875 KF Gas Analyzer

Manual

8.875.8001EN / 2019-11-12

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This documentation has been prepared with great care. However, errors can never be entirely ruled out. Please send comments regarding possible errors to the address above.

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1 Introduction

1.1 Instrument description

The 875 KF Gas Analyzer is a robust, modularly designed analysis system based on ***tiamo***™ for routine analysis at a site.

The system described on the following pages has been devised for the coulometric water content determination according to Karl Fischer in gases and allows the analysis of both liquefied gases and permanent gases. This method is also suitable for very low water contents.

The system comprises an operating unit and an analysis module. The analysis module is equipped with a base plate to convey the gas and with a water content determination cell as well as internally with an 851 Titrando in order to carry out all required analysis steps fully automatically. For this process, an amount of gas defined by the user is precisely measured with the flow meter and fed to the connected coulometer cell. Sample residue and water that might be present in the piping system are rinsed with dry nitrogen. The water is absorbed by the coulometric reagent and determined there by way of Karl Fischer titration. In coulometry, the iodine required for titration is produced by anodic oxidation, and the water content is subsequently determined. For the determination of liquefied gases, the samples are first vaporized in a controlled manner and then conveyed to sample determination.

Please also refer to the manuals and the documentation regarding the individual components (851 Titrando, mass flow controller, individual components) in addition to this documentation of the KF Gas Analyzer.

1.2 System description

- Robust analysis system with high-quality components for routine analysis tailored to the requirements of users.
- Gas-carrying system separate from the electronics and the power supply.
- The base plate with the system components is mounted behind a hood.
- The base plate comprises all components of gas conveyance and preparation as well as the coulometer cell.
- The base plate's gas system is pressure-tested.
- Nitrogen feed line with drying cartridge for predrying and check valve.
- Sample input filter preventing particles from entering the gas system.
- Deaeration bypass for pressure release during gas change.

- Integrated, adjustable vaporizer for liquefied gases.
- Heated oil filter with stainless steel filter element for analyzing used refrigerants with chiller oil contents.
- Rinsing connector for removing oil residue.
- Precise gas measurement with mass flow controller (MFC).
- Automated analysis process thanks to the use of solenoid valves.
- Predefined analysis method with a prerinsing, gas feed and postrinsing phase.
- Coulometric procedure for direct water content determination.
- Industrial PC and TFT panel (available as an option).
- All components except for the TFT panel are contained in one housing.
- Flexible control, user-friendly method creation and management and extensive data management using the ***tiamo***™ software. The operation of tiamo™ is described in the online help. Complete integration and control of all system components via the software.

1.3 System specification

- The system must be operated in a fume cupboard.
- Maximum sample input pressure: 40 bar.
- Maximum vaporization temperature: 80 °C.
- Nitrogen is required as auxiliary gas. The molecular sieve is used for predrying in the 875 KF Gas Analyzer. The input pressure must correspond to the vapor pressure of the samples.
- Gas connectors for nitrogen, rinsing medium, high-pressure waste gas: 6 mm Swagelok ferrule screw connector.
- Sample gas connector: 1/16'' or 6 mm Swagelok ferrule screw connector.
- Gas type: The system is suitable for the liquefied gases and permanent gases listed below. The gas system must be rinsed with nitrogen after each measurement. Additional gases may be approved on request and after testing.
  - Propane, propene, butane, butene, butadiene, LPG, natural gas
  - Dimethyl ether, ethylene oxide
  - Chlorinated hydrocarbons: methyl chloride, ethyl chloride, vinyl chloride
  - Refrigerants: various chlorofluorocarbon (CFC), hydrofluorocarbon (HFC) and chlorinated hydrocarbon (CHC) compounds. Fresh and used refrigerants with chiller oil contents.
- Safety specification: degree of protection IP54.

NOTICE

The materials of the components used have been carefully selected in accordance with the aforementioned gases. According to the current state of technology and the material manufacturers' resistance lists, these materials are resistant to the aforementioned gases.

However, a general guarantee is impossible to give, as we cannot predict how the gas mixtures will behave in the system and we do not know the concentration, degree of purity and aggregate state of the various gases that flow through the system.

1.4 About the documentation

CAUTION

Please read through this documentation carefully before putting the instrument into operation. The documentation contains information and warnings which the user must follow in order to ensure safe operation of the instrument.

1.4.1 Symbols and conventions

The following symbols and formatting may appear in this documentation:

*(5-**12**)*	**Cross-reference to figure legend** The first number refers to the figure number, the second to the instrument part in the figure.
1	**Instruction step** Carry out these steps in the sequence shown.
Method	**Dialog text**, **parameter** in the software
File ▸ New	Menu or menu item
[Next]	**Button** or **key**
	**WARNING** This symbol draws attention to a possible life-threatening hazard or risk of injury.

	**WARNING** This symbol draws attention to a possible hazard due to electrical current.
	**WARNING** This symbol draws attention to a possible hazard due to heat or hot instrument parts.
	**WARNING** This symbol draws attention to a possible biological hazard.
	**CAUTION** This symbol draws attention to possible damage to instruments or instrument parts.
	**NOTE** This symbol highlights additional information and tips.

1.5 Safety instructions

1.5.1 General notes on safety

WARNING

Operate this instrument only according to the information contained in this documentation.

The present system is suitable for processing gases and liquefied gases. In addition, hazardous substances are used in the wet end. Usage therefore requires the user to have basic knowledge and experience in handling liquefied gases, gases and pressurized media. Knowledge with respect to the application of the fire prevention measures prescribed for laboratories is also mandatory. The system may be operated only by trained staff. The operator must be trained both with regard to these operating instructions and the customer's laboratory rules and regulations.

This instrument has left the factory in a flawless state in terms of technical safety. To maintain this state and ensure non-hazardous operation of the instrument, the following instructions must be observed carefully.

NOTICE

Check all connections of the system for leakage at regular intervals and particularly after having made any modifications.

WARNING

The gas system is under pressure. It contains both pressurized gases and liquefied gases.

Before the sample vessel can be changed, the pressure must be released in the piping system and the latter may need to be rinsed with nitrogen.

Observe the applicable regulations.

WARNING

The oven used for vaporizing the liquefied gases and the oil filter downstream of the oven may exhibit a temperature of up to 70 °C. Avoid direct skin contact. Wear heat-insulating gloves, if necessary.

Clean the oil filter and rinse the piping carrying gas through the oven only with the instrument switched off and while it is cold.

1.5.2 Electrical safety

The electrical safety when working with the instrument is ensured as part of the international standard IEC 61010.

WARNING

Only personnel qualified by Metrohm are authorized to carry out service work on electronic components.

WARNING

Never open the housing of the instrument. The instrument could be damaged by this. There is also a risk of serious injury if live components are touched.

There are no parts inside the housing which can be serviced or replaced by the user.

Supply voltage

WARNING

An incorrect supply voltage can damage the instrument.

Only operate this instrument with a supply voltage specified for it (see rear panel of the instrument).

Protection against electrostatic charges

WARNING

Electronic components are sensitive to electrostatic charges and can be destroyed by discharges.

Do not fail to pull the power cord out of the power socket before you set up or disconnect electrical plug connections at the rear of the instrument.

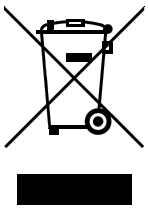
1.5.3 Flammable solvents and chemicals

WARNING

All relevant safety measures are to be observed when working with flammable solvents and chemicals.

- The instrument must be set up in a fume cupboard.
- Keep all sources of flame far from the workplace.
- Clean up spilled liquids and solids immediately.
- Follow the safety instructions of the chemical manufacturer.

1.5.4 Recycling and disposal



This product is covered by European Directive 2012/19/EU, WEEE – Waste Electrical and Electronic Equipment.

The correct disposal of your old instrument will help to prevent negative effects on the environment and public health.

More details about the disposal of your old instrument can be obtained from your local authorities, from waste disposal companies or from your local dealer.

