

**OPERATING MANUAL** 

# TO 7280 | TW 7650 | TM 235 TO

KF Oven System



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Gebrauchsanleitung	Seite	<b>5</b> .	4	6
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#### Important notes:

The operating manual is part of the product. Before initial operation, please carefully read and observe the operating manual and keep it. For safety reasons the product may only be used for the purposes described in these present operating manual. Please also consider the operating manuals for the devices to be connected.

All specifications in this operating manual are guidance values which are valid at the time of printing. However, for technical or commercial reasons or in the necessity to comply with the statuary stipulations of various countries, the manufacturer may perform additions to the product without changing the described properties. A potentially more recent version of this manual is available on our internet website. The German version is the original version and binding in all specifications!

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# 1 Technical Specifications of the Headspace Oven TO 7280 and the Sample Changer TW 7650

### 1.1 Notes to the operating manual

The provided operating manual will allow you the proper and safe handling of the product. For maximum security, observe the safety and warning instructions in the operating manual!

Marning of a general danger:

Non-compliance results (can result) in injury or material damage.

- **i** Important information for device use.
- Refers to another part of the operating manual.

The menu screens shown in this operating manual serve as an example and may differ from what you see!

#### 1.2 Intended Use

The Karl Fischer oven TO 7280 is a Headspace oven, used in conjunction with a coulometric titrator TitroLine<sup>®</sup> 7500 KF *trace* for water determination by the bake-out method.

Der TO 7280 can be used semi-automatically as a stand-alone instrument as well as fully automatically in combination with a Karl Fischer sample changer TW 7650.

The TW 7650 allows serial titrations with automatic sample change to be carried out in combination with the TO 7280. The titrations are carried out in a sealed sample vessel (vial) which is placed in the sample tray. The sample changer is controlled directly by the TO 7280. The TW 7650 has an easily exchangeable round sample plate.

The titration stand TM 235 TO includes a pump and a stirrer. It is used to hold the titration vessel in which the Karl Fischer titration actually takes place. By means of the pump, a volume flow is generated which flushes the liquid evaporating during the heating of the sample in the TO 7280 through the titration vessel and thus triggers a chemical reaction.

#### Solutions to be used:

Special coulometric reagents developed for combination with an oven are recommended.

1 The Karl Fischer oven is not intended for use with potentially biohazardous substances.

#### Do not use the device in hazardous locations!

# General:

The safety guidelines that are applicable to the handling of chemicals have to be observed under all circumstances. This applies in particular to inflammable and/or etching liquids.

# **1.3 Technical Specifications**

#### 1.3.1 Headspace Oven TO 7280

Translation of the legally binding German version

(Release: 22. April 2021)

EMC compatibility according to the Council Directive: 2014/30/EU; applied harmonized standards: EN 61326-1 Low-voltage directive according to the Council Directive 2014/35/EU; Testing basis EN 61 010-1: for laboratory equipment RoHS Council Directive 2011/65/EU

Country of origin: Germany, Made in Germany

**Measuring range**: (µg water absolute) in combination with a TitroLine<sup>®</sup> 7500 KF *trace* Coulometer:

Measuring range	Resolution [µg]	Reproducibility
1 µg 200 mg	0.1	+/- 0.3 μg at 10 – 1000μg 0.3 % at > 1000 μg

#### Temperature range:

 $\mathbf{C} \mathbf{E}$ 

40 °C ... 280 °C (isotherm)

Power supply:by external multi-range power supply from 115 – 230 V, 50/60 Hz<br/>Input voltage: 24 Volt DC, 11000 mA<br/>Power consumption 250 W<br/>Corresponds to protection class III:

# **A** Only use the power supply included in the scope of delivery or a power supply approved by the manufacturer!

Display: 3 colored LED display (blue/red/green). Operating states, error states and operating faults are signaled by color signals and flashing codes

#### **RS-232-C Interface:**

	Daisy chain function available
RS-232-1	for connection to a titrator, Daisy Chain input
	Data bits:fixed 8 BitStop bit:fixed 1 BitParity:fixed noBaud rate:fixed 4800 BaudAddress:fixed 3
RS-232-2	for connection of balances, Daisy Chain output
	Data bit:configurable, 7 or 8 Bit (Default Wert 8 Bit)Stop bit:fixed 1 BitParity:configurable: even / odd / no (Default no)Baud rate:configurable: 1200, 2400, 4800, 9600, 19200, 38400 (Default 4800 Baud)
Housing:	
Material:	Powder coated aluminum, steel, stainless steel
Dimensions:	30 x 45 x 24 cm (W x H x D)
Weight:	approx. 7 kg for basic unit without titrator and titration stand TM 235 TO

#### Ambient conditions:

	${iga \Lambda}$ Do not use the device in hazardous locations!
Climate:	Ambient temperature: + 10 + 40 °C for operation and storage Humidity according to EN 61 010, Part 1: Max. relative humidity 80 % for temperatures up to 31 °C, linear decrease down to 50 % relative humidity at a temperature of 40 °C
Altitude:	Sample changer: No restrictions Power supply: up to 5000 m

Pollution degree: Pollution degree IP 20, indoor use only.

#### 1.3.2 Sample Changer TW 7650

Translation of the legally binding German version

In combination with the headspace Oven TO 7280

(Release: 22. April 2021)

"	EMC compatibility according to the Council Directive: 2014/30/EU;
CC	applied harmonized standards: EN 61326-1: 2013
	Low-voltage directive according to the Council Directive 2014/35/EU;
	Testing basis EN 61 010-1: 2010 for laboratory equipment
	RoHS Council Directive 2011/65/EU

Country of origin: Germany, Made in Germany

#### Number of positions:

1 x 0 - Vial + 49 sample positions

#### Power supply:

Supply directly via the headspace Oven TO 7280

#### Housing:

Dimensions:	42 x 45 x 46 cm (W x H x D) incl. T	O 7280

Weight: approx. 10 kg without TO 7280

#### Ambient conditions:

# **A** Do not use the device in hazardous locations!

Climate:	Ambient temperature: + 10 + 40 °C for operation and storage Humidity according to EN 61 010, Part 1:
	Max. relative humidity 80 % for temperatures up to 31 °C, linear decrease down to 50 % relative humidity at a temperature of 40 °C
Altitude:	Sample changer: No restrictions Power supply: up to 5000 m

Pollution degree: Pollution degree IP 20, indoor use only.

