ON - 900 Oxygen / Nitrogen Determinator





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OPERATION MANUAL ON - 900 as from serial no. 1550xxxxxx

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1 INSTALLATION

1.1 Setting up

Since the analyser weighs about **130 kg** it should be placed on a suitably stable surface. The balance should also be placed free of vibration.

The balance can be placed in any position, positioning it to the right of the analyser has proved to be best suited.

The balance can of course also be placed on a weighing table next to the analyser.

There are no special requirements for setting up the printer and computer; they can be placed on a normal desk.

Below is an example in installation:



Although the analyser's operating environment does not necessarily need to be air conditioned, it is advisable to keep the room temperature between **18°C and 30°C**.

Never run the water pump without any water or else it will be damaged! The pump is activated when the mains switch is set to pos. 2.

First fill up the cooling system with water, according to chapters 1.6 and 1.7.

Under no conditions should the device be placed in direct sunlight ! Avoid places exposed to the wind of air conditioners or to the wind blowing through open windows or doors.

1.2 Front panel illustration



- 1 Current meter
- 2 Furnace inlet flow
- 3 Analytical flow (controlled)
- 4 Purge flow regulator
- 5 Analysis flow regulator
- 6 Sample drop
- 7 Crucible pedestal
- 8 Pneumatic lift
- 9 Upper furnace part
- 10 Lower furnace part

- 11 Dust trap
- 12 Carrier gas pressure gauge
- 13 Compressed air gauge
- 14 Copper oxide catalyst
- 15 Catalyst furnace
- 16 Mains switch
- 17 Carrier gas pre-cleaning
- 18 CO_2/H_2O trap
- 19 IR-cell purge pre-cleaning

1.3 Mains power connections

Since the infrared cell requires about **1 hour** to reach a stable operating temperature, it is advisable to connect the analyser to the **mains power** first and switch on before further installation work is carried out.

This waiting time is only necessary when **installing** the analyser. Since it is normally not switched off, and is always in operating temperature.



- 1 Analyser
- 2 Computer
- 3 Monitor
- 4 Printer
- 5 Balance
- 6 Triple plug
- 7 Analyser mains plug

First connect the analyser to the mains power and switch it on. The switch, located on the fore side of the analyser, is to be set to **position 1**.

1.4 Data interfaces



Rear side of UNI 1.3 board:

- 1 Pear serial interface
- 2 PC connection
- 3 Analog input/output signals
- 4 Digital input/output signals
- 5 Autoloader connection

When all the units are connected to the mains power, then data connections can be made. The plugs are all different from each other, so that they cannot be interchanged. The required data cables are included if the additional units are supplied. These are adapted to the interfaces when the analysers are put into operation in our company.

As the **balance** transfers the weight to the PC, its serial interface must be programmed.

The **computer** is already provided with an operating system and software for controlling the analyser.

<u>NOTE</u>: For all instructions on operating the PC software refer to the Help-function of the software.

1.5 Gas connections



Two gas connections are necessary for the operation of the analyser. The required tubes are included in delivery. **See above drawing**

Tube (8) is for the carrier gas supply, it is soft and transparent.

Tube (6) for the compressed air is harder and opaque.

These are delivered, provided with screw fittings for pressure regulators.

An **R**¹/₄" inner thread as well as the corresponding copper seals are also provided.

Tube fitting (8) connects the analyser with a helium carrier gas bottle

These connections must be very secure, since the operating pressure in the tube is **2-4 bar** (30 to 60 psi). Gas connection **(6)** is for the compressed air supply to the pneumatic furnace lock.

Gas connection (7) is for drawing off outlet gas. It is generally not in use, however, since only low quantities of **CO**₂ and even lower quantities of **N** result from the sample combustion.

When the analyser's **mains switch** is set to **position 2** a valve opens, and the carrier gas flows through the gas tubes. The flow rate is stabilised after several seconds to **15 l/h** and can be read from the lower flow indicator. At the same time the cooling water pump starts.

1.6 Cooling water

The **ELTRA ON-900** has two cooling water circulation systems, a primary and a secondary system.

The primary system provides furnace cooling, and consists of:

- The external barrel.
- The internal section of the analyser, which includes water pump, equalising tank and furnace.

The cooling water circulates from the barrel to the furnace, via the connections (2). It then returns to the barrel, via the connections (1). Inside the barrel, the water is kept cool by the inter-cooling coil of the **primary** system.

The **secondary** system starts from the drinking water tap, the water is then fed to the water flow switch, via connections (5). The water then flows to the inter-cooling coil inside the barrel, via connections (4). Finally, the water flows to the drain, via connection (6)



Connections:

The analyser and the barrel is connected as shown in the **above drawing**.

It is possible to install the barrel away from the analyser. The barrel should not be installed more than **5m** away and **1m** above the instrument. If this is not possible, ask the factory for a modification.



1.7 Filling the cooling water

Before filling the **cooling water**, it is necessary to make sure that the analyser is **properly** connected to the external barrel, see <u>1.6</u>.

