AIRSENSE
ANALYTICS

Trap & Thermal Desorption µ-TD

Handbook

Version 3.0

WMA Airsense Analysentechnik GmbH

12.03.2003

(subject to modifications and amendments)

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The Micro (Trap and) Thermal Desorption

Introduction

Smell intensive compounds in concentrations of less than the detection limit of an analytical instrument need to be enriched before analysis. Gaseous substances are trapped at sampling temperature (ambient) on adsorption tubes and analyzed after thermal desorption. The enrichment factor is related to many different physical and sampling parameters. It can be calculated on the basis of breakthrough volumes known from common tables. Typically, the detection limit can be reduced by a factor of 20 with volatile compounds and up to 1000 with low volatiles (e.g. limonene). Temperatures of the adsorbent during sampling and desorption phases can be adjusted via settings within the software delivered the instrument.

Selective enrichment is possible by choosing suitable adsorbent materials. For example when analyzing beverages, highly volatile compounds, such as CO_2 or even ethanol, are not enriched on the adsorbent "Tenax", while medium- or low-volatile substances, like most flavor compounds are trapped. Thus, the analysis can be limited to the actual flavor substances and the selectivity of the sampling device can be increased.

With applications in very damp environments this hydrophobic polymer has also the advantage that cross influences of humidity on the analysis are avoided.
The combination of heating the samples and adsorption followed by desorption of the tubes is another option that can be very useful in some applications.

Operation Principle

With the Micro-TD the following analytical steps :

1) sampling,
2) postsampling,
3) desorbing,
4) injecting and
5) cleaning

are performed automatically.

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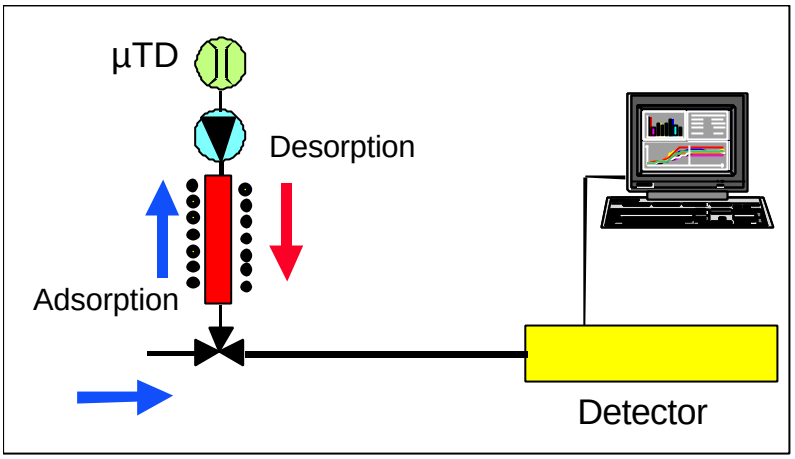

Fig.1: Principle of operation of the µTD

During the sampling step the compounds are trapped at ambient temperatures. The flow during sampling can be adjusted between 50ml/min and 500 ml/min. The maximal sampling time depends on the retention volumes of the analyte on the adsorbent material. The standard trapping material delivered with the system is Tenax-TA (150mg).

After sampling, the tube is heated to its desorption temperature. There is no flow during the heating step. After heating the tube to temperatures $T \geq 160°C$ (T_{max}=280°C) the gas flow is inverted and the trapped compounds are injected into the detector.

Afterwards the tube is cleaned by heating to a higher temperature than the desorption temperature and the tube is rinsed with cleaned air. After cooling down to near ambient temperatures the trap is ready for the next measurement.