#### 1.3.3 Titration stand TM 235 TO

Translation of the legally binding German version

In combination with the titrator TitroLine® 7500 KF trace

CE	EMC compatibility according to the Council Directive: 2014/30/EU; applied harmonized standards: EN 61326-1 Low-voltage directive according to the Council Directive 2014/35/EU; Testing basis EN 61 010-1: for laboratory equipment RoHS Council Directive 2011/65/EU
Country of origin:	Germany, Made in Germany
Pump:	Free flow rate - air externally adjustable between 50 500 ml / min Maximum pressure: 1.5 bar
Valve:	Automatically switchable valve for switching between air and gas flow
Control:	Via USB connection of the titrator TitroLine <sup>®</sup> 7500 KF <i>trace</i>
Stirring Speed:	50 1000 U/min, adjustable via a control dial
Gas flow meter:	Adjustable between 50 500 ml / min
Connections	
Power supply:	Low voltage input 12 V / – on the backside of titration stand Plug connection: plug for low voltage connection – phone jack-, Positive pole at pin contact, inside contact $\emptyset$ = 2.1 mm, USA/Japan, Power supply via titrator TitroLine <sup>®</sup> 7500 KF <i>trace</i>
USB:	For connection to the TitroLine <sup>®</sup> 7500 KF <i>trace</i> (see control unit)
Housing:	
Material:	Polypropylen
Dimensions:	8 x 13 x 25 cm (W x H x D), Height without stand, bottle and titration vessel
Weight:	ca. 1.9 kg
Ambient condition	S:

# ▲ Not suitable for use in explosive environments!

Climate:	Ambient temperature: + 10 + 40 °C for operation and storage Humidity according to EN 61 010, Part 1: Max. relative humidity 80 % for temperatures up to 31 °C, linear decrease down to 50 % relative humidity at a temperature of 40 °C
Altitude:	No restrictions

Pollution degree: Pollution degree IP 20, indoor use only.

(Release: 22. April 2021)

# 1.4 Warning and safety information

#### 1.4.1 General

The devices corresponds to protection class III.

The are manufactured and tested according to EN 61 010 - 1, Part 1 "**Protective Measures for electronic measurement devices**"and control devices and have left the factory in an impeccable condition as concerns safety technology. In order to maintain this condition and to ensure safe operation, the user should observe the notes and warning information contained in the present operating instructions. Development and production is done within a system which meets the requirements laid down in the Norm DIN EN ISO 9001 standard.

For reasons of safety, the devices have only be used for the range of application described in the present operating manual. Nonobservance of the intended proper use of the device may result in personal injury or damage to property.

For reasons of safety, the devices and the power supply must be opened by authorised persons only; this means, for instance, that work on electrical equipment must only be performed by qualified specialists. In case of nonobservance of these provisions the devices and the power supply may constitute a danger: electrical accidents of persons or fire hazard! Moreover, in the case of unauthorised intervention in the devices or the power supply, as well as in the case of negligently or deliberately caused damage, the warranty will become void.

Prior to switching the devices on it has to be ensured that the operating voltage matches the mains voltage. The operating voltage is indicated on the specification plate (underside of the devices and backside of the power supply). Nonobservance of this provision may result in damage to the devices and the power supply, or in personal injury or damage to property!

If the plate rotates, the titration head moves or the horizontal axis moves, the operating display will light up red. In this state, the sample changer must not be touched and no sample vessels must be removed or adjusted.

If it has to be assumed that safe operation is impossible, the device has to be put out of operation and secured against inadvertent putting to operation. In this case please switch the device off, pull plug of the mains cable out of the power supply, and remove the device from the place of work.

Examples for the assumption that a safe operation is no longer possible,

- if the package is damaged,
- if the device shows visible damages,
- if the power supply shows visible damages,
- if the device does not function properly,
- if liquid has penetrated into the casing,
- if the device has been altered technologically or if unauthorized personnel tried or succeeded to open the device as attempt to repair it.

In case that the user operates such a device, all thereof resulting risks are on the user!

**1** The Karl Fischer oven TO 7280 is equipped with integrated circuits (EPROMs). X rays or other high energy radiation may penetrate through the device's casing and delete the program

The devices have not be stored or operated in humid rooms.

#### 1.4.2 Warning signs

Observe all labels and signs attached to the devices. Non-observance may result in personal injury or damage to the devices!





#### 1.4.3 Chemical and biological safety

The relevant regulations regarding the handling of the substances used have to be observed: The Decree on Hazardous Matters, the Chemicals Act, and the rules and information of the chemicals trade. On the part of the user it has to be ensured that the persons entrusted with the use of the unit are experts in the handling of substances used in the devices or that they are supervised by specialized persons, respectively.

When using biohazardous substances, the regulations for handling the substances used must be observed. In such cases, the use is the sole responsibility of the user.

For all work with chemicals: **Always wear protective glasses!** Please observe the memorandums of the employer's liability insurance associations and the safety data sheets of the manufacturers.

Dispose of all used solutions in accordance with national regulations and laws. Select the type of protective equipment according to the concentration and quantity of the hazardous substance at the respective workplace.

#### 1.4.4 Flammable liquids

When handling flammable liquids, make sure that there is no naked flame in the vicinity of the equipment. Adequate ventilation must be provided.

Only small quantities of flammable liquids should be kept in the workplace.

When working with liquids that do not correspond to common reagents, particular attention must be paid to the chemical resistance of the materials of the devices (vgl. 🛄 1.3 Technical Specifications)

# 2 Installation and Commissioning

# 2.1 Operating environment

Deserve the following points to ensure that the device functions properly and has a long service life.