It is absolutely **important** that the inlet of the water pump inside the instrument, is connected to the central tap (2) of the external barrel.

Procedures for filling up and bleeding the cooling system:

- Open the external barrel and put the additive "COOLING AGENT, into it. Fill up the barrel almost completely with water.
- Only drink water quality should be used and not any tap water which can pollute or corrode the cooling system.
- Close the barrel.
- Open the air bleeder on the water pump.
- Pour water into the small plastic water equalising tank above the pump, until some water starts to exit the air bleeder.
- Close the air bleeder.
- Place a graphite crucible into the graphite tip.
- Make sure that the carrier gas supply is closed, in order to avoid unnecessary gas consumption during the following steps.
- Remove the right side panel of the analyser. <u>Caution</u> ! Don't touch electric components.
- Observe the wheel of the water flow detector on the ON-21 board and switch the mains switch to pos. 2 for a couple of seconds only and check during this short period if the water flows.
- Set the mains power switch **immediately** to **position 1** if the water wheel doesn't rotate. It seams that the shaft of the water pump is jammed.

The pump can be damaged when power is applied and the pump will not rotate.

Reopen the air bleeder and add some more water into the equalising tank. Close the air bleeder, switch back to **pos. 2**. Repeat this procedure until the system is completely free of air.

When the cooling system is inactive or dry for a long period of time, it could occur that the water pump doesn't start rotating when the mains switch is set to **pos. 2**

The following instructions will assist you in solving the problems:

- Remove the right side panel of the analyser. <u>Caution</u> <u></u>! Don't touch electric components.
- Remove the screw on the pump's front place.
- The screw that appears next is coaxial to the internal pump shaft, rotate it and the jammed shaft will be loosened. Up to 10 rotations may be needed to restore back to working order.
- Re-install in reverse order.
- Set the mains power switch to **position 2**.
- If the water flow wheel rotates smoothly over a long period of time, the filling-up procedure is complete.
- If the rotation is **erratic**, then there is still some air in the cooling system.
- Switch the mains switch back to **pos. 1**, reopen the air bleeder and add some more water into the equalising tank.

- Close the air bleeder, switch back to pos. 2. Repeat this procedure until the system is completely free of air.
- Close both side panels.



1.8 Adjusting the gas flow

There are two different gas flow systems in the instrument.

Flow meter **(B)** shows the controlled flow in the system, which cannot be changed from the front panel. Flow meter **(A)** shows the total gas flow in the furnace.

There are two different states, which must be adjusted.

- **1.** Only a small flow during the analysis or in normal state with closed furnace.
- **2.** But it needs a higher flow for purging during the out-gas phase or with an open furnace.



Adjust the gas flow as follows:

- Adjust approx. **2 to 4** (30 to 60 psi) on the pressure regulator of the carrier gas/cylinder.
- Enable the carrier gas supply to the analyser.
- Make sure to have a graphite crucible on the graphite tip.
- Set the mains power switch to **pos. 2**.
- Close the furnace by pressing the corresponding button in the software and wait about 10 seconds.

<u>NOTE:</u> For all instructions on operating the PC software refer to the Help-function of the software.

- Adjust the lower flow regulator (D) until the left flow meter (A) shows 30 l/h.
- Open the furnace.
- Adjust the upper regulator (C) until the same left flow meter (A) shows 50 l/h.
- Close the furnace again. If the above settings are unstable, increase both flows by about 10 l/h.
- Regarding the flow meter (B), the gas flow is adjusted in our company. This adjustment is normally not changed. If the blind values of O and N are higher than 20ppm, then the gas flow could be wrong.

CAUTION:

The furnace should be open only as long as absolutely necessary. If the furnace is unnecessarily opened to long, you waste purging gas and additionally the electrodes may oxidise from the air.

2 ANALYSIS

2.1 Working procedure

The **ELTRA ON-900** analyser is designed for the analysis of **metals**. Should other materials be analysed, take care that the furnace and the gas flow are not contaminated by **dust** or other combustion products.

The analysis procedure is described in the following section.

<u>NOTE:</u> For all instructions about operating the PC software refer to the Help-function of the software.

- 1. Ensure that the compressed air and the carrier gas are connected. Turn the mains power switch to **position 2**. The pump starts working and the gas flow is controlled. It takes a few minutes until the analyser is ready to work. Put an empty crucible on the lower electrode and close the furnace.
- 2. A sample of about **1 gram** is weighed. Transfer the sample weight into the PC software by transferring the value from the connected balance or enter it manually. The sample ID may be entered as well. Put the sample with clean tongs into the sample drop on top of the furnace.
- 3. **Start** the analysis. From now on the analysis is performed automatically. At the end of the analysis the results are displayed and all data are saved on the hard disk of the PC.

The analysis procedure consists of several phases following one after another. Depending on the settings (furnace power mode and sample loading mode) the sequence is altered. Here, the sequence in ON-OFF-ON furnace power mode and automatic sample loading mode is described as a basic and only the difference of other modes is specified.

