Ident-Nr.: 25.20-5003

vario MAX CNS

Macro Elemental Analyzer

Operating Instructions

for instruments starting with serial no. 25052016

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Important Instructions

- The **vario MAX CNS** elemental analyzer is a fully automatic instrument which enables a speedy quantitative analysis of CN and CNS. In two operation modes the instrument can determine the elements CSN and CN simultaneously from the weighed-in samples.

- The suitability of the **vario MAX CNS** analyzer for the element analysis depends on the controllable combustibility of the samples or sample quantities. **Analyzing aggressive chemicals, acids, alkaline solutions, solvents, explosives or materials that can create explosive gas mixtures is explicitly forbidden or can be executed at your own risk.** It can lead to damages of the instrument and to injuries of the operating personnel. **For above applications or applications that are especially difficult (**if aggressive combustible products are to be analyzed), **please make use of the counseling possibilities of our Application Laboratory** in order to use special equipment or to work out safe processing procedures. It is also possible that certain substances, as for instance fluorine, phosphate or samples containing heavy metal have a negative influence on the analysis results or the life time of instrument components.

- Operation of the **vario MAX CNS** and peripheral equipment should only be carried out by persons who have been authorized by **E l e m e n t a r Analysensysteme GmbH**. Opening the transport packing by unauthorized personnel can lead to loss of warranty rights! If the supplied instruments show damages, please contact the freight forwarder and the manufacturer immediately.

- For installation, the "Installations Requirements" have to be observed. For details, please refer to the Appendix of this manual (chap. 11.4).

- **Operation, maintenance and repairs should only be carried out by trained personnel.** For a smooth and efficient operation of the **vario MAX CNS**, please read the following operating instructions thoroughly. Please keep the operating instructions for future questions. We reserve the right of deviations in illustrations and technical data described in the operating instructions due to further development. The data shown in the illustrations of the software are only examples and may differ from the actual parameters to be set. Please refer to the installation diskette for the parameters valid for your instrument.

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- The supplied operating instructions explain especially the operation and maintenance of the **vario MAX CNS.** Since the operating software of the **vario MAX CNS** is self-explanatory, we have mentioned only several commands and menus. For further information regarding the software, please read the notes in the PC-online-help.

- The efficiency of the **vario MAX CNS** can be guaranteed only with original consumables as well as original spare parts which can be obtained only through **E l e m e n t a r Analysensysteme GmbH** or authorized dealers. **The use of different consumables or chemicals of an unsuitable standard can lead to injuries as well as instrument damages. It can also falsify the results of the analysis. Unsuitable spares lead to instrument damages and loss of warranty.**

- All the operation dependent maintenance and exchange times mentioned in the operating instructions depend very strongly on the respective test matrix. They are, therefore, only examples **and do not guarantee life times for consumables or wear and tear parts!**

  The analysis parameters described in the following are mean values. The operating parameters that have been optimized for the individual instrument are included in the furnished installation disk. Of course it is possible to optimize the analysis parameters any time for special applications or unusual demands.

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Definitions

- To clarify the input at the PC, we used the following definitions:

Definition in the text	**Meaning**
<Key>	key of the PC keyboard e.g. <ESC> means the Escape-key <CR> means the Return-key <M> means the letter M
{*italic*}	commands to be entered in the PC e.g.: {*vl* <CR>} means, enter the letters "vl" and push the return-key;
Edit or **Edit⇨Input**	dialog/menu or path to open a dialog window; click on menus one after the other. For operation with the keyboard push the underlined letters (”Hot Key”) together with<Alt> ;
[**OK**] or [**Help**]	button (symbol) on the screen to be clicked on with the mouse-cursor;
”*Sleep at sample end*”	definition of a function in a dialog window; e.g.: activate ”*Sleep at sample end*” in the menu **Options⇨Sleep-/Wake-Function**.

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Abbreviations

- The following abbreviations are used in these operating instructions:

DF	daily factor
TCD	thermal conductivity detector
He	helium
O_2	oxygen
H_2O	water
CO_2	carbon dioxide
SO_2/ SO_3	sulfur dioxide/sulfur trioxide
N_2	molecular nitrogen
No_x	certain nitric oxides

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List of initial outfit
List of consumables
List of safety data sheets

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1. Scope of delivery

Smallest operable unit:

- **vario MAX CNS**
  Carbon/Nitrogen analyzer for the determination of C, N and S in solid and liquid samples, **incl. operational software in German or English, initial set of equipment.**
  Initial set of equipment sufficient for inital operation and instruction.
  Order no. 25.20-0000
  (for 230 V, 50/60 Hz, other voltages on request).
  Accessory kit for CN determination:
  Order no. 25.20-0005

- **Control and evaluation unit with German software**
  Order no. 50 008 369
  (for 230 V, 50/60 Hz, other voltages on request)

- **Control and evaluation unit with English software**
  Order no. 50 008 368
  (for 230 V, 50/60 Hz, other voltages on request)

- **Printer and printer cable**
  Order no. 50 031 857
  (for 230 V, 50/60 Hz, other voltages on request)

Accessory requirement:

- **Pressure regulator for oxygen**
  two-stage, 2nd stage 0 - 3 bar with check valve
  for gas quality 99.995 % or better
  Order no. 04 278 800

- **Pressure regulator for helium**
  two-stage, 2nd stage 0 - 8 bar with check valve
  for gas quality 99.995 % or better
  Order no. 25.00-0014

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- **Electronic balance**
  (Readability 0.1 mg)
  with V 24 / RS 232-interface
  Order no. 03 002 633

optional

- **Electronic balance**
  (Readability 0.01 mg)
  with V 24 / RS 232-interface
  Order no. 03 002 634

- **Sets of consumables**
  sufficient for approx. 2000 analyses
  solid samples CNS
  Order no. 25.00-5040
  sufficient for 4000 analyses
  solid samples CN
  Order no. 25.00-5039

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2. Specifications and technical data

2.1 Specifications

Method of analysis	**Elementary analysis of N/CN/CNS from one sample** Catalytic tube combustion of the samples, separation of foreign gases, separation of the desired measuring components, TCD-detection.	
Weigh-in/sample volume	5 mg up to 5 g (1 g organic substance, max. 5 mL, max. 400 mg C	
Dynamic working range		
	CNS	0.02- 30 mg N abs. 0.02- 200 mg C abs. 0.02- 15 mg S abs.
	CN	0.02- 150 mg N abs. 0.02- 400 mg C abs. *)
Precision / Standard deviation	≤ 0.5% relative with approx. 60 mg sulfadiazine	
Recovery rate	> 99.5 % for test substances	
Duration of analysis **N-Mode standard** **CN-Mode standard** **N-Mode precise** **CN-Mode precise** **CNS-Mode**	self optimizing, approx. 5 min. for 250 mg glutamic acid approx. 9 min. for 1 g mineral soil approx. 9 min. for 250 mg glutamic acid approx. 11 min. for 1 g mineral soil approx. 10-14 minutes when using CNS simultaneously	
Calibration	linear and curve adjustment; if necessary in 2 sections	
Data storage respectively data output	storage on hard disk or diskette. LIMS-transfer possible. Data output on monitor or printer.	

*) 0.02 – 200 mg C standard type
0.02 – 400 mg C with C adsorption column, art.-no. 25.00-0091

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2.2 Technical data

Detector	Thermal Conductivity Detector (TCD)
Control and evaluation unit	PC with Windows ® operating system printer (optional)
Software	German or English, menu operated, status display during analysis, real time graphic
Interfaces	RS 232 /V24, low safety voltage according to DIN IEC 380/VDE 0806/08.81
Dimensions	Basic instrument: 780 x 560 x 700 mm (W x D x H) plus 200 mm clearance from the walls
Weight	130 kg
Rated voltage	230 Volt AC ± 10 %, 50/60 Hz other voltages on request
Power consumption	0.8 kW
Connection value	1.6 kVA
Protection class	I, connection for earthed conductor wire
Protective system	IP 20, installation in dry rooms only
Noise level	55 dB (A), acc. DIN 45635 part 1
Ambient temperature	admissible max.: + 35°C min.: + 15°C
Supply gases	**He**, purity 99.996 %, approx. 750 mL/min. using precise mode approx. 850 mL/min. using standard mode **O_2**, purity 99.995 %;
Standard	CE-conformity according to EC guide lines EMC 89/336/EWG and LVD 73/23/EWG

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2.3 Version vario MAX CN(S)

The elemental analyzer **vario MAX CN(S)** corresponds in its design the modification from CNS to CN (see chapter 9 of the operating instructions).
Only the SO_2- adsorption column and some not required tubings are missing.

For a later upgrade to CNS operation all requirements are met. Only the upgrade kit for S determination has to be ordered.
Order no. 25.40-0005.

The operating instructions vario MAX CNS for operating mode CN are valid.

2.4 3-Tube Furnace

Elementar Analysensysteme GmbH grants a 10 years' warranty on the 3-tube furnace. In case of a manufacturing or material damage you will receive a replacement furnace during a 10 years period starting with the date of purchase. This applies for all models ordered from December 1, 2003 on.

Excluded from this warranty are of course damages that are caused by faulty treatment/operation. Therefore, the following instructions for the operation of the 3-tube furnace have to be followed by all means.

2.4.1 Instructions for proper operation of the 3-tube furnace

1. The instrument user has to observe the correct operation of the 3-tube furnace according to the requirements of the operating instructions.

2. The furnace must not be heated above the maximum temperature stated in the operating instructions.

3. The furnace must not be operated with a larger voltage than stated in the operating instructions.

4. For breaks of several hours (e.g. over night) heater 1 shall be set to 900°C (for CHNS, CNS, S mode only). For the operating modes CHN, CN, N and O the corresponding operating temperatures remain the same.

5. For longer breaks over several days (5 days and more) the furnace shall be turned off (set temperature control to 0.00°C).

6. The furnace shall be protected from liquids.

7. Short-circuits in connection with heating spirals and/or thermo elements have to be avoided.

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8. Inner and outer force to the heating spiral, to the thermo elements and to the supply lines have to be avoided.

9. Only original parts by Elementar shall be used (e.g. combustion tubes, reductions tubes, fillings, sealings, etc.)

10. In case of a breakdown, the 3-tube furnace shall be sent to the manufacturer's works to Hanau in order to determine the error. If the inspection clearly shows that the furnace became defective due to improper treatment, the warranty claim expires.

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3. Safety Instructions

Please adhere to the safety instructions and only operate the unit in accordance with manufacturer's instructions. The warnings on the individual instruments and in the appropriate chapter of these instructions are also valid.

General instructions

- The analyzer **vario MAX CNS** is suitable only for the elemental analysis of samples or sample sizes that have a controllable combustibility under combustion conditions, depending on the mode of operation.

  Analyzing aggressive chemicals, acids, alkaline solutions, solvents, explosives or materials that can form explosive gases is explicitly forbidden and must be performed at ones own risk. It can lead to damages of the instrument and to injuries of the operating personnel. For difficult applications, especially if aggressive combustion products can occur, we recommend to use the counseling possibilities of our application laboratory, in order to use certain special equipment and to work out safe operating procedures.

  It is also possible that certain substances, as for instance fluorine, phosphate or samples containing heavy metal have a negative influence on the analysis results or the life time of instruments components.

- **Operation, maintenance and repairs should only be carried out by trained personnel**. For a smooth and efficient operation of the **vario MAX CNS**, please read the following operating instructions thoroughly. Please keep the operating instructions for future questions. We reserve the right of deviations in illustrations and technical data described in the operating instructions due to further development.

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Installation / Start-up

- Operation of the **vario MAX CNS** and peripheral equipment should only be carried out by persons who have been authorized by **E l e m e n t a r Analysensysteme GmbH**.
- **Opening the transport packing by unauthorized personnel can lead to loss of warranty rights!** If the supplied instruments show damages, please contact the freight forwarder and the manufacturer
- **The installation room must be well ventilated!**
  The exhaust gas pipe (connection M) must lead into the open air or into a fume hood because it is possible that during operation toxic gases (e.g. CO during wrong combustion, SO_2) and asphyxiating gases (e.g. per analysis approx. 1 L CO_2) emerge from the instrument. During constant operation the respective gas concentration can exceed the permissible maximum workplace concentration.

- The end of the gas pipe must lead to a sheltered (wind-free) place in the open air to avoid fluctuations of the base line which would lead to detector instabilities.

Measuring process / calibration

- Since the software of the **vario MAX CNS** is self-explanatory and is equipped with an extensive on-line help program, we have mentioned only several commands and menus. For further information regarding the software, please read the notes in the PC-online Help program (**menu HELP⇨Index)**.
- A faultless calibration of the instrument in the selected operation mode, covering the required working range, is of crucial importance of the accuracy of the results. Calibration procedures must, therefore, be carried out with utmost care. **The Operating instructions should be diligently observed.**

- Insufficient oxygen reduces the catalysts and reduces, therefore, their effectiveness and life span. The sample substances that have not been burnt remain inside the crucible and falsify the analysis results of the test samples to follow.

Maintenance / Tube Replacement

- The efficiency of the **vario MAX CNS** can only be guaranteed when original consumables as well as original spare parts which can be obtained only **through Elementar Analysensysteme GmbH** or their authorized dealers, are used. **The use of different consumables or chemicals of an unsuitable standard can lead to injuries as well as to instrument damages. It can also falsify the results of the analysis. Unsuitable spares lead to instrument damages and loss of warranty.**
- All the operation dependent maintenance and exchange times mentioned in the Operating instructions depend very strongly on the respective test matrix. They are, therefore, only examples and **do not guarantee life times for consumables or wear and tear parts!**

  The described mode depending analysis parameters are mean values. The operating parameters that have been optimized for the individual instrument are stored on the

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supplied installation disk. Of course it is possible to optimize the analysis parameters any time for special applications or unusual demands.

- The walls of the **vario MAX CNS** should only be opened by authorized personnel for maintenance and servicing purposes.
- Before opening a side or rear wall of the **vario MAX CNS, disconnect all peripheral equipment and the analyzer itself by turning the main switch and pulling the mains plug.** Attention: **the 3-tube furnace must be cold** otherwise components will become overheated. The side and rear walls must be properly in place before starting a new analysis (check protective conductor connection).

- To prevent leakage around the tube plugs, apply a slight coating of high-vacuum grease to the O-rings and check them for damages. If too much grease is applied C blank values will occur! The O-ring insert grooves in the plugs must be cleaned regularly. The high temperature causes the O-rings to shrink, therefore, a resistance must be felt during O-ring placement otherwise it might leak. Before inserting the plugs, the insides of the tubes must be free of dust.

- The **vario MAX CNS** and the gas pipe system must be checked periodically (at least once per month) for leakage.

- When handling chemicals, observe the safety instructions of the chemical manufacturer (see labels on bottles and safety data sheets)!

  When handling Sicapent® (phosphorpentoxide) and NaOH (sodium hydroxide), it is absolutely necessary to wear safety goggles and protective gloves.

!!!Danger of Cauterization !!!

- All tube fillings must be packed down as tightly as possible to avoid air pockets. To do this, tap the side of the tube lightly with a wooden implement or cork ring while it is being filled.
- **Replacing combustion or post combustion tubes can only be carried out when the tubes are cold.** For this purpose set furnaces to nominal temperature 0°C and let the tubes cool down to room temperature (max. 100°C).
  When working at hot furnaces or tubes there is danger of burning!!
  Therefore**,** always wear the supplied protective gloves (order No. 05 000 094), because it is possible that some components (e.g. heated tube line) are still hot. We are not liable for accidents that are due to disregarding these warnings.

- Before replacing a tube turn off the gas supply into the instrument respectively activate the function **Options ⇨Miscellaneous⇨⇨Replace Comb. or Post Comb. Tube** or **Options ⇨ Miscellaneous⇨Replace Reduction or Drying Tubes**.

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- After a tube exchange, it is necessary to flush out the atmospheric nitrogen that entered the unit for 5 minutes with He. This is accomplished automatically when leaving the function **Options⇨Miscellaneous⇨Replace Comb. or Post Comb. Tube** or **Options ⇨Miscellaneous⇨ Replace Reduction or Drying Tubes**.
- After changing the tubes wait until the 3-tube furnace as well as the new reaction tubes have warmed up (ca. 10 to 15 minutes) before starting the next analysis. In addition, it is necessary to adhere to the mode dependent conditioning rules for the tubes.
- **Before installation into the 3-tube furnace all finger prints must be cleaned off the quartz parts** (for instance with acetone) to avoid crystallization areas which lead to premature aging of the quartz. When handling cold quartz and glass parts wear the supplied leather gloves to avoid danger of injuries in case of broken glass.
- **Do not cut out O-rings with a sharp knife or a scalpel**, since this might damage the sealing area of the plugs etc. through scratches. **There is also the danger of injury in case the knife slips off!!**
- If the supply voltage has been interrupted for more than 15 minutes, the **vario MAX CNS** must be checked for leakage. If necessary, replace all O-rings inside the furnace chamber since they may have been brittle because of the interrupted ventilation.
- The exchange of the gripper arm must only be carried out when it is cooled down. To do so, use function **Options⇨Miscellaneous⇨Replace Gripper** and wait for 15 minutes until the gripper has cooled down.
- Emptying of the crucibles container must only be carried out in the instrument's standby mode. There is the danger of a burning crucible falling into the instrument or in the lab during an analysis run.
- Keep the cover hood locked during routine operation. It is forbidden to work inside the cover hood during operation.

Change of operation mode instrument conversion

- The max. permissible temperature limit values of combustion tube and reduction tube are adjusted to the appropriate mode of operation. They are always approx. 50°C higher than the necessary operating temperatures.

  When changing the mode of operation make sure the tube fillings are adjusted to the respective operating temperatures and cannot overheat. **When overheating the tube fillings they can melt, run into the furnace chamber and destroy the furnace.**

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4. Function of the vario MAX CNS / CN

The elemental analyzer **vario MAX CNS** is a fully automatic instrument for a speedy and quantitative determination of the elements CNS and CN. The instrument can determine simultaneously in two different measuring modes the elements CNS or CN.

The suitability of the **vario MAX CNS** analyzer for the element analysis depends on the controllable combustibility of the samples or sample quantities.

It is also possible that certain substances, as for instance fluorine, phosphate or samples containing heavy metal have a negative influence on the analysis results or the life time of instruments components.

For difficult or contraversive applications (see for instance safety instructions below), especially if aggressive combustible products are to be analyzed, please make use of the counseling possibilities of our Application Laboratory.

Analyzing aggressive chemicals, acids, alkaline solutions, solvents, explosives or materials that can create explosive gas mixtures with the standard equipment of the vario El is explicitly forbidden or can be executed at your own risk. It can lead to damages of the instrument and to injuries of the operating personnel.

4.1 General Measuring Principle

The elemental analyzer **vario MAX CNS** works according to the principle of catalytic tube combustion under oxygen supply and high temperatures. The combustion gases are freed from foreign gases (for instance volatile halogen). The desired measuring components are separated from each other with the help of specific adsorption columns and determined in succession with a thermal conductivity detector (TCD). Helium (He) serves as flushing and carrier gas.

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4.2 Analysis

The automatic control of the analysis procedure is accomplished through the software and the sample specific oxygen dosing. The oxygen dosing can be allocated as up to 8 alpha numeric characters through function **Options⇨Method.**

We will now explain the timely and functional course of a CNS analysis. With the help of illustrations 4.1 and the figures/letters in brackets, it is possible to follow the location of the described process. Illustration 4.1 shows the flow diagram of the CNS mode. Illustration 4.2 shows the flow diagram of the CN mode.

Legend			
V1	switch valve to oxygen during combustion		
V4	O_2 switch/inlet valve	S	water condenser for CN operation only
V6A	H_2O outlet valve, CN mode pressure reduction valve, CNS mode	R	membran gas drying for CN operation only
V6B	He check valve	A	O_2 sensor for CN operation only
V9A,B	switch valves for CO_2 and SO_2 desorption paths for CNS operation	B	furnace (3 tubes)
V10	He flush valve	D	soot filter (filling quartz wool)
V16	He check valve to leak test	E	reference drying tube (filling Sicapent®)
P1	pressure sensor combustion tube inlet	F	combustion tube
P2	pressure sensor measuring MFC inlet	G	post combustion tube
T1	pressure controller measuring side	H	reduction tube
T2	pressure controller flushing side	J	crucible
K1	MFC for lance flush/O_2 dosing	N	detector measuring side
K2	MFC measuring gas	O	detector reference side
L	throttle reference gas flow	left	drying tube, for CN operation only (filling Sicapent®)
C2	SO_2 adsorption column	center	drying tube (filling Sicapent®)
C1	CO_2 adsorption column	right	drying tube (filling Sicapent®)
		P	filter
		Q	throttle

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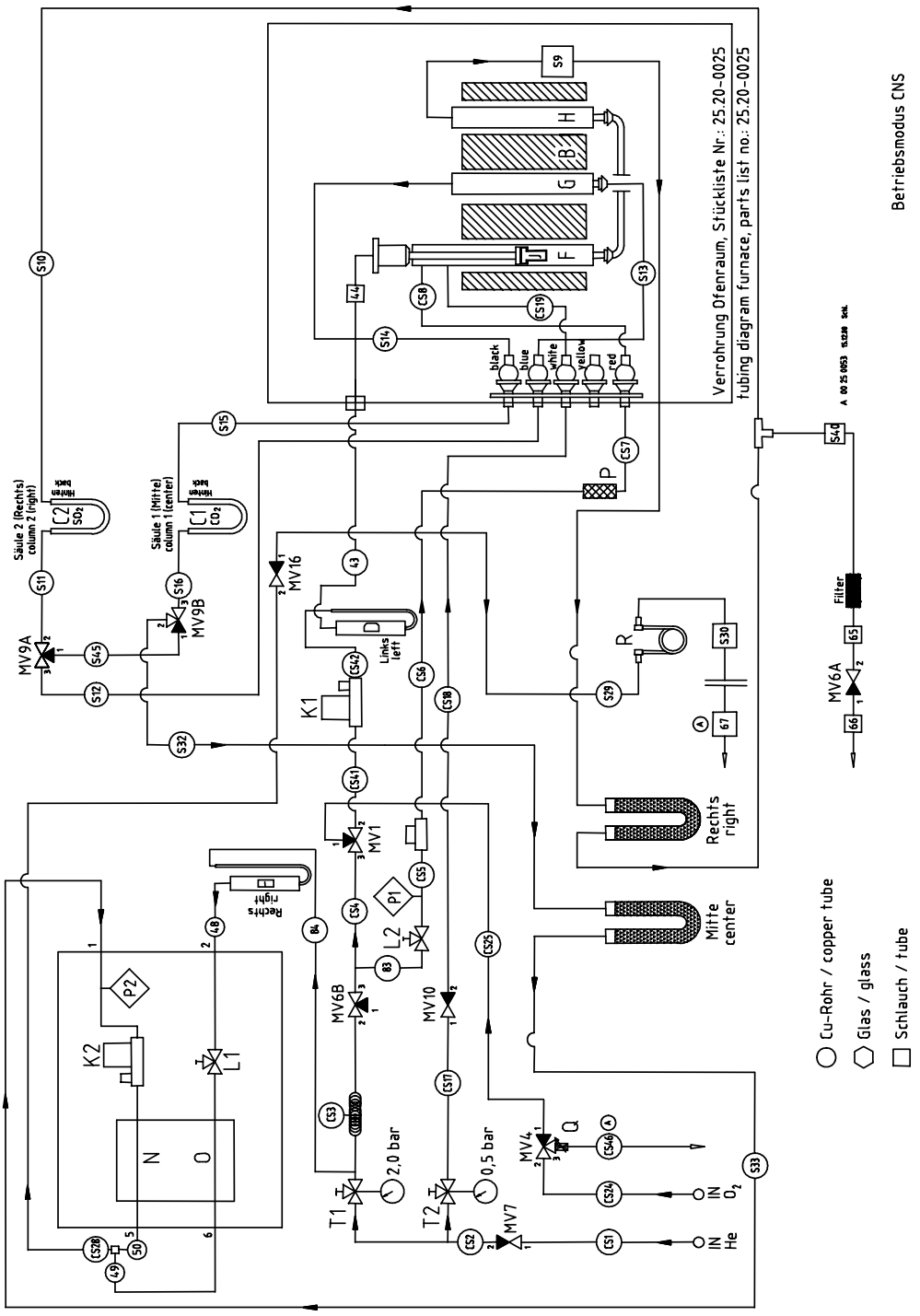

Illustration 4.1: tubing diagram vario MAX CNS (CNS mode)

The consecutive numbers in circles (CS1) state the number of the corresponding tubes and are meant to simplify the procedure of spare parts orders.

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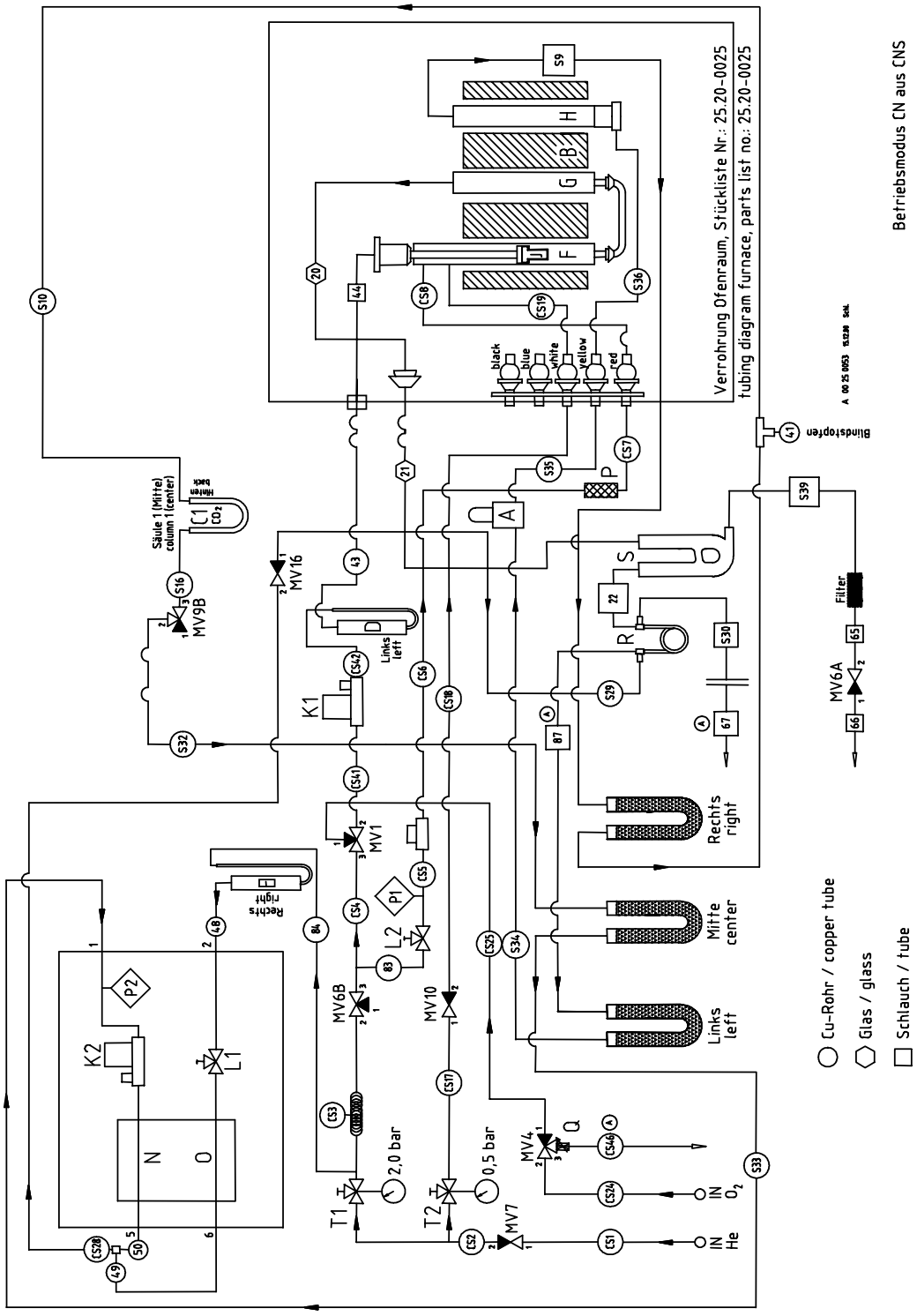

Illustration 4.2: tubing diagram vario MAX CNS (CN mode)

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4.2.1 Sample loading

The homogenized samples will be weighed in reusable crucibles (ceramic) and placed into the plate of the sampler.

