Operating Manual Carbon and Sulfur Analyzer CS-580A (Helios)







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1 Notes on the Service Manual

1.1 Explanations of the safety warnings

In this Operating Manual we give you the following safety warnings

Mortal injury may result from failing to heed these safety warnings. We give you the following warnings and corresponding content.

Type of danger / personal injury

Source of danger

- Possible consequences if the dangers are not observed.
- Instructions on how the dangers are to be avoided.

We also use the following signal word box in the text or in the instructions on action to be taken:

Serious injury may result from failing to heed these safety warnings. We give you the following warnings and corresponding content.



Type of danger / personal injury

Source of danger

- Possible consequences if the dangers are not observed.
- Instructions on how the dangers are to be avoided.

We also use the following signal word box in the text or in the instructions on action to be taken:

▲ WARNING

Moderate or mild injury may result from failing to heed these safety warnings. We give you the following warnings and corresponding content.



Type of danger / personal injury Source of danger

- Possible consequences if the dangers are not observed.
- Instructions on how the dangers are to be avoided.

We also use the following signal word box in the text or in the instructions on action to be taken:

In the event of possible **property damage** we inform you with the word "Instructions" and the corresponding content.



NOTICE

Nature of the property damage

Source of property damage

- Possible consequences if the instructions are not observed.
- Instructions on how the dangers are to be avoided.

We also use the following signal word in the text or in the instructions on action to be taken:

NOTICE



2 Installation

2.1 Setting check up

Since the analyser weighs about 90 kg it should be placed on a suitably stable surface. Due to the balance, the platform should be as free of vibration as possible. The loader is attached to the right side of the analyser. There are no strong recommendations where to place the balance, PC, display and printer. Generally, placing them on the right is recommended. The size of a desktop (approx. 200 cm 80 cm for the analyzer with 130-crucibles autoloader) is sufficient for the entire set-up.

Below is an example of installation:



Fig. 1:Arrangement Suggestion

The environment of the device does not have to necessarily be air-conditioned, although it is best if the room temperature remains between $18^{\circ}C$ and $30^{\circ}C$. *NOTICE*

Under no conditions the device should be placed in direct sunshine!

Avoid places exposed to the wind of air conditioners or to the wind blowing through open windows or doors.



2.2 Front panel illustration



Fig. 1: Illustration front panel

1	Moisture trap
2	Carrier gas purification
3	Mains power switch
4	Compressed air gauge
5	Oxygen pressure gauge
6	Furnace inlet flow adjustment
7	Furnace inlet flow meter
8	Carrier gas flow meter
9	Lance flow meter
10	Lance flow adjustment
11 Furnace low inlet	
12	Dust filter cartridge



2.3 Mains power connections

Since the infrared cell requires about 1 hour for reaching a stable operating temperature, it is advisable to first connect the analyzer to the mains power and to set the mains power switch to position 1 before further installation work is carried out.

This waiting time is only necessary when the analyzer was completely switched off (position 0). It is recommended to keep the analyzer at standby (position 1) during working breaks, to keep the IR-cell operating temperature stable.

The furnace temperature is software controlled.



Fig. 2:Rearview – Mains power connections

1	Analyzer
2	Computer
3	Monitor
4	Printer
5	Balance
6	Quad power socket

At first, only the analyzer (1) is plugged in and switched on. Turn the mains power switch to position 1 (stand-by condition). The switch is on the front panel of the analyzer. See chapter front panel illustration

The mains power cable of the analyzer is mounted inside the analyzer.



All power cables of peripheral devices [computer (2), the printer (4) and the balance (5)] should be connected to the quad mains power socket (6) as shown in the figure.

Remark

Before placing balance, PC, display and printer on the desk for connecting their power cables, the loader should be attached to the analyzer and aligned. Please, refer to "LOADER installation, service and operation manual" for the instructions on installation, connection and alignment of the loader.

2.4 Data Interface



Fig. 3: Microcontroller board

1	PC USB connector	
2	Power and temperature control interface	
3	Analog input/output signals	
4	Pump control	
5	24V connector	
6	Digital input/output signals	
7	Autoloader connection	
8	Regulating valve (not relevant for CS-580A)	

When all units are connected to the mains power, then interface connections can be made. The required interface cables are included in the scope of supply. The supplied additional devices have been already adapted to the interfaces when the analyzers are taken into operation in our company. The plugs are all different from each other, so that they cannot be interchanged. The computer is already provided with operation system and with software for controlling the analyzer.



Please note: As the balance transfers the weight to the PC, its serial interface must be programmed. This is important, if you use a balance, which was not ordered with the analyzer.

NOTICE

For all instructions for using the PC software refer to the Help-function of the software.

2.5 Oxygen connection

The analyzer requires two gas connections: oxygen and compressed air. The necessary tubes for connecting the analyser to the oxygen and compressed air supplies are shipped with the analyzer.



Fig. 4: Oxygen and air connection s- rear side of device

Tube (1) connects the oxygen supply to the analyzer on the fitting of a pressure regulator. This connection must be very secure, considering that the incoming oxygen pressure in the tube is 2 to 4 bar (30 to 60 psi). Do not tighten the nut too much in order to avoid damaging of the tube. Drive the cup nut manually. Don't use any tool.



Compressed air connection 4-6 bar(58-88 psi)

Fig. 2: Compressed air connection

The tube (2) connects the device through a connector with the compressor or compressed air supply. For connecting the tube, simply push it into the connector. This connection must be also very secure, considering that the pressure in the compressed air is 4 to 6 bar (60 to 90 psi).

2.6 Operation modes

There are 4 different positions of the mains power switch possible:



0	Off.	The analyzer is completely switched off.
1	Standby.	The thermostatic control of the IR-cell is switched on. The furnace is switched off and the gas flow is disabled. Communication with the PC is possible.
2	Furnace on.	Furnace is switched on, but the gas flow remains disabled.
3	Gas flow.	All functions of the analyzer are in operation mode.

In case the analyzer was switched off (pos. 0) for long, it should be set to pos. 1 for at least 1 hour, in order for the IR-cell to reach the stable operating temperature.

The furnace heating-up time depends on the set temperature. In order to power the furnace, the analyzer has to be switched to position 2. At this position the gas flow is still disabled so that there is no gas consumption.