2 Overview of the instrument

2.1 Instruments

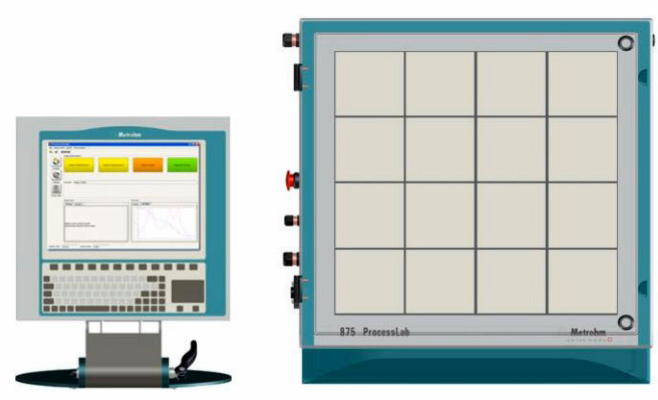


Figure 1 Operating unit and analysis module

2.2 Piping diagram

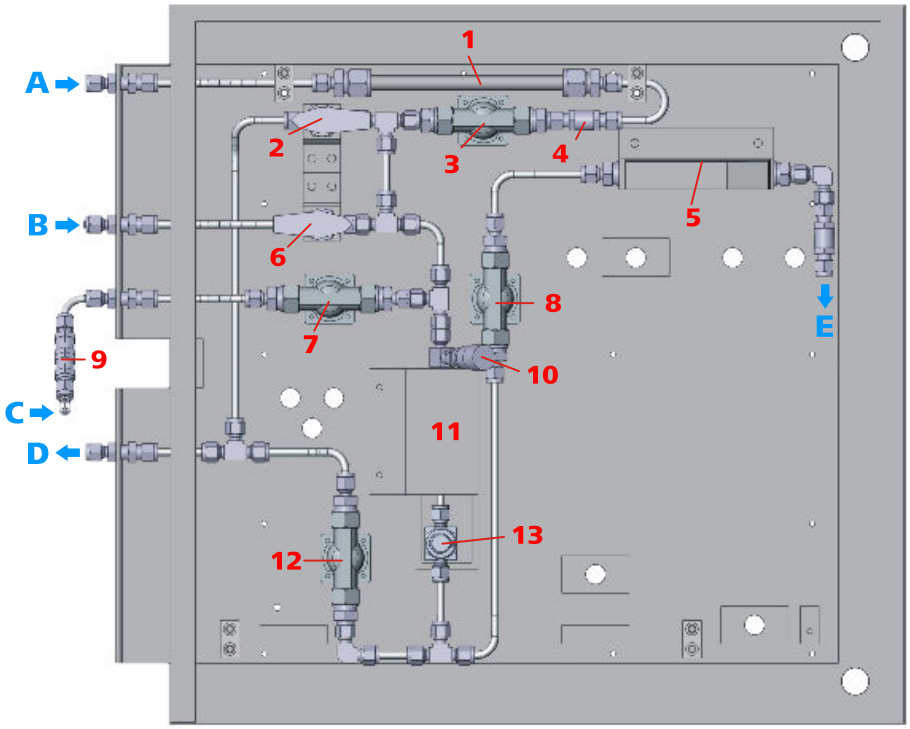


Figure 2 Schematic arrangement of the system

A Nitrogen	**B** Rinsing with solvent

C	**Sample**	**D**	**Waste gas**
E	**To the coulometer cell**		
1	**Drying cartridge (nitrogen)**	**2**	**Stopcock 1 (deaeration)**
3	**Valve 1 (nitrogen)**	**4**	**Check valve**
5	**Mass flow controller**	**6**	**Stopcock 2 (rinsing with solvent)**
7	**Valve 2 (sample)**	**8**	**Valve 4 (measurement)**
9	**Sample input filter**	**10**	**Precision control valve (vaporizer regulator)**
11	**Vaporizer**	**12**	**Valve 3 (waste gas)**
13	**Oil filter, heated**		

2.3 I/O controller

Digital inputs

Table 1 Digital inputs

Terminal	Function	Port	Port description
KL1104-1-1	E1	DigIn_1_1_1	QUICKSTOP
KL1104-1-2	+24 V		
KL1104-1-3	GND		
KL1104-1-4	E3	DigIn_1_1_3	
KL1104-1-5	E2	DigIn_1_1_2	
KL1104-1-6	+24 V		
KL1104-1-7	GND		
KL1104-1-8	E4	DigIn_1_1_4	

Digital outputs and relay outputs

Table 2 Digital outputs and relay outputs

Terminal	Function	Port	Port description
KL2424-2-1	A1	DigOut_1_2_1	Valve1 - N2
KL2424-2-2	GND		
KL2424-2-3	GND		
KL2424-2-4	A3	DigOut_1_2_3	Valve 3 - waste gas

Terminal	Function	Port	Port description
KL2424-2-5	A2	DigOut_1_2_2	Valve 2 - sample
KL2424-2-6	GND		
KL2424-2-7	GND		
KL2424-2-8	A4	DigOut_1_2_4	Valve 4 - measure-ment
Protective ground conductor terminal, 4-pin	Earth	Terminals 1 - 4	Earth for each of the 4 valves
KL2424-3-1	A1	DigOut_1_3_1	-
KL2424-3-2	GND		
KL2424-3-3	GND		
KL2424-3-4	A3	DigOut_1_3_3	MFC
KL2424-3-5	A2	DigOut_1_3_2	Heater
KL2424-3-6	GND		
KL2424-3-7	GND		
KL2424-3-8	A4	DigOut_1_3_4	-

Analog inputs

Table 3 Analog inputs

Terminal	Function	Port	Port description
KL3204-4-1	+I1	AnIn_1_4_1	Oven temperature
KL2424-4-2			
KL2424-4-3	+I3	AnIn_1_4_3	-
KL2424-4-4	GND		
KL2424-4-5	+I2	AnIn_1_4_2	-
KL2424-4-6	GND		
KL2424-4-7	+I4	AnIn_1_4_4	-
KL2424-1-8	GND		

3 Installation

3.1 Setting up the instrument

3.1.1 Packaging

The instrument is supplied in protective packaging together with the separately packed accessories. Keep this packaging, as only this ensures safe transportation of the instrument.

3.1.2 Checks

Immediately after receipt, check whether the shipment has arrived complete and without damage by comparing it with the delivery note.

3.1.3 Location

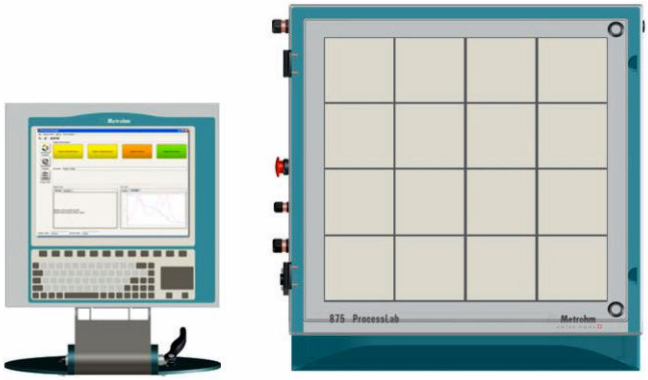
The instrument has been developed for operation indoors and may not be used in explosive environments.

Place the instrument in a location of the laboratory which is suitable for operation and free of vibrations and which provides protection against corrosive atmosphere and contamination by chemicals to the greatest extent possible.

The instrument should be protected against excessive temperature fluctuations and direct sunlight.

The 875 KF Gas Analyzer **must** be set up at a location of the laboratory equipped with a fume cupboard.

The operating unit is set up next to the analysis module.



3.2 General

The 875 KF Gas Analyzer is delivered in a largely preconfigured state.

As a rule, the installation steps described in the individual manuals have been carried out prior to delivery.

Additional notes are described in the subchapters below.

Fill the nitrogen drying cartridge with molecular sieve.

Establish the gas connections for nitrogen and, if required, for rinsing medium with 6 mm Swagelok ferrule screw connectors.

Establish the gas connection for the sample with 1/16'' Swagelok ferrule screw connector.

Connect the high-pressure waste gas and the waste gas of the coulometer cell to the extraction system.

3.3 Connecting the instrument to the power grid

WARNING

Electric shock from electrical potential

Risk of injury by touching live components or through moisture on live parts.

- Never open the housing of the instrument while the power cord is still connected.
- Protect live parts (e.g. power supply unit, power cord, connection sockets) against moisture.
- Unplug the power plug immediately if you suspect that moisture has gotten inside the instrument.
- Only personnel who have been issued Metrohm qualifications may perform service and repair work on electrical and electronic parts.

Connecting the power cord

Accessories

Power cord with the following specifications:

- Length: max. 2 m
- Number of cores: 3, with protective conductor
- Instrument plug: IEC 60320 type C13
- Conductor cross-section 3x min. 0.75 mm^2 / 18 AWG

- Power plug:
  - according to customer requirement (6.2122.XX0)
  - min. 10 A

NOTICE

Do not use a not permitted power cord!

1 Plugging in the power cord

- Plug the power cord into the instrument's power socket.
- Connect the power cord to the power grid.

3.4 Connecting control lines

WARNING

Always disconnect the instrument from the supply voltage.

Only shielded cables may be used for digital outputs, digital inputs, analog outputs and analog inputs.

The cable shielding must be connected to the grounding terminal.

The lines are connected directly to the I/O controller *(see chapter 2.3, page 8)*.

In order to open the contact springs, insert a 2.5 x 0.4 mm screw driver vertically into the rectangular actuation opening and press towards the LED.

A prefabricated cable has to be connected to the computer's network card directly if the 875 KF Gas Analyzer is being integrated into a LAN.