- The ambient temperature must be10 to 40 °C.
- The relative humidity must be below 80 % (without condensation).
- The titration head of the sample changer must be able to move freely.
- Do not use or store the device in dusty, damp or wet places.
- Make sure that no liquids can get into the device and wipe off any liquids on the device immediately.
- Protect the device from vibrations, direct sunlight, corrosive gases and strong magnetic and/or electric fields.
- Use only original spare parts and accessories.

# 2.2 Unpacking and setting up

The devices have been individually assembled for you, therefore there may be deviations with regard to the described scope of delivery and the accessories. Please refer to the enclosed packing list for the exact scope of delivery. If you have any questions, please contact us directly (for the service address, see backside of this operating manual).

The devices and all accessories as well as the peripheral devices have been carefully checked at the factory for function and dimensional accuracy. Please ensure that even the small additional parts are removed from the packaging without leaving any residue.

The units can be set up on any flat surface. Do not push any objects under the devices.

For the operation of the TO 7280 / TW 7650 an additional coulometer module TitroLine<sup>®</sup> 7500 KF *trace* is necessary. The TitroLine<sup>®</sup> 7500 KF *trace* is already included in the scope of delivery of the TM 235 TO M6. For the scope of delivery of the TitroLine<sup>®</sup> 7500 KF *trace* see the according operating manual.

# 2.3 Back panel of the TM 235 TO and the TitroLine<sup>®</sup> 7500 KF *trace*



### Fig. 1

The TM 235 TO is equipped with the following connections (Fig. 1):

- 1) Gas inlet when using the internal pump and ambient air as carrier gas
- 2) Connection of a gas cylinder when using nitrogen as carrier gas
- 3) Gas outlet for connection to the TO 7280
- 4) Power supply of the TM 235 TO
- 5) USB-B connection

The TitroLine<sup>®</sup> 7500 KF *trace* is equipped with the following connections (Fig. 1):

- 6) Connection of the generator electrode TZ 1752
- 7) Connection of the indicator electrode KF 1100

Two RS-232 ports (Mini-DIN):

- 8) RS-1 for connection to the PC
- 9) RS 2 for connection of the TO 7280 / TW 7650
- 10) Connection for power supply of the TM 235 TO

# 2.4 Installation of the TM 235 TO

Set up the TM 235 TO o the left or right of the unit (between the titrator and the oven) (Fig. 2).



# Fig. 2

Connect the TM 235 TO to the rear of the unit (12 V socket "out") with the connection cable TZ 1577 (Fig. 3). To control the TM 235 TO, it must also be connected to the titrator via USB (Fig. 4).





**1** The USB connection of the TM 235 TO **must be** made directly at the titrator and not via an intermediate USB hub. For additional accessories such as keyboard, USB stick or printer, an additional USB hub (TZ 3830) is recommended.

The carrier gas can be either ambient air dried via a molecular sieve or an externally connected gas cylinder (nitrogen). The following pictures show an example of the use of the gas inlet. Nitrogen is used in the same way via the gas inlet (see.  $\square$  Fig. 1).

# The safety regulations for handling gas and pressurized gas cylinders must be observed!

When using ambient air, a connecting hose with dust particle filter TZ 3995 (Fig. 5) is attached (screwed) to the gas inlet (Fig. 6).



Fig. 5



#### Fig. 6

Then screw the stand rod into the thread of the titration stand (Fig. 7).



The scope of delivery includes a holder for the titration vessel (supplied with the coulometer) and a holder for the two drying bottles of the carrier gas (Fig. 8). These are connected to each other via the recess on the bottle holder and the lug on the side of the titration vessel holder (Fig. 9).





#### Fig. 9

The two holders can now be pushed onto the previously mounted stand rod (Fig. 10).



Fig. 10

The drying bottles (Fig. 11) preassembled with hoses and filled with molecular sieve are placed in the holder (Fig. 12 and Fig. 13). The free shorter hose (1) is connected to the outlet of the pump. The longer hose (2) is used for subsequent connection to the flow meter.



Fig. 11





Fig. 13

The titration cell is inserted into the holder (Fig. 14):



#### Fig. 14

The generator electrode is inserted into the rear NS 14.5 opening and connected to the titrator (Fig. 15). The double platinum electrode is also inserted into the rear NS 7.5 (Fig. 16) opening and connected to the titrator (for the connection of the electrodes, see the respective operating manual).





The gas inlet tip is placed in the front right NS 14.5 opening (Fig. 17). The front left NS 14.5 opening is used to fill the titration vessel and is then closed with a gas plug after adding the stirring bar (Fig. 18).



Fig. 17



# 2.5 Back panel of the TO 7280



#### Fig. 19

The TO 7280 is equipped with the following connections (Fig. 19):

- 1) Power supply for connecting the supplied mains adapter
- 2) Main switch for switching on the oven

Two RS-232 ports (Mini-DIN):

- 3) RS-2 (bottom) for connection to a balance
- 4) RS-1 (top) for connection to the titrator
- 5) I/O Port for connecting optional accessories

## 2.6 Installation of the TO 7280

Place the KF Headspace oven TO 7280 to the left or right of the TM 235 TO.

The RS 1 of the oven (Fig. 20) is connected to the RS 2 of the titrator using the TZ 3094 (Fig. 21) cable included in the delivery.



Fig. 20



#### Fig. 21

The flow meter (Fig. 22) is screwed onto the bracket (Fig. 23).





# Fig. 23

The TZ 3993 hose (2 included in the scope of delivery) (Fig. 24) is then attached to the upper outlet of the flow meter (Fig. 25).



Fig. 24



The flow meter prepared in this way is screwed to the oven and the hose connected to the gas inlet of the oven (Fig. 26).



# Fig. 26

With the second TZ 3993 tube, the gas outlet on the needle head (Fig. 27) is screwed to the gas inlet tip on the titration cell (Fig. 28).



Plug the low-voltage cable of the power supply unit into the 24 V socket on the rear of the TO 7280 (Fig. 29). Then plug the power supply unit into the mains socket.

# The power supply must be placed in an easily accessible position so that the device can be disconnected from the mains at any time!

**1** The power switch, located on the back of the TO 7280, is used to switch the device on and off. A blue illuminated operating indicator on the front of the TO 7280 signals the operating state "On".



