting for some damping of the system after one of the cut-off criterion is reached.
 5. “TITRATION SPEED” – it determines dip of a titration curve and influences the total time of the sample titration.
 6. “CELL TYPE” – it determines work with one of two types of cells. (System of acknowledgement of the cell type and automatic installation optimal for this type of the given titration parameters is implemented in the instrument).
 7. “U_i SETPOINT” – it determines a potential value of the indicator system U₀ (baseline) corresponding to necessary excess of free iodine in the working solution.
 8. “POLARIZATION CURRENT”. Indicator electrode polarization current – it determines a current supplied to the pair of indicator electrodes for their polarization.
 9. “MAXIMUM GENERATION CURRENT”. Generator electrodes current – it determines a maximal reached value of the generator electrodes current during titration procedure.
 10. “STIRRER SPEED” – the instrument has 10 speeds of the stirring device (0÷9) that corresponds to the rotor rotation speed from 0 to 200 and then from 200 to 1000 rpm. In the method (Figure 11) the following mixing speeds are set:
 - “WHILE STIRRING” – speed of stirring of the solution with the sample before switching the generator electrodes on after loading the sample to the titration cell;
 - “WHILE TITRATION” – stirring speed during the titration procedure.

The screenshot shows a configuration screen with a green background. It contains the following settings:

- STIRRING TIME:** 15 sec
- SAMPLE WEIGHT UNIT:**
 - ☒ MILLIGRAM (mg)
 - ☐ GRAM (g)
- WATER CONTENT UNIT:**
 - ☒ MICROGRAM (ug)
 - ☐ MILLIGRAM (mg)
- WATER CONCENTRATION UNIT:**
 - ☒ %
 - ☐ ppm

Figure 11

11. "STIRRING TIME" – it determinates the time of mixing of the solution with the sample before switching the generator electrodes on after loading the sample to the titration cell;
12. "SAMPLE WEIGHT UNIT" – measuring units of the sample weight. The sample weight may be stated in milligrams or grams.
13. "WATER CONTENT UNIT" may be stated in micrograms or milligrams.
14. "WATER CONCENTRATION UNIT". Water concentration is stated by the instrument either in percent or in weight content (ppm).
15. "WEIGHT INPUT". Weight input mode with the following variants:
 - "FIXED WEIGHT" – for the dispenser use, the dispenser capacity value is entered into the corresponding edit box;
 - "MANUAL INPUT" – for the weight value input using the virtual keyboard on the every titration;
 - "DIFFERENTIAL INPUT" – for input the weight of syringe with and without a sample and taking a difference.

WEIGHT INPUT:

☒ FIXED WEIGHT 10.0 mg

☐ MANUAL INPUT

☐ DIFFERENTIAL INPUT

DRIFT COMPENSATION:

☒ NONE

☐ MANUAL 0.0 ug/sec

☐ AUTO

FACTOR: 1.0000

COPY DELETE SAVE

Figure 12

ATTENTION! When you enter the sample weight value, weight values of a syringe with the sample and of an empty syringe manually, to receive correct measurement results it is necessary to consider the order of entering values based on measuring units set by the current method. And similarly to it, in the process of automatic work with the balance weight units in the balance must correspond to the measuring units of the current method.

16. "DRIFT COMPENSATION". Drift accounting while calculation: "NONE", "MANUAL" or "AUTO", using preliminarily memorized value of initial drift.
17. "FACTOR" – it is a corrective coefficient by which the received measurement result is multiplied (default value is 1).

There are two types of methods in the instrument – built-in and user methods. Changing parameters in the built-in methods is impossible. You can only copy a built-in method and create a user method on its basis changing the required parameters.

ATTENTION! All methods of the instrument must have unique names. Entering two methods with similar names to the instrument is impossible.

9.4. Choosing a Measurement Method

Touch the method name in the methods list of lot configuration window to choose the method or push the “EDIT” button to configure the method by means of method parameters window.

You can change, copy and delete method using a buttons in the method parameters window (Figure 8).

ATTENTION! Changing parameters is possible only in user methods.

NOTES:

- The name of a new copy must be unique among all names of the instrument methods.
- The copied method becomes the current method of the instrument.
- You may copy any methods: factory and user methods as well.
- Factory methods may be deleted only with a help of service software.

9.5. Lot Measurements

After method and lot configuration you have to push “SAVE” button to return to the main working window and start the lot measurements.

If the instrument is ready for titration (the state “READY” or “STABLE”) you can start the measurements process. For that purpose:

- Press the “START” button in the main window of the instrument;
- Collect the sample for measuring with a help of a syringe;
- Weight the sample and enter the sample weight value into the instrument (the entering order depends on the sample weight entering mode);
- Input the sample to the cell through the sample inlet port;
- Press the “ENTER” button in the entry window, the instrument will be switched to the titration mode, and the main window will be changed to the titration window.

