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KjelROC Analyzer
Operation Manual
Version N

1. Contents

2. Introduction

The KjelROC Analyzer is designed for highly automated distillation and simultaneous titration of mainly Kjeldahl digested samples. Depending on locally available reagents, it can be set to fit almost any requirement. The different screens displays the alternatives possible at any given point will guide the operator. Read this manual to benefit from all features of this instrument.

The built in data handling system allows flexible and safe transfer of weights, volumes and results, either manually via the touchscreen or using network transfer. How to set up the PC network connection is described in a separate Network Connection manual.

As most analyses carried out on this Analyzer involve the use of hazardous chemicals it is important that the operator reads or is informed about what is written in chapter 3, Safety.

To get a brief understanding of how he KjelROC Analyzer is working please refer to chapter 4, Function.

Chapter 5, How to run an Analysis, gives a brief description how analyses are performed using the KjelROC Analyzer.

In chapter 6, Operation, all available functions will be described. Depending on how the installation of your instrument is done, these functions can be different, as the set-up will customize the Analyzer to fit your workload.

To get the best from your instrument it is important to regular check the performance as described in chapter 7, GLP and Performance Tracking. You should also maintain and service your instrument on a regular basis. This is described in chapter 8, Maintenance and Service.

3. Safety

The KjelROC Analyzer is protecting the operator from any hazardous situations, e.g. no steam or alkali can be dispensed without having a tube in place and the protecting door completely closed. However, as the methods described often involve the use of hot corrosive chemicals every user should read the Safety Instructions carefully.

3.1. User Safety

The instrument may only be used by laboratory personnel and other persons who have knowledge and/or experience of doing chemical analysis and dealing with reagents.

Applications not mentioned in this document are improper. In particular, it is forbidden to use the instrument in the following instances:

- Use of the instrument that require ex-protected instruments
- Use of Samples or Reagents which can explode or inflame

It shall be noted that:

- Modifications and upgrades to the instrument shall only be carried out by authorized service personnel
- Service Menus in the Instrument is only to be used by authorized Service Personnel

3.2. Safety Symbols


General Hazard


Corrosive acid


Crushing hazard


Electrical shock hazard


Hot Surface

Explanations used in this manual


Important, Please Note


Please Note, Protection Glasses is recommended


Please Note, Gloves should be used

3.3. Product Safety Systems

The instrument is designed and built in accordance with state-of-the-art technology. Nevertheless, risks to users, property, and environment can arise when the instrument is used carelessly or improperly. If the equipment is not used in a manner specified by this document, the protection provided by the equipment may be impaired.

3.3.1. Maintenance and Service

The Operator is responsible for ensuring that recommended daily and monthly user maintenance is performed on the Instrument. Failure to do so might impair the functionality and/or shorten the lifespan of the instrument.

The operator is responsible to schedule regular Maintenance with authorized service personnel only. Only OPSIS LiquidLINE Spare parts should be used in the instrument.

3.3.2. Safety Sensors

The instrument is equipped with several safety systems:

- A Sensor will identify if a tube is placed in the holder. No automatic operation is allowed without tube in place
- The Safety door (protecting the tube) is monitored and no analysis or manual operation is allowed with the door open. Analysis will stop if opened during analysis.
- The small door protecting the Splash head is monitored and no analysis or manual operation is allowed with the door open.


Please note that the large door is not monitored in order to allow access to Titration Area during analysis. However, caution should be taken if running analysis with open door.

4. Function

This section describes the general function of your KjelROC Analyzer.

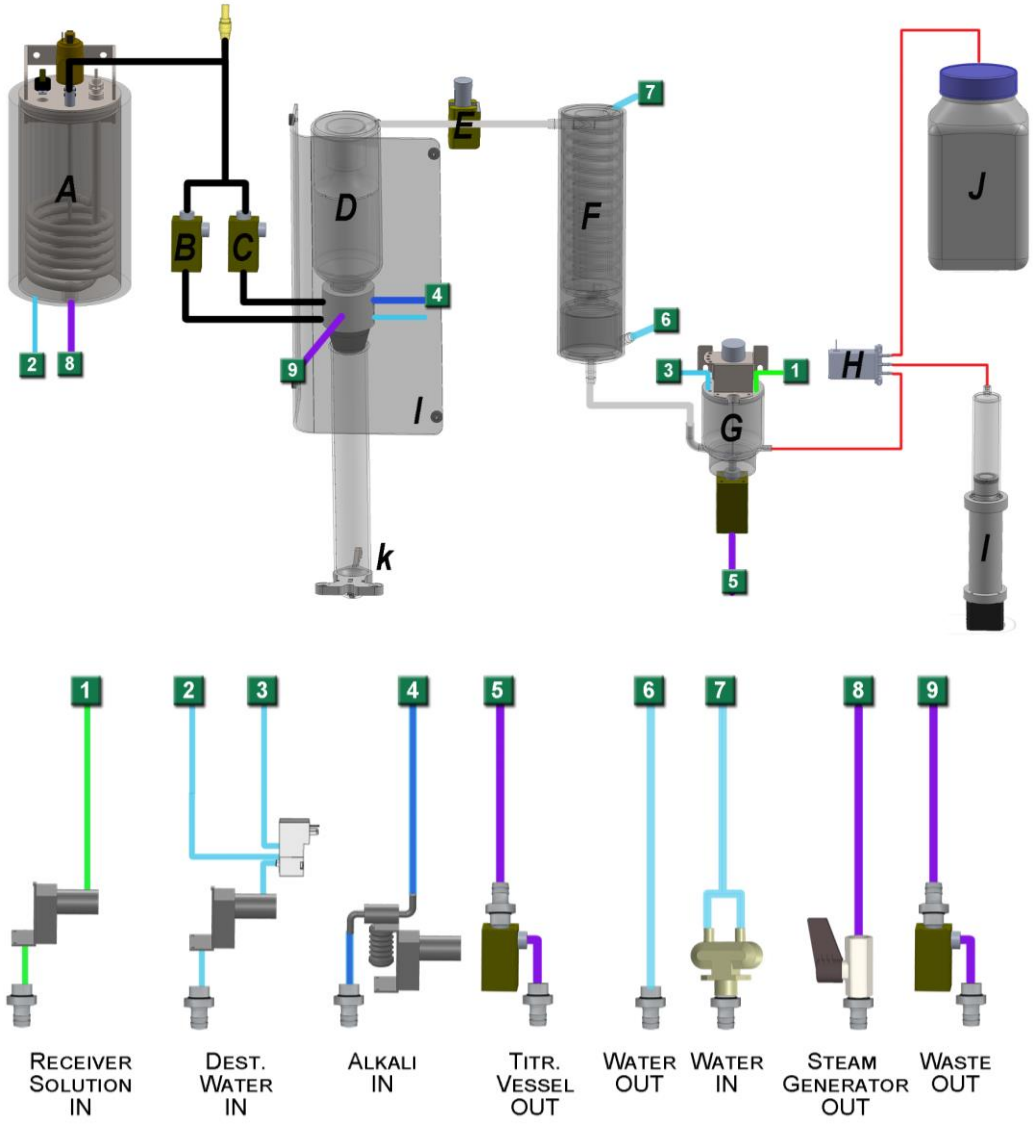

FIG. 1: FUNCTIONAL OVERVIEW OF THE KJELROC ANALYZER

A: Steam generator
B: Valve for steam used during distillation
C: Valve for steam used during tube drainage
D: Splash Head to prevent alkali drops to go with the steam
F: Condenser to cool down the distillate. It is supplied with a sensor to prevent operation without flowing cooling water. This sensor also reduces the water flow if sufficient temperature is obtained.
G: Titration Vessel with colour sensor. Here the titration to the pre-set equilibrium is done.
H: Three-port valve to allow titrant flow either into the burette (refill) or into the Titration Vessel (analysis)
J: Titrant storage flask with volume sensor.
I: Burette dispensing titrant in 1.95µl steps

Volumes and times for the analytical cycle are all pre-set to optimise the use of chemicals and energy. The described method in this section will refer to a Kjeldahl Analysis, since that is the most common method. If other methods are used the reagents and functions might be different. Please refer to the corresponding Application Note.

Please use Figure 1 to identify the parts/functions described below.

At power on the instrument automatically prepares for operation. The Burette (l) is filled up while returning to start position. The pumps for Water (3) and Receiver Solution (1) flush the system for some seconds to assure the tubes are filled. If LOGIN is pre-set, the operator will enter the pin code and thereby reach the allowed level of operation.

Weight/volume is entered directly on the instrument or by using the OPSIS LiquidLINE Transfer Utility program. A Test Tube is put in place and locked with a Tube holder (k). The analytical cycle is started by closing the Safety door (l) and touching the Start Button.

The Valve below the Titration Vessel (G) closes and pump (1) dispenses Receiver Solution. The Water Pump (2) dilutes the digested sample and after a pre-set delay, time Alkali is added with pump (4) The Steam valve (B) opens and the liberated ammonia is transferred by steam through the Splash Head (D), cooled down in the Condenser (F) and finally collected in the Receiver Solution (G).

The Burette (l) operates depending on the colour of the indicator, determined by the sensor below the Titration Vessel (G). Dispensed titrant volume as well as calculated result is displayed on the Screen during the analytical cycle. As soon as the end level or (if chosen) time is reached, the result with all parameters is stored.

The Valve (5) opens to empty the Titration Vessel (G). Steam Valve (B) closes and instead (C) opens. Waste Valve (9) opens and Valve (E) between Splash Head (D) and Condenser (F) closes. The residues from the Test Tube are pressed either into the connected Waste Tank or directly to drain. After some seconds, Steam Valve (C) closes again and Valve (E) opens.

Now next analytical cycle can start. Any time during the analytical cycle weight entry or data handling can be operated.

5. How to run an Analysis

This chapter gives an overview of how to run an analysis with the KjelROC Analyzer. More descriptions and details can be found in the following chapters.

5.1. KJELDAHL

Login into your KjelROC. Standard login code 1111 can be used.

5.1.1. Blank analysis

From the Main menu, select the AUTO BLANK program. Press ▶ until “Express” rack is displayed. Only Racks with sample weights already entered and the “Express” rack will show up.

Use an empty tube for the sequence. Please note, if there is a need to run blank with the chemicals used during digestion then select the Kjeldahl program and a normal blank, without repetition.

Step-by-step

1. Put the tube in place

Fig. 1. Inserting a tube in the KjelROC Analyzer

Start analysis by first closing the Safety door and then pressing START

Fig. 2. Closing the Safety door and starting an analysis

2. Reagents are added and distillation with simultaneous titration until the pre-set end volume. The test tube empties and the first cycle is over.
3. The analysis will automatically repeat until the pre-defined amount of repetitions are completed (default, three times). The last result will thereafter automatically be the selected blank for next analysis.
4. Confirm and if necessary repeat until you have a stable result. A typical blank value should be around 0.1 – 0.2 ml. It depends on how the Receiver Solution is adjusted and the titrant concentration. Please also read the LA1000, Application Guide Kjeldahl Determination.

5.1.2. Sample analysis

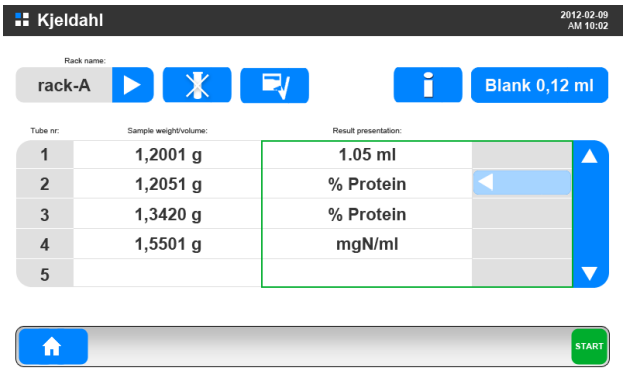

Fig. 3. Analysis menu

Step-by-step

5. Select the desired method
6. Select the Rack with the samples to be analysed in the analysis view. If the displayed blank differs from the one just established, press the blue field and enter the correct value.
7. Put a tube in place and make sure the correct tube no is highlighted. You can select another Tube by pressing on the corresponding row. To scroll in the list you use and buttons
8. Start analysis by closing the Safety door and pressing START (Depending on settings, the cycle can be started by just closing the door)
9. Reagents are added and distillation with simultaneous titration until the pre-set end volume. The test tube empties and the cycle is over.
10. Open the Safety door and continue with next test tube

If a tube cannot be used (e.g. broken during digestion) press the exclude button, .

If for any reason several tubes in a Rack cannot be processed, instead of pressing button for each, just press the complete rack button, , after you have processed the other tubes. When all tubes in the Rack are processed, press

Any time during analysis you can press and select another function, e.g. “Weight entry” the tube in process will continue until cycle is over. Next tube can be analysed by selecting “Kjeldahl” again.

5.2. Closing down and Cleaning

Please follow the instructions and guidance given in chapter 8.1, Daily maintenance and cleaning, on page 36.

- Insert an empty 250 ml sample tube into the KjelROC Analyzer.
- Select MANUAL MODE and then AUTO CLEAN.
- Switch off the instrument and close the water tap after Cleaning.

Note that the function “Tube Drain” should be set in the KjelROC Auto Clean program.

Though the KjelROC Analyzer is designed to resist the chemicals, a clean instrument will always last longer. Therefore, it is recommended to follow below hints.

- Remove the Drip tray and clean tray with warm water and a soft cloth.
- Clean the Safety door with a wet cloth.
- Check the area around the Adapter and Splash head holder and use a wet cloth to wipe any spillages. Check also below the drip tray.
- Use a wet cloth to wipe of any general spillages from the instrument. Alkali and Hydrochloric acid are particular important to clean due to their corroding effects.

6. Operation

The instrument functions can at any time be used in almost any order. However, the most common way to run an analysis is to

- Start up the instrument.
- Enter weight in the Weight Entry menu.
- Run analysis in the program menu and then repeat the analysis until rack or batch is completed.
- Review completed racks and batches in Data handling.

We will follow this structure in this chapter. For easy understanding, each section in the user interface is described under a separate chapter.

6.1. General Guidelines

If a function is not available because another one is in use, the button is faded out.

In all sub sections you can reach the main menu by pressing HOME .

There will always be a visible STOP button, , when running an analysis, which is used if there is a need to stop the process. When running an analysis most menu options will be disabled for safety reasons.

A warning button, , might appear in the lower right corner in case there is a warning from your KjelROC Analyzer. Warnings are to indicate if there is low level of Alkali, water or Boric acid in your tanks, high level in your waste tank or if it is time for maintenance.

It is recommended to use the supplied OPSIS LiquidLINE pen on the touch screen. Select the touch screen stylus.

6.2. START UP

Switch on power to start your KjelROC Analyzer. The instrument prepares for operation by flushing reagent pumps.

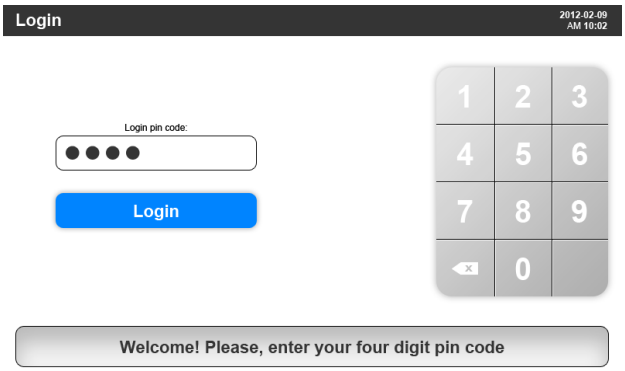

Fig. 4: Login screen

After initial start-up your pin code is requested, unless login has been disabled at set-up.