For running analyses set the power switch to position 3. At this position, the pump is enabled so that air and any moisture which has entered the gas flow system can be purged by the oxygen flow. Keep it there for about 10-15 minutes before starting the first analysis. To save time, the purging can be started before the set temperature has been reached. The slight influence of the oxygen flow on the temperature of the infrared cell is compensated.

At short working breaks (like lunch breaks) the mains switch remains on position 2 (furnace on) to save gas and to keep the set temperature.

During longer breaks, e.g. after finishing the work for the day, the mains switch is set to position 1 (standby). The thermostatic control of the infrared cell is then working, so that no long warm-up time is needed, when re-starting the analyzer.

Energy consumption and wear are negligible on standby. The analyzer is designed for long term use, so that no damage results.

Switch off the analyzer (position 0) is only useful, when working break takes several days or weeks.



2.7 Adjusting the gas flow



Fig. 3: Illustration front panel

1	Moisture trap
2	Carrier gas purification
3	Mains power switch
4	Compressed air gauge
5	Oxygen pressure gauge
6	Furnace inlet flow adjustment
7	Furnace inlet flow meter
8	Carrier gas flow meter
9	Lance flow meter
10	Lance flow adjustment
11	Furnace low inlet
12	Dust filter cartridge



2.8 Temperature adjustment

The furnace temperature is PC-controlled.

The CS-580A is delivered with 1350°C set temperature saved in a software profile. After switching on the instrument at position 2 and starting of the UNI-software, the heating up to the set temperature is started automatically. (The cable connection between instrument and PC is in the scope of supply).

The actual furnace temperature and the set temperature are visible in the window Heater.

You can change the set temperature in the window Configuration.

Configuration Profile Det		Deficiency			
Det	fault				
Auto-number PDF value			-{0}		
		e		0.000	
	Weight			0.0	
4	Heater				
	Alternative gas			Off	
	Maximum current			16	
	Preheat			0	
	Temperat	ure		1350	

Fig. 4: Window Configuration - Heater - Temperature

2.9 Preheating the furnace

NOTICE

For heating up, the mains power switch of the analyzer has to be at position 2 and the PC has to be switched on. The configuration of the BIOS has to be done by the customer as needed.

During heating up to the set temperature, the ceramic combustion tube is under high thermic stress. This stress is increased with increasing speed of the heating up process.

For maximum service life of the combustion tube, the furnace can be heated up slowly with a heating rate of 10°C/min. In order to avoid long waiting time until the furnace heats up, the heating up of the furnace can be started automatically long before the work begins. See chapter Bios Configuration

Please note that the automatic preheating can only be started when the analyzer temperature is below 100°C.

NOTICE

The analyzer is delivered with a default heating rate of 10°C/min. Much higher heating rates are possible; however with respect to the service life of the combustion tube, higher rates should only be used when necessary.

2.9.1 Configuration of preheating

1. Open the window *Default* and choose *Configuration*.



Fig. 5: window Default

2. In the chapter *Furnace* you find *Preheat*.



Alternative gas Catalyst furnace time	Off O	
Catalyst furnace time	0	
Eraptions		
FIECTIONS		
Gas factor 1	1	
Maximum current	13	
Preheat	10	
Temperature	1350	
Heater current limit PID		
CL D	0.1	
CL D max	255	
CL D min	-255	
CI Leon	255	

Fig. 6: Window Configuration - Heater - Preheating

The value shows the chosen heating rate in °C/min. (The preheating function is switched off by entering 0 in this place. Then, the heating occurs with maximum speed after start of the UNI-software.)

- 3. Save your entry with Enter.
- 4. The preheating starts automatically with the next start of the software, if the furnace temperature is lower than 100°C and the analyzer switch is at position 2.

NOTICE

When the preheating function is activated (by any other value except 0) the software automatically creates a shortcut at the startup menu of Windows. This enables the start of the UNI-software with every start of the PC. The shortcut is deleted automatically when the preheating is deactivated.

2.9.2 BIOS-configuration

In order to start the preheating before the work begins, the BIOS has to be configured to start the PC automatically at the selected time. (The preheating starts automatically by starting the UNI-software.)

- 1. Switch on your PC and enter the BIOS by pressing the corresponding key (e.g. F2). The normal booting process is interrupted and the BIOS menu appears.
- 2. Choose Power Management Setup
- 3. Activate "Resume by Alarm" (enabled).
- 4. Enter the required days and times when the heating up of the analyzer should be started.
- 5. Save your entries at Save/Exit with Yes.
- 6. The booting process will be sustained.



3 Analysis

3.1 Working procedure

With the CS-580A (Helios), a wide variety of materials can be analyzed. The analysis methods are therefore diverse. As different materials burn differently, the chosen sample weight, the possible accelerators and the sensitivity of the infrared cells will all be different. The user of the device can receive from us free advice regarding the different methods involved for different materials. The sensitivities of the infrared cells are optimized, free of charge, for each individual purpose.

In the following, the procedures are described for the analysis of coal samples.

Before starting making analyses ensure the following:

- The temperature of the analyzer is stable (at least one to two hours on setting 1).
- The moisture traps are checked and, if necessary, the magnesium perchlorate is replaced. See chapter "Moisture trap replacing furnace".
 - The incoming oxygen supply has a pressure of 2-4 bar (30 to 60 psi).
- The furnace has reached its operating temperature. See chapter "Temperature adjustment".
- The mains switch is set to position 3 for at least 10 to 15 minutes.
- The software is started on the connected PC.

3.1.1 Procedure of carrying out analyses with autoloader

- 1. Place an empty crucible on the balance.
- Tare the balance using the "Tare" button, located on the balance, or "F6-Tare" button in the "Analysis control" window of the software (F6 on the PC-keyboard).
- 3. Put about 250 mg coal sample into the crucible.
- 4. Press the "F4-Balance" button in the "Analysis control" window of the software (or F4 on the PC-keyboard) to transfer the weight value from the balance to the PC. The transfer function is performed regardless how often the button is pressed. This enables a correction of any falsely entered weight.
- 5. Enter the sample ID into the corresponding input field of the "Analysis control" window of the software. This step is optional. If no sample ID is required, the entry may be omitted.
- 6. Press the "F7-Add" button in the "Loader" window. The sample weight and sample ID are transferred to the loader stack and memorized there.
- 7. Take the crucible from the balance and put it on the loader, on the first tray before the pick-up position.
- 8. Mark the "F8-Run/Stop" checkbox to start the analysis.