ON-OFF-ON furnace power and automatic sample loading mode:

- 1. **Outgasing**. The graphite crucible itself (without sample) is heated up in the furnace in order to remove impurities. The carrier gas supply to the furnace is now in the purge mode. The time for outgasing and the furnace power are adjustable.
- 2. **Purging**. The furnace purging is continued after outgasing phase. The furnace is switched off. The duration is adjustable.
- 3. **Stabilising**. The carrier gas supply to the furnace is switched to the analysis mode. This phase is necessary for stabilizing the base lines of the detectors. The duration is adjustable.
- 4. **Sample drop**. The sample drop mechanism rotates and the sample drops into the crucible.
- 5. **Analysis**. The furnace is switched on, the sample is melted and the signals from the detectors are processed. The furnace is switched off after the pre-settable time expires, but the signals processing continues until the comparator level of all detectors or the maximum analysis time is reached. In order to avoid unnecessary signal integration from the moment when the furnace is switched on until the moment when the analytical gas reaches the detectors an integration delay time can be adjusted. All settings are available in the software.

CONTINUOUS furnace power and automatic sample loading mode:

In this mode, the furnace remains switched on after the outgasing phase and its power level is set to the analysis power level. The furnace will be switched off only when the comparator level of all detectors or the maximum analysis time is reached.

ON-OFF-ON furnace power and manual sample loading:

In this mode, the operator is prompted to open the furnace and put the crucible with the sample into the furnace after the **purging** phase. After closing the furnace the analysis is continued.

Please, refer to chapter <u>2.4</u> for instructions about the settings for making analyses of different materials.

2.2 Work breaks

CAUTION:

Keep the **furnace closed** to save carrier gas and to avoid oxidation of the electrodes.

Work breaks, e.g. during **lunch breaks**, the mains switch remains on **position 2**. During **longer** interruptions, e.g. after finishing work for the day, the mains switch is set to **position 1** (standby). The analyser's thermostatic control is then still working and no long warm-up time is needed, when re-starting the analyser. Energy consumption and wear are negligible on standby.

The mains switch is set to **pos.2** for about **10-15 minutes** before starting the first analysis.

Air, and any moisture which has entered the analyser is expelled by the **oxygen flow**. The slight influence which the **oxygen flow** has on the temperature of the **infrared cell** is balanced out. The analyser may only be switched off **(pos. 0)** when it is not used for several days or weeks. The analyser is designed for long term use, so that no **damage** results.

The furnace should always be kept closed during work breaks, so that no moisture can enter. The furnace only remains open when the analyser is completely switched off. The mains switch is only set to zero for safety reasons, the crucible lift is then at the bottom

2.3 Fractional analysis

1. Introduction.

In some cases it is useful not to only know the total content of **one** element in a specific sample, but also the exact amount of a specific **bond** or **structure**.

With the **ELTRA ONH-2000** we are able to separate different **oxygen** and **nitrogen** phases in the material to be tested.

It is necessary to **separate** the different **oxides** in **ores** and **raw** materials, to **guarantee high quality** products and to control the production process more efficiently. Together with the elemental analysis, the engineer obtains information about the **iron**, **silicon**, **aluminium** and **magnesium oxides** in row material. It is therefore possible to calculate the alloy elements and elements in the slag more accurately, before it is melted. The exact amount of different **oxygen** and **nitrogen** phases in a **high alloy steel** is of vital importance, to determine the quality of this product.

There are several reasons why **fractional** analysis is so **important**. Every time when a certain process needs to be optimised or when the quality of a product needs some improvement, there is a demand for more detailed information about the material in use; more than what the simple elemental analysis can offer.

2. Basics.

It is well documented, that the dissociation of various **oxides** and **nitrides**, i.e. the **iron oxides** occurs at approximately 1200°C, of silicon oxides at about 1500°C and of **aluminium oxides** at 2200°C. It is possible therefore to separate these various **oxides**, due to their different dissociation temperature.

There is however one problem:

The **oxides** don't discompose exactly at a certain specific temperature, as it happens for instance when melting a crystal. The velocity of the dissociation increases exponentially with the temperature. This means that below the specific dissociation temperature, an **oxide** discomposes very slowly.

In practice we need mathematical methods to calculate the contents of each **oxide**. It is not possible to obtain the **oxide** value only by temperature analysis. Once the analysis has delivered precise results, the signals from the **oxygen** analyser has to be divided.

3. Preparations.

The first step is to **calibrate** the temperature in the furnace, in accordance to the power setting of the instrument. This temperature depends on the **carrier gas material**, the analytical gas flow, the cooling parameter and the special furnace geometry. Each furnace must be calibrated individually, because the parameters of the instrument are **optimised** for each **customer** individually.

In combination with the power settings, it is possible to **plot** a calibration curve which shows the temperature in relation to the furnace power.

The value of the temperature **inside** the crucible can be obtained, for instance, by melting different materials, using the analyser.