Note:

For safety reasons, never open the door of the cover hood for loading the carrousel! The button next to the cover hood will be used for easy loading of the carrousel. By pushing the button once, the plate is being moved out. Setting back the plate to its original position is being done during the next feeding process. The sample to be analyzed next will be displayed on the PC in the status window, at the bottom on the right.

The weight will either be transmitted online from the balance via an interface or entered manually into the pc.
The sample name and the sample specific oxygen dosage (function **Options⇨Dosing Method**) will be allocated to the sample weight. Subsequently, the analysis can be started.

During measuring start an auto zero adjustment of the detector will be performed followed by a pressure decrease (valve V6A will be opened, valve V6B will be closed).
The gripper arm moves out of the combustion tube, releases the crucible of the previous analysis and takes out the next crucible from the carrousel. At the same time an intense counterflush of the combustion tube via valve V10 is being performed in order to prevent atmospheric nitrogen (⇨ nitrogen blank value) from entering into the open combustion tube. The counterflush is terminated when the gripper arm is back into the combustion tube.
In parallel with the sample feeding procedure also helium is being flushed 300 mL/min via MFC K 1 in order to flush the new crucible free of atmospheric nitrogen.

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4.2.2 Substance digestion and removal of foreign gases

After end of sample feeding, oxygen dosing in the crucible follows through the hollow grip arm (via V4, V1 and O_2-MFC). The moment of turning on the oxygen dosing will be determined by the run time "O_2 delay" in menu **Options⇨Parameters**. The oxygen dosing amount is adjusted via the dosing parameter selected for the sample.

The oxygen dosing will be performed "semi-automatically", i. e. optional

a) in operation mode CNS from a combination of combustion time (⇨ dosing parameters) and peak shape of the N peak.

b) in operation mode CN from a combination of combustion time (⇨ dosing parameters) and the O_2 excess measured at the end of combustion. The O_2 excess will be measured by the O_2 sensor.

Approx. 30-45 seconds after end of sample feeding an integrator reset is being performed which sets the integration display to zero. Some seconds later the N peak and the N integration starts.

During oxidated combustion the elements C, H, N and S produce - in addition to the molecular nitrogen (N_2) - the oxidation products O_2, H_2O, NO_x, SO_2 and SO_3 as well as volatile halogen compounds.

There is a tungsten oxide filling (WO_3-granulate) in the combustion tube. In the following reduction tube (copper filling) nitric oxides and SO_3 will be quantitatively reduced to N_2 and SO_2. The excess oxygen will be bound, halogens will be bound at the silver wool. Directly after the reduction tube the drying of the gases takes place at the Sicapent®.

The gases now flow through the SO_2 adsorption column and afterwards through the post combustion tube (filling CuO + Pt-cat. + copper).

The carbon compounds which were not yet quantitatively oxidized (e.g. CO, CH_4) will be oxidezed quantitatively to CO_2 in the post combustion tube. The CO_2 adsorption column is located behind the post combustion tube. For safety reasons a second fine drying will be performed after the CO_2 adsorption column.

In case of CN operation mode different tube fillings will be used and the gas way changes: combustion tube → post combustion tube → water condenser → membrane drying → fine drying → CO_2 adsorption column. SO_2 and halogens will be chemically bound in the reduction tube.

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4.2.3 Separation of the measuring components

As the thermal conductivity detector cannot specifically distinguish between the different components of the reaction gas, the reaction products N_2, CO_2 and SO_2 must be flushed individually and one after the other by the carrier gas into the measuring module. This separation of measuring components is carried out through adsorption of CO_2 and SO_2 on heatable columns. The nitrogen unaffected by the adsorption columns will be measured by the TCD as the first component.

After the N integration is concluded the integration value will be saved.

In case of CNS measurement an integrator reset is being performed, the CO_2 adsorption column heated (for CO_2 desorption) and the CO_2 peak integrated.

Subsequently, the gas way will be switched over via the valves V9 A/B, (by-pass of the CO_2 column). The S-integrator reset will be performed, the SO_2 column will be heated and the SO_2 peak will be integrated.

Note: for separation of CO_2 traces which are on the SO_2 column the SO_2 desorption will be carried out in two temperature steps.

After the SO_2 integration end the next analysis start will be carried out.

The next analysis start during CN measurement will be carried out after the C integration end. Apart from that, the process remains the same.

4.2.4 Detection

The thermal conductivity detector consists of two measuring chambers. The gas flows through them at constant rate of flow. The carrier gas flow can be turned off during measurement breaks because the measuring chambers are resistant against oxygen. During operation the reference measuring chamber is flushed with pure carrier gas He (reference gas flow) while the measuring gas flow, i.e. the respectively desorbed fraction of the reaction gas (e.g. He/N_2- or He/CO_2 or He-SO_2-mixture) passes through the other one.

The two chambers constitute a measuring bridge and its electrical imbalance provides a direct gauge for the nitrogen content in the carrier gas. The detector output voltage is recorded as a function of time and then digitized, integrated and represented as an integral.

Through a calibration for each element, the integral is allocated to an absolute element content of the sample. From the resulting content and the sample weight, the percentage of the element content is calculated.

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5. Installation and start-up

Installation and operation of the **vario MAX CNS** and peripheral equipment should only be carried out by persons who have been authorized **by E l e m e n t a r Analysensysteme GmbH**.

Opening the transport packing by unauthorized personnel can lead to loss of warranty rights! If the supplied instruments show damages, please contact the freight forwarder and the manufacturer immediately.

5.1 Preparations for operation

These prerequisites are absolutely adhered to for a smooth operation of the **vario MAX CNS**.

The following measures have to be taken before turning on the instrument:

- Suspend weight on the O_2-supply line (the weight is part of the accessories).

Illustration 5.1: Weight for O_2-supply line

- Mount glass line (from post combustion tube outlet to water condenser inlet). The glass tubes are packed separately. It will only be necessary for CN operation.
- Remove transport guard of the furnace.

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5.1.1 Working environment

The whole installation, consisting of the nitrogen analyzer **vario MAX CNS** and the respective peripheral equipment as balance and PC should be protected from draught and direct sources of heat. An air conditioned room is recommended as some parts of the analyzer give off heat.
In addition, appropriate steps are to be taken to avoid electrostatic charges. Electrical discharges on the unit can lead to malfunctions.

placement area	type of protection: IP 20 only in dry, well ventilated rooms; installation of the **vario MAX CNS** only on a heat resistance surface.
space requirement (incl. PC and printer)	length: 2.00 m depth: 0.75 m distance from the walls at least 20 cm at all sides, otherwise the fans of the unit will not function properly.
ambient temperature	constant temperature permissible min.: + 15°C max.:+ 35°C
power supply	230 Volt AC ± 10 %, 50/60 Hz connected load: 1.6 kVA power supply and frequency must match the information on the legend plate of the instrument
connections	2 sockets and multipoint strip with 4 sockets for connection of peripheral instrument or 5 sockets protection class I, connection for earthed conductor wire required fuse protection: 16 A/delay-action fuse
gas connections	gas connections must be available in the immediate vicinity of the instrument. Pressure reducing valves are required for connection. Pressure reducing valves He and O_2, each with two steps (2^{nd} step 0-8 bar He, 0-3 bar O_2) with shut-off valve
exhaust pipes	atmospheric into the open air or into a fume hood (see safety instructions)

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The installation room must be well ventilated. The exhaust gas pipe must lead into the open air or into a fume hood because during operation toxic gases (e.g. CO during wrong combustion as well as SO_2) and ashyxiating gases (e.g. per analysis approx. 1 L CO_2) come out of the instrument. It is possible that during constant operation the gas content of the air exceeds the permissible maximum workplace concentration .

The end of the gas pipe must lead to a sheltered (wind-free) place in the open air since pressure fluctuations, for instance through wind, lead to detector instabilities.

5.1.2 Necessary gases and chemicals

When handling chemicals observe the safety instructions of the chemical manufacturer (see labels on bottles and safety data sheets)!

carrier gas	**helium He** ; purity 99.996 % pre-pressure 3.8 bar consumption: approx. 800 mL/min carrier gas approx. 5 L flushing gas during sample feeding
combustion gas	**oxygen O_2** ; purity 99.995 % pre-pressure ca. 2.5 bar consumption: aprox. 0.4-0.8 liter per analysis
reagents	chemicals for filling the reaction tubes are included in the basic set of consumables. For further analysis we suggest a set of consumables
calibration standards	glutamic acid and sulfadiazine are supplied with the first set of consumables. If desired make other standards available. The substances have to be at least of p.a. quality or certified for elemental analysis.

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5.2 Instrument Design

The walls of the **vario MAX CNS** should only be opened by authorized personnel for maintenance and servicing purposes.

The design of the basic unit of the **vario MAX CNS** is as follows:

5.2.1 Front view

Illustration 5.2: front view of the vario MAX CNS with door open

Legend	1	main switch with control light
	2	open front door
	3	door of furnace room
	4	soot filter (filling filter, 6 pcs.)
	5	reference gas drying tube (filling Sicapent®)
	6	drying tube (filling Sicapent®) for CN operation only
	7	CNS operation: drying tube (filling Sicapent®)
	8	CNS operation: drying tube (filling Sicapent®)
	9	door opens/closes by pressing the door
	10	start button for position "load carrousel"

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5.2.2 Back view

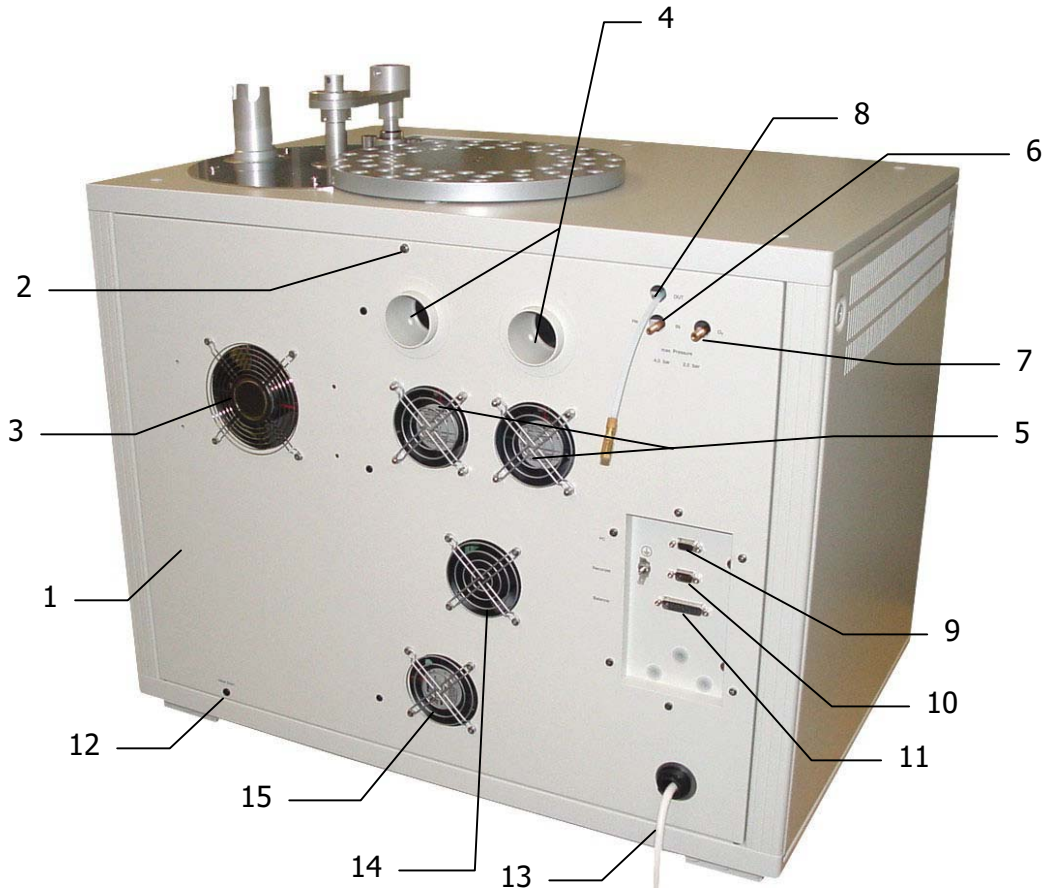

Illustration 5.3: rear view of the vario MAX CNS

Legend	1	back wall
	2	screw for fastening the back wall
	3	vent for cooling the furnace area
	4	outlet of warm air of the adsorption columns
	5	vent of the adsorption columns
	6	helium gas inlet
	7	oxygen gas inlet
	8	gas outlet (measuring gas and reference gas)
	9	connection to PC
	10	connection to recorder (analog outlet)
	11	connection to balance
	12	drain hose for condensate water
	13	power supply cable
	14	vent electronics
	15	vent transformer

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5.2.3 Furnace

The furnace is located behind the right front door (see illustration 5.2) of the **vario MAX CNS.** The door is secured through a magnet and can be removed easily.

The **vario MAX CNS** is equipped with a 3-tube-furnace with three heating tubes, each of which works independently. The temperature for each tube is pre-selected through the PC. All heating tubes are monitored through the software and in case of failure the heating system will be disconnected from all poles of the supplied voltage.

The reaction tubes are inserted in the three-tube furnace - combustion tube in the rear, post combustion tube in the middle and reduction tube in the front. For fast and easy exchange of the reaction tubes, it is possible to pull the three-tube furnace - after loosening of a few holding devices - out of the unit on a rail. The replacement of the reduction tube is also possible when the 3-tube-furnace is inside the instrument.

5.2.4 Safety features

Close cover hood of the **vario MAX CNS** in order to prevent unintended engaging. Danger of injuries by swinging, glowing gripper arm.

- The crucibles container will be monitored by a magnetic sensor. When the container is removed and the right side wall is open, it is not possible to start the feeding system.

- Each heating system is monitored through an internal micro processor control for the following failures:
  1. A heater does not reach its set point temperature during a selected time.
  2. Thermocouple is defect or displays an invalid value.
  3. The selected limit temperature is surpassed.

  These failures will switch off the power supply for all heaters. Similar dangerous failures will cause the furnace transformer to switch off.

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The max. permissible temperature limit values of reaction tubes are always adjusted to the respective mode of operation. They are always approximately 50°C higher than the necessary operating temperatures.

When changing the mode of operation make sure that the tube fillings match the appropriate operating temperatures and do not overheat. **Overheating can cause the tube fillings to melt. The melted material can run into the furnace chamber and destroy the furnace.**

- The **vario MAX CNS** is equipped with a temperature limit control, which turns off all furnaces automatically in case the temperature limit inside has been exceeded (e.g. if a cooler is not working).

- He-supply is only possible when the mains switch is on.

5.3 Start-up

Operation of the **vario MAX CNS** and peripheral equipment should only be carried out by persons who have been authorized by **E l e m e n t a r Analysensysteme GmbH**.

For start-up please follow these instructions successively:
Please also note the shut-down condition of the unit (see chapter 6.5)

a) For transport purposes of the **vario MAX CNS,** pull out the handles at the sides of the unit and tilt them up (see illustration 5.4)

b) Set instrument up (for instructions regarding placement area see chapter 5.1.) and connect peripheral instruments (see chapter 5.3.1).

c) Connect supply gases and install exhaust gas outlets and drain pipes (see chapter 5.1).

d) Loosen transport guards.
The moveable 3-tube furnace must be secured during transport with a screwed on angle piece (see illustration 5.5) to prevent accidental sliding. To loosen the transport guard open the front door and right side panel. The screws holding the transport guard are now accessible and be loosened.
Note: For first start-up remove the cable bundle from the tube linkage pieces inside the combustion chamber.

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e) Do not install the furnished gripper yet.
Caution! There are two different gripper types for the operation modes CNS and CN. Please do not mix up. There is an additional tagging by a ring mark labeled with CNS.
CNS - order no.: 25.20-0004
CN - order no.: 25.00-0029

f) Fill quartz and glass parts and install (see chapter 8ff).

g) Turn PC and printer on and - if necessary - wait for booting procedure to be ended and install software (see chapter 7).

h) Mount cover hood ⇨ Note: Subsequently, lock door and remove key.

i) Turn on main switch of the **vario MAX CNS** (see illustration 5.1). The instrument carries out a reference run of gripper arm and carousel.

j) Turn on operation gases:

He set pre-pressure at the place where pressure is taken (approx. 3.8 bar) so that inlet pressure gauge (gas insert, left) (1) shows 2.0 bar and inlet pressure gauge (2) shows 0.5 bar.
O_2 pre-pressure 2.5 bar (2nd step bottle pressure gauge).

k) Load **vario MAX CNS**-software under Windows® by clicking on the corresponding icon. The software is menu controlled, so that the user sees most of the operational steps displayed on the screen. Moreover, most of the standard operation parameters have been set in the factory (see chapter 5.3.5) which the user does not need to change.

l) Select desired mode of operation in the menu **Mode** (CNS or CN) and the sensitivity (standard or precise) and confirm with [**OK**]. Now confirm all items appearing in the dialog **Change Mode** after checking them and leave the dialog with [**OK**] (see chapter 7). The system is now being initialized from anew, if necessary.

When changing the mode of operation make sure that the tube fillings match the appropriate operating temperatures and do not overheat. **Overheating can cause the tube fillings to melt. The melted material can run into the furnace chamber and destroy the furnace.**

m) Now install the gripper and change via **Options⇨Miscellaneous⇨Gripper** (see also chapter 8.3).

n) Perform leakage test (**Options⇨Miscellaneous⇨Rough Leak Check**).

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o) Start heaters and check parameter setting at the PC (see chapter 5.3.5). Click to the menu item **Options⇨Parameter** and enter your pass word or confirm the factory set parameters by clicking on [**OK**]. In the **Parameter**-dialog the settings should be checked (see chapter 5.3.5) and the operating temperatures must be entered. After confirming the changes with [**OK**] you can observe in the status field how the furnace temperatures rise up. When the heaters have reached the set temperatures (heating time approx. 75 minutes), the **vario MAX CNS** is ready for analyzing procedures.

Note: Changing the instrument parameters is only possible with the corresponding access rights.

p) Check in the menu "**Standards"**, whether or not the desired test substances (for instance glutamic acid and sulfadiazin) have been defined (see chapter 6).

q) Check if calibration coefficients have been loaded (**Math.⇨Coeffs**). With the new **vario MAX CNS** we supply the calibration report of the quality control test. The standard mode as well as the precise, sensitive mode are calibrated at factory.

r) Check functionality of the **vario MAX CNS** (e.g. with "run-in", blank value and standard - measurements), thereby watching the gas flows and pressures (see chapter 6).

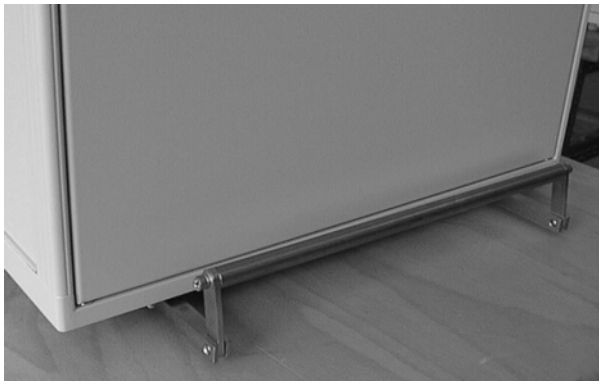

Illustration 5.4: handle carrying position

Illustration 5.5: transport guard 3-tube-furnace

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5.3.1 Connection of peripheral instruments

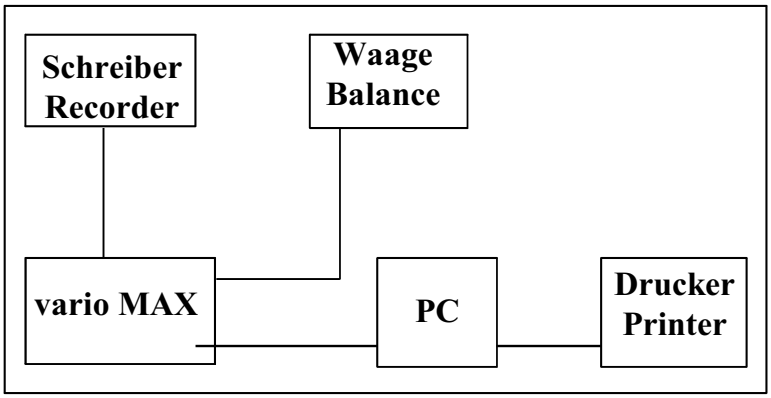

Illustration 5.6: connection of peripheral instruments

As shown in illustration 5.6, the element analyzer consists of the basic instrument **vario MAX CNS** and peripheral equipment. PC and printer are necessary for control and evaluation. Recorder and balance are optional. The peripheral instruments are connected (with the supplied cables) to the appropriate sockets at the back of the **vario MAX CNS** (see illustration 5.4 and inscriptions on the instruments).
As an option, a recorder with the supplied cable can be connected for recording of the following values:

cable inscription	measuring value	recorder setting
TCD comp (±)	TCD-signal, compensated	0 to 10 V according to height of peak
TCD (±)	TCD-signal,	0 to 5 V according to height of peak
rest O_2 (±)	no function	
pressure (±)	system pressure [0 - 2.50 bar]	0 to 5 V, (2 mV/mbar)

These measuring values can also be selected in the PC graphic. Chapter 7.2.4 describes how this graphic can be printed.

5.3.2 Connection of the supply lines

The installation room must be well ventilated. The exhaust gas pipe must lead into the open air or into a fume hood because during operation toxic gases (e.g. CO during wrong combustion as well as SO_2) and ashyxiating gases (e.g. per analysis approx. 1 L CO_2) come out of the instrument. It is possible that during constant operation the gas content of the air exceeds the permissible maximum workplace concentration.The end of the gas pipe must lead to a sheltered (wind-free) place in the open air since pressure fluctuations, for instance through wind, lead to detector instabilities.

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Illustration 5.3 shows the connections at the back of the **vario MAX CNS**. For easy connection of the supply and discharge lines, the left cover plate at the top of the **vario MAX CNS** can be unscrewed and removed.

5.3.3 Succession of instrument start-up

To ensure a smooth operation of the **vario MAX CNS** without any failures, it is absolutely necessary to turn on the basic instruments and the peripheral equipment in a certain succession.

a) Switch on PC with screen and printer and wait for booting procedure to be ended
b) **Switch on the vario MAX CNS** at the main switch (see illustration 5.2) and wait for the reference run..
c) **Load vario MAX CNS-**software by clicking the corresponding icon.
d) Now the gas supply lines He and O_2 can be opened.
   Note: The gas O_2 should only be turned off if no tests are run for a long time (> 1 week).

5.3.4 Instrument settings

These are the suggested standard instrument settings:

gas pressure	Set the O_2-pre-pressure at the reducing valve to 2.5 bar. For He, the pre-pressure is set to 3.8 bar. The inlet pressure gauge 1 will then show 2.0, inlet pressure gauge 2 will then show 0.5 bar (in the instrument, gas insert left)
flow	the flow rates are factory set to: measuring gas outlet: about 700 mL/min (precise mode) about 800 ml/min (standard mode) reference gas outlet: 50 mL/min. - 100 mL/min.

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5.3.5 Parameter setting at the PC

When changing the mode of operation make sure that the tube fillings match the appropriate operating temperatures and do not overheat. **Overheating can cause the tube fillings to melt. The melted material can run into the furnace chamber and destroy the furnace.**

In every mode of the operation the parameters necessary for the analysis procedures are filed in the menu **Options⇨Parameter.** Illustration 5.7 shows the **Parameter** window for the CNS mode with the factory set selections. The following is only an explanation of the individual parameters

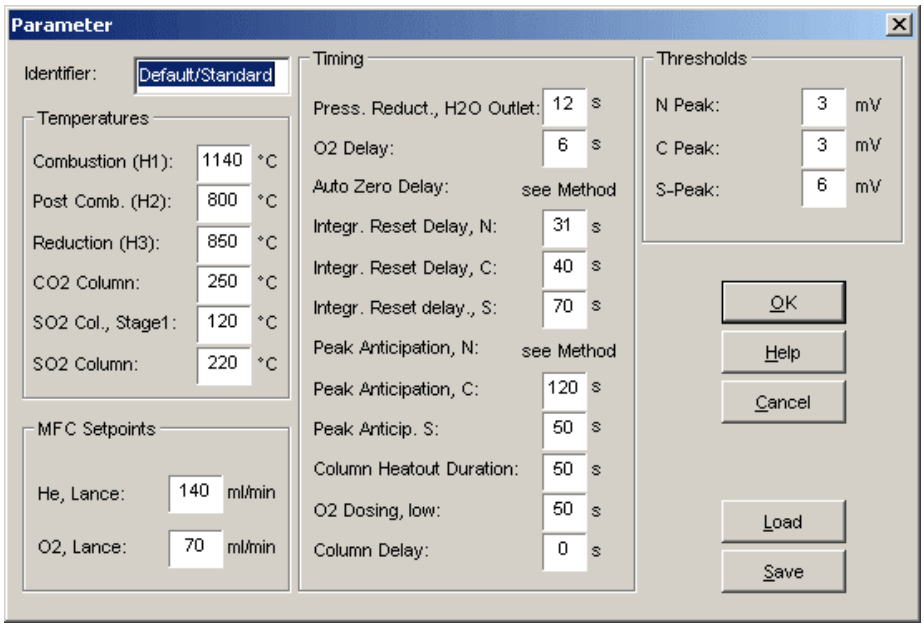

Illustration 5.7: parameter-window in CNS mode

Note: Changing the instrument parameters is only possible with the corresponding access rights.

Note: The mode dependent analysis parameters are average values. The optimized operating parameters of the first start-up are stored on the supplied diskette. These settings can be optimized for special applications or unusual requirement through the operator at any time**.**

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For the meaning of the individual parameters, please note the following:

Parameter	Factory Setting		Meaning
Identifier	**Precise Mode**	**Standard mode**	**Field of remarks of the operator.** A sensible use is, for instance, to enter the name of the stored file.
Temperatures			**Setting the nominal temperatures for the 3-tube furnace**
Furnace 1	CNS 1140°C CN 900°C	CN 900°C	Heater rear, combustion tube
Furnace 2	CNS 800°C CN 900°C	CN 900°C	Heater middle, post combustion tube
Furnace 3	CNS 850°C CN 830°C	CN 830°C	Heater front, reduction tube
CO_2-Column SO_2-Column	250°C Stage 1 120°C Stage 2 250°C		CO_2 desorption temperature SO_2 desorption temperature
Times			**Here the run times of the instrument control are entered**
Pressure decrease H_2O drain	12 s	12 s	During this time, the pressure in the instrument will be decreased and the He supply interrupted.
O_2-Delay	6 s	6 s	Begins after the analysis start and thereafter the O_2-dosing starts. This time is optimized in such a way that for the combustion the best possible initial O_2-concentration is available.
Auto Zero Delay	mode- and matrix dependent		The auto zero adjustment is delayed in order to avoid the temporary fluctuations of the base line after the start of the analysis.
at CN at CNS	45 – 75 s 75 – 120 s	40 – 60 s	
Integrator-Reset-Delay	mode- and matrix dependent		The integrator-reset prior to the integration is delayed so that temporary instabilities of the base line prior to the measuring peak are not integrated.
at CN at CNS	30 – 45 s 60 s	20 – 35 s	

Note: Desorption temperatures are optimized in such a way that at a low concentration of the elements to be determined, a possibly high element concentration occurs in the carrier gas after desorption (Purge and Trap-method) in a possibly short analysis time. This means that narrow and high peaks are formed.

