MAT 253

OPERATING MANUAL

Issue 04/2002

Ident. No. 114 9090

Thermo Finnigan

Übereinstimmungserklärung gemäß EN 45014
declaration of conformity according to EN 45014
Dichiarazione di conformità alla EN 45014

Name des Herstellers:
manufacturers name:
nome produttore:
Thermo Finnigan GmbH

Adresse des Herstellers:
manufacturers address:
indirizzo produttore:
Barkhausenstraße 2
28197 Bremen
Germany

erklärt, daß das Produkt
declares that the following product
dichiara che il seguente prodotto

MAT 253

mit den folgenden Produktspezifikationen übereinstimmt:
complies with the following product specifications:
rispetta le seguenti specifiche del prodotto:

EMV (Störemissionen):
EMC (emissions):
EMC (emissioni):
EN 50081-1,
EN 55022, Kl. B

EMV (Störfestigkeit):
EMC (immunity):
EMC (immunità):
EN 50082-2,
EN 61000-4-2, -3, -4, -5, -6, -11
EN 50204

Elektrische Sicherheit:
electrical safety:
sicurezza elettrica:
EN 61010-1

Ergänzende Informationen:
complementary information:
informazioni complementari:

Dieses Produkt erfüllt die EMV-Richtlinie 89/336/EWG und Niederspannungsrichtlinie 73/23/EWG.
This product complies with the EMC directive 89/336/EEC and the Low Voltage Directive 73/23/EEC.
Questo produtto rispetta la direttiva 89/336/EEC e la direttiva 73/23/EEC.

Bremen, Germany, 26 April 2002

Der Betriebsleiter:
Operations Manager:
Direttore fabrizione:

ThermoFinnigan

Thermo Finnigan MAT GmbH
Postfach 1401 62
28088 Bremen
Germany

Thermo Finnigan

Reparatur-Begleitkarte*)
Repair-Covering Letter

Absender:
Despachter:

_____

_____

Geräte-Type:
Instrument Type:

_____

Service-Nr.:
Service No

Sie erhalten zur Reparatur unter unserer Bestell-Nr.:
You receive for repair under our order no.:

Festgestellte Mängel oder deren Auswirkung:
Established defect or its effect:

Bitte detaillierte Angaben machen / Please specify in detail

Ein Austauschteil haben wir erhalten unter Kommissions-Nr.:
An exchange part already received with commission no.:

Ja/Yes ☐ Nein/No ☐

Die Anlage ist außer Funktion
The system is out of function

Ja/Yes ☐ Nein/No ☐

Durch die nachfolgende Unterschrift bestätige(n) ich /wir, daß die o.g. Teile frei von gesundheitsschädlichen Stoffen sind, bzw. vor ihrer Einsendung an Thermo Finnigan MAT dekontaminiert wurden, falls die Teile mit giftigen Stoffen in Verbindung gekommen sind.

By signing this document I am/ we are certifying that the a. m. parts are free from hazardous materials. In case the parts have been used for the analysis of hazardous substances I/we attest that the parts have been decontaminated before sending them to Thermo Finnigan MAT.

_____
Datum / date

_____
Unterschrift / signature

*) Bitte vollständig ausfüllen / Please fill in completely

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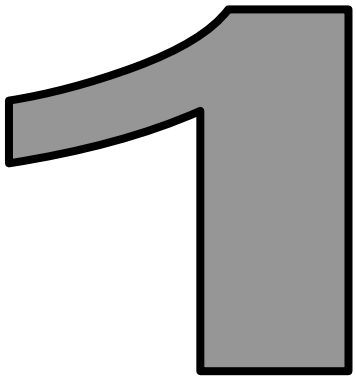

GETTING STARTED

Issue 04/2002

Thermo Finnigan

1.1 INTRODUCTION

This manual describes the functions and the fundamental measuring procedures of your Thermo Finnigan ***MAT 253*** mass spectrometer. In addition, specific manuals for the purchased peripheral equipment are supplied.

To obtain a good understanding of the complete system, it is necessary to study the operating manual and the online-help of the software before starting up your instrument. A basic knowledge of handling computers and of the ISODAT NT software is assumed for proper operation of the Thermo Finnigan ***MAT 253***.

To reach a high level of performance with the Thermo Finnigan ***MAT 253,*** we recommend making use of the operator courses provided by us at our facilities in Bremen, and/or onsite.

For more information, please contact your local Thermo Finnigan MAT service office or contact directly:

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Phone: +49-421-5493-0
Fax: +49-421-5493-396
Internet: www.thermo.com

1.1.1 BASIC INSTRUMENT

With the Thermo Finnigan ***MAT 253***, you can measure gas isotope ratio of

H/D, ^{13}**C**/^{12}**C**, ^{15}**N**/^{14}**N**, ^{18}**0**/^{16}**0**, ^{32}**S**/^{34}**S** (from $\mathbf{SO_2}$ and $\mathbf{SF_6}$), ^{28}**Si**/^{29}**Si** as well as **Ar**, **Kr**, and **Xe**.

The ***MAT 253*** provides a flexible and open platform for the connection of inlet systems and preparation devices. Thermo Finnigan-supplied inlet systems are automatically recognized by

a "plug and measure" concept. In addition, the system is open for easy connection and control of custom inlet/preparation systems.

The basic instrument is equipped with a dual inlet system for classical applications. It has a modular design for the adaptation of different inlet modules. This enables a configuration of the instrument tailored to the requirement of the users.

On the sample side can be connected:

- a second inlet system for variable sample volumes, a multiport with or without a tube cracker installation,
- microvolumes for very small samples,
- multisample inlets for separating and purifying samples or,
- installations for "on-line" coupling to gas chromatographs, elemental analyzer or other peripherals.

The configuration of the inlet systems is described in this manual. Detailed information about other inlet systems such as the "on line" coupling to gas chromatographs or to elemental analyzers you will find in the manual describing the peripheral equipment.

Please make yourself familiar with all the controls on front and all the connection and installations on the rear of your instrument.

Fig. 1-1: Front view of the ***MAT 253***

1.1.2 ISODAT NT SOFTWARE

ISODAT NT is a software suite for system control, data acquisition and data evaluation that is an integral part of the system architecture.

- **System control**

  All aspects of the mass spectrometer are controlled by software, including ion generation, mass separation and ion detection. Control of the ion source allows manual tuning, auto tuning, as well as storage and retrieval of ion source parameters. Different configurations representing different analytical setups can be stored and retrieved. Up to eight simultaneous data acquisition streams are supported.

- **Automation**

  The system is designed to fully automatically execute pre-defined procedures and run sequences of analyses, including customized reporting.

- **Open architecture**

  ISODAT Script Language (ISL) is the tool giving the expert user full access to the mass spectrometer, the inlet systems and additional user-supplied devices. An input-output module allows connection and control of up to five interfaces. Scripts can be developed for customized applications.

- **Data evaluation and display**

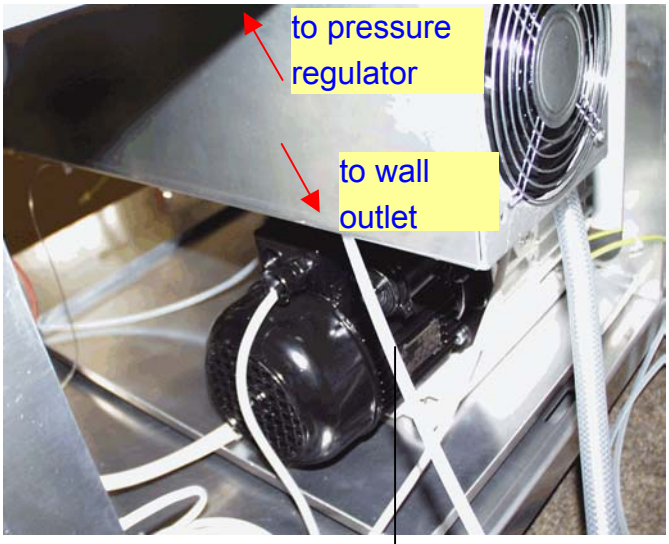
  ISODAT NT provides a comprehensive set of customizable data evaluation routines. Standard report forms are provided according to the application. In addition, reports can be easily customized using ISODAT NT's unique Result Workshop.

1.2 GETTING STARTED: HARDWARE

This paragraph explains the hardware related steps to be performed before any measurement can be started.

First steps

- Unpack your IRMS and arrange it at the desired place in your laboratory.
- Connect the end of the hose for compressed air (i.e. a thin transparent hose) that leaves the IRMS at its rear panel to your wall outlet or a compressor for compressed air. You need at least 5 bar.



hose for compressed air

- leaves IRMS at its rear panel
- leads to wall outlet for compressed air and manometer

The other end of the hose for compressed air must be connected to the pressure regulator behind the front panel of the IRMS as shown below.

hose for com-pressed air

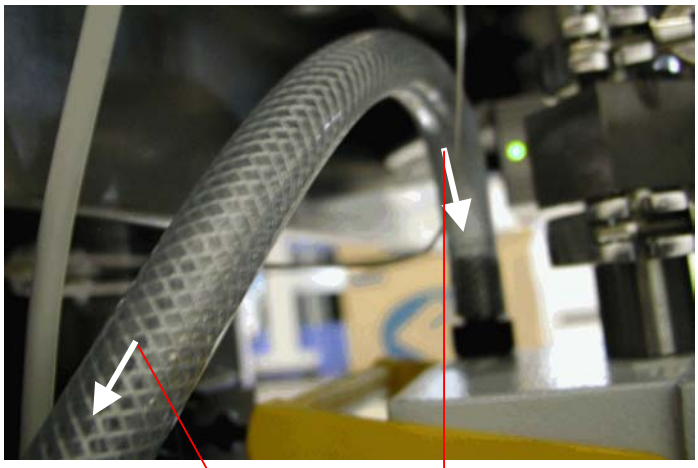
pressure regulator

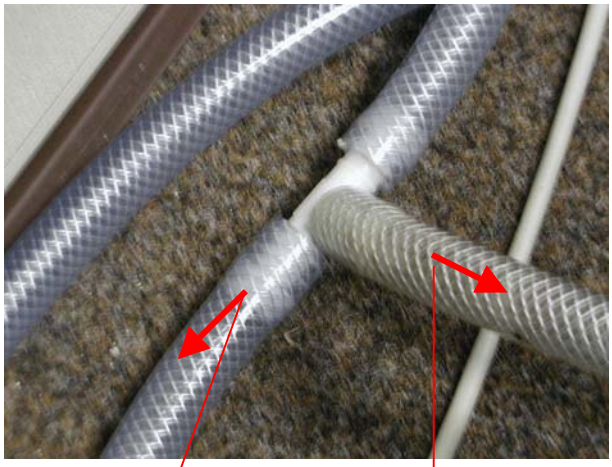
distributor with ports

Usually, the pressure regulator is factory-set via the pressure regulating screw to display 4 bar. To vary the pressure, pull out the pressure regulating screw and turn it.

➢ Insert your peripherals at the distributor (down right behind the front panel). As the ports of the distributor are equivalent, it is unimportant which peripheral to insert at which port.

➢ Lay the waste gas tube (at the output of the vacuum pumps) outdoors in order to prevent accumulation of oil mist and perilous gases (e.g. CO, H_2).

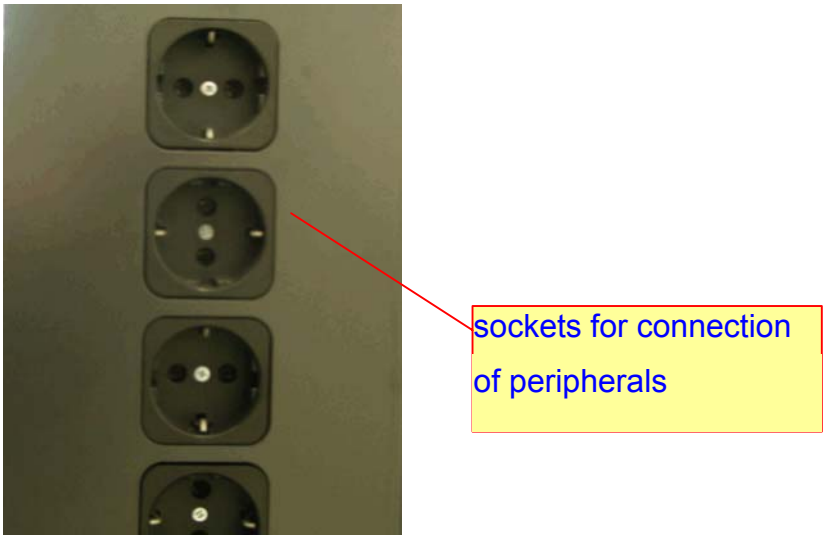




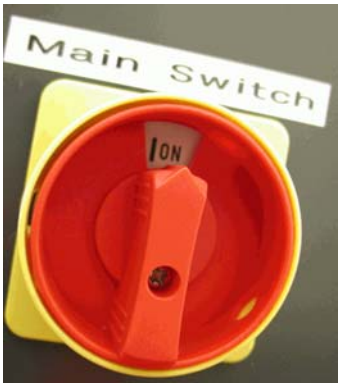
Power supply

➢ Connect your IRMS to the power supply.

- At the rear side of the instruments are sockets to connect peripherals, if available. For first tests, the IRMS is checked without any peripherals connected to it. Later on, the peripherals are connected one at a time.



- Turn on the IRMS by setting the main switch to position ***ON***. The main switch is located at the electronic cabinet's rear panel.



Pumping system

Three Fore Vacuum Pumps are used:

- Pfeiffer “Duo 5” for the Source (**1**)
- Pfeiffer “Duo 2.5” for the Analyzer (**2**)
- Pfeiffer “Duo 2.5” for the Inlet System (**3**)

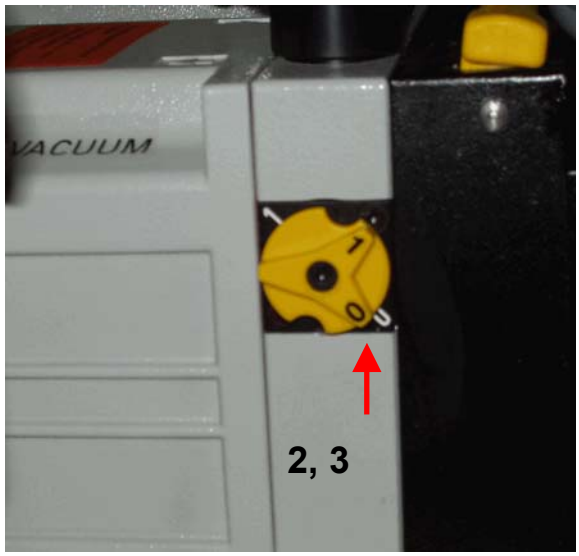
For detailed information - e.g. concerning handling and maintenance - refer to the Pfeiffer Operating Instructions of your pumps.

Before starting the pumping system, it is assumed that:

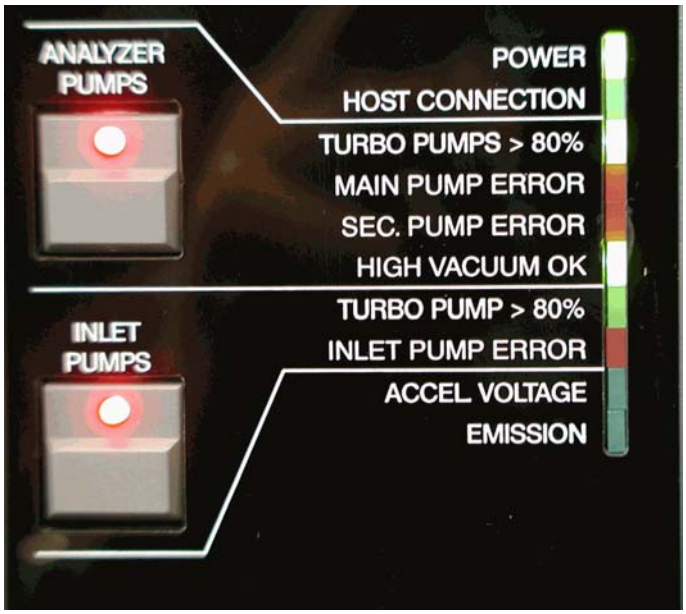
- the Fore Vacuum pumps are filled with oil,
- they are connected to the power supply, and
- their gas ballast is shut.

Normally, the fill level of the oil must range between the upper and the lower line, optimally at half height of the level indicator. A total oil change is recommended twice a year (refer to the Pfeiffer Operating Instructions of your pumps).

The gas ballast is shut by turning the switch to position **0**. In case of Pfeiffer “Duo 5”, i.e. (**1**) the switch is located on top of the pump. In case of Pfeiffer “Duo 2.5”, i.e. (**2, 3**) the switch is located sidewise (refer to the Pfeiffer Operating Instructions of your pumps).



The Control Panel at the front panel of the electronic cabinet shows switches for operating the pumps. The corresponding LEDs are described below.



- Turn on the pumps by pressing the ***Analyzer Pumps*** switch.

  The ***Inlet Pumps*** switch must only be additionally pressed in case of a Dual Inlet System.

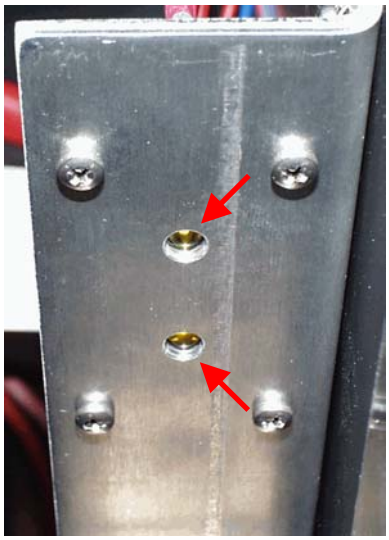
NOTE: ***All the LEDs will still be turned off with the exception of the Power LED, which will be red.***

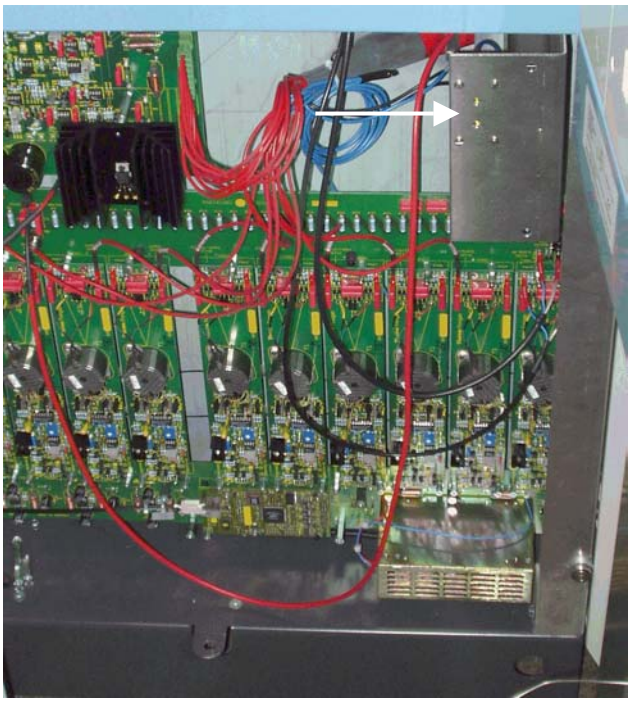
- After 15 min, the green LEDs ***"Analyzer Pumps: Turbo Pumps > 80%"*** and ***"Inlet Pumps: Turbo Pump > 80%"*** must be on.
- If one of the turbo pumps does not reach 80% of the rotation speed after a specific period of time, the pumping system will shut down automatically.
- The red LEDs "Main Pump Error", "Sec. Pump Error" and "Inlet Pump Error" indicate errors concerning the turbo pumps (e.g. after about 15 min, the security threshold of ≈ 3 * 10^{-5} mbar has not been reached).

NOTE: ***If properly functioning, the red LEDs must not be on!***

Source heater

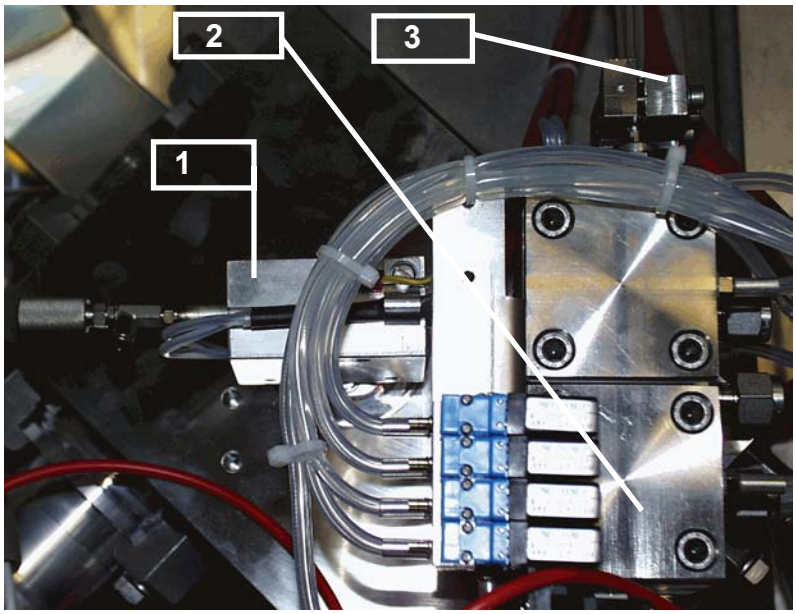
The LEDs of the source heater are located at left side of the IRMS, in the top right corner. When the source heater is turned on, both LEDs must be on. Otherwise, one of the heaters might be defective.





Heater of the Changeover valve and/or needle valve

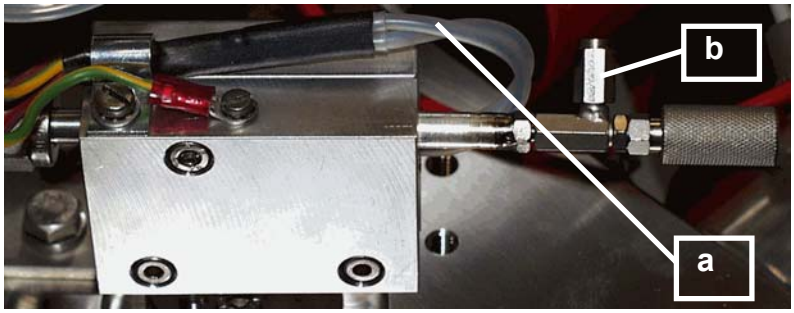
Switch on the heater of the Changeover valve and/or the heater of the needle valve (in case of Continuous Flow applications). This will help to prevent water condensation.



1 needle valve including needle valve heater

2 Changeover valve (COV) including Changeover valve heater

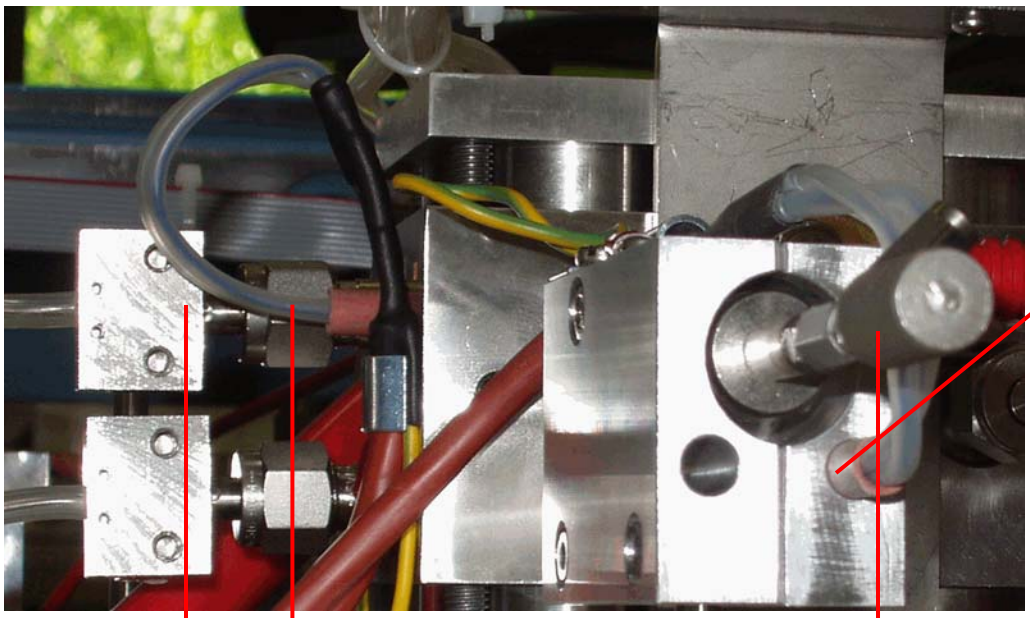
3 device for crimp adjustment



needle valve including needle valve heater (side view)

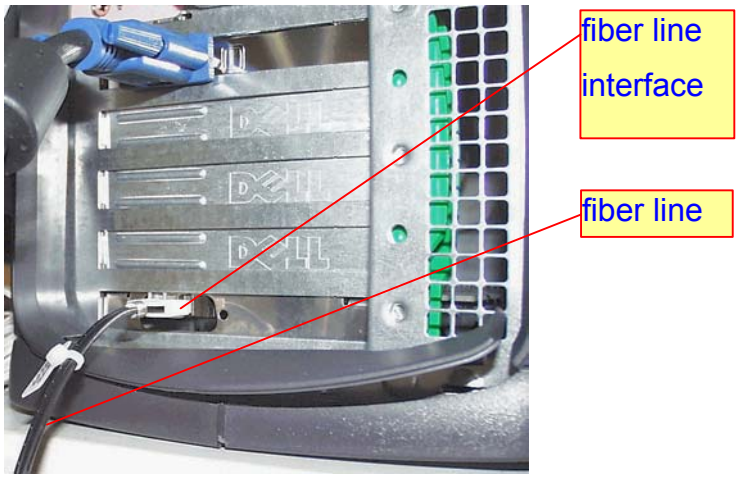
a to needle valve heater

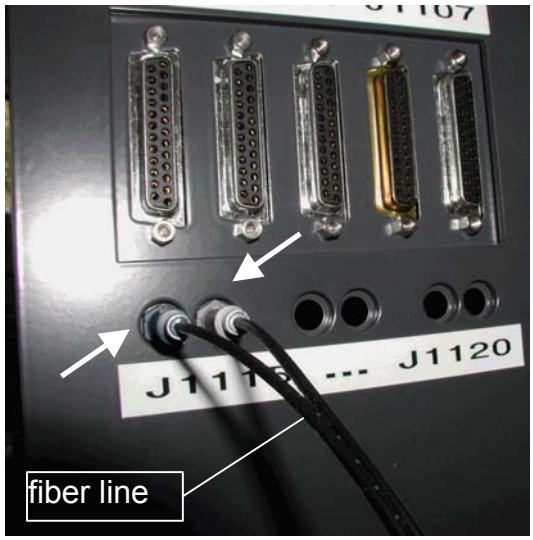
b port for capillary



IRMS - computer connection

To ensure data transfer between IRMS and computer, connect the fiber line to the respective port at the computer's rear panel.





- Connect the other end of the fiber line to the IRMS by inserting the blue plug into the blue connector and the gray plug into the gray connector.

- To verify whether the quality of the established vacuum is sufficient, start ISODAT NT by double-clicking on the ISODAT NT Icon on your desktop.

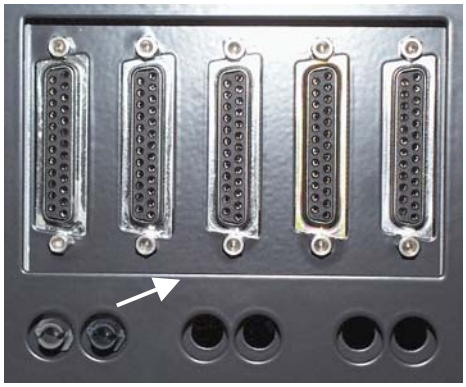
For detailed information refer to the ISODAT NT Help System.

When connected, the LED ***"Host Connection"*** at the front panel will be on.

NOTE: ***If the "Host Connection" LED does not gleam with ISODAT NT being started, no connection between IRMS and computer has been established.***

IRMS-peripherals connection

- To connect peripherals to the IRMS use one of the five identical SUB D ports at the rear panel of the IRMS.



- Peripherals are identified automatically by a ***plug and measure concept*** (see Chapter 4.4).

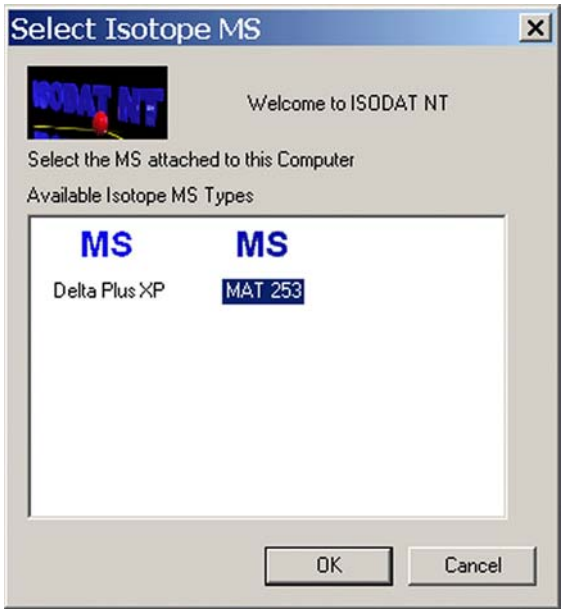
- If ISODAT NT is trying to get access to a peripheral and cannot identify it, an error message occurs (refer to the ISODAT NT Help System).

Start Configurator

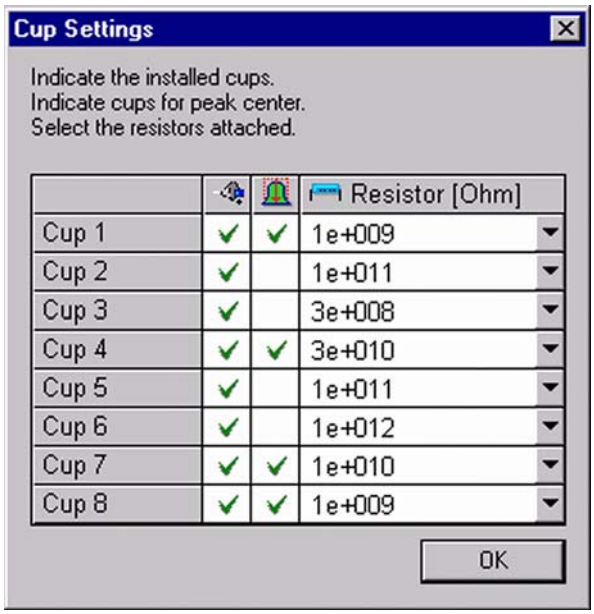
- To select a suitable (Hardware) Configuration containing your hardware equipment, i.e. your IRMS and the peripheral devices, start the ***Configurator*** by a double-click on the ***Configurator*** Icon.

First, ISODAT NT requires some information concerning your hardware equipment:

When starting the Configurator the very first time (or after reset of the IRMS), your type of IRMS is required. Then press ***OK***.

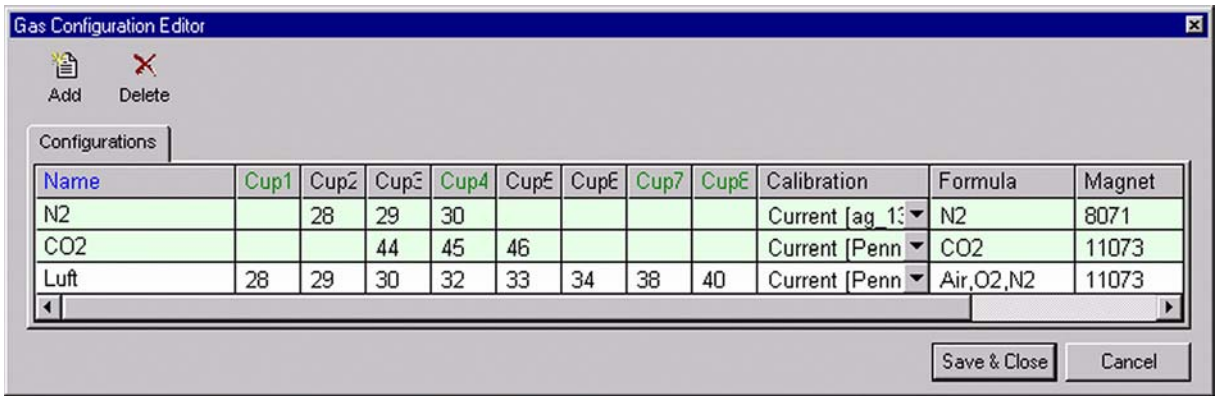


- Select your IRMS type.
- Press ***OK*** to confirm.

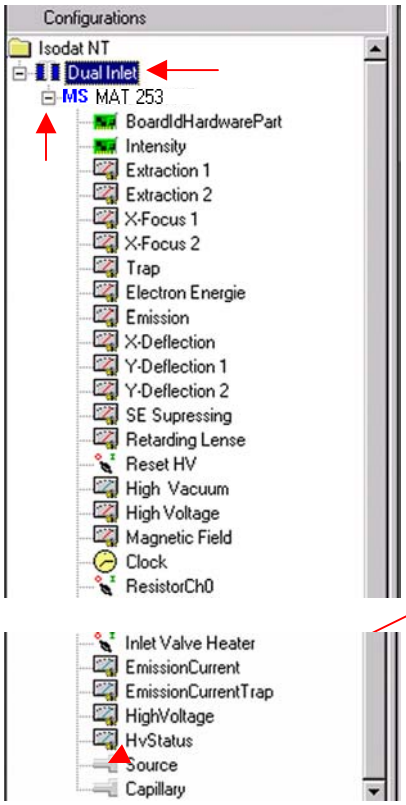


- Check, whether the correct cups are installed.
- Check, whether the correct cups are available for peak center.
- Check, whether the resistor values are correct.
- Confirm by ***OK***.

NOTE: ***Up to eight channels can be used simultaneously!***



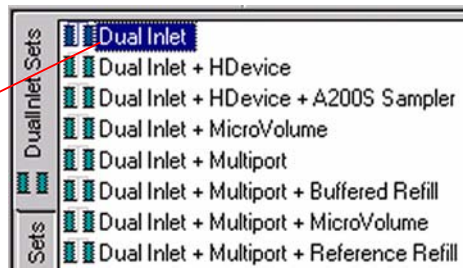
- Check the available Gas Configurations. You can edit them by clicking on the fields (for detailed information refer to the ISODAT NT Help System).
- Finally, press ***Save & Close***.
- Select one of the Configurations preset by Thermo Finnigan MAT or add a ***new Configuration*** using the ***Add Configuration*** button. 



Give it a significant name (e.g. ***"Dual Inlet"*** in case of a ***MAT 253*** with a Dual Inlet System).

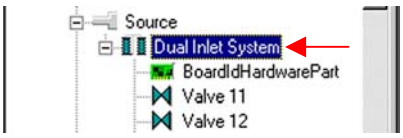
Open the tree structure by a click on ⊞ at ⊞ MS MAT 253

On the right pane, select ***Dual Inlet***.

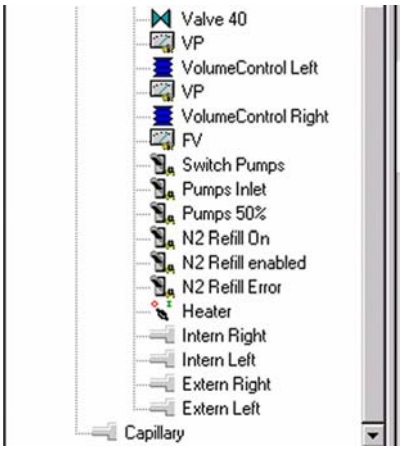


Drag it to the Source port of the new Configuration.

A ⊞ sign appears:



The ***Dual Inlet*** device has been attached to the ***Source***.



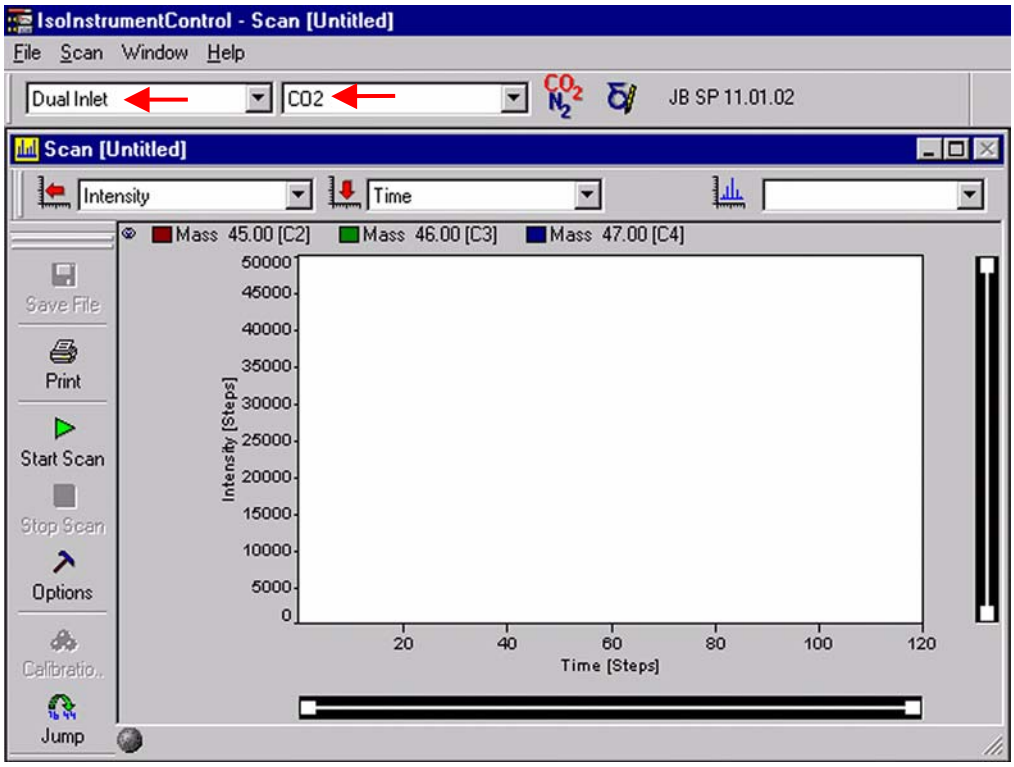
Close the Configurator window.

All settings will be saved automatically.

Start Instrument Control

Start Instrument Control by double-clicking on the ***Instrument Control*** Icon.

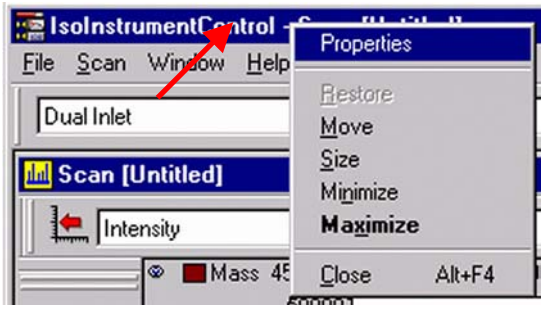
The ***Scan*** window appears.

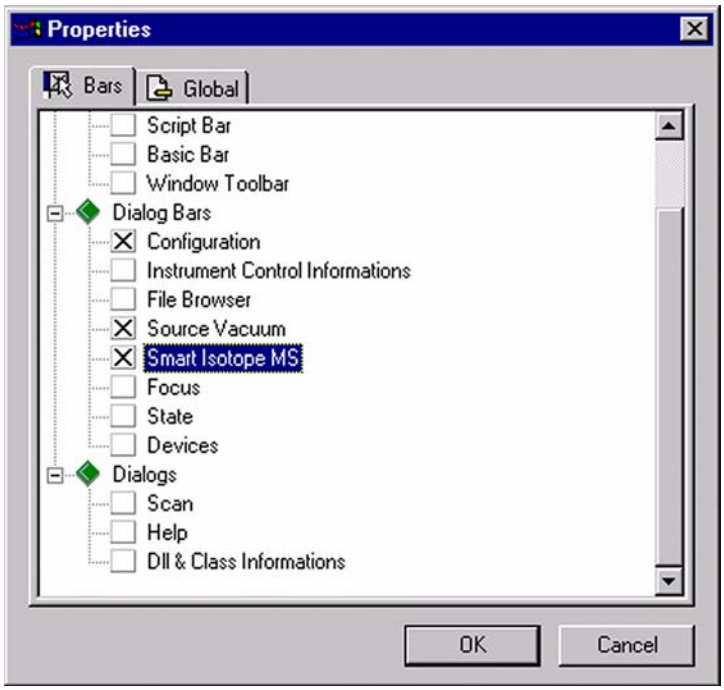


- Select your Configuration, e.g. ***Dual Inlet***.

- Select your Gas Configuration, e.g. ***CO_2***.

- Right-click on the Instrument Control window's ***title bar***. Select ***Properties***.





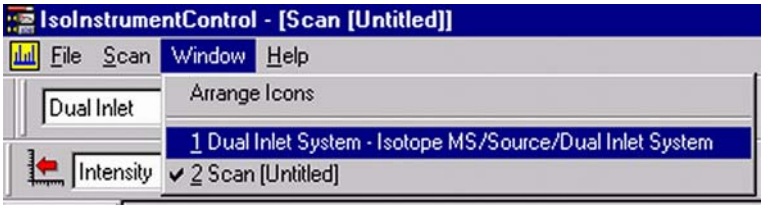
- In the ***Properties*** box, activate ***Configuration***, ***Smart Isotope MS*** and ***Source Vacuum***.

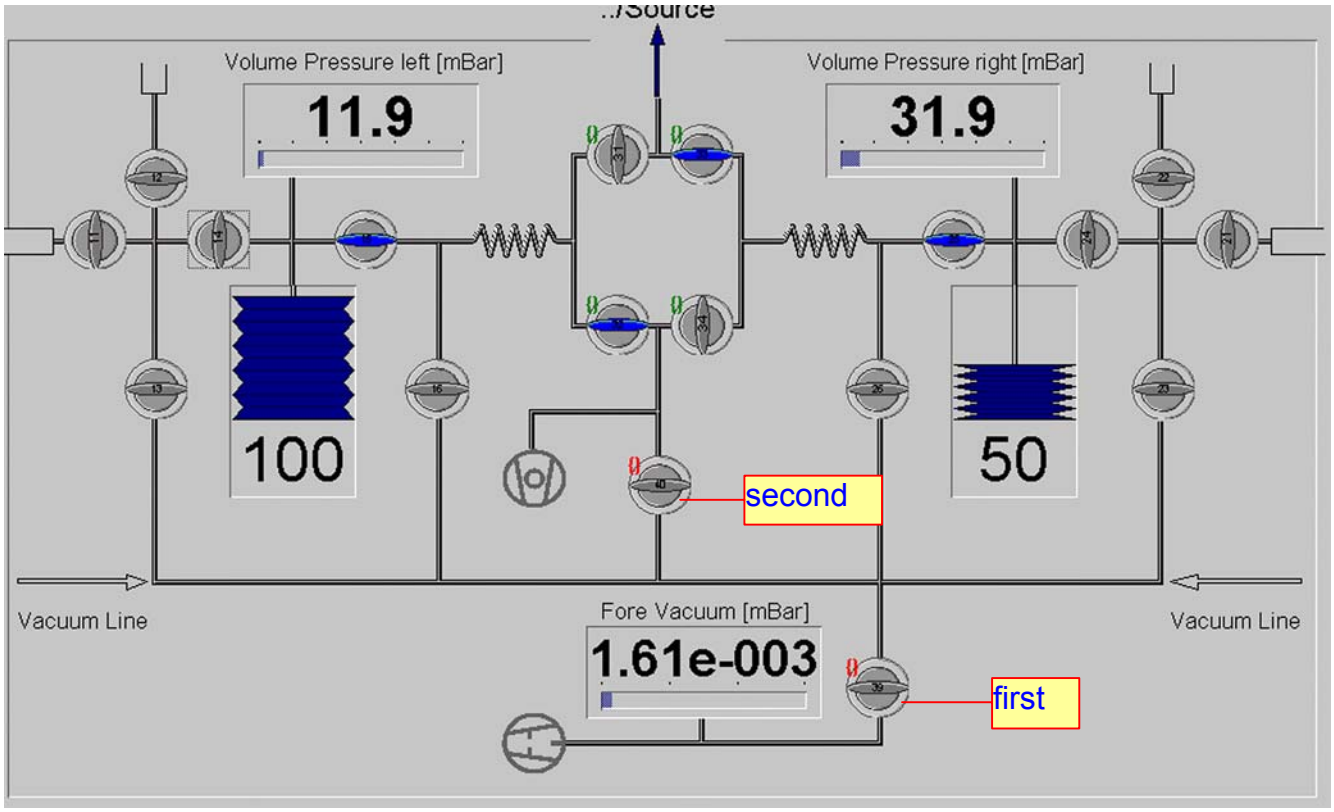
- Then confirm by ***OK***.

- Check the quality of the vacuum being established: the ***HV Source*** display reveals the current source vacuum in mbar.

Evacuating the Dual Inlet system

Activate the ***Dual Inlet System*** window by clicking on ***Window > Dual Inlet System***.





The individual parts of the Dual Inlet System must now successively be evacuated:

- Usually, ***standard*** gas is connected to the ***right*** inlet side, while the ***sample*** gas is connected to the ***left*** inlet side.

- Open the respective valves and evacuate the feed lines between gas reservoir and IRMS (e.g. capillaries), which are still at atmospheric pressure. After evacuation close them again.

- After a small waiting period in order to prevent gas fractionation successively fill the feed lines with CO_2 from the reservoir (e.g. the capillary). Beginning at the outside, proceed stepwise from valve to valve directed inward. It is not recommended to let the gas flow directly into the source.

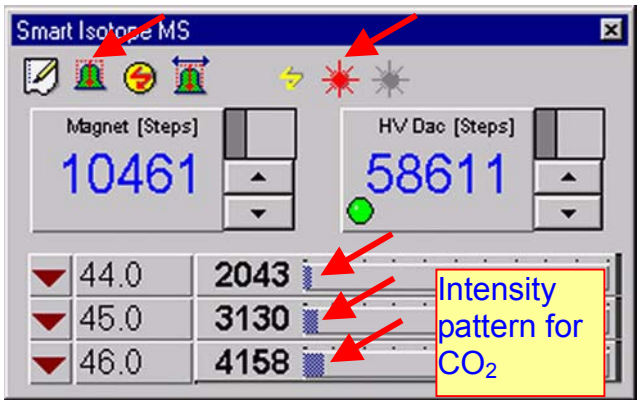
Thus, the gas finally enters the source. The intensity pattern characteristic for CO_2 is displayed at the ***Smart Isotope MS*** bar: the intensity for mass 44 is smaller than the one on mass 45, which in turn is smaller than the one on mass 46.

NOTE: ***Before starting a measurement, after pumping 15 minutes, a pressure between the lower 10 $^{-6}$ mbar and the upper 10 $^{-7}$ mbar range should be reached.***

This pressure should even drop between the lower 10 $^{-7}$ mbar and the upper 10 $^{-8}$ mbar range as pumping continues for a longer time (if all valves are closed).

If after 15 minutes of pumping a pressure of 1 * 10 $^{-5}$ mbar cannot be reached, probably a leak exists. As a safety measure, the cathode turns off at pressures above 3 * 10 $^{-5}$ mbar.

- Open the needle valve (if you have got one).



➢ Then turn on the ion source via the Icon at ISODAT NT's ***Smart Isotope MS*** bar

➢ In case of a Dual Inlet System, open it at one side.

➢ If you got a signal on all cups, perform a peak center by clicking on the Icon. Thus, the signal intensities should yield the pattern type shown above.

If they don't (or if an error occurs during peak center), perform a Calibration using CO_2 (if you calibrated the IRMS before, a peak center may be sufficient).

Principle: Only high voltage (i.e. the HVDAC value) is changed in this subtle HVDAC Scan.
The magnet jumps to a certain BDAC value specified (e.g. 10145 BDAC steps).
As a result, the magnet finds exactly the peak of interest.
The BDAC values of a particular gas (e.g. 12071 BDAC steps in case of CO_2) have first and foremost been stored in the Gas Configuration (e.g. ***CO2***).
The peak center runs according to the BDAC values specified there.
For each mass, at least one peak center will be performed (default: three peak centers for each mass).

Mass Calibration

➢ In Instrument Control's ***Scan Window*** select your (Hardware) Configuration,

e.g. ***Dual Inlet*** in case of a Dual Inlet System. 

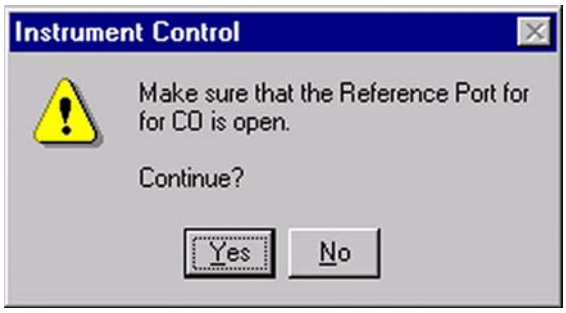
- Select your Gas Configuration, e.g. ***CO2***.

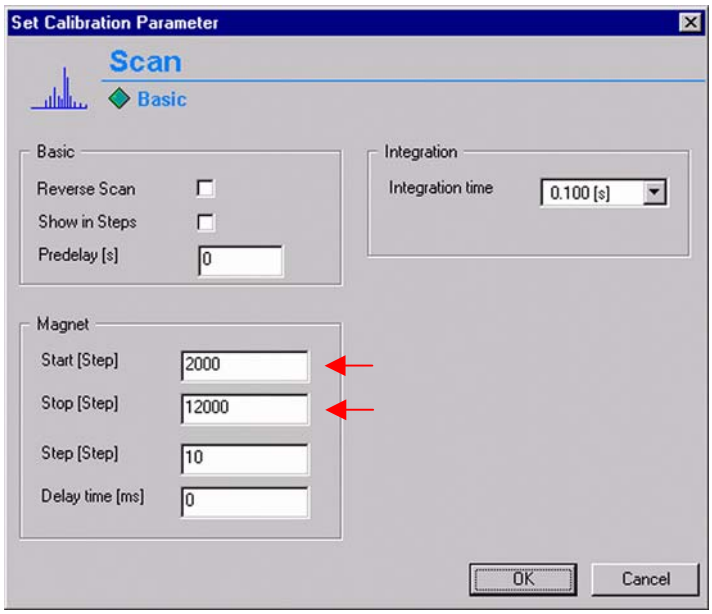
Normally, ***CO2*** is used for the first Mass Calibration, thus serving as a basis for later ones, which possibly may use other gases.

- From the ***Scan*** Menu select ***Calibrate***.

- You are reminded to ensure that Reference Gas is open.

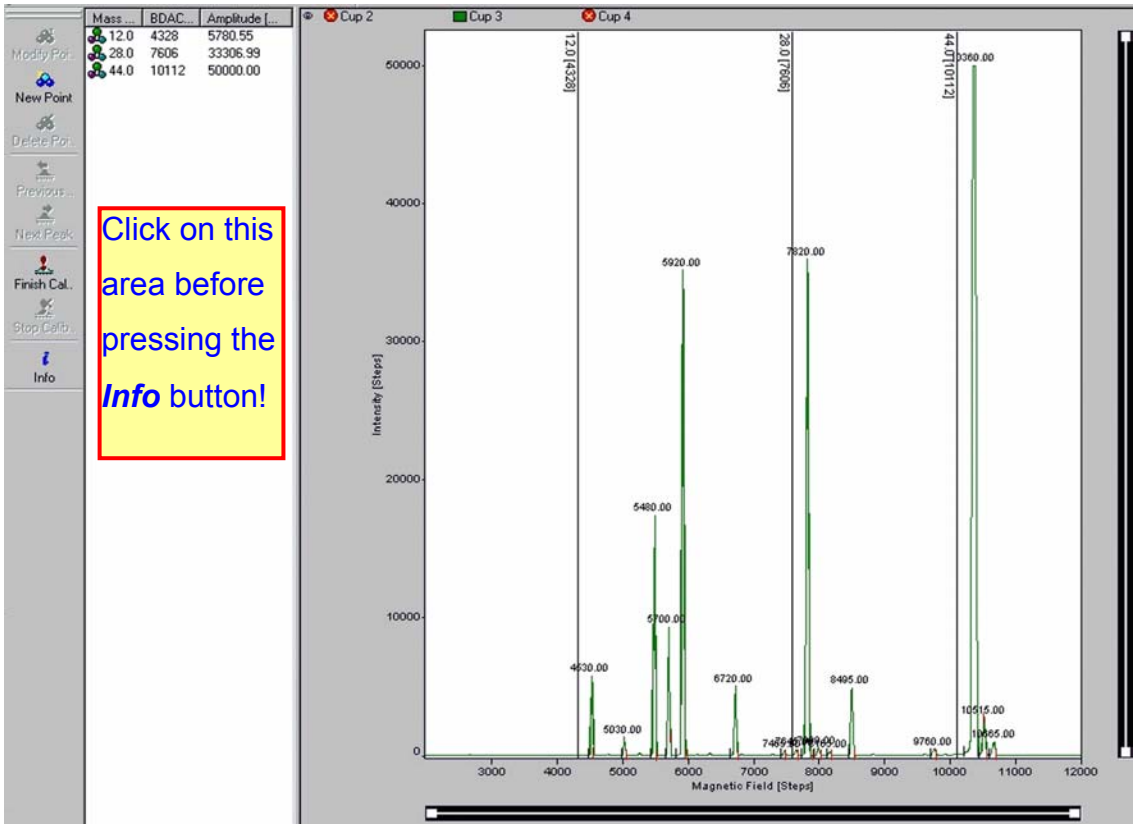
Therefore, check the signal intensity at the ***Smart Isotope MS*** bar. If the signal intensity is sufficient (e.g. > 1 V), click ***Yes***.





- At the ***Scan Basic*** box select 2000 steps and 12000 steps as a range for the Magnet Scan.
- Accept the other default values.
- Finally, press ***OK***.

Principle: The masses 12, 28 and 44 will be searched. A proposal about their position is then offered. On demand, additional masses can be inserted into the proposal list. If the positions don't match satisfactorily, they are slightly moved later.



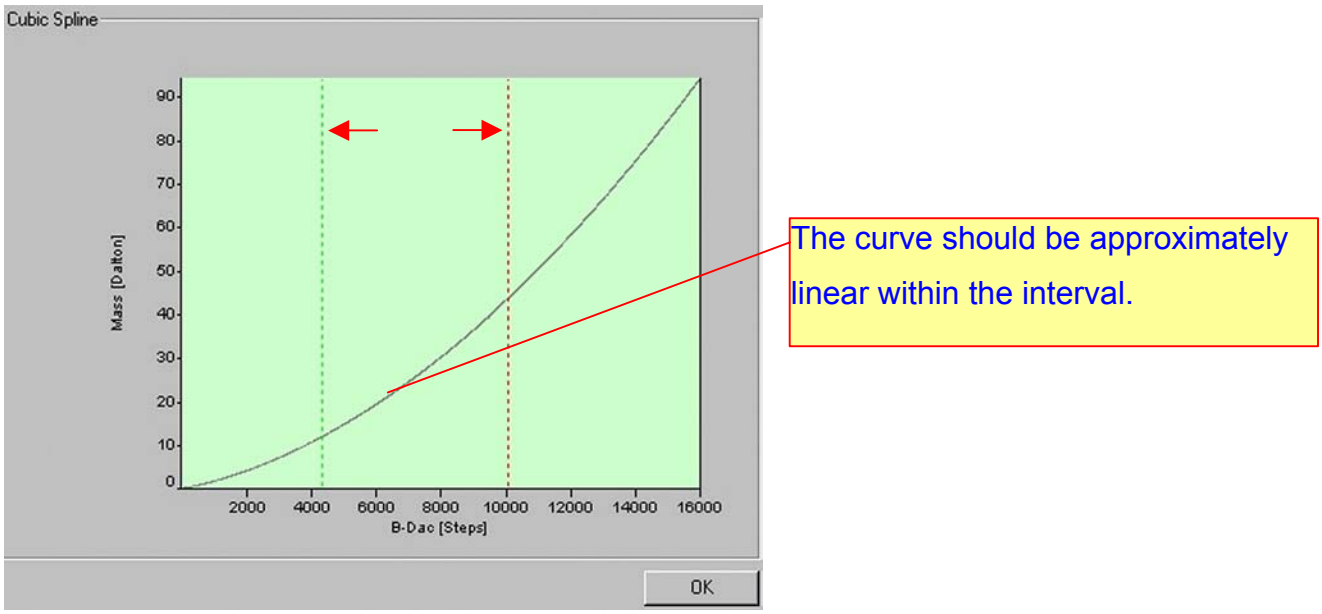
Thermo Finnigan

Sometimes, the proposals for the mass peak positions in the left pane are matching and sometimes they don't. During the very first Calibration, the proposals usually must be corrected: assign the calibration masses to their peaks by drag and drop of the vertical lines using your mouse, snapping the lines to the mass peaks.

During later Calibrations, the proposals mostly don't need to be corrected via drag and drop.

- ***Activate the area containing the mass proposals by clicking on it!***

- Control whether you chose the correct masses using the ***Info*** button.

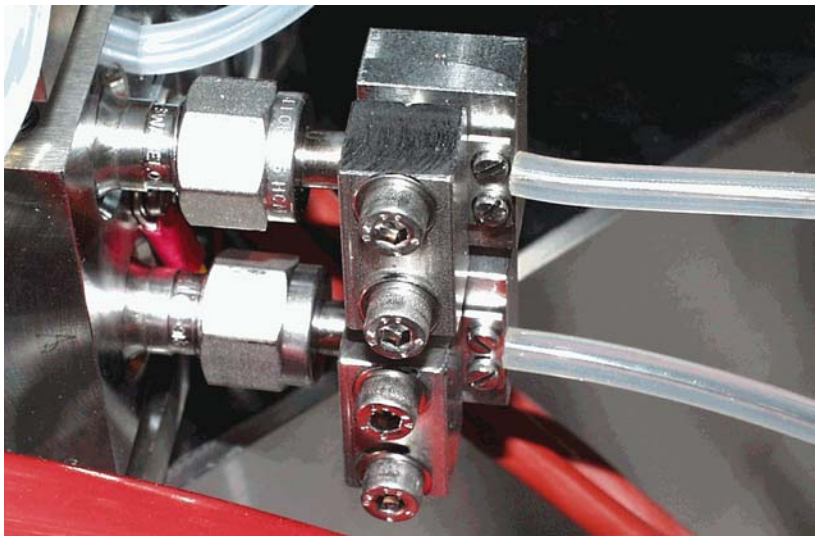


- After having corrected the proposals, press the ***Finish Calibration*** button.

The magnet is alternately set to low and high BDAC values several times. It will try to retrieve the peaks by jumping to them. Due to hysteresis, the peaks can't exactly be retrieved.

- A peak center follows.

Crimp adjustment



- Open the entire Inlet System.
- Let flow 50 mbar of CO_2 into each bellow (“equilibration”).
- Adjust the crimps until this bellow pressure of leads to a signal of 5 V (≈ 10 mbar correspond to 1 V in case of CO_2).

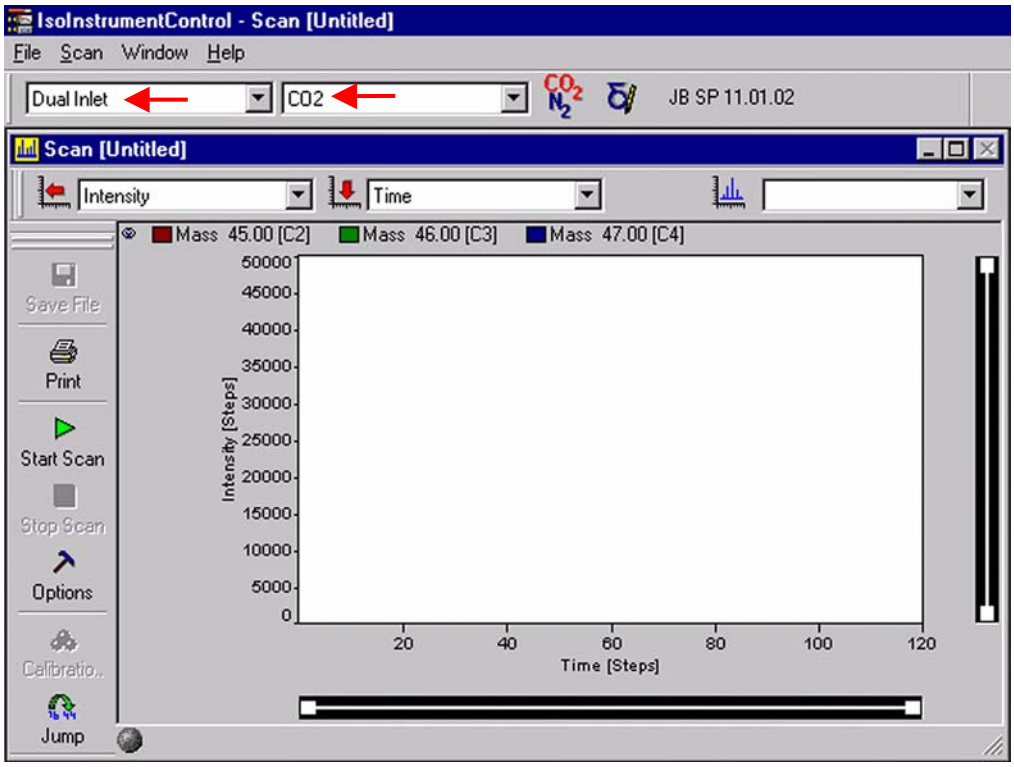
Crimp adjustment ensures an approximately constant and slight gas flow. Therefore, gas consumption will be low.

Bellow Calibration

Prior to a Dual Inlet Acquisition, a Bellow Calibration must be performed as follows:
It is presumed, that the source has been switched on and focusing has been performed.

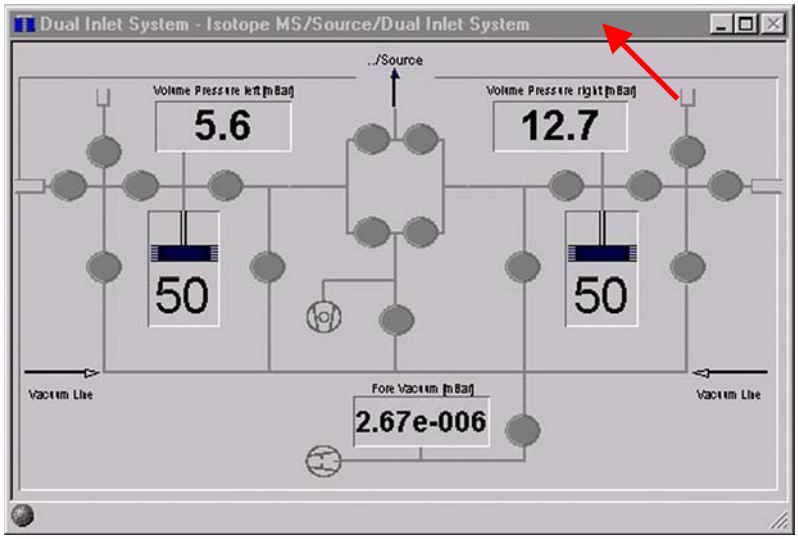
Start Instrument Control by double-clicking on the ***Instrument Control*** Icon.

The ***Scan*** window appears.



➢ Select your Configuration, e.g. ***Dual Inlet***.

➢ Select your Gas Configuration, e.g. ***CO_2***.



NOTE: ***Activate the Dual Inlet System window by a click on its title bar!***

Thus, the title bar will turn from grey (i.e. inactive) to blue (i.e. active).

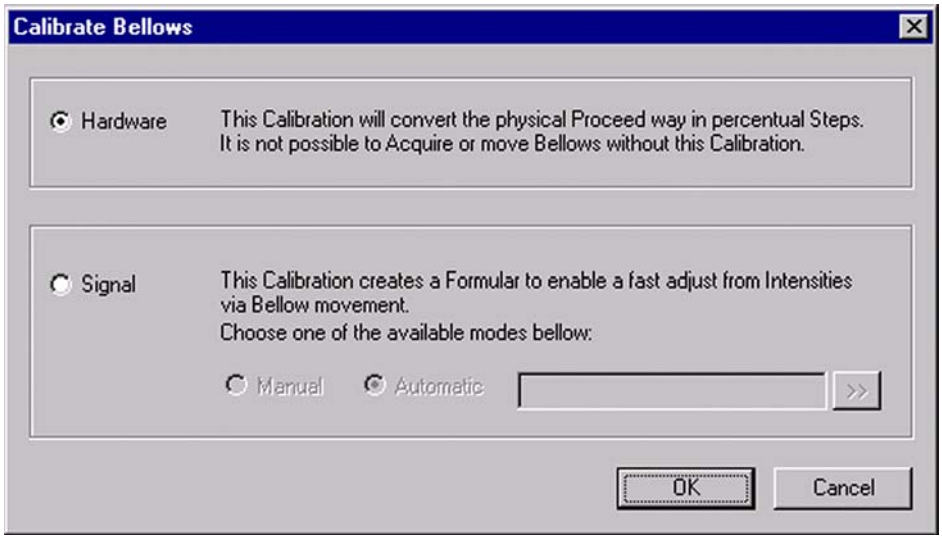
Access to the Calibrate Menu won't be possible while this window is inactive.

➢ From the ***Calibrate*** Menu, select ***Bellows***.

Bellow Calibration must be performed in two steps:

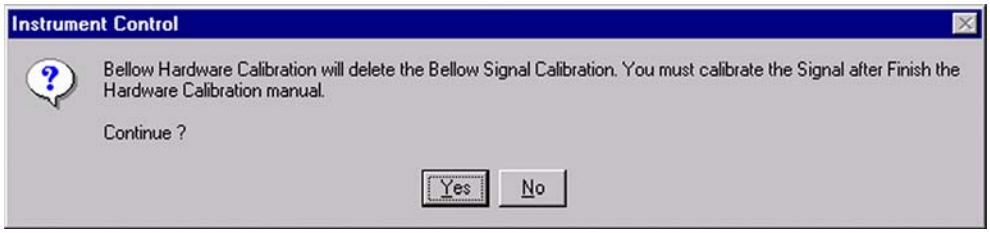
Step 1 *Hardware Calibration of the bellows*

In Hardware Calibration, the bellows are opened as far as it will go (i.e. 100 %) and closed again (i.e. 0 %).



➢ Enable the ***Hardware*** radio button.

➢ Press ***OK***.



NOTE: ***A Hardware Calibration deletes a Signal Calibration. Therefore, Hardware Calibration must always be performed prior to Signal Calibration!***

- Press ***Yes*** to continue.

- The ***Hardware*** Calibration of the bellows has been finished. Press ***OK***.

Step 2 ***Signal Calibration of the bellows***

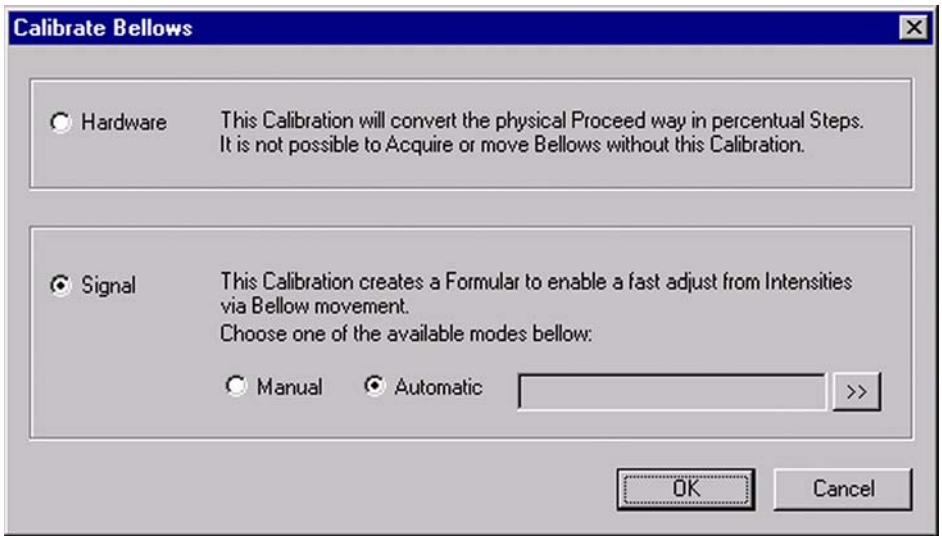
NOTE: ***Signal Calibration requires a signal intensity of at least 3 V!***

WARNING: ***If Signal Calibration has not been performed, the Diagnosis tests can't be run!***

In Signal Calibration, the signal intensity is checked with partially filled bellows (at 25 % and at 75 %). Therefore, Signal Calibration must always be performed after Hardware Calibration.

- Activate the Dual Inlet System window again by a click on its title bar.
- Then, from the ***Calibrate*** Menu, select ***Bellows*** again.

Before starting Signal Calibration, an amount of gas must be let into the bellows, which results in a pressure of about 10 mbar - 20 mbar on each side. This gas amount is necessary to obtain a certain signal intensity. If the gas amount should be either too small (e.g.< 10 mbar) or too large, warning messages will occur.

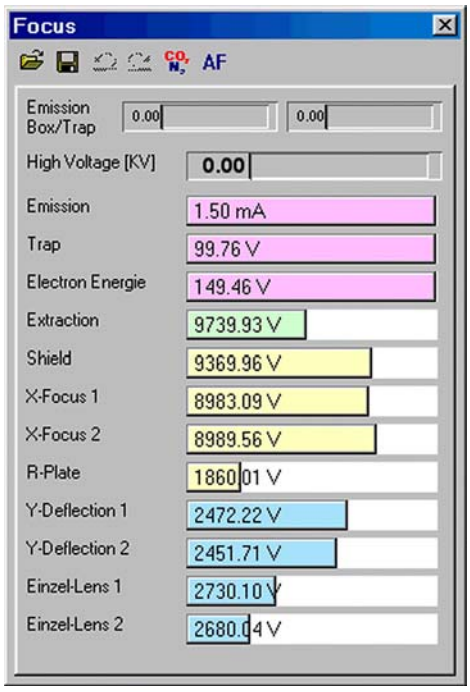


- Enable the ***Signal*** radio button.
- Select ***Automatic*** and press ***OK***.

The signal intensity of the particular gas will be measured depending on the bellow volume.

Focusing

The ion beam must now be ***focused*** (see Chapter 1.4). Thermo Finnigan MAT has already performed a focusing procedure. The obtained parameters, which can be seen on the ***Focus*** toolbar, have been assigned to the respective Gas Configuration.



On top of the Focus window, note the ***Focus Toolbar*** shown below:

Among others, it contains the following buttons:

- ***Load***: opens a “Focus Settings” document (*.fcs).
- ***Save***: saves the actual Focus Settings as “Focus Settings” document (*.fcs).
- ***Undo***: the last command is undone.
- ***Redo***: the last command is redone.
- ***Pass to GC***: attaches the current Focus Setting to the current Gas Configuration.
- ***Reset HV***: switches on high voltage.
- ***Source On***: turns on the entire source electronics.
- ***Source Off***: turns off the entire source electronics.
- ***Autofocus***: calls up an Autofocus Script.

If you changed parameters at the ***Focus*** window, or after you finished your own focusings, you can assign them to the active Gas Configuration by .

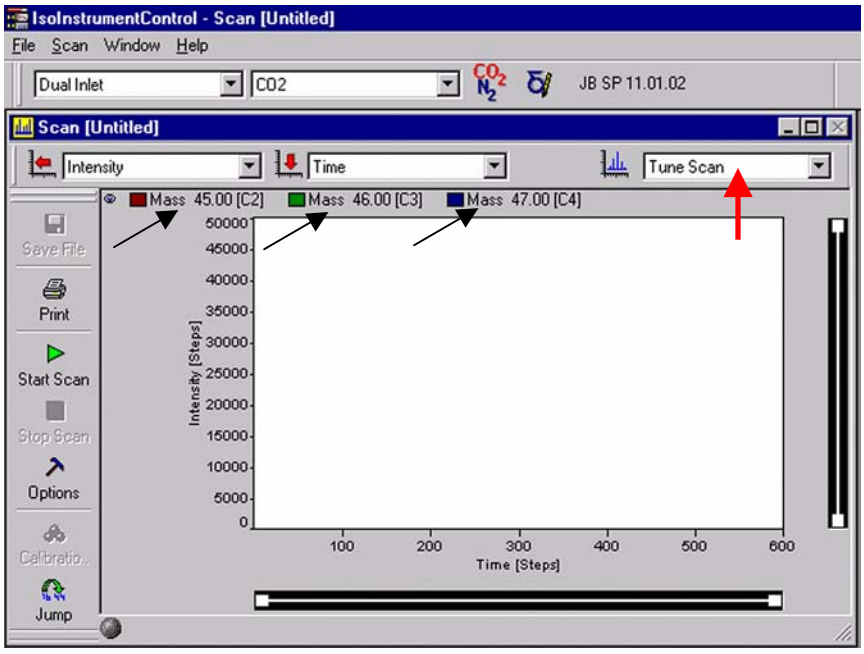
Besides, you can save new parameters by to load them again later by .

NOTE: ***For each Gas Configuration, a specific focus can be set and saved.***

Focusing can now be started:

- In the beginning, you should use the focusing settings Thermo Finnigan MAT has preset (see the ***Focus*** bar). Usually, focusing will be performed automatically (Autofocus). Only trained users should do this by themselves.

- To optimize the focusing settings by yourself, start a Tune Scan, which continues during the whole focusing process. A Tune Scan reveals the course of intensity over time while changing the parameters in the Focus window: At the ***Scan*** window, select ***Tune Scan***.



Furthermore, you can select a particular cup (e.g. mass 44, mass 45 or mass 46).

Example: If you select Cup 1, the intensity at Cup 1 will be monitored over time. For the Tune Scan recommended here, it doesn't matter, which cup is used. Focusing aims at gaining the maximum intensity value (which is revealed by the Tune Scan). In case of Dual Inlet, 100 mV approximately correspond to 1 mbar.

- ***Linearity*** focusing: To achieve maximum linearity, the extraction voltage prevailing at the extraction lens must be increased. It extracts the ions out of the ionization volume by acceleration. Additionally, the ions thus obtain a direction. Therefore, fewer collisions between molecules and ions occur.

- **Sensitivity** focusing: To achieve maximum sensitivity, the extraction voltage (at maximum intensity) must be set to zero. As the acceleration out of the source doesn't exist, more ions stay in the source. The number of ions per molecule is lower. Many ions exist having different energies (secondary ionization processes), providing a wide energy distribution.

➢ Linearity focusing must be performed here: while the extraction voltage is increased, all other lenses are tuned until maximum intensity is reached (monitored by the Tune Scan).

NOTE: ***If linearity focusing is unsuccessful, change the Extraction value at the Focus bar until the intensity pattern of CO_2 is satisfactory.***

The Autofocus settings preset by Thermo Finnigan MAT have been established on the basis of a reasonable pattern. A gas (e.g. CO_2) has its own pattern. Hence, various gases differ with respect to their patterns. The pattern of a particular gas is always the same.

NOTE: ***If Autofocusing has not lead to the desired maximum intensity, perform Focusing manually.***

Diagnosis

Without Bellow Calibration, Diagnosis Tests won't be possible. After the bellows have been calibrated, you can run the ***Diagnosis*** tests. For details, see Chapter 7.

Starting a Measurement

After performing Diagnosis tests, you can start a measurement, i.e. a ***Dual Inlet Acquisition.*** For details, see Chapter 6.

1.3 GETTING STARTED: SOFTWARE

1.3.1 SYSTEM REQUIREMENTS

To use ISODAT NT optimally, meet some system prerequisites. ISODAT NT needs certain

- Software requirements and
- Hardware requirements.

1.3.1.1 SOFTWARE REQUIREMENTS

It is advantageous if your system meets the **recommended** requirements.

Software requirements (minimal)	*Software requirements (recommended)*
Windows NT 4.0 (Intel) operating system	Windows NT 4.0 (Intel) operating system
Pentium class computer (233 MHz)	Pentium class computer (400 MHz or higher)
96 MB RAM	128 MB RAM
100 MB free disk space (only for ISODAT NT, without Backups and Result files)	500 MB free disk space (or higher)
Super VGA monitor (resolution 768 * 1280 pixels)	Super VGA monitor (resolution 1280 * 1024 pixels)
	Windows NT 4.0 supported printer

1.3.1.2 HARDWARE REQUIREMENTS

- COM Port Extension Board
- IEEE Interface Board
- Sound Card
- Joystick requirements
- GC and Autosampler requirements

For the installation of the drivers for the above listed hardware, refer to chapter 8.2.

1.3.2 INSTALLING ISODAT NT

Now install ISODAT NT (shipped on the CD). This can be done via the Startup Screen.

Close all applications (especially older versions of ISODAT NT if already installed and running)!

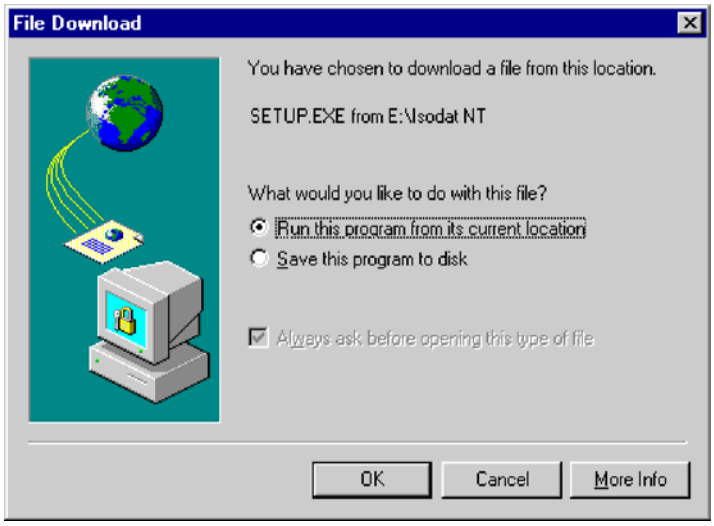
Otherwise, a message will indicate that Setup of ISODAT NT is not possible while older versions of ISODAT NT are running (see: how to shut down the system).

1. Open your CD drive and insert the ISODAT NT CD.
2. Close it.

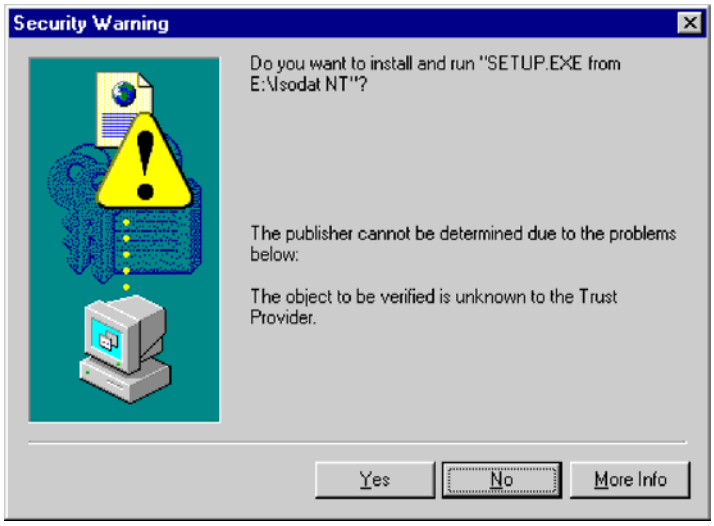
If your CD ROM drive is set to "Autostart", the file "startup.html" on the ISODAT NT CD will be executed and the gray HTML startup screen will appear.

3. From the gray Startup Screen, select "Install ISODAT NT".

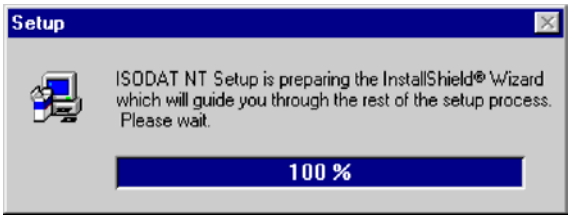
4. Select "Run this program from its current location". Then press ***OK***.



5. Click ***Yes***.

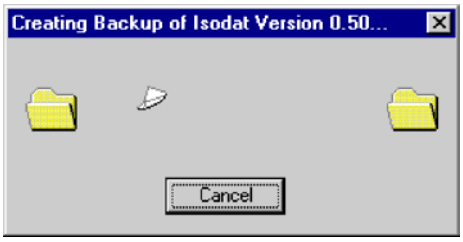


6. Wait while Setup is preparing the InstallShield® Wizard.



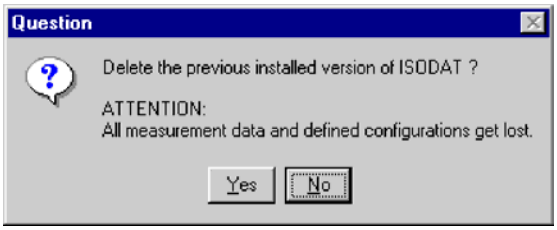
7. Respond with ***Yes*** (***No*** will not create a backup).
Make sure that you have enough free disk space for the backup that includes your Result files.

8. A backup of the current version of ISODAT NT is created and stored in a subdirectory, e.g. "Version_0.50", of the folder C:\Isodat_Backups (if you cancel the backup, a message occurs).



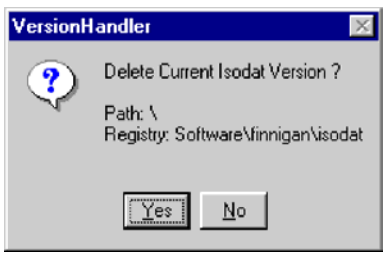
- Each backup includes your Result files and is stored in a subdirectory of its own in the folder C:\Isodat_Backups.
- Backups can be created and restored by using the Version Handler in the folder C:\Isodat_Backups.

9. Respond with ***Yes*** to delete a previous version.

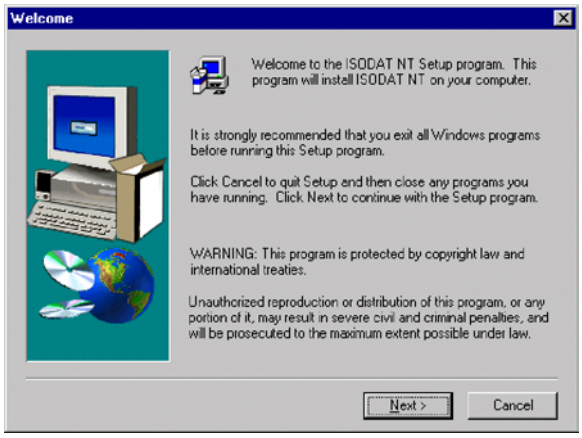


Yes leads to loss of Configurations and measurement data.
No will overwrite the previous version. Data, Configurations and Mass Calibrations will remain untouched.
Even though, it is recommended to delete old versions until unequivocal compatibility between the old and the new version is ensured.

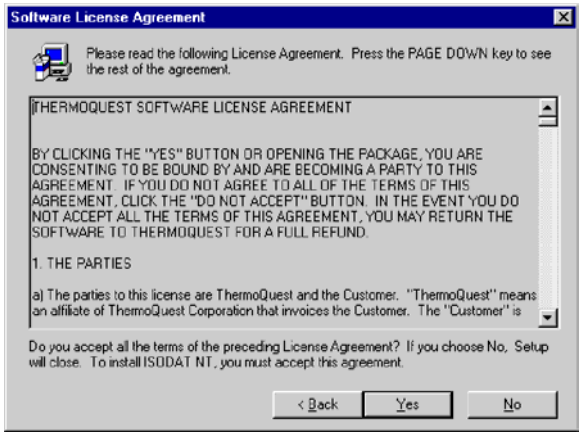
10. ***No*** will overwrite the previous version. Data, Configurations and Mass Calibrations will remain untouched. Even though, it is recommended to delete old versions until unequivocal compatibility between the old and the new version is ensured.
Confirm again with ***Yes*** to delete.



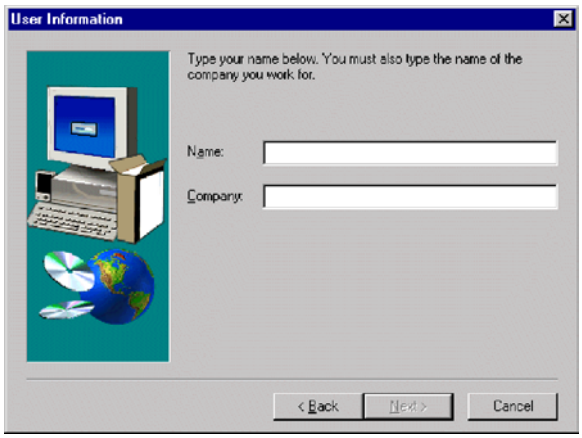
11. Read the text carefully and click ***Next***.



12. Read the software license agreement carefully and click ***Yes***.



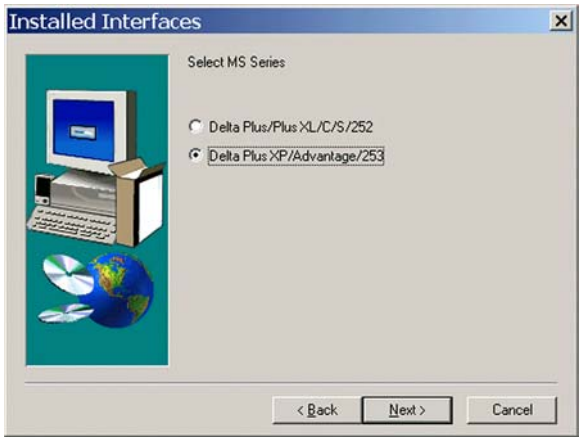
13. Fill in your name and company. Then click ***Next***.



14. On the next window, you have to indicate whether you have interfaces installed.
As default, both checkboxes are selected.
Select the checkbox(es) that correspond to your equipment, and then press ***Next***.

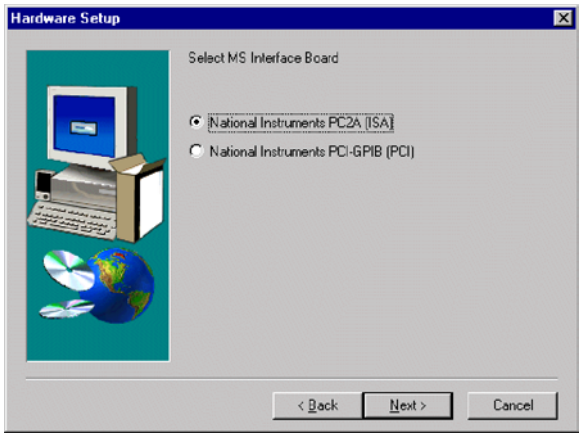
- When you have ticked the checkbox ***Fiber Line Interface***, proceed with step 18.
- When you have ticked the checkbox ***IEEE (GBIB) Interface***, proceed with step 16. This is also the case, when you have ticked both checkboxes.

15. When you have deselected both checkboxes in step 14, the following window appears. Select the radio button ***DeltaPlusXP/Advantage/253*** and press ***Next***. This will install the *Fake Mode*, which can be used for offline computer systems and software testing purposes.
Proceed with step 18.



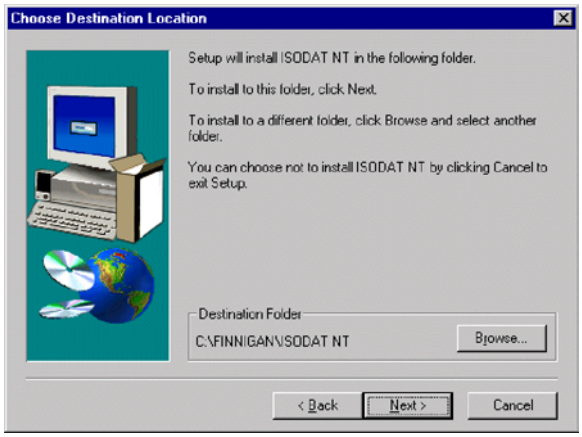
16. Select your MS Interface Board for IEEE connection to the IRMS (on the rear side of your computer). Click on ***Next***.

- The "National Instruments" PC IIA (ISA) driver will automatically be installed.
- The "National Instruments" PCI-GPIB (PCI) driver must be installed separately later.



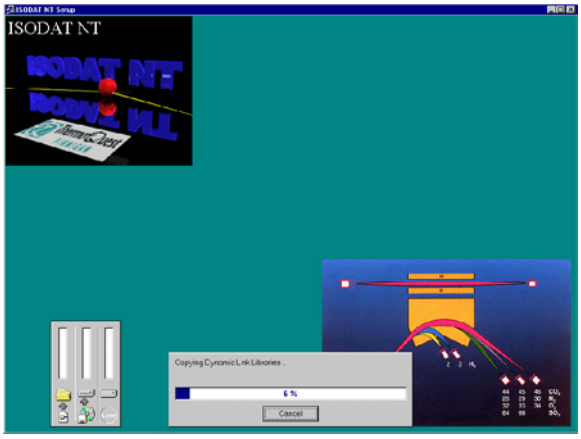
17. Having chosen PCI-GPIB (PCI) in step 16, you are reminded that the "National Instruments" PCI-GPIB driver must be installed separately later using the "National Instruments" CD.
Ensure that the appropriate IEEE Interface board driver has been installed. The required cables are part of the delivered equipment.
Press ***OK***.

18. Accept the proposed folder or choose another one by the ***Browse*** button. Then press ***Next***.



19. Wait while Setup is updating the registry.

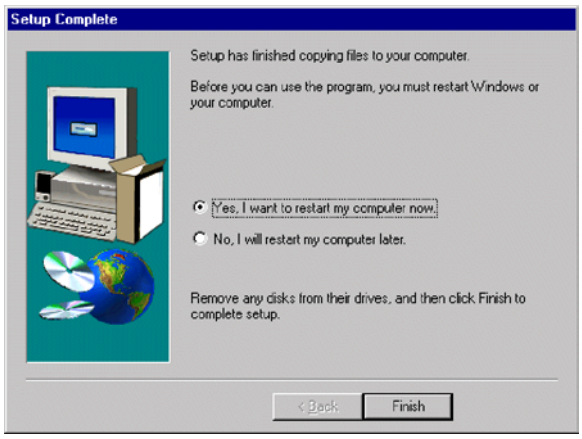
20. Wait while Setup is copying ISODAT NT onto your harddisk.



21. Choose Yes, I want to restart my computer now.

Remove the CD.

Click ***Finish*** to reboot. ISODAT NT is now installed on your computer.



1.4 PRINCIPLE OF FOCUSING

1.4.1 REASONS TO PERFORM FOCUSING

Focusing has to be performed

- to overcome the tiny remaining mechanical tolerances
- to overcome the gradual degradation of the ion source (e.g. due to metal deposition by sputtering)
- to overcome the different ion-optical properties of various gases (e.g. for H_2, a slightly different focusing compared to CO_2 yields better results).
- to optimize the system for various applications (e.g. reduce or increase sensitivity, optimize linearity)

How to perform focusing - (a) Autofocusing

Depending on your choice, all or only selected potentials might be varied.
For detailed information, refer to chapter 2.3.3.

1 It may be advantageous to set some potentials to default values, e.g.

- Trap to maximum
- Electron energy to maximum
- Emission to maximum or lower
- Extraction to about ***2700 V*** for linearity focusing

2 Start Autofocusing by pressing the AF button. AF

3 At ***Autofocus default***, select the potentials to be changed.

4 Press ***OK***.

How to perform focusing - (b) Manual Focusing

NOTE: ***Be careful! In case of doubt, preferably perform Autofocusing! Manual Focusing and Autofocusing can also be performed in turn (e.g. beginning with a gross Manual Focusing which is followed by a fine Autofocusing).***

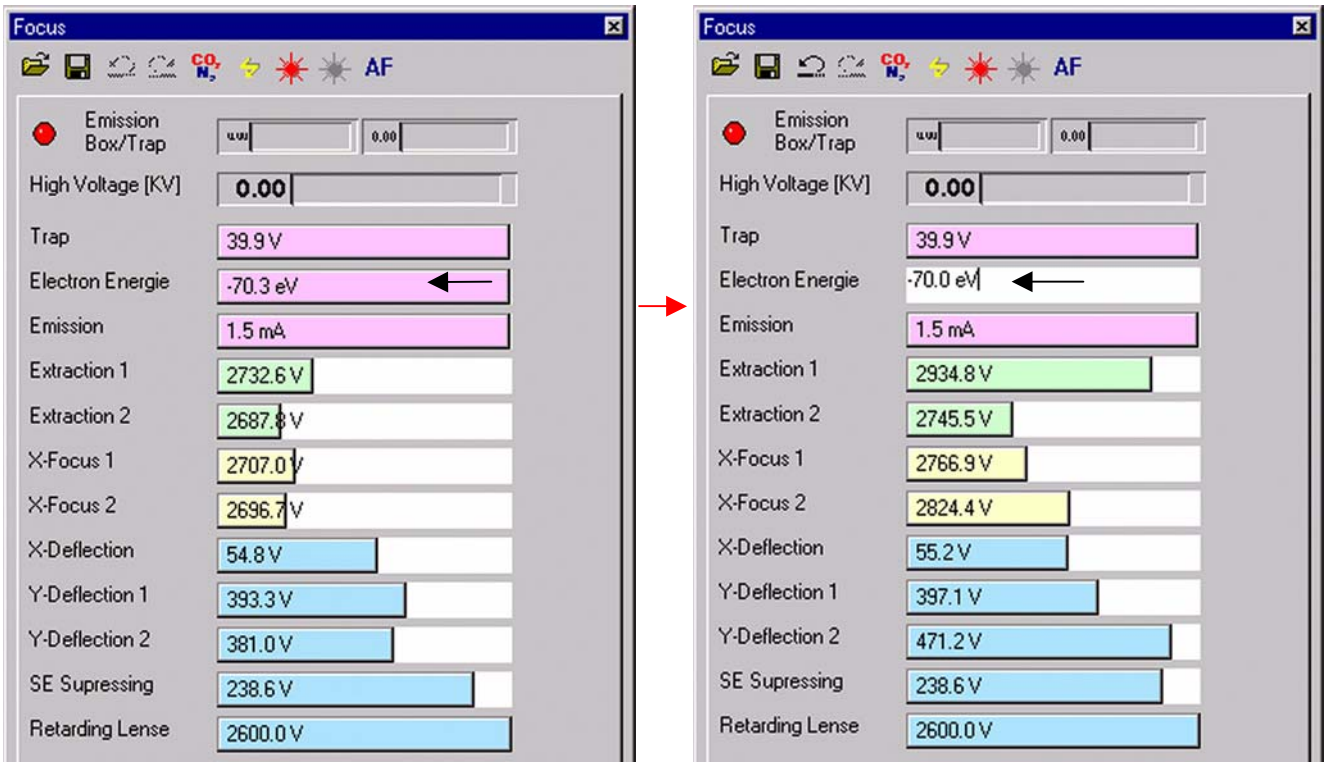
Use the ***"Undo"*** and ***"Redo"*** buttons on the Focus Toolbar to correct inadvertently false edited parameters of the Focus window.

As a parameter value increases, the colored part of its field grows.

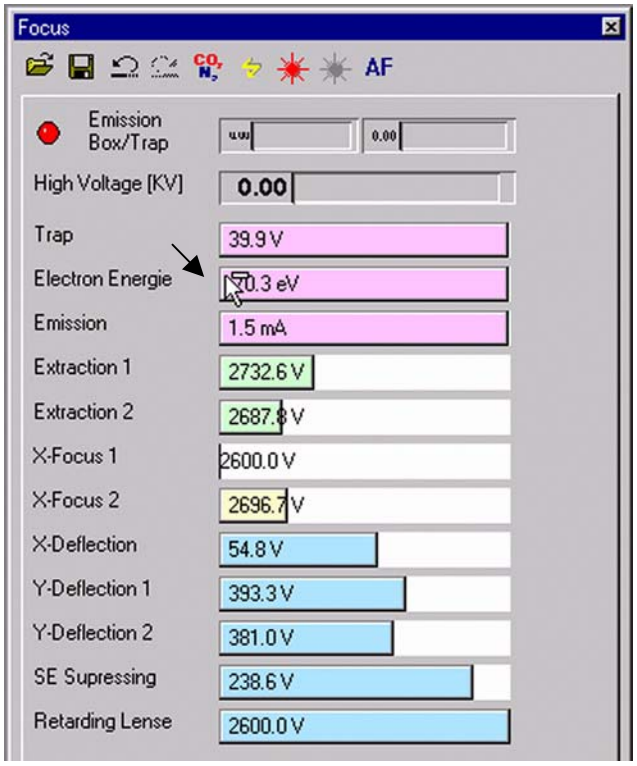
The five following equivalent possibilities are available for data editing:

- Click on the field to be edited. Then use the right or left cursor button to increase or decrease the value.