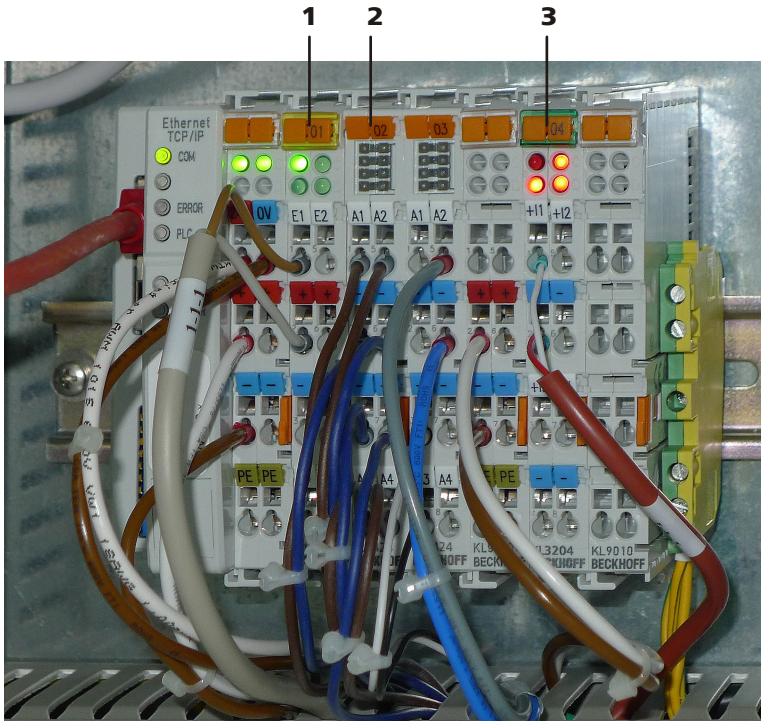


Figure 3 I/O controller

1 Analog output terminals	**2 Digital output terminals**
3 Digital input terminals	

3.5 Connecting the PC and the operating unit

The operating unit is connected directly to the industrial PC at the labeled locations.

The cable entry plate is screwed onto the 875 KF Gas Analyzer's housing.

3.6 Windows passwords

User	Password	Group
Gas Analyzer		User
Administrator	ADMINISTRATOR	Administrator
Metrohm	*******	Administrator

3.7 Gas connections

WARNING

Lines must be laid in such a way that they cannot be pulled out.

Nitrogen, sample, high-pressure waste gas

The connection between sample vessel and the Gas Analyzer's sample input must be as short as possible, have as little dead volume as possible, be absolutely tight and consist of suitable material. Observe the notes in the enclosed assembly instructions from Swagelok on connecting the Swagelok ferrule screw connectors.

Waste gas lines

The waste gas lines must be routed to the exhaust air system with no counterpressure.

3.8 Drying cartridge for nitrogen

Depending on the residual water content of the nitrogen, the cartridge is to be filled with dried molecular sieve.

Secure the filling in place with a glass wool plug on both sides. In addition, insert a sieve disk (enclosed in the delivery as an accessory) on the output side (right).

3.9 851 Titrando

NOTICE

For installation and preparation, refer to the manual of the 851 Titrando.

Both electrodes (indicator electrode and generator electrode) are protected from being pushed out with an SGJ clip.

Given the gas flow, only the adsorber tube with enlarged bore supplied is to be used.

The adsorber tube and the stopper of the gas infeed tip are not secured in order to prevent an uncontrolled pressure rise.

3.10 Shutting down

If the system is shut down for an extended period of time, then the entire gas system (gas flow to the coulometer, waste gas, rinsing and bypass piping) has to be rinsed with nitrogen ("Shut down system" method) and the coulometer cell has to be cleared of reagent and rinsed with dry methanol or ethanol. The cell can then be stored in a dry place.

4 Operation

4.1 Arrangement of the gas-carrying system

The valve arrangement mounted on the front plate of the 875 KF Gas Analyzer permits a safe and complete transfer of the sample and the water contained in it into the coulometric titration cell. The diagram *(see figure 4, page 17)* shows the schematic arrangement of the gas-carrying system.

The sample is introduced into the apparatus via valve 2 *(4-**7**)* and vaporized at the precision control valve (regulator). The heating block *(4-**11**)* compensates the heat that is lost in the system due to the enthalpy of vaporization and thus prevents the water to be analyzed from condensing or cooling.

The gas-carrying components are automatically rinsed with nitrogen that is predried in a drying cartridge *(4-**1**)* via valve 1 *(4-**3**)* before and after the sample is introduced. This nitrogen rinsing completely removes sample gas from the piping, so that no errors resulting from dead volumes can occur. Furthermore, rinsing with inert gas ensures that the water load on seals and internal metal surfaces in the apparatus is equal before and after sample introduction. Memory effects can be ruled out in this way.

The sample amount is metered with a mass flow controller *(4-**5**)*, which records the amount of gas flowing in and regulates the volumetric flow. During the introduction of liquefied gases, no pressure may build up downstream of the precision control valve, as this would entail the risk of sample condensing upstream of and within the mass flow controller and possibly interfere with the flow control and damage the instrument. For this reason, the precision control valve should be adjusted in such a way that the setpoint value for the mass flow controller is not achieved. As an additional safety, the system is equipped with a control that closes the sample input valve if the gas flow exceeds a threshold value defined as common variable.

When a new sample is connected, the feed line is first prerinsed with sample via valve 3 *(4-**12**)*. This is necessary because, initially, the connection fittings of gas bottles generally release water into the passing sample and the results of the first measurement without sample rinsing are generally higher. At the end of the measurement, the user can release the pressure from the sample infeed via stopcock 1 *(4-**2**)* in a controlled manner. The infeed line is then no longer under pressure when the gas container is disconnected.

If samples contain nonvolatile parts, such as oil contaminations, then these parts are held back by the filter element *(4-**13**)*. Contamination of the mass flow controller is thus excluded.

A thermally conductive connection exists between the oil filter and the heating block, which significantly increases the filter temperature. The retarding effect of oils on water is reduced in this way. The filters and the vaporizer are cleaned by rinsing the lines with a suitable solvent via stopcock 2 *(4-**6**)*. The corresponding dosing device forms part of the optional scope of delivery of the 875 KF Gas Analyzer.

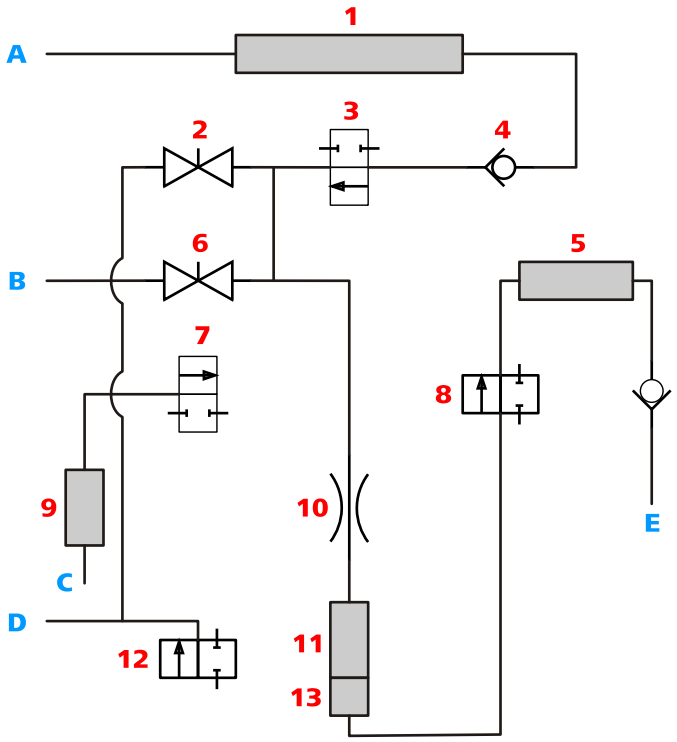


Figure 4 Schematic arrangement of the system

A	**Nitrogen**	**B**	**Rinsing with solvent**
C	**Sample**	**D**	**Waste gas**
E	**To the coulometer cell**		
1	**Drying cartridge (nitrogen)**	**2**	**Stopcock 1 (deaeration)**
3	**Valve 1 (nitrogen)**	**4**	**Check valve**
5	**Mass flow controller**	**6**	**Stopcock 2 (rinsing with solvent)**
7	**Valve 2 (sample)**	**8**	**Valve 4 (measurement)**
9	**Sample input filter**	**10**	**Precision control valve (vaporizer regulator)**

11	**Vaporizer**
12	**Valve 3 (waste gas)**
13	**Oil filter, heated**

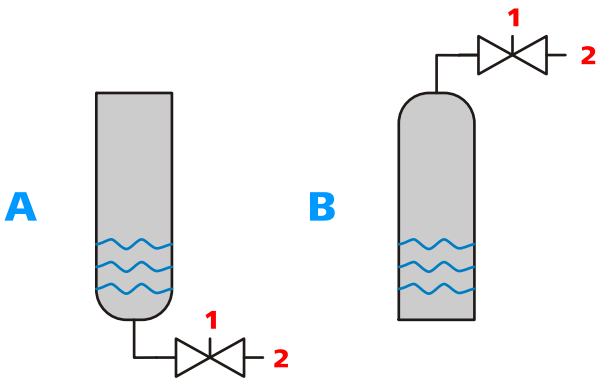


Figure 5 Sample vessel connector

A	**Liquid phase**
B	**Gas phase**
1	**Stopcock**
2	**Sample input to 875 KF Gas Analyzer**

4.2 Methods

WARNING

The gas system is under pressure. It contains both pressurized gas and liquefied gas.