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Parameter	Factory Setting		Meaning
	Precise Mode	**Standard Mode**	
Peak-Anticipation N	240 s	90 s	This time becomes effective after the respective integrator reset. During this time the integrator waits for a measuring peak. If no measuring peak appears during this time, integration is ended and the next element is taken and/or the next analysis begins.
Peak-Anticipation C	195 s	150 s	See Peak-Anticipation N
Peak-Anticipation S			See. Peak-Anticipation N
Heating time N- mode C- mode	 300 s 195 s	 120 s 120 s	This is the minimum column heating time. In N operation mode this time is fixed, in C operation mode it is automatically adjusted to the peak width.
O_2-Dosing low N-mode C-mode	 50 s 50 s	 10 s 10 s	During this time a low amount of O_2 will be dosed at the beginning of the combustion in order to avoid high pressures.
Thresholds			**Here the termination thresholds for peak-end detection are determined.**
N-Peak	3 mV	3 mV	For all sensitivity stages
C-Peak	3 mV 6 mV	3 mV -	For C non-sensitive For C sensitive
S-Peak	6 mV		For all sensitivity stages
MFC-Nominal values			**Setting of various gas flows**
O_2, low	70 ml/min	70 ml/min	This is the low O_2 dosing amount which has to be set at the beginning of the combustion in order to avoid high pressures.
Helium	680 ml/min	780 ml/min N 1000 ml/min C	Carrier gas flow
He, lance	140 ml/min		He flush gas flow through the gripper, if no O_2 is being dosed.

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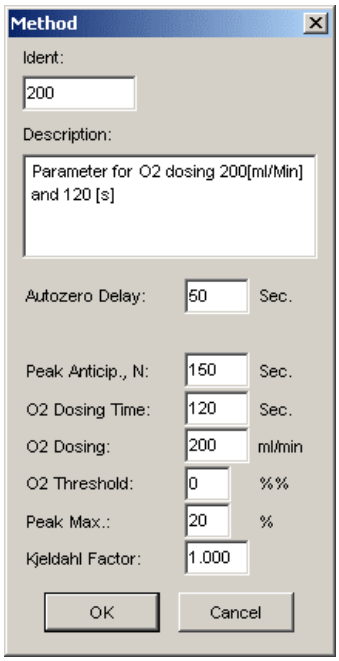

Illustration 5.8: Combustion parameters in CNS mode

The combustion parameters are the O_2 amounts to be dosed for the quantitative combustion of the samples.

This O_2 dosage comes right after the necessary "low dosing" at the beginning of the combustion. A "name" of up to 8 alphanumerical characters can be defined and saved which will be allocated to a dosing amount and a dosing time.

Note: Changing of method parameters is only possible with the corresponding access rights.

Note:

The dosing time is a minimum dosing time. The O_2 dosing will be performed until one O_2 turn-off criterion is fulfilled (exceeding the O_2 threshold, i.e. O_2 excess or integration almost concluded, i.e. recognition of the peak maximum).
In operation mode CNS the O_2 sensor is not integrated. Therefore, only the threshold of the peak maximum can be entered in the operation mode CNS. The value of 20% has proven to be appropriate as a standard value.
Please note that only one turn-off criterion can be entered. The criterion which is not requested (example: the shutting-off via the O_2 threshold) gets the entry 0.
See also chapter 6.1.2, Selection of the oxygen dosing.

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6. Operating the vario MAX CNS

The **vario MAX CNS** analyzer is suitable only for the element analysis of samples or sample quantities which are controlled combustible under mode dependent combustion conditions.

Analyzing aggressive chemicals, acids, alkaline solutions, solvents, explosives or materials that can create explosive gas mixtures with the standard vario MAX CNS equipment is explicitly forbidden. It can lead to damages of the instrument and to injuries of the operating personnel.(see also chapter "Important Notes", point 2)

6.1 Sample preparation

Regarding sample preparation and homogenization, we recommend the special literature that is commonly available. Special attention should be paid to sample grinding and drying, because these procedures bear the danger of contamination and also the loss of volatile components. When preparing the samples for the **vario MAX CNS,** keep in mind that an analysis of the total nitrogen is performed which means that air trappings must be noted. When analyzing carbon contamination could occur through touching the crucibles. Also keep in mind that the moisture content of the samples influences the results of the other elements.

For CNS-determination an additional amount of WO_3 Powder (11.02-0017 or 11.02-0024) must be added to the real samples. This amount depends on the kind and the weight of the sample.

Example: 200-300 mg organic sample (plants etc.)
Additional amount 150–200 mg WO_3
1000-1500 mg soil
Additonal amount approx. 400-500 mg WO_3

Important: The WO_3-powder and the sample should be mixed (e.g. stired with a spattle).

6.1.1 Selection of sample weight

The best sample weight depends on the N, C and S content, the homogeneity and the combustion behavior of the sample. The oxygen consumption and the resulting pressure during combustion determine the maximum sample weight. Combustion of the sample must be timely limited and the pressure must be controllable.

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General Rule:	**The sample weight should be as high as necessary** (C, N and S content well measurable) a**nd as low as possible.** (optimizing use of catalyst and necessary maintenance) The following is valid: **better more measurements than larger sample weights.**

If the sample matrix is not known, the sample weight at the beginning should be 100 mg and the combustion pressure behavior should be watched at the flow meters of the **vario MAX CNS.** Ascertain that during the combustion phase, pressure 1 does not go above 1.7 bar and at the same time the flow control He-comb. does not go below 350 mL/min. Otherwise, soot and tar can be pushed into the gas supply lines (the color of the soot filter turns dark).

If the combustion pressure is too high (He indicator on zero), the gas control unit of the **vario MAX CNS** can become soiled.

In this case, the sample weight has to be reduced by factor 2 to 3. If no pressure deviations occur, the sample weight can be increased step by step to max. 350 mg for organic substances and max. 800-1000 mg for soil. After optimizing the sample weight, the sample matrix can be analyzed without supervision.

6.1.2 Selection of the oxygen dosing

With insufficient oxygen supply the catalysts will be reduced and therefore their effect and lifetime will be limited. The incompletely combusted sample is being removed in the crucible from the instrument but the subsequent analytical result is being falsified.

The oxygen dosing is being performed via an allocation of a method name. The name may consist of up to 8 alphanumerical characters and can be saved. The determination is being carried out under **Options⇨Method⇨Modify Method.**
The method name and the corresponding O_2 dosing (time and amount) will be determined. The dosing time will be the minimum dosing time, the O_2 supply will be turned off when one of the two monitoring functions at the end of the combustion will be recognized (see chapter 6.1.3).

In any case, the O_2-dosing (dosing time and amount) depends on the sample matrix and the weight. Which means that a parameter with sufficient oxygen dosing must be assigned to each sample.

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General rule: 350 mg organic substance with medium carbon content requires approx. 0.30 L to 0.35 L O_2 for combustion!

Note: Changing the method parameters is only possible with the corresponding access rights.

The following table can help with the selection of the appropriate parameters:

dosing time* [s]	dosing amount* [ml/min]	weight [mg]	sample matrix
120	120	approx. 200	sample with medium O_2-requirement (animal feed, cereals, starch, plants)
120	100	approx. 200	test substances (glutamic acid)
120	150	approx. 300 - 350	sample with medium O_2-requirement (animal feed, cereals, starch, plants)
120	50 100	approx. 200 approx. 800	samples with low O_2-requirement soil coal
120	150 175	approx. 200 max. 150	samples with high O_2-requirement (coal, oil)
60	50	10 - 20	test substances (e.g. sulfadiacine)
50	50	fictitious 100	blank value
90	50	200 – 250 µL	aquaeous solutions
120	75	approx. 300 µL	milk
120	75	approx. 200 µL	canned milk 7.5% fat

* factory set parameters

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6.1.3 Monitoring of the oxygen dosing

The table for oxygen dosing shown in chapter 6.1.2 contains only standard values and/or approximate data in order to avoid large mistakes.
The dosing times mentioned in the table are minimum dosing times. The actual necessary dosing time will be determined automatically. The following criteria will be used:

1. Operation mode CN only
   The built-in O_2 sensor measures the excess oxygen resulting from the combustion. At the end of the combustion the value increases (no more O_2 is necessary). The O_2 supply will be turned off if the O_2 threshold value is exceeded (**Options⇨Method**). This procedure can be applied to all sample types – also to such kind of samples which only contain traces of nitrogen.
   Recommended threshold: 15‰ to 17‰.

2. Operation mode CNS and CN
   The O_2 shut-off is controlled by the measuring peak, i.e. the oxygen supply will be concluded if the measuring peak decays to a certain value after reaching its maximum.

   The setting will be done under **Options⇨Method.**

   This procedure can only be applied for samples which have a nitrogen concentration of >0.1%. The advantage is that the residual O_2 amount is smaller and therefore the reduction tube will be protected, versus no. 1. The disadvantage is that every time the optimum value depends on the substance. It is between 30% (plant samples) and 10% (chemicals) and has to be tested.
   The setting of 20% has proven to be adequate as standard value.

   For very low N concentration (N-Peak < 10 mV) this procedure does not work properly anymore.
   Since the O_2 sensor cannot be used in CNS mode the O_2 demand of the sample has to be estimated precisely. This applied especially for pure hydrocarbon (oil, fat).

   Example: oil sample

C-content	85.00%
H-content	14.95%
N-content	0.05%

   sample weight 150 mg, resulting the following absolute amounts C, H and N:

C:	127.50 mg = 10.625 m Mol C
H:	22.425 mg = 11.213 m Mol H_2
N:	0.075 mg

   From stoichiometry there is the following O_2 demand for the combustion of C and H:

   C = 238.0 mL O_2
   H = 125.6 mL O_2
   363.6 mL O_2

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Divided in 2 minutes (120 sec) combustion time there will be an O_2 dosing of 181.8 mL/min. Considering the dosing "low" at the beginning (70mL/min ˉ50 sec.), an O_2 dosing of 175 mL/min. can be set.

6.1.4 Selection of the detector sensitivity

Upon delivery, **vario MAX CNS** is calibrated in analysis mode "standard". The measurement is done with an optimized timing for most applications. For special applications (e.g. nitrogen in low concentrations, see the following table) the **vario MAX CNS** can be switched to the "precise" mode. In this mode, the analyzer has two different sensitivity stages (sensitive, non sensitive) where the sensitive stage is already calibrated from works' side.
The selection of the requested sensitivity is done in the menu **Mode⇨Sensitivity.**

Note:

- The sentitivity **cannot** be changed "automatically" (e.g. via parameters, from sample to sample).

- For GLP-conformity reasons a new file is being created during a sensitivity change.

  Save the previously measured data!

The following sentivity settings are recommended:

type of sample	**mode and sensitivity**
soil and plant samples weight soil 1.5 g weight plants max. 400 mg	N and/or CNS precise/sensitive
soil and plant samples weight soil max. up to 2.0 g weight plants > 400-800 mg	N and/or CNS mode standard
protein determination in food and feed in general weight 300-800 mg	N mode standard
starch samples weight approx. 600 mg	N and/or CNS mode precise / sensitive
protein determination in milk and dairy products weight 300-2000 mg	N mode precise / sensitive

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6.2 Daily measuring routine

Part of the daily measuring routine (after shutdowns for longer than 2 hours) is **conditioning** the **vario MAX CNS** with several so-called "Run-in"-samples. We suggest 1 to 2 analyses of 200 mg ± 10 mg glutamic acid and subsequently 3 to 4 analyses of approx. 10 mg sulfadiacine. Also very good are 2 to 3 analyses of plant or soil standards. During the conditioning phase the correct process of the analysis, the function of the individual components and the flow and pressure behavior of the gases should be checked.

The next step is a **factor determination** (see chapter 6.2.2), to adjust the calibration to the atmospheric conditions of the analysis´point in time. The reproducibility of the measurements and the day-to-day fluctuations of the daily factor are a good criteria to judge the instrument condition (catalyst aging, leaks in the gas lines, blockages etc.).

After **editing the sample data** and assigning the respective parameters the analysis can start (see chapter 6.2.3).
The determination of the **blank value** can be useful for several reasons (see chapter 6.2.1).

6.2.1 Blank value determination

The performance of a measurement with an empty crucible serves mainly as a test of gas tightness of the instrument. High blanks may also be an indicator for a poor gas quality.
The measurement with an empty crucible will be done under the following conditions: 50 sec oxygen dosing / 50 ml/min oxygen amount / 120 sec peak expectation time / 100 mg entering of a fictitious sample weight.
The resulting measuring values are not comparable with real blanks of a sample measurement and may therefore not be used for a measuring value correction. This correction will have to be done via the determination of the daily factor (see. chap. 6.2.2).

For approximate values for blanks – measured according to the selectable **Method "Blank"**- see table below:

integral value	mode / measuring range	instrument version
approx. 25 - 50	standard N mode	
approx. 100 – 250	precise N-sensitive	
approx. 25 - 50	precise N-non sensitive	
approx. 30	precise C-non sensitive	
approx. 100 – 150	S-sensitive	only vario MAX CNS
approx. 200	H-non sensitive	only vario MAX CHN

These values are reached after some measuring value run-in. Are the measured blanks higher than stated and the helium flushing during crucible feeding as well as instrument tightness and gas quality are ok, a falsification of the analysis results may still not occur.

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For measurements in the range of the lower detection limit a very small blank should be the attained. This can be reached e.g. by replacing the tube filling and tubes as well as the crucible material. Please be aware that brand new crucibles have to run through one blank measuring cycle first in order to remove possible contaminations. For additive chemicals and packing material that have be to be combusted as well make sure that they have no or a very low blank. Even for "pure" test substances trace contaminations cannot be excluded. The S content of glutamic acid may have a value of 0.01 – 0.03 % which corresponds to a peak area of approx. 200 – 350.

Usually in daily routine 3 x blank measurements will be performed at the beginning in order to check the instrument state. At least the first value often is far higher and can be evaluated as a "run-in".

6.2.2 Factor determination

The daily factor determination is necessary to adjust the calibration from works side to the daily changing ambient conditions (e.g. air pressure).

Note: For factor computation the used substances must be entered in the **menu standards** and identified as "*Factor sample*".

6.2.2.1 With calibration substances

To adjust the calibration to the daily measuring conditions, it is necessary to measure calibration standards which are very much like the samples in their integral range. Provided from works side: glutamic acid (9.52% N, 40.78% C) and sulfadiacine (22.37% N, 47.99% C, 12.81% S).

The daily factor can be determined in three ways.

a) Compute manually (nominal content/actual content) and assign to real samples [**Edit⇨Input**]
   For this purpose, e.g. glutamic acid can be used as sample.
b) With the function **Math.⇨Factor** the daily correction factor can be computed automatically and assigned to the following samples.
   For this purpose, the daily factor samples must have an identification that is defined in the **Standards**-menu and the standard substance must be activated as "factor sample".
c) In the menu **Math.⇨Setup** mode and degree of automation of the daily factor computation can be set.

   Note: If the daily correction factor is computed automatically we suggest to activate the function "*Standard-Tolerance-Monitoring*" (**Options⇨Parameter**).

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If the measured actual value deviates from the nominal value by more than the determined tolerance (e.g. 10%) in the **menu standard** , the current analysis will be terminated and subsequently, the instrument will be stopped.

6.2.2.2 With standardized test samples

Factory internal test samples or commercially available real standards with known CNS content and sample like matrix can also be used for factor correction.

Procedure	Example
• edit real standard in the menu standards	e.g. wheat standard, e.g. 1.92% N, 40.65% C, 0.155% S
• analyze 4-5 test samples with appropriate parameter	weight: 200 - 210 mg name: “wheat standard“ comb. parameter:120 s / 120 mL/min = "120"
• compute daily factor with **Math⇨Factor**.	

6.2.3 Editing an analysis series

For routine operation of the **vario MAX CNS** we suggest the following sample sequence:

Amount	Identifier	type of sample and description
1-2	"blank"	blank sample, without crucible, weight 100 mg, parameter "bl" (50 s / 50 mL/min)
1	"run-in"	250 ± 10 mg glutamic acid
2	"glutamic acid"	parameter 120 s / 150 mL = 100
2	"run-in"	10 – 30 mg sulfadiacine parameter 60 s / 50 mL/min = 50 short or
3-4	sulfadiacine or real standard	200 – 210 mg real standard parameter 120 s / 120 mL/min = 120
45	corresponding sample name	real samples, weight and parameter acc. to sample matrix (see chapter 5.1)
3-4	sulfadiacine or real standard	10 – 30 mg sulfadiacine, parameter 50 short or 200 – 210 mg real standard, parameter 120

etc.

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Before weigh-in of a new analysis series, sample feeder must be on zero (**Options⇨Sample Feeder)**, so that the running analysis number matches the number of the carousel. Then the substances are weighed in crucibles (see chapter 6.1ff).

Note: In case more than 60 samples shall be measured (= positions in the carrousel), the carrousel can be loaded one position after the other. In this case, position 1 in the carrousel corresponds to no. 61 in the measuring protocol.

The following data must be edited in the PC (**Edit⇨Input**):

- **weight [mg]**
  can either be entered manually or can be transmitted directly from an on-line balance.

- **Name (max. 16 characters)**
  sample name or identifier
  Daily factor and calibration samples must carry the name defined in the **Standards**-menu. Blank samples must be identified with "blank".

- **Parameter**
  corresponds with the name of the stored oxygen dosing (see chapter 6.1.3).

The other data in the **input**-menu (such as protein factor, blank value, daily factor) can be entered before or after the analysis.

6.2.4 Data storage and print-out

The measuring data is displayed on the screen after every analysis and stored in a temporary file. Apart from the measuring data also graphic information (pressure, peaks, etc.), i.e. the timely course of each analysis, are stored in the temporary file

We suggest to save the original measuring file after every analysis series (**File⇨Save** or **File⇨Save as**) and to look at the peak graphics before the next analysis series or print them out.

The graphic information is also stored. Thus, it is made available for a later reload of the document. This not only allows a later observation of the analytical results of the individual samples but also their timely course.
For this purpose the appropriate sample number is entered. It can be displayed in the menu **View⇨Graphic**.
In the menu **View⇨Configuration** the various measuring values can be selected for display. It is possible to show up to 4 measuring values in one graphic. For further information see online-help (**Help⇨Index**).

Of course the saved measuring values can be printed (**File⇨Print**) or worked at any time.

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6.2.5 Statistics

After clicking on **Math.⇨Statistics,** the dialog **Statistics: Define Groups** appears (see chapter 6.1). Here you can form measurement values into groups. The program then calculates the mean value for each of these groups and the standard deviation (see also **Help⇨Index⇨Statistics**). The values are displayed - after closing the dialog - in the sample field of the statistics display (**Display⇨Statistics**).

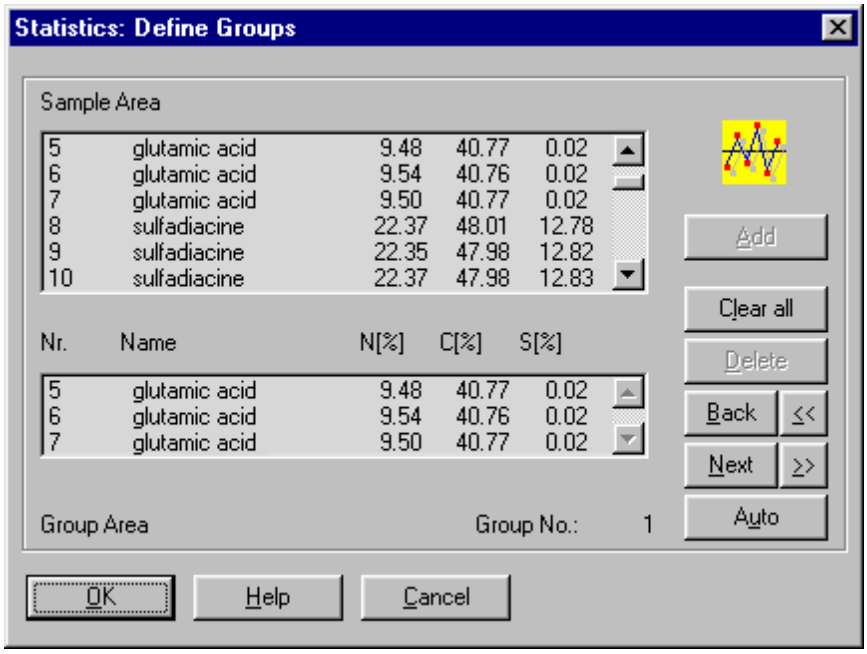

Illustration 6.1: dialog statistics: define groups

The dialog shows two areas, one "*Sample Area*" and one "*Group Area*". The "*Sample Area*" contains all samples currently in the memory. The "*Group Area"* shows the selected samples of a group. On the right side below the group area the "*Group No.*" of the currently displayed group is shown.

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Groups can be built in two ways:

Automatic Group Formation: With the button [**Auto]** the groups with the same name are combined to groups.

Manual Group Formation: The groups are formed individually through selection and group assignment.

Selection Possibilities:

- Selecting the sample by clicking it on.
- Selecting sample successions by clicking on the first one and then by checking on the last sample keeping the <Shift>-key depressed at the same time.
- Selecting individual samples by clicking on the first samples and then clicking on the other samples by keeping the <Ctrl>-key depressed at the same time.

The function [**Add**] adds selected samples to the currently activated group. [**Delete**] removes selected samples from the group area.
The buttons [**Next**], [**Back**], [<<] and [>>] permit opening of several groups.

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6.3 Checklist for analysis start

- Gases turned on?

- Heaters up to nominal temperature? CNS mode
  (combustion tube 1140°C, post-combustion tube 800°C, reduction tube 850°C)

- Pressures o.k.?
  (PC-pressure display for pressure 1 approx. 1.5 bar and pressure 2 approx. 1.2 bar)

- Has flow meter been checked?
  He/O_2 150, carrier gas He 680 (approximate values) in mode "precise"
  carrier gas He 780 (approximate values) in mode "standard"
  He-pressure right (He-Comb.) 530 mL/min (Standard or precise approx. values)

- Enough capacity in drying tubes?
  (last third still white?)

- Soot filter o.k.?
  (see chapter 8.5)

- Have maintenance intervals of the reaction tubes been observed?
  Always check reduction tubes directly.!

- Crucible container empty? Meter in maintenance menue set back to zero?

- Detector signal low and stable?
  (see TCD-signal in the status area)

Now the blank values and “run-in“-samples can be measured!

- Blank values and “run-in“-samples o.k.?

- Has analysis sequence been edited with daily correction factor?

- Parameter assigned correctly?

- Are samples on carousel?

If everything is all right, the analysis can be started with [**System⇨Auto**] or [**System⇨Single**]!

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6.4 Calibration

The elemental analyzer **vario MAX CNS** does not perform absolute measurements, i.e. the connection between the C, N and S content [mg C, mg N and mg S absolute] and the measuring values [C integral, N integral and S integral] must be calculated by means of a calibration. For this purpose, test substances (for instance aspartic acid or glutamic acid, urea and sulfadiacine) with given C, N and S content and different weights are analyzed, so that the desired calibration range is covered. The PC program automatically computes the calibration function (linear or fourth power equation).

From experience, the calibrations of **vario MAX CNS** are stable over a very long period of time. A new calibration is recommended when components which influence the result (e.g. detector) have been replaced. A correct calibration covering the entire requested working area, is decisive for the correctness of the results. Therefore, calibrations must be performed with utmost care. A sudden drift of the daily factor does not have its cause in a change of calibration but in a suddenly appearing error (e.g. leak, etc. see chapter 9).
Observe the Operating instructions diligently!

6.4.1 Analysis of the calibration samples

Note the following instructions:

- **Calibration substances must be defined in the Standards-Menu and identified as "Calibration Sample"**
  Above the sample identification the theoretical N, C and S content is assigned to the calibration samples.

  Factory set standard substance:

Name (Substance)	**$N_{theor.}$ [%]**	**$C_{theor.}$ [%]**	**$S_{theor.}$ [%]**	**Tolerance rel. [%]**
glutamic acid	9.52	40.78	-	5.0
aspartic acid	10.52	36.10	-	5.0
urea	33.33	35.72	-	5.0
sulfadiacine	22.37	47.99	12.81	5.0

- **In the calibration mode, all calibration samples are combusted in a relatively high oxygen excess** to achieve best results.
  This reduces the lifetime of the reduction tube. A freshly filled reduction tube must, therefore, be inserted before starting calibration. Economizing here is out of place!

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- **Determine the sensitivity setting before calibration.**

- **Check and optimize instrument condition before calibration.**
  Example: leak test (see chapter 9), replacement of tube fillings (see chapter 8)

- **Suitable balances are required for weighing the calibration substances.**

sample weight	1.0 mg to 10 mg	readability	0.001 mg
	10 mg to 50 mg		0.01 mg
	above 50 mg		0.1 mg

- **Editing the calibration process can be done in the dialog Edit⇨Input.**
  The following is a calibration recommendation which covers the entire measuring range of the **vario MAX CNS** (sensitivity setting: **N + S sensitive**, **C <u>non sensitive</u>**). Of course, an adjustment to customer specific requirements is possible at any time. The weight can also be entered directly through the balance interface. In that case only the identifiers and the parameters must be edited.

Sample type and number	Weight	Name	O_2 threshold	Peak Max	Detailed Parameter	Parameter input
2 x Aspartic acid	300 mg	Run-in	-	20	120 s / 150 mL	150
2 x Aspartic acid	10 mg	Run-in	-	20	60 s / 50 mL	50 short
2 x Aspartic acid	1 mg	Run-in	-	20	60 s / 50 mL	50 short
<u>Aspartic acid</u>						
1-1.2-1.4-1.6-1.8-2,0 mg		Aspartic	-	20	60 s / 50 mL	50 short
2.2-2.5-3.0-5.0-7.0-10 mg		Aspartic	-	20	60 s / 50 mL	50 short
15-20-25-30 mg		Aspartic	-	20	60 s / 50 mL	50 short
35-40-50-60 mg		Aspartic	-	20	90 s / 75 mL	75
75-100-150 mg		Aspartic	-	20	120 s / 100 mL	100
200-250 mg		Aspartic	-	20	120 s / 125 mL	125
300-350-400 mg		Aspartic	-	20	120 s / 150 mL	150
500-550 mg		Aspartic	-	20	120 s / 200 mL	200
<u>Sulfadiacine</u>						
3 x 1 mg		Run-in	-	20	60 s / 50 mL	50 short
0.5 - 0.5 – 1 – 2 – 3 -		Sulfadiacin	-	20	60 s / 50 mL	50 short
5 – 6 – 8 – 10 – 12 – 15 –		e	-	20	60 s / 50 mL	50 short
20 – 25		Sulfadiacin	-	20	60 s / 50 mL	50 short
30 – 35 – 40 – 45		e	-	20	60 s / 50 mL	75 short
50 – 60 – 80		Sulfadiacin	-	20	120 s / 75 mL	75
100 – 120 – 140		e	-	20	120 s / 100 mL	100
		Sulfadiacin e				
		Sulfadiacin e				
		Sulfadiacin e				

- **Now start calibration run by clicking the menu item System⇨Auto**.

- **After the analysis of all calibration samples the original values should be saved** (with **File⇨Save**).

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6.4.2 Calibration evaluation shown with nitrogen as an example

a) Curve computation

- When all calibration samples have been processed, start the computation of the calibration curve with the function **Math.⇨Calibrate**. At least four calibration samples are required (see chapter 6.4.1, item 1).

- Now the dialog **Configure Calibration** appears. It allows to configure a coefficient computation for each element. It is possible to divide the complete calibration range into one or two ranges. When dividing in two, "range 1" is the lower range and "range 2" is the upper range. The one range setting is displayed by "area 2", "area 1" remains unused. Furthermore, the amount of the polynom order (1…3 for the lower, 1…4 for the upper and/or complete range) can be set for each range. (For further information see **Help⇨Index⇨Calibration**)

Mode of calculation	**Form**	**Remarks**
linear (polynomial 1st order)	$y = a + bx$	Only recommended if the fluctuations in the $N_{abs.}$-content of the samples are very small (i.e. weight and N-content of the samples are always equal). Otherwise only applicable in the low concentration range (0.1 to 0.5 %N).
non-linear (4th order polynomial)	$y = a + bx + cx^2 + dx^3 + ex^4$	Usual evaluation for the entire range ($N_{abs.}$)
2 ranges	low range = ploynomial higher range = polynomial	If high accuracy is also required for very low measuring values ($N_{abs.}$ = low) the working range is divided. **A factor correction must then be performed in both ranges!**

- When the type of computation has been selected, click on [**Compute N**]. Depending on the selected range setting the dialog **Define Lower and Upper Polynomial Range** or **Calibration** appears. (For more information see **Help⇨Index⇨Dialog Overview**).