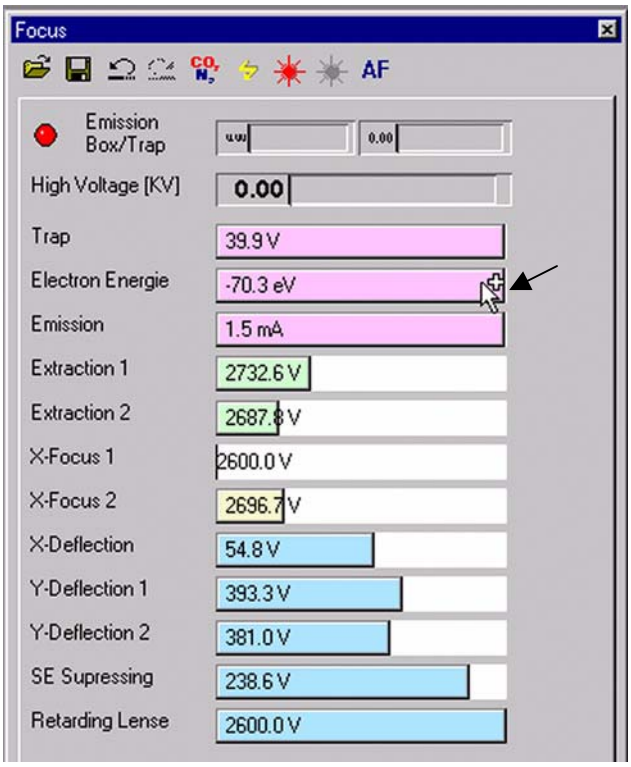
- Double-click the field to be edited. Then, simply type in the new value.



➢ Point to a field leftmost to decrease or rightmost to increase its value.



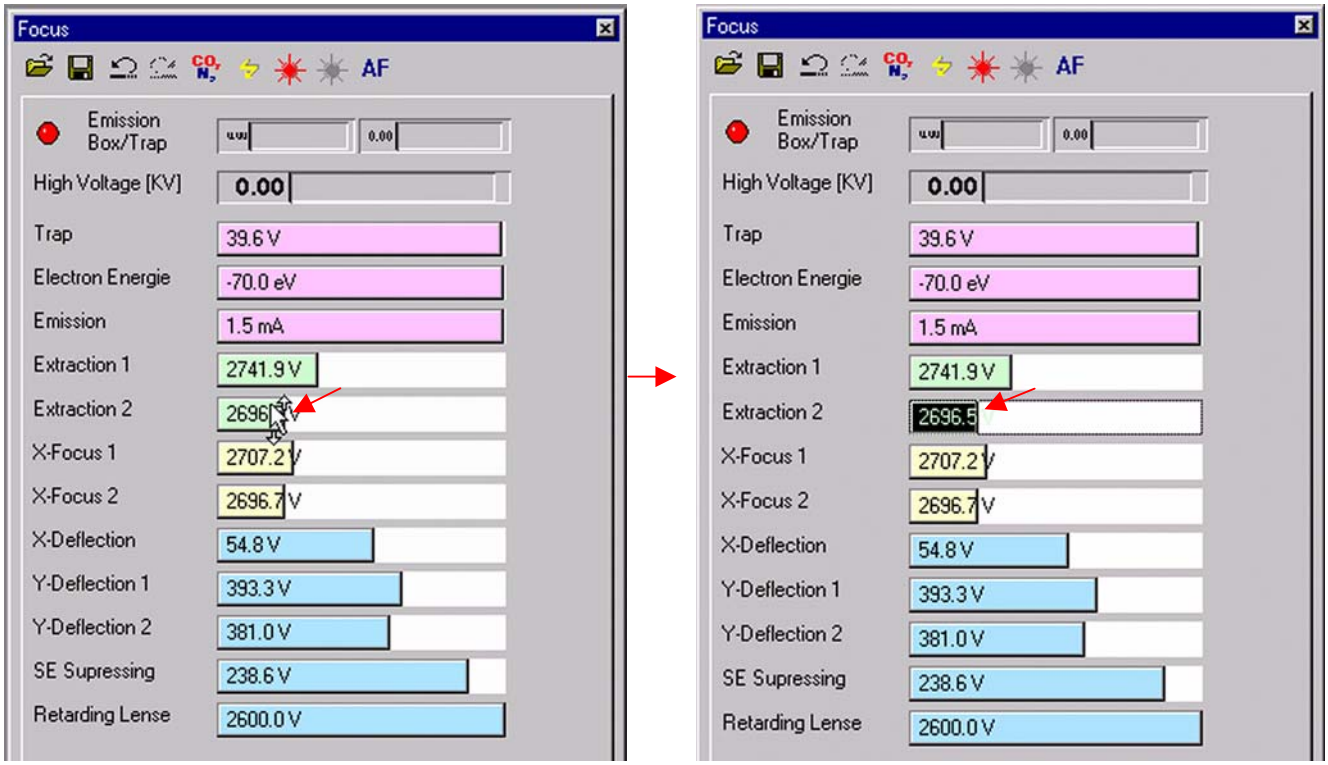
or



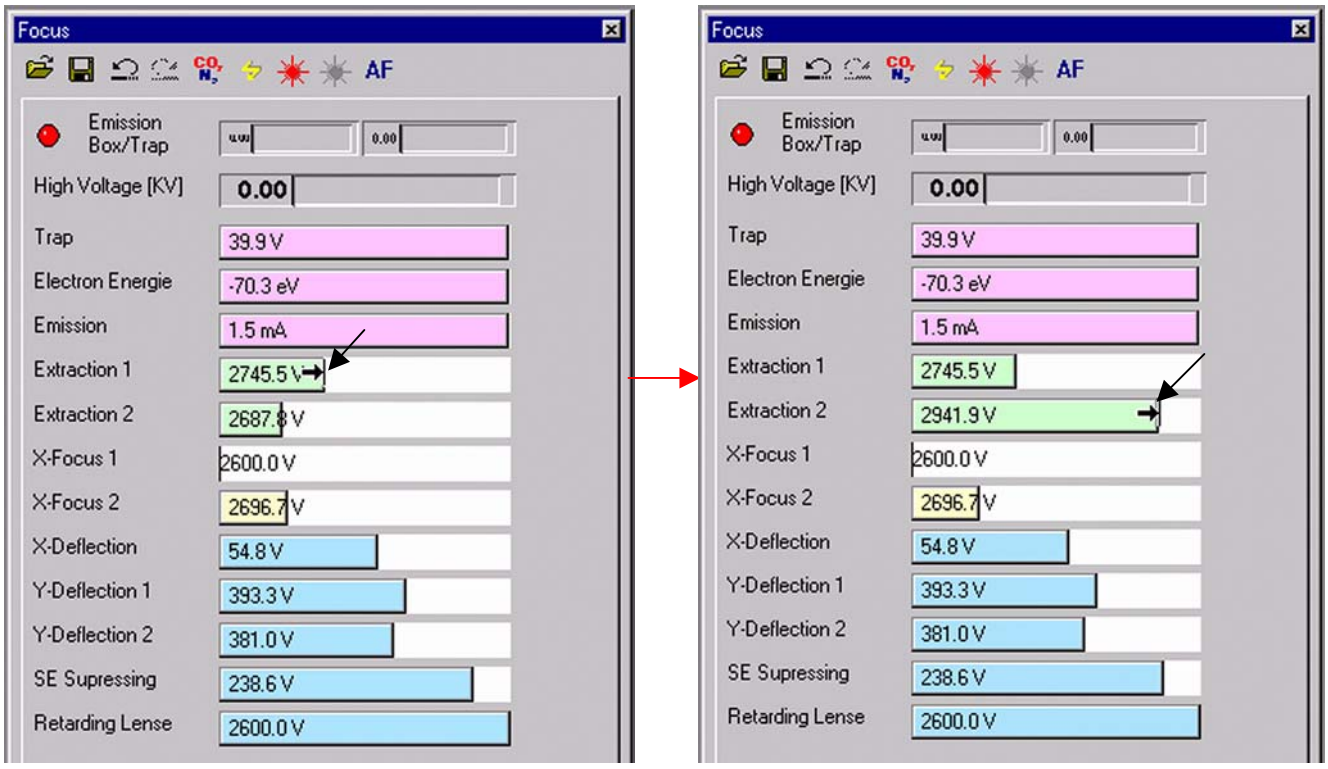
Clicking the right mouse button then allows selecting a step width.

The actual step width is marked.

- Point to the field to be edited. A symbol consisting of two arrows occurs (one bottom-up and one top down). Click into the field. Then move your mouse to change the value. This procedure allows ***subtle*** corrections.



- Point to the separation line between colored area and uncolored area of a field. An arrow occurs. Drag it to the right to increase or to the left to decrease the value. This procedure allows ***rough*** corrections.


Focus
AF
Emission Box/Trap
High Voltage [KV]
0.00
Trap
39.9 V
Electron Energie
-70.3 eV
Emission
1.5 mA
Extraction 1
2745.5 V
Extraction 2
2687.8 V
X-Focus 1
2600.0 V
X-Focus 2
2696.7 V
X-Deflection
54.8 V
Y-Deflection 1
393.3 V
Y-Deflection 2
381.0 V
SE Supressing
238.6 V
Retarding Lense
2600.0 V
Focus
AF
Emission Box/Trap
High Voltage [KV]
0.00
Trap
39.9 V
Electron Energie
-70.3 eV
Emission
1.5 mA
Extraction 1
2745.5 V
Extraction 2
2941.9 V
X-Focus 1
2600.0 V
X-Focus 2
2696.7 V
X-Deflection
54.8 V
Y-Deflection 1
393.3 V
Y-Deflection 2
381.0 V
SE Supressing
238.6 V
Retarding Lense
2600.0 V

MAT 253

OPERATING MANUAL

ANALYZER

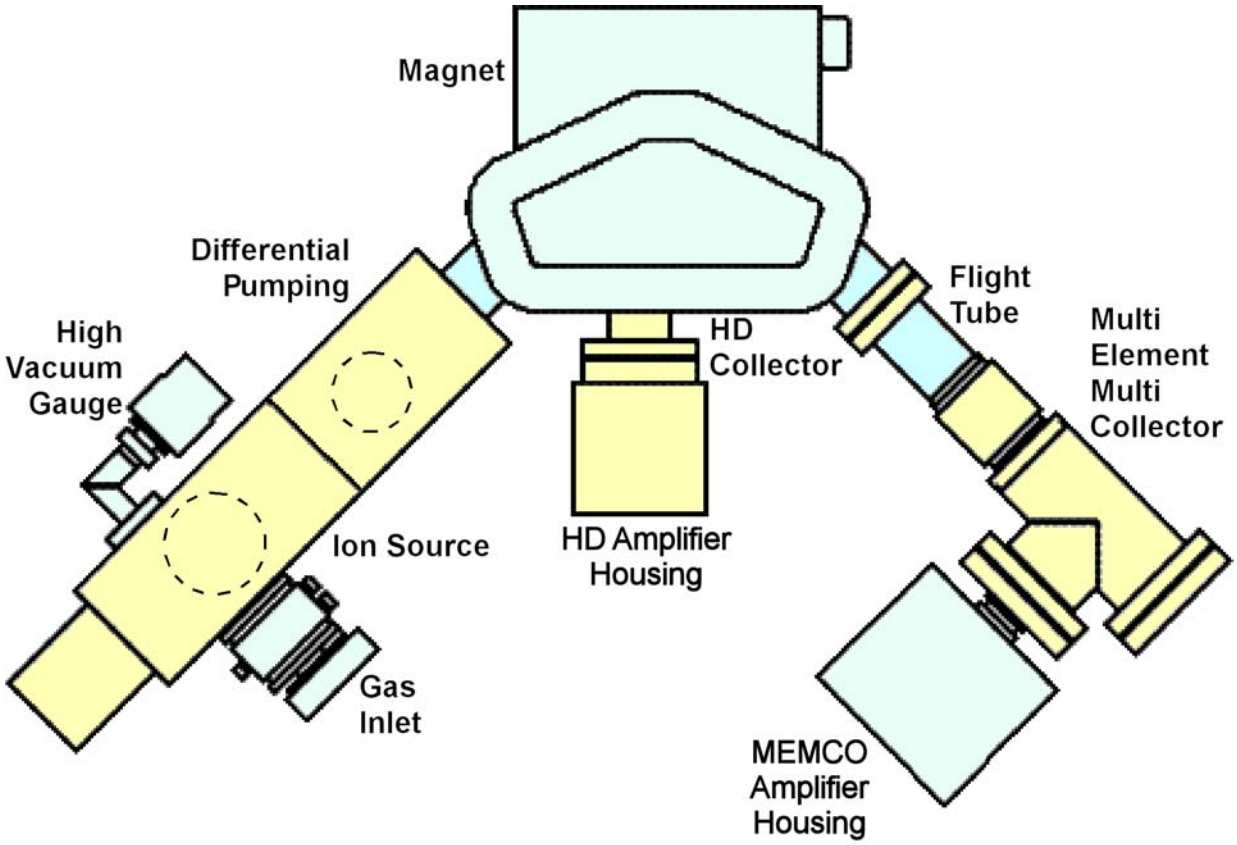
Issue 04/2002

Thermo Finnigan

2.1 GENERAL

The gaseous sample to be analyzed is fed into the ion source via the inlet system. In the ion source, ions are generated in a high vacuum by the impact of electrons. The ions are then accelerated to energies of up to 10 keV and focused by electrostatic lenses to form a beam (for details, see fig. 2-3).

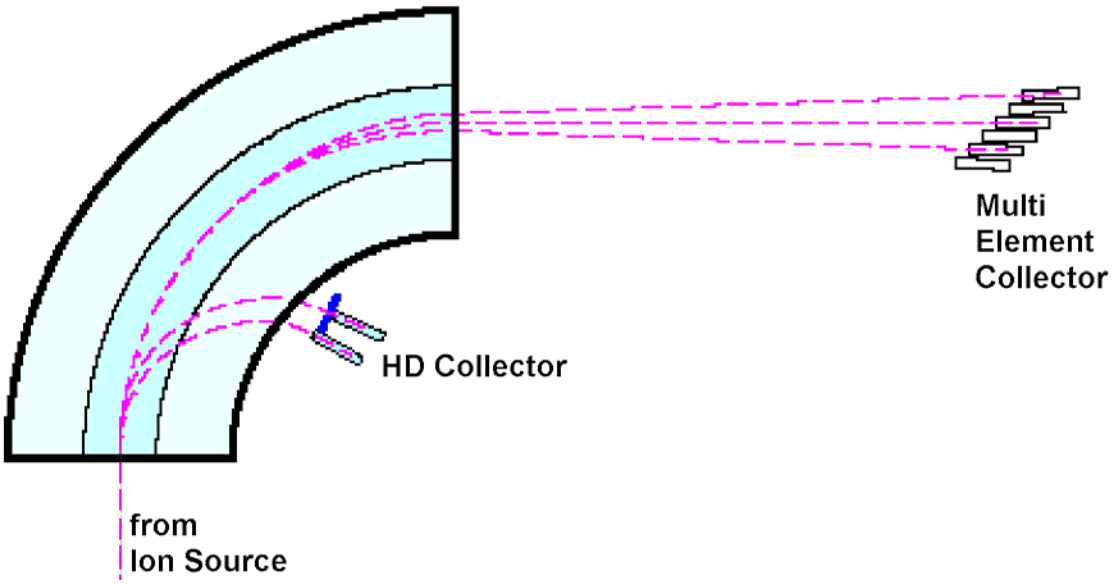
Fig. 2-1 Partial view of the instrument showing the arrangement of the analyzer



The ion beam exits the ion source into the magnetic field through a slit with a fixed width of 0.2 mm. It enters the magnetic field boundary at an angle of 26.5° and traverses the 90° magnetic sector field. A part of the ion beam exits at the same angle of 26.5° (see also fig. 2-2).

Due to shaping and dimensions of the magnet, not only a focusing in X-direction but also in ***Y-direction*** is achieved. The refraction power (X-direction) is half as much as compared to the same magnet without shaping it. The focal length decreases and its value is the same for X- and Y-direction. Consequently, mass dispersion increases compared to the conventional arrangement, where the beam enters and exits the field normal to the boundaries. Thus, the 23 cm radius system has the same mass dispersion as the conventional 46 cm arrangement (where the beam enters and exits the field normal to the boundaries with a sector radius of 46 cm).

Fig. 2-2 The scheme of the ion path



The magnetic sector field is generated by an electro magnet with a maximum field strength of 0.75 Tesla. It covers a mass range up to m/z = 150 at full accelerating voltage.
The mass setting is achieved by variation of the magnetic field strength and/or of the accelerating voltage.

The relation between the mass number m/z of the ions reaching the ion collector and the magnetic field strength H is given by: $m/z = k_M \cdot H^2$

where z: elementary charge

$$k_M = \frac{r^2}{2U} \neq \text{const.}$$

r: nominal radius of ion path (r = 23 cm = const.)

U: accelerating voltage (not constant)

Due to the ***variable*** accelerating voltage U, k_M is also not a constant value but a function of U

Example: For the special case of U = 10 kV, k_M = 26.45 * 10^{-6} m^2/V results.

The HD collectors are positioned in the middle of the flight tube due to the small radius of deflection for the light H and D ions. The minimum exit slit width is 2 mm and results in a resolution of

$$\frac{m}{\Delta m} = 25 \text{ (10\% valley)}$$

The collector slit widths for the universal (CNOS, MEMCO) or user-tailored collectors are available in a range from 0.6 (e.g. for measuring SF_6 or Xe) to 3.5 mm and result in a resolution of

$$\frac{m}{\Delta m} = 200 \text{ (10\% valley) for 1.5 mm cups.}$$

Details on the various collector types are given in chapter 2.5, Ion Detection - Collector System.

The electronic units supply the analyzer system operating voltages and control the analysis. They are described in detail in chapter 2.3, Ion Source Control Unit.

2.2 ION SOURCE

The ion source of the Thermo Finnigan ***MAT 253*** is designed for high sensitivity (due to high gas density) and linearity as well as low H_3^+ production at the same time. To ensure high sensitivity, the ion source is of gas tight design. The sample gas enters the ionization chamber through a ceramic tube and leaves it only via small apertures. These are required as a passage for the electron beam and the ions exiting into the analyzer.

The conductivity of these openings is much lower than the pumping speed of the vacuum pumps. Thus, the pressure within the ionization chamber is about 100 times higher than outside, which leads to high ion yields. The ions are generated in the source by electron impact ionization. The ionizing electrons are emitted by a thermionic cathode. The emission current is held constant by the emission regulator unit (see chapter 2.3).

An additional feature is the Variable Ion Source Conductance (VISC), or "Sulfur window". With the VISC installed, the ion source conductance can be varied from outside without breaking vacuum. High conductance operation is advantageous for SO_2 analyses to reduce memory effects and to achieve short idle times.

A variable conductance is also required to optimize the flow characteristics of carrier gas / sample peak mixtures for dynamic inlet techniques, such as continuous flow isotope ratio monitoring.

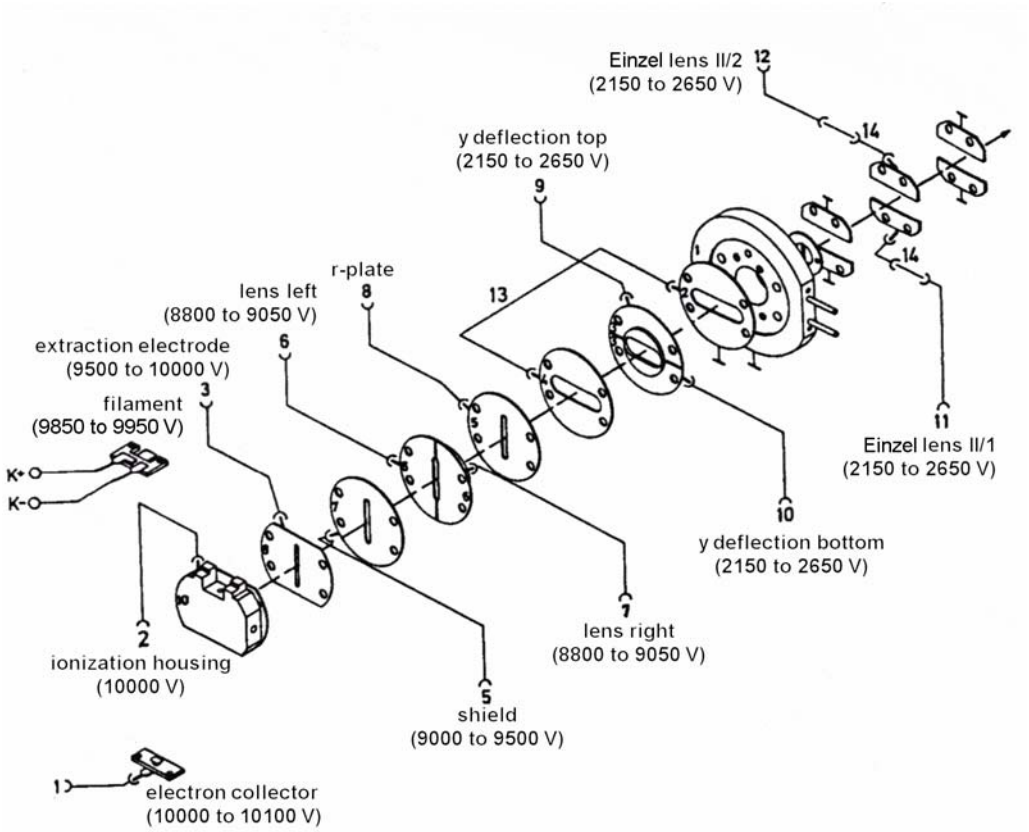
Two small permanent magnets are mounted to the ionization chamber (see figure 2-3), generating a magnetic field parallel to the electron beam. Due to the magnetic field, the electrons are moving along spiral trajectories, which increases their probability in the ionization volume. Thus, the probability to generate an ion with the aid of one single electron increases.

The energy of the ionizing electrons is determined by the potential difference between cathode and ionization chamber. It has a range between 70 and 124 eV.

The electron beam leaves the ionization chamber via a small opening opposite to the cathode. It is collected in the electron trap, which is held on a positive potential relative to the ionization chamber.

Extraction plates accelerate ions out of the ionization chamber. The following lens system of different lenses focuses the ion beam onto the source slit.
Mechanical tolerances might cause a slight out-of-axis deflection of the ion beam. Some of the system's lenses are half sections, which are insulated from each other. This construction allows for compensation by applying different potentials to the halves of the lenses. Fig. 2-3 shows schematically the lens arrangement and the potentials applied to them with an ion accelerating voltage set to 10 kV.

Fig. 2-3 Potentials (vs. ground) of the ion source at 10 kV ion acceleration voltage



Using ISODAT NT, you can set continuous values for the ion accelerating voltage. If it is changed, the lens potentials are changed proportionally with the exception of those of the trap and cathode voltages.

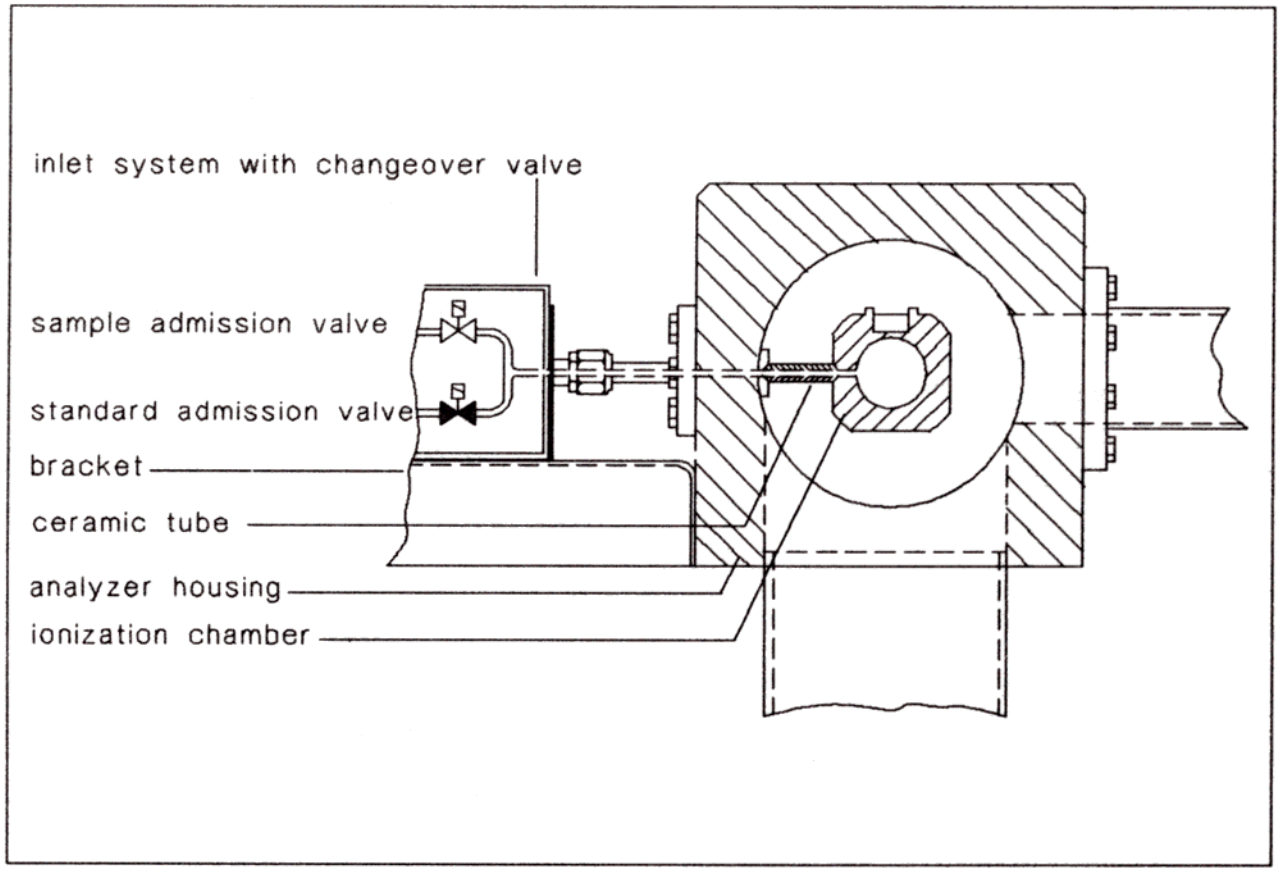
Setting the accelerating voltage to lower values results in an enhancement of the mass range beyond 150. For example: a setting to

8.0 kV results in a mass range up to 190,

6.6 kV results in a mass range up to 230.

The ion source is mounted on the front flange for easy maintenance. A correct alignment of the ion source relative to the analyzer tube is achieved by a mating surface with the analyzer head. Details of the ion source, e.g. the feedthroughs and correlation of lenses, are given in the chapter 8, "Technical Information".

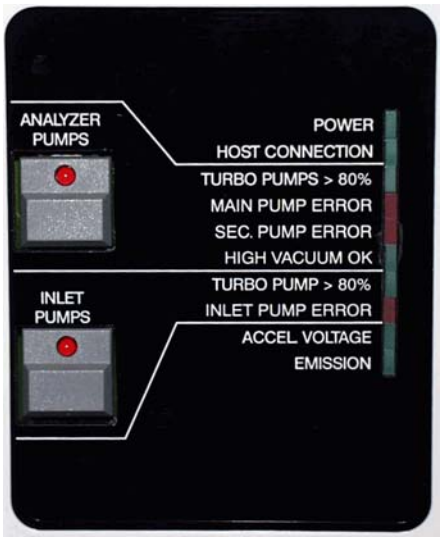
Fig. 2-4 Coupling of the inlet system to the ion source

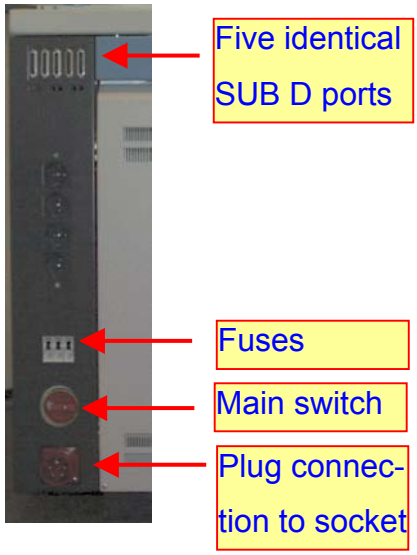


2.3 ION SOURCE CONTROL UNIT

CAUTION: ***Opening the electronics cabinet is only allowed for maintenance purposes by qualified personnel.***

Fig. 2-5 Control panel and main switch of the MAT 253





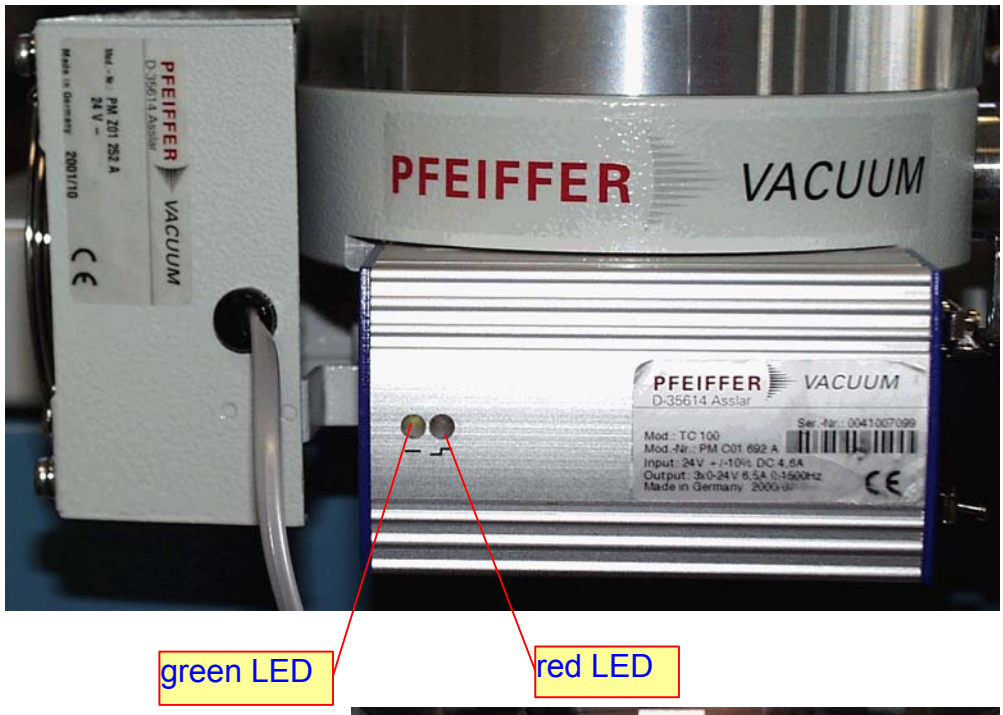
Analyzer Pumps Main pump (i.e. source pump) and secondary pump (i.e. differential pump). They are both connected to a Fore Vacuum pump (e.g. #1).

Power	turns on, if	**1.** the power plug has been inserted into the socket, **2.** the Main Switch has been switched on and **3.** the three fuses above the Main Switch have been switched on.
Host Connection	turns on, if	IRMS and computer have been connected.
Turbo Pumps > 80 %	turns on, if	the turbo pumps have reached 80 % of their rotational speed.
Main Pump Error	turns on, if	an error at the main pump has occurred.

Sec. Pump Error turns on, if an error at the differential pump has occurred.

High Vacuum OK turns on, if a pressure below 10^{-4} mbar has been reached. The penning can only start vacuum measurement below this pressure.

Fig. 2-5 a



➢ Main pump

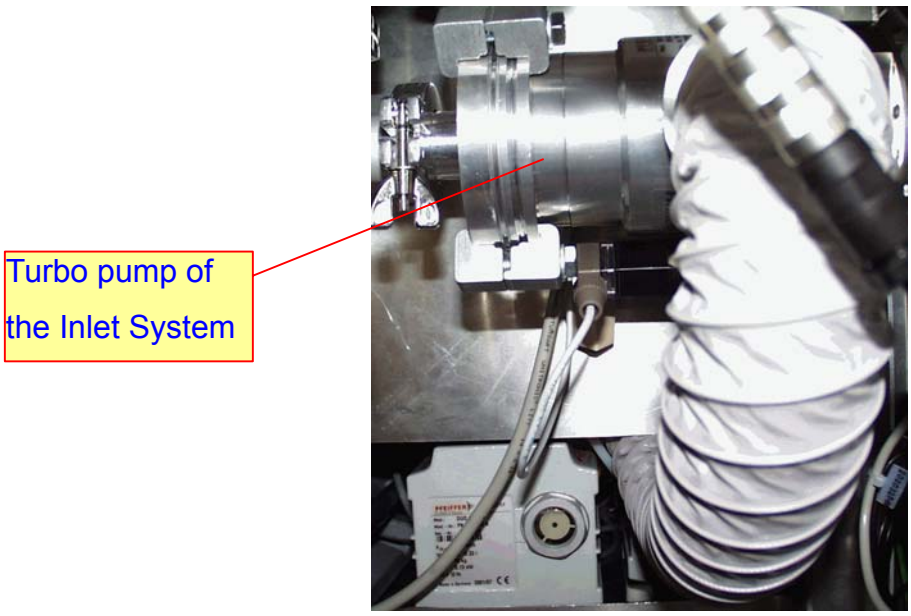
(i.e. source pump)

Fig. 2-5 b

➢ Secondary pump (i.e. differential pump)

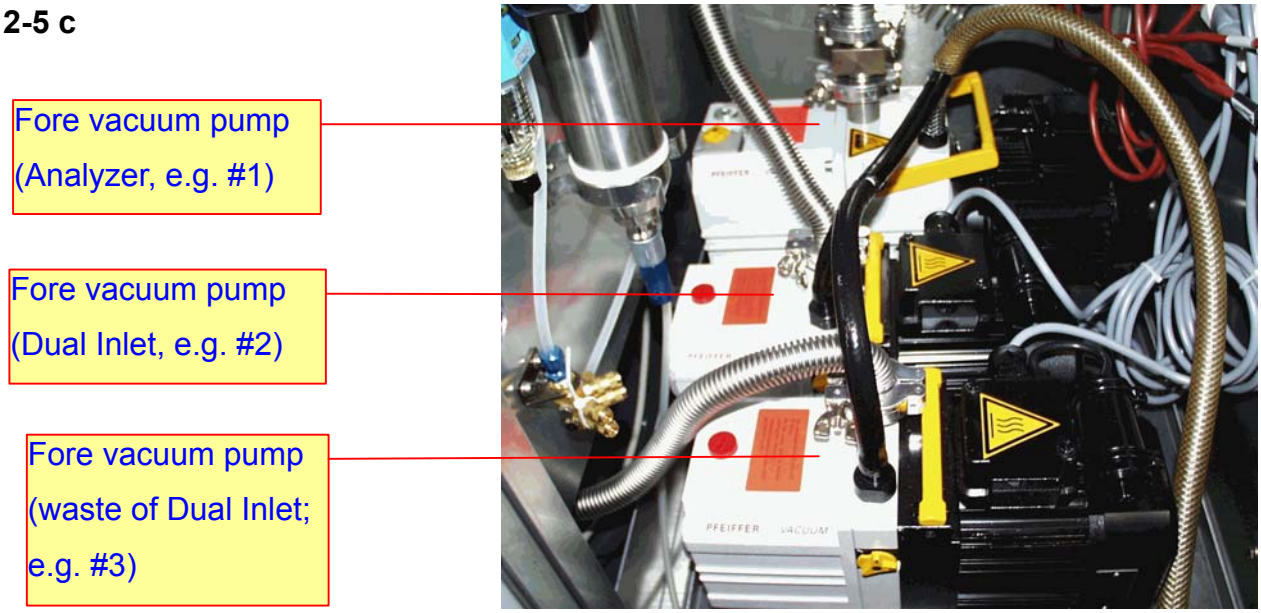
Inlet Pumps Turbo pump of the Inlet System. It is connected to two fore vacuum pumps (e.g. #2 and #3).

Turbo Pump > 80 %	turns on, if	the turbo pump has reached 80 % of its rotational speed.
Inlet Pump Error	turns on, if	an error at the inlet pumps has occurred.
Accel Voltage	turns on, if	the acceleration voltage for the ion source has been switched on.
Emission	turns on, if	the ion source has been switched on for emission.



The turbo pump of the Inlet System (only available in case of Dual Inlet) is located in the lower section of the cabinet's front panel under the bellows block.

Fig. 2-5 c



NOTE: ***Accel Voltage and Emission can only be set via ISODAT NT. The other parameters can be set manually.***

NOTE: ***On the Smart Isotope MS toolbar, both high voltage and emission can be switched on via and switched off via . High voltage can be switched on via and switched off via .***

In case of all three turbo pumps (i.e. turbo pump of the source, differential turbo pump, turbo pump of Inlet System; see LEDs in fig. 2.5 a) note:

- If a pump is off, the ***green*** LED blinks.
- If a pump is on, the ***green*** LED is turned on permanently. No information is revealed about the rotational speed of the pump.
- If a pump did not start up, the ***red*** LED is turned on permanently.

2.3.1 EMISSION REGULATOR

The emission regulator controls the current, which heats the cathode of the ion source and is switched on via ISODAT NT. The ionizing electron current, i.e. emission current, can be selected via ISODAT NT (maximum value: 1.5 mA). The cathode heating current is about 6 A. If the emission regulator is activated, the LED ***Emission*** on the front panel of the instrument turns on.

2.3.2 HIGH VOLTAGE SUPPLY

The high voltage unit provides the ion accelerating voltage. A current overload circuit switches off the high voltage supply, if the load current exceeds 0.2 mA. A switch-off caused by an overload is indicated: the green LED ***Accel Voltage*** located on the front panel then turns off.

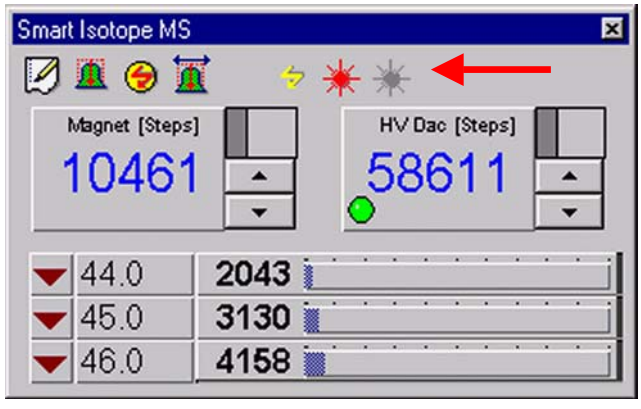
NOTE: ***A switch-off of the high voltage unit in case of an overload is a protective measure! Thus, you must perform troubleshooting:***

A short circuit in the feed line of the source might have occurred (e.g. after cleaning or reinserting the source.

Alternatively, after a longer time the source might have become dirty and must thus be dismantled, cleaned and finally reinserted.

Before you start to adjust the potentials, make sure that you have

- switched on the ion source (button  on the ***Focus*** bar in ISODAT NT),
- switched on Tune Scan (menu ***Scan | Tune***) in ISODAT NT
- admitted gas into the inlet system and a sufficient amount of gas to the ion source. (See also chapter 5, "Dual Inlet System").



Focusing of the ion beam is performed via ISODAT NT.

2.3.3 FOCUSING OF THE MASS SPECTROMETER

You can perform the focusing either manually or with the autofocus (AF). A compressed description of the focusing can be found in chapter 1, "Getting Started". Details about the handling of the software can be found in chapter 6, "Measurements".

NOTE: ***If you are not experienced with focusing we recommend using the autofocus.***

Detailed description of manual focusing

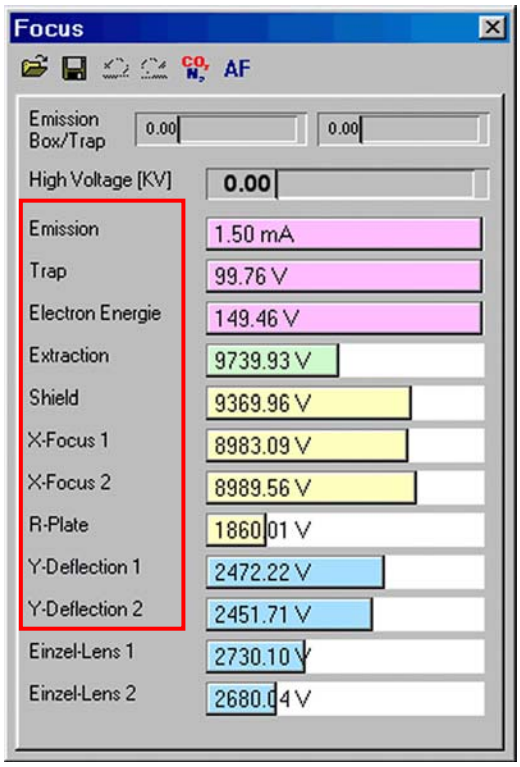
A first type of manual focusing, ***intensity*** focusing (also called ***peak shape*** focusing or ***sensitivity*** focusing) is seldom performed. Sensitivity and peak shape are both improved, when the extraction voltage (i.e. Extraction) is decreased.

In the vast majority of cases however, the so-called ***linearity*** focusing is performed as one particular type of manual focusing. It requires relatively high extraction voltages (i.e. high value of Extraction) in order to extract the ions out of the ionization housing. It will be described below.

1. ***Basic Adjustment of Parameters***

   Before the first focusing run, the following parameters should be preset:

- Set ***Trap*** to 40 V.
- Set ***Electron Energy*** to the maximum value.
- Set ***Emission*** to 1 mA (i.e. to 50 % below the maximum value of 1.5 mA).
- Set ***Extraction*** to a middle position (e.g. to 2600 V).



Thermo Finnigan

- Set ***X-Focus 1*** and ***X-Focus 2*** to a value smaller than middle position.
- Set ***R-Plate*** to a middle position.
- Set ***Y-Deflection 1*** and ***Y-Deflection 2*** to a middle position.

NOTE: ***In case of doubt, it is recommended to accept the default parameter values preset by Thermo Finnigan MAT's Final Test group, if you perform manual focusing!***

2 Ensure that intensity signal is monitored on a cup which can be used for peak centering (e.g. middle cup of universal triple collector cup):

- Be sure that gas is flowing into the source (e.g. CO_2)
- If the mass scale is already calibrated, jump to mass (i.e. 45 on middle cup of universal triple cup). Perform a Peak center. If signal is too low to proceed (below 50 mV), jump to other mass (i.e. 44 on middle cup of universal triple collector cup)
- If no Mass Calibration is available, perform a Mass Scan. Then seek a peak for ion source optimization. Afterwards, perform a Mass Scan again. Finally, perform a Mass Calibration.
- Switch on ***Tune Scan*** in ISODAT NT's ***Instrument Control*** Mode.

3 *Focusing*

The following steps are one proven possibility for achieving the optimum focusing parameters. Other procedures may yield just as satisfying results.

- For all steps where two potentials are simultaneously adjusted for maximum signal intensity (3b, 3c, 3e, 3f): repeatedly adjust both potentials to maximum signal intensity, until signal cannot be increased further
- If the signal exceeds 50V, either

  - decrease the gas flow into the IRMS or
  - use another mass (e.g. mass 44 instead of mass 45).

a ***Intensity*** focusing: Set ***Extraction*** voltage to 50 V below acceleration voltage.

Perform a peak center.

Maximize signal using ***Trap*** Voltage and ***Electron Energy***.

a1 ***Linearity*** focusing: Set ***Extraction*** voltage to 300 V below acceleration voltage.

Perform a peak center.

Set ***Trap*** Voltage and ***Electron Energy*** to maximum. If the signal should become unstable, slightly decrease the ***Electron Energy*** below maximum.

NOTE: ***The signals must always be optimized separately in turn (e.g. X-Focus 1 and X - Focus 2 are changed simultaneously by the same step width). After no more signal increase can be achieved, only change either of them until the maximum is found.***

NOTE: ***In the lens system, proceed outwards starting from the inside (according to fig. 2.3).***

b Maximize the signal using ***Extraction*** lens and ***X-focusing*** voltages.

c Adjust ***X-Focus*** lenses to maximum signal intensity.

d Maximize signal using ***Trap*** voltage and ***Electron Energy***.

e Adjust ***Y-Deflection*** lenses to maximum signal intensity.

f Adjust ***R-Plate*** to maximum signal intensity.

g Repeat steps 3b through 3f until the intensity cannot be increased further

h Try to increase the intensity further by slightly adjusting the ***Extraction*** voltage

i If the sensitivity, which can be measured using Dual Inlet system in ISODAT NT, is too low, increase ***Emission*** up to the maximum value. If the signal should become unstable, decrease ***Emission*** until the signal becomes stable. Then repeat steps 3a to 3h.

2.4 ION DEFLECTION

2.4.1 ELECTROMAGNET

The magnetic field providing the ion deflection is generated by an electromagnet with maximum field strength of 0.75 Tesla. The selection of the different masses is achieved by changing the magnetic field. In addition, the covered mass range can be extended by continuously varying the acceleration voltage.

2.4.2 MAGNET CURRENT REGULATOR

The magnet current regulator provides the current required to generate the electromagnetic field. For further information (e.g. the schematics of the signal path regulating the magnet current), refer to chapter 4, "Electronic Devices".

The relationship between magnet current and mass number is determined and stored by means of the mass calibration procedure. For more details, see the ISODAT NT online help.

2.5 ION DETECTION - COLLECTOR SYSTEM

2.5.1 GENERAL

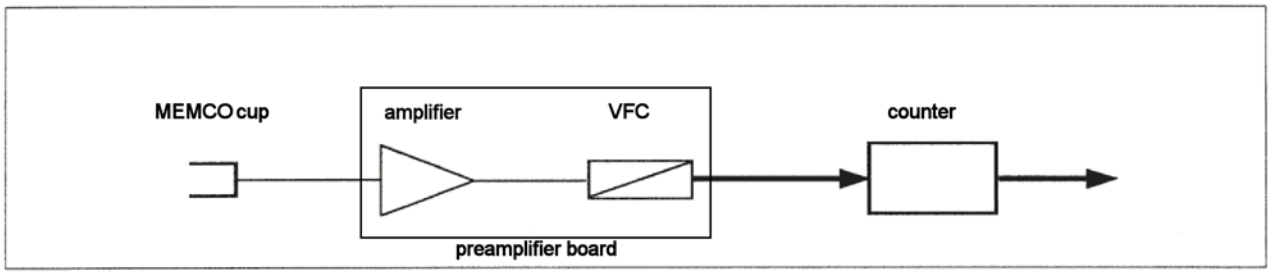
Several configurations of ion collector systems are available, the MEMCO and the universal CNOS collector system. It is also possible to install user-tailored systems. Additional information can be found in chapter 8, "Technical Information".

The collector system is installed in the collector system housing (See fig. 2-1). For HD isotope analysis, an optional collector system with two Faraday collector cups and amplifiers is available. The HD collector system has a separate housing, which is installed in the center of the analyzer region (to be flanged to the flight tube).

Each collector cup has its own amplifier (cp. universal CNOS collector) and the feedback resistor of the amplifier can be matched to the abundance of the isotope to be collected in this cup (see table 2-1).

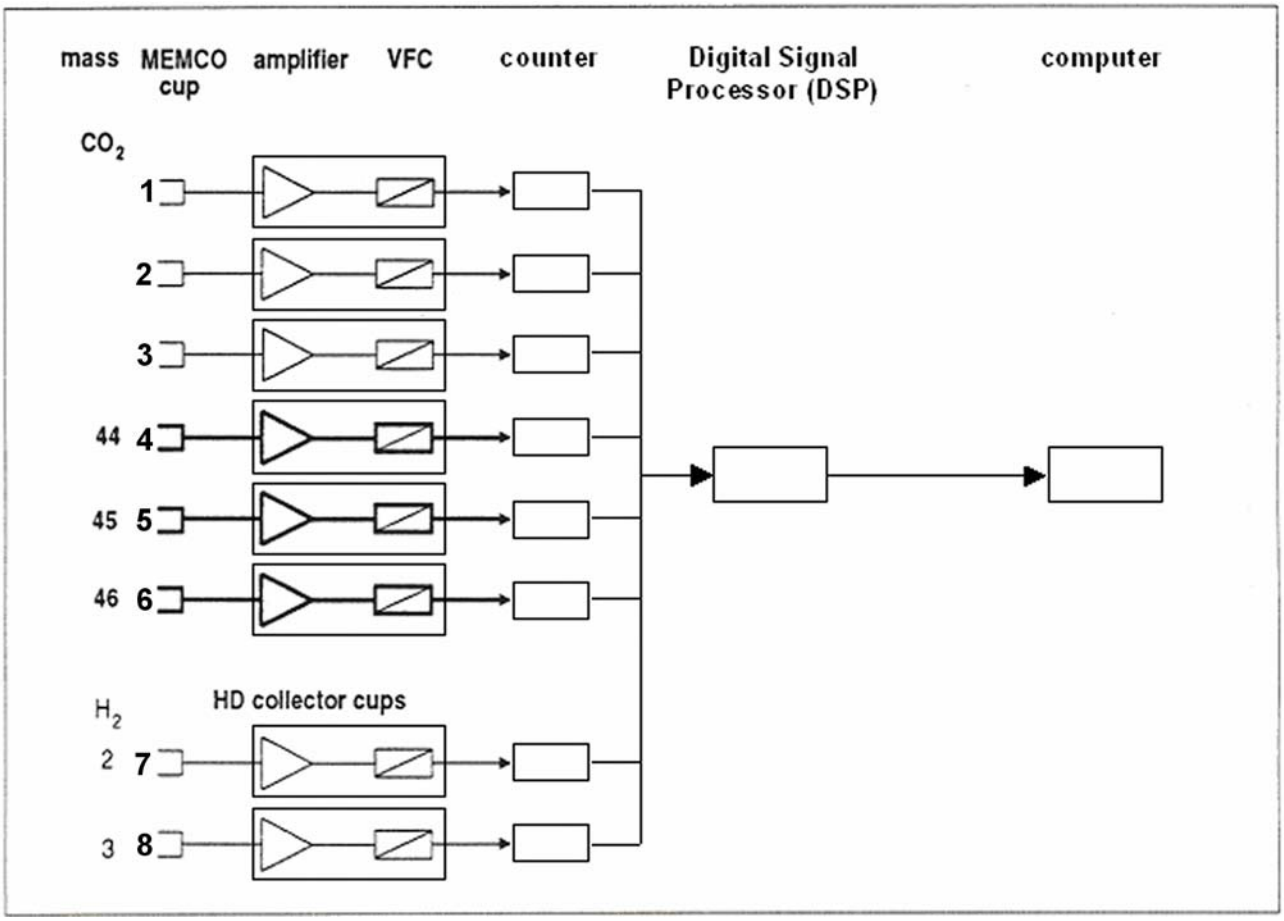
Each collector cup and its amplifier are connected to a voltage-to-frequency converter (VFC). The amplifier and the VFC are located on the preamplifier board. There are up to six VFCs for the MEMCO or CNOS collector system and two VFCs for the HD collector system available. They are allotted to one of the eight counters, so forming a measuring channel as shown in figure 2-6.

Fig. 2-6 Components that form a measuring channel



The converters transform the analog ion current signals into pulses. These pulses are fed to counters for a preselected integration time. At the end of each integration interval, the computer reads the number of counts and calculates the ion current ratios (see fig. 2-7).

Fig. 2-7 Function schematic of the ion detection system



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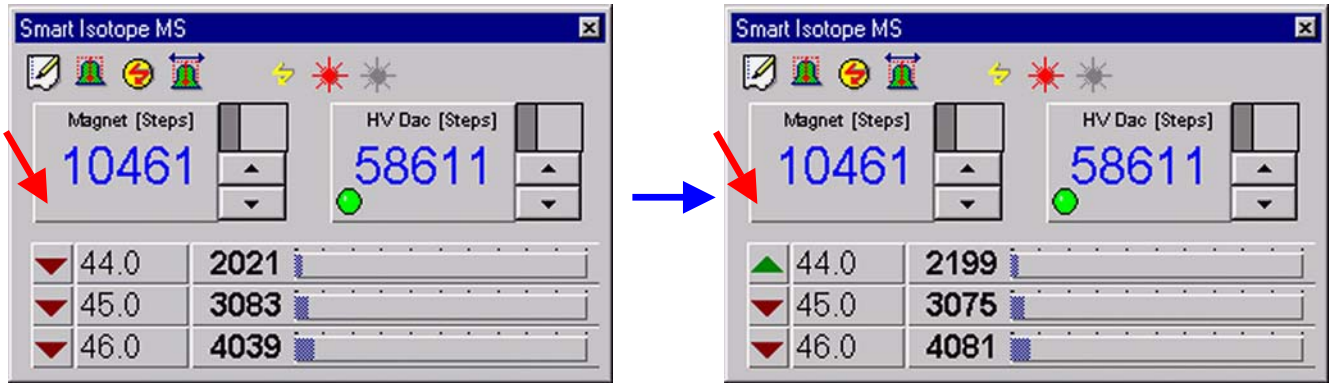
2.5.2 AMPLIFIER

The DC amplifiers have 100% inverse feedback. Their output voltage (50 V maximum) is the product of the input current and a feedback resistor.

The feedback resistor has to match the abundance of the isotope to be collected in the respective collector cup. Table 2-1 shows the resistance values to be used for the isotopes of the different gases.

NOTE: ***Use ISODAT NT's Smart Isotope MS bar shown below to switch between resistors: a click on ▼ (i.e. low resistance) changes the symbol to ▲ (i.e. high resistance) and vice versa.***



To minimize the influence of environmental changes on the amplifier stability, the amplifier housing is evacuated to a pressure below 10^{-4} mbar. This is achieved by connecting the amplifier respectively its housing to the forevacuum pump of the inlet system, and then to the side port of the turbomolecular pump.

Table 2-1 Values of the feedback resistors matching the natural abundance of the listed isotopes

Gas	*m/z*	*Resistor [Ω]*	*Capacity [pF]*
H_2	2	$1 * 10^9$	150
	3	$1 * 10^{12}$	2
N_2	28	$3 * 10^8$	470
	29	$3 * 10^{10}$	5
	30	$1 * 10^{11}$	2
O_2	32	$3 * 10^8$	470
	33	$1 * 10^{12}$	2
	34	$1 * 10^{11}$	2
CO_2	44	$3 * 10^8$	470
	45	$3 * 10^{10}$	5
	46	$1 * 10^{11}$	2
SO_2	64	$3 * 10^8$	470
	66	$1 * 10^{10}$	15

The product R * C is approximately a constant equaling the time constant of the amplifier. Usually, it amounts to 0.15 s - 0.2 s.

CAUTION: ***Never touch the surface of the high ohmic switchable resistors! Already a slight touch of a fingertip would contaminate the resistors resulting in signal instability.***

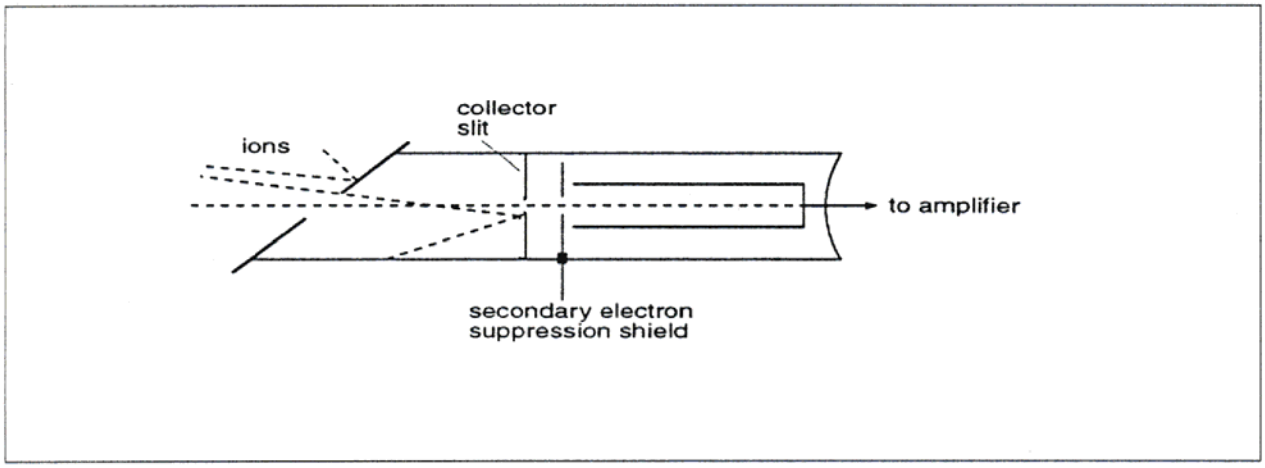
If you nevertheless accidentally once touched them, clean them using alcohol.

2.5.3 COLLECTOR SYSTEMS

The collector systems cover the mass range from 1 to 150 m/z at 10 kV accelerating voltage, allowing a resolution of m/Δm = 200 (10% valley) with 1.5 mm cups.

Owing to the high dispersion of the analyzer system, the distance between the collectors is extremely large (e.g. approx. 4 mm between masses 44 and 45). Thus, it was possible to design the Faraday collectors as deep, shielded buckets with integrated secondary electron suppression shields (fig. 2-8). As a result, effects are eliminated that might degrade the ion current measurement.

Fig. 2-8 Design of a Faraday collector cup

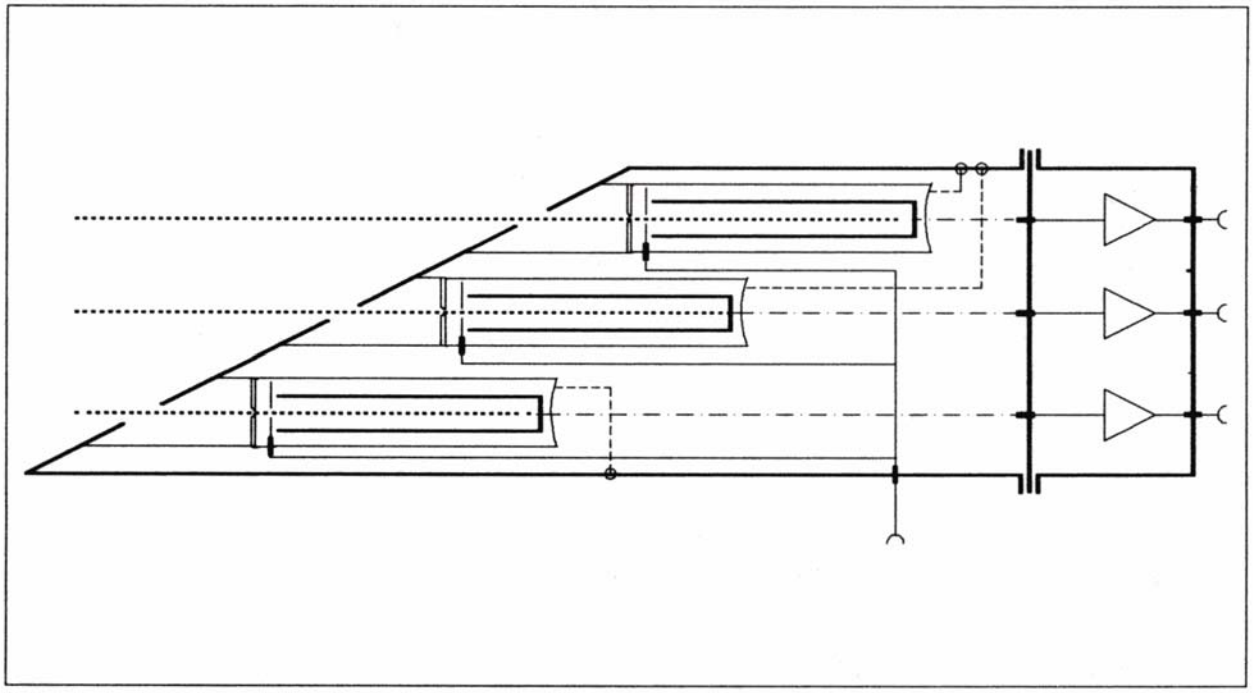


2.5.3.1 MEMCO COLLECTOR SYSTEM

The MEMCO (**M**ulti **E**lement **M**ulti **CO**llector) collector system comprises three to six identical cups.

The 3-cup version (fig. 2-9) allows simultaneous measurement of two isotope ratios from the same sample, e.g. $^{13}C/^{12}C$ and $^{18}O/^{16}O$ of CO_2.

Fig. 2-9 Design of a 3-cup MEMCO



The 6-cup version permits the cup configuration to be preset for the consecutive measurement of different gases. This way, it is not necessary to break vacuum in order to alter the positions of the cups. As with the 3-cup version, simultaneous measurement of two isotope ratios of the same gas can be performed.

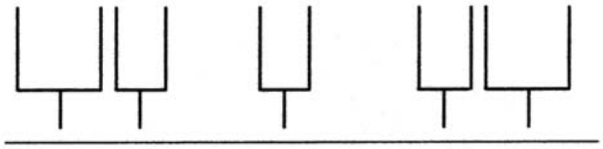
NOTE: ***Different gases may jointly use one cup in order to reduce the total number of cup measuring channels.***

2.5.3.2 UNIVERSAL CNOS COLLECTOR SYSTEM

The universal CNOS collector system is suitable for the measurement of N_2, O_2, CO_2, and SO_2. This collector (see fig. 2-10) consists of five individually shielded deep Faraday cups. A central small cup is combined with two pairs of differing cups. Each pair comprises a small cup on the inner side and a wide cup on the outer side. Both cups of a pair are electrically connected i.e. the cups 1 / 2 are connected with an amplifier and the cups 4 / 5 are connected with another amplifier. The central cup 3 is connected with a third amplifier. This

way the two cups work as one wide cup without having the disadvantages of a single cup with the required width (e.g. insufficient suppression of secondary electrons).

Fig. 2-10 Principle of the universal CNOS collector system



		Cup: 1	2	3	4	5
Experiment/	CO_2		44	45	46	
Mass:	N_2 / CO	28		29		30
	NO	30		31		32
	O_2	32		33		34
	SO_2	64		66		

With this combination, there is no need to change the feedback resistors when e.g. changing from the analysis of CO_2 to that of N_2. The cup configuration is automatically switched by the ISODAT NT software. In case of switching to SO_2 analysis, the amplification factor for mass 66 has to be changed. The resistors are changed and adjusted (including high voltage) via ISODAT NT. Refer to table 2-1.

The CNOS collector system can be accessed together with the optional HD collector system during one experiment (e.g. for background checks).

2.5.3.3 HD COLLECTOR SYSTEM

The HD collector system is a dual Faraday collector assembly for hydrogen isotope measurement on a same ion path, operating in parallel to the MEMCO or universal CNOS collector systems. The HD collector is located in the middle of the analyzer tube and covers the mass range from 1.5 to 14. The HD collector cups are designed like those of the MEMCO system.

MAT 253

OPERATING MANUAL

VACUUM SYSTEM

Issue 04/2002

Thermo Finnigan

3.1 PUMPING SYSTEM

The Thermo Finnigan ***MAT 253*** is supplied with two optional pumping systems. The instrument is equipped either with or without a differential pumping system.

a. The **standard version** comes with a turbomolecular pump to evacuate the analyzer system at a rate of 210 l/s (type: TMH 262, manufacturer: Pfeiffer). The required forevacuum is provided by a rotary pump rated at 5 m^3/h (type: DUO 5, manufacturer: Pfeiffer).
b. The **optional differential pumping system** with an additional turbomolecular pump improves the vacuum in the analyzer system of the instrument. This improvement is required to reduce the high portion of He carrier gas of peripherals such as an elemental analyzer or a GC. The results are better abundance sensitivity, better peak shape and improved signal to background ratio at high ion source pressures.
   A vacuum lock or flow restriction separates the ion source section from the analyzer region. The additional turbomolecular pump evacuates the analyzer region at a rate of 60 l/s (type: TMH 071P, manufacturer: Pfeiffer).
   The required forevacuum for both pumps is provided by a rotary pump rated at 5 m^3/h (type: DUO 5, manufacturer: Pfeiffer).

The inlet system is pre-evacuated by a forevacuum pump rated at 1.3 m^3/h (type: DUO 2.5, manufacturer: Pfeiffer).
The wasteline turbomolecular pump (type: TMH 071P) backed by a forevacuum pump (type: DUO 2.5) is used to provide high vacuum conditions in the inlet system.
For more information, see also paragraph 3.3, Turbomolecular Pump.

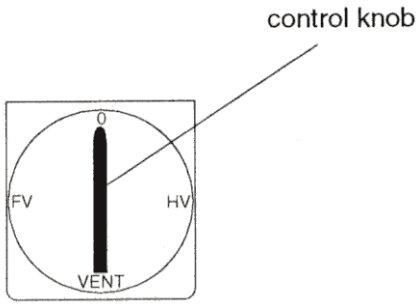
3.2 VACUUM SYSTEM CONTROL UNIT

The high vacuum of the pumping system is monitored by a Penning vacuum gauge, which is attached to the left side of the ion source housing. The forevacuum is controlled by a Pirani vacuum gauge located in the inlet system.

3.2.1 VACUUM IN AMPLIFIER HOUSING

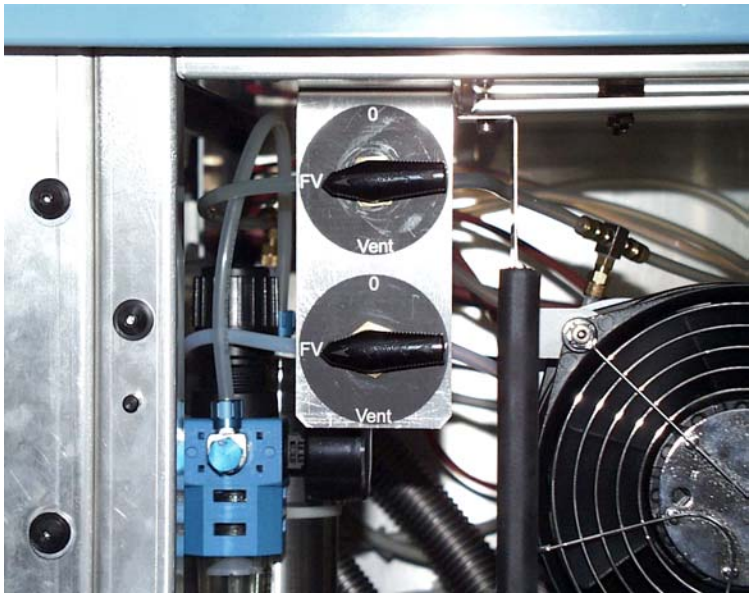
To improve the stability of the amplifiers, the amplifier housings can be evacuated. Using a 3-port valve (see fig. 3-1), the following steps are required:

Fig. 3-1 3-port valve



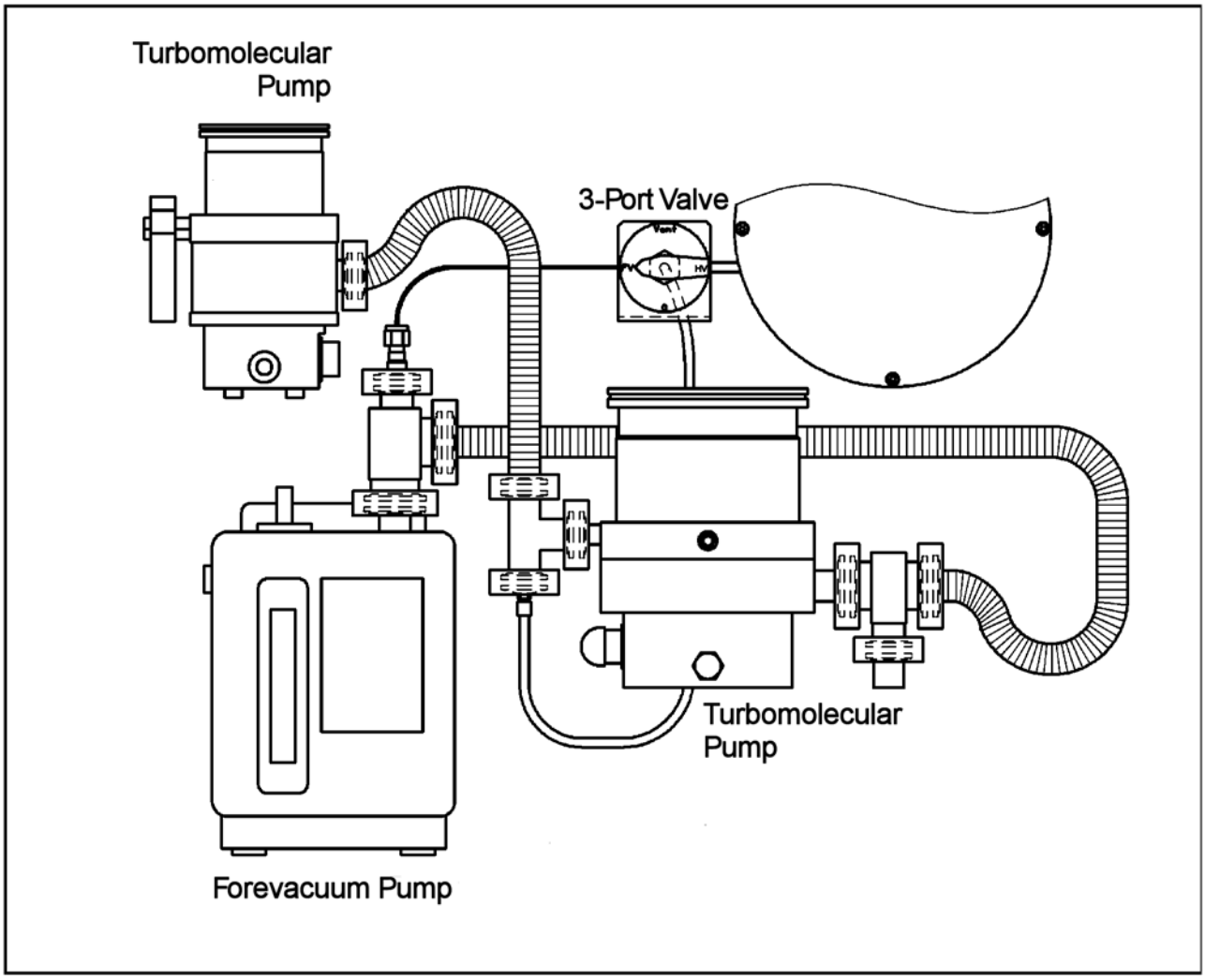
1. Turn control knob anti-clockwise from 0 to FV.
2. After approx. 30 minutes turn control knob clockwise from FV to HV. The housing is evacuated.
3. Before exchanging resistors on the amplifier, turn control knob clockwise from HV to Vent. After having vented the housing, the cover can be removed.
4. In case of a pump breakdown, restart the turbo pump. To do so, press the pump switch twice.

In case the ***MAT 253*** has an HD collector system installed, separate 3-port valves for the HD amplifier housing (bottom) and the MEMCO amplifier housing (top) are available. They are located at the right side of the IRMS.



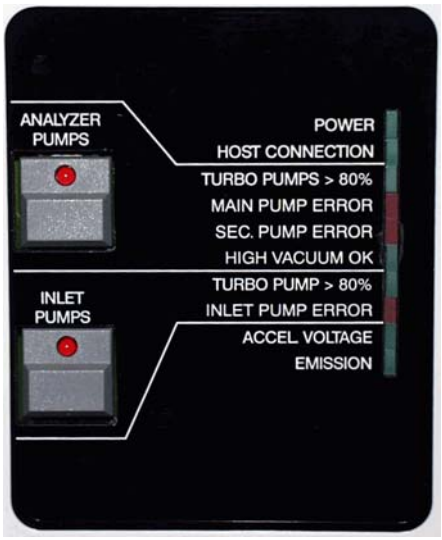
NOTE: ***Each amplifier housing has to be evacuated separately.***

Fig. 3-2 Schematic view of the differential pumping system



Thermo Finnigan

Fig. 3-2 Control Panel



The row of LEDs is lit when the turbomolecular pumps reach 50% of their nominal rotational speed. The green LEDS indicate the proper function of the respective system part. The red LEDs indicate an error in the respective system part.

Pushbuttons switches:

ANALYZER PUMPS	starts pumping system (turbomolecular pumps and forevacuum pump for analyzer system).
INLET PUMPS	starts the turbomolecular pump and the forevacuum pump for the wasteline and the inlet system.

NOTE: ***In case of problems, e.g. a high vacuum > $1O^{-4}$ mbar, the source is cut off automatically.***

LEDs:

	POWER	indicates the supply of main voltage
	HOST CONNECTION	indicates that the DSP program located on the DSP/PCI card on the computer is started **NOTE:** ***If the LED is not lit with ISODAT NT started, no connection between the IRMS and the computer has been established.***
ANALYZER PUMPS	**TURBO PUMPS > 80%**	indicates that the turbomolecular pumps of the analyzer have reached more than 80% of their nominal rotational speed
	MAIN PUMP ERROR	indicates an error of the analyzer main pump
	SEC PUMP ERROR	indicates an error of the secondary analyzer turbo-molecular pump **NOTE:** ***In case the instrument is not equipped with a differential pumping system, this LED is not functional.***
	HIGH VACUUM OK	indicates that the vacuum has fallen below the set point of the Penning gauge **NOTE:** ***The ion source can only be switched on when this LED is lit.***
INLET PUMPS	**TURBO PUMP > 80%**	indicates that the turbomolecular pump of the inlet system has reached more than 80% of its nominal rotational speed
	INLET PUMP ERROR	indicates an error of the inlet turbomolecular pump
	ACCEL VOLTAGE	lit when high voltage is provided to ion source
	EMISSION	only lit when filament of the ion source operates

NOTE: ***If the vacuum system works properly the red LEDs are not lit!***

3.2.1.1 GUIDANCE FOR TROUBLESHOOTING

If either one of the green vacuum control LEDs units is not lit or one of the red LEDS is lit, the instrument should be checked immediately.

HIGH VACUUM OK LED **off**: (or too much back-ground for m/z=28/40 is meas-ured in the mass spectrometer)	**a.** There might be a leak in the system. **b.** There might be a malfunction of the Penning gauge.
ACCEL VOLTAGE LED **on:** EMISSION LED **off:**	The cathode might be burned out. Checking and removal of the fila-ment is described in detail in chapter 8, Technical Information.
ACCEL VOLTAGE LED **off**: EMISSION LED **on:**	The high voltage is tripped when the current exceeds 0.2 mA. This may be caused either by sparking or by a short circuit in the source or the connections to the source.
ACCEL VOLTAGE LED **off**: EMISSION LED **off:**	The ion source is not switched on. Reset and switch on the ion source via software. When LED "HIGH VACUUM OK" is not lit, see above.

3.3 TURBOMOLECULAR PUMP

3.3.1 GENERAL

The turbomolecular pump works completely mechanically by rotor disks imparting momentum to the gas molecules. Baffles or cryogenic traps are not necessary for retention of pump fluid vapors. The vacuum system is roughed by rotary pumps through the turbomolecular pumps. Furthermore, the turbomolecular pumps are started at atmospheric pressure. Hence, this ar-

rangement also obviates the need for a high vacuum pump valve. Thus, the rated capacity of pump speed is available without restriction at the connecting flange of the source housing. Because the molecular pump principle works in the molecular flow region only, the turbomolecular pumps require a forevacuum pump. This pump is roughing the vacuum system through the turbomolecular pump down to the upper limit of the turbomolecular pump operating range.

The turbomolecular pumps installed in the instrument are air-cooled. In case of a mains failure there is a delayed venting provided by a venting valve. When starting the turbomolecular pump the venting valve is closed immediately. After stopping (by mains failure or switch-off), delayed venting is performed. The vent valve remains open until the next start cycle of the electronic unit. In absence of current, the valve is open.

For details regarding function and design of the turbomolecular pumps, see the operating manual of the manufacturers.

3.3.2 CONTROL ELECTRONICS AND POWER SUPPLY

Each turbomolecular pump has an integrated control electronics and power supply. The pumps are started with the switches on the control panel. In addition to the LEDs on the control panel, several LEDS for error indication are directly attached to the turbomolecular pumps. For type of error, see operating instructions of the pump manufacturer (Pfeiffer).

MAT 253

OPERATING MANUAL

ELECTRONIC DEVICES

Issue 04/2002

Thermo Finnigan

4.1 OVERVIEW OF THE ELECTONIC DEVICES

Various electronic installations are required to carry out the procedures of the Thermo Finnigan ***MAT 253***. These comprise the following boards:

- Power Distribution
- Internal & External Inlet Controller
  - Plug & Measure Adapter
- Data Acquisition
  - Data logger
  - Groundplane
  - Preamplifier board
  - 55V Power Supply
- Ion Source Controller
  - Emission Regulator
  - High Voltage Potential Controller
  - High Voltage Supply
  - Bus Controller
- Magnet Current Regulator
- DEL-PCI Controller

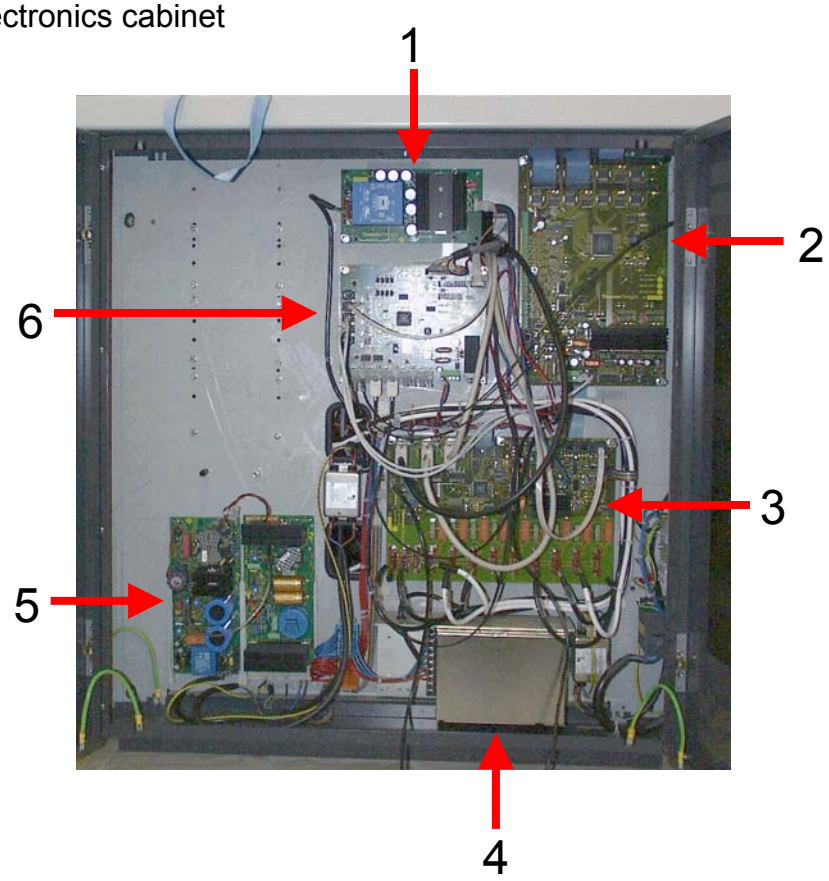
The electronics of the ***MAT 253*** mass spectrometers contain complicated and numerous circuits. Therefore, only qualified and skilled electronics engineers should perform servicing.
It is recommended to call for the Thermo Finnigan MAT Service if servicing is required. It is further recommended to use Thermo Finnigan MAT spare parts only, since many parts are specially selected. When replacing fuses, only use the correct type.
Before calling the Thermo Finnigan MAT Service, please try to localize the defect. A precise description of the defect will ease the repair and reduce the costs.

WARNING: ***Opening the electronics cabinet is only allowed for maintenance purposes by qualified personal. Be careful when removing the protective covers from plugs, cables and other parts.***

DANGER HIGH VOLTAGE!

Most of the electronic boards listed above are parts of the electronics cabinet that is located on the rear side of the IRMS. This chapter describes the basic structure of the electronic equipment. Figure 4-1 shows the electronics cabinet of the Thermo Finnigan ***MAT 253***.

Fig. 4-1 Electronics cabinet



For a description of selected board parts, see the following explanations after the corresponding numbers.

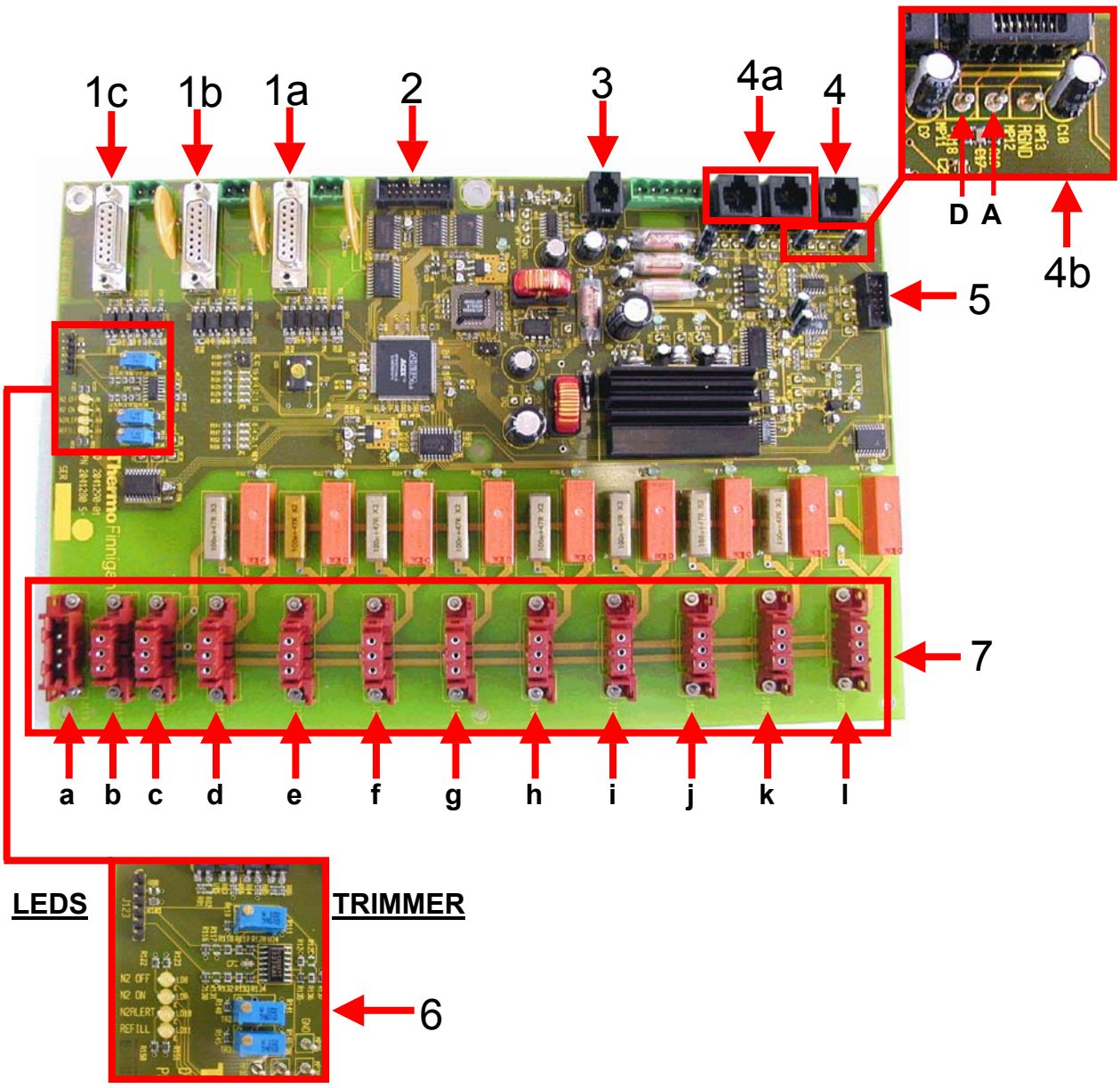
1	Power supply 55 V
2	External Inlet Controller
3	Power Distribution
4	Power supply 24 V
5	Magnet Current Regulator
6	Data logger

On the following pages are described selected boards that are located in the electronics cabinet as well as in other parts of the ***MAT 253***.

4.2 POWER DISTRIBUTION

The power distribution board (Part No. 204 1280) is located in the right side of the electronics cabinet.

Fig. 4-2 Power distribution



For a description of selected board parts, see the following explanations after the corresponding characters.

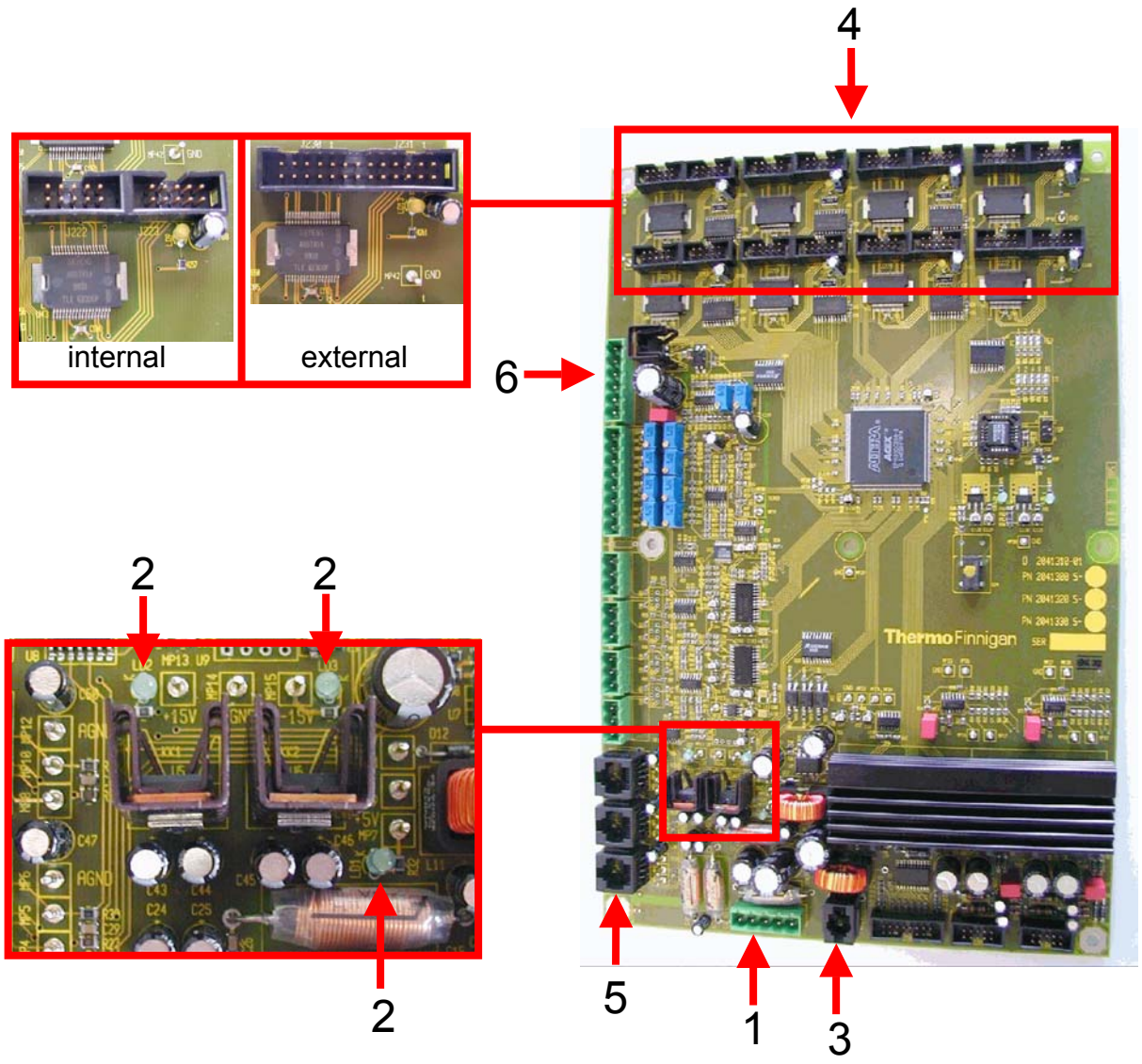
1a	Connection to turbo pump analyzer
1b	Connection to turbo pump ion source
1c	Connection to turbo pump inlet system
2	Connection to front panel
3	Connection to serial interface
4	**Connection to penning gauge** The penning gauge for the high vacuum measurement is connected here. This is important for the proper function of the high vacuum security.
4a	**Vacuum Sensors** Additional vacuum gauges can be connected here. Anyway, the vacuum security is not affected.
4b	**Tapping points for penning gauge** The threshold (**D**) and actual (**A**) current values of the penning gauge can be re-measured here.
5	Connection to magnet current regulator
6	**Controls for refill equipment** The settings of the trimmers determine the trigger thresholds. The LED "N2 OFF" is lit when the liquid nitrogen has reached the maximum level. The LED "N2 ON" is lit when the liquid nitrogen has fallen below the minimum level. The LED "N2ALERT" is lit when the liquid nitrogen has reached a critical level. The LED "REFILL" is lit when the refill is switched on.
7 a b c d e f g h i j k l	**Connections to all 230 V consumers** Current input Power supply 24 V Power supply 55 V →Socket J503 Refill equipment Inlet valve heater →Socket J1112 Ion source controller →Socket J322 Ion source heater →Socket J1114 Source heating Inlet system heater →Socket J1110/J1111 Inlet pump →Socket J1109 Pump source & analyzer →Socket J1108 MS heater →Socket J1113

Thermo Finnigan

4.3 INLET CONTROLLER

The IRMS is provided with two variants of the inlet controller board (Part No. 204 1300). The internal board is located at the top right side of the electronics cabinet. The external board is located on the right side of the IRMS.

Fig. 4-3 Inlet Controller



For a description of selected board parts, see the following explanations after the corresponding numbers.

1	Power supply 24 V
2	These LEDs are lit when the supply current is available.
3	Connection to serial data link
4	Control of the internal/external valve banks
5	Connection to optional pressure sensors
6	Connection to microvolume

The inlet controller board has the following functions:

- Control of the valve banks
- Connection for three additional vacuum gauges
- Connection for the operation device for the cooling trap
- Motion control of the bellows

4.4 THE PLUG AND MEASURE CONCEPT

Each peripheral device has its own plug and measure code.

This code is encoded:

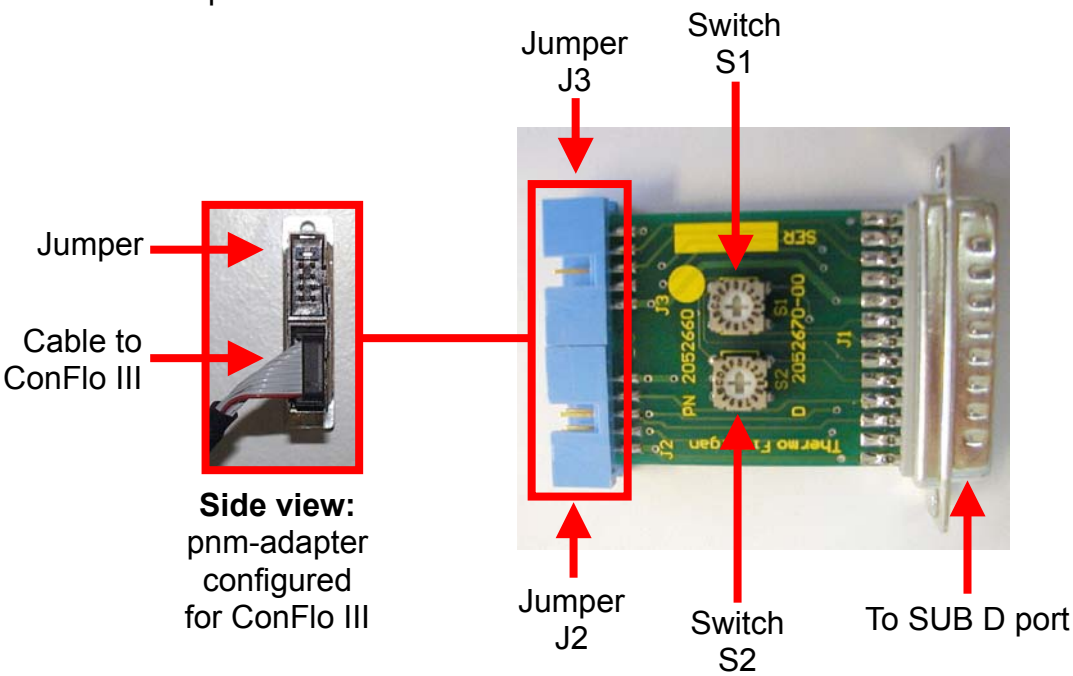
- either in the cable to the device
- or in a plug and measure adapter

  This is also used for downward compatibility using an old peripheral device in combination with the new ***MAT 253*** electronics.

Each peripheral device can be connected to any of the five SUB-D ports (up to five devices simultaneously). The instrument recognizes the kind of peripheral device and the SUB-D port used for it automatically, when a Configuration requires this device. Otherwise (e.g. when the device cable is unplugged accidentally), an error message will be displayed.

4.4.1 THE PLUG AND MEASURE ADAPTER

Fig. 4-4 PNM-Adapter



The plug & measure adapter (pnm-adapter) is used for the connection of the peripherals. It is pre-configured at the Thermo Finnigan factory for a defined option (e.g. ConFlo). A supplementary reconfiguration by the user is not recommended. The plug & measure adapter

is connected to one of the five identical SUB D ports at the rear panel of the IRMS. The peripheral is then connected to the IRMS via the bottom port of the pnm-adapter.
The peripherals are identified by the settings of the turn switches and the jumpers. The turn switches are used to specify the pnm-ID for the peripheral (e.g. for ConFlo set S1 to 2). The jumpers are also used to identify the kind of peripheral that is connected to the IRMS). If indicated, the lowest two contacts of the plug socket at the pnm-adapter, i.e. either socket J3 or J2, are cut short (from the outside of the pnm-adapter).

NOTE: ***Some external options are connected to the SUB D ports without using the plug and measure adapter.***

4.4.2 GROUNDING CABLE FOR PERIPHERALS

Those peripheral devices, which are operated by using a plug and measure adapter, have to be connected to the IRMS with a grounding cable. It is not necessary for peripheral devices, which already run using a new cable (e.g. PreCon, GP Interface).

The peripheral is connected with a green-yellow PE cable to the IRMS. The grounding contact is a screw at the right side of the main switch at the lower part of the electronics cabinet.

4.4.3 CONFIGURING THE PLUG AND MEASURE DEVICES

- ***Internal options:*** each option has a dedicated plug socket (with the exception of the tube cracker second bank). The software recognizes whether an option is connected to that socket or not.
- ***External options:*** peripheral devices. Five SUB-D ports are located at the instrument's rear side. The option is recognized, because it is encoded with a pnm-ID. The electronics recognize:
  - that there ***is*** an option connected to a certain port
  - which ***kind*** of option it is

Peripheral	pnm-ID	2nd ID	Jumper pin 9/10	New cable
ConFlo2/3	0x02	0x03	J3	
GCC2/3	0x04	0x05	J3	
GasBench	0x08	0x09		
PreCon	0x0A	0x0B		Yes
ProRef	0x0C	0x0D	J3	
MultiInlet	0x0E	0x0F		
AcidPump	0x18	0x19	J2	
GC/GP	0x10	0x10		Yes
Dual Inlet				
MP1				
MP2				
TC1				
TC2	0x12	0x13		

pnm-ID: by switches inside the pnm-adapter or by shortcuts inside the cable

2nd ID: if two instruments of the same type are installed

Jumper pin 9/10: if indicated, the lowest two contacts of the plug socket at the pnm-adapter, i.e. either socket J3 or J2, are cut short (from the outside of the pnm-adapter).

New cable: No pnm-adapter. Instead, an exchange of the cable is necessary.

NOTE: ***The Tube Cracker (TC) second bank is applied to two ports of the inlet board for external option. Therefore, it has a pnm number.***

4.4.4 USING THE OPTIONS GAS BENCH, PRECON AND TUBECRACKER WITH ANOTHER MASS SPECTROMETER

The three options Gas Bench, PreCon or TubeCracker are available in two versions:

- One for the ***DELTA^{plus}XP***, ***DELTA^{plus}Advantage*** or ***MAT 253*** and
- one for the ***DELTA^{plus}***, ***DELTA^{plus}XL*** or ***MAT 252***.

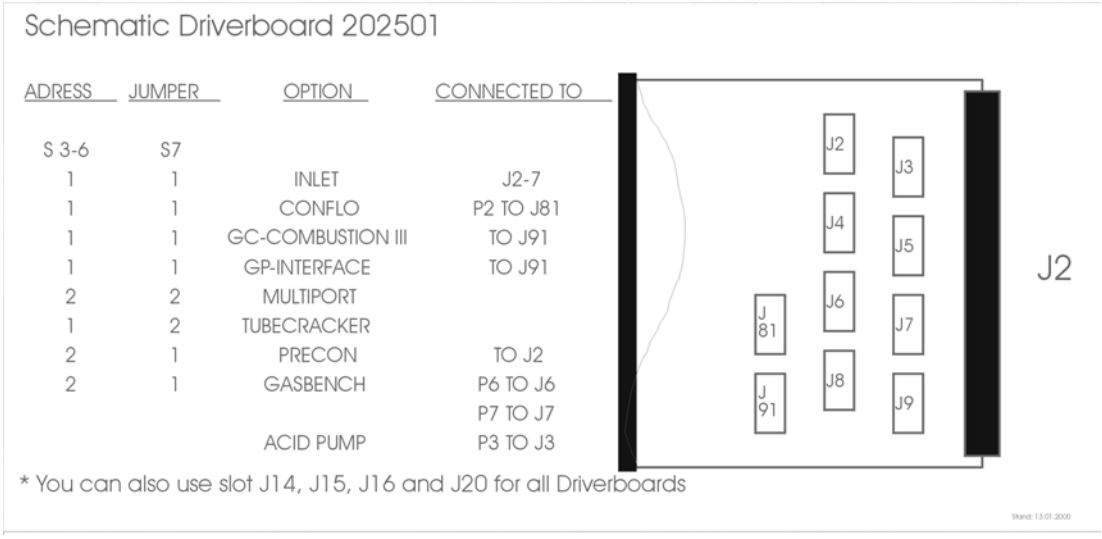
Praxis has shown that in some laboratories are not always connected to the same mass spectrometer, especially the continuous flow options like the Gas Bench and PreCon. Depending on the analytical problem, they are sometimes transferred from one mass spectrometer to another. It is possible to switch between the newer generation mass spectrometers (***DELTA^{plus}XP***, ***DELTA^{plus}Advantage*** or ***MAT 253***) and the older generation, when the subsequent **important guidelines** are followed.