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Hard and Software requirements

Requirements for the instrument µ-TD :

- 110 ... 250VAC (12VDC optional)
- Injection gas, He, N2 or clean air, pressurized, externally flow controlled. As a different solution, the detector (µ-GC) may suck the sample itself during injection with a pump
- Purge gas and Cleaning gas (optional, if different from cleaning gas), He, N2 or clean air, not pressurized
- Unit to be operated within ambient temperature range of 0°C ... 35°C

Hardware Requirements for Software (MicroTD.exe Terminal V3.0) :

- Pentium, min 90Mhz, 16MB Memory, min. 30MB free disk space (HD)
- Colour screen, min VGA
- CD-Rom
- Windows 95, 98, ME or Windows NT 4.0 min. SP3, Windows 2000

Warnings

- **The enrichment unit is an analytical instrument! Avoid extreme environments and do not throw the instrument**
- **At the transfer ports, do not connect directly gases with over-pressure to the system if not specified (use T-connectors)**
- **Never introduce liquids into the system! Severe damage possible**
- **Avoid dusty environments (also liquid droplets of e.g. oil and water). Severe damage possible**
- **Keep in mind, that liquids (especially water) may condense onto the adsorbent material, if the adsorbent temperature is much below the sample gas temperature. Overload with liquids disturbs the sampling procedure significantly and may destroy internal parts**

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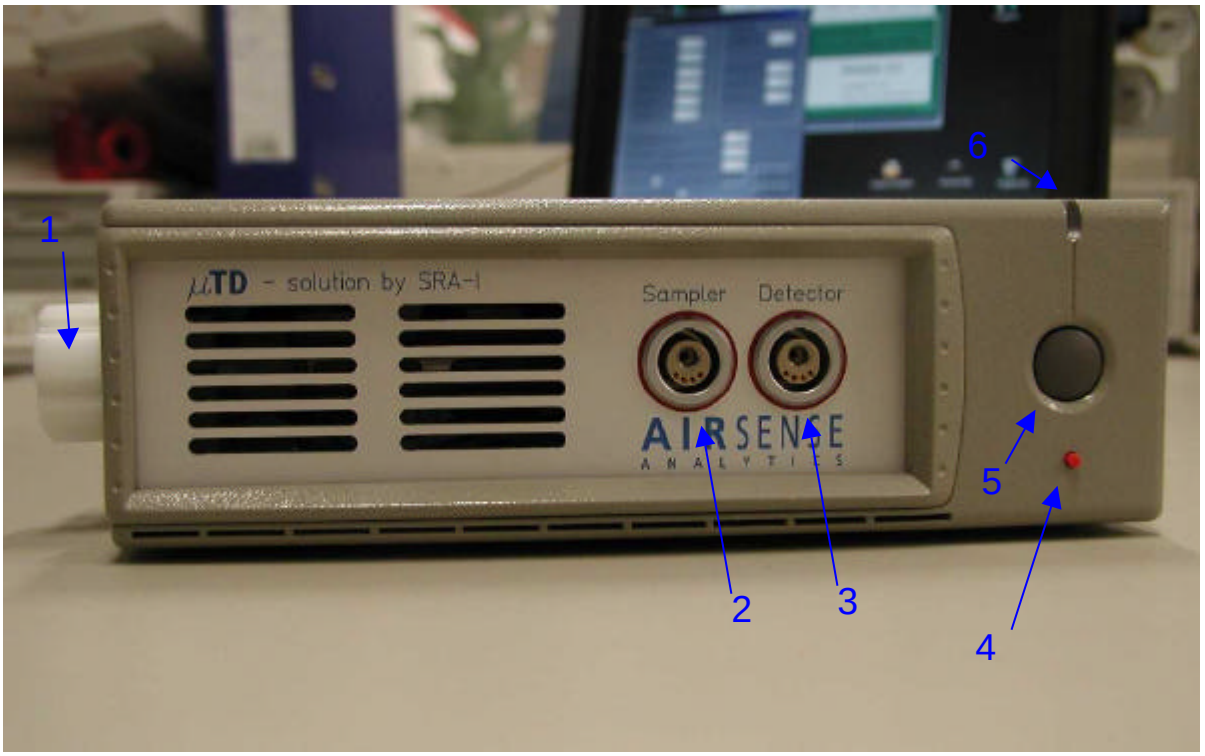
Hardware Installation Guide