The analysis procedure begins. From now on, no operator intervention is required. The furnace is opened automatically, the crucible of the previous analysis is picked up from the pedestal and disposed, and the crucible is taken from the loader and put on the pedestal for analysis. The furnace closes. At the end of analysis, the results are displayed on the PC-screen and saved in the database of the software.

A series of analyses can be run with the CS-580A (Helios) analyzer. To do this, repeat steps 1 to 7 for every sample in the series, while each crucible is placed as next after the last one placed on the loader. This can be done at any time, with or without running analyses.

When putting the samples on the loader, the order of positioning the crucibles on the trays must be respected, i.e. the crucibles must be placed on the loader exactly



in the same order in which they are weighed and memorized in the loader stack. It is allowed to leave one or more empty trays on the loader, for example in order to separate groups of crucibles carrying the same sample material or a calibration standard etc.

3.1.2 Procedure of carrying out analyses with manual loading of the sample

Although the CS-580A (Helios) is equipped with an autoloader and normally analyses are carried out using this autoloader, it is possible to load the sample manually for analysis.

- Place an empty crucible on the balance.
- Tare the balance using the "F6-Tare" button located on the balance, or the "Tare" button in the "Analysis control" window of the software (F6 on the PC-keyboard).
- Put around 250 mg of coal sample into the crucible.
- Press the "F4-Balance" button in the "Analysis control" window of the software (F4 on the PC-keyboard) to transfer the weight value from the balance to the PC. The transfer function is performed regardless of how often the button is pressed. This enables a correction of any falsely entered weight.
- Enter the sample ID into the corresponding input field of the "Analysis control" window of the software. This step is optional. If no sample ID is required, the entry may be omitted.
- Press the "F2-Furnace" button to open the furnace (if it is closed).
- Take the crucible from the balance and put it on the pedestal.
- Press the "F5-Start" button to begin the analysis.

From now on, no further operator intervention is required. The furnace closes and the analysis is carried out automatically. At the end of each analysis, the results are displayed on the PC-display and saved in the database of the software.

Remarks

The sulfur range should be deactivated when only carbon is required. This avoids undue delays of the analysis caused by sulfur compounds which are difficult to burn, like it is in case of cement analysis. Accelerators are also not necessary.

The sample weights are valid when the detectors have appropriate sensitivities and the right path lengths of the IR-cells. If not, the analysis condition can be improved by sample weight variation.

Generally, the weight has an optimum size when the peaks on the screen reach the middle of the range, i.e. the peak maximum go up to 4 to 6 volts.

The sample weight should be reduced when the IR-cell is saturated. However, when the weight is lower than 100 mg, the accuracy will be reduced due to the samples being not homogeneous and due to lower weighing accuracy.



3.2 TIC-determination

3.2.1 TIC-module

Due to the modular design of the CS-580 (Helios), the analyzer can be upgraded by a module for Total Inorganic Carbon (TIC) without further modification. For the TIC determination, the sample is treated with acid in the TIC module.

3.2.2 TIC analysis

The sample is treated with acid in an Erlenmeyer flask inside the TIC-module. The acid decomposes the carbonates in the sample, creating CO2. The oxygen flow purges the CO2 out of the flask through the gas flow system to the infrared detector.



Fig. 7: Installation of TIC-module





2	CO2 outlet
3	Glass distributor
4	Connection to the furnace
5	Acid supply
6	50 ml glass flask
7	Heater with magnetic stirrer
8	Elevator with variable height
9	Acid
10	Furnace
11	Analyzer
12	Moisture trap
13	TIC/TC toggle

3.2.3 TIC-module installation

- 1. The TIC module is placed next to the analyzer.
- 2. Connect the mains power plug to a power socket.
- 3. Connect the TIC module and the analyzer with the provided tubes. The connectors are located at the backside of the analyzer and of the TIC-module.
- 4. The outlet of the furnace is connected via the H_2O trap to the connection (2) and finally to the inlet (1) of the TIC module.
- 5. Inside the TIC module, this tube is connected to the input of the glass distributor (**3**) (connection on top tube 4).
- 6. The bottle of acid with dispenser (9) is placed to the right of the platform (8) and it is connected to the connection (5) of the glass distributor.
- The glass distributor (3) with the glass flask (6) is adjusted properly when the whole surface of the bottom of the glass flask (6) touches the surface of the heater (7).
- 8. The heater (7) is switched on and the temperature is set to 75° C.
- 9. The stirrer is set to 400.







Fig. 8: Scheme CS580 with TIC module

3.2.4 TIC-module Operation procedure

- 1. Place the empty glass flask (6) on the balance.
- 2. Press tare.
- 3. Put the sample into the flask and enter the weight into the analyzer (F4button). When powder sample sticks in the flask neck, add 2 ml of water (but don't transfer the weight of the water!)
- 4. Place a magnetic stirrer into the flask and attach the flask to the distributor (3).
- 5. Raise the adjustable platform to support the flask. Check, and if necessary, readjust the flask so that its bottom lies flat on the heated platform.
- 6. Switch to TIC-mode with the TIC/TC toggle (13).
- 7. Start analysis (F5 or click START).
- 8. Inject acid in two or three doses, when the word "Analyzing" appears.
- 9. When all the CO2 has been released from the sample, the analyzer's signal will return to the baseline level and the analysis will be terminated.

10. switch to TC-mode with the TIC/TC toggle (13).

NOTICE

The rotary speed of the stirrer should be kept low. The rotary speed and the acid dosing should be done in a way to avoid sample particles being pushed up and stick on the inner glass surface.

The heater must be switched on. Do not allow boiling or evaporation of the solution in the flask!



The table below shows approximate sample weight and acid volume depending on the expected TIC content in the sample.