#### Fig. 29

#### 2.7 Installation of the TW 7650

The sample changer TW 7650 is connected directly to the oven TO 7280. The further installation is carried out analogous to 🛄 section 2.6 Installation of the TO 7280.

For the use of the TW 7680 the PC software TitriSoft is required.

The KF oven/changer is supported from TitriSoft version 3.5.

The connection contacts for the oven are located on the sample changer (Fig. 30).

# When connecting the TW 7650, the TO 7280 must be switched off and disconnected from the mains. Only switch on the TO 7280 after complete installation!





In the rear area of the sample changer there are recesses for the feet of the furnace as well as screws for firm fixation. The furnace is placed on top and firmly screwed in place (Fig. 31).



#### Fig. 31

On the holder for the sample plate there is a nose which engages in a recess located on the plate to fix the plate in place (Fig. 32).



If the plate is placed correctly (Fig. 33), the nose can be seen in the area between the upper and lower part of the plate (Fig. 34).



#### Fig. 34

The installation of the oven changer TW 7650 is now carried out analogously to  $\square$  section 2.6 Installation of the TO 7280.

#### 2.8 Switching on the unit and initial conditioning

- Set the stirring speed on the TM 235 TO. Please do not forget the stirrer bar.
- Now switch on the TitroLine<sup>®</sup> 7500 KF *trace*:

ithe device starts the conditioning immediately.

If the anolyte has been freshly filled in, the conditioning may last 10 - 20 minutes. The drift indication is > 1500  $\mu$ g/min at the beginning. When the drift has dropped below 10  $\mu$ g/min, the TitroLine<sup>®</sup> 7500 KF *trace* is ready for the first measurements with the oven.

# 3 Working with the TO 7280 and the TW 7650

# 3.1 General

### 3.1.1 The vials and the crimping tool

The measurements using a headspace oven are carried out in so-called sample vials. The vials have a capacity of 5 ml (Fig. 35). The samples to be measured are weighed directly into the vial and sealed with an ND 20 aluminum cap (Fig. 36). In the case of liquids such as oil samples, it must be ensured that the filling level in the vial is sufficient for the needle of the needle head to protrude into the sample and the gas flow can thus pass through the sample.

The crimping tool (Fig. 37) included in the scope of delivery is used for manual closing of the vials.



Fig. 35



Fig. 36



Fig. 37

For closing, the cap is placed on the vial (Fig. 38) and closed by means of the forceps jaws (Fig. 39) by squeezing the forceps handles (Fig. 40).









#### Fig. 40

1 The crimper can also be used to grip the vials to transfer them to the hot oven or to remove the hot vials from the oven

A The vials can be very hot depending on the bakeout temperature and must be handled with appropriate care!

#### 3.1.2 The 0 – Vial

A 0-vial is a dry-titrated vial that is used for conditioning the entire system including the oven. This principle is analogous to conditioning when working with a TitroLine<sup>®</sup> 7500 KF *trace* without an oven. For the determination, a fresh, empty vial is placed in the oven and conditioned until a constant drift is measured and the start parameters are met. This vial can be used several times. If the start drift is no longer achieved, the 0-Vial must be changed.

The start drift of the system determined in this way is carried out at the beginning of a measurement. The starting conditions are defined in the method parameters.

#### 3.1.3 The blank measurement

Before each sample titration, a blank measurement of the vials is required. This determines how much water is present in the air volume of the vial which is included in the sample measurement. To determine the blank, several blank vials are measured. At least three measurements are recommended. While the samples are weighed, the vials intended for blank measurement are placed near the balance and closed at the same time as the sample vials. The water content is determined before the actual sample measurement. The mean value from the multiple measurement is stored in  $\mu g$  water on a global variable. In the method parameters of the sample method, this global variable is subtracted from the result of the sample titration in the formula for calculating the water concentration.

1 On the subject of global variables and formula editor, see also TitroLine<sup>®</sup> 7500 KF *trace*.

### 3.2 Working with the TO 7280: Standalone

# 3.2.1 Standard methods and method parameters of the TitroLine<sup>®</sup> 7500 KF trace

**I** The menu screens shown in these instructions for use serve as examples and may differ from the actual display! The general operation of the TitroLine<sup>®</sup> 7500 KF *trace* is described in the corresponding

On the TitroLine<sup>®</sup> 7500 KF *trace*, 4 Headspace methods (Fig. 41) are available as standard methods (Fig. 42):

- With Headspace %
- With Headspace Blank
- With Headspace ppm
- With Headspace TitriSoft



Fig. 41

Default method	
With Headspace Blank	<b>A</b>
With Headspace ppm	
With oven %	
With oven ppm	
With TitriSoft	
Selection	$\wedge \vee$
Enter	ОК
Back	ESC
	02/04/21 12:08

#### Fig. 42

If the oven is to be used, it must be activated in the corresponding method. This is already preset in the standard methods. From the main screen (Fig. 43) **<EDIT>** takes you to the method parameters (Fig. 44).

- Main menu	
	7.9
Δ: 0.1 μg/min	ua/min
T: 120.0 °C	- 51
Methode mit Ofen	START
Method parameter	EDIT
Select method / system	MODE
	02/04/21 11:44

Method parameter — With oven ppm	
Edit method New method	
Default method	
Recalculation	▼
Selection	$\land \lor$
Enter	ОК
Back	ESC
	<b>\$</b> 02/04/21 12:07

## Fig. 44

The settings for the oven can be found in the «Titration parameters» (Fig. 45).