- In this view, you will see the current IP address in the upper right corner if connected to the KjelROC network. The IP address is needed when sending and receiving data using Ethernet or Wireless. See section 6.8, Using Network Connection.

- Insert your four-digit pin code and confirm by pressing LOGIN.
  Unless changed in your settings file the default is 1111 for an administrator (admin1). There are also other access codes, such as access to manager menu. These codes are described in a separate document.

- After entering the code, the main menu will appear.

Note that the steam generator will only heat during analysis. It might therefore take a couple of minutes until first analysis can run at full effect.

6.3. Main Menu / Home

After entering the login code, the main menu will be displayed. If login has been disabled then the main menu will be visible immediately after power up.

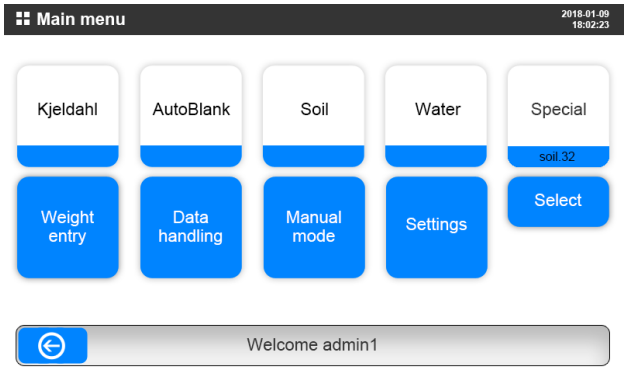

Fig. 5. Main menu screen

The main menu is the central point in the menu structure and you can always reach this menu by selecting HOME .

The options available in the main menu are listed in two rows;
The methods configured in the settings file are displayed in the first row. There are four quick access programs, normally “Kjeldahl”, “Auto Blank”, “Soil” and “Water”. There is also a Special program with a select button. This program allows further selection 60 additional programs. Please see section 5.5. Analysis, on page 19, to get further description of the options when selecting any of these buttons.

In the second row there will be four buttons; weight entry, data handling, manual mode and settings. By pressing the desired button, you will reach the corresponding section.

If you have login enabled you can always logout by selecting the LOGOUT button .

6.4. WEIGHT ENTRY

This chapter describes the weigh entry via the thouchscreen. Weight entry menu can be entered before or during an analysis. Weight entry can also be done through the OPSIS LiquidLINE Transfer Utility, please see 6.8, Using Network Connection, on page 25.

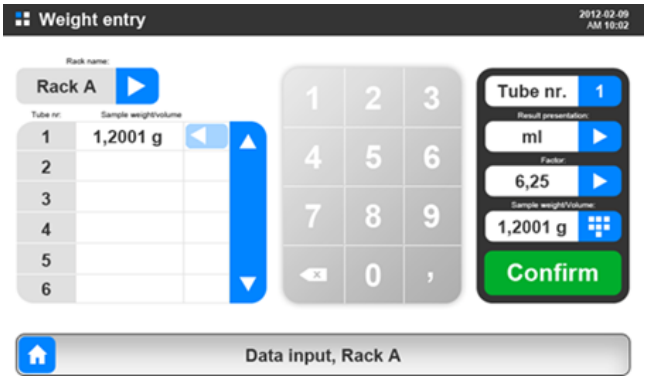

Fig. 6. Weight entry menu

The weight entry menu consists of four different sections; Rack name, Tube list, Number pad and Tube entry section.

Selecting rack
The Rack name and selection of rack is located the upper left part of this view. The amount of racks and the name of the rack are given by your settings file. Default from factory is the special “Express” rack and three racks; rack-a, rack-b and rack-c. Up to 20 racks can be defined in your settings file, please see 6.8.4, The Settings File, on page 25.

Selecting tube
The Tube list is located in the left part of this view. It is possible to select entering tube by selecting corresponding row. It is possible to move freely in this list and change previously entered weights. Default amount of tubes per rack is 20 or 25.

Entering weight
The Number pad is in the middle of this view. By using the number pad, it possible to enter the weight, which will be visible in the Tube entry section. The Tube entry section is to the right in this view. The active tube number will be displayed and it is possible to see the entered weight. Hint: If you have same weight for several samples, you can just press Confirm several times to add same weight.

Selecting Result type
It is possible to select what result type shall apply for each tube. Visible result types are possible to define in your settings file. The different result types can be separated into three groups; automatic Blank, Reference recovery and analytical result types.

The “mlBlank” and “ml” will both give the result in millilitres. However, the “mlBlank” will automatically use this value to update the blank value used in the instrument. The

“ml” result type will give the amount of titrated volume after deduction of the blank value entered in the instrument.

“mlBlank” will result in titrated volume in millilitres.
“ml” will result in titrated volume in millilitres minus the blank.

The “ref-Rec” or Reference recovery will give a recovery result based on OPSIS LiquidLINE reference solution (see separate application instructions in Performance Tracking). In addition, a copy of the result will be stored in a separate file folder in the instrument for easy retrieval by OPSIS LiquidLINE Reference Tracking system. Use the standard “%Rec” result type to perform a recovery test on other substances. The recovery value to use can be set in the settings menu.

The KjelROC Analyzer can also handle %Protein, %N, %N+, mgN/ml, ml, mgN/kg, mgN/g, mgNH3/kg, mgN, mgN/l, mgN/100ml, gN/100ml, mgProtein, mgN/100g, gN/kg and %Rec. If %Protein or N%+ is selected it is further possible to define the applicable factor. The factors that are visible in this menu are defined in the settings file. To simplify it is possible to define a name for each factor that is used.

Step-by-Step

1. Press ▶ until desired Rack is displayed.
2. Make sure the correct Tube number is selected and displayed in the entry area to the right.
3. If the result presentation or factor does not match, press the corresponding ▶ until correct.
4. Key in the sample weight and press CONFIRM. The tube number will automatically increase with one.
5. Continue with next tube as in step one.
6. When ready press ⌂

6.5. ANALYSIS

Up to five different method buttons can be programmed. These five methods will be visible as five different buttons in the main menu and when selecting any of these you will reach the analysis menu. The different methods can contain different options such as time or volume based analysis, Kjeldahl or Direct Distillation analysis, amount of dosing etc.

However, the analysis menu will look and behave similar for all programs options.

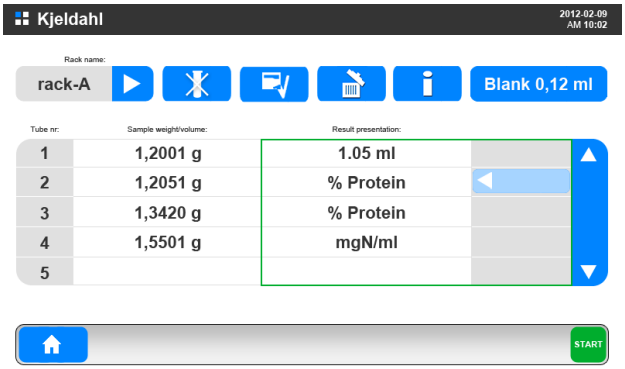

Fig. 7. The Analysis menu

The first row in the Analysis menu will give the possible options for this rack. From left to right you can select rack, exclude tube, complete rack, reset data, get detailed information and define your blank. Below you find a table with all tubes in the selected rack. Selection of tube is done by pressing the corresponding row in the selection area to the right.

It is also possible to toggle between sample name and entered volume/weight by pressing inside the “sample weight/volume” area. Please note that sample names can only be entered inside the OPSIS LiquidLINE Transfer Utility program.

Changing tube rack is done by pressing the  button. Only the Express rack and racks with previous weight entry will be visible. All results in the Express rack will be in ml, which makes it possible to run an analysis without prior weight entry. The express rack is also different compared to other racks since the results will not be stored in the data handling. The express rack is indented for quick analysis and for samples where there is no need for traceability.

It is possible to skip a previous entered weight entry by pressing the exclude button. This may be the case if the tube has broken or if for some other reason the sample is no longer valid. An excluded tube will get an “exclude” flag in the result file but will not be deleted from the system. The exclude tube option is not available in the express rack.

The completed rack option will move the rack into the data handling section. After a rack is moved to data handling, there is no possibility to run previously entered samples in this rack. After a rack has been completed, it is again empty for new weight entries. The completed rack option is not available in the express rack.

Since the express rack does not store any data in the data handling there will be no completed rack button in the express rack, instead it is possible to reset all data by selecting the trash bin. Please note that results in the express rack will not be stored after they have been moved into the trash.

The information button gives detailed information about the currently selected tube and tube results in a separate window. Select between three pages by pressing  at the page indicator. Go back to analysis view by pressing the BACK button.

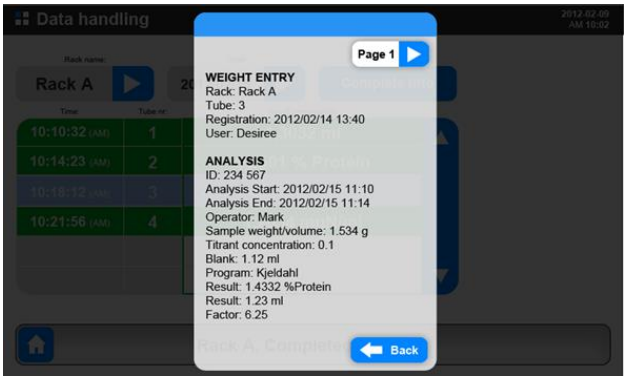

Fig. 8. Detailed Information View

The current blank value will be displayed inside this button. If an analysis with the result type “mlBlank” has been completed then the blank value will automatically be updated with that result. It is always possible to manually change the blank value by pressing this button and enter a new value. Please note that the Blank value is always reset to zero when the KjelROC Analyzer has been switched off.

Step-by-step

1. Press ▶ to select the rack with the samples to be analysed
2. If the displayed blank differs from the one just established, press the BLANK button and enter the correct value in the dialogue
3. Put a tube in place and make sure the correct tube number is highlighted. You can select another tube by pressing on the corresponding row. To scroll in the list you use ▲ and ▼ buttons
4. Start analysis by closing the Safety Door and pressing START (depending on settings the cycle can be started by just closing the door)
5. Reagents will be added automatically and distillation with simultaneous titration will continue until the pre-set end volume has been reached. The test tube will empty automatically when the analytical cycle is over (can be adjusted in your program).
6. Open the safety door and continue with next test tube
   Select the correct tube and press the exclude tube button if a tube cannot be used (e.g. broken during digestion). If for any reason several tubes in a rack cannot be processed, instead of pressing exclude button for each, just press the completed rack button after you have processed the other tubes.
7. When all tubes in the rack are processed, press ⌂

Any time during analysis, you can press ⌂ and continue to enter weights in the “Weight entry” view. Next tube can be analysed by selecting your program again.

6.6. DATA HANDLING

The Data handling menu is the menu where previous analysis results can be retrieved.

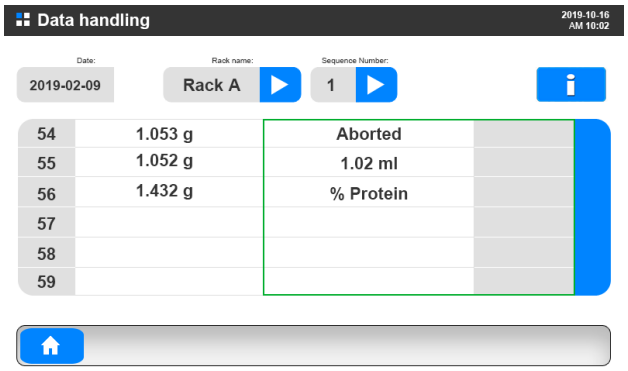

Fig. 9. Data handling menu

When entering data handling a date selection dialog will be shown. You select the desired date by pressing the corresponding year, month and date. If there are no results present for the select date then selection screen will remain, but with that date now removed from possible selections.

The Data handling menu has a header row with several options. From left to right you have the filters for tube rack and instance of rack (if same rack has been used more than once during that day). In addition, there is a detailed information button. Below you find a table with all tubes that matches the chosen filter. Selection of tube is done by pressing the corresponding row, in the selection area to the right.

The information button gives detailed information about the currently selected tube and tube results in a separate window. Select next page by pressing the arrow at the page indicator. Go back to the data handling view by pressing the back button.

Step-by-step

1. Select the date of interest in the date selection dialog
2. Select the rack and instance in the same way
3. The basic information for up to five tubes is displayed. By scrolling up / down information on the remaining tubes can be found
4. Press  when the marker is pointing on a specific tube and the below display will show all available information. By pressing  next page with information will appear. Press BACK to leave the information window

It is recommended to record stored data on a PC on a regular basis. Easiest is to use the OPSIS LiquidLINE Transfer Utility program described in chapter 6.8, Using Network Connection, on page 25.

To prevent slow handling or overfilling the memory, data is stored with a time limit and are thereafter automatically deleted. The default time limit is 1 month but this can be changed in the settings file.

6.7. MANUAL MODE

Manual mode is a possibility to rune several functions of your KjelROC Analyzer manually. Manual mode cannot be selected during an analysis.

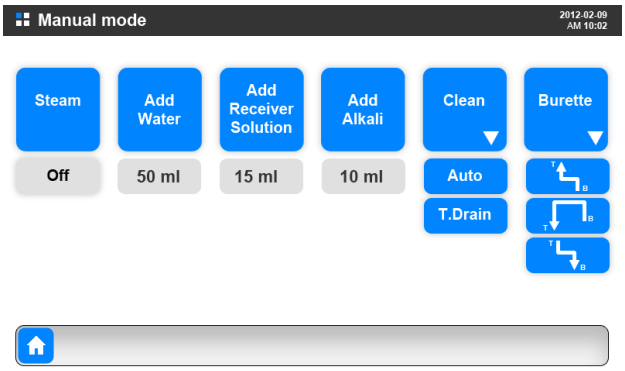

Fig. 10. Manual View

This menu is used to fill up reagent tubing and clean systems when changing reagents or after a longer time not in use. It is also used to check the dispensed volumes of water and alkali. For safety reasons all functions except Receiver solution and Burette requires a tube in place as well as the Safety Door in closed position.

- STEAM, one touch starts the steam production and the flow of cooling water. Condensed water into the Titration Vessel goes to drain, as the valve is open. You can stop steam at any time by selecting .
- ADD WATER, dispenses the pre-set volume into the test tube.
- ADD RECEIVER dispenses the pre-set volume into the Titration Vessel.
- ADD ALKALI, dispenses the pre-set volume into the test tube.
- CLEAN, gives two options. Auto (Auto Clean) and T.Drain (Tube Drain).
    - Auto Clean starts an automatic cleaning cycle. This function requires an empty Test tube and closed Safety Door as well as that Tube Drain is not set to off. Water is dispensed into the Test tube. Steam distillation is on for some minutes and thereafter the tube is drained. See also 8.1.2, Auto Clean Program at the end of the day on page 37.
    - Tube Drain starts a manual tube drain of the sample tube. This is practical to use at any time an analytical cycle has been interrupted and there is remaining waste in the sample tube.