TIC-content	Sample weight	Acid
>5 %	100 - 200 mg	2×2 ml
1-5 %	200 - 500 mg	3×2 ml
<1 %	1000 - 2000 mg	3×3 ml

Used Acids:

Acetic acid 25% concentration or Phosphoric acid 50% concentration. NOTICE

Only the carbon of easily decomposable carbonates can be determined. Carbonates which are difficult to decompose cannot be measured. For example, elementary carbon (graphite, soot) and cyanides cannot be analyzed.

3.3 Applications

This chapter describes the operation modes for analysis of different sample materials. For further materials contact the manufacturer. *NOTICE*

The maximum weights of the samples should not be exceeded!

3.3.1 Coal

Temperature 1350°C

Weight: 300mg-500mg (Depending on the sensitivity of the IR cell and on the C- and S-content of the sample.)

No accelerators are needed. Only in exceptional cases, when the sample contains pyrites, the furnace temperature has to be set at 1500°C or approx. 300mg of iron phosphate have to be spread on top of the sample.

3.3.2 Calcium Carbonate

NOTICE

Calcium carbonate can be used for calibration in the range of 12% C. Temperature higher than 800 °C Weight: 100mg-500mg (Depending on the sensitivity of the IR-cell.)

3.3.3 Graphite

NOTICEAnalysers with a range of 100% C can be calibrated with graphite.Temperaturehigher than 1000 °CWeight:250mg

3.3.4 Limestone

NOTICECalibrate analyser with calcium carbonate of 12% C.Temperature1250 °C or higher.Weight300mg

3.3.5 Oil, Asphalt and Rubber

Temperature	1450 °C
Weight	80mg-100mg (fuel oil and other thick oils)
	Up to 50mg (thin oil analysis)

3.3.6 Wood

Temperature 1300 °C



Weight:350mgSet the minimum analysis time to 50s. Set the comparator level to 20mV.



4 Maintenance

4.1 General information

- Replace the magnesium perchlorate of the moisture trap of the furnace after 150 analyses. See chapter "Front panel illustration" and "Filling the reagent tubes". In order to save material, replace first the upper half. The next time replace the whole of the magnesium perchlorate. This is based on the analysis of coal samples. It is not necessary to replace the glass wool, unless it is penetrated by dust and particles from the magnesium perchlorate.
- The content of the oxygen pre-cleaning glass tube on the analyzer's front panel, see chapter "Front panel illustration", should be replaced every 300 analyses. The depleting of these chemicals depends on the purity of the carrier gas used. The CO2 absorber in the upper half of this glass tube changes its color from black to grey after saturation. The moisture trap in the lower half doesn't change its color. When this chemical is depleted, its particles stick together. It can be recognized visually.

NOTICE

According to our experience in most cases when users report deviation of the results, the problem is solved by replacing the magnesium perchlorate. This means that the importance of the condition of the chemicals is underestimated.

The following chemicals are used:

- Magnesium perchlorate (anhydrone) as moisture absorber
- Sodium hydroxide (ascarite) as CO2 absorber

The chemicals are replaced when they are saturated.

It is not possible to dry the magnesium perchlorate and use it again, as it is chemically changed after reacting with the moisture. The saturation of the sodium hydroxide changes its color (it turns to light grey).

The magnesium perchlorate is saturated if its particles do not move when tapping on the glass tube. It is essential to change the absorber before it becomes cloggy. The moisture absorber should be checked every 100-200 induction analyses and if necessary, it should be replaced (glass tube underneath the metal filter).

Please refer to the following schematics to identify the glass tubes on the analyzer. In addition to the reagents in the glass tube, fill the bottom end of the tube with glass wool. One should pay attention that the glass wool should be only as thick as necessary, otherwise the gas flow can be choked. Under no conditions should the amount of glass wool be less than that shown in the following schematics, otherwise fine particles of magnesium perchlorate can pass through the glass wool layer blocking the hole of the fitting underneath.

It should be pointed out that magnesium perchlorate is a very strong oxidizing material.

At both ends of the glass tube, you should leave sufficient space for the gas connections to be fitted. The free space at the tube ends serve as sealing space. They must be cleaned after filling. The O-rings must be cleaned. Both the O-rings as well as the sealing areas of the tube must be greased with high vacuum silicon grease. This will be easier to assemble or disassemble and further it improves the sealing of the glass tubes.

Make sure that the O-rings are completely sealed around the glass tubes, by looking at the imprint of the O-ring on the inner surface of the glass tube. Check whether there are fibers of glass wool trapped between O-ring and glass tube. This causes leakages.



The O-rings are only replaced when they can no longer adequately seal, due to a damage or age. When removing the old O-rings, be ensure that the sealing areas of the fittings are not damaged.

NOTICE

When replacing O-rings, never grease the new O-rings before installation. Otherwise, the O-rings will turn with the glass tube when trying to remove it.

Remark

There are qualities of magnesium perchlorate and sodium hydroxide which have been especially developed for combustion analyzers and other analytical instruments. The commonly available materials fulfill their specific purposes either inadequately or not at all.

The magnesium perchlorate which is commonly available causes memory effect and affects repeatability. Another typical effect is that the analysis takes too long and it is often not even completed. This effect also occurs with magnesium perchlorate of suitable quality when it is saturated.

The commonly available sodium hydroxide binds CO2 very inadequately at room temperature, whereas the special quality not only perfectly binds CO2 at room temperature but also contains an indicator.

The glass tubes and the O-rings should be lubricated with high vacuum silicon grease and not with ordinary silicone grease.

The user is free to test commonly available materials; the analyzer will not be damaged. If problems should arise, however, suitable materials, in proper, unsaturated condition, should be used, before calling technical service.

Remark

The bottles with chemicals must be closed very tight, immediately after use, so that they do not become saturated with air moisture or CO2.



4.2 Reagent tubes filling

4.2.1 Reagent tube replacing



Fig. 9: Installalling the reagent tubes

The reagent tubes are first lifted and then swung to one side, detached diagonally downwards and emptied.

Remark

The dimensions for filling the glass tubes given in the schematics of chapter "Reagent tubes filling" should be respected in all cases.

When, for example, there is a rest of quartz wool in the bottom of the glass tube, it is possible that dust, forming magnesium perchlorate can fall through and block the fitting below or this can damage the analyser and the infrared cell.