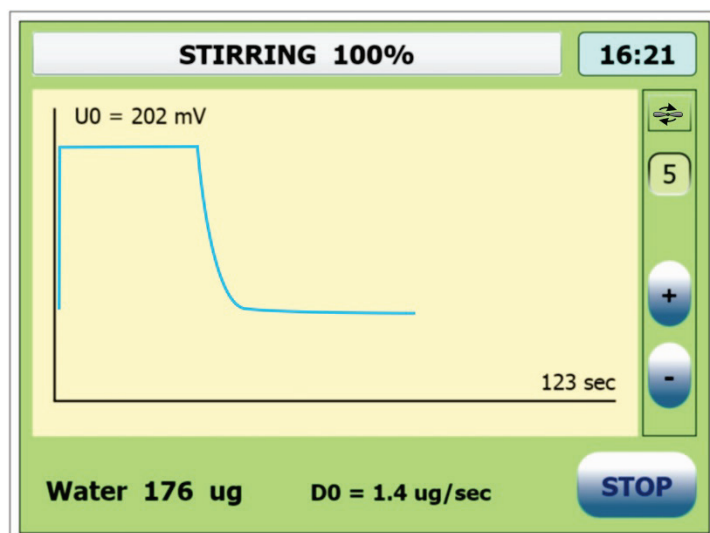


Figure 13

Before the titration process the instrument will stir electrolyte and the input sample. Stirring speed and time are set by the method parameters. When the stirring process is finished the instrument will be automatically switched to the titration mode (Figure 13).

When the titration process of the sample is over the result is displayed in the window of measurement results (Figure 14).

- The following further actions are possible:
- Press the “NEXT” button to continue measurements of this lot. After that the instrument is switched to the main working window, and if it is ready for measurements, a request for the following sample injection will appear. If the instrument is not in the state of readiness for measurements, a request for the sample injection will not appear. In this case wait till the “READY” state and press the “START” button to continue a series of measurements.
- Press the “DELETE” button to delete the received current result. The result will be deleted and the instrument will continue a series of measurements.

OPERATOR:
 LOT: Oil
 METHOD: Default
 SAMPLES : 5
 SAMPLES LEFT: 4

10:22

SAMPLE No: 1
 SAMPLE WEIGHT: 10.0 mg
 WATER AMOUNT: 9999.1 ug
 CONCENTRATION: 100.176 %
 TITRATION TIME: 512 sec
 BLANK: 0.0 ug
 DRIFT COMPENS.: 0.125 ug/sec

LOT RESULTS:

No	Ms mg	Mw ug	C %
1	10.0	9999.1	100.18

STATISTICS:

Ns	Cw %	SD Cw
1	100.176	0.0000

Buttons: RECALC, DELETE, NEXT, PRINT, DELETE, RECALC, EXIT

Figure 14

The overall table of the lot measurement contains the results of the measurement and statistic data.

- Press the “RECALC” button to correct the results of the measurement series. After that choose measurement correction in the appeared window of a request for either method or measurement correction.

- Press the “DELETE” button to delete all lot results.
- Press the “PRINT” button to transfer the measurement result to the printer or PC.
- Press the “EXIT” button to save the results of the current series.

After that the current series of parallel samples measurements for the given lot has been saved or deleted the main window will appear where you can continue or finish operating the instrument.

10. Measurement History

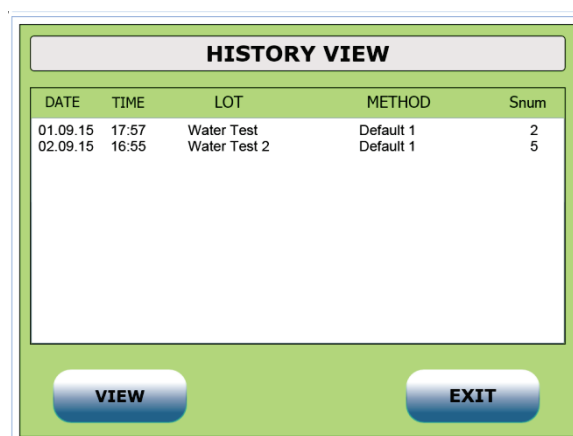
The instrument is equipped with an uninterruptable function of recording all the changes. In order to see the history there is a window in the instrument called “HISTORY VIEW” which is available from lot configuration window (Figure 8) using “HISTORY” button. Moreover, the history is available to see and save to the PC media with a help of a user program for PC.

Each history record has four fields:

- Date – it contains the date of saving of the measurement result.
- Time – it contains time of saving of the measurement result.
- Lot – it contains the name of the lot which the current history measurement belongs to.
- Method – it contains the name of the method which has been used for measuring.
- Snum – it contains a number of samples measured for current lot.

When you press the “VIEW” button, a view window for the current lot (Figure 14) opens where you can see all the measurements for the current lot.

The history capacity is 1000 records; when this value is reached, the history is renovated at the account of the oldest record deleting.



HISTORY VIEW				
DATE	TIME	LOT	METHOD	Snum
01.09.15	17:57	Water Test	Default 1	2
02.09.15	16:55	Water Test 2	Default 1	5

Figure 15

The history view window is designed for simultaneous display of several records. Use a vertical scroll box to scroll the list. When the window is opened the latest records are displayed.

The history is possible to delete only using a special service procedure or external PC software.

11. Instrument Setup

Change over to the Setup menu can be performed from the main window of the software by pressing the “SETTINGS” button (Figure 7).