Adjust the power of the furnace so that a material of a known melting temperature has nearly melted.

A **satisfactory** temperature-power relationship of the instrument, can already be established by melting a **minimum** of only **three** different materials.

4. Detecting the oxygen fractions

First, we need some information about different **oxygen** fractions in a material. An elemental analysis with a **spectrometer** is helpful. We can only get **oxides** from present elements.

We start by treating the sample with furnace power that increases linearly, to determine which **oxides** are present in the sample. A typical pre-selection for furnace power rampfunction, is from **1.0 kW** to **5.0 kW** in **200s**. The **oxygen** profile of the tested material is shown in figure 1



Figure 1

The next step is to separate each **oxygen** phase, as accurately as possible. We change the **temperature** program from a **ramp** function to a **step** function. We choose the power setting of each level in such a way, that the temperature is sufficiently **high** to discompose just one **oxide**, but still too low to brake the next one. The duration of each temperature step should be between **30 and 60 seconds**, depending on the material. The signal of one **oxygen** phase should first fall to the **lowest** level, before the next step can be started. One possible separation is shown in **figure 2**.



Figure 2

At the end of this analysis we obtain the total **oxygen** result. This should be in an **acceptable** range, or else the temperature program needs to be modified.

5. Mathematical separation of the peaks.

The optimised signal from each **oxygen** phase results in a single peak that starts and finishes at the **baseline**. We can integrate each peak individually and get perfect results, as shown in **figure 3**.



Figure 3

In most cases, nature is not very polite. We cannot separate each **oxygen** phase well enough to **treat** the peak as a unique item. We can only be integrated a **peak** correctly if it starts from **zero** and returns to **zero**. Unfortunately, very often before one **peak** has a chance to return to **zero**, the next peak starts to **rise**, causing the reading of the **previous** peak to be **incomplete**. That's why we need to calculate the first **peak** as if it actually descended to **zero**. We have to mathematically "**complete the peak**." See **figure 4**.

Only this way, it is possible to analyse each **oxygen** phase. We have to calculate all the **peaks** out of our "**mountains**" of signals.



Figure 4

The typical shape of a peak includes a **fast rise**, followed by a **slow fall**, because the chemicals and volumes of the system must be **purged out**. We find the same shape for every peak during the heating period.

Figure 4 includes two diagrams:

1. without mathematical correction, as displayed from the analysis, with the peaks **not** returning to **zero**.

2. with the mathematical correction.

By adding up the signals of the first diagram, we obtain the **total** contents of **oxygen** in the sample.

The integration of each peak area gives the content of the particular **oxide**. All the **oxygen phases together** should represent the **total oxygen** content. The difference between the total oxygen content given by the analyser and the sum of the single **oxides** gives a sign for the precision of the mathematical procedure. If the difference is too **large**, then the heating program needs to be optimised. The mathematical treatment cannot compensate for the physical errors.

6. Summary.

The separation of different **oxygen** phases of the temperature profile method, is a way to obtain more information about a material than with the simple elemental analysis. It is possible to **optimise** this method for better temperature programs and better mathematical methods for the **peak calculation**. But the limit is imposed by the nature of this analytical method. With the **x-ray** diffraction analysis, it is possible to analyse the crystal structure directly, without **destroying** the material. The results are more precise, but more time is needed for sample preparation and analysis.

In most cases it is not necessary to know the **exact value** of each **oxygen** phase, it is sufficient to get a "**fingerprint**" of the material. It is very useful to **compare** the fingerprints of different materials to control or check a production process.

2.4 Applications

Nitrogen in baron nitride ceramics

Power mode:	On Off On mode
Sample drop:	Manually
Out-gassing (Power / time)	5.0 kW for 60s
Purge time:	15s
Stabiliser time:	30s
Integration delay:	2s
Analysis power / time:	50 kW for 60s
Min. analysis time:	100s
Accelerator / Flux:	None
Calibration:	Pure chemicals (BN)

Oxygen in magnesium

Power mode:	On Off On mode
Sample drop:	Automatic
Out-gassing (Power / time)	4,5 kW for 45s
Purge time:	10s
Stabiliser time:	20s
Integration delay:	10s
Analysis power / time:	2,8 kW for 30s
Min. analysis time:	50s
Accelerator / Flux:	1g Sn
Calibration:	Standard copper. Furnace cleaning after each analysis

Oxygen in steel

Power mode:	On Off On mode
Sample drop:	Automatic
Out-gassing (Power / time)	5.0 kW for 30s - 45s
Purge time:	10s
Stabiliser time:	20s
Integration delay:	10s
Analysis power / time:	4,0 kW for 30s
Min. analysis time:	50s
Accelerator / Flux:	None
Calibration:	Standard steel O+N in similar range.