Remark

Before the reagent tubes are fitted, both, the O-rings and the inner ends of the tubes are lubricated with high vacuum silicon grease.

The components are refitted in reverse order.

NOTICE

The dimensions for filling the glass tubes should be respected in all cases to secure proper analyzing and to avoid any damages of the device (see chapter filling quantities). When there is not enough glass wool in the lower end of the glass tube, it is possible that dust of magnesium perchlorate can fall through and block the fitting below. This can damage the analyzer and the infrared cell.

Also with the right hand glass tube (moisture trap after the furnace outlet), one must pay special attention that enough magnesium perchlorate is available. Otherwise, water vapor can condense on the inside free glass wall before it reaches the magnesium perchlorate.

NOTICE



Before the reagent tubes are fitted, the O-rings and the inner ends of the tubes should be lubricated with high vacuum silicon grease.

4.2.2 Filling quantities

As different samples can contain different amounts of moisture, it is hard to give a precise number of analyses that can be made before the content of the reagent tubes should be replaced.

A regular check of the reagents is essential to secure a failure free operation of the analyzer.

The moisture stems from the combustion and has two sources. One source is the water content of the sample. By drying the sample in a furnace at about 80 °C, the moisture content can be reduced. The sample is laid in a fairly flat bowl and should be spread as thin as possible. The drying process takes some hours.

The second source is the hydrogen content of the sample itself, available in various hydrogen compounds.

The following chemicals are used

Magnesium perchlorate (Anhydrone) Sodium hydroxide as moisture absorber as CO2 absorber

The reagent tubes have to be replaced when they are saturated. If the absorber particles do not move when tapping on the glass, it is a sign that the magnesium perchlorate is saturated. It is essential to change the absorber before it is completely solid. The sodium hydroxide changes its color after being saturated (it turns to light grey).

It is not possible to dry the magnesium perchlorate and to use it again, as it is chemically modified after reacting with the moisture. It should be pointed out that magnesium perchlorate is a very strong oxidative material.

A clear sign of total saturation is when the absorber's particles do not move freely after tapping at the glass tube. Normally, the moisture absorbers should be checked after 30 to 40 analyses

The grade of the absorber saturation is at its maximum at the gas entrance of the tube and it becomes gradually less toward the glass tube outlet. Therefore, once the upper half only of the magnesium perchlorate can be replaced and next time the complete content of the glass tube.

Please refer to chapter front panel illustration to identify the glass tubes on the analyzer.

Before filling the reagents into the glass tubes, their lower end has to be stuffed using glass wool. One should pay attention that the layer of glass wool should be pressed only as strongly as necessary, otherwise the flow of gas could be choked. Under no conditions the amount of glass wool should be less than that given in the following schematics, as fine particles of magnesium perchlorate could penetrate the glass wool and accumulate at the bottom of the tube blocking the fitting and building deposits in tubes and in the Infrared cell paths.

At both ends of the glass tube, you should leave sufficient space for the gas connections to be fitted. The free spaces at the tube ends serve as sealing areas. They must be cleaned and greased after filling.

Both, the O-rings as well as the sealing areas of the tube are to be greased with high vacuum silicon grease. This enables easy reassembling as well as later removing. In addition, greasing improves the sealing of the tubes to the O-rings. Only the upper end of the right hand glass tube (moisture trap) and the corresponding O-ring should not be greased.

Make sure that the O-rings are completely sealed around the glass tubes!





Fig. 6: Filling quantities

Filling quantities allow tolerances of about ± 20 %.

	Filling	Ordering number
1	Glass wool	90331
2	Magnesiumperchlorat (Anhydrone)	90200
4	Empty	
5	Sodium hydroxide	90210



4.3 Moisture trap replacing (furnace)



Fig. 10: Replacement of moisture trap

- 1. Turn both screws (1) counter clockwise, until the ring (3) touches the part (2).
- 2. Lift the glass tube (5) upwards, tilt it to the side and pull downwards.
- 3. Replace the magnesium perchlorate (Anhydrone). See chapter reagent tube replacing
- 4. Install the glass tube (5) in reverse order after greasing the lower Oring.
- 5. Turn both screws (1) clockwise until the O-ring (4) is properly pressed on the inner surface of the glass tube (5).

Remark

The O-ring imprint on the whole circle length of the glass tube should be of about 2 mm width.

4.4 O-rings replacing

The O-rings are only replaced when they no longer adequately seal, due to severe damage or aging. When removing the old O-rings, be sure that the sealing area of the fittings is not damaged. Grooves where the old O-rings are installed must be cleaned to be completely free of grease.

New O-rings should under no circumstances be greased before installing but only after installation. Otherwise, the O-rings would turn with the glass tubes when trying to remove them at a later stage.

It is recommended not to grease the upper O-ring of the moisture trap.





Fig. 11: Preparation of moisture trap O-rings

4.5 Dust filter cartridge replacing

The dust filter cartridge filters smallest dust particles from the combustion gases. Its saturation depends on the sample material and its combustion characteristics. The filtering material of a new cartridge is white. Replace the dust filter cartridge when the filtering material shows coloration, or at least every 500 analysis.



Fig. 12: Dust filter cartridge - inserting

- 1. Lift the dust filter cartridge (**1**) upwards.
- 2. Tilt its low end to the front and pull downwards.
- 3. Install a new cartridge by acting in reverse order.



Take care for the cartridge body to have the smaller diameter up and the bigger down.

4.6 Combustion tube replacing



Mortal danger from electric shock

Exposed power contacts - High Voltage

- An electric shock can cause injuries in the form of burns and cardiac arrhythmia, respiratory arrest or cardiac arrest.
- Disconnect the mains power plug before opening the analyzer's cabinet.





Fig. 7: Removing the combustion tube

- Switch off the analyzer and disconnect the power plug.
- Remove the cover panel (1) of the cabinet. Unscrew 4 screws (2) on top.
- Remove the tubes (3), (4) and (5) from the upper furnace closure assembly (6/7).
- Remove the 4 screws (8) which hold the assembly (6/7) on the furnace. The 4 screws and nuts holding the disk (7) on the part (6) should remain in place.
- Lift the upper furnace closure. The assembly consisting of the parts (6), (7), (9), (10) and (11) is then taken out of the furnace. Take care



to lift it in straight vertical direction to avoid collision of the lance (11) with the combustion tube (12). If the assembly is not free to be easily lifted, slightly tilt it carefully in different directions until it becomes free.