Since the non-linear polynomial calibration (one range) is sufficient for most applications, we will discuss only this type of calculation.

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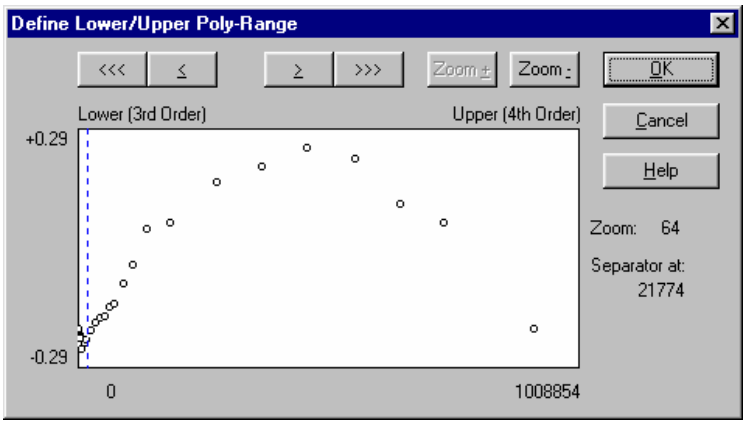

lustration 6.2: Lower/upper range definition dialog

b) Evaluating the curve adjustment

- After computation has been carried out, a dialog consisting of coordinate axis with calibration points and the calibration curve is displayed on the screen. (see illustration 6.2). In this graph the individual calibration points are shown as circles and the curve is shown as a line. The calibration sample with the lowest index is marked at the beginning with a cursor cross.
- This graphic display can be changed in such a way that an optical control of the curve adjustment is possible. This can be done through the functions [**Zoom** +], [**Zoom -**] and [**Center**].
- Through functions [**Prev**] and [**Next**] any sample can be marked with the cursor so that its position to the curve as well as the respective sample information is displayed.

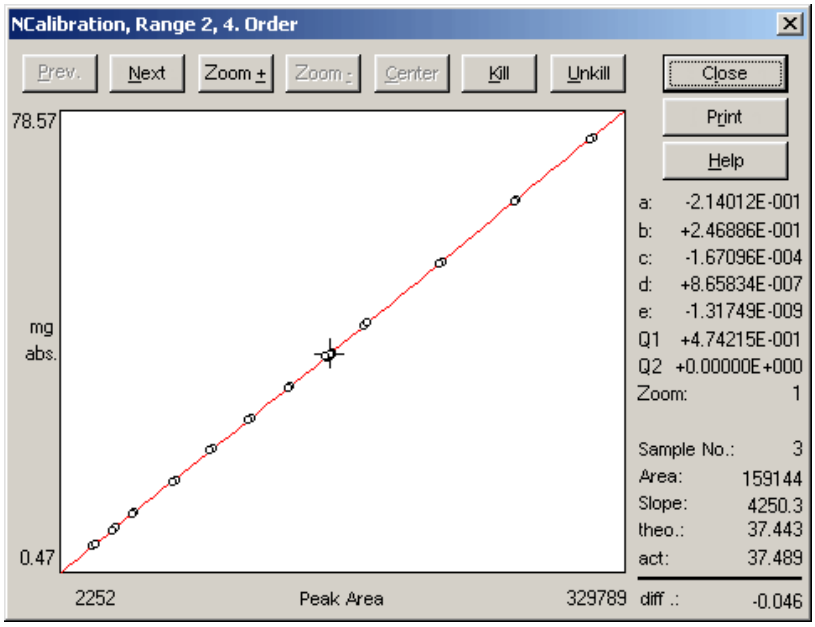

Illustration 6.3: calibration graph

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Sample-Information	**Description**
Sample No.	shows the analysis number (running No.) of the marked sample (with cursor);
Area	integral value of the marked sample
Slope	slope of the curve at the calibration point (marked sample) [integr./mg]
theo.	theoretical N-absolute content of the marked sample [mg]
act.	N-absolute content of the marked sample computed with actual curve [mg]
diff.	difference between "theo" and "act"
Curve-Information	**Description**
a....e	coefficients of the computed polynom.
Zoom	shows the magnified graph (zoom factor).
Q2 (last)	Quality measure for the degree of adaptation of the last curve computation. Serves for comparison with Q1, to check, for instance, the effectiveness of sample elimination.
Q1 (actual)	Quality measure of the degree of adaptation of the actual curve computation. If the computation is linear, **Q is the correlation coefficient (Optimum = 1).If the computation is polynomial, Q is the sum of all error squares (Optimum = 0).** **Achievable in practise: lower section 1 – 3** **upper section < 1**

- The displayed sample information and the quality (Q1) of the curve information provide information for optimizing the curve adaptation.
  For polynom computation the following is valid:
  Obtainable quality values are usually between 1 and 3 (optimum = 0).
  The difference (between theoretical and actual) of the individual samples should be as low as possible.
  The slope of the curve can be used to judge so-called "outliers". The dependence of the integrals on the absolute N-content is a constant function, so that "outliers" will drop out of a defined course of slope which means the "*Slope*" (Integr./mg) of an outlier will be noticed.

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c) Elimination of Outliers

- Illustration 6.4 shows a marked sample, which can be recognized as outlier visibly as well as through the sample information "*Slope*" and "*Diff*".

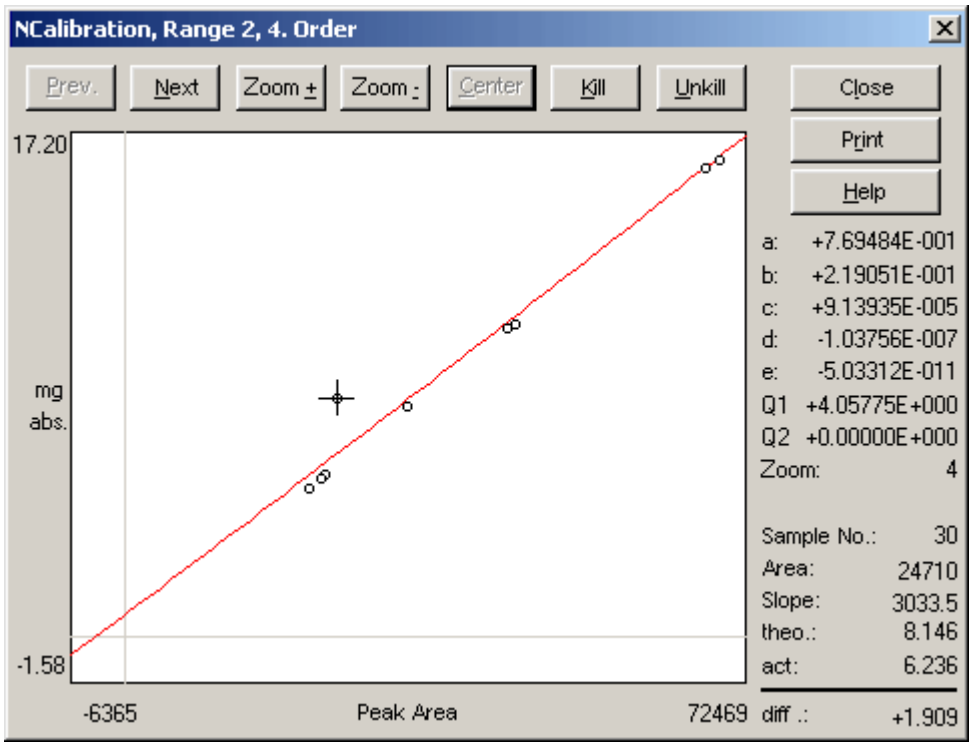

Illustration 6.4: calibration graph with marked outlier

- These marked outliers can be eliminated through the function [**Kill**]. Thereafter the curve is computed again. The effect of the elimination can be evaluated through the new quality (Q1). The eliminated samples are shown as light grey circles and can be reactivated through the function [**Unkill**] if necessary.

d) Closing

- By clicking on [**Close**], the calibration dialog will be closed. Now the dialog shows that calibration coefficients have been computed. With [**OK**] this computation can be accepted. If another mode of computation is desired the calibration data can be processed again but the coefficients of the previous computation that have not been saved will be lost.
- After clicking on [**OK**], the dialog with newly computed **calibration coefficients** appears (see illustration 6.4).
- Through the function [**OK**], the coefficients will be accepted as the actual calibration. Through the function [**Cancel**], the dialog is closed and the coefficients are rejected (for further information see online-help **Help⇨Index⇨Dialog**).

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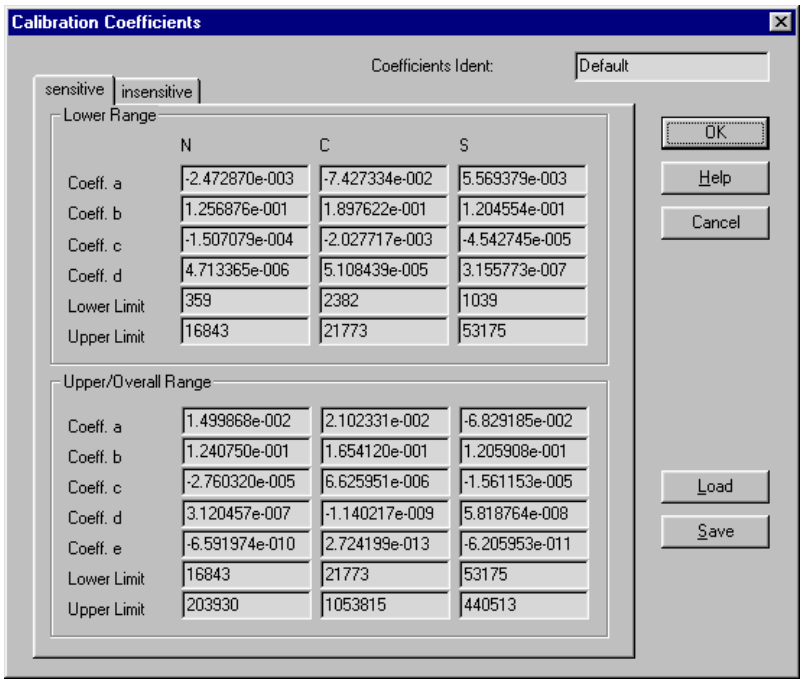

Illustration 6.5: dialog Calibration Coefficients

6.5 Shutdowns during analysis breaks

6.5.1 Short breaks

During analysis breaks for one night or several days (2 - 5 days), the **vario MAX CNS** and the PC remain in a state of readiness. Only the following steps should be taken:

a) Manual

- Reduce temperature of the combustion tube to 900°C [**Options⇨Parameters**]
  Note: Changing the instrument parameters can only be done with the corresponding access rights.
- Turn off He supply. (O_2-supply should remain open for flushing purposes.)
- Turn down screen lighting.

b) Through the software (see also Online-Help)

- Click on **Options⇨Sleep-/Wake-Up-Function**.
  In this dialog (see illustration 6.5) it is possible to select the shut-down conditions.

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- Select *"Activate Gas Reduction*"
- Select *"Activate Sleep Temperature*" and input 900 into the corresponding Edit Box.

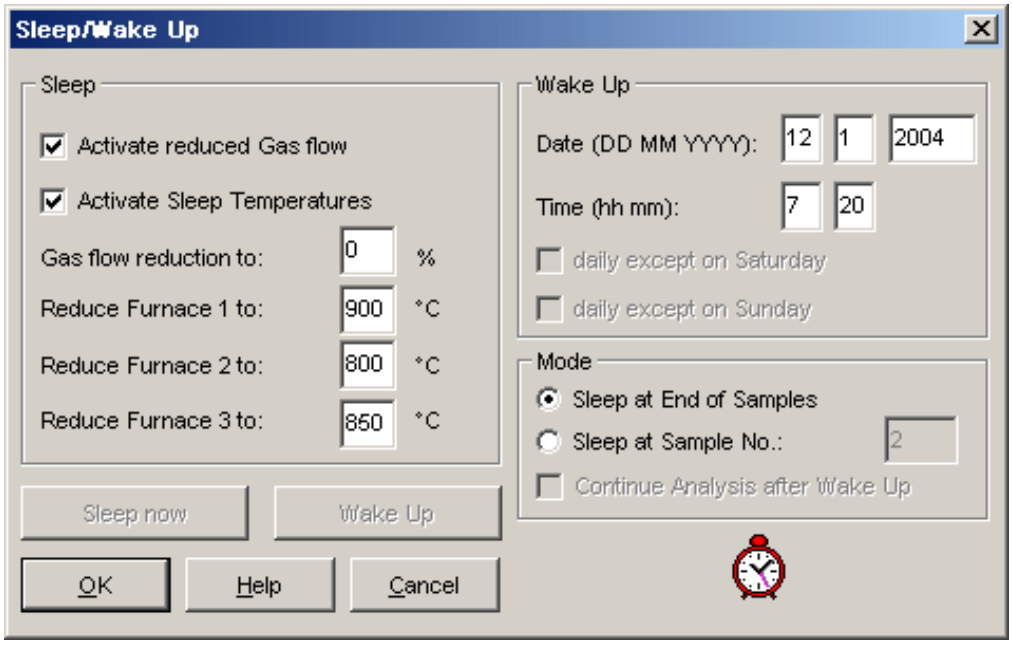

Illustration 6.6: dialog Sleep-Wake-Up-Function

- Ways to activate the **Sleep**-Function:

[**Sleep Now**]	starts the shut-down conditions immediately
"*Sleep after End of Samples*"	The **vario MAX CNS is** brought into shut-down condition after the last sample has been processed.
"*Sleep at Sample No.*"	The **vario MAX CNS** is brought into shut-down condition before the respective sample is processed.

- Ways to activate the **Wake-Up**-Function:

[**Wake-Up**]	Heats up the **vario MAX CNS** to the appropriate operation temperature and flushes the instrument with gas.
"*Wake-Up Time*"	With this function a *"Time"* can be defined at which on a certain "*Date*", "*daily*" or on weekdays Wake-Up is performed. "*Continue Analysis after Wake-Up* " permits the automatic analysis start, provided there are unprocessed samples in the data memory.

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6.5.2 Longer shutdowns

Note: To transport the **vario MAX CNS,** lock the moveable 3-tube-furnace with the supplied transport safety guard (see chapter 5.3 and illustration 5.5)

For longer shutdowns lasting several days or weeks, proceed as follows:

- Cool down all heaters and wait until the temperatures are lower than 100°C (approx. 2 hours). For this purpose set nominal temperatures to 0°C **Options⇨Parameters**.
  Note: Changing the instrument parameters can only be done with the corresponding access rights.

- Exit software by clicking **File⇨Exit**.
- Turn off gas supplies (He and O_2)
- Turn off **vario MAX CNS** and peripheral instruments.

Note: To restart operation of the **vario MAX CNS** proceed in reverse order (see also chapter 5.3.4).

If operation is re-started (see also chapter 5 and 8), tube fillings must be checked respectively renewed.

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7. Software

Since the operating software of the vario MAX CNS is self-explanatory and contains an extensive online help program, we have mentioned only several commands and menus. For further information regarding software or instrument control, please read the notes in the PC-online help program **Help⇨Index** .

7.1 Installation and Update

7.1.1 . Installation of the software

- For the installation of the software insert the furnished CD into the CD-ROM drive
- The CD will start automatically.
- If the CD does not run automatically a start with **CD-START.EXE** will be possible.
- Choose the language.
- Choose **Software Installation** in order to start the setup program.
- Follow the instructions of the appearing installation wizard.

7.1.2 Software update

- Proceed as described under item 7.1.1.

7.1.3 Configuration of the balance

The parameters of the serial interface of a connected balance must match the parameters which are set in the setup-program. The setup-program sets the parameters as follows:

2400	Baud
7	Databit
1	Stopbit
uneven	Parity

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7.2 Main menu of the software

The program is started by clicking on the **vario MAX CNS** icon. Depending on the condition of the initialization files, it will take a while until the main menu appears. If the program finds a connected analyzer, a second window appears with the information that the modules are being initialized.

After program start the main menu looks like illustration 7.1.

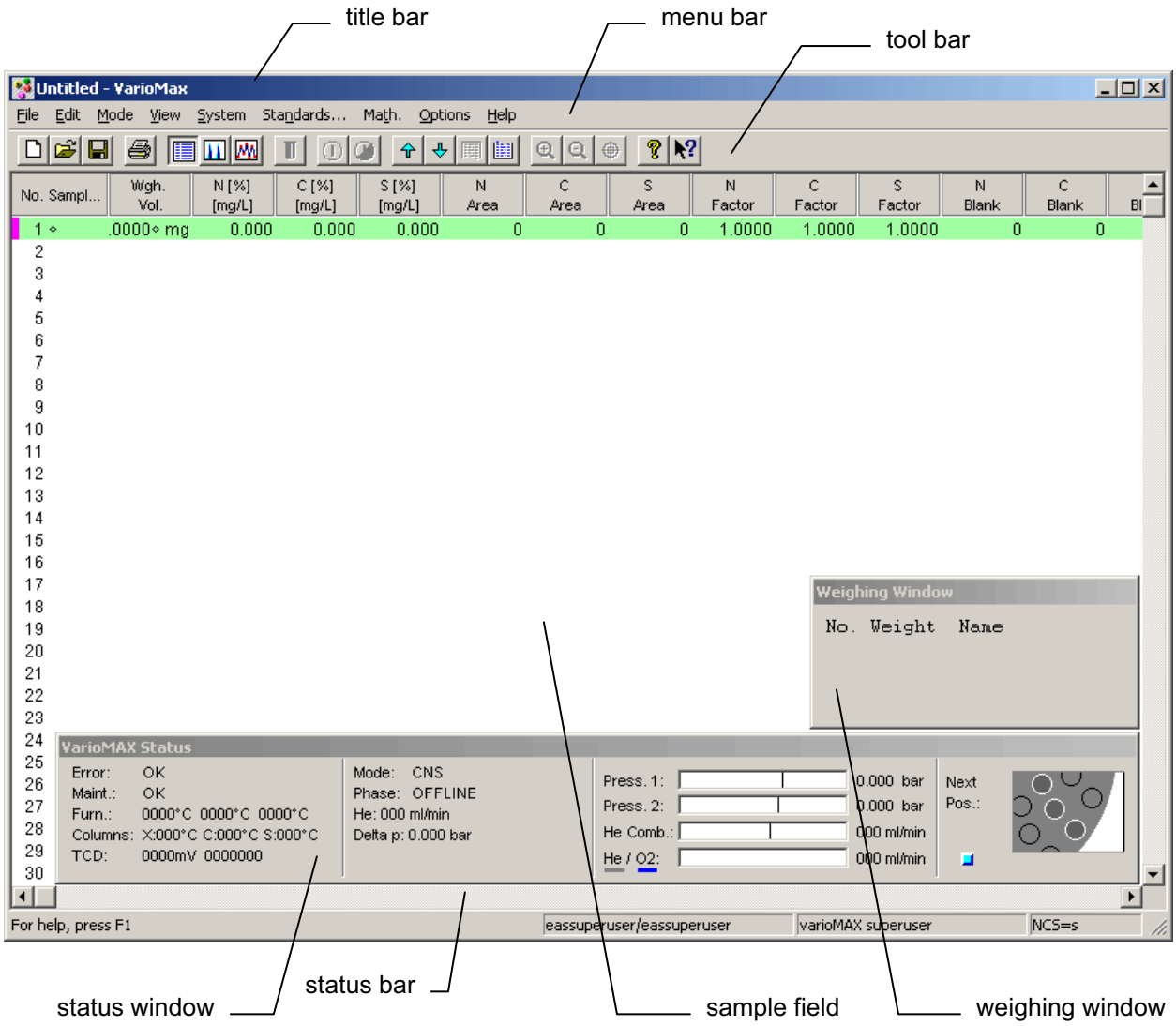

Illustration 7.1: main menu of the vario MAX CNS-software

7.2.1 Title bar

The name of the document of the current process appears in the *title bar*. For a new document the document name is *"untitled"*. The title bar also contains the usual buttons for opening the system menu and for minimizing and maximizing the application window.

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7.2.2 Menu bar

The menu bar shows the top level of the menu structure. The following is a short description of the individual menus:

File⇨New	Creates a new document, the database will be cleared. The name of the new document is set to *Untitled.*
File⇨Open	Opens an existing document. A dialog for selecting the document to be opened appears.
File⇨Save	Actualizes the currently active document. If the document name is *Untitled*, the dialog "**Save As"** will appear.
File⇨Save As	Stores the currently active document under a different name. A dialog appears where an already existing document can be selected or a new document name can be entered.
File⇨Send AQS Data	Sends sample data to the AQS program running in the background for further processing.
File⇨Send LIMS-Data	transmits the database's data of the samples to a overriding LIM-system. The mode of transmission can be selected in the dialog **Options⇨LIMS**.
File⇨Graphics	opens the graphic information of a sample. After a peak graph has been opened a graphical screen representation is possible through **View⇨Graphics** (see chapter 6.2.4).
File⇨Print	creates an analysis report of the currently active document. In an appearing dialog, the operator can perform certain functions as for instance enter a head line, give the document with the current parameters or calibration coefficients first priority etc.
File⇨Protocol Setup	The same dialog appears as described under **File⇨Print** in which printing options can be selected without the actual printing.
File⇨Print Preview	displays the analysis report on the screen.
File⇨Printer Setup	opens a dialog in which the printing options can be selected.

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File⇨Export opens a dialog in which file name, file type and path can be determined. The currently active document will be exported.

File⇨Import opens a dialog in which the required file can be selected. Loads the selected file.

Note: The file to be imported had to be generated either by the vario MAX software via **File⇨Export** or via the balance program "balance".

File⇨User Log On opens a dialog in which one can register as user.

File⇨Exit leaves the software. The menu command is not available as long as an analysis is ongoing.

Edit⇨Pointer opens a dialog in which the current sample and the current weight can be defined. The current sample is the one which is to be processed next (sleep condition) or current (during analysis) respectively. The current weigh-in is the sample which the data from the connected balance are assigned to next.

File⇨Swap enables to swap two samples. Of course, swapping takes place only in the database. Swapping of the original samples in the carousel must be carried out by hand.

File⇨Input opens an application window where sample data can be entered or modified.

Edit⇨Change allows additional changes of already analyzed samples. A dialog appears in which the number of the sample to be changed can be entered. Then, an entry dialog appears where the changes can be done.

Edit⇨Clear Tags removes the markings of all tagged samples.

Mode⇨Analysis Mode opens a dialog where mode of operation can be defined. Changing the operation mode deletes the sample memory automatically and causes an initializing of the module.

During a mode conversion make sure that the tube fillings match the respective operating temperatures and do not overheat. **Overheated tube fillings can cause the tubes to melt. This melt may run into the inside of the furnace and destroy it.**

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Mode⇨Sensitivity	allows the sensitivity setting of the measuring signal control.
View⇨Text	switches the sample field to the regular text display. The sample data of all samples are displayed (see chapter 7.2.4).
View⇨Statistics	switches the sample field to statistics display. The statistical values for each sample group is displayed (see chapter 7.2.4).
View⇨Graphics	switches the sample field to graphics display. Different analysis courses are displayed as a graph. Usually the course of the current sample is displayed. With the function **File⇨Peak Graphic** it is possible during standby mode to display the course of any sample (see chapter 7.2.4).
View⇨Weight	flashes a window in or out (alternating) which shows the weights of the last four samples. This window can be moved to any position on the screen.
View⇨Status Window	flashes the Status Window in and out (alternating).
View⇨Tool bar	flashes the tool bar in or out (alternating).
View⇨Status Field	flashes the Status field in and out (alternating).
View⇨Previous	If the text display is activated, this function scrolls the display field back to the previous sample line. If the graphic display is activated and the peak graphic is open this function changes to the peak graphic of the previous sample.
View⇨Next	If text display is activated, this function scrolls the display field upwards to the next sample line. If the graphic display is activated and the peak graphic is open this function changes to the peak graphic of the next sample.
View⇨Pos 1	If the text display is activated, this function moves the display field to the first position of the database.
View⇨End	If the text display is activated, this function moves the display field to the last position of the database.

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View⇨Zoom In

If the graphic display is activated the enlarging factor of the graphic course can be doubled. Upon clicking on this function the mouse arrow changes into a magnifying glass. If it is placed inside the graphic field and the left mouse key is clicked again, the graphic course is enlarged by the factor two. The pixel at which the left mouse key was clicked moves in the center of the graphic field. It is possible to enlarge up to a zoom factor of 64.

View⇨Zoom Out

If the graphic display is activated, the enlarging factor is cut in half and the graphic course appears anew.

View⇨Center

At activated graphic display and a zoom factor > 1, this function moves a certain point within the graphic field into the center. The mouse arrow changes into a cross. Click again and the point is centered.

View⇨Configuration

If text display is activated, this function enables to define which sample data shall be flashed in or out. Furthermore, the input of three operator-specific formulas are possible. If the graphic display is activated, this function determines the graph that is to be shown with a certain display area.

System⇨Auto

starts a continuous operation. Samples will be analyzed until there are no more weights left. The activated sleep/wake-up function can also stop continuous operation at a particularly defined sample.

System⇨Single

starts a single analysis or terminates a continuos operation.

System⇨Stop

cancels, if possible, a started analysis.

Note: An immediate stop is not always possible, because the **vario MAX CNS** would become contaminated through not combusted samples, which would effect the following tests negatively. **This function should only be carried out before a carousel movement and after completed O_2-dosing.**

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System⇨Feeding Position	moves the carrousel to the sample feeding position. Hole positions which are covered by the protection hood can also be filled. At the next analysis start the carrousel moves to the previous sample position and the sample sequence is being kept the same.
System⇨Normal Position	turns the carrousel from the feeding position to its regular position.
System⇨Module Status	shows a status field, displaying the current state of the individual modules.
System⇨Outputs Status	shows a status field, displaying the current state of the outlet lines for valves, fan, etc.
Standards	opens a dialog for defining standard samples regarding their identification as well as their theoretical contents. Furthermore, it will be determined whether data are calibration or daily factor data. (A change can also be done after the measurement).
Math.⇨Calibrate	performs instrument calibration (see chapter 6.4f). Different dialogs appear for determining the mode of computation, possible determination of the lower and upper calibration areas, visual judgement of the calibration graphs, possible elimination of outlier samples as well as saving the calibration coefficients.
Math.⇨Statistics	Statistical computation (see chapter 6.2.5). Through the appearing dialog the operator forms the respective samples into groups. The program then calculates the mean value for each group and the standard deviation.
Math.⇨Factor	computes the daily factors. Computation is carried out as selected through function **Math⇨Setup**.
Math.⇨Computation Settings	opens a dialog for setting the mode of factor computation and various initialization values.
Math.⇨Coefficients	opens a dialog in which the user can enter calibration coefficients, modify, load or save them (see illustration 6.2).
Math.⇨Recalculate	recalculates the whole content of the database.

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Options⇨Parameters opens a dialog for entering instrument parameters, modify, load or save them (see chapter 5.3.5).
Note: Changing the instrument parameters is only possible with the corresponding access rights.

Options⇨Method allows creating and/or changing the O_2 dosing parameters. The comb.parameter menu is protected by a password.
Note: Changing the method parameters is only possible with the corresponding access rights.

Options⇨Sleep/Wake-Up opens a dialog for activating or modifying the **Sleep/Wake-Up functions** (see chapter 6.3)

Options⇨LIMS opens a dialog for determining how the LIMS transmission is to be carried out. Transmission to an overriding system can either be carried out through an available serial interface or through copying the file into a target path of a computer within the network. In this dialog you can also define which components are contained in one file.

Options⇨Maintenance opens a dialog for defining up to 10 maintenance activities (see chapter 9).
Important: The maximum number of samples for the crucible container is 120. It cannot be altered. If the meter reaches number 120 the instruments stops (danger by crucible jam). The user has to make sure that the crucible container will be emptied in due time and that the meter will be set back to zero.