Gas connections

Before starting please check the sample gas inlet (at the front), the ports at the back of the instrument to be sure that they are clean and free of dust or particles



Front of instrument with elements:

1: Adsorbent tube holder
2: Connector Sample Transfer Line
3: Connector Detector Transfer Line (to Micro-GC)
4: Start Button
5: Main switch (On/Off)
6: Status Lamp

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Connecting the transfer lines

Two heated transfer lines can be connected to the front of the instrument. The first is used as a sampling line, transferring the sample into the trap during sampling. The second one acts as a sample transfer from the trapping unit to the analytical instrument. The transfer line temperatures can be adjusted from the Windows software.

The orientation of the plug of a transfer line is marked by red dots on both sides of the connection. The plug is pushed ***gently***, but with some force, into the female connector. Too much force may destroy the O-ring at the male flow contact (see also chapter Maintenance).

The transfer line is connected at the Micro GC by the 1/16" Swagelok nut. In order to avoid cold spots at the connection, it is important, that the thermal isolation of the transfer line ends close to the Swagelok nut.

The sample may be provided by different ways :

- manually, the trapping unit sucks the sample from a sample vial
- automatically by a headspace sampler, which normally operates with overpressure used to transfer the sample into the adsorbent material.
- automatically from a process line, where self sampling or sampling by overpressure is possible.

The sampling flow can be adjusted from 50 to 500ml/min.

The detector may extract the sample from the adsorption tube after desorption by suction or the sample may be injected with external pressure connected to port "Zero Gas 2"at the back of the instrument. Description and use of the 4 gas ports at the rear front.

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The following 4 gas ports are situated at the backside of the instrument (refer to figure below) :

1. Zero Gas

This port is used for three purposes. *First* the gas for injection is taken from this connection. *Second* this gas is used for cleaning the tube after injection. And *third*, the gas from this port is needed for rinsing the adsorbent tube during the postsampling phase. This is done for purging off remaining amounts of solvent before injection (e.g. sweeping ethanol or water residues off).

In combination with analytical instruments based on metal oxide sensors clean air at a small overpressure (ca. 50hPa) should be applied, if the detector does not suck the sample itself during injection.

(A pressure with 1000 up to 3000 hPa overpressure may be used in combination with a needle valve in order to adjust the injection flow. Alternatively a flow controller (analytical specification) can be connected to this port. Injection flow may be adjusted between 1 and 150ml/min. Please contact Airsense for further support)

2. Waste

Waste gas from a headspace sampler (during injection phase) and the gas during cleaning mode is released through this port.

3. Zero Gas 2

During sampling the detector (electronic nose) may need a continuous flow of carrier gas. This carrier gas (clean air, He or N_2) is provided via this port. The unit will transfer this gas to the detector during sampling mode. Connect the required gas to this port via a needle valve or flow controller in order to maintain the flow, that is needed by the detector.

4. Reserved

Reserved for additional options.

