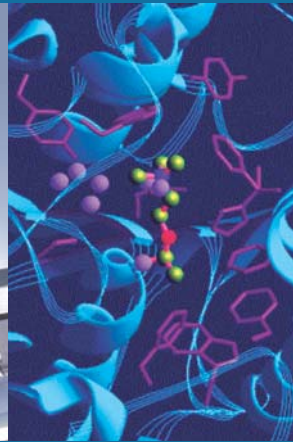
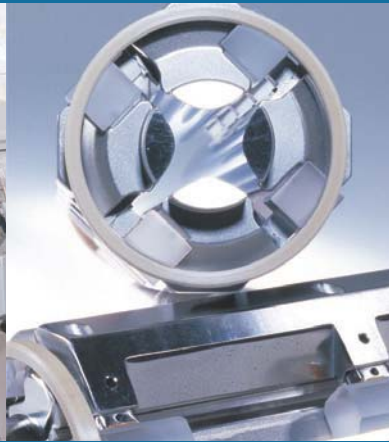


Thermo Fisher Scientific

# **MAT 253** **Operating Manual**

Revision A - 1149090



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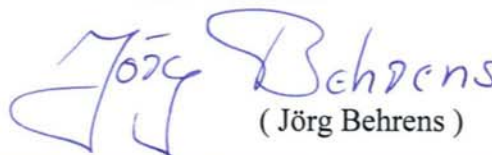
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Bremen, Germany, 2. März 2007

**Technischer Leiter:**  
Director of Operations  
Direttore fabbricazione

**ThermoFisher**  
SCIENTIFIC

  
( Jörg Behrens )



# Read This First

Welcome to the Thermo Scientific MAT 253 system! MAT 253 is a member of the Thermo Scientific family of advanced mass spectrometer (MS) detectors.

## About This Guide

This *MAT 253 Operating Manual* describes the modes of operation and principle hardware components of your MAT 253 instrument. In addition, this manual provides step-by-step instructions for cleaning and maintaining your instrument.

## Who Uses This Guide

This *MAT 253 Operating Manual* is intended for all personnel that need a thorough understanding of the instrument (to perform maintenance or troubleshooting, for example). This manual should be kept near the instrument to be available for quick reference.

## Scope of This Guide

This manual includes the following chapters:

- [Chapter 1: “Getting Started”](#) gives an introduction into both hardware and software of your MAT 253.
- [Chapter 2: “Analyzer”](#) describes the analyzer of the MAT 253.
- [Chapter 3: “Vacuum System”](#) deals with the vacuum system.
- [Chapter 4: “Electronic Devices”](#) describes the electronic devices of the MAT 253.
- [Chapter 5: “Dual Inlet System \(Optional\)”](#) outlines the Dual Inlet system.
- [Chapter 6: “Measurements”](#) outlines how to perform different kinds of sample measurements.
- [Chapter 7: “Diagnosis”](#) gives instructions about performing various types of diagnosis.

**Read This First**  
About This Guide

- [Chapter 8: “Technical Information”](#) outlines the maintenance procedures that you should perform on a regular basis. It also lists the replaceable parts for the MS detector and data system.

## Related Documentation

In addition to this guide, Thermo Fisher Scientific provides the following documents for MAT 253.

The following manuals are available for the peripherals delivered with your instrument:

- *ConFlo IV Operating Manual*
- *GasBench II Operating Manual*
- *Kiel IV Carbonate Device Operating Manual*
- *TC/EA Operating Manual*
- *LC IsoLink Operating Manual*
- *GC IsoLink Operating Manual*

You can access PDF files of the documents listed above from the data system computer. The software also provides Help.

## Contacting Us

There are several ways to contact Thermo Fisher Scientific.

### Assistance

For technical support and ordering information, **visit us on the Web:**

[www.thermoscientific.com/ms](http://www.thermoscientific.com/ms)

Service contact details for customers in Europe are available under:

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### Changes to the Manual

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- Please send your comments (in German or English) to:

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# Typographical Conventions

This section describes typographical conventions that have been established for Thermo Fisher Scientific manuals.

## Data Input

Throughout this manual, the following conventions indicate data input and output via the computer:

- Messages displayed on the screen are represented by capitalizing the initial letter of each word and by italicizing each word.
- Input that you enter by keyboard is identified by quotation marks: single quotes for single characters, double quotes for strings.
- For brevity, expressions such as “choose **File** > **Directories**” are used rather than “pull down the File menu and choose Directories.”
- Any command enclosed in angle brackets < > represents a single keystroke. For example, “press <F1>” means press the key labeled *F1*.
- Any command that requires pressing two or more keys simultaneously is shown with a plus sign connecting the keys. For example, “press <Shift> + <F1>” means press and hold the <Shift> key and then press the <F1> key.
- Any button that you click on the screen is represented in bold face letters. For example, “click on **Close**”.

## Topic Headings

The following headings are used to show the organization of topics within a chapter:

### Chapter 1 Chapter Name

#### Second Level Topics

#### Third Level Topics

#### Fourth Level Topics

## Safety and EMC Information

In accordance with our commitment to customer service and safety, this instrument has satisfied the requirements for the European CE Mark including the Low Voltage Directive.

Designed, manufactured and tested in an ISO9001 registered facility, this instrument has been shipped to you from our manufacturing facility in a safe condition.

This instrument must be used as described in this manual. Any use of this instrument in a manner other than described here may result in instrument damage and/or operator injury.

### Notice on Lifting and Handling of Thermo Scientific Instruments

For your safety, and in compliance with international regulations, the physical handling of this Thermo Scientific instrument *requires a team effort* for lifting and/or moving the instrument. This instrument is too heavy and/or bulky for one person alone to handle safely.

### Notice on the Proper Use of Thermo Scientific Instruments

In compliance with international regulations: If this instrument is used in a manner not specified by Thermo Fisher Scientific, the protection provided by the instrument could be impaired.

### Notice on the Susceptibility to Electromagnetic Transmissions

Your instrument is designed to work in a controlled electromagnetic environment. Do not use radio frequency transmitters, such as mobile phones, in close proximity to the instrument.

## Safety and Special Notices

Make sure you follow the precautionary statements presented in this guide. The safety and other special notices appear different from the main flow of text. Safety and special notices include the following:



**Warning** Warnings highlight hazards to human beings. Each Warning is accompanied by a Warning symbol. ▲

**Caution** Cautions highlight information necessary to protect your instrument from damage. ▲

**Note** Notes highlight information that can affect the quality of your data. In addition, notes often contain information that you might need if you are having trouble. ▲

## Identifying Safety Information

This guide contains precautionary statements that can prevent personal injury, instrument damage, and loss of data if properly followed. Warning symbols alert the user to check for hazardous conditions. These appear throughout the manual, where applicable. The most common warning symbols that appear in Thermo Fisher Scientific manuals are:



**Warning General Hazard.** Danger of injury. This general symbol indicates that a hazard is present that could result in injuries if it is not avoided. The source of danger is described in the accompanying text. ▲



**Warning Electric Current.** Danger of electric shock. High Voltages capable of causing personal injury are used in the instrument. The instrument must be shut down and disconnected from line power before service is performed. Do not operate the instrument with the top cover off. Do not remove protective covers from PCBs. ▲



**Warning Strong Magnetic Field.** Danger of injury or death. Strong magnetic fields are used in the instrument. Keep away with heart pacemakers. Do not bring compressed gas cylinders within close proximity to the instrument. ▲



**Warning Hot Surface.** Danger of burns. Parts of the instrument might be very hot and might cause severe burns if touched. Allow hot components to cool before servicing them. ▲



**Warning Cold Material.** Danger of frostbite. Careless handling of cryogenic liquids might cause severe personal injury including frostbite. Wear protective clothing when operating this equipment including insulated gloves and face shield. ▲



**Warning Corrosive Material.** Danger of injury. Wear gloves when handling toxic, carcinogenic, mutagenic, or corrosive/irritant chemicals. Use approved containers and procedures for disposal of waste solution. ▲



**Warning Laser Radiation.** Danger of injury. Avoid eye or skin exposure to direct or scattered radiation! ▲



**Warning Ultraviolet Radiation.** Danger of injury. Intense ultraviolet radiation is generated by the inductively coupled plasma. This radiation is present within the torch box area. Never attempt to override or defeat the interlock system. ▲



**Warning Radio Frequency Radiation.** Danger of injury. Radio frequency radiation is generated to produce plasma. This radiation is present within the torch box area. Never attempt to override or defeat the interlock system. ▲



**Warning Flammable Gases.** Danger of injury. Use care when operating the system in the presence of flammable gases. ▲



**Warning Explosive Gases.** Danger of injury. Use care when operating the system in the presence of explosive gases. Ventilate the laboratory carefully. ▲

In addition, every instrument has specific hazards. So, be sure to read and comply with all precautions described in this guide. They will help ensure the safe, long-term use of your system.

## General Safety Precautions

Observe the following safety precautions when you operate or perform service on your instrument:

- The system should be operated by trained personnel only. Read the manuals before starting the system and make sure that you are familiar to the warnings and safety precautions!
- Accurate results can be obtained only, if the system is in good condition and properly calibrated.
- Service by the customer should be performed by trained qualified personnel only and should be restricted to servicing mechanical parts! Service on electronic parts should be performed by Thermo Fisher Scientific Service Engineers only!
- Before plugging in any of the instrument modules or turning on the power, always make sure that the voltage and fuses are set appropriately for your local line voltage.
- Only use fuses of the type and current rating specified. Do not use repaired fuses and do not short-circuit the fuse holder.

- The supplied power cord must be inserted into a power outlet with a protective earth contact (ground). When using an extension cord, make sure that the cord also has an earth contact.
- Do not change the external or internal grounding connections. Tampering with or disconnecting these connections could endanger you and/or damage the system.
- The instrument is properly grounded in accordance with regulations when shipped. You do not need to make any changes to the electrical connections or to the instrument's chassis to ensure safe operation.
- Never run the system without the housing on. Permanent damage can occur. When leaving the system, make sure that all protective covers and doors are properly connected and closed, and that heated areas are separated and marked to protect for unqualified personnel!
- Do not turn the instrument on if you suspect that it has incurred any kind of electrical damage. Instead, disconnect the power cord and contact a Thermo Fisher Scientific Service Engineer for a product evaluation. Do not attempt to use the instrument until it has been evaluated. (Electrical damage may have occurred if the system shows visible signs of damage, or has been transported under severe stress.)
- Damage can also result if the instrument is stored for prolonged periods under unfavorable conditions (for example, subjected to heat, water, etc.).
- Always disconnect the power cord before attempting any type of maintenance.
- Capacitors inside the instrument may still be charged even if the instrument is turned off.
- Never try to repair or replace any component of the system that is not described in this manual without the assistance of your Thermo Fisher Scientific Service Engineer.
- Do not place any objects – especially not containers with liquids – upon the instrument. Leaking liquids might get into contact with electronic components and cause a short circuit.

## **Safety Advice for Possible Contamination**

### **Hazardous Material Might Contaminate Certain Parts of Your System During Analysis.**

In order to protect our employees, we ask you to adhere to special precautions when returning parts for exchange or repair.

If hazardous materials have contaminated mass spectrometer parts, Thermo Fisher Scientific can only accept these parts for repair if they have been properly decontaminated. Materials that due to their structure and the applied concentration might be toxic or that are reported in publications to be toxic are regarded as hazardous. Materials that will generate synergetic hazardous effects in combination with other present materials are also considered hazardous.

Your signature on the Health and Safety Form confirms that the returned parts have been decontaminated and are free of hazardous materials. Download the form from [decon.thermo-bremen.com](https://decon.thermo-bremen.com) or order it from the Thermo Fisher Scientific field service engineer.

Parts contaminated by radioisotopes should not be returned to Thermo Fisher Scientific—neither under warranty nor within the exchange part program. If unsure about parts of the system possibly being contaminated by hazardous material, please make sure the Thermo Fisher Scientific field service engineer is informed before the engineer starts working on the system.

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# Chapter 1 Getting Started

This chapter gives an introduction into both hardware and software of your MAT 253. It contains the following topics:

- “Introduction” on page 1-2
- “System Preparation” on page 1-5
- “Software Installation” on page 1-17
- “Starting Isodat the Very First Time” on page 1-25
- “Creating a Gas Configuration” on page 1-28
- “Creating a Configuration” on page 1-32
- “Calibrating/Adjusting the System” on page 1-35

## Introduction

This manual describes the functions and the fundamental measuring procedures of your 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 this manual before starting up your instrument. A basic knowledge of handling computers and of the Isodat software is assumed for proper operation of the MAT 253.

To reach a high level of performance with the MAT 253, we recommend making use of the operator courses at our facilities in Bremen, and/or on-site.

## Basic Instrument

With the MAT 253, you can measure gas isotope ratio of H/D,  $^{13}\text{C}/^{12}\text{C}$ ,  $^{15}\text{N}/^{14}\text{N}$ ,  $^{18}\text{O}/^{16}\text{O}$ ,  $^{34}\text{S}/^{32}\text{S}$  (from  $\text{SO}_2$  and  $\text{SF}_6$ ),  $^{29}\text{Si}/^{28}\text{Si}$  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. Inlet systems supplied by Thermo Fisher Scientific 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, [Figure 1-1](#), 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. In the manual describing the peripheral equipment, you will find detailed information about other inlet systems such as the “on-line” coupling to gas chromatographs or to Elemental Analyzers.

Make yourself familiar with all the controls at the front and all the connection and installations at the rear of your instrument. Refer to “System Preparation” on page 1-5.



**Figure 1-1.** Front view of MAT 253

## 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**

IsodatScript 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**

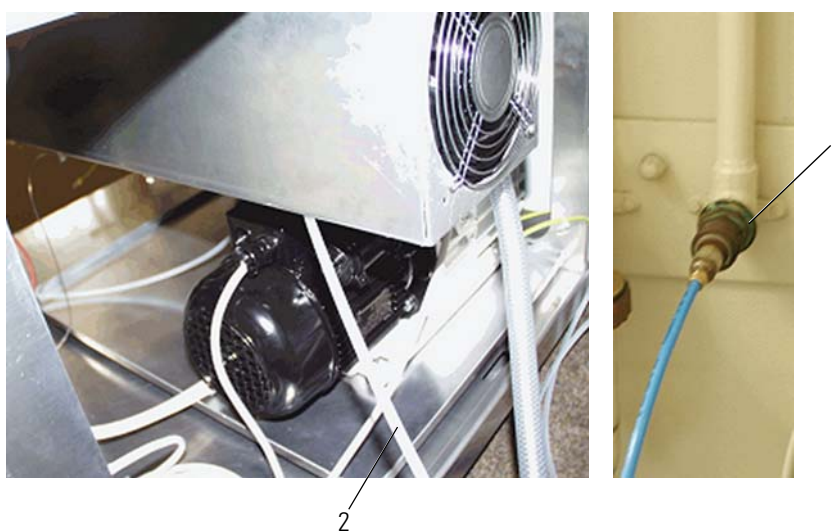
Isodat provides a comprehensive set of customizable data evaluation routines. Standard report forms are provided according to the application. In addition, reports can be easily customized using the unique Result Workshop of Isodat.

## System Preparation

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

### ❖ To install the instrument

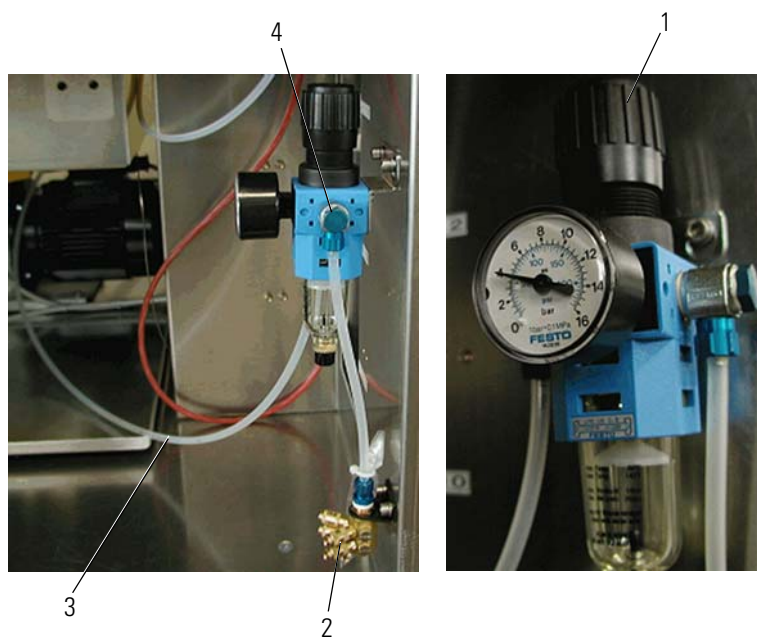
1. Unpack your MAT 253 and arrange it at the desired place in your laboratory.
2. Connect the end of the tube for compressed air (a thin transparent tube which leaves the IRMS at its rear panel, see **2** [Figure 1-2](#)) to your wall outlet or a compressor for compressed air. You need at least 5 bar. See [Figure 1-2](#).



Labeled Components: 1=wall outlet for compressed air, 2=tube to wall outlet for compressed air

**Figure 1-2.** Compressed air connections

3. Connect the other end of the tube for compressed air **3** in [Figure 1-3](#) to the pressure regulator **4** in [Figure 1-3](#) behind the front panel of the IRMS as shown in [Figure 1-3](#).



Labeled Components: 1=pressure regulating screw, 2=distributor with ports, 3=compressed air tube, 4=pressure regulator

**Figure 1-3.** Pressure regulator

4. To vary the pressure, pull out the pressure-regulating screw and turn it.  
Usually, the pressure regulator (**4** in [Figure 1-3](#)) is factory-set via the pressure-regulating screw (**1** in [Figure 1-3](#)) to display 4 bar.
5. Insert your peripherals at the distributor (down right behind the front panel). See [Figure 1-4](#). Because the ports of the distributor are equivalent, it is unimportant which peripheral to insert at which port.



**Figure 1-4.** Distributor with ports

6. Lay the waste gas tube (at the output of the vacuum pumps) outdoors to prevent the accumulation of oil mist and dangerous gases (CO or H<sub>2</sub>, for example). See [Figure 1-5](#).



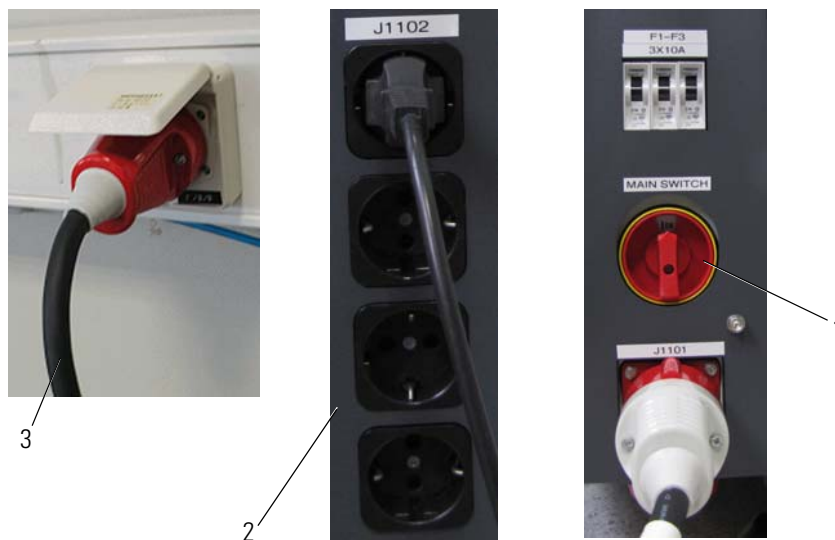
Labeled Components: 1=to fore vacuum pump, 2=to fore vacuum pump, 3=outward, 4=outward

**Figure 1-5.** Waste gas tube

## Power Supply

### ❖ To establish the power supply

1. Connect your IRMS to the power supply. See **3** in [Figure 1-6](#).



Labeled Components: 1=main switch, 2=sockets for peripherals to IRMS, 3=wall socket and plug

**Figure 1-6.** Connecting IRMS and peripherals to power supply

At the rear side of the instrument above the main switch are sockets to connect the power cables of peripherals (see **2** in [Figure 1-6](#)), 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.

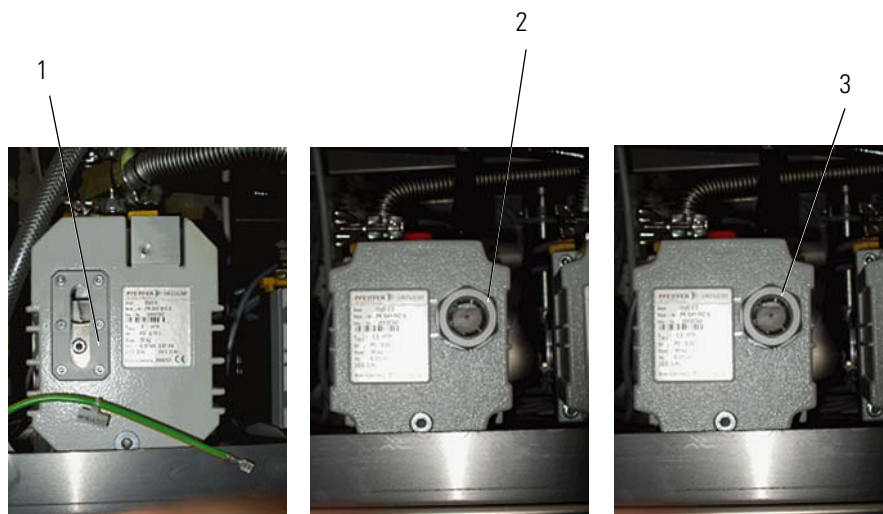
2. Turn on the IRMS by setting the main switch to position ON. The main switch is located at the rear panel of the electronic cabinet, see **1** in [Figure 1-6](#).

## Pumping System

This section describes the main components that generate the vacuum in the analyzer system of the MAT 253.

## Fore Vacuum Pumps

As shown in [Figure 1-7](#), three fore vacuum pumps are used:



Labeled Components: 1=Pfeiffer Duo 5 for the source, 2=Pfeiffer Duo 2.5 for the analyzer, 3=Pfeiffer Duo 2.5 for the inlet system

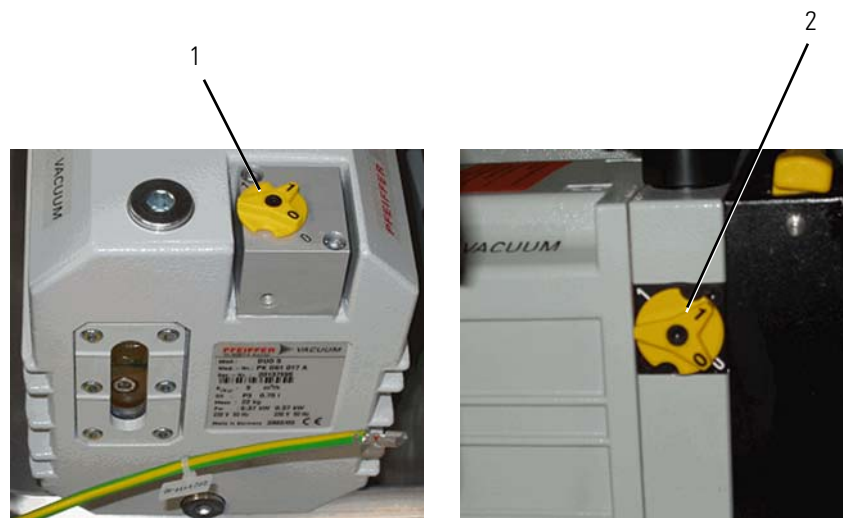
**Figure 1-7.** Fore vacuum pumps in use

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.

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.

The gas ballast is shut by turning the switch to position 0. With the Pfeiffer Duo 5, the switch is located on top of the pump, see **1** in [Figure 1-8](#). With the Pfeiffer Duo 2.5, the switch is located at the side, see **2** in [Figure 1-8](#):



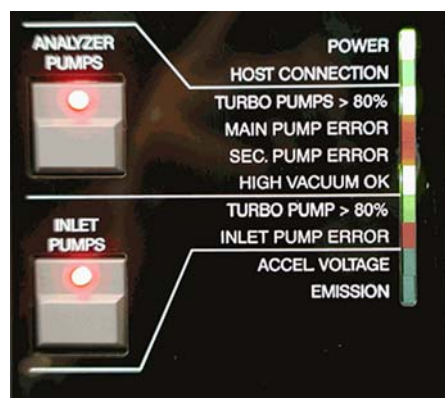
Labeled Components: 1=Pfeifer Duo 5 (switch on top), 2= Pfeiffer Duo 2.5 (switch at the side)

**Figure 1-8.** Pump switches

For detailed information – for example concerning handling and maintenance – refer to the Pfeiffer Operating Instructions of your pumps.

## Pump Switches

The control panel at the front panel of the electronic cabinet shows LEDs and switches for operating the pumps, see [Figure 1-9](#).



**Figure 1-9.** Pump operation via control panel

Turn on the pumps by pressing the ANALYZER PUMPS switch. If an optional Dual Inlet system is operated, press the INLET PUMPS switch.

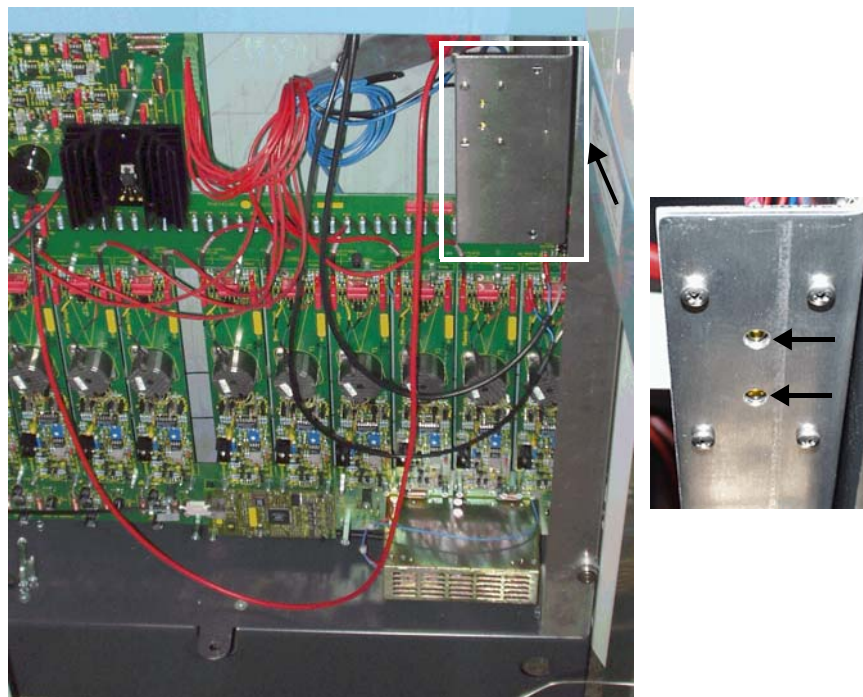
**Note** All the LEDs will remain off with the exception of the Power LED, which will be red. ▲

- 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. For example after about 15 min, the security threshold of  $\approx 3 \times 10^{-5}$  mbar has not been reached.

**Note** If the vacuum is properly established, the red error LEDs remain off. ▲

## Source Heater

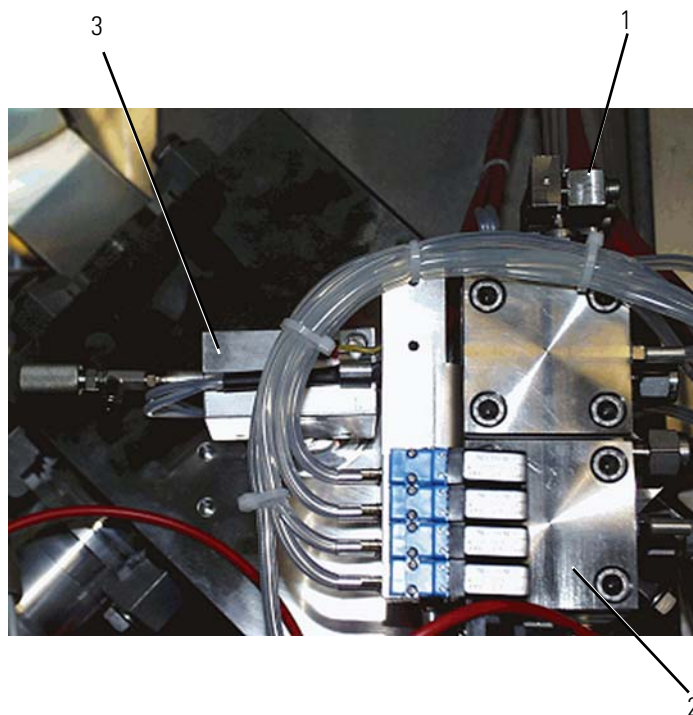
The source heater control LEDs are located at left side of the IRMS, in the top right corner. See [Figure 1-10](#). When the source heater is turned on, both LEDs must be on. Otherwise, one of the heaters might be defective.



**Figure 1-10.** LEDs of source heater

## Heater of Changeover Valve and/or Needle Valve

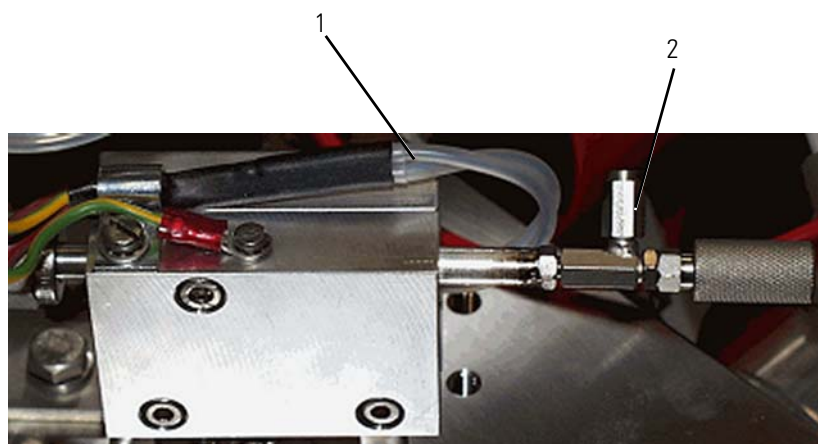
Switch on the heater of the Changeover valve and/or the heater of the needle valve (in case of Continuous Flow applications). This will help to prevent water condensation. See [Figure 1-11](#).



Labeled Components: 1=device for crimp adjustment, 2=Changeover valve (COV) including Changeover valve heater, 3=needle valve including needle valve heater

**Figure 1-11.** Needle valve and Changeover valve

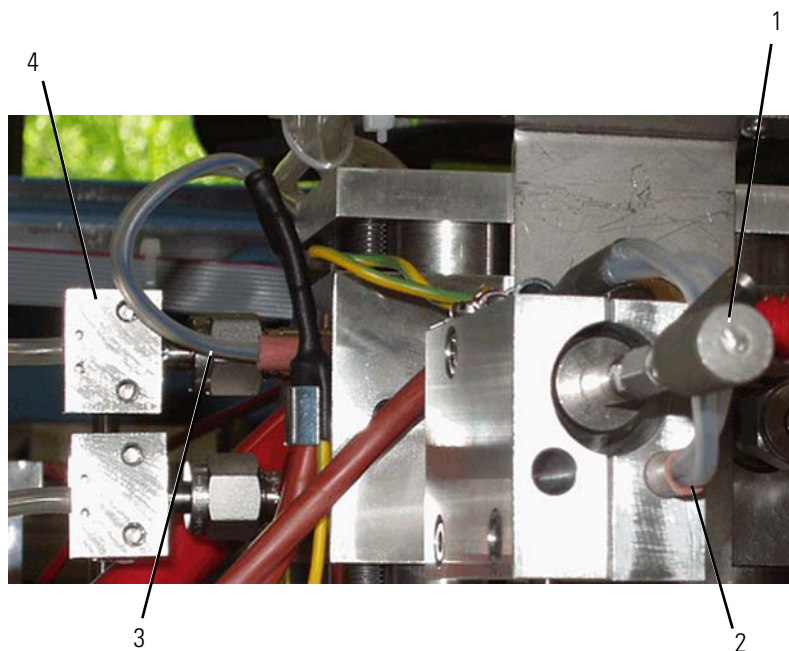
[Figure 1-12](#) shows the needle valve including the needle valve heater in side view:



Labeled Components: 1=to needle valve heater, 2=port for capillary

**Figure 1-12.** Needle valve including needle valve heater (side view)

Figure 1-13 shows the needle valve and the device for crimp adjustment in frontal view.



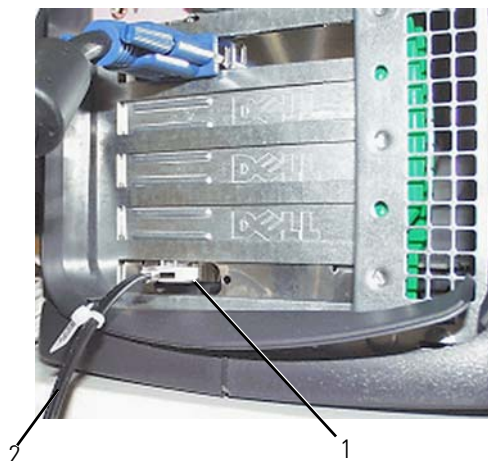
Labeled Components: 1=needle valve, 2=to needle valve heater (inside metal block), 3=to Changeover valve heater (inside metal block), 4=device for crimp adjustment

**Figure 1-13.** Needle valve and device for crimp adjustment

## IRMS-Computer Connection

### ❖ To connect IRMS and computer

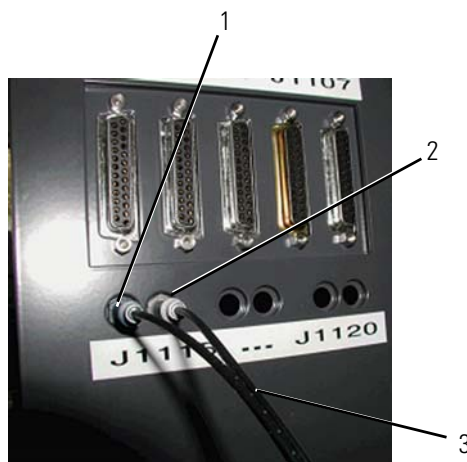
1. Connect the fiber line to the respective port at the rear panel of the computer. See [Figure 1-14](#).



Labeled Components: 1=fiber line connector, 2=fiber line

**Figure 1-14.** Connecting fiber line to computer

2. 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. See [Figure 1-15](#).



Labeled Components: 1=blue connector, 2=gray connector, 3=fiber line

**Figure 1-15.** Connecting fiber line to IRMS

The green LED "Host Connection" ([Figure 1-9](#)) switches ON only when the Isodat has started and verified that the connection between instrument and PC is established. Refer to the chapters "[Software Installation](#)" on [page 1-17](#) and "[Starting Isodat the Very First Time](#)" on [page 1-25](#).

## IRMS-Peripherals Connection

Five identical SUB D ports at the rear panel of the IRMS can be used to connect peripherals. See [Figure 1-16](#).



**Figure 1-16.** SUB D ports at rear panel of IRMS

Peripherals are identified automatically by a plug and measure concept. Refer to “[Plug and Measure](#)” on [page 4-4](#). If Isodat is trying to get access to a peripheral and cannot identify it, an error message will occur.

## Gas Connection

[Figure 1-17](#) shows sample gas connected to a MAT 253.



Labeled Components: 1=right inlet (not used), 2=left inlet connected to gas bottle

**Figure 1-17.** Connecting a gas bottle to the sample side of the dual inlet.

Usually, standard gas is connected to the right inlet side, whereas the sample gas is connected to the left inlet side.

## Software Installation

This chapter explains how to install Isodat.

### Creating a Backup

Installing Isodat deletes the previous version. Be sure to backup the previous version to save your Configurations and measurement data before starting the installation. Backups can be created and restored by using the VersionHandler in the folder C:\Isodat\_Backups.

#### ❖ To create a backup

1. Double-click **VersionHandler.exe** in the folder C:\Isodat\_Backups to open the **VersionHandler**.  
This program was delivered and installed with the previous version of Isodat.
2. Make sure that you have enough free disk space for the backup that includes your Result files.
3. Select **Version > Backup current**.  
A backup of the current version of Isodat is created and stored in a subdirectory, for example “Version\_2.5”, of the folder C:\Isodat\_Backups. If you cancel the backup, a message opens.

Each backup includes your Result files and is stored in dedicated subdirectory of its own in the folder C:\Isodat\_Backups.

### Installing Isodat

**Note** Installation of Isodat deletes previous the version. Isodat configurations and result data will be lost. If Isodat is already installed, refer to chapter “[Creating a Backup](#)” on [page 1-17](#). ▲

#### ❖ To install Isodat (shipped on the CD) via the Startup screen

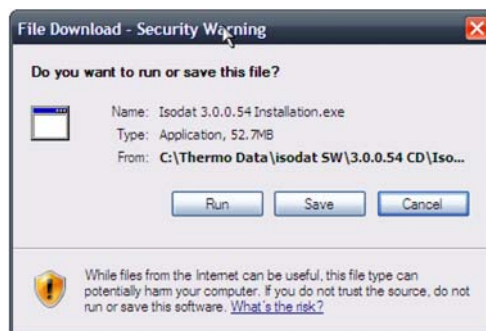
1. Close all applications, particularly previous versions of Isodat if already installed.  
Installation of Isodat is not possible while any part of Isodat is running.
2. Insert the Isodat CD into the CD drive of the Isodat computer.  
If your CD ROM drive is set to “Autostart”, the file “startup.html”

on the Isodat CD is executed and the startup screen appears, see [Figure 1-18](#).



**Figure 1-18.** Isodat startup screen

3. From the Startup screen, select **Install Isodat 3.0**. The File Download dialog opens, see [Figure 1-19](#).



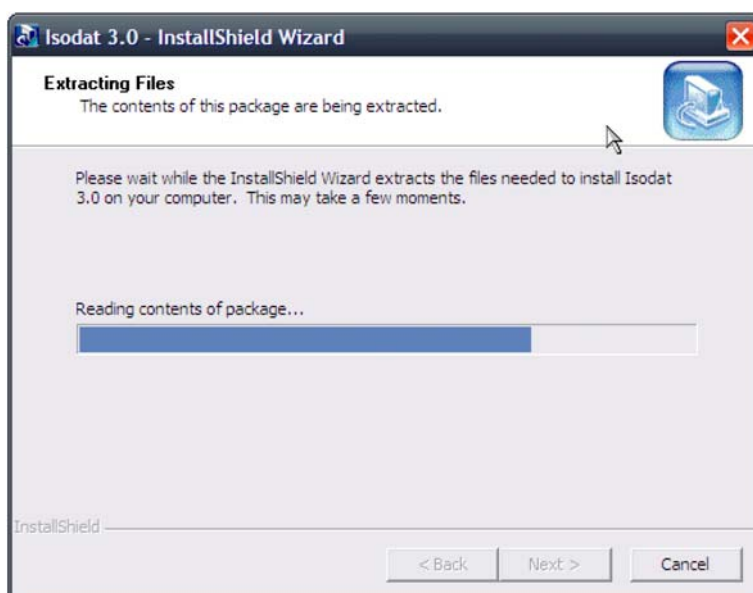
**Figure 1-19.** File download security warning dialog

4. Click **Run**.  
The Internet Explorer dialog opens, see [Figure 1-20](#).



**Figure 1-20.** Internet Explorer security warning dialog

5. Click **Run** to continue the installation.  
The InstallShield® Wizard dialog opens, see [Figure 1-21](#).



**Figure 1-21.** InstallShield wizard extracting files

6. Wait while the InstallShield Wizard is extracting the files.
7. Click **Next**.  
The VersionHandler dialog opens, see [Figure 1-22](#).



**Figure 1-22.** VersionHandler message

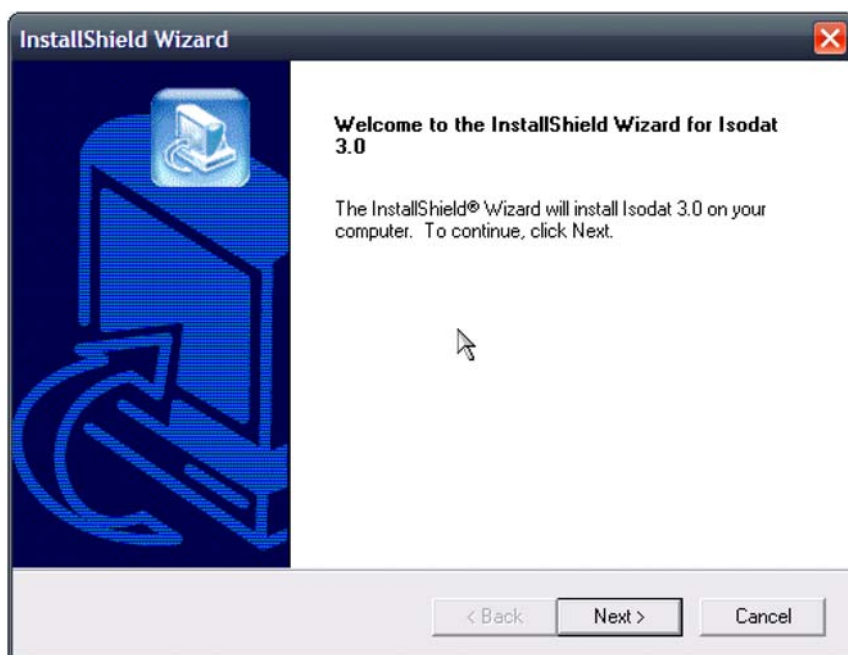
- Click **Yes** to continue.  
The previous version of Isodat is deleted. If you click **No**, the installation is canceled and you can create a backup first, refer to “Creating a Backup” on page 1-17.

A confirmation dialog opens, see [Figure 1-23](#):



**Figure 1-23.** VersionHandler confirmation message

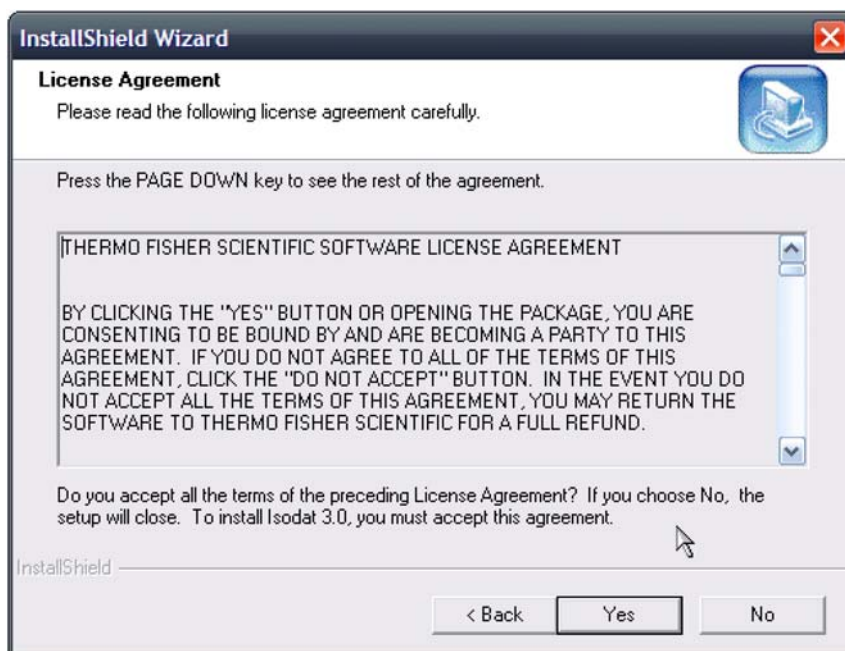
- Confirm with **Yes**.
- The Welcome page of the InstallShield Wizard opens, see [Figure 1-24](#).



**Figure 1-24.** Welcome page of InstallShield Wizard

11. Click **Next**.

The License Agreement dialog box opens, see [Figure 1-25](#).



**Figure 1-25.** Software License agreement

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

The Customer Information dialog box opens, see [Figure 1-26](#).

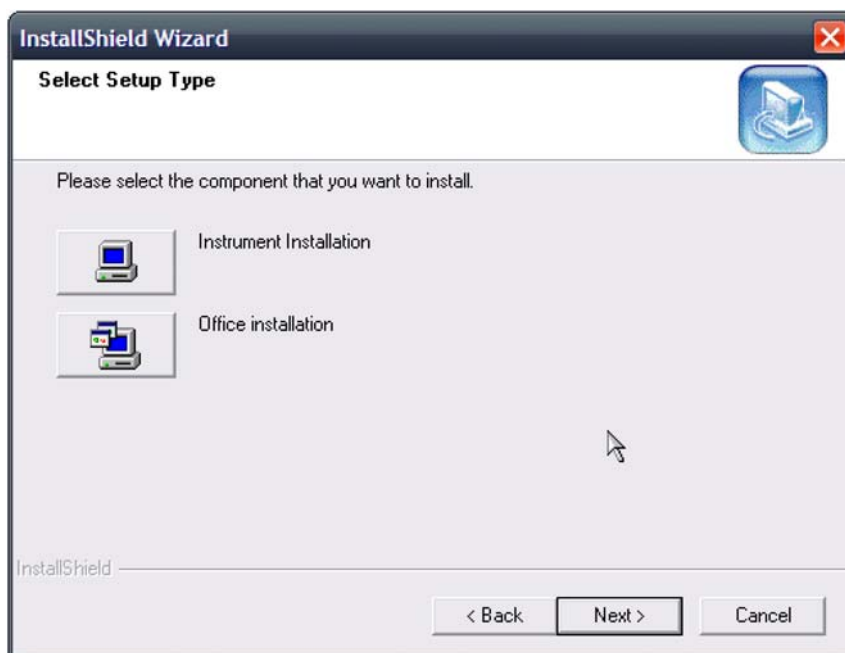


**Figure 1-26.** Entering name and company

13. Enter your name and company.

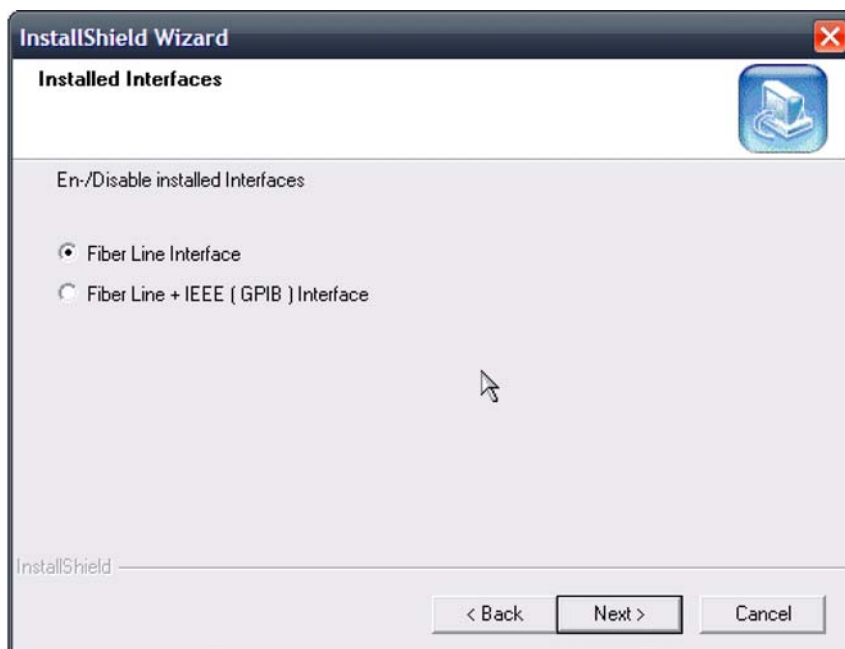
The Next button is activated after you entered the information.