System⇨Acoustic Signals allows fading in and out of various acoustic signals, e.g. audio warning of found errors, transfer of weighing data, etc.

Options⇨ErrorIndex opens a dialog which lists all possible errors of the measuring, I/O, feeding and combustion module. By entering the appropriate error code its meaning is displayed.

Options⇨Error Buffer opens a dialog which lists the errors in chronological order. Up to 50 errors can be stored, thereafter the oldest error is deleted. Double clicking on an error message lets the dialog **ErrorIndex** appear and displays the respective explanation.

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Options⇨Error Handling opens a dialog in which the operator can activate the "Standard Tolerance Control" for defining how many samples in succession can have non-critical error before the analysis series is stopped.

Note: the analysis is always stopped if critical error occur

Options⇨System Test various system components (valves, etc.) can be checked for operability with this command.

Options⇨Sample Feeder allows manual moving of the sample to a desired position where the measuring series can be continued.

Options⇨Configuration serves for editing configuration data.

Caution: Improper changes of the configuration data may lead to severe consequences for the proper operation of the system and even to destruction of system components!

Options⇨Backup serves for configuration and performance of data backup.

Options⇨Clean deletes all sample data of the database files. The configuration data remain unchanged.

Options⇨Restore reads in again the content of a formerly filed database file.

Options⇨Miscellaneous⇨ Replace Gripper cuts off the carrier gas, reduces pressure and permits changing of different spare parts. When the dialog is ended, the carrier gas is turned on again and a gas pre-run will be performed before the system is ready for the next analysis.

Options⇨Miscellaneous⇨ Replace Comb. or Post Comb. Tube cuts off the carrier gas, reduces pressure and permits changing of different spare parts. When the dialog is ended, the carrier gas is turned on again and a gas pre-run will be performed before the system is ready for the next analysis.

Options⇨Miscellaneous⇨ Replace Reduc. or Drying Tube cuts off the carrier gas, reduces pressure and permits changing of different spare parts. When the dialog is ended, the carrier gas is turned on again and a gas pre-run will be performed before the system is ready for the next analysis.

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Options⇨Miscellaneous⇨CO_2 column permits heating the CO_2-adsorption column after a change.

Options⇨Miscellaneous⇨SO_2 column permits heating the SO_2-adsorption column after a change.

Options⇨Miscellaneous⇨Rough Leak Check This function performs a rough leak test of the analysis system.

Options⇨Miscellaneous⇨Fine Leak Check This function performs a fine leak test of the analysis system.

Options⇨Miscellaneous⇨Feeder Adjustment allows the adjustment of the reference values of the feeding system.

Help⇨Index activates the help program which provides assistance through the software.

Help⇨Elementar in the Web opens the internet homepage of Elementar Analysensysteme. This command is only valid if the PC is connected to the internet.

Help⇨Registration shows a dialog with the current registration data (serial number, software ident. number, etc.).

Help⇨vario MAX opens a dialog which displays the different version numbers of the software and modules, the serial number of the instrument as well as the Copyright-remarks.

7.2.3 Tool bar

The tool bar permits access to the most important menu points without going through the menu levels. The following illustration shows the tool bar and the menu points that can be reached.

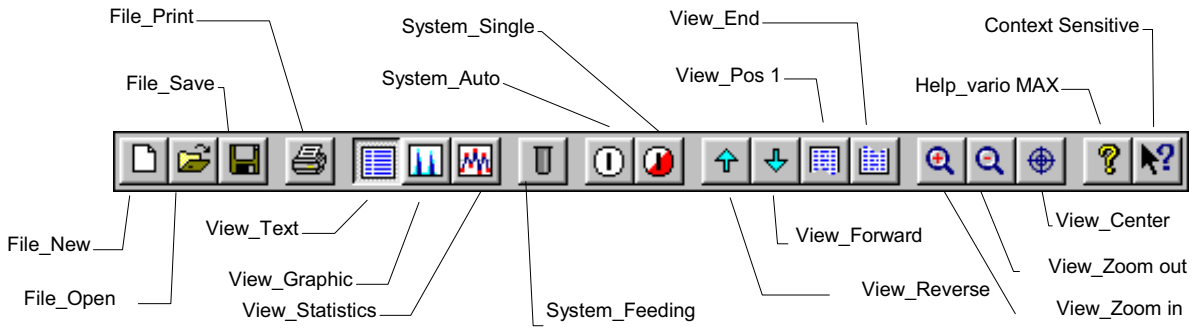

Illustration 7.2: tool bar

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7.2.4 Sample field

The display of the *sample field* depends on the selected display mode (**Menu View**). In the **text**-mode it appears in the *Text Display* with sample specific input or measuring data respectively.
In the **graphic**-mode a coordinates field appears (graphic display) where the timely course of an analysis is displayed.
In the **statistics**-mode (*statistics display*) the statistical data of defined sample groups are listed (see chapter 6.2.5).

7.2.4.1 Text display

In the **text**-mode the sample data is listed line-by-line (see illustration 7.3). With the scroll bar at the right side and the lower edge of the sample field, the operator can move the desired segment as necessary. Listing the data in columns can, as needed, be performed through the menu **View⇨Configuration.** The two left columns, however, "*No.*" and "*Sample name*" cannot be moved.

7.2.4.2 Graphic display

In the **graphic** mode, the coordinates field where the graphic course of an analysis is shown is integrated in the sample field (see illustration 7.3). The right and lower edges of the coordinates field are dimensioned according to the existing settings. In the graphic display, the sample field has no scrolling bars since the entire display is shown completely through standard VGA resolution (640x480 pixels).

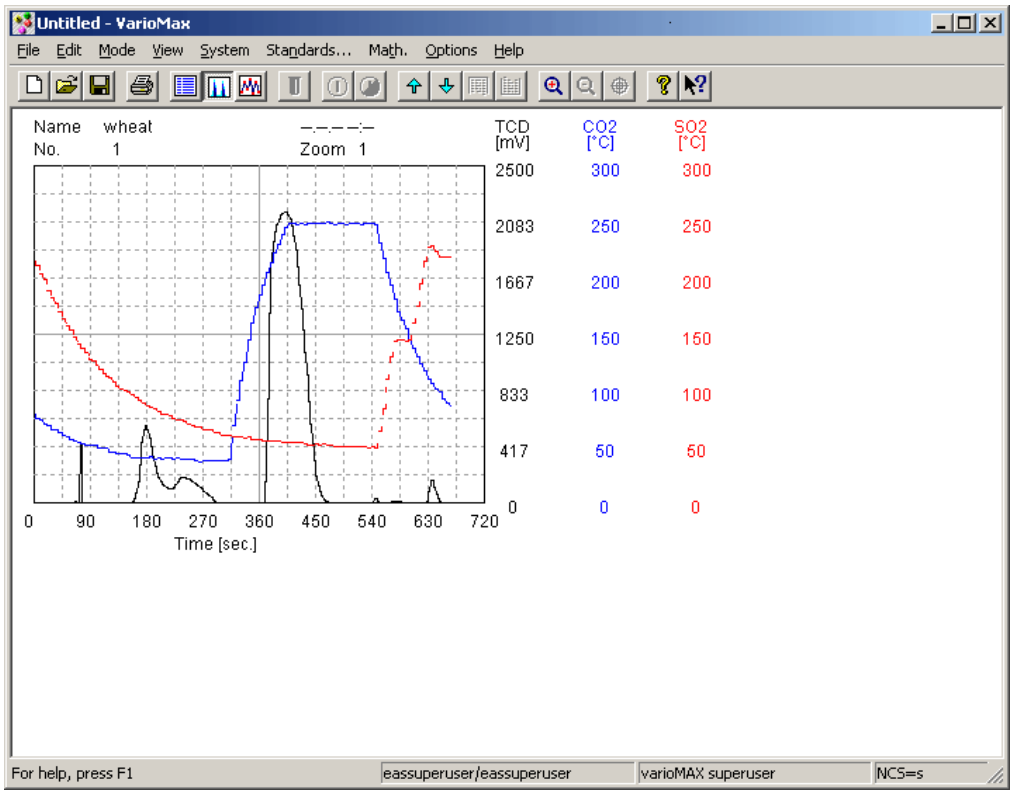

Illustration 7.3: main window with graphic display

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7.2.4.3 Statistic display

In the **statistic** mode, the statistical data of pre-selected sample groups is listed line by line (see illustration 7.4). Through the scrolling bar at the right and lower edges of the sample field, the operator can position the desired segment as needed. A configuration of the column sequence is not possible. For the definition of sample groups see chapter 6.2.5.

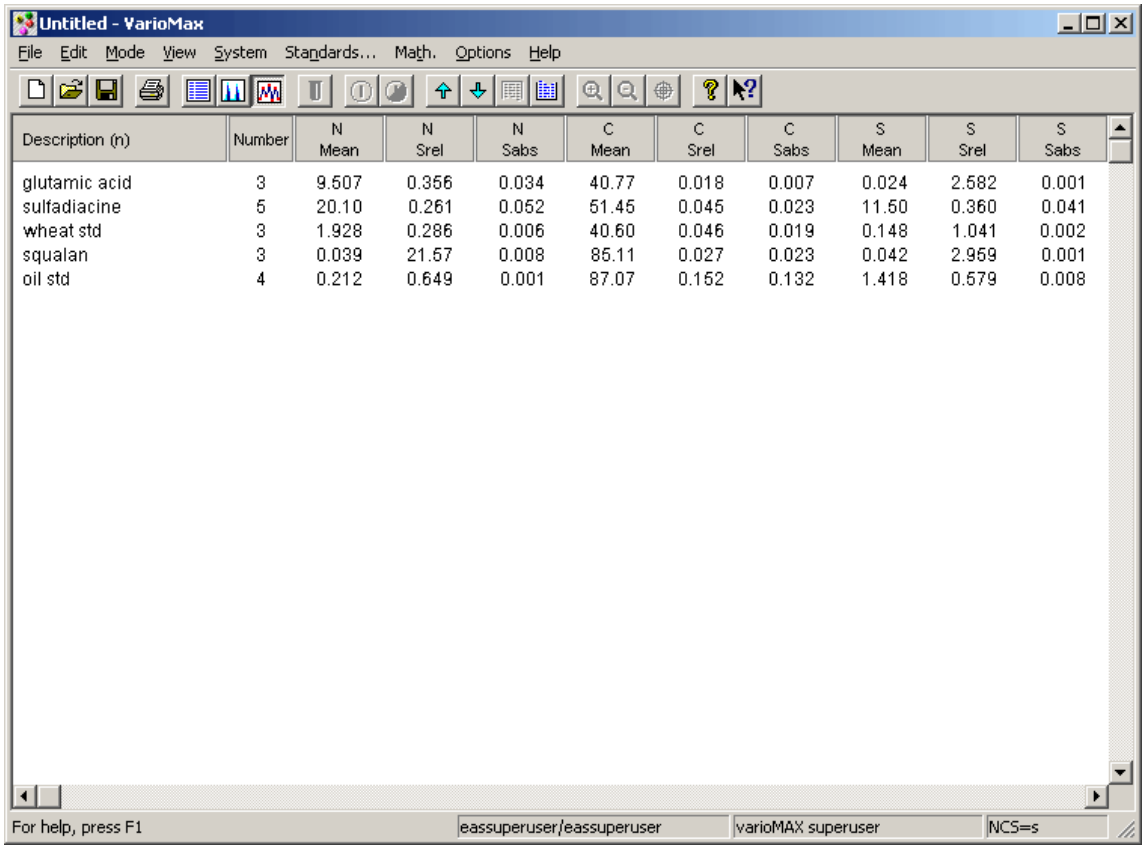

Illustration 7.4: main window with statistic display

7.2.5 Weighing window

The weighing window displays the actual sample weight and the 3 previous sample weights. Fading-in this window can be of help if the weighing datas are being automatically transfered from the balance to the system.

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7.2.6 Status window

The *status window* gives the operator the most important information regarding the current state of the system. It consists of four status lines whereby the upper three lines are subdivided into three columns (see illustration 7.1).

Error		shows that the system recognized module error.
Maint: (here: **OK**):		shows the current state of maintenance. This display is linked to the menu **Options⇨Maintenance** and watches that the set maintenance intervals are adhered to. If "*OK*" is flashing, at least one maintenance procedure will be necessary during the analysis run. If a message is flashing, the respective interval has been reached or surpassed.
Furnaces:	[°C]	shows the current temperatures of the three-tube furnace. (Succession: Furnace 1 = Comb., Furnace 2 = Post Comb. Tube, Furnace 3 = Reduction Tube).
Columns	[°C]	shows the current temperatures of the adsorption columns.
TCD:	[mV] [integral]	shows the current measuring signal in mV and the current integral value during analysis.
Mode:		shows the current mode of operation (CNS or CN). Mode conversions are carried out through the dialog **Mode.** Note: When changing the mode of operation, the tubing scheme and the reaction tube filling must be changed also. **Tube fillings must not be overheated !!**
Phase (here: **Offline**)		"*Offline*" is shown when there is no communication with the analyzer. This is usually the case when no analyzer is connected to the PC. If there is communication with the analyzer, either sleep-state will appear if the system is sleeping or the respective process phase. N integration, C integration, S integration.
He		shows the current flow of the measuring gas (He) through the TCD in mL/min, approx. 680 mL/min.
Delta p		shows the difference between pressure 1 and pressure 2 (indication of blocking).

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Residual O_2		shows the current excess O_2 concentration in ‰. In CNS operation this value is always set to approx. 80‰.
Press.1/Press.2:	[bar]	shows the current system pressure at the pressure sensors. Pressure 1 = comb.tube inlet Pressure 2 = MFC-TCD inlet
HeComb		shows the current value of the carrier gas flow which flows into the combustion tube.
He / O_2		shows the current value of the lance flow. grey = helium flushing blue = O_2 flow during combustion
Next pos.		shows which crucible (sample) is being analyzed next.
		shows by flashing that the program communicates with the analytical instrument. Indication of program crash.

7.2.7 Status bar

Shows a help text on the left side which refers to the currently selected menu of the menu bar or tool bar. If neither a menu of the menu bar nor a menu of the tool bar has been selected, the text *"for help, press F"* appears. By depressing F1 the help system is activated.
In the center area the name of currently logged on user and the group allocation is displayed.

In the right area an information appears with which sensitivity the data processing for each element is done. "s" means sensitive, "i" means insensitive setting.

7.2.8 Recommendations for the software use

As is common through Windows®, it is possible to activate several applications at the same time. Multiple instances of the **vario MAX CNS** program is, however, not possible. If the operator tries to load another instance of this program, an error message will appear. However, it is possible to let the **vario MAX CNS** program run in the background while working on another application. In this case, please note the following:

- programs, which burden the PC drastically (regarding the CPU-time) or which have long access times to reach disk drives can cause disturbances in the program course of the **vario MAX CNS** program.

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- Furthermore, every mouse activity, enlarging or reducing the frame of an application, has the effect that as long as the mouse has picked up the frame, everything else stops. This can also cause disturbances in the program of the **vario MAX CNS**.
- Please note, that programs which (purposely or inadvertently) use the same serial port as the **vario MAX CNS** program will definitely cause a total failure of the **vario MAX CNS** system.

We, therefore, recommend the following:

Avoid simultaneous operation of several programs when using the **vario MAX CNS** program, unless you are absolutely sure that these programs do not have the uncertainties described above. Avoid also unnecessary enlarging, reducing or moving of frames and windows during an analysis.

7.3 Processing of the vario MAX data with MS-Excel

The vario MAX software allows a further processing with MS-Excel® via its export function. Proceed as follows:

1. Load the document to be exported into the sample storage of the vario MAX program by means of the menu command **File⇨Open.**

2. Invoke the menu **File⇨Export.** A dialog "Select document" appears. In the upper field the name of currently open document appears. If it is the document to be exported, confirm by clicking OK, if not, select the requested document from the list and subsequently click OK.

3. The Windows common dialog "File Save As" appears. Select the type "MS Excel-files (*.xls) under "file type". Also select the path under "Save". The recommended file name can be chosen or changed. Then click "Save".

4. Now the vario MAX program generates a MS-Excel legible file and files it in the corresponding folder.

5. Start MS-Excel and open the file generated just now. The appearing worksheet shows all sample data. Now you can fade out the columns that are not of interest for you.
   For further information on how to work with MS-Excel, please refer to the operation manual of Excel.

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8. Replacing consumables

Please note the following safety instructions!!!

- The efficiency of the **vario MAX CNS** can be guaranteed only with original consumables as well as original spare parts which can be obtained only through **E l e m e n t a r Analysensysteme GmbH** or authorized dealers. **The use of different consumables or chemicals of an unsuitable standard can lead to injuries as well as instrument damages. It can also falsify the results of the analysis. Unsuitable spares lead to instrument damages and loss of warranty**.

- All the operation dependent maintenance and exchanges times mentioned in the operating instructions depend very strongly on the respective test matrix. They are, therefore, only examples and **do not guarantee life times for consumables or wear and tear parts!**

- All tube fillings (except tungsten) must be packed down as tightly as possible to avoid air pockets. For this purpose, tap the side of the tube lightly while it is being filled with a wooden implement or a piece of cork.

- To prevent leakage around the tube plugs, apply a slight coating of high vacuum grease to the O-rings and check them for damage before fitting them in. In case too much grease was applied, C blanks may occur.
  The O-ring insert-grooves in the plug must be cleaned regularly.
  Before inserting the plugs, the sealing areas of the tubes (inner walls) should be dusted off.

- When replacing any of the heated tubes, always wear the supplied protective gloves (order No. 05 000 094). **Replacing combustion or post combustion tubes can only be carried out when the tubes are cold.**
  !!! Danger of burning !!!

- Before replacing a tube turn off the gas supply into the instrument respectively activate the function **Options ⇨Miscellaneous⇨Replace comb.tube or post com.tube** or **Options ⇨Miscellaneous⇨Replace reduction or drying tubes**.

- After a tube exchange, it is necessary to flush out the atmospheric nitrogen that entered the unit for 5 minutes with He.This is accomplished automatically through the function **Options ⇨ Miscellaneous⇨Replace comb.tube or post com.tube** or **Options ⇨ Miscellaneous⇨Replace reduction or drying tubes**..

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• After changing the tubes wait until the 3-tube furnace as well as the new reaction tubes have warmed up (approx. 10 to 15 minutes) before starting the next analysis.
• When handling chemicals, observe the safety instructions of the chemical manufacturer (see labels on bottles and safety data sheets)!

To simplify following up on the maintenance and change intervals, it is possible to define ten different replacement or maintenance functions and their due dates through the menu **Options⇨Maintenance.** Illustration 8.1 shows the **Maintenance** dialog. The preset intervals are monitored by the software through the progressive analysis count in the respective *counter*. A message is displayed in the *Status field* (see chapter 7) if there are less than 50 analysis to the preset i*nterval*. After maintenance or a tube replacement has been carried out, the *counter* must be set to zero (for further information see Online-Help).
Caution:
crucible container max. 120 samples, the instrument will stop (danger of crucible jam).

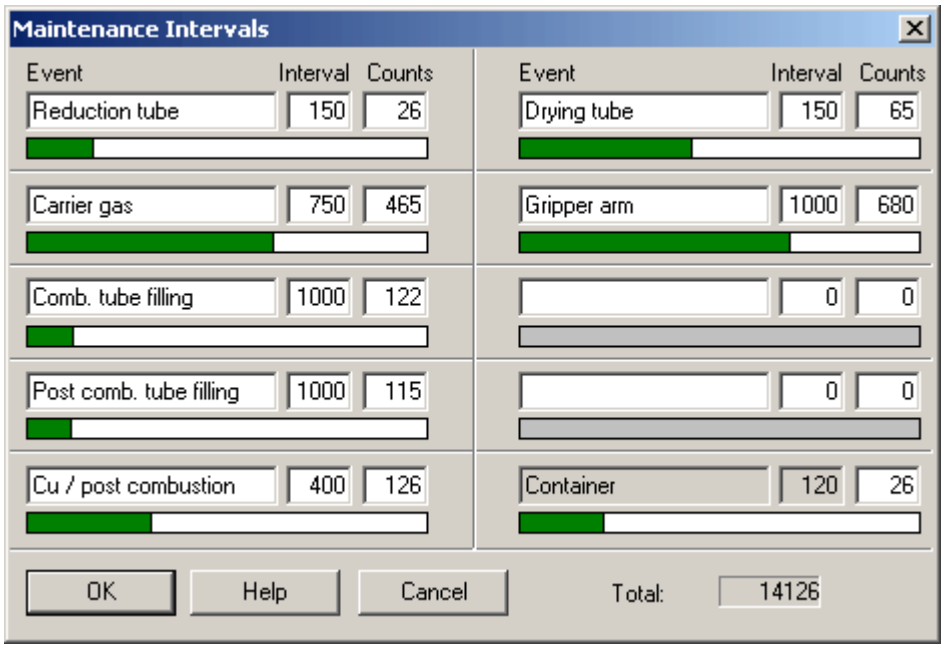

Illustration 8.1: menu Options⇨Maintenance

Before changing a tube, always turn off the carrier gas. This can be accomplished automatically through the function **Options ⇨Miscellaneous⇨Replace comb.tube or post com.tube** or **Options ⇨Miscellaneous⇨Replace reduction or drying tubes.** After the respective part has been changed and the button [**OK**] was activated, the **vario MAX CNS** is flushed for 1 minute with O_2 and for 5 minutes with He. If a tube has been replaced, wait until the new tube has reached the correct temperature.

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8.1 Filling the reaction tubes

All quartz parts have to be cleaned off from fingerprints prior to instalation into the 3-tube furnace, e.g. with acetone. Otherwise it might be possible that crystallation zones will occur which may cause premature aging of the quartz.

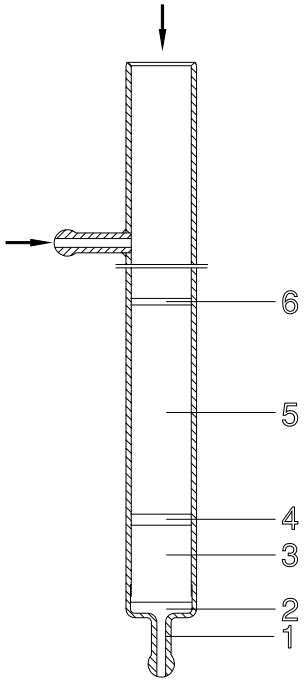
8.1.1 Combustion tube CNS operation (quartz)

Important Note: **Use the supplied measuring tube for filling the combustion tube**, to prevent filling material particles from clinging to the sides of the combustion tube. All measuring heights of the individual chemicals have to be checked since the position of the catalyst (WO_3) is determinative.

Fill the tube with quartz wool. Place the tube in the tube tray and - as described in illustration 8.2 - fill in the chemicals through the plastic measuring tube.

Caution: Prior to its use the plastic tube has to be cleaned with either a dry cloth or a piece of paper to prevent penetration of undesired foreign substances.

The cleaned sealing flange will now be plugged on.



Legend:		
↓		gas inlet
	Ident.-no:	
1	25.00-1028	combustion tube
2	03679908	quartz wool 5 mm
3	50 008 467	corundum balls 35 mm, approx. 15 g
4	03679908	quartz wool 5 mm
5	11.02-0008	WO_3 granulate 95 mm 120 g
6	50 008 467	corundum balls, 3 mm

Illustration 8.2: combustion tube, CNS operation, temperature setting 1140°C

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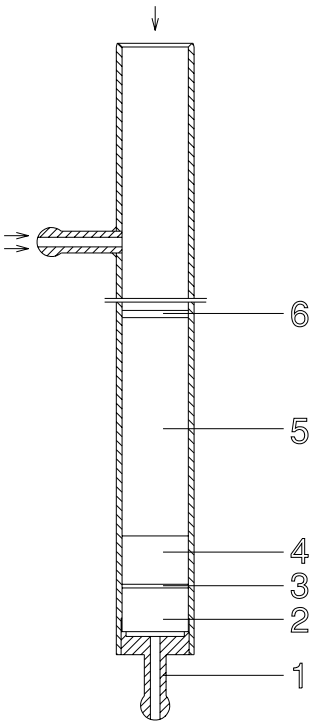
8.1.2 Combustion tube CN operation (steel)

Important Note: **Use the supplied measuring tube for filling the combustion tube**, to prevent filling material particles from clinging to the sides of the combustion tube. All measuring heights of the individual chemicals have to be checked since the position of the catalyst (CuO) is determinative.

Equip the tube with spacer ring and wire mesh, 27 mm diameter. Place the tube in the tube tray as described in illustration 8.2. Fill it with chemicals through the plastic measuring tube. At point 2 fill in copper oxide (CuO) and corundum balls from two separate bottles to get a good mixture. The cleaned sealing flange will now be plugged on.

Caution: Prior to its use the plastic tube has to be cleaned with either a dry cloth or a piece of paper to prevent penetration of undesired foreign substances.

Subsequently, the cleaned sealing flange will be plugged on.



Legend:		
↓		gas inlet
	Ident.-no:	
1	25.00-1030	combustion tube
2	25.00-1138	spacer ring 18 mm
3	50 008 449/4	wire mesh ronde, 26.8 mm diameter
4	50 008 467	corundum balls 20 mm, approx. 8 g
5	05 001 039 50 008 467	copper oxide approx. 92g mixed with approx. 20 g corundum balls, 95 mm
6	50 008 467	corundum balls, 3 mm

Illustration 8.3: combustion tube, CN operation, temperature setting 900°C

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8.1.3 Post combustion tube CNS operation (steel)

Important Note: **Use the supplied measuring tube for filling the combustion tube**, to prevent filling material particles from clinging to the sides of the combustion tube. All measuring heights of the individual chemicals have to be checked since the position of the catalyst (CuO) is determinative.

Fit the sealing plug on top with two Viton o-rings (green). Set the tube into the tube-tray and fill in the chemicals as described in illustration 8.4 . At point 2 fill in copper oxide (CuO) and Pt-catalyst from two separate bottles to get a good mixture. Then seal with upper plug.

Legend		
↑		gas inlet
	Ident. no.	
1	25.00-1048	post combustion tube
2	03679908	quartz wool 5 mm
3	50 008 467	corundum balls 40 mm, approx. 14 g
4	03679908	quartz wool 5 mm
5	05 001 039 03 002 262	CuO + Pt Kat 175 mm = 15 g Pt Kat = 195 g CuO
6	03679908	quartz wool 5 mm
7	05 000 699	copper 90 mm = 100 g
8	22 131 365	silver wool, 30 mm = 6.5 g
9	25.00-1168	plug
10	05 000 095	o-ring, 22 x 2
11	05 000 353	quad ring 7.65 x 1.78

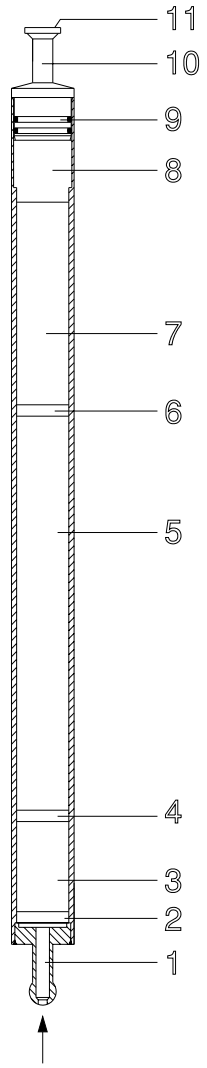

Illustration 8.4: Post combustion tube CNS operation temperature setting 800°C

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8.1.4 Post combustion tube CN operation (steel)

Important Note: **Use the supplied measuring tube for filling the combustion tube**, to prevent filling material particles from clinging to the sides of the combustion tube. All measuring heights of the individual chemicals have to be checked since the position of the catalyst (CuO) is determinative.

Fit the sealing plug on top with two Viton o-rings (green). Set the tube into the tube-tray and fill in the chemicals as described in illustration 8.5. At point 2 fill in copper oxide (CuO) and Pt-catalyst from two separate bottles to get a good mixture. Then seal with upper plug.