- Unscrew the 4 screws (13) by holding the ring (14) to prevent the combustion tube (12) from falling down.
- Remove the ring (14).
- Remove the O-ring (15). If this O-ring and the combustion tube remained in the furnace after lifting the assembly (6/7), take care to prevent the combustion tube from falling down out of the furnace. This can be done by keeping one hand underneath the furnace or by supporting the ring (14) with two manually driven screws (13).
- Lift the combustion tube (12) out of the furnace top. Lift it in straight vertical direction to avoid collision with the heating elements.
- Place a new O-ring (9) on a new combustion tube (12).
- Reinstall in reverse order.

NOTICE

Enter the new combustion tube very carefully in straight vertical direction to avoid collision with the heating elements.

4.7 Lance replacing



Mortal danger from electric shock

Exposed power contacts - High Voltage

- An electric shock can cause injuries in the form of burns and cardiac arrhythmia, respiratory arrest or cardiac arrest.



• Disconnect the mains power plug before opening the analyzer's cabinet.



Fig. 8: Lance replacing

- Switch off the furnace and disconnect the power plug.
- Remove the cover panel (1) of the cabinet. Unscrew 4 screws (2) on top.
- Remove the tubes (3), (4) and (5) from the upper furnace closure assembly (6/7).



- Remove the 4 screws (8) which hold the assembly (6/7) on the furnace.
- Lift the assembly (6/7) in straight vertical direction.
- Unscrew the four screws and nuts which hold the parts (6) and (7) together.
- Replace the lance (11)
- Replace the O-ring (10)
- Replace the O-ring (9)
- Do reassembling in reverse order

4.8 Heating elements replacing

DANGER

Mortal danger from electric shock

Exposed power contacts - High Voltage

- An electric shock can cause injuries in the form of burns and cardiac arrhythmia, respiratory arrest or cardiac arrest.
- Disconnect the mains power plug before opening the analyzer's cabinet.





Fig. 9: Operating procedure for replacement of heating elements

- Remove the combustion tube. See chapter "Combustion tube replacing"
- Remove the two screws (1) fixing the right hand side panel (2) of the analyzer's cabinet.
- Slightly tilt the panel to the right and carefully lift it. The loader should not be shifted otherwise it will lose its mechanical adjustment.
- Unscrew the two screws (3) and remove the heat shield (4).
- Unscrew the four nuts (5) and remove the panel (6).
- Loose the four nuts (7) and remove the two double clamps (8).
- Remove the four ceramic spacers (9).
- Apply the panel (6) in its original place and manually fix it with two nuts (5) diagonally to prevent it from falling down.
- Unscrew the four screws (10) and remove the panel (11).
- Remove the two nuts (12)



- Loose the four nuts (13) and remove the two double clamps (14).
- Pull up the four heating elements (15)
- Reinstall in reverse order using new heating elements.

Remark

Install the clamps (14) in a position which allows the heating elements to move at least 5 mm in vertical (axial) direction. Insert the thermocouple deep enough, to touch the combustion tube and fix it with the spring.



5 Function description

5.1 Measuring principle

The measuring method is based on the principle of sample combustion and the analysis of the combustion gases, using infrared absorption.

A wide variety of sample materials in various forms is possible, like powders, grains, chips, solid pieces and also some materials in liquid form. Typical materials are coal, ashes, steel, cement, soil samples, rubber, oil etc.

During combustion, the sulphur and carbon components present in the sample are oxidized to form SO_2 and CO_2 . Typical combustion temperature is 1350°C.

Combustion is obtained by supplying oxygen which at the same time acts as carrier gas. An electronic flow controller keeps the flow quantity at a constant level of 180 l/h.

Dust traps and a moisture absorber ensure that a dry, dust free gas mixture is supplied to the infrared cells.

The output signals of the infrared cells are selective and correspond to the SO_2 and CO_2 concentrations in the gas mixture. They are electronically linearized, integrated, divided by the sample weight and displayed as % S and % C values.

Since the sample weight is taken into account, the results don't depend on the sample weight. For this purpose, the sample is weighed before analysis and its weight is transferred to the PC. If necessary, blank values can also be entered; the PC takes them into account when calculating results.

The graphical representation of the detectors' signals (peaks) is shown on the PC's screen during and after analysis. At the end of analysis the results are displayed as well. All analyses data for every finished analysis are saved in the PC and remain available for review, results recalculation, calibration, etc. and they can be printed out on a printer or exported to another software, if necessary.



5.2 Gas flow system



Fig. 13: Gasflow (Scheme)

1	Carrier gas inlet	15	Dust filter cartridge	
2	Pressure regulator	16	Pressure regulator	
3	Oxygen stop valve (power switch control)	17	17 Flow adjustment restrictor	
4	Pressure switch and pressure gauge	18	Gas flow meter	
5	Oxygen stop valve (software controlled)	19	19 Infrared cell	
6	Flow adjustment restrictor	20	Gas flow meter	
7	Carrier gas flow meter	21	Attenuator before the pump	
8	Gas pre-cleaning unit	22	Gas flow pump	
9	Oxygen diverting valve	23	Attenuator after the pump	
10	Flow adjustment restrictor	24	Microcontroller	
11	Gas flow meter	25	Computer	
12	Upper furnace closure assembly	26	Monitor	
13	Flow adjustment restrictor	27	Printer	
14	Moisture trap	28	Balance	



The analyzer is operated with pure oxygen. A purity of 99.5% is sufficient. It is normally available in steel bottles. CO2 and H2O impurities in the oxygen are trapped in the trap (8). The upper half of the trap is filled with CO2 absorber and the lower half with moisture absorber.

Magnesium perchlorate (anhydrone) acts as a H2O absorber.

Sodium hydroxide acts as CO2 absorber, preferably with an indicator, so that the degree of saturation can be seen from the coloration.

The oxygen inlet pressure should be 2 to 4 bar, which is then regulated inside the analyzer to 1.5bar, as shown on the pressure gauge (4). Any pressure fluctuation of the external oxygen supply has no influence on the accuracy of the measurements.