14. Click **Next**.  
The Select Setup Type dialog opens, see [Figure 1-27](#)



**Figure 1-27.** Selecting setup type

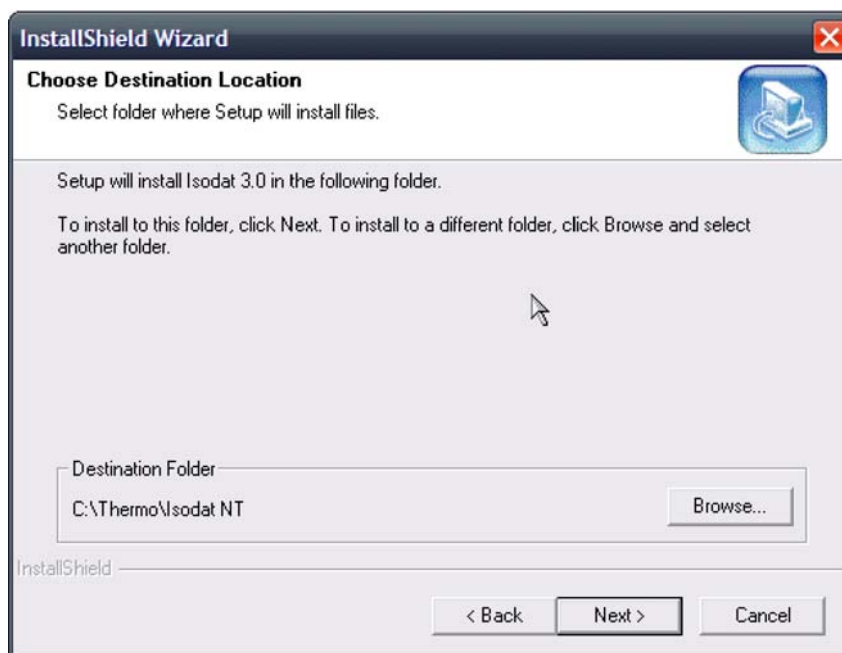
15. Select **Instrument Installation** and click **Next**.  
The Installed Interfaces dialog opens, see [Figure 1-28](#).



**Figure 1-28.** Selecting interfaces

16. Select **Fiber Line Interface** and click **Next**.

The Choose Destination Location dialog opens, see [Figure 1-29](#).

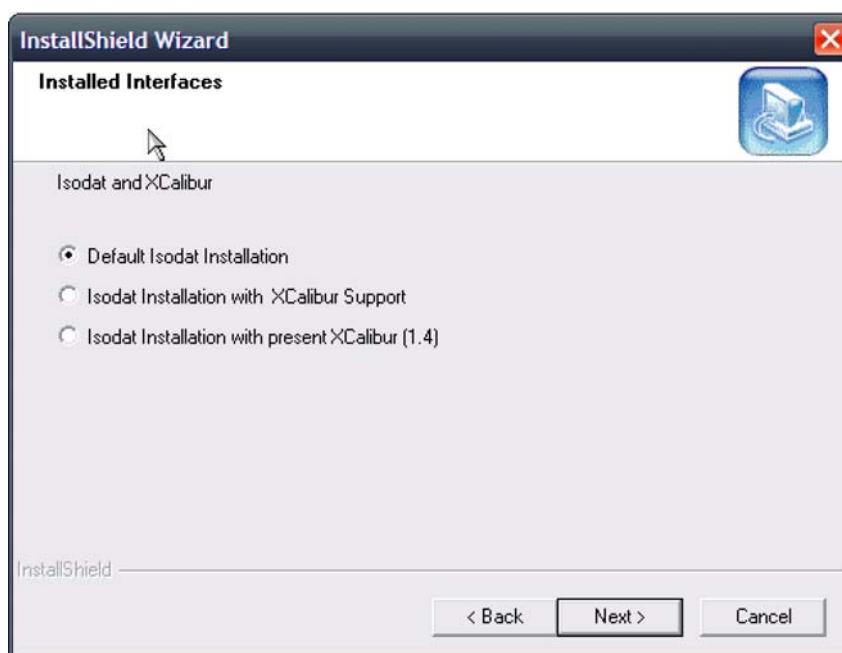


**Figure 1-29.** Defining destination location

17. Accept the proposed folder location or click **Browse** to choose another. Thermo Fisher Scientific recommends to accept the default settings.

18. Click **Next**.

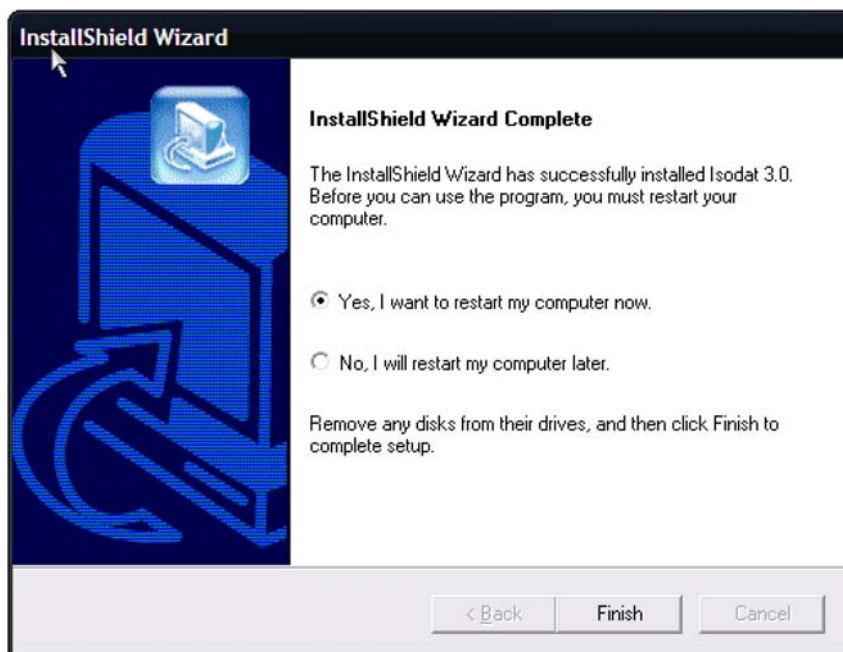
The Isodat and Xcalibur dialog opens, see [Figure 1-30](#).



**Figure 1-30.** Isodat and Xcalibur dialog

The Isodat and XCalibur dialog offers possibility to install XCalibur features needed for some peripherals, for example, LC IsoLink. Refer to the operating manual of your peripherals for details.

19. Select **Default Isodat Installation** and click **Next**.  
The InstallShield Wizard Complete dialog opens, see [Figure 1-31](#).



**Figure 1-31.** Isodat setup complete

20. Select **Yes, I want to restart your computer now**.
21. Click **Finish** to restart.  
After restart Isodat is installed on your computer.

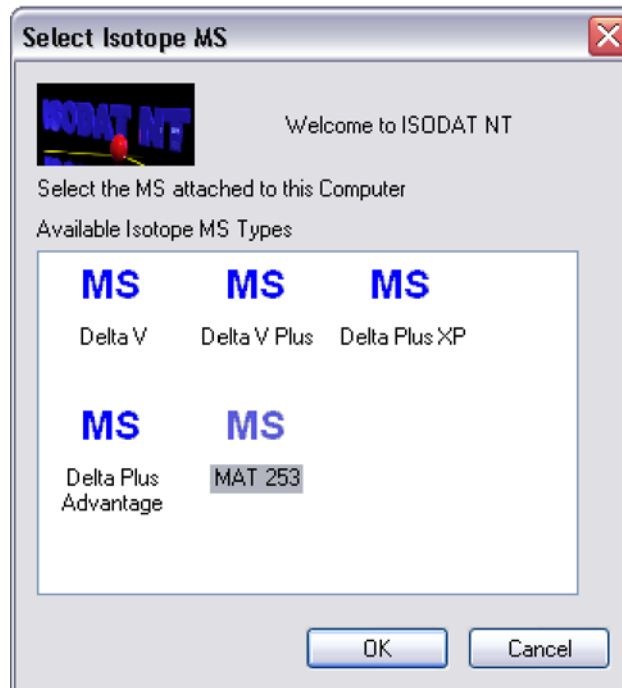
## Starting Isodat the Very First Time

### ❖ To start Isodat

1. After installing, double-click the icon Isodat to start the program.

2. In the Isodat folder, double-click  to open the **Configurator** tool.

The **Select Isotope MS** window opens, see [Figure 1-32](#).



**Figure 1-32.** Selecting IRMS

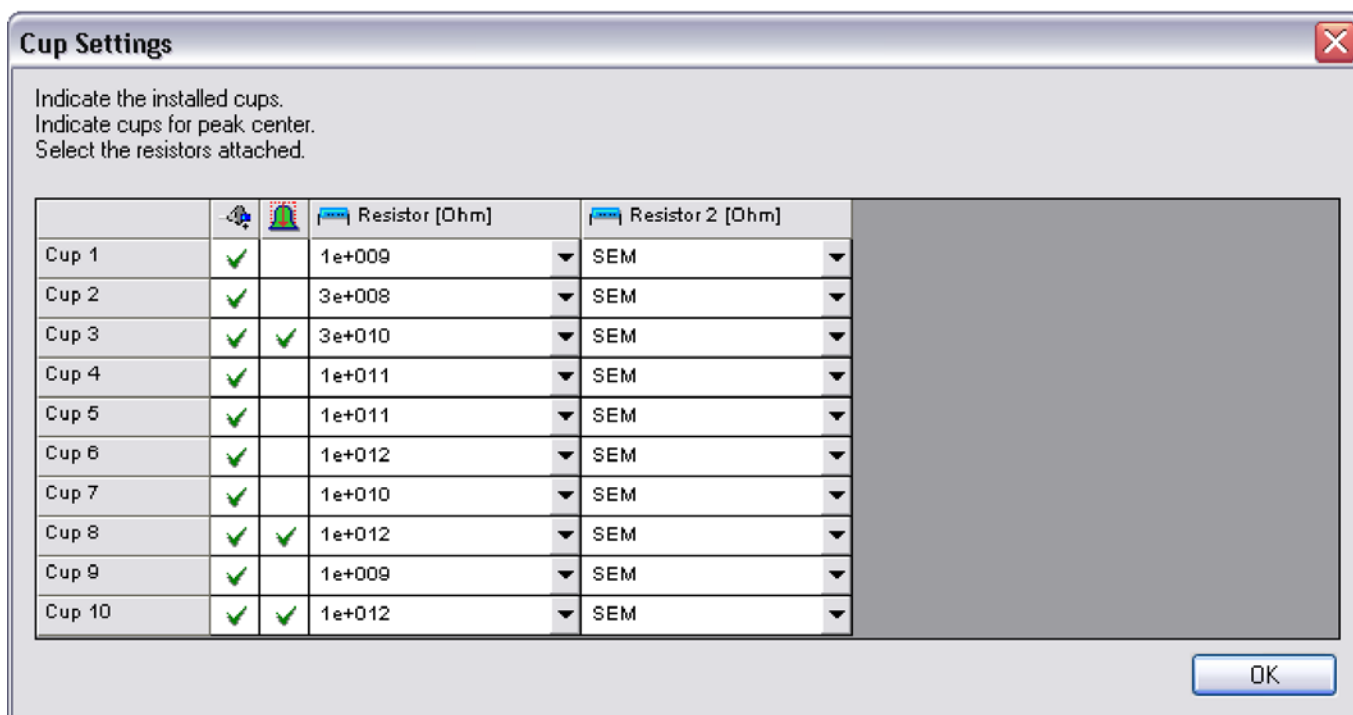
3. Select your **MAT 253**.
4. Confirm with **OK**.

**Note** The window shown [Figure 1-32](#) only appears when starting the system the very first time or after reset of the IRMS. ▲

## Getting Started

### Starting Isodat the Very First Time

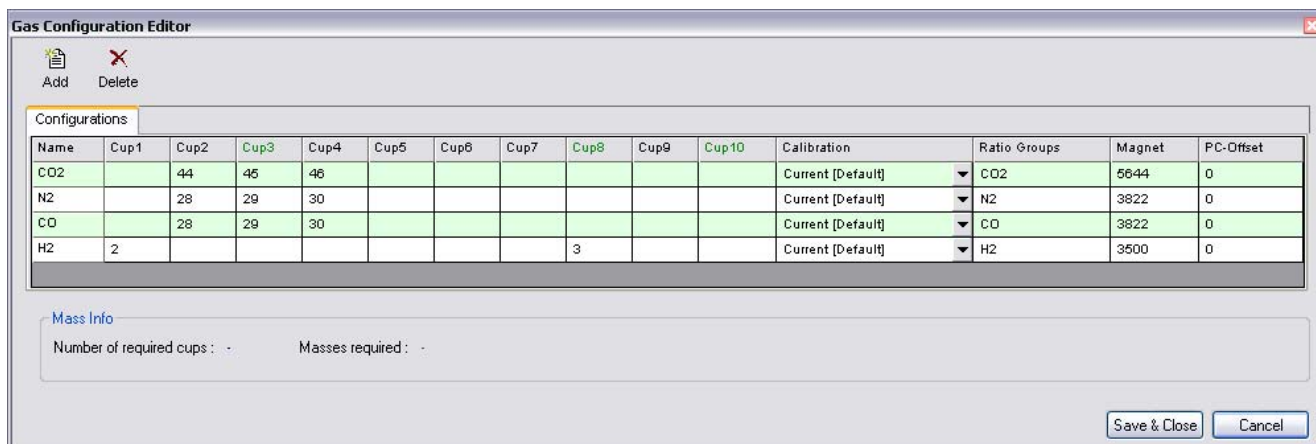
5. Read the instructions in the window **Cup Setting**, see [Figure 1-33](#) and also refer to the cup configuration in your purchase order and other instrument documentation.



**Figure 1-33.** Cup settings

6. Check whether the cups are **Installed** () or **Not Installed** () .
7. Check the **Peak Center** option of the cups () .
8. Check the values for **Resistor [Ohm]** of the cups () .
9. Check the values for **Resistor 2 [Ohm]** of the cups () .
10. Refer to the delivered test protocol and compare it with your order.

- In the **Gas Configuration Editor**, check for your particular configuration, for example, CO<sub>2</sub> (high-lighted in [Figure 1-34](#)), whether the masses are correctly assigned to the cups.



**Figure 1-34.** Gas Configuration Editor

- Check **Calibration**, **Ratio Groups**, **Magnet** position and **PC-Offset**.  
The default values for Magnet position are averaged experience values that cannot be checked and edited here, but later during calibration.
- Under **Mass Info**, the **Number of required cups** (for example, **3** in [Figure 1-34](#)) is displayed together with the corresponding masses  $m/z$ , **44, 45, 46**).
- Finally, click **Save & Close**.

## Creating a Gas Configuration

A Gas Configuration determines a combination of masses which are collected in the cups for evaluation of ratios and eventually  $\delta$  values. The Gas Configuration is specific for the particular gas and is combined with a magnet field value taken from the mass calibration of your IRMS. The ratio groups determine the reported ratios of predefined masses.

For a carbon measurement, a Gas Configuration must be available for m/z 44 (that is  $^{12}\text{C}^{16}\text{O}^{16}\text{O}$ ), m/z 45 ( $^{13}\text{C}^{16}\text{O}^{16}\text{O}$ ) and m/z 46. Otherwise, it must be created as follows.

Prior to defining this Gas Configuration ensure that the connected IRMS has the cups for the simultaneous detection of m/z 44, m/z 45 and m/z 46 and mass calibration for these cups has already been performed.

### ❖ To create a gas configuration

1. Double-click the **Isodat** Icon in the Windows Taskbar to close all open Isodat applications.

2. In the Isodat folder, double-click  to open the **Acquisition** tool.

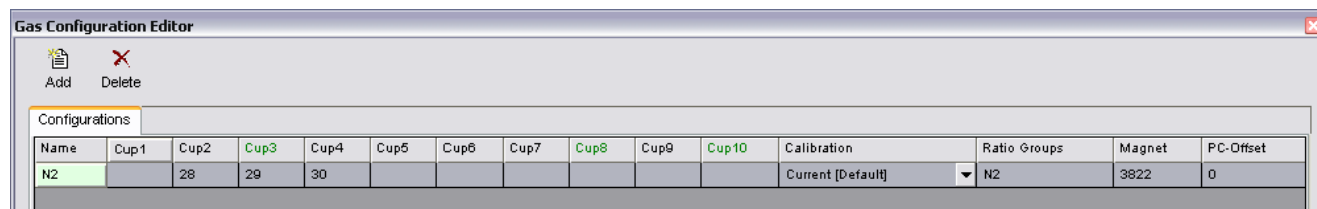
3. Select **Editors**.



4. Click the **Gas Configuration Editor**.

**Note** The Gas Configuration Editor is only available if neither acquisition nor scan are running. ▲

As default, **CO2** is created as the first Gas Configuration. However, if you have deleted CO2, another Gas Configuration, for example, **N2** is created, as shown in [Figure 1-35](#).

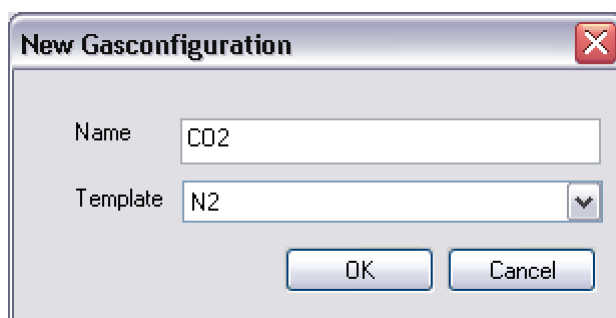


**Figure 1-35.** Creating gas configuration



5. Add a Gas Configuration.

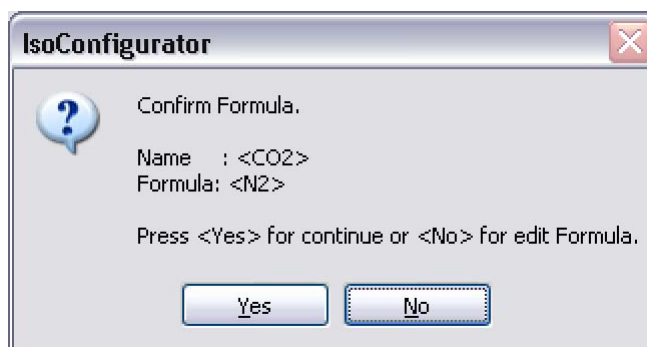
6. Type in **CO2** for **Name**, see [Figure 1-36](#).



**Figure 1-36.** Window New Gasconfiguration

7. Select a Gas Configuration as **Template**.  
In the drop-down menu, all existing Gas Configurations are displayed. When creating the first Gas Configuration, **CO2** is displayed per default.
8. Confirm with **OK**.

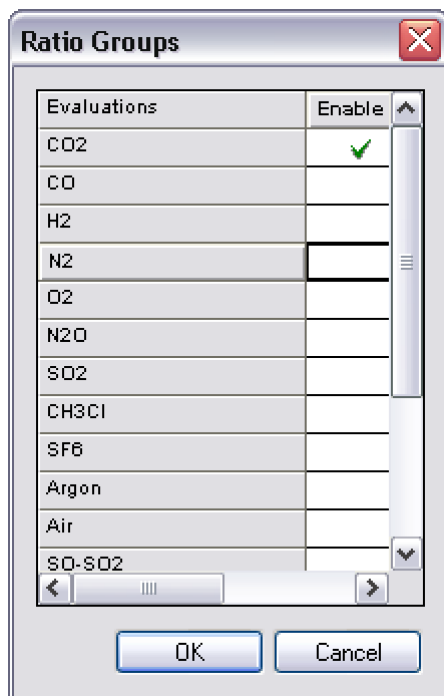
A dialog window opens, see [Figure 1-37](#).



**Figure 1-37.** Confirm Formula window

9. Click **No**.

If you type Yes, this automatically marks the template (for example, N2) instead of CO2 in the **Ratio Groups** window, see [Figure 1-38](#).



**Figure 1-38.** Ratio Groups window

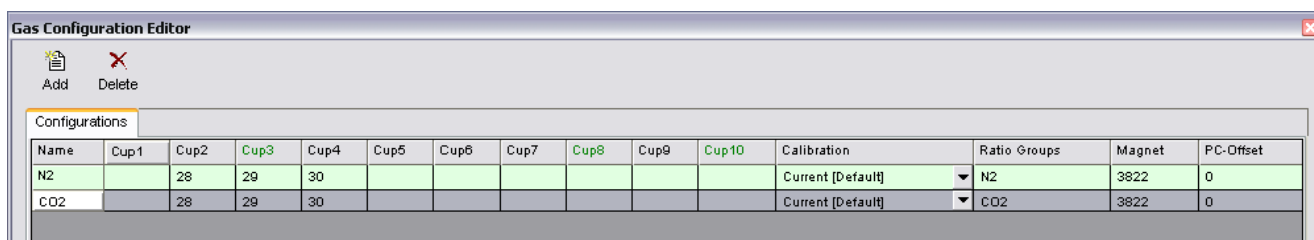
10. Click the column **Enable** for **CO2**.

A check mark  is shown.

11. If **Ratio Groups** other than CO2 are marked, unmark them all.

12. Confirm with **OK**.

The new Gas Configuration CO2 appears in the list as a row of its own, see [Figure 1-39](#).

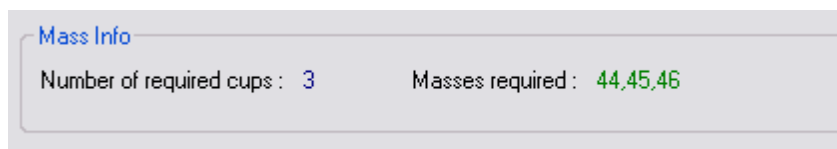


**Figure 1-39.** Creating a New Gas Configuration

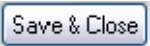
**Note** [Figure 1-39](#) shows a common cup configuration as used in most Delta mass spectrometers, that is universal triple collector. If you have a special cup configuration, the respective masses are collected in other cups. ▲

- In the **Calibration** column, select your current calibration file.  
If no calibration file has yet been created, accept the **Current (Default)** option.
- Fill in the correct masses (m/z 44, m/z 45, m/z 46) to the appropriate cups specific for your IRMS.

When highlighting the specific gas configuration by a click on its row, the number of cups required for measurement is displayed together with the assigned masses, see [Figure 1-40](#).



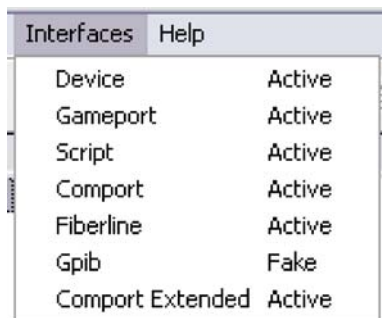
**Figure 1-40.** Mass Info – required cups and masses

- Select the Calibration which is valid for the selected cups.
- Click .


## Creating a Configuration

Before operating, a Configuration containing the MAT 253 Interface needs to be created in **Isodat Configurator**.

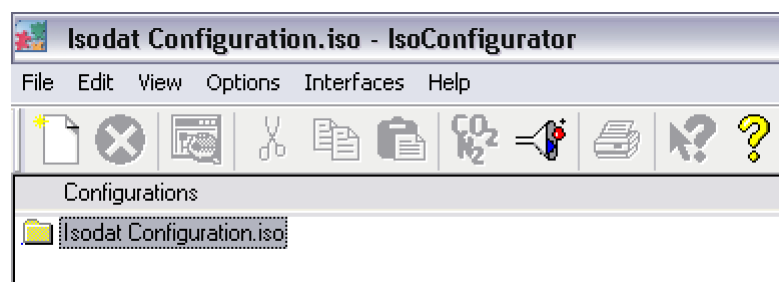
❖ **To create a configuration for the basic unit of MAT 253**



Interfaces	Help
Device	Active
Gameport	Active
Script	Active
Comport	Active
Fiberline	Active
Gpib	Fake
Comport Extended	Active

1. In the Isodat folder, double-click  to open the **Configurator** tool.
2. Click the menu **Interfaces**.
3. Click **Comport** to set the port to **Active**.
4. Click **Comport Extended** to set the port to **Active**.
5. Click **Fiberline** to set the port to **Active**.

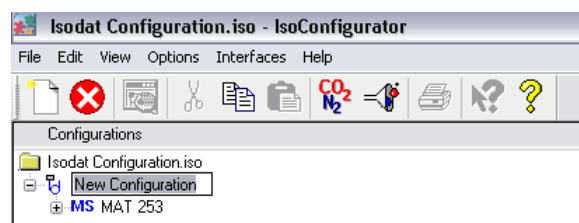
The Configurator opens showing a folder named **Isodat Configuration.iso**, see [Figure 1-41](#).



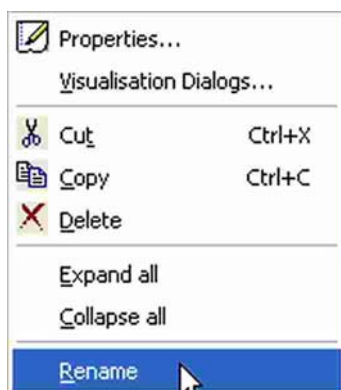
**Figure 1-41.** Configurator showing Configurations

6. Select **Edit > Add Configuration** or click  to add a new configuration.

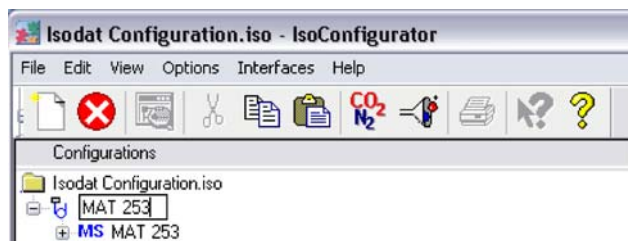
Isodat by default automatically creates a new Configuration, named **New Configuration**, see [Figure 1-42](#).




**Figure 1-42.** New Configuration



- Right-click on **New Configuration** and select **Rename** in the context menu.
- Enter a name for the configuration, for example, **MAT 253**, see [Figure 1-43](#).

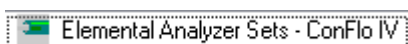


**Figure 1-43.** New Configuration for MAT 253

- Click on the  symbol of MS to expand the complete MS tree, see [Figure 1-44](#).

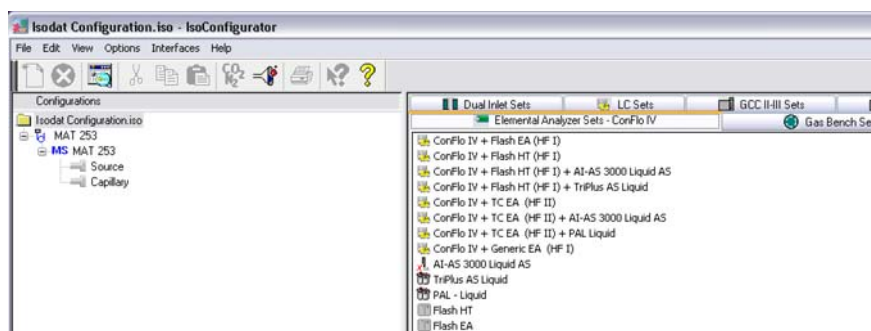


**Figure 1-44.** New Configuration for MAT 253 expanded



- Click the **Elemental Analyzer Sets - ConFlo IV** tab.

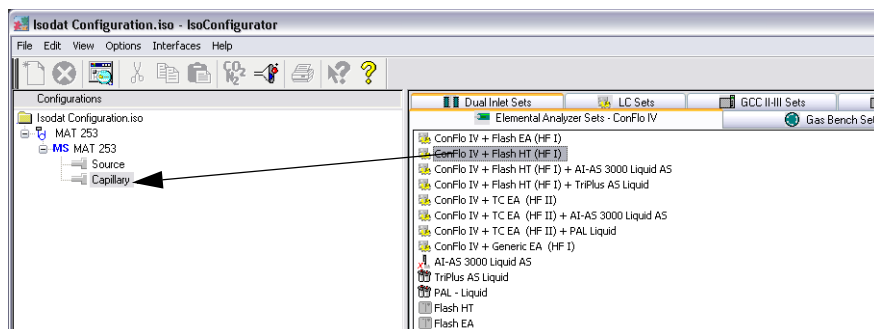
The available **Elemental Analyzer Sets** are displayed in the right pane, see [Figure 1-45](#):



**Figure 1-45.** Elemental Analyzer Sets

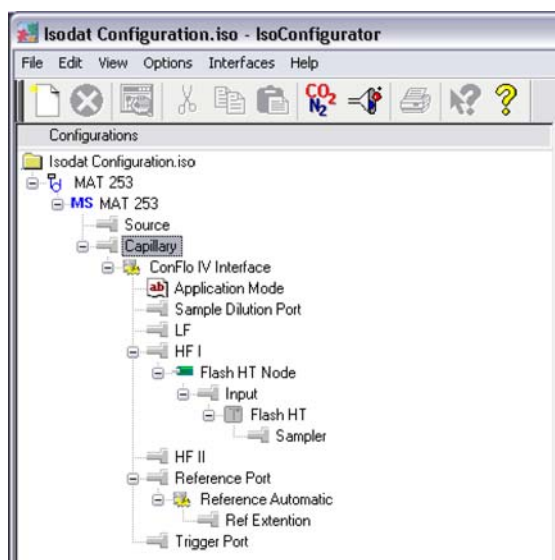
- Click your **Elemental Analyzer Set**, for example, **ConFlo IV + Flash HT (HF1)**.

12. Drag and drop it to **Capillary** port or **Source** port in the left pane, see [Figure 1-46](#).



**Figure 1-46.** Add peripheral Elemental Analyzer Sets to Capillary

[Figure 1-47](#) shows the new peripheral attached to the respective port:



**Figure 1-47.** Peripheral added to Capillary

13. Close the **Configurator** window.  
All settings are saved automatically.
14. Then create a method as described in [“Creating a New Method”](#) on [page 6-2](#).
15. Then create a sequence as shown in [“Creating a New Sequence”](#) on [page 6-3](#).

## Calibrating/Adjusting the System

To be able to work with your system, the whole system must be adjusted/calibrated first. This is done mostly with the Instrument Control tool of Isodat, see the following chapter:

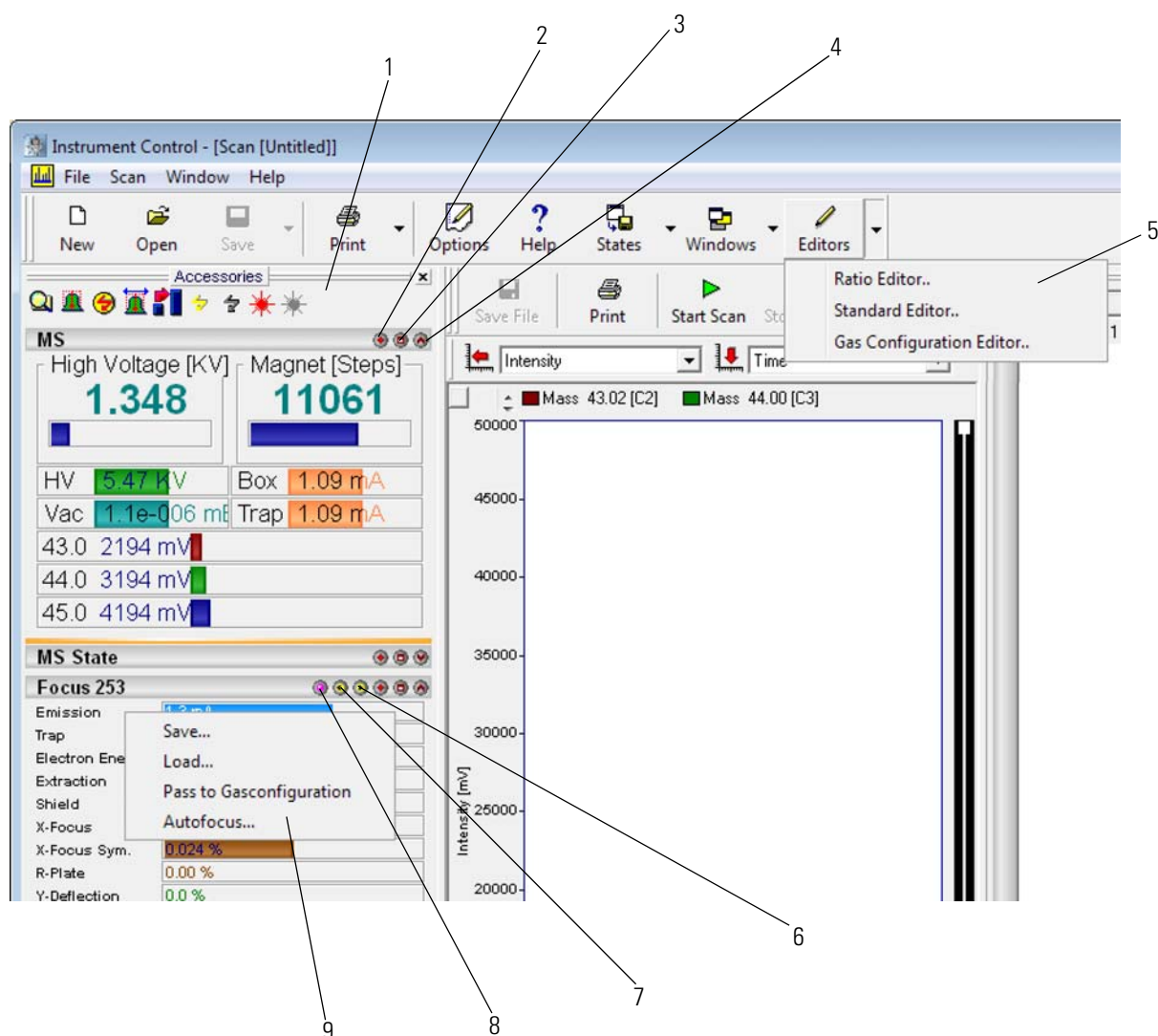
- [“Instrument Control User Interface” on page 1-36](#)
- [“Checking the Vacuum” on page 1-37](#)
- [“Introducing Gas to the Continuous Flow System” on page 1-40](#)
- [“Introducing Gas to the Dual Inlet System” on page 1-41](#)
- [“Mass Calibration” on page 1-44](#)
- [“Crimp Adjustment” on page 1-51](#)
- [“Bellows Calibration” on page 1-53](#)
- [“Focusing” on page 1-56](#)

Furthermore, Thermo Fisher Scientific provides the following functions for focussing:

- [“Auto Focusing” on page 1-59](#)
- [“Manual Focusing” on page 1-60](#)

## Instrument Control User Interface

The Accessories toolbar **1** Figure 1-48 in is located at the top of the panels:













Labeled Components: 1=Accessories toolbar, 2=Hide button, 3=Pop out button, 4=Shrink button, 5=Editors, 6=Redo button, 7=Undo button, 8=Menu button, 9=Focus 253 context menu

**Figure 1-48.** Accessories toolbar and panel buttons

**Note** For details on the Focus 253 panel refer to “Focusing” on page 1-56. ▲

The function available via the Accessories toolbar are listed in [Table 1-1](#):

**Table 1-1.** Accessories toolbar

Icon	Icon name	Comment
	File search	Opens an Isodat File Search dialog box. Enables you to search for Isodat files.
	Peak Centre	Starts the Peak Centering procedure.
	Electronic offset	Opens an Electronic Offset dialog box. Enables you to measure and/or reset electronic offset.
	Peak Centre Offset	Starts the Peak Offset Determination.
	Autofocus	Opens the Auto Focus dialog box. Enables you to select the auto focus parameters and start the auto focus procedure.
	Calibrate Bellows	Opens the Bellows Calibration Dialog Box. Enables you to start the hardware or signal bellows calibration.
	High Voltage ON	Switches high voltage ON.
	High Voltage OFF	Switches high voltage OFF.
	Source On	Switches the entire source electronics on.
	Source Off	Switches the entire source electronics off.

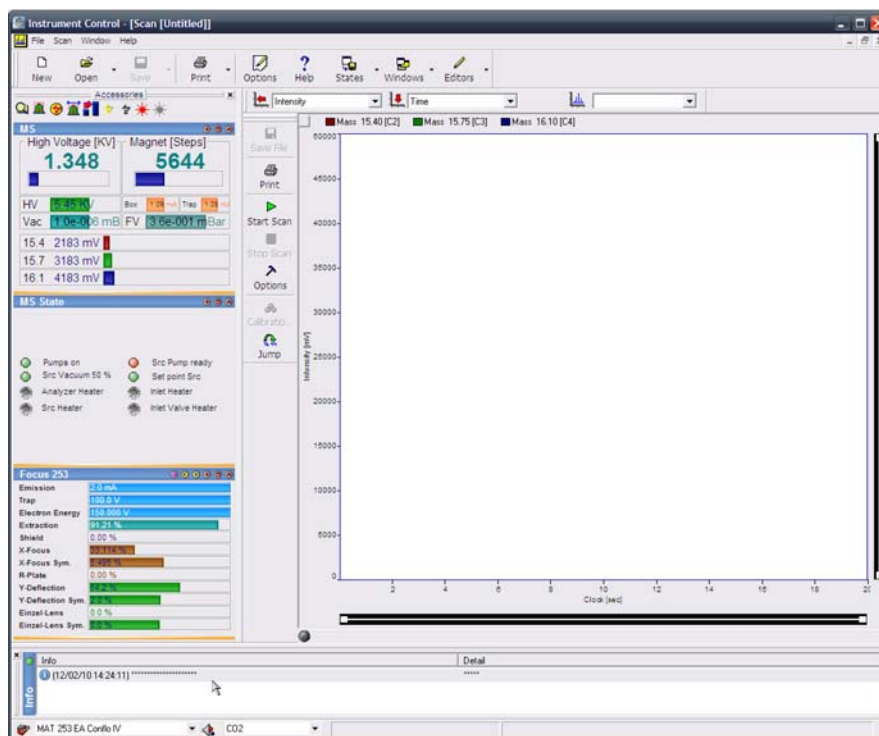
## Checking the Vacuum

Once instrument the instrument is started you need to check the vacuum quality. Checking the vacuum should be a part of daily routine.

### ❖ To check the vacuum

1. In the Isodat folder, double-click  to open the **Instrument Control** tool.

Instrument Control opens showing the Scan window, see [Figure 1-49](#).



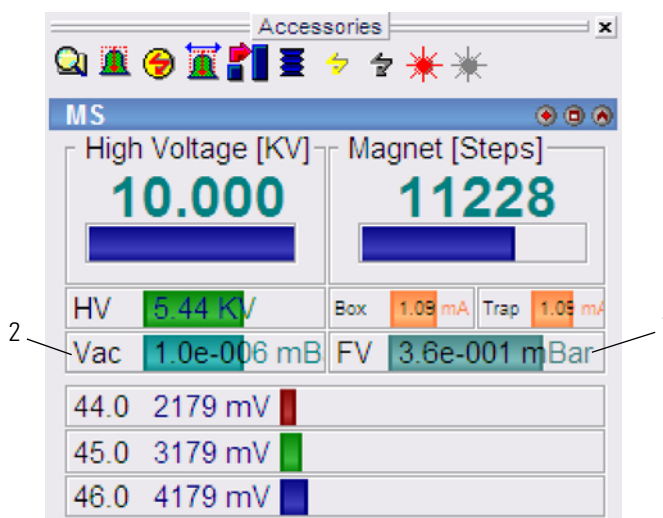
**Figure 1-49.** Scan window of Instrument Control

The Scan Windows allows for different scans, for example, signal over time, high vacuum over time, signal over magnet field, signal over high voltage.



2. In the Scan window, select the name of (hardware) configuration from the drop-down list, for example, **MAT 253 EA ConFlo IV**. Refer to [“Creating a Configuration”](#) on [page 1-32](#) for details on creating a configuration.
3. Select your gas configuration from the drop-down list, for example, **CO2**.

4. Check the quality of the vacuum, see [Figure 1-50](#) for typical values.

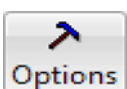


Labeled Components: 1=fore vacuum display, 2=high vacuum display

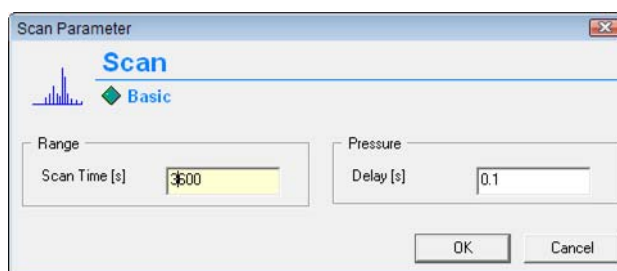
**Figure 1-50.** HV Source display in the MS panel

The MS tab in the Accessories panel displays the high vacuum in the analyzer (Vac, see **2** in [Figure 1-50](#)) and the fore vacuum (FV, see **1** in [Figure 1-50](#)) values in mbar.

5. To monitor the vacuum with the scan feature, in the scan window select **Time** for the X-axis and **High Vacuum** for the Y-axis.

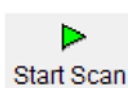
6. Click  **Options**.

The Scan Parameter window opens, see [Figure 1-51](#).



**Figure 1-51.** Scan Parameter window

7. Enter the desired **Scan Time** in seconds (usually 1 hour is sufficient) and leave the **Pressure Delay** on the default value 0.1 s.

8. Click the  **Start Scan** to start the scan.

9. Observe the High Vacuum signal for intermittent spikes and also for absolute value.


Typical pressure value after 24 hours pumping without any gas supplied (needle valve closed or dual inlet with no gas) and no heating of analyzer is  $2 \times 10^{-7}$  mbar or lower.

## Introducing Gas to the Continuous Flow System

Following text describes the steps needed to introduce the helium flow through the needle valve in the mass spectrometer at the time of installation. You can also use the information below if you are starting the instrument after a longer period of no use.

We assume the needle valve is closed and the capillaries are connected to the needle valve. Typically reference and sample capillaries from ConFlo IV are used. It is also recommended the system is under the vacuum for more than 24 hours with the ion source heaters and the inlet valve heaters on.


### ❖ To introduce gas to the Continuous Flow system

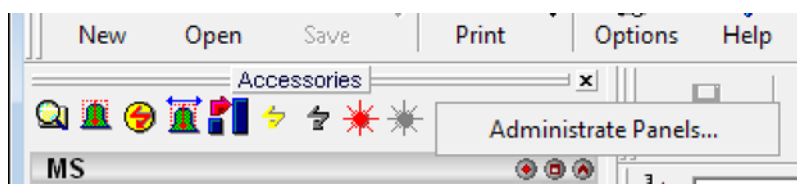
1. In the Isodat folder, double-click  to open the **Instrument Control** tool.
2. Check the vacuum level.  
With the needle valve closed it should be in the range 1 to  $5 \times 10^{-7}$  mbar.
3. Make sure the filament and high voltage are OFF.
4. Make sure you have a flow of helium through all the capillaries installed.  
Refer to the correspondent operating or reference manual of the connected peripheral.
5. Open the needle valve counter-clockwise 1/2 to 3/4 turn.
6. Wait a few minutes. Observe the high vacuum level.  
You can do the time scan - high vacuum vs. time. As soon as the vacuum is stable and the analyzer pressure is below  $3.5 \times 10^{-5}$  mbar you can turn on the high voltage.  
If this range is not reached ( $<3.5 \times 10^{-5}$  mbar), most likely there is a leak on the capillaries connected to the needle valve. Because of risk of filament damage in such case you cannot turn on the high voltage (blocked by Isodat). Close the needle valve and check the capillaries from the peripheral or ConFlo IV to the needle valve and also check the ferrule in the needle valve.

## Introducing Gas to the Dual Inlet System

All the gas paths in the Dual Inlet system must be successively evacuated prior to use.

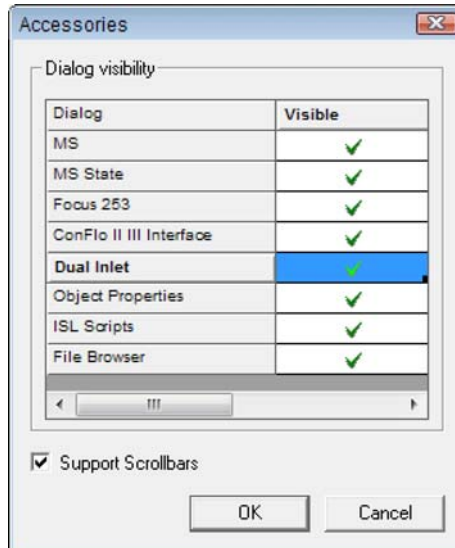
❖ **To introduce gas to the Dual Inlet system**

1. In the Isodat folder, double-click  to open the **Instrument Control** tool.
2. If the **Dual Inlet** panel is not shown, right-click next to the **Accessories** icons to open the context menu, see [Figure 1-52](#).




**Figure 1-52.** Context menu Administrate Panels

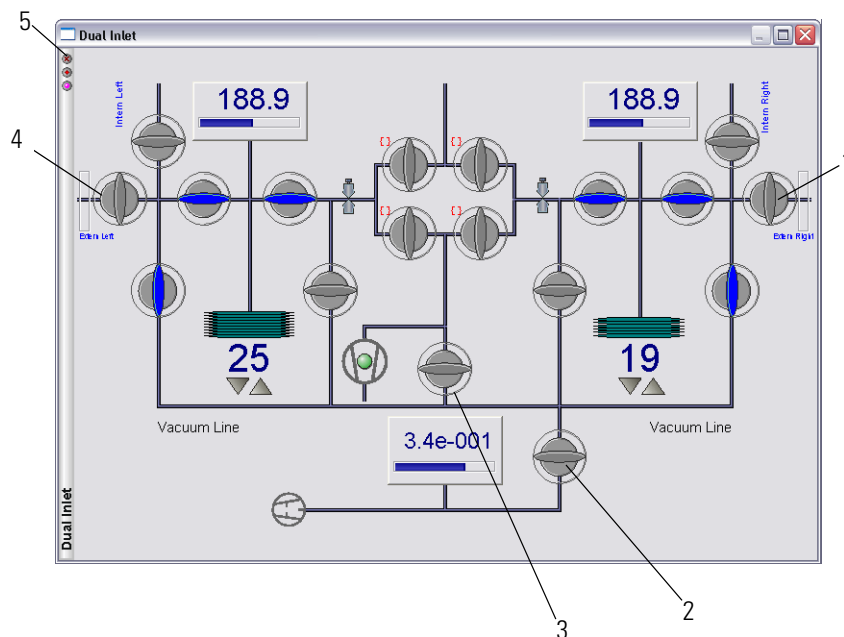
3. Select **Administrate Panels**.  
The Accessories dialog opens, see [Figure 1-53](#).



**Figure 1-53.** Administrate Panel - Accessories dialog

4. In the **Accessories** dialog, click in the column **Visible** in the row **Dual Inlet** to show the panel.
5. Click **OK**.
6. On the **Dual Inlet** panel, click the Pop out button  to open the panel in a separate window, see [Figure 1-54](#).

The size of this window can now be adjusted and the content dynamically changes with the size.





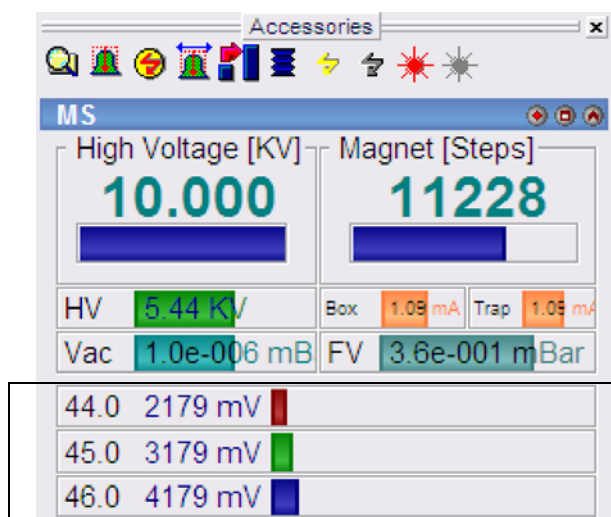
Labeled Components: 1=valve to external gas supplies, 2=rough pump valve, 3=turbo pump valve, 4=valve to external gas supplies, 5=Login button

**Figure 1-54.** Dual Inlet system panel

7. Evacuate the Dual Inlet system and connected tubing from the reference gas supply to the dual inlet with rough vacuum pump. The valve on the external gas supply (see **1** and **4** in [Figure 1-54](#)) needs to remain closed. Commonly used reference gas supply is a 1-liter bottle with manual valve regulator.
8. After evacuation, close the rough pump valve (see **2** in [Figure 1-54](#)).
9. Introduce the gas to the tubing. To do that, successively fill the tubing, feed lines and the bellows with the gas from the gas reservoir. 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<sub>2</sub> is displayed in the MS panel (see [Figure 1-55](#)). The intensity for m/z 44 is smaller than the one on m/z 45, which in turn is smaller than the one on m/z 46.

**Note** Before starting a measurement, after pumping for 15 min, an analyzer 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 while pumping continues for a longer time (if all valves are closed). If after 15 min of pumping a pressure of  $\times 10^{-5}$  mbar cannot be reached, a leak might be the cause. As a safety measure, the cathode turns off at pressures above  $3 \times 10^{-5}$  mbar. ▲

10. Click  below **Accessories** in the Smart Isotope MS bar of Instrument Control (see Figure 1-55) to turn on the ion source.
11. If you got a signal on all cups, click  to perform a peak center. The signal intensities should yield the pattern type shown in Figure 1-55.