Legend		
↑		gas inlet
	Ident. no.	
1	25.00-1048	post combustion tube
2	12.01-1032/4	spacer ring 18 mm
3	12.00-1004/4	wire mesh ronde, 22 mm diam.
4	50 008 467	corundum balls 20 mm, approx. 7 g
5	05 001 039 03 002 262	copper oxide approx. 305 g CuO + 30 g Pt catalyst, 270 mm
6	22 131 365	silver wool, 10 g=45 mm
7	25.00-1168	plug
8	05 000 095	o-ring, 22 x 2
9	05 000 353	quad ring

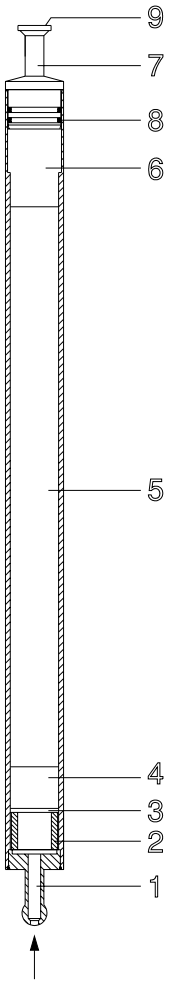

Illustration 8.5: Post combustion tube CN operation temperature setting 900°C

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8.1.5 Reduction tube CNS operation (quartz)

Finger prints must be cleaned off all quartz parts before installing them in the 3 tube furnace, for instance with acetone, otherwise crystallization areas can be created which leads to premature aging of the quartz.

Place the tube into the tube tray and fill in the quartz wool and the corundum balls. 4 zones of copper follow which are separated from each other by a quartz wool layer. As top filling there will be corundum balls and silver wool. The silver wool will be pressed with the plug.

Legende:		
↑	gas inlet	
	Ident.-no.	
1	11.00-1150	reduction tube
2	03 679 908	quartz wool
3	50 008 457	corundum balls 55 mm, approx. 19 g
4	03 679 908	quartz wool 5 mm
5	05 000 699 03 002 262	4 x copper à 50 mm = 60 g, separated each by 5 mm quartz wool
6	50 008 457	corundum balls 20 mm, approx. 7 g
7	22 131 365	silver wool, 25 mm, 14 g
8	03 654 627	o-ring, 20 x 2
9	25.20-1001	plug (Arlon)
10	05 000 353	quad-ring 7.65 x 1.78

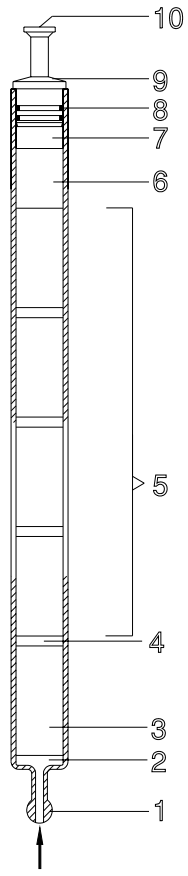

Illustration 8.6:
Reduction tube
CNS operation
Temperature setting 850°C

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8.1.6 Reduction tube CN operation (quartz)

Finger prints must be cleaned off all quartz parts before installing them in the 3 tube furnace, for instance with acetone, otherwise crystallization areas can be created which leads to premature aging of the quartz.

It is mandatory to:

- **replace the reduction tube after 180 analyses even with incomplete consumption (tube may block or crack)**
- **exactly weigh the tungsten filling per zone (□ 20.0 g), otherwise the tube may block or crack.**

Set the tube into the tube-tray and fill in the corundum balls. Illustration 8.7 shows the reduction tube with the 10 filling layers for tungsten. To provide enough room for tungsten expansion, this catalyst must be filled in portions using spacers of wire mesh. For this purpose set the forming tool on the reduction tube and insert the flat spacer into the space in the tool. Now press the spacer into the tube with the filling rod (see illustration 8.8). Now one portion (approx. 20 g) tungsten can be given through the filling tube.

Important: the tungsten has to be weighed exactly. In case of an overdose (> 20.0 g), the reduction tube may block or crack.

Repeat this procedure nine times. At the end insert one more spacer and on this spacer add one layer copper oxide (CuO approx. 50 mm) and one layer copper (Cu approx. 20 mm). Now fill in one layer of silver wool up to the upper plug.

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Legende:		
↑	gas inlet	
	Ident.-no.	
1	25.00-0017	reduction tube
2	12.01-1014/4	plug, bottom
3	03 654 627	o-ring, 20 x 2
4	12.00-1004/4	wire mesh ronde 22 mm diameter
5	50 008 467	corundum balls, 55 mm approx. 18 g
6	12.01-0045	tungsten set, complete (11 x spacers, 10 x 20 g tungsten granulate)
7	05 001 039	copper oxide fine- granular, 0.5 mm, 50 mm □ approx. 65 g
8	05 000 699	copper, wire, 20 mm approx. 25 g
9	22 131 365	silver wool, 40 mm, 10 g
10	25.00-1172	plug
11	03 654 627	o-ring, 20 x 2
12	05 000 353	quad-ring 7.65 x 1.78

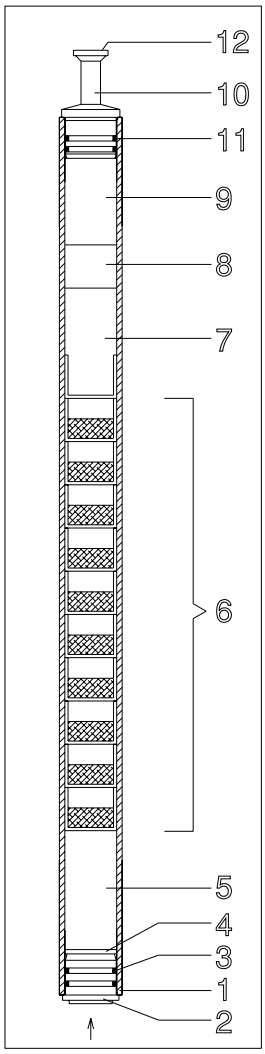

Illustration 8.7: Reduction tube

Temperature setting 830°C

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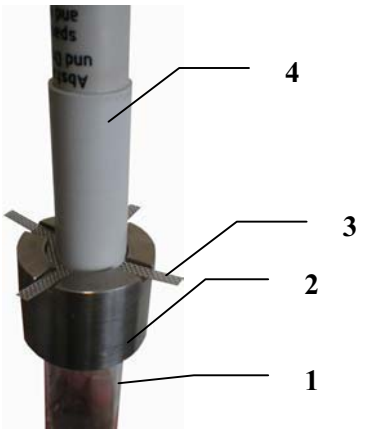

Illustration 8.8: Mounting of spacer

Legende:		
	Ident.-Nr.	
1		reduction tube
2	12.01-1029/4	insert of spacer
3		spacer
4	25.00-0095	filling and measuring tube

8.2 Replacing the tubes in the 3-tube-furnace

Changing the combustion tubes or post combustion tubes can only be performed when the tubes have cooled down.

!!! Danger of burning !!!

8.2.1 Replacement of the reduction tube, operation mode CNS

For the installation and disassembly of the tube turn off the gas supply at the unit [**Options⇨Miscellaneous⇨Replace Reduction Tube**], wait until the pressure is reduced, remove the right front door and open the right side wall.
The replacement of the reduction tube can be carried out without moving the 3-tube-furnace out of the instrument.
The procedure is as follows:

- Open the front door and side wall on the right.
- Loosen butterfly nut of the retaining clip and swing it out of the way.
- Loosen ground-in clamps on top and put tubing no. 6 aside.
- Remove thermal cover on bottom.
- Loosen ground-in ball-and-socket joint on bottom.
- Remove cover lid on top of the casing.
- Turn catcher upside down over the reduction tube (incl. plug!!!) and tighten it
- Pull out the reduction tube towards the top with the catcher, place reduction tube in the tube tray.

The installation will be done in reverse order.

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Illustration 8.9: Front view of installed reaction tubes

Illustration 8.10: Side view of installed reaction tubes

Legend:	
1	Ground-in clamp combustion tube /flushing gas inlet
2	Ground-in clamp reduction tube / Inlet
3	Ground-in clamp reduction tube / outlet
4	Ground-in clamp combustion tube / carrier gas inlet
5	Ground-in clamp post combustion tube / outlet
6	Butterfly nut for fastening the retaining clip
7	Retaining clip
8	O_2 sensor
9	Reduction tube
10	Post combustion tube
11	Combustion tube
12	sealing flange with carrier gas connection, mounted to the combustion tube

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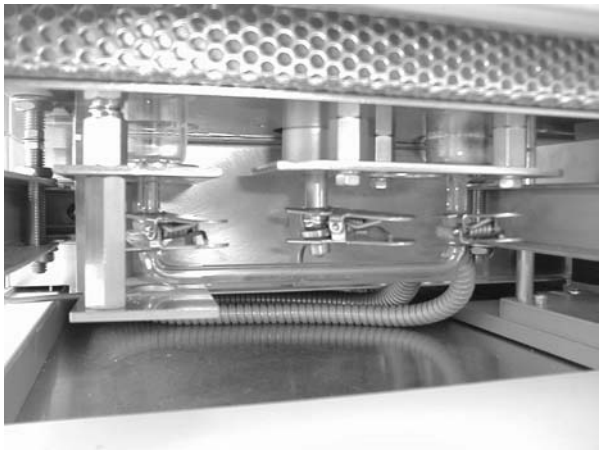

Illustration 8.11: Ground-in clamp, bottom

Illustration 8.12: Mounted catcher

8.2.2 Replacement of the reduction tube, operation mode CN

For the installation and disassembly of the tubes turn off the gas supply at the unit [**Options⇨Miscellaneous⇨Replace Reduction Tube or Drying Tubes**], wait until the pressure is reduced, remove the right front door and open the right side wall.

The replacement of the reduction tube can be carried out without moving the 3-tube-furnace out of the instrument.

The procedure is as follows:

- Open the front door and side wall on the right.
- Loosen butterfly nut of the retaining clip and swing it out of the way.
- Loosen ground-in clamps on top and put tubing no. 9 aside.
- Lift off the plug on the bottom by means of a screw driver.
- Remove cover lid on top of the casing.
- Turn catcher upside down over the reduction tube (incl. plug!!!) and tighten it
- Pull out the reduction tube towards the top with the catcher, place reduction tube in the tube tray.

The installation will be done in reverse order.

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Illustration 8.13: Front view of installed reaction tubes

Illustration 8.14: View from the right of installed reaction tubes

Legend:	
1	Ground-in clamp combustion tube /flushing gas inlet
2	Ground-in clamp reduction tube / Inlet
3	Ground-in clamp reduction tube / outlet
4	Ground-in clamp combustion tube / carrier gas inlet
5	Ground-in clamp post combustion tube / outlet
6	Butterfly nut for fastening the retaining clip
7	Retaining clip
8	O_2 sensor
9	Reduction tube
10	Post combustion tube
11	Combustion tube
12	sealing flange with carrier gas connection, mounted to the combustion tube

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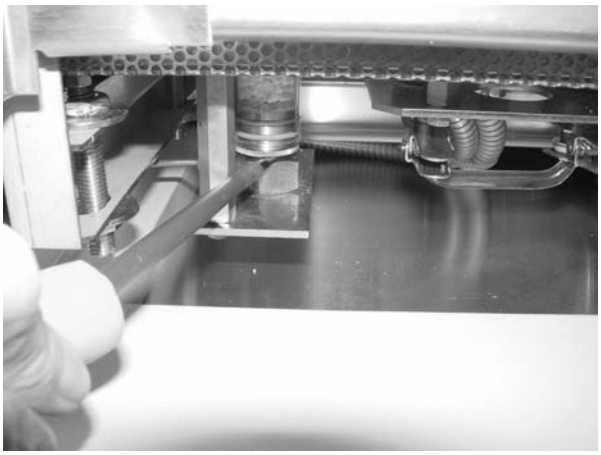

Illustration 8.15: Plug, bottom

Illustration 8.16: Mounted catcher

8.2.3 Replacement of the post combustion tube and the combustion tube

Operation mode CNS and CN

For the installation and disassembly of the tubes turn off the gas supply at the unit [**Options⇨Miscellaneous⇨Replace Combustion Tube and Post Combustion Tube**], wait until the pressure is reduced, the gripper arm has moved out, remove the right front door and open the right side wall.
The replacement of the post combustion tube and the combustion tube can only be carried out when the 3-tube-furnace is moved out of the instrument.

The procedure is as follows:

- Loosen all ground-in balls at the manifold, left (see illustration 8.11).
- Open door of the cover hood, loosen the two fastening screws (knurled head screws) of the combustion tube flange with some turnings and press towards the bottom (a gap between flange and instrument casing will become visible). Keep on loosening the two knurled head screws and remove them.
- Now the 3-tube-furnace can be pulled out of the instrument.
- For replacing the tubes, now the quartz connection bridge on the bottom and the tubings located at the plug on top have to be removed.
- For operation mode CNS also the ground-in ball-and-socket connection at the bottom of the post combustion tube has to be loosened.
- The tubes can now be pulled out of the furnace towards the top. Subsequently, the sealing flange of the combustion tube has to be removed.

The installation will be done in reverse order.

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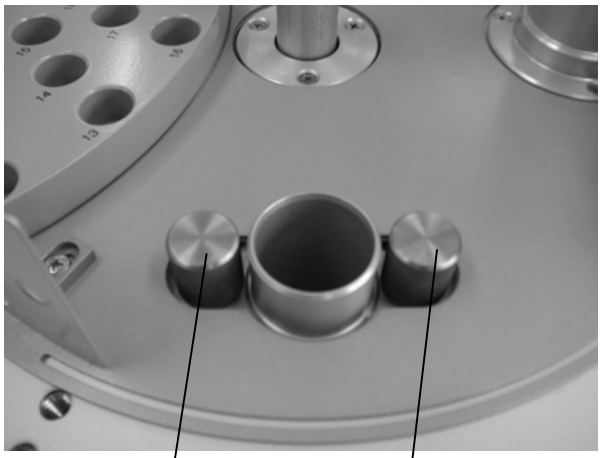

Illustration 8.17: Knurled head screws

Illustration 8.18: "Gap" after loosening the knurled head screws

8.3 Replacement of the gripper arm

Changing the gripper arm can only be performed when it has cooled down.

!!! Danger of burning !!!

For the exchange of the gripper arm turn off the gas supply at the unit [**Options⇨Miscellaneous⇨Replace Gripper Arm**], wait until the pressure is reduced and the gripper arm has moved out. The gripper swings a bit to the right and stops.

For safety reasons, please wait for 15 minutes until the gripper has cooled down !

Subsequently, open the plexiglass slide door by releasing the lock.

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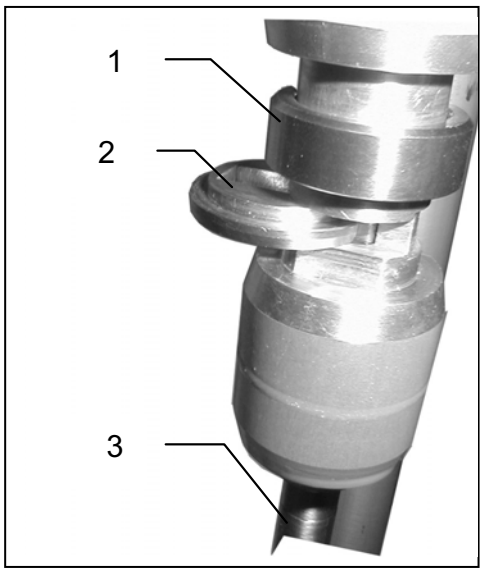
For removal of the gripper, please proceed as follows:

- Pull the safety ring up.
- Remove safety disc by pressing it from the side.
- Pull gripper toward bottom.



Legende:	
1	safety ring
2	safety disk
3	gripper

Illustration 8.19: Gripper

The installation of a new gripper will be done in reverse order.

Prior to this, the following steps are necessary:

- Mount 3 o-rings 7x1.5 red (03 002 395) into the nuts located at the gripper
- Grease lightly o-rings with silicon grease.

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8.4 Drying tubes

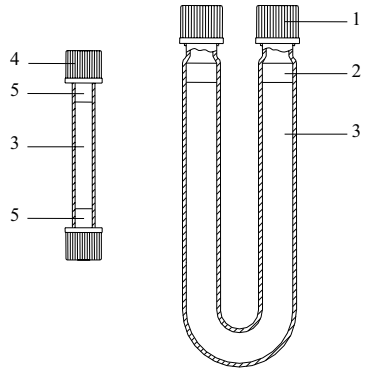
When using Sicapent® (Phosphorpentoxide) and NaOH, it is absolutely necessary to wear safety goggles and protective goggles
!!!Danger of Causticization!!!

Please note:

Operation mode CN and CNS: installation of three drying tubes ⇨ left, center, right

8.4.1 Drying tube filling + reference drying tube

The U-shaped drying tube is filled with Sicapent® and sealed off on both sides with 2 filters. The straight reference drying tube is filled with Sicapent and closed on both sides with a filter. (see illustration 8.20). Please check if there is resulting a free space between filter and filling material while operating the U-shaped tubes (may be possible due to compaction of the filling material). Fill this new free space with filter too. Take care to fill the bends of the drying tubes with sufficient absorbent. Sicapent® must be cleaned off from the tube ends. Finally the connection pieces are fitted into the gaskets (black), the caps are put on and the connection with the glass tube is made.



Legend:		
	Ident.-no.	
1	11.01-0015/4	U-shaped tube, GL 18, compl.
2	03 002 399	filter 2 pcs each
3	03 960 352	Sicapent®
4	22 160 200/4	drying tube
5	03 002 399	filter 1 pc each

Illustration 8.20: Drying tube

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8.4.2 Installation / Disassembly

After opening the left front door the U-shaped tubes, the soot filter and the straight reference drying tube can be seen in the upper half of the front side (see illustration 8.21).

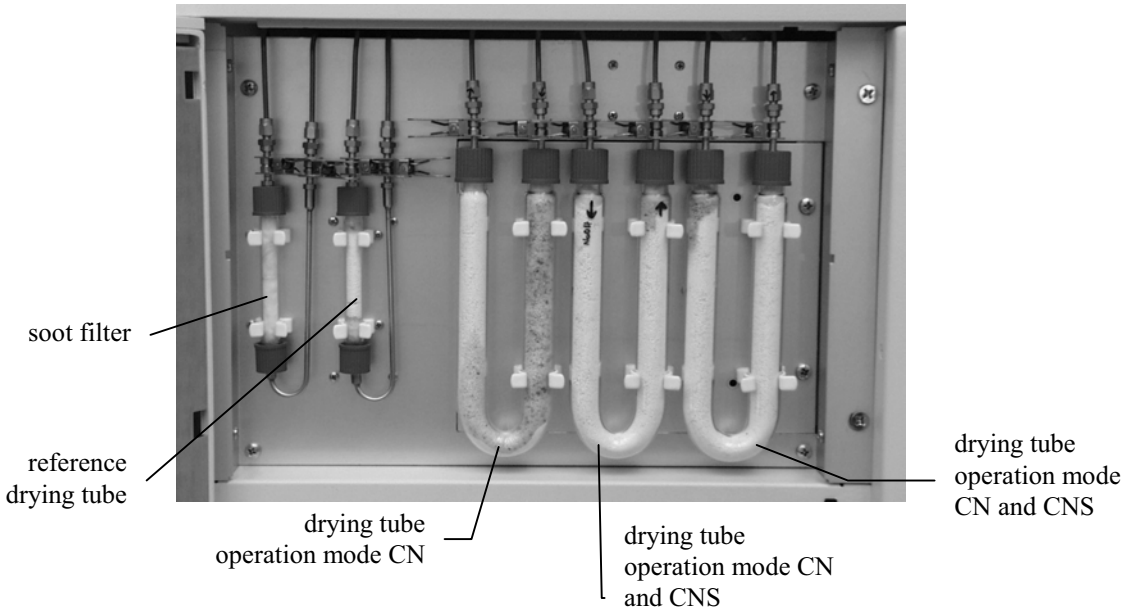

Illustration 8.21: Arrangement glass tubes front side

The following is a description of installing the U-shaped tubes. The procedure for the disassembly is in reverse order.

Disassembly or installation of the glass tubes is accomplished by opening or closing of the two ground-in clamps.

- Hold the U-tubes at the bottom and push them carefully backwards and upwards into the mounting brackets (see illustration 8.22).

- The U-tubes are then turned inwards by 90° (see illustration 8.22).

- Subsequently a connection is carried out by means of the ground-in clamps (see illustration 8.22).

Change intervals: An indicator has been added to the Sicapent® so that used up capacity is shown through blue or red color. Check before every analysis if sufficient unused Sicapent® is left. If 80% of the filling volume is discolored, a change is necessary.

Caution: **Not absorbed moisture can falsify the measuring results!**

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Illustration 8.22: Insertion of the drying tubes into the holders.

8.5 Soot filter

The soot filter serves to protect the oxygen dosing system and is installed into the O_2-supply line. It will change its color to black if the combustion pressure is too high and has then to be replaced immediately.

8.5.1 Filling

The straight glass tube is filled with 6 filters (03 002 399) Then the connection pieces are fitted into the gaskets (black), the caps are put on and the connection to the glass tube is made.

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8.5.2 Changing the soot filter

The soot filter is on the left front side (see illustration 5.2) in the O_2-connection. Disassembly or installation is performed through opening or closing the two ground-in clamps.

Change intervals: Only in the course of explosive combustion is the soot pressed into the filter (see chapter 7.1ff), which means its color turned dark. In this case, renew the filtes in the soot filter and optimize the weight or combustion parameters of the respective sample.

8.6 Emptying of the crucible container

The crucible container is meant for the used crucibles. It is able to contain approx. 120 crucibles.

The container has to be emptied prior to each analysis series in order to avoid a crucible blocking and possible damages at the gripper arm connected with it.

The automatic analysis run will be stopped if the meter of the crucible container has reached 120 in the Menu "Maintenance".

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For emptying, please proceed as follows:

- Open sidewall a bit to the right.

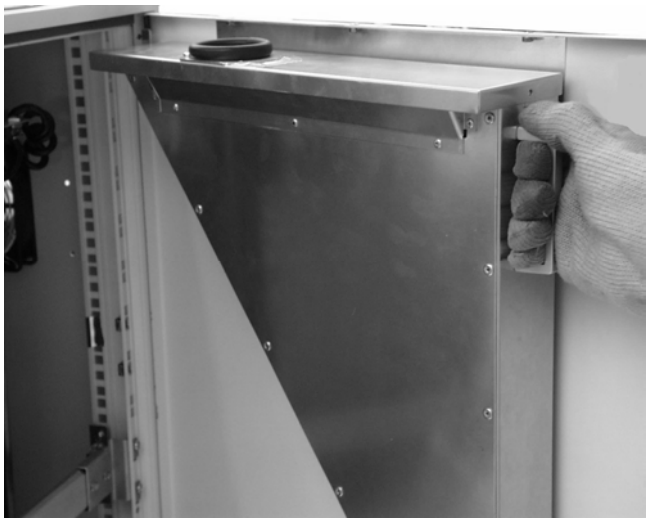

Illustration 8.23: Crucible container

- Unhinge the container.
- Remove lid from the container.
- Empty container.

There might be hot crucibles in the container left.

The installation will be done in reverse order. It shall be taken care that the container locks in the guiding rails in order to guarantee the right position.

Illustration 8.24: Guiding rails

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9. Modification from operation mode CNS to CN

The modification is be easily performed. Generally, the furnace will be equipped with the CN reaction tubes and the corresponding gas ways shall be activated by reconnecting the ground-in ball-and-socket joint connections.

The modification procedure is as follows:

- Cool off the furnaces, set the nominal values to 0°C under **Options⇨ Parameters**.

Only perform exchange of the reaction tube when cooled down!

Danger of burnings!

- Turn off gas supply to the instrument (at the supply location).

- The movable 3-tube-furnace has to be swung out of the instrument.
  In addition, the gripper arm has to be exchanged (type CNS ⇒ type CN)
  The CNS gripper is labeled with CNS. (Position of labeling between the o-rings), art.no. 25.00-0029.

- Evoque **Options ⇨ Miscellaneous ⇨ Replace Gripper** and wait until the pressure is reduced and the gripper arm has moved out. The gripper arm moves a bit to the right and stops.

For safety reasons, wait 15 minutes until the gripper has cooled down.
Then open the plexi glass door by unlocking the lock. Also open the front door on the right and the right side wall.

- Loosen all ground-in balls at the manifold, left, and at the reduction tube and post combustion tube on top (see illustration 9.1).
- Loosen the two fastening screws (knurled head screws) of the combustion tube flange with some turnings and press towards the bottom (a gap between flange and instrument casing will become visible). Keep on loosening the two knurled head screws and remove them (see illustration 9.2).
- Now the 3-tube-furnace can be pulled out of the instrument.
- For replacing the reaction tubes, now the thermal cover and the quartz connection bridge on the bottom and the tubings located at the plug on top have to be removed.

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Illustration 9.1: Front view on installed reaction tubes

Illustration 9.2:"Gap" after loosening of the knurled head screws operation mode CN

- Remove CNS reaction tubes out of the furnace and put in tube tray.
- Remove tube connection device for the Cu reduction tube below the furnace (illustration 9.3)

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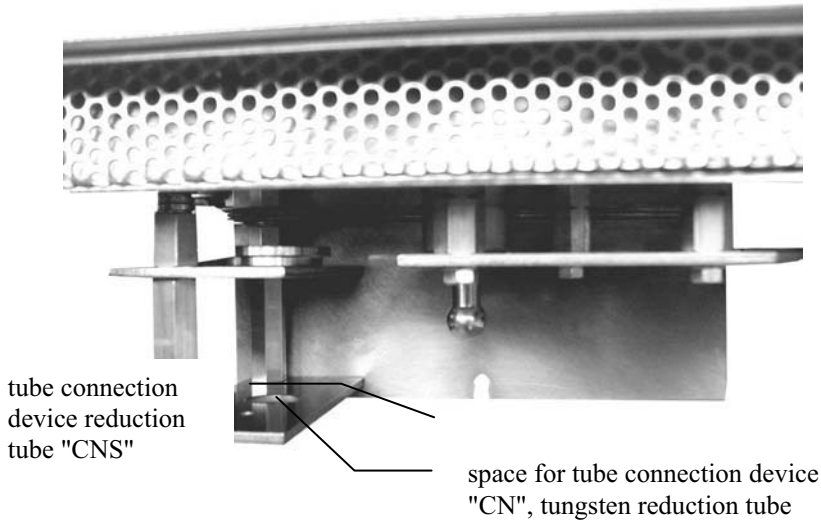

Illustration 9.3: View tube connection device

- Mount tube connection device for the tungsten reduction tube below the furnace..
- Fill CN reaction tubes (see chapter 8) and equip furnace with them.
  - Connect combustion tube outlet and post combustion tube inlet with the short quartz bridge.
  - Connect tube connection device tungsten reduction tube inlet with tubing no. 36. (Remove tubing no. 13). See illustration 9.4.