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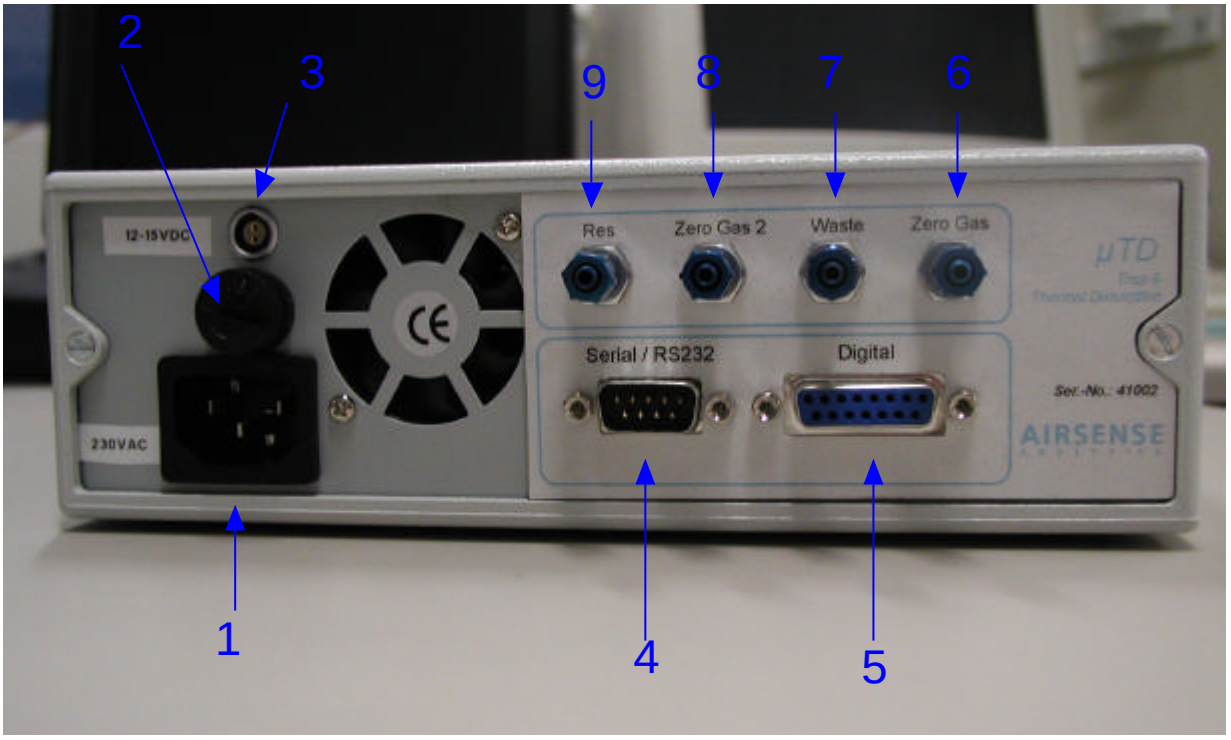

Backside of instrument with elements:

1: Connector Main Power (110-230VAC)
2: Fuse (5 A slow)
3: Connector 12VDC (optional)
4: Serial Interface (to PC)
5: Digital Port (see sec. Dig. Interf.)
6: Gas Port “Zero Gas 1” (tube 3mm)
7: Gas Port “Waste” (tube 3mm)
8: Gas Port “Zero Gas 2” (tube 3mm)
9: Gas Port (Reserved)

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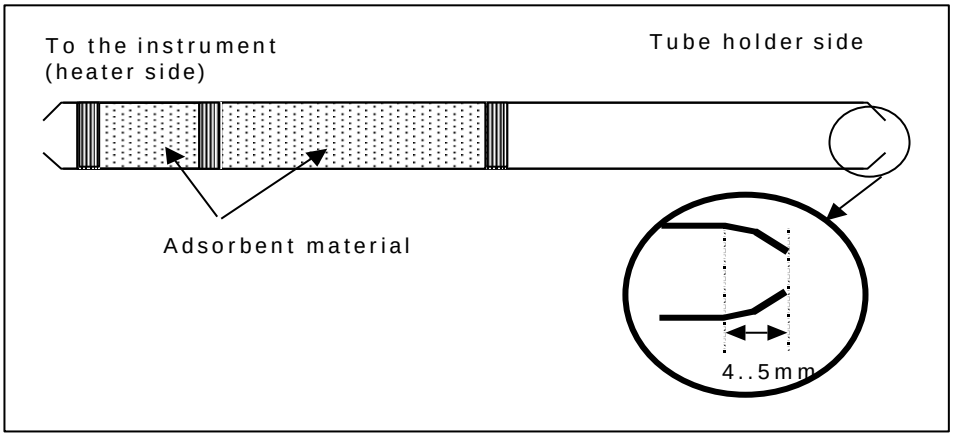
Adsorbent Tubes