- BURETTE has three options to move the burette piston. By pressing the buttons the burette piston will move until next touch or until it reaches the upper/lower end position. Also the three port valve will automatically open to ether the Titrant Storage Flask or the Titration vessel. This is helpful when changing the titrant or when removing bubbles that might develop inside the burette.
  -  , piston moves up. Burette is connected to the Storage Flask.
  -  , piston moves up. Burette is connected to the Titration Vessel.
  -  , piston moves down. Burette is connected to the Storage Flask.

6.8. USING NETWORK CONNECTION

The KjelROC Analyzer is equipped with an Ethernet Interface and supplied with a KjelROC wireless configured router. This simplifies transfer of weights to the instrument and for retrieval of results.

6.8.1. Network Setup

The KjelROC Analyzer can communicate to a PC network via the Ethernet Interface (RJ-45). Simply connect a network cable between the KjelROC Analyzer and your Ethernet network. The KjelROC Analyzer will connect and acquire an IP address automatically.

It is also possible to connect the supplied KjelROC Wireless router to the Ethernet Interface. Simply connect the supplied network cable between the KjelROC router and the KjelROC Analyzer. The instrument will connect and acquire an IP address automatically. It thereafter possible to access the KjelROC Analyzer via the KjelROC Wireless network (WiFi).

Please read the separate KjelROC Analyzer Network Connection Guide for further explanations, proposed set-up for your laboratory and OPSIS LiquidLINE supplied programs.

6.8.2. How to send Weight Entry to the KjelROC Analyzer

Use the OPSIS LiquidLINE Transfer Utility to send weights and volumes to the KjelROC Analyzer. These programs can be downloaded from internet or are on the supplied USB stick.

6.8.3. How to retrieve results from the KjelROC Analyzer

All results stored in data handling are also available for transfer to an external device such as an iPhone/iPad, PC or Mac. Use the OPSIS LiquidLINE Transfer utility to retrieve these results. Please note that the Transfer Utility for iPhone/iPad requires a Wireless KjelROC network as previously described.

6.8.4. The Settings File

A settings file is stored in your KjelROC Analyzer and this contains additional possibilities to customize your instrument. The settings file can be transferred to the KjelROC by using the Restore function in OPSIS LiquidLINE Transfer Utility program.

The following options are possible to customize in the settings file:

- Preferences with language, time format, date format and decimal vs dot for numbers. It is possible to change the default mane language (English) to other languages. Time format can be either as 18:00 or as AM/PM system.

- Possibility to start analysis automatically by closing safety door (default behaviour is that analysis start when user press START).

- Advanced Titration settings can be set. However, please consult your OPSIS LiquidLINE representative before modifying these settings.

- Amount of racks and their names to be used in the menus. Default is three racks but up to 20 racks can be defined, each with a unique name and from 1-99 tubes.

  

  Please note that space should not be used in rack names. It is also recommended to keep rack-a/b/c if the Transfer Utility Program is used.

- Possible results types to be used in the menus. All result types are available in the user interface by default. However, it is easy to remove those not desired from the menus.

- Four pre-defined factors for %Protein and %N+ are available in the KjelROC Analyzer. The default values and names of these can be changed.

- It is possible to change the name of each program that is used in the KjelROC Analyzer.

- It is possible to change the user names, password codes and their access levels (operator or full access). This is modified in the userdata.ini file.

6.9. SET-UP

The KjelROC Analyzer comes pre-set for all needed functions but sometimes there might be a need to change some of the default settings. The KjelROC Analyzer has three different settings;

- Settings views on the touch screen with basic adjustments for administrators and operators. This view will be explained in this section.
- A specific Managers menu with more detailed analytical adjustments. This menu is explained in the KjelROC Analyzer Managers Manual.
- A Settings file that can only be modified via a network connection, for complete control and adjustments. This settings file is described in 6.8.4, The Settings File, on page 25 and in the KjelROC Analyzer Network Connection Guide.

By using the above options, you will get an Analyzer tailor made for your operation. However, in most cases the set-up adjustments described in this section will be sufficient for complete control. To prevent mistakes all settings can be password protected.

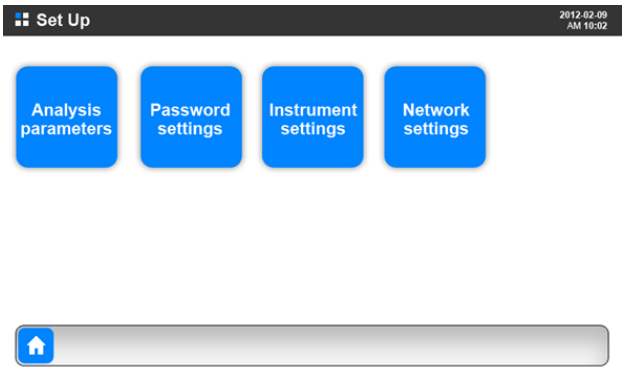

Fig. 11. Settings menu

You can adjust the Analysis parameters, Password, Instrument and Network settings from this menu.

6.9.1. Set-Up Analysis parameters

When entering Analysis Parameter view a selection dialog will be shown. You select among the four quick access programs and the currently selected “special” program. You will have to change the selected program in the main menu if you want to adjust another “special” program.

The Analysis Parameters view shows the values for the different methods that are available in the instrument. Each method can be configured as on or off, i.e. if they should be visible in the main menu.

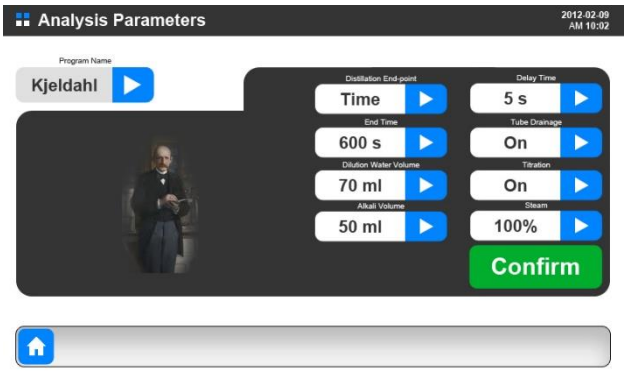

Fig. 12. Analysis Parameters Setting - Kjeldahl

Depending on analytical sequence (see below), the following parameters can be adjusted;

- Distillation Endpoint – time or volume;
  Default for a Kjeldahl sequence is end volume (100ml) which is adjusted with the outer level pin in the Titration Vessel, see adjustment of End point level pin in the KjelROC Analyzer Managers manual. When the liquid level during analysis reaches this pin, analysis is interrupted if the correct end point for the titration is fulfilled. However, before final stop steam distillation will continue for the time necessary to compensate for the titrant volume. This means that all samples independent on Nitrogen level will be distilled the same way and that the system (Splash head and Condenser) always are rinsed with the same amount of water.
  If Distillation Endpoint is set to time (0-999 seconds), distillation will stop at this time independent if the titration has reached end point or not.

- Dilution Water Volume;
  Enough water, to avoid a violent reaction when alkali later is dispensed, should be added. If dilution is done manually, the volume can be set to zero.

- Alkali Volume;
  Dependent on the concentration enough volume to neutralise the acid has to be dispensed. Note that the Auto clean sequence always should have Alkali volume set to zero.

- Delay Time;
  To allow the alkali and water to mix before steam distillation starts. A longer time, several minutes, is often used in combination with Dewardas Alloy when analysing fertilizers. Please see separate Application Note.

- Tube Drainage;
  If set to “on” the test tube will be automatically emptied after analysis. If the sample contains solid particles, e.g. soil or boiling beads it is recommended to switch of the tube drainage. Otherwise the valve might be blocked by particles.

- Titration;
  Can be set to ON, ON Low, D->T, D->T Low and OFF. ON is the standard titration algorithm and is recommended for most analyses.
  ON Low uses a titration algorithm with less aggressive dosing, more suitable for samples with very low levels of Nitrogen.
  D->T titration is separating the Distillation and Titration into two steps, the distillation will then first complete before the titration starts. This is also more suitable for samples with low levels of Nitrogen. D->T Low is using both separate Distillation and Titration as well as a lower speed on titration.

- Steam, (10-100%);
  In Kjeldahl 100% is recommended.

- Repeat Counter;
  This parameter is only available together with an Auto Blank sequence. The repeat counter will define how many blanks shall be repeated.

If any parameter is changed then press “Confirm” to save your new setting.

The instrument has four different types of analytical sequences, that can be set via the settings file.

Kjeldahl sequence
The Kjeldahl sequence is used to run a classical Kjeldahl analysis. Two Kjeldahl program sequences are supplied by default with the instrument (Kjeldahl and Dewarda) and up to five different Kjeldahl programs can be programmed.

Auto Blank sequence
The auto blank sequence allows several blanks to be run automatically after each other without user intervention. This saves time when starting the instrument and helps when establishing a correct blank value.

It is possible set the repeat counter, i.e. how many times will the blank be repeated.

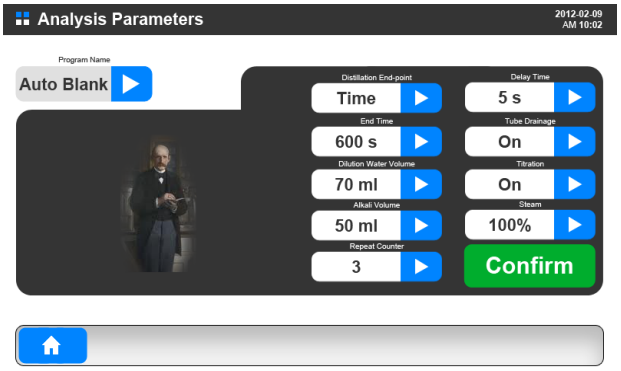

Fig. 13. Analysis Parameters Setting – Auto Blank

Auto Clean sequence

The sequence for the Auto Clean program can be adjusted in this menu. Enter the manual menu to start your Auto Clean program.

DD, Direct Distillation sequence

It is possible to use the instrument for direct distillation. Visible sample types, intercepts and slopes are defined in the settings file. For details, please check the corresponding Application Note.

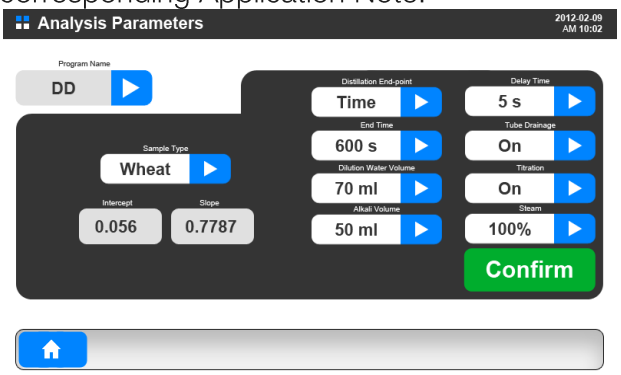

Fig. 14. Analysis Parameters Setting – DD

6.9.2. Set-Up Password

Fig. 15. Password Setup

- Select if login should be active or not

- Selection of name where you want to adjust access level or password. The actual name that is displayed can be changed in the settings file.

- Selection of access level (administrator or operator) can be done in this menu. The administrator has access to all functions in the instrument while the operator only can enter weight data and run analysis. Please also note than an operator can only see their own analysis information and not weights and analysis results done by other operators.

If any parameter is changed then press “Confirm” to save your new setting.

6.9.3. Set-Up Instrument settings

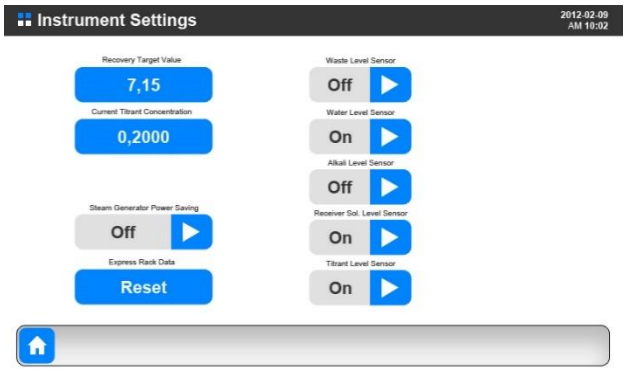

Fig. 16. Instrument Settings

This menu allows set-up of specific parameters in the instrument.

- Recovery target value for Rec analysis allows entering the target value for a standard Recovery analysis.

- Titrant concentration can be defined in this menu.

- The use of level sensors can be enabled or disabled in this menu.

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6.9.4. Set-Up Network settings

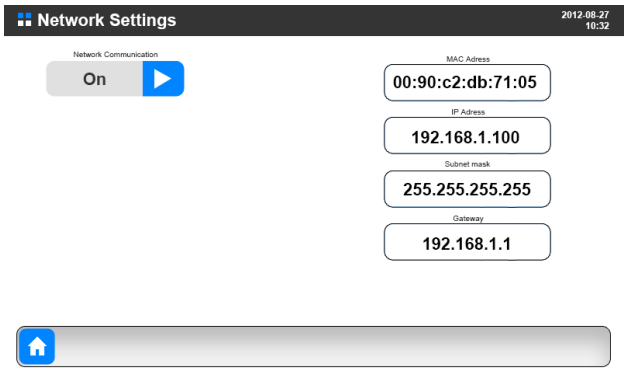


Fig. 17. Network Settings

The Network settings view display the current IP address acquired in the KjelROC Analyzer.

If the IP address is 0.0.0.0 then please try to re-establish a new connection by selecting the on/off button. If that is not successful, then switch off and thereafter switch on your KjelROC Analyzer. If this does not work then please follow the instructions in the KjelROC Network Connection Guide to troubleshoot your connection.

7. GLP and Performance Tracking

To meet Good Laboratory Practice (GLP) demands there are several ways to verify the function of a KjelROC Analyzer. For a complete instrument check a Performance Verification, described below, is recommended.

Often the request to calibrate the instrument is raised. However, as the Kjeldahl method is the reference method used to calibrate other techniques for nitrogen/protein determination e.g. infrared. A KjelROC Analyzer used for Kjeldahl analysis never can or should be calibrated. However, certain modules of the instrument can be calibrated which is described in this section.

7.1. Calibration - verifying Modules

The built in burette is factory calibrated and can be checked. The software is standard in the KjelROC, but an optional kit is needed. Please note that a calibration check according to ISO 8655 also demands extensive control of temperature and humidity. The procedure to use the KjelROC software for burette calibration is described in the Managers Manual.

The built in reagent pumps (water, receiver and alkali) are calibrated/checked before delivery and can at any time be re-checked in an easy way. All steps are described in the Managers Manual.

7.2. Performance Tracking – Verifying the Instrument

As most KjelROC Analyzers are used for Kjeldahl determination this paper will give some hints how to optimise the instrument for this analysis. Often by just carrying out a couple of distillations, it is easy to pinpoint were the reason for not accurate results can be found. These are not always related to the instrument but can be found in other parts of the analytical chain!