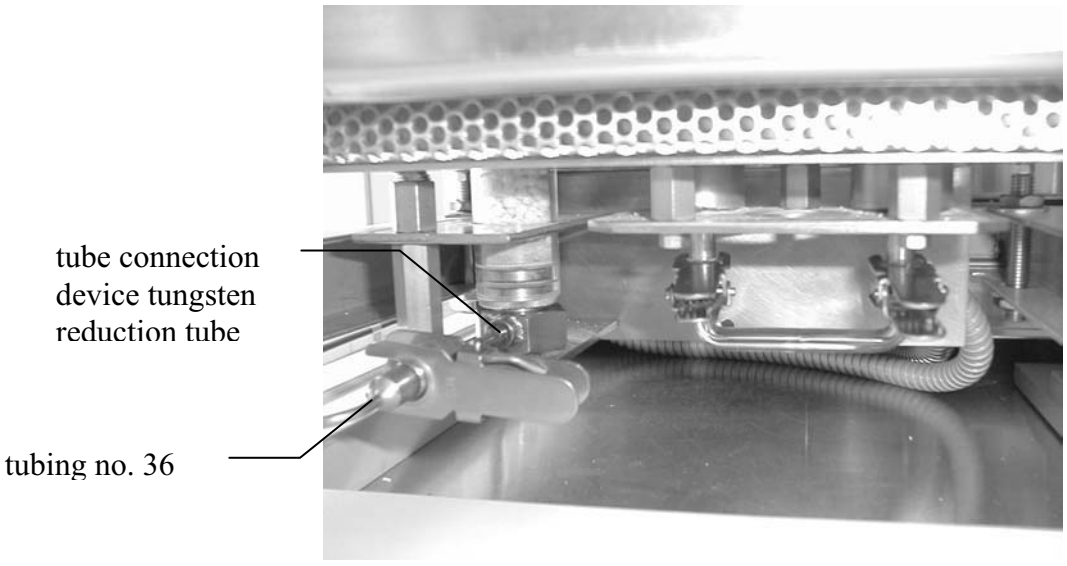

Illustration 9.4: View installed reduction tube "CN", tungsten reduction tube

- Connect water condenser outlet to valve 6A. Exchange tubing no. 39 and tubing no. 40 at the filter, in front of the valve.

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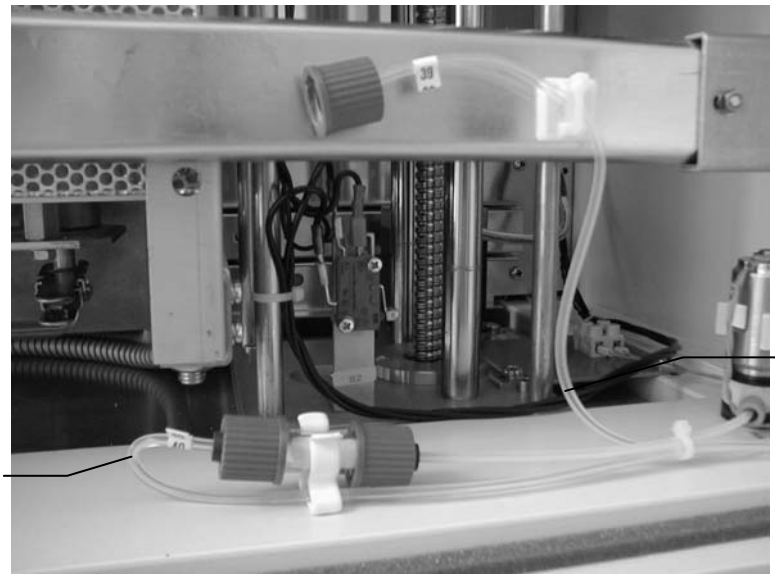

Illustration 9.5: Side view on MV 6A

- Equip drying tube "left".
- Connect tubing at the gas insert.
  - Unscrew carrousel (2 x Phillips screw)
  - Remove instrument lid "left" (remove 2 x Phillips, pull up slightly lid at the left rim and turn to the left).
  - Close T-piece "pressure drop" with a plug, prior to this, remove the pressure drop tube (no. 40), put aside in the gas insert

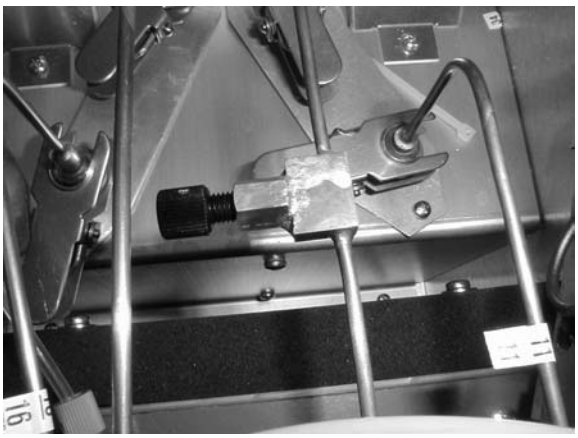

Illustration 9.6: Blind plug for CN mode

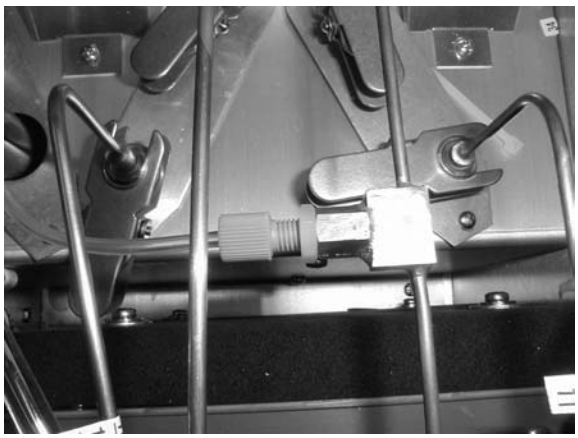

Illustration 9.7: Hose connection for CNS mode

- Connect tube for CO_2 adsorption column.

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Connect tubing no. 10 from the inlet of the SO_2 column to the inlet of the CO_2 column. Prior to this, disconnect tubing no. 15 which leads to the CO_2 column inlet and close with a blind pan.

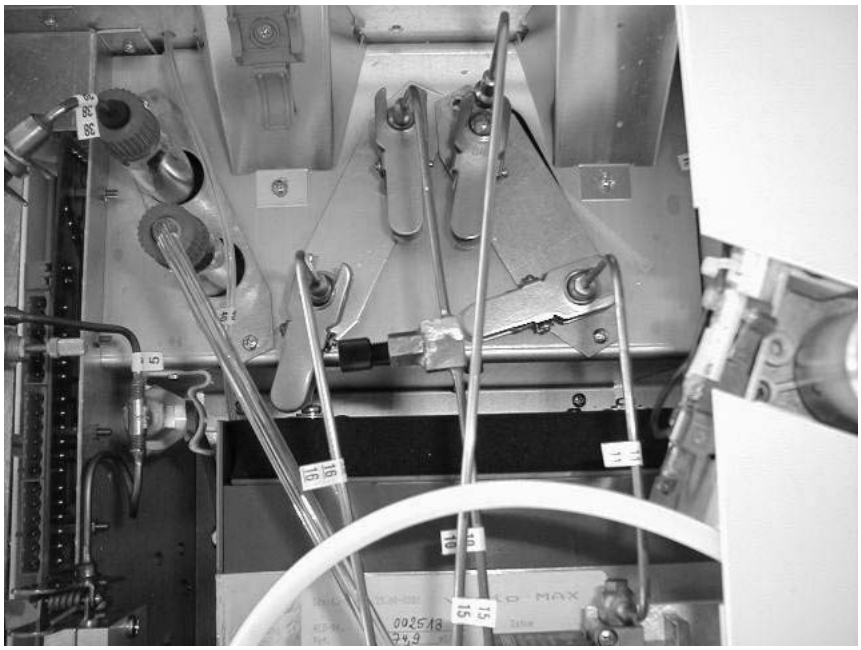

Illustration 9.8: Adsorption columns

Close inlet of SO_2 column with blind ball.

- Put on instrument lid and fasten it.
- Put on carrousel and fasten it.

- Finish tubing in furnace area.
  - Fasten tubing no. 8 and tubing no. 19 to the combustion tube .
  - Move furnace into the instrument.
  - Fasten combustion tube flange with the two knurled head screws.
  - Close ground-in ball-and-socket joints at the manifold, see illustration 9.9 and 9.10.
  - Install glass tube no. 20 from the post combustion tube outlet to glass tube no. 21 "water condenser". Close ground-in ball-and-socket joint.
  - Fasten PFA hose no. 9 at the reduction tube outlet.
  - Close front door on the right and right side wall.
- Now exchange gripper arm from type CNS to CN. Refer to chapter 8.3.

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- Leave the menu "Replace Gripper" with o.k.. Gripper and carrousel will perform a reference run. Check during immersion of the gripper into the combustions tube whether the gripper immerses in the center.
- Change from operation mode CNS to CN in menu "Mode".
- Check the parameters for CN, see chapter 5.3.5.
- Open supply gases at the place where they are taken.
- Watch at the PC the pressure and flow rates. In general the former values of the CNS operation will be set. Only pressure P2 will be a bit higher, Δ p will be lower, since in CN operation mode the resistance to flow (drop in pressure) of the SO_2 adsorption column is missing.
- If necessary – and also for safety reasons – a leak test should be performed (see chapter 10.2).
- After performance of the leakt test the furnaces can be heated up.
- The sequence of the analyses and/or measuring series can be performed as described in the example of chapter 6.2.3. The measurement of a sulfur test substance can omitted.

The modification from CN to CNS will be performed in reverse order.

Illustration 9.9:
Front view of reaction tubes

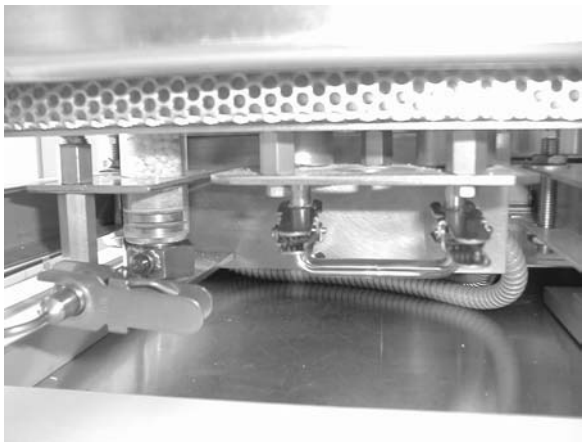

Illustration 9.10:
Ground-in clamp, bottom

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10. Maintenance / Service

Before opening a side or rear wall of the vario MAX CNS, disconnect all peripheral equipment and the vario MAX CNS itself by turning the main switch and pulling the mains plug. Attention: the 3-tube furnace must be cold otherwise components will become overheated. The side and rear walls must be properly in place before starting a new analysis (check safety linkage).

10.1 Troubleshooting

After a power supply failure of more than 15 minutes, the **vario MAX CNS** must be checked for leaks. If necessary replace all o-rings inside the furnace chamber since they may have become defect due to the missing ventilation.

10.1.1 Failure messages from the PC

The unit is equipped with an internal failure diagnosis. When the diagnosis recognizes a failure, it will be displayed, and either the unit will be stopped immediately (critical failure) or after a defined number of samples (non-critical failure). For non-critical failures, the quantity of permitted repetitions is defined on the menu **Options⇨Error Handling**. The occurring failures are displayed and also saved in the report as error code. The chronological listing of the errors (up to max. 50) is done in the menu **Options⇨Error-Buffer.** The error code can be decoded any time in the menu **Options⇨Error Index** (see illustration 10.1).

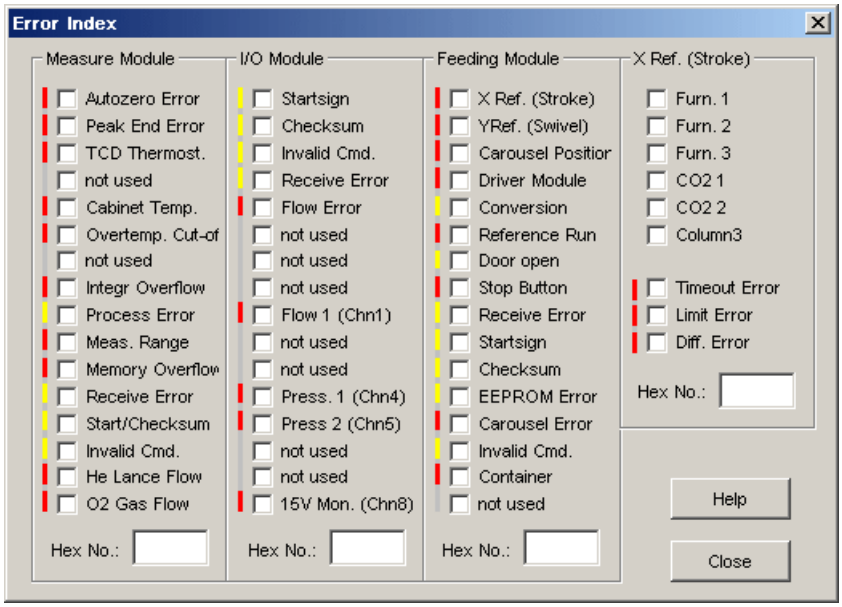

Illustration 10.1: **Menu Options⇨Error Index**

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We will now mention several error messages and the remedies that can be activated by the operator:

Failure message from the measuring module	Cause:	Remedy
Auto-Zero Error	The change of the zero point of the detector is higher than 2mV in 10 seconds That happens when the unit is started immediately after turning on the gas.	After a completely new start of the vario MAX CNS wait approx. 1.5 hours. After turning on the gas when the vario MAX CNS is already switched on, wait approx. 15 minutes.
Peak End Error	At the end of the peak the detector signal does not become smaller than the *N-Peak-End* defined in the menu **Options⇨Parameters**. The integration is aborted after 8 minutes.	• check stability of the base line of the detector • check combustion • check threshold
TCD thermostat	TCD-temperature not stable. The deviation from the nominal value is more than ! 12°C.	contact service;
Cabinet temperature too high **The heaters will be switched off parallel to this failure message.**	• Fan defect. Internal temperature of the vario MAX CNS is higher than 70°C. • Front door was open too long.	push thermal switch above 3-tube furnace back in, then restart the vario MAX CNS and PC (see chapter 5.3.3); if unsuccessful contact service.
Furnace turn-off	The furnace monitoring system has responded and switched of the furnace transformer (see chapter 5.2.4) Cause for this is usually an additional failure message of the combustion module (see following pages).	• Restart unit and software and adhere to the prescribed succession (see chapter 5.3.3) • correct failure message of the combustion module. • if unsuccessful contact service.
Pressure Error	• Operating pressure at the pressure sensor < 0,5 bar during analysis • gas bottle empty? • leakage • defective pressure sensor	• change gas bottle • Leak test • check the reaction tubes
Integration overflow	• Integral too high • Auto-Zero value too low	• insufficient gas flush before analysis start • check base line during standby condition • see remedy at "Auto Zeror Error"

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Failure message from measuring module	Cause	Remedy
Process error	• temporary disturbance of the serial interface • Start command incomplete or new start of an analysis during an analysis	if this error occurs after a re-start, contact service.
Measuring range exceeding	TCD signal was higher than 10 V	• sample weight too high • leakage
Measure memory overflow	• Analysis takes too long • Occurs mostly in combination with other failures	• check parameters and sample weight • check gas supply He + O_2
Reception error	Short failures of the serial interface	• check grounding of the data cables on both ends • check power supply • if it occurs frequently, contact service
Start sign	see reception error	see reception error
Check sum	see reception error	see reception error
Illegal command	see reception error	see reception error
Illegal command at measuring module	• Succession of switching on wrong. Error of serial interface	• keep switiching succession (see chapt. 5.3.3) • contact service
O_2 error	not enough oxygen was supplied during oxygen dosing	• O_2 bottle empty • blocking

Failure message from combustion module	Cause	Remedy
Timeout error; heaters 1 - 6	heater 1-3 or the columns did not reach the nominal temperatures during a defined time. • voltage too low • heater is defective • defective solid state relay	• heat up heaters in two steps • heater power supply defective. Check fuses • wrong nominal temperature in the parameter file? • contact service if the respective heater remains totally cold.
Limit error heaters 1-6	a defined limit temperature was exceeded • defective thermocouple electrical connection of adsorption column or blind plug not plugged in .	• check connections of ad-sorption columns. • wrong nominal temperature in the parameter file? • contact service.
Difference error heaters 1-6	Actual temperature is not approaching nominal temperature. Difference > 70°C during overheating	• short circuited thermocouple • defective heater • contact service

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Failure message from I/O module	Cause	Remedy
Start sign **Check sum** **Illegal command** **Reception error**	Temporary disturbances of the serial interface	• check mass connections of the data cables on both ends • check power supply • if it occurs frequently, contact service
Mass flow controller	the flow control is outside the set tolerance	• leak in the system; carry out a leak test • gas bottle empty • defective flow controller, contact service
Flow 1	the flow is outside the set tolerance	• leak in the system; carry out a leak test • gas bottle empty • defective flow sensor, contact service
Pressure 1 **Pressure 2**	the specified pressure sensor is outside the set tolerance	• leak in the system; carry out a leak test • gas bottle empty • defective flow sensor, contact service
+ 15 V Monitoring	the +15V voltage is outside the set tolerance	• possible defective power supply unit; contact service

Failure message from the sample feeding unit	Cause	Remedy
X-Ref (lift)	one of the lift end switches are activated during an analysis run	• start instrument from anew • if it occurs again, contact service
X-Ref (pivot)	the pivot reference switch is activated during an analysis run	• see above
Calculation	internal error during ramp computation	• usually no interference during an analysis run
Reference run	error occurs during reference run	• start instrument from anew • if it occurs again, contact service
Stop button	the stop button on the interface board is pressed	• release stop button on the board

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Failure message from the sample feeding unit	Cause	Remedy
Reception error Start sign Check sum Illegal command	Temporary disturbances of the serial interface	• check grounding of the data cables on both ends • check power supply • if it occurs frequently, contact service
EPROM error	problems during saving of the set values	• contact service
Carrousel error	an illegal carrousel position has been selected	• contact service

Failure message PC-monitoring	Cause	Remedy
Threshold values for test substances are exceeded "*Standard-Threshold-Control*" must be activated [Options⇨Parameters].	The threshold defined in the standards menu has been exceeded	• insufficient run-in samples? • check combustion • perform leakage test • chosen threshold too narrow? • weight error

10.1.2 Help to recognize failures

Failure	Cause	Remedy
Variations in the measuring values, bad peak shaping, especially for C	**poor sample combustion**	Has the O_2 dosing to be increased? ⇨ combustion parameters. Does the oxygen get to the crucible? ⇨ check Pt jet in the gripper. How old are the reaction tubes and their fillings? ⇨ If necessary replace combustion tube and post combustion tube incl. fillings.
Long peak shaping for S, too low recovery for S	**memory effects for S due to aging of gripper, reaction tubes**	⇨ If gripper > 700 samples, replace it ⇨ If tube fillings (combustion tube) > 500, replace them

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Failure	Cause	Remedy
Variations in the N measuring values which are also too low	**a) C-content of the sample too high** **b) CO_2 adsorption too fast** **c) first sign of blocking**	Watch the pressure display P2 (MFC inlet) during combustion. The pressure must not decrease below 0.45...0.50 bar. to a) and b): The CO_2 adsorption is too fast, the system will lose to much gas and P2 decreases. ⇨ Decrease sample weight. to c): The gas and/or pressure loss resulting from the CO_2 adsorption will not be compensated fast enough by the flowing helium due to higher flow resistance (blockings) in the area of the combustion tube/post combustion tube/reduction tube. ⇨ Watch P1 and P2 in standby mode. If the pressure difference is higher than 0.15 bar, there is a beginning blocking. ⇨ Check the quartz bridge. ⇨ Check the age of the tube fillings, if necessary replace them.
Too low N results, variations in N results P1 increases to > 2.0 bar during combustion P2 is at only 1.1 bar and decreases to < 0.5 bar during combustion / CO_2 adsorption	**Blockings**	⇨ Look for blockings in the area of the combustion tube – post combustion tube – reduction tube (see above).
Too low C, N and S results P1 and P2 are too low; flow display too high	**Leak**	⇨ Untight retaining ring in the combustion tube flange; put new grease on flange and retaining ring, if necessary replace it. ⇨ Has the sealing flange any scratches? ⇨ Check water drain valve V6A for tightness (put water drain hose into water). ⇨ Perform leak test and look for leaks.
Steadily increasing N measuring results. At the end of the N-peak there is a peak which is getting bigger and bigger sample by sample.	**Reduction tube consumed**	⇨Replace reduction tube filling. ⇨Check cupper in the post combustion tube, if oxidized, replace it.

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High C and N blank results when using brand new crucibles.	**Crucible blank value, only occuring once**	⇨ Glow all new crucibles in a furnace (1000°C, 30 minutes) or perform a complete blank value run.
Operation mode CN	**Oxydation agent in the reduction tube used up**	⇨ Replace the CuO layer in the reduction tube.
P1 and P2 too low. The other parameters are o.k. (flow rates, Δp, measuring values).	**Throttle of pressure setting out of adjustment**	Adjust throttle in the gas insert from anew: remove left casing cover, the throttle is opposite valve V6B. In standby mode P1 will be set to approx. 1.50 bar
Variations in the measuring values of all elements which means: CNS value always too high or too low. Stable CN relation	**Probably a weighing error**	Check the balance with a test weight regarding stability and consistency. Avoid electrostatic charging during weighing. Contact service if the problem persists.

10.1.3 Replacement of adsorption columns

Replacement of adsorption columns can be necessary if:
Aging of the column filling (e.g. through fluor compounds) which decreases the adsorption/desorption ability and causes extreme peak widening.
All columns are available as a new part or replacement part (only with a new filling).

For replacement of adsorption columns, furnaces and columns must have been cooled down and the vario MAX CNS must have been shut off. For this purpose set furnaces and adsorption columns to nominal temperature 0°C and let them cool down to room temperature (max. 100°C).
Turn off vario MAX CNS after it has cooled down so that it does not overheat because of missing ventilation.

Procedure for the replacement of adsorption columns:

a) Set furnaces and adsorption columns to nominal temperature 0°C and let them cool down to room temperature (max. 100°C). For this purpose set nominal temperatures in **Options⇨Parameters** to 0°C and wait until they have cooled off (approx. 2 hours).

b) Leave **vario MAX CNS**-software with **System⇨Offline** and **File⇨Exit**.

c) Turn main switch of **vario MAX CNS** off (see illustration 5.2).

d) Remove left cover plate from instrument top by removing two Philips screws. The separation part of the **vario MAX CNS** is now accessible. Illustration 10.2 shows the lay-out of the adsorption columns and their electrical connection sockets.

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1 remains free
2 electrical connection for adsorption column CO_2
3 electrical connection for adsorption column SO_2
4 adsorption column CO_2
5 adsorption column SO_2
6 water condenser

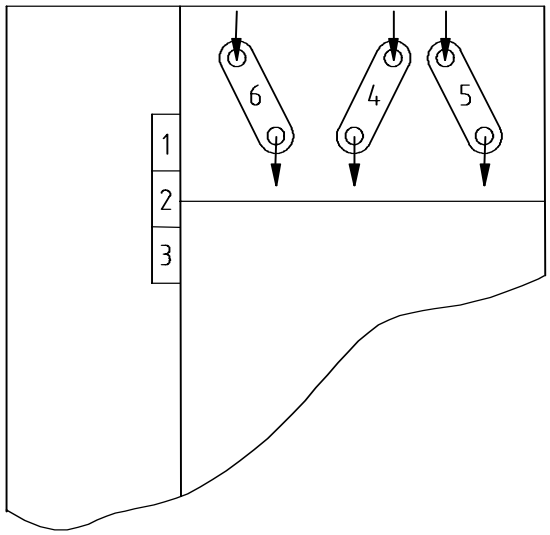

Illustration 10.2: lay-out of the adsorption columns (top view)
CO_2 art.-no. 25.11-0001 / SO_2 art.-no. 25.20-0009

e) **Disassembly of the adsorption column for replacement (art.no.: 25.00-0091 for 400 mg C, 25.11-0001 for 200 mg C)**:

- When replacing column 2, unscrew the carrousel (2 x Philips screws)
- Loosen ground-in screws at inlet and outlet.
- Loosen two Philips screws of the holding device.
- Pull plug from the appropriate socket.
- Remove adsorption column.
- Close adsorption column with blind stopper.
  Store adsorption column in a cool, dry place where it is not subjected to vibrations or send in to be refilled.

f) **Installation of a new adsorption column**:

- Insert adsorption column into its retainer.
- Fasten holding device with two screws.
- Plug the adsorption column in (see illustration 9.2).
  The electrical connection sockets must be coded so that a mistake is not possible.
- Remove blind stoppers and close the ground-in connections at the inlet of the adsorption column according to the tubing diagram (see illustration 4.1).
- The outlet of the adsorption column remains open because the new adsorption column must be heated up before operation.

g) **Heating the new adsorption column**:

- Turn **vario MAX CNS** on (see chapter 5).
- Reduce He-prepressure at the source to 0.5 – 0.6 bar.
- Load **vario MAX CNS** software and start the heating program with function

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Options⇨Miscellaneous⇨CO_2-Column).

- After heating time is over close the outlet of the adsorption column according to the tubing diagram (see chapter 4.1).

h) Reset He-prepressure at the source to 3.8 bar and remount the cover plate on the top of the unit.

i) The **vario MAX CNS** is now ready for operation.

Note: If after an adsorption column replacement the daily factor is not within the range of 0.9 to 1.1, a calibration becomes necessary (see chapter 6).

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10.1.4 Customer maintenance

Maintenance	Recommended interval	Reference
Changing fillings or replacing tubes with simultaneous checking of the plugs and O-rings	for changing intervals see maintenance menu **Options⇨Maintenance**	chapter 8
Cleaning or checking the quartz glass bridge between combustion and reduction tube incl. checking of the o-rings	at every tube replacement	chapter 8
Checking or replacing the absorption-U-tubes and/or drying tubes	daily sight check	chapter 8
Checking the capacity of the hard disk, saving the data to a diskette, if necessary	backup each week, saving on disk, if necessary	
Checking the supply gases for correct pre-pressure and sufficient volume	before every test series	chapter 5
Leak test of the **vario MAX CNS** and the supply lines for the gases	min. every two months	
Check of the gripper arm: a) state of the jaws of grab, clean with a wire brush, if necessary b) state of the O_2 outlet jet made of platinum, clean with drill 0.8 mm diam., if necessary	 daily sight check daily sight check (with mirror)	 chapter 8.3 chapter 8.3
Grease driving spindle of the gripper arm (WD40 initial outfit) Clean and grease shaft for rotating motion (WD40 initial outfit)	after 1000 samples (dusty laboratory conditions) otherwise after approx. 3000 samples after 3000 samples (or two weeks)	
Grease retaining ring sealing of the combustion tube	weekly	
Replace retaining ring sealing of the combustion tube	after 8,000 samples	
Empty crucibles' container	after 120 samples (⊂ 2 carrousel)	

Recommendation: an annual maintenance contract for a complete maintenance saves time and money.
For more information contact your service.

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10.1.5 Replacing fuses

Note: If the overload releases jumped out, please always inform your respective service. The causes could be serious.

Only fuses of the mentioned type must be used. In case fuses with higher fusing current data are being used, liability will not be valid. Prior to replacement of the fuses, turn off the instrument and pull out the power supply plug.

To replace the fuses, the left side wall of the **vario MAX CNS** must be removed.

Table of the vario MAX CNS fuses			**Ident. no.**
F1	furnace 1	42 V AC; M 15 A	05 000 455
F2 + F3	furnace 2, furnace 3	42 V AC; M10 A	05 000 454
F4	adsorption columns	42 V AC; M 10 A	05 000 454
F5	heater for the tubes	13 V AC; T 8 A	05 001 028
F7	TCD-heater	24 V AC; T6,3 A	05 001 029
F8	power supply	230 V AC; T2 A	05 001 031
F9 + F10	furnace transformer	230 V AC; M15 A	05 000 455
F11 + F12	electronic-transformer	230 V AC; M15 A	05 000 455

10.1.6 Replacing the O-rings

The sealings in the drying tubs, in the plugs of the combustion and reduction tubes and in the quartz glass bridge are particularly susceptible to chemical corrosion and mechanical and thermal stress, which alter their material properties, so that after a while they become leaky.

Sealings in the drying tubes
The sealings are immediately accessible after removing the sealing plugs. They only have to be slipped over the cylindrical tube connections.
A replacement is advisable when the sealings show cracks or permanent damage caused by the sharp edges of the drying tube.