There are five groups of internal settings in the instrument (Figure 16):

- “SYSTEM SETTINGS” are the settings which have common meaning: setup of an internal clock, verification date, inventory number as well as view of the current serial number of the instrument and configuration of network parameters and interface language.
- “INSTRUMENT SETTINGS” are the settings which provide operation of the touch screen and display.
- “PRINT FORMAT” is the settings of type of connected printer and report format.
- “BALANCE SETTINGS” – it means choice of a brand of the balance connected to the instrument.
- “SPECIAL SETTINGS” – setup of additional instrument parameters, concerning some titration parameters and reset of service limit of used electrolytes.

All the settings are saved in the permanent memory of the instrument.

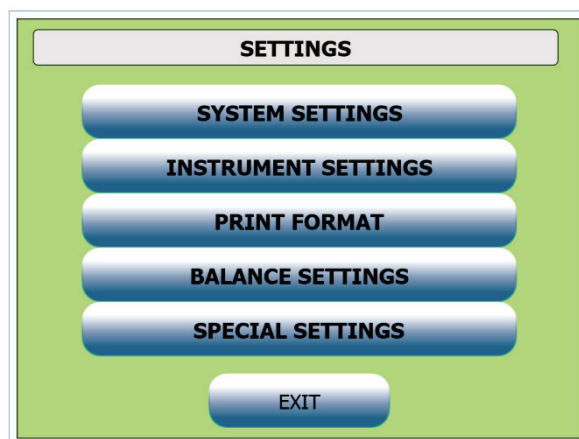


Figure 16

11.1. System Settings

The setting group “System Settings” (Figure 17):

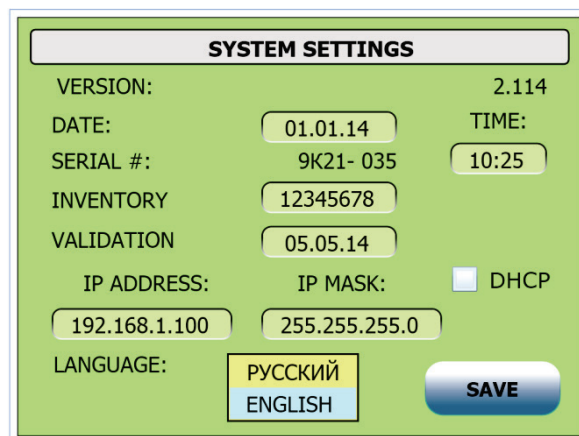


Figure 17

- Shows the version of instruments firmware.

- Sets the current date in the entry field “Date”. When this field is activated, the virtual keyboard window for digital entry will appear. The date must be entered in the format “dd:mm:yy”, where “dd” is a day (01-31 depending on the month), “mm” is a month (01-12), “yy” is two last symbols of the year (00-99).
- Sets the current time in the entry field “Time”. When this field is activated, the virtual keyboard window will also appear with a help of which you can set the current time in the format “hh:mm”, where “hh” is the current hour (00-23), “mm” is a current minute (00-59).
- Provides the instrument serial number view in the view window “Serial number”. The serial number of the instrument is factory set and must not be changed.
- Sets an inventory number in the entry field “INVENTORY”. Choosing this field opens the virtual keyboard window to enter an unspecified number with maximum 12 characters.
- Sets the validation date in the entry field “Validation Date”, similar to the field “Date”.
- IP ADDRESS – a field for the manual setting of network IP-address.
- IP MASK – a field for the manual setting of network IP-mask.
- DHCP – turns on an internal DHCP-server for PC-connection using Ethernet cable.
- Language – a choice of instruments interface language.

Setup changes are saved and applied, if you press the “Save” button.

11.2. Instrument Settings

This setting group (Figure 18) provides:

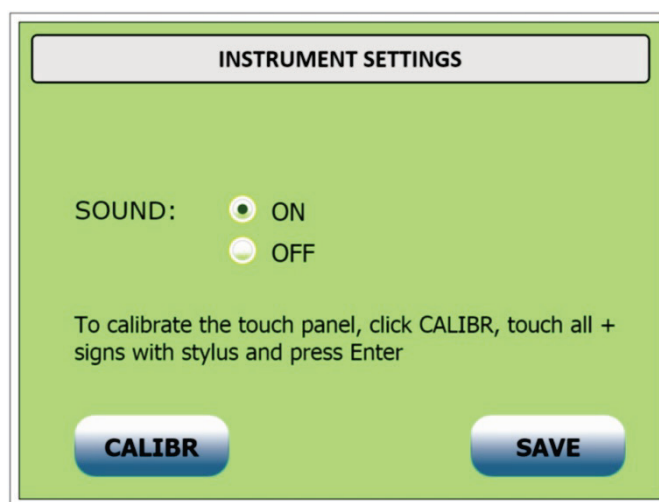


Figure 18

- Turn on/off the sound when touching the touch screen buttons - turn on or off the checkbox in the field "On" or "Off".
- Calibration of the touch screen for more comfortable work with screen objects. The instrument is fitted with an analog touch screen; that is why errors of matching of sensor and screen coordinates may appear. Initially the instrument has been calibrated by the manufacturer, but during the operation process discrepancy between screen and touch panel coordinates may appear. To remove these errors the touch panel must be calibrated. For calibration four coordinate targets “+” placed in the corners of the screen are used. Calibration is performed with a stylus by alternative choice of all four targets ap-

pearing in the corners of the screen one by one. When all four targets have been chosen the message “Calibration performed!” will be displayed in the bottom of the window.