Oxygen and nitrogen in titanium

Power mode:	On Off On mode
Sample drop:	Automatic
Out-gassing (Power / time)	5,8 kW for 45s
Purge time:	15s
Stabiliser time:	30s
Integration delay:	10s
Analysis power / time:	5,0 kW for 45s
Min. analysis time:	50s
Accelerator / Flux:	(250mg) sample placed in a 1g nickel basket
Calibration:	Standard titanium 100 ppm. N / 1000 ppm O. High crucibles required.

Oxygen nitrogen in zirconium

Power mode:	On Off On mode
Sample drop:	Automatic
Out-gassing (Power / time)	5.5 kW for 30s - 45s
Purge time:	10s
Stabiliser time:	30s
Integration delay:	10s
Analysis power / time:	4,5 kW for 45s
Min. analysis time:	50s
Accelerator / Flux:	(250mg) sample placed in a 1g nickel basket
Calibration:	Standard titanium or zirconium. High crucibles required.

Oxygen and nitrogen in ceramics

Power mode:	On Off On mode
Sample drop:	Manually
Out-gassing (Power / time)	5,0 kW for 45s
Purge time:	10s
Stabiliser time:	30s
Integration delay:	2s
Analysis power / time:	3,0 kW for 60s
Min. analysis time:	100s
Accelerator / Flux:	None
Calibration:	Pure chemicals in similar range. 10 – 100mg sample weight depending on oxygen concentration.

2.5 Gas conservation

Saving carrier gas:

When the **ON-900** is in analysis mode, but it has nor been used for a while, the gas consumption will automatically be reduced to a very **low level**. This low flow keeps the gas flow system **flooded** with **carrier gas**, preventing the air from penetrating the system. As soon as the operator starts using the analyser again, the normal flow will be **automatically** restored. The analyser will be ready again for operation in a very short time, due to the purging with the low flow during the break.

This function can be activated in the software, if needed.

3 MAINTENANCE

3.1 General information

Every 50 analyses or at least 2 times a day:

■ Clean the furnace and the electrodes. See <u>3.6</u> and <u>3.7</u>.

Every 500 analyses:

■ Replace all chemicals. See <u>3.2</u> and <u>3.3</u>.

Every 1000 analyses or if 1/3 of the material turned grey:

■ Change the graphit tip. See <u>3.7</u>.

Every 3000 analyses:

Replace the copper oxide in the catalyst furnace, take the electrodes from the furnace chamber out to clean them with a brush.

Remark:

The above is related to steel analyses and helium 99.995% pure.

3.2 Installing and removing the reagent tubes



6-1-1

To replace the reagent tubes:

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

IMPORTANT:

The dimensions for filling the glass tubes given in the schematic of <u>3.3</u> 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.

NOTE:

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

With **catalyst furnaces**, the copper oxide is replaced after about **3000** analyses. See <u>3.1</u>. It is safer, but not absolutely essential, to switch the analyser off.

The components are refitted in reverse order.

ATTENTION:

It should be remembered that the furnace temperature is about 450°, and protective gloves must be worn.

Only the outside grid of the furnace is to be handled; the quartz reagent tubes must only be heldat the ends.



- B The quartz tube (1) of the furnace (2/3) is raised as far as it will go.
- C It is then swung out together with the furnace (2/3).
- D The quartz tube (1) is pulled put diagonally downwards
- E The furnace (2), together with the grid (3) is removed.

The components are refitted in reverse order.

3.3 Filling the reagent tubes

The following chemicals are used:

Magnesium perchlorate (anhydrone)	as moisture absorber.
Sodium hydroxide (ascarite)	as CO ₂ absorber.
Copper oxide catalyst, schutzes reagent	as oxidiser (CO \rightarrow CO ₂)

The reagent tubes 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 it's colour (**it turns to light grey**). If the absorber particles do not move (e.g. tapping on the glass), then this is a sign that the **magnesium perchlorate** is saturated. It is essential to change the absorber before it is completely solid. The moisture absorber should be checked after **100-200** analyses and if necessary replaced.

Please refer to the following schematics to identify the glass tubes on the analyser. In addition to the reagents in the glass tube, fill the bottom end of the tube with **unleaded glass wool**. One should pay attention that the glass wool should be only as **thick** as necessary, otherwise the flow of gas can be **choked**. Under no conditions should the amount of glass wool be less than that given in the following schematics, since fine particles of **magnesium perchlorate** can pass through the wool and collect itself at the bottom of the tube, causing severe damage.

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

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

IMPORTANT:

There are qualities of chemicals such as **anhydrone**, **ascarite**, **copper oxide**, **tungsten granules**, **iron chips**, **copper chips** etc. which have been specially developed for analysing instruments. The commonly available materials serve 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 is often not even completed. This effect also occurs with magnesium perchlorate of suitable quality if it is over saturated.
- The commonly available sodium hydroxide binds CO₂ very inadequately at room temperature, whereas the special quality not only binds extremely well 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 analyser will **not** be damaged. If problems should arise, however, suitable materials, in proper, unsaturated condition, should be used, **before** calling technical service.

The chemical containers must be closed **very tightly, immediately** after use, so that they do not become contaminated with air moisture or CO_2 .