**Figure 1-55.** Intensity pattern for CO<sub>2</sub>

12. If you do not see this pattern or if an error occurs during peak center, perform a calibration using CO<sub>2</sub>.  
If you calibrated the IRMS before, a peak center may be sufficient.

**Peak Center Principle:** Only the HVDAC value (high voltage) is changed in this subtle HVDAC scan. The magnet jumps to a certain BDAC value specified, for example, 10145 BDAC steps. As a result, the magnet exactly finds the peak of interest. The BDAC values of a particular gas, for example 12071 BDAC steps in case of CO<sub>2</sub>, have first and foremost been stored in the gas configuration, for example, CO<sub>2</sub>. The peak center runs according to the BDAC values specified there. For each mass, at least one peak center will be performed. Default value is three peak centers for each mass.

## Mass Calibration

The mass calibration of MAT 253 is performed with Instrument Control. It is assumed that the mass spectrometer is coupled either with a Dual Inlet system or a ConFlo IV device to supply the required reference gas.

When using a ConFlo IV interface, make sure the helium inlet pressure is set to 1 bar and the CO<sub>2</sub> reference gas is set to 0.5 bar on the respective gauge. The sample dilution should be set to maximum to avoid any effects from the sample side of the ConFlo IV. See also “[Introducing Gas to the Continuous Flow System](#)” on page 1-40.


When using a Dual Inlet system as the gas source for calibration, it is required to fill one of the bellows with reference gas. See also “[Introducing Gas to the Dual Inlet System](#)” on page 1-41.

### Mass Calibration Process

During the mass calibration process, a mass scan over the selected mass range is performed. The result is an approximate pattern of the gases present in the mass spectrometer. MAT 253 offers a proposal of the position of the gases. These positions need to be checked, adjusted and accepted before the mass calibration can be finished.

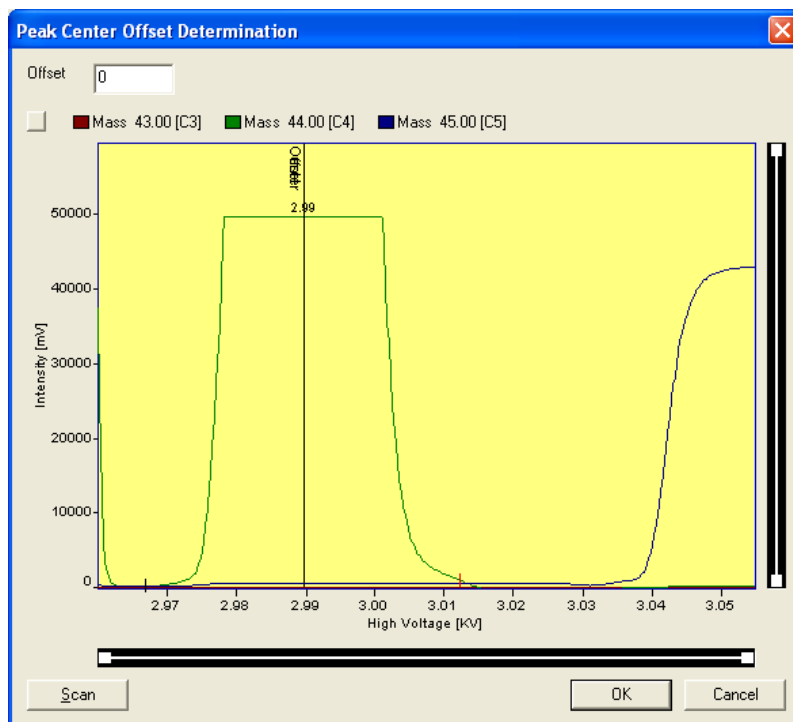
Sometimes, the proposals for the mass peak positions are matching and sometimes they are not. During the very first calibration, the proposals usually must be corrected. The calibration masses are assigned to their peaks by drag and drop, snapping the lines to the mass peaks.

Once the calibration is completed, it is important to perform the Peak Centre Offset and pass on this Offset value to the Gasconfiguration PC offset value.

**Peak Centre Offset**  performs the voltage scan. In a separate window, the Peak Centre and the Offset value are displayed graphically. It is possible to change the display with the mouse.


The Offset value changes the PC offset value in the Gasconfiguration window.

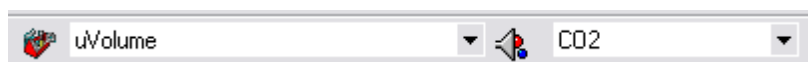
In the [Figure 1-56](#) the **Offset** is 0 and the peak centre and the cursor position are matching.



**Figure 1-56.** Peak center offset determination

❖ **To perform a mass calibration**

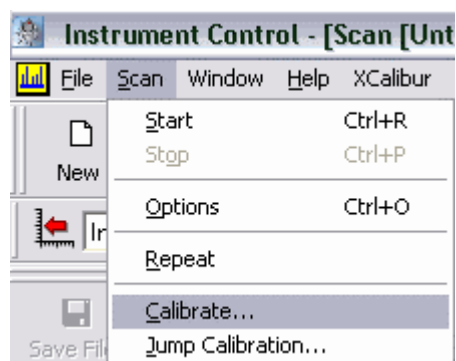
1. In the Isodat folder, double-click  to open the **Instrument Control** tool.
2. Select hardware configuration and gas configuration as required for the calibration, see [Figure 1-57](#).



**Figure 1-57.** Hardware and gas configuration

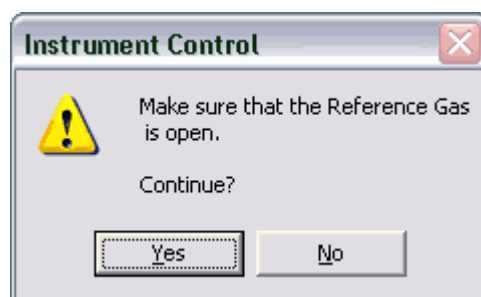
Calibration is usually performed using CO<sub>2</sub> as reference gas, but it is also possible to calibrate on other gases. If you want to use another gas than CO<sub>2</sub>, make sure you know all the mass peaks that this gas will produce in the source.

3. Select **Scan > Calibrate**, see [Figure 1-58](#).



**Figure 1-58.** Scan menu Calibrate

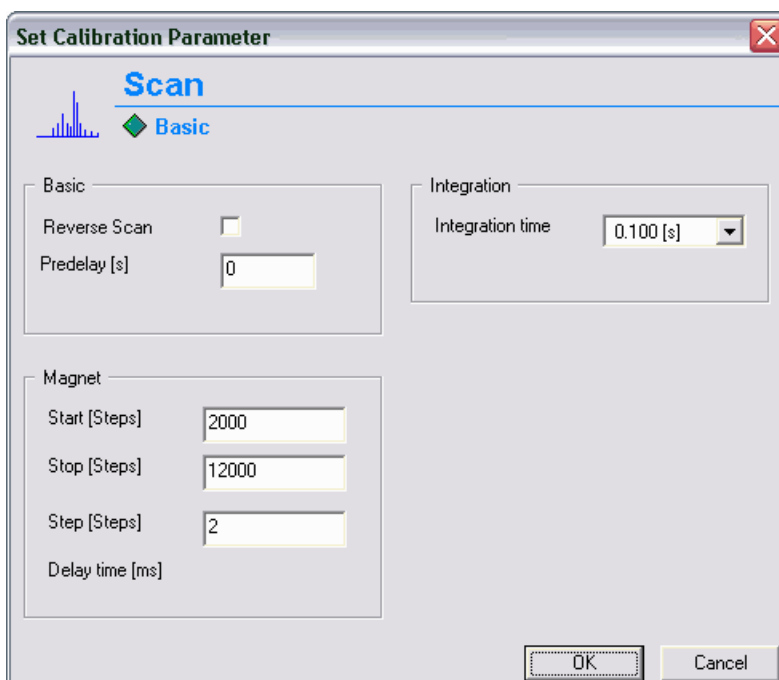
A message window opens, see [Figure 1-59](#).



**Figure 1-59.** Message window to ensure that reference gas is opened

4. Make sure the reference gas is open and click **Yes**.

5. In the **Set Calibration Parameter** window in the field **Magnet**, enter **218** for **Start [Steps]** and **12000** for **Stop [Steps]** as a range for the magnet scan, see [Figure 1-60](#).

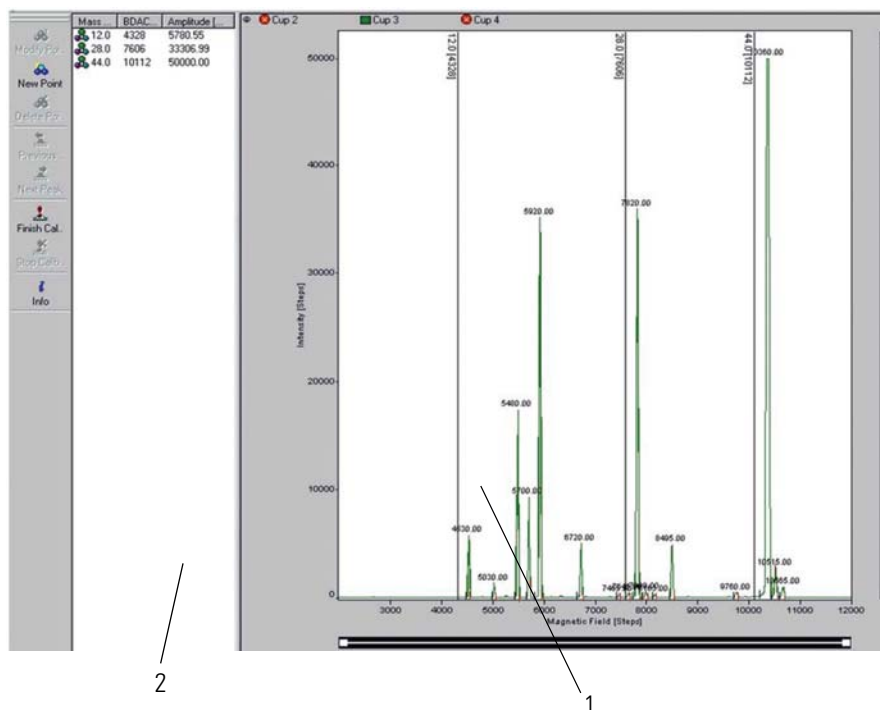


**Figure 1-60.** Set Calibration Parameter window

6. Enter **2** for **Step [Steps]** to catch the correct peak height for the various peaks, see [Figure 1-60](#).
7. Confirm with **OK**.  
A mass scan is started.
8. When the mass scan is finished, click **Calibration**.



The calibration window opens, see [Figure 1-61](#):



Labeled Components: 1=plot area, 2= calibration area

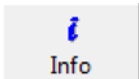
**Figure 1-61.** Proposal of peak positions

The mass proposals in the calibration area **2** in [Figure 1-61](#) shows CO<sub>2</sub> masses at 44, 28, and 12 for the selected reference gas as well as some water masses 16, 17, 18. The masses 12, 28 and 44 need to be identified in order to proceed. A proposal about their position is then offered. On demand, additional masses can be inserted into the proposal list. If the positions do not match satisfactorily, they are slightly moved later.

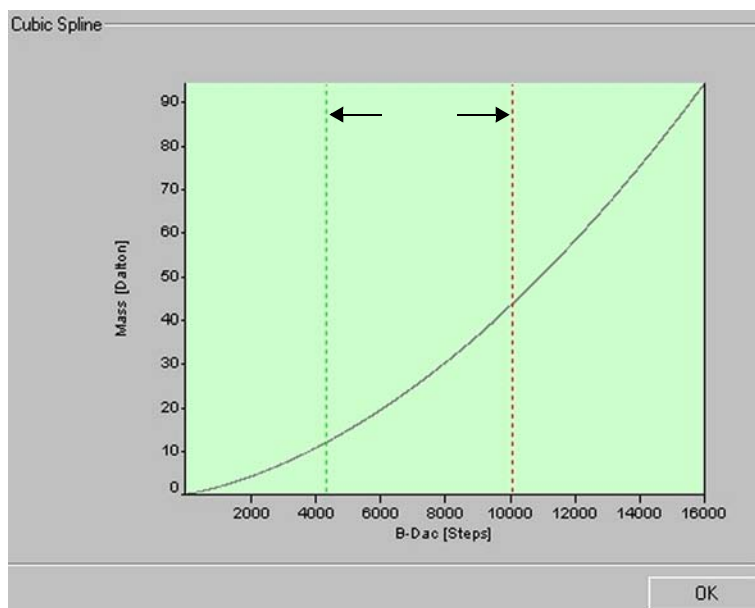
9. Click in the calibration area (**2** in [Figure 1-61](#)) to activate it.
10. Double-click on **Cup 3** above the plot area (**1** in [Figure 1-61](#)) to select the centre cup.  
The display of the other cups changes as shown in [Figure 1-62](#).



**Figure 1-62.** Cup 3 selected as center cup


11. Click  to check your selection of masses for consistency.

The curve in Figure 1-63 shows the quadratic dependence of the magnetic field (proportional to mass units) generated by the current, the latter given in steps of the DAC that controls the current in the magnet:



**Figure 1-63.** Controlling mass proposals

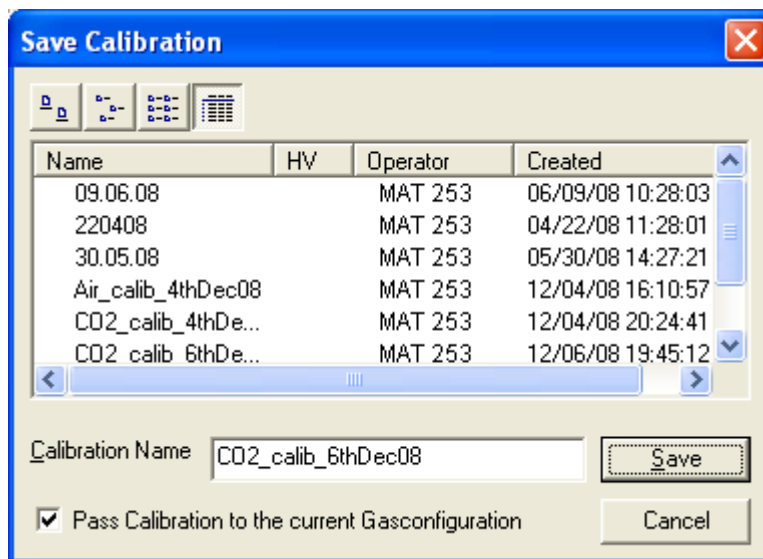
12. Identify the masses 44, 28 and 12 in the plot area of the scan and adjust the proposed values by dragging and dropping the vertical lines to the correct value.

13. When you finished the corrections, click  .  
The calibration takes approximately 15 min.


Isodat now attempts to scan the peaks in detail to determine the exact positions on the magnet current scale. It is important to check in the calibration window and peak list that the peak height shown there matches the mass number selected.

**Note** After a successful calibration, the calibration window presents a new picture where the mass position markers are no longer coincident with the mass peaks. This is not an error but it reflects the magnet hysteresis, a physical effect that is inherent to the magnet and can not be avoided. ▲

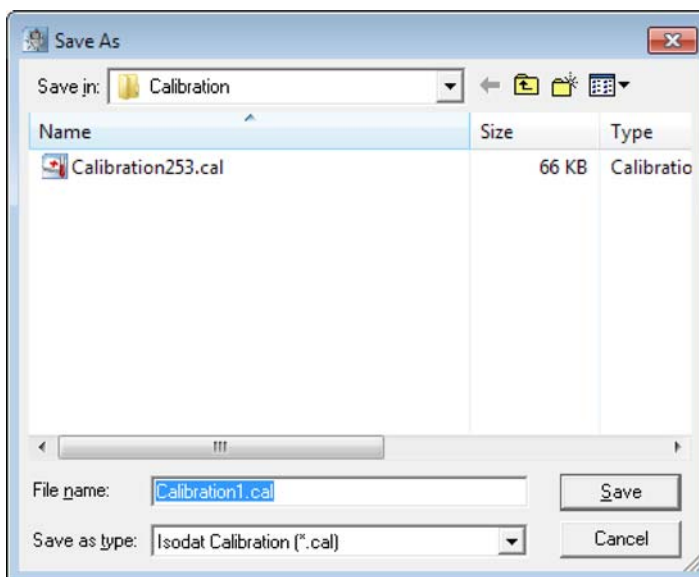
When the calibration process is finished, the **Save Calibration** window opens automatically, see [Figure 1-64](#):



**Figure 1-64.** Save Calibration window

14. Select the check box **Pass Calibration to the current Gasconfiguration**.
15. Enter a **Calibration Name**.
16. Click **Save**.  
The calibration is saved in the Isodat Calibration file (not as a single file) and passed to the gas configuration. This particular gas configuration is loaded with this particular calibration information.
17. Click  in the system bar to save the calibration.

The **Save As** window opens, see [Figure 1-65](#).

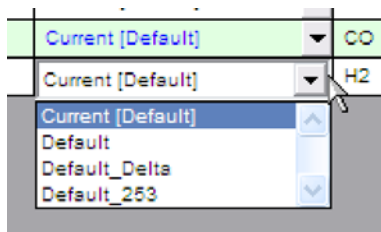


**Figure 1-65.** Save As Calibration window

18. Enter the **File name** for the calibration.

19. Click **Save**.

The calibration is saved with the name entered. This file is not used for the mass identification. It can be used for mass recalibration if necessary.



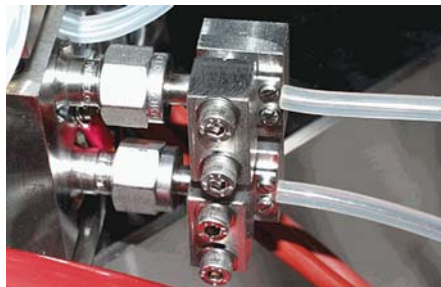
**Note** Check the Gas Configuration Editor (refer to [“Creating a Gas Configuration”](#) on [page 1-28](#)) for selected calibration.

It is possible to use the same calibration multiple times - for more than one gas configuration. It is also possible to have several calibrations for specific gas configuration. ▲

## Crimp Adjustment


Crimp adjustment ensures an approximately constant and slight gas flow into the source and in most cases ensures the same flow from both bellows. Adjusting this flow is essential for high performance operation of the Dual Inlet system and has to be done with utmost care. Crimp adjustment is typically performed on CO<sub>2</sub> and this procedure is described below.

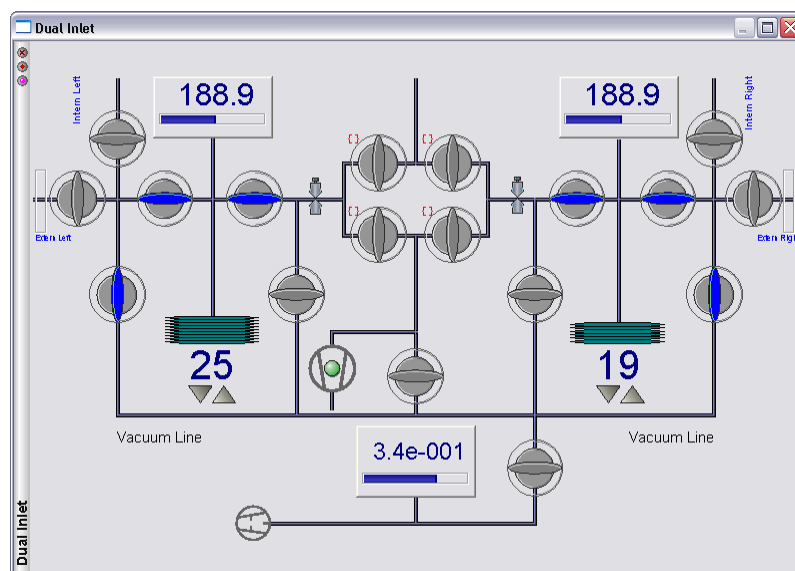
At the time of installation the crimping is performed by the Thermo Fisher Scientific field service engineer and there is no reason to change it. However, after some time of operation it might be required to bake the instrument and subsequently to control the crimping of the inlet capillaries, see [Figure 1-66](#).



**Figure 1-66.** Crimp adjustment

❖ **To perform a crimp adjustment**

1. In the Isodat folder, double-click  to open the **Instrument Control** tool.
2. Open the valves that connect the two bellows in the inlet system ([Figure 1-67](#)) and pump out the dual inlet using the fore vacuum and high vacuum pumps.



**Figure 1-67.** Visualization of Dual Inlet

3. Close the pumps.
4. Admit approximately 10 mbar of CO<sub>2</sub> into each bellows.


5. Wait for at least 180 seconds to allow for equilibration of the gas.
6. Carefully adjust the crimps. The 10 mbar for CO<sub>2</sub> must yield 2 V on mass 44.
7. In order to check the current signal level, switch the Change Over Valve repeatedly between left and right bellows. Allow for the signal to settle a few seconds before taking the reading. The difference between the two inlet capillaries should be smaller than 50 mV.

## Bellows Calibration

Prior to a Dual Inlet acquisition, a bellows calibration must be performed. It is presumed, that the source has been switched on and focusing has been performed.

### ❖ To start bellows calibration



1. In the Isodat folder, double-click  to open the **Instrument Control** tool.

The Scan window opens (see [Figure 1-49](#)).



2. Select a Dual Inlet configuration from the list of available configurations that appears in the bottom left corner of the Instrument Control window.



3. Click **Calibrate Bellows** in the Accessories toolbar.
4. Bellows calibration must be performed in two steps, continue with “[Step 1 - Hardware Calibration of Bellows](#)” on [page 1-53](#) and “[Step 2 - Signal Calibration of Bellows](#)” on [page 1-55](#).

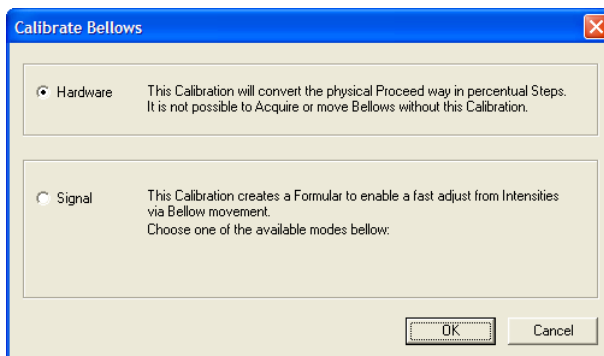
### Step 1 - Hardware Calibration of Bellows

In hardware calibration, the bellows are completely opened (that is 100%) and closed again (that is 0% and these positions are stored). The hardware calibration deletes any previous signal calibration. After hardware calibration, the signal calibration “[Step 2 - Signal Calibration of Bellows](#)” on [page 1-55](#) must be performed.

**Caution** Hardware calibration must always be performed prior to signal calibration! ▲

❖ **To perform hardware calibration of bellows**

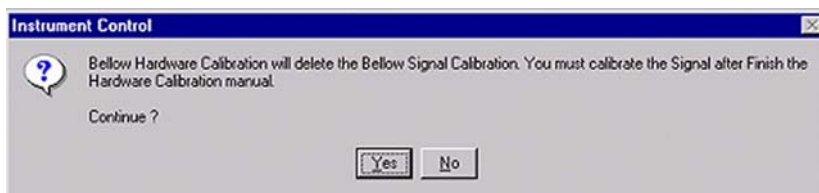
1. Select the **Hardware** radio button. See [Figure 1-68](#).



**Figure 1-68.** Hardware radio button

2. Click **OK**.

The hardware calibration deletes the signal calibration. A message window opens. See [Figure 1-69](#).



**Figure 1-69.** Hardware calibration deletes signal calibration

3. Click **Yes** to continue.

The hardware calibration takes up to 3 minutes.

When the hardware calibration of the bellows is completed, a message window opens, see [Figure 1-70](#).



**Figure 1-70.** Bellows calibration complete

4. Click **OK**.

## Step 2 - Signal Calibration of Bellows

**Note** Signal calibration requires a signal intensity of at least 3 V with the bellows opened at 100%. If signal calibration has not been performed, the Diagnosis tests cannot be run. ▲

In signal calibration, the signal intensity is checked with partially compressed bellows (at 25% and at 75%). Therefore, signal calibration must always be performed after hardware calibration.

### ❖ To perform a signal calibration

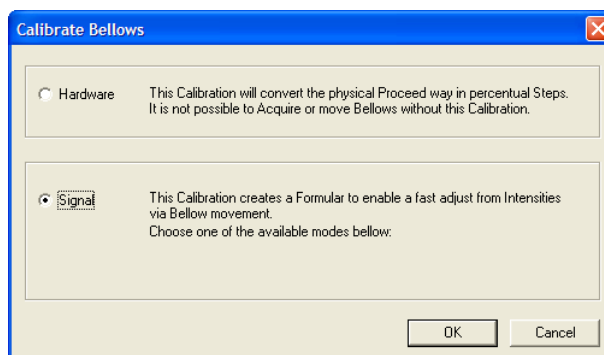


1. In the Isodat folder, double-click **Instrument Control** to open the **Instrument Control** tool.  
The Scan window opens (see [Figure 1-49](#)).
2. Select a Dual Inlet configuration from the list of available configurations at the bottom left corner of the **Instrument Control** window.
3. Choose **Calibrate Bellows** in the **Accessories** toolbar.



Before starting signal calibration, an amount of gas must be let into the bellows, which results in a pressure of about 10 to 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 (for example, less than 10 mbar) or too large, warning messages will occur.

4. Select the **Signal** radio button. See [Figure 1-71](#).



**Figure 1-71.** Signal radio button

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

Without bellows calibration, Signal Pressure Adjustment and Diagnosis tests will not be possible. After the bellows have been calibrated, you can run the Diagnosis tests. For details, refer to [“Diagnosis”](#) on [page 7-1](#).

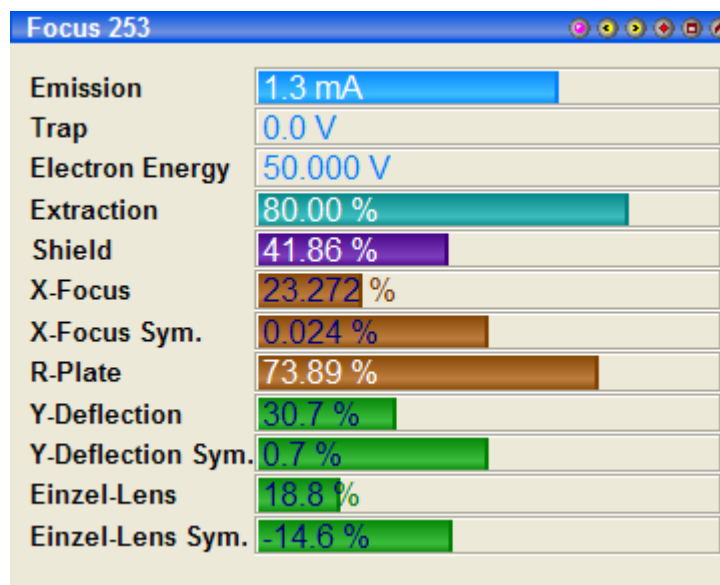
After performing Diagnosis tests, you can start a measurement (that is a Dual Inlet acquisition). For details, refer to “Measurements” on page 6-1.

## Focusing

Focusing has to be performed:

- to overcome the tiny remaining mechanical tolerances
- to overcome the gradual degradation of the ion source (for example due to metal deposition by sputtering)
- to overcome the different ion-optical properties of various gases (for example, for H<sub>2</sub> a slightly different focusing compared to CO<sub>2</sub> yields better results).
- to optimize the system for various applications (for example, reduce or increase sensitivity, optimize linearity)

The obtained parameters which can be seen in the **Focus** window [Figure 1-72](#) are assigned to the respective gas configuration.



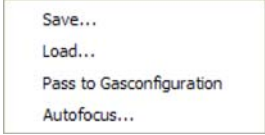
**Figure 1-72.** Parameters on Focus window

**Note** Specific focus settings are recommended for each gas configuration. ▲

## Context Menu

The function available via the context menu are listed in [Table 1-2](#):

**Table 1-2.** Context menu of the Focus 253 panel

Icon	Icon name	Comment
	Load	Opens a Focus Settings document (*.fcs). Used to load saved parameters.
	Save	Saves the actual focus settings as Focus Settings document (*.fcs). Used to save changed parameters.
	Pass to Gasconfiguration	Assigns the current focus settings to the active gas configuration. This command replaces previously assigned focus settings.
	Autofocus	Opens the Auto Focus dialog box. Enables you to select the autofocus parameters and start the autofocus procedure.

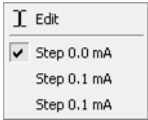
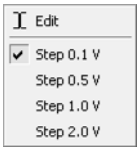
### ❖ To open the context menu

1. Right-click in the **Focus 253** window or click the **Menu** button (8 [Figure 1-48](#)) to open the context menu (9 [Figure 1-48](#)).
2. See [Table 1-2](#) for details on the context menu.

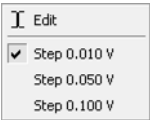
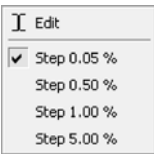

## Editing Focus Parameter Values

Each Focus parameter value of the Focus 253 panel can be adjusted manually either continuously by clicking and dragging up or down or with single clicks. A Single click changes the value according to the step selected for this field with a right-click, see context menus in [Table 1-3](#).

**Table 1-3.** Focus 253 context menus on value side

Context menu	Focus parameter
	Emission
	Trap

**Table 1-3.** Focus 253 context menus on value side, continued

Context menu	Focus parameter
	Electron Energy
	Extraction Shield R-Plate
	X-Focus X-Focus Sym. Y-Deflection Y-Deflection Sym. Einzel-Lens Einzel-Lens Sym.


If you move the mouse over the values on the right side of the panel the mouse changes its appearance according to the functionality available (up and down arrows or a plus sign). If you selected Edit in the context menu, the respective value can be typed in directly.

**Note** Refer to “Auto Focusing” on page 1-59 and “Manual Focusing” on page 1-60 for details on performing focusing. ▲

❖ **To edit Focus parameter values**

**Caution** Only experienced users should perform manual focusing. ▲



1. In the Isodat folder, double-click  to open **Instrument Control**.
2. In the focus 253 panel, move the mouse over the value to be changed.  
The mouse changes its appearance to arrows or a plus sign at the end of the row.
3. When the plus sign shows, click once to change the value according to the step selected in the context menu, see [Table 1-3](#).
4. If you want to change the value continuously, click and drag the mouse when the mouse shows the arrows.

5. Right-click the value and select **Edit** from the context menu if you wish to enter the new value directly.  
As a parameter value increases, the colored part of its field grows.

## Sensitivity Focusing

To achieve maximum sensitivity, the extraction voltage (at maximum intensity) must be set to zero. Because the acceleration out of the source does not exist, more ions stay in the source. The number of molecules per ion is lower. Many ions exist having different energies (secondary ionization processes), providing a wide energy distribution.

## 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.

Linearity focusing must be performed here. While the extraction voltage is increased, all other lenses are tuned until maximum intensity is reached.


**Note** If linearity focusing is unsuccessful, change the Extraction value at the Focus bar until the intensity pattern of CO<sub>2</sub> is satisfactory. ▲

The focus settings preset by the factory have been established on the basis of a reasonable pattern. Each gas (for example CO<sub>2</sub>) has its own pattern. Hence, gases differ with respect to these patterns. The pattern of a particular gas is always the same.

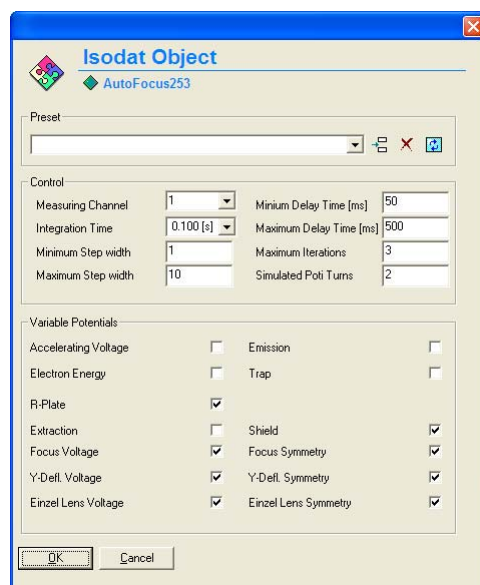
**Note** If Autofocus has not lead to the desired maximum intensity, perform focusing manually. ▲

## Auto Focusing

### ❖ To perform Auto Focusing

1. In the Isodat folder, double-click  to open **Instrument Control**.  
The Scan window opens (see [Figure 1-49](#)).

- Click  to start **Auto Focus**.  
The **AutoFocus253** window opens, see [Figure 1-73](#).



**Figure 1-73.** Autofocus details

- Click **OK**.

The default settings can usually be accepted. The potentials (see [Figure 1-72](#)) are adjusted accordingly.

By deselecting one of the **Variable Potentials** you decrease the possibilities of changing the signal. This is very useful for fine tuning. If you know, for example, your desired focus voltage, you set it manually and then in the **Autofocus** window deselect the **Focus Voltage** option and perform the autofocus.

- Save** the focus settings or **Pass to Gas Configuration** (see [Table 1-2](#)).

## Manual Focusing

The instrument has been focused in Thermo Fisher Scientific factory during the testing procedure you only need the fine tuning according to your needs.

**Caution** Only experienced users should perform manual focusing. ▲

**Caution** Be careful! In case of doubt, preferably perform Autofocusing! Manual Focusing and Autofocusing can also be performed in turn (for example beginning with a coarse manual focusing that is followed by a fine Autofocusing). ▲

All or only selected potentials might be varied. It may be advantageous to set some potentials to default values, for example:


- Trap to maximum
- Electron energy to maximum
- Emission to maximum or lower
- Extraction to about 80% for linearity focusing

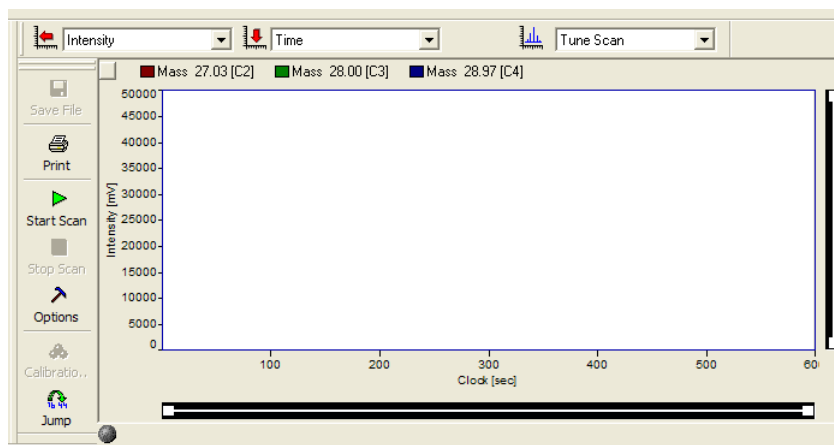
❖ **To perform manual focusing**



1. In the Isodat folder, double-click **Instrument Control**.

The Scan window opens (see [Figure 1-49](#)).

2. In the Scan window, select **Tune Scan** for , see [Figure 1-74](#).  
A Tune scan reveals the course of intensity over time while changing the parameters in the Focus window. It continues during the whole focusing process.



**Figure 1-74.** Selecting Tune Scan

3. If you wish select a particular cup (for example, mass 44, mass 45 or mass 46).  
The intensity of this mass/cup will be monitored over time.
4. Change the values as described in “[Editing Focus Parameter Values](#)” on [page 1-57](#).
5. **Save** the focus settings or **Pass to Gas Configuration**. See [Table 1-2](#).

## Getting Started

### Calibrating/Adjusting the System

**Note** For the Tune scan recommended here, it does not 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. ▲

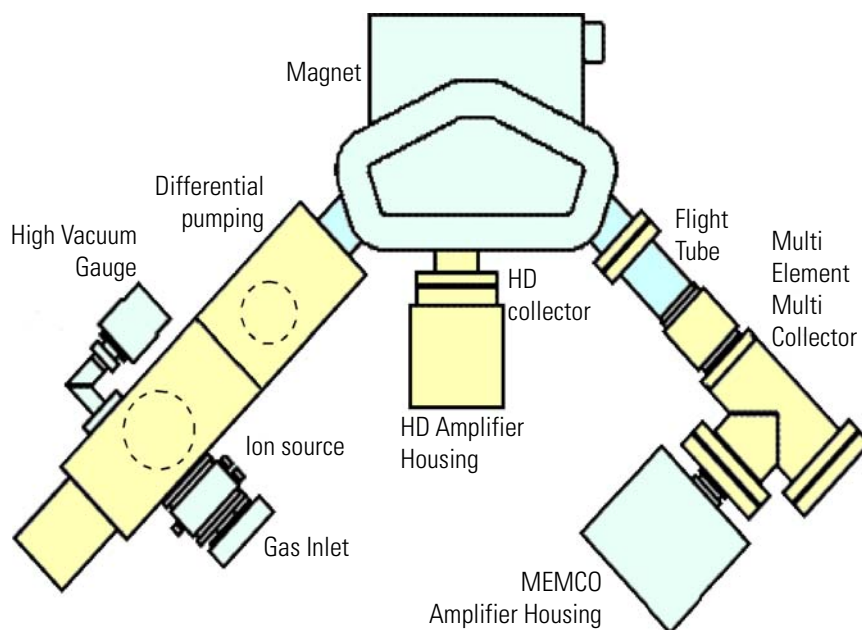
## Chapter 2 Analyzer

This chapter describes the analyzer of the MAT 253. It contains the following topics:

- “Overview” on page 2-2
- “Gas Inlet” on page 2-5
- “Ion Source” on page 2-6
- “Ion Source Control Unit” on page 2-8
- “Ion Deflection” on page 2-14
- “Ion Detection – Collector System” on page 2-15

# Overview

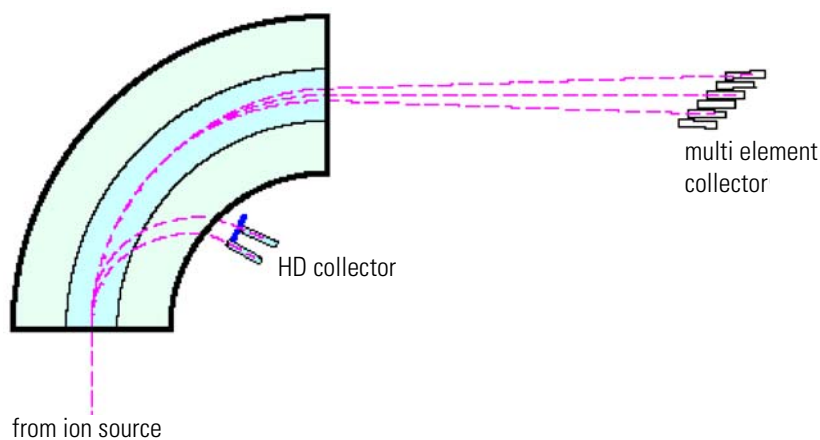
Figure 2-1 shows the arrangement of the analyzer.



**Figure 2-1.** Schematic view of instrument showing analyzer arrangement

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.

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 Figure 2-2.



**Figure 2-2.** Schematics of ion path

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).

The magnetic sector field is generated by an electromagnet with a maximum field strength of 0.75 T. It covers a mass range up to  $m/z$  150 at full accelerating voltage. The mass setting is achieved by varying the magnetic field strength and/or 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:

$$m/z = k_M \times H^2$$

with  $z$ : elementary charge

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

where

- $r$  Nominal radius of ion path ( $r = 23 \text{ cm} = \text{const.}$ )
- $U$  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  $U = 10 \text{ kV}$ ,  $k_M = 26.45 \times 10^{-6} \text{ m}^2/\text{V}$  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 (for measuring  $\text{SF}_6$  or Xe, for example) to 3.5 mm and result in a resolution of:

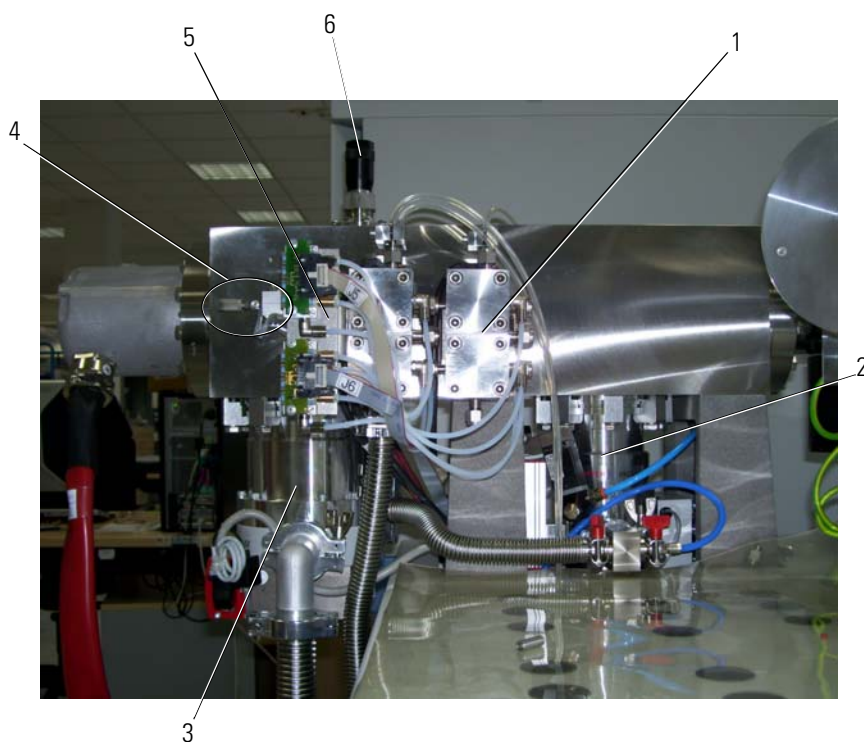
$$\frac{m}{\Delta m} = 200 \text{ (10\% valley) for 1.5 mm cups}$$

Details on the various collector types are given in “[Ion Detection – Collector System](#)” on [page 2-15](#).

The electronic units supply the analyzer system operating voltages and control the analysis. They are described in detail in “[Ion Source Control Unit](#)” on [page 2-8](#).

## Gas Inlet

Figure 2-3 shows the gas inlet system with several optional components:



Labeled Components: 1=changeover extension (optional), 2=differential pump (optional), 3=turbomolecular vacuum pump, 4=needle valve with heater, 5=changeover valve (only with Dual Inlet system), 6= actuator for VISC

**Figure 2-3.** Source section gas inlet

The turbomolecular vacuum pump **3** is switched on at the main panel, see ANALYZER PUMPS in Figure 1-9. All continuous flow peripherals are connected to the needle valve **4**. For dedicated continuous flow peripherals Thermo Fisher Scientific recommends to use an additional differential pump, see **1** in Figure 2-3.

The changeover valve **5** is the inlet for an optional Dual Inlet system, refer to “Dual Inlet System (Optional)” on page 5-1. The changeover extension **2** in Figure 2-3 can optionally be used to connect two Dual Inlet systems. The actuator **6** in Figure 2-3 adjusts the conductance, as described in “Ion Source” on page 2-6.

## Ion Source

The ion source of the 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 gastight 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. Refer to “[Ion Source Control Unit](#)” on [page 2-8](#).

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 2-4](#) on [page 2-7](#)), 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.

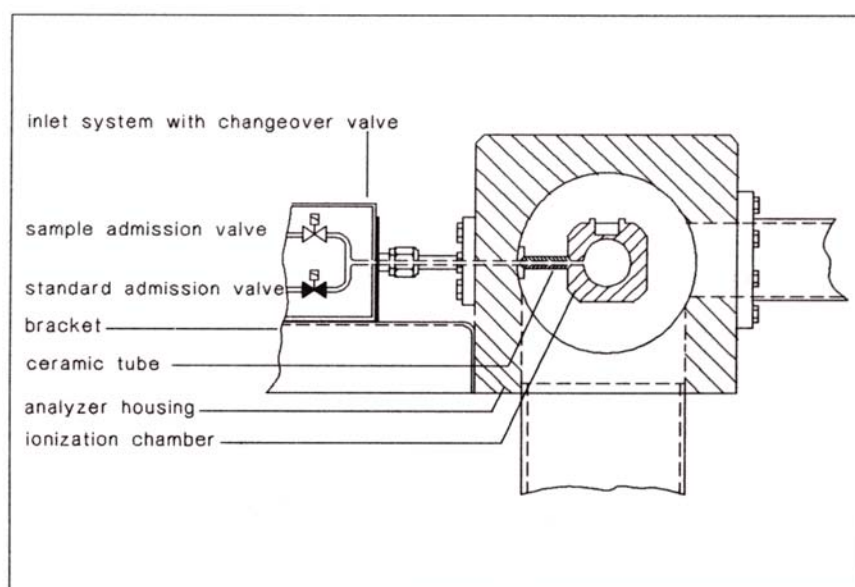
Mechanical tolerances might cause a slight out-of-axis deflection of the ion beam. Some of the lenses of the system are half sections that are insulated from each other. This construction allows for compensation by applying different potentials to the halves of the lenses. See “[Connections to Ion Source](#)” on [page 8-32](#) for details.

Using Isodat, 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.

**Example:** A setting to 8.0 kV results in a mass range up to 190, whereas a setting to 6.6 kV results in a mass range up to 230. ▲

The ion source is mounted on the front flange for easy maintenance. Correct alignment of the ion source relative to the analyzer tube is achieved by a mating surface with the analyzer head. See also [Figure 2-4](#). Details of the ion source, for example the feedthroughs and correlation of lenses, are given in [Chapter 8: “Technical Information”](#).



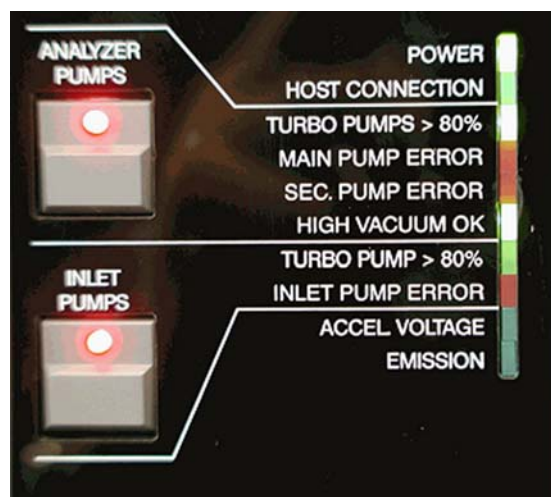
**Figure 2-4.** Coupling inlet system to ion source

## Ion Source Control Unit



**Warning Electric Current.** Danger of electric shock. Opening the electronics cabinet is only allowed for maintenance purposes by qualified personnel! ▲

Figure 2-5 shows the control panel at the front of the MAT 253:



**Figure 2-5.** Control panel at front of MAT 253

Table 2-1 summarizes a description of the ANALYZER PUMPS LEDs at the control panel:

**Table 2-1.** Control panel LEDs of ANALYZER PUMPS

LED	Description	
ANALYZER PUMPS	Main pump (source pump) and secondary pump (differential pump). They are both connected to a Fore Vacuum pump (for example, #1 in Figure 2-10).	
POWER	turns on, if	the power plug has been inserted into the socket the main switch has been switched on 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.

Table 2-2 summarizes a description of the INLET PUMPS LEDs:

**Table 2-2.** Control panel LEDs of the inlet system turbo pump

LED	Description	
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.

**Note** ACCEL VOLTAGE and EMISSION can only be set via Isodat. The other parameters can be set manually. ▲



On the Smart Isotope MS toolbar, both high voltage and emission can be switched on via these icons.



High voltage can be switched on and off via these icons.