- Adsorbent tubes should not be touched with bare fingers. Use a towel or clean lab glove.
- New adsorbent tubes have to be cut at the front and end, avoiding too long edges (see fig.2). Use proper glass cutting instruments. Be careful, injuries might occur. Do not insert tubes with sharp edges. Broken pieces will remain inside the desorber and influence the desorption procedure or even destroy the seals.
- To insert the tubes please A) open the adsorber by turning the tube counter-clockwise. B) First insert the adsorbent tube into the tube holder (the side without adsorbent resin!). C) Introduce the adsorbent tube together with the holder into the desorber and close it by pushing it gently and turning the holder at 45° to the right (the side with the resin should be introduced first).
- If a new tube is applied, it first has to be conditioned. This can be done by running at least three measurement cycles using the current µTD method.



a) attach tube to Holder

b) push tube into Desorber

c) shut desorber by turning clock-wise

Fig.: Adsorption tube (L=91 ± 1 mm, o.D.=8 ± 0.1 mm,
edge max. length = 4.5 ± 0.5mm, edge o.D.= 4.8 ± 0.3mm)

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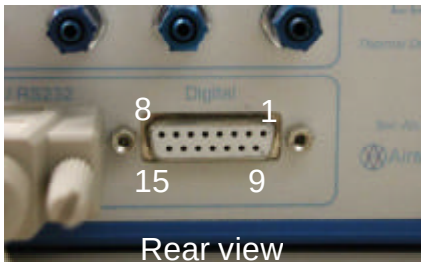
Electrical connections

- connect the serial interface cable (9-pin) to the serial port of the computer
- connect the digital interface (15-pin) to the Agilent Micro GC. (Different electrical setups may be recommended in specialized applications)
- connect the instrument to either 110VAC or 230VAC
- **Important:** replacement of fuse only with 20x5mm (IEC 127-2-3); 5 Ampere, slow.

Digital interface

- The Gas Chromatograph (Agilent 3000 Micro GC) just has to be connected with the digital interface cable provided.
- The system can be connected to different further sampling instruments or analytical equipment (GCs, detectors) by shortcut detection or TTL signals (see fig.3 for connector configuration).



15 Pin SUB-D
Connector
(female)

Fig. 3. Connector configuration

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Pin		Timing	Electrical Character-istic
11	GND		GND see Pin 3
3	INPUT START Shortcut -> Start TTD cycle	min 200msec	Shortcut input , at-tach relay contact
9	GND		GND see Pin 1
1	OUTPUT INJECTION Shortcut -> µTD injecting µTD switches valves for injection. Signal to be used to start aquisi-tion at Detector.	Contact closes at beginning of injection and opens at end of cool down phase /OR/ contact closes for five sec-onds (see chapter „parameter adjustment“)	Shortcut output, relay contact
10	GND (TTL)		GND see Pin 2
2	OUTPUT INJECTION „Low to High“ -> µTD injecting µTD switches valves for injection same function as Pin 1	Signal switches to „High“ at beginning of injection and „Low“ at end of cool down phase /OR/ signal switches to „High“ for five seconds (see chapter „parameter ad-justment“)	TTL output
12	GND (TTL)		GND see Pin 4
4	INPUT START optocoupled „High“-> Start TTD cycle	Impulse min. 100msec	TTL Input optocoupled Max.+5V, min. 1.6mA
13	GND (TTL)		GND see Pin 5
5	INPUT INJ-MOD optocoupled Low during start-> µTD master mode µTD injection will be carried out according to time settings High during start-> µTD slave mode (sytem waits after desorption until signal turns to Low (ready). Then injection is started)	Low continuously or Low Impulse min 100msec	TTL Input optocoupled Max.+5V, min. 1.6mA

Pin 5: ... may be used as „ready“ signal achieved from detector as described

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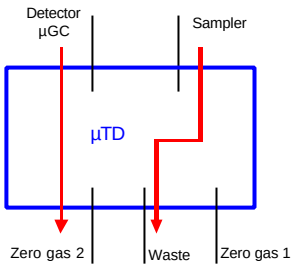
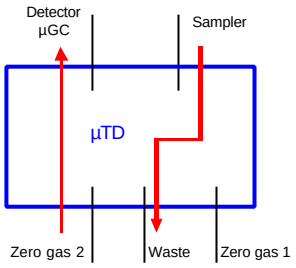
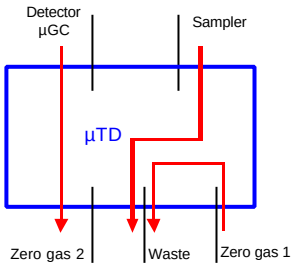
Flow configuration (with Agilent 3000 MicroGC)