1. **Applying an older peripheral device to a newer generation mass spectrometer**
   Use a plug and measure device, in case of the PreCon and the GP-Interface use a new connection cable.
   a. Each option delivered after June 2002 is delivered with the necessary hardware.
   b. For older devices, order a plug and measure device (Part No. 205 2660) or a connection cable.
2. **Applying a peripheral device ordered with or for a newer generation mass spectrometer to an older generation mass spectrometer**
   Connect the connection cable to the driver board (see figure 4-4a).
   a. For most peripheral devices the driver board supplied with the older generation mass spectrometer can be used directly
   b. For devices like Gas Bench, PreCon or TubeCracker it is recommended to order a dedicated driver board (Part No 202 5001).
   Alternatively, a new address can be assigned to the driver board delivered with the older generation mass spectrometer (see figure 4-4a).

ATTENTION: ***NEVER put the plugs of the peripheral device connection cable into the wrong socket on the driver board; serious damage of the driver board may occur, which is not covered by any warranty.***
On the other hand, if the jumpers a set to a wrong address, the device cannot be addressed, but nothing will be damaged.

3. **Applying an option using an IEEE interface (Carbonate device/ HDO device) delivered with an older generation mass spectrometer to a newer generation one**
   Install an additional IEEE interface (Part No 079 9130).

Figure 4-4a Driver board



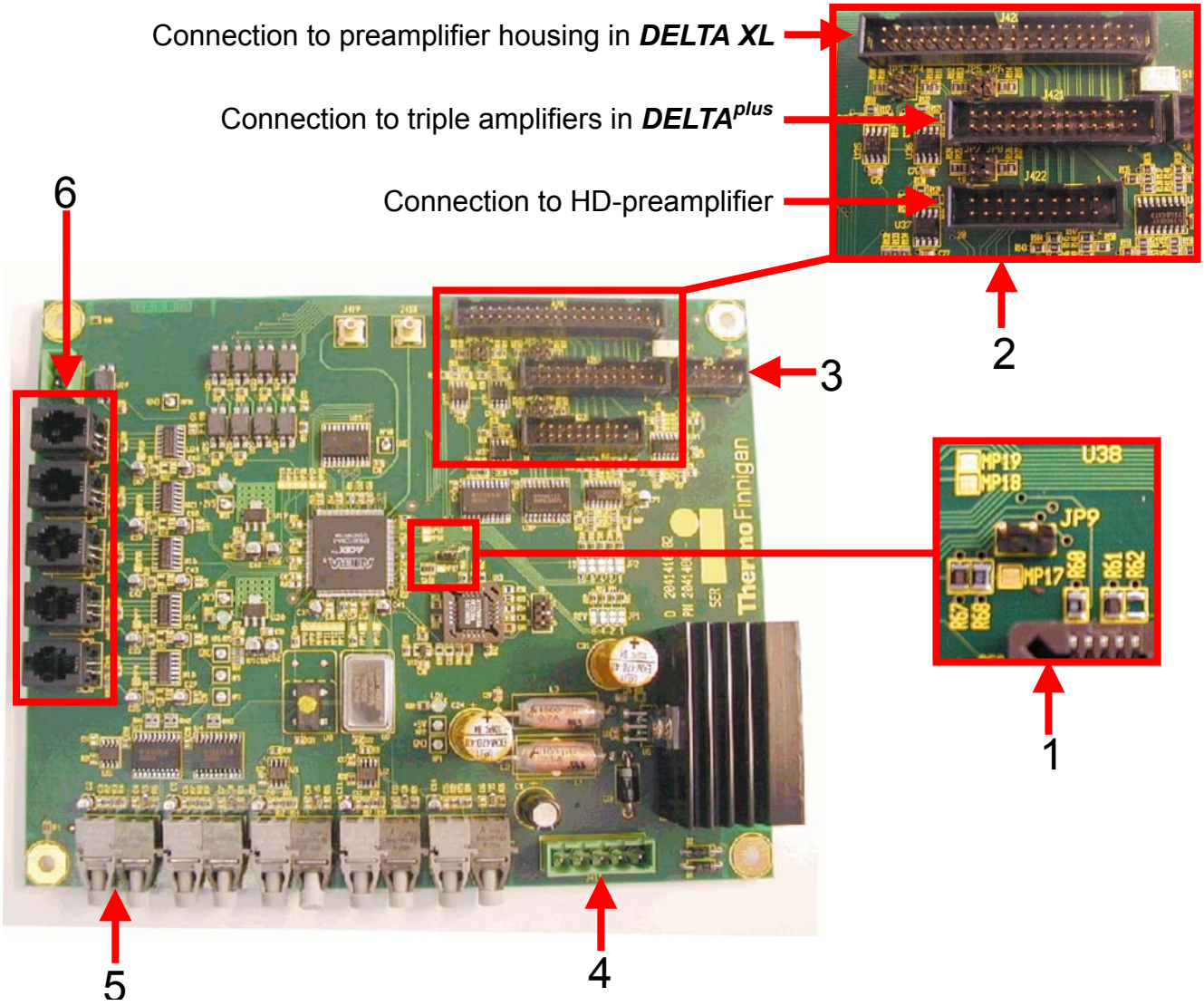
4.5 DATA ACQUISITION

Several boards are involved in the data acquisition process. These boards are described in the following.

4.5.1 DATA LOGGER

The data logger board (Part No. 204 1400) is located in the electronics cabinet. It is used for recording measurement readings and serves as distributor for the serial data link.

Fig. 4-5 Data logger



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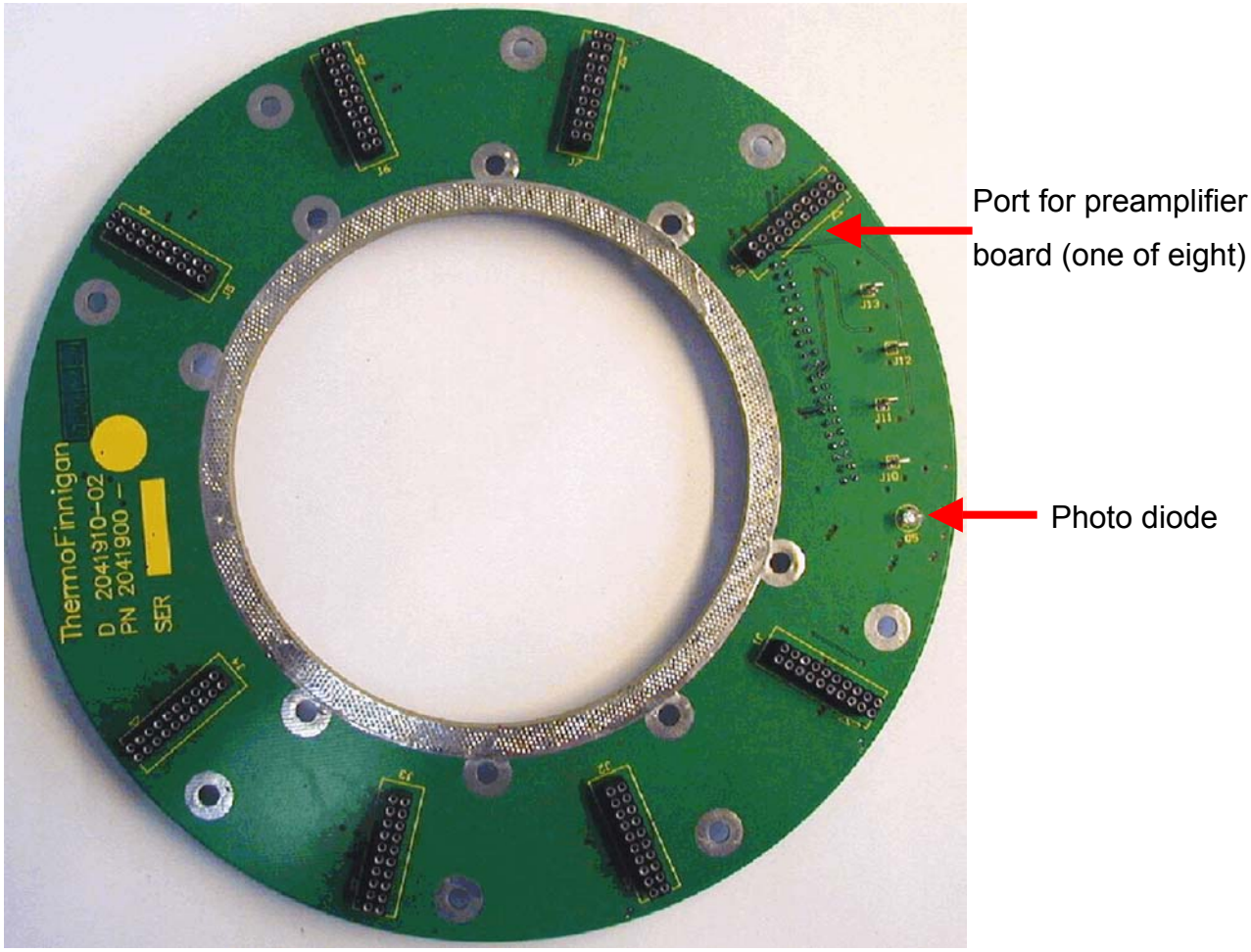
For a description of selected board parts, see the following explanations after the corresponding characters.

1	This jumper (JP9) is used to switch on the power supply of the amplifiers when the amplifier housing is open. **ATTENTION:** ***The amplifiers can be damaged when they are exchanged while the power supply is switched on.***
2	Connections to other boards
3	Connection to 55 V power supply
4	Connection to 24 V supply
5	Connection from serial interface to PC (DEL-PCI controller) via optical fiber
6	Serial data link to all other boards (e.g. power distributor, inlet control)

4.5.2 GROUNDPLANE

The groundplane (Part No. 204 1900) is located in the amplifier housing. It carries the eight preamplifier boards with amplifiers and VFCs. It connects the amplifiers to the data logger. The photo diode switches off the supply voltage when the lid of the amplifier housing is removed. It serves as an additional precaution and only works properly when the workplace is sufficiently lit.

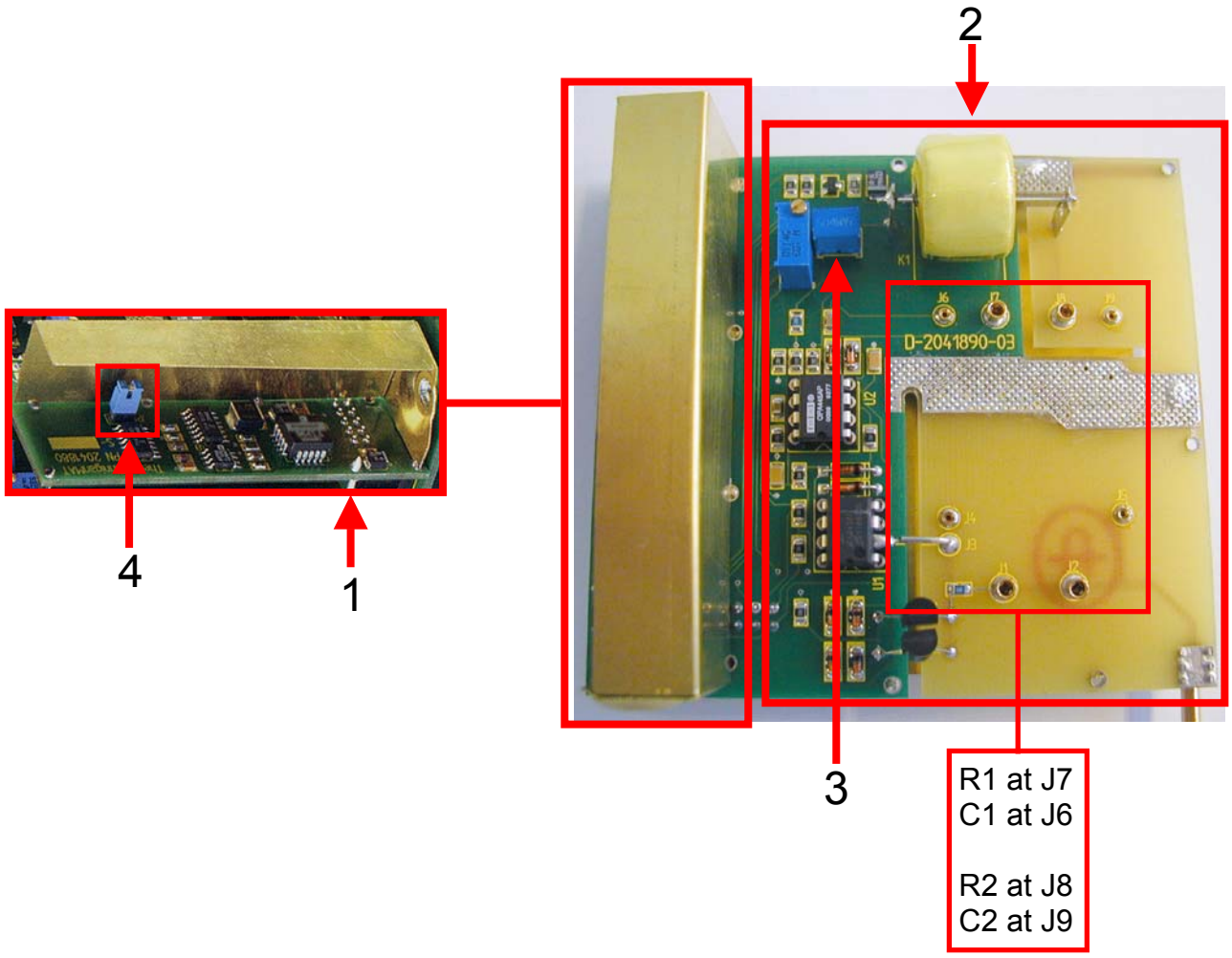
Fig. 4-6 Groundplane



4.5.3 AMPLIFIER & VFC

For each of the eight acquisition channels a preamplifier board (Part No. 204 1880) is attached to the groundplane. It carries the amplifier extended by a resistor switch as well as the voltage-to-frequency converter (VFC). Here, the signals coming from the cups are amplified and converted and then transferred to the counters.

Fig. 4-7 Preamplifier board



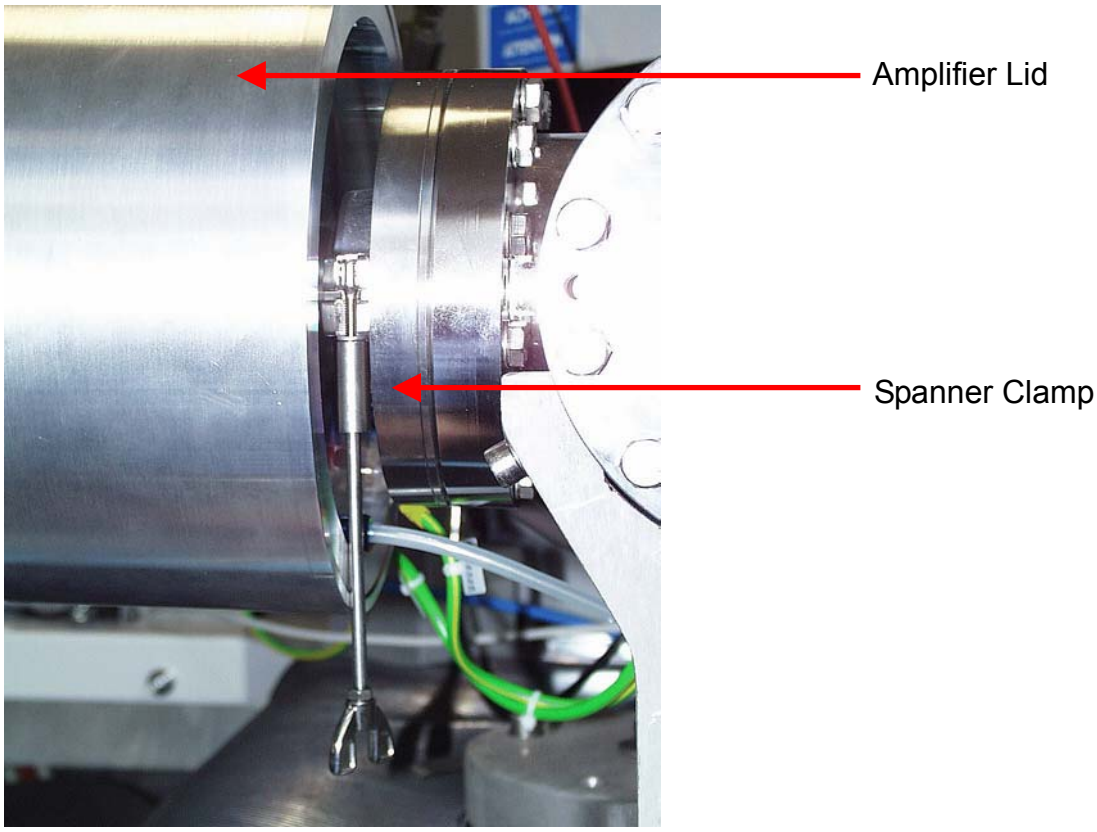
For a description of selected board parts, see the following explanations after the corresponding numbers.

1	VFC
2	Amplifier
3	Setting of the time constant
4	Jumper to disconnect amplifier and VFC (for service purposes)

4.5.4 CHANGING THE AMPLIFIER

When working with the amplifier, take care of the spanner clamp: it must be fixed at the flange beneath the amplifier housing and closed.

Fig. 4 - 8: Bottom side of amplifier housing



NOTE: ***If the spanner clamp is not fixed attach it to the flange before continuing your work.***

This ensures that the amplifier cover can be removed without stripping the amplifier ground-plane with the preamplifier boards from the collector.

4.5.4.1 WORKING WITH THE COLLECTOR - REMOVING THE AMPLIFIER HOUSING

If it is necessary to work directly at the collector or its housing, proceed as follows:

1. Control, whether the spanner clamp fits correctly.
2. Remove the amplifier lid.

3 Remove each preamplifier board from the groundplane separately and mark its position.

4 Unclip the spanner clamp by tearing or pressing slightly.

5 If the amplifier bottom has to be removed totally, unfix the data cable and the vacuum tube.

4.5.4.2 WORKING WITH THE COLLECTOR - MOUNTING THE AMPLIFIER AGAIN

To mount the amplifier again, proceed as follows:

1 Position the open spanner clamp on the flange.

2 Attach the data cable and the vacuum tube to the amplifier bottom.

3 Place the amplifier bottom precisely upon the flange and secure it with the spanner clamp,

4 Carefully re-insert the preamplifier boards upon the corresponding contact pin of the collector and the port of the groundplane (as it was arranged before).

CAUTION: ***Remove the plug of the 55 V power supply before you re-insert the preamplifier board on the groundplane. Otherwise, a damage may be caused when the preamplifier board is not exactly positioned on the corresponding contact pin.***

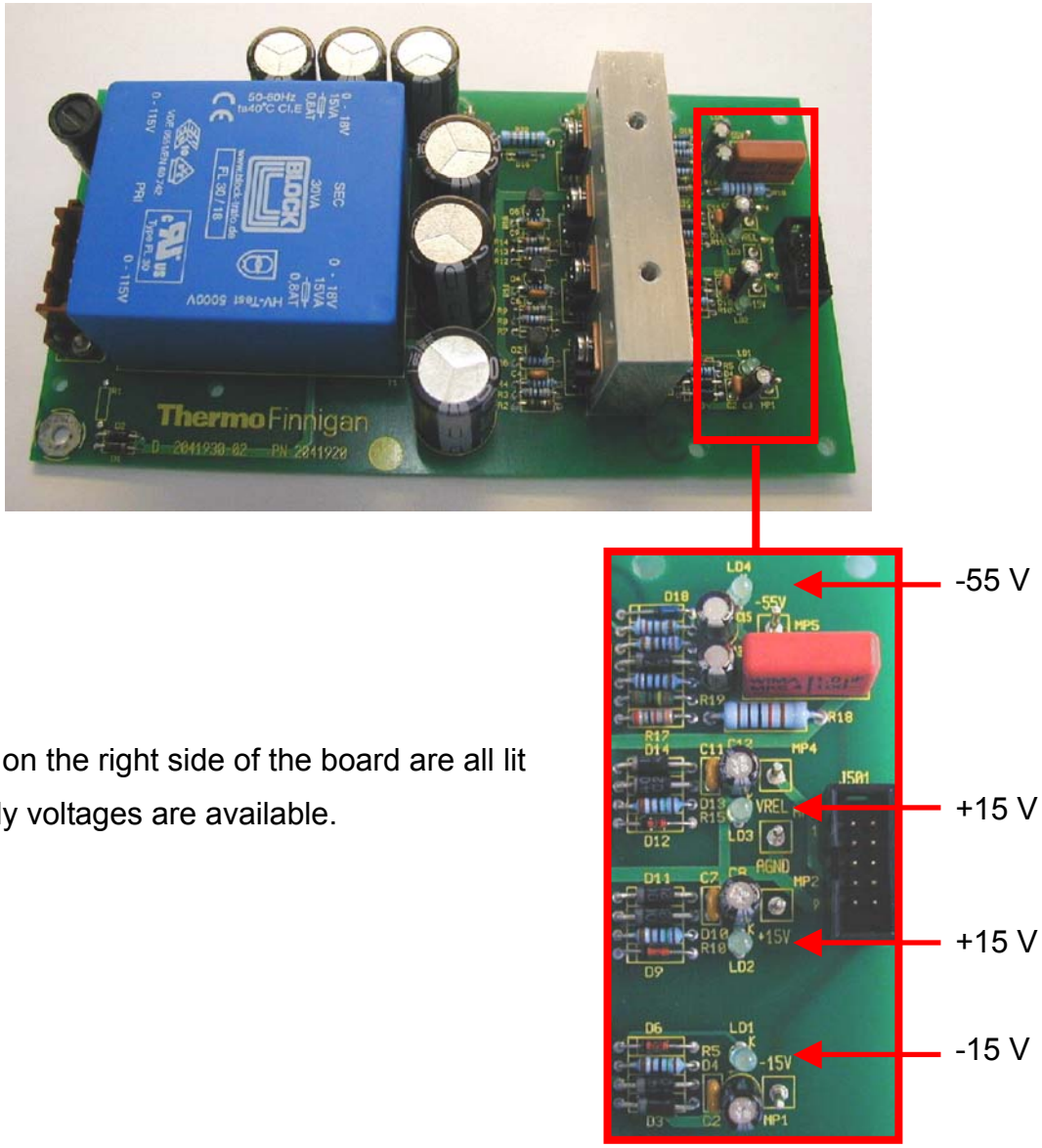
5 Secure the preamplifier boards by screws.

6 Put on the amplifier lid again.

4.5.5 POWER SUPPLY 55 V

The 55 V power supply (Part No. 204 1920) is located at the top middle of the electronics cabinet. It provides the voltages (-55 V, +15 V, -15 V) that are required by the amplifiers.

Fig. 4-9 Power supply 55 V



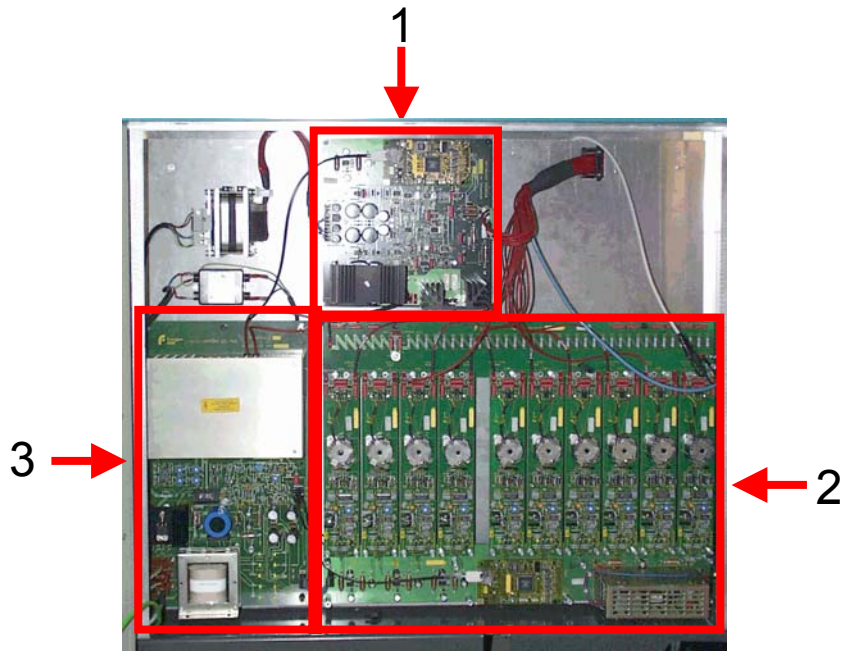
The four LEDs on the right side of the board are all lit when the supply voltages are available.

4.6 ION SOURCE CONTROLLER

The ion source controller (Part No. 204 5880) is located on the left side of the IRMS. The ion source controller comprises three main parts (see fig. 4-10) that have the following functions:

- Emission regulation (see fig. 4-10a)
- Creation of all required potentials as X-focus, deflection, etc. (see fig. 4-10b)
- High voltage supply (see fig. 4-10c)

Fig. 4-10 Parts of the ion source controller



For a description of selected parts of the ion source controller, see the following explanations after the corresponding numbers.

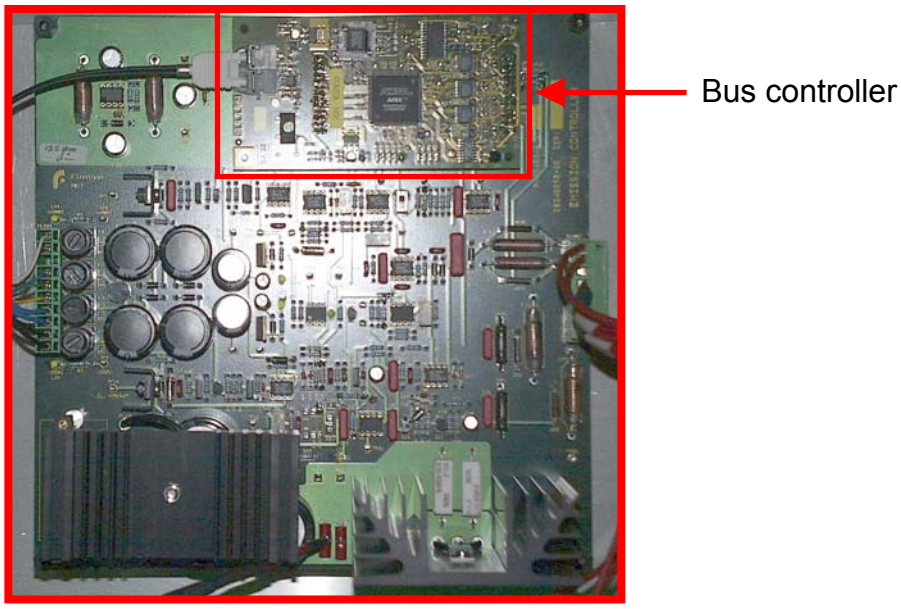
1	Emission regulator
2	High voltage potential controller
3	High voltage supply

On the next pages, you will find a more detailed description of the parts of the ion source controller of the ***MAT 253***.

4.6.1 EMISSION REGULATOR

The emission regulator (Part No. 204 2040) keeps the current constant that heats the cathode of the ion source. It is located on the top of the ion source controller. A bus controller (Part No. 204 1450) provides the connection via data logger to the DEL-PCI controller in the computer.

Fig. 4-10a Emission controller



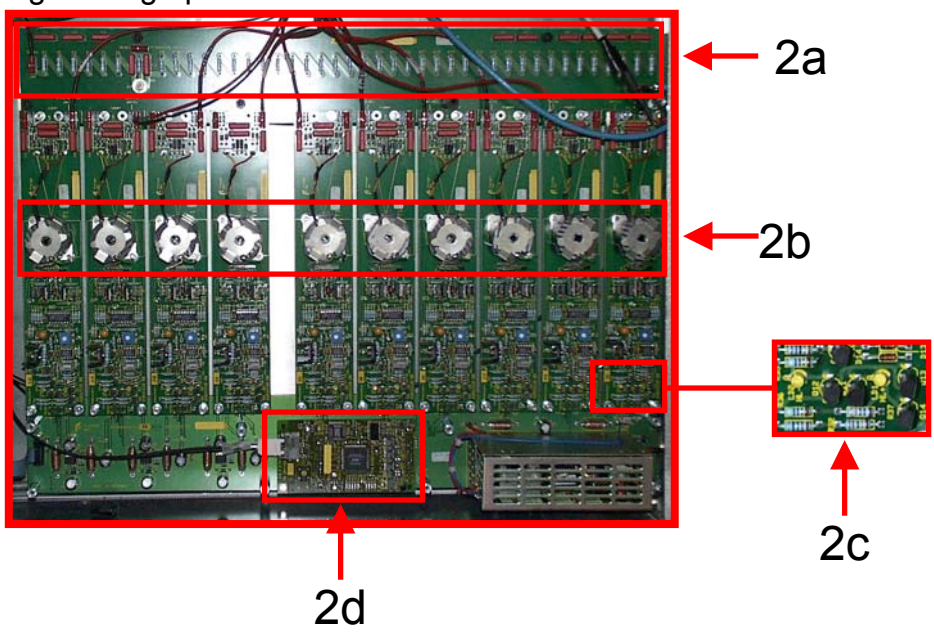
4.6.2 HIGH VOLTAGE POTENTIAL CONTROLLER

Various components (e.g. extraction plates, lens systems) focus the ion beam by adjustable electric potentials. The high voltage potential controller (Part No. 204 1520) provides the power supplies for the ten high voltage potential modules. It controls these modules and the high voltage supply with the help of the bus controller board. It communicates with the connected boards via analogous and digital controlling and feedback signals.

The high voltage potential module (Part No. 204 1500) enables the ground-potential based computer-controlled activation of a focusing potential for ion sources. It is used for the potential separation (in the transmitter) up to 10 kV and supplies a maximum of either 250 V or 500 V alternatively. The polarity of the output voltage is determined by the corresponding connection of the voltage divider.

On each module, a voltage divider (Part No. 204 1480) generates the corresponding potential from the high voltage. The voltage divider is connected to the 10 kV accelerating voltage and creates the base potentials for the focusing of the ion source. When the yellow control LEDs are lit the potential of the component is off range. Then use the software to set the potential to a middle position. If the LEDS are still lit the corresponding component has to be replaced.

Fig. 4-10b High voltage potential controller



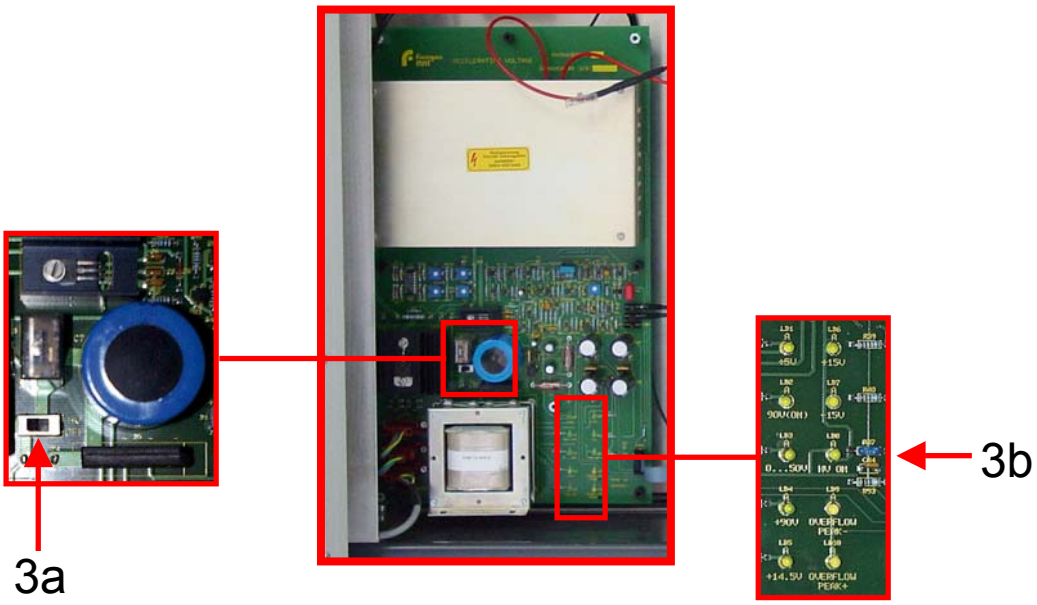
For a description of selected parts of the high voltage potential controller, see the following explanations after the corresponding numbers.

2a	Voltage divider
2b	Potentials controls
2c	Control LEDs
2d	Bus controller

4.6.3 HIGH VOLTAGE SUPPLY

The high voltage supply (Part No. 204 2020) creates an adjustable high voltage up to 10 kV for the ion source, the voltage divider and the emission controller. It is located on the left side of the ion source controller. Green LEDs are lit when the high voltage is switched on.

Fig. 4-10c Parts of the high voltage supply



For a description of selected parts of the high voltage supply, see the following explanations after the corresponding numbers.

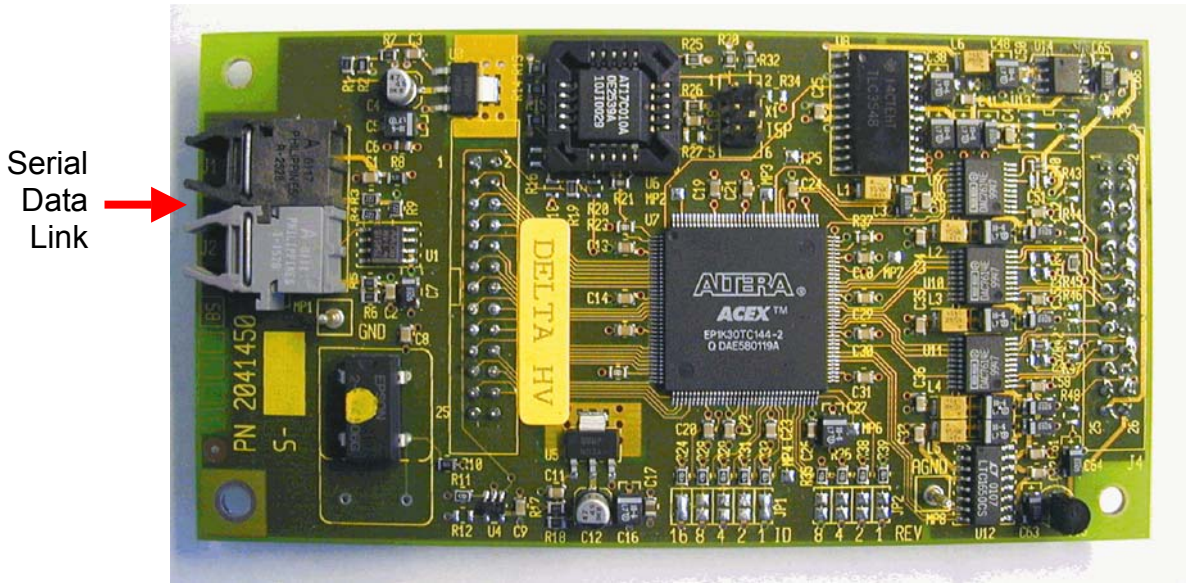
3a	On/off switch
3b	Control LEDs

4.6.4 BUS CONTROLLER

The bus controller is a universal control unit. Two boards of this type are located on the ion source controller, the first one (Part No. 204 1450) on the emission regulator and the second one (Part No. 205 5020) on the high voltage potential controller.

The serial data link provides two ports that are connected to the data logger. One port is dedicated to emission, the other one for the potentials. By using optical fibers instead of metallic conductors, the signals for the two ports are galvanically isolated from the rest of the electronics. This design obviates mutual interferences of the conductors especially in case of line surges.

Fig. 4-11 Bus controller



4.7 MAGNET CURRENT REGULATOR

The magnet current regulator (Part No. 205 2860) is located in the bottom left side of the instrument's electronic cabinet. It provides the current required to generate the electromagnetic field. The current is computer-controlled via the DAC printed circuit board (digital-to-analog converter).

The computer feeds the information of the specified mass number via the optical bus and the data logger to the microprocessor, which controls the DAC on the power distribution. Here, this information is converted into an output voltage, which controls the power supply of the magnet current regulator.

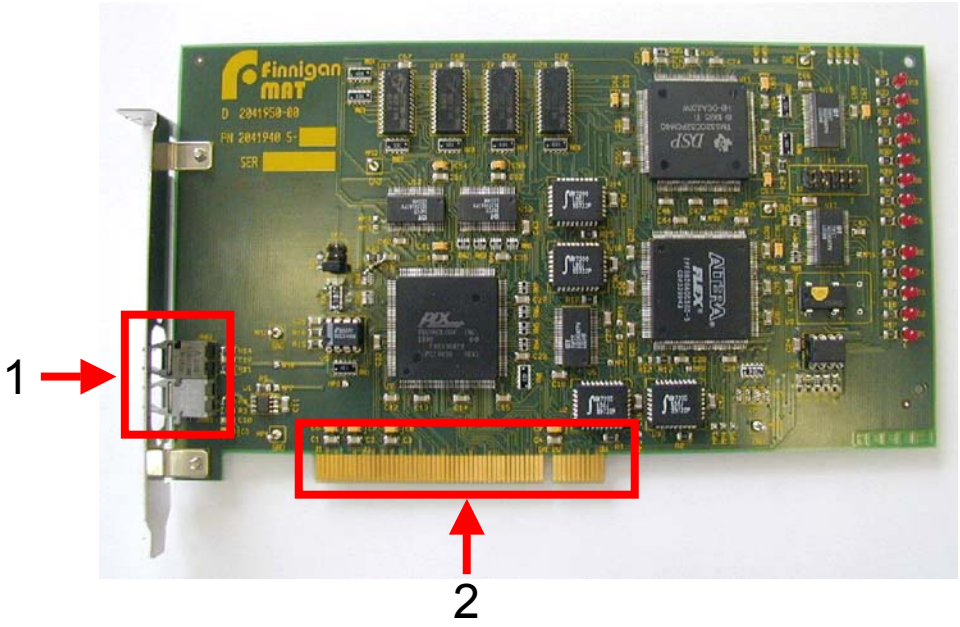
Fig. 4-12 Magnet current regulator

WARNING; ***Parts of the board are at 230 V voltage. Never touch this board without safety measures.***

4.8 DEL-PCI CONTROLLER

The DEL-PCI controller (Part No. 205 2580) is plugged in a PCI slot of the computer that is delivered with the IRMS. It is connected to the bus controllers in the IRMS via optical fibers. This board is the instrument controller and contains the front-end processor.

Fig. 4-13 DEL-PCI controller



For a description of selected parts of the DEL-PCI controller, see the following explanations after the corresponding numbers.

1	Serial data link to IRMS via optical fibers
2	Connection to PCI slot of computer

MAT 253

OPERATING MANUAL

DUAL INLET SYSTEM

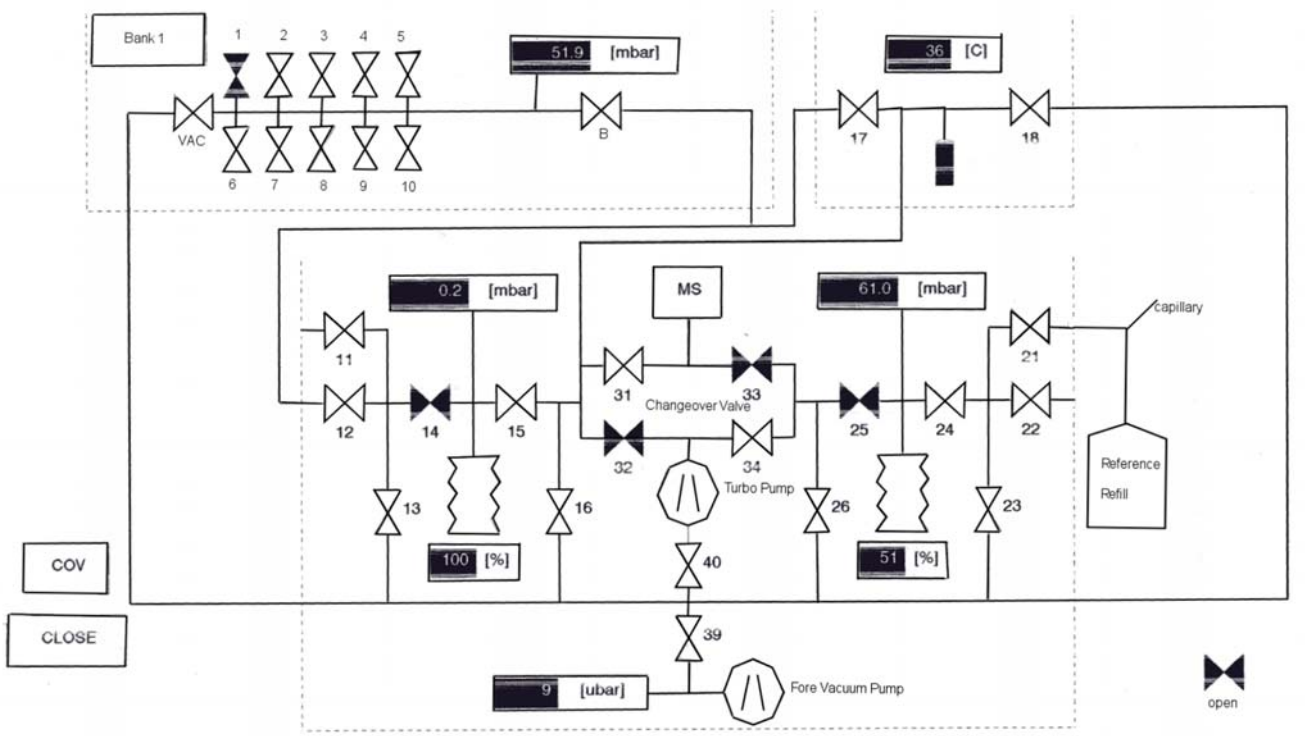
Issue 04/2002

Thermo Finnigan

5.1 GENERAL DESCRIPTION OF THE DUAL INLET SYSTEM

The Dual Inlet system of the ***MAT 253*** isotope ratio mass spectrometer has a symmetric design to allow alternating measurements of a sample and a standard gas.
If the instrument is equipped with a Dual Inlet system, the configuration is identical for the sample side and the standard side, which enables a balanced flow. Each inlet side has two ports and a variable volume (bellow) with the respective inlet capillary leading to the changeover valve. For very small samples, a Microvolume with its own capillary is installed.

Fig. 5.1 Dual Inlet System



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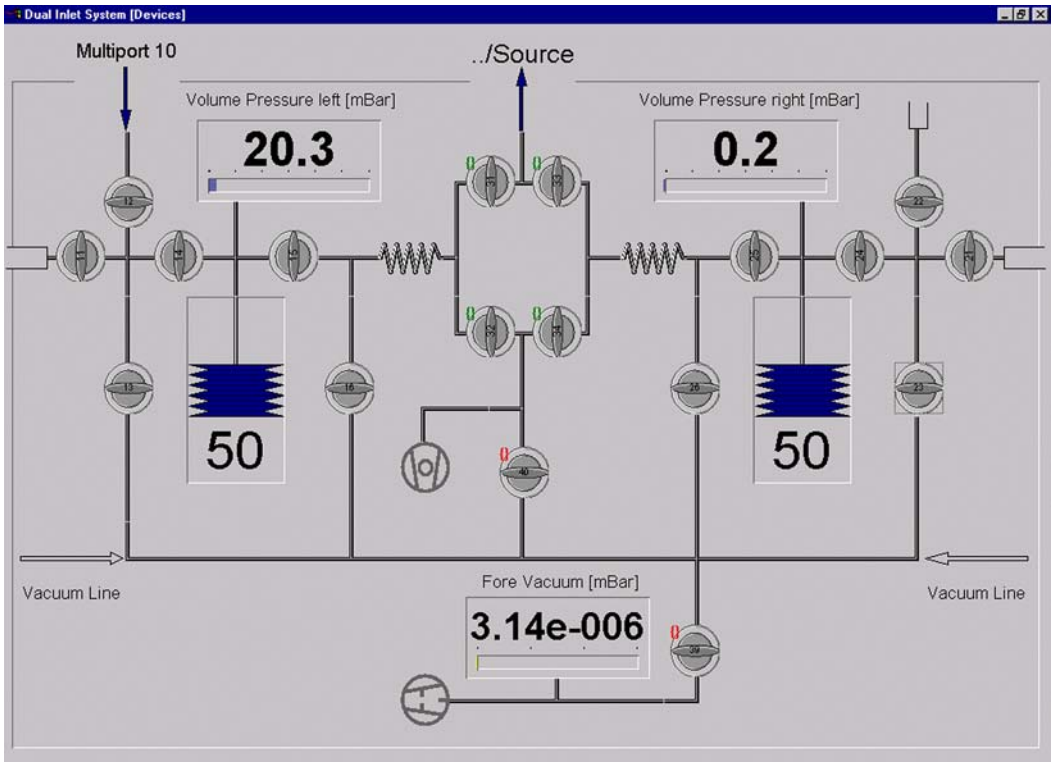
Before measurements can be performed and results be compared, equal gas conditions - as pressure and flow - must be provided for both sample and standard gas to obtain a balanced ion beam intensity. Pressure adjustment for sample and standard gas is performed in reservoirs (bellows), which are adjustable in volume. These variable volumes are bellows, which are operated by software-controlled motors. An automated procedure balances the volumes to such an extent that the ion beam intensity of a selected mass attains a preset value. As it is not possible via computer in some cases, balancing of the volumes can also be performed manually. The bellows are adjustable from about 3.5 ml to 40 ml each.

Fig. 5.2 Schematic of the Dual Inlet System



Precise isotope ratio determination via Dual Inlet requires a stable gas flow into the ion source. To obtain this, bellow balancing of both sides is essential (the ISODAT NT software description for bellow balancing is called ***Pressure Adjust***). If sample gas flows into the ion source, an equal amount of a standard gas is evacuated simultaneously by the wasteline pump system - or vice versa. The flow conditions thus remain identical during measurement.

Flow conditions are also matched by adjusting the flow resistance through the capillaries to the ion source. The flow resistance is set to equal conditions by crimping the capillaries in front of the inlet port of the changeover valve.

The crimps of the capillaries are factory-set, but must be set new when a capillary is replaced. How to crimp a capillary to a specific flow resistance is described in Chapters 7.4 (“Heating Inlet Capillaries”) and 7.5 (“Replacing Inlet Capillaries”).

The variable volumes adjust the pressure for larger samples (> 50 barµl). Very small samples, as low as 5 barµl, can be analyzed using the optional Microvolume inlet. For more details, refer to Chapter 5.5 (“Microvolume”).

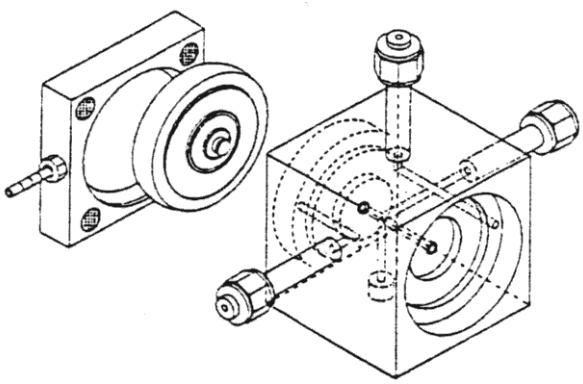
To avoid any condensation, to remove impurities or to measure SO_2, the Inlet system including the changeover valve and the ion source housing can be heated to a temperature of up to 80°C.

5.2 VALVE SYSTEM

The inlet system is operated by pneumatic valves with a nominal closing pressure of 4 bar. The compressed air is supplied either by an optional compressor attached to the Mass Spectrometer or by a user supplied pressure-air line.

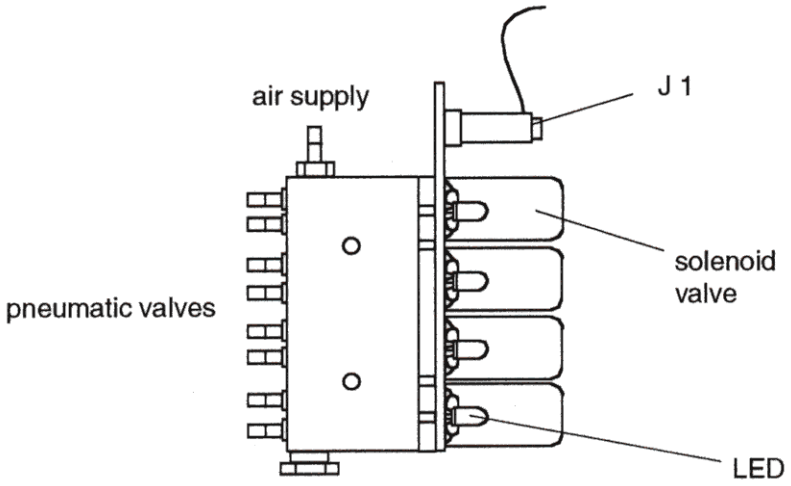
The metal valves are equipped with gold gaskets and gold seats acting on knife-edges. Up to six valves are machined into one monoblock thus considerably reducing the volume in plumbing as well as possible leakage of the installation. This type of valve blocks is used throughout all inlet modules. For plumbing the valve blocks are fitted with 1/4" Swagelok connectors. Compressed air is fed to the pneumatically operated valves by solenoid valves. These are controlled by dedicated electronics linked to the computer via a data bus.

Fig. 5.3 Schematic view of a double valve block



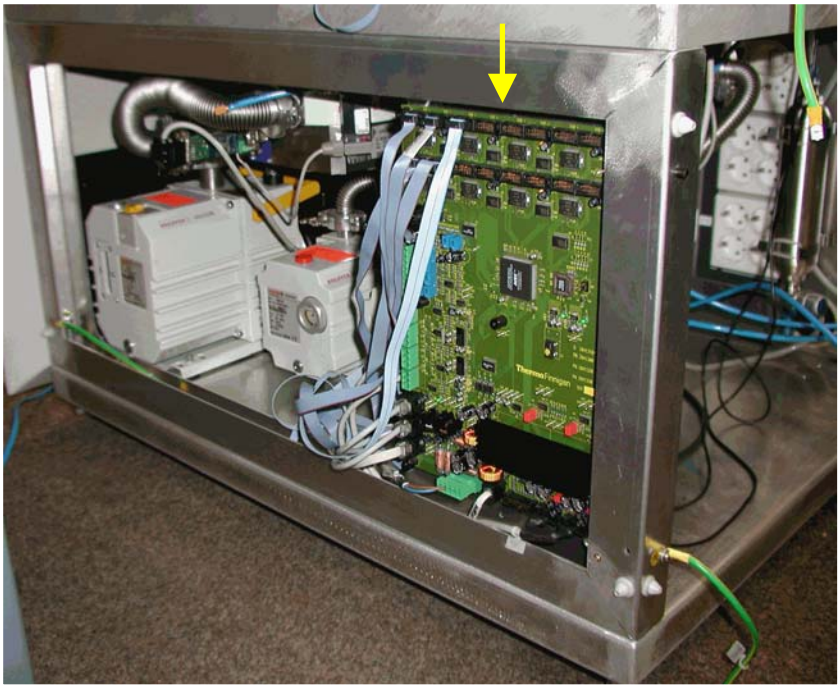
Four of the solenoid three-way valves are located on a "Manifold" block. The solenoid valves are operated with a voltage of 24 V. The voltage is supplied by one of the inlet boards. The solenoid valves are normally open and the working condition is signaled by a red LED located on the printed board.

Fig. 5.4 A "Manifold" block with 4 solenoid valves.



The cable connections are as follows (for the appropriate valve connections, refer to the Service Manual). In case of a power failure, all solenoid valves open automatically. Thus, the pneumatic valves close avoiding contamination of the inlet system.

Fig. 5.5 Driver board with distributor function



NOTE: ***Both Inlet System and Options are now arranged on one single board, i.e. the Inlet board for the Internal Options. (no longer on several boards).***

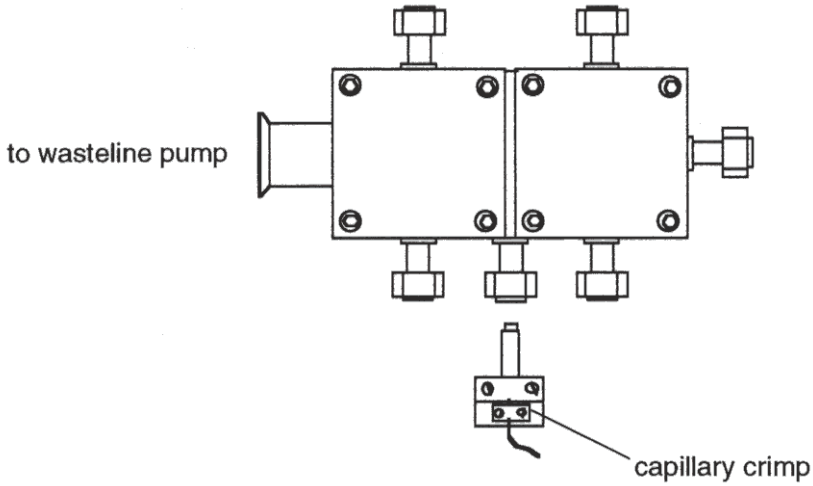
NOTE: ***Only two further cables connect the remaining electronics to the board: control line and power supply.***

5.3 CHANGEOVER VALVE

5.3.1 BASIC MODULE

The basic module of a changeover valve consists of a single block attached to the analyzer housing and accepts the coupling of capillaries for sample and standard gas. The changeover valve is operated automatically by ISODAT NT or manually via the monitor display of the inlet schematic.

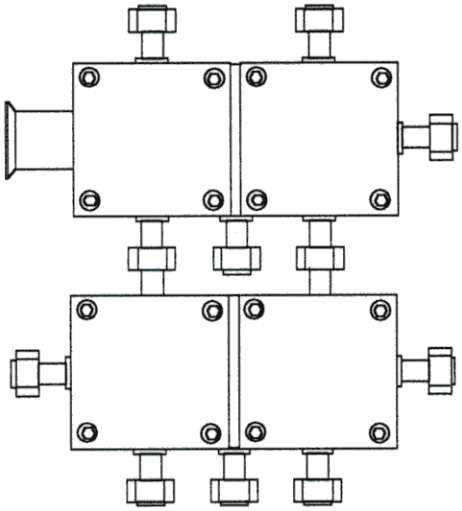
Fig. 5.6 Basic module of changeover valve block



5.3.2 EXTENSION MODULE

An extension module may be added to the changeover valve as an option. The extension module is flanged to the changeover valve by ¼" Swagelok connectors. It provides two additional inlet ports allowing the coupling of further inlet system options, e.g. a carbonate system. To monitor and to operate the extended changeover valve the respective ISODAT NT routine must be activated.

Fig. 5.7 Extension module attached to the basic module of the changeover valve



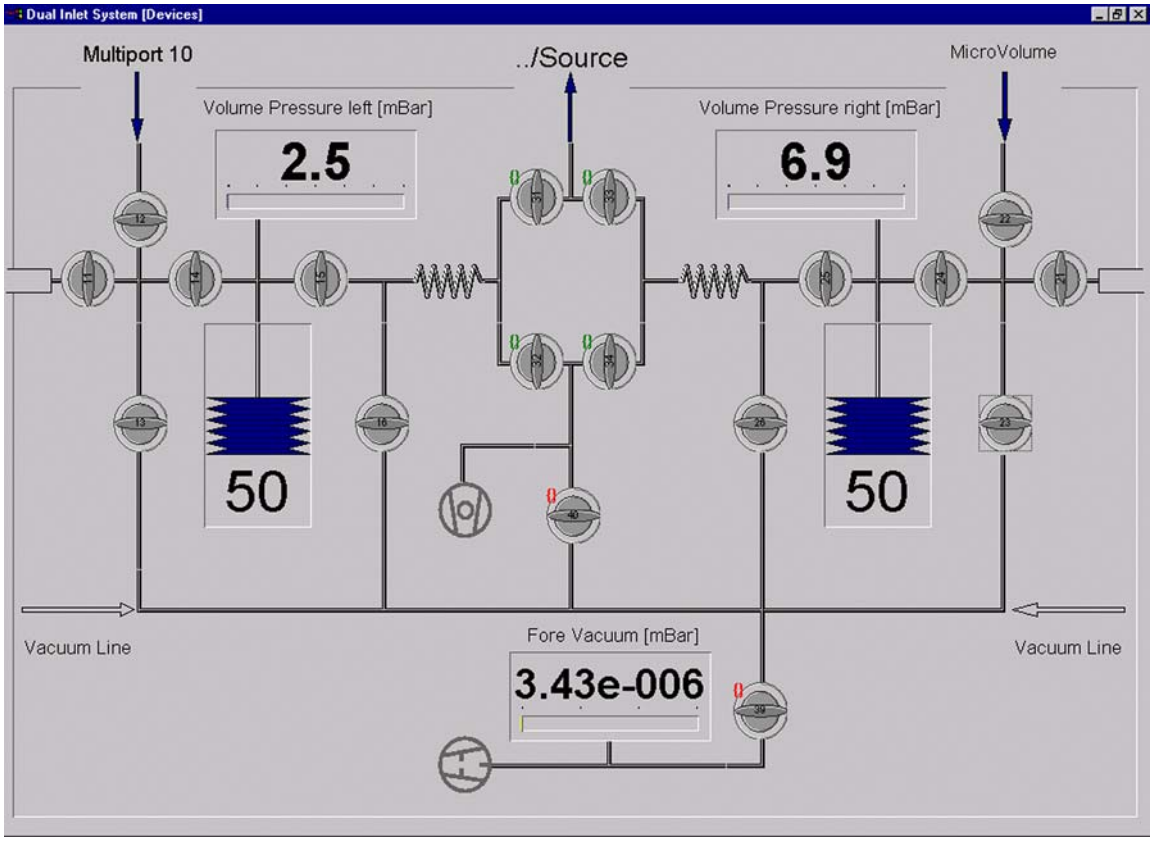
5.4 MULTIPORT

The Multiport is a sample manifold inlet system consisting of one or two banks of 10 ports each. It may be optionally equipped with tube crackers (see Chapter 8). When using the Multiport as an inlet system, the Multiport is connected directly to the sample side of the Inlet System valve # 12 (left inlet port). The Multiport valve system is shown in the following figure.

The valves of the Multiport are operated the same way as the components of the Dual Inlet system, i.e. automatically by the computer or manually via the monitor display.

To monitor and to operate the Multiport valve system on screen the respective ISODAT NT routine has to be activated.

Fig. 5.8 Schematic of a Dual Inlet System with Multiport and Microvolume



Measurement using a Multiport as an Inlet System is described in chapter 6.4.

5.5 MICROVOLUME

The Microvolume or "cold finger" is an optional Inlet module for very small samples and may be installed in combination with a Dual Inlet system or a Multiport. In both cases, the Microvolume is connected to the left port (valve # 12) of the Dual Inlet system (see Fig. 5.2 ”Schematic of the Dual Inlet System” and Fig. 5.8 “Schematic of a Dual Inlet System with Multiport and Microvolume” above).

Fig. 5.8a Schematic of a Multiport

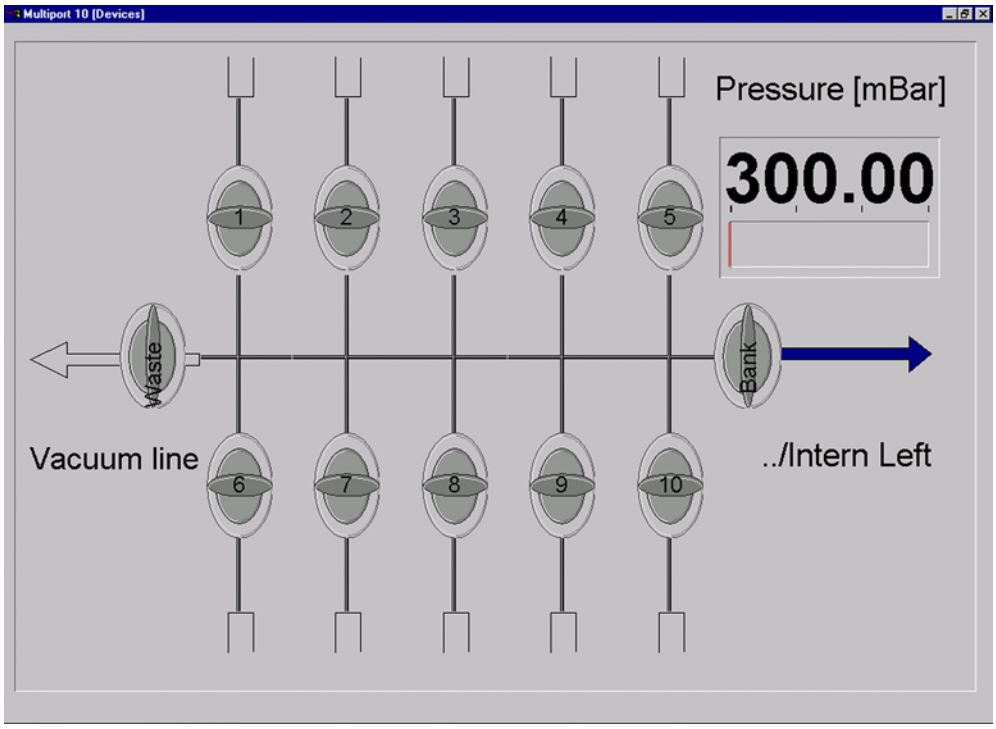
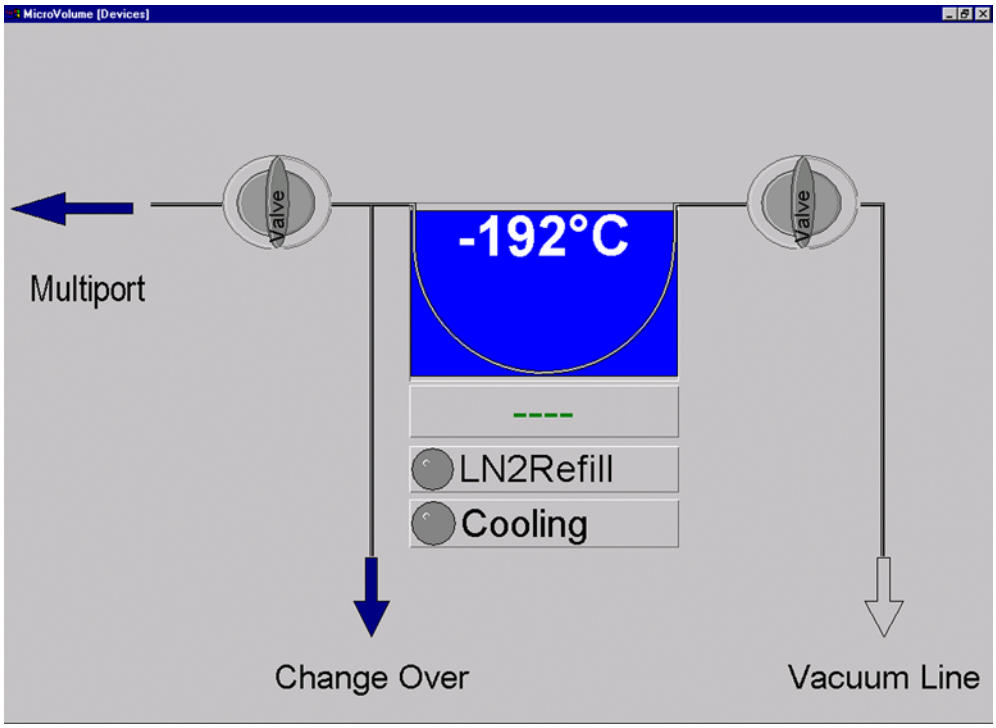
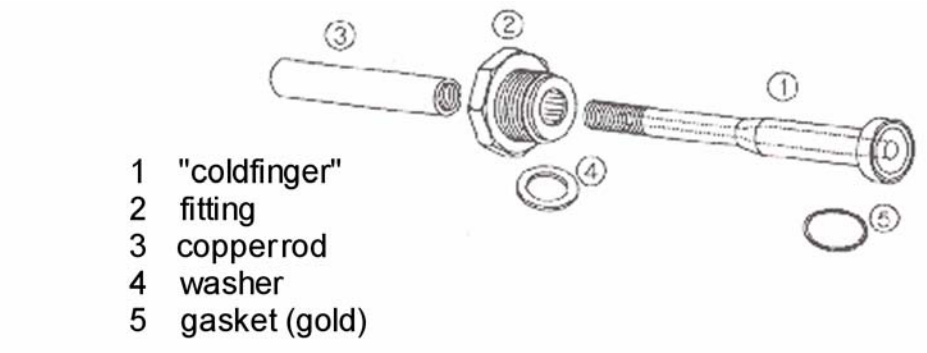


Fig. 5.8b Schematic of a Microvolume



A Microvolume consists of a "cold finger", a valve block, an Autocool unit and a separate capillary. This capillary leads directly to the changeover valve of the system.

Fig. 5.9 Parts of a Microvolume to be inserted into an Autocool unit



The total volume in front of the capillary crimp, i.e. "cold finger" volume plus the connections including the capillary, is about 250 µl. Due to the viscous flow conditions, which require a pressure of at least 15 mbar in front of the capillary, a sample of 5 to 50 barµl must be concentrated into a small volume. The "cold finger" volume can be reduced (for even smaller samples) by inserting small steel spheres. The concentration in a Microvolume is performed by freezing the small sample using liquid nitrogen and expanding it again by subsequent heating.

Two different types of Microvolumes can be used depending on the gas to be measured. For CO_2 a smaller Microvolume is required, and for N_2 a larger one is used. The larger one contains a molecular sieve to freeze out N_2 at liquid nitrogen temperature.

The valves of the Microvolume are operated in the same way as the other components of the Dual System, i.e. automatically via computer or manually via monitor display. Using the Autocool unit the temperature can be set individually within a range of about -180 °C and +155 °C.

5.5.1 AUTOCOOL UNIT

The temperature of the Autocool unit, which cools the "cold finger", can be set via ISODAT NT's Microvolume control (i.e. via the "Instrument" tab's "Microvolume" part; refer to chapter 6.5). A temperature range between -180 °C and +155 °C can be covered.

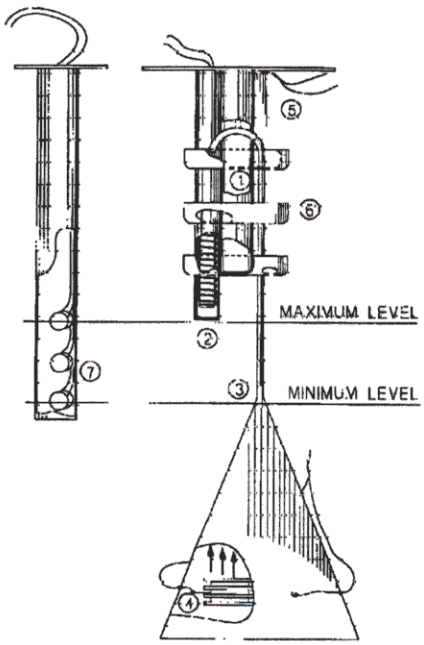
The time to get from: + 50 °C down to - 180 °C is less than 2 min,

- 180 °C up to + 50 °C is about 1 min.

The Microvolume fits into a thermal contact pipe attached to the lid of a Dewar. The Dewar contains liquid nitrogen. An electrical heater element, a temperature sensor and a cascade arrangement of three small bowls are fitted to the contact pipe (see Fig. 5.10). All parts of the assembly are made of a material of high thermal conductivity and are placed in close thermal contact to each other. Thus, a quick changeover from one temperature to another is provided. To heat the Microvolume to a defined temperature, the heater element is activated. The heating phase is controlled by the temperature sensor. The three thermistors (7) are Pt 100 resistors (i.e. 100 Ω).

Fig. 5.10 Schematic view of an Autocool unit

1 thermal contact pipe
2 heater element
3 hood and standpipe
4 immersed heater element
5 temperature sensor
6 cascaded arrangement of bowls
7 level sensor with 3 thermistors



To cool the Microvolume, another electrical heater element immersed in liquid nitrogen is activated and causes both evaporation and agitation.

Above the heater element, a funnel-shaped hood of a standpipe is positioned, which leads to the uppermost bowl of the cascaded arrangement. This arrangement enables about one droplet of liquid nitrogen per second to be carried by the stream of evaporated nitrogen.

Small holes in the bottom of the bowls yield a constant trickle of liquid nitrogen back into the Dewar, and the continuous flow of liquid nitrogen rapidly cools down the Microvolume. By suitable balancing of the liquid nitrogen flow and heating the Microvolume, any temperature within the range can be attained.

Due to the very small quantity of liquid nitrogen held in the cascaded bowl arrangement the Microvolume temperature rises very quickly when the immersion heater is switched off and the pipe heater is switched on. A constant liquid nitrogen level in the Dewar vessel is maintained by means of the liquid nitrogen refill device.

5.5.2 AUTOCOOL REFILL DEVICE

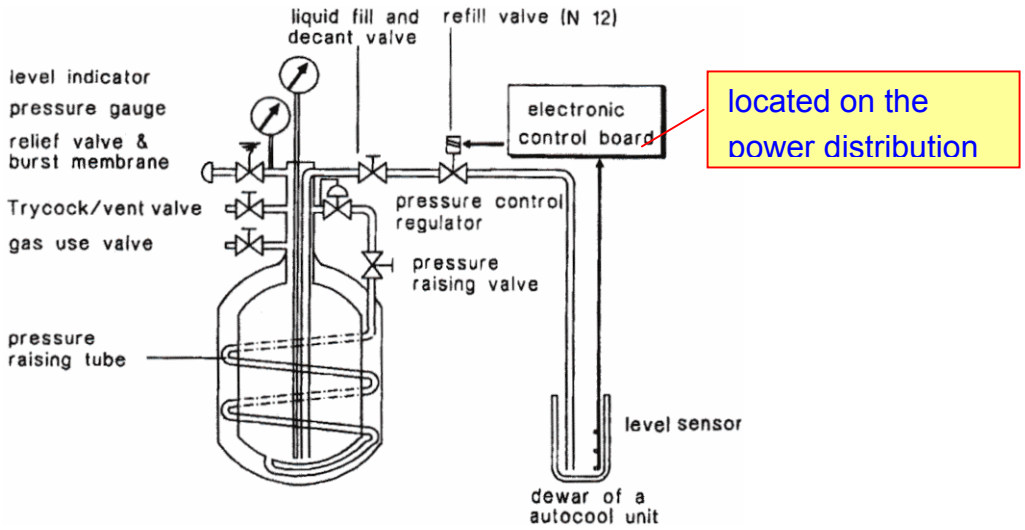
5.5.2.1 GENERAL REMARKS

The refill device provides a constant level of liquid nitrogen in the Dewar of the Autocool unit. It consists of a storage Dewar of 75 l or 25 l capacity and is equipped with the safety devices, valves and pressure gauges required for a safe handling of liquid nitrogen.

The transfer line to the Dewar of the Autocool unit is controlled by a solenoid-operated refill valve (N 12). The refill valve (N 12) is directly connected to the liquid fill and decant valve of the refill device.

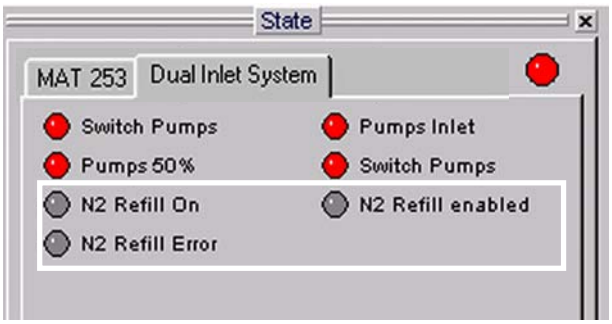
The refill device is activated by a level sensor installed in the Dewar of the Autocool unit. The level sensor consists of three sensing thermistors, one each for the maximum, the minimum and the alert level.

Fig. 5.11 Schematic view of a refill device



The power distribution board of the refill device evaluates the signals of the sensors and activates the refill valve (N 12) to start or to end the nitrogen transfer.

On the power distribution board, three yellow LEDs indicate which thermistor is sensing, and the status of the liquid nitrogen level of the Autocool unit can be controlled. In addition, these three LEDs are visualized in ISODAT NT.'s ***State*** window as follows:

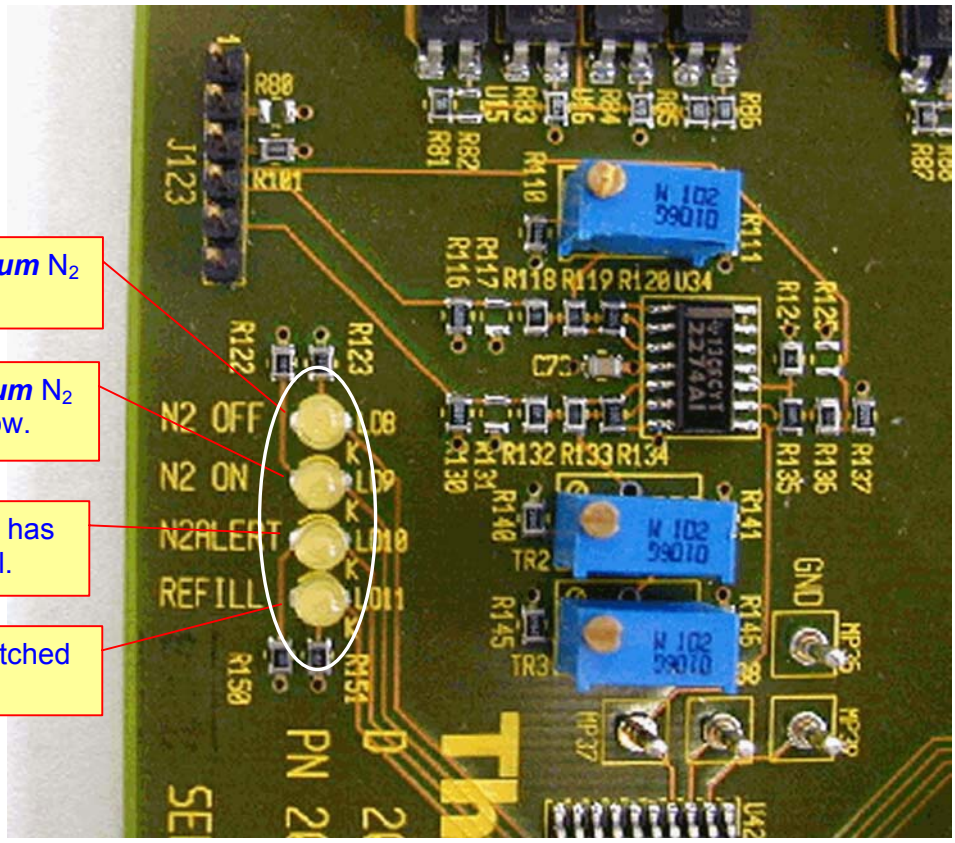


- yellow LED signals (upper position):
  high liquid nitrogen level, indicates ***end*** of refill.
- yellow LED signals (middle position):
  low liquid nitrogen level, indicates ***start*** of refill.
- yellow LED signals:
  alarm level, ***no*** refill has been performed.

If the signal of the thermistor positioned in the middle does not result in a refill and the lowest level is reached (i.e. the storage Dewar of the refill device is also empty) the level sensor activates:

- the yellow LED on the control board panel,
- a warning message of the system on the monitor,
- an interruption of the measurement

5.5.2.2 CONTROL OF THE REFILL DEVICE



5.5.2.3 SAFETY WARNINGS

NOTE: ***Keep in mind: Safety First!***

- Before operating the refill device, carefully read these notes as well as the manufacturer's handling instructions.
- The refill device contains extremely cold liquid gas. Careless handling might cause severe personal injury including frostbite.
- Wear protective clothing when operating this equipment, including protective gloves and face shield. Do not overfill or tilt the refill device. Prevent spills.
- Use the refill device only in well-ventilated areas. Poor ventilation might cause suffocation. Follow correct First Aid procedures. If gas was inhaled, remove victim to fresh air. If necessary give artificial respiration and seek medical assistance immediately.
- Make sure that only authorized and fully trained operators use this equipment and they are fully conversant with these safety notes.
- Only use the cryogenic liquid specified on the label on the refill device.

5.5.2.4 WORKING PRINCIPLE

The transfer of liquid nitrogen is affected by a pressure build-up in the self-pressurizing Dewar of the refill device. The pressure builds up by vaporization of liquid nitrogen in a coiled pressure raising tube located in the Dewar's vacuum interspace when the gas vent valve is closed and the pressure-raising valve is opened.

A pressure gauge monitors the pressure. A pressure of 5 psi should be sufficient to transfer liquid nitrogen. A pressure of 10 psi will transfer liquid at about 10 l/min. A higher pressure is not necessary and even squandering.

As soon as a preset pressure is reached, the pressure regulator installed in the circulation cuts the flow through the coiled pressure raising tube. The working pressure can be set to an optimal level using the pressure regulator.

The blow-off valve is set to a limit of about 1.5 bar. An additional burst membrane prevents from building up a dangerous pressure. The gas vent valve allows bleeding excessive pressure, if necessary. The function schematic of the liquid nitrogen refill device is shown in Fig. 5.11.

The 75 l storage Dewar is equipped with a level indicator monitoring the liquid nitrogen content.

5.5.2.5 MAINTENANCE

Hardly any routine maintenance is required. However, the components of the refill device should be regularly checked for damage or possible freeze-up. If it is necessary to dry and clean items or to replace them, make sure that they are thoroughly degreased and dried, as moisture or lubricants will freeze at cryogenic temperatures. Do not use thread-sealing compounds. Use PTFE tape, e.g. Teflon®, or other approved oxygen-safe compounds instead. Occasionally, the pressure should be increased up to the relief valve setting to ensure satisfactory functioning of this safety device. Setting the pressure control regulator is performed as follows:

Step 1 Loosen hexagon locking nut below adjusting screw.

Step 2 Rotate adjusting screw counter-clockwise to set to zero.

Step 3 Close vent valve and liquid valve and open pressure building valve.

Step 4 Rotate adjusting screw clockwise to increase vessel pressure until gas escapes through the vent hole. Rotate adjusting screw counter-clockwise again until gas stops escaping through the vent hole.

Step 5 The pressure is now set. Tighten locking nut to prevent further rotation or tampering of the adjusting screw.

5.5.2.6 CHECKING THE LIQUID NITROGEN EVAPORATION RATE

If you suspect that the evaporation rate of the refill device is excessive, note down the decrease of the liquid nitrogen level for some time. To check the loss rate, close the pressure raising valve and open the Trycock/vent valve. After the contents are fully vented down to atmospheric pressure, measure the boil off rate using a simple flow meter. The normal boil-off rate for nitrogen is:

1 liter per minute for gas, i.e. about 2 liters of liquid per day.

The reason for a higher rate might be an abnormal cold or frost formation at the lower dished end of the outer casing, which should be removed.

5.5.2.7 OPERATING INSTRUCTIONS

As a quick reference, the arrangement of valves for filling, dispensing and storage of liquid nitrogen is shown below (refer to Fig. 5.11):

Operation	*Liquid fill and decant valve*	Gas use valve	*Pressure-raising valve*	*Trycock/vent valve*
Filling Liquid	open	closed	closed	open
Dispensing Liquid	open	closed	open	closed
Gas Withdrawal	closed	open	open	closed
Storage: -Short Term -	closed	closed	open	closed
Storage:- Long Term -	closed	closed	closed	open

Before using the Microvolume, check the content of the refill device. If filling is required, proceed as follows:

1. Open the Trycock/vent valve.
2. Close the pressure-raising valve.
3. Close the gas use valve.
4. Fill via the opened liquid fill and decant valve.

After having checked the refill device, first fill the Dewar of the Autocool unit with liquid nitrogen roughly to the required level. To enable the automated and computer controlled refill operation, follow the steps below:

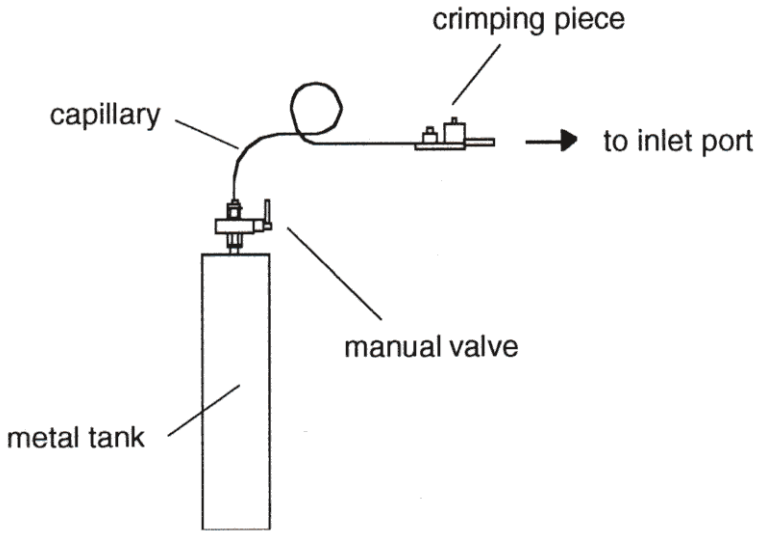
1. Check the pipe connection leading to the Dewar of the Autocool unit.
2. Close the gas vent valve.
3. Open the liquid fill and decant valve.
4. Open the pressure-raising valve.

The refill device is now connected to the Dewar of the Microvolume via the opened liquid fill and decant valve. The flow is controlled via the refill valve N 12. The pressure-building valve may be closed when the working pressure is reached, or it may remain open - provided the pressure regulator is set to a suitable working pressure.

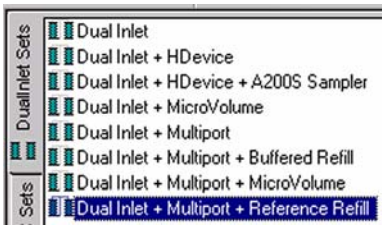
5.5.3 STANDARD REFILL (REFERENCE GAS REFILL)

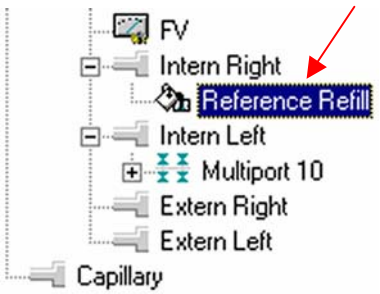
When working with a Multiport Inlet System, a reference gas refill may be necessary in order to avoid running out of reference gas during measurements. Reference Refill provides the reference gas supply to the Inlet System. Reference Refill is a hardware option. It consists of a metal tank (its capacity is approximately 5 l) with a manual valve connected via a capillary to one of the Inlet ports on the standard side (see Fig. 5.12). With the Reference Refill selected, the standard side of the inlet system is completely pumped out before it is filled again for the next measurement Sequence.

Fig. 5.12 Reference Gas Refill



- To create a ***Configuration*** containing Reference Refill, at ***Dual Inlet Sets*** select the predefined ***Dual Inlet + Multiport + Reference Refill*** device and drag it to the Source. Thus, the Reference Refill will automatically be connected to the ***Internal Right*** port (at Internal Right port, two possibilities exist for connection. Either of them can be used). For detailed information, refer to page 6.7.



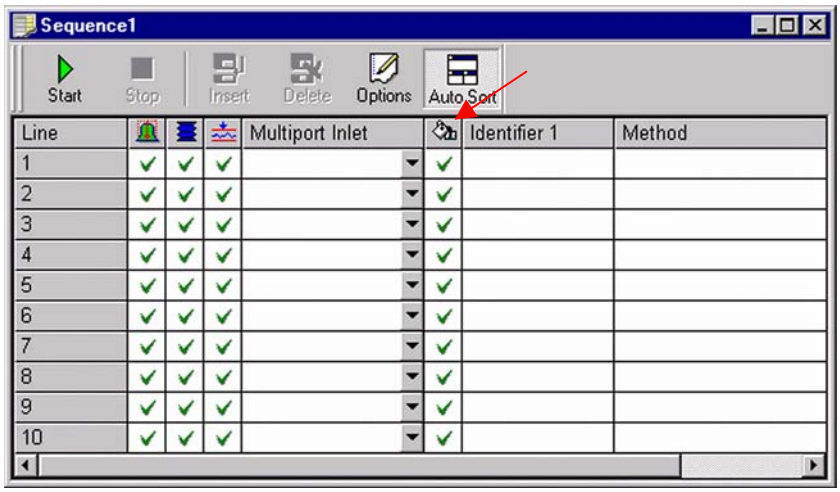


- Creating a new ***Method*** or using a predefined one, note the ***Instrument*** tab, where some peculiar information concerning Reference Refill is summarized:

For detailed information, see Chapter 6.7.

- Creating a new ***Sequence*** or using a predefined one, note the Sequence list.

If Reference Refill is to be used, a checkbox must be activated per sample by a ✓ in the column.



MAT 253

OPERATING MANUAL

MEASUREMENTS

Issue 04/2002

Thermo Finnigan

6.1 GENERAL REMARKS

6.1.1 PROCEDURE BEFORE STARTING ANY MEASUREMENTS

Before starting any measurements, three steps must be performed:

1 Define a (Hardware-) ***Configuration***

2 Define a ***Method***

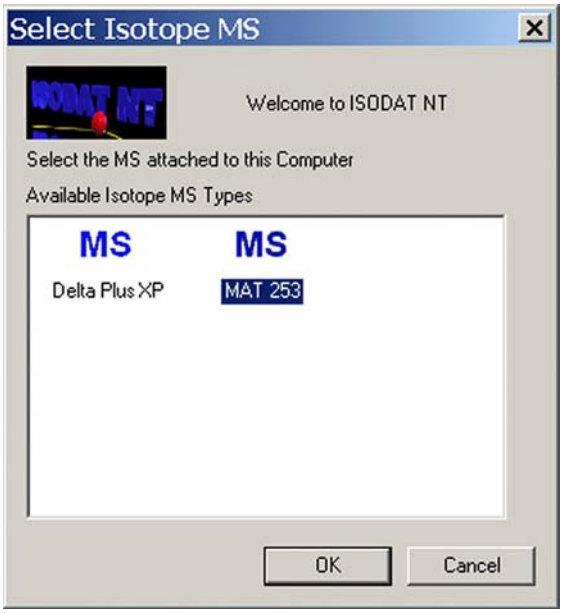
3 Define a ***Sequence***

These three basic steps will be described chronologically for several peripherals in this chapter.

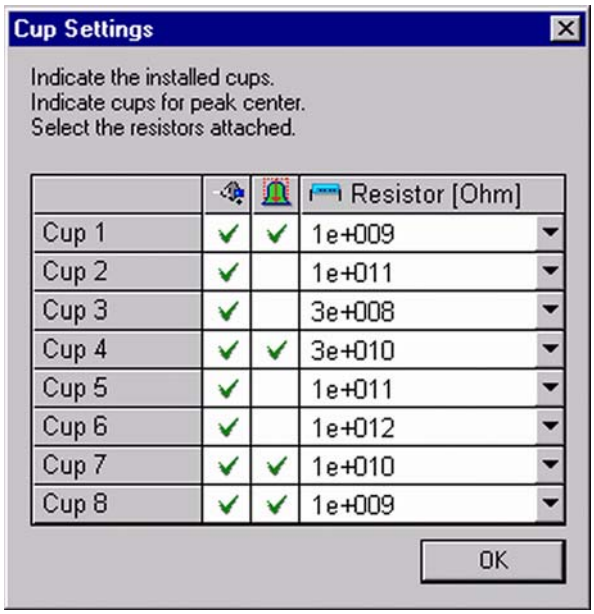
6.1.2 IF NO CONFIGURATION IS AVAILABLE

As first step, a ***Configuration*** containing the Dual Inlet System and your connected peripheral(s) must exist.

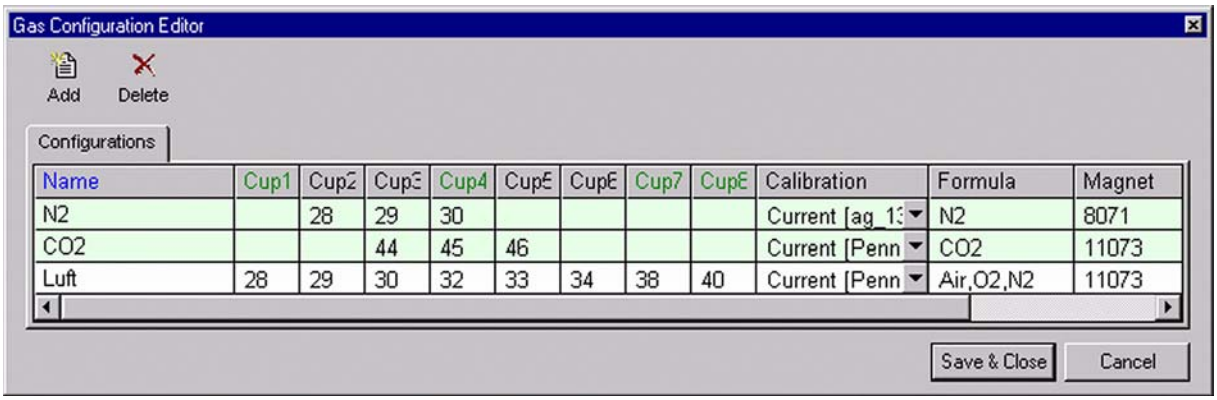
If ***no Configuration at all*** is available (e.g. you have either never before created one or you have just reset your IRMS), ISODAT NT requires some information about your hardware equipment first:



- Your type of IRMS is required.
  Select ***MAT 253***.
- Press ***OK***.



- Check, whether the correct cups are installed.
- Check, whether the correct cups are available for Peak Center.
- Check, whether the resistor values are correct.
- Confirm with ***OK***.



- Check the available Gas Configurations. You can edit them by clicking on the fields (refer to the ISODAT NT Help System).
- Finally, press ***Save & Close***.

You can now proceed creating a ***new Configuration*** using the ***Configurator***.

6.1.3 HOW TO CREATE A NEW GAS CONFIGURATION

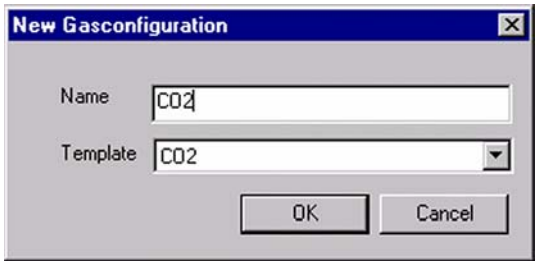
Prior to defining a new Gas Configuration ensure the connected IRMS has the cups set for the simultaneous detection of masses 44, 45 and 46 and mass calibration for these cups has already been performed.

For a measurement, a new Gas Configuration has to be created for the masses 44 ($^{12}C^{16}O^{16}O$), 45 ($^{13}C^{16}O^{16}O$) and 46 ($^{12}C^{16}O\,^{18}O$).

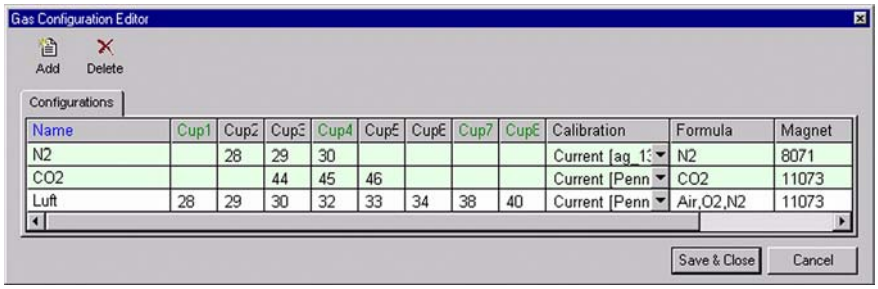
- To create a new Gas Configuration, open the ***Dual Inlet*** module.

- Then open the ***Gas Configuration Editor***.

- ***Add*** a Gas Configuration.



- Type ***CO_2*** for the Name.
- Select Gas Configuration ***CO2*** as ***Template***.
- Confirm with ***OK***.



- Select a ***Calibration*** valid for the selected cups.
- Press ***Save & Close***.

The Dual Inlet application software allows fully automated isotope ratio determination of different elements of bulk samples (C, N, O, S). All parameters relevant for data acquisition of a sample are stored in a ***Method***. Two different ways exist to get access to a Method or a Sequence:

- Select a ***predefined*** Method and Sequence using the File Browser or
- Create a ***new*** Method and Sequence on your own.

Selecting a ***predefined*** Method and Sequence is the usual way described in detail for the different measurement types later in this chapter. The following information is needed, if you want to create a ***new*** Method or Sequence:

6.1.4 HOW TO CREATE A NEW METHOD

- Open the ***Dual Inlet*** module.

- Select the ***Configuration*** for your application (e.g. “Dual Inlet”).

- Select the ***Gas Configuration*** (e.g. ***CO2***).

- Press the ***New*** button.

- Press the ***Method*** Icon.

The new Method is structured in Tab pages: Instrument, Peripherals, Evaluation, Printout and Dyn. Externals. Methods are described in detail later in this chapter.

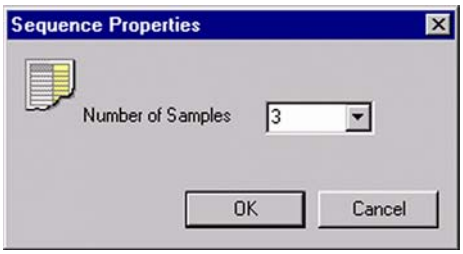
6.1.5 HOW TO CREATE A NEW SEQUENCE

After having defined a new Method, a ***new Sequence*** can be created as follows:

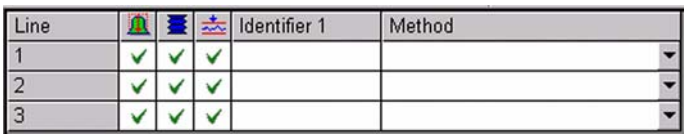
- Press the ***New*** button.

- Press the ***Sequence*** Icon.

- Define the number of samples (e.g.: 3).



- Edit the Sequence list.



Peak Center Enable ✓ to perform a Peak Center prior to measurement.

Press Adjust Enable ✓ to perform a pressure adjust.

Background Enable ✓ to perform a background measurement.

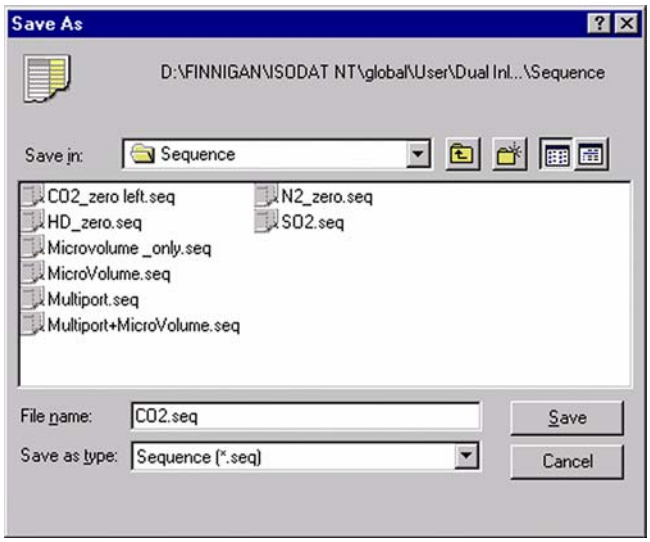
Identifier 1 Edit text to identify sample.

Method Select IRMS Method (for each sample, e.g. “CO2.met”).

- Press the ***Start*** button.

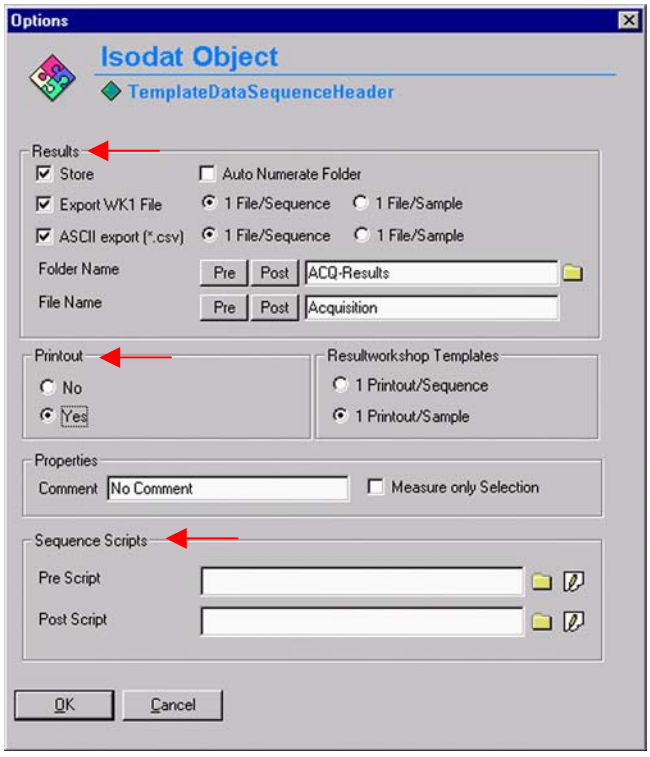
- Type a ***new*** significant File name retaining the extension .seq (e.g. “CO2.seq”).