Edit method parameter - With Headspace ppm	
Method name Result	
Titration parameter	
Sample ID	man 🔻 🛛
Selection	$\wedge \vee$
Enter	ОК
Back	ESC
	<b>\$</b> 02/04/21 12:08

# Fig. 45

Under «Oven parameters» the oven is activated if necessary (Fig. 46),

<b>Edit titration parameter</b> With Headspace ppm	
Stirrer control	free
Oven control	On
Control factor	4
start condition	•
Selection	$\land \lor$
Enter	ОК
Back	ESC
	<b>1</b> 02/04/21 12:0

## Fig. 46

and the settings for the oven are made (Fig. 47)

Oven control With Headspace ppm	
deactivate	
Temperature	150.0 °C
Temperature deviation	0.5 °C
automatic fan control	On▼
Selection	$\land \lor$
Enter	ОК
Back	ESC
	02/04/21 12:09

You have the following options:

- **«Temperature»**: The working temperature of the oven is set
- «Temperature deviation»: Delta criterion from which the temperature reached is accepted
- «Automatic fan control»: When the automatic fan is switched on, a fan switches on to accelerate cooling processes
- **«Gas supply**»: Selection between pump (ambient air) or gas (nitrogen)

Via **<ESC>** you get back to the titration parameters (Fig. 48).

Edit titration parameter With Headspace ppm	
Stirrer control	free
Oven control	On
Control factor	4
start condition	T
Selection	$\land \lor$
Enter	ОК
Back	ESC
	<b>\$</b> 02/04/21 12:10

#### Fig. 48

Here you can set the start conditions of the titration (Fig. 49).

• Edit titration parameter With Headspace ppm	
Start drift	15.00 µg/min
interval	20 s
stability	0.3 µg/min
Selection	$\land \lor$
Enter	ОК
Back	ESC
	12:10

#### Fig. 49

You have the following options:

- «Startdrift»: Drift that must be reached before a method can be started
- «Intervall»: Period over which the stability is determined
- «Stability»: Stability criterion for the start drift

The general stop criteria of a coulometric Karl Fischer titration are described in the operating manual of the TitroLine<sup>®</sup> 7500 KF *trace* and also apply when working with a headspace oven.

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#### 3.2.2 Initialization of the TO 7280 / TW 7650

If a standard method with an activated oven is selected or the oven is activated in an existing method and switched back to the main screen, the oven is automatically scanned for and, once found, initialized (Fig. 50).

<b>Oven</b> Change vial Oven is initialized. Please wait!	
	<b>1</b> • 02/04/21 11:45

#### Fig. 50

After initialization, a prompt is displayed asking whether there is a 0-vial in the oven (Fig. 51).

TO7280 Change vial Please confirm if a via inserted into the over	al has been 1
Continue	<b>START</b>

Fig. 51

#### 3.2.3 Conditioning

If there is a 0-vial in the oven, press  $\langle OK \rangle$  to confirm. The needle head moves down and the gas supply is switched on. If there is already a vial in the oven and a new one is to be inserted, this is confirmed by  $\langle START \rangle$  the gas supply is switched off and the needle head is raised. The following prompt is displayed (Fig. 52):

T <b>O7280</b> Change vial Please confirm if a v inserted into the ove	ial has been n
Continue	<b>START</b> ) • 02/04/21 11:48

Press **<START>** to confirm when a 0-vial has been inserted in the oven. The display changes to the main screen and conditioning begins (Fig. 53). Once the start conditions are reached, the method name is no longer greyed out and a sample measurement can be started via **<START>** (Fig. 54).

- Main menu 3	7.6
Δ: 9.7 μg/min Τ: 120.0 ℃ ↑	µg/min
Methode mit Ofen Method parameter	START) EDIT MODE
Select method / system	∲ 02/04/21 11:4
Select method / system	¢02/04/21 11:4
Select method / system Main menu A: 0.1 µg/min	¢02/04/21 11:4
Select method / system - Main menu Δ: 0.1 μg/min T: 120.0 °C	<pre></pre>
Select method / system Main menu Δ: 0.1 μg/min T: 120.0 °C Methode mit Ofen	<sup>ψ 02/04/21 11:4</sup> 7.9 μg/min start
Select method / system Main menu Δ: 0.1 μg/min T: 120.0 °C Methode mit Ofen Method parameter	02/04/21 11:4 <b>7.9</b> μg/min START EDIT



#### Fig. 54

**1** At the end of each titration, a new sample measurement can be started directly from the results screen via  $\langle$ **START** $\rangle$  without measuring a new 0 – vial. The previously determined basic drift will also be used for the new titration. If the result screen is exited via  $\langle$ **ESC** $\rangle$ , a new 0 – vial can be inserted or the last titrated sample can be used as the new 0 – vial.

Change vial Is there a O-vial in the over be inserted?	n or one to
Insert 0-vial	START
0-vial in the oven	ОК
	<b>1</b> 04/28/21 14:10

#### Fig. 55

A new 0 - vial can be inserted via <**START**>. The gas supply switches off and the needle head moves up. Via <**OK**> the titrated sample is used as a new 0 – vial. The display switches back to the main screen and the new titration can be started as soon as the start conditions are met.

A vial in the oven can be changed at any time by pressing  $\langle$ **SHIFT** $\rangle + \langle$ **F10** $\rangle$ . The following security question is displayed (Fig. 56):

r TO2360	
Change vial	
Do you want to c	hange the 0-vial now?
Yes	ОК
No	ESC
	<b>1</b> 04/28/21 14:10

#### Fig. 56

After confirmation via < OK > the gas supply is switched off and the needle head moves up. The vial is removed and the new 0 – vial is inserted.

#### 3.2.4 Carrying out a sample measurement / blank measurement

When all start conditions are met, the measurement can be started by pressing **<START>**. The gas supply switches off and the needle head moves upwards (Fig. 57).



#### Fig. 57

The 0 – vial is removed from the oven and the sample vial is inserted (Fig. 58).

TO7280 Change vial Please confirm if a vial has been inserted into the oven
Continue <b>START</b>

#### Fig. 58

After confirmation via **<START**>, the needle head moves down again, the gas supply is switched on and the measurement starts (Fig. 59 and Fig. 60).



Fig. 59



#### Fig. 60

As soon as the end criteria of the measurement are met, the titration stops automatically (Fig. 61 and Fig. 62). A description of the end criteria can be found in the of the TitroLine<sup>®</sup> 7500 KF *trace*.