The reagent tubes are filled as followed:

See following pages:

For the chemicals to be retained in the reagent tubes the lower end is filled with glass wool. Do not stuff the glass wool to tight, other wise the gas flow is blocked. The rest of the tube is filled with the appropriate chemicals.

The lower half of the reagent tube for the **oxygen** pre-cleaning tube is filled with **anhydone** and the upper half with **sodium hydroxide**. The chemicals are separated by a quantity of glass wool. The tube at the furnace outlet is filled with **glass wool**.

Sufficient **space** must be left at **both ends** of the tube so they can be attached to the glass fittings. The free inner surface at the ends of the tubes serve as sealing surfaces. And must be cleaned after filling.

The **O-rings** also have to be clean. Both the **O-rings** and the **sealing surfaces** on the tube should be greased with **silicon grease**. This simplifies the fitting and particularly the removal of the tube, and ensures proper **sealing**.

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



Each filling quantity carries a tolerance of \pm 20 %

1	Quartz wool	90330
2	Copper oxide on rare soils	90289
3	Anhydrone	90200
4	Sodium hydroxide	90210
5	Glass wool	90331

Gas cleaning furnace:

Some time there is a small **oxygen** concentration in the carrier gas. This oxygen causes a **high blank value** in oxygen. At the same time the blank value in **nitrogen** is low.

In this case it is helpful to use a **gas cleaning furnace**. In this furnace are **copper** turnings which **catches** the oxygen out of the carrier gas.



3.4 Replacing the O-rings

For the reagent tubes:

The **O-rings** are only replaced when they can no longer adequately seal, due to severe damage or age. When removing the **old** O-rings, be ensure that the sealing area of the fittings are not damaged. The groove in which the old O-rings sat must be cleaned, so that it is completely **free of grease**.

The new O-rings should under **no-circumstances** be greased before installing, **only** after installation. Otherwise, **the O-rings will turn with the glass tubes** when trying to remove it. See **fig. 1**.

Furnace: to make the assembly easier grease the O-rings for the furnace slightly. See fig 2.



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3.5 Cleaning the dust trap

The dust trap is the glass tube, filled with quartz wool. If this quartz wool is dirty it must be replaced, see 3.2 and 3.3.

3.6 Cleaning the furnace



Older version

Newer version

The quantity of dust in the furnace depends on the power setting. The furnace should be cleaned after **50 analyses**.

If there is more dust then usual, then the furnace should be cleaned in shorter intervals.

- Open the furnace with the pneumatic lift
- Pull the sample drop (2) out of the furnace
- Clean the gas channel with the big brush (3)
- Clean the inside of the furnace and the upper electrode with a soft cloth or a paper serviette (4). Do not use a metal brush, it may cause damage!
- Clean the lower electrode with the small brush (5) or a soft cloth.
- Put the sample drop back into the furnace
- Shut the furnace

3.7 Replacing the electrodes

Due to the **high temperature** in the furnace, the **graphite tip** and the **upper electrode** will wear out, so they need to be changed from time to time. If there is no contact between the crucible and upper electrode, the current flow fails and the analysis starts without any current. **The graphite tip and the upper electrode need to be changed**.

Older furnace version:

Replacing the upper electrode:

To remove the upper electrode (7), turn the four screws (6) anticlockwise by three to four turns and then remove the upper electrode with the special provided tool (4)

Replacing the electrode tip:

- Remove the four screws (2) on the lower electrode (1), and then the graphite tip (3).
- Reassemble the new graphite tip and both the electrodes in the reverse order. Secure the screws holding the electrodes well.



Newer furnace version:

Replacing the upper electrode insert:

- Remove the gas inlet tube (1).
- Remove the sample drop unit (2).
- Remove the two screws on top of the upper furnace unit (3) with a 5 mm allen key.
- Remove the upper furnace unit (4).
- While holding a hand below the combustion chamber, loosen the four screws (5), with a 5 mm allen key, until the electrode insert (7) falls into the hand.

There is no need to pull out the screws, make sure that the coil spring washers (6) don't get lost.

Replace electrode insert (7) and reassemble in reverse order. Secure the screws (5) well.





- Remove the four screws (8) with a 3 mm allen key, remove the electrode plate (9), and finally replace the graphite tip (10).
- Reassemble in reverse order. Secure the screws (8) well.

4 DESCRIPTION OF FUNCTIONS

4.1 Measuring principle

The sample falls from the sample drop into the graphite crucible. There it is melted by high temperature caused by the current flowing through the crucible. The carrier gas takes the **helium** and **nitrogen** out of the sample. The **oxygen** is converted to **CO** at the surface of the hot graphite crucible. The pump sucks the gas through the catalyst furnace. There it is converted from **CO** to **CO**₂. After that the **CO**₂ is detected in the **IR-cell**. The **nitrogen** is detected by a thermal-conductivity-cell.