- Press ***Save***.



- Define parameters for:
  - Results Export
  - Printout
  - Sequence Scripts

- Press ***OK*** to start Sequence Acquisition.

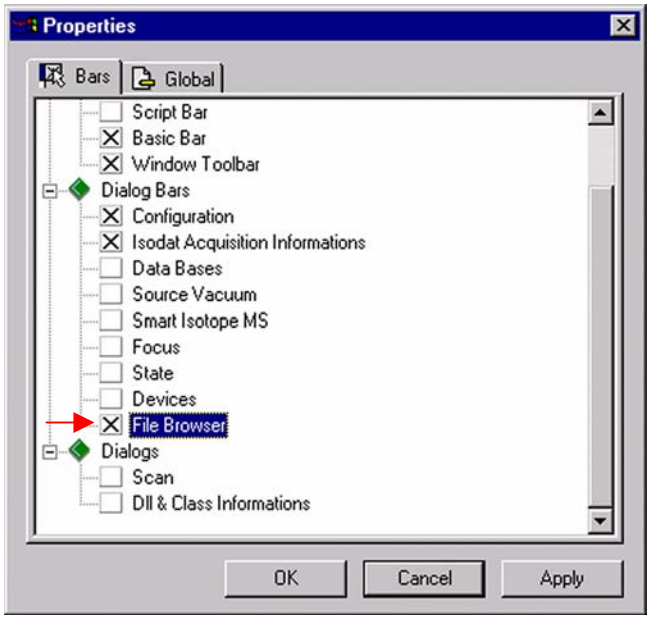


6.2 ZERO ENRICHMENT (STANDARD ON / OFF TEST)

We assume that the user already has working experience with the Dual Inlet System, the Dual Inlet devices and with IRMS. It is recommended to perform a simple check in order to test the analytical condition of the complete system before measuring any samples. Proceed as follows for zero enrichment:

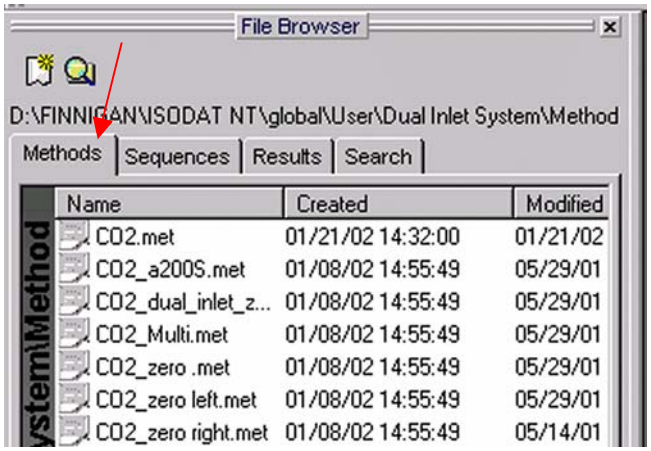
- If the File Browser cannot be seen, press the ***Options*** button.

- Activate the ***File Browser*** check box.

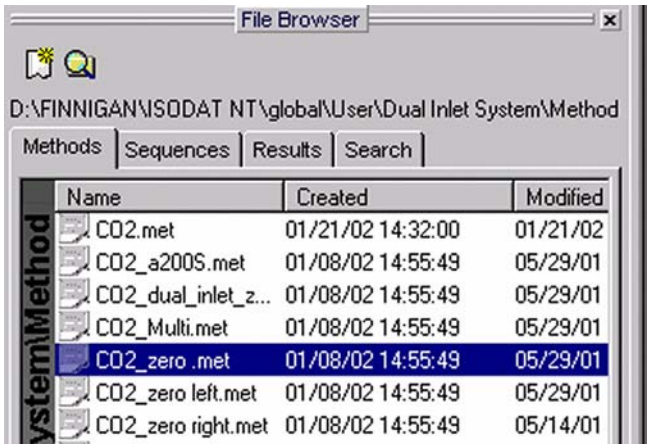


- Click ***OK***.

- On the File Browser, select the ***Methods tab*** (default).



- From the predefined Methods choose ***"CO2_zero.met"*** by double-click.



NOTE: ***The Method "CO2_zero.met" for zero enrichment will be described here. It must be used, when both bellows are measured against each other.***

The Method "CO2_zero left.met" is used, when only the left bellow is measured (against itself).

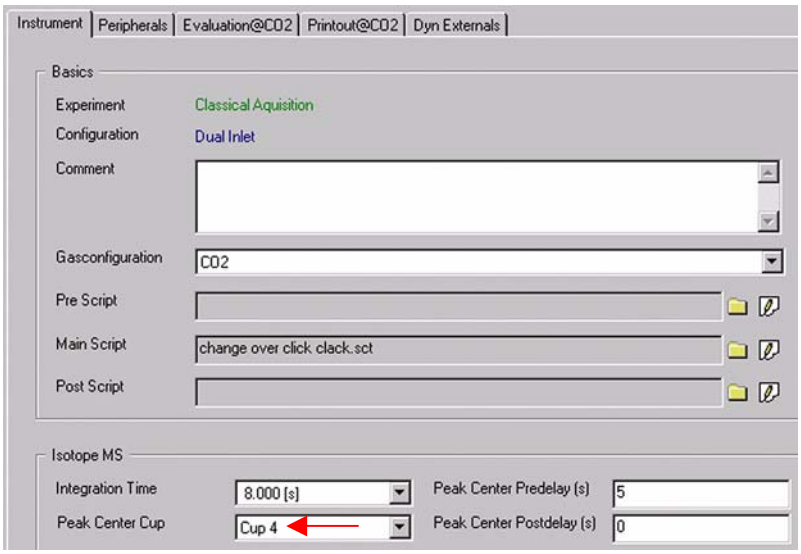
The Method "CO2_zero right.met" is used, when only the right bellow is measured (against itself).

The three Methods only differ from the Script.

- If the Standards in the Method to be opened are older than the actual ones in the Standard Database, or if the user has changed the Method, the warning beside appears.

- Press ***OK***. Thus, the Method will be corrected: the actual values of the Standard Database (e.g. the laboratory Standard has changed or been replaced by another one) are transferred to the Method.

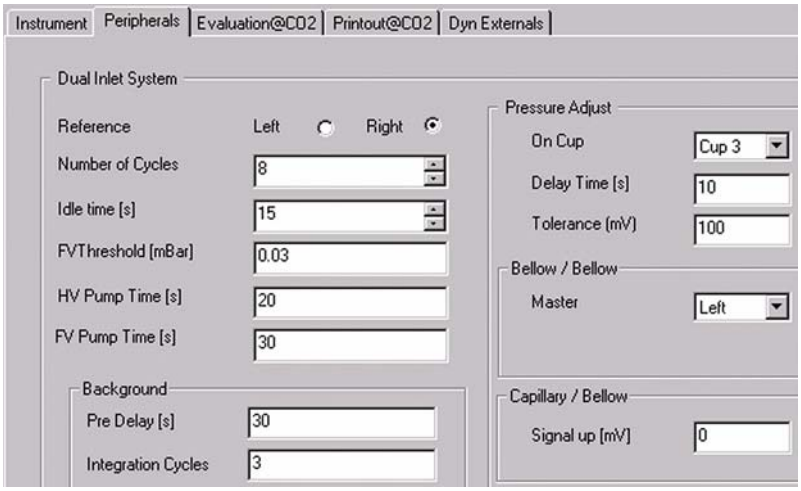
- The ***Instrument tab*** of the Method “CO2_zero.met” appears.



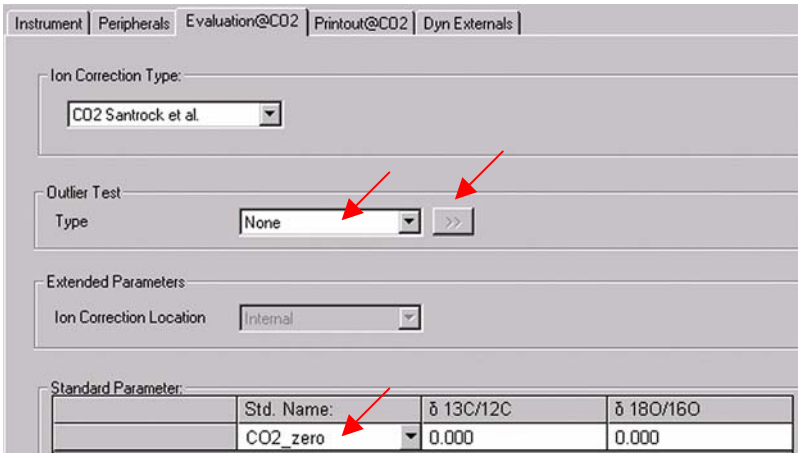
- For details, see chapter 6.3.

NOTE: ***Ensure that the proper peak center cup is selected.***
If necessary, correct the default cup (e. g. Cup 4)!

At ***Peripherals tab***, see chapter 6.3 for details.



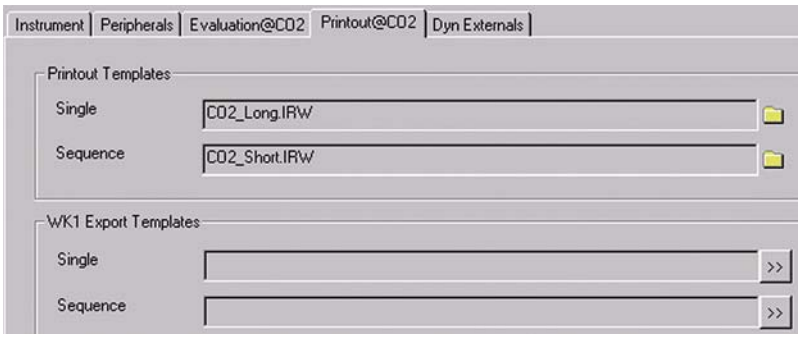
- At ***Evaluation@CO2*** tab, see chapter 6.3 for details.



- Select “None” or “Sigma” as Outlier test.
- In case of “Sigma”, specify the k-fold of the standard deviation using >>.

NOTE: ***At Std. Name, choose a suitable Standard!***

At ***Printout tab***, see chapter 6.3 for details.



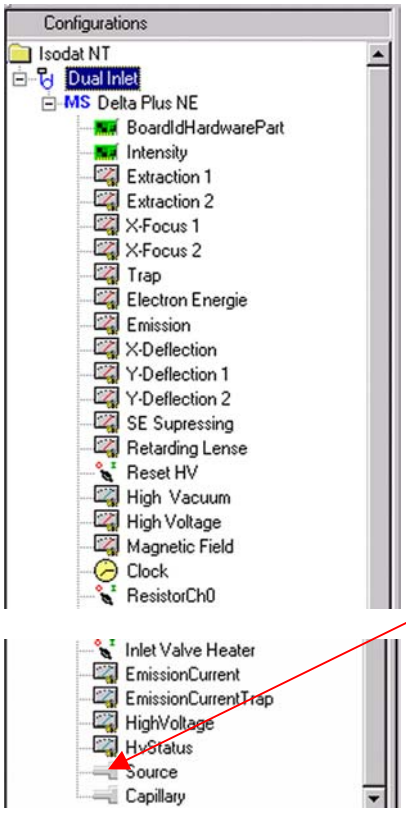
6.3 SIMPLE DUAL INLET MEASUREMENT

6.3.1 DEFINING A CONFIGURATION

➢ Before operating, a ***Configuration*** containing the simple Dual Inlet System must be created in the ***Configurator*** as follows.

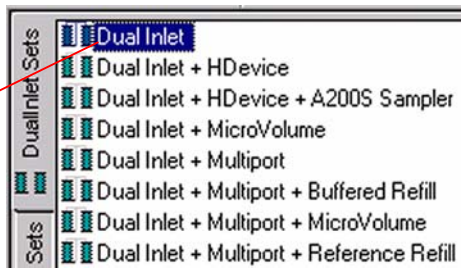
➢ Add a new Configuration using the ***Add Configuration*** button.

➢ Give it a significant name, e.g. ***"Dual Inlet"***.



➢ Open the tree structure by a click on ⊞ at ⊞ MS Delta Plus NE

➢ On the right pane, select ***Dual Inlet***.



➢ Drag it to the Source port ⊐ of the new Configuration.

A ⊞ sign appears:

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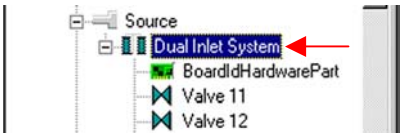
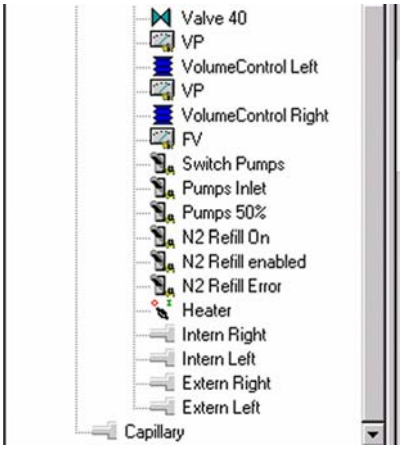


➢ The ***Dual Inlet*** device has been attached to the ***Source***.



➢ Close the Configurator window.

All settings will be saved automatically.

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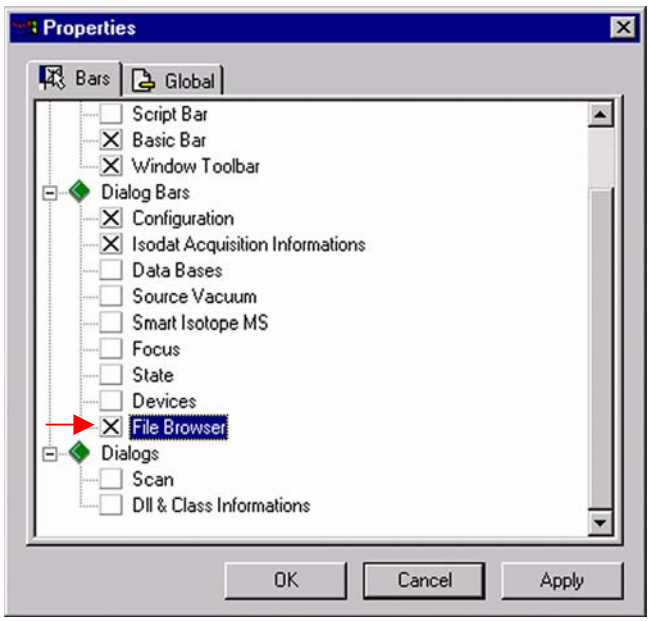
6.3.2 USING A PREDEFINED METHOD

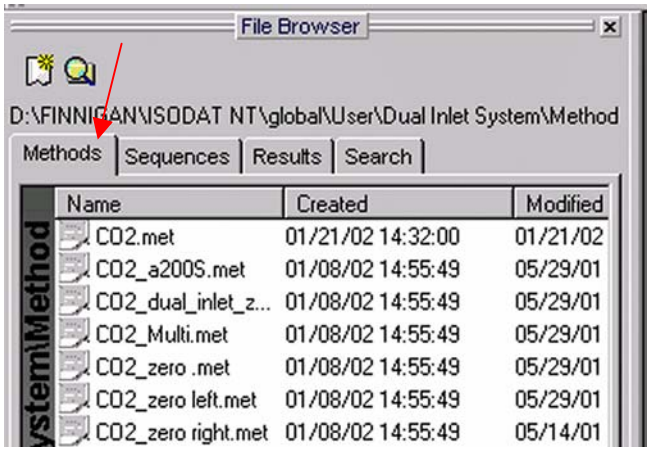
- If the File Browser cannot be seen, press the ***Options*** button.

- Activate the ***File Browser*** check box.

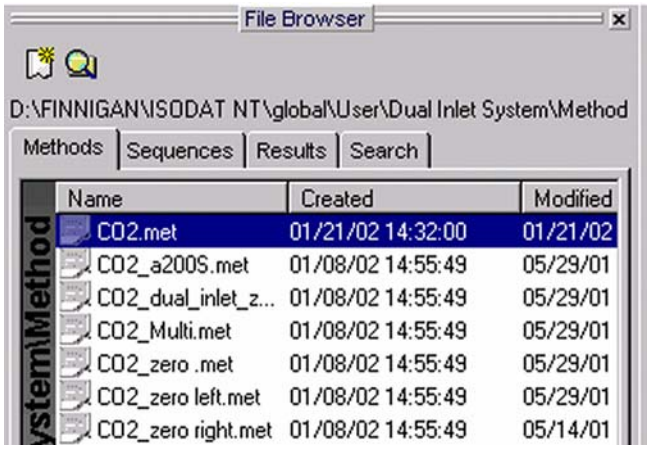


- Click ***OK***.

- On the File Browser, select the ***Methods tab*** (default).



- From the predefined Methods choose a suitable one by double-click (e.g. ***"CO2.met"***).

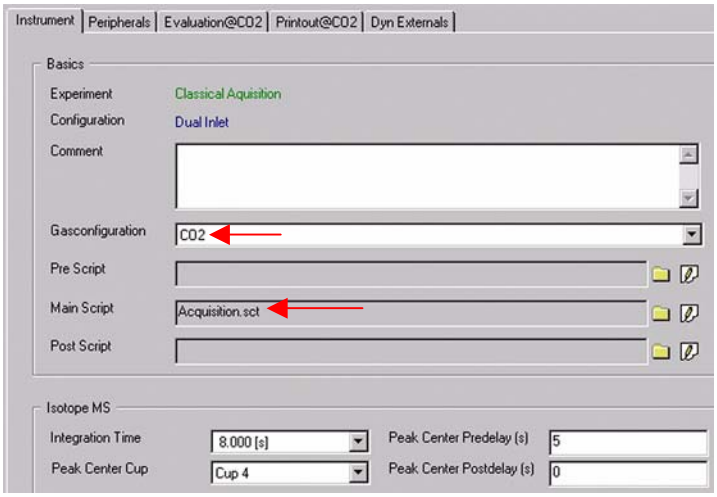


- If the Standards in the Method to be opened are older than the actual ones in the Standard Database, or if the user has changed the Method, the warning beside appears.

- Press ***OK***. Thus, the Method will be corrected: the actual values of the Standard Database (e.g. the laboratory Standard has changed or been replaced by another one) are transferred to the Method.

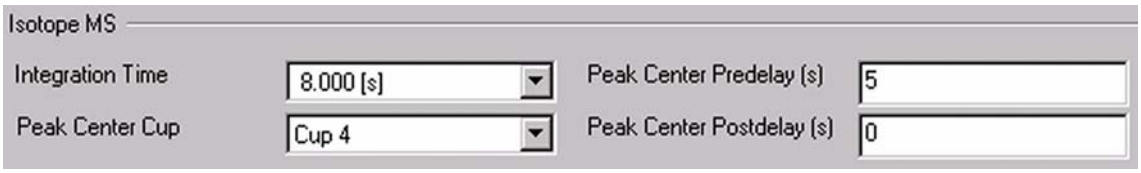
Instrument

- Select the Gas Configuration for your determination type (e.g. "CO2").
- The ***Main Script*** controls the acquisition cycle.



NOTE: ***It should only be edited by users trained on script editing.***

ISOTOPE MS



- Select the ***Peak Center Cup***, e.g. **Cup 4** for a universal triple collector on a ***MAT 253*** (narrow cup for m/z 45).

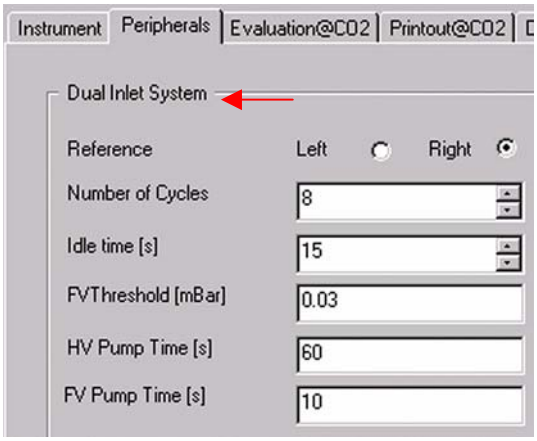
- ***Peak Center Predelay*** is the time the system waits between activation of the reference gas and start of the peak center cycle (e.g. **5** s).

- ***Peak Center Postdelay*** is the time the system waits between the end of the peak center cycle and the start of the data acquisition (e.g. **0** s).

- ***Integration time*** is the time integrated to form a data point triplet (e.g. **8** s). It is needed to measure each individual ion intensity of the masses 44, 45 and 46.

Peripherals

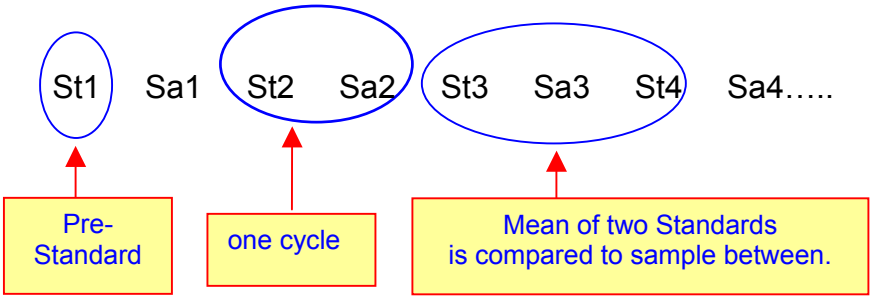
Dual Inlet System



- ***Reference*** denotes, which bellow will contain the standard gas (default: right bellow). The other bellow will contain the sample.

- ***Number of cycles*** indicates, how often a sample will be measured successively (e.g. 8 cycles means, that eight times the standard and eight times the sample is measured).

- A ***cycle*** is a series consisting of a standard measurement followed by a sample measurement. Thus, two standards flank every sample, one before it and one after it:



  The first standard is called pre-standard (i.e. St1). The mean of two consecutive standards is calculated and compared to the flanked sample (e.g. mean of St1 and St2 is compared to Sa1).

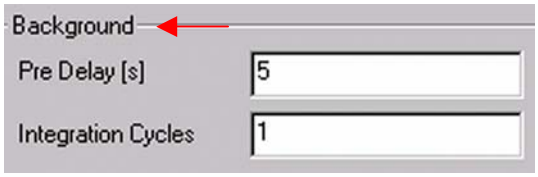
- ***Idle time*** is the waiting time after having switched the changeover from sample to standard side (or vice versa) before the ion intensities of the masses 44, 45 and 46 will be integrated. The gas needs a certain time to flow away, before the measurement can be started.

- ***FV pump time*** is the time interval during which the Fore Vacuum pump will be active after it has started the pumping process (e.g. 10 s).

- ***FV threshold*** is the pressure value that must be fallen below during Fore Vacuum pump activity (e.g. 0.03 mbar). If this value has been reached during FV pump time, the High Vacuum pump starts. If this value has not been reached, the system stops.

- ***HV pump time*** is the time interval during which the High Vacuum pump is active after the FV threshold has been reached (e.g. 60 s).

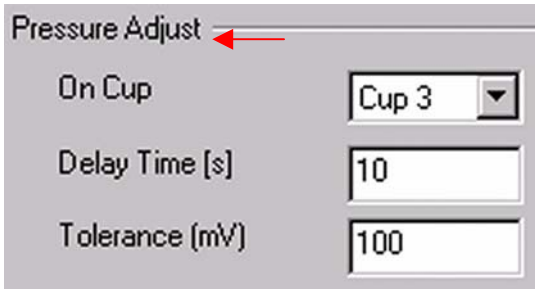
Background



- ***Background Predelay*** is the waiting time after closing the changeover valves until the background measurement begins.
- ***Background Integration Cycles*** denotes, how long the background measurement will be performed (default: 1 cycle). k integration cycles specify that background measurement spans the k-fold integration time.

Pressure Adjust

If standard pressure and sample pressure are varying considerably, a pressure adjust will be necessary. It ensures that standard and sample are measured nearly at the same intensity.



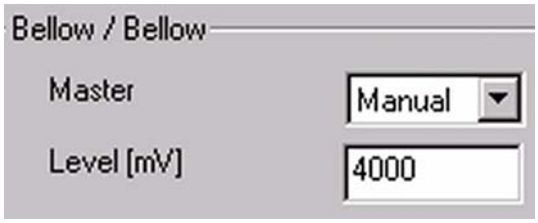
- ***on cup***: cup on which the pressure adjust will be performed. Normally, it is the cup, where mass 44 is measured.
- ***Delay time*** is the waiting time after changing from sample to standard gas (or vice versa) before matching the standard bellow to sample ion intensity.
- ***Tolerance*** denotes the precision of the pressure adjust. It is the maximally acceptable deviation of the ion intensities between the sample signal and the standard signal after matching. If the deviation is less than or equals the value (e.g. 100 mV, this means ± 50 mV), the press adjust is finished successfully.

Bellow/Bellow

➢ ***Master:*** the bellow that predefines the pressure, whereas the other bellow has to follow. Choose between “Left”, “Right” and “Manual”.

In most cases, the sample bellow is defined as master. Thus, the sample enters the system with a certain predefined pressure p to which the standard will be compressed.

NOTE: ***The standard bellow will be adjusted to the level of sample ion intensity minus signal up ± tolerance.***

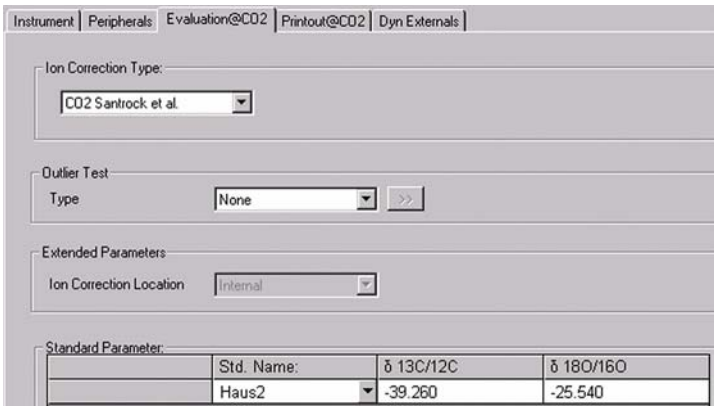


➢ If you select “Manual”, you can edit the pressure level (intensity) that must be reached in both bellows.

Capillary/Bellow

➢ ***Signal up***: match the ion intensity of the standard gas less than the given value before closing valve 25 and starting data acquisition.

Evaluation

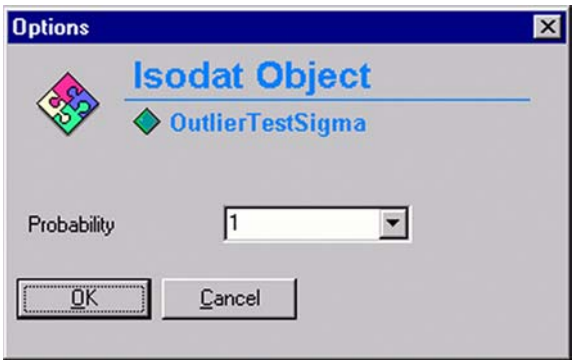


- Select an ***Ion Correction type*** (e.g. ***"CO2 Santrock et al."***).
- Choose the type of ***Outlier Test***. (Rejection criterion for possible outliers: None or Sigma).

Std. Name: Select a Standard Name or edit the related delta values (user defined). The standards are predefined in the standard table. These values are isotope ratios of carbon and oxygen of your standard gas.

Outlier Test: If you select "None", only mean and standard deviation of the obtained delta values will be calculated. Outliers will not be identified.
If you select "Sigma", those delta values beyond k times of the standard deviation will be identified and rejected.

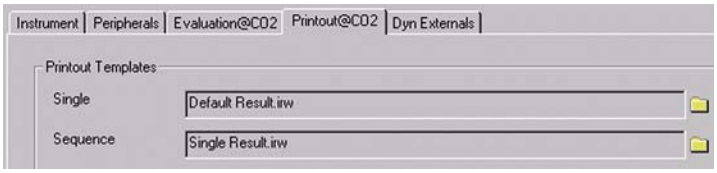
- If you selected "Sigma", the button on the right side becomes available. Click it. 



- Select the k-fold standard deviation (default: k = 1).

Printout

Printout Templates

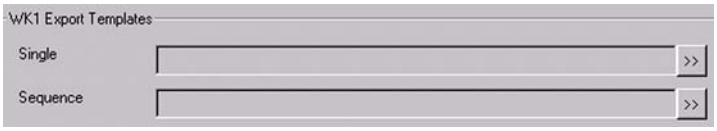


- ***Single Print*** selects a print template from the Result Workshop for an individual printout per sample.

- ***Sequence Print*** selects a print template from the Result Workshop for a reduced print per sample within a sequence summary.

WK1 Export Templates

As with the above Printout Templates, WK1 Export Templates can be selected for an Excel-Export of the data.



- ***Single Print*** selects an Excel-Export Template from the Result Workshop for an individual printout per sample.

- ***Sequence Print*** selects an Excel-Export Template from the Result Workshop for a reduced print per sample within a sequence summary.

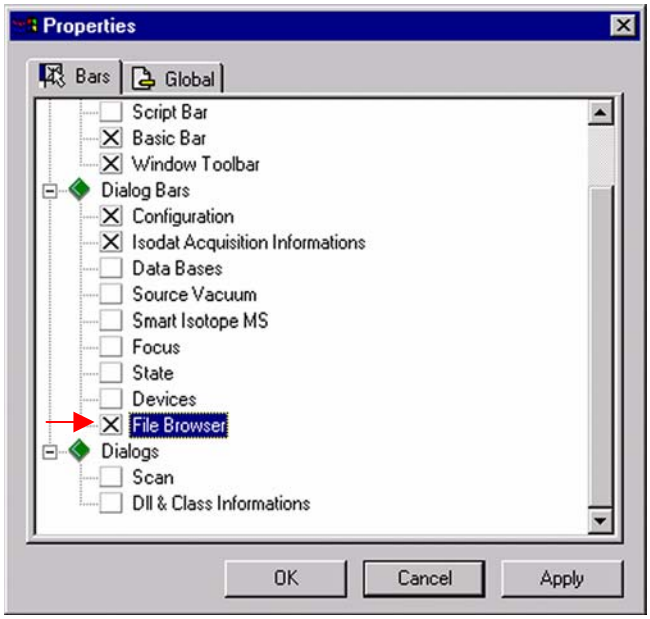
Dyn. Externals: will follow soon.

The ***Method*** for measurements using a simple Dual Inlet has now been established. As next step, either use a ***predefined Sequence*** (refer to chapter 6.3.3) or create a ***new Sequence*** (refer to chapter 6.1.5).

6.3.3 USING A PREDEFINED SEQUENCE

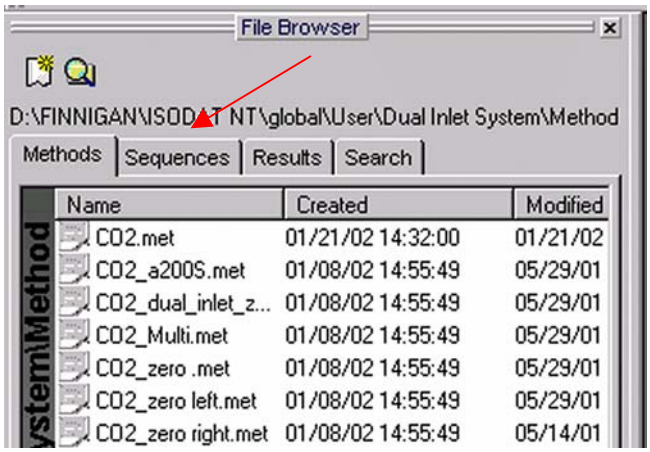
- If the File Browser cannot be seen, press the ***Options*** button.

- Activate the ***File Browser*** check box.

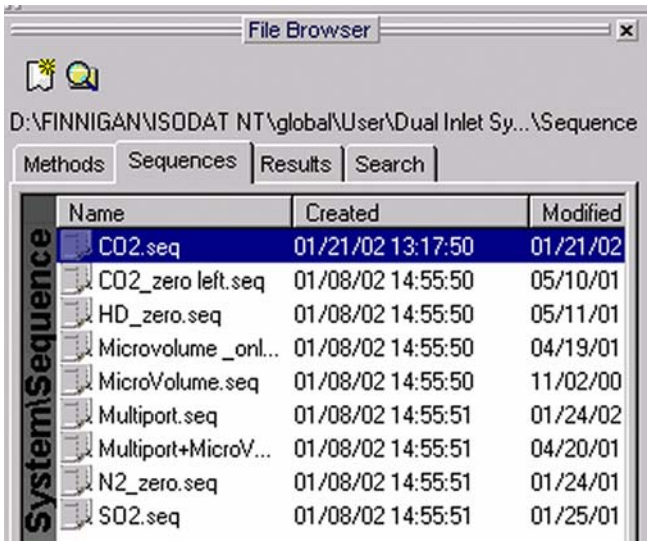


- Click ***OK***.

- On the File Browser, select the ***Sequences tab*** (default: Methods tab).



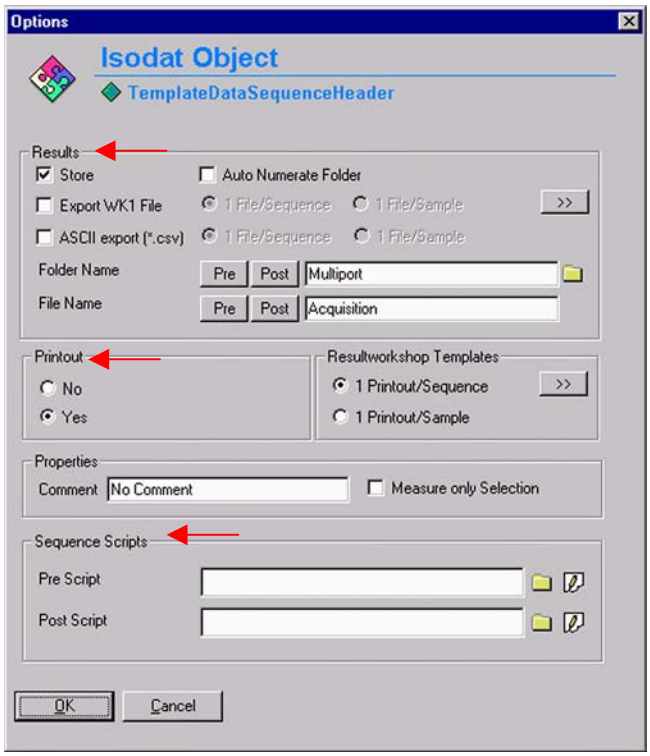
- From the predefined Sequences, choose a suitable one by double-click (e.g. ***"CO2.seq"***).



- In the "Method" column, the Method chosen in chapter 6.3.2 occurs as default ("CO2.met").

Normally, the Sequence List needs not to be edited further. It is possible, however, to select another Method from the "Method" column.

- Press the ***Start*** button.



- Define parameters for:
  - Results Export
  - Printout
  - Sequence Scripts

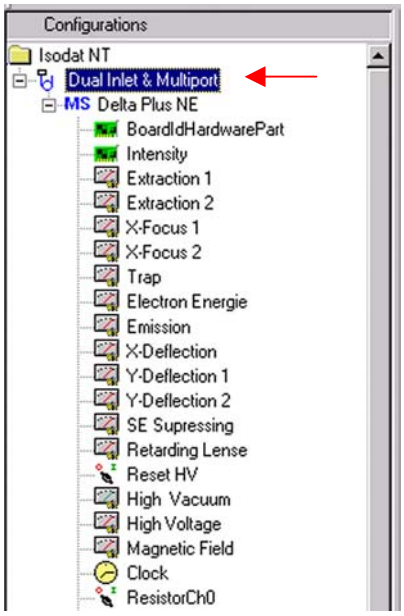
- Press ***OK*** to start Sequence Acquisition.

6.4 DUAL INLET MEASUREMENT INCLUDING MULTIPORT

6.4.1 DEFINING A CONFIGURATION

- Before operating, a ***Configuration*** containing the Dual Inlet System and the Multiport must be created in the ***Configurator*** as follows.

- Add a new Configuration using the ***Add Configuration*** button.

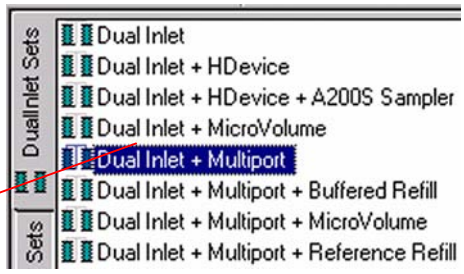


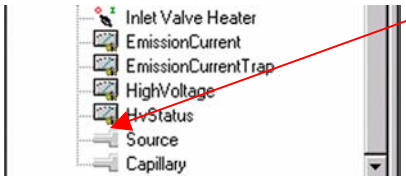
- Give it a significant name, e.g. ***"Dual Inlet & Multiport"***.

- Open the tree structure by a click on ⊞ at ⊞ MS Delta Plus NE

- On the right pane, select

  Dual Inlet + Multiport.





- Drag it to the Source port of the new Configuration.

  A ⊞ sign appears:

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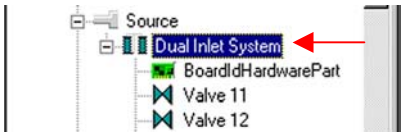
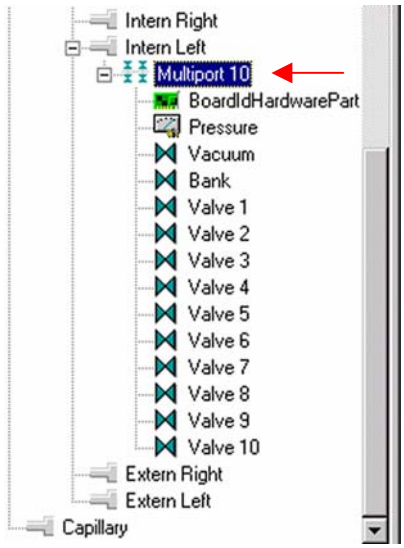


➢ The ***Dual Inlet*** device has been attached to the ***Source***.



➢ The ***Multiport*** has been attached to the ***Intern Left*** Port.

➢ Close the Configurator window.

All settings will be saved automatically.

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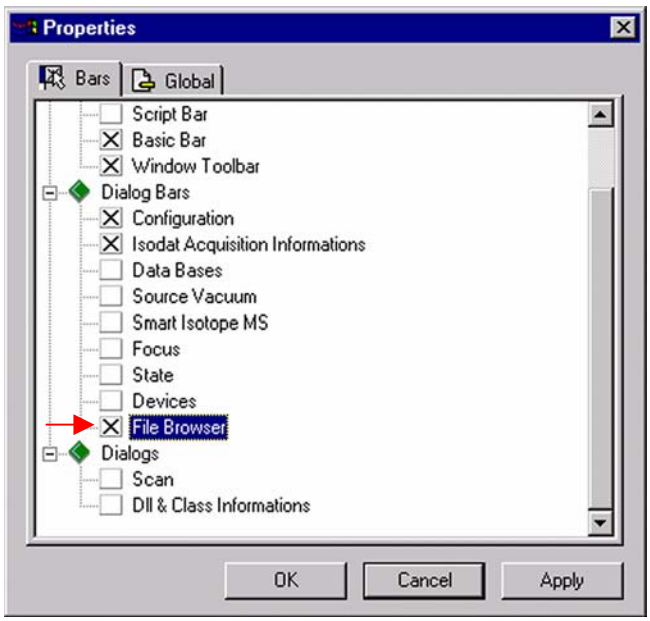
6.4.2 USING A PREDEFINED METHOD

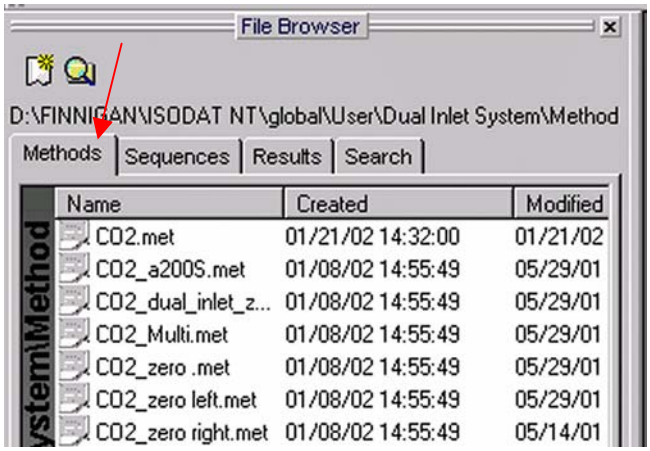
- If the File Browser cannot be seen, press the ***Options*** button.

- Activate the ***File Browser*** check box.

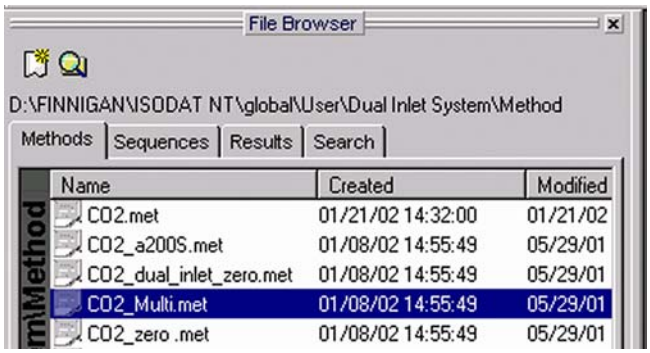


- Click ***OK***.

- On the File Browser, select the ***Methods tab*** (default).



- From the predefined Methods choose ***"CO2_multi.met"*** by double-click.

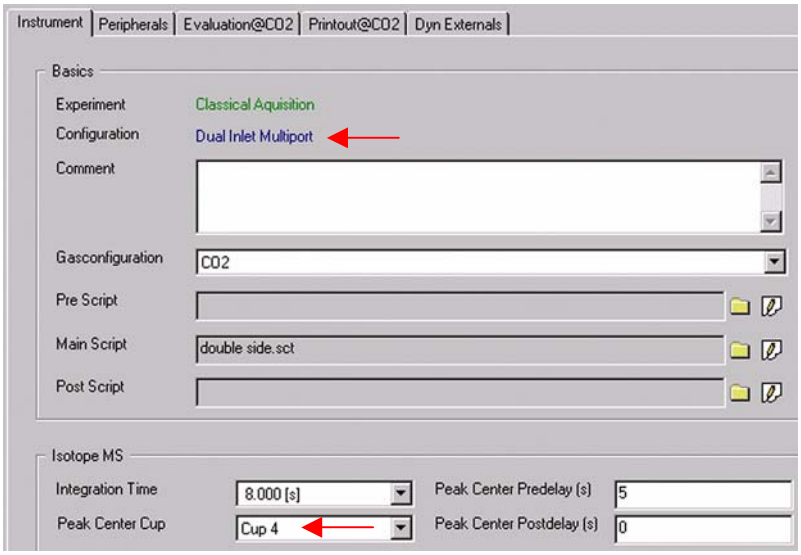


- If the Standards in the Method to be opened are older than the actual ones in the Standard Database, or if the user has changed the Method, the warning beside appears.

- Press ***OK***. Thus, the Method will be corrected: the actual values of the Standard Database (e.g. the laboratory Standard has changed or been replaced by another one) are transferred to the Method.

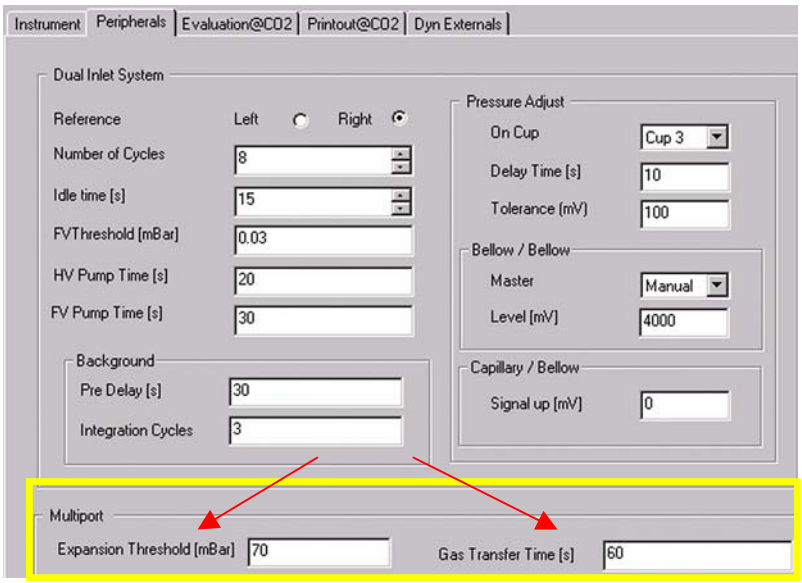
- The ***Instrument tab*** of the Method "CO2_multi.met" appears.

For details, see chapter 6.3.2.



NOTE: ***Ensure, that the proper peak center cup is selected. If necessary, correct the default cup (i.e. Cup 4)!***

➢ See chapter 6.3.2 for details at ***Peripherals tab***.

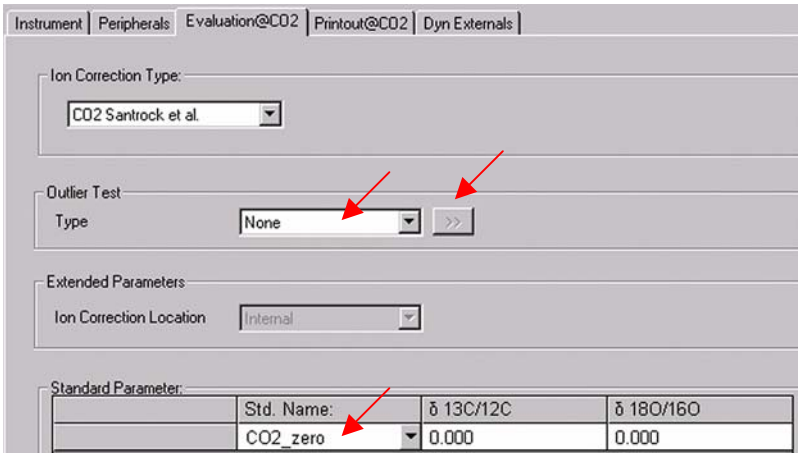


NOTE: ***For measurements including Multiport two additional parameters occur:***

- ***Gas Transfer Time:*** ***time the sample gas needs to pass from the Multiport to the sample bellow (e.g. 60 s). The valves "bank", "12", "14" and "15" are opened.***

- ***Expansion Threshold:*** ***when the sample has entered its bellow, the sample gas pressure within it must not exceed the Expansion Threshold at bellow 100 % open (e.g. 70 mbar). If the pressure exceeds the threshold, sample gas will be pumped off stepwise in at the most 10 consecutive trials. If the pressure still exceeds the threshold after these trials, the system stops. In this case, use less substance.***

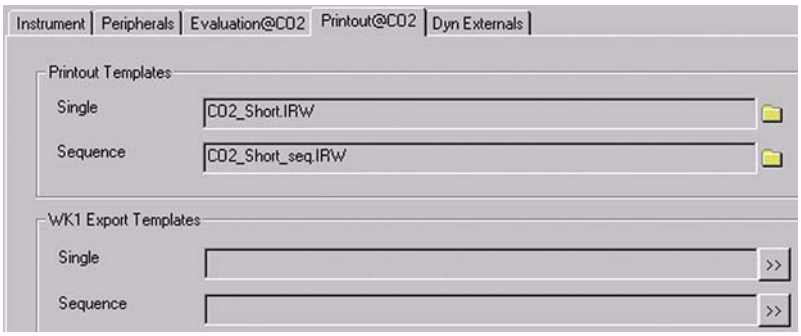
- At ***Evaluation @CO2*** tab, see chapter 6.3.2 for details.



- Select “None” or “Sigma” as Outlier test. In case of “Sigma”, specify the k-fold of the standard deviation using >>.

NOTE: ***At Std. Name, choose a suitable Standard!***

- At ***Printout tab***, see chapter 6.3.2 for details.

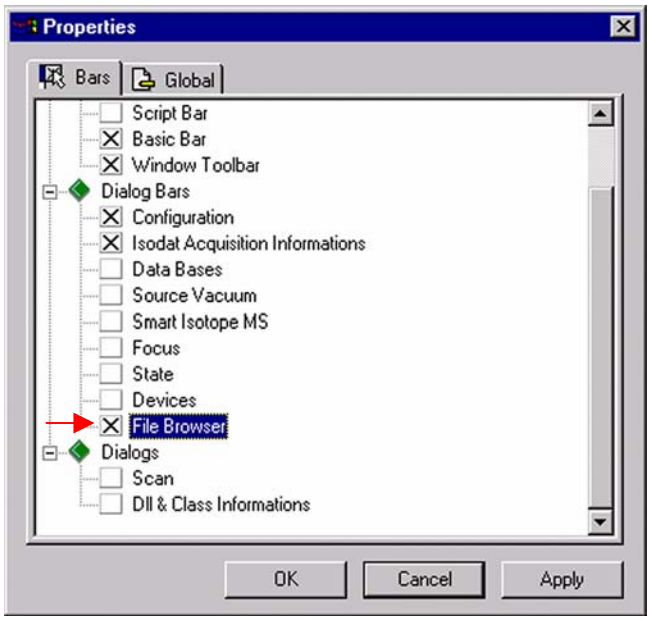


The ***Method*** for measurements using Dual Inlet in combination with a Multiport has now been established. As next step, either use a ***predefined Sequence*** (refer to chapter 6.4.3) or create a ***new Sequence*** (refer to chapter 6.1.5).

6.4.3 USING A PREDEFINED SEQUENCE

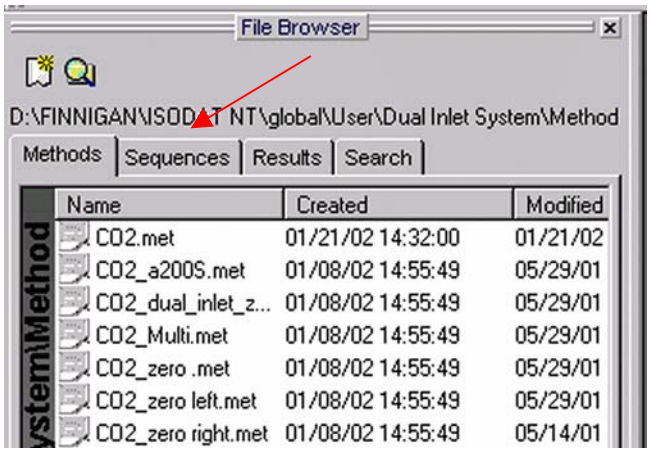
- If the File Browser cannot be seen, press the ***Options*** button.

- Activate the ***File Browser*** check box.

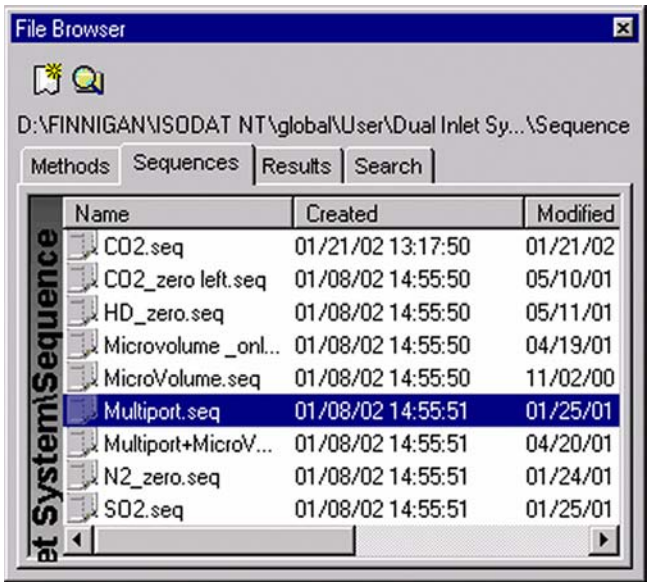


- Click ***OK***.

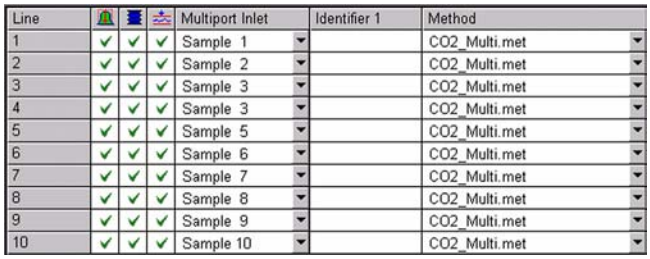
- On the File Browser, select the ***Sequences tab*** (default: Methods tab).



- From the predefined Sequences choose ***"Multiport.seq"*** by double-click.



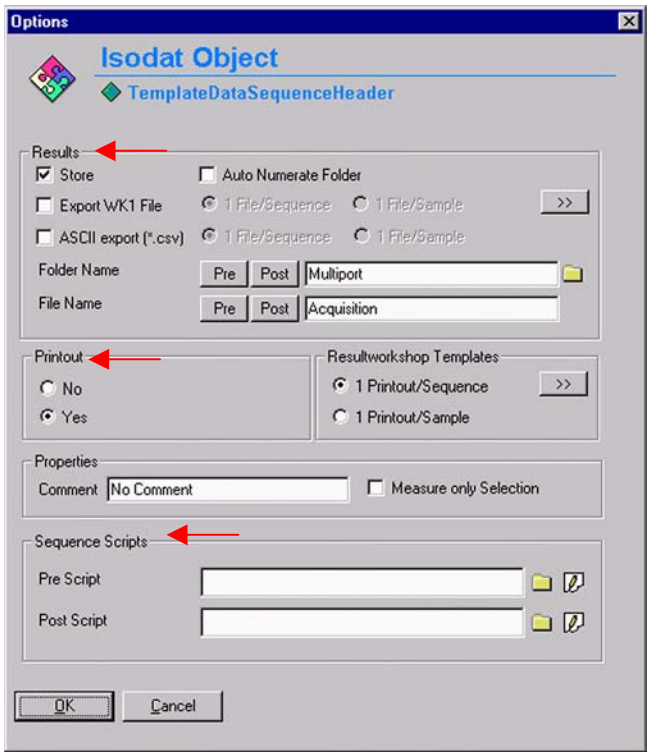
- In the "Method" column, the Method chosen in chapter 6.4.2 occurs as default ("CO2_Multi.met").



Normally, the Sequence List needs not to be edited further. It is possible, however, to select another Method from the "Method" column.

NOTE: ***A new column, "Multiport Inlet", occurs. It defines, which Multiport Inlet port will be connected to which sample (e.g. Inlet port 8 to Sample 8). It is possible to measure a sample repeatedly (e.g. connect both Sample 3 and Sample 4 to the Multiport Inlet port 3).***

- Press the ***Start*** button.



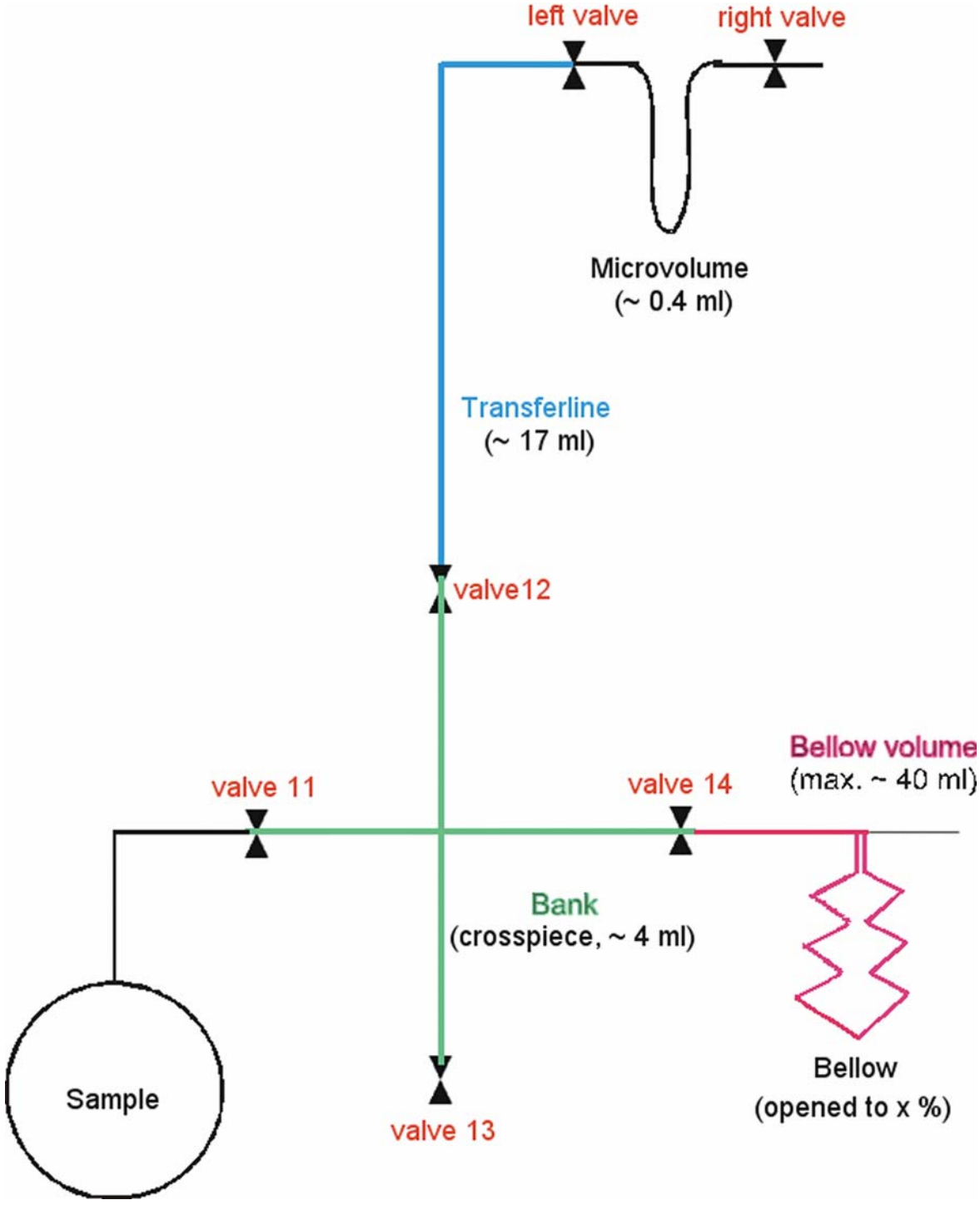
- Define parameters for:
  - Results Export
  - Printout
  - Sequence Scripts

- Press ***OK*** to start Sequence Acquisition.

6.5 DUAL INLET MEASUREMENT INCLUDING MICROVOLUME

6.5.1 MICROVOLUME - MEASUREMENT PRINCIPLE

6.5.1.1 MICROVOLUME WITHOUT MULTIPORT - MEASUREMENT PRINCIPLE



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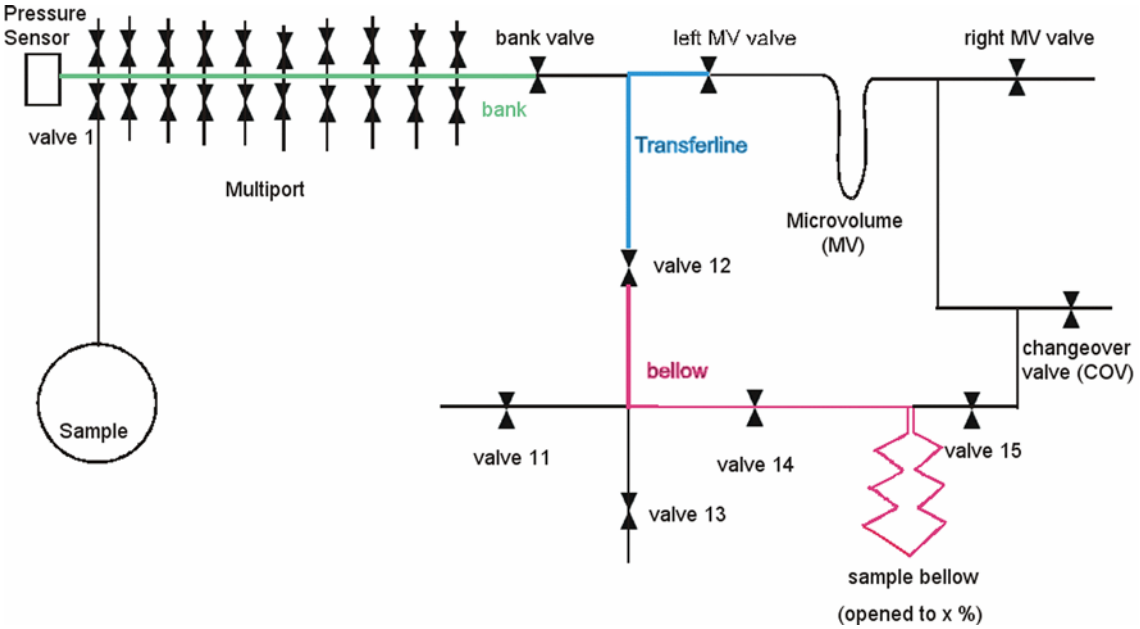
6.5.1.2 MICROVOLUME VIA MULTIPORT - MEASUREMENT PRINCIPLE



Two fundamental measuring modes exist for using a Microvolume (via Multiport):

- ***Expansion*** mode
- ***Freeze direct*** mode

6.5.1.3 EXPANSION MODE – GENERAL REMARKS

Expansion mode is used, if the amount of gas to be measured is unknown. Depending on the unknown gas amount, the gas can be expanded into three different volumes:

- ***Bank***: Volume between valves 11, 12, 13 and 14
- ***Bank and Transferline***: Bank + volume between valve 12 and left valve of Microvolume
- ***Bank, Transferline and Bellow***: Bank + Transferline + Bellow, which is opened to x % (i.e. Bellow Pos. [%]).

6.5.1.4 EXPANSION MODE – BEFORE MEASUREMENT

1 Attach the sample to be measured to a sample valve (e.g. valve 1).

2 Pump the volume between valve 1 and the sample valve. Close valve 1 and open sample valve.

3 Ensure that on the Standard side enough gas is available.

4 If Reference Refill is planned, activate  in the Sequence list.

5 Start the Acquisition.

6 After starting the Acquisition, the bellow of the Sample side (i.e. mostly left) will be opened fully, i.e. to 100 %.

7 A first evacuation step by means of the fore vacuum pump takes places over the baseline (if after a certain time, which you can edit via a Script, the vacuum has not reached FV Threshold, the measurement will be canceled. An error message then appears).

8 If FV Threshold has been reached, the fore vacuum pump continues to pump until FV Pump Time (e.g. 10 s) has elapsed.

9 The bank valve, wasteline valve, left valve of Microvolume, right valve of Microvolume are opened as well as the valves 12, 13, 14, 15, 16 and 39.

10 These opened valves are evacuated stepwise by means of the fore vacuum pump: first, only the bellow is evacuated. When FV Threshold has been reached in the bellow, the bank valve and then all other valves are evacuated one by one. To proceed evacuation, attainment of FV Threshold is required on every step: if FV Threshold is not reached on one of the steps, measurement will be canceled. An error message then appears.

11 A switch to High Vacuum is performed. A second evacuation step takes now place by means of the high vacuum pump until HV Pump Time has elapsed (e.g. 60 s). All valves are opened with the exception of valve 39. Valve 40 is set to “Toggle Switch”. ISODAT NT opens valve 39 while closing valve 40 and vice versa.

12 Bank valve, wasteline valve, left valve of Microvolume, right valve of Microvolume are closed as well as valves 12, 13, 14, 16 and 40. Only valve 15 remains opened.

13 The sample valve of the Multiport (e.g. valve 1) is opened. The system waits until Sample Equilibration Time has elapsed (e.g. 60 s). The pressure p is measured via the sensor.

14 If you selected only Expansion mode (i.e. “Bank”), the sample gas will only be transferred into the bank volume.

If you selected “Bank and Transferline”, the bank valve must be opened additionally in order to transfer the sample gas into bank volume plus transferline.

If you selected “Bank, Transferline and Bellow”, the bank valve, valve 12 and valve 14 must be opened to transfer the sample gas throughout the whole system.

15 The pressure p is determined using a pressure-meter (i.e. sensor).

6.5.1.5 EXPANSION MODE – MEASUREMENT WITH BANK

1 The pressure p is measured using a pressure-meter (i.e. sensor).

2 If ***p > Freeze Bank Threshold*** (e.g. p > 6 mbar), the pressure is too high.

3 To reduce p, the gas in the bank volume has to be pumped off. Therefore, the sample valve, e.g. valve 1, is closed (all valves for the other samples, which currently are out of interest, are closed anyway).

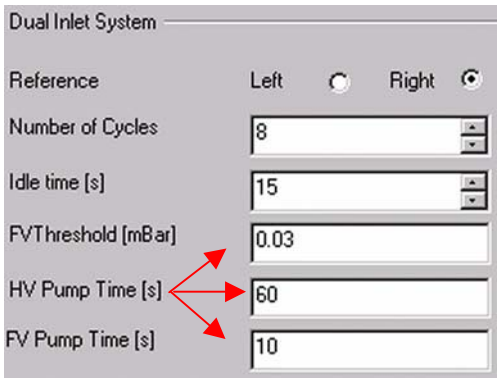
4 The gas in the bank volume is pumped off.

5 The sample valve, e.g. valve 1, is opened (of course, all other sample valves remain closed). The sample gas expands into the bank volume.

6 It is waited until Sample Equilibration Time has elapsed (e.g. 60 s).

7 The pumping time depends on the three parameters

- FV Threshold [mbar]
- HV Pump Time [s]
- FV Pump Time [s]



8 After pumping the sample valve (e.g. valve 1) is opened. Again, the gas expands into the Bank volume. Again, it is waited, until the Sample Equilibration Time (e.g. 60 s) has elapsed.

9 The decreased pressure p is measured again and compared to the Freeze Bank Threshold.

Case 1: If ***Freeze All Threshold < p < Freeze Bank Threshold***, the sample valve (e.g. valve 1) is closed. The transfer process of the sample into the Microvolume - which is explained elsewhere - starts. Measurement begins.

Case 2: If ***p < Freeze All Threshold***, only so few gas is available that it can be transferred immediately and measured as a whole (the sample valve, e.g. valve 1, will not be closed any longer).

6.5.1.6 EXPANSION MODE – MEASUREMENT WITH BANK AND TRANSFERLINE

The working principle is the same as described at “Expansion Mode - Bank”. Only the route of the sample gas is longer.

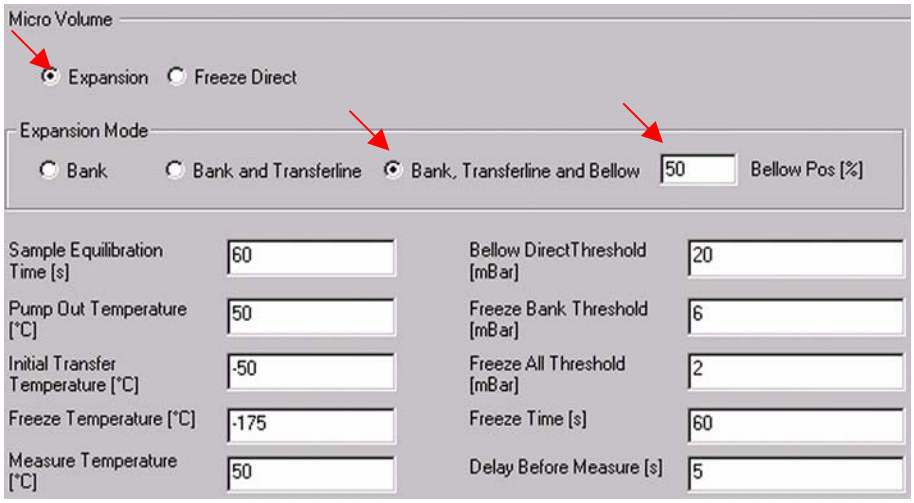
Example

1 If ***Freeze All Threshold < p < Freeze Bank Threshold***, the sample gas in the Transferline is pumped out to prevent Transferline gas from reaching the Microvolume.

2 Only the sample gas in the Bank volume will be measured.

6.5.1.7 EXPANSION MODE – MEASUREMENT WITH BANK, TRANSFERLINE AND BELLOW

This expansion mode is used if large amounts of sample gas are available.



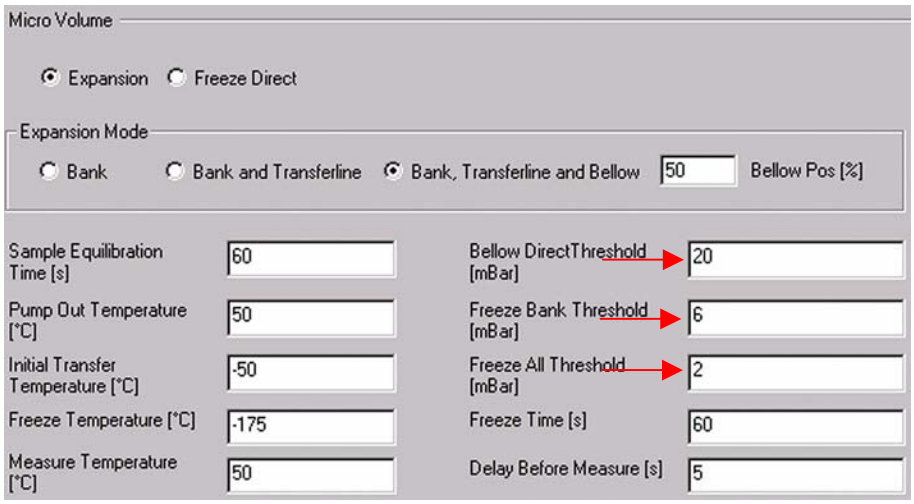
1 The bellow is opened to the specified percentage (e.g. 50 %).

2 The sample valve (e.g. valve 1) is opened.

3 Bank valve, valve 12 and valve 14 are opened.

4 The sample gas is transferred up to the bellow (i.e. the whole volume is filled with gas).

5 The pressure p is measured.



Case 1: If ***p > Bellow Direct Threshold***, the sample is measured via the bellow.

Case 2: If ***Freeze Bank Threshold < p < Bellow Direct Threshold***, the sample valve (e.g. valve 1) is closed. The whole gas is pumped out. If nevertheless Freeze Bank Threshold < p < Bellow Direct Threshold is still valid, the process will be repeated.

Case 3: If ***Freeze All Threshold < p < Freeze Bank Threshold***, Bank valve and sample valve (e.g. valve 1) are closed. After pumping off a part of the gas, only the gas in the bank volume will be frozen.

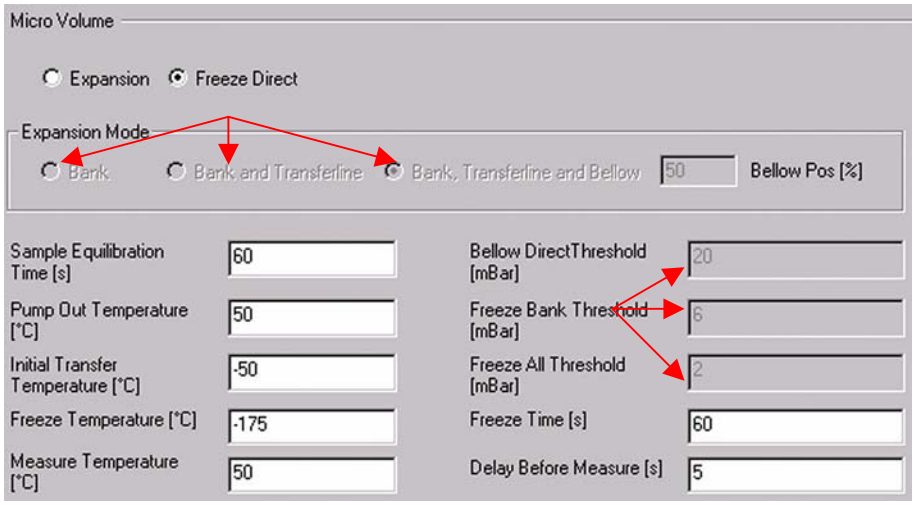
Case 4: If ***p < Freeze All Threshold***, only few gas is available. Therefore, the whole gas amount will be frozen.

6.5.1.8 FREEZE DIRECT MODE

Freeze direct mode is used

- if the amount of sample gas to be analyzed is known
- in case of very small gas samples

The sample gas will not be expanded but transferred to the Microvolume directly. There, it will be frozen according to the parameter values and finally be measured. Therefore, note that the fields related to Expansion mode are inactive:



1 The sample valve (e.g. valve 1) is opened.

2 The gas in the bank volume is equilibrated until Sample Equilibration Time has elapsed.

3 Simultaneously, the Microvolume is heated to Pump Out Temperature (e.g. 50 °C). At this slightly elevated temperature, evaporation of impurities and water is facilitated. They are pumped off. Thus, the Microvolume is cleansed.

4 The Microvolume is cooled down until Initial Transfer Temperature (e.g. - 50 °C) is reached.

5 At Initial Transfer Temperature (e.g. -50 ºC), bank valve and left valve of Microvolume are opened. Thus, the sample gas is transferred to the Microvolume.

6 The sample gas having just entered the Microvolume is cooled down further until Freeze Temperature (e.g. -175 ºC) is reached.

7 The system waits, until Freeze Time has elapsed (e.g. 60 s).

8 Then, bank valve and left valve of Microvolume are closed.

9 The Microvolume is heated up to Measure Temperature (e.g. 50 ºC).

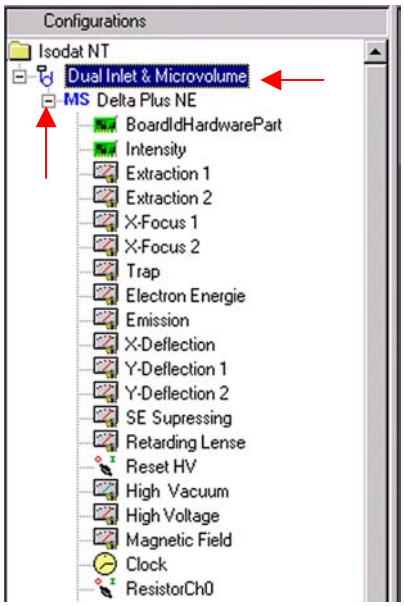
10 The system waits until Delay Before Measure has elapsed.

11 Measurement starts.

6.5.2 DEFINING A CONFIGURATION

➢ Before operating, a ***Configuration*** containing the Dual Inlet System and the Microvolume must be created in the ***Configurator*** as follows.

➢ Add a new Configuration using the ***Add Configuration*** button.

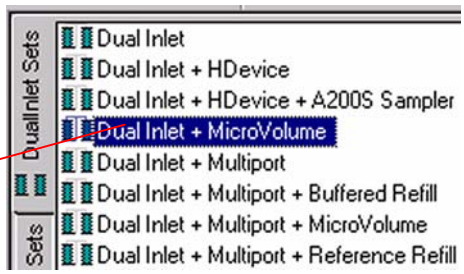


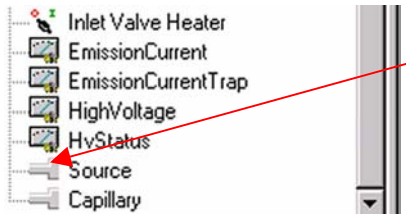
➢ Give it a significant name, e.g. ***"Dual Inlet & Micro-volume"***.

➢ Open the tree structure by a click on ⊞ at ⊞ MS Delta Plus NE

➢ On the right pane, select

"Dual Inlet + Micro-volume".





➢ Drag it to the Source port ⊣ of the new Configuration.

A ⊞ sign appears:

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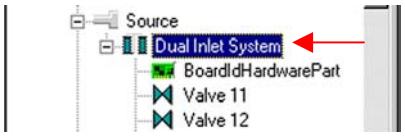
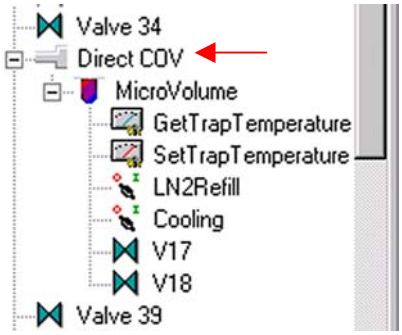


- The ***Dual Inlet*** device has been attached to the ***Source***.



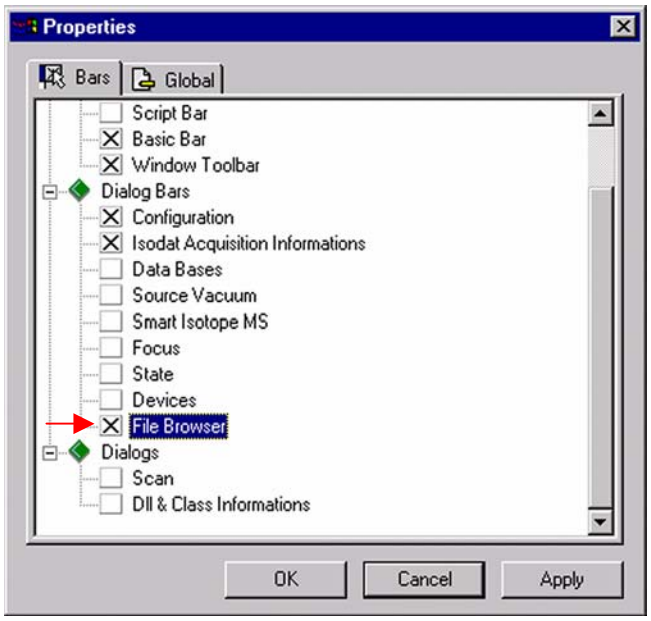
- The ***Microvolume*** has been attached to the ***Direct COV*** Port.

- Close the Configurator window.
  All settings will be saved automatically.

6.5.3 USING A PREDEFINED METHOD

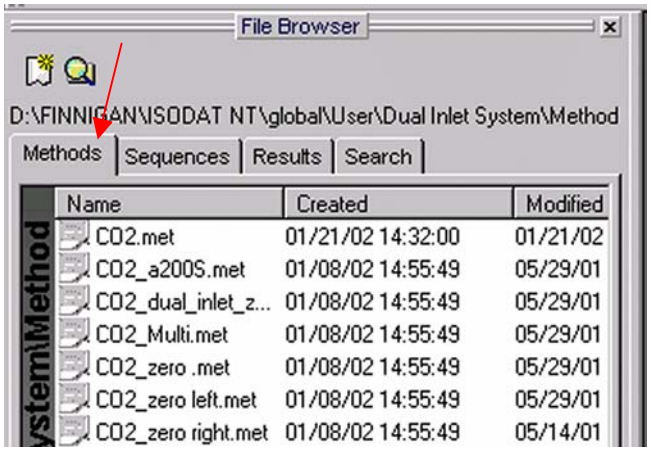
- If the File Browser cannot be seen, press the ***Options*** button.

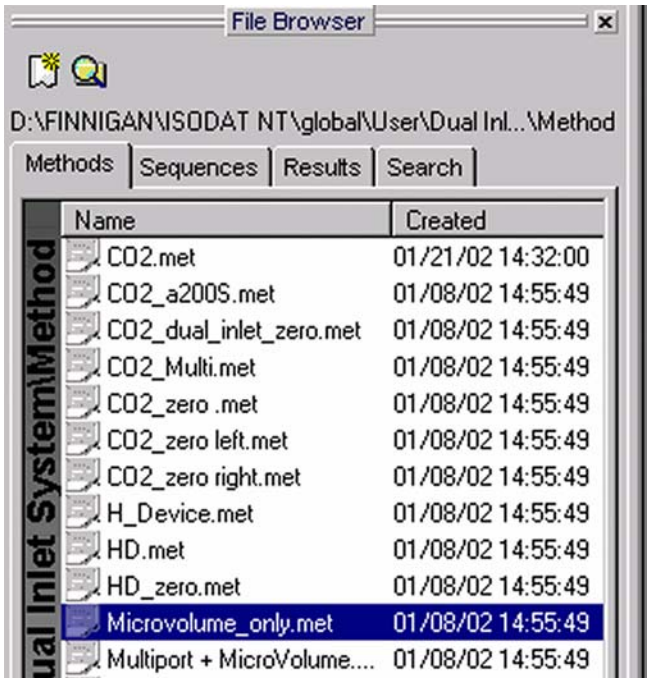
- Activate the ***File Browser*** check box.



- Click ***OK***.

- On the File Browser, select the ***Methods tab*** (default).





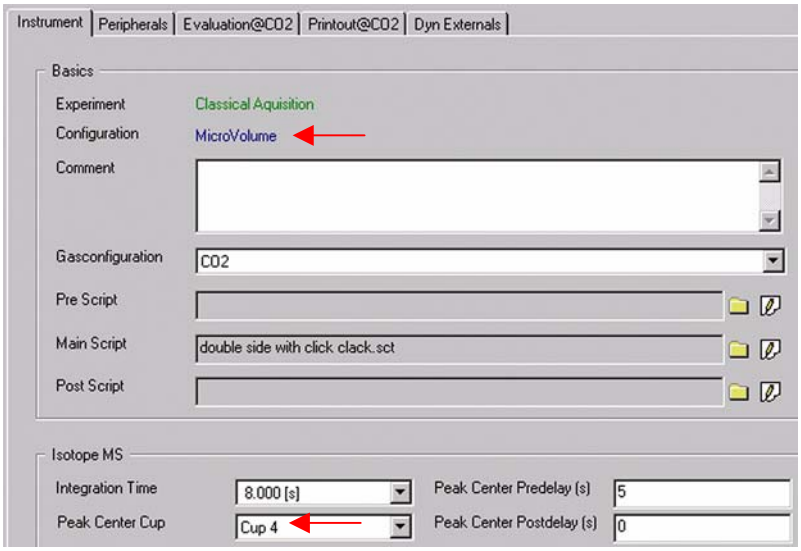
- From the predefined Methods choose ***"Microvolume_only.met"*** by double-click.

- If the Standards in the Method to be opened are older than the actual ones in the Standard Database, or if the user has changed the Method, the warning beside appears.

- Press ***OK***. Thus, the Method will be corrected: the actual values of the Standard Database (e.g. the laboratory Standard has changed or been replaced by another one) are transferred to the Method.

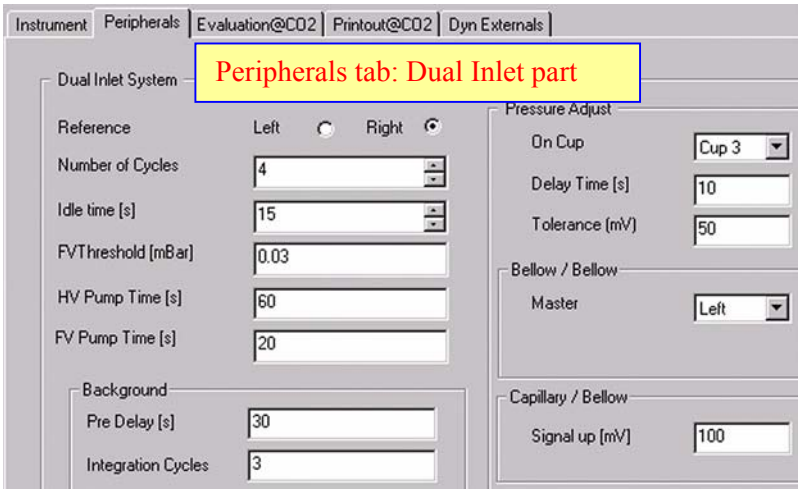
- The ***Instrument tab*** of the Method "Micro-volume_only.met" appears.

- For details, see chapter 6.3.2.

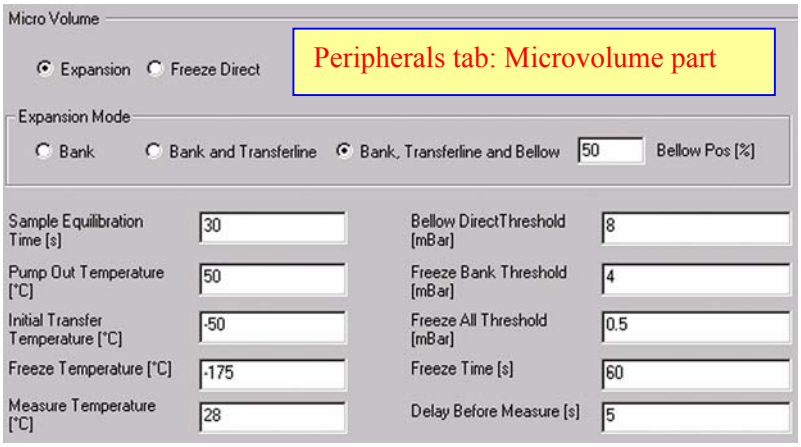


NOTE: ***Ensure, that the proper peak center cup is selected. If necessary, correct the default cup (i.e. Cup 4)!***

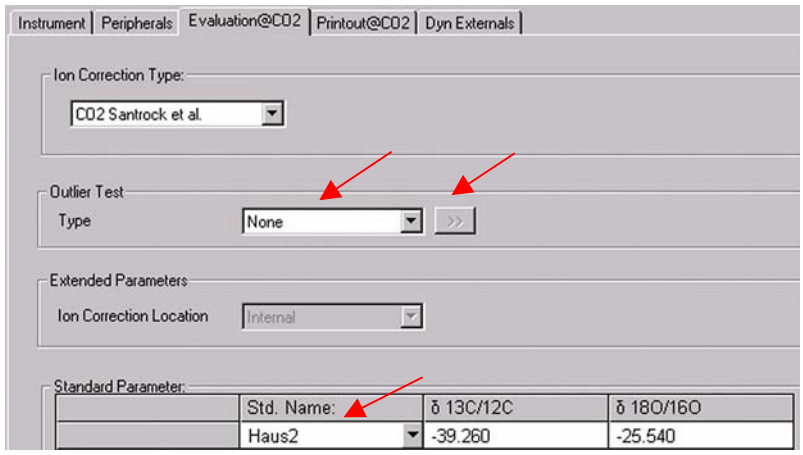
- For details about the Peripherals tab's ***Dual Inlet*** part, see chapter 6.3.2.



- For details about the Peripherals tab's ***Microvolume*** part, see chapter 6.3.2.



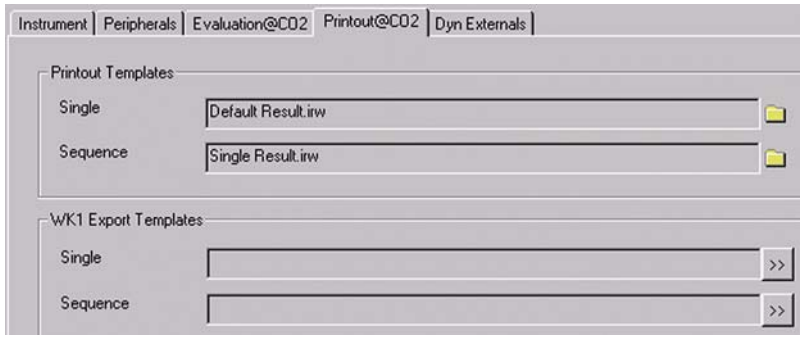
- At ***Evaluation @CO2 tab***, select an Ion Correction type (e.g. “CO2 Santrock et al.”).
- Select “None” or “Sigma” as Outlier test. In case of “Sigma”, specify the k-fold of the standard deviation using >>.



For details, see chapter 6.3.2.

NOTE: ***At Std. Name, choose a suitable Standard!***

- For details at the ***Printout tab***, see chapter 6.3.2.

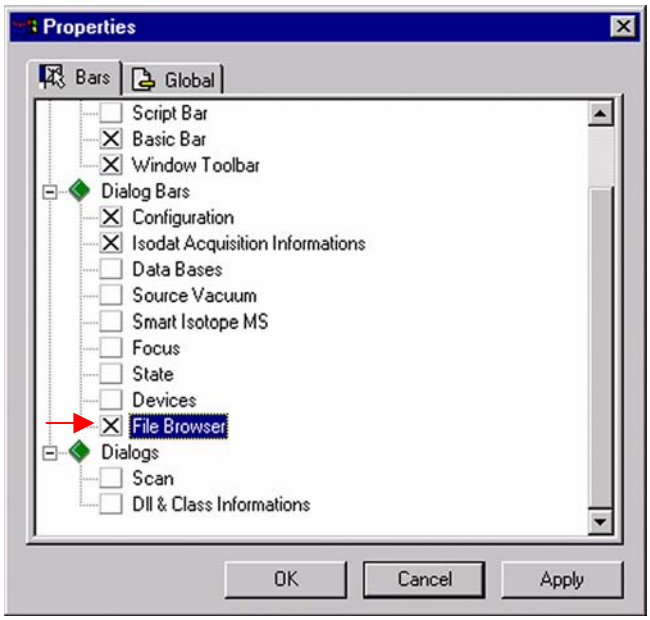


The ***Method*** for measurements using Dual Inlet in combination with a Microvolume has now been established. As next step, either use a ***predefined Sequence*** (refer to chapter 6.5.4) or create a ***new Sequence*** (refer to chapter 6.1.5).

6.5.4 USING A PREDEFINED SEQUENCE

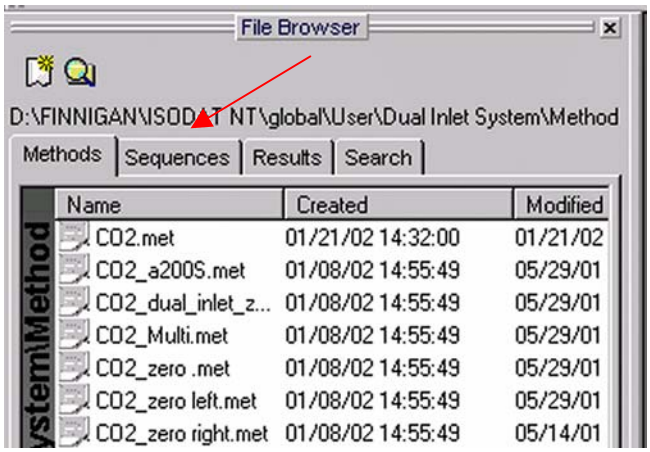
- If the File Browser cannot be seen, press the ***Options*** button.

- Activate the ***File Browser*** check box.

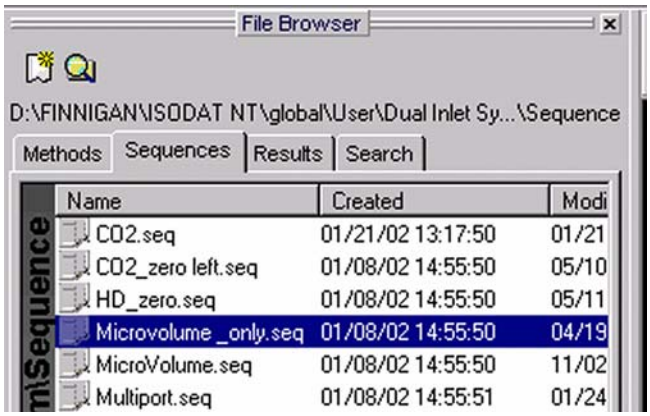


- Click ***OK***.

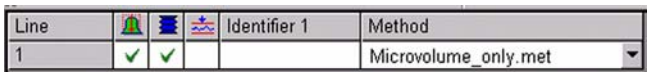
- On the File Browser, select the ***Sequences tab*** (default: Methods tab).



- From the predefined Sequences choose ***"Microvolume_only.seq"*** by double-click.

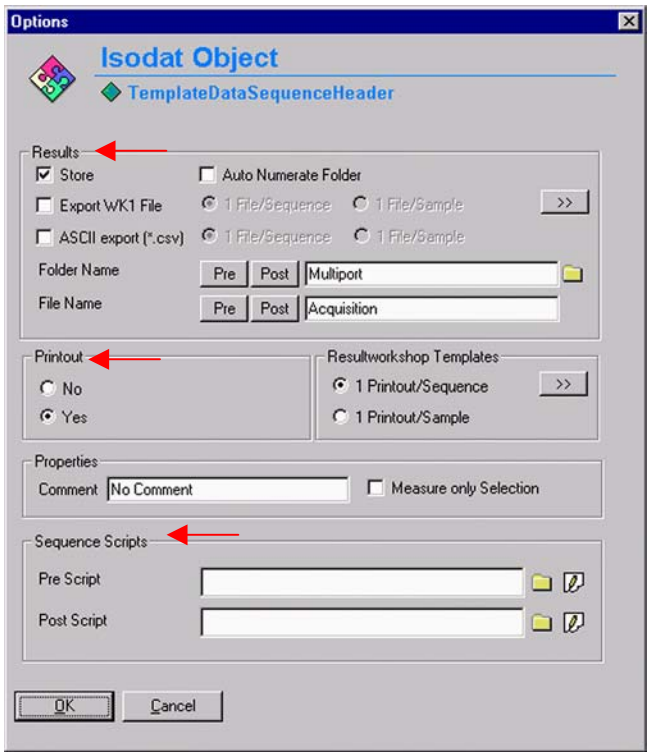


- In the "Method" column, the Method chosen in chapter 6.5.3 occurs as default ("CO2_Multi.met").



Normally, the Sequence List needs not to be edited further. It is possible, however, to select another Method from the "Method" column.

- Press the ***Start*** button.



- Define parameters for:
  - Results Export
  - Printout
  - Sequence Scripts

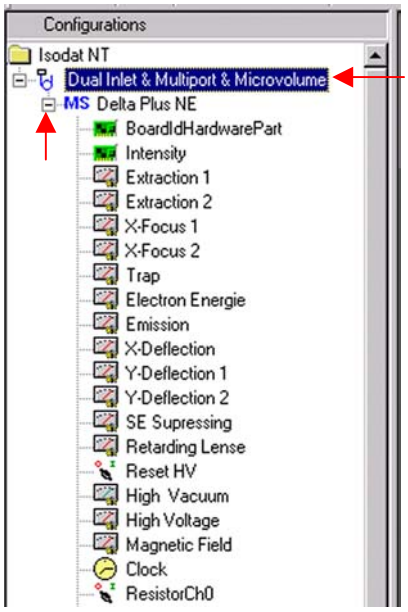
- Press ***OK*** to start Sequence Acquisition.

6.6 DUAL INLET MEASUREMENT INCLUDING MULTIPORT AND MICROVOLUME

6.6.1 DEFINING A CONFIGURATION

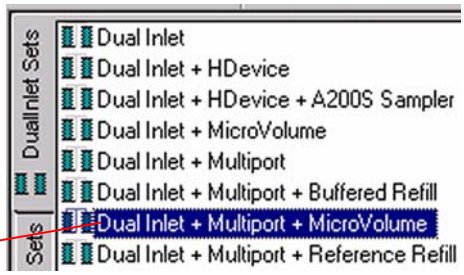
➢ Before operating, a ***Configuration*** containing the Dual Inlet System, the Multiport and the Microvolume must be created in the ***Configurator*** as follows.

➢ Add a new Configuration using the ***Add Configuration*** button.



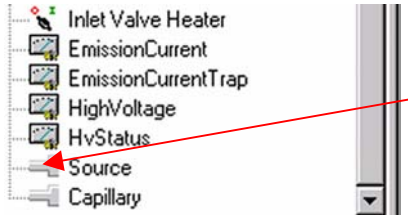
➢ Give it a significant name, e.g. ***"Dual Inlet & Multiport & Microvolume"***.

➢ Open the tree structure by a click on ⊞ at ⊞ MS Delta Plus NE



➢ On the right pane, select

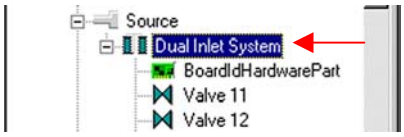
Dual Inlet +Multiport + Microvolume.



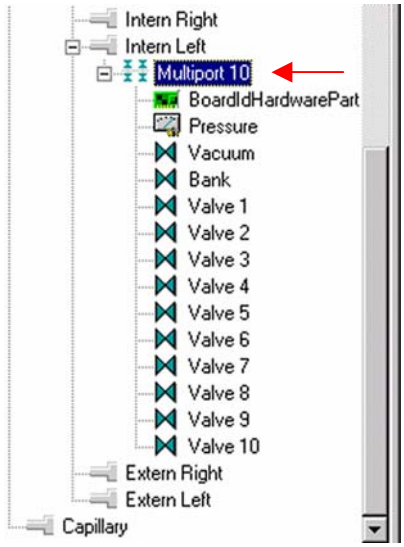
➢ Drag it to the Source port of the new Configuration.

A ⊞ sign appears:

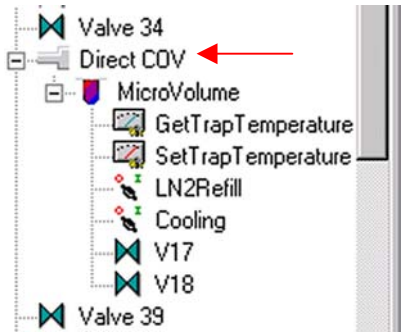
Thermo Finnigan



➢ The ***Dual Inlet*** device has been attached to the ***Source***.