Press the “Save” button to save changed settings of this group in the permanent memory.

11.3. Print Format

A setting group “Print Format” (Figure 19) provides a choice of connected printer and the report type. The report meets the requirements of a GLP standard group.

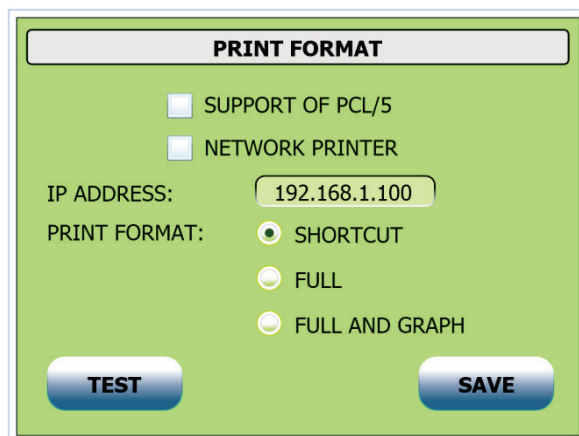


Figure 19

- Shortcut report includes:
 - Measurement date and time (time of the latest series measurement);
 - Parameters displayed in the main working window of the instrument;
 - Results of the measurement series displayed in the window of titration termination in the series.
- Full report includes besides the standard report parameters all parameters of the used method and parameters of measurement setup (all parameters of the window “Lot” except for the method name).

Reports are sent on command to the printer connected to the instrument or to the personal computer via interface USB after every series of measurements and processed by the user program.

11.4. Balance Settings

The group “Balance Settings” (Figure 20) provides a choice of a type of the balance used for weighing samples and sets parameters of serial connection interface.

ATTENTION! The instrument has been adjusted for work with the balance in which parameters of interface RS-232 are set as default. Before switching on the balance make sure that parameters of interface RS-232 of the balance and the instrument correspond to each other. If necessary adjust parameters in the balance settings.

The instrument software supports work with analytical balance of the following manufacturers:

- AXIS;
- KERN & Sohn;

- SARTORIUS;
- Ohaus;
- SHIMADZU;
- Mettler-Toledo;
- A&D Instruments.

BALANCE SETTINGS	
SELECT BALANCE:	PORT SETTINGS:
AXIS	SPEED: 4800 bod
A&D	LENGTH: 8 bit
KERN	PARITY: none
SARTORIUS	STOP BITS: 1 bit
OHAUS	
SHIMADZU	
METTLER	
TEST	EXIT

Figure 20

Connection of the balance of other manufacturers is unintended and possible only on request.

To choose the required balance type, touch corresponding manufacturers mark in the list of the applied balance types. In the window of the balance setting the information on the current settings of the interface RS-232 of the instrument for the chosen balance will appear.

Save the settings by pressing the “Exit” button, you also can test the input from the balance if press “Test” button.

SPECIAL SETTINGS	
READY DRIFT LEVEL:	1.0 ug/sec
STABLE DRIFT LEVEL:	0.1 ug/sec
MAX TITRATION TIME:	600 sec
OPERATOR:	
EXIT to OS	EXIT
ANOLYTE CAPACITY:	1000 mg
ANOLYTE CAPACITY LEFT:	1000 mg
CATHOLYTE CAPACITY:	300 mg
CATHOLYTE CAPACITY LEFT:	300 mg
	RESET

Figure 21

11.5. Special Settings

A setting group “Special Settings” (Figure 21) lets you adjust the following parameters:

- “READY DRIFT LEVEL”. A drift in the READY state – it determines the drift value at which the instrument is considered to be ready to measurements.
- “STABLE DRIFT LEVEL”. A drift in the STABLE state – it determines the drift value at which the instrument is considered to be ready to measurements and provides the specified accuracy of measurements.
- “MAX TITRATION TIME”. A maximal titration time – after this time the instrument stops the titration independently on cut-off criterion.
- “OPERATOR”. Operator name.
- Limit values of catholyte and anolyte capacity. It may be edited by entering digital values of the relevant capacity into the entry fields. Entry of the catholyte capacity value is not available for the titration cell “Type 1”.
- Reset (sets the initial value which coincides with the limit value) capacity values of catholyte and anolyte. It may be performed by pressing the “Reset” button.
- View the rest of the catholyte and anolyte capacity. It is performed in the fields “Anolyte capacity” and “Catholyte capacity”. For the titration cell “Type 1” the current capacity of the catholyte is always 0 and is not important.

12. Preparation of the Titration Cell

12.1. Disassembly and assembly of the Titration Cell

Assemble the titration cell before the first use and after disassembling of it.

NOTE: normally the instrument is equipped with cells of “type 1”, and in this section the cell preparation is described in connection with this type of cells. Cells of “type 2” are supplied on request and equipped with separate operational documentation.

A cell is a complex precision device; accuracy of the parameter measurements and service life of reagents used for titration depend on correct preparation thereof for work and observance of the operation rules.