4.3 Infrared cell

The **measuring principle** is based on the **infrared radiation** absorbing property 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.



electrically heated and radiates **broad-band infrared radiation**. The light beam is interrupted by a rotating vane (chopper) (3), resulting in an alternating light. The chopper motor (2) is quartz controlled, so that the infrared radiator chopper frequency is highly stable. The **infrared radiation** then passes through the measuring IR-paths (10, 11), through which the sample gas/carrier gas mixture flows. Depending on the composition of the gas mixture, certain **infrared spectra** are absorbed; the absorption is stronger or weaker, depending on the concentration of the gases present. As the **infrared beam** leaves the measuring covet, passes through a filter (6, 9), which only lets a narrow band of infrared light through. The broad-band wavelength is selected which corresponds to the wavelength where the gas to be measured shows its maximum absorption capacity. The intensity of the beam after the filter thus provides information about the concentration of a specific gas in the path. The beam finally strikes a semiconductor infrared detector (7, 8), which emits an electrical signal in proportion to the intensity of the beam.

Since the beam is interrupted by the rotating chopper, as mentioned above, the detector emits an alternating signal. Temperature and ageing influences of the detector, as well as noise, are thereby suppressed.

The signal thus obtained is amplified and rectified, so that it leaves the **infrared cell** as d.c.

The **infrared cell** also ensures that the **zero point** and the sensitivity of the analyser are constantly automatically corrected, thus completely eliminating the need for manual checking and readjustment.

The infrared analyser is thermostatically controlled, so that the sample gas which flows through it is brought to a constant temperature.

4.4 Thermoconductivity cell

The **thermoconductivity cell** detects changes of the contents by measuring the thermal conductivity. The thermal conductivity is **not** absolutely measured, but in relationship to a reference, to get a better resolution. This reference is normally the pure carrier gas.



- 1 Thermo stabilised cell
- 2 Measuring channel
- 3 Reference channel
- 4 Thermistors
- 5 Amplifier
- 6 Microcontroller UNI 1.3

There are **two** channels in the cell (1); the measuring channel (2), in which the analysis gas streams, and the reference channel (3) with the pure carrier gas.

In each channel there are **two** thermistors **(4)**, which detect every change of the thermal conductivity of the surrounding with a change of their electric resistance. The thermistors are connected as a Wheatstone-bridge for a measurement with a constant current.

The signal of the **thermistors** is amplified by an amplifier **(5)** and get into the microcontroller **(6)**. The base line is automatically set to **zero**, so that no adjustment is necessary. The thermo stabilisation takes care, that the surrounding has no influence on the **cell**.

5 MISCELLANEOUS

5.1 Ordering numbers

Front panel:

11062	Reagent tube
11064	Reagent tube
11480	Adjustable restrictor
15083	Gas flow indicator 15 l/h
15085	Gas flow indicator 130 l/h
20002	Catalyst furnace
20040	Catalyst tube
70210	O-ring
70230	O-ring
72010	Pressure gauge
77411	Panel meter 5A
78015	Mains power switch





- 05020 Chopper blade
- 05030 Chopper blade holder
- 05065 Chopper motor
- 05048 Infrared source (emitter)
- 70280 O ring
- 70330 O ring
- 75120 Spring
- 75130 Retaining washer
- 75190 Washer



- 04007 TC board TC1.3
- 05067 IR path for high oxygen
- 05068 IR path for low oxygen
- 05254 Chopper housing
- 05270 Threaded rod (advise the length)
- 05274 Infrared preamplifier
- 05584 TC sensor
- 06210 Cable
- 06421 Infrared power supply assembly
- 06670 IRT zero adjustment board
- 06748 Infrared electronic board IR41(2channels)
- 06749 Infrared electronic board IR41(1channel)
- 15205 Gas flow sensor

Analysator – Trafo Seite



Thyristor block HF-filter 250 V

77033

77051

Combustion chamber

Sample drop mechanism

Abdeckung Wasserkühlung 77140

31246

31325

31331

Analysator – IR/TC cell side



Furnace:

		\bigcirc
31220 31230 31246 31250 31325 31331 31360 31365 31380 31393 70405 70410 70415 70430 70435 71010 71029 72100 72101 72102 90191	Lower furnace assembly Upper furnace part Combustion chamber Upper electrode insert Sample drop mechanism Cooling cover Graphite tip foraphite tip holder Isolating ring Lower power connector O-ring O-ring O-ring O-ring Cleaning brush Furnace cleaning brush Screw Screw Screw Graphite crucible	70435 70430 70435 70430 70410 700 700 700 700 700 700 700 700 700 7
		31360 70405 70405

-31393

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31220

70405 31380

06-5

Analyser right-hand side



- 31325 Sample drop mechanism
- 31450 Lower bride
- 31450 Upper bride
- 31466 Eccentric wheel
- 31480 Support for motor bracket
- 33120 Tube for dust trap
- 77423 Temperature sensor
- 77460 Micro switch
- 78054 Motor for sample drop

5.2 Packing



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

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

The analyser and furnace should be wrapped in plastic foil, especially when you use chips or any other kind of material in small pieces. The glass tubes must be empty.