The prescribed analysis procedure may not be modified. Users must have detailed knowledge of the gas conveyance in order to use the manual operation. Uncontrolled operation of the valves may result in a sudden vaporization of the liquefied gas or in pressure surges.

NOTICE

The correct position of the precision control valve has a decisive effect on the precision of the analysis. The exact position has to be determined for each gas type.

As standard, the 875 KF Gas Analyzer is delivered with the following methods (control programs of the ***tiamo***™ software):

- Sample measurement
- Reference measurement
- Precision control valve setting
- Gas calibration_liquefied gas

- Gas calibration_gas
- Shut down system
- Drift diagnosis
- System preparation

The following methods form part of the optional scope of delivery:

- Rinsing with solvent
- Reagent replacement
- Addition of methanol

NOTICE

Please note:

The ***tiamo***™ method can only be run if the **Flow** program has been started.

4.2.1 Sequence of the "Sample measurement" method

The water content determination of the samples is controlled by the **Sample measurement** method, which basically consists of three steps:

- Prerinsing the line route with nitrogen
- Feeding in the sample
- Postrinsing with nitrogen

The method is designed in such a way that the pressure prevailing in the area before the regulator (line volume between precision control, nitrogen and sample valve) is released during the change from prerinsing to sample introduction and from sample introduction to postrinsing. In this way, a mixing of nitrogen and sample that could result in faulty measurements is prevented. The entire sequence is shown in *(see table 4, page 19)*.

The flow diagrams of the analysis are visualized in figure 6. Some partial steps are only run through if the corresponding scans are set to "yes" in the sample table. The dosing device for methanol addition and reagent replacement as well as for the automated rinsing with solvent is an optional equipment of the 875 KF Gas Analyzer.

Table 4 Gas conveyance and valve control during the analysis

Partial step	Condition	Opened valves	Stop condition
Prerinsing with sample	Method variable "first sample measurement?" is set to "yes"	Sample valve Waste gas valve	90 seconds expired

Partial step	Condition	Opened valves	Stop condition
Draining of the sample that flowed into the area upstream of the regulator	Method variable "first sample measurement?" is set to "yes"	Waste gas valve	60 seconds expired
Rinsing out the waste gas line with nitrogen	Method variable "first sample measurement?" is set to "yes"	Nitrogen valve Waste gas valve	45 seconds expired
Prerinsing with nitrogen	None	Nitrogen valve Measurement valve	Status message from the coulometer "Conditioning OK", but at least 60 seconds
Pressure release nitrogen	None	Measurement valve	20 seconds expired
Sample introduction	None	Sample input valve Measurement valve	Value entered for minimum sample amount (mg) in the method variable is achieved
Pressure release sample	None	Measurement valve	Gas flow falls below 30 mL/min for more than 6 seconds
Postrinsing with nitrogen	None	Nitrogen valve Measurement valve	Stop criteria of the coulometric KF titration are met (extraction time and relative drift)
Relieving the sample infeed	Method variable "disconnect gas container after measurement?" is set to "yes"	Sample valve Stopcock 1	

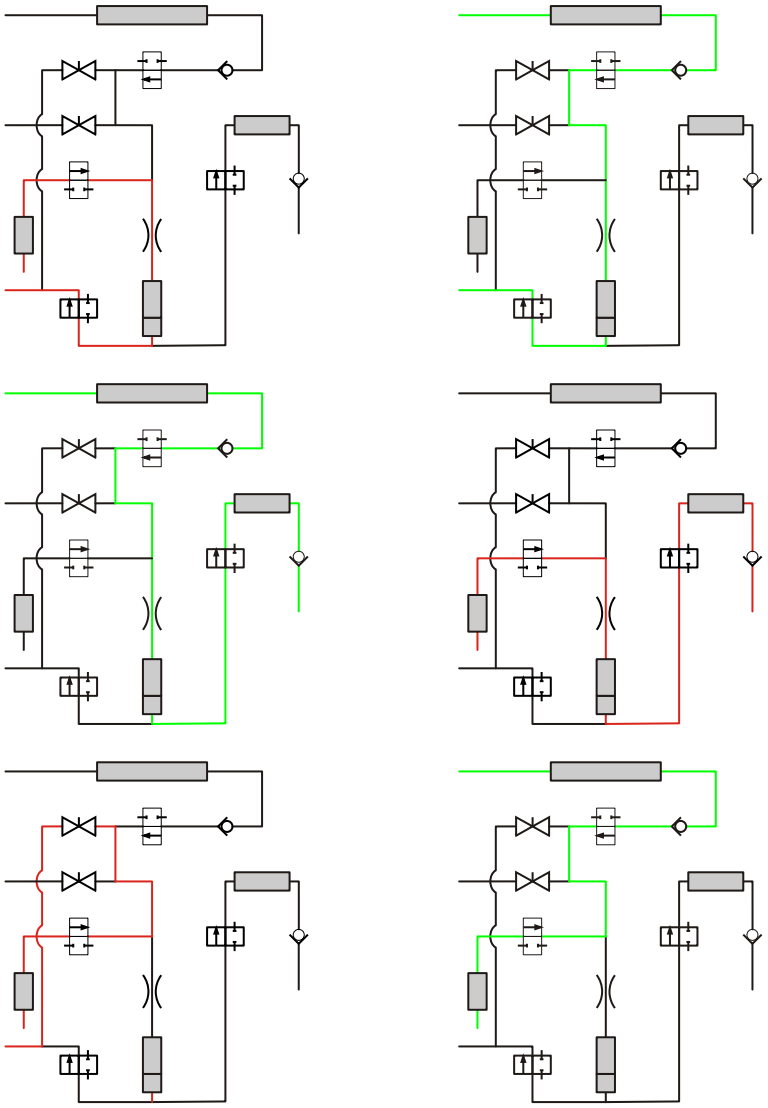
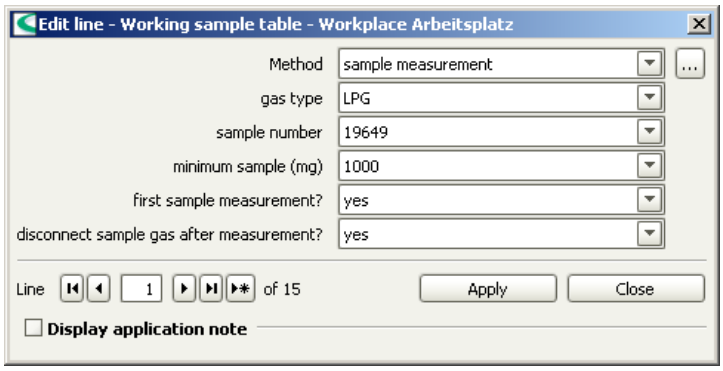


Figure 6 Schematic representation of the gas flows during an analysis

	Red marking = sample flow		Green marking = nitrogen
1	Prerinsing with sample	2	Rinsing out the waste gas line with nitrogen
3	Prerinsing and postrinsing with nitrogen	4	Sample introduction
5	Relieving the sample infeed	6	Rinsing the feed line with nitrogen

4.2.2 Working steps for carrying out a measurement

Load the sample table **Standard sample table gas measurement** in the run window of your ***tiamo***™ workplace under **Determination series ▸ Sample table ▸ Load**. This sample table is preset in such a way that you can make the entries that are relevant for you. The input window opens by double-clicking in the first line of the table template.



Gas type

Designation of a sample (substance or substance mixture), such as butadiene or propane, selected from the drop-down bar. The gas type is linked to the calibration factor that is stored under the same name as common variable.

Sample number

Sample ID used to identify your sample. The designation may be changed. It is also possible to assign further sample identifications. These must be created in the method and in the sample table.

Minimum sample amount

Valve 2 closes after the amount of sample entered in this field has been fed in.

Sample infeed is only completed after the sample contained in the area upstream of the regulator has flowed out.

Recommended range: approx. 1,000 to 2,500 mg, depending on the water content.

First sample measurement?

(yes/no)

Enter **yes** here in the case of the first measurement after a gas bottle has been connected. In this case, the feed line is rinsed with sample first.

Disconnect gas container after measurement?

(yes/no)

Enter **yes** here if you would like to disconnect the gas bottle after the measurement. The pressure is then released from the feed line via valve 1 in a controlled manner after the analysis and the feed line is subsequently rinsed with nitrogen.

4.2.3 Explanations regarding the shape of the gas flow and titration curves

The analysis procedure described in *(see chapter 4.2.1, page 19)* results in a characteristic shape of the gas flow and titration curves. The sample infeed phase concludes with the gas flow dropping to a value close to zero. The titration rate (drift) follows this drop with a delay of approx. 10 seconds. If the gas flow is below a threshold value defined as common variable for 6 seconds, then the nitrogen valve opens and postrinsing commences.