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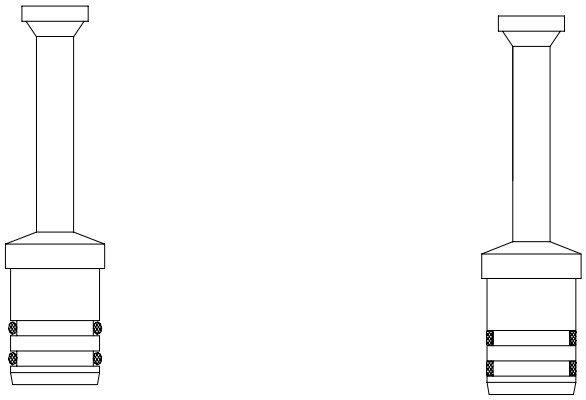
O-rings in the quartz glass bridge
After disassembly of the quartz glass bridge the o-rings are freely accessible in the ground-in connections of the quartz glass bridge.
Always replace the o-rings in the quartz glass bridge if only a slight pressure resistance or none at all can be felt on closing the ground ball connector and/or if the o-rings are mechanically deformed.

O-rings in the combustion tube flange or in the sealing plug of the reduction tube and the post combustion tube
These o-rings are subject to long-term thermal wear and tear. In the course of time they are pressed flat and become leaky. During cooling-off the plug / flange shrinks a little and if the o-rings are old and not elastic anymore they cause leaks or the sealing plug or flange can fall out of the tube. For disassembly and installation of the combustion tube flange see chapter 8.1.

Always replace the o-rings in the sealing plugs if only a slight pressure resistance can be felt on inserting the plug into the tube. A visual check is helpful, to see if the o-rings still protrude from the recesses of the plugs (see illustration 10.3).



O-rings are in good condition

O-rings must be replaced

Illustration 10.3: condition of o-rings in sealing plugs

To remove an o-ring from a sealing plug, push it with thumb and index finger sideways out of the plug until it forms a loop at the side (see illustration 10.4). Grip this loop with a pair of tweezers and pull the o-ring off the plug completely.

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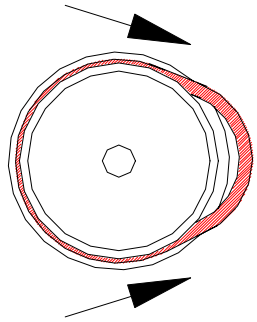

Illustration 10.4: removing the o-ring

Using a sharp knife or scalpel to cut out o-rings can damage the sealing surface underneath the o-ring through scratches. There is also the danger of injuries through slipping!!

10.1.7 Replacing the retaining ring sealings in the combustion tube flange

The complete combustion tube flange including sealing has to be replaced by an exchange flange.

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10.2 Leak test

Leak tests can be performed via the menu commands **Options⇨Miscellaneous⇨Rough Leak Check** and/or **Options⇨Miscellaneous⇨Fine Leak Check.**
The different leak test steps are self explanatory and menu guided.

10.2.1 General

For the rough leak test and the precision leak test (entire instrument) the user only has to reduce the He prepressure at the place where pressure is taken (see menu leak test). Further auxiliaries are not required, the leak tests run automatically.

In case of a required leak search in the precision leak test, the latter will be divided in steps. The steps are marked by different colors in the menu. The user has to install blind plugs or by-pass lines at the corresponding places.
The corresponding parts are included in the leak test set, ident.-no. 25.00-5037.

10.2.2 Rough leak test

No leak rate is being identified during the rough leak test. It will only be stated whether the He flow into the instrument is being reduced to ⊄ zero with a closed outlet valve.

The rough leak test is recommended after replacement of tubes etc. in order to exclude severe mistakes during assemblance of the equipment (forgot O-rings, etc.?).

10.2.3 Precision leak test

The precision leak test will be performed with computing the leak rate, i.e. only a certain pressure loss within a certain time is permitted. The result is divided in:

- non permissible leak (red bar)
- tolerable leak (yellow bar)
- no leak within tolerance (green bar)

If a leak is being identified during a precision leak test, the equipment has to be divided in different segments ("steps") in order to locate the leak. Blind plugs or by-pass lines have to be installed at certain places. The leak test menu contains the corresponding help.

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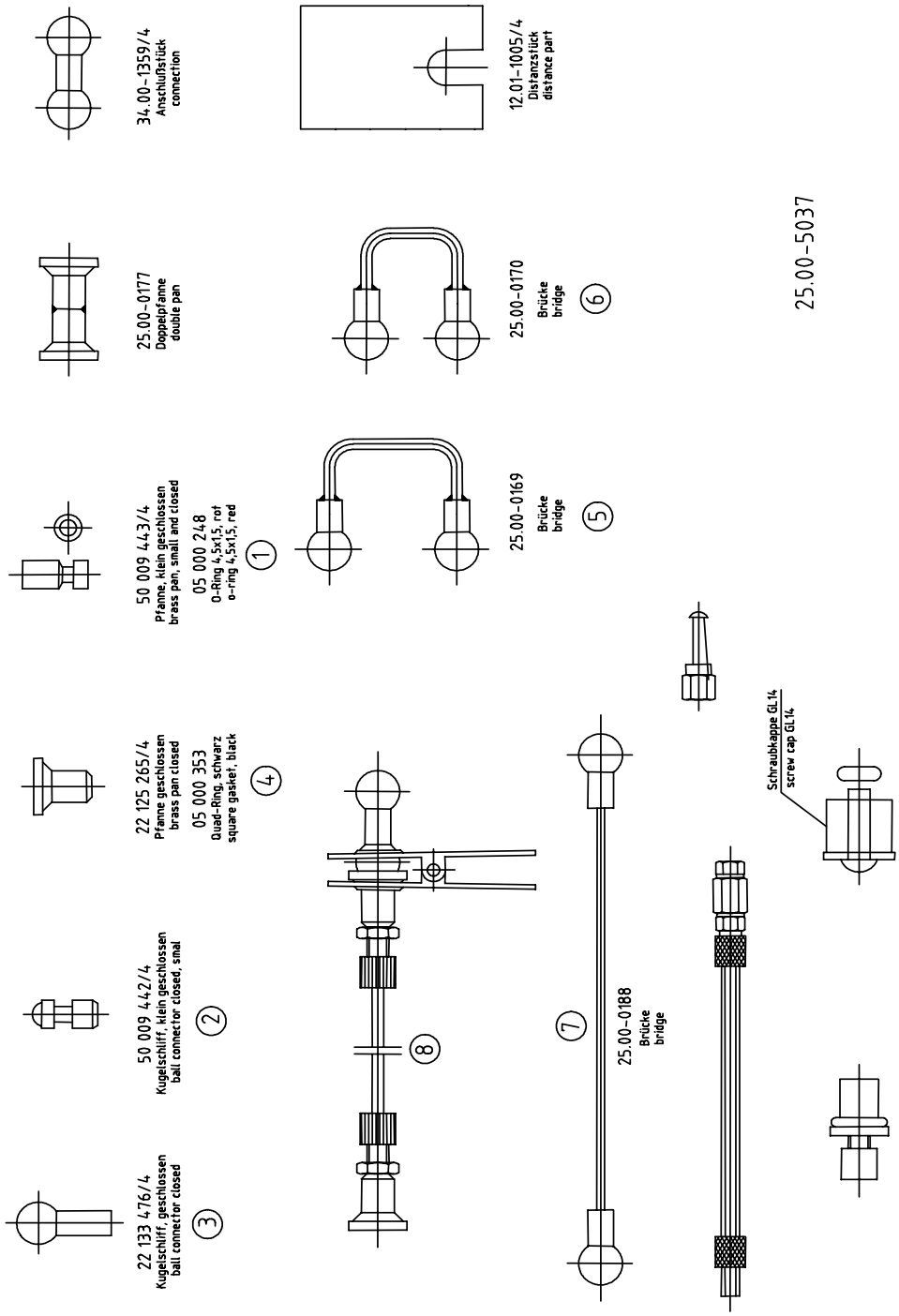

Illustration 10.5: Leakage Set

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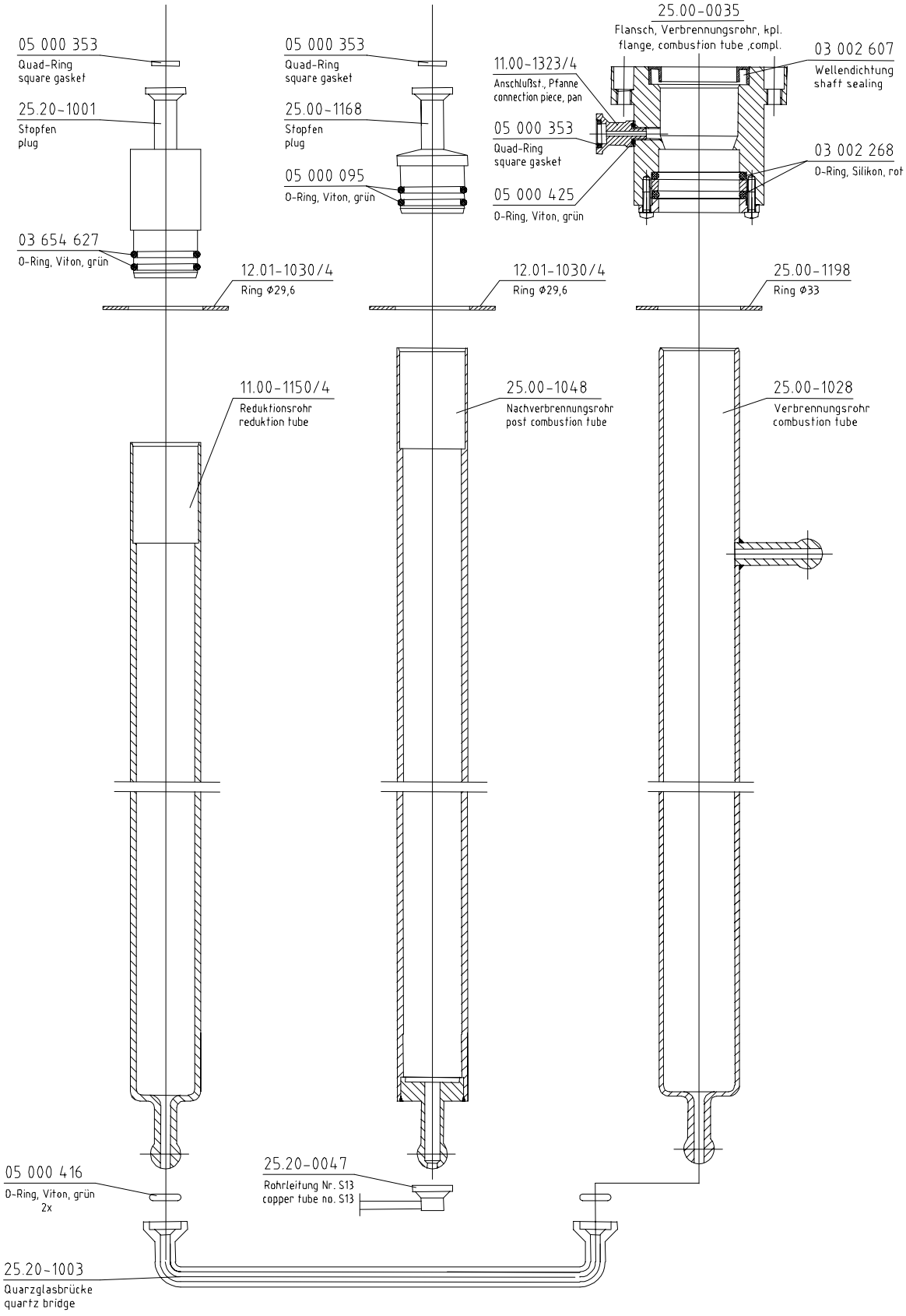

Illustration 10.6 : Tubes and plugs vario MAX CNS

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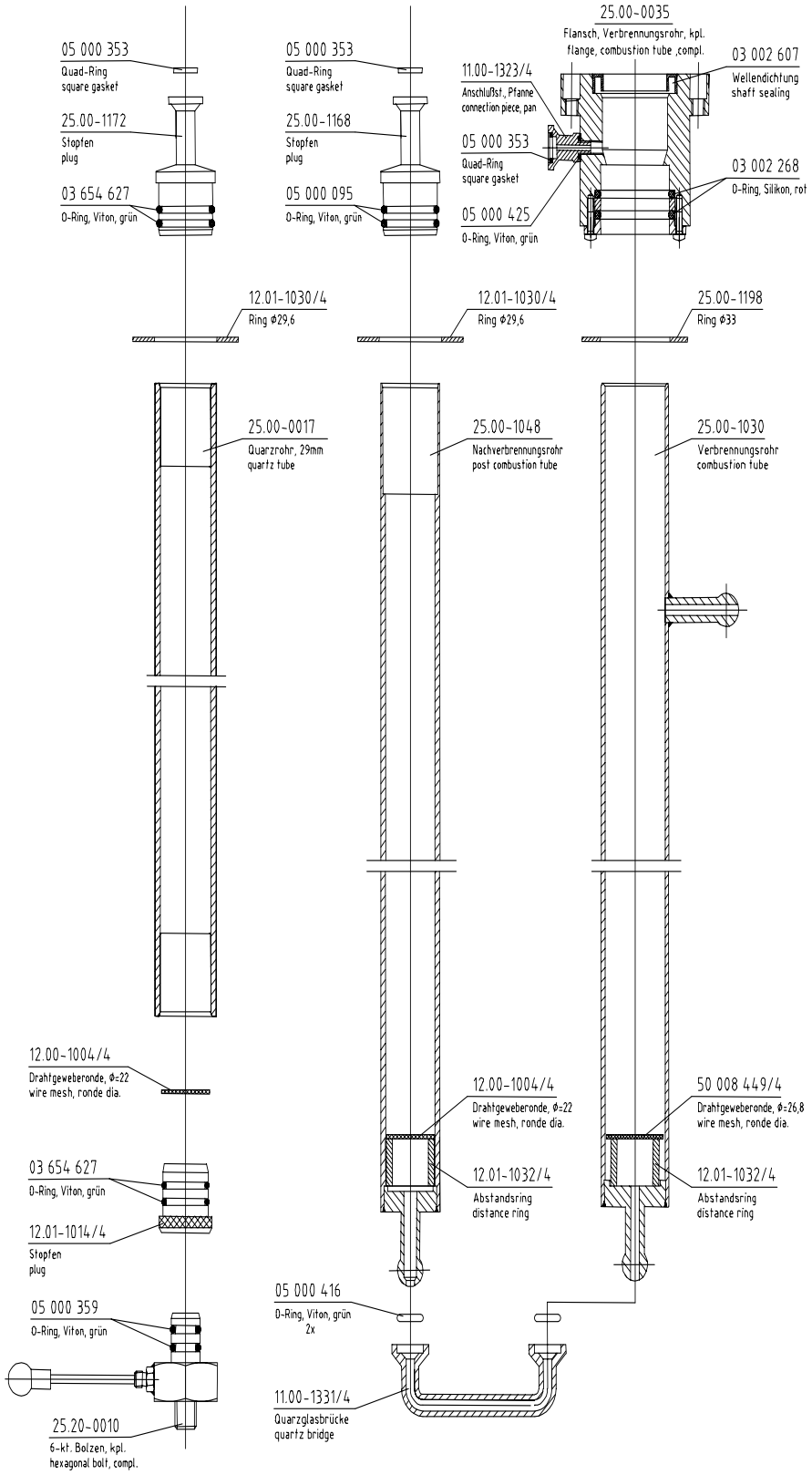

Illustration 10.7: Tubes and plugs vario MAX CN - CNS

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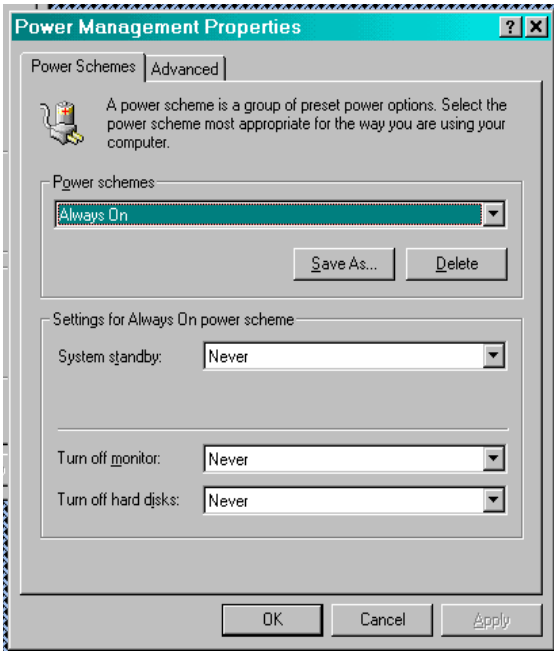
11.2 Solving PC problems

Occasionally, there are problems with the PCs which probably are in connection with the Elementar instrument software. Possible problems may be occasional system crashes or a freeze of the instrument software. The cause can be explained by the connection of the settings in the operation system for the power management of the PC. The settings shall be checked in the power management under Control Panel-> Power Option Properties and/or Power Schemes. The options for "Turn off monitor", "Turn off hard disks", "System standby" and "System hibernates" shall be set to "Never". If possible, also the power management in the BIOS of the PC shall be deactivated.



If the PC is connected to a network a further measure can be the deactivation of the network. If possible, deactivate the network adapter under Control Panel -> System Manager or remove the network cable and restart the PC without network.

The vario MAX software exclusively runs on the operating systems Windows 2000® and Windows XP Professional®.

An operation on older systems like Windows® 9x / ME / NT and Windows XP Home Edition® is not possible.

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11.3 Special Applications

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11.3.1 Analysis of strong salt containing samples

The following is meant for samples with a high amount of common salt or pickle of up to 60%, e.g. seasoning, instant soup concentrates, soup cubes, instant sauces, etc.

The salt melts and sublimes at 900°C under regular combustion conditions. It will be slowly transported into the instrument and blocks the combustion and post combustion tube.
This procedure is being avoided if the combustion temperatur will be decreased to 750°C (the salt remains almost 100% in the crucible). The combustion parameters will not be changed (O_2 dosing).

The remaining salt should be washed out with hot water after the analysis. The crucibles must not be used uncleaned at 900°C.
In case of pure pickle, it should be worked with an cellulose additive, similar to the application for fertilizers.

If there is a large variety of sample matrices, it has to be tested whether all samples can be analyzed at a low combustion temperature.
It might be that the samples have to be sorted in such a way that e.g. one sample package will be analyzed at a combustion temperature of 750°C and another sample package at a combustion temperature of 900°C.
In case of larger series, the silver wool in the combustion tube has to be checked every 250 analyses.

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11.3.2 Analysis of inorganic fertilizers

Anorganic fertilizers often contain NO_3.

For the tranformation of nitric oxides into N_2, in case of anorganic fertilizers one needs the presence of carbon since carbon strongly supports the reduction of NO_X to N_2 as formed CO.

Carbon will be mixed as an additive as an organic nitrogen-free compound with the fertilizer sample to be analyzed. The amount of the additive depends on the carbon content of the used substance.

Example:

200 mg	$NH_4 NO_3$	+	spprox.. 200 mg	± 20 mg benzoic acid
400 mg	$NH_4 NO_3$	+	approx. 400 mg	± 20 mg benzoic acid
200 mg	$NH_4 NO_3$	+	approx. 300 mg	± 20 mg cellulose
400 mg	$NH_4 NO_3$	+	approx. 600 mg	± 20 mg cellulose

Note:

- Glucose and other sugar in some cases did not prove to be suitable as an additive.
- Cellulose does not necessarily combust 100%. Carbon containing residues remain in the crucible which have to be removed (only in N mode).
- Preferable grain size ≤ 0.5 mm.

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11.4 Installation requirements

Dear User,

We thank you for your order and the confidence expressed to our representative and Elementar by this decision.

You have made you choice for a CN/CNS/CHN and N analyzer of highest analytical performance and reliability. In order to secure a fast and smooth installation of the instrument in your laboratory, we kindly ask you to take the following hints into consideration:

Installation location

- The analyzer can be installed under normal laboratory conditions, i.e. no fume hoods or air condition (ambient temperature between +15°C max. +35°C) are necessary.
- However, like for all analytical equipment direct sun beams, draft or other inconvenient conditions for precision instruments should be avoided.
- For the installation of the unit (without PC, printer and balance) a stable laboratory table with heat resistant surface is sufficient.
    - Minimum dimensions: length 1.20 m * depth 0.80 m
    - Weight vario MAX approx. 130 kg.

Gas supply

For the operation of the analyzer the following gases are required which should be kept ready by you on the spot for installation:

- helium (purity 99.996 %)
- oxygen (purity 99.995 %)

Unless delivered from us, the availability of two-step high precision pressure reducers (Air Liquide or similar) is necessary. Pressure reducers of inferior quality will influence negatively the analysis results!

! Pay attention to the bottle pressure !

Instrument control

If PC and balance were not delivered from Elementar, we reserve the right to perform the installation with own instruments. Possible modifications to our analyzer have to be done by the customer.

We would like to emphasize that the PC is the central control system of the analyzer. The operating ability of the analyzer depends on a failure-free operation of the PC. Due to experiences in the past with the use of control PC in mixed operation as office workplace impairments due to software instabilities may occur.

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Therefore, we recommend to only use programs on this PC for operation and data evaluation of this analyzer.

Control PC

In case the PC was not delivered by us, the following minimum requirements have to be met:

For operation without modem:

- PC with Pentium III Processor 500 MHZ (or equivalent) or faster
- Operating system
  - Windows 2000 Professional (SP3 or later), with 128 Mbyte RAM
    or
  - Windows XP Professional (SP1 or later), with 256 Mbyte RAM
- At least 2 GB fee memory on hard disc
- 1 free serial port RS 232
- CD-ROM or DVD-ROM drive

For operation with modem:

- 2 free serial ports RS 232
- ***CAUTION:*** The PC mouse must not occupy any of these ports.
- 1 analog telephone socket TAE-6N in 6 m periphery from the PC.
- This connection is necessary to guarantee the remote control maintenance of the instrument via an analog modem.
- 1 analog modem with at least 56 kBaud transfer rate.
- Remote control maintenance software Symantec PC-Anywhere 10.5 or later.

Installation requirements for the instrument software:

To install the instrument software correctly, administrator rights for the PC have to be made available.

Requirements for the correct operation of the instrument software:

The instrument software has to be operated under a user account that is provided with writing rights in the instrument software directory (e.g. c:\variomax) and in the system directory (e.g. c:\windows).

The PC where the instrument software will be installed should be checked for settings in the control panel under Control Panel⇨Power Option Properties and/or Power Schemes. Setting should be made as follows: "Turn off monitor", "Turn off hard disks", "System stand by" and "System hibernates" should always be set to "Never". If possible, also deactivate the power management in the BIOS of the PC.

As screen saver use only the Windows standard screen saver.

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Generally, it is not recommended to overload the PC where the instrument software is installed with other software programs. This may lead to instable operation of the instrument software.

Balance

Requirements for the balance: readability of 0.1 mg, alternatively 0.01 mg.

weighing range 10 g

tara 10 g

If the balance was not furnished by us, we reserve the right to enter the weights manually. Possible adjustments to our analyzer have to be made by the customer.

General remarks

The instrument will be installed by a factory trained service engineer and the analytical performance will be demonstrated by max. 10 standard calibration samples.

In case of specific handling and application questions, you are kindly invited to contact our representative for assistance. The elaboration of specific operation procedures can be ordered there against separate charge.

The instrument is calibrated over a standard measuring range from works`side which is stable for a long time. If specific applications are not within this range, the user can perform an extended calibration by himself.

Our agent and Elementar will of course assist you at all arising questions.
Please do not hesitate to contact us under:

Service:	+49/6181/91 00-16
Application Lab:	+49/6181/91 00-26
Sales Department:	+49/6181/91 00-12

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Specifications:

1. Electrical connection values for complete analyzer unit:

Power connection value:	16A	
Voltage value:	100V AC	± 10%
	120V AC	± 10%
	230V AC	± 10%
	50/60 Hz	

A separate static bonding connection (safety earth) is necessary.

Connector: 1 x connector (customary in place)

Mode of protection: IP 20, installation in dry rooms only.

2. Gas supply:

Emission-free parts should be used in the entire gas supply (copper, stainless steel, etc.)

Pressure reducer:

Output pressure:	2,5 bar	adjustment range:	0-3 bar/0-300 kPa oxygen
Output pressure:	3,8 bar	adjustment range:	0-8 bar/0-800 kPa helium

two step precision reducer

Example: Type AIR LIQUIDE HSB 315/3 for oxygen
HSB 315/8 for helium

!CAUTION: Pay attention to the bottle pressure!

Necessary gas connection behind the pressure reducers 2 x clamping ring screwing for tube diameter 6 mm /Swadgelock, Serto)

Initial operation and instructions:

Please make sure that the direct user of the Instrument is available for instrument delivery and training over the whole period of time.

If additional instructions or training time by our service technician are required, they will have to charge separately.

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Initial outfit vario MAX (CNS) for installation and training				
Erstausstattung vario MAX (CNS) für Inbetriebnahme und Einweisung				
art.-no.:	25.20-0031	Date:	31.1.03	(Te)
			Menge	
article-no.	description	Artikelname	units	
03 002 262	Pt-catalyst 5%, 30g	Pt Katalysator 5%, 30 g	1	pcs
03 002 341	funnel D = 65 mm	Trichter, D = 65 mm	1	pcs
03 002 395	o-ring 7 x 1.5, red	O-Ring 7 x 1,5, rot	3	pcs
03 002 399	filter 18 x 20 mm	Filter 18 x 20 mm	40	pcs
03 651 010	pressure hose	Druckluftschlauch 5 x 1	25	m
03 654 627	o-ring 20 x 2, green	O-Ring 20 x 2, grün	4	pcs
03 674 010	clamp, large	Schliffklammer, groß	2	pcs
03 679 908	quartz wool, 10 g	Quarzwolle	1	pcs
03 968 006	vacuum grease 100 gr	Vakuumfett 100 g	1	pcs
04 000 080	spiral drill 0,8 mm	Spiralbohrer 0,8 mm	1	pcs
04 266 100	tool holding device 100 mm	Werkzeughalter 100 mm	1	pcs
05 000 094	gloves, temp. resistant	Handschuhe, 1 Paar	1	pcs
05 000 095	o-ring 22 x 2, green	O-Ring 22 x 2, grün	4	pcs
05 000 353	square gasket, black	Quad-Ring, schwarz	2	pcs
05 000 358	leather gloves, 1 pair	Lederhandschuhe	1	pcs
05 000 416	o-ring 7.5 x 2.5, green	O-Ring 7,5 x 2,5, grün	2	pcs
05 000 699	copper, wire 100 g	Kupfer, drahtförmig	18	pcs
05 000 934	mouse pad	Mouse - Pad	1	pcs
05 001 039	Copper oxide, wires, 250g	Kupferoxid, drahtform, 250g	1	pcs
05 001 067	glutamic acid, purity > 99%	Glutaminsäure Reinheit >99%	1	pcs
05 001 137	Special oil WD 40	Sprühdose WD 40	1	pcs
05 001 144	sulfadiazine	Sulfadiazin	1	pcs
05 001 255	hexagon socket screw key	Inbuschlüssel Gr. 4	1	pcs
11.00-0500/4	manually operation drilling	Hand-Ausbohrwerkzeug	1	pcs
11.00-1150/4	combustion tube, reduct. tube	Verbrennungs-, Reduktionsrohr	1	pcs
11.02-0008	tungsten(VI)-oxide,60 g	Wolfram(VI)-Oxid Granulat 60 g	2	pcs
11.02-0024	tungsten(VI)-oxyd powder, 90g	Wolfram(VI)-Oxid Pulver, 90g	2	pcs
22 131 365	silver wool, 50 g	Silberwolle	1	pcs
25.00-0037	plate for 18 samples	Probentablett für 18 Proben	1	pcs
25.00-0042	tube extractor, compl.	Rohrgreifer, kpl.	1	pcs
25.00-0095	filling and measure tube	Füll.- und Messrohr	1	pcs
25.00-0099	set of glass tubes	Glasleitungssatz	1	pcs
25.00-0171	tubing, instrument	Rohrleitung,Geräte-Gasanschluß	1	pcs
25.00-0172	tubing, instrument	Rohrleitung,Geräte-Gasanschluß	1	pcs
25.00-1028	Combustion tube quartz	Verbrennungsrohr,Quarz	1	pcs