Packing is done as follows:

Front view:

a. Place the analyser directly on the pallet with the right side towards the middle of the pallet, because the furnace and the transformer are the heaviest parts of the analyser.





Top view

b. Shift the analyser to the exactly required position.

c. Foam:

Tilt the analyser to the furnace side and place a piece of foam at the right position.



Tilt the analyser to the other side and place the second piece of foam at the right place. If necessary, a third piece of foam can be placed on to the pallet.



5.3 Trouble shooting

High blank value in O and N:

in some cases the gas flow is not **high enough** to clean the furnace from air. See <u>**1.8**</u> <u>**"Adjusting the gas flow"**</u>.

The analysis does not start:

The start of the analysis depends on many conditions. If there is no **water flow**, or the **temperature** is too high, a message appears on the display. A wrong adjustment of the flow rate too prevents the analysis form starting.

If one or more values are higher than **+/-3V**, then this problem will occur. Maybe the gas bottle is empty, or some air got into the analyser. Check and change the chemicals if necessary.

If only the value for the thermoconductivity cell is lower than **6V**, then the cause is some air inside the reference cell. Open the right door and shut the gas inlet of the furnace for **10s**. After that, the value must be higher.

No current during the analysis:

If there is no current during an analysis, then there is a problem with the electric contact in the furnace. There must be a gap between the upper furnace and the plastic disc of the lower furnace. If there is no gap, change the **lower electrode**. If this is not the problem, change the **upper electrode**.

"No water flow" or furnace overheat message:

If the analyser recognises no water flow in the beginning of the analysis, then the following message will appear:

No water flow!

The system will then shut down the electric current for the analysis. The analysis will be stopped.

Behind the right door of the analyser is a flow indicator on a board. If this shows **no water flow**, look for a cracked tube. If a water flow is detected, but the analyser does not recognise it, change the cooling water, it may be muddy. Otherwise call a service engineer.

If the furnace becomes too hot during the analysis, the following message will appear:

TEMPERATURE TOO HIGH!

Make sure that the temperature is less than **70°C**. Test the secondary cooling system. If the temperature is not above **70°C**, an electric fault is the problem, call a service engineer.

5.4 ON-900 Pre-installation guide

Following requirements apply, when installing the Analyser Eltra ON-900:

Compressed air	4 - 6 bar (60 - 90 psi)
Carrier gas	Helium 99.99% pure; 2 - 4 bar (30 - 60 psi)
Mains power supply	400 VAC \pm 10%; 50/60 Hz; 3 phase and neutral; fuse = 32 A.
	Use DIN 49462/63 mains power socket, if locally applicable
Analyser dimension	55 x 80 x 60 cm
Analyser weight	ca. 135 Kg. It is important to install the instrument on a stable
	place

- The balance should rest on a vibration free support.
- Connections for helium and compressed air; outer diameter = $R\frac{1}{4}$ ". (The tubes supplied together with the analyser, carry a connector with $G\frac{1}{4}$ " inner diameter).
- Use tap water for cooling, with at least 4 bar (60 psi) pressure. Connector for water supply: outer diameter = R³/₄". (The tubes supplied together with the analyser, carry a connector with G³/₄" inner diameter).

Approved Methodologies to Which ELTRA Instruments Conform

ASTM (ANALYTICAL SOCIETY FOR TESTING MATERIALS)

Metals

Instruments	Method	Elements	Materials
CS-2000 CS-800	ISO-9556	С	Steel & Iron
CS-2000 CS-800	ISO-4935	S	Steel & Iron
CS-2000 CS-800 ON-900 ONH-2000	ASTM E-1019	C, S, N, O	Steel, Iron, Nickel/Cobalt Alloys
CS-2000 CS-800 ON-900 ONH-2000	E-1587	C, S, N, O	Refined Nickel
ON-900 ONH-2000	E-1409	0	Titanium and Titanium Alloys
ON-900 ONH-2000	E-1569	0	Titanium
ON-900 ONH-2000	E-1937	N	Titanium and Titanium Alloys
OH-900 ONH-2000	E-1447	Н	Titanium and Titanium Alloys
CS-2000 CS-800 CS-500	E1915-97	C, S	Metal Bearing Ores and Related Materials (f.e. tailings, waste rock)
CS-800 CS-2000	UOP-703-98	C,S	Catalysts

Organics

Instruments	Method	Elements	Materials
CS-2000 CS-500	ASTM D-1552	S	Oils & Petroleum Products
CS-2000	D-4239	S	Coal & Coke
CS-500	D-5016	S	Coal & Coke Ash
CS-2000 CS-500	D-1619	S	Carbon Black
CS-2000 CS-500	PN-93 G-04514/17	S	Coal & Coke
CS-2000 CS-500	DIN EN 13137	тос	Waste
CS-2000 CS-800 CS-500	ISO-10694	TC/TOC	Soil samples