The gas flow of the instrument in connection with the GC instrument is displayed in the following figures. Cleaned external air is required. Please connect the external gas supply (Zero gas and zero gas 2 port) as indicated in the following pictures. During analysis with the sampler the following different operating states are used:

Standby : The GC inlet is connected to port Zero Gas 2. The gas connection from the sampler is connected with the waste outlet. The adsorption tube is closed.	
Sampling: the internal pump is switched on and drives the sampling flow through the tube (flow rate defined by software). Compounds are trapped within the tube. The internal pump from the detector re-leases cleaned air.	
Postsampling: the internal pump is switched on and zero gas from the port Zero Gas 1 is passed through the tube to the waste outlet in order to remove gase-ous compounds with low boiling points (and water) from the adsorbent. The GC is connected to port Zero Gas 2. The gas connection from the sample is connected with the waste outlet.	

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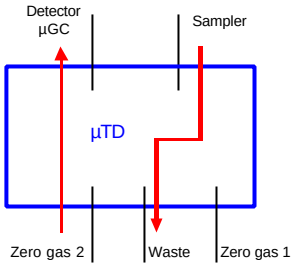
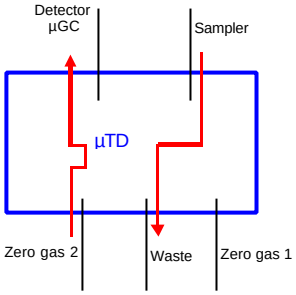
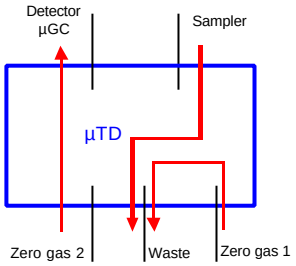
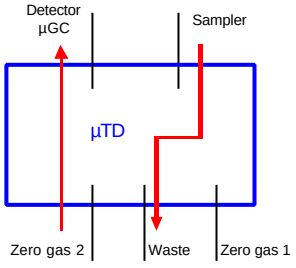
Desorption: the trapped compounds are released by heating the adsorbent tube to a defined temperature. During desorption there is no gas flow in the tube. The eventual gas flow from the sample(r) is released through the waste port. The detector (GC) is connected to port Zero Gas 2.	
Injection: the trapped compounds are transferred from the tube into the detector (GC) due to the sampling pump of the GC. (Alternatively, this flow may be driven by a flow controller attached to port Zero Gas 2) The eventual gas flow from the sample(r) is released through the waste port.	
Cleaning and Postcleaning: the temperature of the trap is increased according to the given cleaning temperature. The internal pump forces reverse flow of clean gas through the tube to the waste outlet. The eventual gas flow from the sampler is directed to waste. During a postcleaning step possible internal contamination is removed.	
Cooling: cooling fans are used to cool the tube until a defined temperature is reached for starting the next sampling step. The gas flow directions are the same as in the standby step.	

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Software Installation

Please end all programs that may be open on the computer system.

Insert the installation CD into the CD-player of the computer and run ***"setup.exe"*** from its top layer. This is achieved by opening the windows explorer and the view related to the CD. Double click on the icon for setup.exe.