The amount of water detected in the postrinsing phase increases if the samples contain nonvolatile components that remain in the vaporizer and the oil filter. The distribution of the liquid and the gas phase balances out during the infeed phase, so that, at the end of the infeed phase, a part of the water contained in the sample is still present in the instrument's piping. Postrinsing serves to remove the retained water. Hydrophilic, nonvolatile sample components, such as glycol ester oils used in the refrigerant industry, for instance, therefore lead to a flattening of the drift curve during the infeed phase and as a result to an extension of the analysis time. As a general rule, the minimum titration time (extraction time) has to extend beyond the beginning of the postrinsing phase, as the titration would otherwise be finished in the "trough" between infeed and postrinsing. The control program uses the following formula to calculate the extraction time:

$$t_e = \frac{60 \cdot (m + 1000mg)}{v} + \frac{t_n \cdot m}{6000mg}$$

Figure 7 Formula for calculating the extraction time

te	**Extraction time**	**m**	**Minimum sample amount in mg**
v	**value in mg/min saved under CV.mean mass flow**	**tn**	**Value in sec entered under CV.time for postrinsing**

The default value of the **time for postrinsing** common variable is 3 minutes. If a sample requires a longer postrinsing phase, then the value must be increased accordingly.

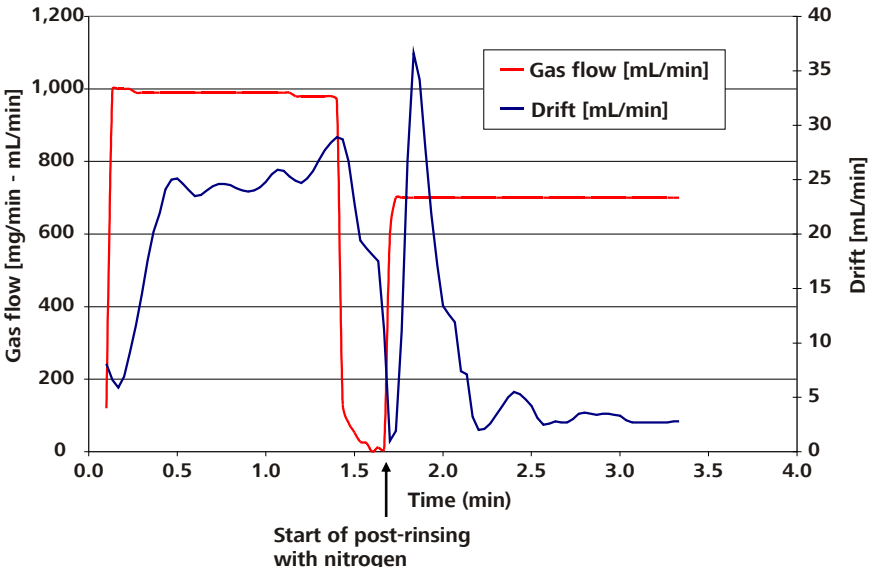


Figure 8 Typical shape of the gas flow curve and drift curve

Gas type Propene	**Sample amount** 1.25 g
Minimum sample amount 0.5 g	**Vaporization temperature** 70 °C

4.2.4 Method "Reference measurement"

The trueness of the analysis can be checked by measuring water-spiked reference gases using the **Reference measurement** method.

Control gases with certified water contents are commercially available.

The **Reference measurement** method relies on the nitrogen calibration of the mass flow controller integrated in the instrument; i.e., it only delivers correct values if nitrogen is used as reference gas. The procedure for reference measurement is the same as the one applied for **sample measurement**. The result is indicated as a recovery rate in percent.

4.2.5 Changing the gas type

If the measurement of a new sample coincides with a change of the gas type, then the flow rate of the precision control valve has to be adjusted to the current sample using the **Precision control valve setting** method. This method sets the setpoint value at the MFC to the maximum value of 5 L/min and graphically displays the current flow by utilizing the internal nitrogen calibration. In order to prevent a pressure rise in the area after the regulator, the precision control valve has to be set in such a way that its vaporization rate is lower than 5 L/min and the setpoint value is not reached at the MFC. After the start of the method, follow the instructions of the text messages and adjust the precision control valve so that the gas flow is within the required limits (definition by common variable).

NOTICE

Please note:

This method does not use the calibration factor that is assigned to this gas type. The mass flow displayed during the subsequent analysis may therefore considerably deviate from the value that was set when the precision control valve was adjusted.

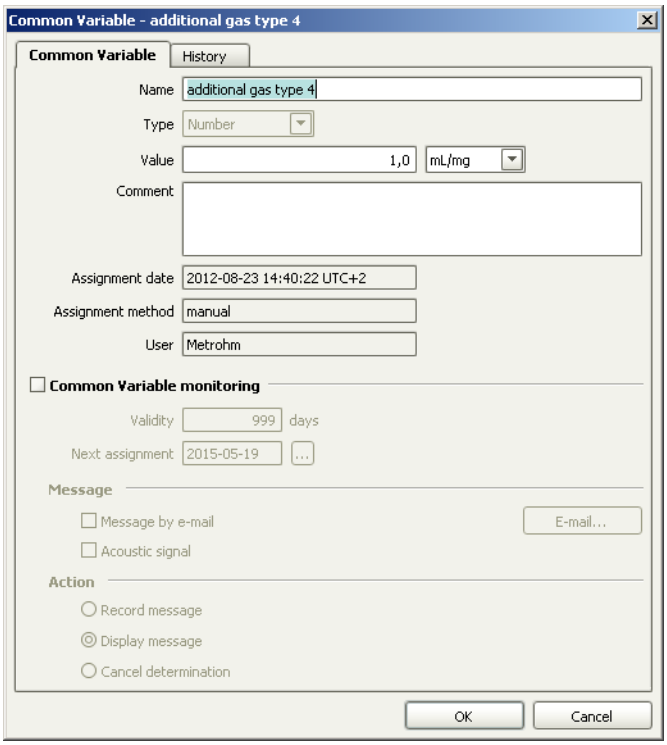
4.2.6 Calibrating a new gas type

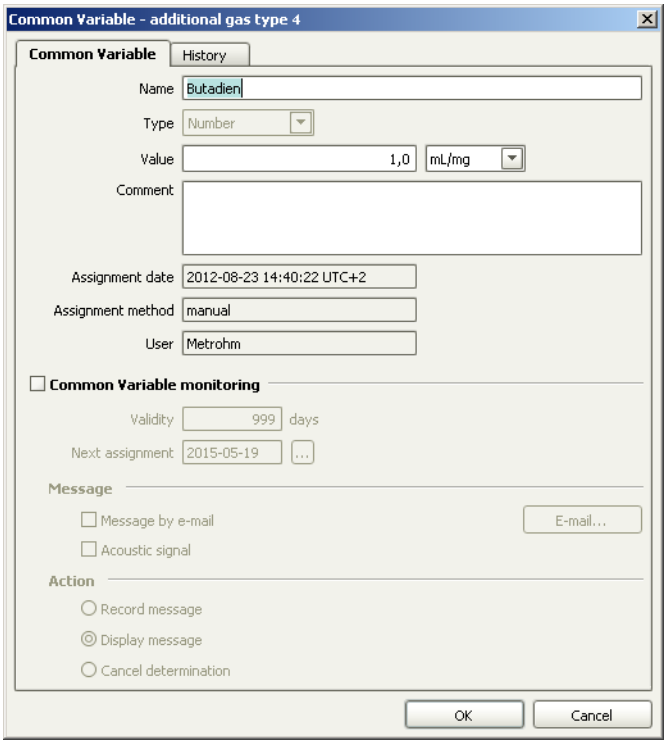
At the factory, the mass flow controller is calibrated to nitrogen. If the instrument is to be operated with a different gas, then the flow value has to be corrected by an appropriate factor. These correction factors are determined gravimetrically by letting larger amounts of gas flow through the MFC and monitoring the weight reduction of the gas container. The quotient of the gas volume indicated and the weight difference is the correction factor. This factor is in the range between 0.5 and 1.5 mL for most liquefied gases. The correction factors have to be individually determined for each flow controller using the **Gas calibration** method. This method saves the correction factor in the ***tiamo***™ configuration as common variable. In order to achieve a sufficient level of accuracy, the sample weight difference should have at least three significant places. The balance used therefore has to offer a corresponding resolution and maximum weight in accordance with the gas bottle size. For the determination of the calibration factor, the gas container has to be connected to the 875 KF Gas Analyzer with the flexible plastic capillary (OD 1/16'') enclosed in the scope of delivery, as steel capillaries transmit vibrations to the balance.

Samples should be taken from the gas phase **Gas calibration_gas** rather than the liquid phase of the gas container **Gas calibration_liquefied gas** for calibrations, because the flow pattern is much more uniform if vaporization does not take place in the 875 KF Gas Analyzer. The **Gas calibration_liquefied gas** method is only to be used if a water content determination is to be done for the same gas container after calibration.