For the turbo pump of the source, differential turbo pump, and turbo pump of the inlet system the following applies:

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

**Note** For information on pump switches refer to “[Pump Switches](#)” on [page 1-10](#). ▲

**Note** For information on turbopump controls refer to “[Turbopump Controls](#)” on [page 3-5](#). ▲

Figure 2-6 shows the rear panel and the main switch of the MAT 253:

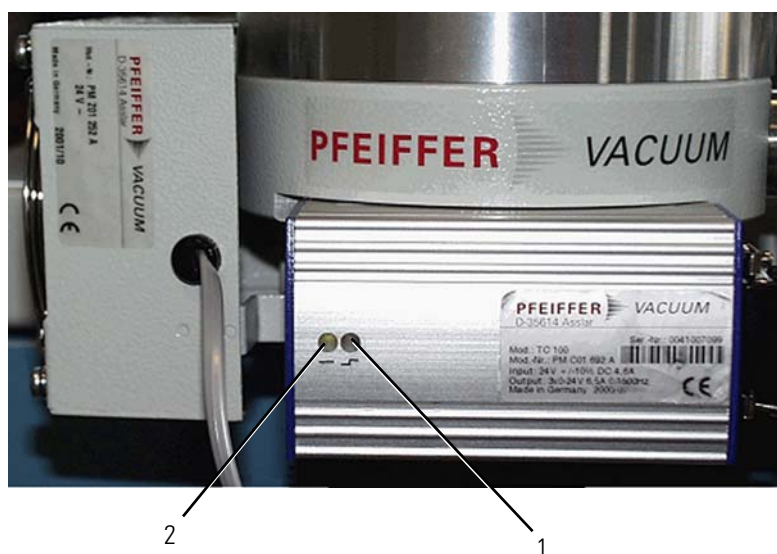


Labeled Components: 1=SUB D ports (five), 2=connections for power cables of peripherals (four), 3=fuses, 4=main switch, 5=power plug in socket

**Figure 2-6.** Rear panel of MAT 253

The control panel [Figure 2-5](#) is located at the front of the instrument on the top left corner (see [Figure 1-1](#)). The main switch **4** in [Figure 2-6](#) is located at the rear panel of the system.

Figure 2-7 shows the main pump or source pump:



Labeled Components: 1=red LED, 2=green LED

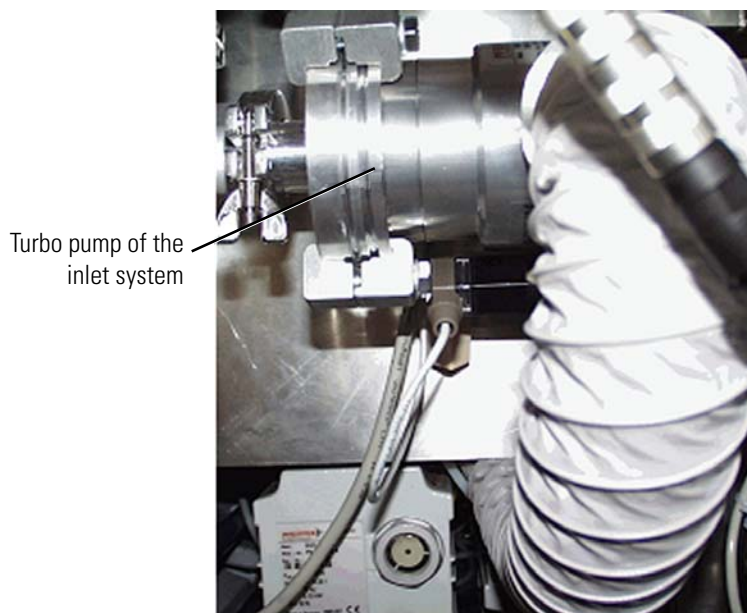
**Figure 2-7.** Main pump

Figure 2-8 shows the secondary or differential pump:



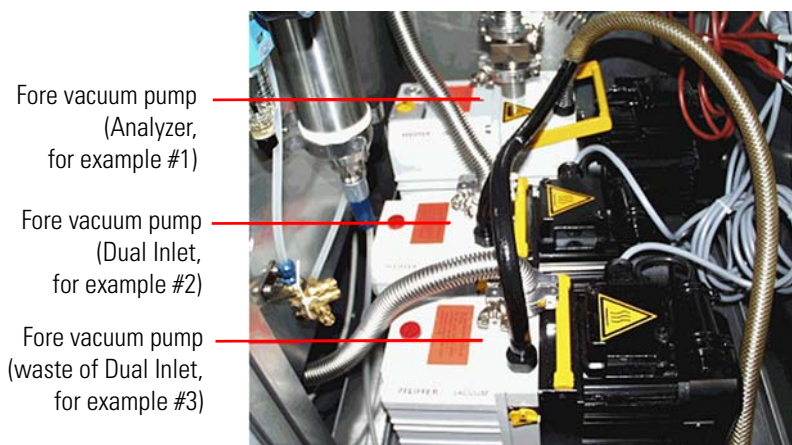
**Figure 2-8.** Secondary pump

The turbo pump of the inlet system, [Figure 2-9](#), is connected to two fore vacuum pumps (for example, #2 and #3 in [Figure 2-10](#)). It is available only in case of Dual Inlet system and is located in the lower section of the front panel of the cabinet under the bellows block.



**Figure 2-9.** Turbo pump of inlet system

[Figure 2-10](#) shows the fore vacuum pumps.



**Figure 2-10.** Fore vacuum pumps

## Emission Regulator

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

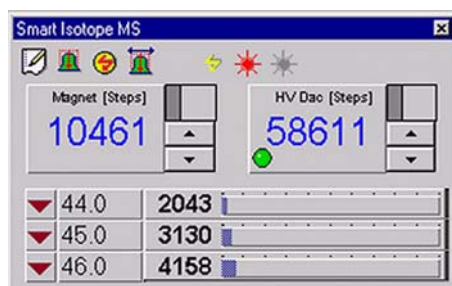
## High Voltage Supply

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 by the green Accel Voltage LED located at the front panel which turns off.

**Caution** A switch-off of the high voltage unit in case of an overload is a protective measure! You must perform troubleshooting: a short circuit in the feed line of the source might have occurred (after cleaning or reinserting the source, for example). Alternatively, after a longer time the source might have become dirty and must be dismantled, cleaned and finally reinserted. ▲

Before you start adjusting the potentials, make sure that you have:

- switched on the ion source by clicking the  button on the Focus toolbar in Isodat shown in [Figure 2-11](#),



**Figure 2-11.** Focus toolbar with button to switch on ion source

- switched on Tune Scan by selecting **Scan > Tune** in Isodat,
- admitted gas into the inlet system and a sufficient amount of gas to the ion source. Refer to “[Dual Inlet System \(Optional\)](#)” on [page 5-1](#) as well.

Focusing of the ion beam is performed via Isodat.

## **Ion Deflection**

This chapter describes the ion deflection system.

### **Electromagnet**

The magnetic field providing the ion deflection is generated by an electromagnet with maximum field strength of 0.75 T. 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.

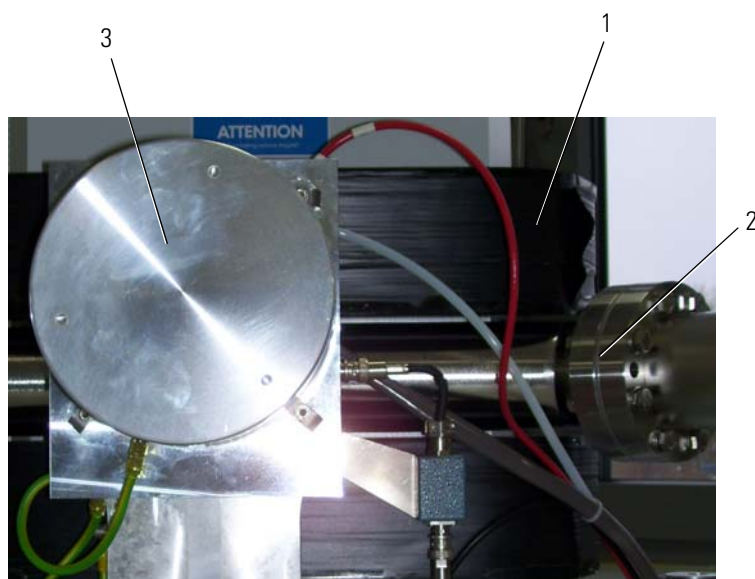
### **Magnet Current Regulator**

The magnet current regulator provides the current required to generate the electromagnetic field. For further information (for example 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.

## Ion Detection – Collector System

This chapter describes the collector system of the ion deflection, see [Figure 2-12](#).



Labeled Components: 1=magnet, 2=vacuum chamber, 3=electronics for HD collector

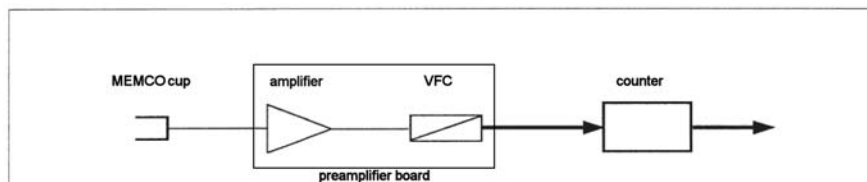
**Figure 2-12.** Magnet section with HD collector

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. For additional information, refer to [Chapter 8: “Technical Information”](#).

The collector system is installed in the collector system housing. See [Figure 2-1](#) on [page 2-2](#). 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).

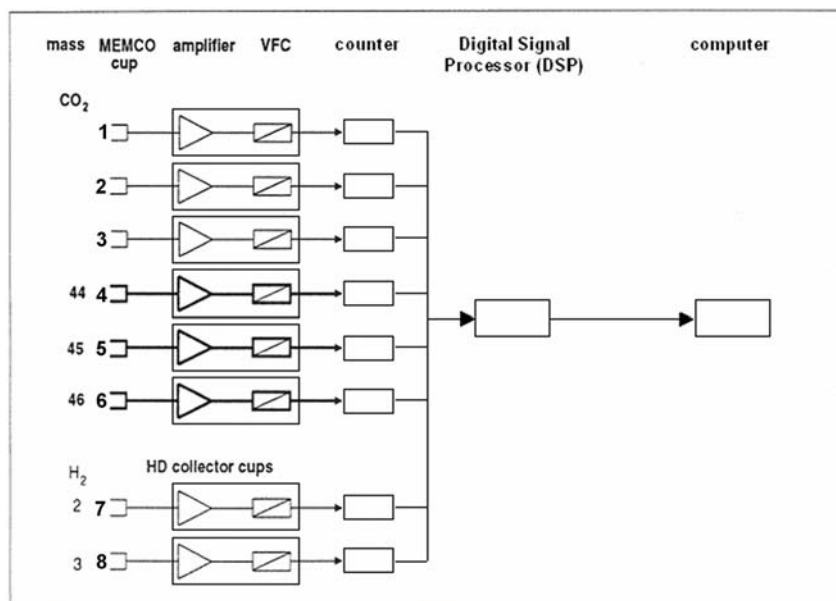
Each collector cup has its own amplifier (compare universal CNOS collector), and the feedback resistor of the amplifier can be matched to the abundance of the isotope to be collected in this cup. See [Table 2-3](#).

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 available for the MEMCO or CNOS collector system, and two VFCs for the HD collector system. They are allotted to one of the eight counters, so forming a measuring channel as shown in Figure 2-13.



**Figure 2-13.** Components forming a measurement channel

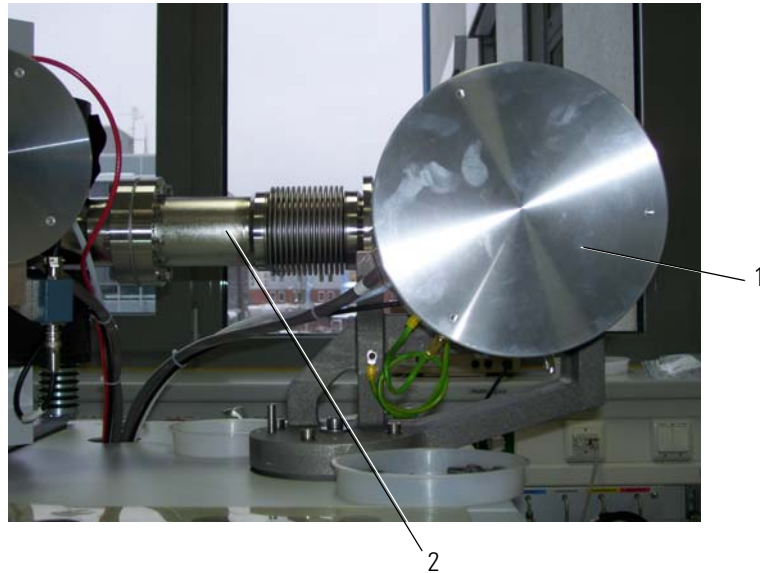
The converters transform the analog ion current signals into pulses. These pulses are fed to counters for a preselected integration time. At the end of each integration interval, the computer reads the number of counts and calculates the ion current ratios. See Figure 2-14.



**Figure 2-14.** Schematic of ion detection system

## 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. See [Figure 2-15](#).

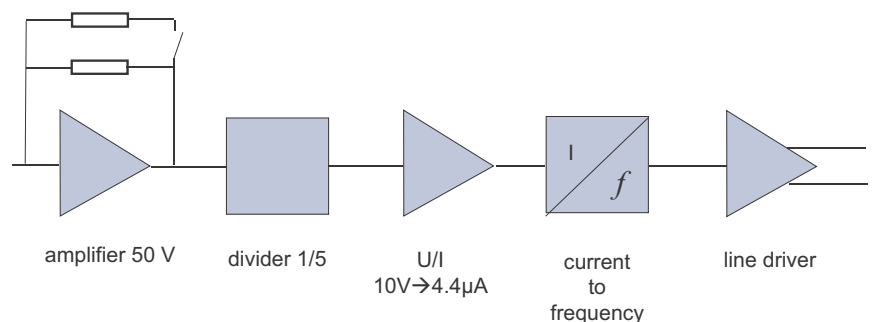


Labeled Components: 1= amplifier electronics for CNOS collector, 2=vacuum chamber

**Figure 2-15.** Collector and amplifier

## Collector Cup

Each collector cup has its own amplifier and the feedback resistor of the amplifier can be matched to the abundance of the isotope to be collected in this cup. Each collector cup and its amplifier are connected to a voltage-to-frequency converter (VFC). Up to ten amplifiers can be used to support up to ten cups. They are allotted to one of the ten counters, so forming a measuring channel as shown in [Figure 2-16](#).



**Figure 2-16.** Components forming a Measuring Channel

## Additional Resistor

All amplifiers allow the addition of a second resistor to alter amplification. The second resistor is added to the first resistor:

$$\frac{1}{R_{\text{sum}}} = \frac{1}{R_1} + \frac{1}{R_2}$$

Example: The first resistor of  $10^{12} \Omega$  is fixed. The additional second resistor of  $10^{10} \Omega$  is switchable. Two resistor values can be gained:

- Only the first fixed resistor is employed, value  $10^{12} \Omega$
- The second resistor is added to the first. The sum total - according to the equation given - with both resistors employed is  $0.9 \times 10^{10} \Omega$

**Note** It is not possible to switch between two resistors. ▲

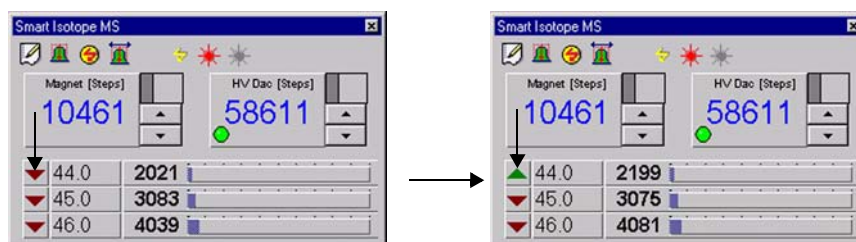
Table 2-3 shows the resistance values to be used for the isotopes of the different gases.

**Table 2-3.** Values of feedback resistors matching natural abundance of the listed isotopes

Gas	m/z	Resistor [ $\Omega$ ]	Capacity [pF]
H <sub>2</sub>	2	$1 \times 10^9$	150
	3	$1 \times 10^{12}$	2
N <sub>2</sub>	28	$3 \times 10^8$	470
	29	$3 \times 10^{10}$	5
	30	$1 \times 10^{11}$	2
O <sub>2</sub>	32	$3 \times 10^8$	470
	33	$3 \times 10^{11}$	2
	34	$1 \times 10^{11}$	2
CO <sub>2</sub>	44	$3 \times 10^8$	470
	45	$3 \times 10^{10}$	5
	46	$1 \times 10^{11}$	2
SO <sub>2</sub>	64	$3 \times 10^8$	470
	66	$1 \times 10^{10}$	15



Use the Smart Isotope MS bar of Isodat, [Figure 2-17](#), to switch between resistors. Clicking the respective icon changes between low resistance and high resistance and vice versa.



**Figure 2-17.** Switching between resistors via Smart Isotope MS bar

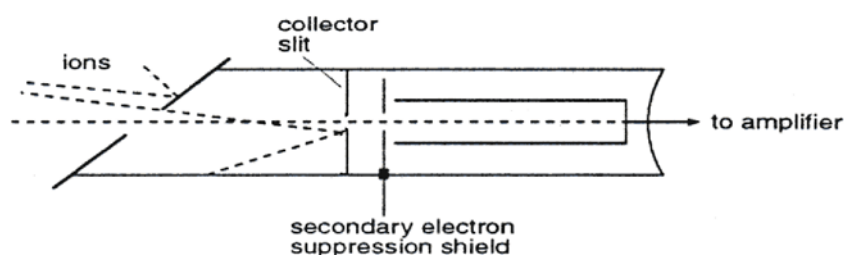
To minimize the influence of environmental changes on the amplifier stability, the amplifier housing is evacuated to a pressure below  $10^{-4}$  mbar. This is achieved by connecting the amplifier respectively its housing to the fore vacuum pump of the inlet system, and then to the side port of the turbomolecular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

The product  $R \times C$  is approximately a constant equaling the time constant of the amplifier. Usually, it amounts to 0.15 to 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. ▲

## Collector Systems

The collector systems (see [Figure 2-18](#)) cover the mass range from 1 to 150 m/z at 10 kV accelerating voltage, allowing a resolution of  $m/\Delta m = 200$  (10% valley) with 1.5 mm cups.



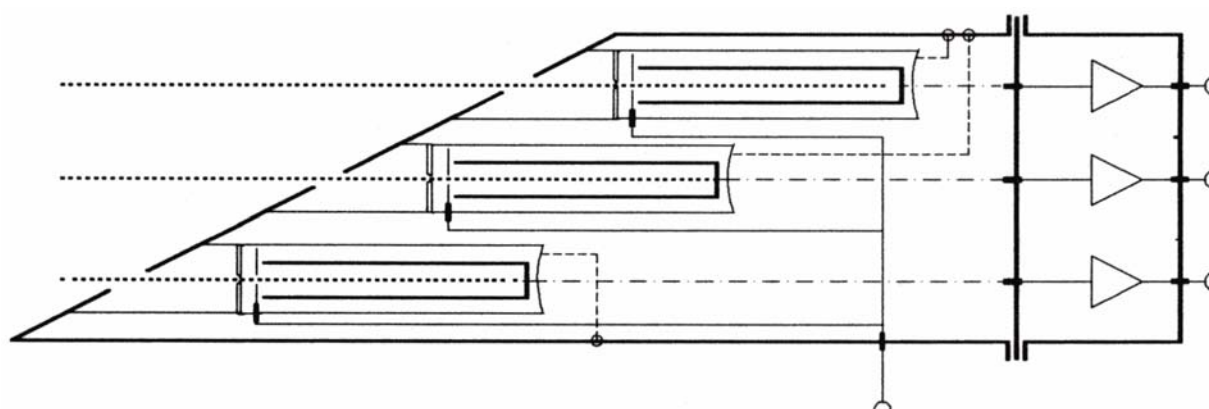
**Figure 2-18.** Design of Faraday collector cup

Owing to the high dispersion of the analyzer system, the distance between the collectors is extremely large (for example approximately 10 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. See Figure 2-18. As a result, effects that might degrade the ion current measurement are eliminated.

### MEMCO Collector System

The MEMCO (Multi Element Multi Collector) collector system comprises three to six identical cups. The 3-cup version, Figure 2-19, allows simultaneous measurement of two isotope ratios from the same sample, for example  $^{13}\text{C}/^{12}\text{C}$  and  $^{18}\text{O}/^{16}\text{O}$  of  $\text{CO}_2$ .



**Figure 2-19.** Design of 3-cup MEMCO

This way, it is not necessary to break vacuum 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. ▲



## **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.

## Chapter 3 Vacuum System

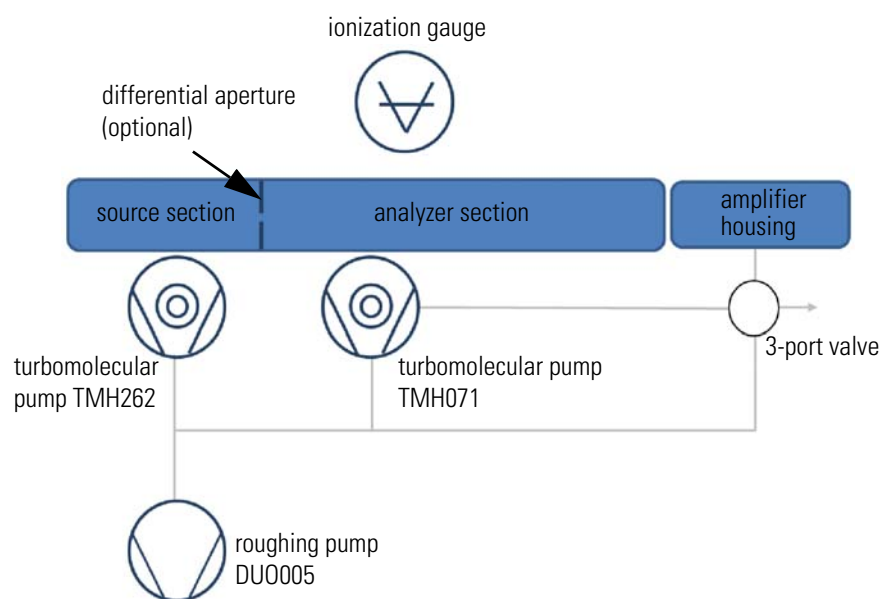
This chapter describes the vacuum system of the MAT 253 system. It contains the following topics:

- “Pumping System” on page 3-2
- “Vacuum System Control Unit” on page 3-4
- “Turbomolecular Pumps” on page 3-8
- “Pirani Gauge (Optional)” on page 3-9

## Pumping System

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

- The standard version comes with a turbomolecular pump to evacuate the analyzer system at a rate of 210 L/s (TMH262 of Pfeiffer, see [Figure 3-1](#)). The required fore vacuum is provided by a rotary pump rated at 5 m<sup>3</sup>/h (DUO005 of Pfeiffer, see [Figure 3-1](#)).
- The optional differential pumping system with an additional turbomolecular pump improves the vacuum in the analyzer system (see [Figure 3-1](#)) of the instrument. This improvement is required to reduce the high portion of helium 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. [Figure 3-1](#) schematically shows the differential pumping system.



**Figure 3-1.** Schematic of differential pumping system

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 (TMH071P of Pfeiffer, see [Figure 3-1](#)).

The required fore vacuum for both pumps is provided by a rotary pump rated at 5 m<sup>3</sup>/h (DUO005 of Pfeiffer, see [Figure 3-1](#)).

The inlet system is pre-evacuated by a fore vacuum pump rated at 1.3 m<sup>3</sup>/h (DUO 2.5 of Pfeiffer). The turbomolecular pump (TMH 071P of Pfeiffer) backed by a fore vacuum pump (DUO 2.5 of Pfeiffer) is used to provide high vacuum conditions in the inlet system. For more information, refer to “[Turbomolecular Pumps](#)” on [page 3-8](#).

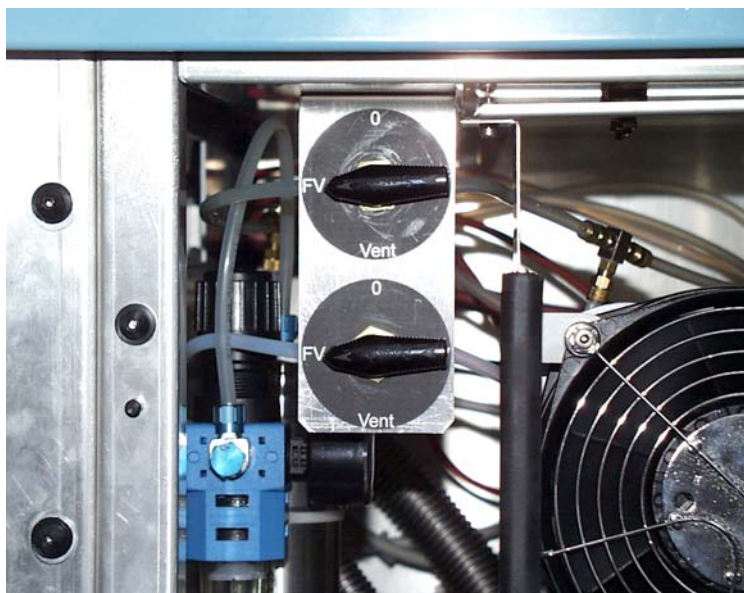
## Vacuum System Control Unit

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 fore vacuum is controlled by a Pirani vacuum gauge located in the inlet system.

## Vacuum in Amplifier Housing

To improve the stability of the amplifiers, the amplifier housings can be evacuated.

If 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. See [Figure 3-2](#).

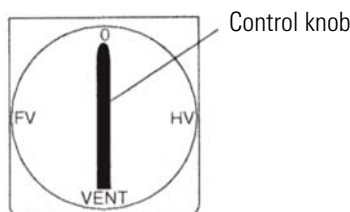


**Figure 3-2.** 3-port valves for HD amplifier housing and MEMCO amplifier housing

**Note** Each amplifier housing has to be evacuated separately. ▲

❖ **To evacuate the amplifier housings**

1. Turn the control knob of the 3-port valve (Figure 3-3) anti-clockwise from 0 to FV.

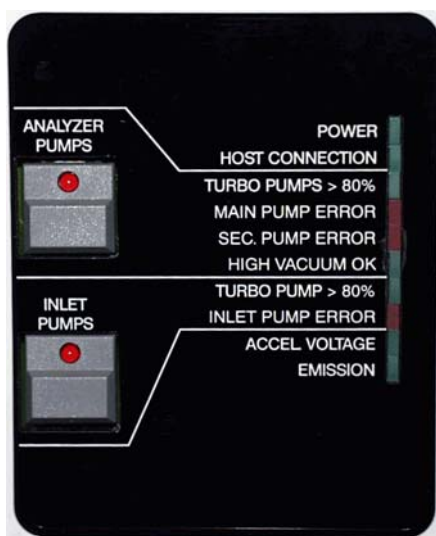


**Figure 3-3.** 3-port valve

2. After approximately 30 min, turn the control knob clockwise from FV to HV. The housing is evacuated.
3. Before exchanging resistors on the amplifier, turn the 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 by pressing the pump switch twice.

## Turbopump Controls

Figure 3-4 shows the control panel with the switches and LEDs of the analyzer pumps and the inlet pumps.



**Figure 3-4.** Control panel with pump switches and LEDs



The ANALYZER PUMPS switch starts the pumping system, that is the turbomolecular pumps and the fore vacuum pump for the analyzer system.



The INLET PUMPS switch starts the turbomolecular pump and the fore vacuum pump for the inlet system.

**Note** For information on pump switches, refer to “[Pump Switches](#)” on [page 1-10](#). ▲

**Note** For information on the ion source control unit, refer to “[Ion Source Control Unit](#)” on [page 2-8](#). ▲

The LEDs are on when the turbomolecular pumps reach 50% of their nominal rotational speed. A green LED indicates proper function of the respective system part. A red LED indicates an error in the respective system part.

**Note** If the vacuum system works properly, the red LEDs are off. In case of problems, for example a high vacuum > 10<sup>-4</sup> mbar, the ion source will be switched off automatically. ▲

[Table 3-1](#) summarizes the meaning of the LEDs of the control panel.

**Table 3-1.** LEDs at control panel

Pump	LED	Description
	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 If the LED is off with Isodat started, no connection between IRMS and 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 turbomolecular pump 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 The ion source can only be switched on when this LED is on.

**Table 3-1.** LEDs at control panel, continued

Pump	LED	Description
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	is on when high voltage is provided to the ion source
	Emission	is only on when the filament of the ion source operates

## Guidance for Troubleshooting

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

[Table 3-2](#) summarizes information for troubleshooting of the LEDs at the control panel.

**Table 3-2.** Troubleshooting for LEDs at control panel

LED on/off	Comment for troubleshooting
High Vacuum OK LED off	There might be a leak in the system. There might be a malfunction of the Penning gauge. Too much background for m/z 28 and/or m/z 40 is measured in the mass spectrometer.
Accel Voltage LED on Emission LED off	The cathode might be burned out. For checking and removal of the filament, refer to <a href="#">Chapter 8: "Technical Information"</a> .
Accel Voltage LED off Emission LED on	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 Isodat.

## Turbomolecular Pumps

Turbomolecular pump work 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 arrangement 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 fore vacuum 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 vent valve. When starting the turbomolecular pump, the vent 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 electric power, the valve is open.

For details regarding function and design of the turbomolecular pumps, refer to the operating manual of the manufacturer.

## Control Electronics and Power Supply

Each turbomolecular pump has an integrated control electronics and power supply. The pumps are started by the switches at the control panel. In addition to the LEDs on the control panel, several LEDs for error indication are directly attached to the turbomolecular pumps. See [Figure 2-7 on page 2-11](#). In case of an error, refer to the operating instructions of the pump manufacturer.

## Pirani Gauge (Optional)

The Pirani gauge monitors the fore vacuum pressure within the mass spectrometer. In case the customer upgrades the MAT 253 with the Pirani gauge, it is installed by a Thermo Fisher Scientific field service engineer. The installation is done vertically to minimize contamination with oil.

The optional Dual Inlet system contains a Pirani gauge to monitor fore vacuum pressure within the Dual Inlet system. Refer to “[Dual Inlet System \(Optional\)](#)” on [page 5-1](#) for details.

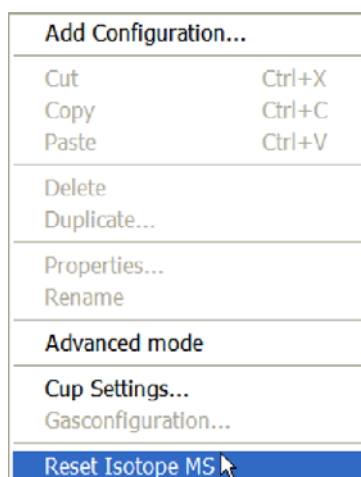
Isodat supports the Pirani gauge with SP 3.0.0.64 or higher. If the Pirani gauge has been purchased separately, the system software needs to be reset to identify the Pirani gauge, see for example [Figure 3-12](#).

## Resetting Mass Spectrometer

**Caution** Upon reset of the mass spectrometer all configurations including the cup settings are deleted. Remember the settings for later reconfiguration. ▲

### ❖ To reset the mass spectrometer

1. Double-click  to open the **Configurator** tool.
2. Select **Edit > Reset Isotope MS**, see [Figure 3-5](#).

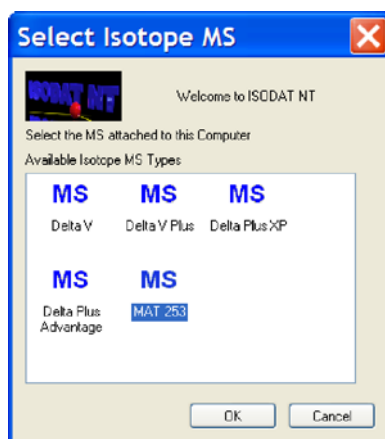


**Figure 3-5.** Selecting Reset Isotope MS



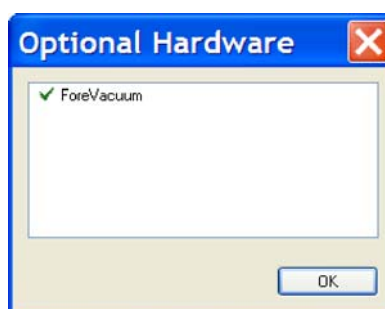
**Figure 3-6.** Reset deletes all configurations

3. Confirm with **Yes** to continue the reset. See [Figure 3-6](#).



**Figure 3-7.** Selecting MAT 253 as mass spectrometer

4. Select your mass spectrometer, MAT 253, for example. See [Figure 3-7](#).
5. Click **OK**.  
The **Optional Hardware** window opens, see [Figure 3-8](#).

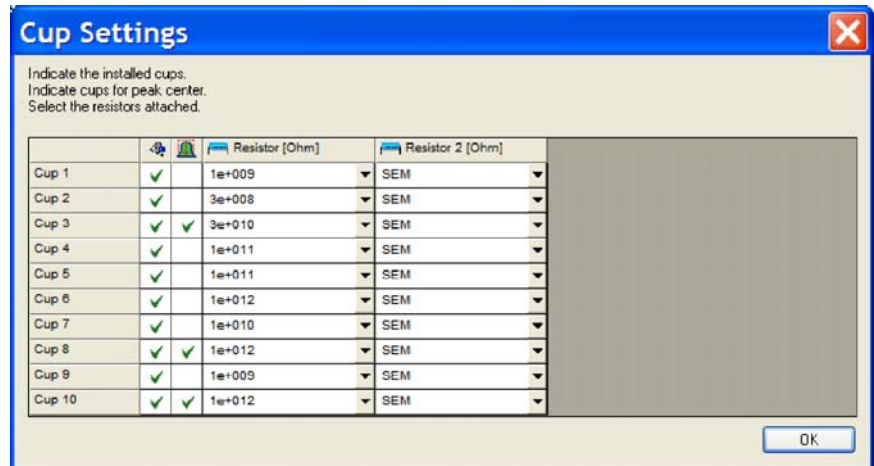


**Figure 3-8.** Selecting Pirani gauge

6. Click **ForeVacuum**.
7. Click **OK**.  
The **Cup Settings** window opens, see [Figure 3-9](#).

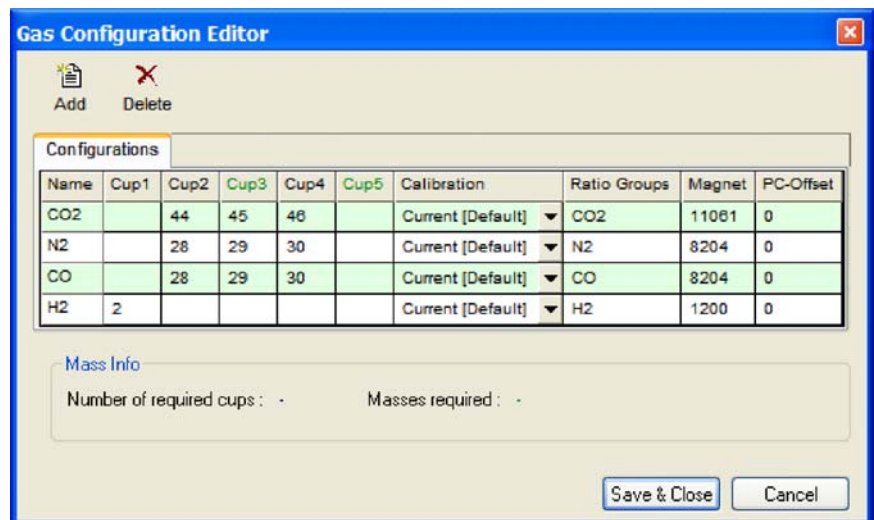
❖ **To configure cup settings and gas configuration**

**Note** The default cup configuration installed on your MAT 253 system is shown on the Collector Configuration Sheet supplied with your instrument. The Collector Configuration Sheet can also be requested from Thermo Fisher Scientific (Bremen). ▲



**Figure 3-9.** Default cup settings

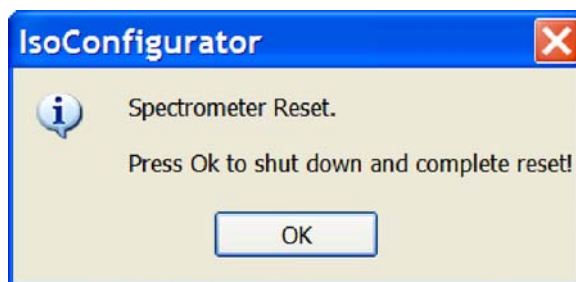
1. Check the cup settings. Confirm with **OK**.  
The **Gas Configuration Editor** opens, see [Figure 3-10](#).



**Figure 3-10.** Default gas configurations in Gas Configuration Editor

2. Check the individual gas configurations.
3. Click **Save & Close**.  
You are informed that your mass spectrometer has been reset.

4. To shut down Isodat and thereby complete the process of resetting the mass spectrometer, confirm with **OK**. See [Figure 3-11](#).

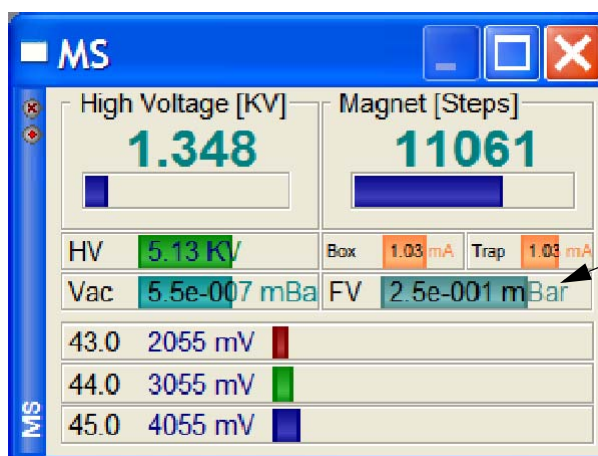


**Figure 3-11.** Shutdown of Isodat to complete reset

## Pirani Gauge in MS Panel

The Pirani gauge is shown in the MS panel of the Acquisition mode (or Instrument Control) of Isodat, see [Figure 3-12](#).

If the Pirani gauge has previously been selected (see [Figure 3-8](#)), it will be symbolized as **FV** in the MS panel of the Accessories toolbar. The current value of the fore vacuum is monitored there. See arrow in [Figure 3-12](#).



**Figure 3-12.** MS panel if a Pirani gauge is available

## Chapter 4 Electronic Devices

This chapter describes the electronic devices of the MAT 253. It contains the following topics:

- “Overview” on page 4-2
- “Plug and Measure” on page 4-4
- “Data Acquisition” on page 4-7

## Overview

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

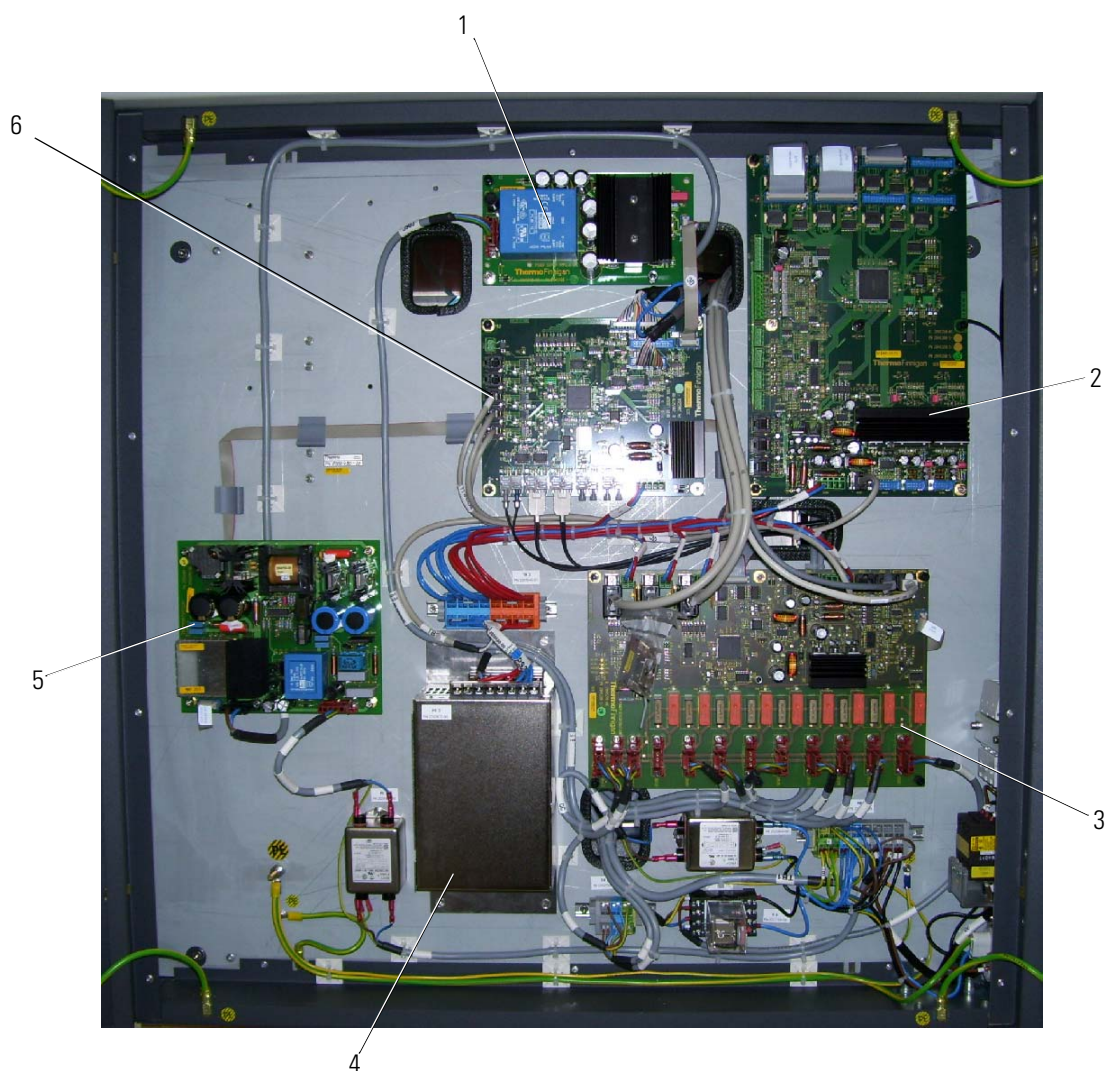
- Power Distribution
- Internal & External Inlet Controller (Plug & Measure Adapter)
- Data Acquisition (data logger, groundplane, preamplifier board, 55 V power supply)
- Ion Source Controller (emission regulator, high voltage potential controller, high voltage supply, bus controller)
- Magnet Current Regulator
- DEL-PCI Controller

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



**Warning High Voltage!** Opening the electronics cabinet is only allowed for maintenance purposes by qualified personnel. Be careful when removing the protective covers from plugs, cables and other parts. ▲

Most of the electronic boards are parts of the electronics cabinet that is located on the rear side of the IRMS. This section describes the basic structure of the electronics equipment. [Figure 4-1](#) shows the electronics cabinet of the MAT 253.



Labeled Components: 1=power supply 55 V, VFC and amplifier, 2=external inlet controller, 3=power distribution, 4=power supply 24 V, 5=magnet current regulator, 6=data logger

**Figure 4-1.** Electronics cabinet of MAT 253

Selected boards, which are located in the electronics cabinet as well as in other parts of the MAT 253, will be described in the following sections.

## Plug and Measure

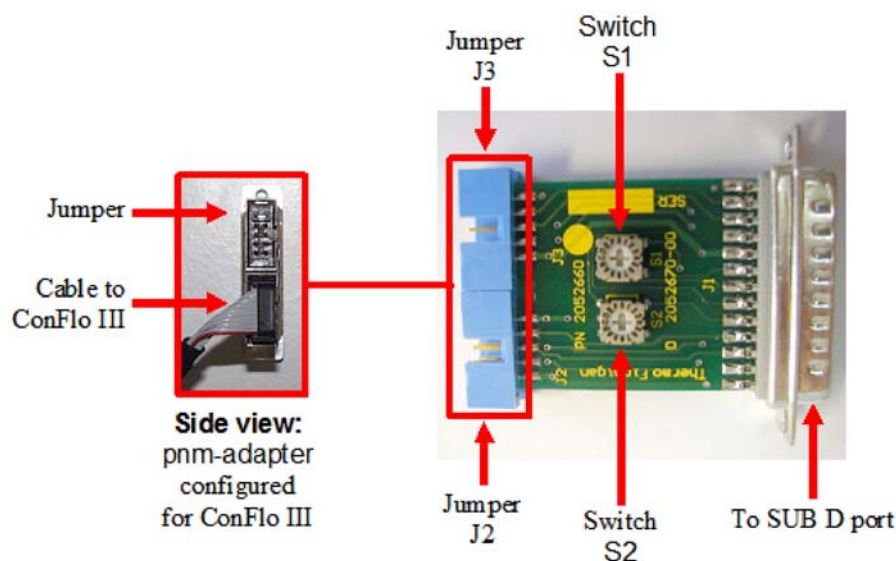
Each peripheral device has its own plug and measure code. This code is encoded either in the cable to the device or in a plug and measure adapter.

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 (when the device cable is unplugged accidentally, for example), an error message will be displayed.

### Plug and Measure Adapter (PNM Adapter)

The plug and measure adapter (pnm adapter), [Figure 4-2](#), is used for connecting the peripherals. It has been pre-configured by Thermo Fisher Scientific for a defined option (ConFlo III, for example). A supplementary reconfiguration by the user is not recommended. The plug and 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 (for example, for ConFlo III 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, that is either socket J3 or J2, are cut short from the outside of the pnm adapter.



**Figure 4-2.** Plug and measure adapter (pnm adapter)

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

## 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 however, for peripheral devices which already run using a new cable (PreCon, GP Interface, for example).

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. See arrow in [Figure 4-3](#).



**Figure 4-3.** Grounding bolt right to main switch

## Configuring 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 rear side of the instrument. The option is recognized, because it is encoded with a pnm-ID. The electronics recognizes that there is an option connected to a certain port and which kind of option it is.

[Table 4-1](#) shows the peripherals and some important parameters.

**Table 4-1.** Peripherals and pnm-IDs

Peripheral	pnm-ID	Second ID	Jumper pin 9/10	New cable
ConFlo III	0x02	0x03	J3	
GCC II/III	0x04	0x05	J3	
GasBench	0x08	0x09		
PreCon	0x0A	0x0B		Yes
Acid Pump	0x18	0x19	J2	
GC/GP	0x10	0x10		Yes

**Table 4-1.** Peripherals and pnm-IDs, continued

Peripheral	pnm-ID	Second ID	Jumper pin 9/10	New cable
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, that is either socket J3 or J2, are cut short (from the outside of the pnm-adapter).
- New cable: no pnm-adapter. Instead, it is necessary to exchange the cable.

**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. ▲

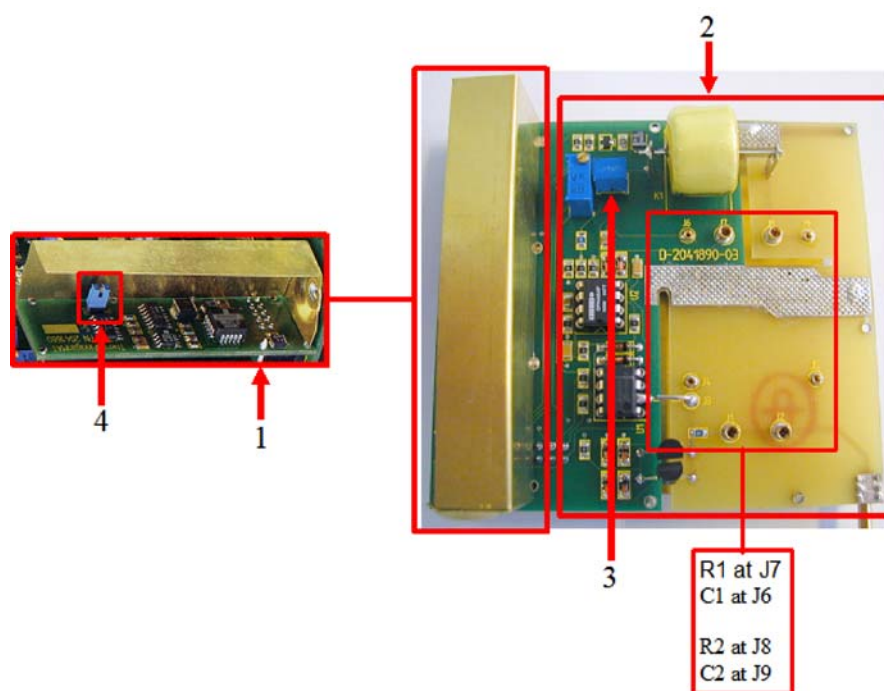
## Data Acquisition

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

### Amplifier and VFC

**Caution** The amplifiers might be damaged when they are exchanged while the power supply is switched on! ▲

For each of the eight acquisition channels, a preamplifier board (P/N 2041880, shown in Figure 4-4) 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.