The cell comes standard with:

- Glass jar;
- Carrying platform with a hard-wired generator chamber with desiccant unit;
- Attachment point of the indicator electrode;
- Sample inlet port;
- Fasteners of the carrying platform on the jar;
- O-rings from fluorine rubber;
- Replaceable seal (septum);
- Magnetic stirring rotor;

A titration cell is supplied in the assembled condition (Figure 22). Before operation it is necessary to clean a working area of the cell. For that purpose disassemble it.

NOTE: it's strictly forbidden to demount a generator chamber which will result to generator electrodes damage.

Partially disassemble the cell (Figure 22) in the following order:

1. Loosen the fixing screws (pos. 1) of the platform (pos. 2) on the jar (pos. 3).
2. Draw aside the grips (pos. 4) and remove the top supporting ring (pos. 5) and the bottom supporting ring (pos. 6) with a retaining ring (pos. 7).
3. Remove the carrying platform assembly (pos.2) with an O-ring (pos. 8).
4. Take out a magnetic stirring rotor from the jar (pos. 10).
5. Wash the jar, inner surface of the carrying platform, rotor, and operation surfaces of the indicator electrode and generator chamber with methanol. Persistent residues can be removed with a detergent and following rinsing with a distilled water. Carefully dry all the parts with a soft tissue.
6. Take off a top cap of the desiccant (pos. 12) and fill the desiccant tube with fresh molecular sieves. Put a top cap back.
7. Dry all the parts of the titration cell for several hours in the drying oven at the temperature of 70°C or using any warm air blower.

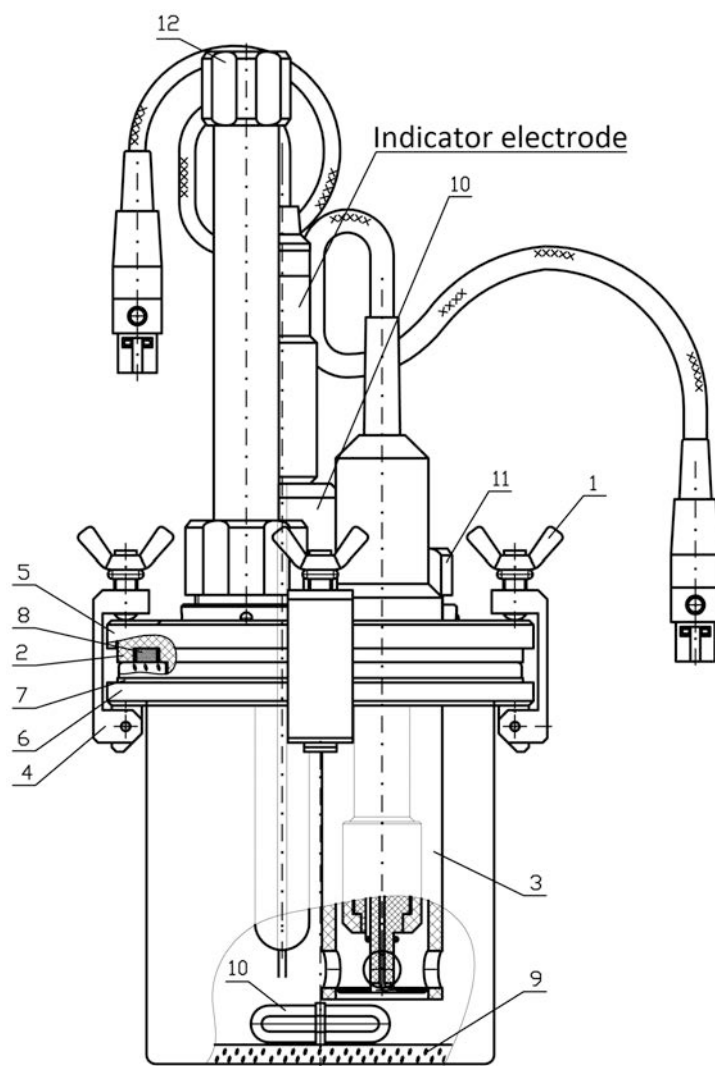


Figure 22

8. Put a magnetic stirring rotor to the jar and fill the jar with about 100 ml of fresh analyte solution.
9. Assemble the cell in reverse order. Diametric screws must be tightened one by one providing leak tightness of the inner volume at the account of an O-ring deformation (pos. 8).
- 10.²Insert the indicator electrode until bumping to the electrode attachment point (pos. 13 Figure 22). Do not turn an outer cap (pos. 7 Figure 25), otherwise, leak tightness of the whole unit may be broken;
11. Make sure visually that a septum is installed in the sample inlet port unit (pos. 11 Figure 22).
12. The cell is ready for installation into the instrument and operation.

12.2. During-Operation Maintenance

12.2.1. Septum replacement

The septum is manufactured from flexible material which provides leak tightness of the cell during operation. It is recommended to replace the septum not less than after 100 pinholes. In order to replace the septum take off the cap (pos. 1 Figure 23). Take out a clamping sleeve (pos. 2). Take out the septum (pos. 3). Wash the clamping sleeve and install a new septum from a spare parts set. While installation of the septum and clamping sleeve make sure they are not warped, otherwise it can result in leak tightness breaking of the unit. Turn the cap (pos. 1) by force necessary for the septum deformation by 0.5÷1.0 mm which provides leak tightness of the unit.