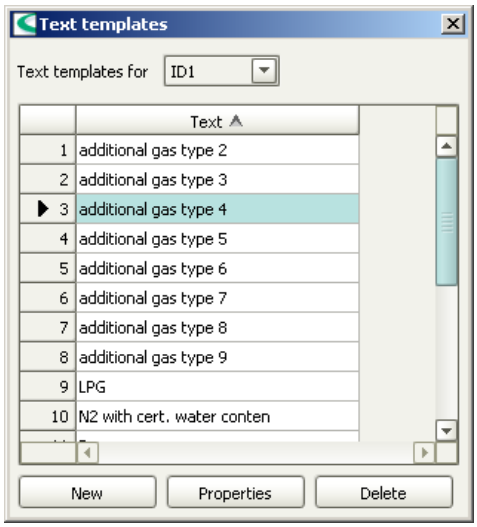
The procedure to determine the calibration factor is described below step by step using butadiene as an example:

1. You can find the correction factors for the gases you have used so far in the Common Variable subwindow in the ***tiamo***™ configuration. Templates with the designation "additional gas type x" (x = 1 to 9) are stored for adding further gases. The common variables can be rendered editable via **Edit ▸ Properties**. Replace the blank variable **additional gas type x** with the lowest number x by the term butadiene.

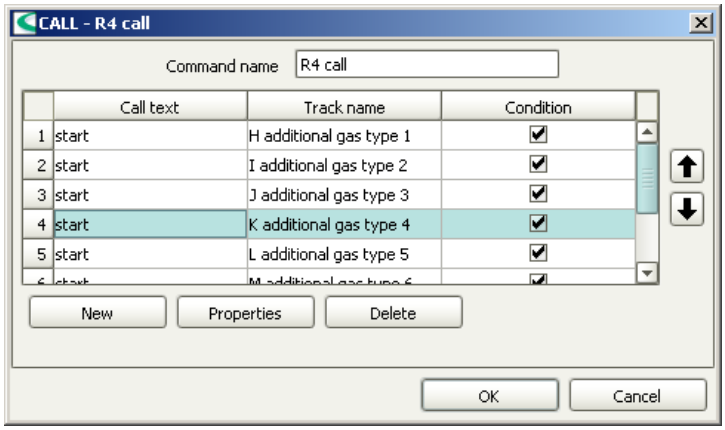


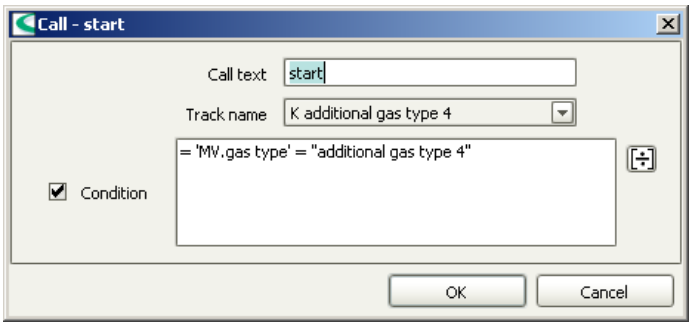


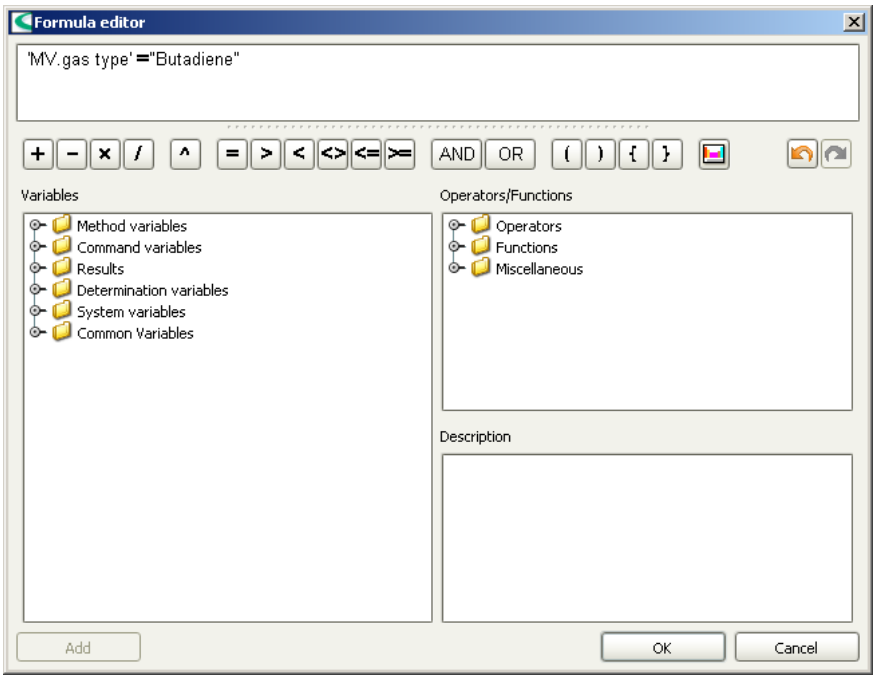
2 Enter the name of your gas type also in the **additional gas type x** text template under **Tools** ▸ **Text templates** ▸ **Text templates for ID** in the workplace of ***tiamo***™.



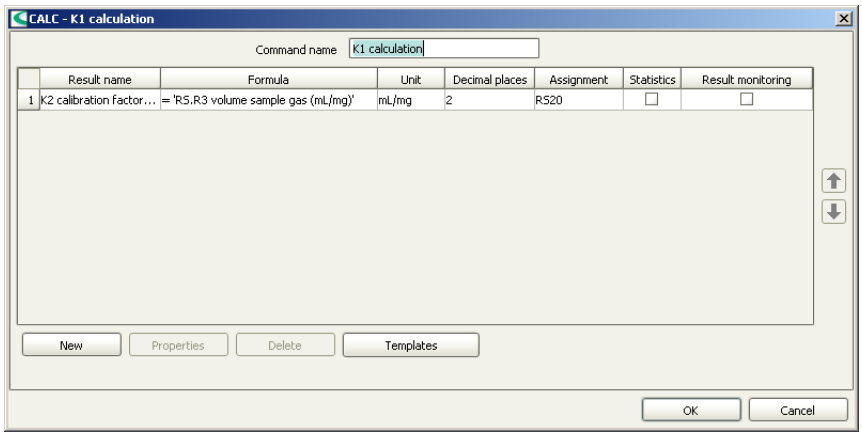
3 Open the **Gas calibration_liquefied gas** method under **File ▸ Open** in the Methods part of ***tiamo***™. The method consists of tracks that run from the top to the bottom. Each track is labeled with a letter. The individual commands are numbered consecutively from the top to the bottom. Search the **R4 call** command in the exit track. Double-click on the command to edit it. Overwrite the first line saying **additional gas type** by editing the line via the properties. Click on the ÷ symbol to open the formula editor. Replace the term **additional gas type x** in inverted commas with butadiene.