End of titration 2 of 3 With Headspace % - sample	
Start drift	13.4 µg/min
Start temperature o	119.7 °C
End drift	20.3 µg/min
End temperature oven	119.9 °C
nevt Dane	MODE
next raye	
Back	ESC
NO USB Stick	<b>W</b> 04/28/21 14:55



#### Fig. 62

Pressing **<START**> from the results screen will raise the needle head and the next sample measurement can be taken. By pressing **<ESC**> a new basic drift is determined.

#### 3.2.5 Communication error with the oven

If no TO 7280 is found when scanning for a furnace, an error message appears (Fig. 63).



#### Fig. 63

Press <**ESC**> to return to the main screen. The oven that was not found is indicated by three black lines in the temperature display (Fig. 64).

⊤Main menu	
	9.0
Δ: 0.8 μg/min	ua/min
T:	µy/mm
Methode mit Ofen	START
Method parameter	EDIT
Select method / system	MODE
	02/04/21 11:58

#### Fig. 64

If the connection to the oven is lost during operation, this is indicated by a red temperature display (Fig. 65).



#### Fig. 65

Please check that the oven is correctly connected to the titrator and that it has been switched on. In the oven service menu area, a re-initialization of the oven can be triggered after checking. If the oven is still not found, please restart it and try again.

#### 3.2.6 The oven service menu

The key combination **<SHIFT> + <F7>** takes you to the oven service menu (Fig. 66).

- Service menu oven '	
Reinitialize	
Needle head	
Temperature oven	
RS232 settings	
Sample changer	
Selection	$\wedge \vee$
Enter	ОК
Back	ESC
	02/04/21 11:45

#### Fig. 66

The following actions can be performed here:

- «Reinitialize»: Starting a reinitialization of the oven.
- «Needle head»: Manual up and down movement of the needle head. The movement also automatically controls the gas flow. When driving up, this is first interrupted, when driving down, it is switched on again.
- **«Temperature oven**»: Setting an oven temperature. Furthermore, a switch-on temperature can be defined that is automatically set when the oven is switched on. Furthermore, the automatic fan can be switched on and off.
- «RS 232 settings»: Setting the RS 1 and RS 2 ports.
- «Sample changer»: Internal Service interface.

#### 3.3 Working with the TO 7280 / TW 7650 and TitriSoft

Optionally, the TO 7280 can also be operated via the PC software TitriSoft. For general information on working with the PC software, see the operating instructions for the corresponding TitriSoft version. Working with the oven and the changer is possible from TitriSoft 3.5.0. The following examples are based on TitriSoft 3.5.0 and may differ from the actual display.

The Examples database included in the scope of delivery contains example methods for working with a headspace oven and changer and can be used directly. Adaptations to the specific sample, e.g. temperature, may be necessary.

If the TO 7280 / TW 7650 is to be controlled via TitriSoft, no method with activated oven must be selected on the titrator, otherwise the communication between TitriSoft and oven is blocked! The method «with Headspace TitriSoft » is available as default method.

#### 3.3.1 The titration command «Coulometer»

Methods are created in the analysis center and the analysis procedures are programmed. A titration module is available for working with the TO 7280 / TW 7650, which automatically regulates the titration procedure (Fig. 67).

Titration loop
Calculation
Measurement
Dose Chemical
Delay time
Stirring speed
Clear curve
IO box
Direct command
ISE
Coulometer

#### Fig. 67

The «Coulometer» command is selected and built into the method (Fig. 68).



#### Fig. 68

In the "**Type**" and "**Result**" area, the variable is defined and named on which the absolute result of the titration in  $\mu g$  water is stored. As a rule, the variable "**Type**" is "Result" and the "**Result**" name is "Water [ $\mu g$ ]" (Fig. 69).

Name	Value
Name	value
🔁 Coulometer	
Туре	Result
Result	Water [µg]
KF Trace	
Oven	
🕀 Drift parameter	
Start Drift	15
Start Drift Tolerance	0,3
Start Drift Tolerance Interval	20
Stop Drift Delta	5
Stop Drift Tolerance	0,02
Minimal Time	90
Maximal Time	1200
Stop delay	5
Working Point	300
Control Factor	4

#### Fig. 69

Under **«KF Trac**e» the titrator to be used for the titration is selected. In the **«Oven**» section, the oven to be used is selected. Depending on the connected system, the TO 7280 or the TW 7650 can be selected.

#### 3.3.2 Working with the TO 7280

After selecting the oven, the additional oven options open (Fig. 70).

Name	Value
🖯 Coulometer	
Туре	Result
Result	Water [µg]
KF Trace	Titroline 7500 KF Trace:RS1:01
Oven	T07280:RS1:03
Drift parameter	
Start Drift	15
Start Drift Tolerance	0,3
Start Drift Tolerance Interval	20
Stop Drift Delta	5
Stop Drift Tolerance	0,02
Minimal Time	90
Maximal Time	1200
Stop delay	5
Working Point	300
Control Factor	4
Oven Options	
Gas Source	Pump
Oven Temperature	150
Automatic fan control	$\checkmark$
Oven temperature delta	0,5
Keep head down after method	

#### Fig. 70

You have the following options:

٠	«Gas source»:	Selection between pump (ambient air) or gas (nitrogen).
•	«Oven temperature»:	Setting the working temperature of the oven.
•	«Automatic fan control»:	When the automatic fan is switched on, an additional fan switches on to accelerate cooling processes.
•	«Oven temperature Delta»:	Delta criterion above which the temperature reached is accepted.
	«Koon bood down	After completion of a titration, the people hand remains down as that the

• **«Keep head down after method»**: After completion of a titration, the needle head remains down so that the titrated sample vial can be used to measure a new base drift.

I For general setup of a worklist see the TitriSoft .

Once the worklist is set up and started, the following action prompt appears (Fig. 71).

Oven action	
Select next action Change 0-vial O-vial in oven Insert sample (use last 0-vial value)	
	ok

#### Fig. 71

You have the following options:

•	«Change 0 –via»:	A vial is in the furnace and is to be exchanged for a new 0 - vial. Go to point 1 of the following process description.
•	«0 – vial in oven»:	There is a 0 - vial in the furnace, which is used for the next conditioning. Go to point 2 of the following process description.
•	«Insert sample (use last 0 – vial value)»:	No new determination of the basic drift is made. The last measured base drift is taken over for the next measurement. Go to point 3 of the following process description.