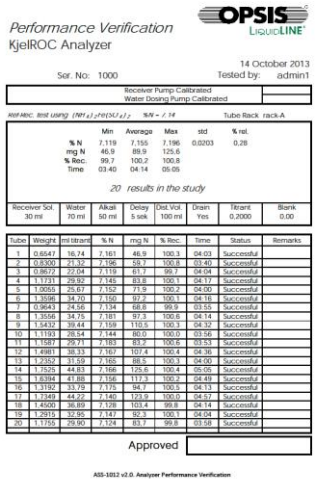
It is well known that. Independent of sample type. The final substance of interest in a Kjeldahl determination is Ammonium Sulphate, $(NH_4)_2SO_4$. This is during the distillation step converted to Ammonia, NH_3, which after it has been collected in a receiver is titrated. The titrant is an acid with well-defined concentration. The amount found Nitrogen is often calculated to percent protein by multiplying with a sample dependent standard factor.

$$\%\ Nitrogen = \frac{(ml\ titrant\ - ml\ blank) * N\ * 14.007}{g\ sample\ * 10}$$

N = the titrant concentration in normality. If a two proton acid is used (e.g. H_2SO_4) N=2 the molarity 14.007 is the atomic weight of Nitrogen*

7.2.1. Recovery test (added product test)

As an option, it is possible to order a KjelROC Analyzer with Performance Test at Factory. This means that the KjelROC Analyzer will be tested using the result presentation “Ref-Rec” during standard Kjeldahl distillation. All settings as when analysing normal samples but to make cleaning easier a less concentrated alkali used. At least six samples, covering the range 10 – 200 mg Nitrogen, are used in the test. A printed form from this is delivered together with the instrument and the individual results are as well stored in the KjelROC memory for future use. A typical form is shown below;



Performance Verification
KjelROC Analyzer

OPSIS LIQUIDLINE

14 October 2013
Ser. No: 1000 Tested by: admin1

Receiver Pump Calibrated
Water Dosing Pump Calibrated

Ref-Rec: Test using: $(NH_4)_2Fe(SO_4)_2$ %N = 7.14 Tube Rack rack-A

	Min	Average	Max	std	% rel.
% N	7.119	7.155	7.196	0.0203	0.28
mg N	46.9	88.9	125.6		
% Rec.	99.7	100.2	100.8		
Time	03:40	04:14	05:05		

20 results in the study

Receiver Sol. 30 ml	Water 70 ml	Alkali 50 ml	Delay 5 sek	Dist.Vol. 100 ml	Drain Yes	Titrant 0.2000	Blank 0.00

Tube	Weight	ml titrant	% N	mg N	% Rec.	Time	Status	Remarks
1	0.6547	16.74	7.161	46.9	100.3	04:03	Successful	
2	0.8300	21.32	7.196	59.7	100.8	03:40	Successful	
3	0.8672	22.04	7.119	61.7	99.7	04:04	Successful	
4	1.1731	29.92	7.145	83.8	100.1	04:17	Successful	
5	1.0055	25.67	7.152	71.9	100.2	04:00	Successful	
6	1.3595	34.70	7.150	97.2	100.1	04:16	Successful	
7	0.9643	24.56	7.134	68.8	99.9	03:55	Successful	
8	1.3556	34.75	7.181	97.3	100.6	04:14	Successful	
9	1.5432	39.44	7.159	110.5	100.3	04:32	Successful	
10	1.1193	28.54	7.144	80.0	100.0	03:56	Successful	
11	1.1587	29.71	7.183	83.7	100.6	03:53	Successful	
12	1.4981	38.33	7.167	107.4	100.4	04:36	Successful	
13	1.2352	31.59	7.165	88.5	100.3	04:00	Successful	
14	1.7529	44.83	7.166	125.6	100.4	05:05	Successful	
15	1.6394	41.88	7.156	117.3	100.2	04:49	Successful	
16	1.3192	33.79	7.175	94.7	100.5	04:13	Successful	
17	1.7343	44.22	7.140	123.9	100.0	04:57	Successful	
18	1.4500	36.89	7.128	103.4	99.8	04:14	Successful	
19	1.2915	32.90	7.147	92.3	100.1	04:04	Successful	
20	1.1755	29.90	7.124	83.7	99.8	03:58	Successful	

Approved

ASS-1012 v2.0. Analyzer Performance Verification

Any Ammonium salt can be used to test the function. However, to minimise errors when weighing in, Ammonium iron (II) sulphate, or Mohr's Salt, with the formula $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ is recommended. The Nitrogen level for this substance is configured in the KjelROC and used to calculate the recovery when “Ref-Rec” is selected.
(If any other Ammonium salt or even a standard sample is used when including the Digestion step the simple “Rec” result presentation should be used instead. The theoretical/expected value should be entered in the Settings menu of your KjelROC Analyzer)

7.2.2. Verifying the function

It is recommended to carry out recovery tests now and then to make sure the results from the KjelROC are still accurate. Depending on the Nitrogen content of the samples analysed and the titrant concentration used, other Nitrogen ranges might be actual than the one described in the attached form. However, the test will inform about the instrument status.

- Results from the recovery test are on target and the relative error is low. (<1%)
    - The KjelROC is in order and eventual errors should be found elsewhere. Check the sample preparation, the balance and the digestion procedure. (Temperature, salt/acid ratio, time)

- Results from the recovery test are below or above target but the relative error is still low.
    - The titrant concentration should be checked.

- Results from the recovery test are below target and the relative error is too high.
    - Look for leakages. Check the tube adapter and the tubing between splash head and condenser

- Results from the recovery test are now and then above target and the relative error is too high.
    - Clean the system carefully as most likely deposits in the splash head now and then go into the titration vessel. If the problem continues the splash head should be replaced.

8. Maintenance and Service

8.1. Daily maintenance and cleaning

It is recommended to follow below hints for daily care of your instrument, to avoid break-downs of your instrument or faulty results.

8.1.1. Clean spillages

- Remove the Drip tray (A) and clean tray with warm water and a soft cloth (B).
- Remove the Service door protecting the Splash head (C).
- Release the Safety door stop/hatch by turning the holding screw counter clockwise (D)
- Move the Safety door upwards to remove it from your KjelROC Analyzer (E).

- Clean the Safety door with a wet cloth (F).

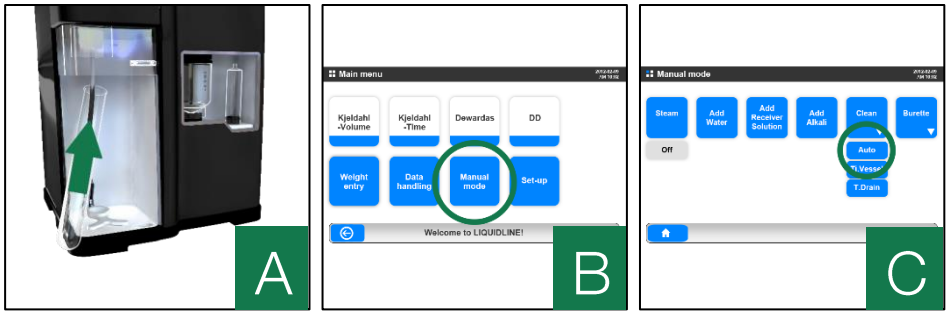
If there are cracks in the Safety door then these might be a result of leakage between the sample tube and the adapter. Please check your tubes and adapter as well as take care when inserting your sample tubes.

- Check the area around the Adapter and Splash head holder and use a wet cloth to wipe any spillages (G). Check also below the drip tray.

- Use a wet cloth to wipe of any general spillages from the instrument. Alkali and Hydrochloric acid are particular important to clean due to their corroding effects.

- Put back Safety and Service door into the KjelROC Analyzer. Take care to move back the Safety door stop (D).

8.1.2. Auto Clean Program at the end of the day



It is recommended to run the Auto Clean Program at the end of the working day to avoid crystallization of boric acid in the titration vessel during the night.

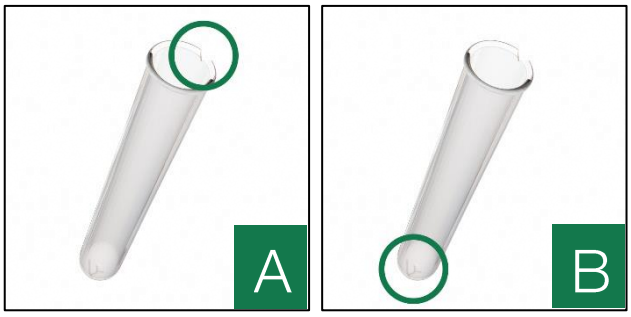
- Insert an empty 250 ml sample tube into the KjelROC Analyzer (A).

- Select MANUAL MODE (B) and then AUTO CLEAN (C).

- After addition of water into the test tube a steam cycle is started. It will continue until the overflow pin inside the Receiver vessel is reached. The test tube and Receiver vessel are thereafter drained.

- Switch off the instrument and close the water tap after Cleaning.

8.1.3. Check Digestion Tubes



- Check the rims of the digestion tubes for uneven surfaces (A). In particular, look for cracks and chips. A damage in this area can cause leakage and loss of nitrogen during analysis.

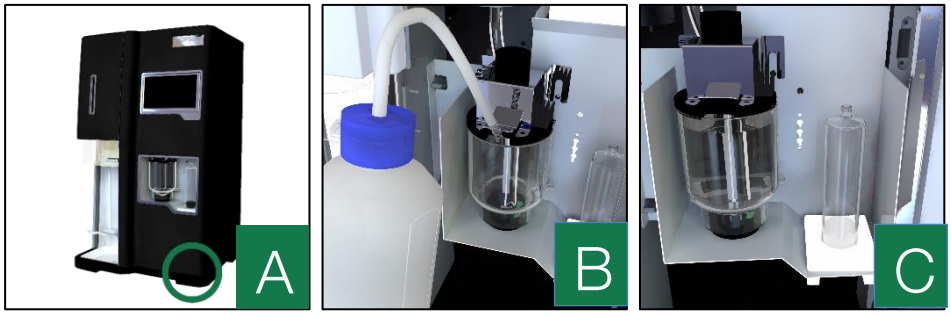
- Check the bottom of the digestion tubes for cracks (B). Most tubes becomes discoloured or “white” in the bottom, which normally is no problem. However, if cracks are discovered then these samples tubes should be discarded.

It is recommended to use OPSIS LiquidLINE tubes. If other tubes are used, take care that they follow the same dimensions (inside diameter, length of tubes) and tolerances as original tubes to avoid leakages or cracks.

8.2. Monthly Maintenance and Cleaning

Once a month an extended user maintenance should be performed. Please follow these instructions to ensure an instrument that will work and perform as expected over time.

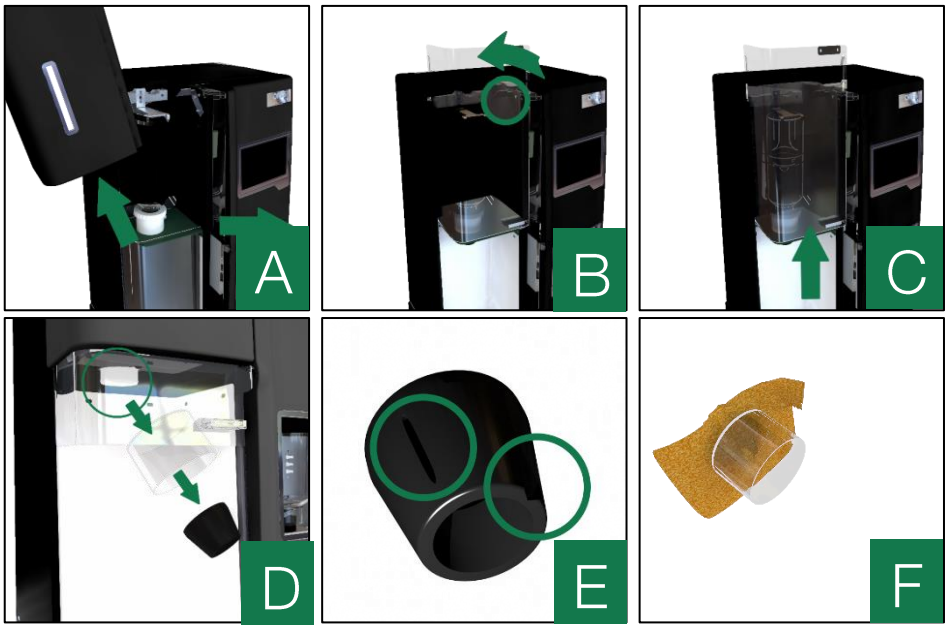
8.2.1. Rinse Receiver Vessel



It is recommended to clean the receiver vessel at regular intervals. This is easily done by adding distilled water in the vessel during the night.

- Switch off the KjelROC Analyzer (A).
- Add distilled water into the receiver vessel (B).
- Leave the vessel with water during the night (C).
- Switch on the KjelROC Analyzer. The receiver vessel will automatically be emptied when switching on the instrument.

8.2.2. Check Rubber Adapter and Clean Protection Cover



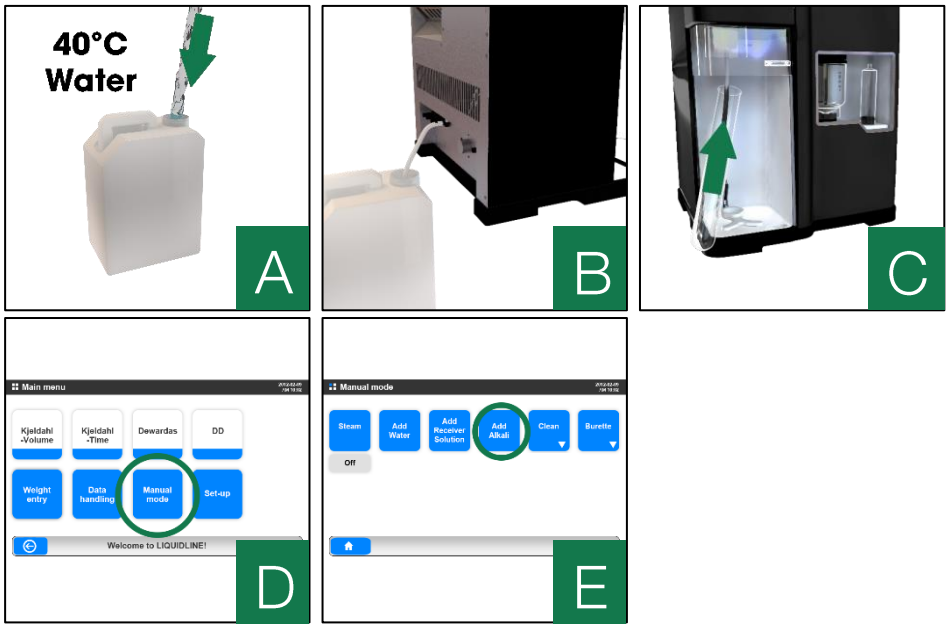
A leaking or damage Tube Cone Adapter will affect your results and also increases the risk for damages to your instrument. It is therefore important to remove the adapter at regular intervals for inspection.

- Remove the Service and Safety Door following the same procedure as in the daily maintenance (A)(B)(C).
- Remove the adapter by pulling it down, also remove the transparent cover (D).
- Inspect the Adapter rubber and notice if there are any scratches or cracks (E). Check also that the adapter has not lost its softness. If something is noted then exchange the adapter against a new Tube Cone Adapter.
- Clean the transparent cover with a wet cloth or wash it with warm water (F).
- Take care when returning the adapter and cover to the instrument. Some force might be necessary in order to put the Tube Cone Adapter in the correct position.

  Hint, put the Tube Cone Adapter in hot water during two minutes. It will make the Adapter softer and easier to mount.

- Put back Safety and Service door into the KjelROC Analyzer. Take care to move back the Safety door stop and lock it by turning the screw clockwise (A)(B)(C).