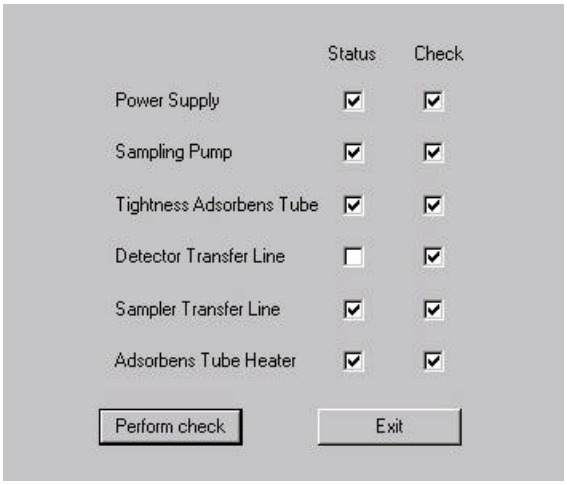
Follow the instructions during the setup process.
There is no need to reboot the computer after the software installation.

Trapping with µTD

Start of instrument, Self Check, Equilibration

If the computer shall be attached during operation (for supervision or parameter adjustment), the instrument has to be switched on in advance of starting the software.

After switching on the instrument, it performs a self check procedure. This is a supervision of the instruments status, like tightness of the flow system, power supply function, transfer line heaters and so on. If the self check – lasting for ca. 2 minutes ends successfully the instrument enters the initial equilibration phase (ca. 5 minutes). All temperatures are being adjusted to their given values. The adsorbent tube maintains sampling temperature.



If both phases end successfully, the light at the front turns green and the instrument is ready for operation. If not, the light stays red and an error message is displayed within the controlling program. The start up procedure is carried out in any case - disregarding whether a PC is attached to the device or not.

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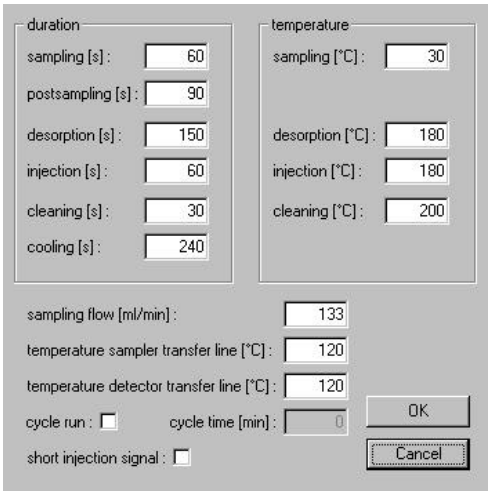
Parameter Adjustment, Start of Measurement Cycle

By simply connecting the inlet port of the µTD via its transfer line with the sampler, process or environment, automated measurements are possible.

- switch on the µTD instrument
- start the program from the Windows “Start” menu

To adjust parameters and to start the system for a **single run**:

- wait until the systems ready signal (“green light”)
- Check the sampling parameters displayed under the menu items ***<Options>*** and then ***<Parameter>***. The values of the parameters are displayed within a dialog box. The sequence of these parameters correlates with the steps of the for the trap/thermal desorption procedure.



- Adjust the parameters according to your application if required.
- Disable the check button “cycle run”
- Press “Ok” – the software checks the adjustments and may inform about invalid or inadequate adjustments. If no faults are detected, the software prompts for download of the parameters to the instrument.
- Carry out adjustments at the detector (MicroGC) accordingly and put it into standby operation mode.
- Start the system either by clicking on the button "GO" in the taskbar or by pressing the start button at the front of the instrument. The front light of the instrument turns red and the procedure starts with the sampling phase.

The µTD performs the steps of the Trap and Thermal Desorption cycle. At the beginning of the injection phase, the GC procedure (sampling and measurement) is started by a start signal provided by the µTD.

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Note: The duration of the start signal for the detector is depending from the switch "short injection signal". With this switch turned off the signal lasts until the whole TTD cycle and may enforce the GC to start its procedure a second time (see chapter "digital interface").

The TTD cycle ends, when the sampling temperature is reached during cool down. The "ready" signal will return and the cycle may be started again.
If the sampling temperature is not reached during cool down – due to high environmental temperatures – the cycle will end when the cool down time (entered as a parameter) elapses.

Note: It is important to enter a value high enough for the cool down phase in order to ensure, that samples are trapped always at the same temperature.