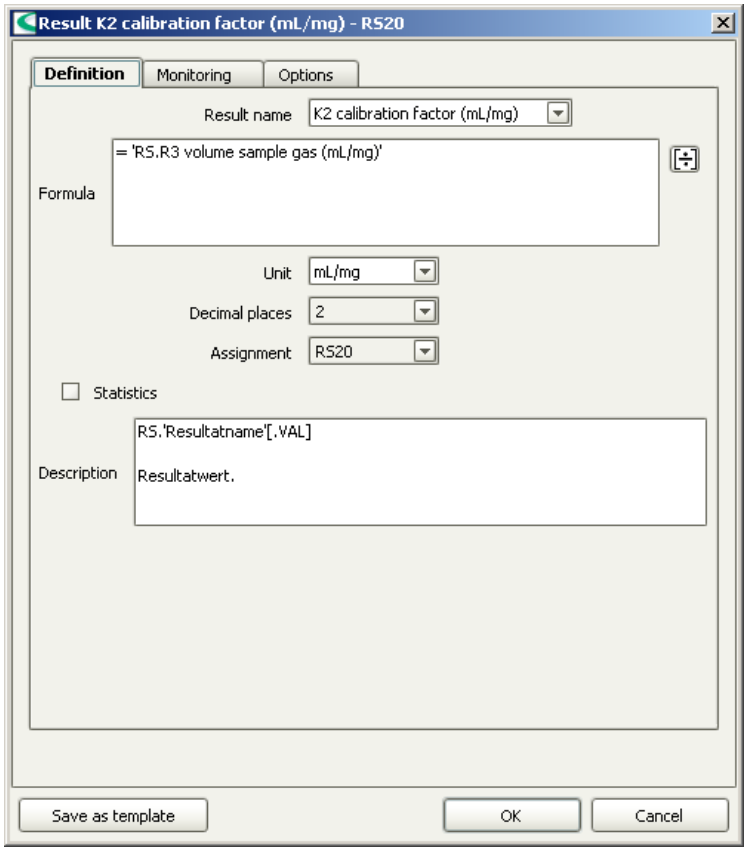


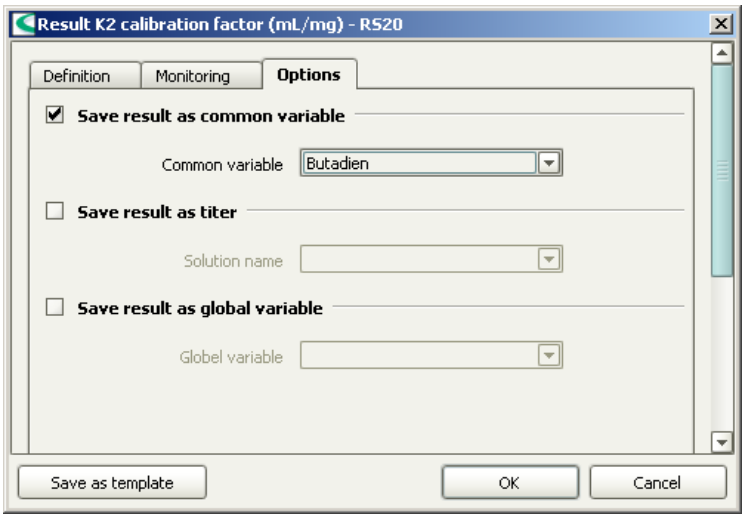




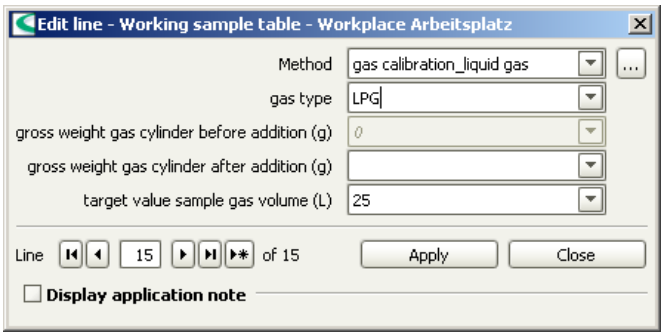
4 Edit the CALC command of the track to which the previously modified call command refers (in the example above, the track name was K additional gas type 4). Double-clicking in the calculation line opens a subwindow for the result properties. Click on the **Options** tab, select butadiene as common variable and then save the method with **File ▸ Save**.


CALC - K1 calculation
Command name
K1 calculation
Result name
Formula
Unit
Decimal places
Assignment
Statistics
Result monitoring
1
K2 calibration factor...
= 'RS.R3 volume sample gas (mL/mg)'
mL/mg
2
RS20
New
Properties
Delete
Templates
OK
Cancel

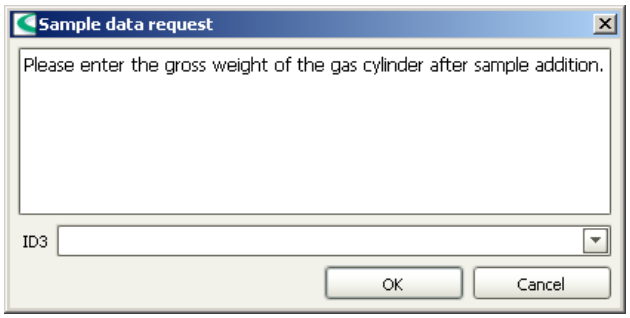

Result K2 calibration factor (mL/mg) - RS20
Definition
Monitoring
Options
Result name
K2 calibration factor (mL/mg)
Formula
= 'RS.R3 volume sample gas (mL/mg)'
Unit
mL/mg
Decimal places
2
Assignment
RS20
Statistics
Description
RS.'Resultatname'[.VAL]
Resultatwert.
Save as template
OK
Cancel



5 Now load the **Gas calibration_liquefied gas** method in the sample table of your ***tiamo***™ workplace. Select the designation of the gas type that was newly added and enter a target value for the sample gas volume (recommended range: approx. 20 L). This is the value that is displayed with the internal nitrogen calibration and not the actual gas volume of your sample. This value should be approx. 1.5 times the gas amount (in grams) which you want to convey through the instrument.



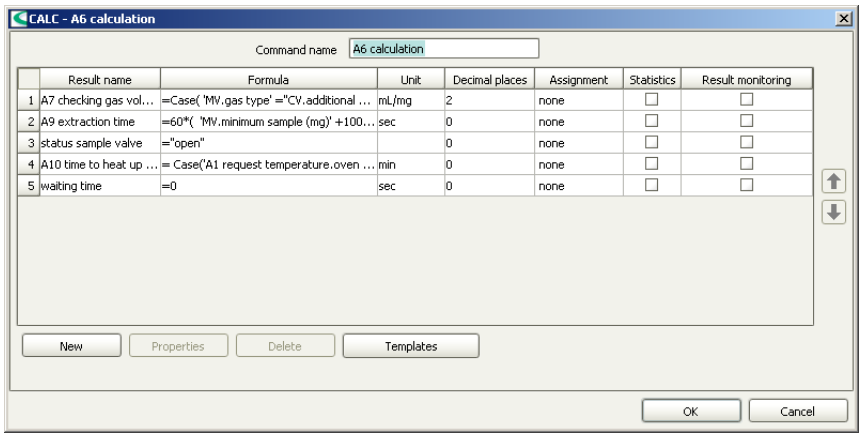
6 Tare the balance and start the method. After the target value has been reached, a prompt appears in which you have to enter the weight difference after gas infeed. The prefix does not matter for this.

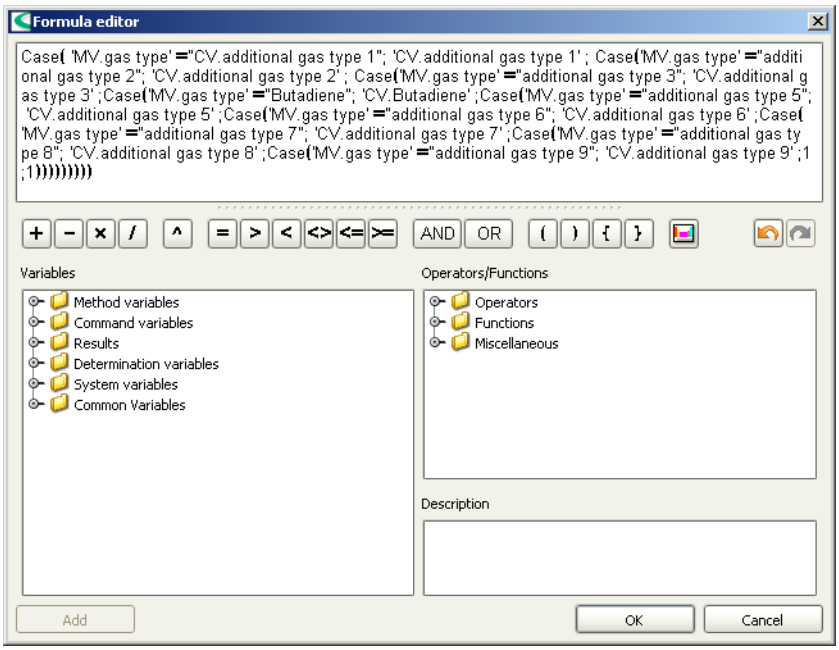


7 Check whether a valid value is entered under the corresponding common variable in the configuration.

8 Load the **Sample measurement** method in the ***tiamo***™ Methods part with **File ▸ Open** and double-click on the **A6 calculation** command to open it.

9 Edit the line **A7 checking gas volume** via the properties and open the formula editor by clicking on the ÷ symbol. An if-then query (nested CASE function) then opens; in this query the additional gas type x that you have replaced with butadiene is listed twice in a row. Replace the term additional gas type x with butadiene also here and save the method using **File ▸ Save**. You can now select your new gas type for the subsequent analyses, and the method automatically uses the appropriate correction factor for the calculations.





4.2.7 Automatic addition of methanol, automatic reagent replacement (optional accessories)

The anolyte in the coulometer cell consists mainly of methanol, which is removed to a considerable extent by the sample gas and the rinsing gas. The fill level of the measuring solution therefore decreases by approx. 8 mL per hour under normal operating conditions. In order to avoid malfunctions and faulty measurements, the missing methanol must be added regularly. This can be done manually with a syringe. Alternatively, the KF Gas Analyzer can be equipped with a dosing device to add methanol cyclically that is part of the optional scope of delivery. The rate at which the fill level decreases depends on the composition and temperature of the anolyte. The fill level can be increased if necessary using the **Addition of methanol** method. The **Reagent replacement** method is used for a complete exchange of anolyte.

4.2.8 Rinsing with solvent (optional accessories)

If liquefied gases contain nonvolatile components, these components precipitate in the piping of the KF Gas Analyzer. This is particularly the case for used refrigerants, which are usually contaminated with compressor oils. To prevent the sensitivity of the mass flow controller's sensors being compromised by such substances, an oil filter made of sintered stainless steel is located beneath the vaporizer. However, an infeed of larger amounts of oil results in a measurable retardation of the water in the piping and additionally increases the flow resistance of the oil filter, as its pores are covered by the oil. If samples contaminated with oil are to be measured, the system has to be rinsed with a suitable solvent from time to time.

The rinsing medium has to fulfill the following requirements:

- It has to be a suitable solvent for the nonvolatile residues.
- It has to exhibit a low boiling point, as it can be removed from the piping only by nitrogen rinsing.