➢ The ***Multiport*** has been attached to the ***Intern Left*** Port.



➢ The ***Microvolume*** has been attached to the ***Direct COV*** Port.

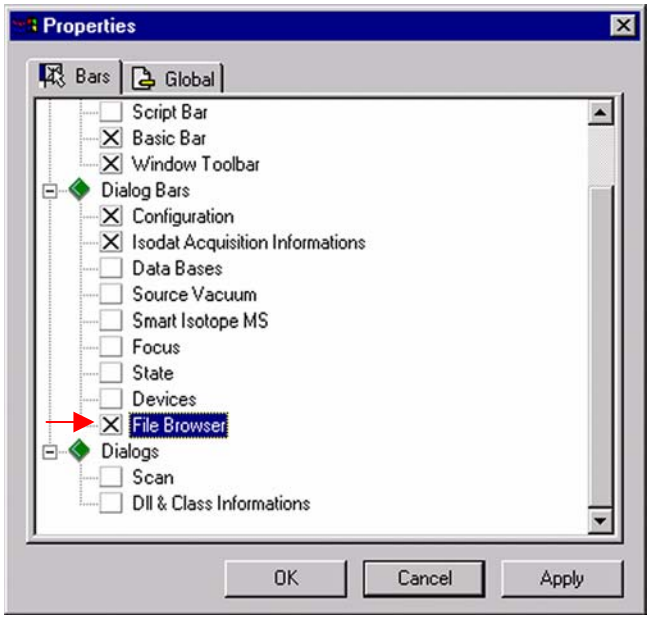
➢ Close the Configurator window.

All settings will be saved automatically.

6.6.2 USING A PREDEFINED METHOD

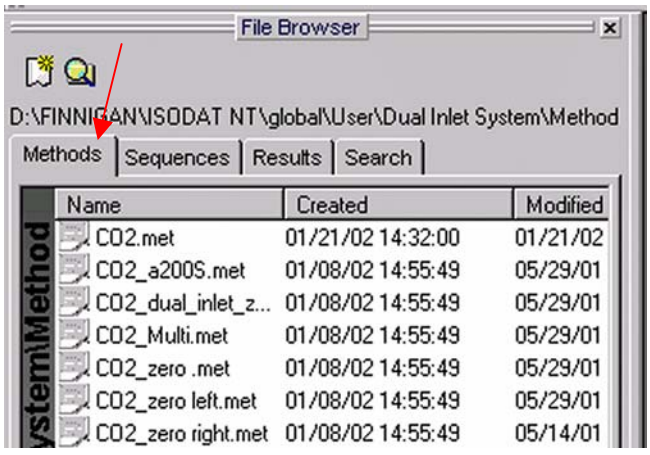
- If the File Browser cannot be seen, press the ***Options*** button.

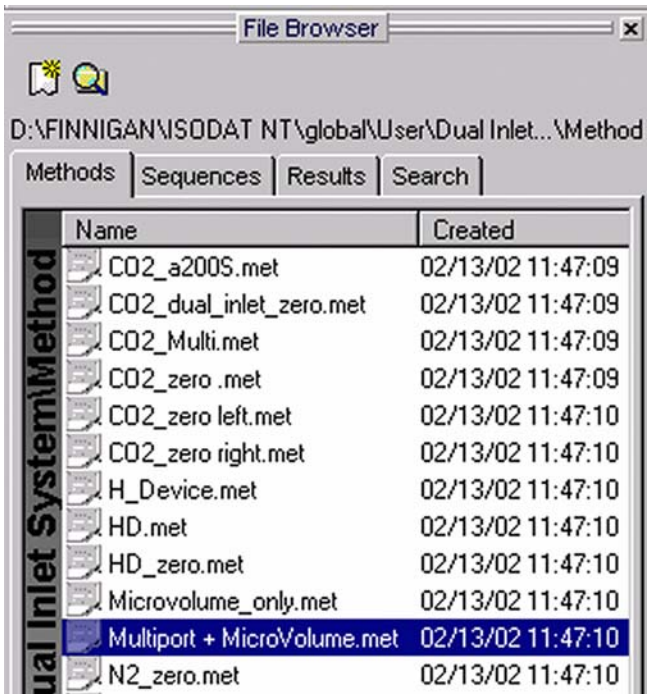
- Activate the ***File Browser*** check box.



- Click ***OK***.

- On the File Browser, select the ***Methods tab*** (default).



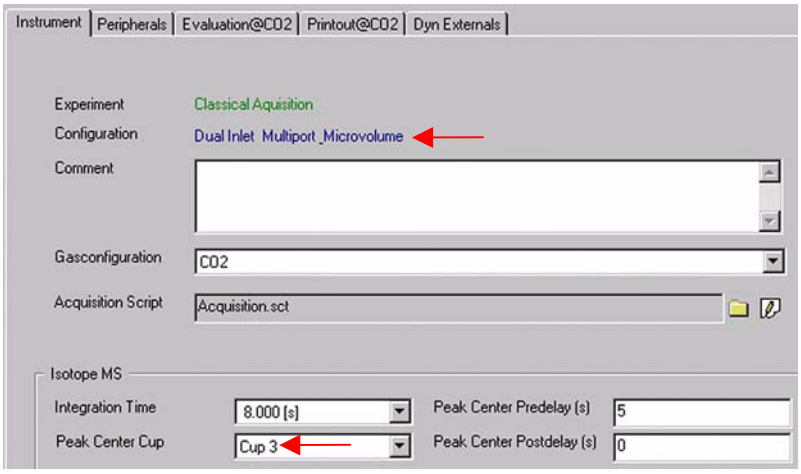


- From the predefined Methods choose ***"Multiport + Micro-volume.met"*** by double-click.

- If the Standards in the Method to be opened are older than the actual ones in the Standard Database, or if the user has changed the Method, the warning beside appears.

- Press ***OK***. Thus, the Method will be corrected: the actual values of the Standard Database (e.g. the laboratory Standard has changed or been replaced by another one) are transferred to the Method.

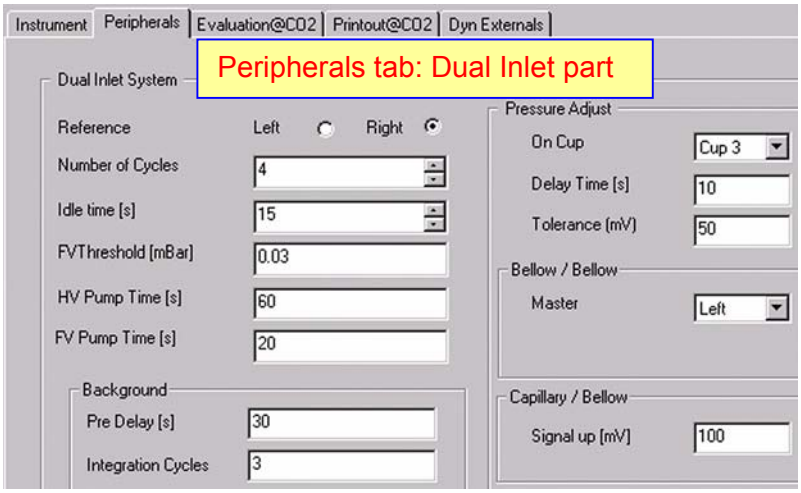
- The ***Instrument tab*** of the Method "Multiport + Micro-volume.met" appears.



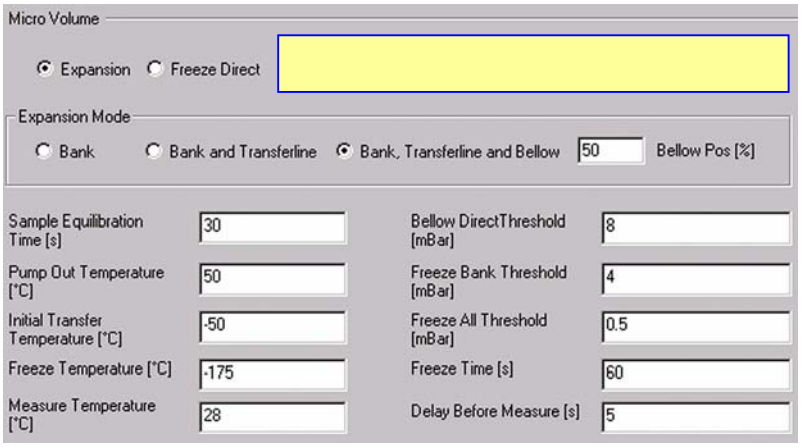
- For details see page chapter 6.3.2

NOTE: ***Ensure, that the proper peak center cup is selected. If necessary, correct the default cup!***

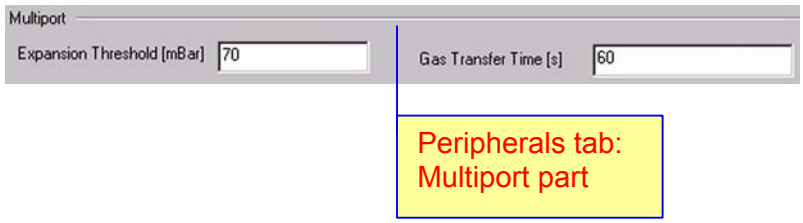
- For details at the Peripherals tab's ***Dual Inlet*** part, see chapter 6.3.2.



- For details at the Peripherals tab's ***Microvolume*** part, see chapter 6.5.3.



- For details at the Peripherals tab's ***Multiport*** part, see chapter 6.4.2.

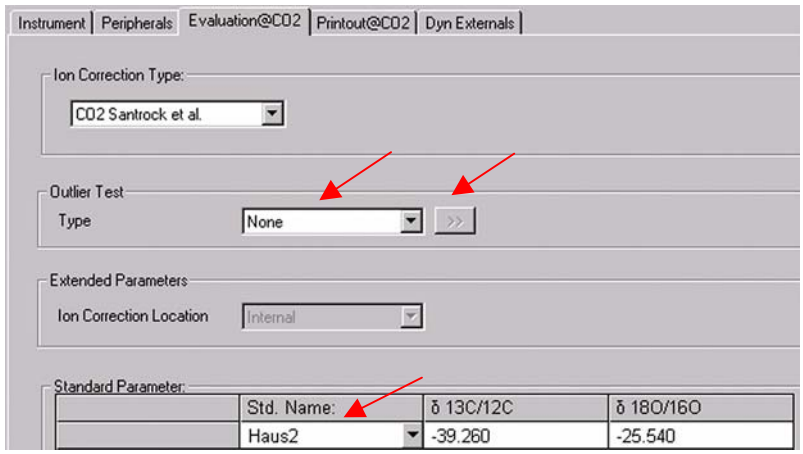


- At ***Evaluation @CO2 tab***,

  Select an Ion Correct type,

  e.g. "CO2 Santrock et al.".

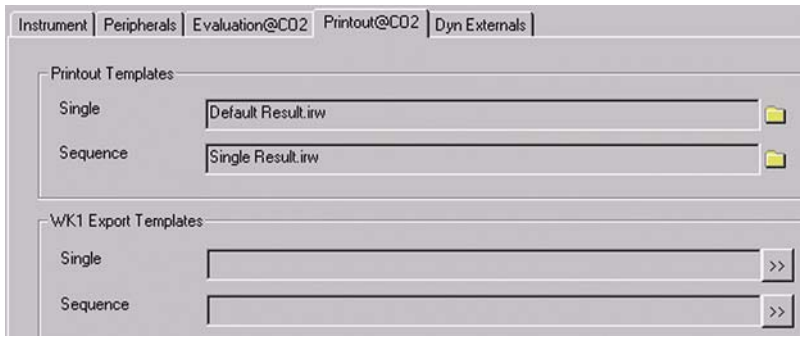
- Select "None" or "Sigma" as Outlier test. In case of "Sigma", specify the k-fold of the standard deviation using >>.



  For details, see chapter 6.3.2.

NOTE: ***At Std. Name, choose a suitable Standard!***

- For details at ***Printout tab***, see chapter 6.3.2.

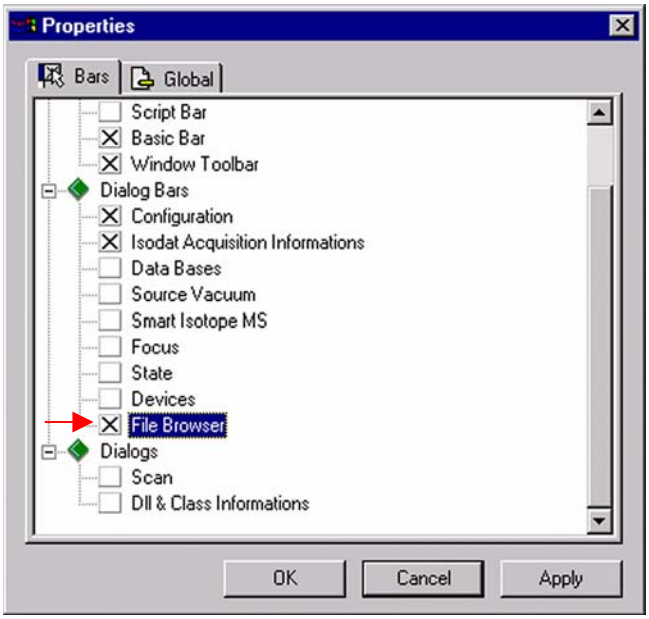


The ***Method*** for measurements using Dual Inlet in combination with a Multiport and a Micro-volume has now been established. As next step, either use a ***predefined Sequence*** (refer to chapter 6.6.3) or create a ***new Sequence*** (refer to chapter 6.1.5).

6.6.3 USING A PREDEFINED SEQUENCE

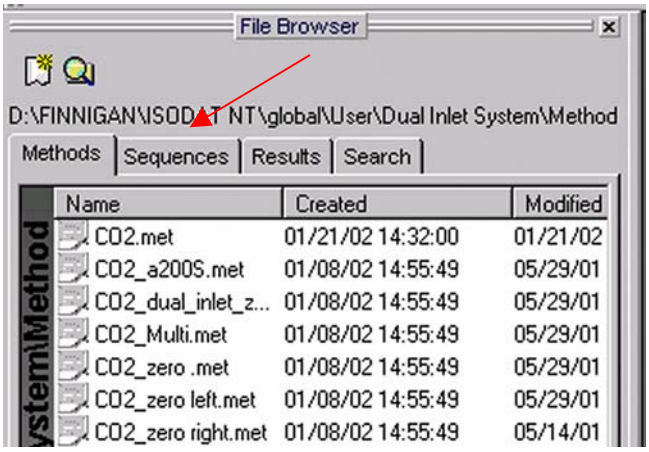
- If the File Browser cannot be seen, press the ***Options*** button.

- Activate the ***File Browser*** check box.

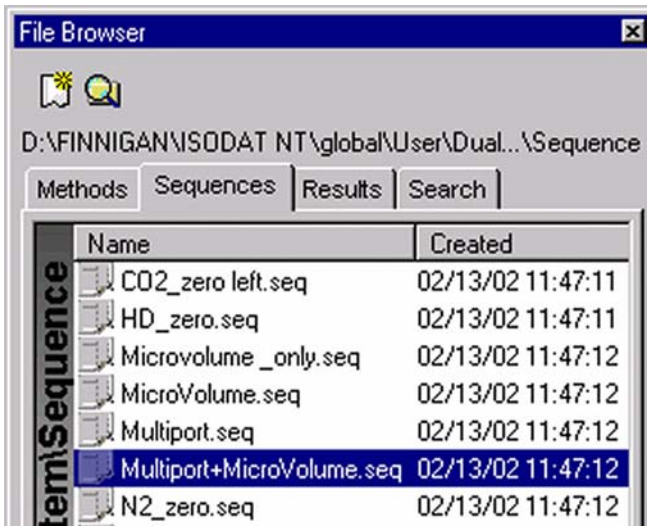


- Click ***OK***.

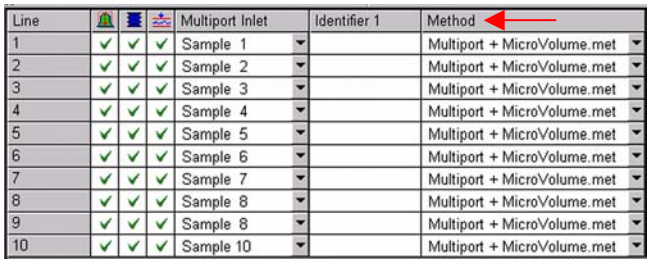
- On the File Browser, select the ***Sequences tab*** (default: Methods tab).



- From the predefined Sequences choose ***"Multiport + Micro-volume.seq"*** by double-click.



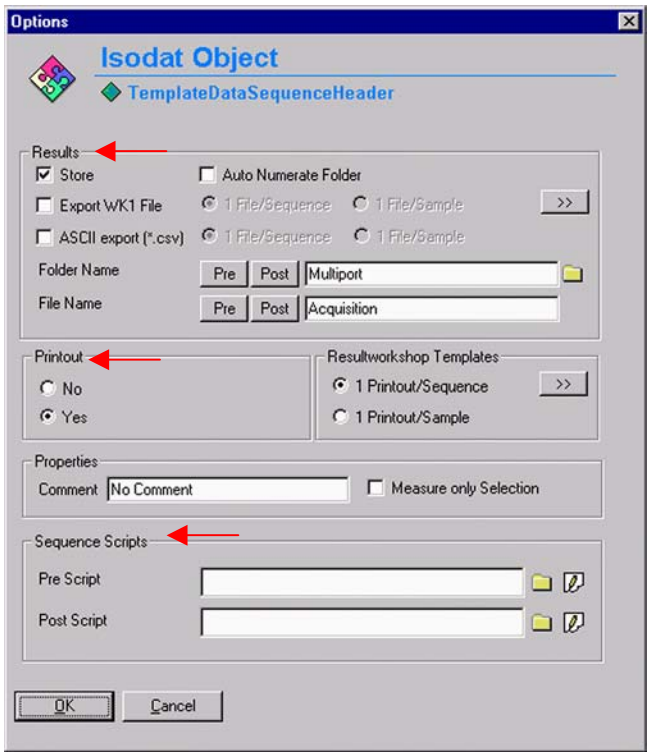
- In the "Method" column, the Method chosen in chapter 6.6.2 occurs as default ("Multiport + Micro-volume.met").



Normally, the Sequence List needs not to be edited further. It is possible, however, e.g. to select another Method from the "Method" column or to modify the "Multiport Inlet" column. For details, see chapter 6.3.3.

- Press the ***Start*** button.

- Define parameters for:
  - Results Export
  - Printout
  - Sequence Scripts



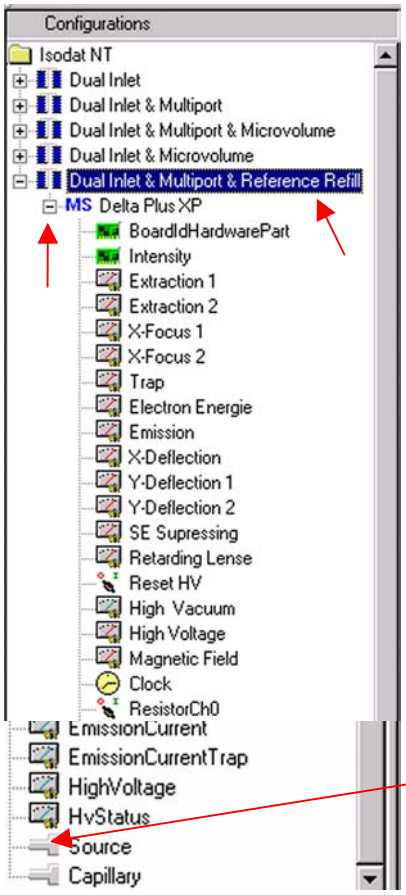
- Press ***OK*** to start Sequence Acquisition.

6.7 DUAL INLET MEASUREMENT INCLUDING MULTIPORT AND REFERENCE REFILL

6.7.1 DEFINING A CONFIGURATION

➢ Before operating, a ***Configuration*** containing the Dual Inlet System, Multi-port and Reference Refill must be created in the ***Configurator*** as follows.

➢ Add a new Configuration using the ***Add Configuration*** button.

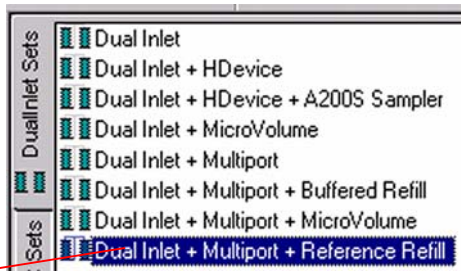


➢ Give it a significant name, e.g. ***"Dual Inlet & Multiport & Reference Refill"***.

➢ Open the tree structure by a click on ⊞ at ⊞ MS Delta Plus NE

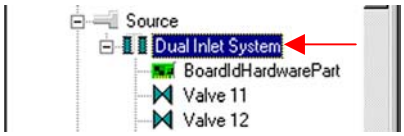
➢ On the right pane, select

Dual Inlet + Multiport + Reference Refill.

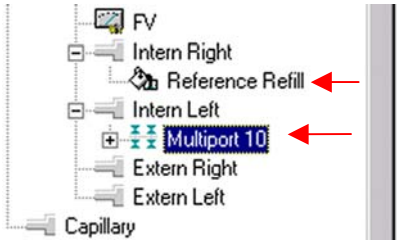


➢ Drag it to the Source port ⊣ of the new Configuration.

A ⊞ sign appears:



- The ***Dual Inlet*** device has been attached to the ***Source***.



- The ***Reference Refill*** device has been attached to the ***Intern Right*** Port.

  It is always connected to the ***right*** side (i.e. the Standard side) - internal or external.

- The ***Multiport*** has been attached to the ***Intern Left*** Port.

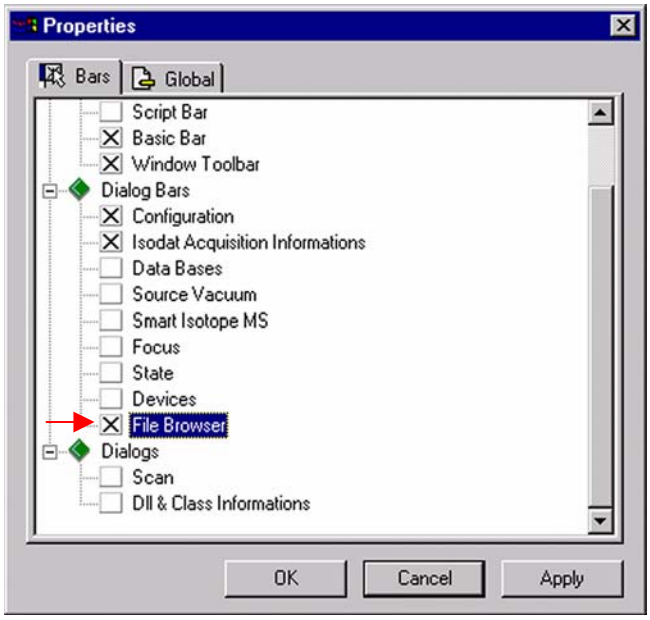
- Close the Configurator window.

  All settings will be saved automatically.

6.7.2 USING A PREDEFINED METHOD

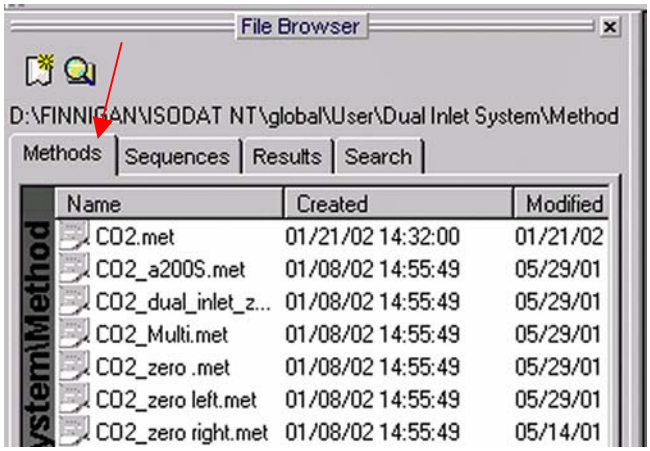
- If the File Browser cannot be seen, press the ***Options*** button.

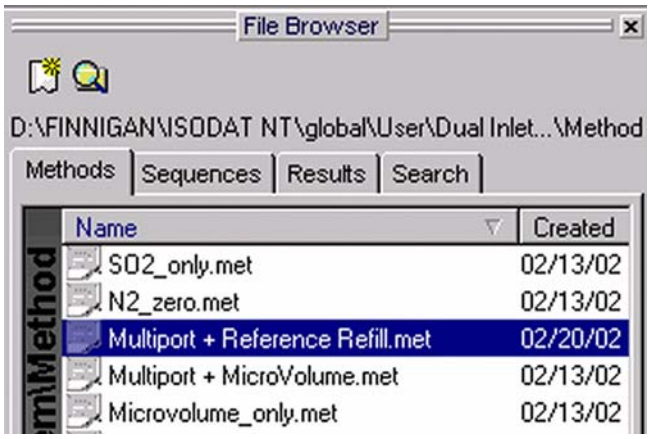
- Activate the ***File Browser*** check box.



- Click ***OK***.

- On the File Browser, select the ***Methods tab*** (default).





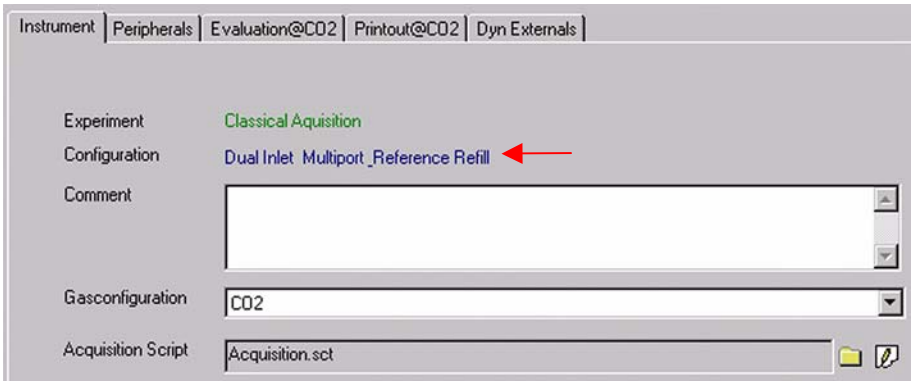
- From the predefined Methods choose a suitable Method (e.g. ***"Multiport + Reference Refill.met"***) by double-click.

- If the Standards in the Method to be opened are older than the actual ones in the Standard Database, or if the user has changed the Method, the warning beside appears.

- Press ***OK***. Thus, the Method will be corrected: the actual values of the Standard Database (e.g. the laboratory Standard has changed or been replaced by another Standard) are transferred to the Method.

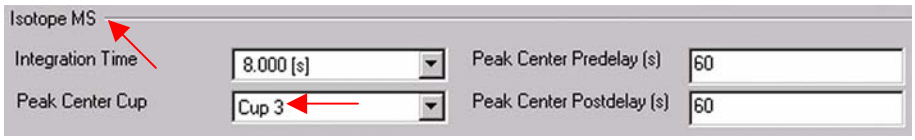
Instrument tab

The ***Instrument tab*** of the Method "Multiport + Reference Refill.met" appears.

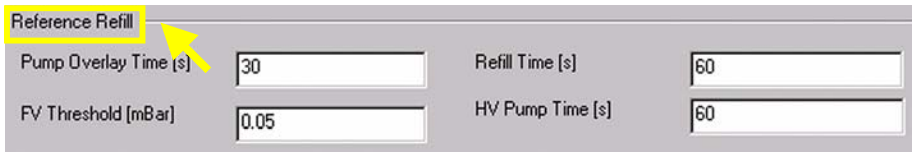


- As ***Gasconfiguration*** select "CO_2".
- The ***Main Script*** controls the acquisition cycle.

NOTE: ***It should only be edited by users trained on script editing.***



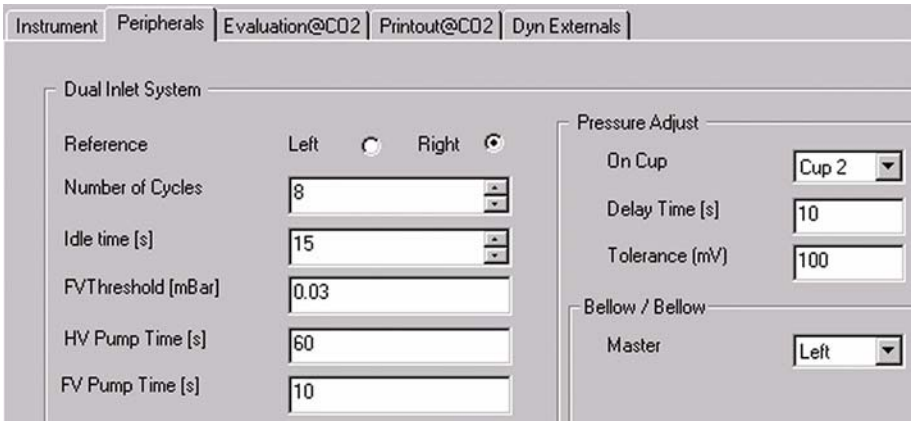
- ***Integration time:*** The time needed to measure each individual ion intensity of the masses 44, 45 and 46.
- Select the ***Peak Center Cup*** (e.g. Cup 3).
- ***Peak Center Pre Delay:*** Waiting time between activation of reference gas and start of peak center cycle (e.g. 60 s).
- ***Peak Center Post Delay:*** Waiting time between end of peak center cycle and start of data acquisition.



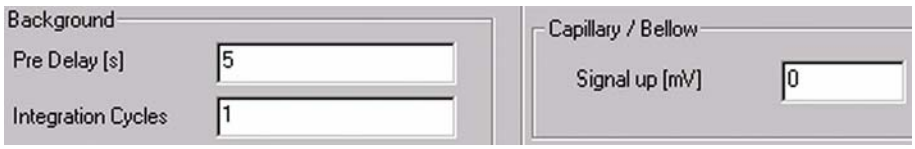
NOTE: ***The above information is specific for Reference Refill applications. The values depend - among others - on the diameter of the Reference Refill capillary.***

- ***Pump Overlay Time***: Capillary pump out time of Reference Refill tank.
- ***FV Threshold***: Minimum pressure of standard bellow including the valves and tubes evacuated using fore vacuum pump before continuing pumping using turbo molecular pump.
- ***Refill Time***: Gas flow time from Reference Refill tank into inlet port of standard bellow.
- ***HV Pump Time***: Pumping time of bellow including the valves and tubes using turbo molecular pump.

Peripherals tab



For details, see chapter 6.3.2.

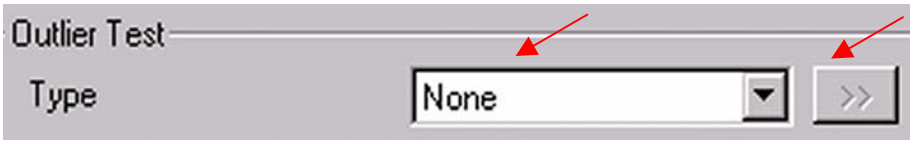


For details, see chapter 6.3.2.

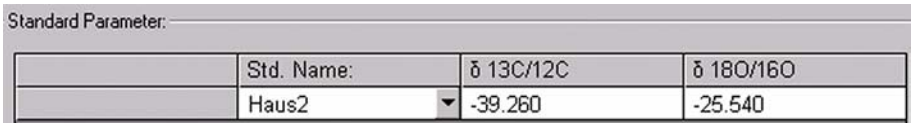
For details, see chapter 6.3.2.

Evaluation tab

- Select the ***Ion Correction Type*** (e.g. “CO2 Santrock et al.”).

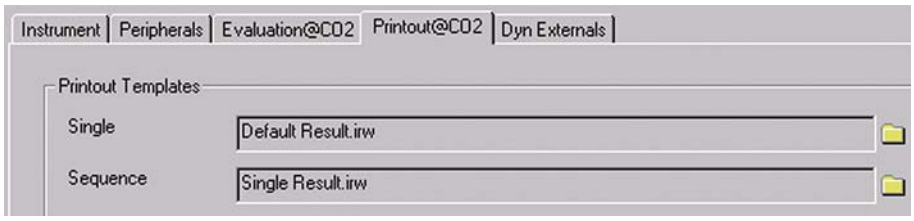


- As ***Outlier test*** select “None” or “Sigma”. In case of “Sigma”, specify the k-fold of the standard deviation using >>. For details, see chapter 6.3.2.

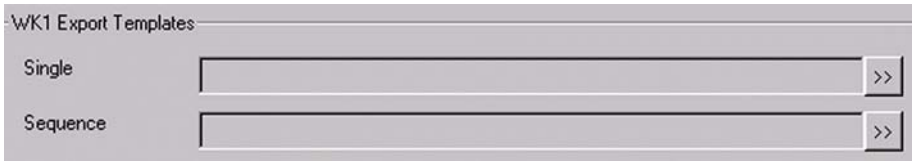


- At ***Std. Name***, choose a suitable Standard.

Printout tab



For details, see chapter 6.3.2.



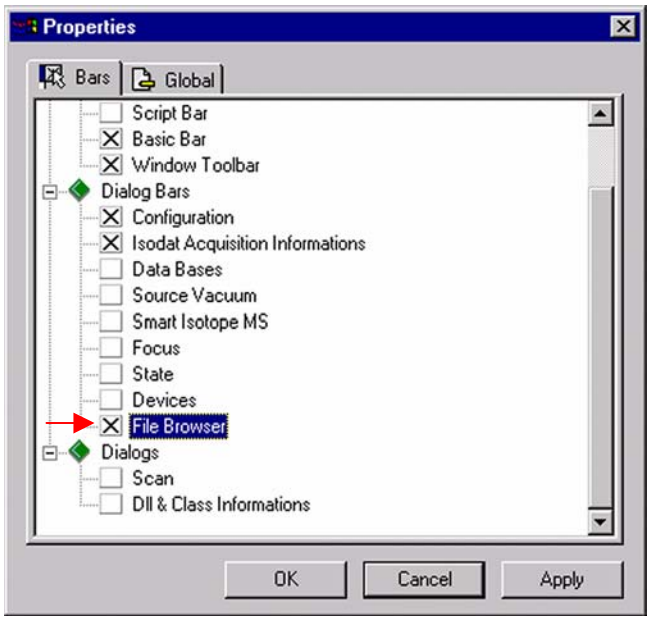
For details, see chapter 6.3.2.

The ***Method*** for measurements using Dual Inlet in combination with a Multiport and a Reference Refill has now been established. As next step, either use a ***predefined Sequence*** (refer to chapter 6.7.3) or create a ***new Sequence*** (refer to chapter 6.1.5).

6.7.3 USING A PREDEFINED SEQUENCE

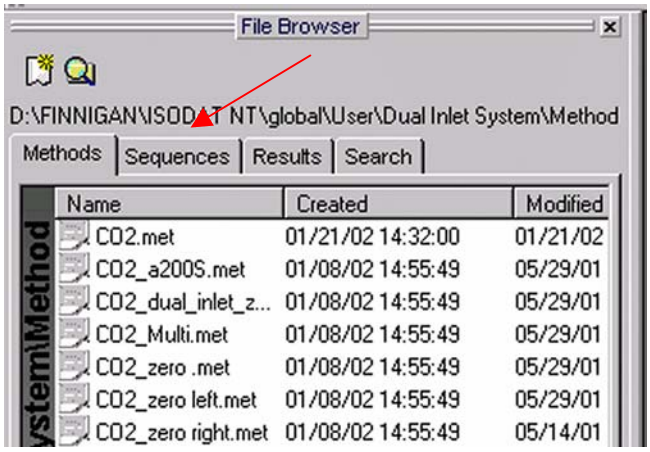
- If the File Browser cannot be seen, press the ***Options*** button.

- Activate the ***File Browser*** check box.

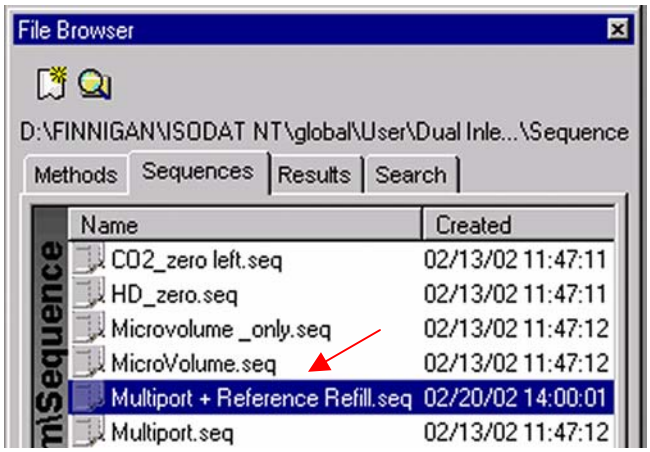


- Click ***OK***.

- On the File Browser, select the ***Sequences tab*** (default: Methods tab).



- From the predefined Sequences choose ***"Multiport + Reference Refill.seq"*** by double-click.



Line				Multiport Inlet		Identifier 1	Method
1	✓	✓	✓	Sample 1	✓	Lab. gas	Multiport + Reference Refill.met
2	✓	✓		Sample 2		Lab. gas	Multiport + Reference Refill.met
3	✓	✓		Sample 3		Lab. gas	Multiport + Reference Refill.met
4	✓	✓		Sample 4		Lab. gas	Multiport + Reference Refill.met
5	✓	✓		Sample 5		Lab. gas	Multiport + Reference Refill.met
6	✓	✓		Sample 6		Lab. gas	Multiport + Reference Refill.met
7	✓	✓		Sample 7		Lab. gas	Multiport + Reference Refill.met
8	✓	✓		Sample 8		Lab. gas	Multiport + Reference Refill.met
9	✓	✓		Sample 9		Lab. gas	Multiport + Reference Refill.met
10	✓	✓		Sample 10		Lab. gas	Multiport + Reference Refill.met

- In the ***Method*** column, the Method chosen in chapter 6.7.2 occurs as default ("Multiport + Reference Refill.met").

- Normally, the Sequence List needs not to be edited further. It is possible, however, e.g. to select another Method from the "Method" column or to modify the "Multiport Inlet" column.

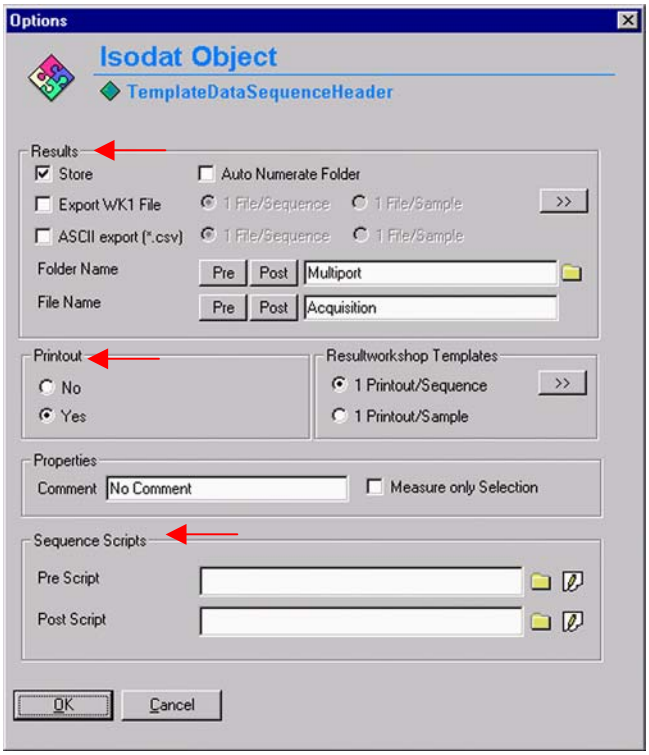
- The ***Multiport Inlet*** column defines the Multiport Inlet Port from which a particular sample is taken (e.g. Sample 2 enters via Multiport Inlet Port 2).
  If enough sample is available, it is possible to measure repeatedly out of the same port.

- If a ***Reference Refill*** is to be performed, the column must be activated by ✓ for the particular sample (e.g. for Sample 1).

- Press the ***Start*** button.

- Define parameters for:
  - Results Export
  - Printout
  - Sequence Scripts

- Press ***OK*** to start Sequence Acquisition.



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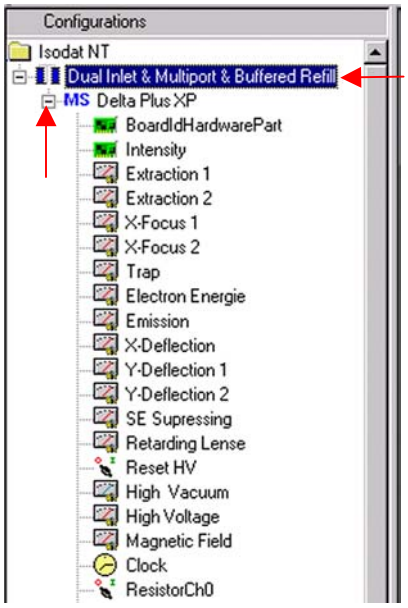
6.8 DUAL INLET MEASUREMENT INCLUDING MULTIPORT AND BUFFERED REFILL

6.8.1 DEFINING A CONFIGURATION

➢ Before operating, a ***Configuration*** containing the Dual Inlet System, Multiport and Buffered Refill must be created in the ***Configurator*** as follows.

➢ Add a new Configuration using the ***Add Configuration*** button.

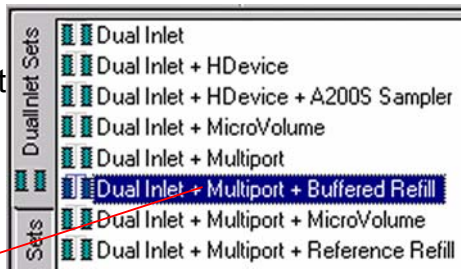


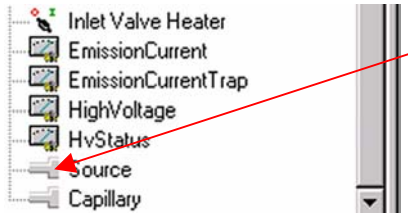
➢ Give it a significant name, e.g. ***"Dual Inlet & Multiport & Buffered Refill"***.

➢ Open the tree structure by a click on ⊞ at ⊞ MS Delta Plus NE

➢ On the right pane, select

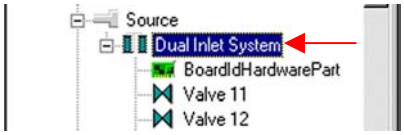
Dual Inlet + Multiport + Buffered Refill.



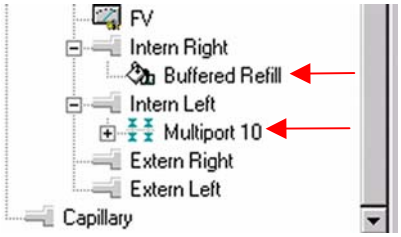


➢ Drag it to the Source port of the new Configuration.

A ⊞ sign appears:



- The ***Dual Inlet*** device has been attached to the ***Source***.

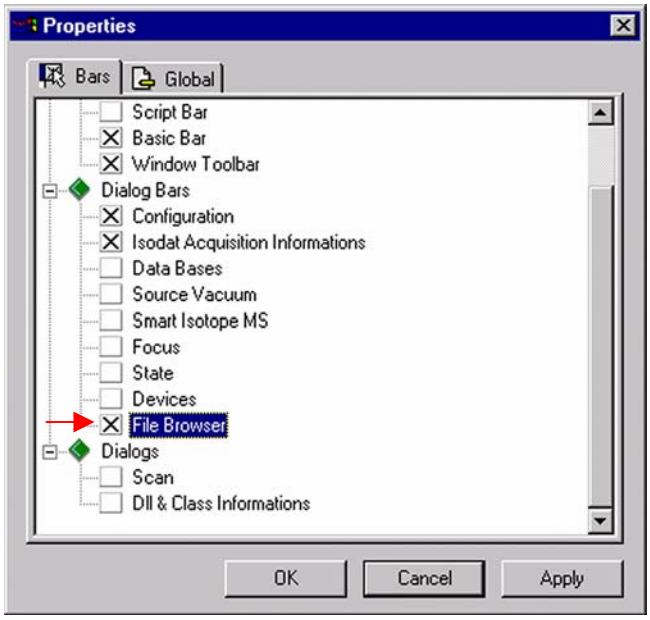


- The ***Buffered Refill*** device has been attached to the ***Intern Right*** Port.
- The ***Multiport*** device has been attached to the ***Intern Left*** Port.
- Close the Configurator window.
  All settings will be saved automatically.

6.8.2 USING A PREDEFINED METHOD

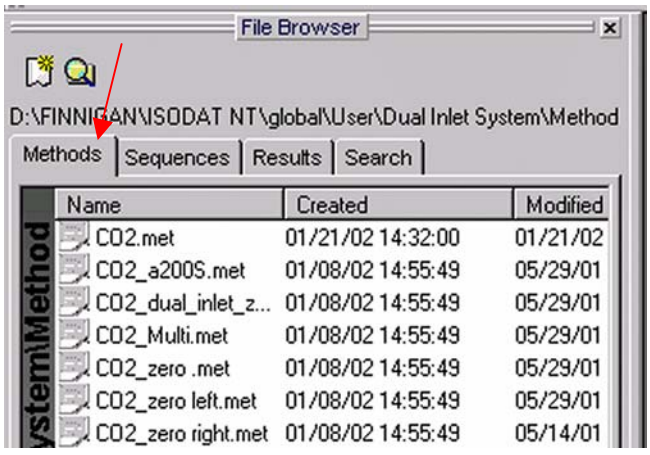
- If the File Browser cannot be seen, press the ***Options*** button.

- Activate the ***File Browser*** check box.

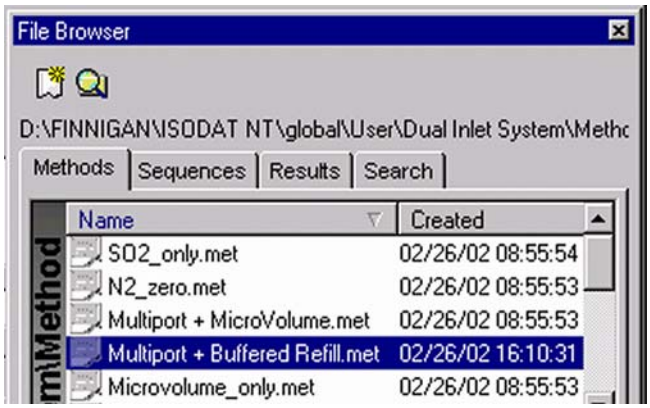


- Click ***OK***.

- On the File Browser, select the ***Methods tab*** (default).



- From the predefined Methods choose a suitable Method (e.g. ***"Multiport + Buffered Refill.met"***) by double-click.

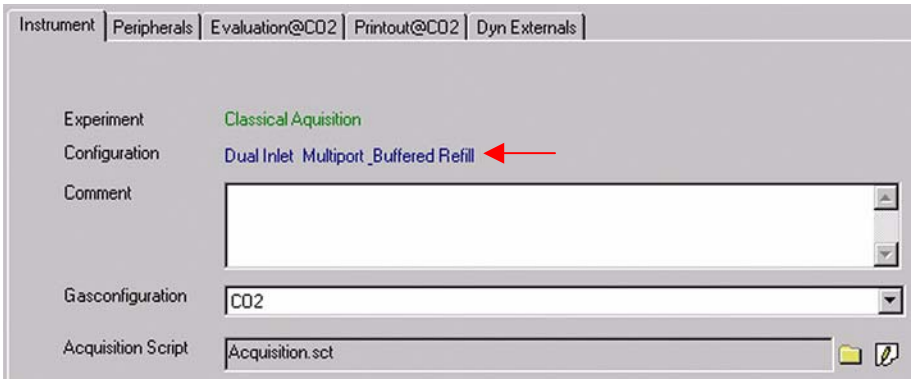


- If the Standards in the Method to be opened are older than the actual ones in the Standard Database, or if the user has changed the Method, the warning beside appears.

- Press ***OK***. Thus, the Method will be corrected: the actual values of the Standard Database (e.g. the laboratory Standard has changed or been replaced by another Standard) are transferred to the Method.

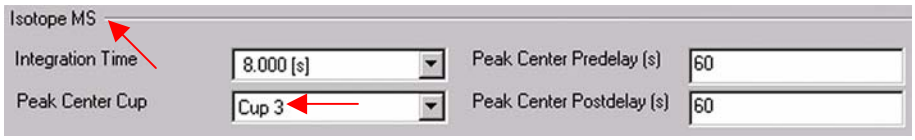
Instrument tab

The ***Instrument tab*** of the Method "Multiport + Buffered Refill.met" appears.

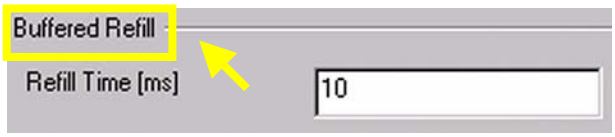


- As ***Gasconfiguration*** select "CO_2".
- The ***Main Script*** controls the acquisition cycle.

NOTE: ***It should only be edited by users trained on script editing.***



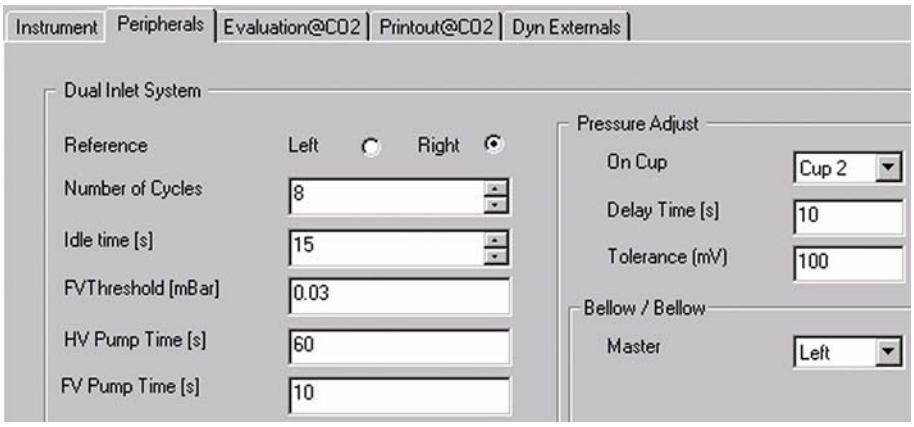
- ***Integration time***: The time needed to measure each individual ion intensity of the masses 44, 45 and 46 (e.g. 8 s).
- ***Peak Center Cup:*** Select the Peak Center Cup (e.g. Cup 3).
- ***Peak Center Pre Delay:*** Waiting time between activation of reference gas and start of peak center cycle (e.g. 60 s).
- ***Peak Center Post Delay:*** Waiting time between end of peak center cycle and start of data acquisition (e.g. 60 s).



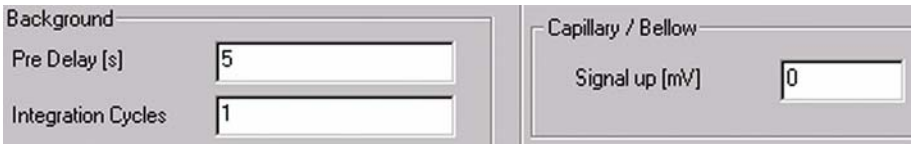
NOTE: ***The above information is specific for Buffered Refill applications.***

- ***Refill Time***: Gas flow time from Buffered Refill tank into inlet port of standard bellow (e.g. 10 ms).

Peripherals tab



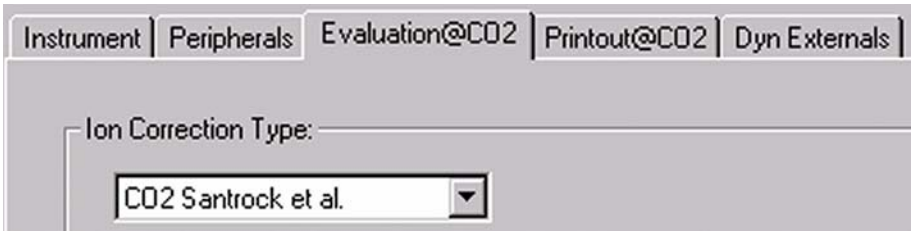
For details, see chapter 6.3.2.



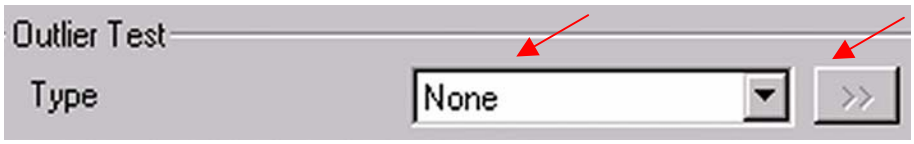
For details, see chapter 6.3.2.

For details, see chapter 6.3.2.

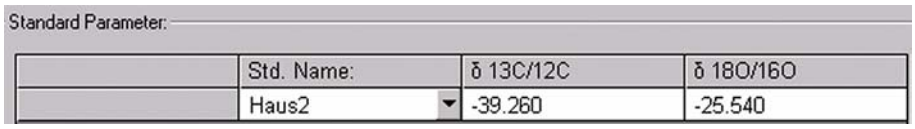
Evaluation tab



- Select the ***Ion Correction Type*** (e.g. “CO2 Santrock et al.”).

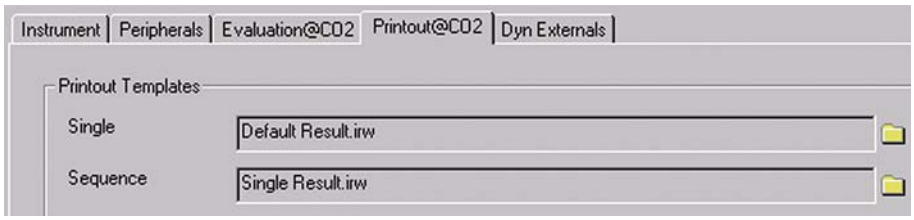


- As ***Outlier test*** select “None” or “Sigma”. In case of “Sigma”, specify the k-fold of the standard deviation using >>.

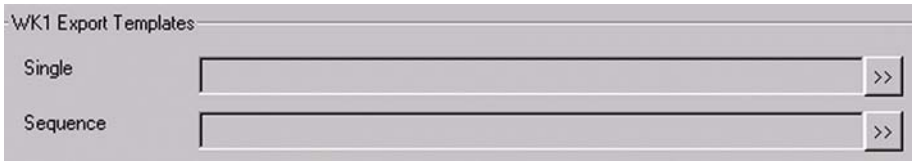


- At ***Std. Name***, choose a suitable Standard.

Printout tab



For details, see chapter 6.3.2.



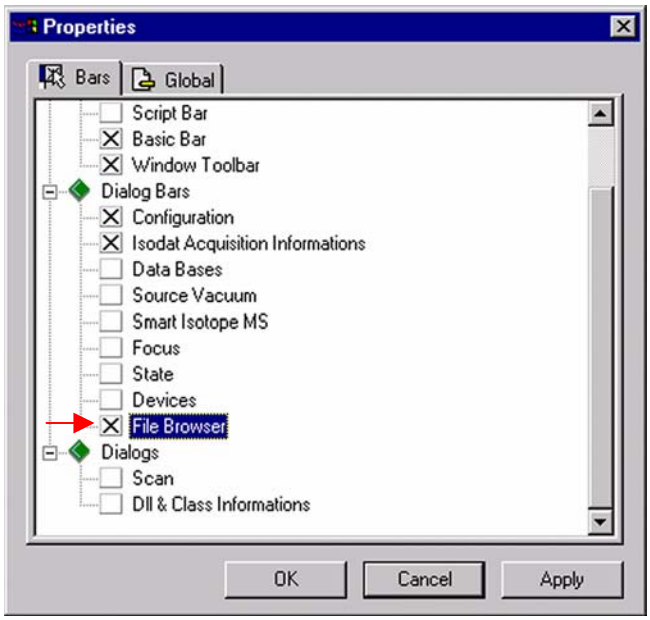
For details, see chapter 6.3.2.

The ***Method*** for measurements using Dual Inlet in combination with a Multiport and a Buffered Refill has now been established. As next step, either use a ***predefined Sequence*** (refer to chapter 6.8.3) or create a ***new Sequence*** (refer to chapter 6.1.5).

6.8.3 USING A PREDEFINED SEQUENCE

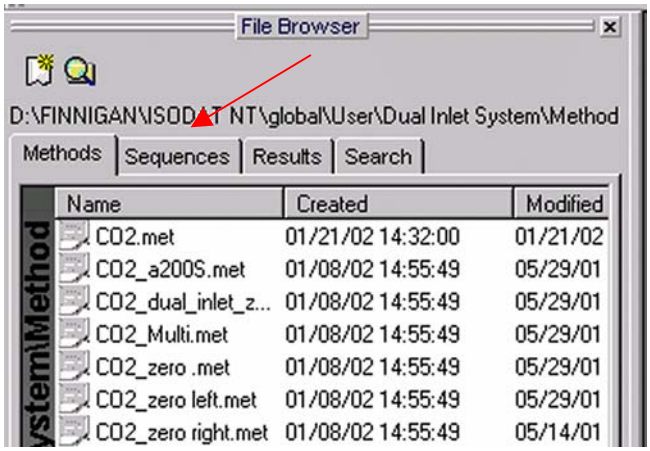
- If the File Browser cannot be seen, press the ***Options*** button.

- Activate the ***File Browser*** check box.

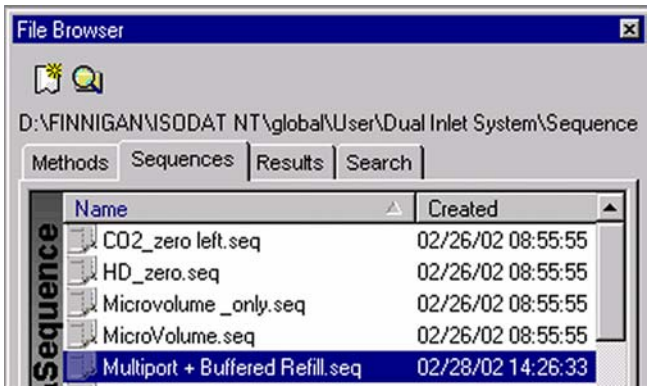


- Click ***OK***.

- On the File Browser, select the ***Sequences tab*** (default: Methods tab).



- From the predefined Sequences choose ***Multiport + Buffered Refill.seq*** by double-click.



Line	Multiport Inlet	Identifier 1	Method
1	Sample 1	Lab. Gas	Multiport + Buffered Refill.met
2	Sample 2	Lab. Gas	Multiport + Buffered Refill.met
3	Sample 3	Lab. Gas	Multiport + Buffered Refill.met
4	Sample 4	Lab. Gas	Multiport + Buffered Refill.met
5	Sample 5	Lab. Gas	Multiport + Buffered Refill.met
6	Sample 6	Lab. Gas	Multiport + Buffered Refill.met
7	Sample 7	Lab. Gas	Multiport + Buffered Refill.met
8	Sample 8	Lab. Gas	Multiport + Buffered Refill.met
9	Sample 9	Lab. Gas	Multiport + Buffered Refill.met
10	Sample 10	Lab. Gas	Multiport + Buffered Refill.met

- In the ***Method*** column, the Method chosen in chapter 6.8.2 occurs as default (“Multiport + Buffered Refill.met”).

- Normally, the Sequence List needs not to be edited further. It is possible, however, e.g. to select another Method from the “Method” column or to modify the “Multiport Inlet” column.

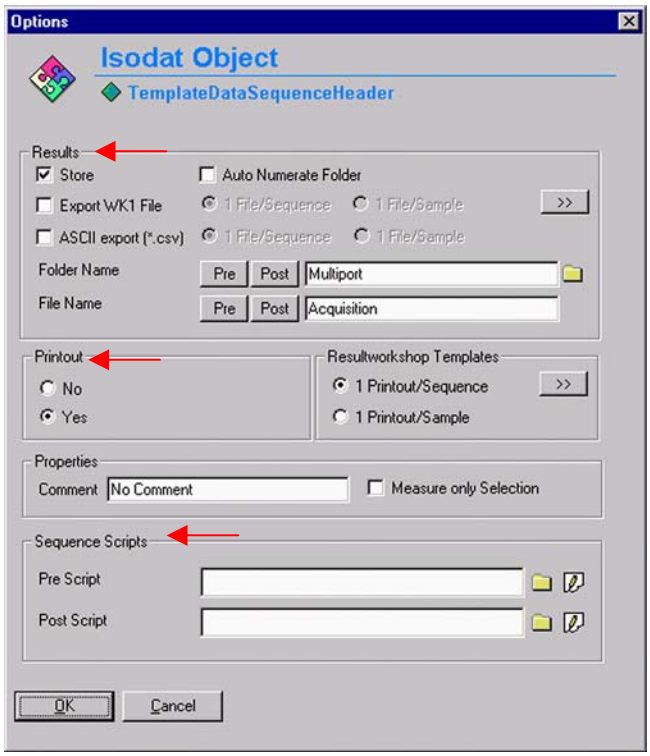
- The ***Multiport Inlet*** column defines the Multiport Inlet Port from which a particular sample is taken (e.g. Sample 2 enters via Multiport Inlet Port 2).
  If enough sample is available, it is possible to measure repeatedly out of the same port.

- If a ***Buffered Refill*** is to be performed, the column must be activated by ✓ for the particular sample (e.g. for Sample 1).

- Press the ***Start*** button.

- Define parameters for:
  - Results Export
  - Printout
  - Sequence Scripts

- Press ***OK*** to start Sequence Acquisition.



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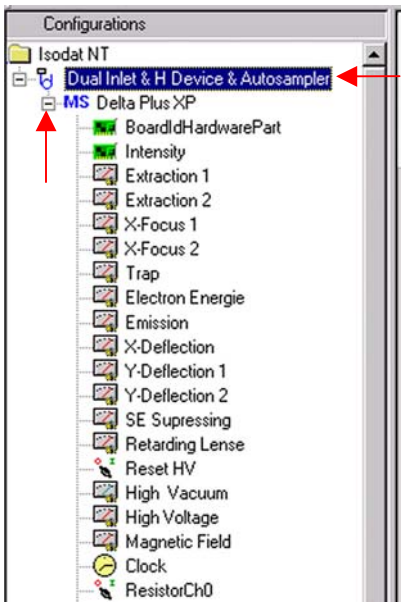
6.9 DUAL INLET MEASUREMENT INCLUDING H-DEVICE AND AUTOSAMPLER A200S

6.9.1 DEFINING A CONFIGURATION

- Before operating, a ***Configuration*** containing the Dual Inlet System, H-Device and Autosampler must be created in the ***Configurator*** as follows.

- Add a new Configuration using the ***Add Configuration*** button.

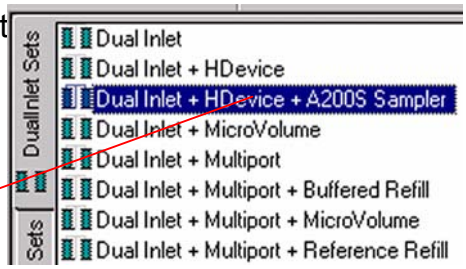


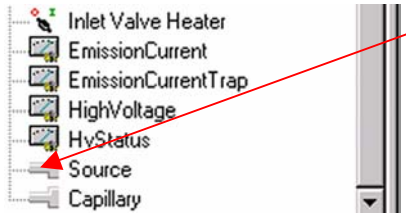
- Give it a significant name, e.g. ***"Dual Inlet & HDevice & Autosampler"***.

- Open the tree structure by a click on ⊞ at ⊞ MS Delta Plus NE

- On the right pane, select

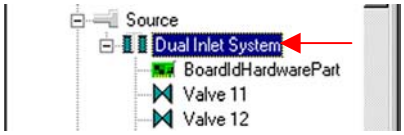
  Dual Inlet + HDevice + A200S Sampler.



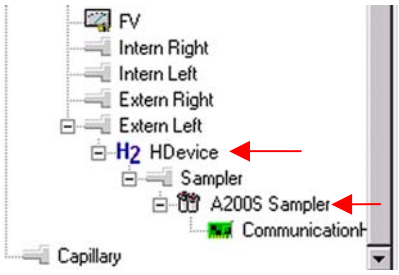


- Drag it to the Source port ⊟ of the new Configuration.

  A ⊞ sign appears:



➢ The ***Dual Inlet*** device has been attached to the ***Source***.



➢ The ***H-Device*** has been attached to the ***Extern Left*** Port.

➢ The ***A200S Autosampler*** has been attached to the ***Sampler*** Port.

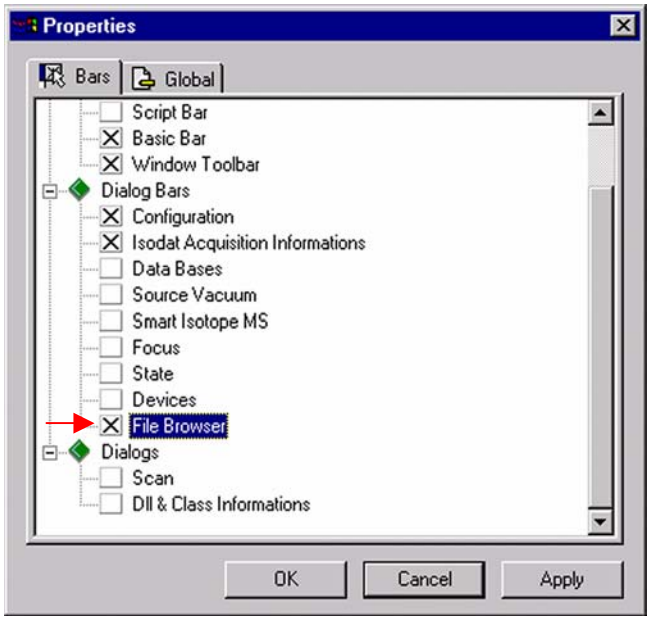
➢ Close the Configurator window.

All settings will be saved automatically.

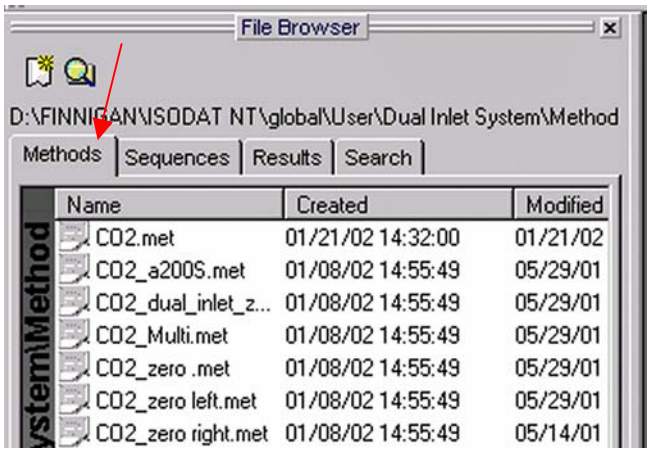
6.9.2 USING A PREDEFINED METHOD

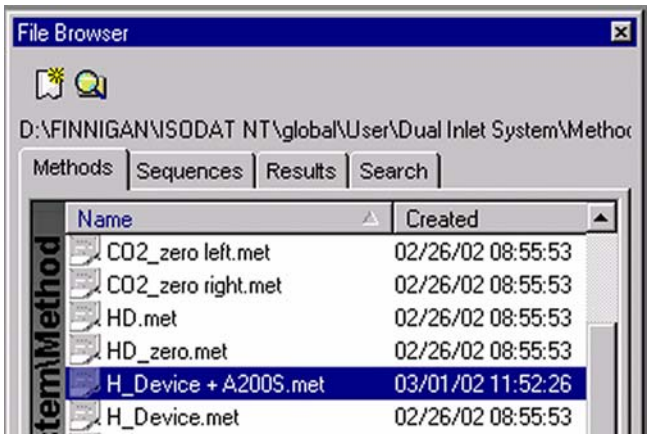
- If the File Browser cannot be seen, press the ***Options*** button.

- Activate the ***File Browser*** check box.
- Click ***OK***.



- On the File Browser, select the ***Methods tab*** (default).





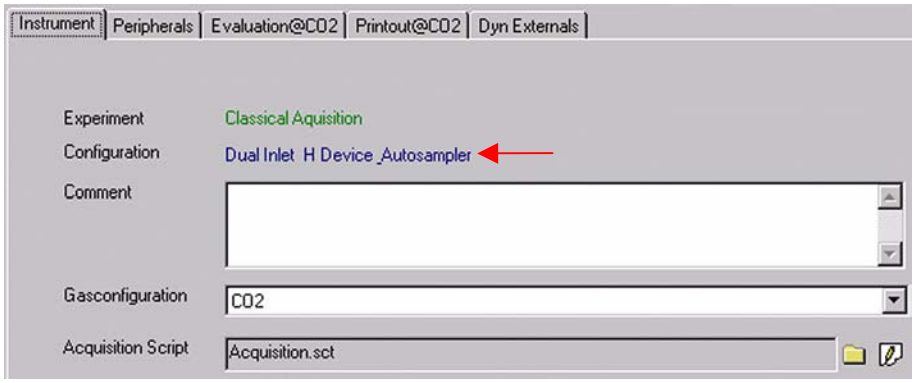
- From the predefined Methods choose a suitable Method (e.g. ***"H_Device + A200S.met"***) by double-click.

- If the Standards in the Method to be opened are older than the actual ones in the Standard Database, or if the user has changed the Method, the warning beside appears.

- Press ***OK***. Thus, the Method will be corrected: the actual values of the Standard Database (e.g. the laboratory Standard has changed or been replaced by another Standard) are transferred to the Method.

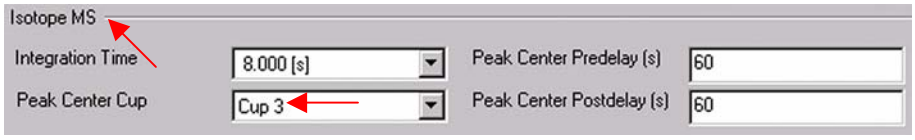
Instrument tab

The ***Instrument tab*** of the Method "H_Device + A200S.met" appears.



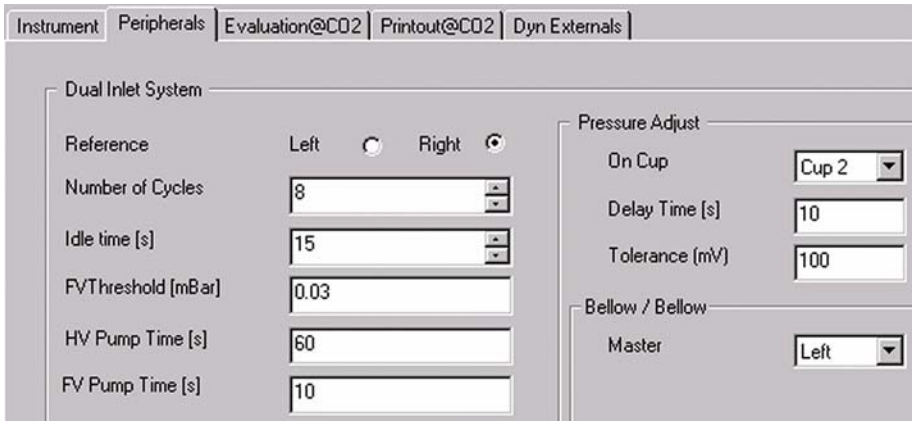
- As ***Gasconfiguration*** select "CO_2".
- The ***Main Script*** controls the acquisition cycle.

NOTE: ***It should only be edited by users trained on script editing.***

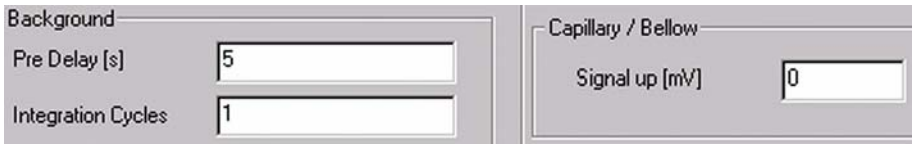


- ***Integration time***: The time needed to measure each individual ion intensity of the masses 44, 45 and 46 (e.g. 8 s).
- ***Peak Center Cup***: Select the Cup for peak center (e.g. Cup 3).
- ***Peak Center Pre Delay***: Waiting time between activation of reference gas and start of peak center cycle (e.g. 60 s).
- ***Peak Center Post Delay***: Waiting time between end of peak center cycle and start of data acquisition (e.g. 60 s).

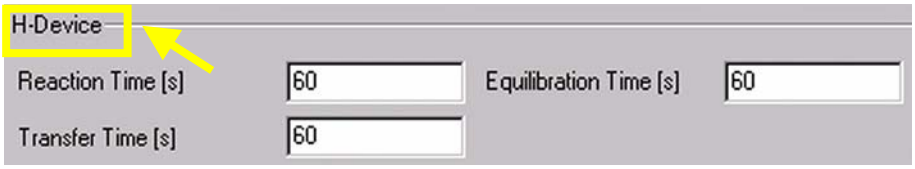
Peripherals tab



For details see chapter 6.3.2.



For details, see chapter 6.3.2.



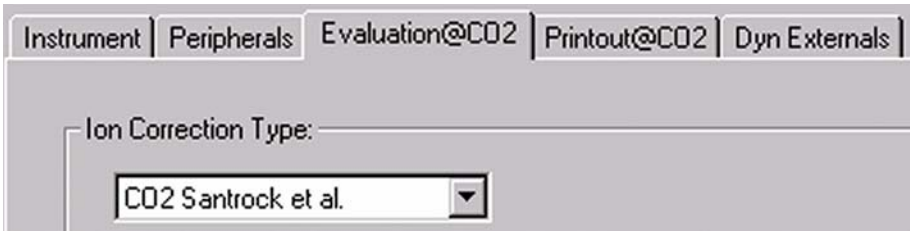
NOTE: ***The above information is specific for H-Device applications.***

- ***Reaction Time***: Time for the injected substance to react with chromium (e.g. 60 s). Reaction time differs between substances. Water only needs 20 to 60 s to react, whereas organic compounds need longer times, which must be determined empirically.

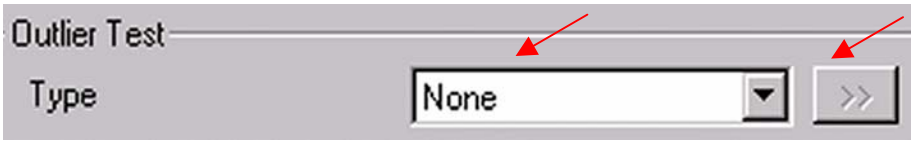
NOTE: ***Valve 11 must be closed!***

- ***Transfer Time***: Time for the gas to expand from the expansion volume into the bellow (e.g. 60 s).
- ***Equilibration Time***: Valve 11 is opened and the gas mixture is allowed to expand into the intermediate or expansion volume (connecting metal hose plus valve body). An equilibration time of 30 s is recommended for 800 ºC to 850 ºC. For other temperatures, this needs to be tested empirically.

Evaluation tab

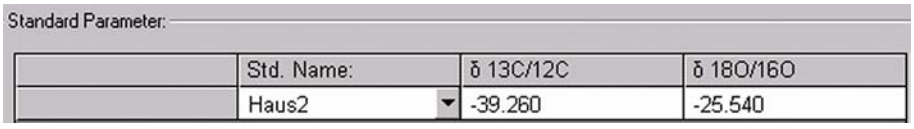


- Select the ***Ion Correction Type*** (e.g. “CO2 Santrock et al.”).



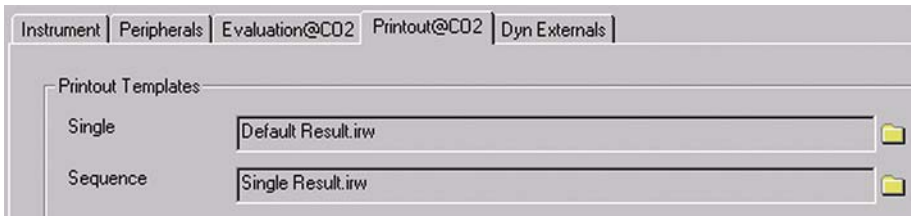
- As ***Outlier test*** select “None” or “Sigma”. In case of “Sigma”, specify the k-fold of the standard deviation using >>.

For details, see chapter 6.3.2.

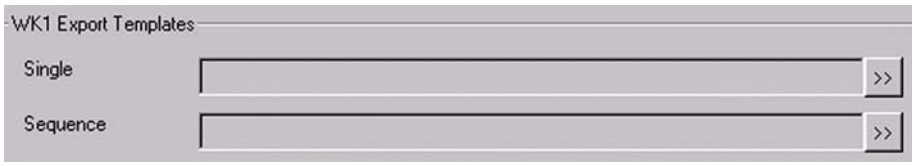


- At ***Std. Name***, choose a suitable Standard.

Printout tab



For details, see chapter 6.3.2.



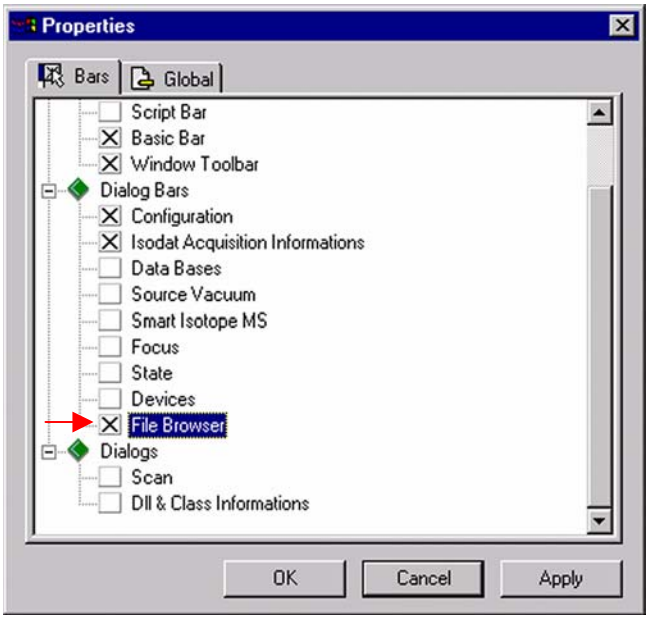
For details, see chapter 6.3.2.

The ***Method*** for measurements using Dual Inlet in combination with a H-Device and an A200S Autosampler has now been established. As next step, either use a ***predefined Sequence*** (refer to chapter 6.9.3) or create a ***new Sequence*** (refer to chapter 6.1.5).

6.9.3 USING A PREDEFINED SEQUENCE

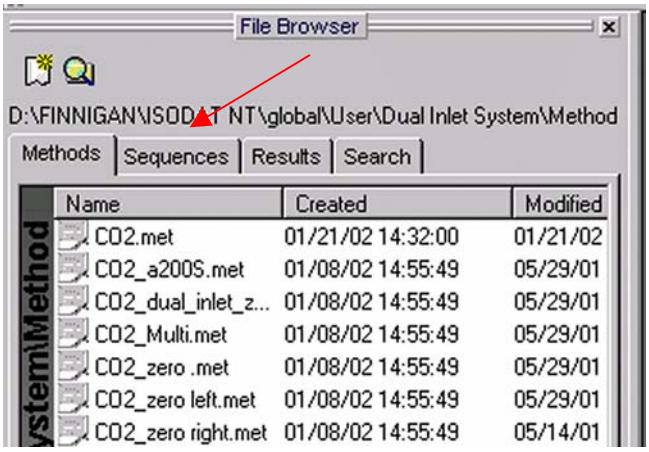
- If the File Browser cannot be seen, press the ***Options*** button.

- Activate the ***File Browser*** check box.

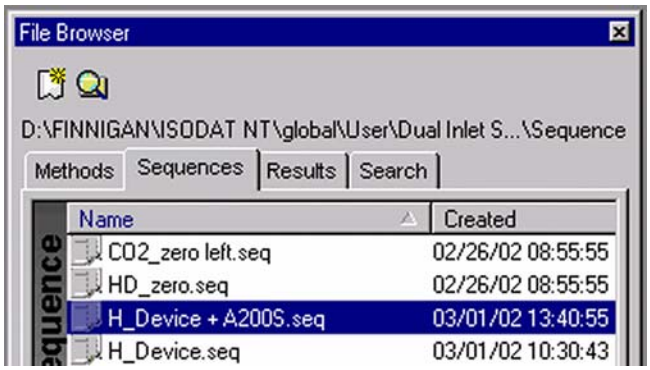


- Click ***OK***.

- On the File Browser, select the ***Sequences tab*** (default: Methods tab).



- From the predefined Sequences choose a suitable one (e.g. ***H_Device + A200S.seq***) by double-click.



Line				AS Sample	AS Method	Identifier 1	Method
1	✓	✓	✓	1	1	Lab. Gas	H_Device + A200S.met
2	✓	✓		2	2	Lab. Gas	H_Device + A200S.met
3	✓	✓		3	3	Lab. Gas	H_Device + A200S.met
4	✓	✓		4	4	Lab. Gas	H_Device + A200S.met
5	✓	✓		5	5	Lab. Gas	H_Device + A200S.met
6	✓	✓		6	6	Lab. Gas	H_Device + A200S.met
7	✓	✓		7	7	Lab. Gas	H_Device + A200S.met
8	✓	✓		8	8	Lab. Gas	H_Device + A200S.met
9	✓	✓		9	9	Lab. Gas	H_Device + A200S.met
10	✓	✓		10	10	Lab. Gas	H_Device + A200S.met

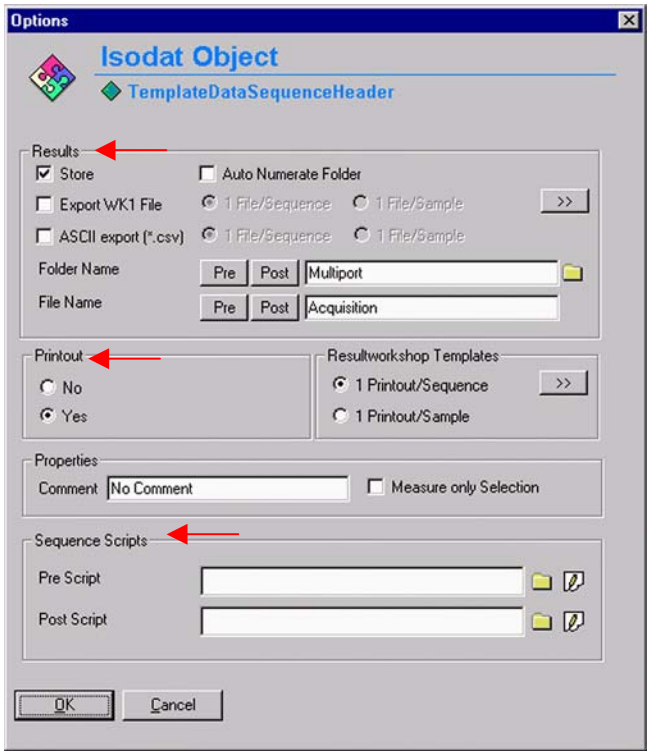
- Normally, the Sequence List needs not to be edited further. It is possible, however, e.g. to select another Method from the “Method” pulldown menu.

- ***AS Sample***: Type in the sample position in the Autosampler tray.
- ***AS Method***: Select Autosampler Method.
- ***Identifier 1***: Edit text to identify the sample.
- ***Method***: Select IRMS Method. The Method chosen in chapter 6.9.2 occurs as default (“H_Device + A200S.met”).

➢ Press the ***Start*** button.

➢ Define parameters for:

- Results Export
- Printout
- Sequence Scripts



➢ Press ***OK*** to start Sequence Acquisition.

6.10 FUNDAMENTAL PROCEDURES BEFORE ANY MEASUREMENT

Before starting any acquisition make sure, the following procedure has been performed:

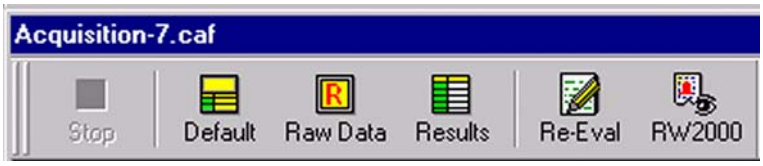
1. Electronic offset has been measured and saved.
2. All peripherals have been configured.
3. The Mass Calibration for all gases to be measured has been performed.
4. In case of a Dual Inlet application, the Bellow Calibration has been performed.
5. In case of hydrogen collectors, the H3 factor for Continuous Flow and Dual Inlet application has been determined.
6. In case of Elemental Analyzer application for C/N or H/O analysis, the Jump Calibration has been performed and saved.
7. In case of a Liquid Autosampler, the COM port and the other parameters are set.

6.11 HANDLING OF RESULTS

Toolbar Commands

After the measurement has been started by 

, a ***classical acquisition file***, i.e. ****.caf***, is generated (e.g. “Acquisition-7.caf”). The Acquisition window opens. It contains the toolbar shown below with the following Icons:



- A running Dual Inlet data acquisition can be stopped. 

- The data are shown ***both*** in a data grid and graphically (i.e. both in medium size). 

- The ***graphical*** representation of the raw data is shown in maximum size (i.e. without data grid). 

- The ***result grid*** is shown in maximum size (i.e. without graphical representation). 

- Some parameters of the Method can be changed (not the Method itself). Afterwards, the recorded data are reevaluated using the new parameter values. 

- Opens a Result Workshop Template containing the data (e.g. for preparing a printout). 

Sequence line

Line					Fill + EQ	Unit 1 Port	Unit 1 Bank	Identifier 1	Method
47	✓	✓			No	5	3	water	CO2_EQ.met

A particular line of the chosen Sequence is shown. It denotes the currently measured sample.

The tabs of the Acquisition window

The Acquisition window offers the following tabs for data interpretation:

Raw tab

Raw<CO2> | Evaluated<CO2> | Errors | Extended | Method

	Int.44 Sample [mV] 1	Int.45 Sample [mV] 1	Int.44 Standard [mV] 2	Int.45 Standard [mV] 2	rR 45CO2/44CO2 3	rDelta 45CO2/44CO2 [‰] vs. CO2_zero 4	rDelta 46CO2/44CO2 [‰] vs. CO2_zero 4	Delta 13C/12C [‰] vs. PDB 5	Delta 18O/16O [‰] vs. V-SMOW 5	AT% 13C [%] 6
Pre			5397.264	6239.374						
1	5364.022	6294.031	5393.776	6235.185	1.1733790	15.024	20.507	15.347	20.495	1.128
2	5359.753	6289.017	5390.523	6231.409	1.1733782	15.038	20.472	15.362	20.460	1.128
3	5355.706	6284.090	5387.564	6228.014	1.1733449	15.008	20.467	15.331	20.454	1.128
4	5352.059	6279.971	5384.796	6224.809	1.1733749	15.032	20.476	15.356	20.463	1.128
5	5348.574	6275.926	5382.165	6221.931	1.1733829	15.026	20.477	15.350	20.465	1.128
6	5345.251	6272.043	5379.417	6218.645	1.1733860	15.025	20.473	15.348	20.460	1.128
7	5341.927	6268.162	5376.827	6215.697	1.1733896	15.033	20.478	15.357	20.466	1.128
8	5338.647	6264.314	5374.151	6212.760	1.1733898	15.016	20.523	15.338	20.511	1.128

In case of ***Dual Inlet*** applications, ***intensity*** values are determined (while in case of ***Continuous Flow*** applications, ***peak areas*** are calculated).

In the leftmost column, the ***sample number*** is displayed (e.g. “Pre” and “1” to “8”; cf. the lines of the chosen Sequence). Besides, the raw data grid contains the following columns:

1. **Int Sample** — **Sample intensity** of a particular mass in mV (e.g. mass 44). Each sample mass has a column of its own.
2. **Int. Standard** — **Standard intensity** of a particular mass in mV (e.g. mass 44). Each standard mass has a column of its own.

3. **rR** — **Raw ratio** of two masses, i.e. ratio of the intensities of these two masses (e.g. rR 45/44).

4. **rDelta** — **Raw Delta** of two molecular masses (in ‰) vs. a secondary standard - i.e. vs. a user standard. E.g.: rDelta [45 CO2/44 CO2] vs. CO2_zero. These values are **molecule** deltas and are calculated from the raw ratios.

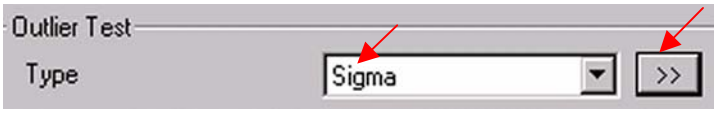
5. **Delta** — **Delta** value of two element isotopes (in ‰) vs. a primary standard. E.g.: Delta [13C/12C] vs. V-PDB or Delta [18O/16O] vs. V-SMOW. These values are **element** deltas and are calculated from the true element sample ratios and the true element standard ratios. The true element standard ratios are read from primary standards defined in the primary standard database.

6. **At %** — **Atomic percentage** value of an isotope, e.g.: At % [13C] or At % [18O]. It is calculated from the element delta and the corresponding absolute element ratio of the standard.

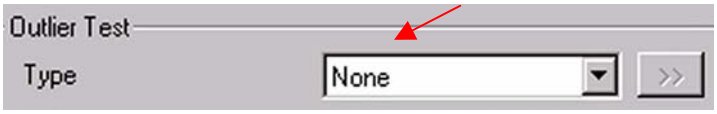
Kinds of data in the raw data grid

- A red value indicates that during integration the timeout criterion was reached. Red numbers only occur in case of intensities, i.e. in the columns ***Int. Sample*** and ***Int. Standard***.

- A black value on a pink background is an outlier relating to the particulars given at the “Evaluation” tab’s “Outlier Test”: 15.362



If ***Sigma*** was selected there and specified by >> , outliers may occur and are marked as shown above. They only occur in case of ***Delta*** values, e.g. Delta [13C/12C] vs. V-PDB



If ***None*** was selected there, no outliers are calculated at all. Therefore, no black values on a pink background will occur.

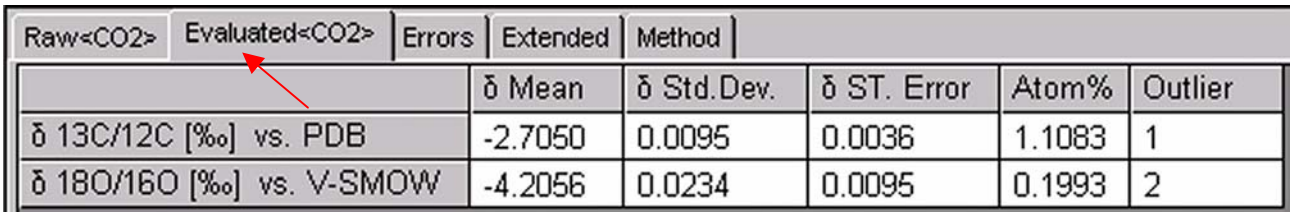
Suppose, that outliers have occurred (i.e. the case of ***Sigma***). If you then decide to reevaluate these data using a changed Method (yia the ***Method*** tab) selecting ***None*** at the “Evaluation” tab’s “Outlier Test”, no more outliers will be calculated. Thus, the values will turn into normal black ones:

15.362 → 15.362

- A black value on a green background is a Normal value falling into the k-fold of σ (i.e. no outlier). They only occur in case of ***Delta*** values, e.g. Delta [13C/12C] vs. V-PDB or Delta [18O/16O] vs. V-SMOW.

20.454

Evaluated tab



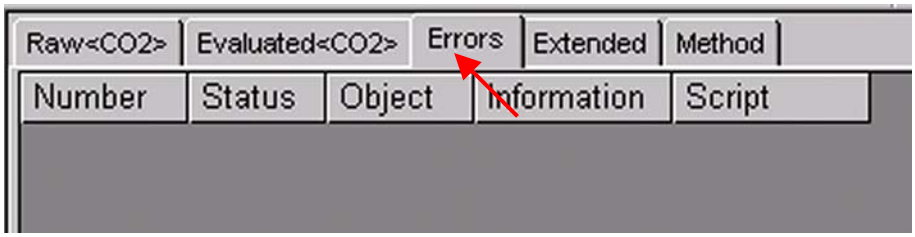
	δ Mean	δ Std.Dev.	δ ST. Error	Atom%	Outlier
δ 13C/12C [‰] vs. PDB	-2.7050	0.0095	0.0036	1.1083	1
δ 18O/16O [‰] vs. V-SMOW	-4.2056	0.0234	0.0095	0.1993	2

The ***Delta*** values (i.e. δ 13C/12C vs. V-PDB and δ 18O/16O vs. V-SMOW) marked pink or green in the raw data grid's columns **5** undergo a statistical analysis here:

- ***δ Mean*** Mean of the ***Delta*** values noted in column **5** (e.g. Mean of all Delta 18O/16O values in column **5**).
- ***δ Std. Dev.*** Standard deviation of the ***Delta*** values noted in column **5**. (e.g. Standard deviation of all Delta 18O/16O values in column **5**).
- ***δ St. Error*** Standard error of the ***Delta*** values noted in column **5** (e.g. Standard error of all Delta 18O/16O values in column **5**).
- ***Atom-%*** Atomic percentage value of the heavier isotope. It is calculated from the ***δ Mean*** value (e.g. Atom-% of ^{13}C is calculated from δ [13C/12C]).

- ***Outlier*** Number of outliers in column **5** entering into calculation of δ Mean, δ Std. Dev and δ St. Error (e.g. 2).

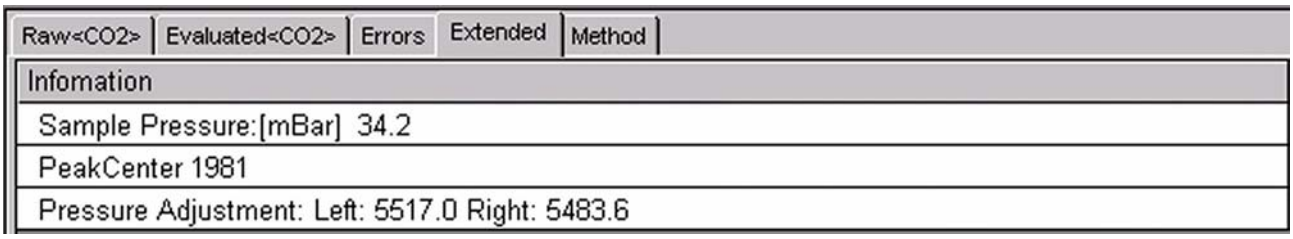
Errors tab



The "Errors" tab contains detailed information about errors that occurred during Data Acquisition:

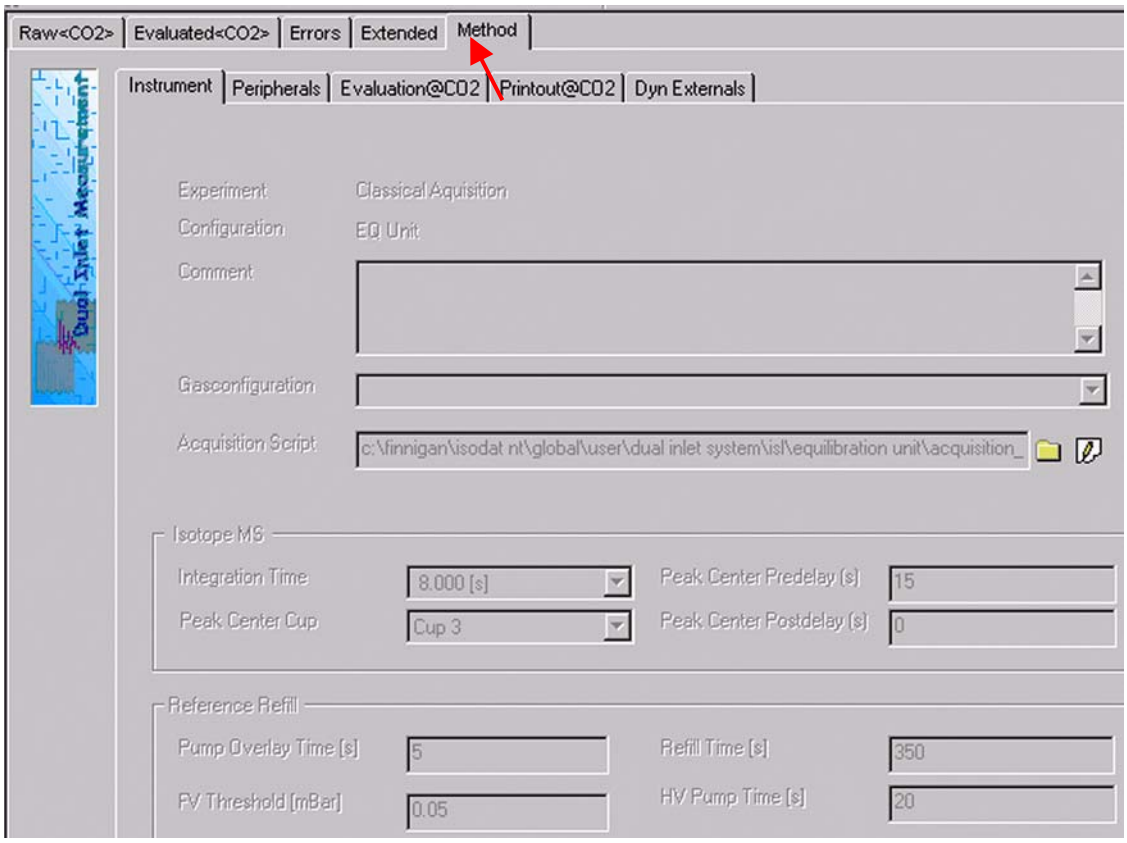
- ***Number*** The consecutive number of the error (i.e. 1; 2; 3…)
- ***Status*** The quality or gravidity of the error (e.g. fatal; warning; error)
- ***Object*** Object in the Script that caused the particular error
- ***Information*** Explanation concerning the error
- ***Script*** Script that caused the error

Extended tab



In the “Extended” tab, a measurement protocol is presented: detailed information is given about particular results during measurement progression (e.g. Peak Center results or Pressure Adjustment results). The kind of information depends on the selected ***Configuration***; e.g. different information in case of “Dual Inlet + Microvolume” compared to “Dual Inlet + H-Device”.