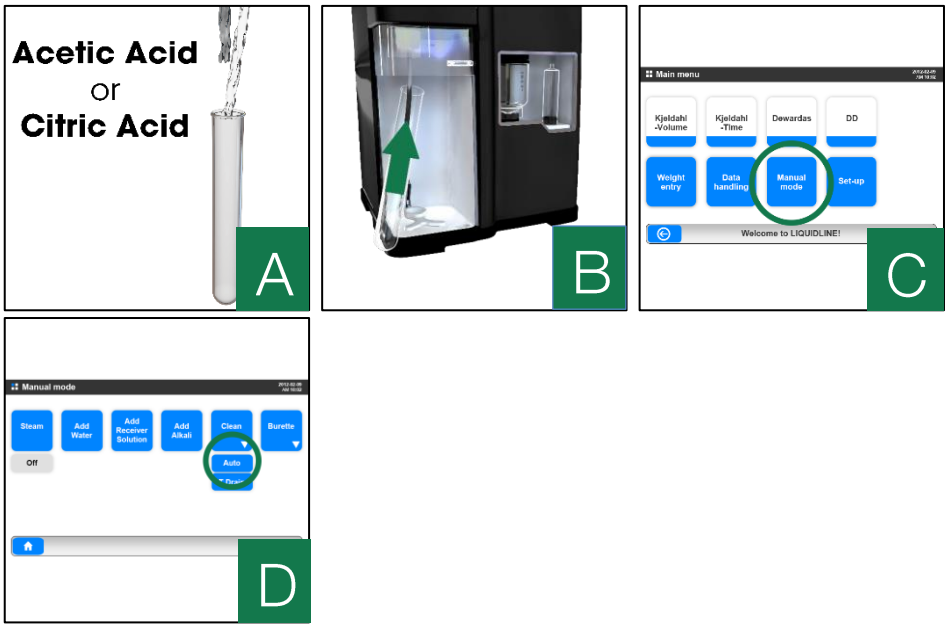
8.2.3. Clean Alkali Pump



It is necessary to clean the Alkali pump at regular intervals to ensure that your pump will last over time.

- Fill distilled warm water, approximately 40°C, in a tank (A).
- Connect the KjelROC Analyzer to the tank by placing the tube from the Alkali inlet inside the tank (B).
- Insert an empty 250 ml sample tube in the KjelROC Analyzer (C).
- Select MANUAL MODE (D) and then ADD ALKALI (E) to pump water into the sample tube. Several key presses might be necessary in order to fill a tube.
- Repeat until at least one, preferably two or three, tube(s) have been filled with water. Exchange sample tube whenever it is full.
- Remove the warm water tank. Select ADD ALKALI two to three times to ensure that all water is removed from the system.
- Reconnect your Alkali tank and select ADD ALKALI until you see Alkali added into the sample tube.

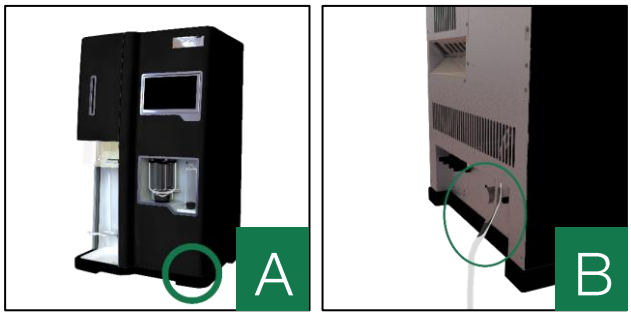
8.2.4. Clean Splash head



The Splash head might become dirty during analysis, which normally is not a problem for the instrument or your analysis results. However, regular check your instrument performance and if carry-over effects are noticed then cleaning of the Splash head is recommended.

- Mix 25 ml of distilled water with 25 ml of concentrated acetic acid (CH_3COOH) in an empty 250 ml sample tube (A). You can also mix a solution of 25 ml distilled water and 1g of citric acid.
- Insert the sample tube inside the KjelROC Analyzer and close the Safety door (B).
- Select MANUAL MODE (C) and then AUTO CLEAN (D). Leave steam on until it automatically stops after 600 seconds (default setting).
- Replace the tube with a new sample tube containing a new mixture of water and acetic acid. Repeat this at least three times.
- Replace the tube with a new sample tube containing only water and run steam to clean.

8.2.5. Rinse Steam Generator



It is necessary to rinse the steam Generator at regular intervals to remove any residues or deposits inside.

- Switch off the KjelROC Analyzer (A)
- Open Steam Generator Drain valve to empty the Steam Generator (B).
- Switch on the KjelROC Analyzer and let the pump refill the generator.

8.3. Monthly Performance Tracking and Analytical Adjustments

Continue to use the “Ref-Rec” results type and running analysis with the same test substance at regular intervals. This ensures that your instrument continues to perform satisfactorily. See separate chapter 7 GLP and Performance Tracking on page 33.

If necessary, adjust the analytical parameters by following the below instructions.

8.3.1. Adjusting the Titration Vessel Level Pins

The KjelROC Analyzer is equipped with level pins to ensure flexible adjustment of the analysis cycle. Sometimes it might be necessary to adjust these pins for better performance.

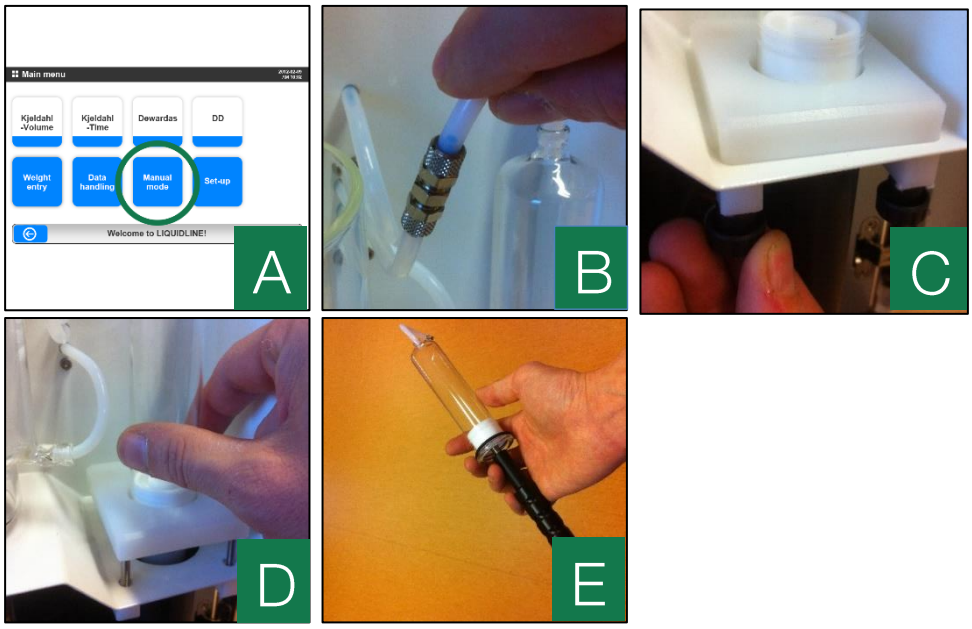
Fig. 18. Titration Level Pins

The KjelROC Analyzer has the following Level Pins;

- Overflow Pin: When liquid reaches this level pin then the overflow warning will set in and the Analysis will be stopped. By adjusting this pin the timing of the overflow can be adjusted.
- Ground Level Pin: This is used as a reference by the instrument. This pin should be as low as possible (as close as possible to the glass).
- Receiver Solution Level Pin: This is the reference used when calibrating the amount of receiver solution that will be used. Adjust this pin while operating the instrument in the Managers menu (see separate manual)
- End Point Pin: Adjust this pin to achieve a correct end-point volume. Adjust this pin while operating the instrument in the Managers menu (see separate manual)

Take care when moving the pins up and down so that the Teflon tubing does not cover the end of the pin, this might result in less reliable signal.

8.3.2. Changing Titrant in the KjelROC Analyzer



Sometimes it is necessary to exchange the titration used in the KjelROC Analyzer. Please follow this procedure.

- Go into the MANUAL MENU (A) and select the button to remove as much as possible of the titrant in the burette.
- Remove the Titrant tank and select the button to move the burette piston down.
- Select and let the piston move up a short distance (approx. 5-10 mm)
- Switch-off the KjelROC Analyzer
- Open the front door to access the Burette and un-lock the Teflon tubing going from the burette to the burette valve (B).
- Remove the four screws below the burette (C).
- Remove the burette by turning the Burette glass anti-clockwise (D).
- Remove the piston and remove the last titrant by using the supplied Burette tool (E).
- Change the Titration tank to your desired solution
- Remount the Burette by following the above steps in the opposite direction. I.e, insert the piston into the Burette glass again, re-mount the Burette by turning clockwise, attach the four screws and re-connect the tube from the Burette valve.

- Switch-on the KjelROC Analyzer

- Please do not forget to access the Instrument Settings and change your titration concentration to the new value. Refill the burette by using the manual menu.

8.3.3. Further Adjustments

The KjelROC Analyzer is prepared with a specific Managers menu for calibration of pumps, calibration of burette, selection of indicator and adjusting of end-point, changing time/date and factory restore. Please see the Manager Manual for a description of the features in this menu.

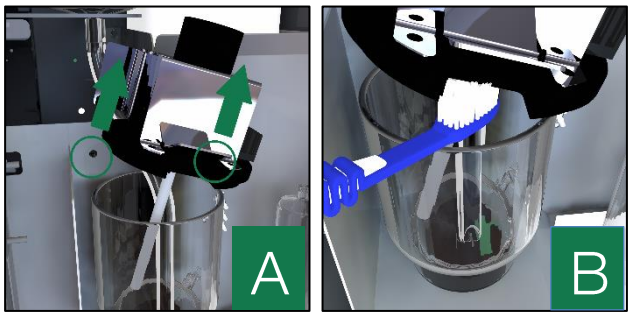
It is also possible to adjust all parameters of the instrument via settings file. In addition of the options in the set-up menu this includes setting date format, set amount of numbers in results, adjusting behaviour of Start and other buttons etc. Please see the KjelROC Analyzer Network Connection Guide or contact your distributor for further instructions how to adjust these settings.

8.4. Additional Maintenance

Every two or three months a more complete user maintenance should be performed. Please consult your OPSIS LiquidLINE representative and follow these instructions to ensure an instrument that will work and perform as expected.

Never open any rear plates without prior approval by an authorized service engineer. Always disconnect the KjelROC Analyzer from mains power before any maintenance. Please always also ensure that you have necessary components available before starting to remove or detach any parts from the KjelROC Analyzer.

8.4.1. Clean Receiver Vessel

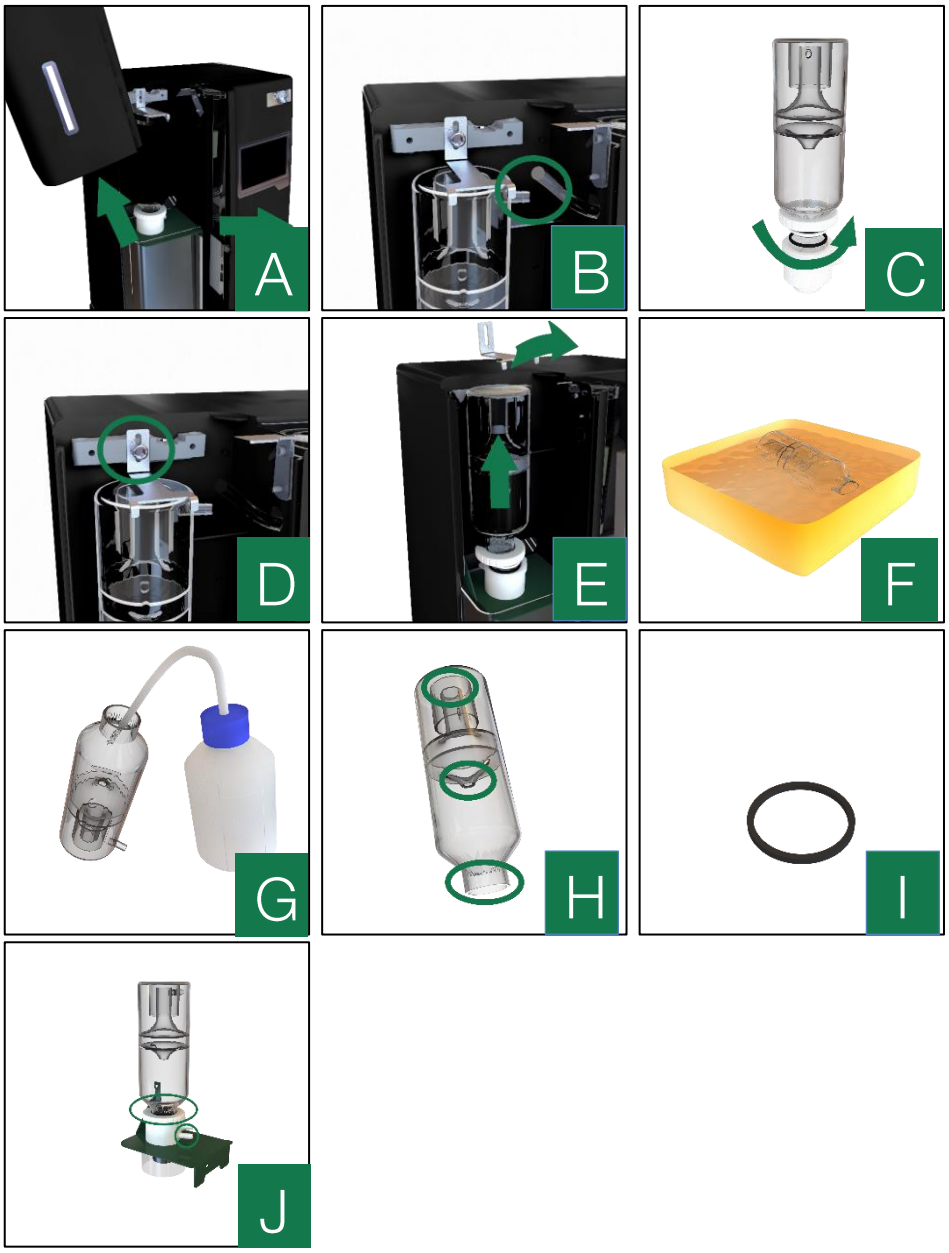


Normally there is no need for additional cleaning of the Receiver vessel if the monthly routine is followed. However, in some cases residues might appear below the top cover or at the upper part of the vessel. If this is the case a more complete cleaning is recommended.

- Remove the Receiver vessel cover by loosening the two screws holding its place (A).
- Carefully move the cover upwards to the right. Take care of the tube from the condenser and the Level sensor cables.

  Clean the Receiver vessel by using a small brush such as a toothbrush (B). Clean the area below the cover and as well the inside.
- Reattach the Receiver vessel cover (A).
- Reconfirm that the level pins are in the correct position. See also chapter 8.3.1, Adjusting the Titration Vessel Level Pins.

8.4.2. Control and Washing of Splash head



The Splash head is the area above the sample tube that ensures that only nitrogen is released to the receiver vessel. In addition to the monthly cleaning it might also be necessary with a more complete cleaning as well as exchange of this part.