Petroleum ether with a boiling range between 40 °C and 60 °C is recommended for oil contaminations. The rinsing medium is dosed with a dosing device that is optionally available. The system can be cleaned with the **Rinsing with solvent** method. The precision control valve must be entirely open during rinsing. For the subsequent sample measurements, the precision control valve has to be adjusted to the corresponding sample again using the **Precision control valve setting** method.

4.3 QUICKSTOP module

The red button on the left side of the housing resets all modules that are connected to the I/O controller to their default state (this usually means switched off), e.g., heater, valves and potential-free signal contacts.

The button locks in place and has to be pushed again to unlock.

Dosinos, stirrers and other devices that are connected directly to the 851 Titrando are not affected. They must be stopped directly in the software.

If an automatic analysis is running, then the quickstop module input can be queried in this ***tiamo***™ method. Thus, the devices connected to the 851 Titrando can also be stopped in this method.

5 Operation and maintenance

5.1 Care

WARNING

Appropriate personal safety measures must be taken for any work during which hazardous substances may be released (e.g., removing connection piping, disassembling or modifying the gas-carrying system). Examples of these safety measures include wearing personal protective equipment in accordance with the laboratory regulations: protective glasses, gloves and clothing.

Rinse with nitrogen and release the pressure from the system prior to carrying out work on the gas system.

The 875 KF Gas Analyzer requires appropriate care. Excess contamination of the instrument may result in functional disruptions and a reduction in the lifetime of the otherwise sturdy mechanics and electronics.

Spilled chemicals and solvents should be removed immediately. Above all, the plug connectors on the rear of the instrument (in particular the power socket) should be protected from contamination.

CAUTION

Although this is largely prevented by design measures, the power plug should be unplugged immediately if aggressive media have penetrated the inside of the instrument, so as to avoid serious damage to the instrument electronics. In such cases, Metrohm Service must be informed.

The molecular sieve of the predrying cartridge must be exchanged at regular intervals (in accordance with the residual water content of the nitrogen used).

Please refer to the 851 Titrando manual for information on maintenance and care of the coulometer cell.

A careful visual inspection of the gas-carrying system and the wet end has to be performed before an analysis series is started (e.g., status of the coulometer cell, gas connections and exhaust lines, leak-tightness). Check all connections of the system for leakage at regular intervals and particu-

larly after having made any modifications. If leakage is detected, this has to be eliminated immediately so as to prevent instrument damages.

If the necessity to clean the oil filter should arise periodically as a result of analyzing liquefied gases with nonvolatile components, the **rinsing with solvent** *(see chapter 4.2.8, page 32)* option is particularly recommended. Given the automated rinsing, no mechanical work is required on the gas-carrying system. The risk of leakage is thus eliminated. If the filter is cleaned manually, the system's tightness should be checked again after the filter is built in, like after any changes to the gas system.

NOTICE

The nitrogen inlet's check valve, which is a safety feature in case of an operating error, must be subjected to a functional check at least once a year. It has to be checked whether an additional check valve is required for the nitrogen supply.

5.2 Maintenance by Metrohm Service

Maintenance of the 875 KF Gas Analyzer is best carried out as part of an annual service, which is performed by specialist personnel of the Metrohm company. If you are frequently working with caustic and corrosive chemicals, we recommend a shorter maintenance interval.

Metrohm Service offers every form of technical advice for maintenance and service of all Metrohm instruments.

6 Troubleshooting

A low and constant drift is a prerequisite for correct and precise water content determination in the trace range. In the case of a carrier gas-flooded coulometric titration cell, this drift consists of the measuring cell's own basic drift (cell drift) and the water contained in the carrier gas. Therefore, to the extent possible, the nitrogen used for prerinsing and postrinsing must be water-free. Molecular sieve is capable of reducing the residual water content to approx. 1 to 2 µg/L, which is sufficient for the operation of the 875 KF Gas Analyzer. If the water concentration of the inert gas used for rinsing is higher, then the gas has to be dried with molecular sieve. A molecular sieve cartridge is located on the front plate of the Gas Analyzer before valve 1. With 15 mL, however, its capacity is rather limited, and therefore the cartridge only serves as a safety measure.

In an equilibrated state, the cell drift lies in a range between 1 and 3 µg/min. If a volumetric stream of 1 L/min of nitrogen that has been dried through the molecular sieve is set for the titration cell, the cell drift increases to approx. 2 to 4 µg/min.

A drift rise is attributable either to an increase in cell drift or an increased water infeed via the carrier gas *(see table 5, page 36)*.

The carrier gas' share in the total drift can be determined with the **Drift diagnosis** method. This share should not exceed 2 µg/min.

Table 5 Possible causes for a drift rise

Cause	Remedy
Cell drift rise due to the infeed of reactive matrix components	Exchange the anolyte
Cell drift rise due to the accumulation of water and H_2S in the catholyte	Exchange the catholyte
Water concentration rise in the rinsing gas due to exhaustion of the molecular sieve	Check the nitrogen quality, exchange the molecular sieve cartridge
Retardation of the water due to accumulation in the vaporizer and the oil filter	Rinse the gas-carrying system with solvent

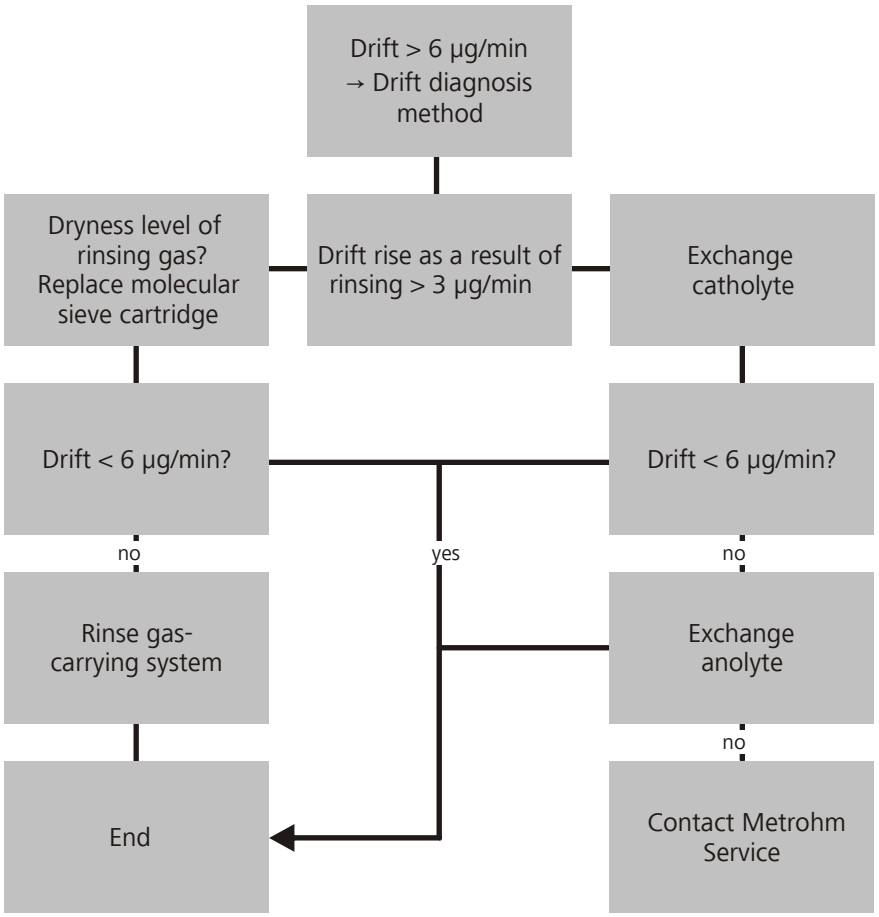


Figure 9 Systematic procedure for identifying the cause of drift rises

7 Technical specifications

7.1 Temperature ranges

Vaporization oven and oil filter	maximum 80 °C

7.2 Pressure ranges

Input pressure	maximum 40 bar

7.3 Supply voltage

Nominal voltage range	110 V or 230 V, adjustable at the power supply unit
Frequency	50 or 60 Hz
Power consumption	maximum 2,200 W
Fuse	10 ATH (slow-acting)

7.4 Dimensions

Analysis module	
Width	670 mm
Height	600 mm
Depth	470 mm
Operating unit	Values in brackets with pedestal.
Width	440 mm (550 mm)
Height	433 mm (433 mm)
Depth	95 mm (450 mm)

7.5 Weight

Analysis module	56.0 kg
Operating unit	21.7 kg

8 Accessories

Up-to-date information on the scope of delivery and optional accessories for your product can be found on the Internet. You can download this information using the article number as follows:

Downloading the accessories list

1. Enter *https://www.metrohm.com/* into your Internet browser.
2. Enter the article number (e.g. **875**) into the search field.
   The search result is displayed.
3. Click on the product.
   Detailed information regarding the product is shown on various tabs.
4. On the **Included parts** tab, click on **Download the PDF**.
   The PDF file with the accessories data is created.

NOTICE

Once you have received your new product, we recommend downloading the accessories list from the Internet, printing it out and keeping it together with the manual for reference purposes.

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