Method tab



For Data Reevaluation purposes, some parameters of the Method selected prior to measurement start can be modified (mainly in the “Peripherals” tab and the “Evaluation” tab; click the tabs in order to determine the changeable parameters and then type in the new corrected values). After the parameters of interest have been modified, reevaluation of the data can be started. Therefore, press the ***Re-Eval*** button.

6.12 EXCEL EXPORT OF RESULTS

6.12.1 PRINCIPLE OF EXCEL EXPORT

Principle: Measurement parameters and results are exportable as an Excel file. The Excel export can easily be tailored to your individual needs. Therefore, rules must be established to determine how to perform the export. These export rules are described in an export ***template*** (i.e. a ***wke*** file).

Various ***selectors***, which group Identifiers, are used to create an export template. For an export, more than one template can be selected. Multiple templates can be used simultaneously. Every single template (i.e. every wke file) finally leads to an export file of its own, its ***wk1*** file. Every wk1 file is created according to the rules of the wke file.

Advantages: Although measurement data are stored in one result file, the data concerning ***every single gas type*** can be exported separately to an export file of their own (e.g. multigas measurement).

Single ***parts*** of a measurement can be exported to an export file of their own (e.g. only the Method parameters).

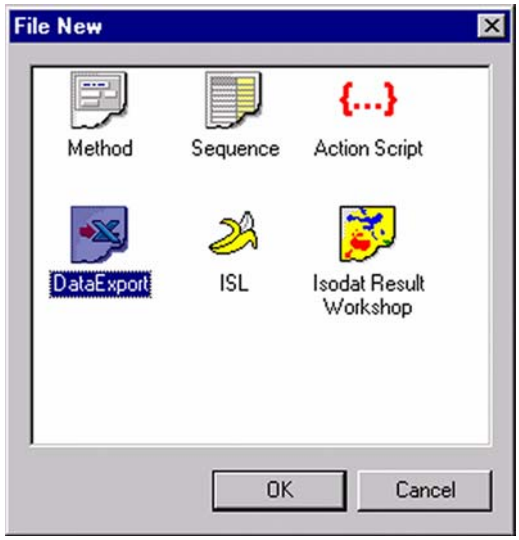
Some types of data can be specified further using ***query tabs***, where sophisticated export properties are defined (e.g. peak relevant data).

6.12.2 CREATING AN EXPORT TEMPLATE

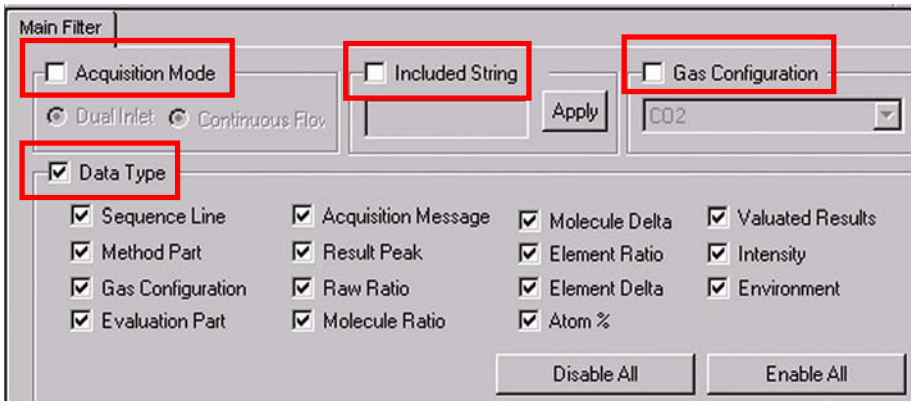
- On the Dual Inlet window's toolbar, press the ***New*** button.

- Click ***Data Export*** and confirm by ***OK***.

  Thus, a new export template can be edited.

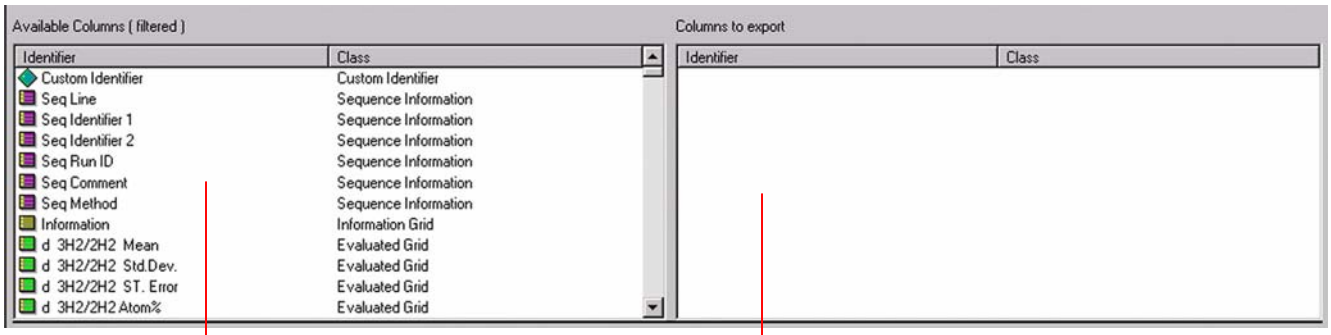


- Four selector types can be selected.



The four selector types for template creation

Selectors facilitate the Excel-Export: the list of exportable data can be shortened (see the Identifiers in the left pane). Without selectors, all exportable data are visible.



available pool of Identifers (columns)

Identifiers (columns) to be exported

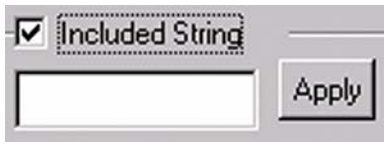
NOTE: ***Selectors are always combined by a conjunction (i.e. by an AND relation).***

NOTE: ***(De-)activation of Selectors can be changed during template creation. The export columns will not be affected.***

1 *Acquisition Mode*

If Acquisition Mode is activated, choose via click between parameters, which are only relevant for Dual Inlet Acquisition and those only relevant for Continuous Flow Acquisition.
As default, both parameter types are selected, i.e. no selector.

2 *Included String*

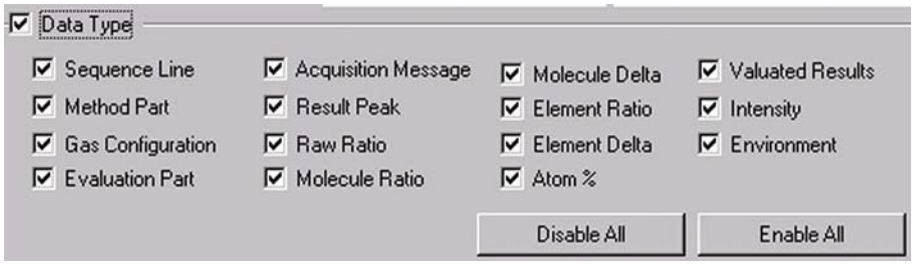


If Included String is activated and you type a string (e.g. “Delta”), the entries in the left pane (“pool”) will be selected according to that string after pressing ***Apply***. The entries that match the string will be displayed in the right pane.

3 *Gas Configuration*

If Gas Configuration is activated, choose a Gas Configuration from the list (e.g. “CO2”). All relevant data for this Gas Configuration will be shown in the right pane.

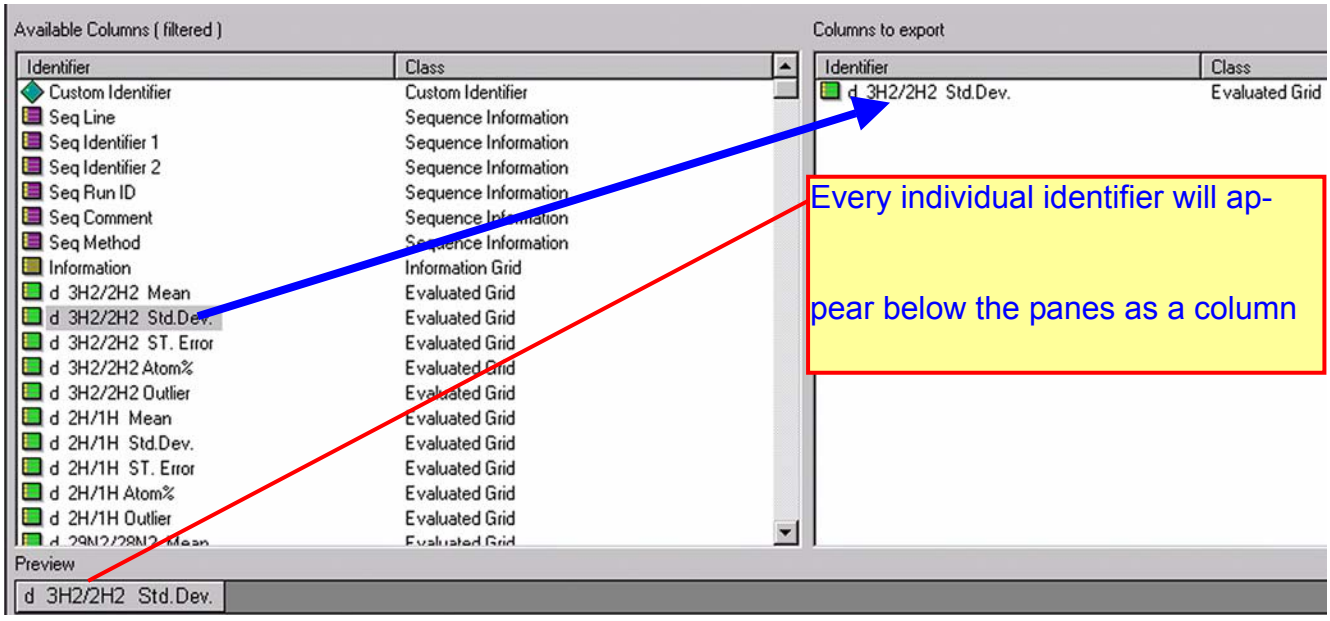
4 *Data Type*



If activated, you can select according to data type. Activate one or more selection criteria (e.g. if only “Raw Ratio” is selected, merely entries concerning raw ratios will be displayed in the right pane). As default, all data types are activated. Use ***Disable All*** or ***Enable All*** to ease your work with the Data Type selector.

Individual Identifiers

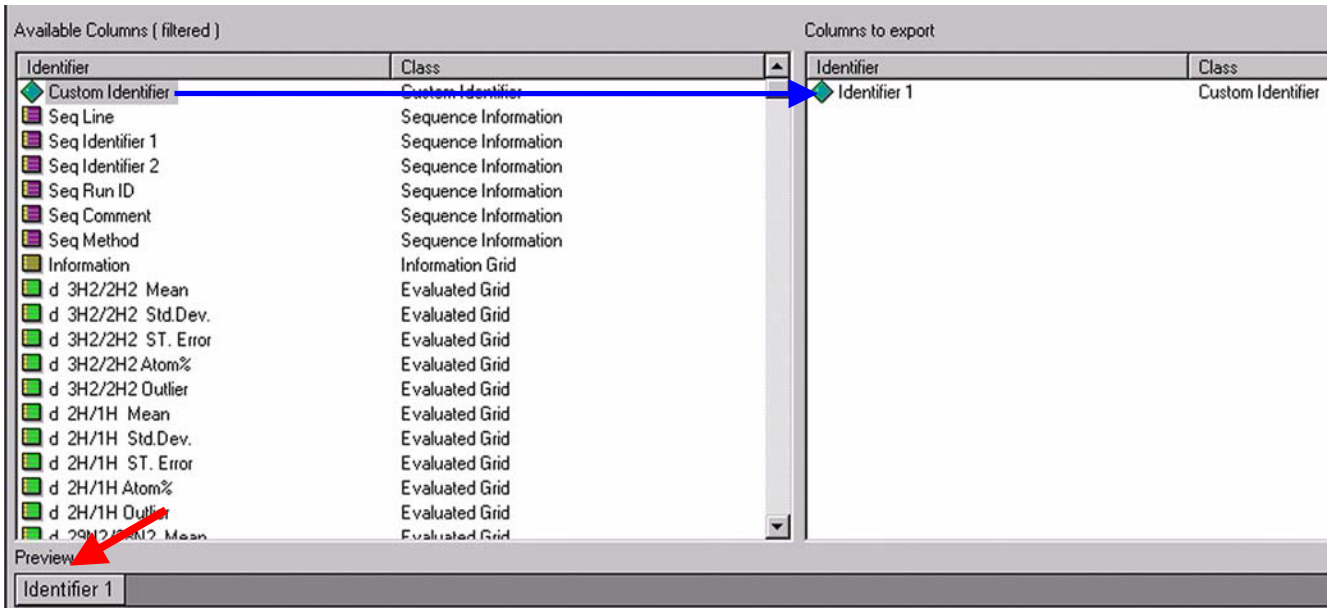
In addition to selectors drag and drop ***individual*** Identifiers to the right pane to export them:



Thermo Finnigan

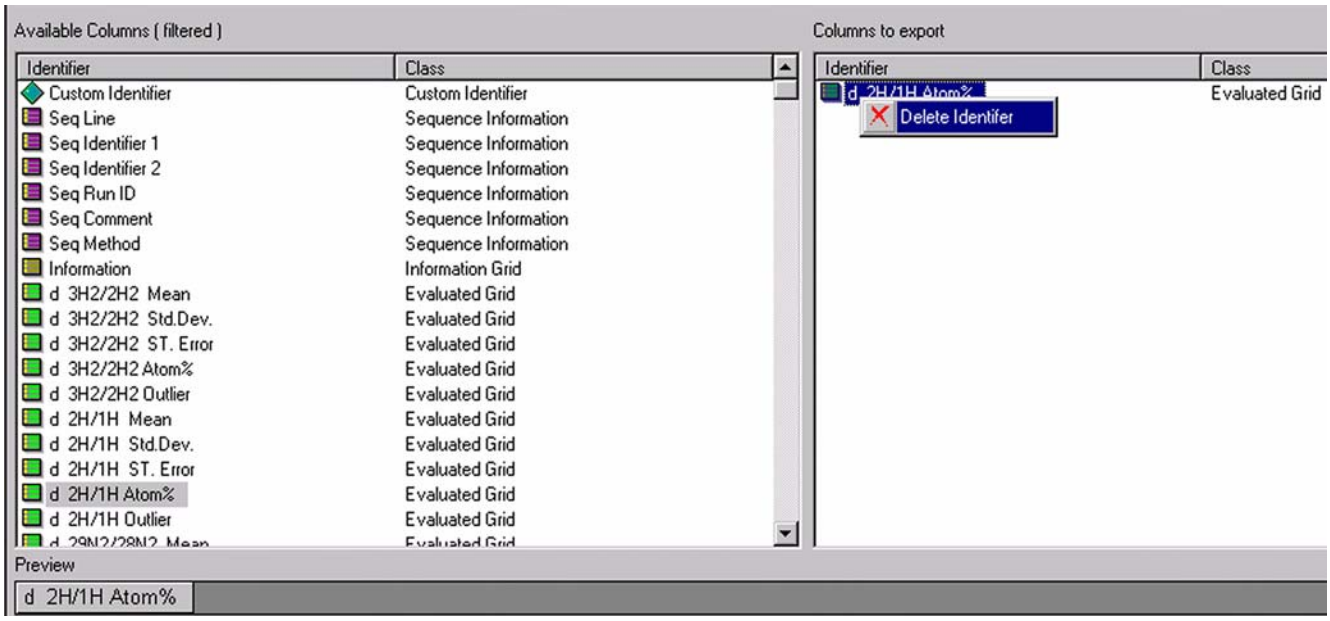
Drag and drop of the ***Custom Identifier*** allows inserting a column that contains no data. You can give it a name of your own choice. Later on, you can, for example, import data into this column. During drag and drop of the Custom Identifier from the left to the right pane, the box shown below appears.

- Type a significant name for the custom identifier (e.g. “Identifier 1”).
- Press ***OK***.



The Custom Identifier also appears as a column in the wk1 Excel sheet. To create additional empty columns, proceed with the next Custom Identifier(s) by repeating the steps above.

To remove one or more Identifiers (Custom Identifiers or other ones) from the right pane, mark them there. Then right-click them.



Select ***Delete Identifier***.

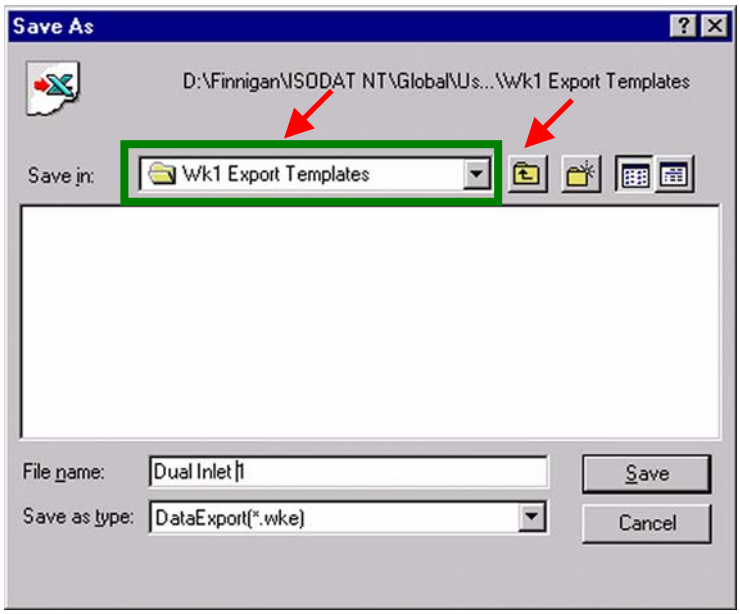
➢ To remove the Identifier(s) confirm by ***Yes***.

The selected Identifier(s) will be removed from the right pane together with the provided empty columns in the Excel sheet.

6.12.3 SAVING AN EXPORT TEMPLATE

After creating an export template (i.e. a wke file) it must be saved. Therefore, on the Dual Inlet window's toolbar press the ***Save*** or ***Save As*** button 

.



➢ ***Save the export template in the folder Wk1 Export Templates!***

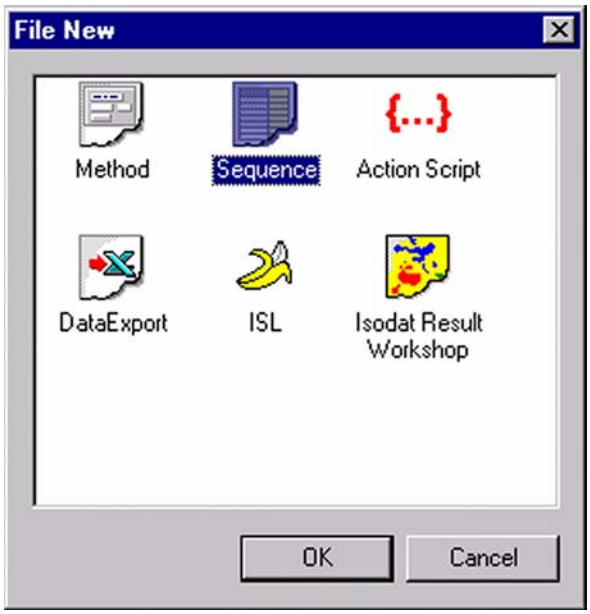
➢ In the ***File Name*** field, type a significant name for the export template (e.g. Dual Inlet 1).

➢ In the ***Save as type*** field, accept the extension .wke and press ***Save***.

6.12.4 APPLYING AN EXPORT TEMPLATE

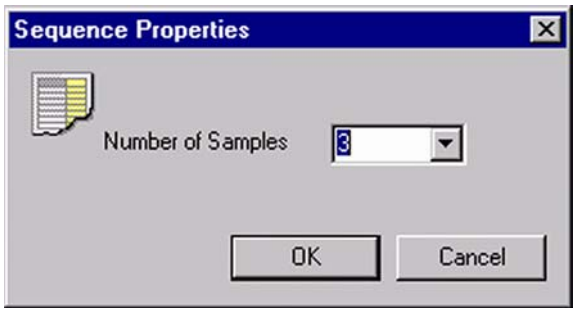
A Sequence is always exported as several samples can thus be bundled into a single export file. To apply an export template, open a new Sequence via the ***New*** button 

.

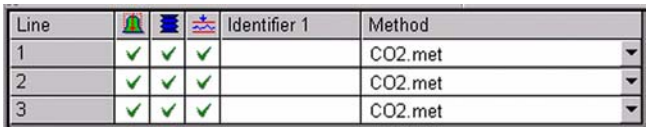


➢ Then click the ***Sequence*** Icon.

➢ Confirm by ***OK***.

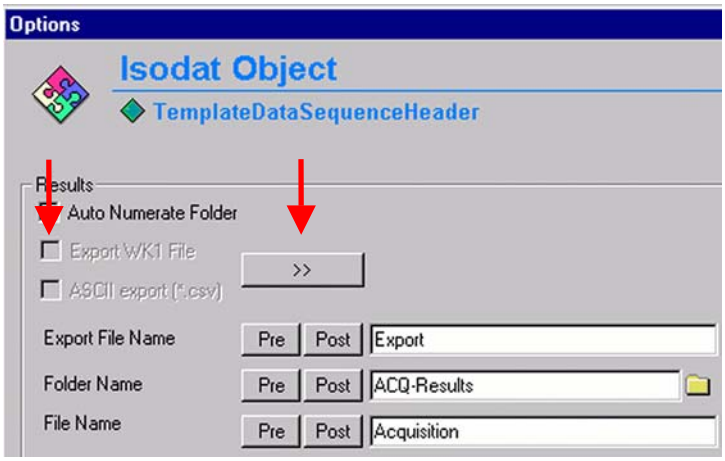


- Type your number of samples (e.g. 3).
- Confirm by ***OK***.



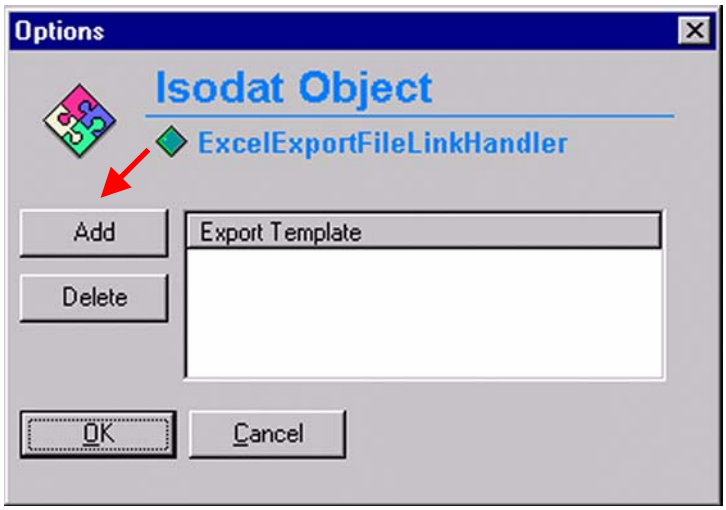
- The Sequence of your choice is displayed.
- Press the ***Start*** button 

.



- Since no export templates have been selected yet, the ***Export WK1. File*** box is inactive.
- Press the 

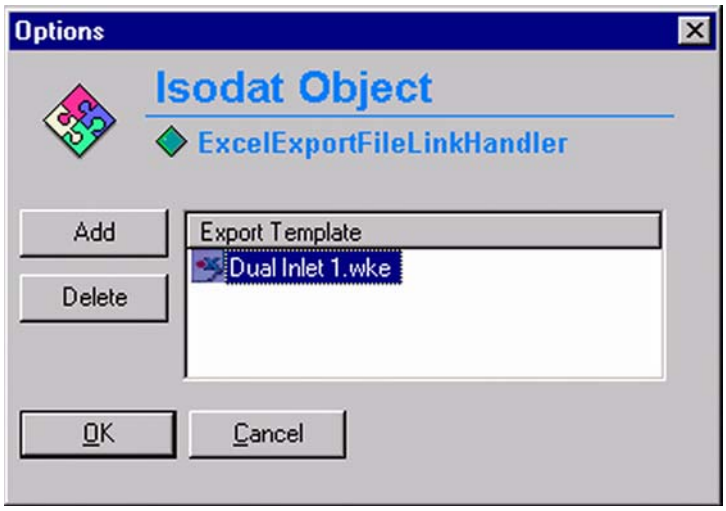
button.



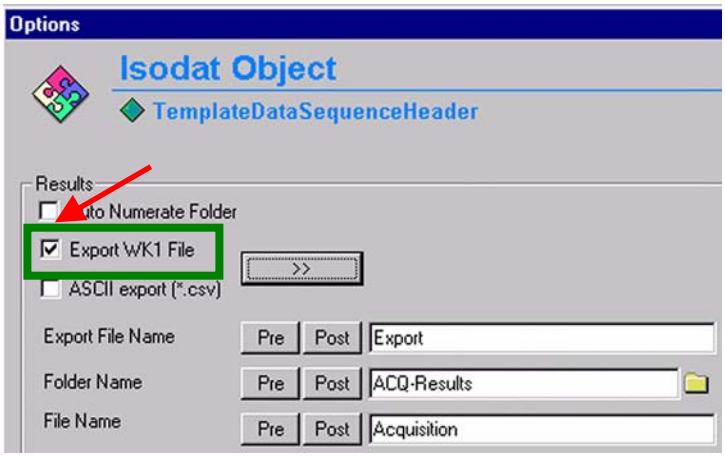
- Since no export templates have been selected yet, the Export Template list is empty.
- Press the 

button.

- In the folder ***WK1 Export Templates***, select your Export Template (e.g. Dual Inlet 1.wke).
- Press ***Open***.



- Select your Export Template (e.g. Dual Inlet 1.wke).
- Confirm by ***OK***.



- Note that the ***Export WK1 File*** box has now become active.
- Confirm by ***OK***.

The options specified in the Sequence are now set and Data Acquisition starts.

NOTE: ***The Excel Export will be performed online, i.e. during the measurement. This is different from a Re-evaluation, where the Excel Export takes place offline, i.e. after a measurement.***

MAT 253

OPERATING MANUAL

DIAGNOSIS

Issue 04/2002

Thermo Finnigan

7.1 CHECKING PERFORMANCE DATA

Thermo Finnigan MAT has developed several test routines to check the performance data of the ***MAT 253*** isotope ratio mass spectrometer.
For user's convenience, the program ***"Diagnosis"*** covering these test routines is included in the supplied version of ISODAT NT. It must be noted that operating some of the test routines requires technical knowledge of the instrument's internals. In addition, successful execution of some of the tests depends upon instrument preconditions.

When running the test routines, a highly sensitive focusing of your instrument will lead to the best specifications results (see chapter 1.4 about how to focus the ion source).

The program ***Diagnosis*** contains the following test routines:

1 Absolute Sensitivity

2 Abundance

3 Amplifier Test

4 Compression Factor

5 Linearity

6 Peak Flatness

7 Relative Sensitivity

8 Resolution

9 Signal Stability

10 System Stability

NOTE: ***Reference gas for all performance data is CO_2.***
Make sure to have properly filled CO_2 reservoirs attached to the inlet system before starting the "Diagnosis" program.

7.2 HOW TO START DIAGNOSIS

- Start ISODAT NT by double-clicking the Icon on your desktop.

- Double-click the ***Diagnosis*** Icon.

- Mark the test routine to be performed (e.g. “Abundance”).

- Press ***OK***.

7.2.1 ABSOLUTE SENSITIVITY

NOTE: ***Testing Absolute Sensitivity requires a Dual Inlet system***

Absolute Sensitivity is defined as the number of molecules needed to generate an ion, which is then registered at the collector (e.g. one ion of mass 44 at the corresponding collector cup). It is thus dimensionless and measured in molecules per ion.

Based on a defined volume, the ion current is determined during a defined time period as a function of sample consumption (i.e. sample loss). The small, defined volume is located between valve 25 of the inlet system and the inlet capillary. It amounts to approximately 250 µl.

Integrating the ion current over time yields the number of ions. From the signal drop during measurement the number of molecules necessary to generate this ion amount is calculated. To obtain Absolute Sensitivity, the number of molecules is divided by the number of ions.

Positively charged ions are produced in the ion source by electron bombardment. This electron impact (EI) ionization is described by:

$$AB + e^- \rightarrow AB^+ + 2e^- \qquad \text{(ionization)}$$

$$AB + e^- \rightarrow A^+ + B + 2e^- \qquad \text{(ionization and dissociation)}$$

Definition of Absolute Sensitivity AS:

$$AS = \frac{\Delta s}{n_{ion}} \; (molecules \; / \; ion)$$

where Δs: sample gas consumption

n_{ion}: number of detected ions

The number of consumed sample molecules n is calculated via the ideal gas law:

$$p\,V = n\,R\,T$$

where:
- R: universal gas constant
- T: temperature
- p: pressure
- n: number of sample molecules
- V: volume (here 250 µl)

The amount of detected ions n_{ion} in the collector cup can be calculated via the electrons needed to neutralize the positive ions:

$$Q = \int_{t_1}^{t_2} I\,dt$$

where:
- Q: charge
- I: intensity

To test this parameter:

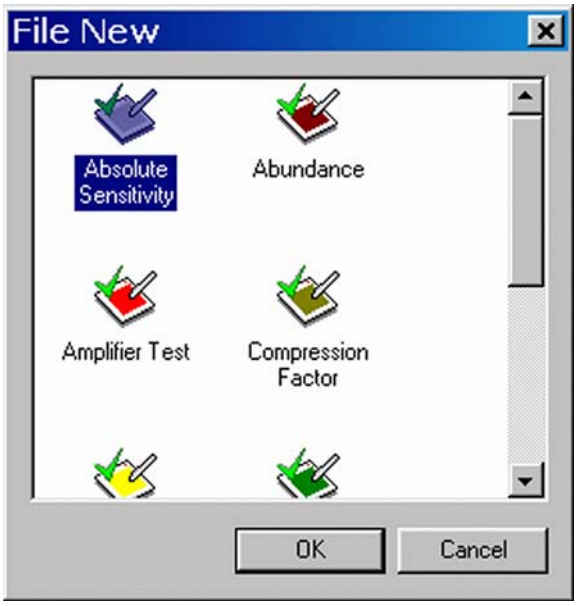
1 Measurement starts with a determination of pressure and intensity.

2 The volume is reduced to the defined volume by closing valve 25 of the inlet system. The system is in a waiting position until the start pressure and intensity are reached.

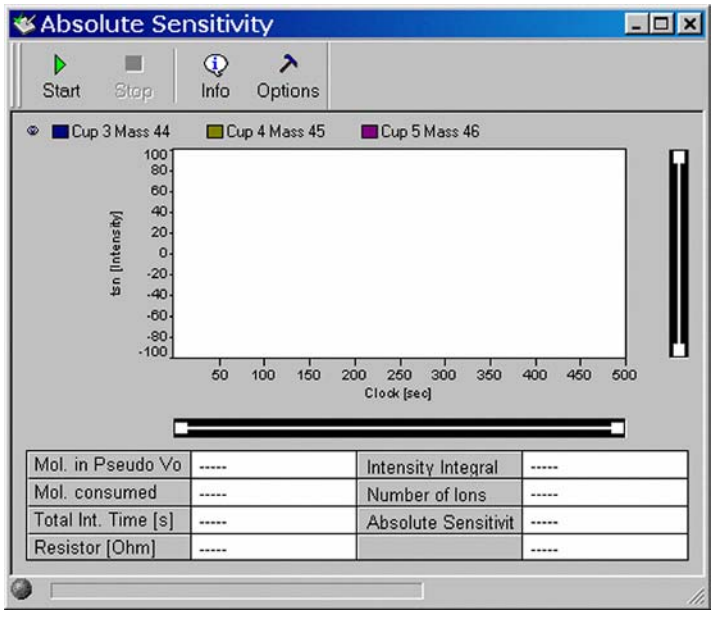
3 The ion current is measured for the preset time.

NOTE: ***Absolute Sensitivity should be about 800 molecules CO_2 per mass 44 at the collector cup.***

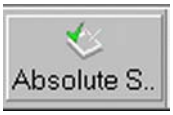
Testing Absolute Sensitivity



- Click on the ***Absolute Sensitivity Icon*** and press ***OK*** (or double-click on the Icon).



- On the "Window" toolbar, the ***Absolute Sensitivity Icon*** becomes visible.



- Press the ***Options*** button.

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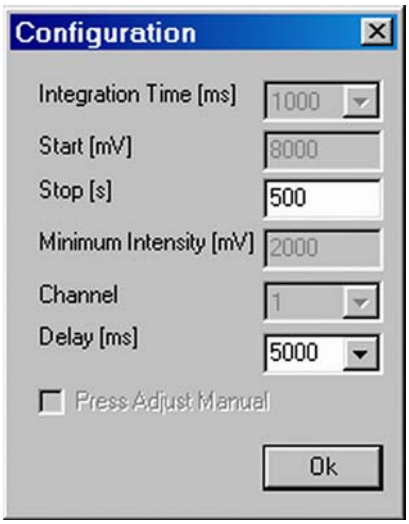
- Accept the defaults or from the pulldown menus, enter a value for "Stop [s]" and choose a value for "Delay [ms]" between 100 ms and 20000 ms from the list.



- In ***Advanced Mode***, all values can be edited:

  Integration time in ms

  Start Intensity value ("Start [mV]")

  Minimum Intensity [mV]

  Channel: a recording channel consists of an amplifier, a voltage-frequency (VF) converter and a counter.

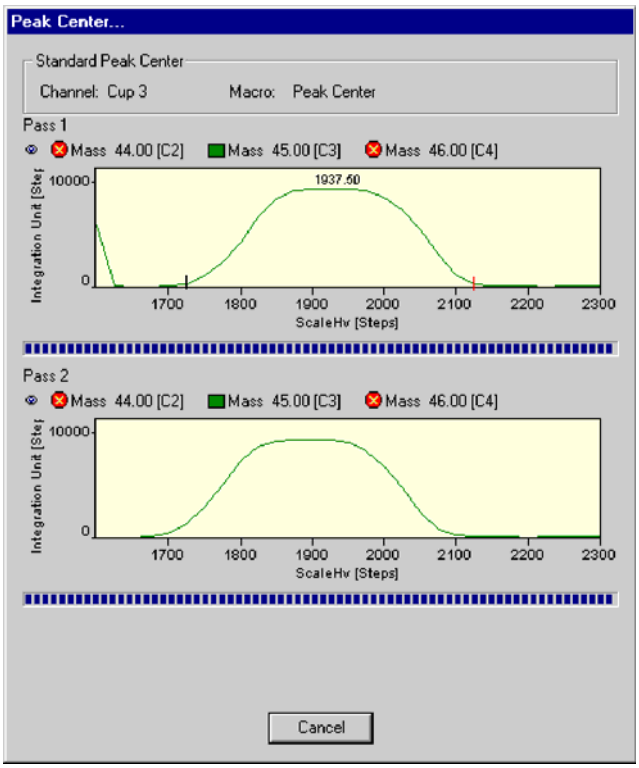
  "Press Adjust Manual": activate it, if you wish to adjust pressure and peak center manually (if it is activated, you are requested to adjust to e.g. 8000 mV manually).

➢ Finally, click ***OK***.

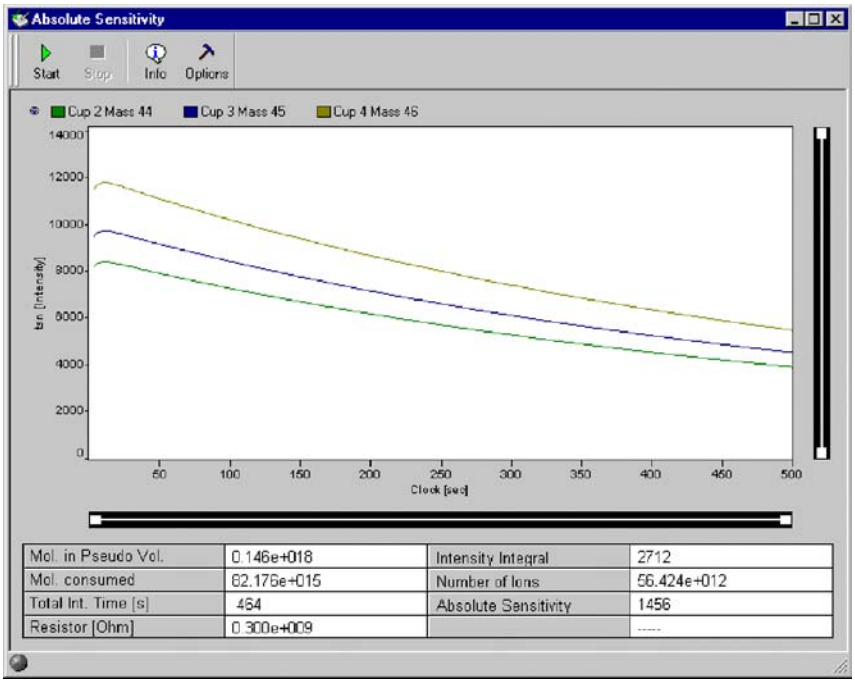
➢ Press the ***Start*** button.

- Testing starts with a Peak Center on the selected Cup, e.g. mass 45, Cup3.

  The bellow is reduced stepwise until the start level intensity (e.g. 8000 mV) is reached. Then, intensity and pressure are measured for the preset time (e.g. 500 s).



- Finally, Absolute Sensitivity and other parameters are calculated.



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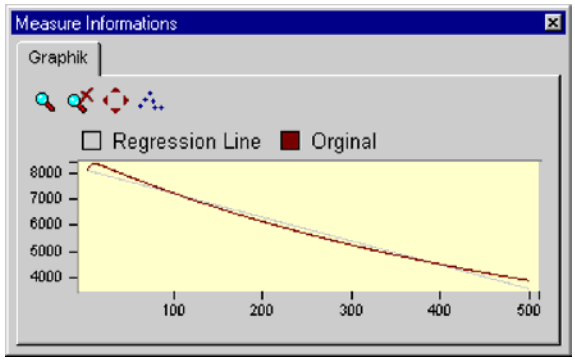
- Press the ***Info*** button.

- Original measurement and regression line are shown.



- When closing the "Absolute Sensitivity" window you are asked, whether to save the changes.

7.2.2 ABUNDANCE

Abundance denotes the contribution of a mass to a neighbor mass (e.g. the amount of ions for mass 44 falling into cup 45). Thus, the intensity of a mass (e.g. mass 44) is compared to the intensity of the neighboring peak (e.g. mass 45).

In case of CO_2, divide the amount of ion current of mass 44 falling into the mass 45 cup by the ion current of mass 44 falling into the mass 44 cup.

Measured as ratio of two ion currents it is dimensionless and quoted in % or ppm. It should not exceed $2 * 10^{-6}$ for a ***MAT 253*** with Dual Inlet system. The Abundance test is performed with CO_2, and the device must be calibrated. Peak center is performed on Channel 2 (i.e. narrow cup) before the measurement starts.

To test this parameter

1 Signal height (i.e. intensity) on mass 44 is determined in the appropriate collector cup (e.g. Cup 3).

2 The background signal on mass 45 is measured (i.e. electronic noise with no gas) from mass 44 to mass 45.5.

3 With CO_2 as sample gas, the magnetic field is scanned from mass 44 to mass 45.5, and the intensities are measured on the neighboring Cup of mass 45 (e.g. Cup 4, with bigger resistor value in order to keep the signal in the detection range).

4 Extrapolate the abundance (of mass 44 onto mass 45) from the signal to the left and to the right of mass 45 peak.

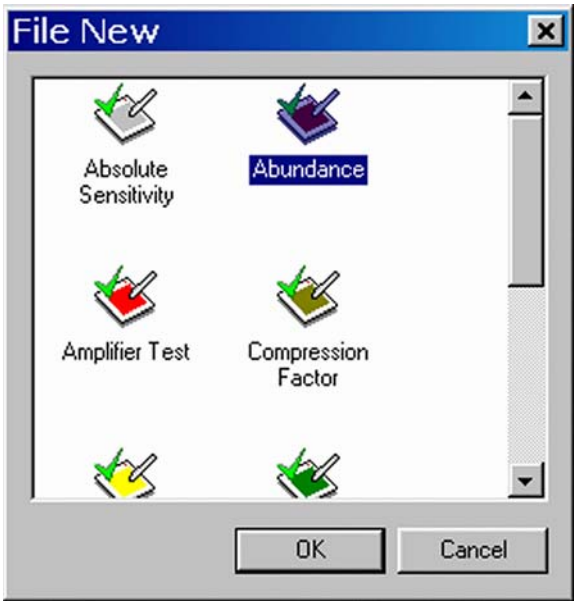
5 Calculate the abundance as described above.

Sources of error

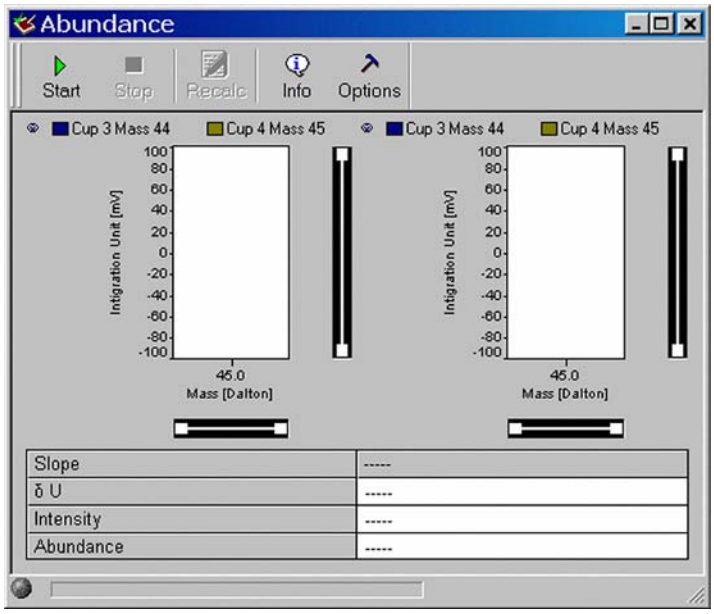
- Resistor values are not configured correctly.
- Due to electrons on the left and on the right side of the peak, a negative signal may result. This problem can be overcome by manual adjustment.

Testing Abundance

- Click on the ***Abundance Icon*** and press ***OK*** (or doubleclick the Icon).



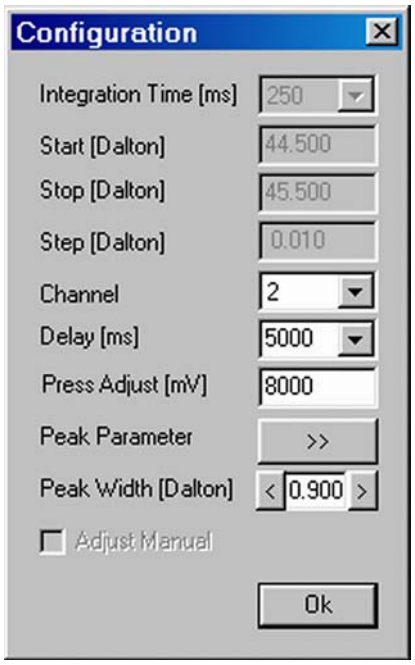
- The left window detects mass 44 ions in the correct cup. The right window detects mass 44 ions in the neighbor cup (i.e. cup of mass 45).



- On the "Window" toolbar, the ***Abundance Icon*** becomes visible.

- Press the ***Options*** button.

  - Accept the defaults, or type appropriate values for "Press Adjust [mV]" and "Peak Width [Dalton]". The selected cup for the Abundance test is connected with the indicated recording channel (e.g. choose channel 2 from the pulldown list). Choose a "Delay [ms]" value between 100 ms and 20000 ms. The measurement start will be delayed by it.

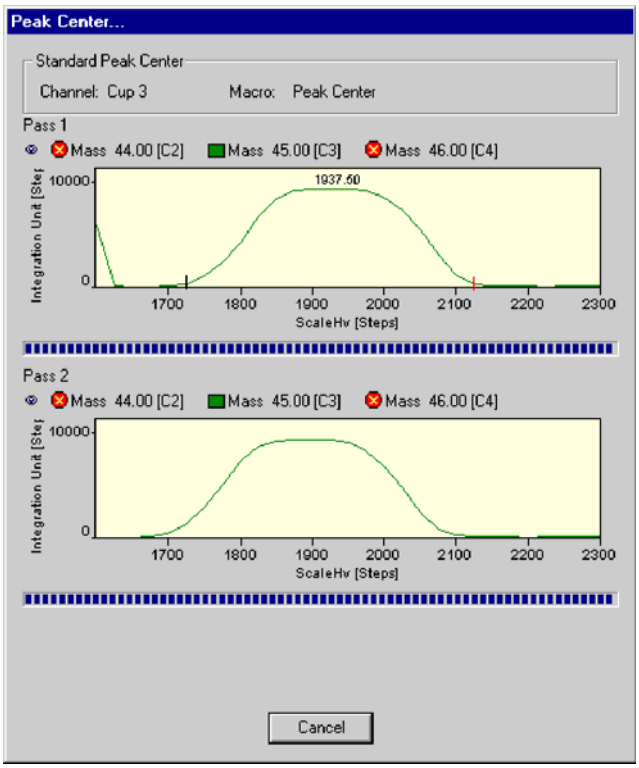


    In ***Standard Mode***, the inactive, i.e. gray, variables cannot be edited. The ***Advanced Mode*** allows experienced users to set them all:

    - the start mass for the magnet field scan ("Start [Dalton]"),
    - the end mass, where the magnet stops scanning ("Stop [Dalton]")
    - the step width for the magnet scan ("Step [Dalton]")
    - the "Adjust Manual" check box - if it is activated, signal intensity and peak center must be performed manually.

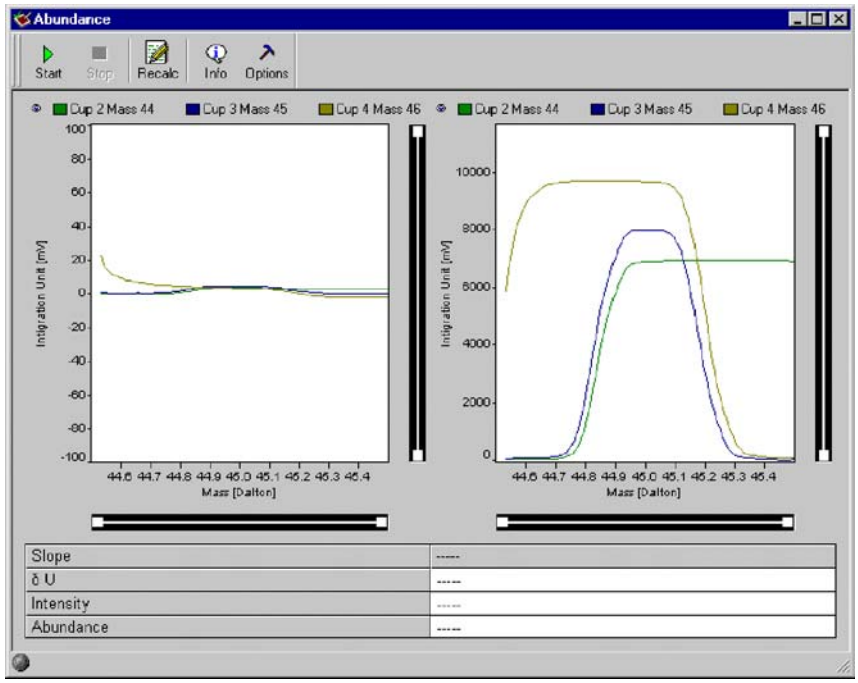
- Finally, click ***OK***.
- Start the Abundance test by the ***Start*** button.

- Testing starts with a Peak Center on the selected cup (e.g. mass 45, cup 3).



It might be helpful to activate the ***Advanced Mode***, press the ***Options*** button and enhance the interval between "Start" and "Stop" values (i.e. decrease "Start" and increase "Stop" values).

- Values for Abundance, Intensity, δU and slope are displayed.



- When closing the "Abundance" window you are asked whether to save the changes.

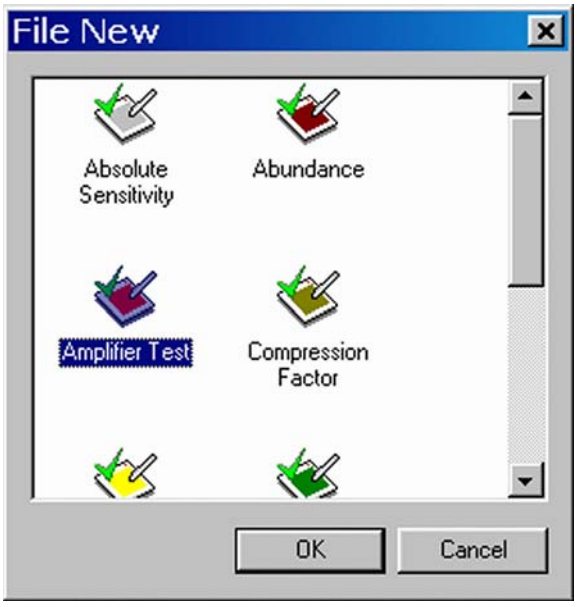
7.2.3 AMPLIFIER TEST

The Amplifier Test (formerly called UFC-Test) checks the ion detection performance of the IRMS with no ions present. It thus informs about the background noise of electronic devices.

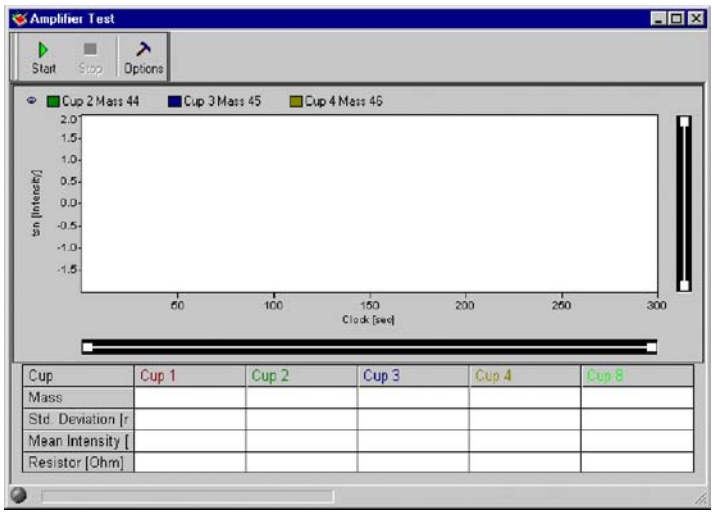
The amplifier baseline must be determined without an interfering signal. Thus, the ion source is switched off before measurement starts (i.e. ion current equals zero). The signal intensity of every cup is individually measured at least 200 times for a defined integration time. Finally, mean and standard deviation are calculated.

Testing Amplifiers

- Click the ***Amplifier Test Icon*** and press ***OK*** (or doubleclick the Icon).



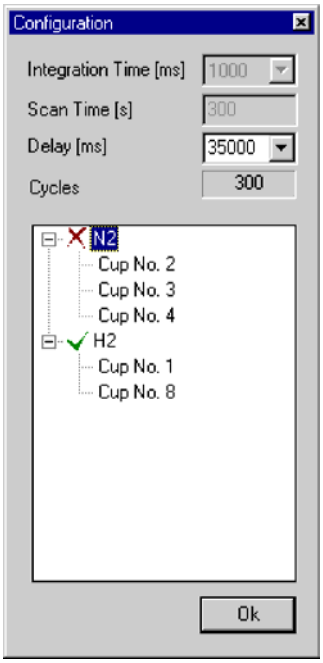
- Wait, while the available Gas Configurations are scanned.



- On the "Window" toolbar, the ***Amplifier*** becomes visible.

- Press the ***Options*** button.

- With the exception of ***Delay***, all parameters are preset and not changeable in Standard Mode.
  - The Advanced Mode allows experienced users to modify all parameters.
  - Accept the default value of ***Integration Time*** (in ms).
  - ***Scan Time*** (in s) denotes the duration of the scan.
  - The measurement start is delayed by the displayed default value of ***Delay*** time (in ms). Accept it, or from the pulldown menu, select a suitable value between 100 ms and 35000 ms.



NOTE: ***In one of the next versions, the process will be simplified: all amplifiers can then be measured using one single Scan.***

- ***Cycles*** denotes the number of measurements. The intensities of the channel are measured for indicated cycles with a definite cycle time.

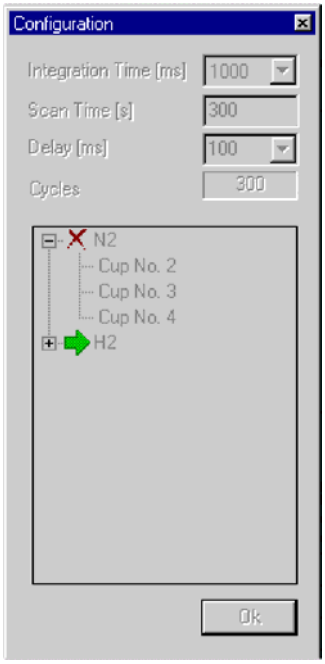
- Finally, press ***OK***.

- Press the ***Start*** button.

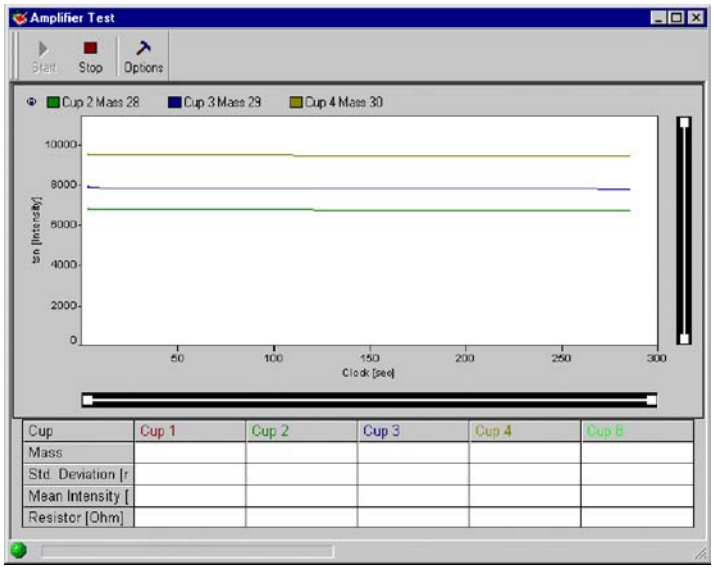
- ***Switch*** off High Voltage (on the ***Focus*** bar). Then press ***OK***.

- The amplifiers of the cups activated in step 5 are being tested, e.g. cup 1, mass 2, brown and cup 8, mass 3, green.

- If you press the ***Options*** button during measurement, a green arrow informs you about what is currently done.

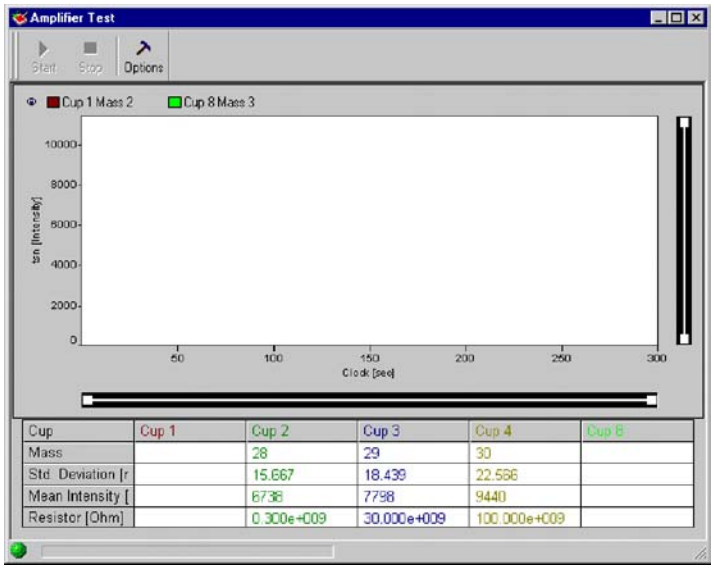


- The correspondent intensity vs. time diagram is shown.



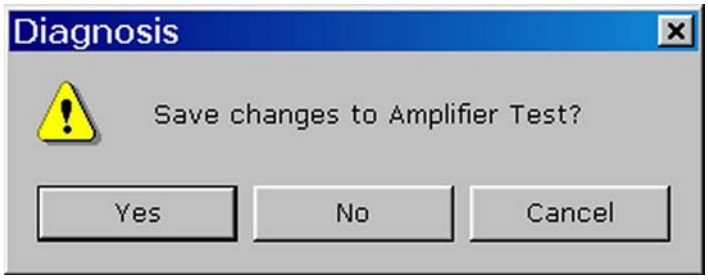
- The baselines of the cups are shown with no ions present.

  Standard deviation [mV], mean [mV] and resistance [Ohm] of each selected cup are displayed.



- The procedure is repeated for the other masses.

- When closing the "Amplifier Test" window you are asked, whether to save the changes.



7.2.4 COMPRESSION FACTOR

NOTE: ***Testing Compression Factor requires a Dual Inlet system.***

The Compression Factor, formerly called ***Pressure Ratio***, is defined as intensity ratio and is thus dimensionless [mV/mV]:

$$Comp = \frac{Int_{end}}{Int_{start}}$$

where:

Int_{end}: Intensity at the end of measurement

Int_{start}: Intensity at measurement start

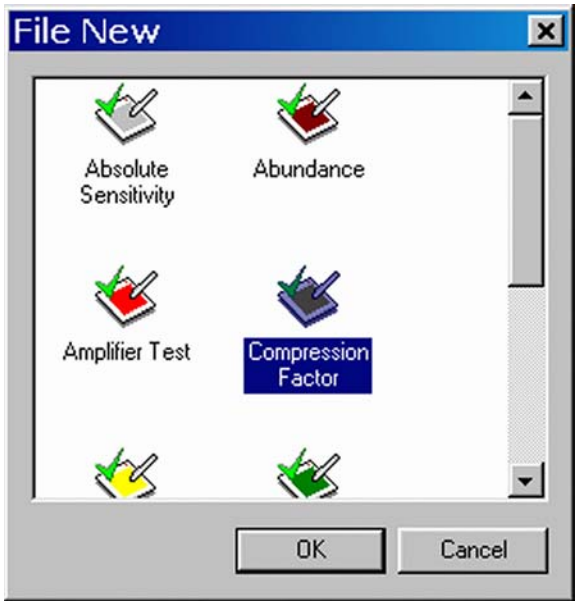
The Compression Factor determines the dynamic range of the two bellows informing about their tightness and linearity. The ion signal (i.e. intensity) is measured at different bellow compressions: an intensity vs. volume diagram results. The standard deviation around the signal's mean is calculated.

NOTE: ***The bellows must be calibrated before performing the test.***

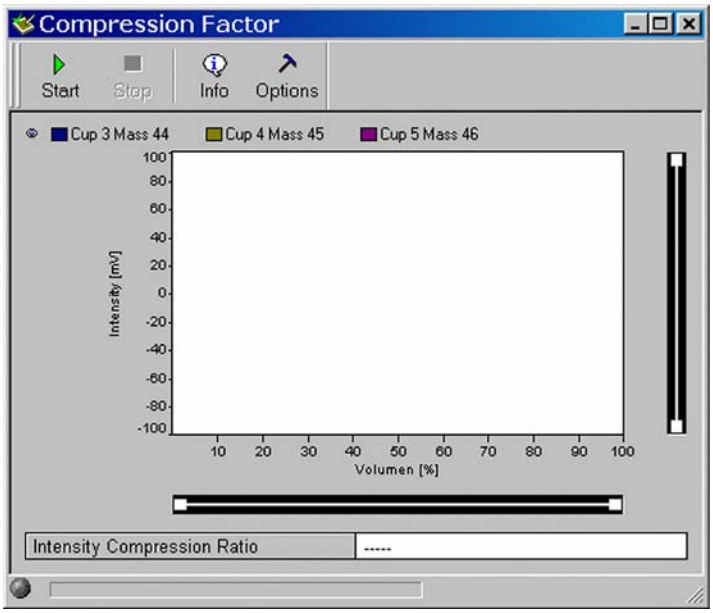
To test this parameter, the peak intensity for a mass (e.g. 44) is measured starting at the maximum (i.e. 100 %) down to the minimum (i.e. 0 %). A minimum death volume of about 3 ml is still remaining at 0 % volume. A certain level (e.g. 200 mV) serves as starting point of the measurement. The signal for the bellow expanded to maximum should be at this level. If it is not the case, the inlet system is expanded and pumped automatically until the reference level is reached.

NOTE: ***The Compression Factors should be about the same for both bellows: at least 1:10 or higher.***

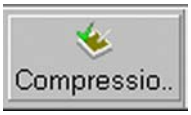
Testing the Compression Factor



- Click on the ***Compression Factor Icon*** and press ***OK*** (or double-click the Icon).



- On the "Window" toolbar, the ***Compression Factor Icon*** becomes visible.

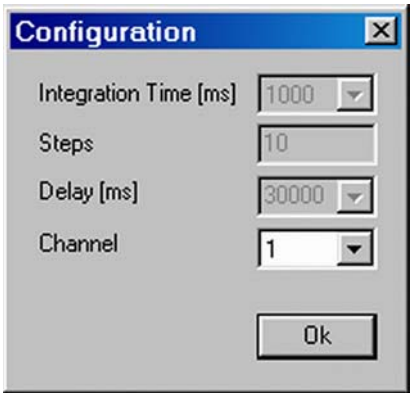


- Press the ***Options*** button.

- In ***Standard Mode***, you are only able to select a channel from the pulldown menu.

  In ***Advanced Mode***, also the other variables can be edited:

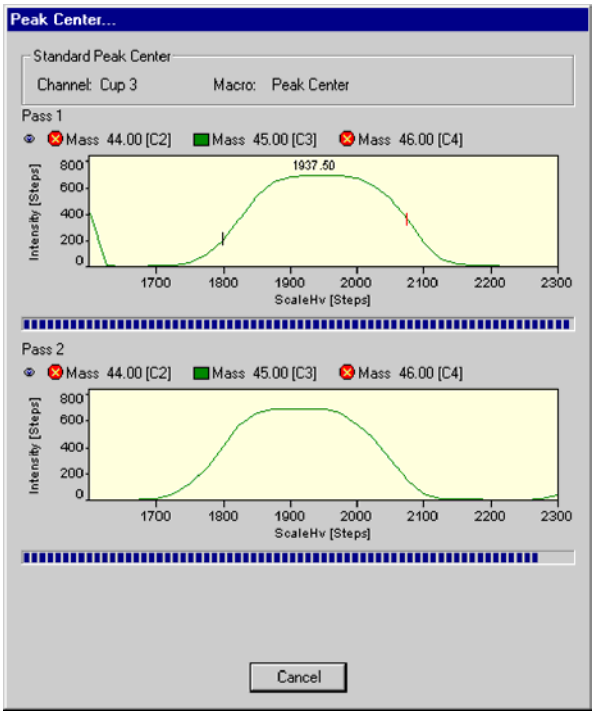
  - Integration time [ms],
  - Steps: the volume of the bellows is varied stepwise, e.g. in 10 steps,
  - Delay [ms]: the measurement start is delayed by the displayed value.



- Finally, press ***OK***.

- Press the ***Start*** button.

- Testing starts with a Peak Center for the first bellow (e.g. Sample).



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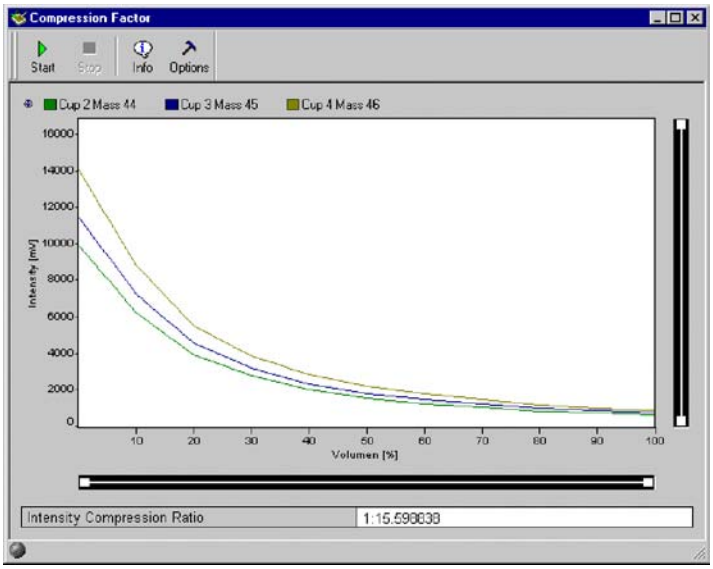
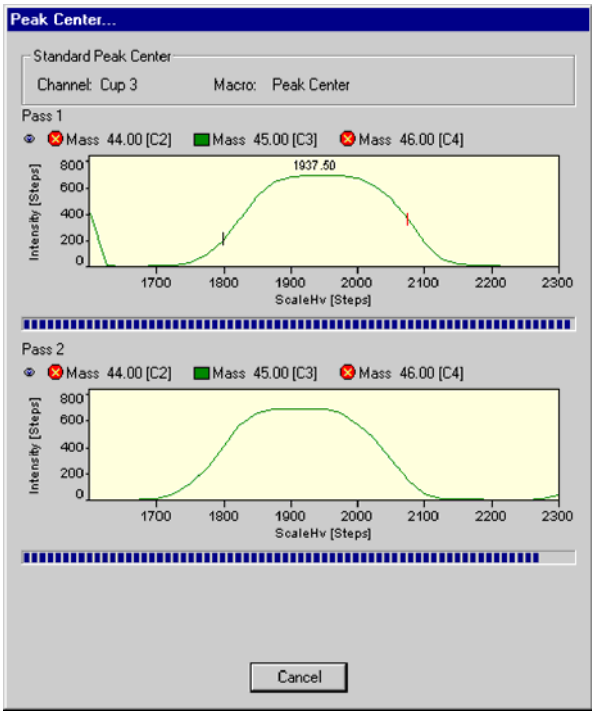
- When the Peak Center is finished, signal intensity is shown during the volume change of the bellows (for the different masses).

  Finally, the Intensity-Compression ratio is calculated for the first bellow, e.g. Sample.



- The procedure is repeated for the second bellow (e.g. Standard): a Peak Center is performed.



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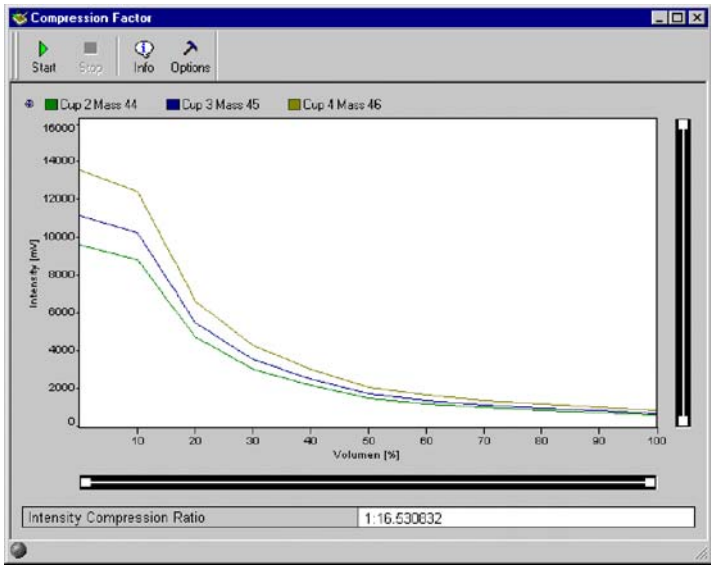
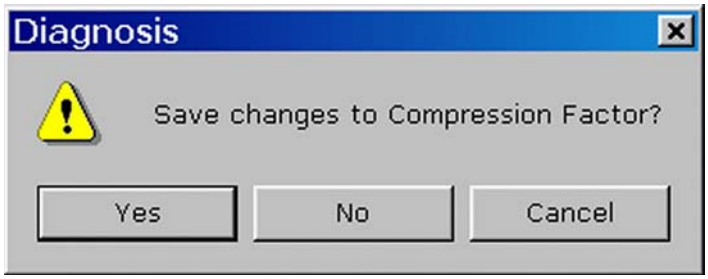
- The procedure is repeated for the second bellow (e.g. Standard).

  When the Peak Center is finished, an Intensity vs. Volume diagram for the different masses is shown. Finally, the Intensity Compression ratio is calculated for the second bellow (e.g. Standard).



- When closing the "Compression Factor" window you are asked, whether to save the changes.

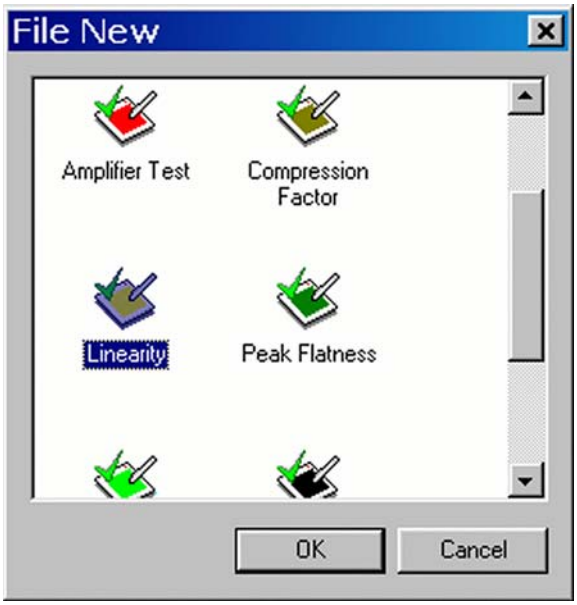


7.2.5 LINEARITY

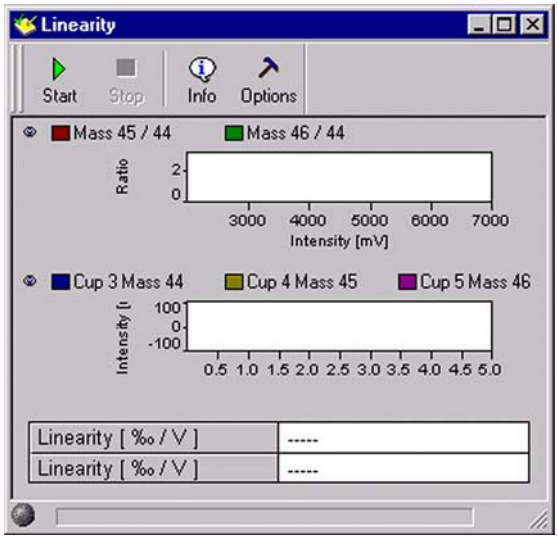
At Linearity test, synonymously called ***Ratio Linearity***, over a range of varying signals, signal linearity is checked vs. beam intensity (i.e. intensity of the main ion current). The signal intensity is measured, and the isotope ratios are displayed vs. beam intensity. Linearity is calculated as slope of the regression line [‰/V]. The ratios are monitored between 2 V and 8 V in 1 V steps. For each data point, the background is subtracted.

Source of error: Resistor values must be configured correctly.

Testing Linearity



➢ Click the ***Linearity*** Icon and press ***OK*** (or double-click the Icon).

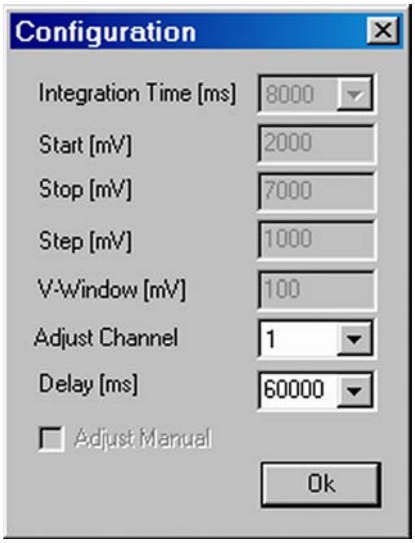


- On the "Window" toolbar, the ***Linearity*** Icon becomes visible.

- Press the ***Options*** button.

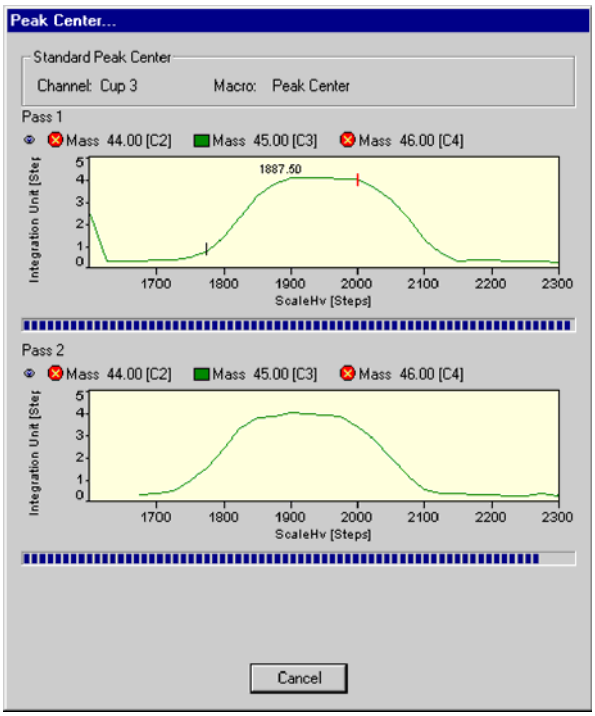
- From the pulldown menus select:
  - a channel and a value between 100 ms and 20000 ms for "Delay [ms]".

    The other variables are preset in Standard Mode (to be accepted), but editable in Advanced Mode:
  - Integration time [ms], Start [mV], Stop [mV] Step width [mV], V-Window [mV]
  - Adjust Manual: activate it, if the intensities (i.e. voltage steps) between 2 V and 8 V are to be set manually.



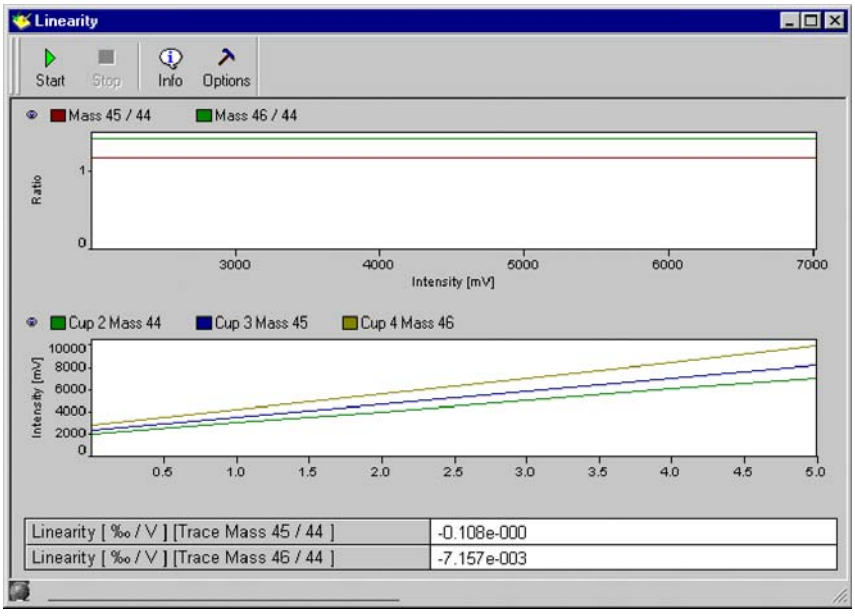
- Finally, press ***OK***.

- Press the ***Start*** button.



- Testing starts with a Peak Center on cup 3 as the narrow cup.

  If no peak could be found, the measurement will be terminated.

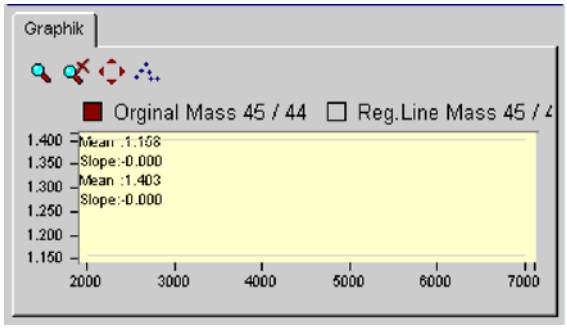


- Two linearity values [‰/V] are calculated: one for mass 45/44 and one for mass 46/44.

NOTE: ***Linearity must be less than 0.06 ‰/V!***

➢ Press the ***Info*** button.

➢ Mean and slope values are displayed.



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7.2.6 PEAK FLATNESS

As slope of the peak plateau, Peak Flatness reflects the quality of the ion stream. A correction is necessary to eliminate effects of descending peak plateau with increasing high voltage. This is done by measuring the peak twice - first with increasing and then with decreasing high voltage. The resulting peak represents the mean values of both runs. The measured intensity is a function of the acceleration voltage (i.e. ion energy). Therefore, during a high voltage scan the intensity is slightly affected by this effect. To overcome this an "energy correction" is performed. The ion intensity on top of the peak (i.e. at a parameterized mass range around the center) is measured. Peak Flatness can be determined for different gases and different collector cups (e.g. for a CO_2-peak at mass 45 at Cup 2).

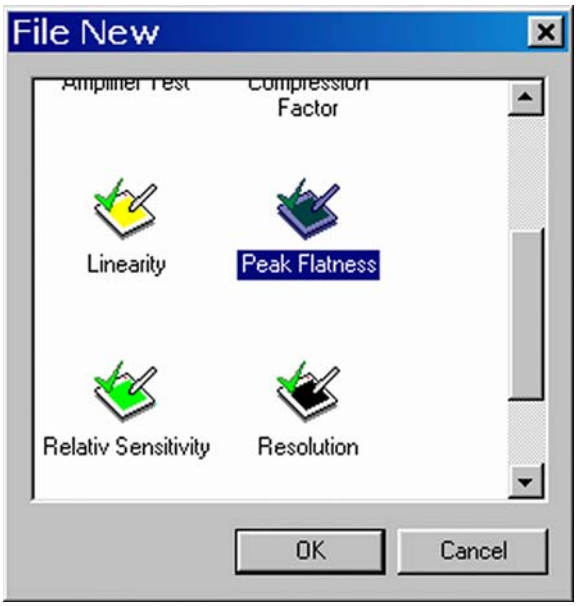
Two results are obtained:

- Maximal intensity deviation divided by the intensity
- Slope of the regression line [1/Da]

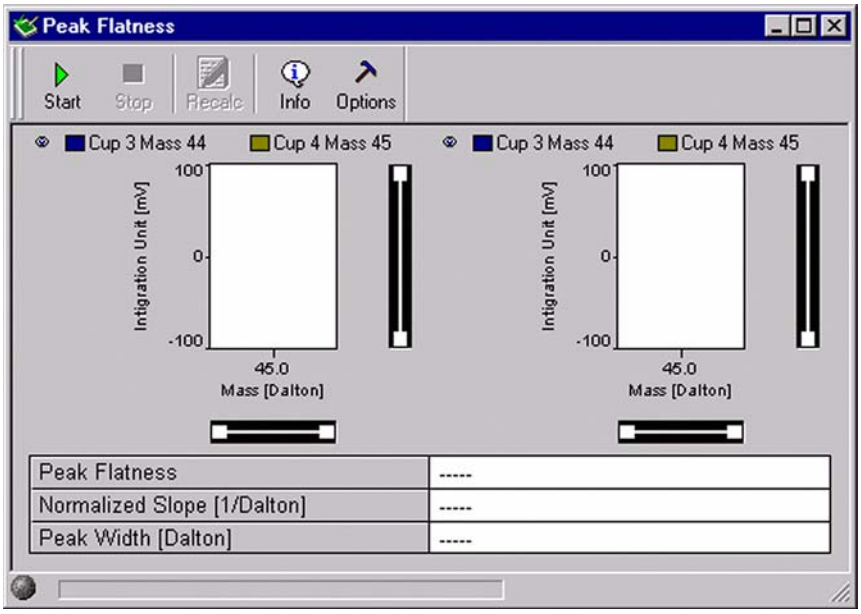
Differences to ISODAT Old

- ***Slope of the regression line as additional result***
  Dividing System Stability by this slope yields a measure for Signal Stability with respect to high voltage or magnet current instabilities.
  It is complementary to the Diagnosis parameter Signal Stability that is also sensitive to emission fluctuations.
- ***Parameterized mass range***
  In ISODAT Old, a certain proportion of the peak top (30%) is used instead of a parameterized mass range. However, to know how much the measurement is affected by a certain system drift, remember that this is a proportion of mass scale rather than of cup width. The default mass range is set in such a way that the final result is comparable to the one of ISODAT Old using a typical cup width.

Testing Peak Flatness



- Click on the ***Peak Flatness Icon*** and press ***OK*** (or doubleclick on the Icon).



- On the "Window" toolbar, the ***Peak Flatness Icon*** becomes visible.

- Press the ***Options*** button.

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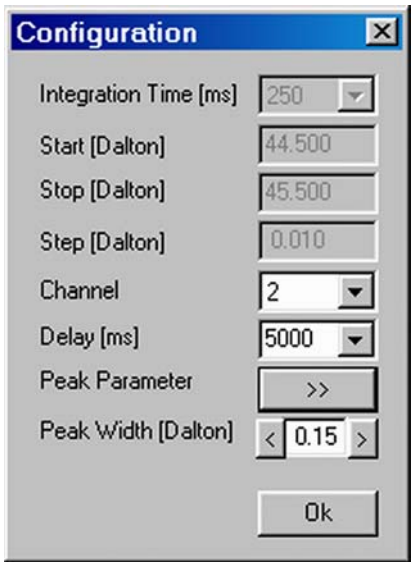
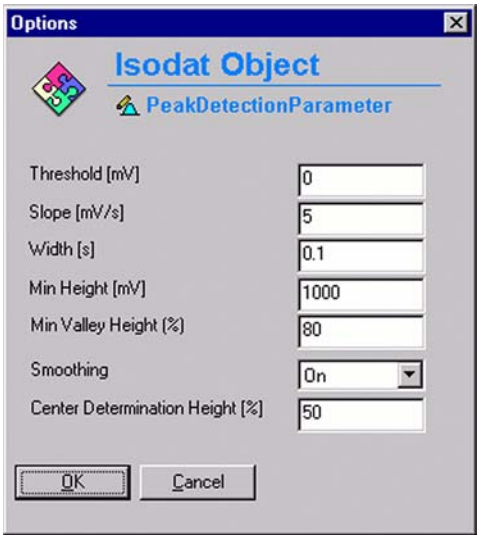
- Accept the defaults or from the pulldown menus, select a Channel and a Delay value between 100 ms and 20000 ms.



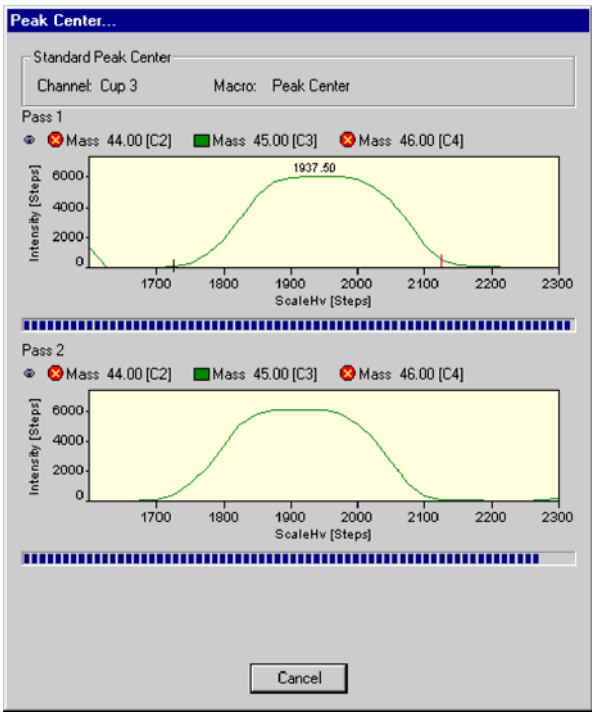
- Accept the default, or choose an appropriate Peak Width value by the < or > buttons.
- If you simply want to accept the Peak Detection parameters without a glance, do **not** press the ***Peak Parameter*** button. Instead, click ***OK***.
- However, as a sophisticated user, you may want to look at the Peak Detection parameters. To do so, press the ***Peak Parameter*** button.

➢ Change the values according to your needs.



➢ Then click ***OK***.

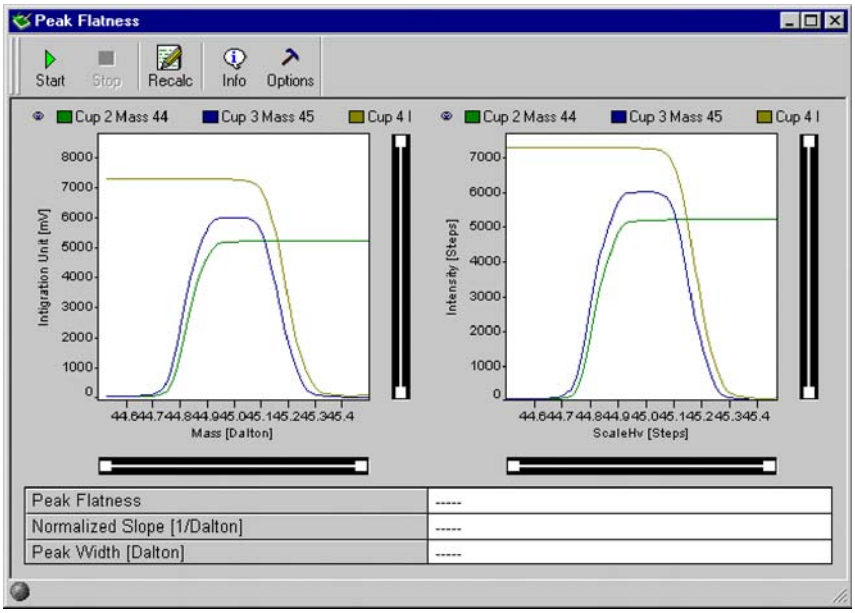
➢ Press the ***Start*** button.

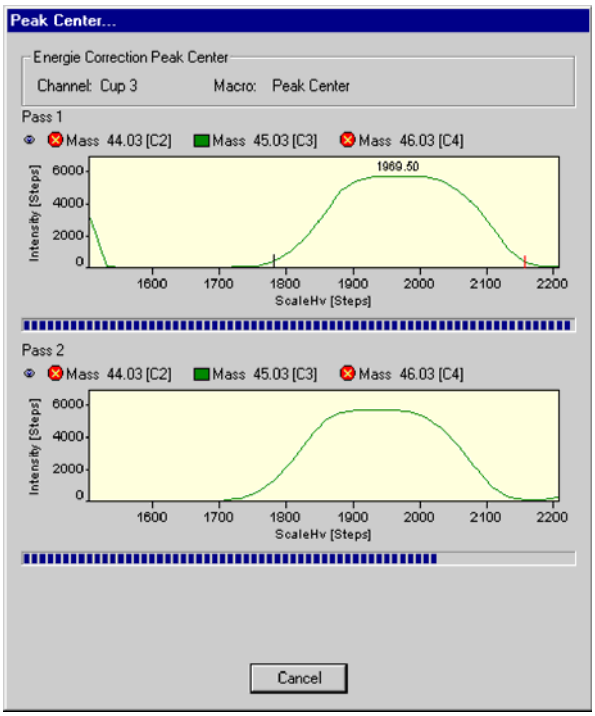


- Testing starts with a Peak Center on the selected cup (e.g. mass 45, cup 3).

If no peak can be found, the measurement will be stopped.

In this case, press ***Cancel*** to proceed.

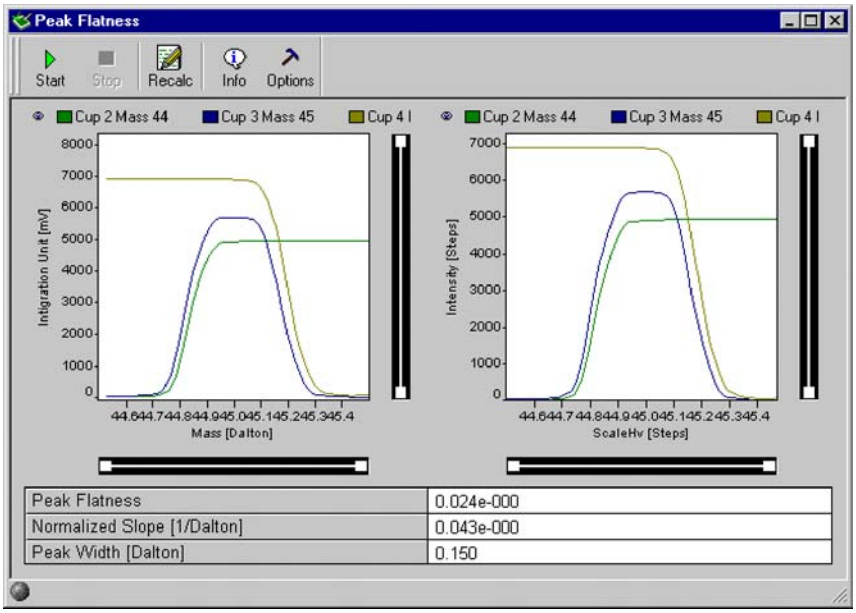




- A Peak Center for Energy Correction is performed.

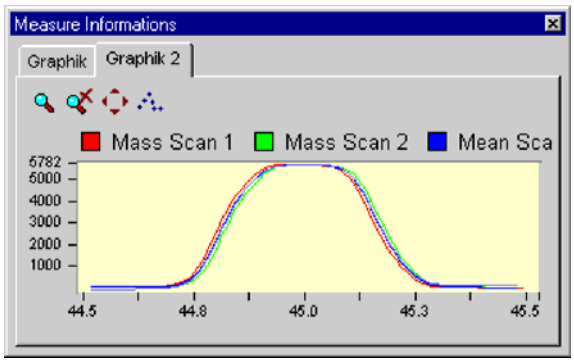
  If no Peak Center can be found, the measurement will be stopped.

  In this case, press ***Cancel*** to proceed.

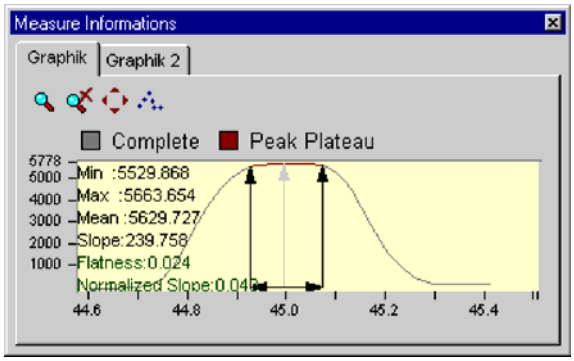


- Values for Peak Flatness, normalized slope [Da^{-1}] and Peak Width [Da] are calculated.

- Press the ***Graphic*** tab to view relevant data.



- Press the ***Graphic 2*** tab to view relevant data.



- When closing the "Peak Flatness" window you are asked whether to save the changes.

7.2.7 RELATIVE SENSITIVITY

Relative Sensitivity (S_{rel}) describes the dependency of signal intensity (i.e. ion current) on the ion source pressure and is thus given in A/mbar:

$$S_{rel} = \frac{1}{0.69}\,\frac{1}{p}\,\frac{U}{R}$$

where:

U: voltage measured at the amplifier of the collector cup (e.g. Cup 3, mass 44)

R: resistor value (e.g. 3 * 10^8 Ω for mass 44). This value is the same for N_2 and CO_2 as reference gases. It needs to be changed in special cases only.

Δ p: pressure difference between a measurement with and without reference gas.

0.69: correction factor for CO_2. The ion gauge is calibrated with N_2, however, which has a different ionizing probability. The correction factor takes this into account. To calculate it, intensities (i.e. ion currents) and pressures are measured with and without reference gas.

NOTE: ***Two different Relative Sensitivity values exist depending on whether the instrument is equipped with a differential pumping system or not. The difference is due to different pressure readings at the same flow. The Absolute Sensitivity (given in molecules/ion) however, is the same.***

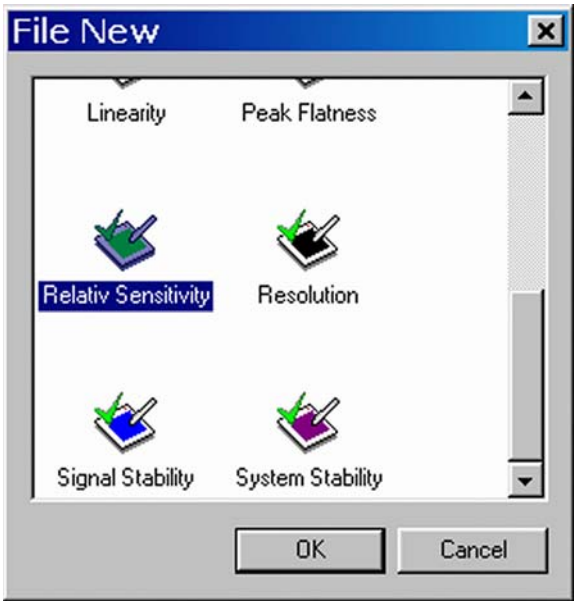
After a cycle of e.g. three measurements and calculations of Relative Sensitivity, a mean value is displayed. This value should be about 0.2 A/mbar for a standard system and about 0.5 A/mbar in case of a differentially pumped system. They depend on the pumping capacity of the turbomolecular pumps. Each cup (i.e. each mass) is characterized by a Relative Sensitivity value of its own.

Different values of Relative Sensitivities can only be compared, if pumping speed, conductance, location of the ion gauge etc. are identical. Therefore, it is senseless to compare different types of instruments.

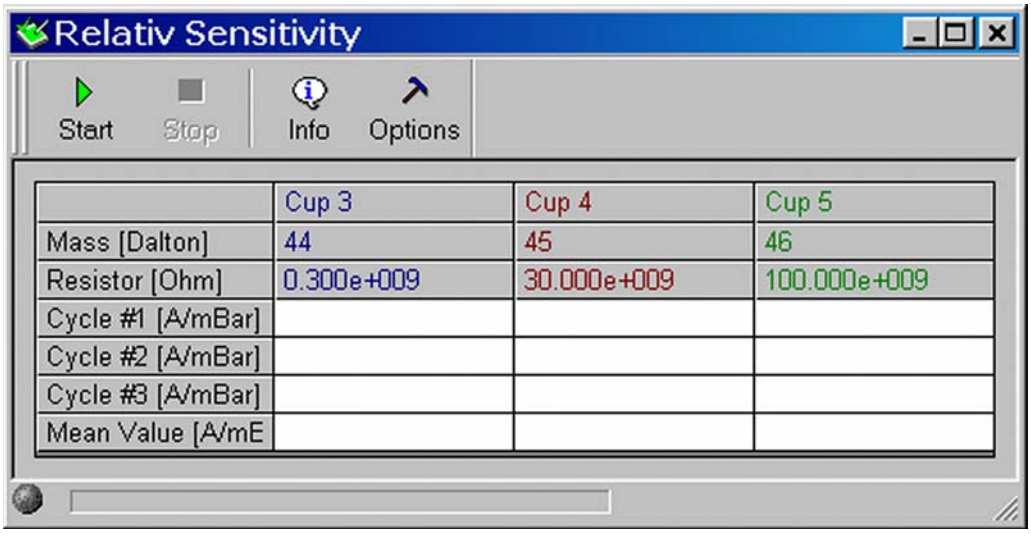
Sources of error

- Resistor values must be configured correctly.
- Relative Sensitivity depends on the accuracy of the high vacuum pressure gauge's accuracy, which is limited.

Testing Relative Sensitivity



- Click the ***Relative Sensitivity Icon*** and press ***OK*** (or double-click the Icon).



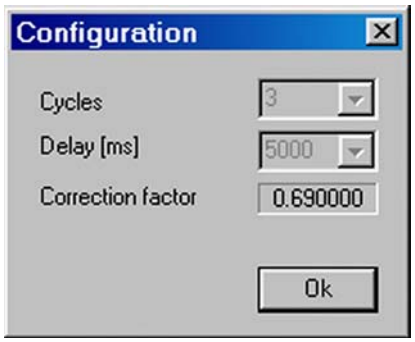
- The cups with the corresponding masses and resistor values are shown.

- On the "Window" toolbar, the ***Relative Sensitivity Icon*** becomes visible.