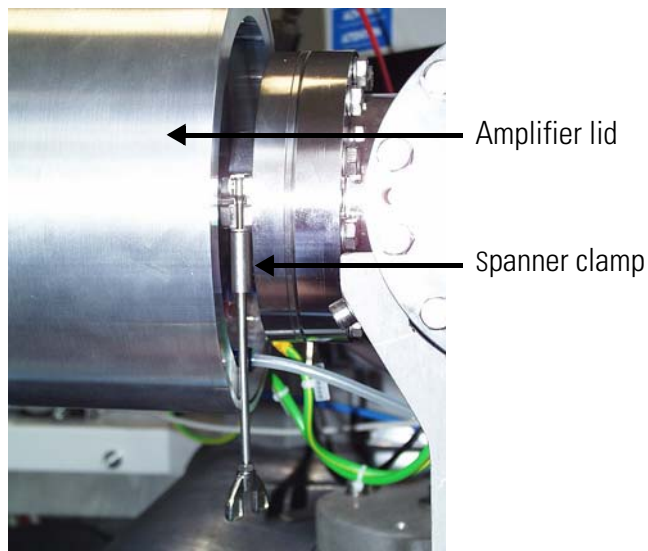


Labeled Components: 1=VFC, 2=amplifier, 3=setting of time constant, 4=jumper to disconnect amplifier and VFC (for service purposes)

**Figure 4-4.** Preamplifier board

## Exchanging 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. See [Figure 4-5](#).



**Figure 4-5.** 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 groundplane with the preamplifier boards from the collector. ▲

### ❖ To disassemble the amplifier

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

### ❖ To assemble the amplifier again

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.

- 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. A damage may be caused when the preamplifier board is not exactly positioned on the corresponding contact pin. ▲

- Secure the preamplifier boards by screws.
- Put on the amplifier lid again.



## Chapter 5 Dual Inlet System (Optional)

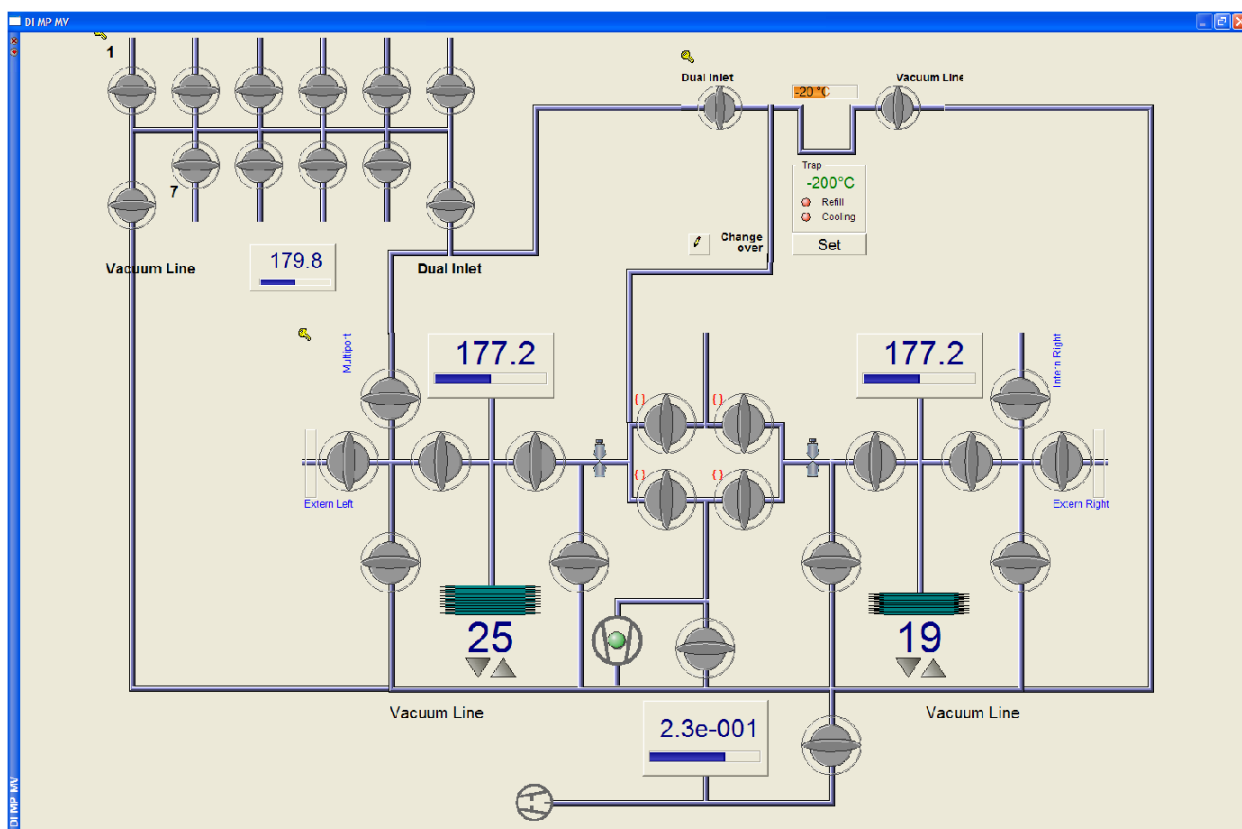
This chapter describes the Dual Inlet system. It contains the following topics:

- “General Description” on page 5-2
- “Valve System” on page 5-4
- “Changeover Valve” on page 5-6
- “Multiport” on page 5-8
- “Microvolume” on page 5-10

## General Description

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.

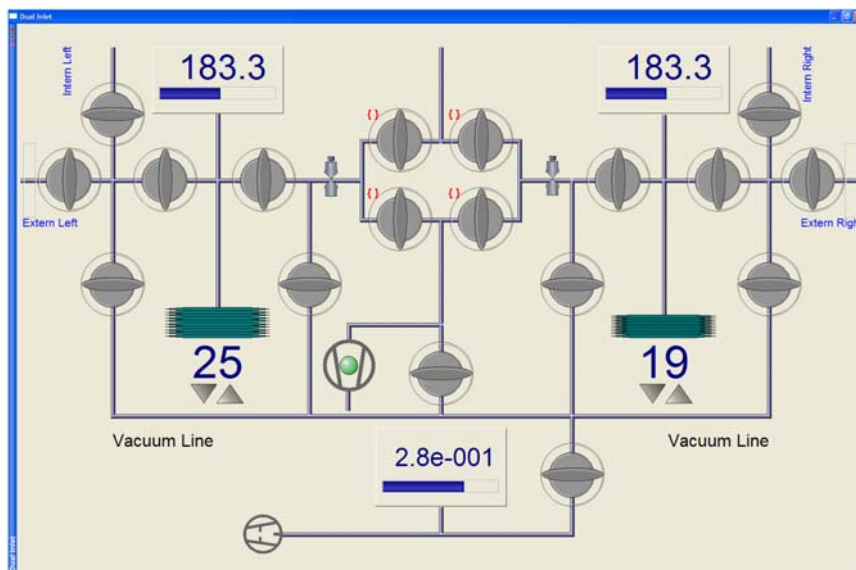
For instruments equipped with a Dual Inlet system, the configuration is identical for the sample side and the standard side. This enables a balanced flow. Each inlet side has two ports and a variable volume (bellows) with the respective inlet capillary leading to the changeover valve, see Figure 5-1. For very small samples, a Microvolume with its own capillary is installed.



**Figure 5-1.** Schematics of Dual Inlet system in Isodat

Before measurements can be performed and results be compared, equal gas conditions – such 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) adjustable in volume.

These variable volumes are bellows that are operated by software-controlled motors. An automated procedure balances the volumes so that the ion beam intensity of a selected mass attains a preset value. Balancing of the volumes can also be performed manually. The bellows are adjustable from about 3.5 mL to 40 mL each. [Figure 5-2](#) shows the Dual Inlet system in Isodat.



**Figure 5-2.** Dual Inlet system in Isodat

Precise isotope ratio determination via Dual Inlet requires a stable gas flow into the ion source. To obtain this, bellows balancing (**Pressure Adjust** in Isodat) of both sides is essential. If sample gas flows into the ion source, an equal amount of a standard gas is evacuated simultaneously by the vacuum line 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 anew when a capillary is replaced. How to crimp a capillary to a specific flow resistance is described in [“Heating Inlet Capillaries”](#) on page 7-60 and [“Replacing an Inlet Capillary”](#) on page 7-62.

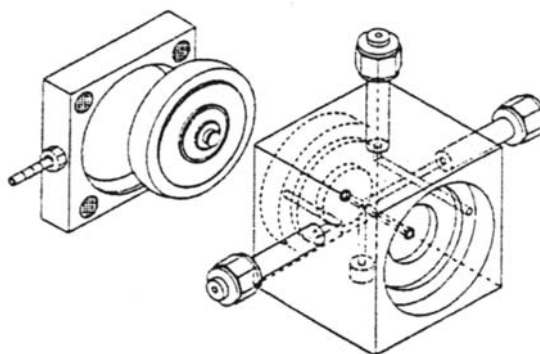
The variable volumes adjust the pressure for larger samples ( $> 50 \text{ bar} \times \mu\text{L}$ ). Very small samples, as low as  $5 \text{ bar} \times \mu\text{L}$ , can be analyzed using the optional Microvolume inlet. For more details, refer to [“Microvolume”](#) on page 5-10.

To avoid any condensation, to remove impurities or to measure  $\text{SO}_2$ , the Inlet system including the changeover valve and the ion source housing can be heated up to  $80 \text{ }^\circ\text{C}$ .

## Valve System

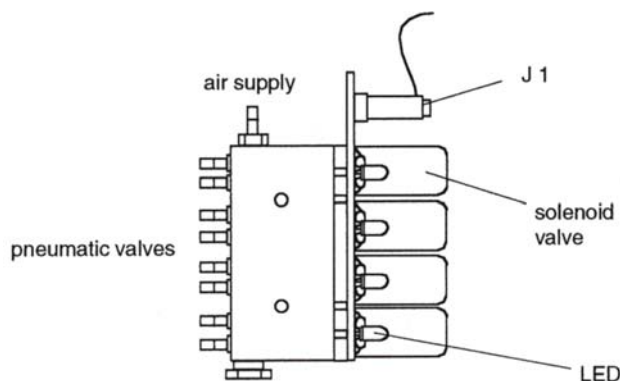
The inlet system is operated by pneumatic valves with a nominal closing pressure of 4 bar. The compressed air is supplied either by an optional compressor attached to the mass spectrometer or by a user-supplied pressure air line.

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-in 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. [Figure 5-3](#) schematically shows a double valve block.



**Figure 5-3.** Schematic of double valve block

Four of the solenoid three-way valves are located on a manifold block. See [Figure 5-4](#). 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.



**Figure 5-4.** Manifold block with four solenoid valves

In case of a power failure, all solenoid valves open automatically. Thus, the pneumatic valves close avoiding contamination of the inlet system. [Figure 5-5](#) shows the driver board with distributor function.



**Figure 5-5.** Driver board with distributor function

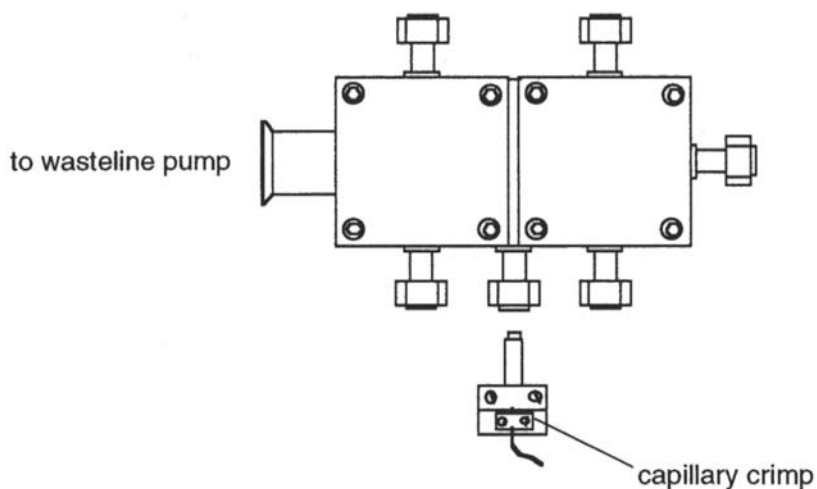
**Note** Both inlet system and options are arranged on the inlet board for the internal options. Only two further cables connect the remaining electronics to the board (control line and power supply). ▲

## Changeover Valve

This section describes the changeover valve.

### Basic Module of Changeover Valve

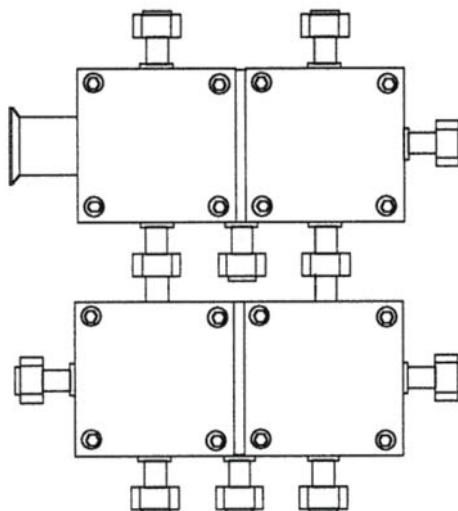
The basic module of a changeover valve, [Figure 5-6](#), consists of a single block attached to the analyzer housing and accepts the coupling of capillaries for sample and standard gas. The changeover valve is operated automatically by Isodat or manually via the monitor display of the inlet schematic.



**Figure 5-6.** Basic module of changeover valve block

## 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 1/4-in Swagelok connectors, see [Figure 5-7](#).



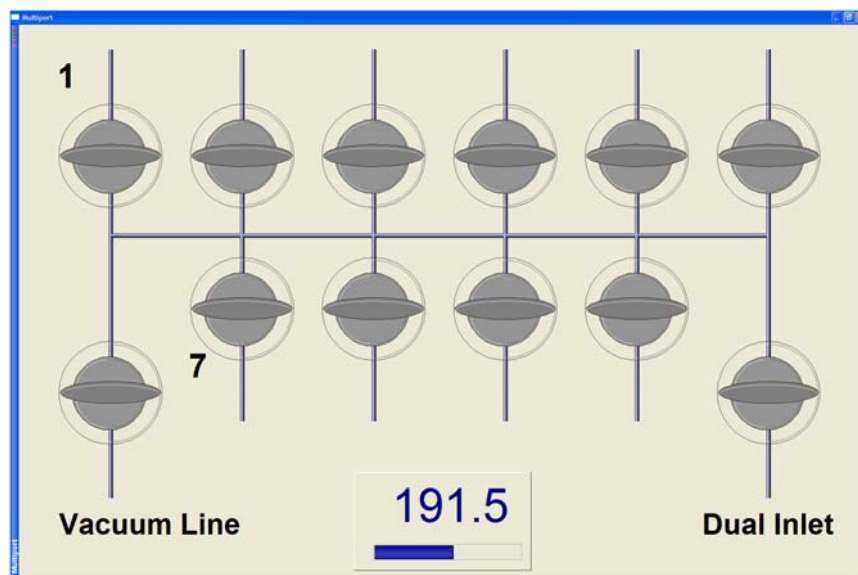
**Figure 5-7.** Extension module attached to basic module of changeover valve

The extension module provides two additional inlet ports allowing the coupling of further inlet system options, for example a carbonate system. To monitor and operate the extended changeover valve, the respective Isodat routine must be activated.

## Multiport

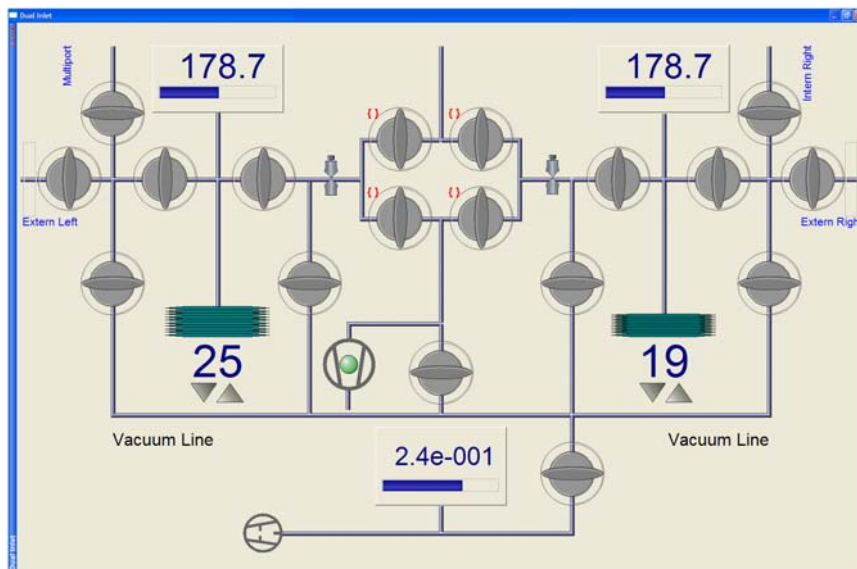
The Multiport is a sample manifold inlet system consisting of one or two banks of 10 ports each. It may be optionally equipped with tube crackers. When using the Multiport as an inlet system, it is connected directly to the sample side of the inlet system (**Intern Left** in [Figure 5-2](#)).

The valves of the Multiport are operated the same way as the components of the Dual Inlet system, that is either automatically by Isodat or manually via the monitor display. To monitor and operate the Multiport valve system on screen, the respective Isodat routine has to be activated. [Figure 5-8](#) shows the schematic of a Multiport in Isodat.



**Figure 5-8.** Multiport schematic in Isodat

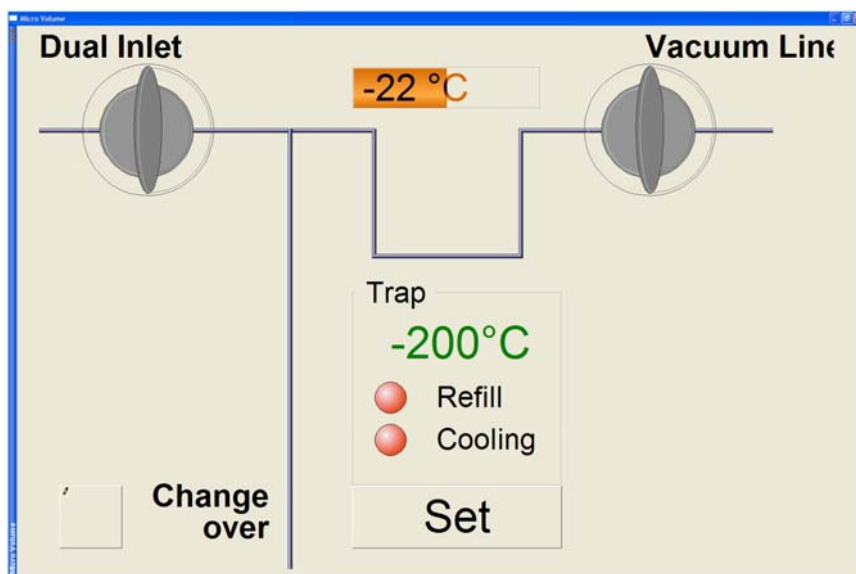
Figure 5-9 shows a Dual Inlet system with Multiport and Microvolume in Isodat. Measurement using a Multiport as an inlet system is described in “Dual Inlet Measurement Including Multiport” on page 6-24.



**Figure 5-9.** Dual Inlet system with Multiport and Microvolume

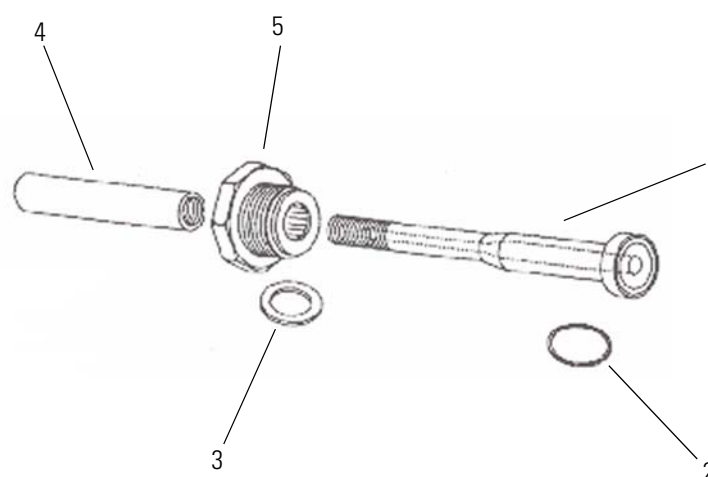
## Microvolume

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 (**Intern Left** in [Figure 5-2](#)) of the Dual Inlet system. [Figure 5-10](#) shows its schematic in Isodat.



**Figure 5-10.** Microvolume schematic in Isodat

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. [Figure 5-11](#) shows parts of a Microvolume to be inserted into an autocool unit:



Labeled Components: 1=cold finger, 2=gasket (gold), 3=washer, 4=copper rod, 5=fitting

**Figure 5-11.** Parts of Microvolume to be inserted into autocool unit

The total volume in front of the capillary crimp, that is “cold finger” volume plus the connections including the capillary, is about 250  $\mu\text{L}$ . 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  $\text{bar} \times \mu\text{L}$  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  $\text{CO}_2$  a smaller Microvolume is required, and for  $\text{N}_2$  a larger one is used. The larger one contains a molecular sieve to freeze out  $\text{N}_2$  at liquid nitrogen temperature.

The valves of the Microvolume are operated in the same way as the other components of the Dual Inlet system, that is either automatically via Isodat or manually via monitor display. Using the autocool unit the temperature can be set individually within a range of about  $-180\text{ }^\circ\text{C}$  and  $+155\text{ }^\circ\text{C}$ .

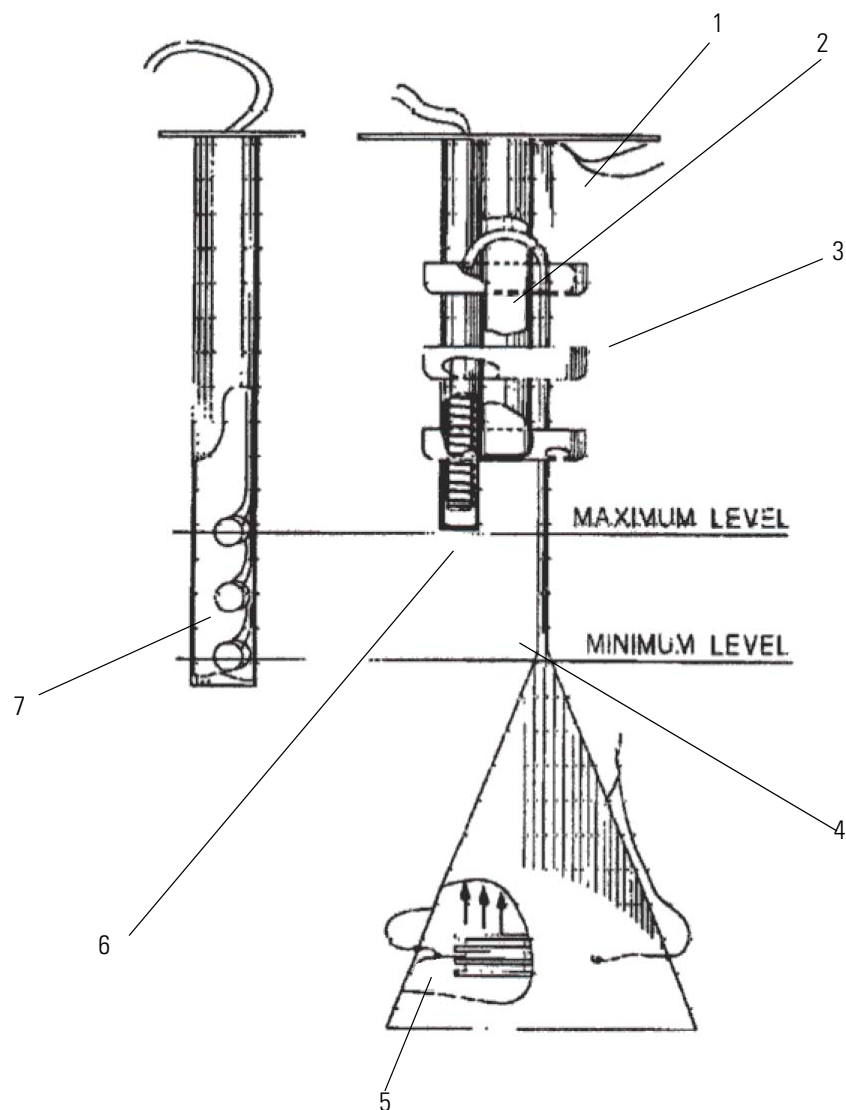
## Autocool Unit

The temperature of the autocool unit, which cools the “cold finger”, can be set via the Microvolume control of Isodat (that is via the Microvolume part of the Instrument tab). Refer to “[Dual Inlet Measurement Including Microvolume](#)” on page 6-34. A temperature range between  $-180\text{ }^\circ\text{C}$  and  $+155\text{ }^\circ\text{C}$  can be covered.

The time to get from  $+50\text{ }^\circ\text{C}$  down to  $-180\text{ }^\circ\text{C}$  is less than 2 min. The time to get from  $-180\text{ }^\circ\text{C}$  up to  $+50\text{ }^\circ\text{C}$  is about 1 min.

The Microvolume fits into a thermal contact pipe attached to the lid of a Dewar vessel. The Dewar vessel 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 [Figure 5-12](#).

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 in Figure 5-12, are Pt 100 resistors, that is 100  $\Omega$ .



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

**Figure 5-12.** Schematic of autocool unit

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 vessel, 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.

## Autocool Refill Device

This section describes the autocool refill device.

### Safety Instructions

**Note** Safety First! Make sure that only authorized and fully trained operators use this equipment and that they are fully familiar with these safety notes. ▲

Before operating the autocool refill device, carefully read these notes as well as the handling instructions of the manufacturer.



**Warning Cold Liquid.** The autocool refill device contains extremely cold liquid gas. Careless handling might cause severe personal injury including frostbite. Only use the cryogenic liquid specified on the label on the autocool refill device. ▲



Wear protective clothing when operating this equipment, including face shield. Do not overfill or tilt the autocool refill device. Prevent spills. ▲



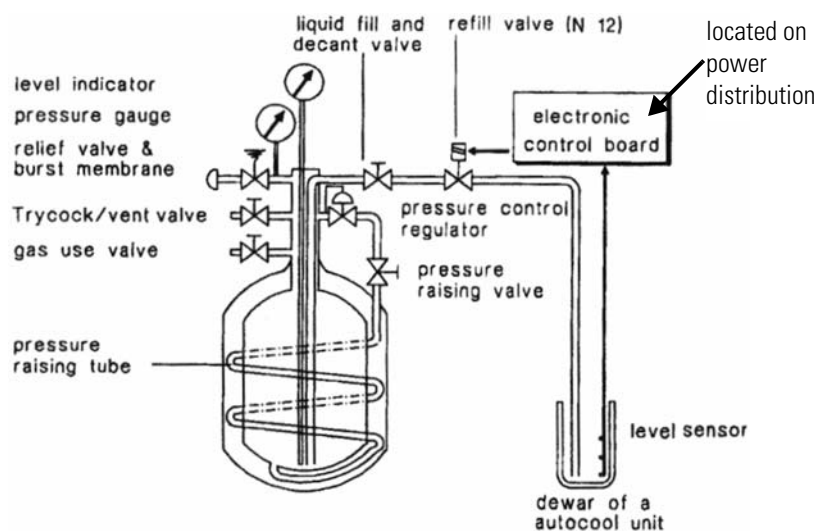
Wear protective clothing when operating this equipment, including protective gloves. Do not overfill or tilt the autocool refill device. Prevent spills. ▲



**Warning Toxic Gases.** Use the autocool 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. ▲

## General Remarks

The autocool refill device shown in [Figure 5-13](#) provides a constant level of liquid nitrogen in the Dewar vessel of the autocool unit. It consists of a Dewar vessel of 75 L or 25 L capacity and is equipped with the safety devices, valves and pressure gauges required for safely handling liquid nitrogen.



**Figure 5-13.** Schematic of autocool refill device

The transfer line to the Dewar vessel 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 autocool refill device.

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

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

## Working Principle of Autocool Refill Device

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

A pressure gauge monitors the pressure. A pressure of 0.35 bar (5 psi) should be sufficient to transfer liquid nitrogen. A pressure of 0.7 bar (10 psi) will transfer liquid at about 10 L/min. A higher pressure is not necessary and even wasteful.

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 autocool refill device is shown in [Figure 5-13](#). The 75 L Dewar vessel is equipped with a level indicator monitoring the liquid nitrogen content.

## Maintenance of Autocool Refill Device

Hardly any routine maintenance is required. However, the components of the autocool refill device should be regularly checked for damage or freeze-up. If it is necessary to dry and clean items or to replace them, make sure that they are thoroughly degreased and dried, because moisture or lubricants will freeze at cryogenic temperatures. Do not use thread-sealing compounds. Use PTFE tape, for example 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.

### ❖ To set the pressure control regulator

1. Loosen the hexagon locking nut below the adjusting screw.
2. Turn the adjusting screw counter-clockwise to set to zero.
3. Close the vent valve and liquid valve and open the pressure raising valve.
4. Turn the adjusting screw clockwise to increase the vessel pressure until gas escapes through the vent hole. Turn the adjusting screw counter-clockwise again until gas stops escaping through the vent hole.

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

## Checking Liquid Nitrogen Evaporation Rate

If you suspect that the evaporation rate of the autocool refill device is excessive, mark down the decrease of the liquid nitrogen level for some time.

### ❖ To check the loss rate

1. Close the pressure raising valve.
2. Open the Trycock/vent valve.
3. After the contents are fully vented down to atmospheric pressure, measure the boil-off rate using a simple flow meter.

**Note** The normal boil-off rate for nitrogen is 1 L/min for gas, that is about 2 L of liquid nitrogen 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.

## Operating Instructions

As a quick reference, the arrangement of valves for filling, dispensing and storage of liquid nitrogen is given in [Table 5-1](#). See [Figure 5-13](#) as well.

**Table 5-1.** Valve arrangement for liquid nitrogen supply

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
short term storage	closed	closed	open	closed
long term storage	closed	closed	closed	open

Before using the Microvolume, check the content of the autocool refill device.

### ❖ If filling is required

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 autocool refill device, first fill the Dewar vessel of the autocool unit with liquid nitrogen roughly to the required level.

❖ **To enable the automated and computer-controlled refill operation**

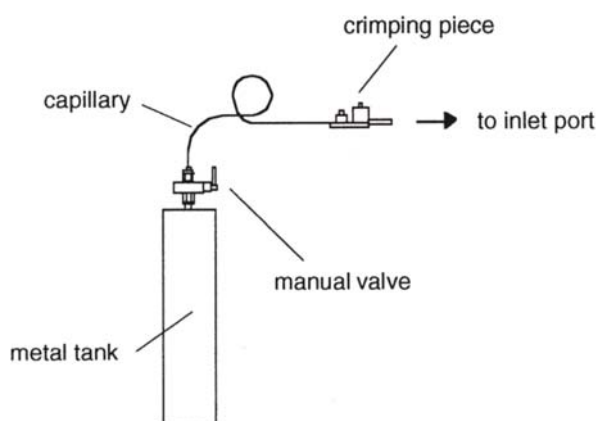
1. Check the pipe connection leading to the Dewar vessel of the autocool unit.
2. Close the gas vent valve.
3. Open the liquid fill and decant valve.
4. Open the pressure raising valve.

The autocool refill device is now connected to the Dewar vessel of the Microvolume via the opened liquid fill and decant valve. The flow is controlled via the refill valve N12. The pressure raising valve may be closed when the working pressure is reached, or it may remain open, provided the pressure regulator has been set to a suitable working pressure.

## Reference Gas Refill (Standard 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 gas refill provides the reference gas supply to the inlet system. Reference gas refill is a hardware option.

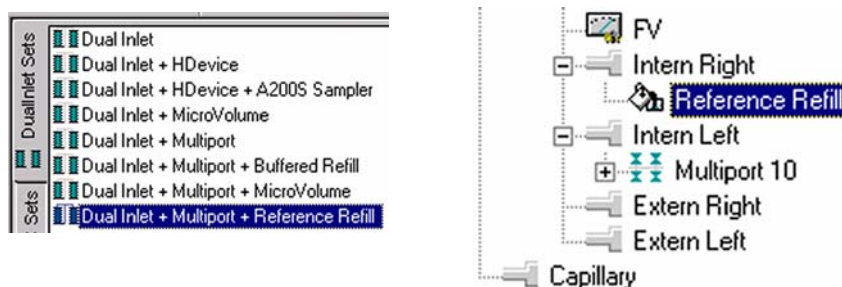
It consists of a metal tank with a capacity of approximately 5 L with a manual valve connected via a capillary to one of the inlet ports on the standard side. See [Figure 5-14](#). With the reference gas refill selected, the standard side of the inlet system is completely pumped out before it is filled again for the next measurement sequence.



**Figure 5-14.** Schematic of reference gas refill

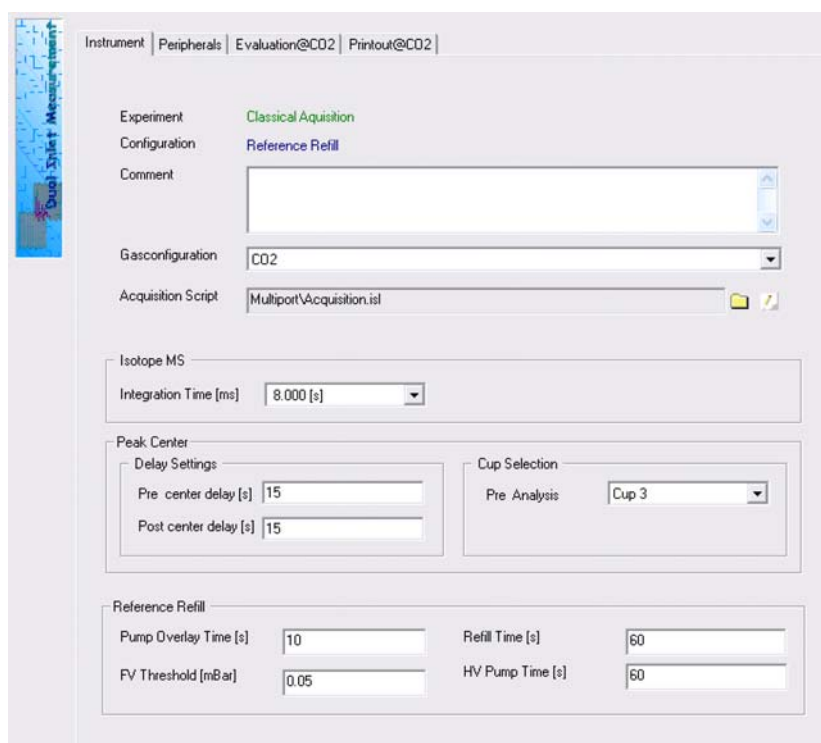
The reference gas refill is by default connected to the Intern Right port, see [Figure 5-15](#).

To create a configuration containing reference gas refill, at **Dual Inlet Sets** select the predefined **Dual Inlet + Multi-port + Reference Refill** device. See [Figure 5-15](#), left. Then, drag it to the source. See [Figure 5-15](#), right.






































**Figure 5-15.** Creating a configuration containing reference gas refill

When creating a new method note the Instrument tab in the method window. See [Figure 5-16](#) for the default values.



**Figure 5-16.** Reference gas refill on Instrument tab of method

If reference gas refill is to be used in the sequence, the row for the sample must be selected  in the Reference Refill column. See [Figure 5-17](#).

Row				Reference Refill	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 ▼

**Figure 5-17.** Sequence for reference gas refill

**Dual Inlet System (Optional)**  
Microvolume

## Chapter 6 Measurements

This chapter outlines how to perform different kinds of sample measurements. It contains the following topics:

- “Preparing Measurement” on page 6-2
- “Continuous Flow Measurement” on page 6-6
- “Zero Enrichment (Standard On/Off Test)” on page 6-7
- “Simple Dual Inlet Measurement” on page 6-11
- “Dual Inlet Measurement Including Multiport” on page 6-24
- “Dual Inlet Measurement Including Microvolume” on page 6-34
- “Dual Inlet Measurement Including Multiport and Microvolume” on page 6-54
- “Dual Inlet Measurement Including H-Device and Autosampler A200S” on page 6-65
- “Basic Procedures Before Measurement” on page 6-77
- “Viewing Results in the Acquisition Window” on page 6-92
- “Excel Export of Results” on page 6-98

## Preparing Measurement

Before starting measurement you must perform the following steps:

- “Creating a Gas Configuration” on page 1-28
- “Creating a Configuration” on page 1-32
- “Creating a New Method” on page 6-2
- “Creating a New Sequence” on page 6-3

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 ( $^{12}\text{C}^{16}\text{O}^{16}\text{O}$ ), 45 ( $^{13}\text{C}^{16}\text{O}^{16}\text{O}$ ) and 46 ( $^{12}\text{C}^{16}\text{O}^{18}\text{O}$ ).

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.



There are two different ways to access a Method or a Sequence:

- Selecting a predefined Method and Sequence using the File Browser
- or
- Creating 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, see “Creating a New Method” on page 6-2 and “Creating a New Sequence” on page 6-3.

## Creating a New Method

### ❖ To create a new method

1. In the Isodat folder, double-click  to open the **Acquisition** tool.
2. Open the Dual Inlet module. 
3. Select the configuration for your application (for example **Dual Inlet**).
4. Select the gas configuration, for example **CO2**.




5. Click the  **New** button.



6. Click the **Method** icon.  
The example values and parameters in the tabbed pages Instrument, Peripherals, Evaluation, Printout and Dyn. Externals of the new Method can be accepted.




7. Click .  
The Save As window opens.
8. Enter a name for the method.
9. Click **Save**.

## Creating a New Sequence

After having defined a new Method, you can create a new Sequence.

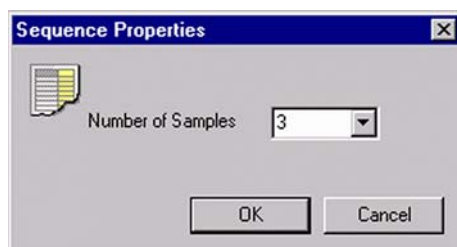
### ❖ To create a new sequence

1. In the Isodat folder, double-click  to open the **Acquisition** tool.

2. Click the  **New** button.









3. Double-click the **Sequence** icon.
4. Define the number of samples (for example: 3). See [Figure 6-1](#).



**Figure 6-1.** Defining the number of samples


5. Edit the Sequence List. See [Figure 6-2](#).

Line				Identifier 1	Method
1					
2					
3					

**Figure 6-2.** Editing the Sequence List

6. Check  to perform a peak center prior to measurement.


7. Check  to perform a pressure adjustment.

8. Check  to perform a background measurement.

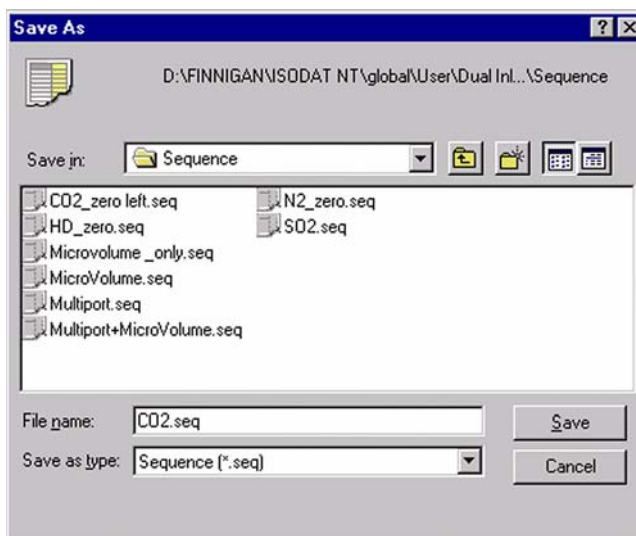
9. Edit text in column **Identifier 1** to identify the sample.

10. Select a IRMS **Method** for each sample, for example **CO2.met**.



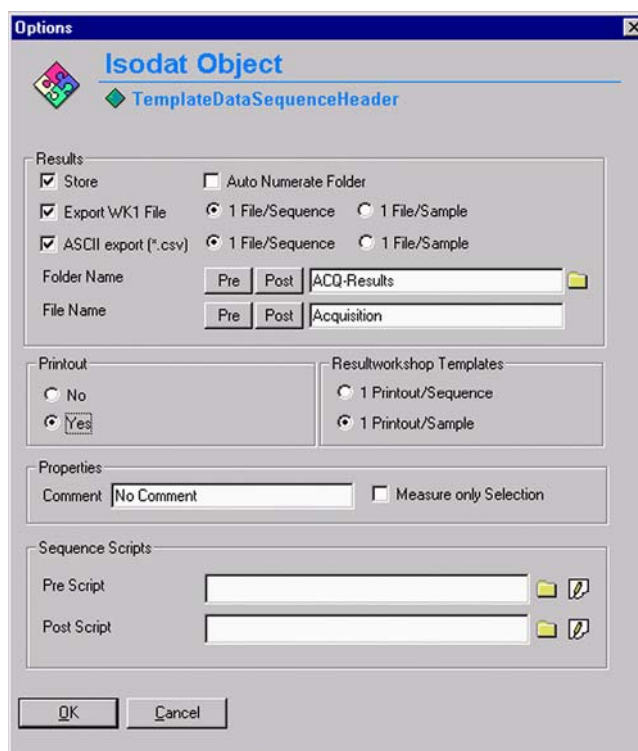
11. Click the  button.

12. Enter a File name with the extension .seq, for example **CO2.seq**, see [Figure 6-3](#).



**Figure 6-3.** Saving a sequence

13. Click **Save**.  
The Options dialog opens, see [Figure 6-4](#).



**Figure 6-4.** Defining sequence parameters

14. Select parameters for **Results**.
15. Select parameters for **Printout**.
16. Select parameters for **Sequence Scripts**.
17. Click **OK** to start Sequence acquisition.

## **Continuous Flow Measurement**


Continuous flow measurement is described in detail in the operating manuals of Thermo Fisher Scientific IRMS peripherals, for example, LC IsoLink, Gasbench II and ConFlo IV. Refer to these operating manuals for description on continuous measurement.

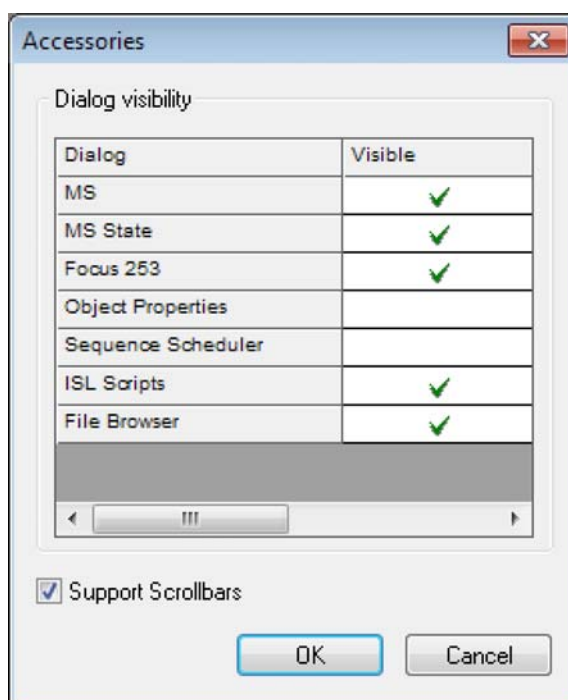
## Zero Enrichment (Standard On/Off Test)

The description is intended for users with 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.

The Method CO2\_zero.met for zero enrichment is 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 bellows is measured (against itself). The Method CO2\_zero right.met is used when only the right bellows is measured (against itself). The difference of the three Methods lies in the script.

### ❖ To perform zero enrichment

1. In the Isodat folder, double-click  to open the **Acquisition** tool.
2. Right-click one of the panel title bars.
3. In the context menu, click the **Administrate Panels**.  
The **Accessories** window opens, see [Figure 6-5](#).



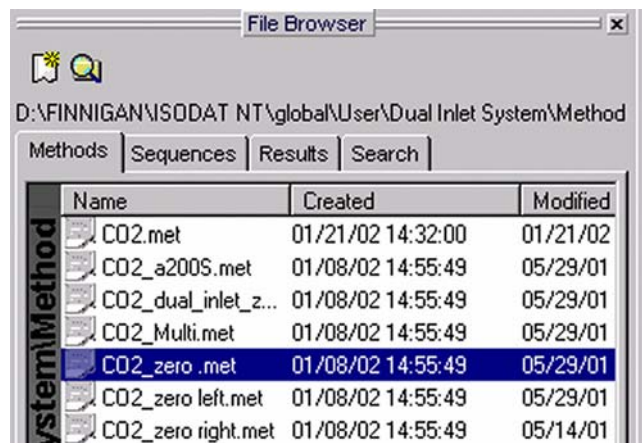
**Figure 6-5.** Activating dialogs in the Accessories window

The **Accessories** window allows to activate dialogs with ✓. When you select a dialog, the respective window is shown as a part of the **Accessories** toolbar.

## Measurements

### Zero Enrichment (Standard On/Off Test)

4. In the **Visible** column, click the field next to **File Browser**.
5. Click **OK**.
6. In the **File Browser**, click the **Methods** tab.
7. Double-click the predefined Method **CO2\_zero.met**. See [Figure 6-6](#).



**Figure 6-6.** Selecting the Method CO2\_zero.met

If the Standards in the Method to be opened are older than the current ones in the Standard Database or if the user has changed the Method, the warning shown in [Figure 6-7](#) appears.

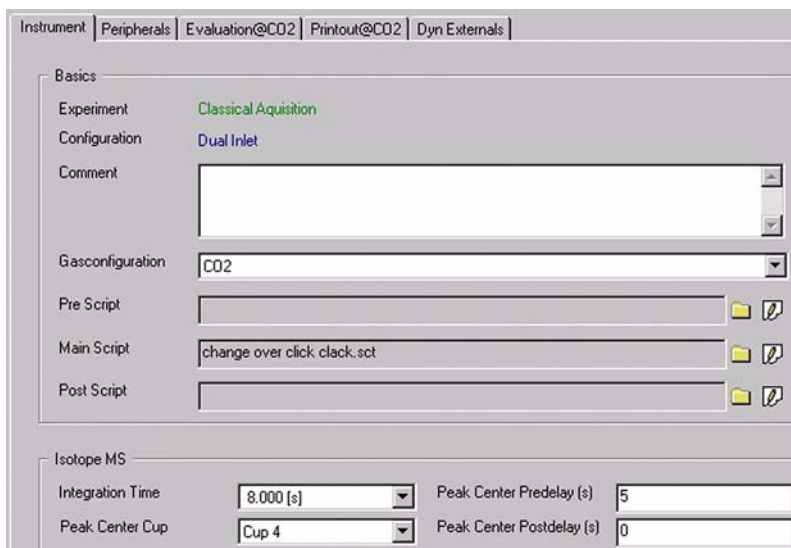


**Figure 6-7.** Updating the Standard values

8. Click **OK**.

The Method is corrected. The current values of the Standard Database – for example, the laboratory Standard has changed or has been replaced – are transferred to the Method.

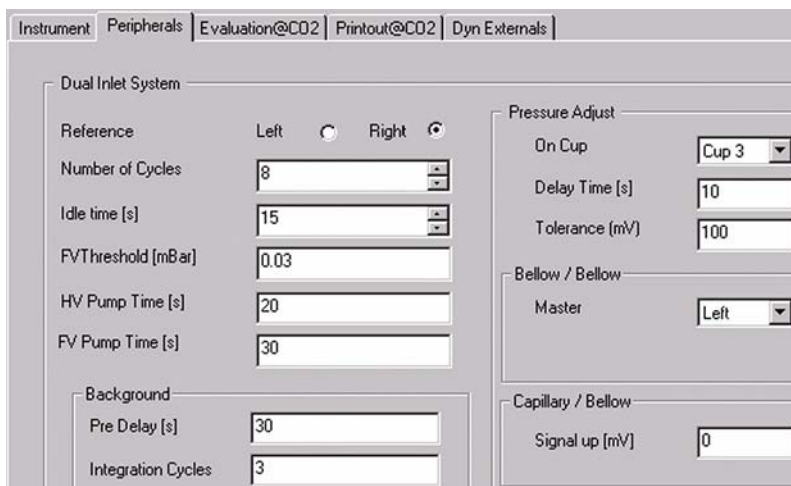
The Instrument page of the Method CO2\_zero.met appears. See [Figure 6-8](#).



**Figure 6-8.** Instrument page

For details, refer to [“Simple Dual Inlet Measurement”](#) on [page 6-11](#).

9. On the **Instrument** page, ensure that the proper **Peak Center Cup** is selected. If necessary, correct the default cup (that is Cup 4).
10. Select and enter all data for the **Peripherals** page. See [Figure 6-9](#). Refer to [“Simple Dual Inlet Measurement”](#) on [page 6-11](#) for details.