#### 1) Add the 0 – Vial

The following window (Fig. 72) is only displayed if the **«Change 0 –via»** option is selected and a new background drift is to be recorded. After confirmation, the gas supply is switched off and the needle head moves up.

TO7280 Oven	
Add the 0-vial	
	ok

#### Fig. 72

The vial in the oven is removed, the new 0 - Vial is inserted and confirmed with < OK >. The needle head is moved down and the gas supply is switched on again. The status of the sample changes from «Active» to «Conditioning».

#### 2) Conditioning phase

If the **«0 – vial in oven**» option is selected, the vial in the oven is used for the conditioning phase that now follows.

Worklist: Water with TO 280											
no	Status	Analysis	Identification	Amount	Date	User	Comment	Curve	Wasser [µg]	Start Drift	End Drift
1	Conditioning	With Headspace Sample	Plastic	0,234	08.03.20	o≨ad					

#### Fig. 73

As soon as the start parameters are observed, the gas supply is switched off and the needle head is moved upwards.

#### 3) Place sample in the oven

If the «Insert sample (use last 0 - vial value)» option is selected, the sample is placed directly in the oven and the procedure continues as described from this point onwards.



#### Fig. 74

The 0 – Vial is removed from the oven, the sample vial is inserted and confirmed with **<OK**> (Fig. 74). The status of the sample changes to «Active» (Fig. 75) and the measurement begins (Fig. 76).

no	Status	Analysis	Ident	ification	Amount	Date	User	Comment	Curve	Wasser [µg]	Drift Drift	
1	Active	With Headspace Samp	ole Plast	с	0,234	08.03.202	ad					
5												
5												
5												
5												
5 Work	list: Water with	TO 280					_					
5 Work	list: Water with	TO 280 Analysis it	dentification	Amount	Date Use	r Comm	ent	Curve	_	Wasser [µ	Start End g] Drift Drift	t

#### Fig. 76

At the end of the titration, a prompt to remove the sample vial is displayed after the gas supply has been turned off and the head has been moved upwards. (Fig. 77).

10 sec



#### Fig. 77

If the «Leave head down at end of method» option is activated in the method, this message is not displayed; the gas supply remains and the needle head down.

If more samples have been added to the list, you will be asked if you want to continue with the work list (Fig. 78).



## Fig. 78

After confirmation with **<YES**>, a selection can be made again via the 0 – Vial.

Oven action
Select next action Change 0-vial 0-vial in oven Insert sample (use last 0-vial value)
ok

Fig. 79

#### 3.3.3 Working with the TW 7650

After selecting the changer, the additional changer options will open (Fig. 80).

Coulometer	
Name	Value
🖯 Coulometer	
Туре	Result
Result	Water [µg]
KF Trace	Titroline 7500 KF Trace:RS1:01
Oven	TW7650:RS1:03
Drift parameter	
Start Drift	15
Start Drift Tolerance	0,3
Start Drift Tolerance Interval	20
Stop Drift Delta	5
Stop Drift Tolerance	0,02
Minimal Time	90
Maximal Time	1200
Stop delay	5
Working Point	300
Control Factor	4
Oven Options	
Gas Source	Pump
Oven Temperature	150
Automatic fan control	$\checkmark$
Oven temperature delta	0,5
0-vial interval	Always
0-vial on method change	

You have the following options:

- «Gas source»: Selection between pump (ambient air) or gas (nitrogen).
- «Oven temperature»: Setting the working temperature of the oven.
- **«Automatic fan control**»: When the automatic fan is switched on, an additional fan switches on to accelerate cooling processes.
- «Oven temperature delta»: Delta criterion above which the reached temperature is accepted.
- **«0 Vial Interval**»: Choice, after how many measured samples a new 0 Vial measurement is performed.
- **«0 Vial on Method change**»: Setting whether a new 0 Vial is to be measured independently of the interval selection when the method is changed. Activation is recommended.

If the sample changer is to be used, it must also be selected in the worklist settings (Fig. 81 and Fig. 82).



#### Fig. 81

Name	Value	
	Value	
- General		
Name	Water with TW7650	
Category		
Worklist type	Normal	
Created by	Administrator	
Created date	20.10.2020	
Last modified	09.04.2021	
🔿 Report Properties		
Report style	Sample list	
Report title		
Is Landscape		
Report on finish		
Eims Import Properties		
Automatic import type		
Export Properties		
Export on finish	NUTT	
Include date in export the name		
Changer Properties		
Changer	TW7650:R\$1:03	
Continue with first position last position is ready		

#### Fig. 82

Here you can also select whether to start again with the first sample after a complete run through the sample plate.

The plate of the sample changer has 50 positions, whereby the first position is marked 0. This position is reserved for the 0 - Vial Thus, up to 49 samples can be measured by means of the changer. The procedure is completely automated. Depending on the setting (0 - Vial interval), the 0 - Vial is retrieved from the 0 position and placed in the oven before the actual sample measurement. After the start conditions have been met, the conditioning phase is completed, the 0 - Vial is transported back into the changer and then the sample vial of the corresponding position is placed in the oven.

#### 3.3.4 Quick Access to the devices via TitriSoft

The quick access menu (Fig. 83) can be used to send important direct commands to the TO 7280, TW 7650 and the TitroLine<sup>®</sup> 7500 KF *trace*.



#### Fig. 83

#### 1) TitroLine<sup>®</sup> 7500 KF trace

Titroline 7500 KF Trace:RS1:01		Pump on
T07280:RS1:03	•	Pump off
Stirrer on		Gas on
Stirrer off		Gas off

#### Fig. 84

You have the following options:

- «Pump on»: Manually switches on the pump when using air as carrier gas.
- «Pump off»: Manually switches off the pump when using air as carrier gas.
- «Gas on»: Manually switches on the valve, the nitrogen is supplied to the cell.
- «Gas off»: Turns the valve off manually, the nitrogen is no longer supplied to the cell.
- 2) TO 7280

Titroline 7500 KF Trace:RS1:01	Interview Provide Automatic
T07280:RS1:03	
Stirrer on	Head Up
Stirrer off	Head Down
11313	

#### Fig. 85

You have the following options:

- «Init»: Starts a re-initialization of the TO 7280.
- **«Head Up»**: Moves the needle head to the top position. The gas supply must be switched separately.
- **«Head Down»**: Moves the needle head to the lowest position for sampling. The gas supply must beswitched separately.