It is recommended that the OPSIS LiquidLINE maintenance program is followed for this part. When inspecting and replacing, please follow these instructions:

- Disconnect the Power Supply.
- Remove the Service door protecting the Splash head (A).
- Cut the tube between the Splash head and condenser. Remove the tube from the KjelROC Analyzer (B).
- Remove the Splash head carefully from the holder by turning the Top cover counter clockwise (C). Remove the stop above the Splash head by turning the screw counter clockwise (D). Move the Splash head upwards (E).
- Prepare a container with a solution of 50% distilled water and 50% concentrated acetic acid (CH_3COOH). A solution with distilled water and citric acid can also be used. Place the Splash head inside the container and leave it there for 5-10 minutes (F). If necessary spray distilled water inside for additional cleaning (G).
- Inspect the Splash head (H)
    - Check for signs of glass that glass has disappeared inside the Splash head. If this is the case then there is no need for immediate replacement. However, this is a sign that Splash head should be replaced soon.
    - Check for signs of leakage at the top area. If there are any holes then Splash head should be replaced.
    - Check for signs of a rim in the bottom area (the part that is covered by the holder). This might indicate an increased risk for leakage in the future. No immediate replacement but if leakage is detected then Splash head should be replaced.
- Put the Splash head back inside the instrument.
    - Put a new tube from the condenser, through the pinch valve.
    - Put the Splash head in place and secure it with the Top cover and the stop above the Splash head (C)(D).

      A new sealing ring should be used when reattaching to avoid leakages (I).

    - Secure the tube from the condenser to the Splash head.
    - Put back the Service door (A)
- Confirm that there is no leakage (J) after reattaching the Splash head by running a couple of blank analyses. You will need to remove the Service door to observe any leakage.

8.4.3. Clean Steam Generator

The frequency how often you will need to clean the Steam generator depends on the environment around the laboratory. In particular the quality of water which should always be distilled, with few particles and at a neutral pH level. Higher hardness of the water will require more frequent cleaning, since more salt deposits will build-up inside the generator.

Always use distilled water for the Steam Generator. However, please note that a certain conductivity is necessary in order for the level pins to work correctly.

- Disconnect the Power Supply.
- Empty the Steam generator by opening the drain valve on the back (A).
- Prepare a solution of 100g Citric Acid in 800ml hot distilled water (B).
- Fill-up the generator by connecting your citric solution to the water inlet. Switch on the KjelROC and let the pump fill the steam generator with the citric solution.
- Leave the solution in the KjelROC Analyzer overnight.
- Open the drain valve and empty the steam generator.
- Connect the Power supply and switch on the KjelROC Analyzer. The Analyzer will now refill the steam generator with water automatically. Switch off the instrument and empty the steam generator. Repeat two to three times.

8.5. Maintenance Programs By OPSIS LiquidLINE

The KjelROC Analyzer has a built-in service system to inform when it is time for service. Intervals will depend on amount of analyses and elapsed time since last service.
To keep your instrument in good shape it is wise to call for service technician when the warning is displayed.

Some parts are recommended to be replaced on a regular basis to assure proper operation. Please consult your authorized service technician. Original parts from OPSIS LiquidLINE should always be used.

9. Installation

Normally we recommend that the installation and first training of the laboratory personal should be done by and authorized OPSIS LiquidLINE technician. However, by carefully following the instructions below it also can be done by others.

9.1. Installation Requirements

- Laboratory bench 0.6 m free space
- Electrical supply 230V +/- 10%
- Power Consumption 2200W
- Water Supply: Cold water tap and drain within 1.5 m from the instrument, 1.75 l/min tap water at 20°C

9.2. Unpacking and assembly

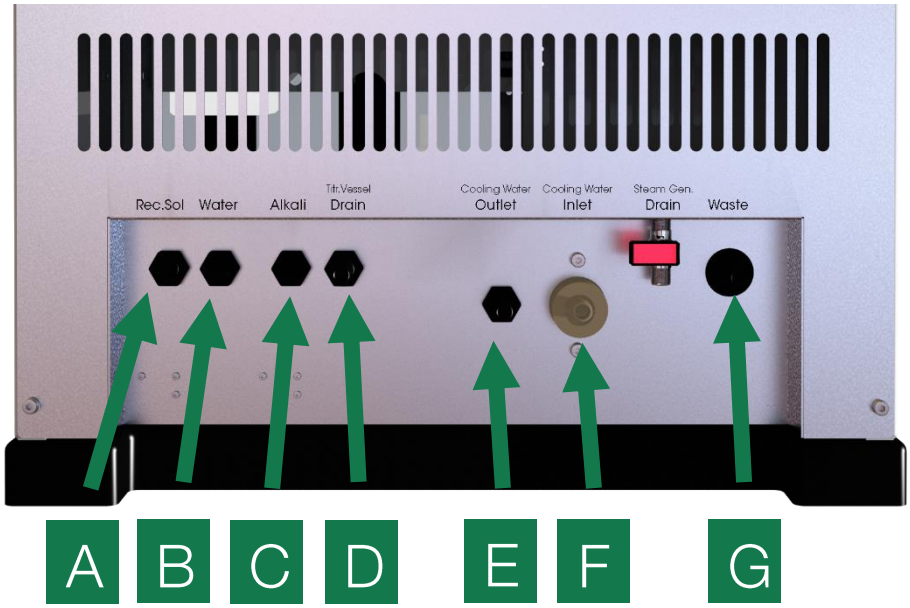
Carefully remove the packing material and make sure there are no transport damages. If so you should directly contact your OPSIS LiquidLINE representative.

To avoid breakage during transport there will be packaging material inside the KjelROC Analyzer. Also remove this.

Depending on choice, either the delivered reagent tanks or locally supplied should be connected. Make sure the tube with the level sensor is adjusted to fit the height of the tank. If too long, shorten it.

If local regulations permit waste (alkaline residues) in the drain there is no need for a waste tank. If so the waste level sensor should be deactivated.

9.3. CONNECTING THE KJELROC ANALYZER



- Connect All tubes to the KjelROC Analyzer
  - Add Titrant Solution into the supplied Titrant Tank and place it inside the Analyzer
  - Add Receiver Solution to Receiver Solution Tank. Connect tube to back plate of Analyzer (A).
  - Add distilled Water to a tank with Water. Connect tube to back plate of Analyzer (B).
  - Add Alkali to the Alkali Tank. Connect tube to back plate of Analyzer (C).

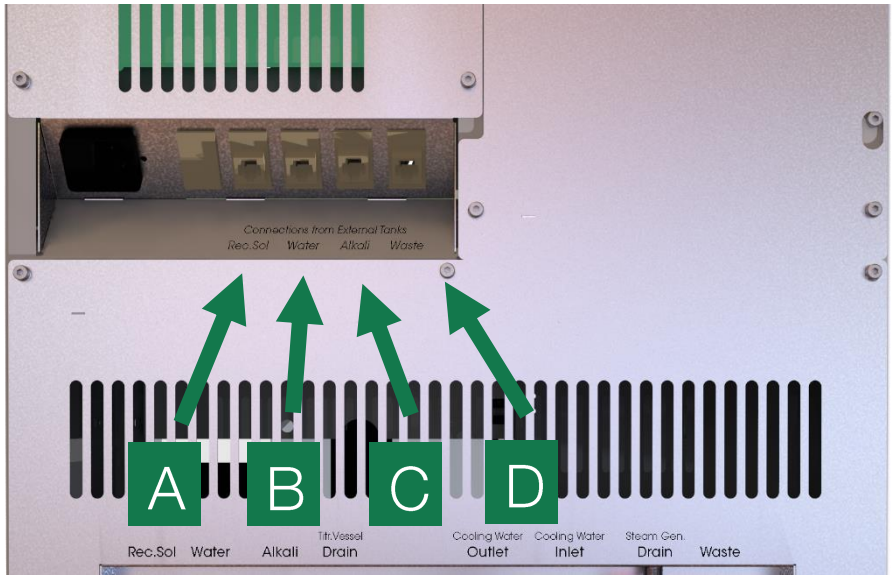
    Please note, Water tank should have the supplied Water filter. The filter looks like a small sponge and should be added at the inlet of the tube that is inserted into the water tank.

  - Connect the first Waste tube to the back plate of the Analyzer (D).
  - Connect the second Waste tube to back plate of the Analyzer (G).

    Ensure that waste is going to the drain or a waste tank. Make sure that outlet from the Waste is in air and not in any liquid (if so then you will get problem with the automatic Tube Drain).

  - Ensure that cooling water IN is be connected to a cold water tap (F).
  - Ensure that cooling water OUT is going to the drain (E).

- Connect the Power to the KjelROC Analyzer
- If included in the package, connect the signal from the External Level Pins to the RJ-connectors. If not used then switch off these sensors in the software (MENU->SETTINGS->INSTRUMENT SETTINGS)



  - Connect the Receiver Solution signal to the KjelROC Analyzer (A).
  - Connect the Water signal to the KjelROC Analyzer (B).
  - Connect the Alkali signal to the KjelROC Analyzer (C).
  - Connect the signal from the Waste to the KjelROC Analyzer (D).
- Ensure that there is an empty tube inserted into the Analyzer

9.4. Starting the KjelROC Analyzer

- Switch on the KjelROC Analyzer and login (default code 1111 can be used)
- Fill burette with Titrant Solution and ensure that titrant solution is inside the tube to the Receiver Vessel. Remove any Air bubbles. (MENU->MANUAL->BURETTE)
- Check Receiver Pump (MENU->MANUAL->RECEIVER PUMP). You should get receiver solution into the Receiver Vessel. Repeat until success.
- Check Water pump (MENU->MANUAL->WATER PUMP). You should get water into the Tube. Repeat until success.
- Check the Alkali pump (MENU->MANUAL->ALKALI). You should get Alkali into the Tube. Repeat until success.

- If you plan to use the automatic Tube Drain then please confirm this function (MENU->MANUAL->TUBE DRAIN). Make sure that the tube is empty before proceeding to the next step.

It is important that the outlet of the waste tube is in the air and not in any liquid, since that might prevent the Auto Drain to function correctly.

- Run an Auto Blank (MENU-> AUTO BLANK) if you have automatic Tube Drain enabled, otherwise run at least three manual blanks.

- Check that programs and user access is set-up according to your requirements (i.e., alkali dosing, program names, login codes etc). Switch-off instrument and re-start to confirm that changes are registered into the system.

Run Performance Check

Run Performance Check together with the customer. See chapter 7.2, Performance Tracking – Verifying the Instrument, on page 33.

- Run Auto blank to establish a stable blank. Repeat until the blank is stable.
- Run 5x Ammonium Iron Sulphate ($(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ is recommended) . Register result to send back to OPSIS LiquidLINE. Check that result is satisfactory.
- Demonstrate how to verify the analytical performance of their instrument. Give the customer the Application Instruction LA1000 – which instructs the user how to prepare reagents.

Run Daily Maintenance

Run Daily Maintenance and Monthly Maintenance together with the customer. See chapter 8, Maintenance and Service, on page 36.

- Demonstrate how to maintain their instrument

It is critical to demonstrate and show the customer how to prepare reagents and how to maintain their instrument. Failure in this area is the most common cause for after sales support.

9.5. Connection to a Network

Please study the KjelROC Analyzer Connection Guide before setting-up the network connection.

9.5.1. Connect using Wireless

- Place the KjelROC Router near the Analyzer and connect the supplied Ethernet cable between the Analyzer and the router. Connect the router to a power outlet.
- Switch on the KjelROC Analyzer
- Check that the Analyzer connects to the KjelROC network (MENU->SETTINGS->NETWORK SETTINGS). If everything is fine then there will be an IP address for the Analyzer.
- You are now ready to transfer files between the Analyzer and a computer, using Wireless.

9.5.2. Connect using Ethernet

- Connect an Ethernet cable between your PC network and the KjelROC Analyzer. Please note that the KjelROC Analyzer expects connection to a DHCP router. Direct connection between a computer and the Analyzer will not work.
- Switch on the KjelROC Analyzer
- Check that the Analyzer connects to the KjelROC network (MENU->SETTINGS->NETWORK SETTINGS). If everything is fine then there will be an IP address for the Analyzer.
- You are now ready to transfer files between the Analyzer and a computer, using Ethernet.

It is recommended that you make a copy on your computer of settings.ini, service.ini, userdata.ini and wifi.ini (for later support issues). You can also make a copy of your reference analysis data (to be sent to OPSIS LiquidLINE) in the Ref-Rec folder. You can use the backup feature of the OPSIS LiquidLINE Transfer Utility program.

It is recommended to make a backup copy of the system before leaving the installation. This can simplify future support.

Depending on your preferences; it is possible to copy the settings.ini file from the settings folder to the backup folder on the Analyzer. This means that when you select “Factory Restore” in the Managers menu the instrument will NOT be restored to factory defaults but rather installation defaults.

10. Technical Data

Operating Temperature	5˚C - 40˚C
Relative humidity	max 80 %
Power Supply	190-240 VAC, 50-60 Hz, 10A
Power consumption	2200 W
Dimensions (WxHxD)	430 x 700 x 330 mm
Weight	30 kg

11. Declarations and Requirements

11.1. Declaration of conformity

OPSIS

CE

Declaration of Conformity

Identification of apparatus:	KjelROC Distillation Unit KD-200 KjelROC Distillation Unit KD-210 KjelROC Analyzer KD-310
Model/type:	Kjeldahl Analyzer
Manufacturer:	OPSIS AB Box 244, SE–244 02 Furulund, Sweden Phone: +46 46 72 25 00

The undersigned hereby declares that the above-referenced product, to which this declaration relates, is in conformity with the provisions of:

- Council Directive 2014/30/EU (February 26, 2014) on Electromagnetic compatibility (EMC),
- Council Directive 2014/35/EU (February 26, 2014) on Electrical Safety: low-voltage electrical equipment,
- Council Directive 2006/42/EC (June 9, 2006) on Safety of Machinery,
- Council Directive 2011/65/EU (June 8, 2011) on Restriction of the use of hazardous substances (RoHS 2).

The below harmonised standard specifications have been applied:

Safety:

ANSI/ISA-61010-1 (November 5, 2012) Safety Requirements for Electrical Equipment for Measurement, Control, and Laboratory Use - Part 1: General Requirements

Electromagnetic Compatibility:

Emission: EN 61000-6-3 (2007)

Immunity: EN 61000-6-2 (2005)

October 8, 2018

Svante Wallin

President OPSIS AB

OPSIS AB Box 244, SE-244 02 Furulund, Sweden Phone +46 46 72 25 00 E-mail info@opsis.se www.opsis.se

11.2. FCC REQUIREMENTS (FOR USA AND CANADA)

English:

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to both Part 15 of the FCC Rules and the radio interference regulations of the Canadian Department of Communications. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications.

Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

> Caution – Exposure to Radio-Frequency Radiation.
>
> To comply with FCC RF exposure compliance requirements, for mobile configurations, a separation distance of at least 20 cm must be maintained between the antenna of this device and all persons.
>
> This device must not be co-located or operating in conjunction with any other antenna or transmitter.

Français:

Cet appareil a été testé et s'est avéré conforme aux limites prévues pour les appareils numériques de classe A et à la partie 15 des réglementations FCC ainsi qu'à la réglementation des interférences radio du Canadian Département of Communications. Ces limites sont destinées à fournir une protection adéquate contre les interférences néfastes lorsque l'appareil est utilisé dans un environnement commercial.

Cet appareil génère, utilise et peut irradier une énergie à fréquence radioélectrique, il est en outre susceptible d'engendrer des interférences avec les communications radio, s'il n'est pas installé et utilisé conformément aux instructions du mode d'emploi. L'utilisation de cet appareil dans les zones résidentielles peut causer des interférences néfastes, auquel cas l'exploitant sera amené à prendre les dispositions utiles pour pallier aux interférences à ses propres frais.