**Figure 6-9.** Peripherals page


## Measurements

### Zero Enrichment (Standard On/Off Test)

11. Select and enter all data for the **Evaluation@CO2** page. See [Figure 6-10](#).  
Refer to “Simple Dual Inlet Measurement” on page 6-11 for details.

Std. Name:	δ 13C/12C	δ 18O/16O
CO2_zero	0.000	0.000

**Figure 6-10.** Evaluation@CO2 page

12. Select **None** or **Sigma** as Outlier test.
13. In case of **Sigma**, click  to specify the k-fold of the standard deviation.
14. For **Std. Name** (see [Figure 6-10](#)), choose a suitable Standard.
15. Select and enter data for the **Printout** page. See [Figure 6-11](#).  
Refer to “Simple Dual Inlet Measurement” on page 6-11 for details.

Printout Templates
Single: CO2_Long.IRW
Sequence: CO2_Short.IRW

WK1 Export Templates
Single: [ ] >>
Sequence: [ ] >>

**Figure 6-11.** Printout@CO2 page





## Simple Dual Inlet Measurement

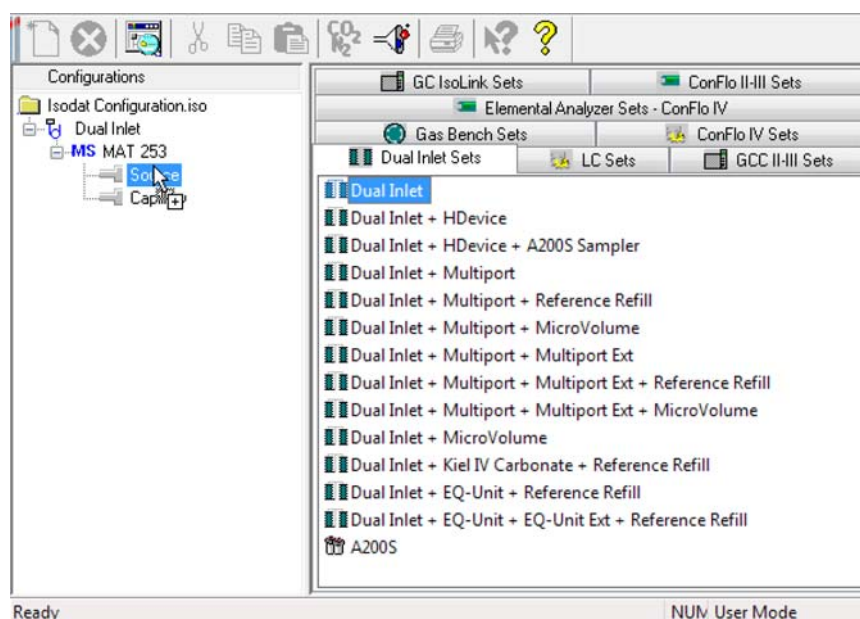
This chapter describes how to define a configuration for the Dual Inlet measurement, and how to select predefined Method and Sequence.

### Defining a Configuration for the Dual Inlet System

Before operating, a Configuration containing the simple Dual Inlet system must be created in the Configurator.

❖ **To define a Configuration for the Dual Inlet system**

1. In the Isodat folder, double-click  to open the **Configurator** tool.
2. Click  to add a new Configuration.
3. Enter a name, for example, **Dual Inlet**.
4. Select **Dual Inlet Sets**.  
The Dual Inlet sets are listed in the right pane, see [Figure 6-12](#).
5. Select **Dual Inlet**.
6. Drag and drop it to the **Source** port  of the new Configuration. A  sign appears, see [Figure 6-12](#).

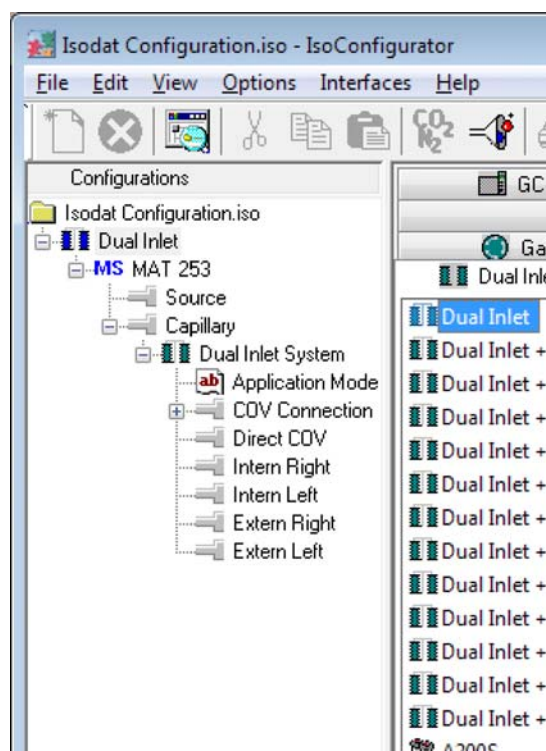


**Figure 6-12.** Defining a Configuration with Dual Inlet system

## Measurements

### Simple Dual Inlet Measurement

**Dual Inlet System** is attached to **Source**. See [Figure 6-13](#):



**Figure 6-13.** Dual Inlet device attached to the Source

7. Close the **Configurator** window.  
All settings are saved automatically.

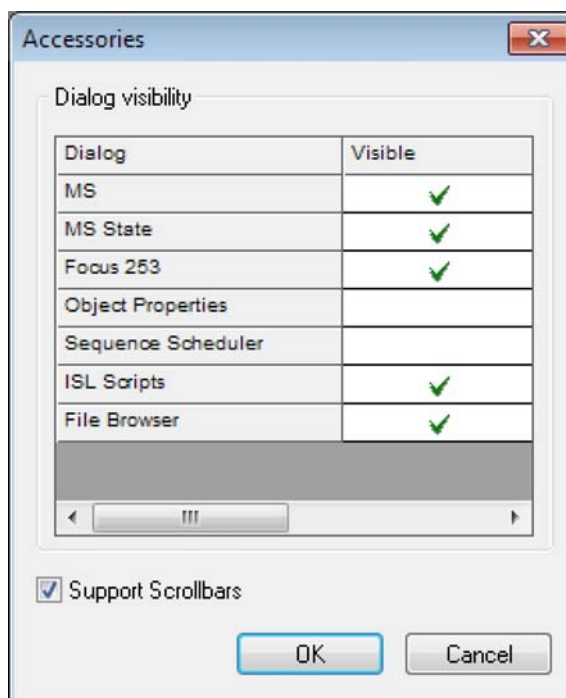
## Using a Predefined Method for the Dual Inlet System

- ❖ **To use a predefined method for the Dual Inlet system**

1. In the Isodat folder, double-click  to open the **Acquisition** tool.
2. Right-click one of the panel title bars.



3. In the context menu, click the **Administrate Panels**.  
The **Accessories** window opens, see [Figure 6-14](#).



**Figure 6-14.** Activating dialogs in the Accessories window

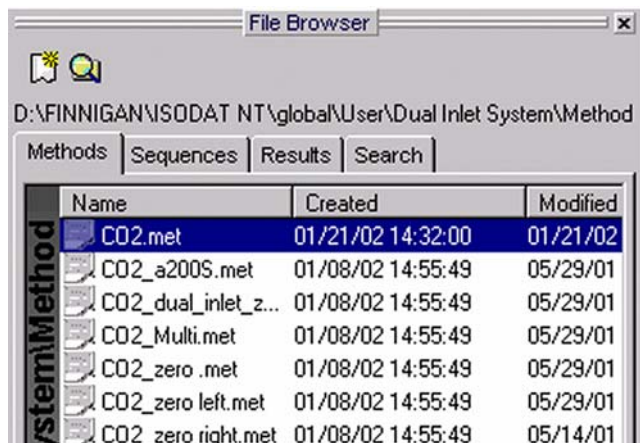
The **Accessories** window allows to activate dialogs with ✓. When you select a dialog, the respective window is shown as a part of the **Accessories** toolbar.

4. In the **Visible** column, click the field next to **File Browser**.
5. Click **OK**.
6. In the **File Browser**, click the **Methods** tab.

## Measurements

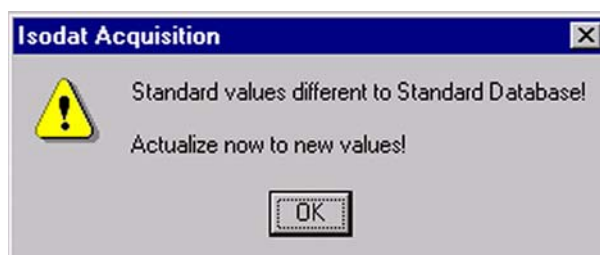
### Simple Dual Inlet Measurement

7. Double-click a predefined Method, for example **CO2.met**. See [Figure 6-15](#).



**Figure 6-15.** CO2.met

If the Standards in the Method to be opened are older than the current ones in the Standard Database or if the user has changed the Method, the warning shown in [Figure 6-16](#) appears.

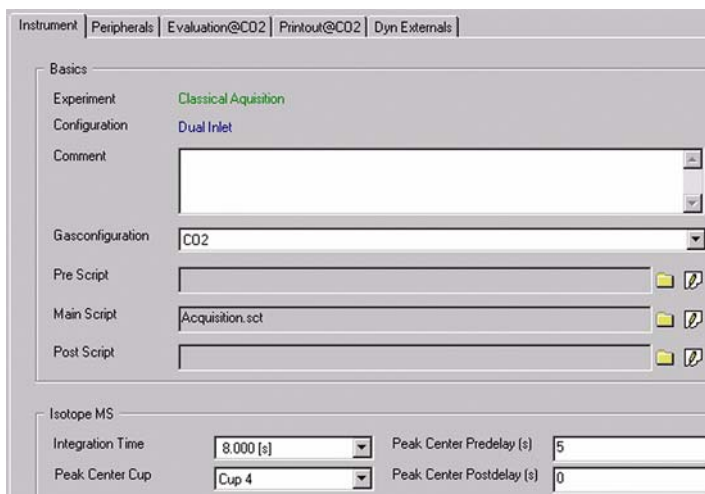


**Figure 6-16.** Actualizing the Standard values

8. Click **OK**.

The Method is corrected. The current values of the Standard Database – for example, the laboratory Standard has changed or has been replaced – are transferred to the Method.

- On the **Instrument** page, select the **Gasconfiguration** for your determination type (for example **CO2**). See [Figure 6-17](#).



**Figure 6-17.** Instrument page

**Note** The Main Script should be edited only by users trained on script editing. ▲

The Main Script controls the acquisition cycle.

- For **Isotope MS**, select the **Peak Center Cup**, for example, Cup 4 for a universal triple collector on a MAT 253 (narrow cup for  $m/z$  45).

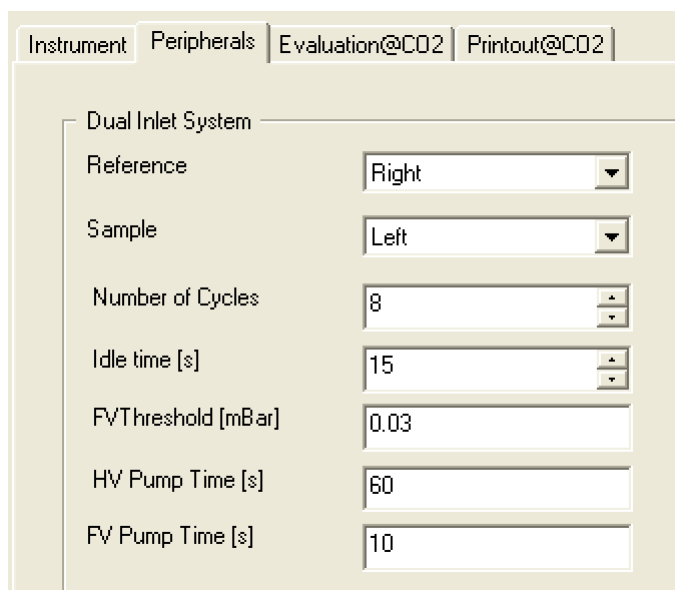
**Integration Time** is the time integrated to form a data point triplet (for example 8 s). It is the time needed to measure each individual ion intensity of the masses 44, 45, and 46. **Peak Center Predelay** is the waiting time between activation of reference gas and the start of the peak center cycle (for example, 5 s). **Peak Center Postdelay** is the waiting time between the end of the peak center cycle and the start of data acquisition (for example 0 s). See [Figure 6-18](#).



**Figure 6-18.** Isotope MS area

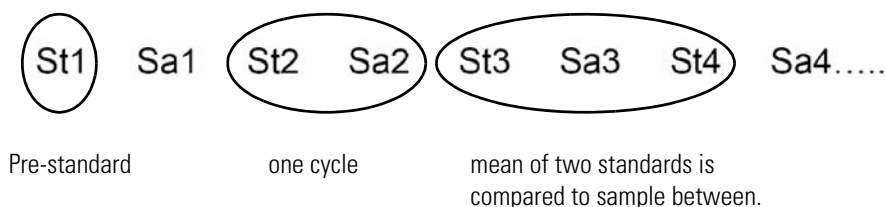
- On the **Peripherals** page, define the Dual Inlet system, see [Figure 6-19](#).  
**Reference** denotes which bellows contains the standard gas (default: right bellows). **Sample** denotes which bellows contains the sample. **Number of Cycles** indicates how often a sample is measured successively. For example 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.



**Figure 6-19.** Peripherals page

Thus, two standards flank every sample, one before it and one after it. See [Figure 6-20](#). The first standard is called pre-standard (that is St1). The mean of two consecutive standards is calculated and compared to the flanked sample, for example, mean of St1 and St2 is compared to Sa1.



**Figure 6-20.** Cycle

**Idle time [s]** in [Figure 6-19](#) 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 are integrated. The gas needs a certain time to flow away before the measurement can be started. The pressure must fall below the **FV Threshold** during fore vacuum pump activity (for example 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 [s]** is the time interval during which the High Vacuum pump is active after the FV threshold has been reached (for example 60 s). **FV Pump Time [s]** is the time interval during which the fore vacuum pump will be active after it has started the pumping process (for example 10 s).

12. Enter the **Background** values, see [Figure 6-21](#).

Background **Predelay [s]** 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.

Background	
Pre Delay [s]	120
Integration Cycles	1

**Figure 6-21.** Background area

13. Select and enter the **Pressure Adjust** values.

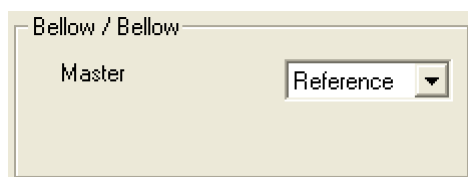
If standard pressure and sample pressure are varying considerably, a pressure adjustment is necessary. It ensures that standard and sample are measured nearly at the same intensity. See [Figure 6-22](#).

Pressure Adjust	
On Cup	Cup 2
Delay Time [s]	10
Tolerance [mV]	100

**Figure 6-22.** Pressure Adjust area

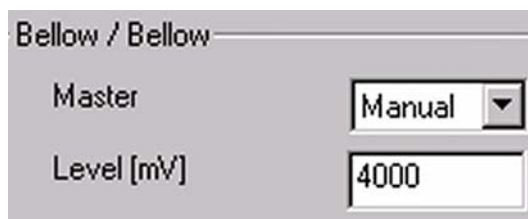
**On Cup** is the cup on which the pressure adjustment is performed. Normally, it is the cup where mass 44 is measured. **Delay time [s]** is the waiting time after changing from sample to standard gas (or vice versa) before matching the standard bellows to sample ion intensity. **Tolerance [mV]** denotes the precision of the pressure adjustment. 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 (for example 100 mV, this means  $\pm 50$  mV), the pressure adjustment is finished successfully.

14. Select the value for **Bellow/Bellow**. See [Figure 6-23](#).  
The standard bellows is adjusted to the level of sample ion intensity minus signal up  $\pm$  tolerance.



**Figure 6-23.** Bellow/Bellow area

Master is the bellows that predefines the pressure, whereas the other bellows has to follow. Possible values are **Sample**, **Reference** and **Manual**. If Manual is selected, the pressure level (intensity) that must be reached in both bellows can be entered, see [Figure 6-24](#).



**Figure 6-24.** Editing the pressure level in the bellows

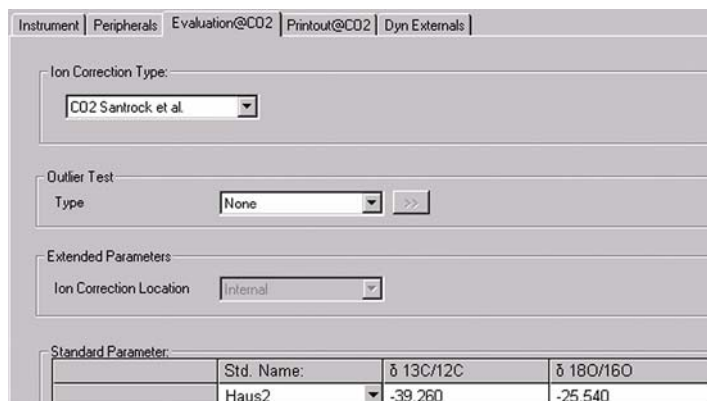
In most cases, the sample bellows is defined as master. Thus, the sample enters the system with a certain predefined pressure  $p$  to which the standard is compressed.

15. Enter the value for **Capillary/Bellow**. See [Figure 6-25](#).  
When closing valve 25, the mechanical movement causes the pressure to rise. Measure once and calculate the percentage, then enter the value here.



**Figure 6-25.** Capillary/Bellow

16. On the Evaluation page, select an **Ion Correction Type**, for example, **CO2 Santrock et al.**, see [Figure 6-26](#).




**Figure 6-26.** Evaluation page

17. Choose the **Type** of **Outlier Test**. (Rejection criterion for possible outliers: **None** or **Sigma**).
18. Select a standard name **Std. Name** or edit the related delta values. The standards are predefined in the standard table. These values are isotope ratios of carbon and oxygen of your standard gas.
19. Select a value for **Outliers Test** from the drop-down menu, see [Figure 6-27](#).  
If you select **None**, only mean and standard deviation of the obtained delta values will be calculated. Outliers are not identified. If you select **Sigma**, those delta values beyond  $k$  times of the standard deviation are identified and rejected.



**Figure 6-27.** Outlier Test: Sigma

If you selected **Sigma**, the  button on the right side becomes available.

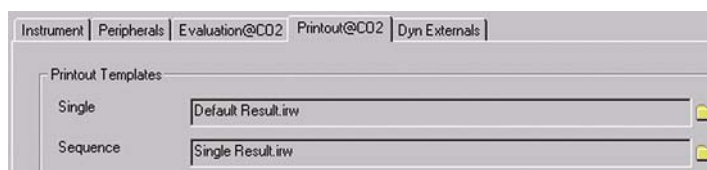
20. Click .

21. Select the k-fold standard deviation (default: k = 1). See [Figure 6-28](#).



**Figure 6-28.** k-fold standard deviation

22. Select the **Printout Templates** on the Printout@CO2 page.  
For **Single** you select a print template from the Result Workshop for an individual printout per sample.  
For **Sequence** you select a print template from the Result Workshop for a reduced print per sample within a sequence summary. See [Figure 6-29](#).



**Figure 6-29.** Printout Templates

23. As with the Printout Templates, select **WK1 Export Templates** for an Excel-Export of the data.  
For **Single** you select an Excel-Export Template from the Result Workshop for an individual printout per sample.  
For **Sequence** you select an Excel-Export Template from the Result Workshop for a reduced print per sample within a sequence summary. See [Figure 6-30](#).




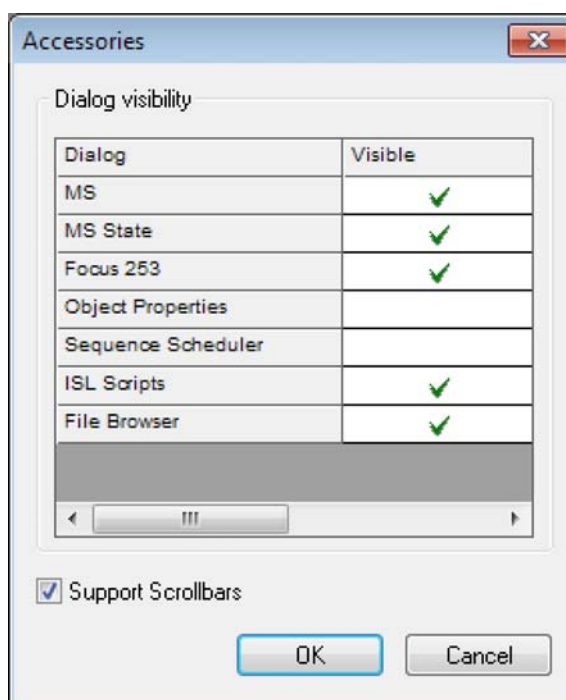
**Figure 6-30.** WK1 Export Templates

The Method for measurements using a simple Dual Inlet has now been established. As next step, either use a predefined Sequence (refer to [“Using a Predefined Sequence for the Dual Inlet System”](#) on page 6-21) or create a new Sequence (refer to [“Creating a New Sequence”](#) on page 6-3).

## Using a Predefined Sequence for the Dual Inlet System

❖ To use a predefined sequence for the Dual Inlet system

1. In the Isodat folder, double-click  to open the **Acquisition** tool.
2. Right-click one of the panel title bars.
3. In the context menu, click the **Administrate Panels**.  
The **Accessories** window opens, see [Figure 6-31](#).

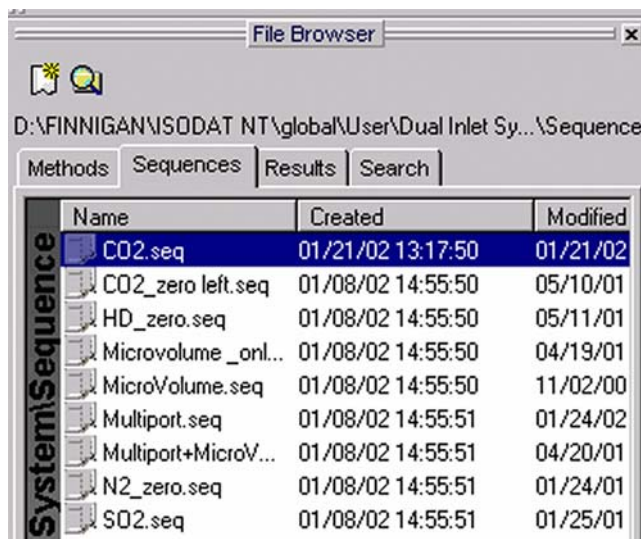


**Figure 6-31.** Activating dialogs in the Accessories window

The **Accessories** window allows to activate dialogs with ✓. When you select a dialog, the respective window is shown as a part of the **Accessories** toolbar.

4. In the **Visible** column, click the field next to **File Browser**.
5. Click **OK**.

- In the File Browser, click the **Sequences** tab (Methods tab is shown by default, see [Figure 6-32](#)).



**Figure 6-32.** Sequences page

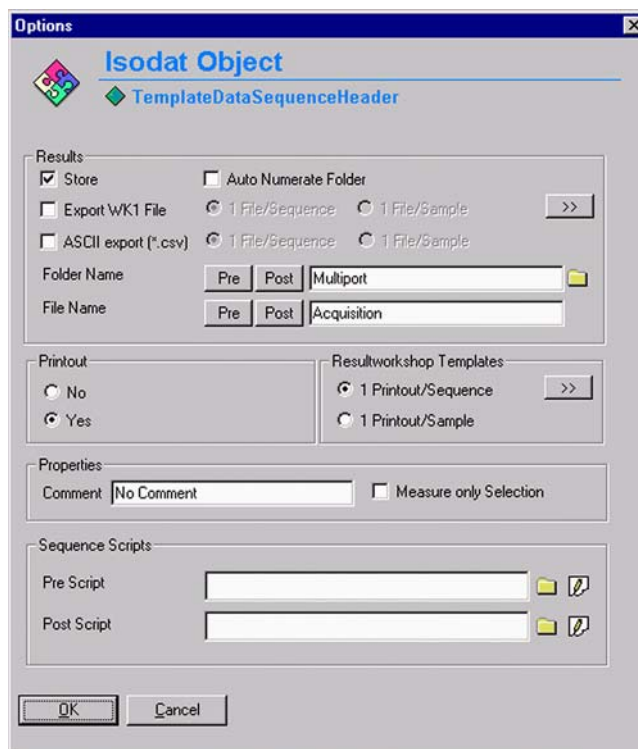
- Double-click a predefined Sequence, for example **CO2.seq**. The Sequence list opens, see [Figure 6-33](#).

Line			Identifier 1	Method
1	✓	✓		CO2.met
2	✓	✓		CO2.met
3	✓	✓		CO2.met

**Figure 6-33.** Method column

In the Method column, the Method chosen in “[Using a Predefined Method for the Dual Inlet System](#)” on [page 6-12](#) appears as default (CO2.met). It is possible, however, to select another Method from the Method column.

8. Click the  button.  
The **Options** dialog opens, see [Figure 6-34](#).



**Figure 6-34.** Options for sequence

9. Select parameters for **Results**.
10. Select parameters for **Printout**.
11. Select parameters for **Sequence Scripts**.
12. Click **OK** to start Sequence acquisition.





## Dual Inlet Measurement Including Multiport

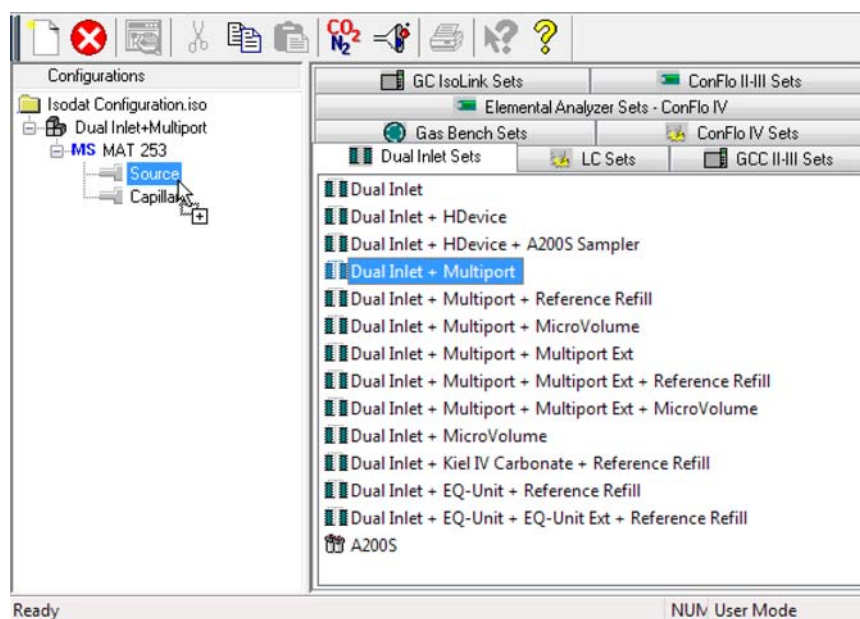
This chapter describes how to define a configuration for the Dual Inlet measurement with Multiport, and how to select predefined Method and Sequence.

### Defining a Configuration for Dual Inlet System and Multiport

Before operating, a Configuration containing the Dual Inlet system and the Multiport must be created in the Configurator.

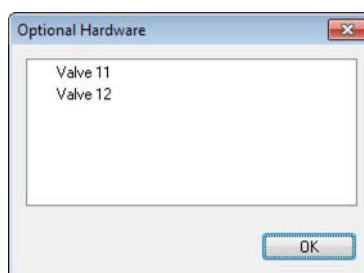
#### ❖ To define a Configuration for Dual Inlet system and Multiport

1. In the Isodat folder, double-click  to open the **Configurator** tool.
2. Click  to add a new Configuration.
3. Enter a name, for example, **Dual Inlet+Multiport**.
4. Select **Dual Inlet Sets**.  
The Dual Inlet sets are listed in the right pane, see [Figure 6-35](#).
5. Select **Dual Inlet + Multiport**.
6. Drag and drop it to the **Source** port  of the new Configuration. A  sign appears, see [Figure 6-35](#).



**Figure 6-35.** Defining a Configuration with Dual Inlet system and Multiport

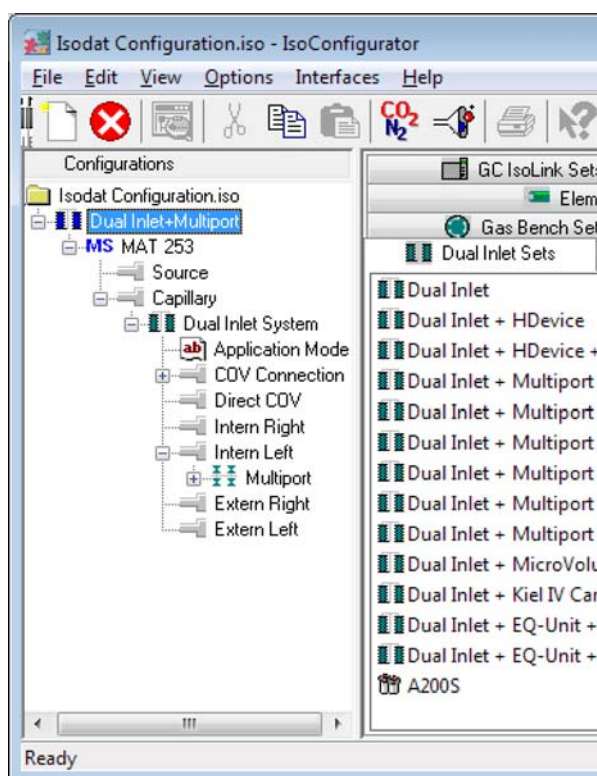
The **Optional Hardware** dialog opens, see [Figure 6-36](#):



**Figure 6-36.** Optional Hardware dialog

7. If you use a multiport with more than 10 ports, select **Valve 11** or **Valve 12**, as appropriate.  
If you do not select a valve in this window, the standard multiport with 10 port is configured.
8. Click **OK**.

**Dual Inlet System** is attached to **Source**. **Multiport** is attached to the **Intern Left** port. See [Figure 6-37](#):




**Figure 6-37.** Dual Inlet device attached to the Source

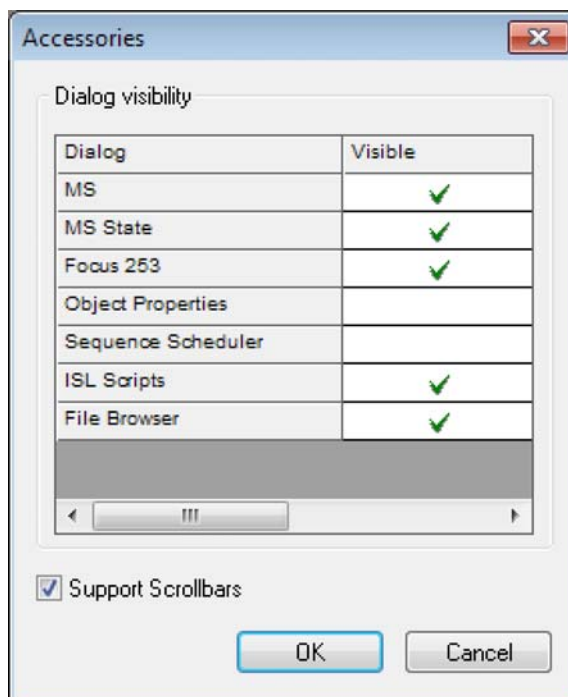
9. Close the **Configurator** window.  
All settings are saved automatically.

## Using a Predefined Method for Dual Inlet System and Multiport

- ❖ To use a predefined method for Dual Inlet system and Multiport



1. In the Isodat folder, double-click  to open the **Acquisition** tool.
2. Right-click one of the panel title bars.
3. In the context menu, click the **Administrate Panels**. The **Accessories** window opens, see [Figure 6-38](#).



**Figure 6-38.** Activating dialogs in the Accessories window

The **Accessories** window allows to activate dialogs with ✓. When you select a dialog, the respective window is shown as a part of the **Accessories** toolbar.

4. In the **Visible** column, click the field next to **File Browser**.
5. Click **OK**.
6. In the File Browser, click the **Methods** tab (default).

7. Double-click the predefined Method **CO2\_Multi.met**. See [Figure 6-39](#).



**Figure 6-39.** CO2\_Multi.met

If the Standards in the Method to be opened are older than the current ones in the Standard Database or if the user has changed the Method, the warning shown in [Figure 6-40](#) appears.



**Figure 6-40.** Actualizing the Standard values

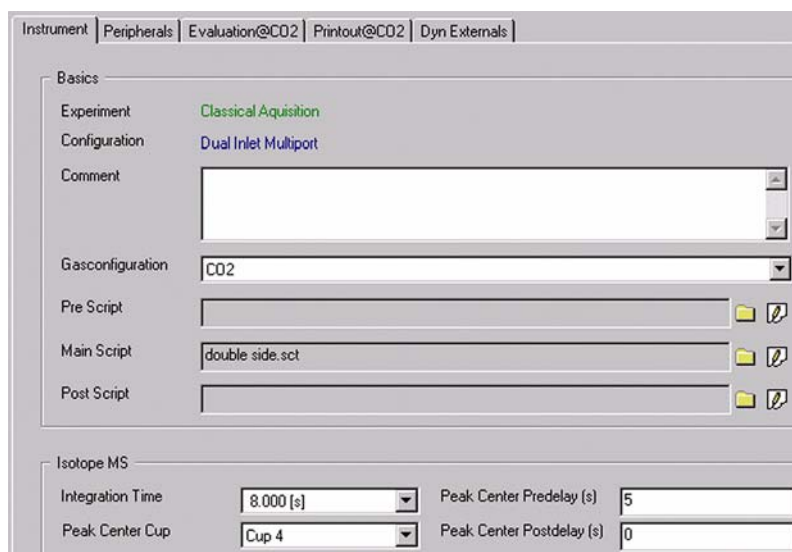
8. Click **OK**.

The Method is corrected. The current values of the Standard Database – for example, the laboratory Standard has changed or has been replaced – are transferred to the Method.

## Measurements

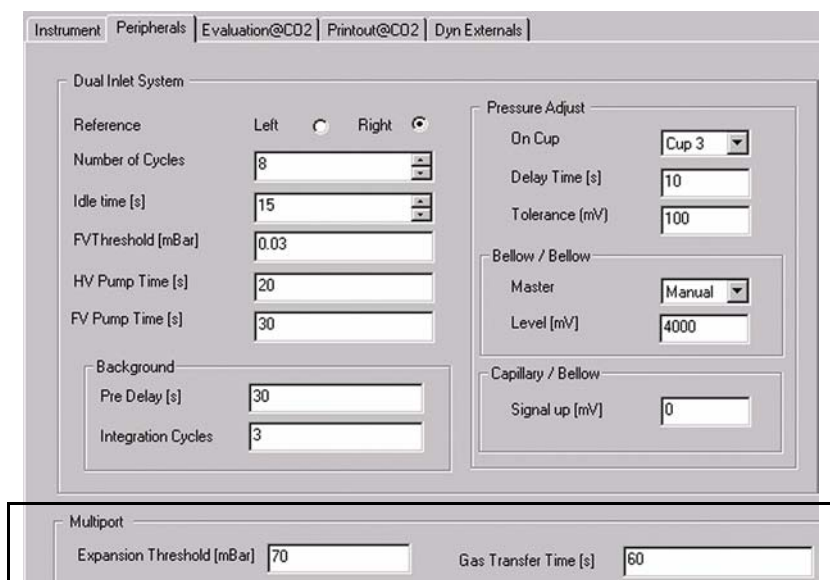
### Dual Inlet Measurement Including Multiport

The Instrument page of the Method CO2\_Multi.met opens, see [Figure 6-41](#). For details, refer to “Using a Predefined Method for the Dual Inlet System” on page 6-12.



**Figure 6-41.** Instrument page of CO2\_Multi.met

9. On the **Instrument** page, ensure that the proper **Peak Center Cup** is selected. If necessary, correct the default cup (that is Cup 4)!
10. Click the **Peripherals** tab, see [Figure 6-42](#).



**Figure 6-42.** Peripherals page

For measurements including Multiport two additional parameters show in the Peripherals page. **Gas Transfer Time** is the time the sample gas needs to pass from the Multiport to the sample bellows (for example 60 s). The valves “bank”, “12”, “14” and “15” are

opened.


When the sample has entered its bellows, the sample gas pressure within it must not exceed the **Expansion Threshold** at bellows 100% open (for example 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.

Refer to “Using a Predefined Method for the Dual Inlet System” on page 6-12 for details on the Peripherals page (see Figure 6-42).

11. Click the Evaluation@CO2 tab, see Figure 6-43.

Standard Parameter:			
	Std. Name:	$\delta$ 13C/12C	$\delta$ 18O/16O
	CO2_zero	0.000	0.000

**Figure 6-43.** Evaluation@CO2 page

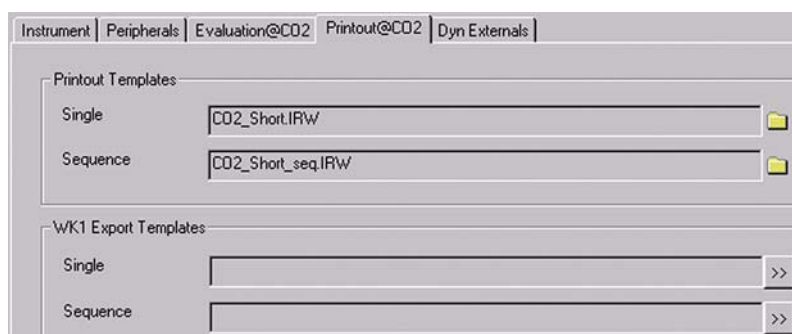
12. Select **None** or **Sigma** as **Outlier Test**.
13. In case of **Sigma**, click  to specify the k-fold of the standard deviation.
14. For **Std. Name**, choose a suitable standard.

For details on the Evaluation@CO2 page (see Figure 6-43), refer to “Using a Predefined Method for the Dual Inlet System” on page 6-12.

## Measurements

### Dual Inlet Measurement Including Multiport

15. Click the **Printout** tab, see [Figure 6-44](#).



**Figure 6-44.** Printout page

For details on the Printout page (see [Figure 6-44](#)), refer to “[Using a Predefined Method for the Dual Inlet System](#)” on page 6-12.

The Method for measurements using Dual Inlet in combination with a Multiport has now been established.

16. As next step, either use a predefined Sequence (refer to “[Using a Predefined Sequence for Dual Inlet System and Multiport](#)” on page 6-30) or create a new Sequence (refer to “[Creating a New Sequence](#)” on page 6-3).

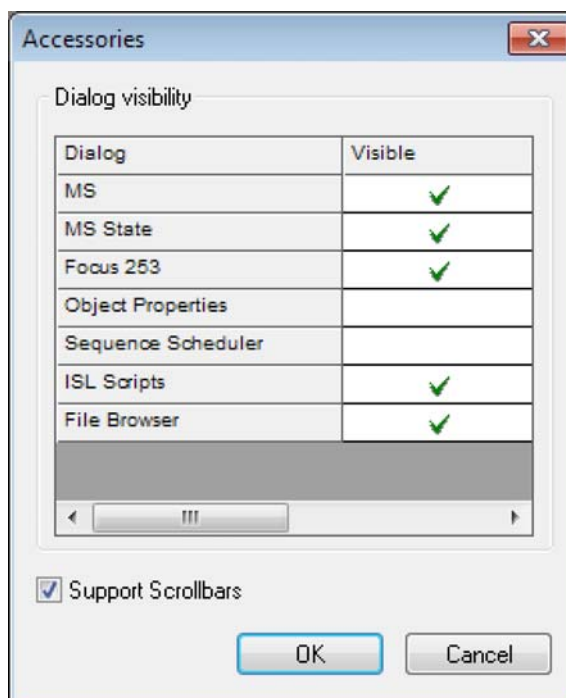
## Using a Predefined Sequence for Dual Inlet System and Multiport

- ❖ **To use a predefined sequence for Dual Inlet system and Multiport**

1. In the Isodat folder, double-click  to open the **Acquisition** tool.
2. Right-click one of the panel title bars.



- In the context menu, click the **Administrate Panels**.  
The **Accessories** window opens, see [Figure 6-46](#).

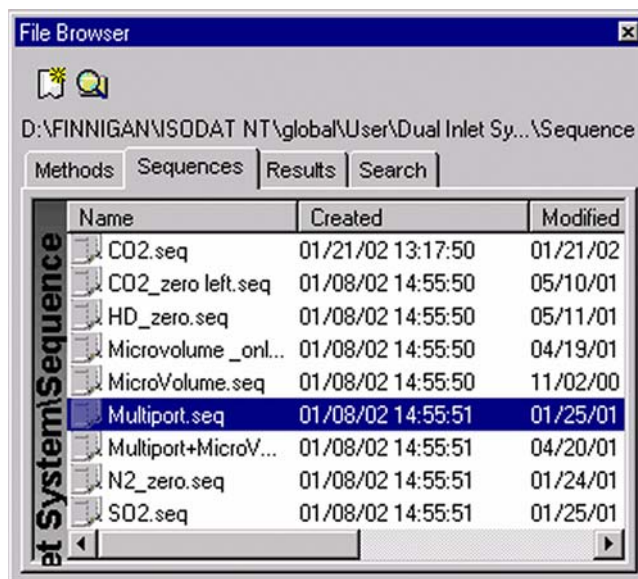


**Figure 6-45.** Activating dialogs in the Accessories window

The **Accessories** window allows to activate dialogs with ✓. When you select a dialog, the respective window is shown as a part of the **Accessories** toolbar.

- In the **Visible** column, click the field next to **File Browser**.
- Click **OK**.
- In the File Browser, click the **Sequences** tab (default: Methods tab).

7. Double-click the predefined Sequence **Multiport.seq**. See [Figure 6-46](#).



**Figure 6-46.** Sequences page: Multiport.seq

In the Method column, see [Figure 6-47](#), the Method chosen in “Using a Predefined Method for Dual Inlet System and Multiport” on [page 6-26](#) appears as default (CO2\_Multi.met). It is possible, however, to select another Method from the Method column.

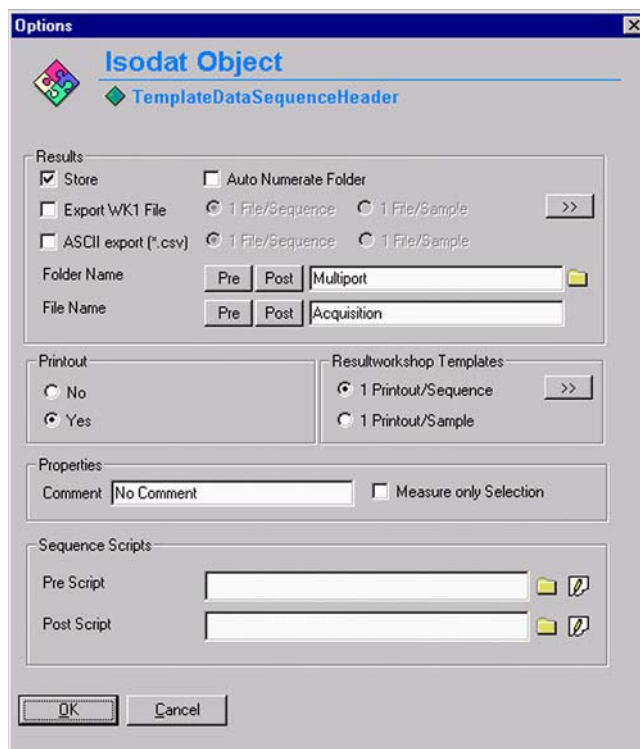
Line			Multiport Inlet	Identifier 1	Method
1	✓	✓	Sample 1		CO2_Multi.met
2	✓	✓	Sample 2		CO2_Multi.met
3	✓	✓	Sample 3		CO2_Multi.met
4	✓	✓	Sample 3		CO2_Multi.met
5	✓	✓	Sample 5		CO2_Multi.met
6	✓	✓	Sample 6		CO2_Multi.met
7	✓	✓	Sample 7		CO2_Multi.met
8	✓	✓	Sample 8		CO2_Multi.met
9	✓	✓	Sample 9		CO2_Multi.met
10	✓	✓	Sample 10		CO2_Multi.met

**Figure 6-47.** Sequence List

**Note** A new column, Multiport Inlet, is shown. It defines which Multiport inlet port will be connected to which sample (for example inlet port 8 to Sample 8). It is possible to measure a sample repeatedly (for example connect both Sample 3 and Sample 4 to the Multiport inlet port 3). ▲



- Click the **Start** button.  
The **Options** dialog opens, see [Figure 6-48](#).



**Figure 6-48.** Options for Sequence

- Select parameters for **Results**.
- Select parameters for **Printout**.
- Select parameters for **Sequence Scripts**.
- Click **OK** to start Sequence acquisition.

## Dual Inlet Measurement Including Microvolume

This chapter describes how to define a configuration for the Dual Inlet measurement with Microvolume, and how to select predefined Method and Sequence.