A pressure switch (4) becomes conductive at around 1 bar (15 psi). This information is passed to the software which energizes the valve (5) and starts the gas pump (22) as long as the pressure is higher than 1 bar. This ensures that the pump runs only when sufficient pressure of oxygen is available, so that no air enters the gas flow system, if there is no input oxygen pressure.

With the help of the adjustable restrictor (6) the inlet flow is set at a level of at least 200 l/h. The flow rate is shown on the lower flow meter (7) on the front panel. This flow rate enters the furnace. During analysis when the furnace is closed, the inlet flow rate is split. One part enters the furnace top to exit the lance over the crucible. This flow is shown on the middle flow meter (11). The rest of the oxygen flow enters the furnace at the low end. The ratio of the two flows can be adjusted with the restrictors (10) and (13).See chapter "Adjusting gas flow".

The flow at the outlet of the furnace is created by a pump (22) at the end of the gas flow system, which sucks a flow of 180L/h out of the furnace.

A tube connects the furnace outlet with the moisture trap (14). The gases that come out of the furnace normally contain some moisture. This particularly happens when analyzing coal samples. The dried gases pass thru the dust filter (15) and then enter the Infrared cells which are accommodated in the cell module (19), where the measurements of CO2 and SO2 take place.

The gas flow through the cells is shown on the upper flow meter (20) on the front side of the analyzer. The displayed value must be at constant 180 l/h. The attenuator (21) makes the pulsing flow of the pump (22) smooth. The flask (23) is a silencer for the pump exhaust.





Fig. 14: Gas flow



5.3 Infrared cell

The measuring principle is based on the infrared radiation absorbing properties of many gases. Each of these gases absorbs specific characteristic spectral wavelengths of infrared radiation. The absorption spectrum is determined by the number, configuration and type of the atoms in the gas molecules.

Graphic: Infrared cells with flexible measuring range



Fig. 15: Infrared cell

An infrared source is electrically heated emitting wide band infrared radiation. The radiation beam is interrupted by a rotating chopper blade, resulting in alternating light. The rotary speed of the chopper is crystal controlled, so that the chopper frequency is very stable. The infrared radiation then passes through the measuring IR-paths, through which a mixture of combustion gases and carrier gas flows.

Depending on the composition of the gas mixture, certain frequencies of the infrared spectrum are absorbed. The rate of absorption depends on the concentration of the gases.

As the infrared beam leaves the IR-path, it passes through an infrared filter, which allows only a certain narrow band infrared radiation to pass. This narrow band must correspond to the IR wavelength for which the gas to be detected has its maximum absorption capability.

The intensity of the radiation after the filter thus corresponds to the concentration of a specific gas in the path. The beam finally strikes a solid state infrared sensor, giving an electrical signal corresponding to the intensity of the beam.

As the beam is interrupted by the rotating chopper, the detector receives an alternating radiation creating an AC electrical signal. Temperature and aging influences of the detector, as well as noise are thereby strongly reduced. The signal obtained is amplified, rectified and passed thru a low pass filter so that it leaves the infrared cell as a DC voltage.

The infrared cells utilize solid state sensors combined with infrared filters.

The lengths of all four cells can be individually optimised to obtain maximum precision for the target analysis levels of each customer. Each of the cells can be installed with infrared absorption lengths ranging from 1mm to 320mm. The



infrared cell rack is temperature controlled so that the sample gas flowing through it is kept at a constant temperature.

5.4 Micro-controller unit and PC Software

The microcontroller unit (MCU) contains all components for signal processing and control sequencing. It is working under the control of the PC software, collecting and processing signals and sending data to the PC-software. The communication between MCU and PC is taking place via USB interface.

The first stage of signal processing in the PC is the linearization. This is necessary because the output signal of the IR cells as a function of the sample gas concentration have an exponential characteristic.

The linearization produces a correction which is exactly opposite to the characteristic of the detector, so that a linearized analysis signal is available.

The second stage is integration. It starts at the beginning of the combustion and it ends after a pre-set minimum time. In case the combustion takes longer, the integration is prolonged. Then, the detector signal is compared to a comparator level. The integration continues as long as the IR-output signal is higher than the comparator level. When it comes down below this level, the integration is stopped. Should the IR-signal remain above the comparator level, the analysis is interrupted by a set maximum time. Then the integration is terminated.

The integrated value is directly proportional to the SO_2 resp. CO_2 content in the combustion gases. The blank value is deducted from the integrated value.

The integrated value is then multiplied by the calibration factor. After dividing by the sample weight, a result is obtained which does not depend on the sample weight.

With the multipoint calibration the results can be calculated to match exactly the values of certified standard materials.

Finally the results are directly shown as % S or % C on the PC screen.

For instructions of operating the PC-software, please refer to the Help-function of the Software.



6 Ordering numbers

6.1 Analyszer front view





Part Nr.	Description
09090	Reagent tube
09092	Alternative reagent tube with bigger capacity
09093	Alternative reagent tube with maximum capacity
11170	Dust filter cartridge
11480	Adjustable restrictor
15087	Flow meter 300l/h
15095	Flow meter 600l/h
36700	Upper reagent tube holder
36710	Lower reagent tube connector
48067	Lower reagent tube connector
48069	Upper reagent tube connector
70320	O-ring



72010	Pressure gauge 2.5 bar for oxygen
72020	Pressure gauge 10 bar for compressed air
78017	Mains power switch

6.2 Rear side (outside view)



Fig. 11: Rear side inside view

Part Nr.	Description
14618	Microcontroller board UNI 1.4
15268	Pump control boart PC1
35490	Pump support
15270	Gas pump
35325	Attenuator volume
35336	Attenuator volume
35456	Blower
60234	Pneumatic valve
60522	Fitting 90°



60552	Adjustable restrictor
77032	Circuit breaker
77140	Mains power filter
77145	Mains power filter
77460	Switch

6.3 Bottom (outside view)



Fig. 12: Bottom view

Part Nr.	Description
11035	Cooling fan
11492	Oxygen pressure regulator



6.4 Left side view (inside)



Fig. 16: Left side view (inside)