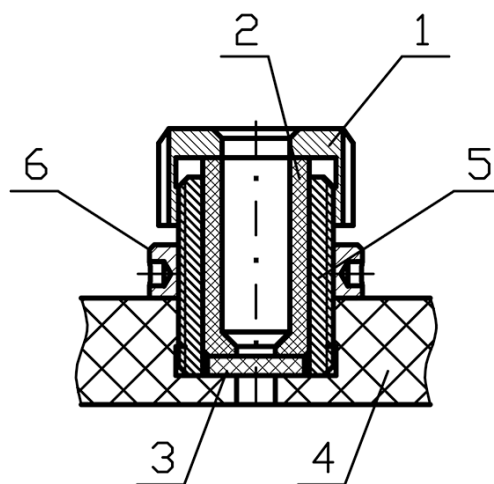


Figure 23

12.2.2. O-ring Replacement

An O-ring (pos. 8 Figure 22) is manufactured from flexible resistance material – fluorine rubber and does not require replacement during long-term operation.

² In case of the electrode was unmounted.

It is recommended to check flexibility of the O-ring once a month when replacing anolyte solution. When the O-ring has lost its flexibility it must be replaced.

12.2.3. Replacement of Molecular Sieves

If the cell drift is kept on a very high level fill the desiccant tube with fresh molecular sieves. In order to replace molecular sieves unscrew a cap (pos. 1) Figure 24, take out a tube (pos. 2) together with caps (pos. 1 and 3). Do not take out the O-ring (pos. 4). Take off a cap (pos. 3) from the tube and take out used molecular sieves. Clean and blow out the tube. Clean and blow out the cap (pos. 3). Fill the tube with fresh molecular sieves. Assemble the unit in the same way. The cap (pos. 1) has to be tightened firmly to ensure a hermeticity of joint.

It is recommended to check flexibility of the O-ring regularly in compliance with the instructions of molecular sieve replacement. If the O-ring has lost its flexibility it must be replaced. In order to regenerate molecular sieves it is necessary to keep them in the drying oven for 24 hours at temperature 170–260°C.

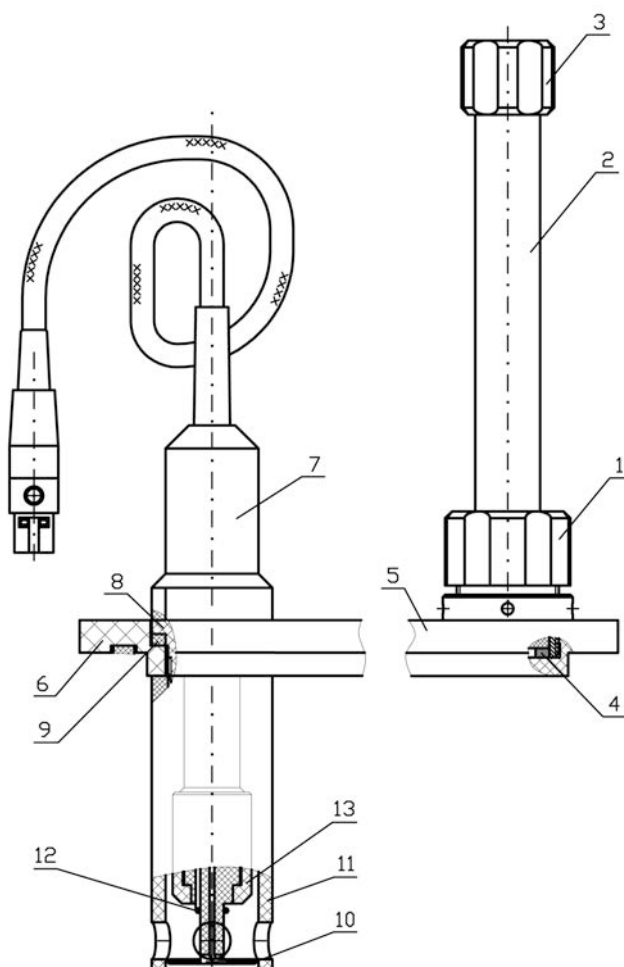


Figure 24

12.2.4. Replacement of O-rings in the Indicator Electrode Unit

The unit has been adjusted in such a way to provide firm tight connection of a glass housing of the electrode with O-rings which provides required leak tightness of the unit. While in long-term operation O-ring flexibility may be changed. That is why if any changes in connec-

tion firmness are found during installation of the electrode, it is necessary to adjust the cap (pos. 1) position Figure 25.

In order to replace O-rings:

- loosen the cap (pos. 1) for 2-3 turns;
- take out the electrode, take off the cap (pos. 1), take out intermediate sleeves (pos. 2) and O-rings (pos. 3);
- clean an alighting sleeve (pos. 4) and intermediate sleeves (pos. 2);
- replace O-rings and assemble the unit.

During installation of the O-rings and the sleeves make sure they are not warped, otherwise it can result in leak tightness breaking of the unit. Obtain leak tightness of the unit with a help of installation of the cap (pos.1) position.