### Microvolume Without Multiport – Measurement Principle

The measurement principle of Microvolume without Multiport is shown in Figure 6-49:

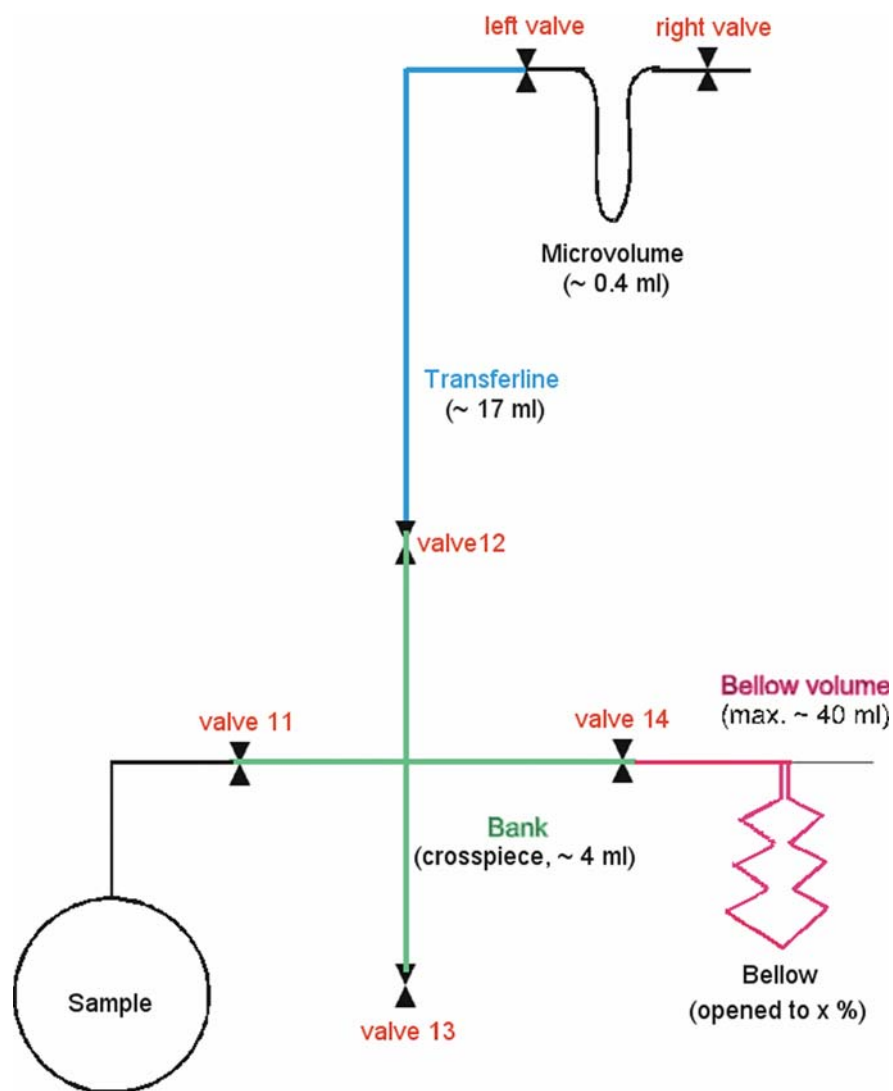
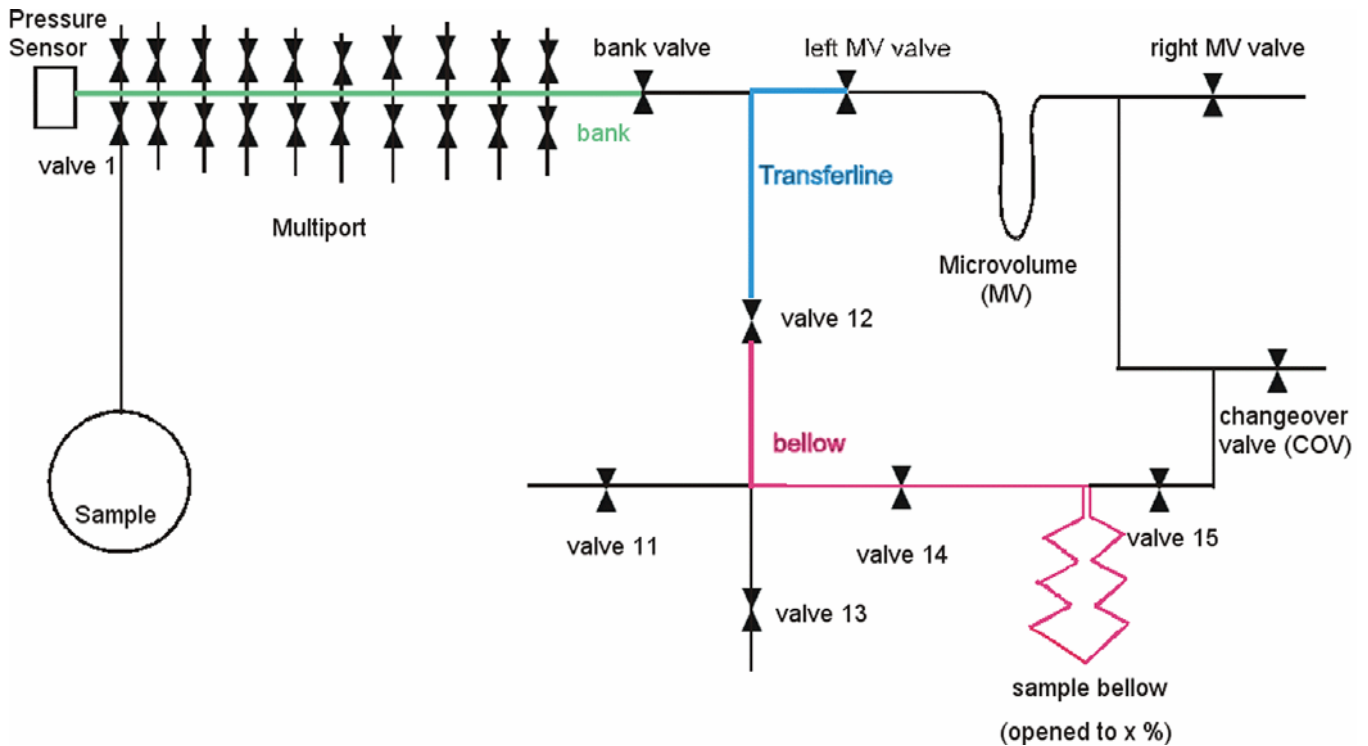


Figure 6-49. Microvolume without Multiport – measurement principle

## Microvolume via Multiport – Measurement Principle

The measurement principle of Microvolume via Multiport is shown in Figure 6-50:



**Figure 6-50.** Microvolume via Multiport – measurement principle

Two fundamental measuring modes Expansion mode and Freeze Direct mode, see Figure 6-51, exist for using a Microvolume (via Multiport):



**Figure 6-51.** Measuring modes for Microvolume

## Microvolume Expansion Mode

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, see Figure 6-52:



**Figure 6-52.** Options for Expansion Mode


## Measurements

### Dual Inlet Measurement Including Microvolume


With **Bank**, the gas is expanded into valves 11, 12, 13 and 14. The volume between valve 12 and the left valve of the Microvolume are used if **Bank and Transferline** is selected. With **Bank, Transferline and Bellows**, the gas is expanded to the bank, Transferline and Bellows. In this case, the value for **Bellow Pos. [%]** must be entered as well. The bellows is opened to the percent value entered for **Bellow Pos. [%]**.

## Expansion Mode - Before Measurement

### ❖ To prepare a measurement

1. In the Isodat folder, double-click  to open the **Acquisition** tool.
2. Connect the sample container with the sample to be measured to a port of the multiport unit, for example, **valve 1** in [Figure 6-50](#).
3. Pump the volume between **valve 1** in [Figure 6-50](#) and the sample container.
4. Open valve 1 manually and open all valves necessary to connect to the vacuum line (valve 1, bank valve, valve 12 and valve 13).
5. Close valve 1 manually.
6. Open the sample container.
7. Ensure that enough gas is available on the Standard side.
8. If Reference Refill is planned, click the appropriate row in the column **Reference Refill** to activate in the Sequence list of the **Acquisition** tool.



9. Click  to start the acquisition.

Measurement sequence:

- After starting the acquisition, the bellows of the Sample side (mostly left) is opened fully to 100%.
- A first evacuation step by means of the fore vacuum pump takes place over the vacuum line. 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.
- If FV Threshold has been reached, the fore vacuum pump continues to pump until FV Pump Time (for example, 1 s) has elapsed.

- The bank valve, vacuum line valve, left valve of Microvolume, right valve of Microvolume are opened as well as the valves 12, 13, 14, 15, 16 and 39.
- These opened valves are evacuated stepwise by means of the fore vacuum pump. First, only the bellows is evacuated. When FV Threshold has been reached in the bellows, 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.
- 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, for example 60 s. All valves are opened with the exception of valve 39. Valve 40 is set to “Toggle Switch”. the Isodat software opens valve 39 while closing valve 40 and vice versa.
- Bank valve, vacuum line 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.
- The sample valve of the Multiport (for example valve 1) is opened. The system waits until Sample Equilibration Time has elapsed, for example 60 s. The pressure  $p$  is measured via the sensor.
- If you selected only Expansion mode (that is, **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.
- The pressure  $p$  is determined using a pressure-meter (that is, sensor).

## Measurements

### Dual Inlet Measurement Including Microvolume

## Expansion Mode – Measurement With Bank

This chapter describes the measurement sequence for **Bank** (see [Figure 6-53](#)):

The screenshot shows a control panel for 'Micro Volume'. At the top, there are two radio buttons: 'Expansion' (selected) and 'Freeze Direct'. Below this is a section for 'Expansion Mode' with three radio buttons: 'Bank' (selected), 'Bank and Transferline', and 'Bank, Transferline and Bellow'. To the right of these buttons is a text input field containing '50' and a label 'Bellow Pos [%]'.

**Figure 6-53.** Expansion Mode – Measurement With Bank

- The pressure  $p$  is measured using a pressure-meter sensor.
- If  $p > \text{Freeze Bank Threshold}$ , for example,  $p > 6$  mbar, the pressure is too high. See [Figure 6-54](#).

The screenshot shows a settings panel with two columns of input fields. The first column contains: 'Sample Equilibration Time [s]' (60), 'Pump Out Temperature [°C]' (50), 'Initial Transfer Temperature [°C]' (-50), 'Freeze Temperature [°C]' (-175), and 'Measure Temperature [°C]' (50). The second column contains: 'Bellow DirectThreshold [mBar]' (20), 'Freeze Bank Threshold [mBar]' (6, highlighted with a black border), 'Freeze All Threshold [mBar]' (2), 'Freeze Time [s]' (60), and 'Delay Before Measure [s]' (5).

**Figure 6-54.** Freeze Bank Threshold

- To reduce  $p$ , the gas in the bank volume has to be pumped off. Therefore, the sample valve, for example valve 1, is closed (all valves for the other samples, which currently are out of interest, are closed anyway).
- The gas in the bank volume is pumped off.
- The sample valve, for example valve 1, is opened (of course, all other sample valves remain closed). The sample gas expands into the bank volume.

- The system waits until **Sample Equilibration Time** has elapsed, for example 60 s, see [Figure 6-55](#).

Sample Equilibration Time [s]	60	Bellow DirectThreshold [mBar]	20
Pump Out Temperature [°C]	50	Freeze Bank Threshold [mBar]	6
Initial Transfer Temperature [°C]	-50	Freeze All Threshold [mBar]	2
Freeze Temperature [°C]	-175	Freeze Time [s]	60
Measure Temperature [°C]	50	Delay Before Measure [s]	5

**Figure 6-55.** Sample Equilibration Time

- The pumping time depends on the three parameters **FV Threshold [mbar]**, **HV Pump Time [s]**, and **FV Pump Time [s]**, see [Figure 6-56](#).

Dual Inlet System	
Reference	Left <input type="radio"/> Right <input checked="" type="radio"/>
Number of Cycles	8
Idle time [s]	15
FVThreshold [mBar]	0.03
HV Pump Time [s]	60
FV Pump Time [s]	10

**Figure 6-56.** Parameters for pumping time

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

## Measurements

### Dual Inlet Measurement Including Microvolume

- The decreased pressure  $p$  is measured again and compared to the **Freeze Bank Threshold** (see [Figure 6-57](#)). If **Freeze All Threshold**  $< p <$  **Freeze Bank Threshold**, the sample valve, for example valve 1, is closed. The transfer process of the sample into the Microvolume starts and measurement begins.

Sample Equilibration Time [s]	60	Bellow DirectThreshold [mBar]	20
Pump Out Temperature [°C]	50	Freeze Bank Threshold [mBar]	6
Initial Transfer Temperature [°C]	-50	Freeze All Threshold [mBar]	2
Freeze Temperature [°C]	-175	Freeze Time [s]	60
Measure Temperature [°C]	50	Delay Before Measure [s]	5

**Figure 6-57.** Freeze Bank Threshold and Freeze All Threshold

- If  $p <$  **Freeze All Threshold**, only as much gas is available as can be transferred immediately and measured as a whole. The sample valve, for example valve 1, will not be closed any longer.

### Expansion Mode – Measurement With Bank and Transferline

The measurement sequence for **Bank and Transferline** (see [Figure 6-58](#)) is the same as described in “[Expansion Mode – Measurement With Bank](#)” on [page 6-38](#). Only the route of the sample gas is longer or Bank and Transferline.

Micro Volume	
<input checked="" type="radio"/> Expansion	<input type="radio"/> Freeze Direct
Expansion Mode	
<input type="radio"/> Bank	<input checked="" type="radio"/> Bank and Transferline
<input type="radio"/> Bank, Transferline and Bellow	<input type="text" value="50"/> Bellow Pos [%]

**Figure 6-58.** Expansion Mode – Measurement With Bank And Transferline

### Example

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

## Expansion Mode – Measurement With Bank, Transferline, and Bellows

The expansion mode **Bank, Transferline and Bellows** (see [Figure 6-59](#)) is used if large amounts of sample gas are available.

Micro Volume			
<input checked="" type="radio"/> Expansion <input type="radio"/> Freeze Direct			
Expansion Mode			
<input type="radio"/> Bank <input type="radio"/> Bank and Transferline <input checked="" type="radio"/> Bank, Transferline and Bellow <input type="text" value="50"/> Bellow Pos [%]			
Sample Equilibration Time [s]	<input type="text" value="60"/>	Bellow DirectThreshold [mBar]	<input type="text" value="20"/>
Pump Out Temperature [°C]	<input type="text" value="50"/>	Freeze Bank Threshold [mBar]	<input type="text" value="6"/>
Initial Transfer Temperature [°C]	<input type="text" value="-50"/>	Freeze All Threshold [mBar]	<input type="text" value="2"/>
Freeze Temperature [°C]	<input type="text" value="-175"/>	Freeze Time [s]	<input type="text" value="60"/>
Measure Temperature [°C]	<input type="text" value="50"/>	Delay Before Measure [s]	<input type="text" value="5"/>

**Figure 6-59.** Expansion Mode – Measurement with Bank, Transferline, and Bellows

Measurement sequence:

- The bellows is opened to the specified percentage, for example 50%.
- The sample valve (for example valve 1) is opened.
- Bank valve, valve 12 and valve 14 are opened.
- The sample gas is transferred up to the bellows. The whole volume is filled with gas.

## Measurements

### Dual Inlet Measurement Including Microvolume

- The pressure  $p$  is measured (see Figure 6-60). If  $p > \mathbf{Bellow\ Direct\ Threshold}$ , the sample is measured via the bellows.

The screenshot shows the 'Micro Volume' control panel. At the top, there are radio buttons for 'Expansion' (selected) and 'Freeze Direct'. Below this is the 'Expansion Mode' section with radio buttons for 'Bank', 'Bank and Transferline', and 'Bank, Transferline and Bellow' (selected), along with a 'Bellow Pos [%]' input field set to 50. The main area contains two columns of input fields for various parameters:

Sample Equilibration Time [s]	60	Bellow DirectThreshold [mBar]	20
Pump Out Temperature [°C]	50	Freeze Bank Threshold [mBar]	6
Initial Transfer Temperature [°C]	-50	Freeze All Threshold [mBar]	2
Freeze Temperature [°C]	-175	Freeze Time [s]	60
Measure Temperature [°C]	50	Delay Before Measure [s]	5

**Figure 6-60.** Bellow Direct Threshold, Freeze Bank Threshold and Freeze All Threshold

- If  $\mathbf{Freeze\ Bank\ Threshold} < p < \mathbf{Bellow\ Direct\ Threshold}$ , the sample valve (for example valve 1) is closed. The whole gas is pumped out. If nevertheless  $\mathbf{Freeze\ Bank\ Threshold} < p < \mathbf{Bellow\ Direct\ Threshold}$  is still valid, the process will be repeated.
- If  $\mathbf{Freeze\ All\ Threshold} < p < \mathbf{Freeze\ Bank\ Threshold}$ , Bank valve and sample valve (for example valve 1) are closed. After pumping off part of the gas, only the gas in the bank volume will be frozen.
- If  $p < \mathbf{Freeze\ All\ Threshold}$ , only little gas is available. Therefore, the whole gas amount will be frozen.

## Microvolume Freeze Direct Mode

The **Freeze Direct** mode (see [Figure 6-61](#)) is used if the amount of sample gas to be analyzed is known, and in case of very small gas samples.

The screenshot shows the 'Micro Volume' control panel. At the top, there are two radio buttons: 'Expansion' (unselected) and 'Freeze Direct' (selected). Below this is the 'Expansion Mode' section, which is inactive. It contains three radio buttons: 'Bank' (unselected), 'Bank and Transferline' (unselected), and 'Bank, Transferline and Below' (selected). To the right of these is a text box containing '50' and a label 'Bellow Pos [%]'. The main configuration area consists of two columns of input fields:

Sample Equilibration Time [s]	60	Bellow DirectThreshold [mBar]	20
Pump Out Temperature [°C]	50	Freeze Bank Threshold [mBar]	6
Initial Transfer Temperature [°C]	-50	Freeze All Threshold [mBar]	2
Freeze Temperature [°C]	-175	Freeze Time [s]	60
Measure Temperature [°C]	50	Delay Before Measure [s]	5

**Figure 6-61.** Freeze Direct mode

The sample gas is not expanded but transferred to the Microvolume directly. There, it is frozen according to the parameter values and finally be measured.

**Note** The fields for **Expansion Mode** are inactive when **Freeze Direct** is selected. ▲

Measurement sequence:

- The sample valve (for example valve 1) is opened.
- The gas in the bank volume is equilibrated until Sample Equilibration Time has elapsed.
- Simultaneously, the Microvolume is heated to Pump Out Temperature (for example 50 °C). At this slightly elevated temperature, evaporation of impurities and water is facilitated. They are pumped off. Thus, the Microvolume is cleansed.
- The Microvolume is cooled down until Initial Transfer Temperature (for example - 50 °C) is reached.
- At Initial Transfer Temperature (for example -50 °C), bank valve and left valve of Microvolume are opened. Thus, the sample gas is transferred to the Microvolume.
- The sample gas having just entered the Microvolume is cooled down further until Freeze Temperature (for example -175 °C) is reached.

## Measurements



### Dual Inlet Measurement Including Microvolume

- The system waits, until Freeze Time has elapsed (for example 60 s).
- Then, bank valve and left valve of Microvolume are closed.
- The Microvolume is heated up to Measure Temperature (for example 50 °C).
- The system waits until Delay Before Measure has elapsed.
- Measurement starts.

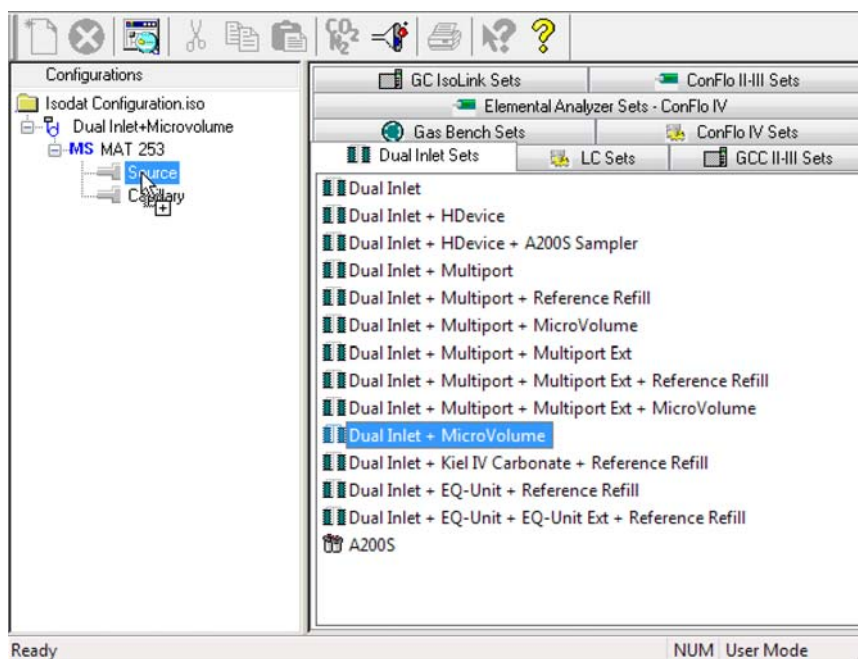
## Defining a Configuration for Dual Inlet System and Microvolume

Before operating, a configuration containing the Dual Inlet system and the Microvolume must be created in the Configurator.

### ❖ To define a Configuration for Dual Inlet system and Microvolume

1. In the Isodat folder, double-click  to open the **Configurator** tool.
2. Click  to add a new Configuration.
3. Enter a name, for example, **Dual Inlet+Microvolume**.
4. Select **Dual Inlet Sets**.  
The Dual Inlet sets are listed in the right pane, see [Figure 6-62](#).
5. Select **Dual Inlet + Microvolume**.

6. Drag and drop it to the **Source** port  of the new Configuration. A  sign appears, see [Figure 6-62](#).

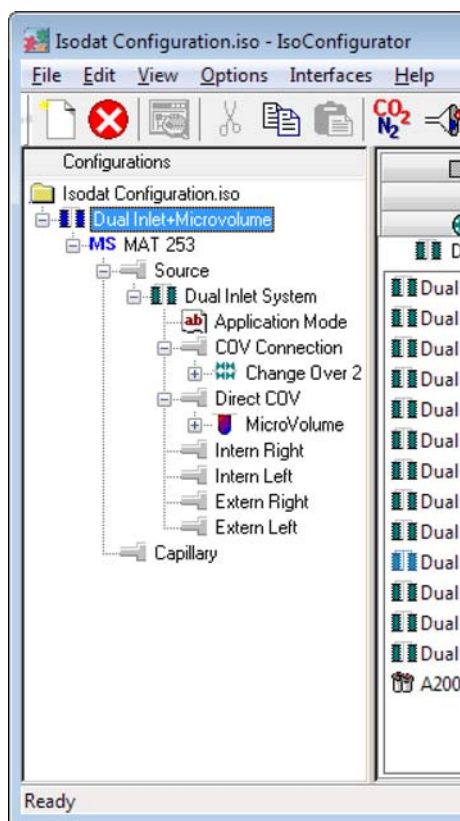


**Figure 6-62.** Defining a Configuration with Dual Inlet system and Microvolume

## Measurements

### Dual Inlet Measurement Including Microvolume

**Dual Inlet System** is attached to **Source**. **Microvolume** is attached to the **Direct COV Port**. See [Figure 6-63](#):




**Figure 6-63.** Dual Inlet device attached to the Source

7. Close the **Configurator** window.  
All settings are saved automatically.

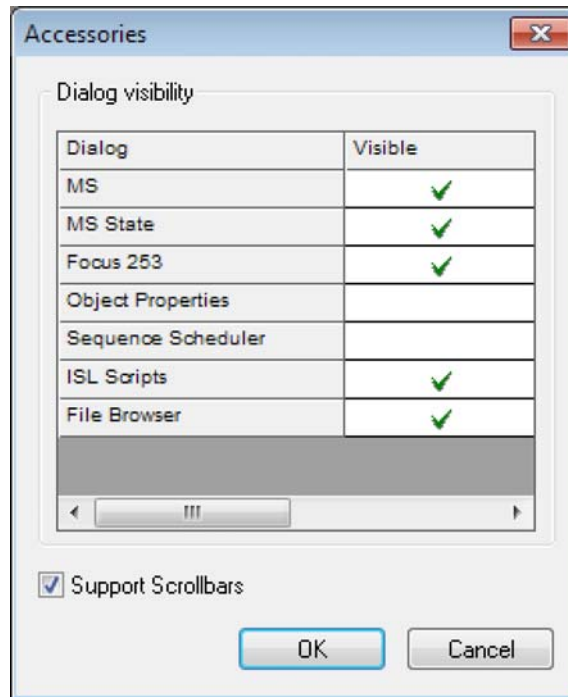
## Using a Predefined Method for Dual Inlet System and Microvolume

- ❖ **To use a predefined method for Dual Inlet system and Microvolume**

1. In the Isodat folder, double-click  to open the **Acquisition** tool.
2. Right-click one of the panel title bars.



3. In the context menu, click the **Administrate Panels**.  
The **Accessories** window opens, see [Figure 6-64](#).



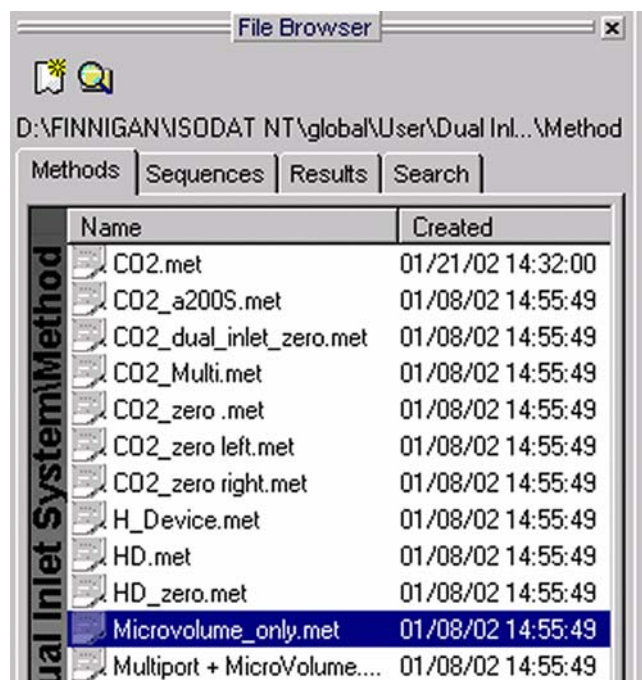
**Figure 6-64.** Activating dialogs in the Accessories window

- The **Accessories** window allows to activate dialogs with ✓. When you select a dialog, the respective window is shown as a part of the **Accessories** toolbar.
4. In the **Visible** column, click the field next to **File Browser**.
  5. Click **OK**.
  6. In the File Browser, click the **Methods** tab (default).

## Measurements

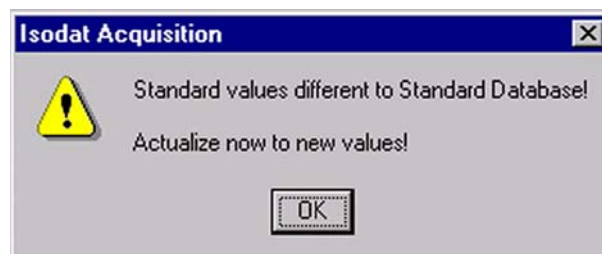
### Dual Inlet Measurement Including Microvolume

7. Double-click the predefined Method **Microvolume\_only.met**. See [Figure 6-65](#).



**Figure 6-65.** Microvolume\_only.met

If the Standards in the Method to be opened are older than the current ones in the Standard Database or if the user has changed the Method, the warning shown in [Figure 6-66](#) appears.

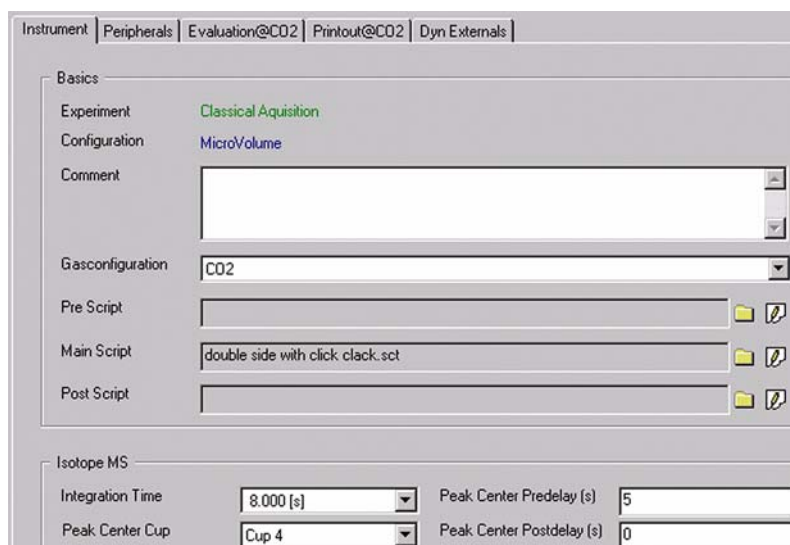


**Figure 6-66.** Actualizing the Standard values

8. Click **OK**.

The Method is corrected. The current values of the Standard Database – for example, the laboratory Standard has changed or has been replaced – are transferred to the Method.

The Instrument page of the Method Microvolume\_only.met opens. See [Figure 6-67](#).



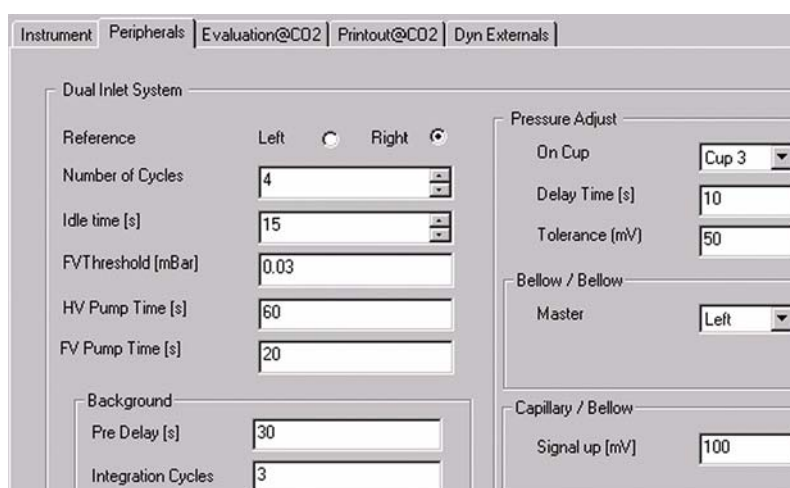
**Figure 6-67.** Instrument page of the Method Microvolume\_only.met

9. On the **Instrument** page, ensure that the proper **Peak Center Cup** is selected. If necessary, correct the default cup (that is Cup 4)!

For details on the Instrument page (see [Figure 6-67](#)), refer to “Using a Predefined Method for the Dual Inlet System” on page 6-12.

10. Click the **Peripherals** tab.

For details on Dual Inlet in the Peripherals page (see [Figure 6-68](#)), refer to “Using a Predefined Method for the Dual Inlet System” on page 6-12.



**Figure 6-68.** Peripherals page: Dual Inlet part

## Measurements

### Dual Inlet Measurement Including Microvolume

For details on Microvolume in the Peripherals page (see [Figure 6-69](#)), refer to “Using a Predefined Method for the Dual Inlet System” on page 6-12.

The screenshot shows the 'Micro Volume' configuration window. It includes radio buttons for 'Expansion' (selected) and 'Freeze Direct'. Under 'Expansion Mode', there are radio buttons for 'Bank', 'Bank and Transferline', and 'Bank, Transferline and Bellow' (selected), along with a 'Bellow Pos [%]' field set to 50. Below these are several input fields for parameters: Sample Equilibration Time [s] (30), Pump Out Temperature [°C] (50), Initial Transfer Temperature [°C] (-50), Freeze Temperature [°C] (-175), Measure Temperature [°C] (28), Bellow Direct Threshold [mBar] (8), Freeze Bank Threshold [mBar] (4), Freeze All Threshold [mBar] (0.5), Freeze Time [s] (60), and Delay Before Measure [s] (5).

**Figure 6-69.** Peripherals page: Microvolume part

11. Click the **Evaluation@CO2** tab, see [Figure 6-70](#).

The screenshot shows the 'Evaluation@CO2' configuration page. It has tabs for 'Instrument', 'Peripherals', 'Evaluation@CO2' (selected), 'Printout@CO2', and 'Dyn Externals'. The 'Ion Correction Type' dropdown is set to 'CO2 Santrock et al.'. The 'Outlier Test' section has a 'Type' dropdown set to 'None' and a '>>' button. The 'Extended Parameters' section has an 'Ion Correction Location' dropdown set to 'Internal'. The 'Standard Parameter' table is shown below:

	Std. Name:	δ 13C/12C	δ 18O/16O
	Haus2	-39.260	-25.540

**Figure 6-70.** Evaluation@CO2 page

12. On the **Evaluation@CO2** page, select an **Ion Correction Type** (for example **CO2 Santrock et al.**).

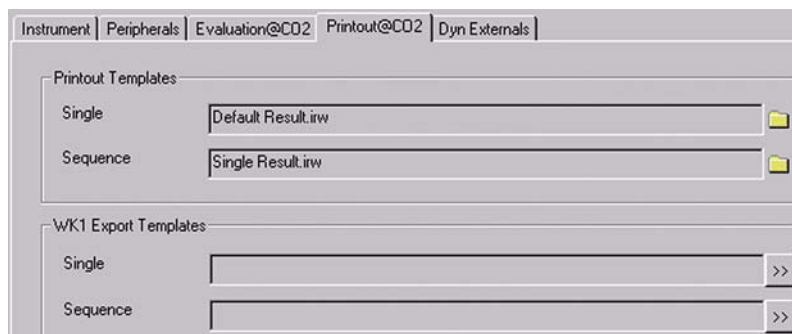
13. Select **None** or **Sigma** as **Outlier Test**.

14. In case of **Sigma**, click  to specify the k-fold of the standard deviation.

For details, refer to “Using a Predefined Method for the Dual Inlet System” on page 6-12.

15. For **Std. Name**, choose a suitable Standard.

For details on the Printout page (see [Figure 6-71](#)), refer to “[Using a Predefined Method for the Dual Inlet System](#)” on [page 6-12](#).



**Figure 6-71.** Printout page

The Method for measurements using Dual Inlet in combination with a Microvolume has now been established.

16. As next step, either use a predefined Sequence (refer to “[Using a Predefined Sequence for Dual Inlet System and Microvolume](#)” on [page 6-51](#)) or create a new Sequence (refer to “[Creating a New Sequence](#)” on [page 6-3](#)).

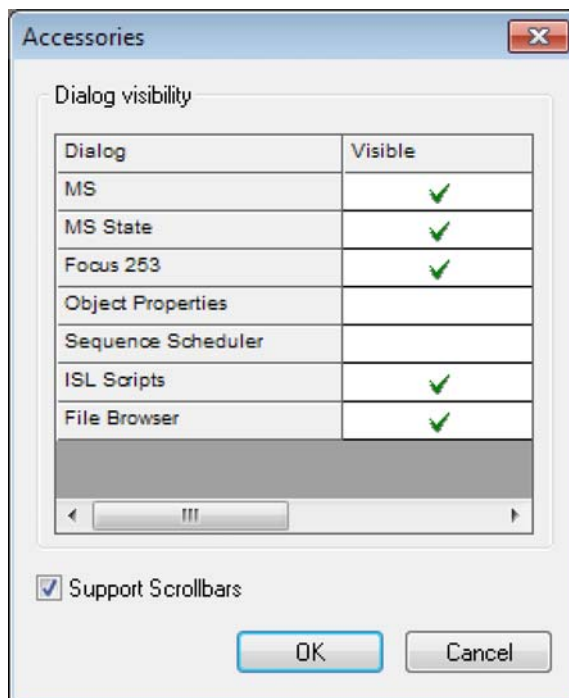
## Using a Predefined Sequence for Dual Inlet System and Microvolume

- ❖ **To use a predefined sequence for Dual Inlet system and Microvolume**

1. In the Isodat folder, double-click  to open the **Acquisition** tool.
2. Right-click one of the panel title bars.



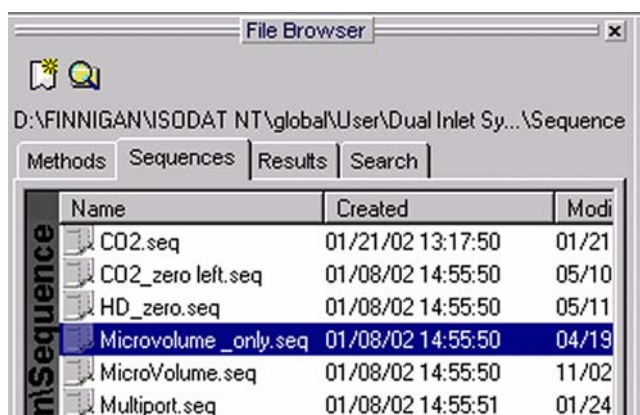
- In the context menu, click the **Administrate Panels**. The **Accessories** window opens, see [Figure 6-72](#).



**Figure 6-72.** Activating dialogs in the Accessories window

The **Accessories** window allows to activate dialogs with ✓. When you select a dialog, the respective window is shown as a part of the **Accessories** toolbar.

- In the **Visible** column, click the field next to **File Browser**.
- Click **OK**.
- In the File Browser, click the **Sequences** tab (default: Methods tab).
- Double-click the predefined Sequence **Microvolume\_only.seq**. See [Figure 6-73](#).




**Figure 6-73.** Sequences page: Microvolume\_only.seq

In the Method column, see [Figure 6-74](#), the Method chosen in “Using a Predefined Method for Dual Inlet System and Microvolume” on page 6-46 appears as default (Microvolume\_only.met). It is possible, however, to select another Method from the Method column.

Line				Identifier 1	Method
1					Microvolume_only.met

**Figure 6-74.** Sequence List



8. Click the  button.  
The **Options** dialog opens, see [Figure 6-75](#).

**Figure 6-75.** Options for sequence

9. Select parameters for **Results**.
10. Select parameters for **Printout**.
11. Select parameters for **Sequence Scripts**.
12. Click **OK** to start Sequence acquisition.



## Dual Inlet Measurement Including Multiport and Microvolume

This chapter describes how to define a configuration for the Dual Inlet measurement with Multiport and Microvolume, and how to select predefined Method and Sequence.

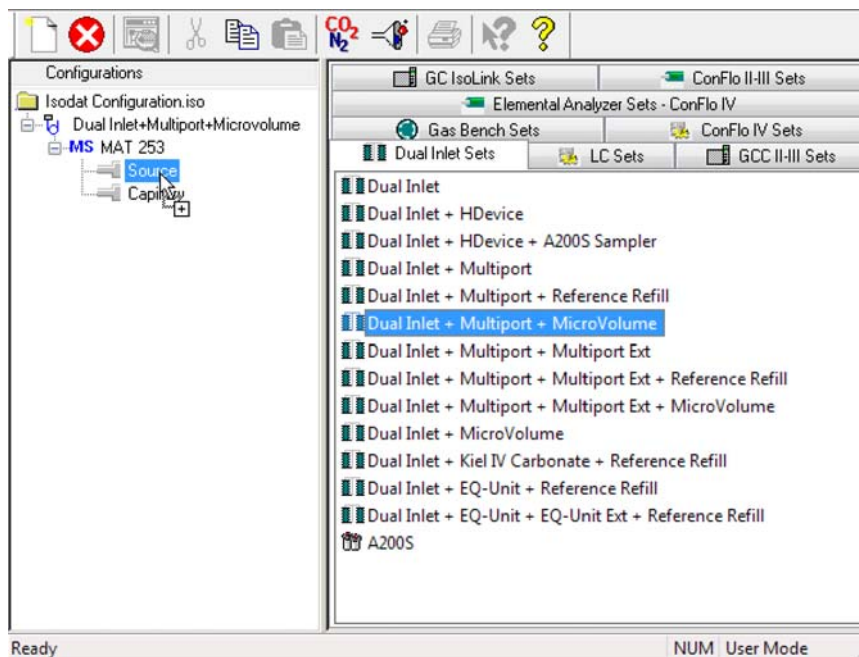
### Defining a Configuration for Dual Inlet System with Multiport and Microvolume

Before operating, a configuration containing the Dual Inlet system, Multiport and Microvolume must be created in the Configurator.

❖ **To define a Configuration for Dual Inlet system with Multiport and Microvolume**

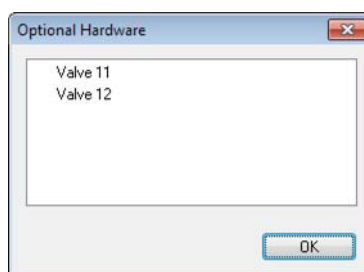
1. In the Isodat folder, double-click  to open the **Configurator** tool.
2. Click  to add a new Configuration.
3. Enter a name, for example, **Dual Inlet+Multiport+Microvolume**.
4. Select **Dual Inlet Sets**.  
The Dual Inlet sets are listed in the right pane, see [Figure 6-76](#).
5. Select **Dual Inlet + Multiport + Microvolume**.

6. Drag and drop it to the **Source** port  of the new Configuration. A  sign appears, see [Figure 6-76](#).



**Figure 6-76.** Defining a Configuration with Dual Inlet system with Multiport and Microvolume

The **Optional Hardware** dialog opens, see [Figure 6-77](#):



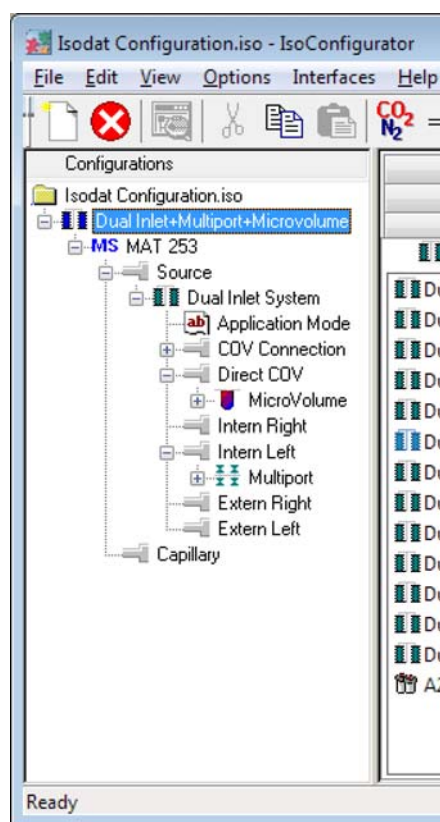
**Figure 6-77.** Optional Hardware dialog

7. If you use a multiport with more than 10 ports, select **Valve 11** or **Valve 12**, as appropriate.  
If you do not select a valve in this window, the standard multiport with 10 port is configured.
8. Click **OK**.

## Measurements

### Dual Inlet Measurement Including Multiport and Microvolume

**Dual Inlet System** is attached to **Source**. **Multiport** is attached to the **Intern Left** port. **Microvolume** is attached to the **Direct COV** Port. See [Figure 6-78](#):



**Figure 6-78.** Dual Inlet device attached to the Source

9. Close the **Configurator** window.  
All settings are saved automatically.

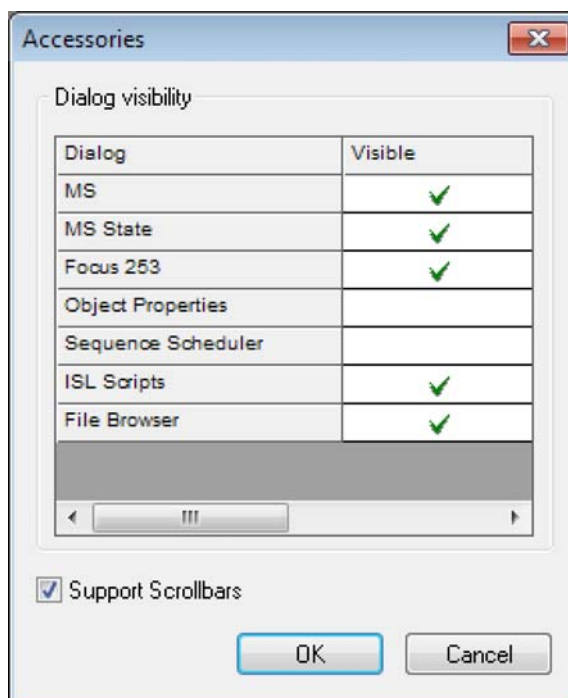
## Using a Predefined Method for Dual Inlet with Multiport and Microvolume

- ❖ **To use a predefined method for Dual Inlet with Multiport and Microvolume**

1. In the Isodat folder, double-click  to open the **Acquisition** tool.
2. Right-click one of the panel title bars.



- In the context menu, click the **Administrate Panels**.  
The **Accessories** window opens, see [Figure 6-79](#).



**Figure 6-79.** Activating dialogs in the Accessories window

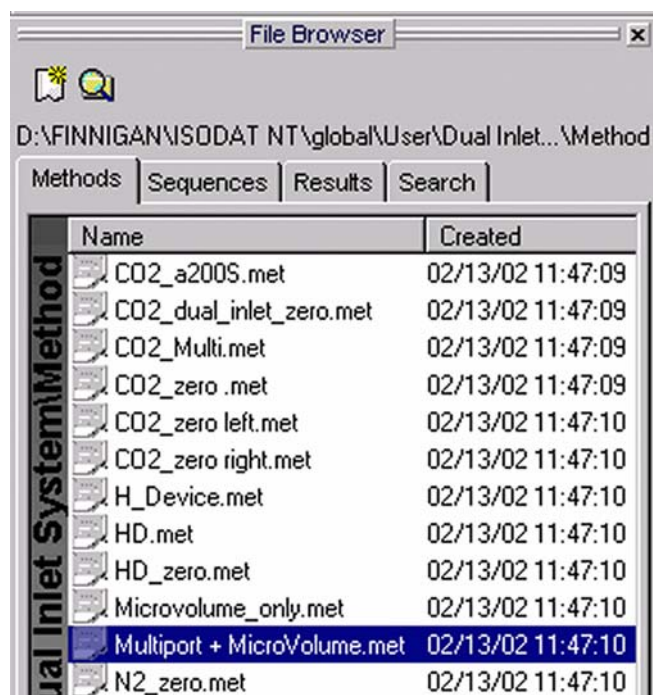
The **Accessories** window allows to activate dialogs with ✓. When you select a dialog, the respective window is shown as a part of the **Accessories** toolbar.

- In the **Visible** column, click the field next to **File Browser**.
- Click **OK**.
- In the File Browser, click the **Methods** tab (default).

## Measurements

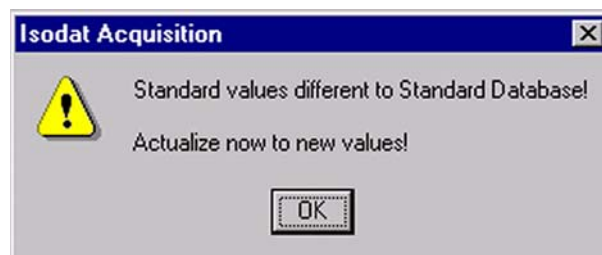
### Dual Inlet Measurement Including Multiport and Microvolume

7. Double-click the predefined Method **Multiport + MicroVolume.met**. See [Figure 6-80](#).



**Figure 6-80.** Multiport + MicroVolume.met

If the Standards in the Method to be opened are older than the current ones in the Standard Database or if the user has changed the Method, the warning shown in [Figure 6-81](#) appears.



**Figure 6-81.** Actualizing the Standard values

8. Click **OK**.

The Method is corrected. The current values of the Standard Database – for example, the laboratory Standard has changed or has been replaced – are transferred to the Method.

The Instrument page of the Method Multiport + MicroVolume.met opens, see [Figure 6-82](#).

Instrument | Peripherals | Evaluation@CO2 | Printout@CO2 | Dyn Externals

Experiment: Classical Acquisition  
 Configuration: Dual Inlet Multiport Microvolume  
 Comment:   
 Gas configuration: CO2  
 Acquisition Script: Acquisition.sct

Isotope MS  
 Integration Time: 8.000 [s] | Peak Center Predelay (s): 5  
 Peak Center Cup: Cup 3 | Peak Center Postdelay (s): 0

**Figure 6-82.** Instrument page of Multiport + MicroVolume.met

For details on Instrument page, refer to “[Using a Predefined Method for the Dual Inlet System](#)” on [page 6-12](#).

9. On the **Instrument** page, ensure that the proper **Peak Center Cup** is selected. If necessary, correct the default cup (that is Cup 4)!
10. Click the **Peripherals** tab.

For details on Dual Inlet in the Peripherals page (see [Figure 6-83](#)), refer to “[Using a Predefined Method for the Dual Inlet System](#)” on [page 6-12](#).

Instrument | Peripherals | Evaluation@CO2 | Printout@CO2 | Dyn Externals

Dual Inlet System  
 Reference: Left  Right   
 Number of Cycles: 4  
 Idle time [s]: 15  
 FV Threshold [mBar]: 0.03  
 HV Pump Time [s]: 60  
 FV Pump Time [s]: 20

Pressure Adjust  
 On Cup: Cup 3  
 Delay Time [s]: 10  
 Tolerance [mV]: 50

Bellow / Bellow  
 Master: Left

Background  
 Pre Delay [s]: 30  
 Integration Cycles: 3

Capillary / Bellow  
 Signal up [mV]: 100

**Figure 6-83.** Peripherals page

## Measurements

### Dual Inlet Measurement Including Multiport and Microvolume

For details on Microvolume in the Peripherals page (see [Figure 6-84](#)), refer to “Using a Predefined Method for Dual Inlet System and Microvolume” on page 6-46.

The screenshot shows the 'Micro Volume' configuration window. At the top, there are radio buttons for 'Expansion' (selected) and 'Freeze Direct'. Below this is the 'Expansion Mode' section with radio buttons for 'Bank', 'Bank and Transferline', and 'Bank, Transferline and Bellow' (selected). A text box next to it contains the value '50' and is labeled 'Bellow Pos [%]'. The main area contains several input fields for various parameters:

Sample Equilibration Time [s]	30	Bellow DirectThreshold [mBar]	8
Pump Out Temperature [°C]	50	Freeze Bank Threshold [mBar]	4
Initial Transfer Temperature [°C]	-50	Freeze All Threshold [mBar]	0.5
Freeze Temperature [°C]	-175	Freeze Time [s]	60
Measure Temperature [°C]	28	Delay Before Measure [s]	5

**Figure 6-84.** Peripherals page – Microvolume part

For details on Multiport in the Peripherals page (see [Figure 6-85](#)), refer to “Using a Predefined Method for Dual Inlet System and Multiport” on page 6-26.

The screenshot shows the 'Multiport' configuration window. It contains two input fields: 'Expansion Threshold [mBar]' with the value '70' and 'Gas Transfer Time [s]' with the value '60'.

**Figure 6-85.** Peripherals page – Multiport part

11. On the **Evaluation@CO2** page, select an **Ion Correction Type**, for example **CO2 Santrock et al.**, see [Figure 6-86](#).

The screenshot shows the 'Evaluation@CO2' configuration page. It has several sections:

- Ion Correction Type:** A dropdown menu with 'CO2 Santrock et al.' selected.
- Outlier Test:** A dropdown menu with 'None' selected and a '>>' button.
- Extended Parameters:** A dropdown menu with 'Internal' selected.
- Standard Parameter:** A table with the following data:

	Std. Name:	δ 13C/12C	δ 18O/16O
	Haus2	-39.260	-25.540

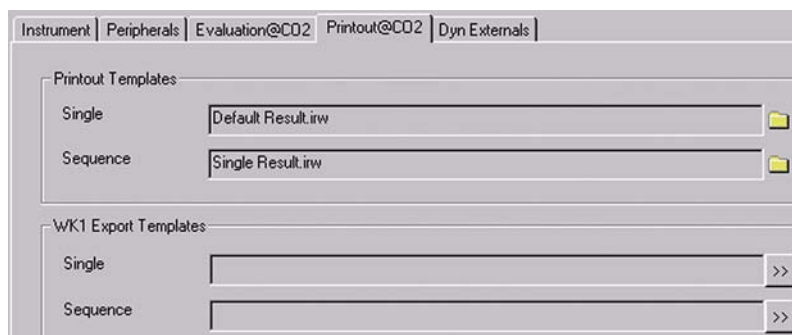
**Figure 6-86.** Evaluation@CO2 page

12. Select **None** or **Sigma** as **Outlier Test**.

13. In case of **Sigma**, click  to specify the k-fold of the standard deviation.

For details, refer to “[Using a Predefined Method for the Dual Inlet System](#)” on page 6-12.