Part Nr.	Description
06441	IR module cable
11390	2/2-way valve 220VAC
11435	3/2-way valve 24VDC



11480	Adjustable restrictor
11490	Pressure regulator
11492	Oxygen pressure regulator
15083	Flow meter 15 L/h
16114	Power supply
18468	Microcontroller board UNI2
26320	Insulator
35536	2/2-way valve 24VDC
35779	Power/temperature control cable
36284	Heating element connector
36798	Transformer
36904	Temperature control board TH 44
36914	Thermocouple
36915	Power/temperature control board
60353	Piston sensor
77051	Thyristor block
77052	Solid state relay
78051	Pump control cable
78052	USB cable for PC interface
78072	Current transformer



6.5 Furnace side (inside view)



Fig. 13: Furnace inside view (inside)

Part Nr.	Description
35408	Lower plate insulation
35462	Upper plate
35512	Ceramic plate
35540	Combustion tube
35541	Ceramic lance
35542	Pedestal
36284	Heating element connector
36289	Heating element clamp



70150	O-ring
70427	O-ring
70428	O-ring
77501	Heating elements, 4pcs
77505	Ceramic spacer
90150	Crucible



6.6 TIC-module



Fig. 17: TIC-module [35543-9008]



1	Glass stopper (connection to the analyzer)	38225
2	Upper moisture trap connector	11042
3	O-ring 9*3	70230
4	Moisture trap	11064
5	Glass distributor	38200
6	50 ml glass flask	90090
7	Heater with magnetic stirrer	71070
8	Support with variable height	38400
9	O-ring 9*3	70230
10	Lower moisture trap connector	11045
11	Geared mechanism	38850
12	Glass stopper (connection to the furnace)	38227
13	Glass stopper (connection to the acid supply)	38220
14	Glass support	38340
15	Dispenser Assimat	71065
16	Acid bottle	71060
17	Bottle support	38677



6.7 Packaging



Fig. 14: Packing

Before packing, the analyzer must be wrapped in plastic foil to protect it from moisture and dust, and then be placed in a wooden case. The wrapped analyzer should be surrounded by a layer of plastic foam (or at least chips) of at least 10cm, in order to avoid any damage during transport.

Especially the foam where the analyzer is put on is very important. It should neither be too hard nor too soft. When the foam is too soft, the analyzer will practically touch the wood. Fix the foam on the bottom of the wooden case by gluing.

The small loader (with 36 trays) can be packed in the wooden case together with the analyzer. The bigger loaders (for 104 and 130 crucibles) however, are to be packed in separate carton boxes and protected by foam or another shock absorbing material. The transportation of these carton boxes has to be made on pallet.

The glass tubes must be empty.



7 Pre-installation guide

Following requirements apply, when installing the analyzer:Carrier gasOxygen 99.95 % pure; 2 - 4bar (30 - 60psi)Mains power supply230VAC ±10%, 50/60Hz; 20A fuse
CEE-Plug 230V, 32A



Analyzer dimension	550 x 970 x 600mm
Loader dimension	850 x 350 x 450mm (depending on version)
Analyser weight	approx. 90kg
Loader weight	approx. 55kg
_ , , , , , , ,	

The balance should rest on a vibration free support.

Loader dimensions: Version 36p: 500x250x460mm Version 130p: 900x500x460

Gas connections: The supplied tubes carry a connector with G¹/₄" inner diameter ".



Fig. 15: Carrier gas tube

Connections for compressed air;

The tubes supplied together with the analyzer, carry a connector with $G^{1\!\!\!/}_{4}$ inner diameter.



Fig. 16: Compressed air tube



8 Approved methodologies to which Eltra instruments conform

8.1 Inorganic materials (Metals)

Norm	Elements	Materials	Instruments
DIN EN ISO 9556:2002-04	с	Steel and Iron	CS-800 CS-2000
ISO 4935:1989 DIN EN 24935:1992-07	s	Steel and Iron	CS-800 CS-2000
ASTM E 1019:2011	C,N,O,S	Steel, Iron, Nickel / Cobalt Alloys	CS-800 CS-2000 ON-900 OH-900 ONH-2000
ASTM E 1587:2010	C,N,O,S	Refined Nickel	CS-800 CS-2000 ON-900 OH-900 ONH-2000
ASTM E 1409:2013	N,O	Titanium and Titanium Alloys	ON-900 OH-900 ONH-2000
ASTM E 1569:2009	0	Tantalum	ON-900 OH-900 ONH-2000
ASTM E 1937	N	Titanium and Titanium Alloys	ON-900 ONH-2000
ASTM E 1447:2009	н	Titanium and Titanium Alloys	OH-900 ONH-2000
ASTM E1915 - 13	C,S	Metal Bearing Ores and Related Materials (i.e. tailings, waste rock)	CS-580 CS-800 CS-2000
UOP703 - 09	с	Catalysts	CS-580 CS-800 CS-2000



8.2 Organic materials (Oil, Coal, foodstuffs)

Norm	Elements	Materials	Instruments
ASTM D 1552:2008	S	Oil and Petrolium Products	CS-580
			CS-2000
ASTM D 4239:2013;	S	Coal and Coke	CS-580
			CS-2000
ASTM D 5016:2008	S	Coal and Coke Ash	CS-580
			CS-2000
ASTM D 1619:2011	S	Carbon Black	CS-580
			CS-2000
PN-G-04514-17:1993P	S	Coal and Coke	CS-580
			CS-2000
DIN EN 13137:2001-12	С	Waste	CS-580
			CS-2000
DIN ISO 10694:1996-08	С	Soil samples	CS-580
			CS-2000



9 Disposal

Please observe the respective statutory requirements with respect to disposal. Information on disposal of electrical and electronic machines in the European Community.

Within the European Community the disposal of electrically operated devices is regulated by national provisions that are based on the EU Directive 2002/96/EC on Waste Electrical and Electronic Equipment (WEEE).

Accordingly, all machines supplied after 13.08.2005 in the business-to-business area to which this product is classified, may no longer be disposed of with municipal or household waste. To document this they have the following label:



Fig. 18: Disposal label

Since the disposal regulations within the EU may differ from country to country we would request you to consult your supplier.



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