KjelROC Analyzer
Managers Manual
Version F

1. Contents

2. Introduction

The KjelROC Analyzer is designed to facilitate Kjeldahl analysis and Protein determination in a lab. Care has been taken to allow full traceability in the system and therefore there are many levels of access into the instrument. The KjelROC Analyzer differentiates between the following scenarios;

2.1.1. Login disabled

There is no login into the instrument. This makes it easy to use the instrument but unfortunately it also means that there is no traceability of operator activity. When login is disabled there is still a code required to enter the settings menu.

2.1.2. Login enabled - Operator access

Login is enabled but the user has only operator level privileges. This allows the user to enter weights (unless this has been disabled via the settings file) and run an analysis. The operator can also retrieve his/her own results but is unable to see other operator results. The operator cannot access the settings menu.

2.1.3. Login enabled - Administrator access

Login is enabled and the user has administration level privileges. In addition to the operator privileges this level also allows access to the settings menu and it is possible to retrieve all results on the instrument.

2.1.4. Managers Menu

There is a Managers menu which will be described in this manual. This menu is for calibration or adjustment of the instrument. The Managers menu is independent of access level and uses its own login code.

Please note that to access the Manager menu the login needs to be enabled (the login code for managers menu is via the login screen).

3. Safety

The KjelROC Analyzer is protecting the operator from any hazardous actions. e.g. no steam ore alkali can be dispensed without having a tube in place and the protecting door completely closed. However, as the methods described often involve the use of hot corrosive chemicals every user should read the Safety Instructions or be instructed by the laboratory manager. Below you can find the instruction in all EU languages.

3.1. User Safety

The instrument may only be used by laboratory personnel and other persons who have knowledge and/or experience of doing chemical analysis and dealing with reagents.

Applications not mentioned in this document are improper. In particular, it is forbidden to use the instrument in the following instances:

- Use of the instrument that require ex-protected instruments
- Use of Samples or Reagents which can explode or inflame

It shall be noted that:

- Modifications and upgrades to the instrument shall only be carried out by authorized service personnel
- Service Menus in the Instrument is only to be used by authorized Service Personnel

3.2. Safety Symbols


General Hazard


Corrosive acid


Crushing hazard


Electrical shock hazard


Hot Surface

Explanations used in this manual


Important, Please Note


Please Note, Protection Glasses is recommended


Please Note, Gloves should be used

3.3. Product Safety Systems

The instrument is designed and built in accordance with state-of-the-art technology. Nevertheless, risks to users, property, and environment can arise when the instrument is used carelessly or improperly. If the equipment is not used in a manner specified by this document, the protection provided by the equipment may be impaired.

3.3.1. Maintenance and Service

The Operator is responsible for ensuring that recommended daily and monthly user maintenance is performed on the Instrument. Failure to do so might impair the functionality and/or shorten the lifespan of the instrument.

The operator is responsible to schedule regular Maintenance with authorized service personnel only. Only OPSIS LiquidLINE Spare parts should be used in the instrument.

3.3.2. Safety Sensors

The instrument is equipped with several safety systems

- A Sensor will identify if a tube is placed in the holder. No automatic operation is allowed without tube in place
- The Safety door (protecting the tube) is monitored and no analysis or manual operation is allowed with the door open. Analysis will stop if opened during analysis
- The small door protecting the Splash head is monitored and no analysis or manual operation is allowed with the door open.


Please note that the large door is not monitored in order to allow access to Titration Area during analysis. However, caution should be taken if running analysis with open door.

4. Managers Menu

4.1. Accessing the Managers menu

The Managers menu is accessed via the main login screen, see Fig. 1. Login to the Managers Menu. Please note that the login screen is only visible if the login function is enabled in the system.

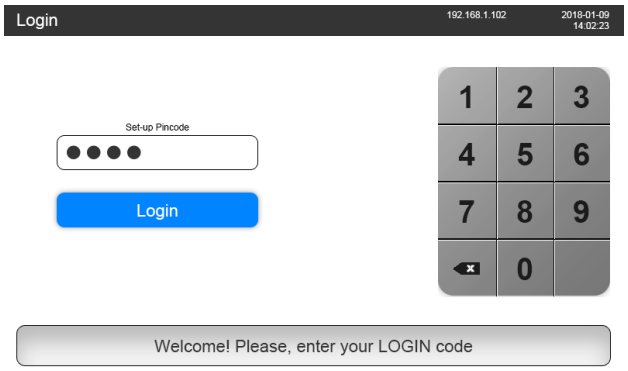


Fig. 1. Login to the Managers Menu

The code used to enter the Managers menu is 1 2 3 1 .

4.2. The Managers menu

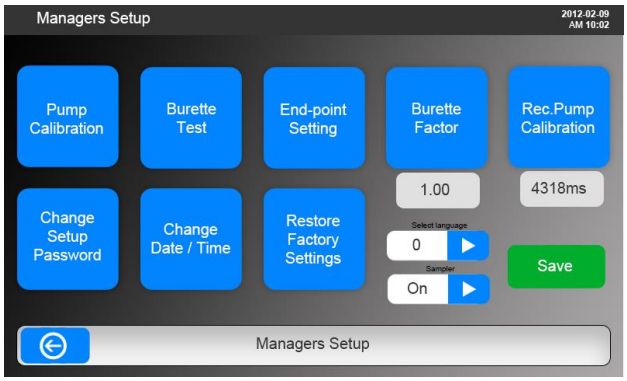


Fig. 2. Managers menu

The Managers menu consists of;

Water Pump Calibration (dosing of water) – in case you want to calibrate the dosing given by the Water pump.

Burette Test and adjustment of Burette Factor – It is not recommended to adjust the Burette dosing. However, in case you are required to do this by your quality assurance system, then these two menus allows this adjustment

End-point setting – It is possible to adjust the end-point of the KjelROC Analyzer as well as changing the indicator used with the instrument

Receiver Pump Calibration (dosing of receiver solution – in case you would like to calibrate the dosing given by the Receiver Solution pump.

Change Setup Password – change the default password for entering the settings menu

Change Date/Time – change the date and/or time in the instrument

Restore Factory Settings – reload the default settings file into the instrument. Your results will not be affected.

Select Language – change the text language in the KjelROC Analyzer. Please consult OPSIS LiquidLINE to find the correct language code (Zero is always used for English Language).

Select Sampler on/off – this will allow a user to disable Sampler functionality. This might be necessary in case maintenance is due on the sampler while more samples needs to be run on the Analyzer.

4.3. Water Pump Calibration

The Water pump calibration menu will guide you through the calibration of the water pump.

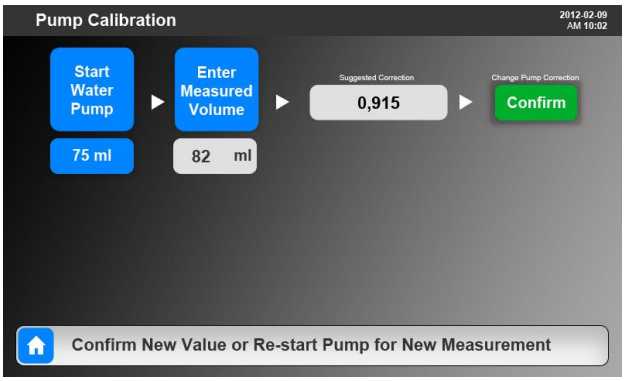


Fig. 3. Pump Calibration Menu

Follow these steps to calibrate your pump;

1. Insert an empty tube into the KjelROC Analyzer
2. If you want a different amount of dosing water (default is 70 ml) then please press the “ml” button to change. Otherwise press the “Start Water Pump” button and dosing will come into the tube.
3. Measure the dose amount and enter value by pressing on the second “ml” button.
4. Press the “Enter Measured Volume” button and the KjelROC Analyzer will suggest a calibration of your pump. If you agree with the suggestion then press Confirm. If you do not want any change then press the Home button instead.

4.4. Burette Test

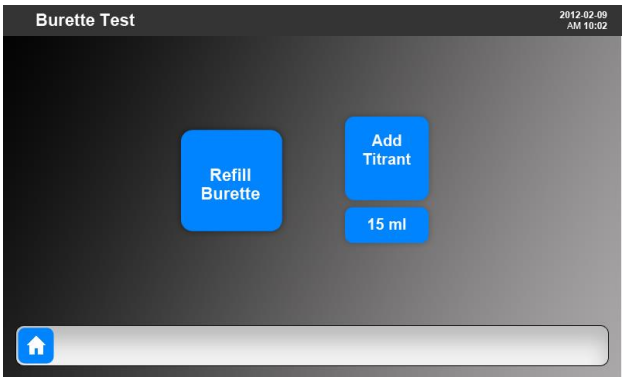


Fig. 4.Burette Test Menu

The calibration of the Burette requires that the connection between the burette and the Receiver vessel is opened. Place the tube coming from the burette into your selected vessel.

There are two functions in this menu;

- Refill Burette: Will force the burette to go to end position and therefore fill itself with liquid.
- Add Titrant: Will dose the indicated amount of titrant into your vessel. The amount of titrant can be adjusted by pressing corresponding button.

4.5. Burette Factor

By pressing the Burette Factor button in the Managers menu it is possible to change the factor used for dosing Titrant.

We do not recommend changing of the Burette factor since it can severely impact your results. This feature should only be used by a qualified chemist.

4.6. END POINT SETTING

4.6.1. Setting the End Point

The End-point settings menu allows change of end-point as well as change of indicator solution. Default indicator is a change between green and red but other colors are possible. The menu consist of two areas, the selection area to the right and the titration/checking area to the left.

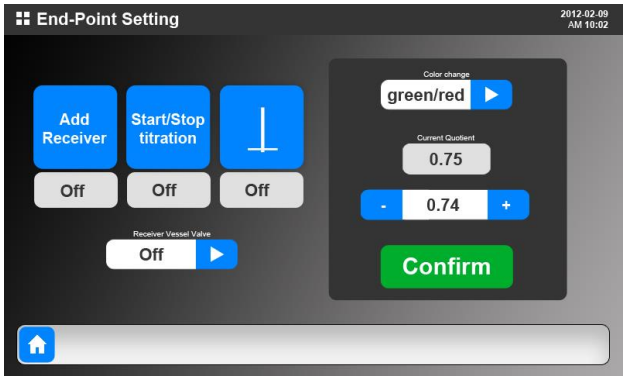


Fig. 5. End Point Settings menu

Follow these steps to adjust your End-point and/or your indicator solution;

1. In the selection area. Please select desired indicator solution in the upper right corner. You can toggle between different pre-configured options.
2. The current quota is given in the selection area and below this you are allowed to adjust the color quota. In this example (green/red) a higher value would mean a slightly more green color for the end-point and a lower value would mean a slightly more red color for the end-point.
3. An easy check of the color quota can be performed if you have the possibility to fill the receiver vessel with Boric acid and very little Alkali (too much will mean that excessive amount of Titrant will be needed). Please ensure that receiver vessel valve is on so that the vessel is closed. Add you solution of Boric acid and Alkali.
4. Press the Start/Stop titration the KjelROC Analyzer will dose Titrant in the receiver vessel until the end-point has been reached.
5. Check that the end-point is to your satisfaction. If not, repeat steps 2 to 4.

It might be difficult to finally verify the color quota in this menu. It is recommended to run several analyses prior to deciding if your new color quota is to your satisfaction.

4.6.2. Adjusting the End-Point Volume/Level

The KjelROC Analyzer has the four Level Pins in the Titration Vessel;

1. Overflow Pin: When liquid reaches this level pin then the overflow warning will set in and the Analysis will be stopped. By adjusting this pin the timing of the overflow can be adjusted.
2. Ground Level Pin: This is used as a reference by the instrument. This pin should be as low as possible (as close as possible to the glass).
3. Receiver Solution Level Pin: This is the reference used when calibrating the amount of receiver solution that will be used.
4. End Point Pin: Adjust this pin to achieve a correct end-point volume.

Step to Step to adjust the End-point

- Enter Managers menu and the End-Point Settings View
- Close the titration vessel (lower left button)
- Measure 100 ml of water (typical volume in Kjeldahl) and pour this into the Titration Vessel
- Adjust the End Point Pin (number 4 in picture above) until the pin is at the same level as the water
- When done, open the titration vessel again

4.7. Receiver Pump Calibration

The receiver pump will automatically calibrate against the level pin in the Receiver vessel when this button is selected. The amount of milliseconds that it will take until receiver solution reaches the Receiver Level Pin will automatically be used as the correct dosing value.

Adjust the amount of receiver solution by moving the receiver level pin (pin number 3 in picture below) up or down.

Make sure that the receiver solution always covers the outlet from the cooler.

4.8. Change Setup Password

By pressing this button you can change the default Setup password to something else. Default password for the settings menu is 1234.

4.9. Change Date/Time

The Date/Time menu allows you to change the date and time in the instrument.

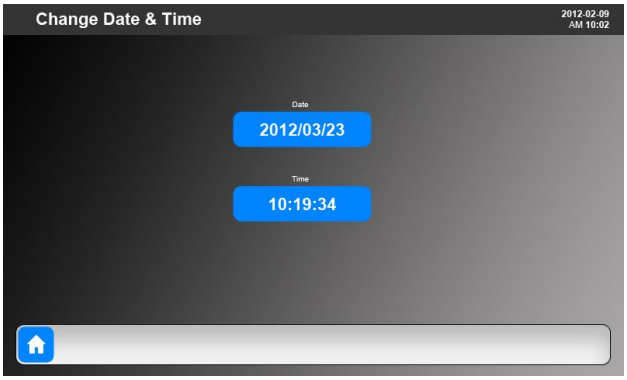


Fig. 6. Date/Time menu

4.10. Restore Factory Settings

The Restore Factory settings button allows you to restore the KjelROC Analyzer to the default settings. Unless your service engineer has updated the default settings file to your default installation then this will be factory settings. The restore factory settings will not erase any analytical data, user data or wireless settings.

Please note that after pressing this button you should log out to ensure that the factory settings file is restored. The instrument will thereafter reboot itself with the new settings file.

The Restore Factory Settings will only restore the settings file in the instrument. Results, user information, service information and wireless will remain intact. Please consult your technician in case a full restore is required.

4.11. Change Language

It is possible to change the language in all menus. The selected number will refer to the numbers that are used inside the language folder on the KjelROC. Zero will refer to language0.txt, one will refer to language1.txt etc. Up to 9 different languages can reside simultaneously in the KjelROC Analyzer.

4.12. Sampler Enable/Disable

It is possible to disable and enable the sampler in the software. This is intended for situations when an Autosampler is used and it is necessary to disable all sampler functionality (sampler waiting for service etc).

It is not recommended to have sampler enabled in the software, without any Sampler present.

5. References

5.1. Login to Managers Menu

To enter Managers menu: Please press  in the login screen. If login is disabled then you need to enable this first.

5.2. Login to Settings Menu

To enter the settings menu: Default code is 1234.

5.3. Login via the Network

To enter the KjelROC Analyzer via the Ethernet interface: Use “admin” as user and “admin” as password. Use “operator” and “operator” for network login as an operator.

Application Guide Kjeldahl Determination

Using KjelROC Digestion Systems and the KjelROC Analyzer

As the Kjeldahl method involves use of highly corrosive and hazardous chemicals at high temperature it is strongly recommended to use protective glasses and gloves. The safety section in the KjelROC Operation Manual should be read before starting any analysis work.

Introduction

Since the seventies Block Digestion followed by Steam Distillation in the same test tube has more or less become a standard in the Kjeldahl analyses. Many different application hints are available and often a combination can be the best. Dependent on sample type and reason for doing the analysis must decide which application to follow. This Guide is describing the method in a general way and will help to use the KjelROC Digestion systems in combination with the KjelROC Analyzer. To benefit from all features it is recommended to read the different KjelROC User Manuals. Here details e.g. wireless and the possibilities to integrate with LIMS are described. Also more sample specific applications are available as “KjelROC Quick Guides”.