NOTE: it's strictly forbidden to remove a ring (pos. 5) and an alighting sleeve (pos. 4) from the bearing platter (pos. 6).

12.2.5. Replacement of O-rings in the generator chamber assembly

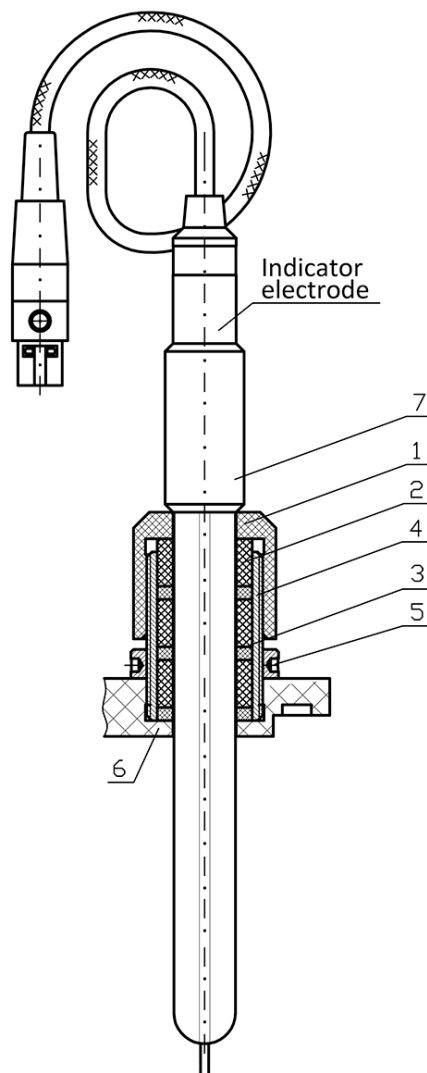


Figure 25

12.2.6. Anolyte Replacement

- Disassemble the cell (see p.12.1);

- Pour anolyte solution from the glass jar of the cell;
- Wash thoroughly all elements of the cell which are in contact with its inner volume with methanol; persistent residues can be removed with a detergent and following rinsing with a distilled water;
- Wipe the washed parts of the cell with a lint free cloth;
- Dry all the parts of the titration cell for several hours in the drying oven at the temperature of 70°C or using any warm air blower.
- Put a magnetic stirring rotor to the jar and fill the jar with about 100 ml of fresh anolyte solution.
- Assemble the cell.
- Make sure visually that a septum is installed in the sample inlet port unit.

13. Karl Fischer Reagents

For Karl Fischer titration it is necessary to choose suitable reagents in compliance with chemical composition of the analysed sample. See the reagents used for different sample types in the table below.

Sample \ Reagent	Honeywell	Mitsubishi	Notes
	Coulomat	Aquamicon	
General titration Alcohols Hydrocarbons Simple ethers Compound ethers	Coulomat AG Coulomat CG	Aquamicon AX Aquamicon CXU Aquamicon AX Aquamicon CXU	Reagents Coulomat AG/CG, Aquamicon AX/CXU are not appropriated for organic chlorine-containing compounds
Ketones	Coulomat AG Coulomat CG-K Coulomat AK Coulomat CK	Aquamicon AKS Aquamicon CXU Aquamicon AKS Aquamicon CKS	Only formaldehyde may be titrated from aldehydes. Coulomat CG-K и Amicon CXU are not appropriated for organic chlorine-containing compounds.
Gases	Coulomat AG Coulomat CG	Aquamicon AX Aquamicon CXU Aquamicon AX Aquamicon CXU	
Fats and oils	Coulomat AG-H Coulomat CG	Aquamicon AX Aquamicon CXU Aquamicon AX Aquamicon CXU	Reagents Coulomat AG/CG, Aquamicon AX/CXU are not appropriated for organic chlorine-containing compounds.
Amines	Coulomat AG Coulomat CG To use Coulomat AG add 20% of acetic, salicylic or benzoic acid to 100 ml of Coulomat AG	Aquamicon AX Aquamicon CXU To use Aquamicon AX add 10 g of salicylic acid to 100 ml of Aquamicon AX	Amine in a base form must be neutralised with acid.

14. Utilization Information

Upon expiration of the service life the titrator does not pose threat to life and health of people or environment, and does not require special utilisation methods.

An utilisation order is determined by the organisation which operates the instrument.

15. Storage and Transportation Rules

The titrator must be stored in the closed room in the factory package at the ambient air temperature between +5 and +40°C and maximum relative humidity 80%.

Without package the instrument must be kept at the ambient air temperature between +10 and +35°C and maximum relative humidity 80%.

The titrator may be transported by all types of transport in closed vehicles within the temperature range from -40 to +50°C and maximum relative humidity 95%.

16. Warranty

ECROSKHIM Co. Ltd. guarantees that the titrator corresponds to the characteristics specified in paragraph 3.3 of the present document provided that operation, transportation, and storage conditions are observed.

The warranty period of the titrator operation is 12 months since the date of shipment to the customer determined by a consignment note.

Warranty maintenance is performed only by authorised service centers of the manufacturer.

During the warranty period the instrument may be repaired or replaced free of charge. The warranty period of the instrument operation may be prolonged for the time during which the instrument has not been used due to found damages.