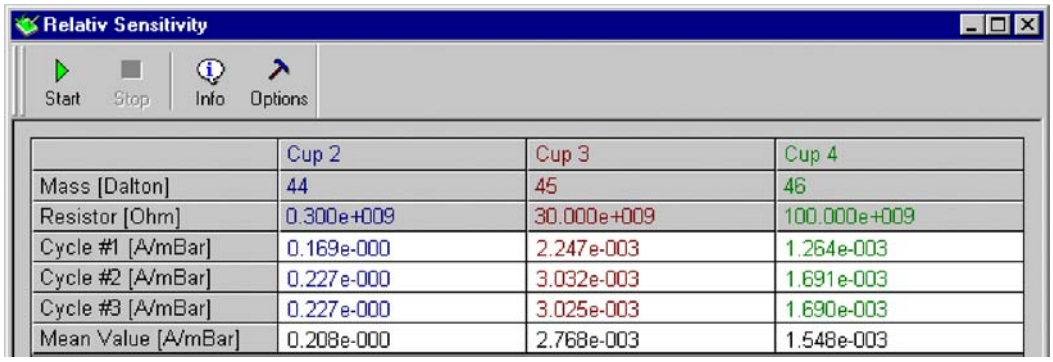
- Press the ***Options*** button.

- Press ***OK***.

  Experienced users can change the values via the ***Advanced Mode***.



- Click the ***Start*** button.



	Cup 2	Cup 3	Cup 4
Mass [Dalton]	44	45	46
Resistor [Ohm]	0.300e+009	30.000e+009	100.000e+009
Cycle #1 [A/mBar]	0.169e-000	2.247e-003	1.264e-003
Cycle #2 [A/mBar]	0.227e-000	3.032e-003	1.691e-003
Cycle #3 [A/mBar]	0.227e-000	3.025e-003	1.690e-003
Mean Value [A/mBar]	0.208e-000	2.768e-003	1.548e-003

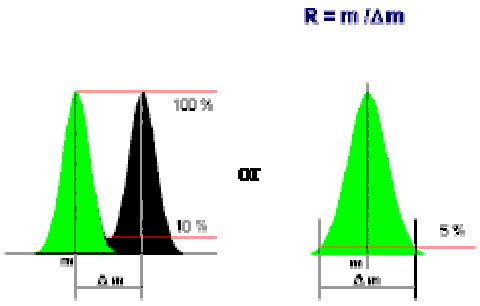
- Finally, for each cup, the values of three cycles and their mean are displayed.

- When closing the "Relative Sensitivity" window you are asked, whether to save the changes.

7.2.8 RESOLUTION

Resolution describes the masses, which can be separated from each other (i.e. the minimal relative distance between two masses, which can be resolved). Different definitions of resolution are used in mass spectrometry.

- The **10%** valley definition, commonly used for double focusing sector field mass spectrometers, means:



Resolution can be defined as:

1 Mass divided by the mass difference of two neighboring peaks, if the valley between the peaks drops to 10% of the peak height, **or** as

2 Mass divided by the peak width (in Dalton) at 5% of peak height.

According to the 10% valley definition, it is dimensionless (given in m/Δm):

$$R = \frac{m}{\Delta m}$$

where:

R: Resolution
m: Mass of the interesting isotope
Δm: Mass difference between neighboring peaks

In ***Dual Peak mode***, the distance between Peak Centers of two neighboring peaks is measured [Da]. The peak width of one peak is measured at 5% peak height.
In ***Single Peak mode***, the distance between neighboring peaks is set to 1 Da. In both modes, Resolution can be calculated as follows (the mass difference is usually 1 Da):

$$R = \frac{m}{\Delta m}\,\frac{a}{b}$$

where:

a: distance between peak centers

b: peak width of the isotope of interest

Resolution is determined using the narrowest cup (i.e. usually Cup 2). Mass 45 is used.

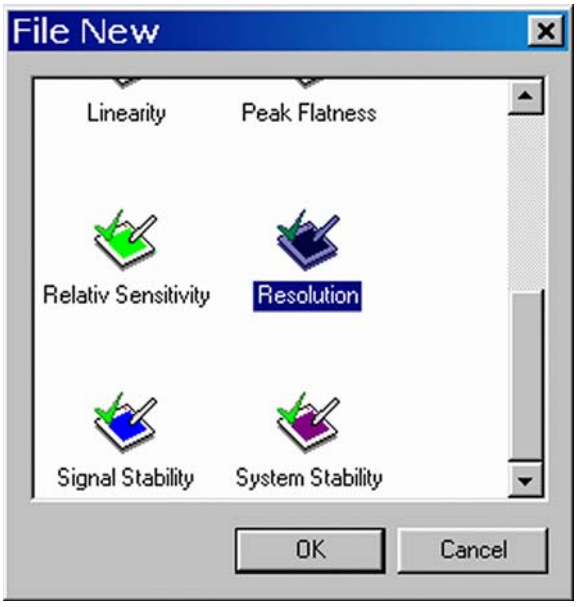
Example

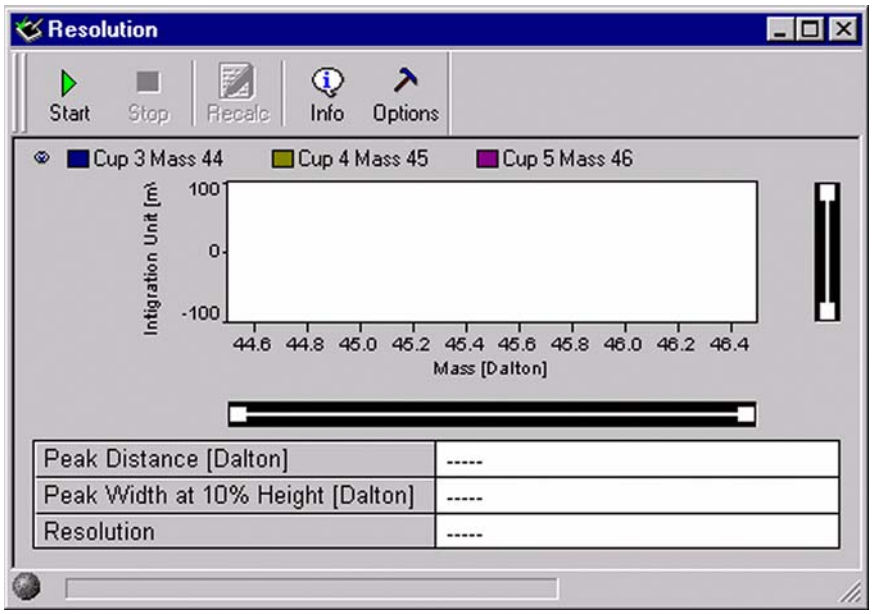
For a resolution of 88 and mass 44, a peak with a distance of (44/88) Da = 0.5 Da could be resolved using the 10% criterion.

Start mass and end mass of the magnetic field scan can be edited. In case of CO_2, the mass ranges from about 43 to 45.5. The BDAC values referring to the masses 44 and 45 are determined and the Resolution is calculated.

Testing Resolution

- Click on the ***Resolution*** Icon and press ***OK*** (or double-click the Icon).

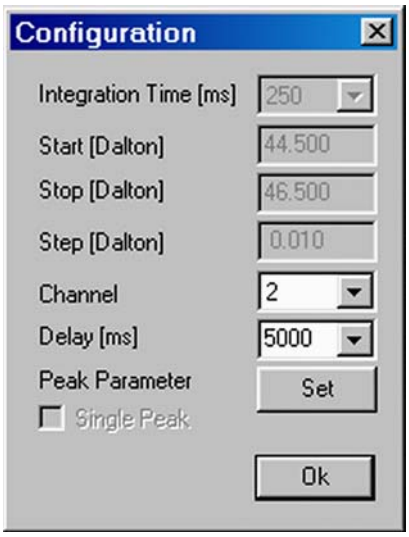


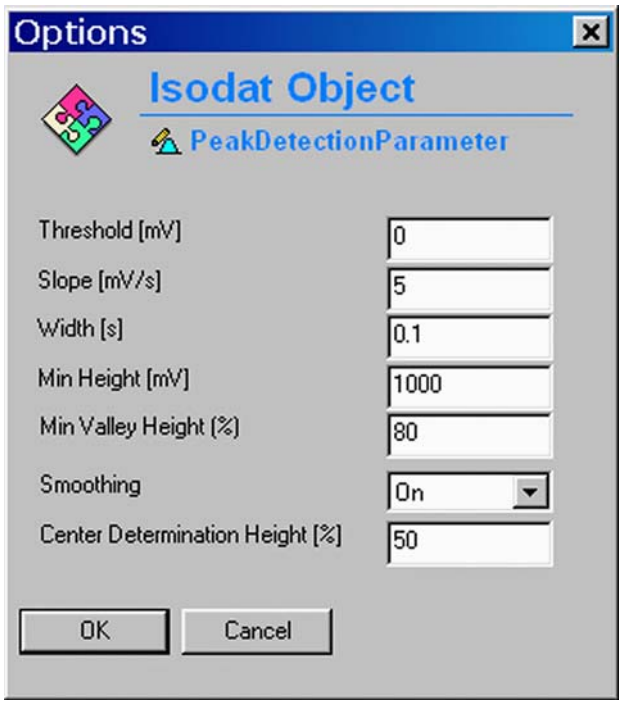


- On the "Window" toolbar, the ***Resolution*** Icon becomes visible.

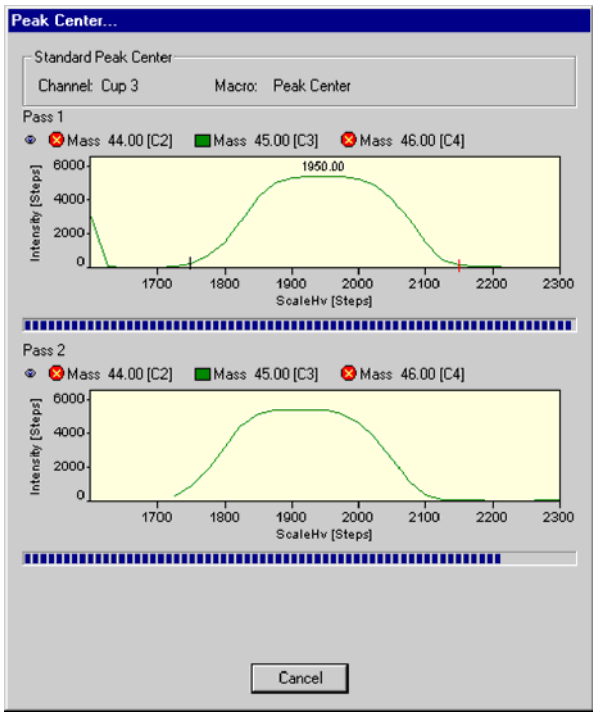
- Press the ***Options*** button.

- Accept the defaults or from the pulldown menus, select a Channel and a Delay value between 100 ms and 20000 ms.
- If you simply want to accept the Peak Detection parameters without a glance, do not press the ***Set*** button. Instead, click ***OK***.
- However, as a sophisticated user, you may want to look at the Peak Detection parameters. To do so, press the ***Set*** button.

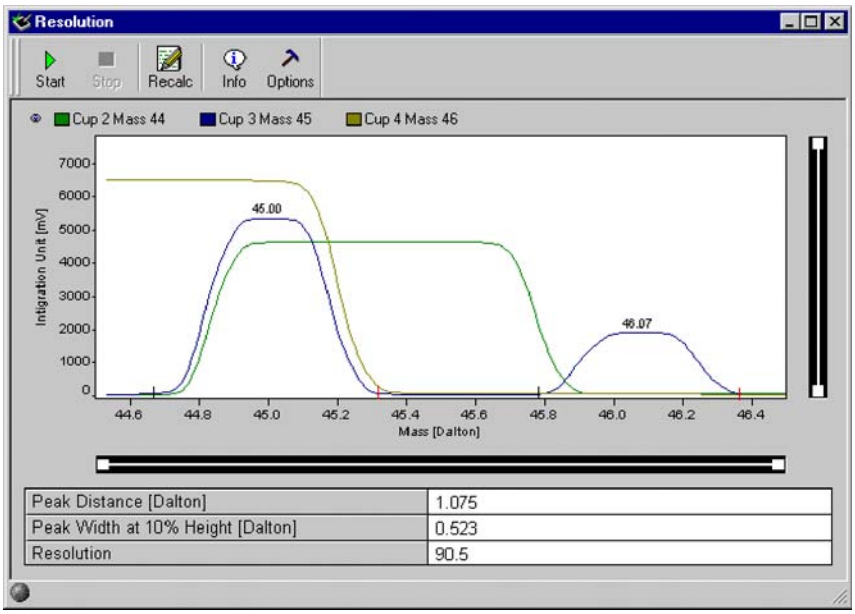




- Change the values or accept the defaults.
- Then click ***OK***.
- Press the ***Start*** button.



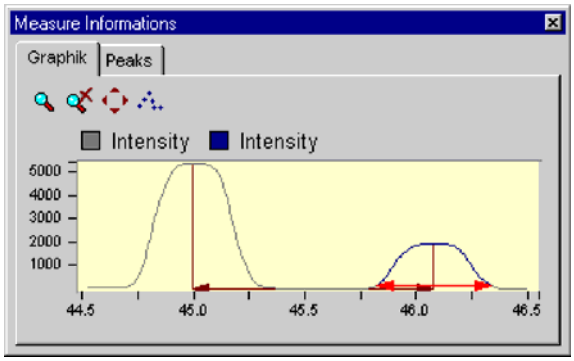
- Testing starts with a Peak Center on the selected cup (e.g. mass 45, cup 3).
  - If no peak could be detected, the measurement is stopped.
  - If not enough peaks could be detected, an error message occurs.



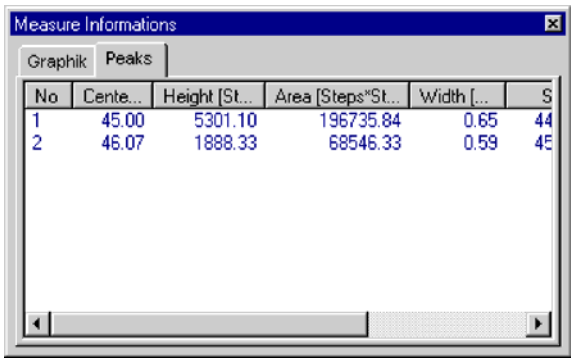
- Finally, the values of Peak Distance [Da], Peak Width at 10% Height [Da] and Resolution are displayed.

- Click the ***Info*** button.

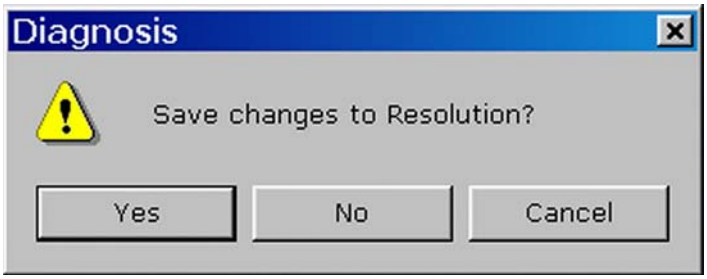
- Press the ***Graphik*** tab.



- Press the ***Peaks*** tab.



- When closing the "Resolution" window you are asked, whether to save the changes.



7.2.9 SIGNAL STABILITY

Signal Stability describes the stability of the intensity (i.e. peak height). The intensity on top of the peak is measured for a limited period of time (e.g. 5 min). Note the similarity to the System Stability, but here, the stability is not measured at the peak flank, but at the peak center. The value of Signal Stability should be about $2*10^{-4}$ (for 5 min).
Two results are obtained:

- Slope of the regression line (normalized by the intensity)
- Standard deviation of the regression line (normalized by the intensity)

Testing Signal Stability requires a signal of 3 V or more!

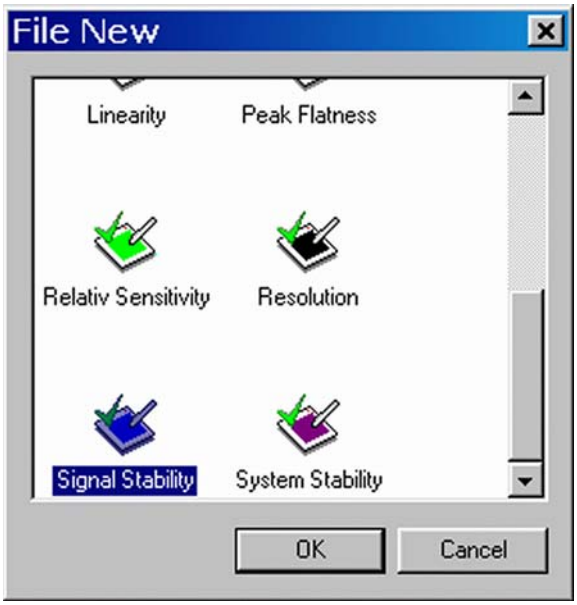
SOURCES OF ERROR

- The slope is usually due to gas consumption during measurement. However, it should be checked, if an unusual result is obtained.
- Instabilities of the emission may cause an unstable signal although a stable high voltage and magnetic field are given.
- Pressure fluctuations (check the oil of the forevacuum pumps!) or temperature fluctuations particularly at the crimps.

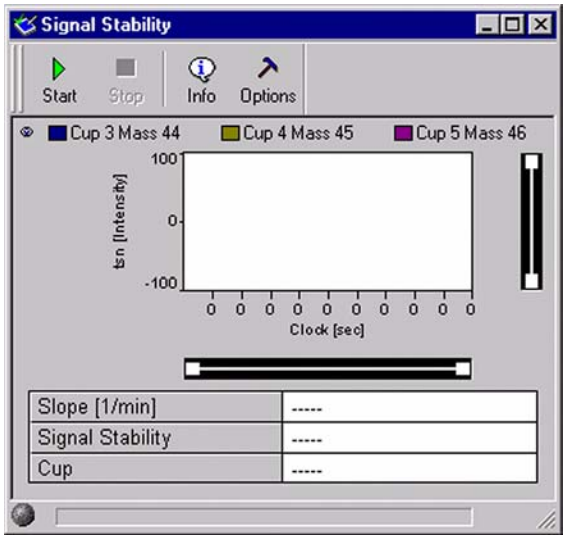
DIFFERENCES TO ISODAT OLD

1. In ISODAT Old, maximum deviation is reported instead of standard deviation.
2. However, for a statistical process standard deviation is more relevant as it is less sensitive to measuring time and outliers.
3. Conversion of results to the ISODAT Old scale: Multiplication by a factor of approximately four.

Testing Signal Stability



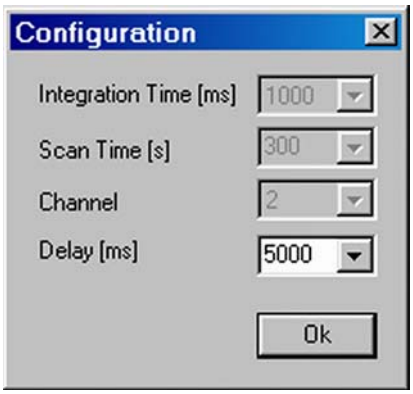
- Click the ***Signal Stability*** Icon and press ***OK*** (or double-click the Icon).



- On "Window" toolbar, the ***Signal Stability*** Icon becomes visible.

- Press the ***Options*** button.

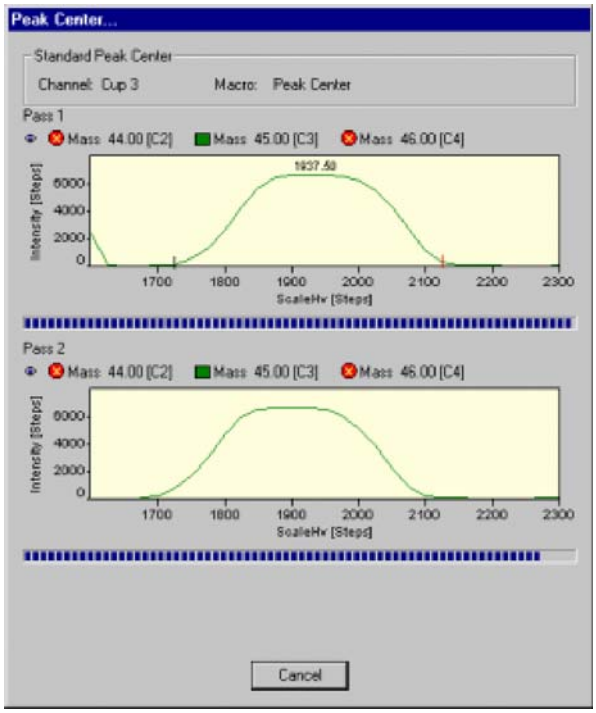
- From the pulldown menu, select a delay value between 100 ms and 20000 ms.

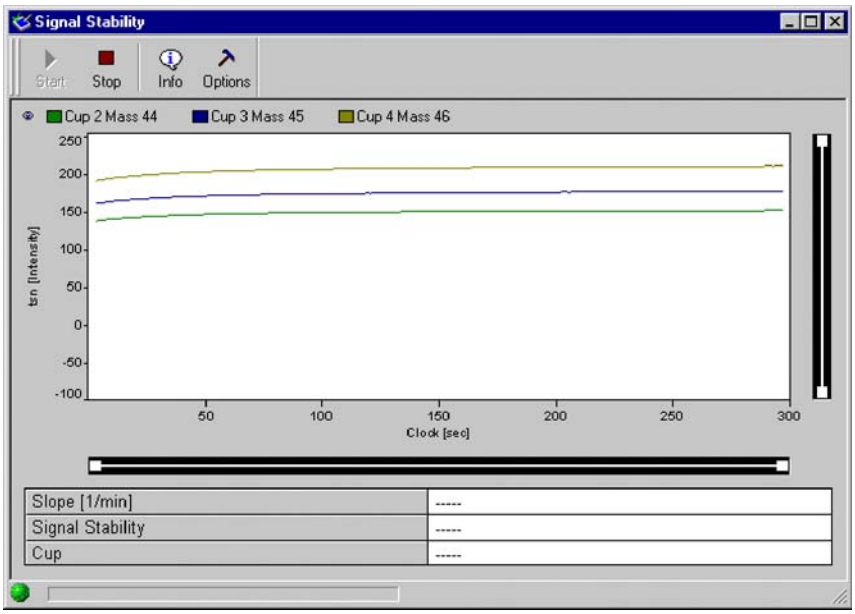


- Click ***OK***.

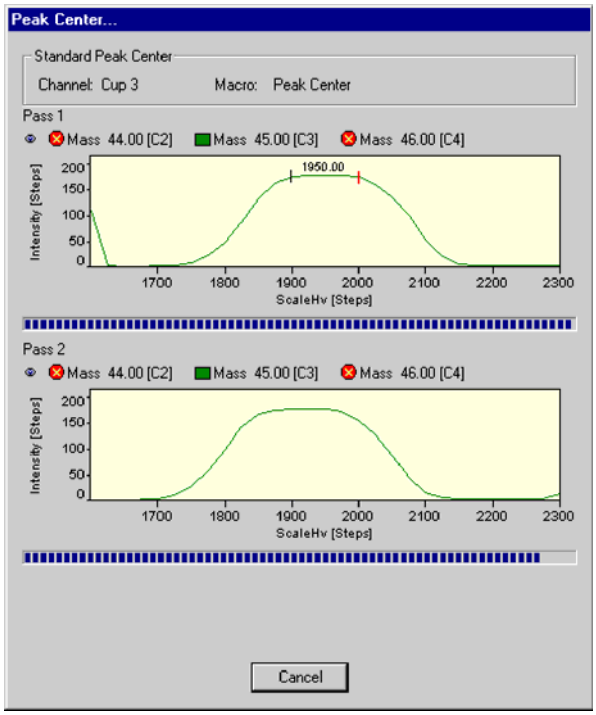
- Press the ***Start*** button.

- Testing starts with a Peak Center on the selected Cup (e.g. mass 45, Cup 3).





- Another Peak Center is performed.



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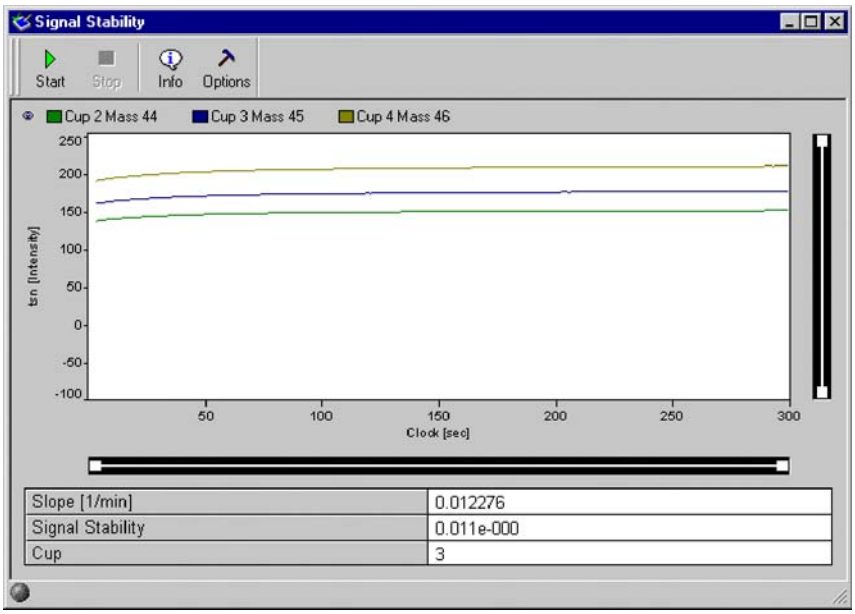
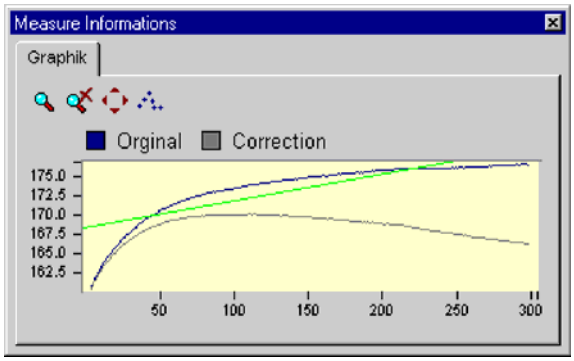


- Finally, values of slope [1/min], Signal Stability and the Cup number are displayed.

- Press the ***Info*** button.

- Press the ***Graphik*** tab.



- When closing the "Signal Stability" window you are asked whether to save the changes.

7.2.10 SYSTEM STABILITY

System Stability informs about high voltage stability and thus magnetic field stability. Already small variations of high voltage or magnetic field dramatically influence signal intensity: They cause peak shifts. The fluctuations of high voltage or the magnetic field strength are measured at the peak flank, because they exert a much higher impact on peak intensity at the flank than on top.

The System Stability test comprises the following steps:

- Determination of peak center and peak flanks
- Set magnetic field to 50% of peak height (at the peak flank).
- Measurement of signal intensity (high voltage fluctuation) at the peak flank for a defined period of time (e.g. 15 min).
- New peak center procedure
- Calculation of System Stability [min^{-1}] and Relative Mass Drift [min^{-1}] (either electronic or to magnetic drift) using the slope of the peak flank.

Testing System Stability requires a signal of 3 V or more!

Two results are obtained:

1. Slope of relative mass drift vs. time (time drift)
2. Standard deviation of this slope (scatter of the mass)

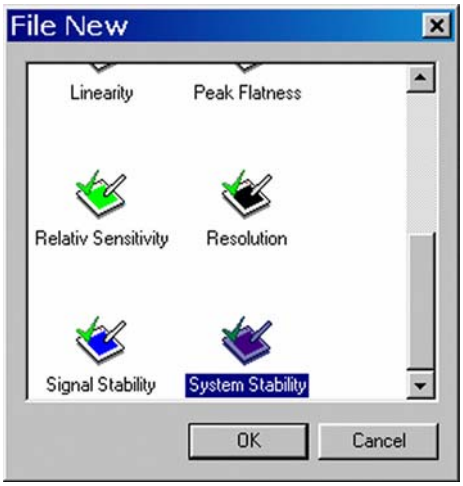
A value of $5*10^{-4}$ measured for a period of 15 min reflects a good System Stability.

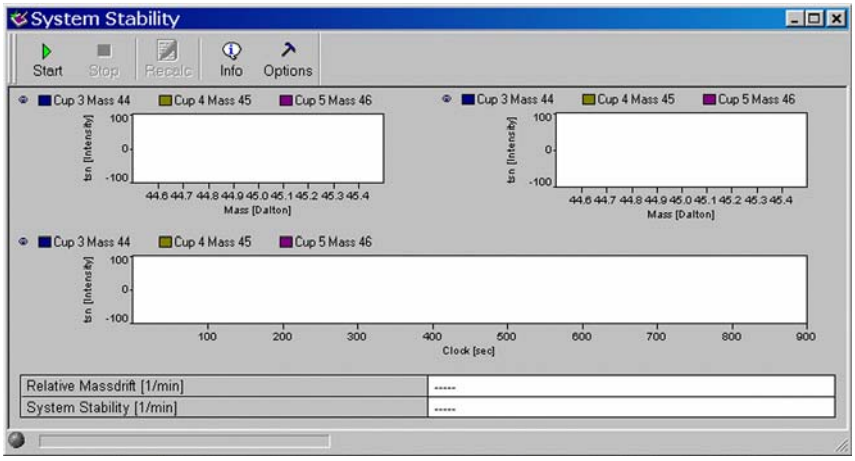
Differences to ISODAT Old

- The slope is a new, but important parameter, as it shows the effect of slow fluctuations in temperature, supplied voltage etc.
- In ISODAT Old, maximum deviation is reported instead of standard deviation. However, for a statistical process, standard deviation is more relevant as it is less sensitive to measuring time and outliers.
- Conversion of the Results to the ISODAT Old scale: multiplication by a factor.

Testing Signal Stability

- Click the ***System Stability*** Icon and press ***OK*** (or double-click the Icon).





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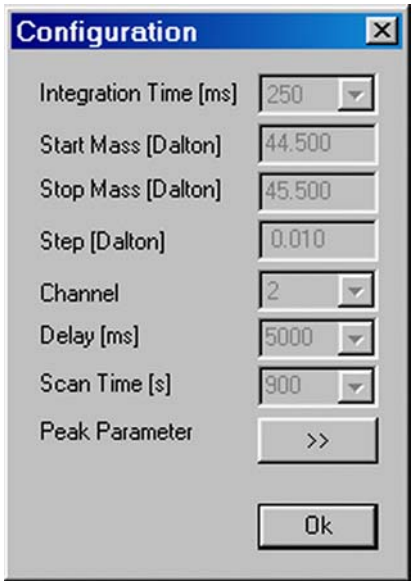
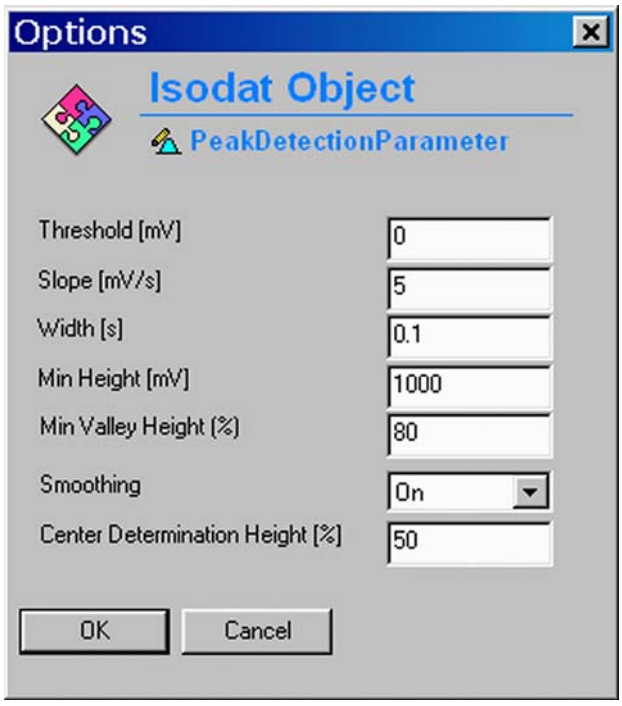
- On the "Window" toolbar, the ***System Stability*** Icon becomes visible.

- Press the ***Options*** button.



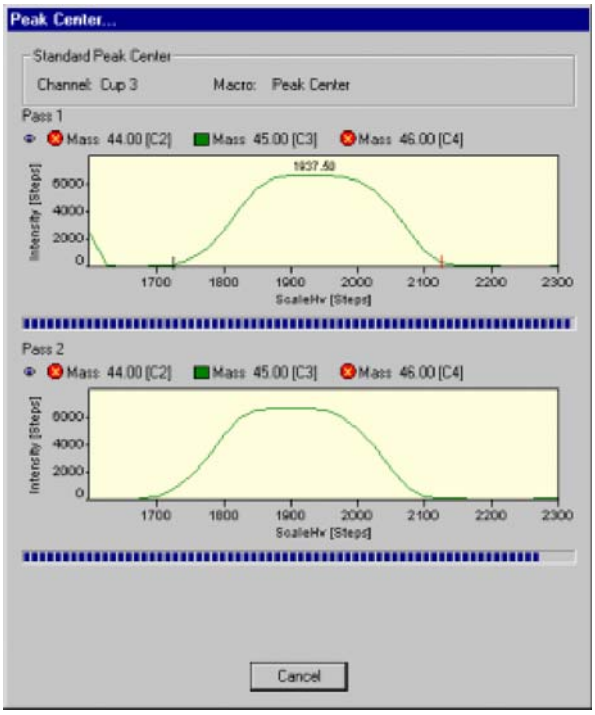
- If you simply want to accept the Peak parameters without glancing at them, do not press the ***Peak Parameter*** button. Instead, click ***OK***.
- However, as an experienced user, you may want to look at the Peak parameters. To do so, press the ***Peak Parameter*** button.
- Experienced users may further want to change the gray default values (from Integration Time to Scan Time). This can be achieved via the ***Advanced Mode***.

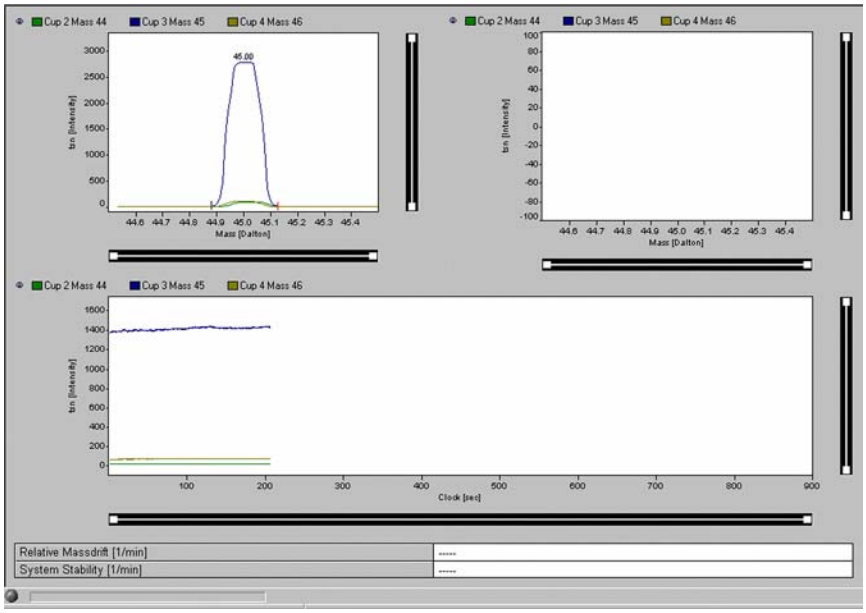


- Make your changes or accept the defaults.
- Then press ***OK***.

- Press the ***Start*** button.

- Testing starts with a Peak Center on the selected Cup, e.g. mass 45, Cup 3.





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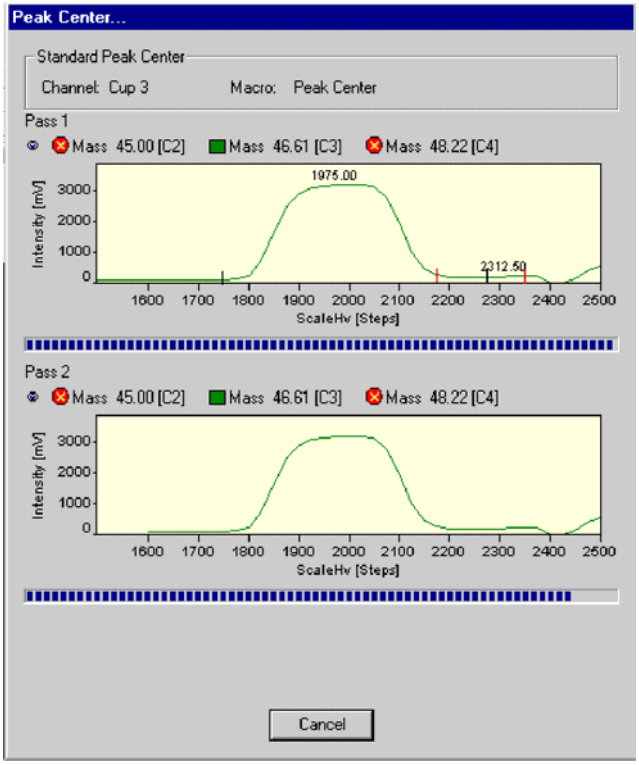
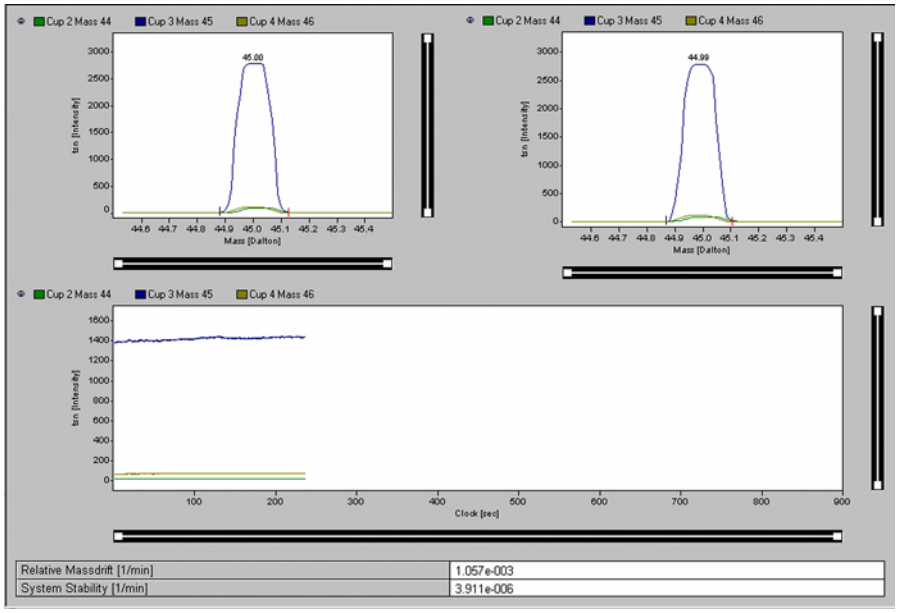


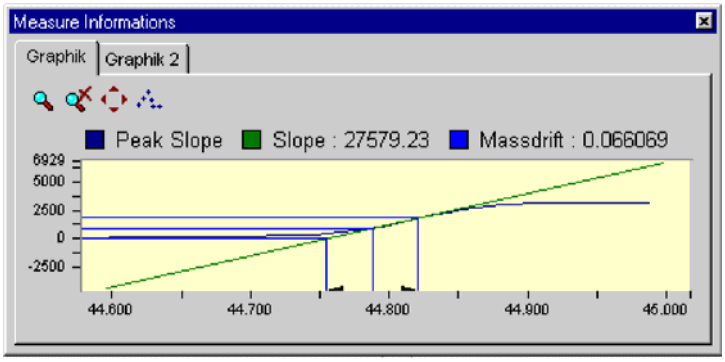
- Testing continues with another Peak Center on the selected Cup.



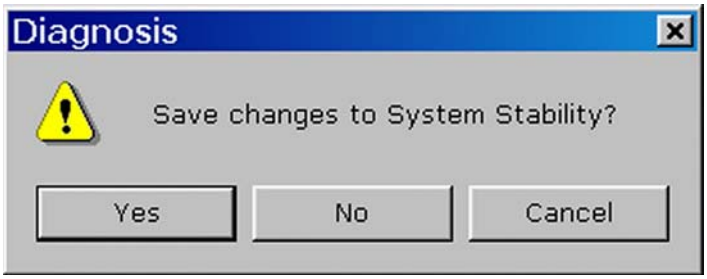
- Finally, values for System Stability [min^{-1}] and Relative Mass Drift [min^{-1}] are displayed.

➢ Press the ***Info*** button.

➢ Pressing the ***Graphik*** tab, the diagram for calculating Slope and Mass Drift is shown together with the respective values.



➢ When closing the "System Stability" window you are asked whether to save the changes.



7.3 CONVERSION TO OTHER GASES

Depending on the number of collector cups installed it may be necessary to switch the feedback resistor of an amplifier when intending to analyze different gases. This is done automatically. An example of a collector assignment with six cups is given below:

Example: Assignment of a collector with six cups (i.e. MEMCO 6)

Measuring Channel	1	2	3	4	5	6
VF-converter element and mass	1	2	3	4	5	6
N_2	28	29	30			
CO_2				44	45	46
SO_2			64	66		

With this combination, there is no need to switch the feedback resistors when changing from CO_2 analysis to N_2 analysis. The switching is done automatically by ISODAT NT changing the cup configuration. Only in case of switching to SO_2 analysis, it is necessary to switch the amplification factor (done automatically by switching the Gas Configuration).

7.4 HEATING INLET CAPILLARIES

NOTE: ***The heating system can be regulated via ISODAT NT.***

NOTE: ***ISL Scripts can be used to create time programs for the heating process***

If a contamination of the instrument is detected, also the inlet capillaries should be heated. Together with the instrument, a transformer (90VA / 220V/ 7. 7 V/12 A) is supplied for heating purposes. To heat all capillaries simultaneously, it is advisable to acquire additional heating transformers.

Step 1 Before heating the capillary itself, the surrounding of the capillary has to be heated to approximately 80 °C for about 30 to 60 minutes, i.e. the valves of the inlet system (incl. the valves of a Multiport, if in use), the changeover valve, the ion source and the analyzer housing.

During the heating period, all valves must be open. Swagelok connectors should be heated separately for a short while using a flame or a heat gun.

Step 2 Plug two cables parallel to one socket of the transformer and clip on the ends with the alligator clips to each end of the capillary to be heated or to the related Swagelok connector.

Plug the third cable to the second socket and connect the clip to the middle of the capillary. Capillaries supplied by Thermo Finnigan MAT have a metal sleeve attached in the middle providing better electrical contact.

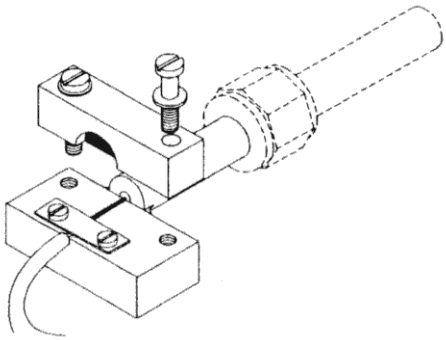
CAUTION: ***After the capillary has been wired make sure, the capillary (with or without insulation) has no contact to any plastic surface of hoses, housings, cables etc. to avoid melting or smoldering caused by a hot capillary.***

Step 3 Connect the transformer to the mains and switch it on. The heating phase should be controlled by monitoring the signal intensity of H_2O (mass 18, measured on the channel for mass 45; see “Instrument Control”).

It is recommended to heat also the crimped part of the capillary separately for a few minutes using a flame or a heat gun.

NOTE: ***Before removing the upper crimp block mark the parts of the crimping device to avoid mismatch when reassembling.***

Fig. 7-20 Crimping device at the end piece of a capillary



Step 4 When fitting the crimp block again, make sure the capillary is exactly placed in the grove of the base and the die of the upper block in the crimp of the capillary. After heating the capillary, the flow resistance of the crimp has to be checked and reset to 1 Volt per 10 mbar if required (see “Replacing an Inlet Capillary”).

During the start phase of the heating, the signal intensity increases but it decreases and stabilizes later. The best results of decontaminating capillaries are achieved by heating for approximately 6 to 8 hours. With stabilized signal intensity lower than the first signal, a successful decontamination can be assumed. The result can be checked by a zero-measurement, i.e. measuring the same gas on sample and standard side.

7.5 REPLACING AN INLET CAPILLARY

Replacement of an inlet capillary may become necessary in case of contamination or mechanical damage. After replacement, the flow rate of the new capillary has to be set by crimping. The crimping device consists of two metal blocks. The base is attached to the end piece of the new capillary, which has to be fitted to the changeover valve. The second block, to be bolted on top of the base block, holds a metal pin in a spacing, which will squeeze the capillary when bolting the two blocks together. To replace a capillary proceed as follows:

NOTE: ***Make sure that all valves are closed before venting the surrounding area of the capillary, which is to be exchanged.***

Step 1 Vent the parts of the inlet system and the changeover valve, which are connected by the capillary.

Step 2 Loosen the Swagelok fittings holding both end pieces of the capillary to be exchanged. New capillaries are delivered with close ends. Use a diamond file to cut a capillary end at opposite sides before breaking off the tip. Then smoothen the end of the capillary.

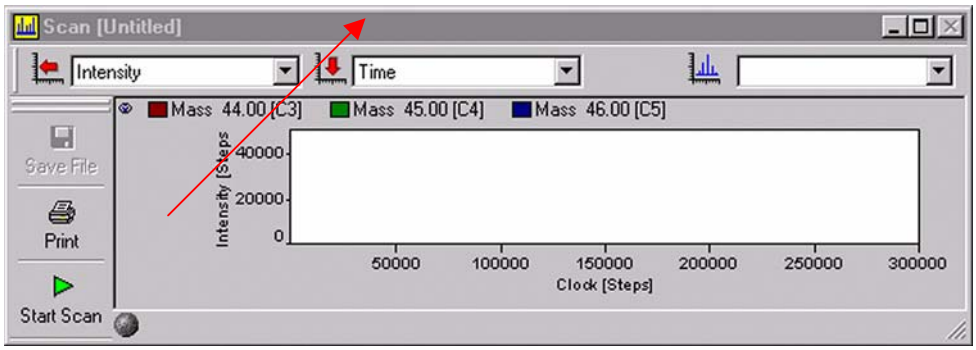
Step 3 Fasten the end pieces of the new capillary with the Swagelok connectors. The end piece with the crimp block has to be connected to the changeover valve. Fasten the upper crimp block loosely onto the base with the capillary.

Step 4 Pump out the inlet system.

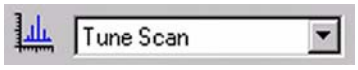
Step 5 Admit a proper amount of CO_2 into the inlet system, so that the storage reservoir pressure is about 20 mbar on both sides of the capillary.

Step 6 Start ISODAT NT's ***Instrument Control***.

- Activate the ***Scan*** window by a click on its frame.



- Select ***Tune Scan***.



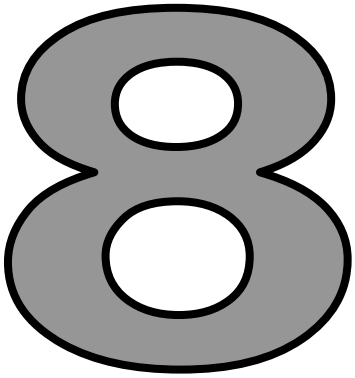
- Press the ***Start Scan*** button.

Step 7 Tighten the screws of the crimp block carefully and squeeze the capillary until the output signal reaches 1 Volt per 10 mbar with CO_2 used for measurement.

Step 8 After crimping the capillary has to be heated (see Chapter 7.2.12 “Heating Inlet Capillaries”).

MAT 253

OPERATING MANUAL



TECHNICAL INFORMATION

Issue 4/2002

Thermo Finnigan

8.1 INTRODUCTION

This chapter contains information required for maintenance and repair of the ***MAT 253*** mass spectrometer. It also includes schematic drawings and spare parts lists as well as instructions for the installation of various hardware drivers.

8.1.1 GENERAL REMARKS

The ***MAT 253*** mass spectrometer contains elaborate and expensive components. Only qualified and skilled personnel, therefore, should perform servicing. It is recommended that the Thermo Finnigan MAT service by called if there are any uncertainties or if difficulties arise. It is further recommended to use original Thermo Finnigan MAT spare parts only. Note that many adjustments can be made only by the use of special tools and instruments, which are not supplied with the system. (See for references within the manuals.)

Before starting maintenance and repair, please read the appropriate chapters of the manuals.

Before calling the Thermo Finnigan MAT service, please try to localize the defect! A precise description of the defect will ease the repair and reduce the costs.

WARNING: ***Some parts of the MAT 253 mass spectrometer are at dangerously high voltages! Therefore, opening the electronics cabinet is only allowed for maintenance purposes by qualified service personal.***

CAUTION: ***When replacing fuses, only use the correct types!***

Be careful when servicing the vacuum system. Abrupt opening to atmosphere might destroy the filament or damage the collector system and other expensive parts.

When working with solvents might and sample residuals, please consider your regional safety instructions!

8.1.1.1 MAINTENANCE

To maintain optimum mass spectrometer performance, the user must perform routine preventive maintenance. Please, see chapter 8.3 of this manual.

8.1.1.2 SPARE PARTS LISTS

In the diagrams attached to the spare parts lists, the individual spare parts are shown with item numbers. The parts are classified by these item numbers in the corresponding lists and provided with a designation and a part number.

When ordering a part, always give the designation and the part number. Please, also provide the service number of the instrument in question. In this way, the processing of your order will be expedited.

8.1.1.3 SERVICE INSTRUCTIONS

Wherever necessary, service instructions supplement the spare parts lists.

8.1.1.4 REPAIR REQUEST FORMS

If parts are to be returned to the factory for repair, please fill in the repair request form and enclose it with the shipment. In this way, you will help to avoid delays frequently caused by inquiries, which become necessary if parts are returned to us without reference to the instrument to which they belong or without any details about the faults observed.

8.1.1.5 SHIPMENT LISTS

Because of our modular instrument concept it might happen that parts are listed which are not supplied with your instrument. Please note that listing a part does not imply that this part is necessary required for your particular instrument configuration.

8.1.1.6 TECHNICAL MODIFICATIONS

Continuous improvement of the performance of our products may result in technical modifications.

We are always striving to have the spare parts lists up-to-date.

8.1.2 SAFETY RULES FOR THE REPAIR SERVICE

Thermo Finnigan MAT mass spectrometers frequently are used for analyzing materials, which are insalubrious. In these cases, usually certain parts of the system will be contaminated.

To protect the health of our employees we ask you for some special precautions when returning those parts for exchange or repair.

Mass spectrometer parts, which have been contaminated by hazardous materials, we can accept only if they have been decontaminated prior to return. Hazardous materials are those materials listed up on the MAK list (Maximale Arbeitsplatzkonzentration) and on the EPA (Environmental Protection Agency) priority list.

Additionally such materials are enclosed which due to their structure and the applied concentration might be toxic or which in publication are reported to be toxic. Finally, such materials are concerned which in combination with other present materials will generate synergetic hazardous effects.

Please, take care, that vacuum pumps and all other parts which had been in contact to hazardous materials, will be properly **decontaminated** prior to return to Thermo Finnigan MAT.

Parts contaminated by radioisotopes are not subject to return to Thermo Finnigan MAT neither under warranty nor under the exchange part program.

When returning parts to Thermo Finnigan MAT the use of our repair-covering letters is obligatory.

Please, state by your signature on this repair-covering letter, that the returned part had been decontaminated and is free of hazardous materials.

We hope that the analysts of our customers will understand these safety regulations.

8.2 INSTALLATION OF BOARD DRIVERS

The following pages contain descriptions for the installation of drivers for the various extension boards of the computer.

8.2.1 "SERIAL SOLUTIONS" COM PORT EXTENSION BOARD DRIVER

The PCI Dual RS 232 Card is already part of your computer. Its driver by "Serial Solutions Products" makes available two additional COM Ports. Thus, install it only, if your computer is equipped with merely one COM Port.

Install it once only, namely when you newly install your operating system.

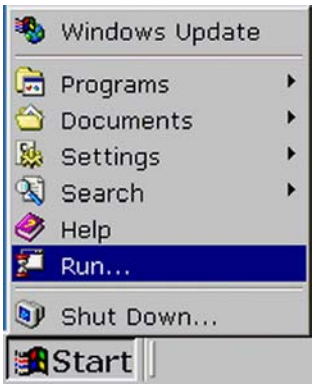
Close all other applications! Otherwise, an error message might occur.

NOTE: ***If your computer hangs up during either installation or reboot, shut it down and try to install again.***

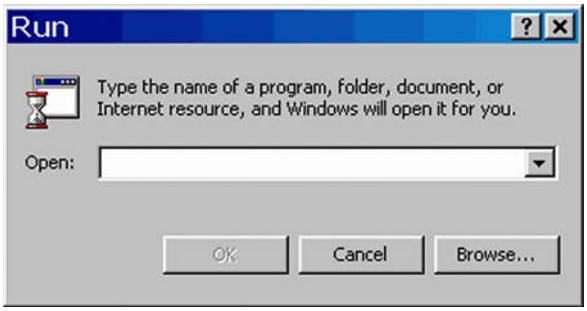
If your system hangs up again, plug the PCI Dual RS 232 Card to another slot. Then only start your system without reinstallation.

- To install the driver, insert the "Serial Solutions" CD into your drive and close it. The CD is shipped with your system.

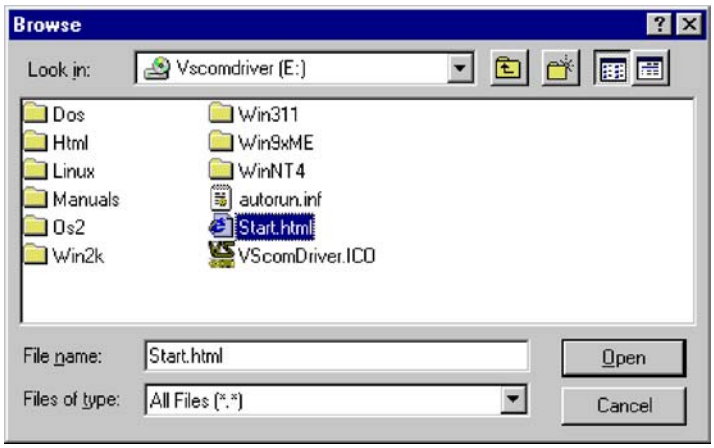
- If Auto Run is deactivated on your computer, press the ***Start*** button and then ***Run***. If Auto Run is activated on your computer, proceed with Step 2 **(8)**.



- Click ***Browse***.

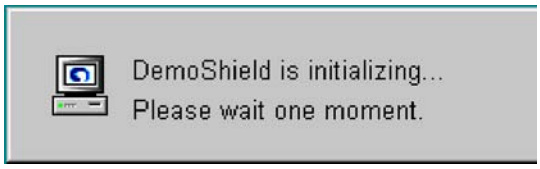


- Select your CD drive e.g. "E:\Vscomdriver". From the "Files of type" pulldown menu choose "All Files (*.*)". Click the file "Start.html" and then ***Open*** (or double-click "Start.html").



- Press "***OK***".

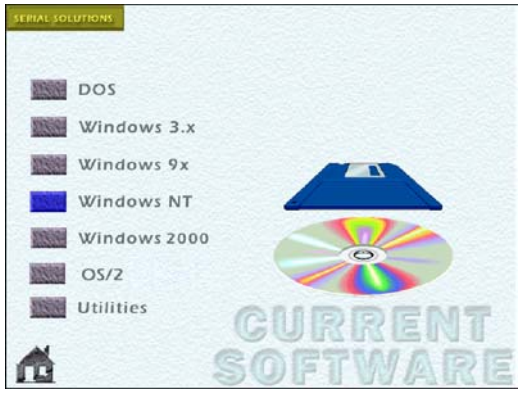
- Wait, while the DemoShield is initializing.



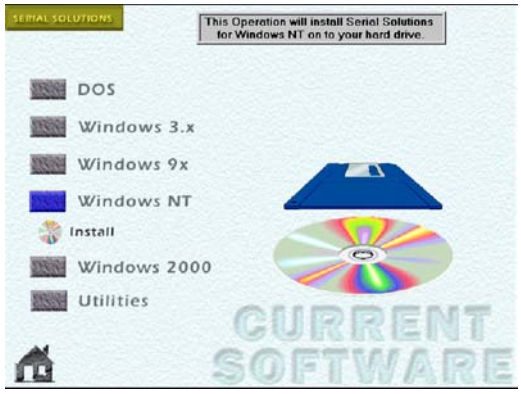
- On the Installation Screen, click ***Software***.

➢ Press ***Current***.

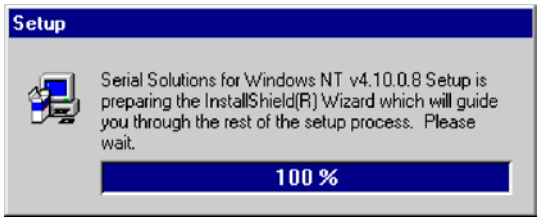
➢ Click ***Windows NT***.



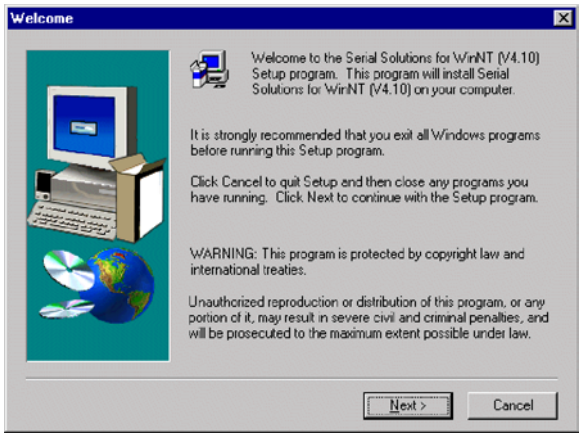
➢ Press ***Install***.



- Wait, while Setup is preparing the InstallShield® Wizard.

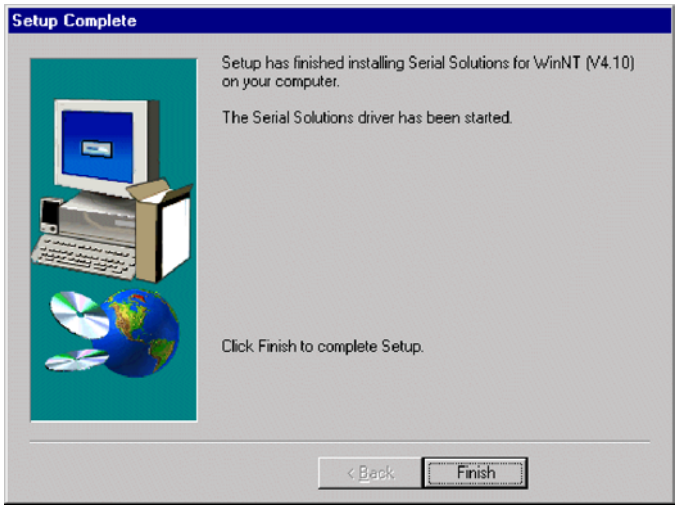


- Carefully read the instructions about closing all applications and copyright law. Click ***Next*** to continue.
  (If Setup has found a previously installed version, continue as described below).



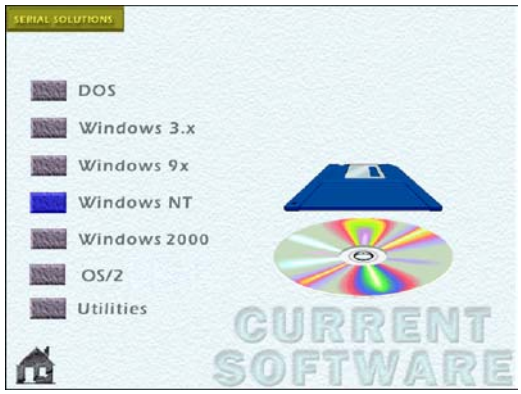
- Wait, while Setup is copying the files.

- "Serial Solutions for Win NT" has been installed on your computer.



- Click ***Finish*** to complete Setup.

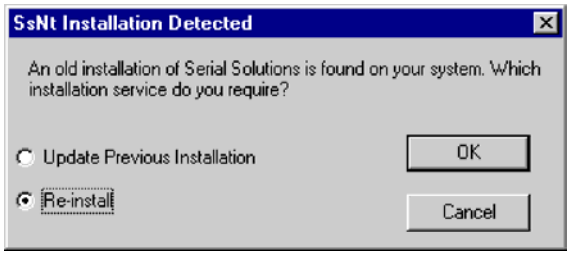
- Press the ***House*** button.



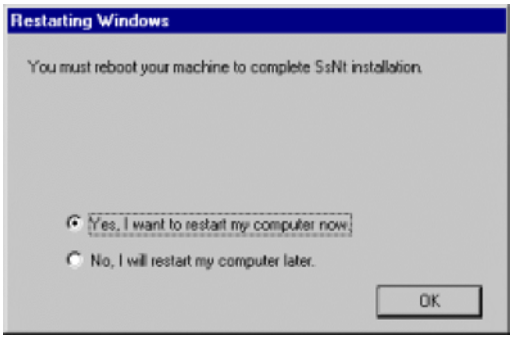
- Click ***Quit***. Then reboot your system.

8.2.1.1 REINSTALLING A PREVIOUSLY INSTALLED "SERIAL SOLUTIONS" DRIVER

- "Serial Solutions" Setup has detected a previously installed version.



- Choose ***Re-install***.



- Select ***Yes, I want to restart my computer now*** and press ***OK*** to reboot.

NOTE: ***If your computer hangs up during either installation or reboot, shut it down and try to install again.***
If your system hangs up again, plug the PCI Dual RS 232 Card to another slot. Then restart your system without reinstallation

8.2.2 INSTALLING IEEE INTERFACE BOARD DRIVERS

Some peripherals (e.g. equilibration unit, carbonate device) require the installation of an IEEE board in the computer. If this is the case, follow the subsequent instructions.

1. Install "National Instruments" PCIIA IEEE Interface Card or
2. Install "National Instruments" GPIB-PCI IEEE Interface Card

8.2.2.1 "NATIONAL INSTRUMENTS" GPIB PCIIA ISA IEEE INTERFACE BOARD

Do not use Disk Kit, National Instruments NI-488.2 Software!

During the installation of ISODAT NT, a driver for this interface card is installed. To run this card under Windows NT some settings must be changed in the BIOS.

1. Therefore, restart the computer.
2. During the initial BIOS initialization phase press F1 to enter the BIOS editor. At "Advanced", the entry "Resource Configuration" becomes visible. Here, the interrupt 7 can be locked exclusively for ISA.
3. Switch the entry from "Available" to "Reserved" (i.e. position the cursor on the line of interrupt 7 and press Enter).
   Interrupt 7 is normally reserved for printer.
4. Therefore, leave the "Resource Configuration" with Esc and enter the "Peripheral Configuration". At the entry "Parallel Port", the subentry "Interrupt" is visible. It is normally set to "IRQ7". After reserving the IRQ 7 for the PCIIA card, this entry shows an asterisk indicating a resource conflict.
5. Change the interrupt to "IRQ5". The asterisk should disappear. Leave all other parameters unchanged.
6. Quit the "Peripheral Configuration" Menu and the "Advanced" Menu by "Esc" and enter "Exit" Menu.
7. Exit the BIOS editor by "Exit Saving Changes". The PCIIA card is jumpered to interrupt 7 as default.

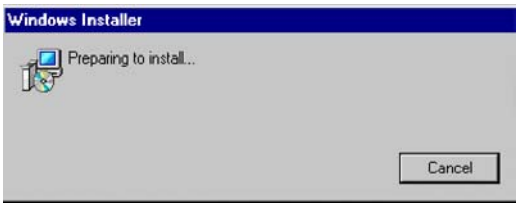
8.2.2.2 "NATIONAL INSTRUMENTS" GPIB PCI IEEE BOARD - INSTALLATION

Install the driver from the **"National Instruments" NI 488.2 CD** shipped with your system.

If **Adobe Acrobat Reader** is not installed on your computer, it can be installed later. It will be needed to read "National Instruments" Help files.

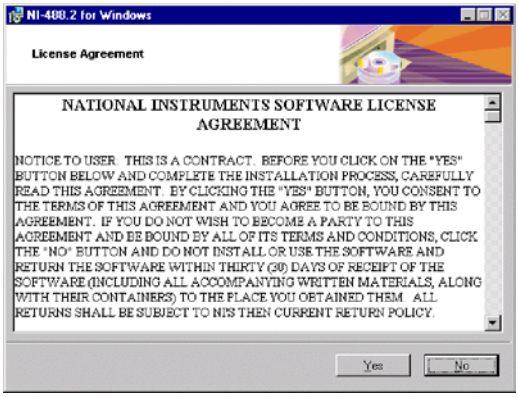
- **Close all other applications!**
  Insert the CD into your CD drive and close it. The box shown above appears.
- Click "Install NI-488.2 Software for Windows" (turning into blue).

- Wait, while Installation is being prepared.

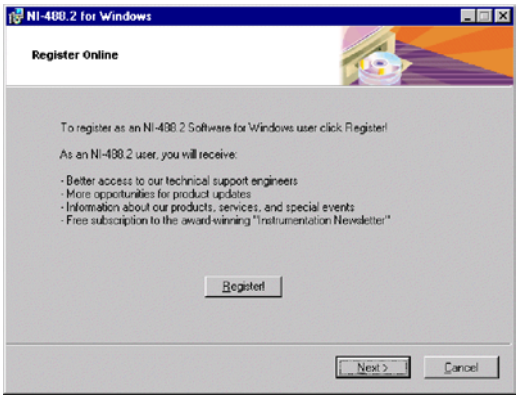


- Wait, while the NI-488.2 Installation Wizard appears and searches for previously installed applications.

- Click ***Next***.

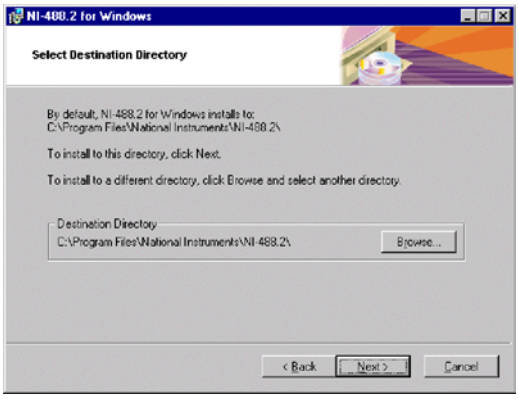


- Carefully read the "National Instruments" Software License Agreement. To continue press ***Yes***.

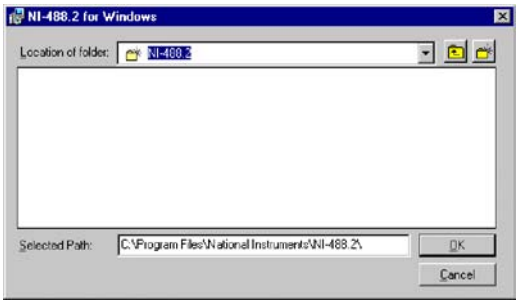


- Take your registration decision. Then press ***Next***.

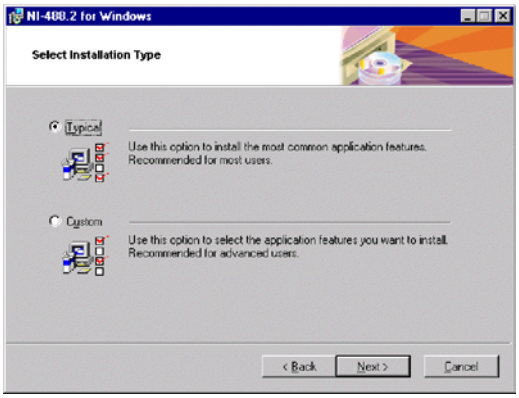
- To select the destination directory, either accept the proposed path and press ***Next*** or select another one by the ***Browse*** button.



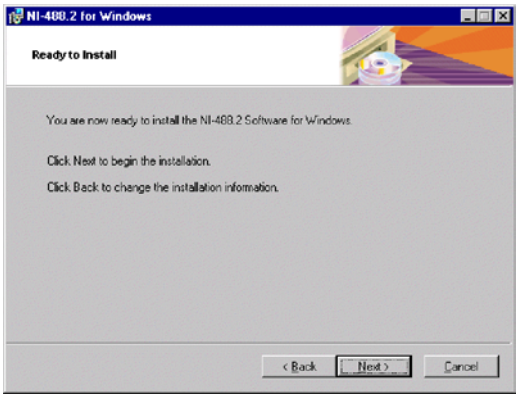
- If you pressed the ***Browse*** button (**8b**), a box appears. From the ***Location of folder*** pulldown menu, choose the alternative destination folder. Note the according entry in the ***Selected Path*** box. Finally, press ***OK***.

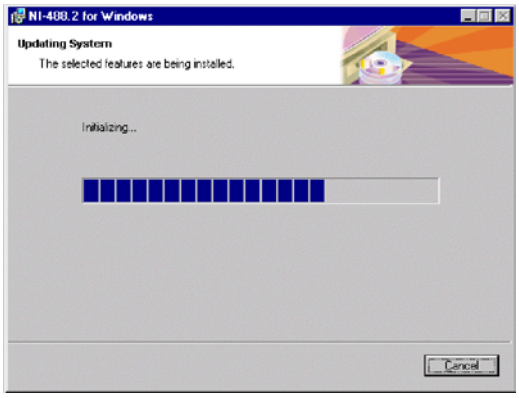


- Select the Installation type.
  Typical is recommended for most users, while ***Custom*** is recommended for advanced users. Finally, press ***Next***.

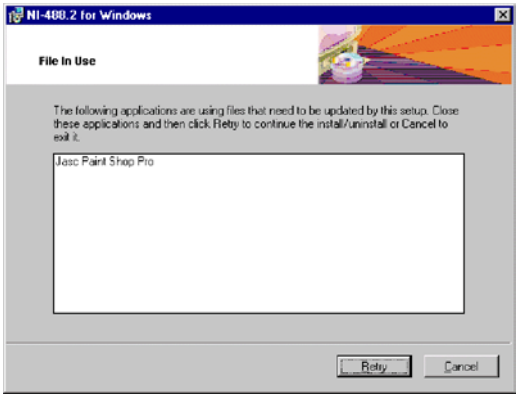


- The NI-488.2 software is now ready to be installed. To begin the Installation press ***Next***.

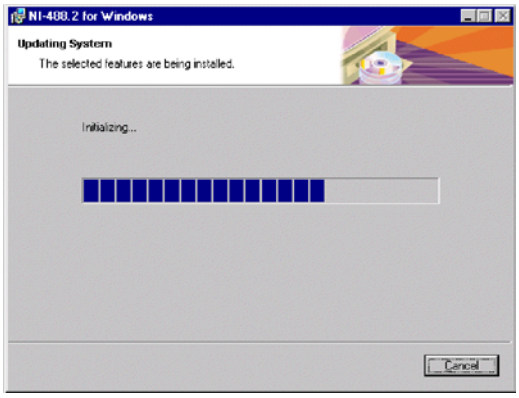




- Wait, while Initialization begins.

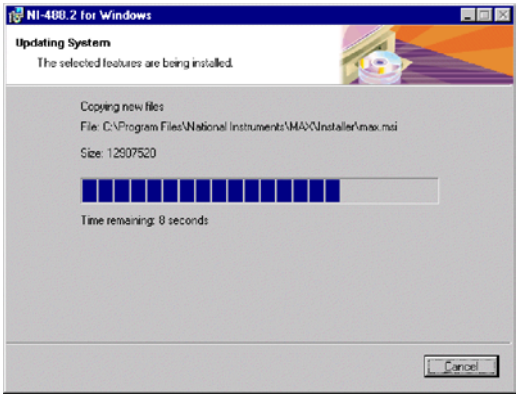


- If you did not close all other applications yet (as was recommended in **1**), you are reminded to do it now: Click on the open applications' entries right to the ***Start*** button and close all of them.
  Finally, press ***Retry***.

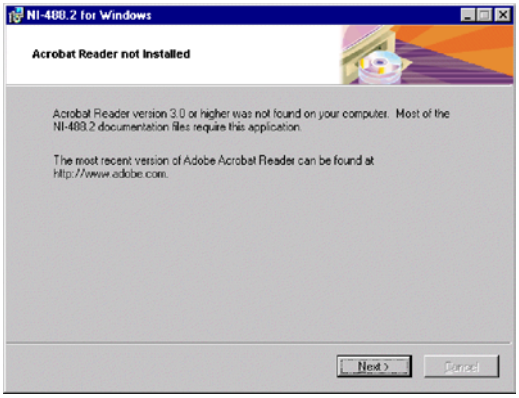


- Wait while Initialization is terminated.

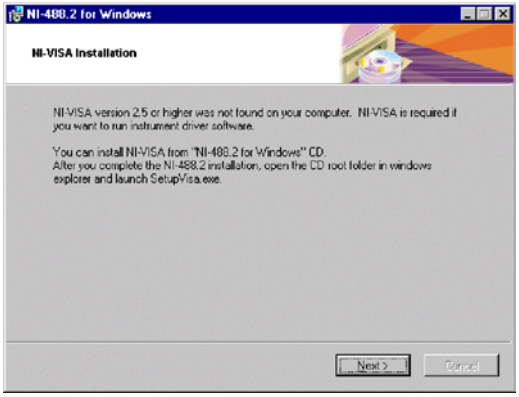
- Wait, while the files are copied.



- Possibly, you might be reminded that Acrobat Reader 3.0 (or higher) was not found on your system.
  It is necessary to read "National Instruments" documentation files, but you can install them later.
  Press ***Next***.



- This box informs about the separate NI-VISA Installation. This will be done after this installation is finished (see chapter 8.2.2.5).
  Click ***Next***.



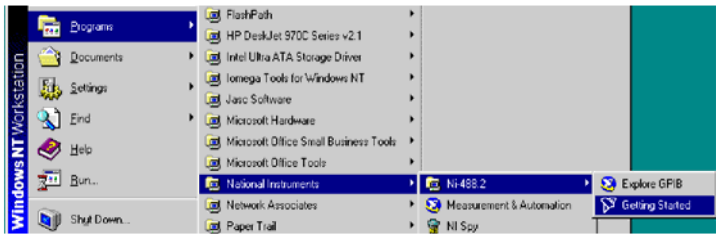
- The NI-488.2 Software has been successfully installed. Click ***Finish***.

- On your desktop, note the new NI-Icon.

8.2.2.3 "NATIONAL INSTRUMENTS" GPIB PCI IEEE BOARD - CONFIGURATION

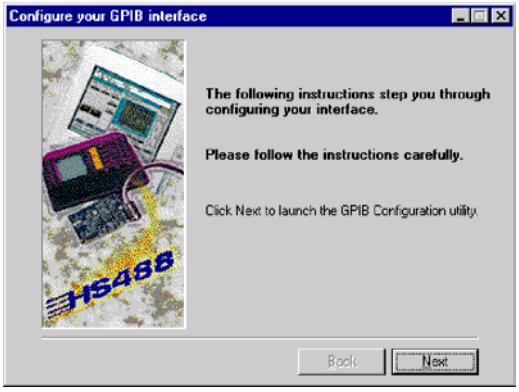
After having installed the GPIB PCI driver, **configure** it now as shown below.

- To configure it press ***Start > Programs > National Instruments > NI 488.2 > Getting Started***.

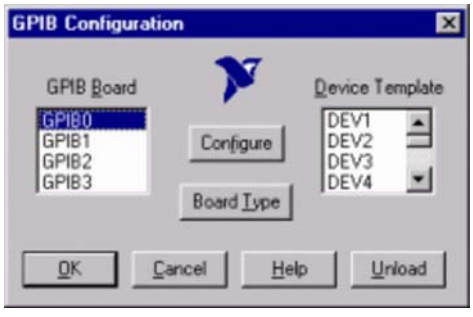


- Point with the mouse at ***Configure your GPIB interface*** and click.

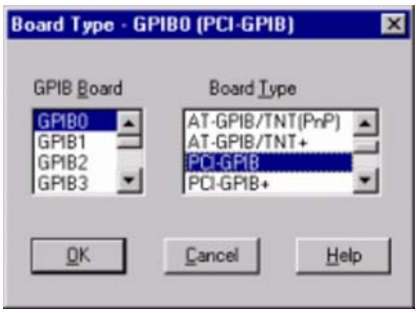
- On the next window, click on the ***Next*** button to begin the Configuration.



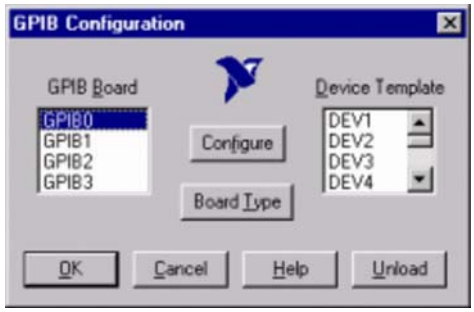
- On the following window, press the ***Board Type*** button.



- From the ***Board Type*** pulldown list on the right, select ***PCI-GPIB***. Press ***OK***.



- Press ***Configure***.



- Press ***Software >>***.

- On the next window, tick the checkboxes as shown in the figure right. Then click the ***OK*** button and confirm with ***OK*** on the following window (see above).

The configuration is finished now and the system starts the verification of the board driver (see chapter 8.2.2.4).

Under some circumstances, the following window may appear.

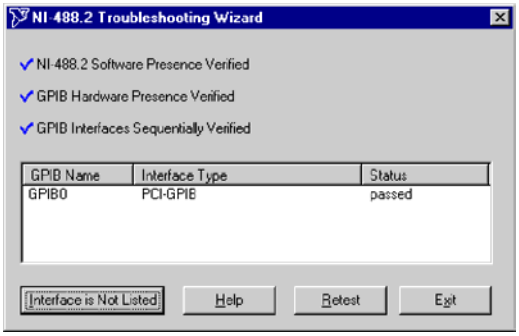
The error statement shown on the message box in the figure right indicates that the software cannot be started. Press ***OK*** to close the window.
Check whether your Interface Card is properly inserted and then repeat the configuration of the GPIB-PCI driver.

8.2.2.4 "NATIONAL INSTRUMENTS" GPIB PCI IEEE BOARD - VERIFICATION

After installing and configuring your GPIB-PCI driver, **verify** it now as shown below.

- Point at ***Verify your hardware and software installation*** and click.

- The NI-488.2 Software, GPIB Hardware and GPIB Interface(s) are successively being verified.
  A blue mark (✓) indicates successful verification. A red mark (✗) indicates that verification failed.
  If the red mark indicates that verification of your GPIB interface failed, press ***Help*** and finally ***Retest***.

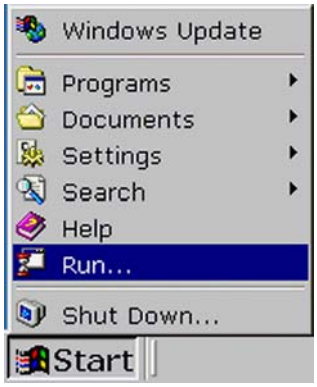


8.2.2.5 "NATIONAL INSTRUMENTS" GPIB PCI IEEE BOARD - VISA INSTALLATION

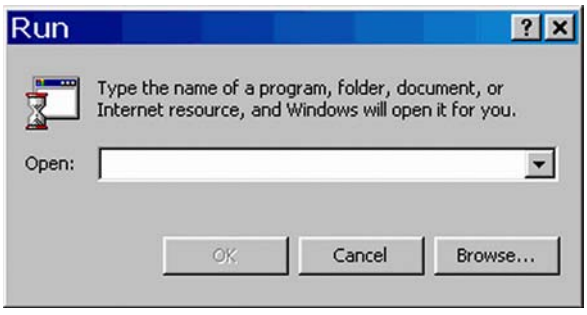
After having installed the NI-488.2 Software, the program "NI-Visa" will now be installed.

Close all applications!

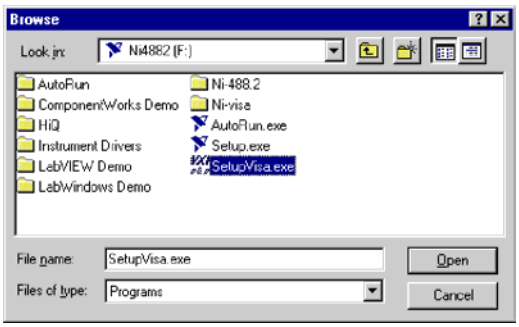
- Press the ***Start*** button on the taskbar.



- Press ***Browse***.



- At the "Look in" pulldown menu, select your CD drive [e.g. Ni4882 (F:)].



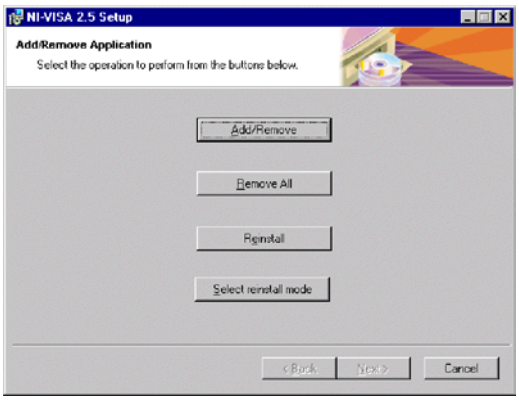
- Select "SetupVisa.exe" and press ***Open*** (or double-click "SetupVisa.exe").

- The path to "SetupVisa.exe" appears. Click ***OK***.

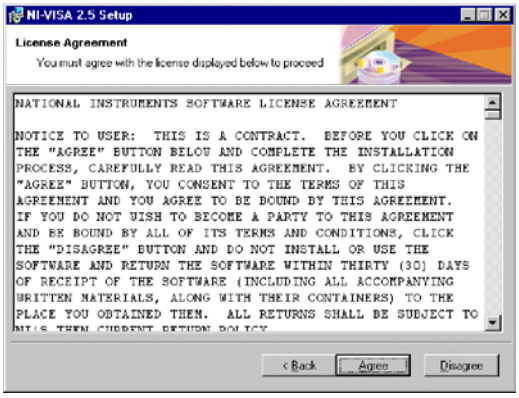
- Wait, while the Installation program is being prepared.

- You are reminded to close all applications.
  To do so, if necessary, see how to exit Visa Setup.
  Then press ***Next***.

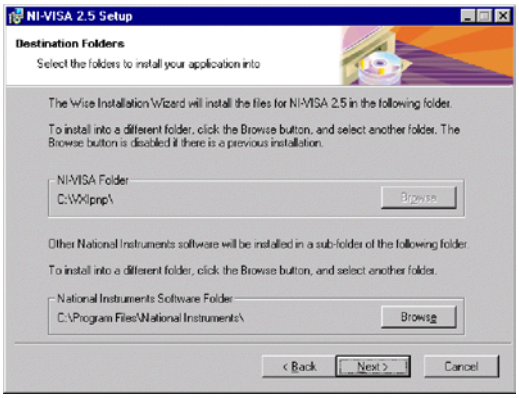
- If the Setup program detects previously installed NI-Visa components, either add / remove some of them or choose reinstall / reinstall mode.



- Read the License Agreement carefully and click "Agree" to proceed.
  "Disagree" would lead to exit Visa Setup.

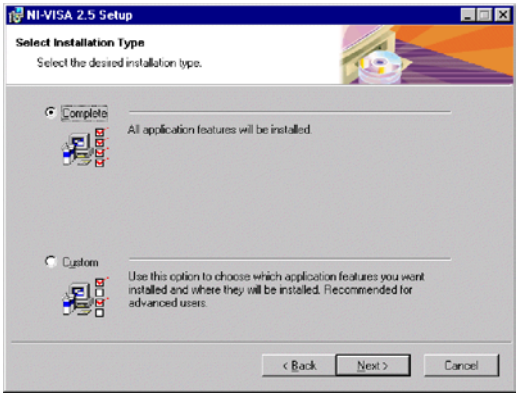


- The Installation program proposes a destination folder for the **NI-Visa** Software (e.g. C:\VXIpnp\). To choose another folder, press the upper "Browse" button.
  In case of a previous NI-Visa installation, this button is disabled and the NI-Visa Software will be installed in the proposed folder.
- Beneath it, another folder is proposed for the installation of **other NI** Software (e.g. C:\Program Files\National Instruments\).
  To choose another folder use the lower ***Browse*** button. Finally, click ***Next***.

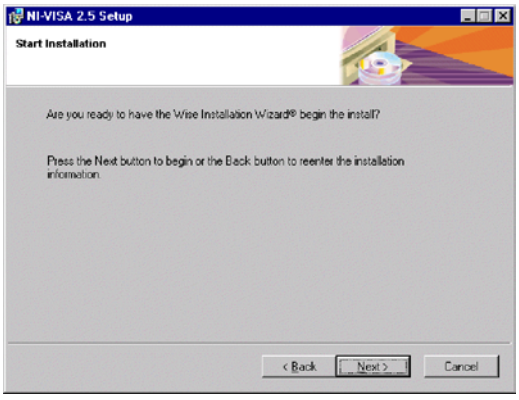


- Choose a folder (for the **other** NI Software). In the "Folder Name" box, the respective path is displayed. Press ***OK***.

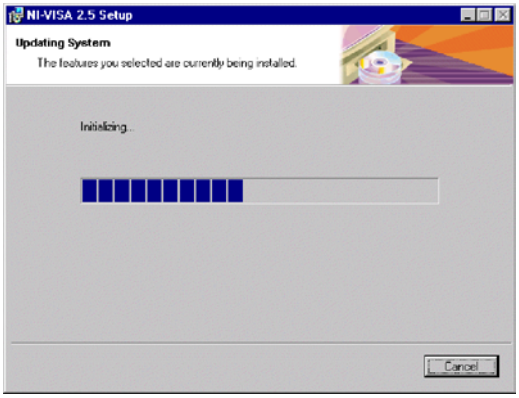
- To install all features select ***Complete*** (recommended usually) and press ***Next*** or use ***Custom*** (for advanced users) to install selected features and press ***Next***.



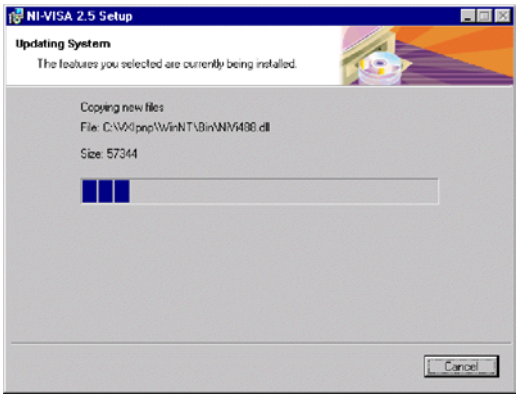
- To start installing the files press ***Next***.



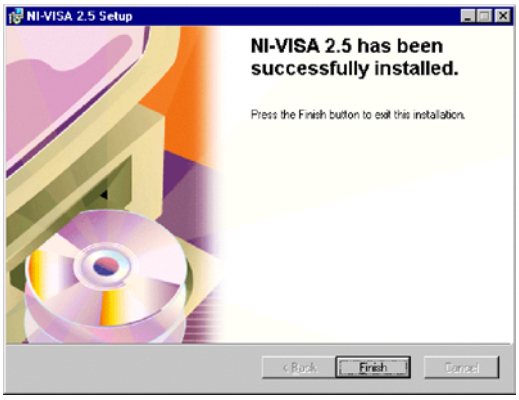
- Wait, while the initializing takes place.



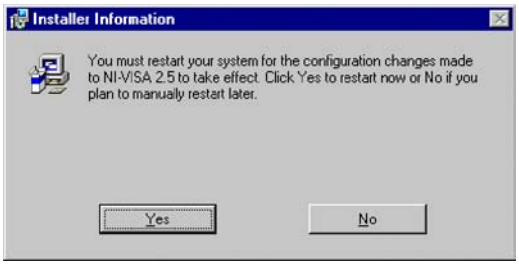
- Wait, while the files are being copied.



- After NI-Visa has been successfully installed press ***Finish***.



- Click ***Yes*** to reboot your system now.



8.2.3 INSTALLATION OF THE SOUND CARD DRIVER

The installation of a sound card is required when a GC application is used as peripheral. The GC communicates with the IRMS via the joystick port of the sound card.

- Insert the sound card into slot #5.
- For the installation of the respective sound card driver, follow the instructions in the manual of the sound card manufacturer.

8.2.4 INSTALLATION OF THE "ANALOG JOYSTICK" DRIVER

The following GC applications additionally require the **"Analog Joystick"** driver. The GC communicates with the IRMS via the joystick port of the sound card.

- HP 6890 GC
- Trace GC
- any other "generic" GC

NOTE: ***The successful installation of a sound card with the appropriate driver is a prerequisite for the installation of the joystick driver.***

For Joystick driver handling follow the steps below:

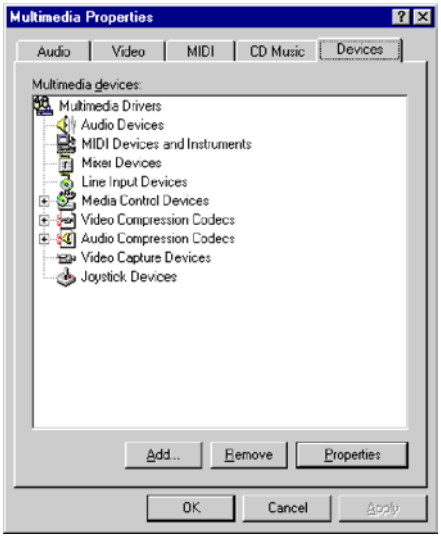
To install it insert the **"Operating System Backup CD"** and follow the steps below.

- Close the appearing box via the button (⊠) at the top right corner.

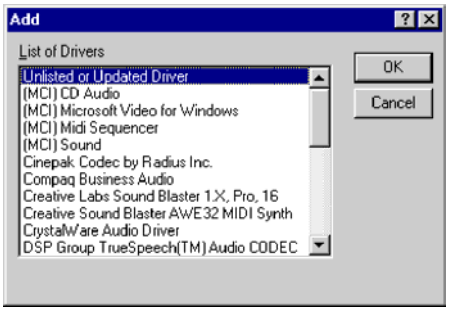
- Press ***Start > Settings > Control Panel***.

- Double-click the ***Multimedia*** Icon.

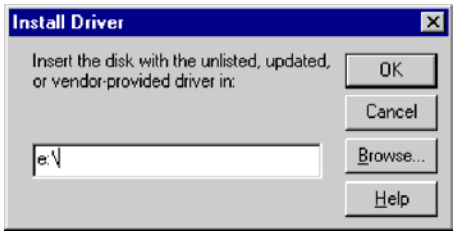
- Click the ***Devices*** tab.



- Press the ***Add*** button.

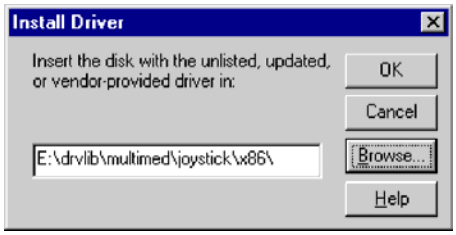


- Select ***Unlisted or updated driver*** and click ***OK***.

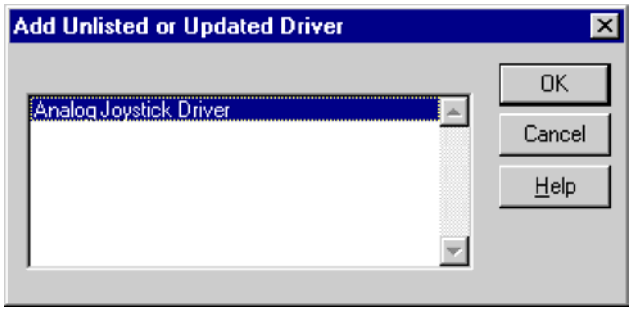


- Insert the **"Operating System Backup CD"** containing the driver - as was already recommended in Step 1. Type the letter of your CD drive (e.g. e:\) and click ***Browse***. From the ***Drives*** pulldown list select your CD drive (e.g. e: NTWKS 40A). In the ***Directories*** pulldown list double-click the folders, which successively lead to the Joystick drivers (e.g. e:\drvlib\multimed\joystick\x86).
  Click ***OK***.

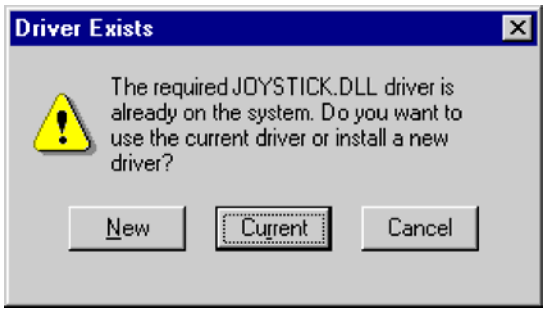
- Click ***OK***.



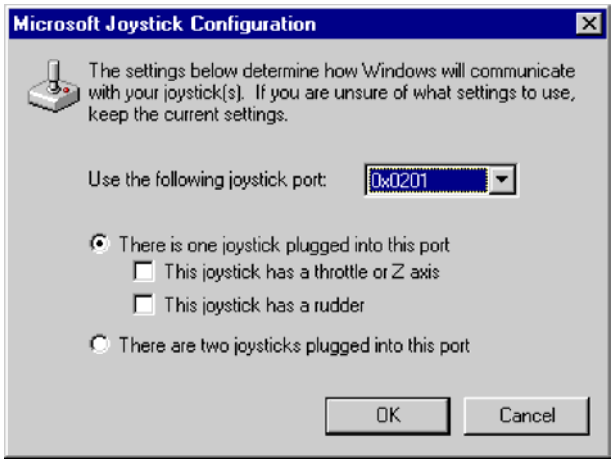
- Select ***Analog Joystick Driver*** and click ***OK***.



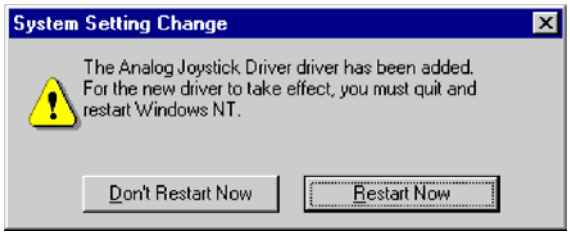
- If the required joystick.dll driver is already installed:
  press ***New*** to install a new driver, or press ***Current*** to use the current driver.



- In both cases, the box above appears.
  The different settings determine the communication of Windows with the joystick(s).
  If you are unsure of which settings to use, keep the default settings.
  Then click ***OK***.



- Click ***Restart Now***.

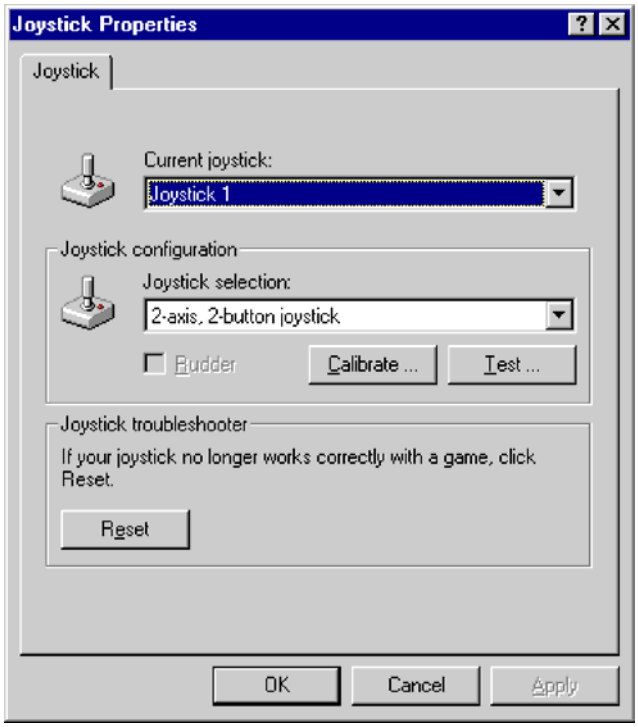


8.2.4.1 CALIBRATING THE JOYSTICK DRIVER

After the installation of the Joystick driver, it must now be calibrated and finally tested.
To calibrate it, follow the steps below.

- Press ***Start > Settings > Control Panel***.

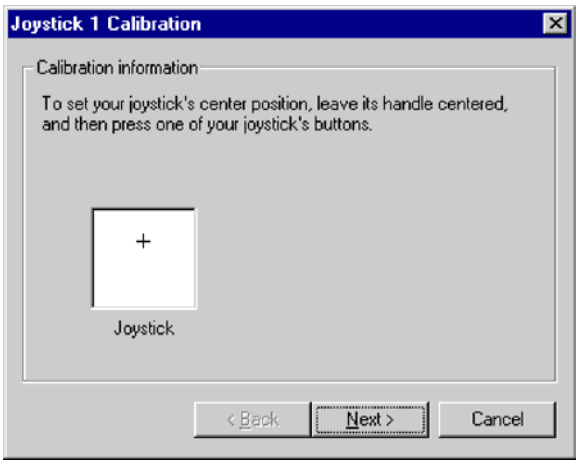
- Double-click on the ***Joystick*** icon.



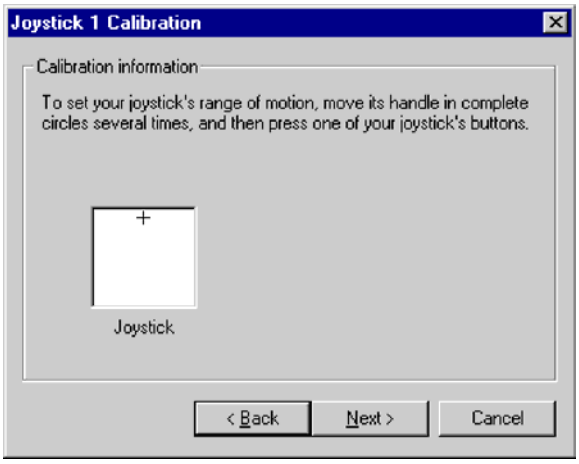
- At ***Joystick Configuration***, select the ***Calibrate*** button.