Note: If the sampling temperature for the TTD cycle is changed, the system will enter a new equilibration phase. After that, the instrument will return to standby mode, showing a green light at the front

To start the system for **cyclic operation**:

- Follow the same steps as for single cycle operation described above.
- enable the check button "cycle run" and enter a value for the cycle time, which means the time between two repetitions of the trap and thermal desorption cycle. If the time for the repetition is shorter than the trap and thermal desorption cycle the software will display a message, accordingly.
- The whole cyclic operation is started by the button "GO" from the task bar or by pressing the start button at the front of the instrument.

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Hints and Troubleshooting

What sampling material should I use and how do I know the efficiency of the trap?
For most applications Tenax is the adsorbent material we recommend. Depending on the interaction of the substance to analyze and the sampling material, different safe sampling volumes can be determined. These breaktrough volumes (defined as sampled gas volume per adsorbent material (l/g) before 50% of the original concentration is seen at the detector) are listed for different analytes and adsorptive materials. For a specific compound it is possible to choose the optimal trapping material. It is important to keep in mind that when using other adsorbent material condensation of water could be a problem. The new adsorptive material should be approved for thermal desorption.

When do I have to change the sampling tube?
When using Tenax generally many hundred sampling and desorption cycles are possible. Please check for the color of the adsorptive material, which should be white. If there are yellow spots (due to extreme temperatures) or other visible changes (soot, etc.) please change the tube. By using a defined standard (e.g. 10 ppm Toluene in air) and performing the same measurement in greater intervals the efficiency of the trapping material over time can be also monitored.

How can I check the system for tightness?
The system itself checks most of the flow system for tightness by running the self check. However, some pathways - like the transfer lines can not be checked automatically. The best way is to test these connections is to look with a flow meter for the correct flow rates, during sampling or injection. If no flow meter available it is also possible to hear if the noise and frequency of the sampling pump is increased during the sampling phase when a restriction closes the front of the sampling line (inlet).

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Maintenance

A) Tightness of transfer lines

If the sampling flow decreases or is less than adjusted in the parameter dialog, the o-ring at the flow contact in the plug of a transfer line may be damaged. This is due to installation of the transfer line with too much force. The exchange of the O-ring can be carried out easily – please contact the manufacturer for a description and spare parts.

B) Broken sampling tubes

Broken glass within the desorber may cause leakage of the flow system and damage of the tube sealing. It is important to remove parts and particles of glass from the tube holder or the internal desorber. This can be done by the use of small tools without sharp edges.

C) Cleaning

When using the system with very dirty samples most of the pollution will remain inside the (sampler) transfer line. This may be cleaned by rinsing it with toluene and methanol followed by intensive drying with clean air (filtered by active charcoal). It is important that all liquids are removed prior to reinstallation of the transfer line. The further parts of the system are cleaned by operating the system at elevated temperatures for a number (e.g. 5) of cycles – providing clean gas at the inlets (sampler transfer line and zero gas ports) and having a prolonged cleaning time (e.g. 300 seconds)

If still pollution can be seen by running measurements with clean gas, it is recommended to contact the supplier for a service.

D)

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Technical Data

Adsorbent tube	one tube, can be easily replaced
Adsorbent material	different, commercially available tubes, commonly Tenax TA® , 125mg
Desorption temperature	Adjustable, up to 300°C
Sampling temperature	Adjustable, min 30°C
Sampling flow	Adjustable, 0,05 – 0,5L/min
Desorption flow	Adjustable, 2ml/min - 200ml/min
Power	max. 80W, 110-250VAC, (12VDC opt.)
Size	210x260x60, 2.3kg
Sampling Inlet	Plug with electrical and gas contact, transfer line
Interface electrical	TTL or relays digital signal port for communication with devices attached to the unit
Heating rate	80°C/sec (heater, temp needs around 90sec to penetrate adsorbent bed)
EMC, CE	Electrical security is provided by built in power supply following the standards: IEC61010-1

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