17. Reclamation Information

If any faults are identified during the guarantee period or if the customer finds that the instrument is not complete while unpacking it, he should send a reclamation report to the manufacturer's address:

ECROSKHIM Co. Ltd.

22 17-liniya V.O, build. "I", office 406, 199178, Saint-Petersburg

Telephone (812) 322-96-00, fax (812) 448-76-00

E-mail: info@ecohim.ru URL: www.ecohim.ru

A reclamation report is not submitted:

- If the warranty period is expired;
- If the customer has violated operation, transportation, and storage rules specified in the operation documentation.

18. Acceptance Certificate

Automatic coulometric moisture titrator PE-9210, factory number **9K21-**_____ has been checked in compliance with technical specifications БКРЕ.414315.002ТУ, obligatory requirements of national standards and approved for operation.

Manufacturing date _____

QC stamp

Inspector _____

Annex 1. Diagnostic Messages and Relevant Actions

The instrument has a developed system of self-diagnostics of the unit working capacity which provides the instrument testing upon switching on and working capacity testing during the measurement process as well.

Full diagnostics is conducted immediately after switching on the instrument. Most of errors reported by the instrument diagnostic system on the initialisation stage are critical and require to be eliminated under the terms of specialised maintenance service.

Critical errors are those which have been found by the diagnostic system immediately after switching on the instrument and reported by the following diagnostic messages:

- **CLOCK FAILURE** – a failure connected with breakdown of elements of a built-in real-time clock;
- **NO CONNECTION WITH MU** – a failure of the measurement unit or elements of communication between the measurement and interface units;
- **MU FAILURE: XX** – a failure of the measurement unit;
- **POWER FAILURE** – a failure of the power supply unit.

ATTENTION! The instrument which has errors of initial diagnostics except non-critical errors must not be used till these errors are eliminated. Non-critical errors are those which have been found by the diagnostic system immediately after switching on the instrument and reported by the following diagnostic messages:

- **FACTORY METHODS ERROR** – factory methods loaded to the instrument have incorrect meaning of parameters (while reading them out from the permanent memory check sum has not matched). If this error occurs the instrument may be operated after pressing the “IGNORE” button, but only one factory method will be loaded to the instrument;

NOTE: when this error occurs, user methods are stored as they are recorded in the different area of the permanent memory.

- **PARAMETER ERROR** – factory settings loaded to the instrument have incorrect meaning of parameters (while reading them out from the permanent memory check sum has not matched). If this error occurs the instrument may be operated after pressing the “IGNORE” button, but only default settings will be loaded to the instrument which do not change the instrument characteristics significantly.

NOTE: if non-critical errors occur, it is recommended to call the service center to reload instrument constants.

During the measurement process the built-in diagnostic system can give some messages about errors which influence significantly the measurement process and require immediate elimination actions from the operator. They contain the following errors and error messages:

- **Connect the INDICATOR electrode** – this diagnostic message can appear for the following reasons:

- The plug of the indicator electrodes is not connected or poorly connected to the relevant port of the instrument. The operator must check and correct the fault by connecting the plug correctly.
- The analyte solution level is not enough for dipping the indicator electrodes into it. Fill the titration cell with analyte till the required level.
- An indicator electrode is damaged. Replace the electrode by a new one.
- An input circuit of the indicator system of the instrument is damaged. This fault may be eliminated only in the service center.
- **GENERATOR electrode error** – this diagnostic message can appear for the following reasons:
 - The plug of one of the generator electrodes is not connected or poorly connected to the relevant port of the instrument. The operator must check and correct the fault by connecting the plug correctly;
 - Intermediate connections of the generator electrodes are not connected or poorly connected to the relevant electrodes. The operator must check and correct the fault by connecting intermediate connections in compliance with requirements of this manual;
 - One of the generator electrodes is damaged. Replace the electrode by a new one;
 - Intermediate connections of the generator electrodes are damaged. Replace intermediate connections by new ones;
 - The generator unit of the instrument is damaged. This fault may be eliminated only in the service center.
- **Pre-titration error** – this diagnostic message can appear for the following reasons:
 - Defective electrolyte. In this case the instrument is not able to get the titration cell to readiness for use. To eliminate this error, replace electrolyte;
 - Maximal titration current is not correctly matched (too little). To eliminate this error, change the current method or change the parameter of the method “Current mode” for a larger limit titration current;
 - Increased drift of the cell because of poor tightness of joints. To eliminate this error, check tightness and if necessary replace all seals in the titration cell joints;
 - The parameter “Potential” is set incorrectly. To eliminate this error, check the potential values in the “Special” menu and set the value in compliance with requirements of this instruction.
- **Titration is not possible** – this diagnostic message can appear for the following reasons:
 - Defective electrolyte. In this case the instrument is not able to terminate the titration process. To eliminate this error, replace electrolyte;
 - Maximal titration current is not correctly matched (too little). To eliminate this error, change the current method or change the parameter of the method “Current mode” for a larger limit titration current;
 - The titration method is chosen incorrectly. The titration stop mode is chosen incorrectly for the given electrolyte. To eliminate this error, change the current method or change the parameter “Stop mode”, or set the relevant parameter of the chosen stop mode.

Annex 2. Information on Repairs Made

Date	Fault description	Repaired by	Note