  An error message occurs, if the joystick is not correctly connected.

- To set the joystick's position, leave its handle centered.
  Then press one of the joystick's buttons.
  Click ***Next***.

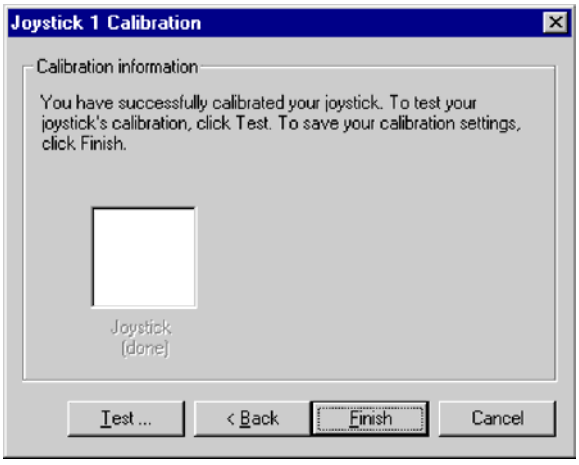


- To set the joystick's motion range, move the handle in complete circles several times.
  Then press one of the joystick's buttons.



- The joystick has been calibrated successfully.
  Click ***Finish*** to save your calibration settings.

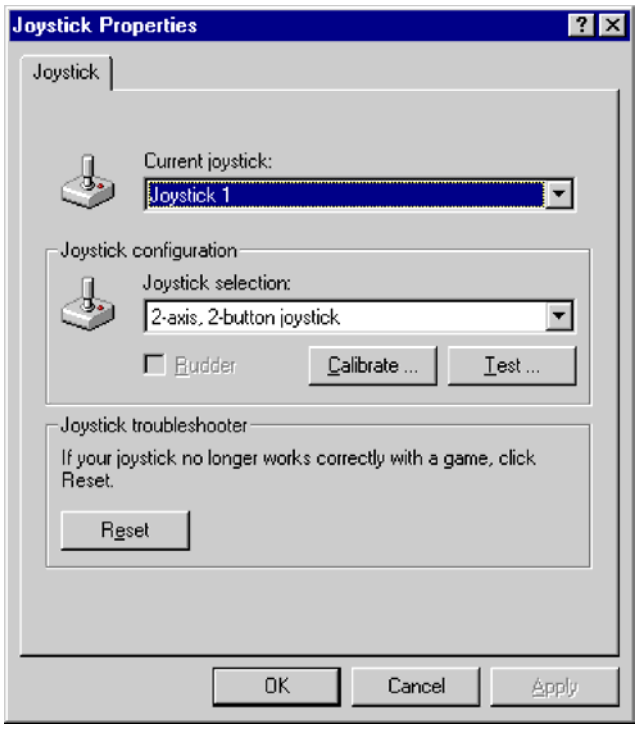
- Test the calibration.



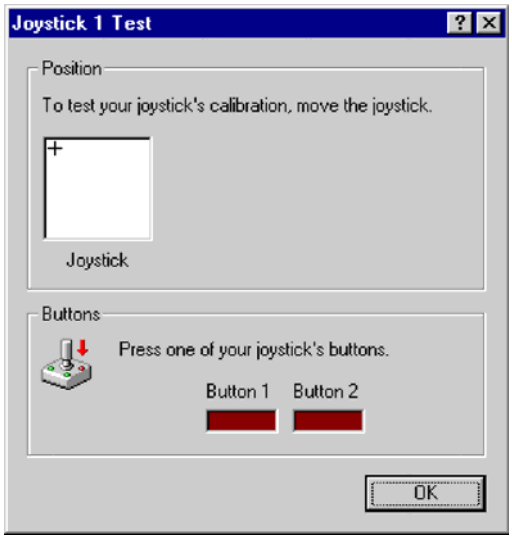
8.2.4.2 TESTING THE JOYSTICK'S CALIBRATION

- Press ***Start > Settings > Control Panel***.

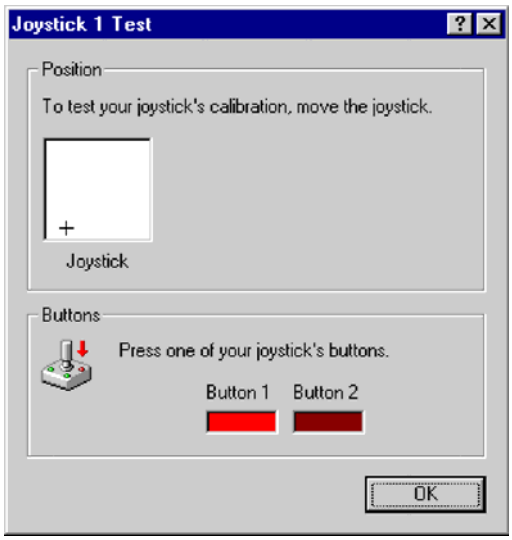
- Double-click on the ***Joystick*** icon.



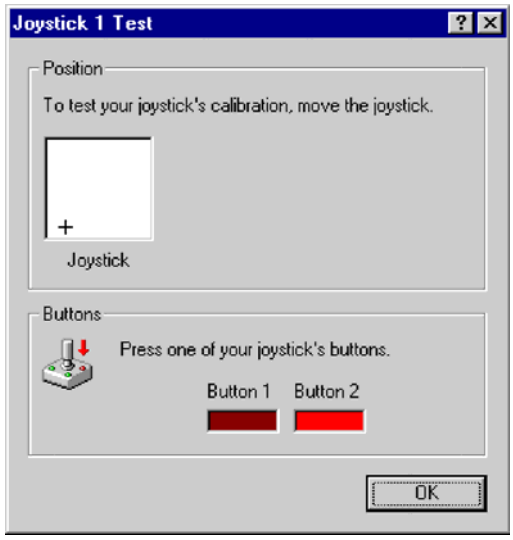
- At ***Joystick Configuration***, select the ***Test*** button.
  An error message occurs, if the joystick is not correctly connected.



- To test the Joystick's calibration, move the Joystick.
  Click ***OK***.
  Press one of the Joystick's buttons.



- One of the fields is lighted red (e.g. "Button 1").
  Press the other button.



- The other field is lighted red (e.g. "Button 2").
  Press ***OK***.

8.2.5 GC AND AUTOSAMPLER REQUIREMENTS

8.2.5.1 HP 6890 GC

- Gameport Interface (in the PC)
- Gameport cable (ATT: modified version, two resistors)
- Serial cable from PC to HP 6890 (Null Modem 2X3) - Default: COM 2

8.2.5.2 TRACE GC

- Gameport Interface (in the PC)
- Gameport cable from PC to Trace GC
- Serial cable from PC to Trace GC (Null Modem 2X3) - Default: COM 2

8.2.5.3 ANY OTHER "GENERIC" GC

- Gameport Interface (in the PC)
- Gameport cable from PC to any other "generic" GC (only Trigger Mode)

8.2.5.4 A200S OR PAL AUTOSAMPLER

- Serial cable from PC to A200S Autosampler - Default: COM 1
- Cable for "GC ready" status from Autosampler to GC

8.3 MAINTENANCE

Regular maintenance including functional checks and service routines is required to maintain trouble free and continuous operation.

These maintenance operations are listed in the table.

Part	Maintenance Operation	Frequency
Rotary pump	Check oil level Exchange oil Refer to manufacturer's instructions	Refer to manufacturer's instructions
Turbomolecular pump	Exchange oil reservoir Refer to manufacturer's instructions	Refer to manufacturer's instructions
Compressed air service unit	Check oil level Drain condensate and clean filter	Monthly If required
Safety circuit	Check function	Every 6 months

8.3.1 BASIC RULES FOR CLEANING

Do not economize with the cleaning agent! Renew the cleaning bath frequently.

Method of checking highly volatile cleaning agents (cyclohexane, acetone): Dip a polished metal plate into the bath and take out again so that as much liquid as possible remains behind on the plate (preferable bend the plate to a concave shape). Allow the bath liquid to evaporate. No residue should be left on the plate. Otherwise, change the bath liquid.

(Use slightly contaminated bath liquids for preliminarily washing severely contaminated parts!).

Do not rinse with water if using volatile cleaning agents!

Rinse with a lot of water if using cleaning agents in aqueous solution! Use warm water, finally rinse with distilled (or de-ionized) water!

Dry the parts, which have been rinsed in water, well in a dust free area, preferably in a vacuum drying oven! In the case of drying ovens with forced air circulation, the air must be free of dust. If necessary, wrap the parts loosely in tissue paper and place them in the oven.

Because contaminations on ceramic parts consist of evaporated-on metal layers, they cannot be removed with volatile cleaning agents. For this reason, use a high percentage aqueous solution of RBS 50 (Trade name of the firm Carl Roth, Karlsruhe).

The use of an ultrasonic bath increases the probability of a successful removal of the contaminations.

Even with RBS 50, a successful removal of contaminations from ceramic parts may not always be possible. In this case, replace these parts by new ones.

If new parts are not available, strongly adhering layers can be removed as follows:

- By filing down with a diamond file. After being filed, the parts must be washed. The diamond file can easily be cleaned again with an erasing rubber.
- By annealing at red heat in a propane-oxygen flame. Too high temperature may cause distortion of ceramic parts. Therefore, avoid the white heat range!

  Do not wash any parts after they have been annealed.

  CAUTION: ***Ceramic parts, which cannot be disconnected from metal parts, must not be annealed.***

When re-assembling, do not touch the washed parts with naked hands! Use non-fibrous gloves and clean tools!

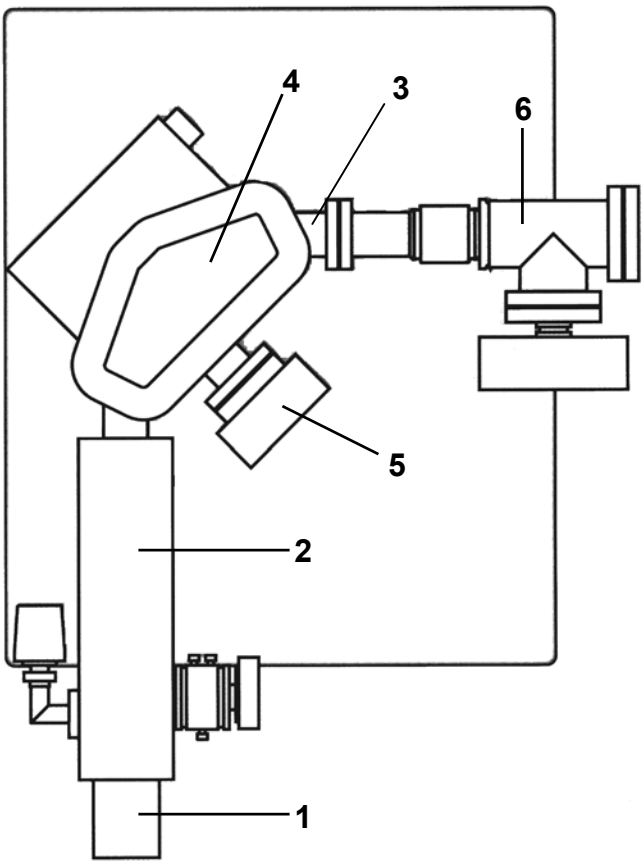
In the case of difficult assembly jobs, it may be necessary to work without gloves. Then thoroughly wash your hands and remove any fat or grease from the fingertips with a solvent. The parts should then be touched only with degreased fingertips.

In order to avoid any damage to the skin, rub the fingertips over with a fatty skin cream when the work is completed!

8.4 ANALYZER SYSTEM

This chapter contains drawings and spare part lists of the analyzer system. For the analyzer control, see chapter 4.

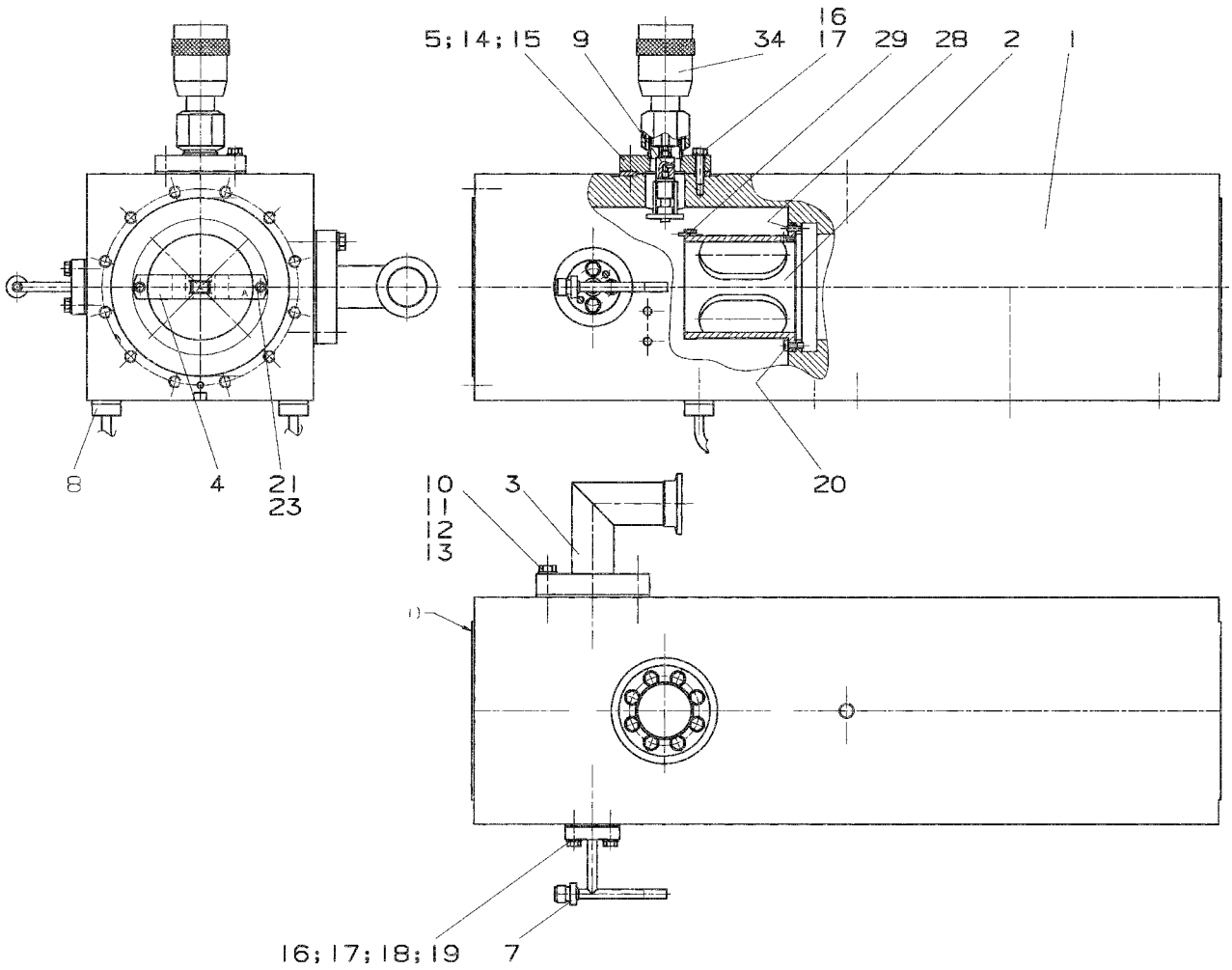
Fig. 8 - 1 Basic Unit (Part No. 113 6000)



Pos. 7 not shown

No.	Qty.	Designation	Part No.
1	1	Ion source with flange (see chapter 8.6)	113 1480
2	1	Analyzer head (see chapter 8.4)	113 1460
3	1	Flight tube	114 1700
4	1	Electro magnet	114 2490
5	1	HD measuring equipment	114 8100
6	1	Multi collector system	113 6010
7	1	Vacuum system (see chapter 8.5)	-

Fig.: 8-2 Analyzer Head (Part No.: 113 1460)



No.	Qty.	Designation	Part No
1	1	Analyzer housing	113 1470
2	1	Source carrier	100 3540
3	1	Angle	111 8480
4	1	Entrance plate	100 3670
5	1	Nipple	100 3590
7	1	Gas inlet	111 3980
8	2	Heater	079 2990
9	1	Adapter	100 3570
10	1	Spacer ring, NW 32	095 1170
11	1	Gasket, gold; 49,5 NW 32	055 1700
12	8	Screw, M 6x25 DIN 933 A4	045 4380
13	8	Washer, 6,4 DIN 125 A2	047 0060
14	1	Spacer ring, NW 25	095 1250
15	1	Gasket, gold; 38	054 5270
16	8	Screw, M 5x20 DIN933 A4	045 4500
17	8	Washer; 5,3 DIN125 - 1.4301	047 0060
18	1	Spacer, NW 6	095 1600
19	1	Gasket, gold; 11,5 NW 6	055 1240
20	4	Screw, M 4x10	064 3010
21	2	Washer; 3,2 DIN 125 A4	047 0030
23	2	Screw, M 3x6 DIN 84 A4	045 0750
25	12	Washer; 8,4 DIN 125 A2	047 0070
28	1	Pin, 3x10 DIN7 A4	048 0110
29	1	Pin, 2x8 DIN7 A4	048 0050
30	1	Spacer ring, NW 100	095 1380
31	1	Gasket, gold; 117,5 NW 100	055 1600
32	12	Screw, M 8x35 DIN 931 A4	045 4400
34	1	Variable ion source conductance	055 9900
36	1	Flange sealing, DN 63	069 3220
37	1	Flange sealing, DN 100	056 9119

8.5 VACUUM SYSTEM

This chapter contains remarks, drawings and spare part lists for the vacuum system.

8.5.1 GENERAL REMARKS

The final pressure should be checked daily. About 24 hours after start, without sample inflow the pressure should be in the 10^{-8} mbar range.

The main reasons of troubles with the vacuum system are leaks and contaminations:

Leaks may be caused:

- If flange connections and sealings are not properly treated during service operations.
- Accidentally during cooling down periods after baking.
- If heavily treated sealing components are worn out.

As leak detection is rather time-consuming, make sure that flange connections are carefully and properly assembled during service.

Additionally, watch the pressure during cooling down after baking. If necessary, retighten the flanges.

Contaminations may be caused:

- By water vapor stemming from the sample preparation devices.
- By use of improper elastomer gaskets especially when operated at elevated temperatures. We recommended ordering spare gaskets from Thermo Finnigan MAT only.
- By introducing solvents which had not been removed carefully after cleaning.

For distinguishing, whether elevated pressures are caused by leaks or by contamination, take a background spectrum.

WARNING: ***Never use silicon oils or silicon greases! Silicon layers lead to surface charges and are difficult to eliminate!***

8.5.1.1 FLANGES WITH METAL GASKETS

Before mounting the flange connection, make sure that the sealing surface of the flanges are clean and that the gaskets are clean and intact.

Afterwards put the spacer ring onto the protruding edge of the flange, place the metal gasket inside the spacer ring and insert the counter flange into the spacer ring. Make sure, that the

flanges are not canted, that flanges and spacer ring are centered in the right way and have equal distances. Then insert and manually tighten the screw. Afterwards use a wrench and tighten the screw crosswise within at least two procedures. Check, that flanges and spacer ring have equal distances. Stop tightening, when the flanges touch the spacer ring.
If using the metal gaskets for more than one time, do not take off the gasket from the spacer ring. Make sure that the gaskets are not damaged. Note that the probability for leakage increases with increasing use.

Note: ***Do not re-use a baked metal gasket.***

8.5.1.2 FLANGES WITH VITON GASKETS

When changing the Viton gasket, do not use other materials. The Viton used must be of good and preheated quality. For this reason, we recommended to order spare gaskets from Thermo Finnigan MAT only.
Before using the gasket, make sure that the gasket and the sealing surfaces are clean and that the gasket surfaces are not injured. For Cleaning, wipe the gasket with clean paper. Do not use vacuum grease, and do not clean Viton gaskets with solvents.
Note that Viton must not be operated at temperatures above 1500 °C.

8.5.1.3 PVC-HOSES OF FORE VACUUM LINES

If the hoses are contaminated by fore pump oil, replace with new hoses. Never clean PVC hoses with solvents.
Note that PVC-hoses are sensitive to elevated temperatures. Therefore, make sure that these hoses do not contact hot parts.

8.5.1.4 CLEANING THE VACUUM COMPONENTS

Cleaning the parts of the high vacuum regions is described in section 8.6 (ion source).
For cleaning parts of the vacuum region, many different methods are known.

The best cleaning is obtained when using a hot ultra sonic bath.

The following recommendations should be observed:

- Use pure solvents only.
- When cleaning with solvents, use a hood.
- Pay attention to your country's safety regulations.
- After cleaning, dry the parts carefully.
- Do not touch the cleaned parts with your fingers (use lint-free gloves).

8.5.1.5 LEAK DETECTION

Two different methods are recommended:

1. Mass spectrometric leak detection can be applied if the leak permits mass spectrometer operation (pressure ≤ 6 x 10^{-4} mbar). Use a container with suitable leak detection gas (e.g. argon). Connect the reducing valve to a PVC hose and the open end of the hose to a fine capillary (e.g. glass or metal tubing). Set the mass spectrometer to the argon peak (m/z=40) and blow a fine argon beam onto those parts, where leaks are suspected. The argon peak will rise when the leak has been detected.

   Note that at small leaks (creeping leaks) it might last a certain time until the test gas has entered the vacuum system. In case of very small leaks, it might be necessary to enclose the suspicious part with a plastic sheet, to fill the plastic sheet with testing gas and to wait for peak rising.

   If the instrument is equipped with the HD collector, helium may be used as the test gas. In this case, set the analyzer system to mass 4.
2. For leak detection in the forevacuum section, or if the pressure is too high for mass spectrometer operation, proceed as follows:

   Use a small washing bottle with ethanol and spray a fine ethanol beam onto those connections where a leak is suspected. When hitting the leak, the pressure, after a short time of delay, will decrease at first and afterwards will increase rapidly. Use the ion gauge for pressure reading.

Warning: ***Ethanol vapors are explosive. Do not smoke or handle with open fire during leak detection. After leak detection, ventilate the laboratory carefully.***

8.5.1.6 ION GAUGE

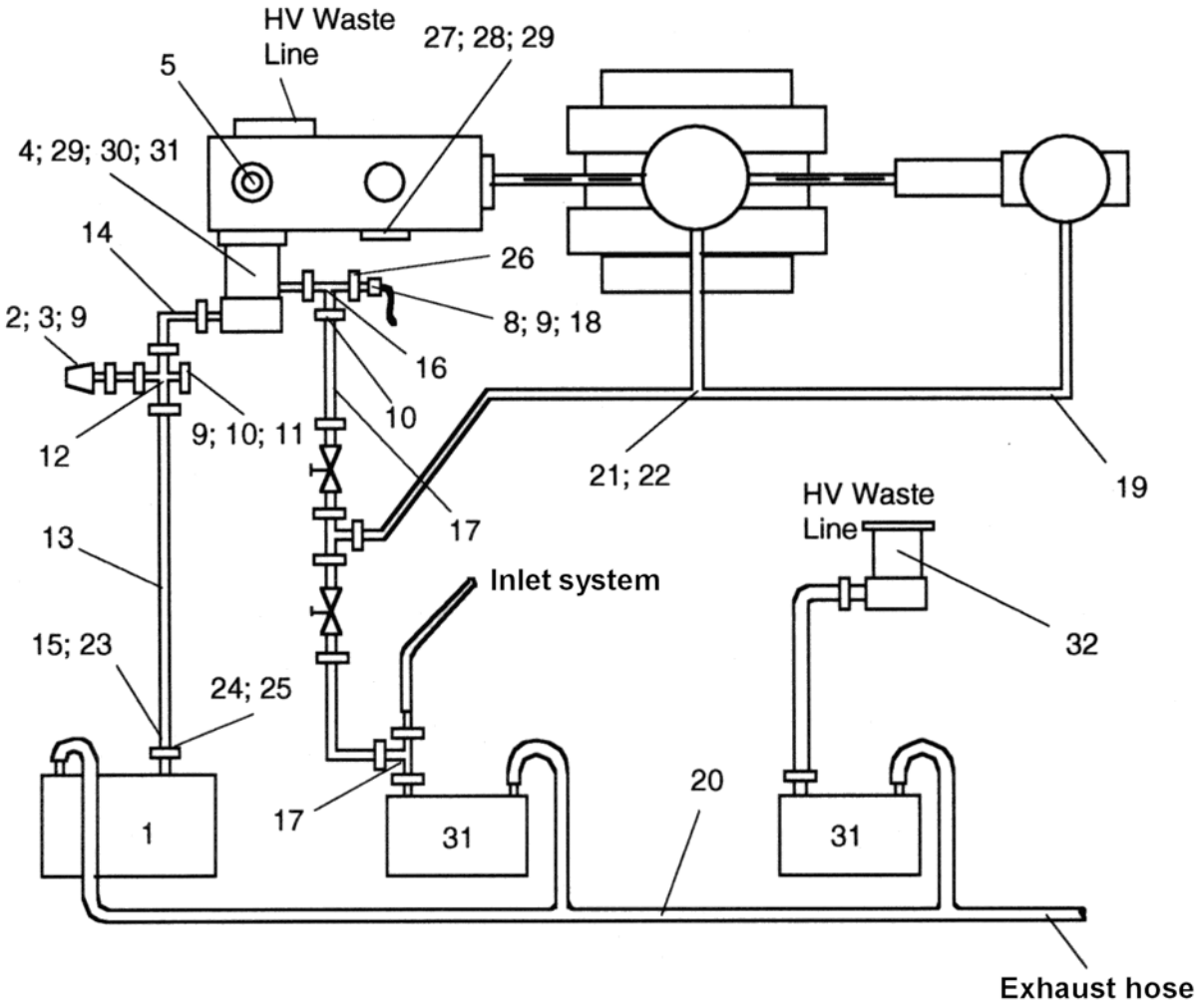
The ion gauge is used for vacuum protection. As soon as the filament (cathode) of the ion gauge is burnt out, the vacuum protected part of the mass spectrometer is turned off automatically.

In this case, replace the ion gauge tube with a new one. When replacing the ion gauge tube, take care that the filament is placed in the downward direction. Otherwise a short circuit may occur.

A general description of cleaning, generally used parts, expendable materials and tools is given in chapter 8.3.1.

8.5.2 PUMPING SYSTEM

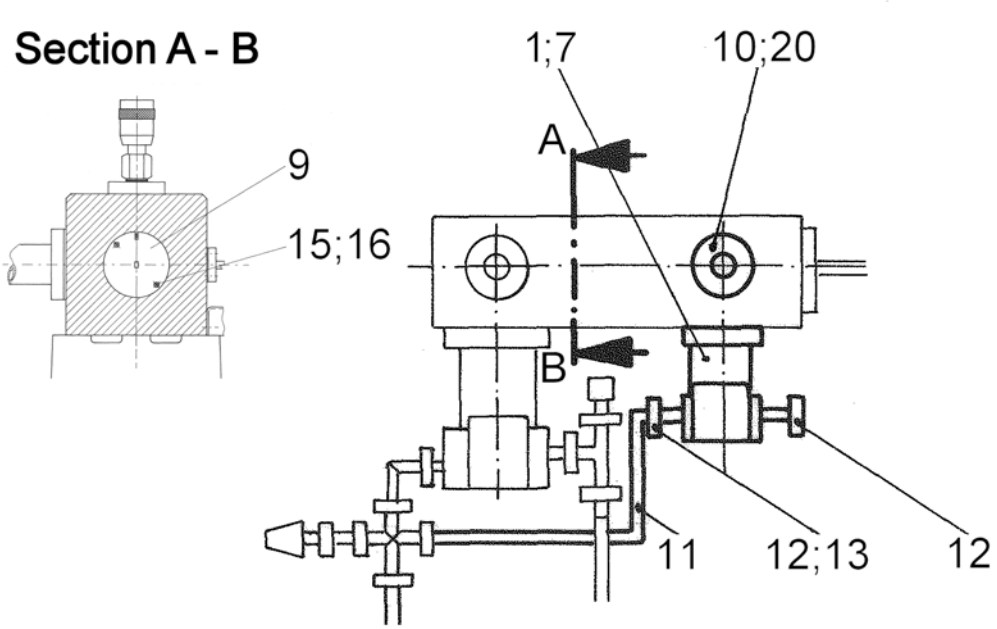
Fig.: 8-3 Pumping Scheme



Thermo Finnigan

No.	Qty.	Designation	Part No
1	1	Fore vacuum pump, DUO 005	109 7510
2	1	Vacuum gauge, Pirani	102 7320
3	1	Connection cable for pos. 2	103 9280
4	1	Turbomolecular pump, TMH 262	114 1600
5	1	Vacuum gauge, Penning	111 8320
6	1	Air cooling f. pos. 4	069 4150
8	1	Vent valve	108 4890
9	3	Centering ring, NW 16/10	052 2200
10	7	Clamping ring, NW 10/16 KF	052 1830
11	1	Blind flange	052 2090
12	1	Cross piece	100 3780
13	1	Hose	069 1290
14	1	Line	102 9540
15	1	Hose clamp	037 0190
16	2	T piece	095 3460
17	2	Hose	052 2110
18	4	Centering ring	052 2140
19	5 m	Hose: 7 x 3.0, PVC	0690230
20		Hose, 13 x 3.5	0690720
21	1	T piece, 8 mm	052 0350
22	4	Centering ring	052 2170
23	2	Hose clamp	051 2344
24	4	Clamping ring	052 1560
25	4	Centering ring	052 2150
26	1	Flange	108 7620
27	1	Flange	065 2620
28	1	Edge sealing	069 3220
29	16	Clamping screw	102 8380
30	2	Edge sealing	056 9110
31	2	Fore vacuum pump, DUO 2.5	109 5950
32	1	Turbomolecular pump TMH 071 P	114 1500

Fig. 8 - 4: Differential Pumping System (Part No.: 111 1121)



No.	Qty.	Designation	Part No.
1	1	Turbo molecular pump TMH 071 P	114 1500
7	1	Air cooling for pos. 1	079 2900
9	1	Differential pressure plate	100 3710
10	1	Sealing centering ring: NW 32/40, aluminum	055 2940
11	1	Metal hose, KF NW 16 x 500	053 4500
12	2	Clamping ring, NW 10 /16 KF	052 1830
13	2	Centering ring, NW 16/10	052 2200
15	2	Screw; M 2 x 4, DIN 84	045 0690
16	2	Washer; 2,5 DIN 125	047 0090
20	1	Clamping ring	047 2510

8.6 ION SOURCE

The following chapter describes the ion source connections, the removal and the insertion of the ion source, the replacement of the filament, the disassembling of the ion source, the cleaning procedure and the reassembly of the ion source.
The exploding views and the spare parts lists indicate the location and the ordering numbers of the different ion source parts.

8.6.1 REMOVAL OF THE ION SOURCE

1. Pull out the ion source plug on the ion source flange.
2. Unscrew the 12 bolts of the ion source flange and remove the ion source carefully, without knocking against the ion source housing.
3. In case the ion source shall be removed for a longer period of time, e.g. for cleaning, close the opening of the analyzer head by suitable means in order to prevent foreign particles from entering the ion source chamber.

8.6.2 REINSTALLATION OF THE ION SOURCE

When the ion source section has been baked repeatedly or for a longer period at maximum baking temperature, a new gold wire gasket must be used. In case of no baking or baking at moderate temperature the gasket can be used repeatedly, provided it has not been damaged during removal.

1. Inspect the gasket carefully for absence of scratches and lint.
2. Place the spacer ring (see fig. 8 - 8, pos. 5) on the flange of the analyzer head and insert the gasket (pos. 4). Insert the source, with the inlet line connector upside, into the analyzer head. Take care that the pin in the flange points upwards. Engage this pin with the hole of the analyzer housing. When inserted, ion source flange and making flange should be aligned in parallel.
3. Press the ion source in the direction of the analyzer and tighten the screws hand tight. Check for short circuits. Afterwards use a wrench and tighten the screws crosswise within at least two procedures. Check, that flanges and spacer ring are equally spaced. Do not proceed tightening if the flanges touch the spacer ring!

8.6.3 EXCHANGE OF THE CATHODE UNIT

NOTE: ***Use lint-free gloves and clean tools for the exchange.***

1. Remove the ion source as described in chapter 8.6.1.
2. Unscrew the supply line K connecting plug (see fig. 8 - 6).
3. Unscrew the two cylinder screws (pos. 6, see fig. 8 - 11) and take off the magnet and the cathode (pos. 7).
4. Insert a new cathode and the magnet.
5. Reconnect the supply lines.
6. Perform the source check as described in chapter 8.6.7.
7. Reinstall the ion source as described in chapter 8.6.2.

8.6.4 DISASSEMBLY OF THE ION SOURCE

1. Preparing the disassembly:
   a. Use a table with clean and smooth surface.
   b. Put a white and clean paper onto this table.
   c. Prepare receptacles for the small ion source parts. Separate metal and ceramic parts.
2. Remove the ion source as described in chapter 8.6.1.
3. Remove the cathode as described in chapter 8.6.3.
4. Remove the electron collector (pos.10) and the magnet (pos. 8, see fig. 8 - 11).
5. Pull off carefully the connector from the ion source and bend the lead wires somewhat apart. Take care not to alter the curved shape of the lead wires as this facilitates correct reconnection.
6. If not necessary, do not pull off the lead wires from the feedthrough connectors of the flange. This will save time and will ease the later assembly.
7. Unscrew the cylinder head screws (pos.1, see fig. 8 - 8) and remove the ion source from its supporting rods.
8. Unscrew the screws (pos. 19) and take off carefully the spacing piece (pos. 15). Pull out the ceramic tube (pos. 22) (see fig. 8 - 10).
9. Slacken the four threaded sleeves (pos. 1) (see fig. 8 - 10).

10. Pull out the four cylinder pins (pos. 5) (see fig. 8 - 11), which fix the other end of the ceramic rods (pos. 2, see fig. 8 - 10) to the ionization housing (pos. 4).

11. Remove the ionization housing (pos. 4) from the ceramic rods.

12. Remove all plates from the ceramic rods.

NOTE: ***Remove the plates without tilting them. If the plates are tilted, it is very difficult to remove them. It also gives rise to metal abrasion on the ceramic rods, which causes leakage currents between the plates. There is also the danger that the ceramic rods will break.***

13. Unscrew the cylinder screws (pos.9); remove the slit (pos.13) from the ion source base (pos.6, see fig. 8 - 9).

8.6.5 CLEANING THE ION SOURCE PARTS

8.6.5.1 CLEANING THE METALLIC PARTS

Grind all contaminated and discolored metal parts with fine emery paper (use 360 grain or finer).

A chemical cleaning as described in the following must follow the mechanical cleaning described above.

For chemical cleaning, distinguish between the different types of metallic parts:

All parts must be cleaned within a grease-dissolving bath (e.g. cyclohexane). Use a bath temperature of about 60 °C. An ultrasonic bath will improve and accelerate the cleaning procedure.

CAUTION: ***Do not use ultrasonic when cleaning the ion source magnets! The basic rules for cleaning are described in chapter 8.3.1. The cleaning described there will be sufficient for all parts with minor contamination, which are out of the ion and the electron paths. Afterwards all parts made of stainless steel should be cleaned in a 30% RBS solution at 60 °C for about 1 or 2 hours. Before drying, the RBS cleaned parts must be thoroughly rinsed twice within distilled water at 60 - 70 °C. Additionally see for the basic rules of cleaning in chapter 8.3.1.***

CAUTION: ***The following parts must not be cleaned in RBS:***
- Parts, which are not made of stainless steel.
- The ion source magnets.
These parts must be cleaned with cyclohexane as described above.

After cleaning in RBS and in distilled water, the parts should be dried chemically within a Freon bath or pure alcohol.
After chemical drying, the parts should be additionally dried by a fan or a drying oven to eliminate solvent residuals.

8.6.5.2 CLEANING THE CERAMIC INSULATORS

Use a clean diamond file for eliminating metal abrasion and evaporated films. Use this method only for cleaning the ceramic insulator of the cathode and the electron collector. Heavily contaminated ceramic parts may be cleaned later in RBS solution (do not use RBS for the cathode and the electron collector).
Anneal the ceramic spacer rings to soft red heat. Use a propane/oxygen flame or a muffle furnace for annealing (maximum temperature: 800 - 1000 °C).

CAUTION: ***At too high temperature (white heat) the parts may be deformed!***
Do not anneal ceramic parts containing metal connections like the ceramic rods!
Do not touch annealed parts with bare fingers and prevent wetting!

8.6.6 ASSEMBLING THE ION SOURCE

Touch the ion source parts with clean, lint-free gloves only. Use clean tools only!
Before assembling the ion source, arrange the parts and the tools in a clear manner on white clean paper.

1. Assemble the ion source base according to fig. 8 - 9.
2. Loosely screw on the threaded sleeves (see fig. 8 - 10, pos. 1) to the ceramic rods and insert the rods from the lower side into the ion source base plate.
3. Pass the four ceramic rods (pos. 2) through the hoes of the ion source base (pos. 5) such that the punched numbers are visible from above.

4. Afterwards insert the spacer tubes, the ion source plates and ionization housing to the ceramic rods as shown in fig. 8 - 10 and 8 - 11.
   - Insert the parts carefully
   - Avoid metallic abrasion
   - Spacer rings and plates must fit tightly together
   - The base plate, the plates and the ionization housing are numbered. The numbers must be arranged with ascending sequence and in the same direction.
5. Turn the four ceramic rods (fig. 8 - 10, pos. 2, see) such that the slot grinded into their upper end is aligned with the boreholes in the ionization housing (fig. 8 - 11, pos. 4), allowing the four pins (pos. 5) to be pushed in smoothly.
   Tighten the four threaded sleeves carefully with the special tool contained in the instrument tool kit as fast as it is necessary to have the parts of the ion source clamped firmly together.
6. Insert the bush (fig. 8 - 10, pos. 21) into the support (pos. 18). Position the convex surface as shown on fig. 8 - 10.
7. Slip the spring (pos. 17) over the bush (pos. 21) and then fasten the spring (pos. 17) to the support (pos. 18) with the screws (pos. 20) and nuts (pos. 16).
8. Insert the tube (pos. 22) into the bush (pos. 21).
9. Mount the support (pos. 18) and the spacing piece (pos. 15) with the cylinder screw (pos. 19) to the ion source base (pos. 5).
10. Assemble the parts of the electron collector pos. 11 to 15 (see fig. 8 - 11).
11. Attach the rod magnets (pos. 8) together with the electron collector resp. the filament (pos. 7) with the cylinder screw pos. 17 and 7. Pay attention to correct polarity!
12. Screw on the four threaded rods (fig. 8 - 12, pos. 2) into the ion source base.
13. Slip the parts pos. 3 to 7 onto the threaded rods (pos. 2) and screw on the nuts (pos. 8) (see fig. 8 - 12).
14. The ion source now must have its correct height. The distance between the base plate and the middle of the bush part must be 60 ± 0.1 mm.
15. Connect the ion source connection leads to the pins of the source (see fig. 8 - 6). The plugs must fit tightly onto the pins. If necessary, compress the plugs a little.

16. Connect the ion source connection leads (if they had been disconnected) to the feedthroughs of the ion source flange (see fig. 8 - 5).

NOTE: ***Hold the pins of the feedthroughs with small pliers while inserting the plugs.***

CAUTION: ***The feedthroughs contain delicate ceramics. Do not break!***

8.6.7 CHECKING THE ION SOURCE

Carefully check the position of the connection wires. The wires must **not**:

- contact each other,
- contact the ionization housing or the ion source plates,
- contact the inner walls of the analyzer head after insertion. To avoid this contact, the wires must be inside a virtual cylinder, which is defined by the central axis of the supporting rods.

Use an ohmmeter and check the connection between the pins of the ion source socket and the ion source electrodes. The schematic diagram for the ion source connection is shown in fig. 8 - 6.

Check the filament of the cathode unit: The resistance between pin +K and -K source socket must be about 0.1 Ω.

The insulation resistance of the ion source plates, with respect to each other and to ground must exceed 10^{12} Ω (unless these are interconnected internally).

NOTES: ***The insulation resistances may be decreased by humidity. In this case, the resistance increases as soon as the ion source is evacuated and warmed up.***

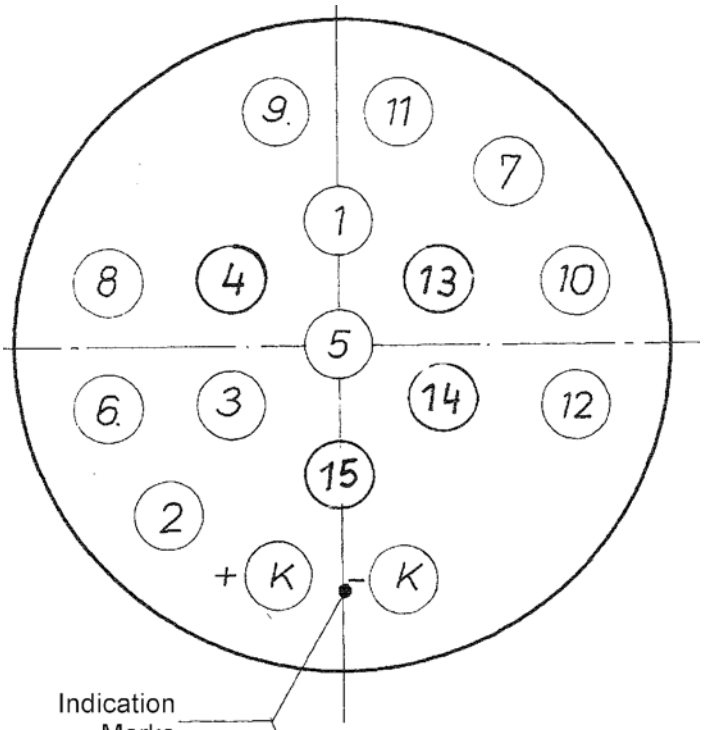
If no ohmmeter is available for measuring such high resistances, at least check the source for short circuits.

Check the gasket and the sealing surfaces of the ion source flange before inserting the ion source into the analyzer head.

8.6.8 PARTS OF THE ION SOURCE

Fig. 8 - 5: Source Feedthroughs

Feedthrough, view from ion source (from inside)



Indication Marks

Feedthrough, view towards ion source (from outside)

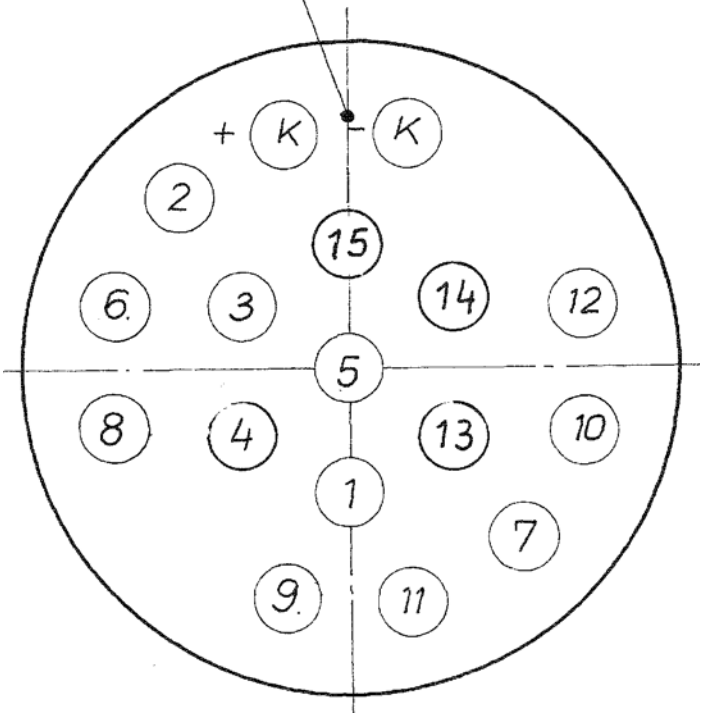
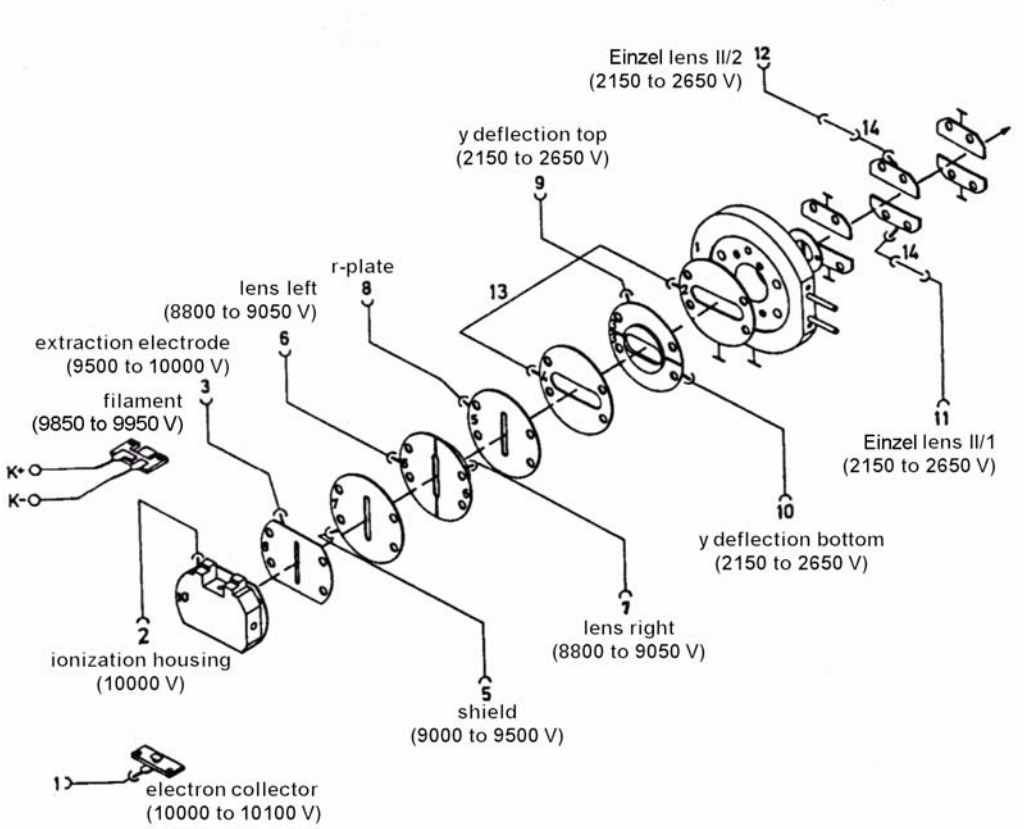
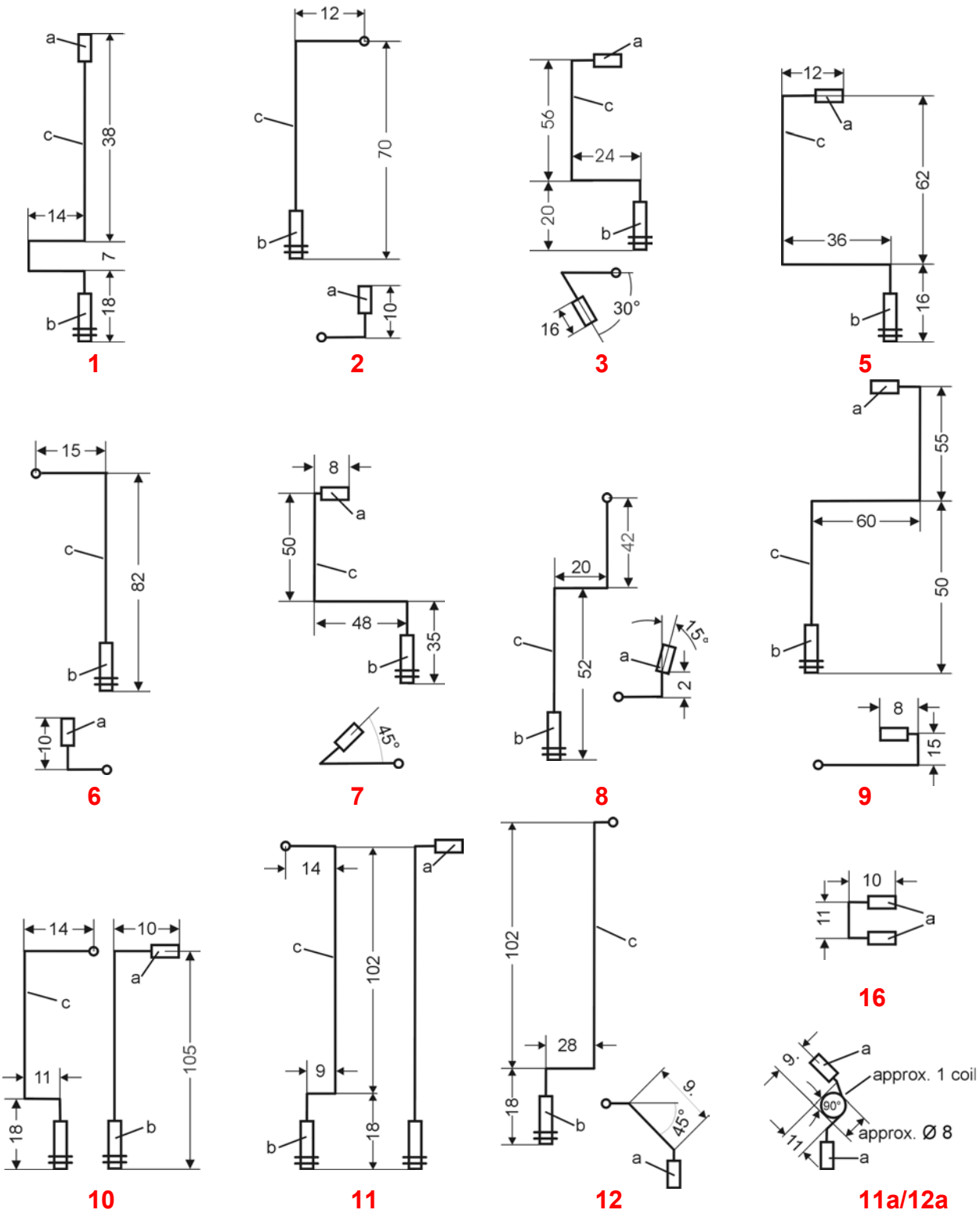


Fig. 8 - 6: Connection Schematics of the Ion Source



Thermo Finnigan

Fig. 8 - 7: Connection Lines (Part. No. 114 7580)

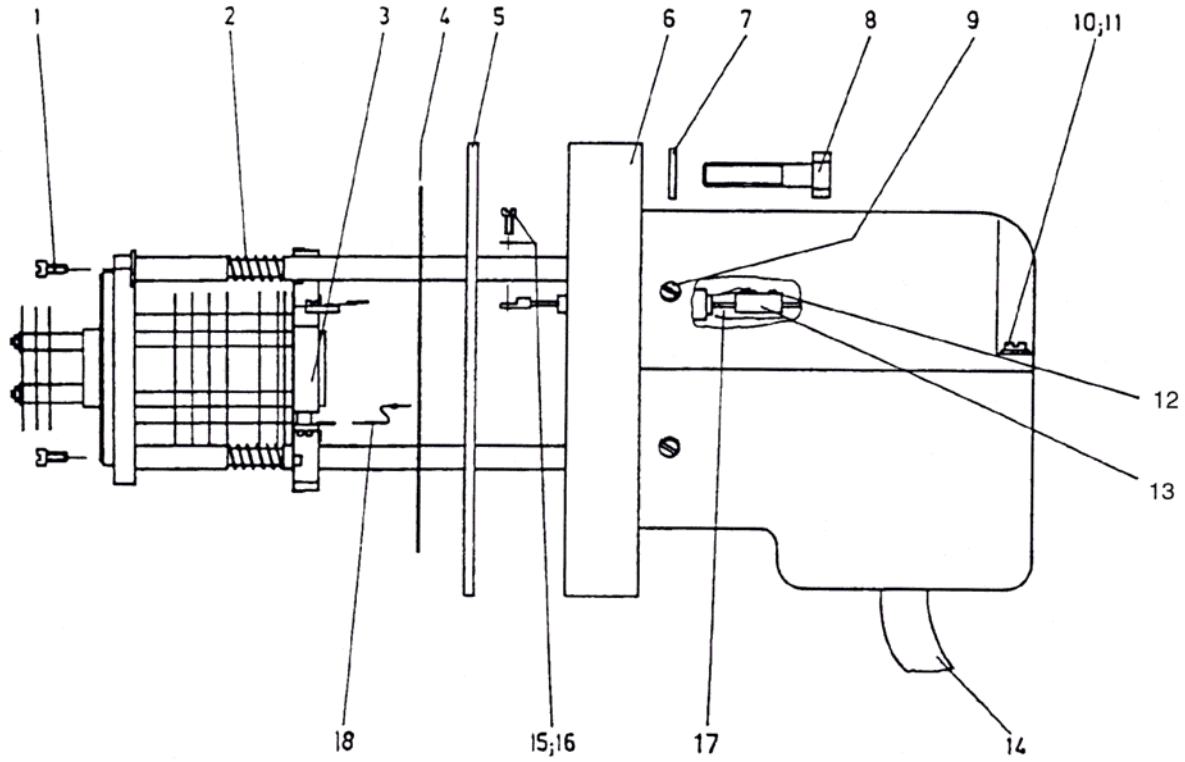


Thermo Finnigan

No.	Qty.	Designation
1	1	Wiring connection Electron collector
2	1	Wiring connection Ionization housing
3	1	Wiring connection Extraction plate
5	1	Wiring connection Shield
6	1	Wiring connection Lens, left
7	1	Wiring connection Lens, right
8	1	Wiring connection R - plate
9	1	Wiring connection Z - deflection, above
10	1	Wiring connection Z - deflection, below
11	1	Wiring connection Einzel lens
11a	1	Wiring connection Einzel lens
12	1	wiring connection Einzel lens
12a	1	Wiring connection Einzel lens
16	1	Wiring connection grounding plate
a	1	Bush
b	1	Bush with ring
c	1	Wire, nickel 0,8 (specify length required)

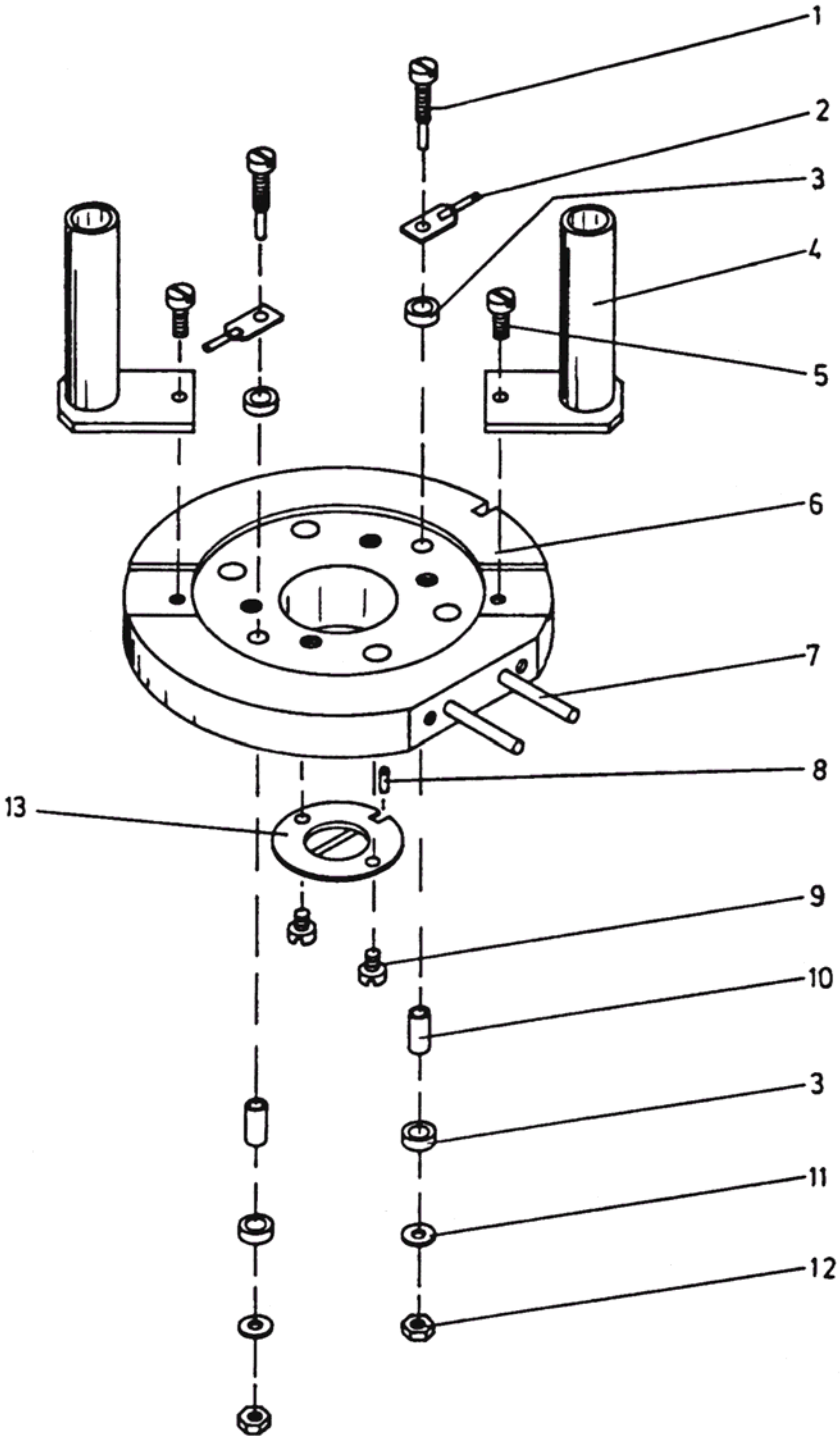
NOTE: ***Drawings on page 8-56 do not scale. All dimensions are in mm.***

Fig. 8 - 8: Ion Source with Flange (Art. No.: 113 1480)



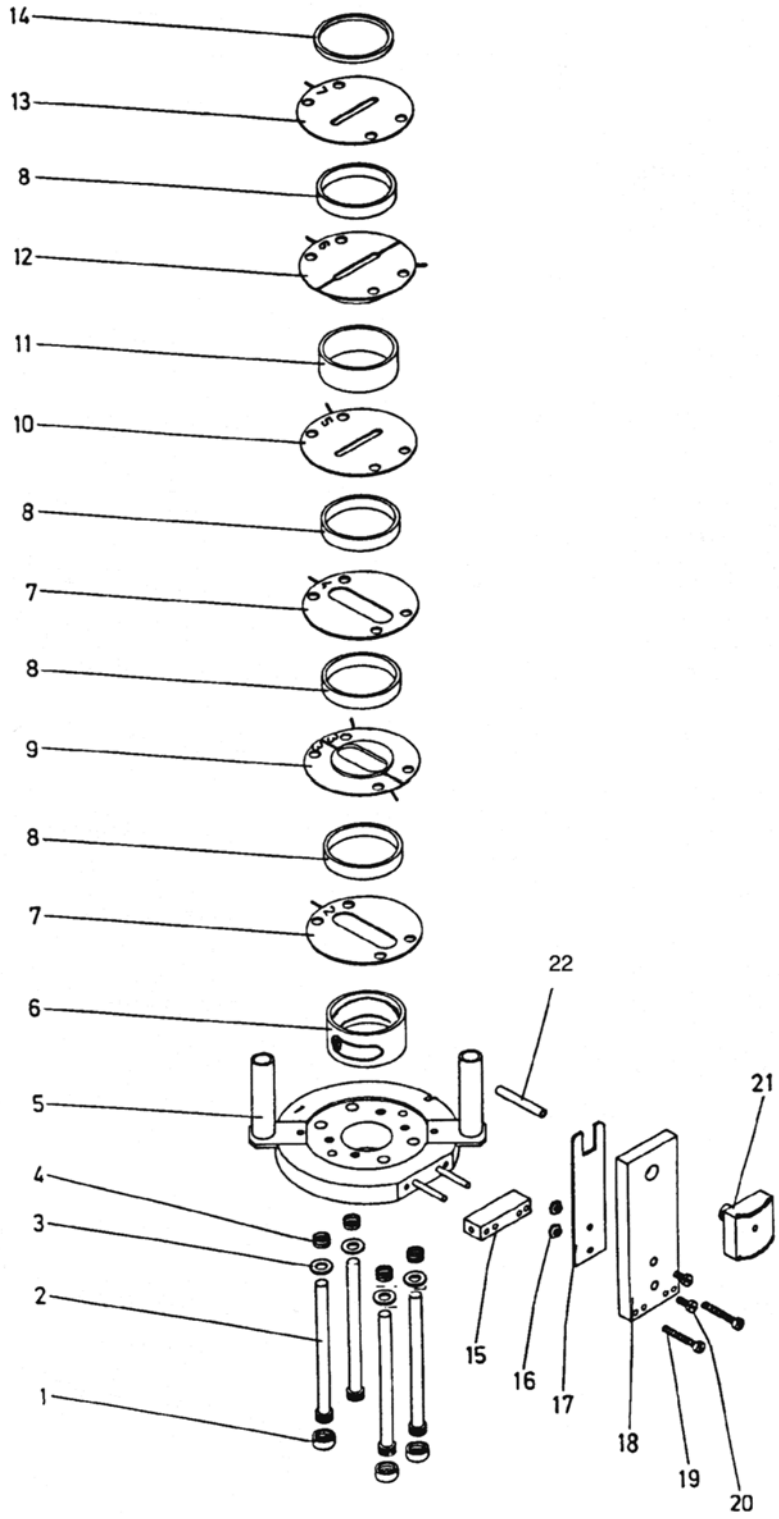
No.	Qty	Designation	Part No.
1	2	Cylinder screw, stainless steel	045 0780
2	2	Compression spring	043 0570
3	1	Ion source, see fig. 8 - 9 to 8 - 12	113 1490
4	1	Gasket; gold, DN 100	055 1600
5	1	Spacer ring, DN 100	095 1380
6	1	Feedthrough flange, with pos. 1, 2, 4, 5, 7 to 10	046 9080
7	13	Washer; stainless steel, I.D. 8.4	047 0070
8	13	Hexagonal screw; stainless steel, M 8x 35	045 4400
9	3	Cylinder screw; stainless steel, M 3 x 6	045 0750
10	2	Cylinder screw; stainless steel, M 4 x 12	045 0810
11	2	Washer; stainless steel, I.D. 4.3	047 0940
12	34	Threaded pin	045 2330
13	17	Clamp	032 1560
14	1	Connection cable with plug	101 5650
15	2	Cylinder screw; stainless steel, M 2 x 6	045 0710
16	2	Washer; stainless steel, I.D. 2.2	047 0010
17	1	Silicon hose supplied by the meter	034 0330
18	-	Wiring connections, see figure 8 - 7	114 7580

Fig. 8 - 9: Ion Source Base



No.	Qty.	Designation	Part No.
1	2	Contact screw	043 9760
2	2	Connection lug with pin	050 5990
3	4	Spacer tube; ceramic, 6 x 4.2 x 4	056 0780
4	2	Supporting tube	016 8170
5	2	Cylinder screw; stainless steel, M 3 x 6	045 0750
6	1	Ion source base	043 9600
7	2	Cylinder pin; stainless steel, 2 x 18 long	048 0360
8	1	Cylinder pin; 2 x 4 long	048 0350
9	2	Cylinder screw; stainless steel, M 2 x 4	045 0690
10	2	Spacer tube; ceramic, 4 x 2.1 x 10	056 0570
11	2	Washer; stainless steel, I.D. 2.5	047 0090
12	2	Nut; stainless steel, M 2	046 0060
13	1	Slit, 0.5 mm	043 9550

Fig. 8 - 10: Ion Source, Lower Part



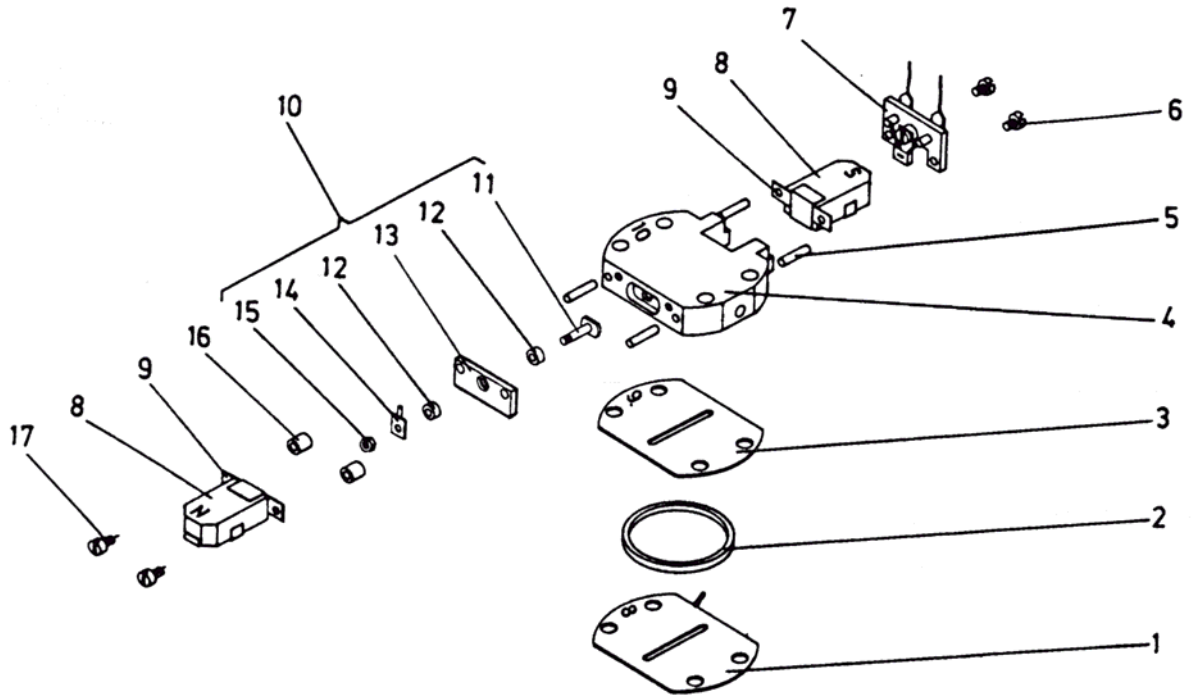
Thermo Finnigan

No.	Qty.	Designation	Part No.
1	4	Threaded sleeve; stainless steel, M 8 x 0.5	091 0450
2	4	Rod; ceramic, length 69.5	043 9530
3	4	Washer; stainless steel,	044 6150
4	4	Compression spring	043 0680
5	1	Ion source base	-
6	1	Spacer tube with slide	079 4750
7	2	Grounding plate	043 9640
8	4	Spacer tube; quartz glass, 35.6 x 30 x 5	078 3920
9	1	Plate, z - deflection	043 9650
10	1	Plate, r - deflection	043 9620
11	1	Spacer tube; quartz glass, 35.6 x 30 x 10	078 3910
12	1	Lens	043 9540
13	1	Shield	043 9630
14*	2	Spacer tube; quartz glass, 35.6 x 30 x 2	078 3930
15	1	Holding block	043 9720
16	2	Nut; stainless steel, M 2	046 0060
17	1	Spring plate	044 6300
18	1	Support, stainless steel	043 9710
19	2	Cylinder screw; stainless steel, M 2 x 16	045 5400
20	2	Cylinder screw, M 2 x 8	045 0720
21	1	Bush, stainless steel	043 9700
22	1	Tube; quartz glass, length 23 cm	078 3900

ATTENTION: ***Open the spacer tube (Pos. 6) for SO_2 operation!***

NOTE: ***The parts marked with an asterisk (*) frequently break. It is recommended to provide for replacements before the source is opened.***

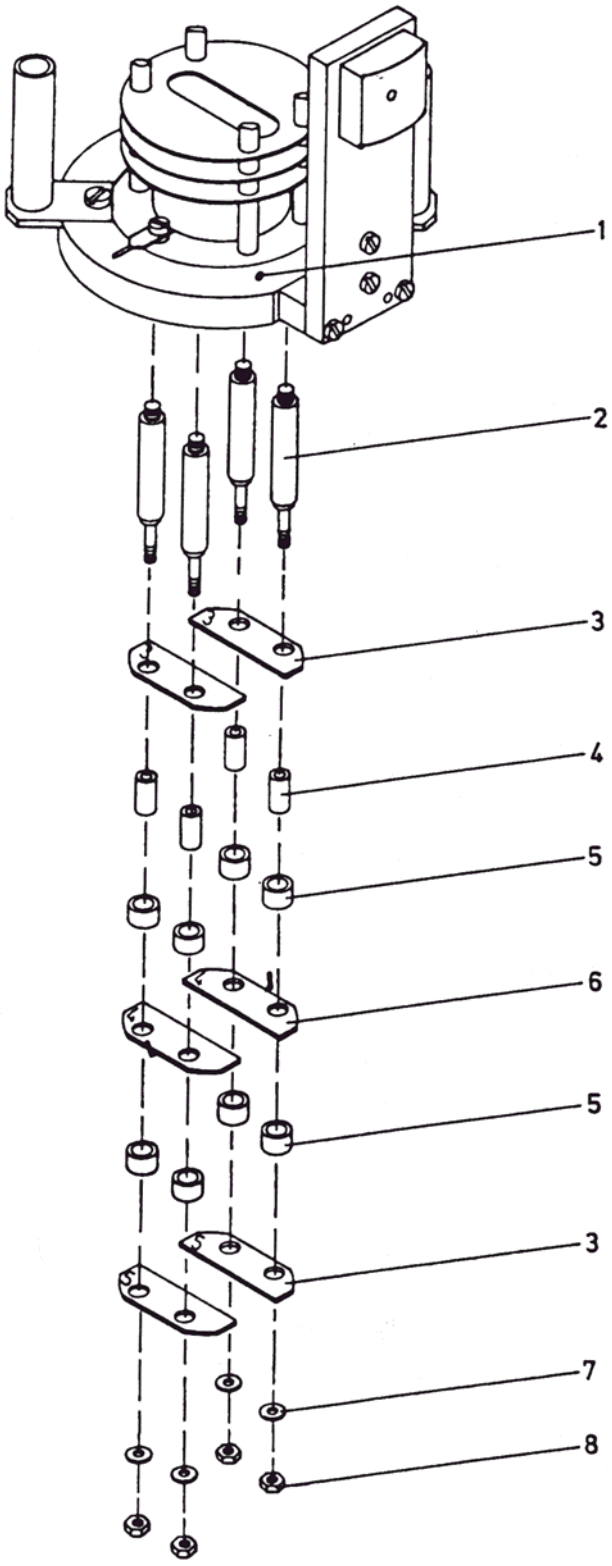
Fig. 8 - 11: Ion Source, Upper Part



No.	Qty.	Designation	Part No.
1	1	Extraction plate	043 9610
2*	1	Spacer tube; quartz glass, 35.6 x 30 x 2	078 3930
3	1	Cover plate	043 9680
4	1	Ionization housing	113 5710
5	4	Cylinder pin; stainless steel, 2 x 12	015 6600
6	2	Cylinder screw; stainless steel, M 2 x 6	045 0710
7*	1	Cathode unit (Filament), tungsten wire	067 2460
8	1	Source magnet (one pair)	044 8030
9	1	Magnet support	043 9820
10	1	Electron collector with pos. no. 11 to 15	043 9790
11	1	Collector (Trap)	043 9810
12*	2	Spacer tube; ceramic, 3.9 x 2.2 x 2	056 1180
13	1	Collector plate	043 9800
14	1	Connection lug	111 9260
15	1	Nut; stainless steel, M 2	046 0060
16	2	Spacer tube, stainless steel	043 9490
17	2	Cylinder screw; stainless steel, M 2 x 8	045 0720

NOTE: ***The parts marked with an asterisk (*) frequently break. It is recommended to provide for replacements before the source is opened.***

Fig. 8 - 12: Ion Source with Grounding Plates



No.	Qty.	Designation	Part No.
1	1	Ion source	-
2	4	Rod, stainless steel	043 9770
3	2	Grounding plate	043 9660
4*	4	Spacer tube; ceramic, 4 x 2.1 x 8	056 0760
5*	8	Spacer tube; ceramic, 6 x 4.2 x 4	056 0780
6	1	Einzel lens	043 9670
7	4	Washer; stainless steel, I.D. 2.5	044 6150
8	4	Nut; stainless steel, M 2	046 0060

NOTE: ***The parts marked with an asterisk (*) frequently break. It is recommended to provide for replacements before the source is opened.***

Fig. 8 -13: Connection Scheme for Lamps

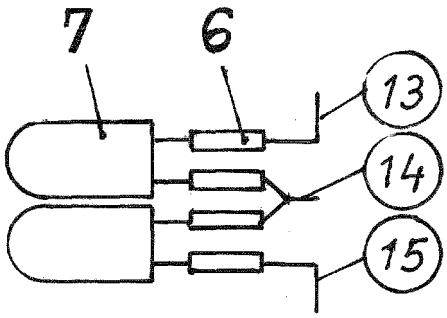
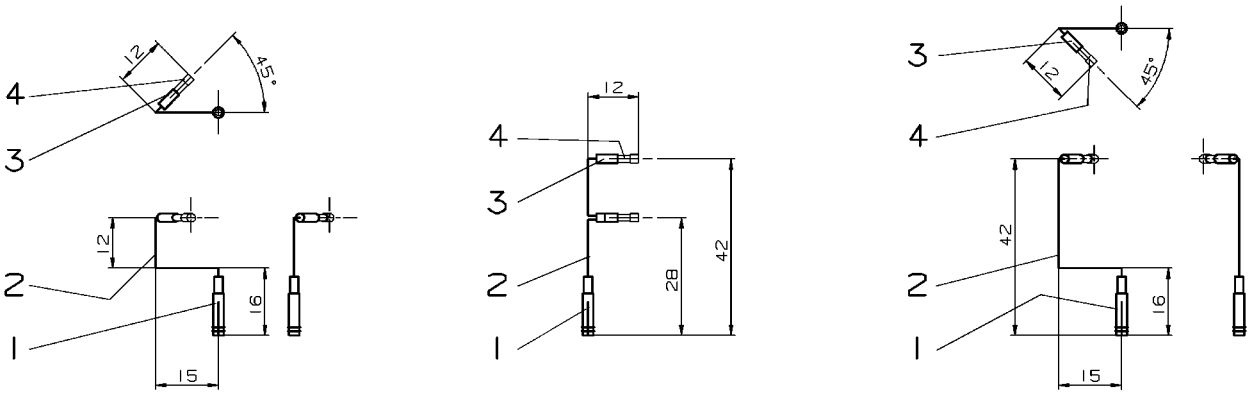


Fig. 8 - 14: Connection Lines for Lamps



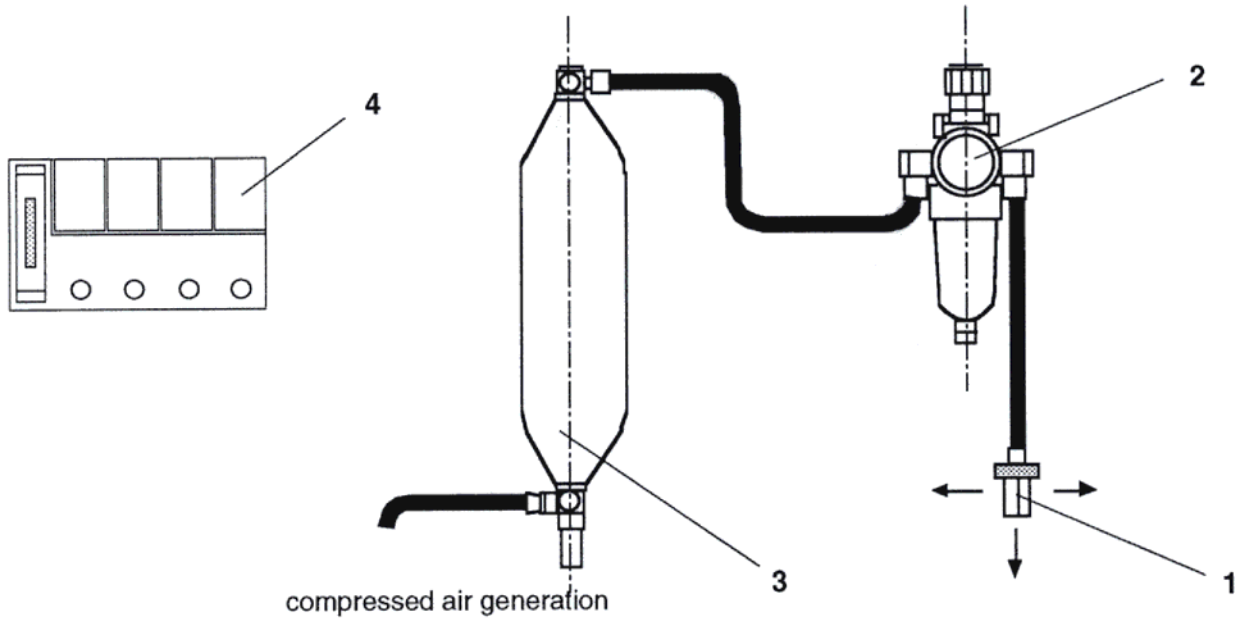
15 **14** **13**

NOTE: ***Drawings do not scale. All dimensions are in mm.***

Pos. No.	Qty.	Designation	Part No.
1	3	Terminal, female 14 long	032 1280
2	100 cm	Wire: 0.5 thick, Ni	062 0500
3	4	Capillary tube, 0.5 x 6 mm	060 5560
4	4	Wrap connection	113 2140
6	1	Connection lines, see above	114 7320
7	2	Halogen lamp 10 W, 12 V	108 5040

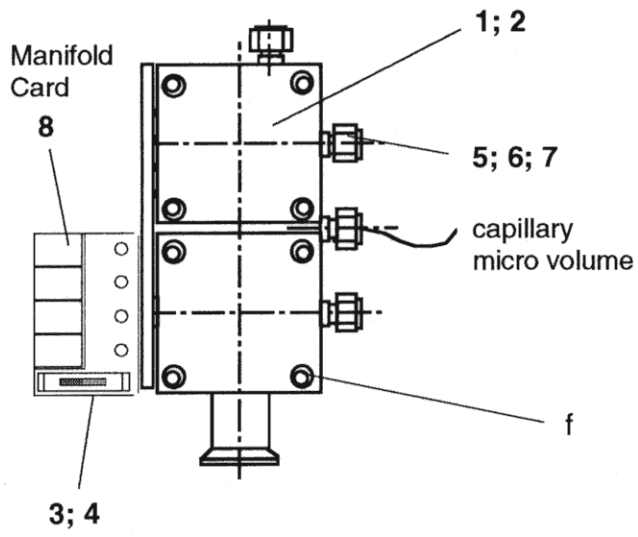
8.7 INLET SYSTEMS

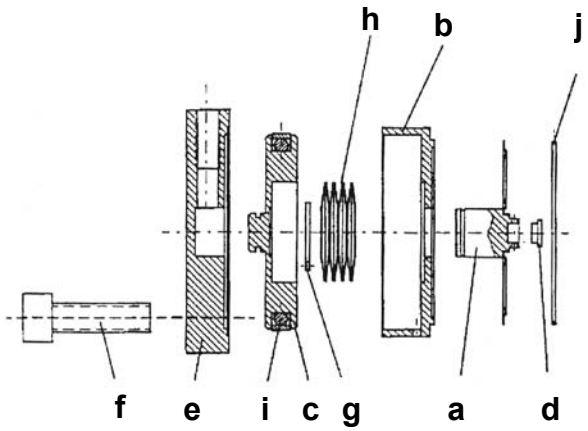
Fig. 8 - 15: Compressed Air Supply (Part No. 108 2520)



Pos. No.	Qty.	Designation	Part No.
1	1	Compressed air distributor	106 8410
2	1	Compressed air service unit	052 1630
3	1	Compressed air store	049 3830
4	5	Valve control unit manifold card	108 3241
		Accessory	
	2	Swivel joint	052 1730
	1	Quick connection	052 1620
	6 m	Hose	069 0740
	4	Screw	045 2130
	4	Washer	047 0050
	11	Sealing ring	050 5260
	5	Blind stopper	052 1950
	4	Coupling compl.	067 4652

Fig. 8 - 16: Basic Module Changeover (Part No. 108 2630)

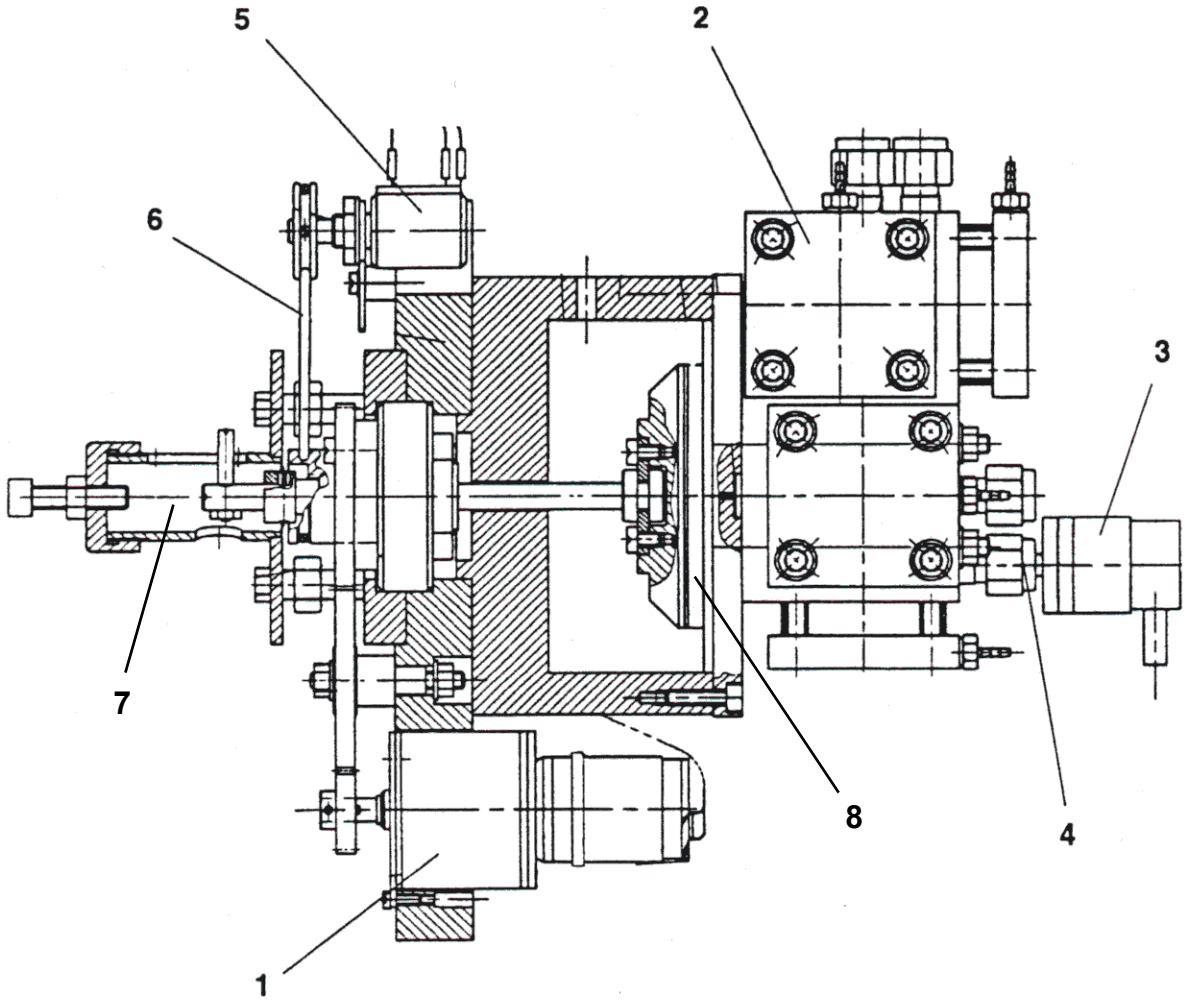




Valve unit, pos. 2

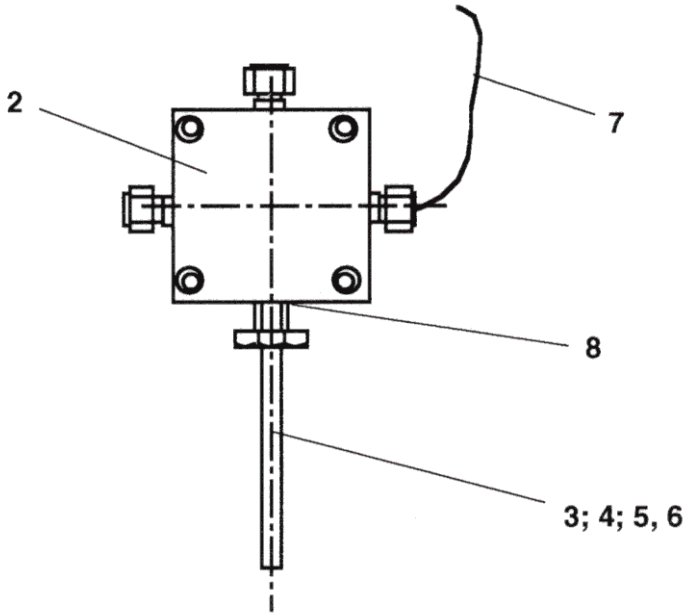
Pos. No.	Qty.	Designation	Part No.
1	1	Valve body	108 2620
2	4	Valve unit	065 3001
a	1	Membrane	065 3010
b	1	Pressure unit	065 3050
c	1	Piston	065 3030
d	1	Stamp, gold	065 3041
e	1	Cover	065 3060
f	4	Screw; M 6 x 20	045 3420
g	1	Guard ring; 10 x 1	047 3430
h	8	Plate spring; 20 x 10.2 x 0.4	043 1570
i	1	Jacket ring	055 3140
j	1	Gasket; gold, 38	054 5270
k	1	Lithium fat	079 1140
3	4	Connecting fitting	070 3780
4	4	Gasket	050 5260
5	8	Cap nut	052 1160
6	8	Front ferrule	079 2800
7	8	Back ferrule	079 2810
8	1	Manifold Card	108 3241

Fig. 8 - 17: Variable Volume (Part No. 108 2681)



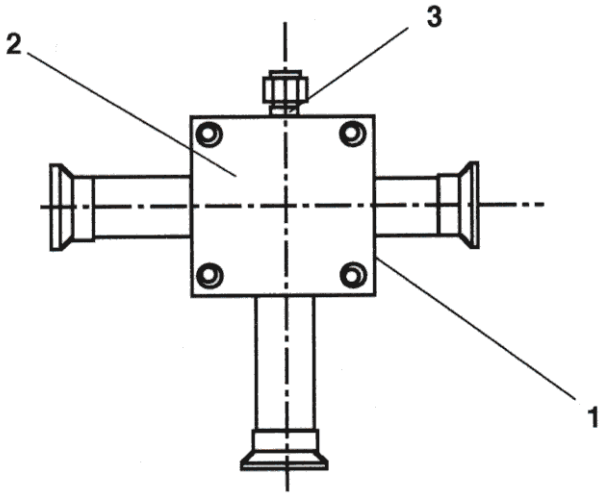
Pos. No.	Qty.	Designation	Part No.
1	1	Motor, compl.	070 3330
2	1	Valve unit	108 2670
3	1	Manometer	101 6802
4	1	Capillary	067 1182
5	1	Potentiometer	070 3540
6	1	O ring	055 3180
7	1	Limit stop	070 3610
8	1	Bellows unit, small	070 3310

Fig. 8 - 18: Micro Volume (Part No. 108 2900)



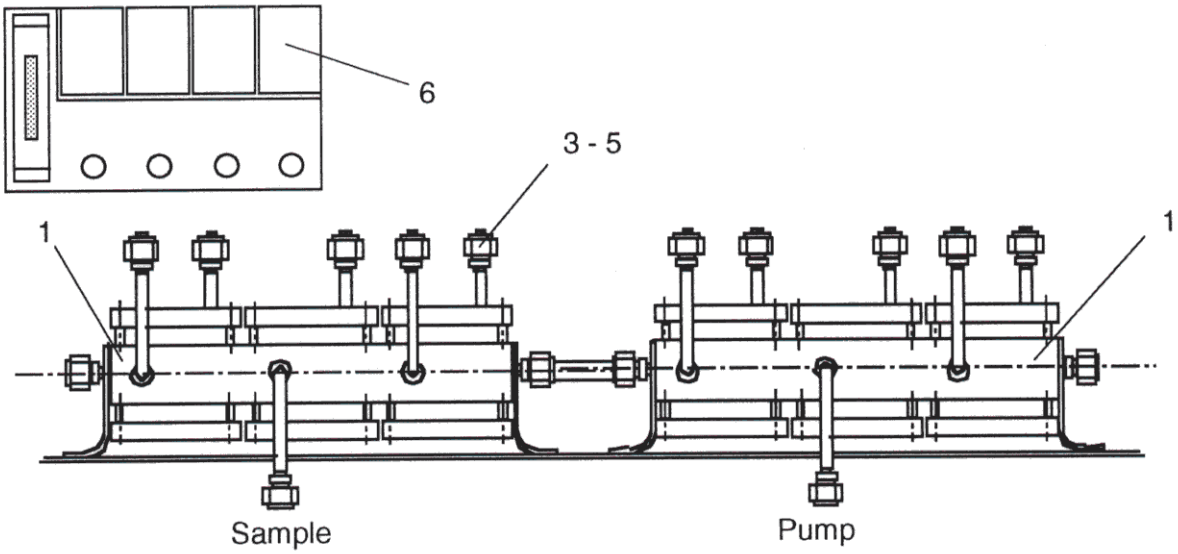
Pos. No.	Qty.	Designation	Part No.
2	1	Valve	106 8970
3	1	Cold finger; CO_2	078 3330
4	1	Cold finger; N_2	078 3340
5	1	Cooling plate; CO_2	041 2300
6	1	Cooling plate; N_2	058 3290
7	1	Capillary tube, heatable	067 1182
8	1	Gasket; gold, 6.3	055 1010

Fig. 8 - 19: Pumping Unit (Part No. 108 3300)



Pos. No.	Qty.	Designation	Part No.
1	1	Valve body	059 3410
2	2	Valve unit	065 3001
3	2	Gasket	050 5260

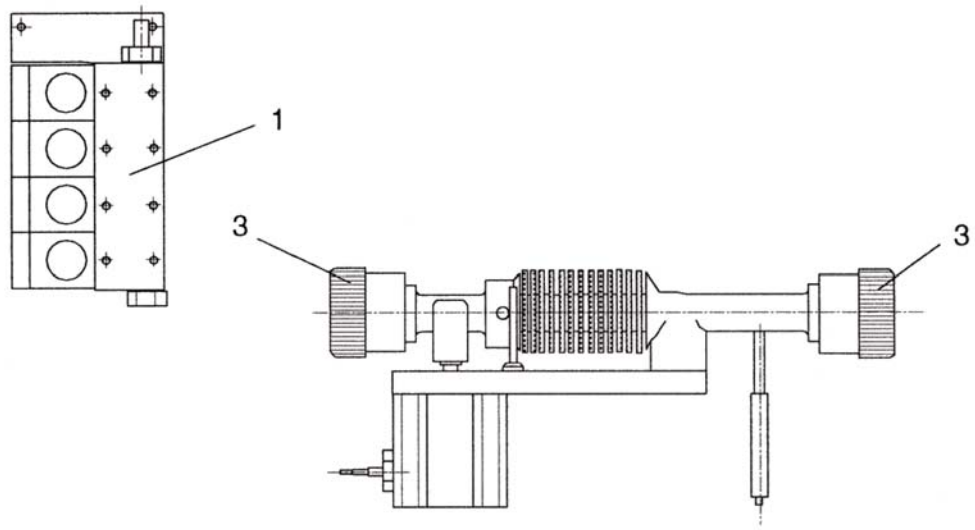
Fig. 8 - 20: Multiport Module (Part No. 108 3200)



Pos. 2 and 7 are not shown

Pos. No.	Qty.	Designation	Part No.
1	2	Valve bloc	106 8770
2	1	Manometer	101 6801
3	5	Nut	052 1160
4	5	Front ferrule	079 2800
5	5	Back ferrule	0792810
6	3	Manifold card	108 3241
7	5 m	Silicon hose	101 5830

Fig. 8-21: Tube Cracker (Part No. 108 2840)

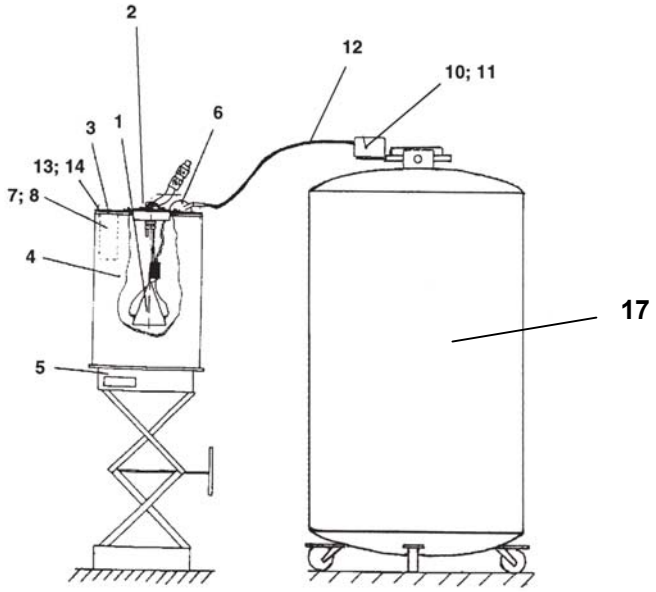


Pos. 2 is not shown

Pos. No.	Qty.	Designation	Part No.
1	3	Manifold Card	109 7750
2	5 m	Silicon hose	101 5830
3	2	O ring	055 2180

8.7.1 AUTOCOOL

Fig. 8 - 22: Autocool (Part No. 049 3661)

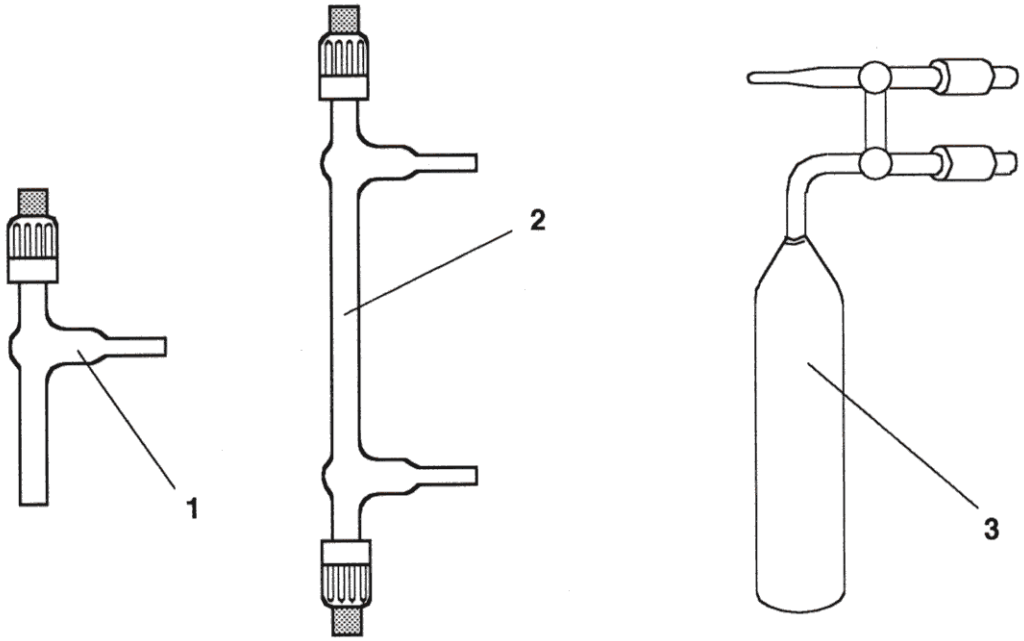


Pos. 16 is not shown

Pos. No.	Qty.	Designation	Part No.
1	1	Cooling unit	079 2400
2	1	Blank flange	059 7380
3	1	Cover	059 6530
4	1	Insulating case	054 2830
5	1	Lifting device	060 9260
6	1	Filler cap	112 8230
7	1	Sleeve	075 4850
8	1	Insert	075 4710
9	1	Refill device	039 5441
10	1	Magnet valve, for LN_2	041 4130
11	1	Manifold, for LN_2	048 2610
12	1	Hose for refill box	059 9610
13	1	Screw; M 4 x 10	045 088
14	4	Nut; M 4	046 0220
15	2	Screw; M 4 x 25	045 0500
16	1	Connection cable f. trap	025 3791
17	1	Container 75 l, f. LN_2	079 4700

8.7.2 ACCESSORIES

Fig. 8 - 23: Accessories



Pos. No.	Qty.	Designation	Part No.
1	1	Sample vial (5 ml)	100 3560
2	1	Sample vial (10 ml)	100 3840
3	1	Sample vial (200 ml)	025 4650

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