A ground and well homogenised sample is weighed and put into the Test Tube. Catalyst Tablet containing salt and a catalyst as well as concentrated sulphuric acid is added making sure the acid is wetting the same before putting the tube into the Digestor. Amount of sample and the needed time for digestion can vary a lot dependent on sample type. However, all protein in the sample is during the digestion converted to ammonium sulphate.

After cooling the Test Tube with the digested sample is put into the KjelROC Analyzer. Dilution water and alkali is dispensed into the tube and thereafter steam distillation starts. The liberated ammonia is collected in a receiver solution (often 1% boric acid) and titrated with an acid during the distillation. The added titrant volume in combination with the concentration and earlier recorded sample weight allows the Analyzer to calculate the result and present it on the touch screen. After completed analysis the test tube is automatically drained and the KjelROC Analyzer is ready for next sample.

Reagents

For digestion

- Kjeldahl Tablets, different types are available see LA 1001"The importance of the catalyst"
- Sulphuric acid, concentrated p.a. quality
- Alkali, ≈20% to be used in the Scrubber bottle no.2

For proper work liquids should be added to the Scrubber Flasks, 800 ml of tap water in the left flask (no 1) and a mixture of 400 ml water plus 400 ml alkali (30-40%) plus 5 ml of BTB (bromothymol blue) indicator (100 mg in 100 ml methanol) in the second. As long as the liquid in flask no 2 is alkaline the air coming out from the Scrubber will be odourless. The pH of the liquid can easily be checked by just adding a small amount of the mixed indicator used for the receiver solution. The number of batches that can be analysed before changing the liquids depends on the suction effect applied as well as the volume added acid. During "normal" conditions the contents in the flasks should be replaced after digestion of 60-80 tubes. The color of the solution in flask no 2 will turn from blue to yellow when it is time to change.

The Scrubber motor is not affected by eventual acidic gases. However, the stink from evaporated gases if the outlet tube from the Scrubber is not placed inside a fume cupboard is neither pleasant nor healthy.

For distillation

- Distilled or deionised Water for dilution and steam production. If the tap water is free from Nitrogen it can be used. However, cleaning of the Steam Generator has to be done on a frequent basis if the water contains calcium or other minerals building up deposits.
- Alkali, 30 – 40% Sodium Hydroxide
- Titrant acid, HCl (or H_2SO_4) with a well-defined concentration e.g. 0.1000 or 0.2000 Mol/L The concentration used in the KjelROC Analyzer is the Normality which if HCl is used is the same as the Mol/L but two times this value if H_2SO_4 is used.

Often the titrant can be prepared from ampoules.

If the instructions for dilution are followed the accuracy normally is enough for routine work.

Sometimes when a large volume at low price is desired the titrant is prepared out from concentrated, constant boiling, hydrochloric acid. This method requires careful standardisation of the acid, often by using sodium carbonate. The procedure can be found in chemical hand books or on the Webb.

Ready-made titrant with certificated concentration is often a good choice for accurate and convenient work.

- Receiver solution, 1% boric acid with mixed indicators.
- Ready-made receiver solutions are sometimes available but often it is more economical to prepare it in the laboratory, if so please follow the below instruction.

Preparation of Receiver Solution with mixed Indicators

10L is prepared as follows;

1. Dissolve 100 g boric acid in approximately 1 L boiling water. Add about 7 L more water and let cool.
2. Solve 100 mg bromocresol green and 70 mg methyl red in 170 ml methanol (or ethanol)
3. Pour the indicator solution into the boric acid solution and make up to 10 L with water.
4. Take 25 ml of the receiver solution in a beaker or E-flask and add 100 ml water. If the colour turns slightly green or just neutral grey the solution is OK. If still red add weak alkali (≈0.1M) until it turns grey. Calculate the volume needed for all 10 L. After eventual alkali addition the colour should be checked again. The colour must never stay red after addition of water as it is impossible to know how far from the equilibrium it is and this will influence the result.A blank value between 0.02 to 0.10 ml is optimal.

Dependent of workload it might be better to prepare a larger volume as the time to check and the eventual adjustment of colour with alkali will be the same.

The shelf time for the prepared receiver solution depends on the temperature in the laboratory. If repeated blank distillations start to vary it is often a signal to discharge the old liquid, clean the storage tank and prepare new.

Sample preparation and weighing

Sampling and preparation of the sample prior to analysis is important for the result. Dry samples should be ground, the finer the better. Semi solid samples, like meat, can be minced using a kitchen mixer or a proper homogenizer.

Weigh the sample using an analytical balance, if liquid samples use a pipette or measuring cylinder. Note the sample weight/volume in a form or directly key it into a computer for later transfer to the KjelROC Analyzer. During weighing the result presentation is selected. To make it simple it is recommended to only have the actual activated. If LIMS is used the easiest might be to just select the titrant volume in ml as result, the calculations probably already are installed in the LIMS.

A high sample weight is often better as it is giving a more representative result. However, the reagents used must match the nitrogen level. If the titrant concentration is 0.2000 Mol/L it covers the nitrogen range 10 – 200 mg with a good precision. This is routine levels in e.g. a laboratory analysing raw materials as grain, soya and fish meal. For samples with lower nitrogen levels a weaker titrant is recommended. Please see the table at the end of this Application Guide.

The KjelROC burette volume is 50ml and if necessary it makes refill during the analysis.

As high fat content samples consume more sulphuric acid during the digestion, either a lower sample weight or a higher dosing of the acid is needed.

One or several blanks, all chemicals used for digestion but no sample, often is put on a random place in the Rack. Dependent on GLP used this is done for every Rack, once a day or only when a new batch chemicals is begun.

Digestion

Put the weighed sample into the Test Tube. Add the required amount salt/catalyst mixture. For convenient and reproducible conditions often Kjeldahl Tablets are used. The salt, most common potassium sulphate K_2SO_4, is added to rise the boiling temperature and thereby shorten the digestion time. Different catalysts like cupper, selenium or titanium are used to speed up the reaction. The most efficient Mercury is nowadays banned in most countries due to environmental rules. In Opsis LA 1001, the efficiency of different Kjeldahl Tablets was compared. No significant difference could be seen and when environmental influence is of importance the Missouri tablets with 0.3% cupper (order no.KT-211A) are recommended.

Add the sulphuric acid H_2SO_4 and make sure the sample is wet by the acid by gently swirling the tube before putting it back into the Tube Rack. For dry samples like grain, feed mixes and raw materials as fish meal 10 to 12 ml acid is often enough. If the sample has a high fat content more acid is necessary as fat will consume some during the digestion. If the digest mixture directly after completed digestion, when still hot, is a liquid it indicates that the added acid has been enough. However, if the digest when raised from the Digestor looks like a solid cake, more acid is needed for this type of sample. Another reason can be that too much suction has been used. The Scrubber / water aspirator should always be adjusted to a minimum after the initial ten minutes.

Some recommend the use of hydrogen peroxide, 30% H_2O_2, to speed up the reaction and also reduce the foaming some sample types might create. However, the time saved is often not more than what the extra addition takes. Also the handling of this chemical can be troublesome as the reaction with many samples is quite violent. Foaming problems can often be overcome by using slightly more acid or start at a lower temperature.

Dependent on the Digestor alternative available (KjelROC Digestor Advanced with Motor Lift or Auto with or without Manual Stand) the tubes are connected to the Exhaust and loaded into the Digestor and the operation started /stopped as described in the actual Instruction Manual.

Before starting a digestion the Scrubber or water aspirator always should be adjusted to full capacity. After the first ten minutes the exhaust capacity should be adjusted to a minimum but still high enough to prevent gas to escape. This is to minimise the acid consumption. If too much acid is evaporated the salt concentration will be too high and there is a great risk for Nitrogen losses. Only if the sample contains a lot of liquid the exhaust should be fully on for a longer time. Please refer to the Quick Guide for the specific sample.

Many samples can be digested starting directly with a pre-heated Digestor e.g. with a temperature of 420°C. Some needs more attention. All Opsis Digestors come with pre-set programmes for different sample types. Water is an example where the first step is at a lower temperature to avoid violent bumping during the reduction of the start volume, approximately 100 ml. Thereafter the temperature is raised to the final level for the true digestion. To collect evaporated water an optional flask, available from Opsis, should be connected between Exhaust and Scrubber.

The time needed to complete the digestion can vary a lot dependent on sample type, salt/acid ratio and the catalyst used. Some claim that the digest should be completely clear others that an internal standard, often an amino acid e.g. glycine or lysine, must reach the theoretical result. As many samples can be completed earlier than a pure amino acid routine work can be done more efficient by testing the Nitrogen / Protein level of the actual samples after different times e.g. 45, 60, 75 and 90 minutes. The time where all the samples of interest have reached a stable level is the optional time. To give a margin of extra ten minutes can be wise. Sometimes it is impossible to obtain a “clean digest”. E.g. for samples like soil or when tablets with titanium oxide are used as they contain insoluble matters.

On all Opsis LiquidLINE Digestors temperature and time can easily be set by the user to fit any individual need.

Distillation including simultaneous titration and calculation of the result

Note: After digestion the test tubes with hot digest has to cool for at least ten minutes to avoid violent reaction during water dilution and alkali addition.

The distillation step is compared with the digestion very simple as all samples can be handled the same. Also the distillation, including the titration and calculation, is easy to confirm by performing a recovery test using an ammonium salt as standard. Please refer to KjelROC Analyzer Operation Manual chapter 7.2.1 “Recovery test”.

When switching on the KjelROC Analyzer it automatically prepares for analysis. Dependent on settings the operator has to log-in before selecting the function. The pre-set programmes are displayed.

Note: If new titrant is used the concentration has to be entered as described in KjelROC Analyzer Operation Manual chapter 5-9-3 Set-Up Instrument settings

If any “Refill Alarms” are displayed, fill up the corresponding storage tank. If an alarm is activated during distillation normally the Rack can be completed before action is taken.

If the instrument has not been used for a while it is recommended to run a couple of blanks. The easiest way is to select “Auto blank”. Three, or as many as pre-set, blanks will be processed. It is quite normal that the first blank will differ but when the Analyzer is warm and all tubes are filled with fresh reagents the blank values will stabilise. The last blank value will be stored and used in the coming calculations. If preferred the average of all or selected blanks can be entered.

The real blank, all digestion chemicals but no sample, is normally analysed together with the other samples. If the chemicals used do not affect the blank many laboratories save labour by just using the “auto blank”, only dilution water and alkali in the test tube during distillation.

When good quality chemicals are used no difference between the two blank alternatives can be seen.

If not already transferred via the PC, to save time it is recommended to key in the sample weights as the “Auto blanks” are running. New weights can also be entered when previous sample(s) are distilled.

Select “Kjeldahl” and connect the first test tube to be distilled. Close the safety door and press start. (Optionally just close the door)

1. 30 ml receiver solution is added into the titration vessel
2. 70 ml dilution water is added into the test tube
3. 50 ml alkali is dispensed into the test tube
4. Steam distillation starts and continues until end volume and end-point colour is reached. Dependent on how much titrant was added, distillation will continue to compensate for this.
5. When the analysis is ready the test tube will automatically drain.
6. (Optionally no drain can be selected. This is recommended if the digested sample contains solids like boiling beads in water samples or sand from soil samples)
7. The automatic cycle stops and next tube can be processed.

During distillation the titrant volume as well as the result is displayed. During distillation it is possible to enter more weights.

All volumes above are the default factory-set volumes and should be set differently if other concentrations and conditions are selected. Here are some hints.

Receiver solution: The condenser outlet should be below the liquid surface. The receiver volume is controlled by the second level pin in the titration vessel. It is quite normal that the liquid is sucked into the lower part of the condenser during dilution/alkali addition due to the heat. However, the volume is easily held in the condenser, working as a trap for any ammonia liberated.

Dilution water: If a larger volume of digestion acid is used it might be necessary to increase the water volume. However, it might be better to investigate if the digestion procedure is optimised. Too much acid will longer the time and does cost money.

Alkali: The sulphuric acid remaining after digestion has to be neutralised and an access of alkali is needed to liberate the ammonia. Theoretically 1ml concentrated H_2SO_4 needs 3.64 ml of 40% (10M) NaOH to be neutralised. If the alkali concentration is lower e.g. 32% (8 M) instead 4.28 ml is required. Please consider that not all acid added at the digestion remains, some is consumed and some lost through evaporation/ exhaust.

Distillation volume: 100 ml gives very good results for all levels of nitrogen, allowing enough steam to rinse splash-head/condenser between samples. If samples with almost the same and relatively low nitrogen level e.g. grain samples, a lower distillation volume will save time and still the results will be satisfying.

The table below will give some indications on what titrant concentrations to use. As the burette dispenses 1.9 µl per step also relatively low titrant volumes, around 1ml, still give accurate results. Also the automatic refill function of the burette during analysis allows a wide nitrogen range
without changing titrant.

Titrant concentration N	Titrant Volume ml	mg N	%N if 1g sample	%Protein (6.25) if 1g sample
0.2000	5 - 45	14 - 125	1.4 - 12	9 - 79
0.1000	5 - 45	7 - 63	0.7 - 6	4.5 - 40
0.0050	5 - 45	3.5 - 30	0.35 - 3	2 - 20
0.0010	5 - 45	0.7 - 6	0.07 - 0.6	0.4 - 4

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LA 1000
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List of Application Notes

Updated December 2020, Please contact your OPSIS LiquidLINE representative in case a more updated list is required.

Kjeldahl method and/or KjelROC instrument

LA1000 Application Guide (included in manual)

LA1001 The Importance of Catalyst in Kjeldahl

LA1009 Determination of total nitrogen in milk

LA1010 Determination of nitrogen in wheat

LA1011 Determination of nitrogen in pet food

LA1012 Determination of nitrogen in fish meal

LA1013 Determination of nitrogen in corn (maize) starch

LA1018 Determination of nitrogen in rice

LA1019 Determination of nitrogen in water

LA1022 Determination of protein in soy sauce

LA1023 Determination of nitrogen in soil

LA1024 Determination of TVB-N in fish

LA1026 Determination of protein in nuts

LA1028 Determination of protein in pasta

LA1031 Determination of SO_2

LA1031 Determination of SO_2 according to Chinese National Standards (CNS)

LA1033 Determination of alcohol in wine

LA1036 Determination of protein in Wine

Hot Solvent methods and/or SoxROC Instrument

LA1002 Application Guide (included in manual)
LA1003 Extraction of palm oil
LA1004 Extraction of fat in potato chips
LA1005 Extraction of total fat in pet food
LA1006 Extraction of fat in biscuits
LA1007 Extraction of fat in chocolate
LA1008 Extraction of total fat in chocolate
LA1014 Determining the gel content of Ethylene Vinyl Acetate
LA1015 Extraction of total fat in hard Cheese
LA1016 Extraction of fat in coconut milk
LA1017 Extraction of fat in fish Meal
LA1020 Extraction of dioxins and SVOC in food
LA1021 Extraction of SVOC and PAH in soil
LA1025 Extraction of fat in nuts
LA1029 Extraction of crude and total fat in poultry feed
LA1034 Determination of fat in Waste water
LA1035 Extraction of Total fat in Milk powder

FiberROC instrument

LA1027 Determination of crude fiber in cattle feed