3) TW 7650

Volumetrisch	Head Down
enses ↓ Modified	Head Up
Stirrer off	Set Vial
Stirrer on	Get Vial
TW7650:RS1:03	Init
Titroline 7500 KF Trace:RS1:01	ration 2 Konfig

#### Fig. 86

You have the following options:

- «Init»: Starts a re-initialization of the TW 7650.
  «Get Vial»: Fetches a vial from the changer and places it in the oven.
- **«Set Vial**»: Take a vial out of the oven and place it in the changer. The sample plate position is queried at the beginning.

The sample plate position is queried at the beginning.

- **«Head Up»**: Moves the needle head to the top position. The gas supply must be switched separately
- **«Head Down»**: Moves the needle head to the lowest position for sampling. The gas supply must be switched separately.

#### 3.4 Error messages

Error number	Description	Troubleshooting
401	The TO 7280 / TW 7650 is not ready for operation	Check if the oven is switched on and the cables are correctly connected to the titrator and PC?
402	The TO 7280 / TW 7650 is in a non-initialized state or stop state, e.g. after a work list has been aborted.	The oven must be reinitialized manually.
404	Due to a device error, the system is in the stop state.	The oven must be reinitialized manually. If the error still occurs, please contact our service department.
405	There is a serious internal device error	Please contact our service.

# 4 Service and maintenance

To maintain the functionality of the device and the correctness of the volume, regular inspection and maintenance work must be performed.

Regular checks are a prerequisite for the correctness of the measured water content. The correctness is determined by the condition of the lines carrying carrier gas and their seals as well as the needle head system. Hoses, seals and the needle are wearing parts.

For maintenance of the coulometer, see the corresponding.

#### We recommend the following inspection and maintenance work:

Inspection Step	Interval
Easy cleaning: • External wiping of chemical splashes	Always when in use or when necessary
Check screw connections for tightness on TM 235 TO Flowmeter Neadle head Gas inlet tube on the titration cell	Weekly and on reactivation
Check the hose system for damage and contamination.	Weekly and on reactivation
Check the needle for damage (bent) and contamination (blockage due to septum residues, adherence of sample residues) and clean if necessary.	Weekly
Replace or dry the desiccant	Monthly or when the start drift is no longer achieved

All inspections and maintenance work can also be defined differently depending on the application. The individual intervals can be extended if no complaint occurs, they must be shortened again as soon as a complaint has occurred.

A regular check of the entire system by means of standards specially suited for oven measurement is recommended.

# 5 Guarantee

We provide guarantee for the device described for two years from the date of purchase. This guarantee covers manufacturing faults being discovered within the mentioned period of two years. Claim under guarantee covers only the restoration of functionality, not any further claim for damages or financial loss. Improper handling/use or illegitimate opening of the device results in loss of the guarantee rights. The guarantee does not cover wear parts, as lobes, cylinders, valves and pipes including the thread connections and the titration tips. The breach of glass parts is also excluded. To ascertain the guarantee liability, please return the instrument and proof of purchase together with the date of purchase freight paid or prepaid.

# 6 Storage and transportation

If the devices have to be stored over some time, or to be transported, the use of the original packing will be the best protection. However, in many cases this packing will not be available anymore, so that one will have to compose an equivalent packaging system. Sealing the devices in a foil is hereby recommended. The devices should be stored in a room with a temperature between + 10 and + 40°C, and the (relative) humidity of the air should not exceed 70 %.

# 7 Recycling and Disposal



Please observe the applicable local or national regulations concerning the disposal of "waste electrical and electronic equipment".

The devices and their packaging have been manufactured as far as possible from materials that can be disposed of in an environmentally friendly manner and recycled properly. If you have any questions about disposal, please contact our service department (see back of these operating manual).

# 8 EC – Declaration of Conformity

The corresponding declaration of conformity of the device can be found on our homepage. It will also be made available to you on request.

#### Bescheinigung des Herstellers

Wir bestätigen, dass oben genanntes Gerät gemäß DIN EN ISO 9001, Absatz 8.2.4 "Überwachung und Messung des Produkts" geprüft wurde und dass die festgelegten Qualitätsanforderungen an das Produkt erfüllt werden.

#### Supplier's Certificate

We certify that the above equipment has been tested in accordance with DIN EN ISO 9001, Part 8.2.4 "Monitoring and measurement of product" and that the specified quality requirements for the product have been met.

#### Certificat du fournisseur

Nous certifions que le produit a été vérifié selon DIN EN ISO 9001, partie 8.2.4 «Surveillance et mesure du produit» et que les exigences spécifiées pour le produit sont respectées.

#### Certificado del fabricante

Certificamos que el aparato arriba mencionado ha sido controlado de acuerdo con la norma DIN EN ISO 9001, sección 8.2.4 «Seguimiento y medición del producto» y que cumple con los requisitos de calidad fijados para el mismo.



a xylem brand

Hersteller (Manufacturer) Xylem Analytics Germany GmbH Am Achalaich 11 82362 Weilheim Germany

SI Analytics Tel. +49(0)6131.66.5111 Fax. +49(0)6131.66.5001 E-Mail: si-analytics@xylem.com www.XylemAnalytics.com

Service und Rücksendungen

(Service and Returns) Xylem Analytics Germany Sales GmbH & Co.KG SI Analytics

Gebäude G12, Tor Rheinallee 145 55122 Mainz Deutschland, Germany

Tel. +49(0)6131.66.5042 Fax. +49(0)6131.66.5105 E-Mail: Service-Instruments.si-analytics@xylem.com

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