14. For **Std. Name**, choose a suitable standard.
15. Click the **Printout@CO2** tab, see [Figure 6-87](#).



**Figure 6-87.** Printout page@CO2

For details on the Printout@CO2 page, refer to “[Using a Predefined Method for the Dual Inlet System](#)” on page 6-12.

The Method for measurements using Dual Inlet in combination with a Multiport and a Microvolume has now been established.

16. As next step, either use a predefined Sequence (refer to “[Using a Predefined Sequence for Dual Inlet System with Multiport and Microvolume](#)” on page 6-61) or create a new Sequence (refer to “[Creating a New Sequence](#)” on page 6-3).

## Using a Predefined Sequence for Dual Inlet System with Multiport and Microvolume

- ❖ **To use a predefined sequence for Dual Inlet with Multiport and Microvolume**

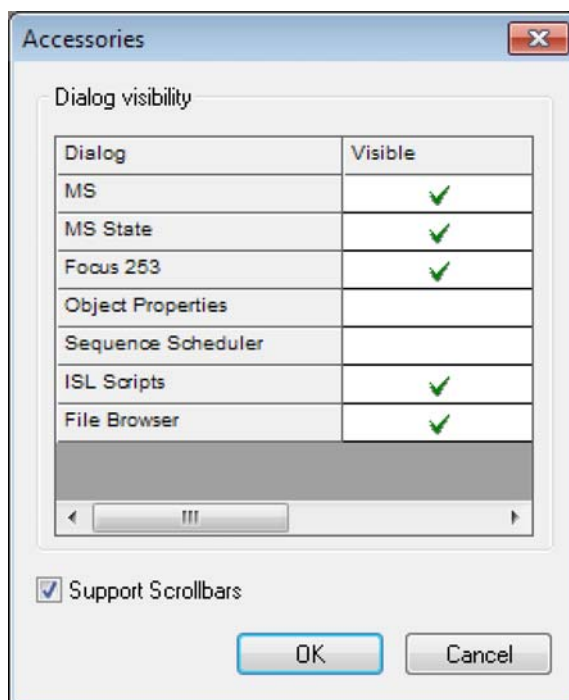
1. In the Isodat folder, double-click  to open the **Acquisition** tool.
2. Right-click one of the panel title bars.



## Measurements

### Dual Inlet Measurement Including Multiport and Microvolume

3. In the context menu, click the **Administrate Panels**. The **Accessories** window opens, see [Figure 6-88](#).

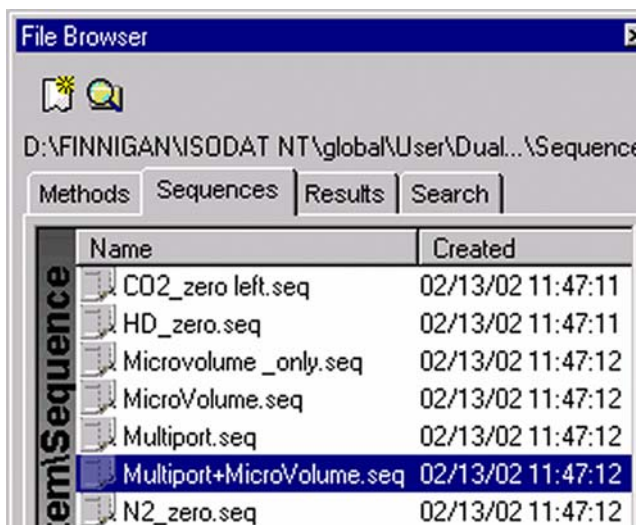


**Figure 6-88.** Activating dialogs in the Accessories window

The **Accessories** window allows to activate dialogs with ✓. When you select a dialog, the respective window is shown as a part of the **Accessories** toolbar.

4. In the **Visible** column, click the field next to **File Browser**.
5. Click **OK**.
6. In the File Browser, click the **Sequences** tab (default: Methods tab).

- Double-click the predefined Sequence **Multiport+Microvolume.seq**. See [Figure 6-89](#).



**Figure 6-89.** Sequences page: Multiport + Microvolume.seq

In the Method column, see [Figure 6-90](#), the Method chosen in “Using a Predefined Method for Dual Inlet with Multiport and Microvolume” on [page 6-56](#) appears as default (Multiport + MicroVolume.met). It is possible, however, for example to select another Method from the Method column or to modify the Multiport Inlet column.

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

**Figure 6-90.** Sequence List

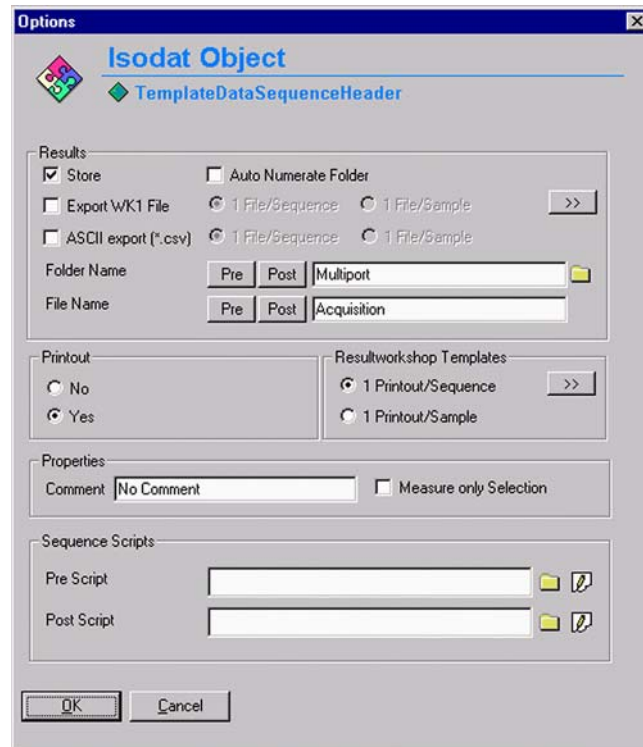
For details, refer to “Using a Predefined Sequence for the Dual Inlet System” on [page 6-21](#).

## Measurements

### Dual Inlet Measurement Including Multiport and Microvolume



- Click the **Start** button.  
The **Options** dialog opens, see [Figure 6-91](#).



**Figure 6-91.** Options for sequence

- Select parameters for **Results**.
- Select parameters for **Printout**.
- Select parameters for **Sequence Scripts**.
- Click **OK** to start Sequence acquisition.



## Dual Inlet Measurement Including H-Device and Autosampler A200S

This chapter describes how to define a configuration for the Dual Inlet measurement with H-Device and autosampler A200S, and how to select predefined Method and Sequence.

### Defining a Configuration for Dual Inlet System, H-Device, and Autosampler



Before operating, a configuration containing the Dual Inlet system, H-Device, and Autosampler must be created in the Configurator.

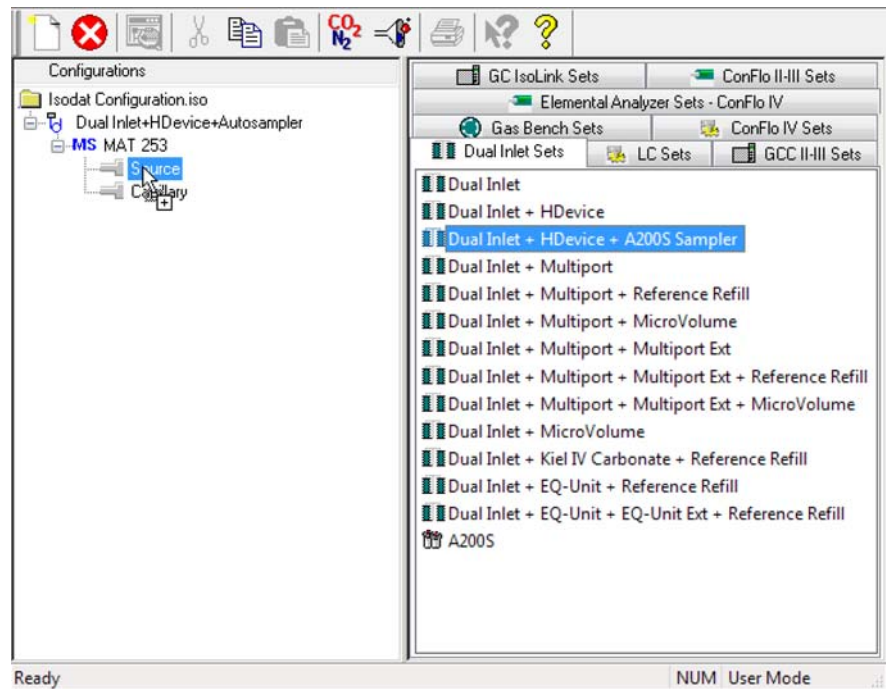
#### ❖ To define a Configuration for Dual Inlet system, H-Device, and Autosampler

1. In the Isodat folder, double-click  to open the **Configurator** tool.
2. Click  to add a new Configuration.
3. Enter a name, for example, **Dual Inlet+HDevice+Autosampler**.
4. Select **Dual Inlet Sets**.  
The Dual Inlet sets are listed in the right pane, see [Figure 6-92](#).
5. Select **Dual Inlet + HDevice + Autosampler**.

## Measurements

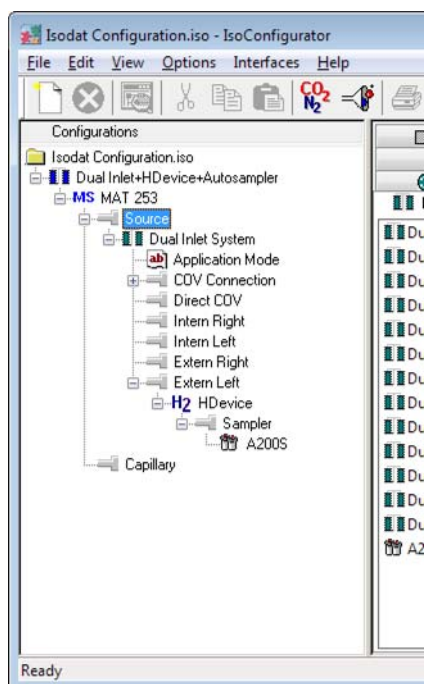
### Dual Inlet Measurement Including H-Device and Autosampler A200S

6. Drag and drop it to the **Source** port  of the new Configuration. A  sign appears, see [Figure 6-92](#).



**Figure 6-92.** Defining a Configuration with Dual Inlet system and HDevice and Autosampler

**Dual Inlet System** is attached to **Source**. **H-Device** is attached to the **Extern Left** port. **A200S** is attached to the **Sampler** port. See [Figure 6-93](#):




**Figure 6-93.** Dual Inlet device attached to the Source

7. Close the **Configurator** window.  
All settings are saved automatically.

## Using a Predefined Method for Dual Inlet System, H-Device, and Autosampler

- ❖ To use a predefined method for Dual Inlet system, H-Device, and Autosampler

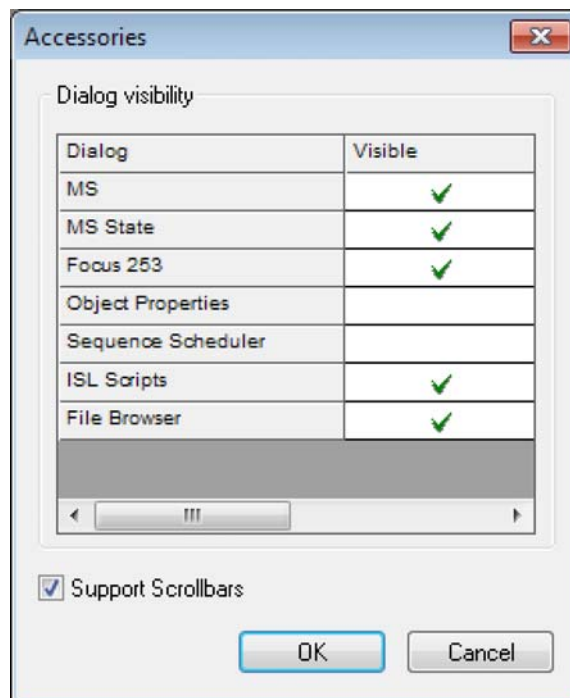
1. In the Isodat folder, double-click  to open the **Acquisition** tool.
2. Right-click one of the panel title bars.



## Measurements

Dual Inlet Measurement Including H-Device and Autosampler A200S

3. In the context menu, click the **Administrate Panels**. The **Accessories** window opens, see [Figure 6-94](#).

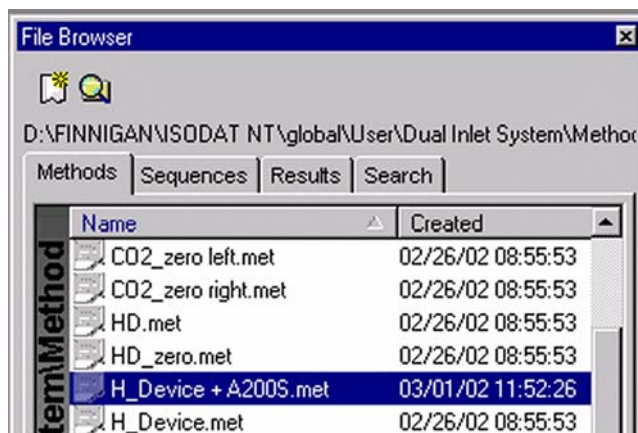


**Figure 6-94.** Activating dialogs in the Accessories window

The **Accessories** window allows to activate dialogs with ✓. When you select a dialog, the respective window is shown as a part of the **Accessories** toolbar.

4. In the **Visible** column, click the field next to **File Browser**.
5. Click **OK**.
6. In the File Browser, click the **Methods** tab (default).

7. Double-click a predefined Method, for example **H\_Device + A200S.met**. See [Figure 6-95](#).



**Figure 6-95.** H\_Device + A200S.met

If the Standards in the Method to be opened are older than the current ones in the Standard Database or if the user has changed the Method, the warning shown in [Figure 6-96](#) appears.



**Figure 6-96.** Actualizing the Standard values

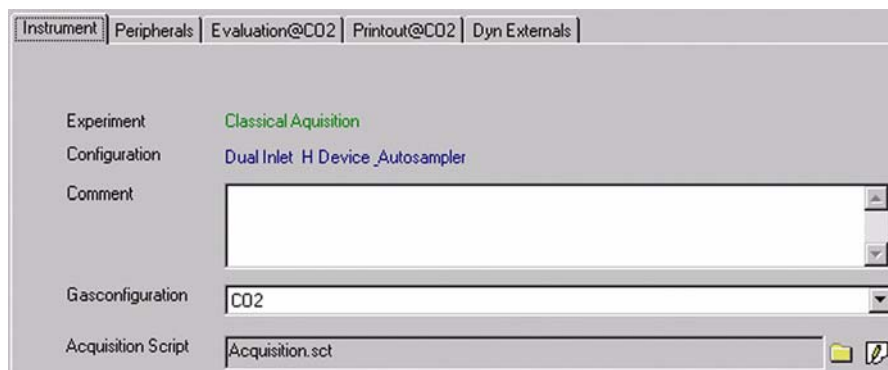
8. Click **OK**.

The Method is corrected. The current values of the Standard Database – for example, the laboratory Standard has changed or has been replaced – are transferred to the Method.

## Measurements

### Dual Inlet Measurement Including H-Device and Autosampler A200S

The Instrument page of the Method H\_Device + A200S.met opens. See [Figure 6-97](#).



**Figure 6-97.** Instrument page of H\_Device + A200S.met

9. For **Gasconfiguration**, select **CO2**.

**Note** The **Acquisition Script** controls the acquisition cycle. The **Acquisition Script** should only be edited by users trained on script editing. ▲

10. Select the **Peak Center Cup**, for example Cup 3, for example, 8 s, see [Figure 6-98](#).

**Integration Time** is the time needed to measure each individual ion intensity of the masses 44, 45, and 46. **Peak Center Predelay** is the waiting time between activation of reference gas and the start of the peak center cycle (for example, 60 s). **Peak Center Postdelay** is the waiting time between the end of the peak center cycle and the start of data acquisition.



**Figure 6-98.** Isotope MS area

11. Click the **Peripherals** tab, see [Figure 6-99](#).

The screenshot shows the 'Peripherals' tab selected. The 'Dual Inlet System' section has the following settings: Reference set to 'Right', Number of Cycles at 8, Idle time at 15s, FV Threshold at 0.03 mBar, HV Pump Time at 60s, and FV Pump Time at 10s. The 'Pressure Adjust' section has On Cup set to 'Cup 2', Delay Time at 10s, Tolerance at 100 mV, and Bellow / Bellow Master set to 'Left'.

**Figure 6-99.** Peripherals page

For details on the Peripherals page, refer to “[Using a Predefined Method for the Dual Inlet System](#)” on page 6-12.

[Figure 6-100](#) shows the values for **Background**.

The screenshot shows the 'Background' area with Pre Delay set to 5s and Integration Cycles set to 1. The 'Capillary / Bellow' section has Signal up set to 0 mV.

**Figure 6-100.** Background area

For details on Background, refer to “[Using a Predefined Method for the Dual Inlet System](#)” on page 6-12.

[Figure 6-101](#) shows the values for **H-Device**.

The screenshot shows the 'H-Device' parameters with Reaction Time, Equilibration Time, and Transfer Time all set to 60s.

**Figure 6-101.** H-Device parameters

**Reaction Time** is the time for the injected substance to react with chromium (for example 60 s, valve 11 must be closed). Reaction time differs between substances. Water only needs 20 to 60 s to react, whereas organic compounds need longer, which must be determined empirically.

**Transfer Time** is the time for the gas to expand from the expansion volume into the bellows (for example 60 s).

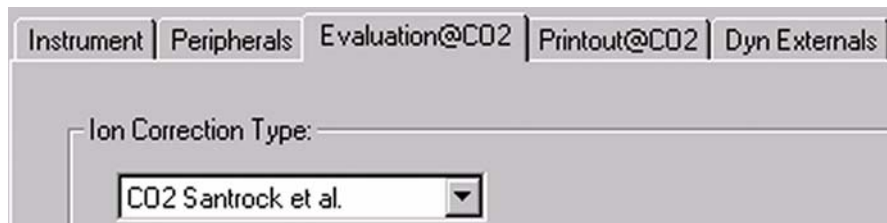
Valve 11 is opened and the gas mixture is allowed to expand into the intermediate or the expansion volume (connecting metal tube plus

## Measurements

### Dual Inlet Measurement Including H-Device and Autosampler A200S

valve body). An **Equilibration Time** of 30 s is recommended for 800 °C to 850 °C. For other temperatures, this needs to be tested empirically.

- Click the **Evaluation@CO2** tab, see [Figure 6-102](#).




**Figure 6-102.** Ion Correction Type

- Select the **Ion Correction Type** (for example **CO2 Santrock et al.**).
- Select **None** or **Sigma** as **Outlier Test**. See [Figure 6-103](#).

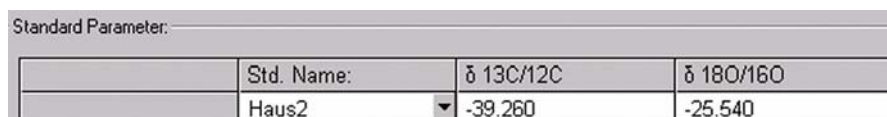


**Figure 6-103.** Outlier Test

- In case of **Sigma**, click  to specify the k-fold of the standard deviation.

For details, refer to “[Using a Predefined Method for the Dual Inlet System](#)” on [page 6-12](#).

- For **Std. Name** (see [Figure 6-104](#)), choose a suitable standard.



Standard Parameter:			
	Std. Name:	δ 13C/12C	δ 18O/16O
	Haus2	-39.260	-25.540

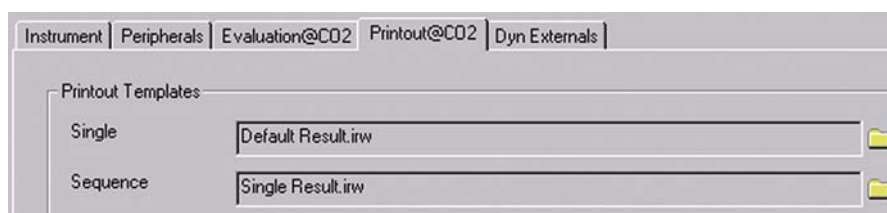
**Figure 6-104.** Standard Name

[Figure 6-105](#) shows the values for **Extended Parameters**.



**Figure 6-105.** Extended Parameters area

17. Click the **Printout@CO2** tab, see [Figure 6-106](#).



**Figure 6-106.** Printout@CO2 page

For details on the Printout page, refer to [“Using a Predefined Method for the Dual Inlet System”](#) on [page 6-12](#).

For details on the WK 1 Export Template page (see [Figure 6-107](#)), refer to [“Using a Predefined Method for the Dual Inlet System”](#) on [page 6-12](#).



**Figure 6-107.** WK1 Export Templates page


The Method for measurements using Dual Inlet in combination with a H-Device and an A200S Autosampler has now been established.

18. As next step, either use a predefined Sequence (refer to [“Using a Predefined Sequence for Dual Inlet System, H-Device, and Autosampler”](#) on [page 6-73](#)) or create a new Sequence (refer to [“Creating a New Sequence”](#) on [page 6-3](#)).

## Using a Predefined Sequence for Dual Inlet System, H-Device, and Autosampler

- ❖ **To use a predefined sequence for Dual Inlet system, H-Device, and Autosampler**



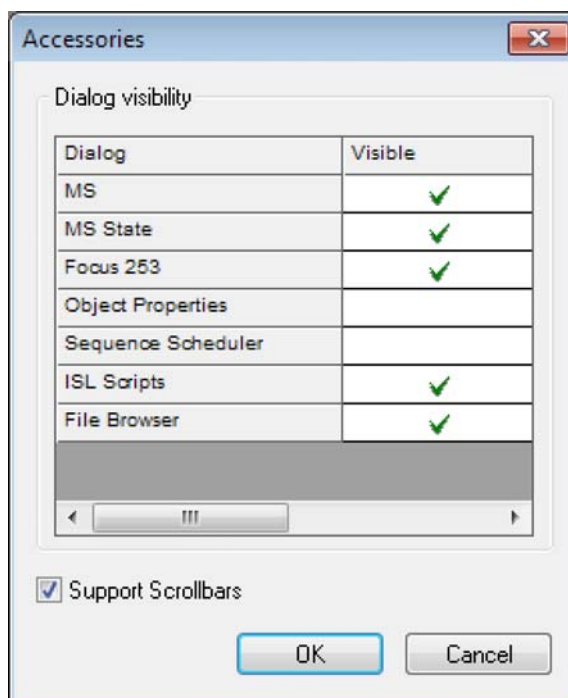
1. In the Isodat folder, double-click  to open the **Acquisition** tool.
2. Right-click one of the panel title bars.



## Measurements

### Dual Inlet Measurement Including H-Device and Autosampler A200S

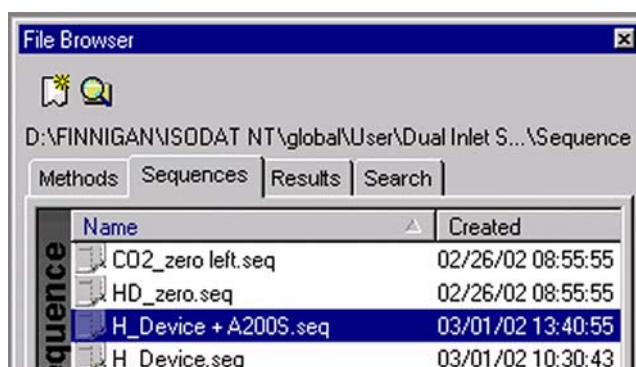
3. In the context menu, click the **Administrate Panels**.  
The **Accessories** window opens, see [Figure 6-108](#).



**Figure 6-108.** Activating dialogs in the Accessories window

The **Accessories** window allows to activate dialogs with ✓. When you select a dialog, the respective window is shown as a part of the **Accessories** toolbar.

4. In the **Visible** column, click the field next to **File Browser**.
5. Click **OK**.
6. In the File Browser, click the **Sequences** tab (default: Methods tab).
7. Double-click a predefined Sequence, for example **H\_Device + A200S.seq**. See [Figure 6-109](#).



**Figure 6-109.** Sequences page: H\_Device + A200S.seq

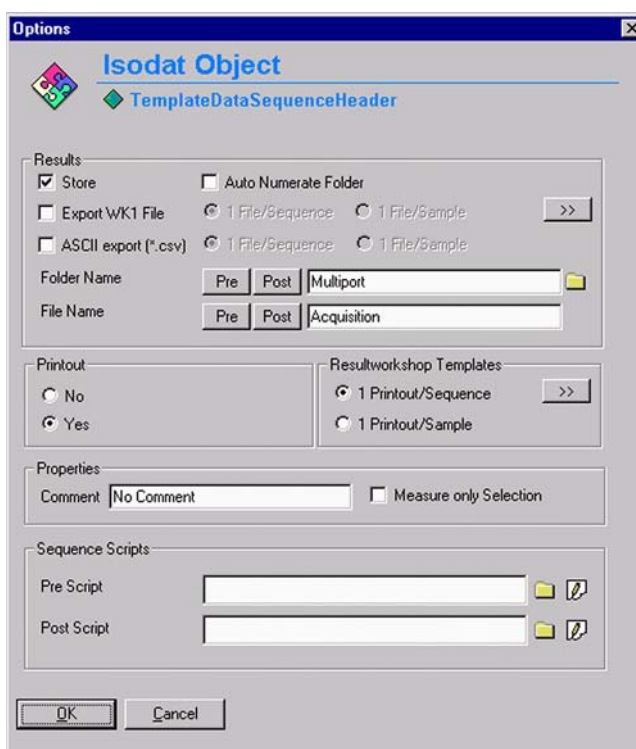
In the Method column, see [Figure 6-110](#), the Method chosen in “Using a Predefined Sequence for Dual Inlet System, H-Device, and Autosampler” on page 6-73 appears as default (H\_Device + A200S.met). It is possible, however, to select another Method from the Method column, to enter sample positions (AS Sample) or select another autosampler method (AS Method).

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

**Figure 6-110.** Sequence List for H\_Device + A200S.seq



- Click the  button.  
The Options dialog opens, see [Figure 6-111](#).



**Figure 6-111.** Options for sequence

- Select parameters for **Results**.

## Measurements

Dual Inlet Measurement Including H-Device and Autosampler A200S

10. Select parameters for **Printout**.
11. Select parameters for **Sequence Scripts**.
12. Click **OK** to start Sequence acquisition.

## Basic Procedures Before Measurement

Before starting any acquisition, make sure that the following procedure has been performed:


- Electronic offset has been measured and saved.
- All peripherals have been configured.
- The mass calibration for all gases to be measured has been performed.
- In case of a Dual Inlet application, the Bellows Calibration has been performed.
- In case of hydrogen collectors, the H3 factor for Continuous Flow and Dual Inlet application has been determined.
- In case of Elemental Analyzer application for C/N or H/O analysis, the Jump Calibration has been performed and saved.
- In case of a Liquid Autosampler, the COM port and the other parameters are set.

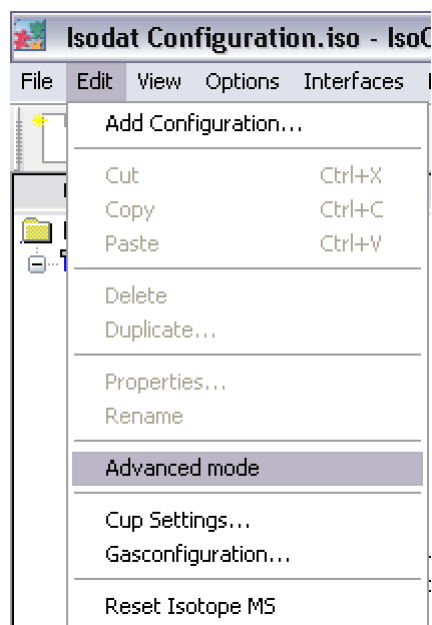
## Editing the Base Instrument via Advanced Mode

This chapter describes how to edit the base instrument configuration in Advanced mode.

**Note** Editing configurations is only possible in **Advanced mode** and should only be performed by personnel trained by Thermo Fisher Scientific. ▲

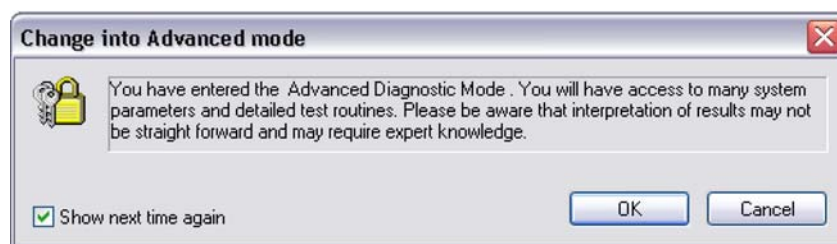
### ❖ To edit base instrument configuration

1. In the Isodat folder, double-click  to open the **Configurator** tool.
2. Select **Edit > Advanced mode**, see [Figure 6-112](#)



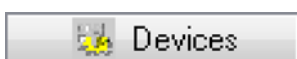
**Figure 6-112.** Menu Edit in the Configurator

The **Change into Advanced mode** window opens, see [Figure 6-113](#):



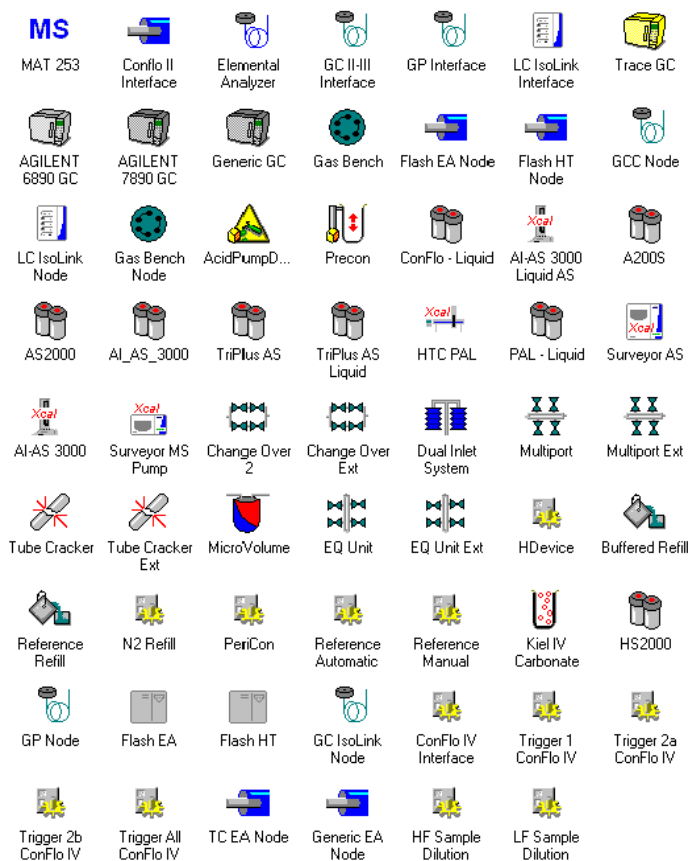
**Figure 6-113.** Change into Advanced mode

3. Click **OK**.



4. Click the **Devices** tab.

Base instruments and all peripherals are displayed in the right pane, see [Figure 6-114](#):

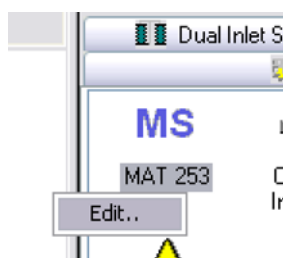


**Figure 6-114.** Peripherals in Device tab

5. Right-click the icon **MAT 253**.

6. Click **Edit**.  
The **Device Editor** opens.

7. Continue with [“Device Tab”](#) on [page 6-80](#).



**Note** The **Edit** command that allows editing a configuration is only accessible via the **Devices** tab, not via the **Elemental Analyzer Seta** tab. ▲

## Measurements

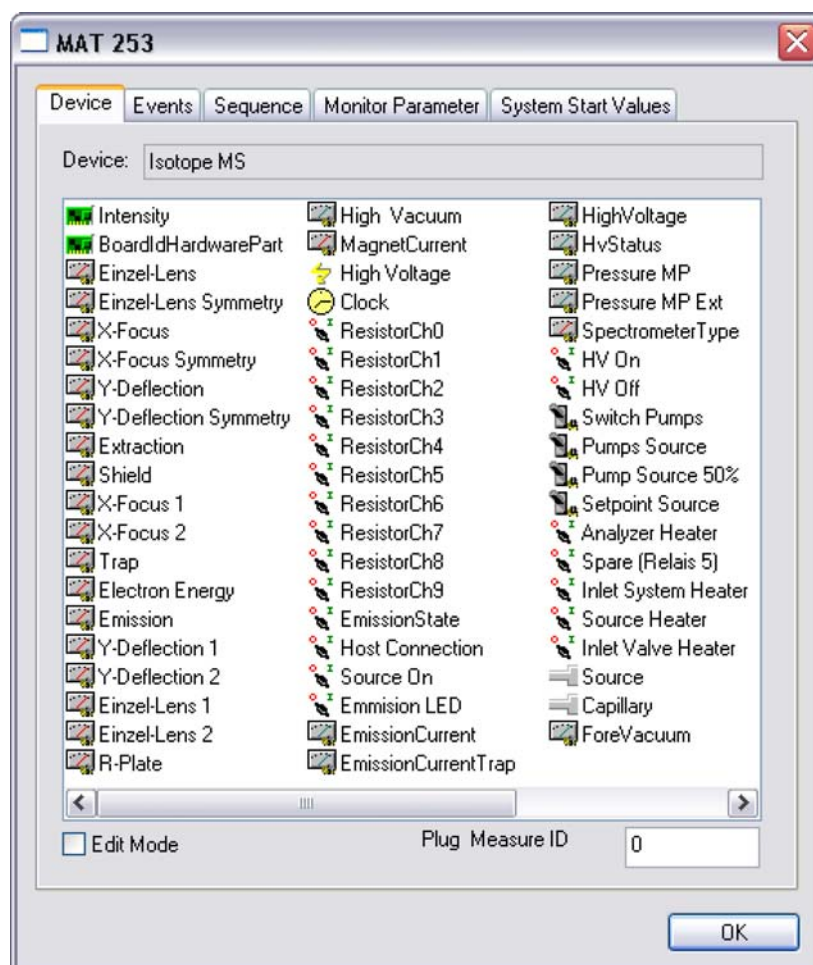
Editing the Base Instrument via Advanced Mode

## Device Tab

The hardware database provides the pool of all available hardware elements. During installation of Isodat, the hardware database is checked for new hardware. As a subset, the installed hardware components of the device are listed here.

The device as a whole is retrieved on the board via its Plug Measure ID (that is, a board address). The board can manage more than one device, not only MAT 253. Other, additional devices can be assigned.

The **Edit** window for the currently edited Device **Isotope MS** is shown in [Figure 6-115](#):



**Figure 6-115.** Device tab for Isotope MS

The **Edit Mode** allows modifying a device by adding additional hardware components from the hardware database.

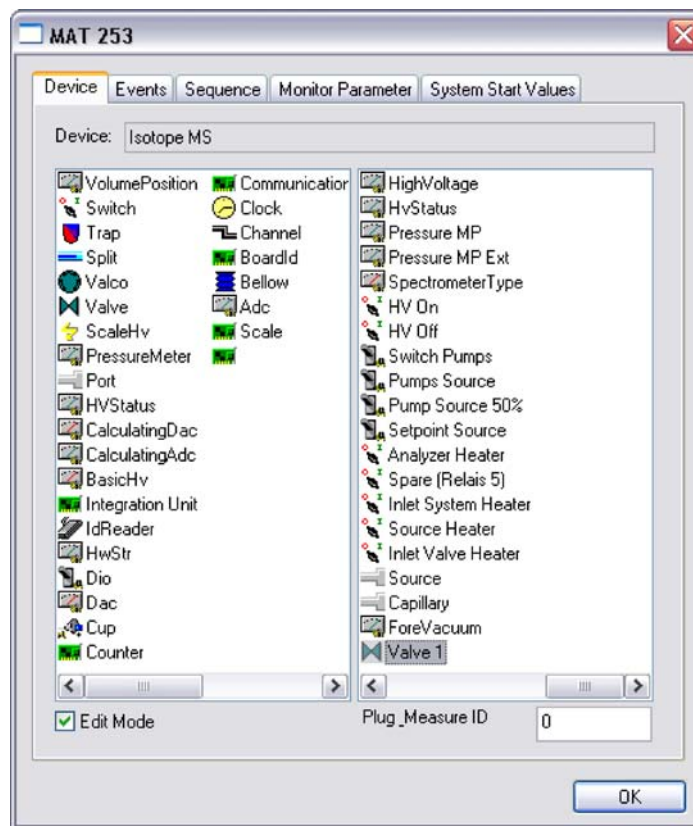
The **Plug Measure ID** is the ID assigned to the current device, for example, **0** for MAT 253 in [Figure 6-115](#).



## Measurements

Editing the Base Instrument via Advanced Mode

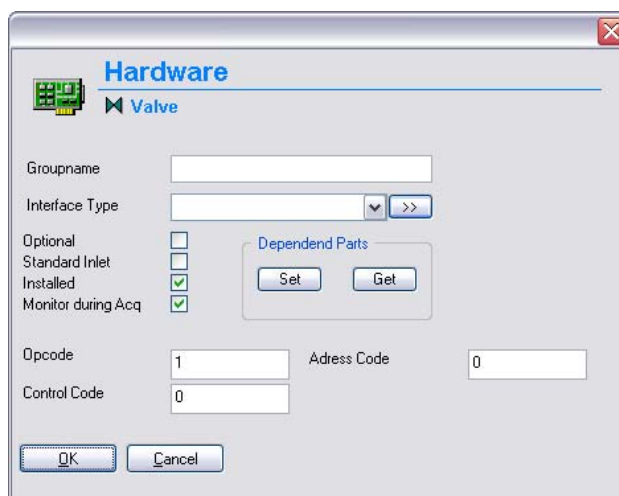
4. Drag and drop the hardware component, for example, **Valve**, into the right pane. See [Figure 6-117](#).



**Figure 6-117.** Hardware component added

The new hardware component **Valve** is added to the right pane. Any added new component must be configured.

5. Double-click on the new hardware component in the right pane, for example, **Valve**.  
The **Hardware** window opens, see [Figure 6-118](#).



**Figure 6-118.** Hardware properties for Valve

6. Configure the newly installed hardware component by assigning its properties.
7. Confirm with **OK**.  
The hardware component is defined via Isodat software.
8. Continue with “[Events Tab](#)” on [page 6-83](#)

## Events Tab

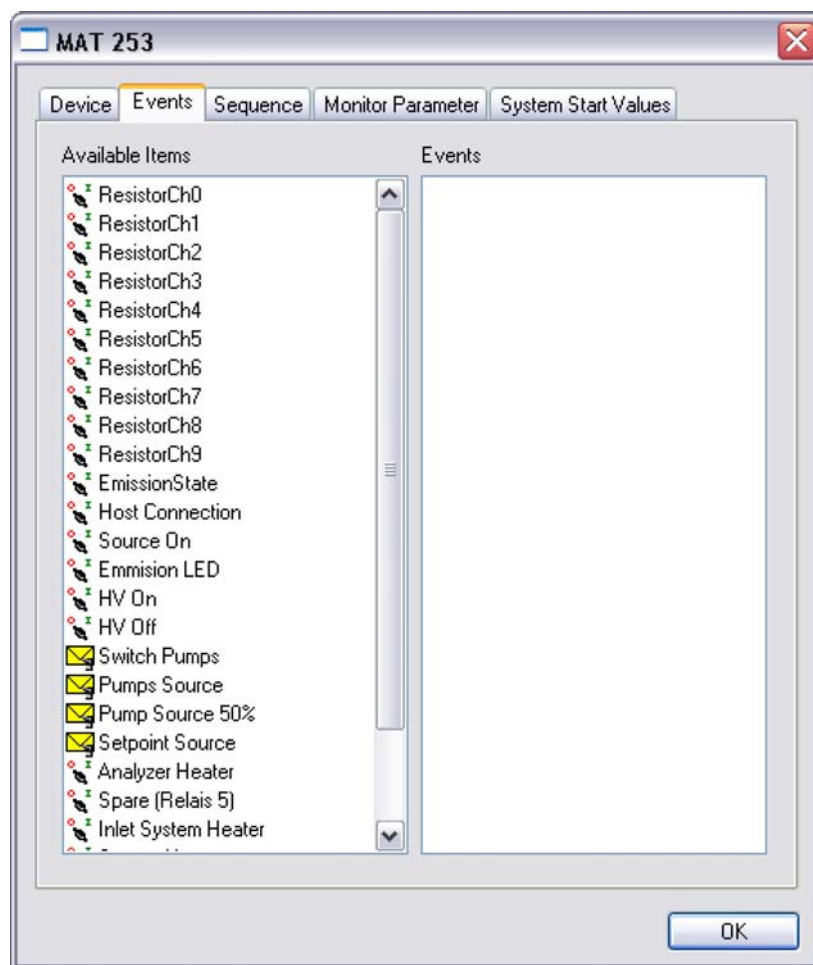
### ❖ To configure Events for a hardware component

1. In the Configurator, open the Advanced mode and open the Edit window of the Device MAT 253 as described in “[Editing the Base Instrument via Advanced Mode](#)” on [page 6-78](#).

## Measurements

Editing the Base Instrument via Advanced Mode

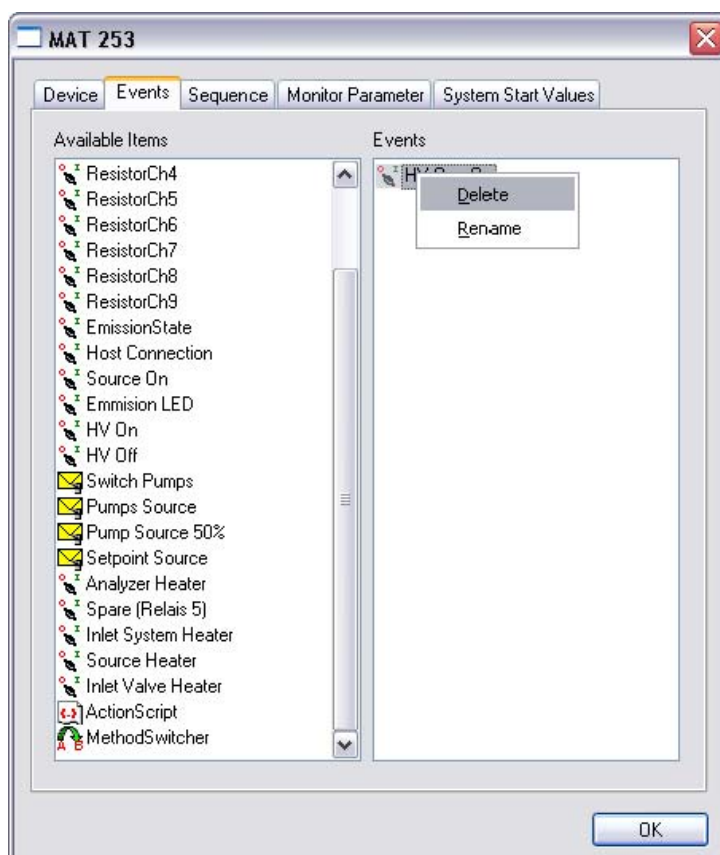
2. Click the **Events** tab in the **Edit** window for the currently edited device, for example, **Isotope MS**, see [Figure 6-119](#).



**Figure 6-119.** Event tab

3. Drag and drop elements from the left pane to the right to add them to the **Time Events** list.

- Right-click on the item added to the **Events** list.  
The context menu opens, see [Figure 6-120](#).



**Figure 6-120.** Delete selected object

- From the context menu, select **Rename** to rename an item, or select **Delete** to delete an Event.
- If you selected **Delete**, the message window opens. Confirm with **Yes**.  
The Event is deleted from the pane.

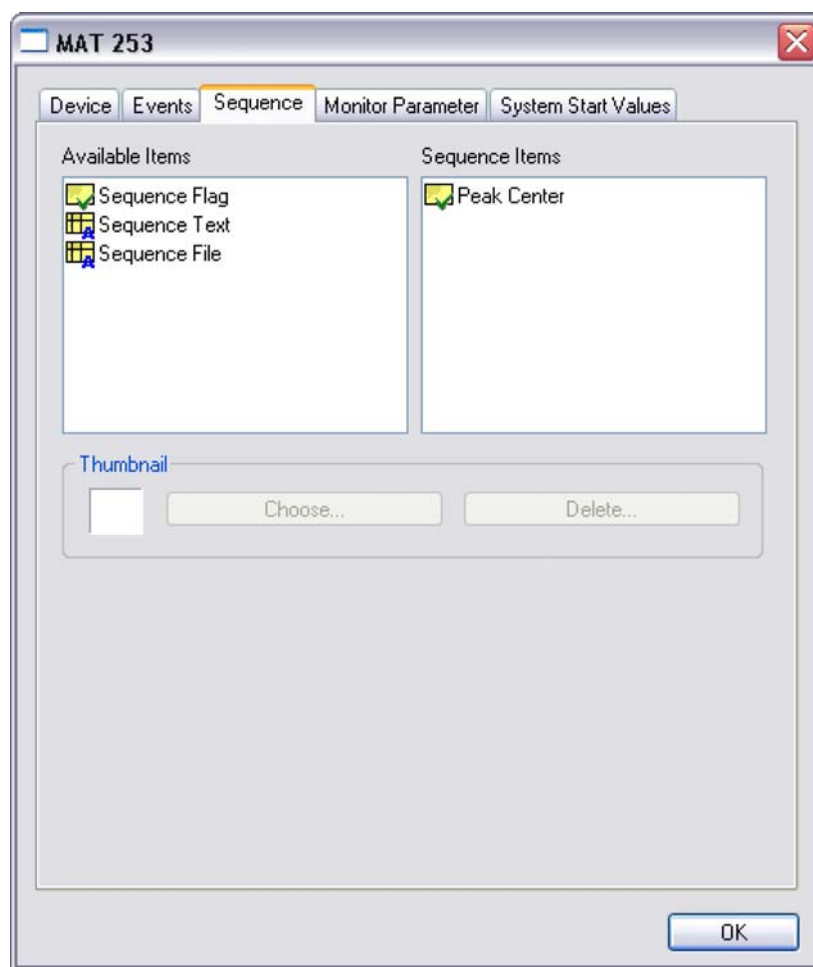
**Note** After you changed Events of the Time Events list by adding, renaming or deleting them, you must create new methods and sequences. In general, after changing a database, create new methods and sequences. ▲

- If you added or changed an item, continue with “[Sequence Tab](#)” on [page 6-86](#).

## Sequence Tab

### ❖ To configure Sequence for a hardware component

1. In the Configurator, open the Advanced mode and open the Edit window of the Device MAT 253 as described in “[Editing the Base Instrument via Advanced Mode](#)” on page 6-78.
2. Click the **Sequence** tab in the **Edit** window for the currently edited Device, for example, **Isotope MS** see [Figure 6-121](#).



**Figure 6-121.** Sequence tab

3. Drag and drop an item from the left pane to the right to add a new item to the **Sequence Item** list.
4. Select, for example, **Sequence Cmd** to let a command be executed.
5. Select **Sequence Flag** to activate or deactivate an action, for example, **Interface Pump Control**.
6. Select **Sequence File** and define method files, for example for autosampler or GC. These methods can then be selected in a sequence.

7. Select **Sequence Text** to add a text column, for example, for a comment.

By dragging and dropping you can add as many flags as you like to a sequence. They are read out in the particular measurement scripts. By activating/deactivating the sequence items, certain parameters defined in the measurement script are set or not (for example, delays or flow rates).

Often, for the first sample within a sequence, a flag is activated. It is deactivated for the following samples of the sequence.

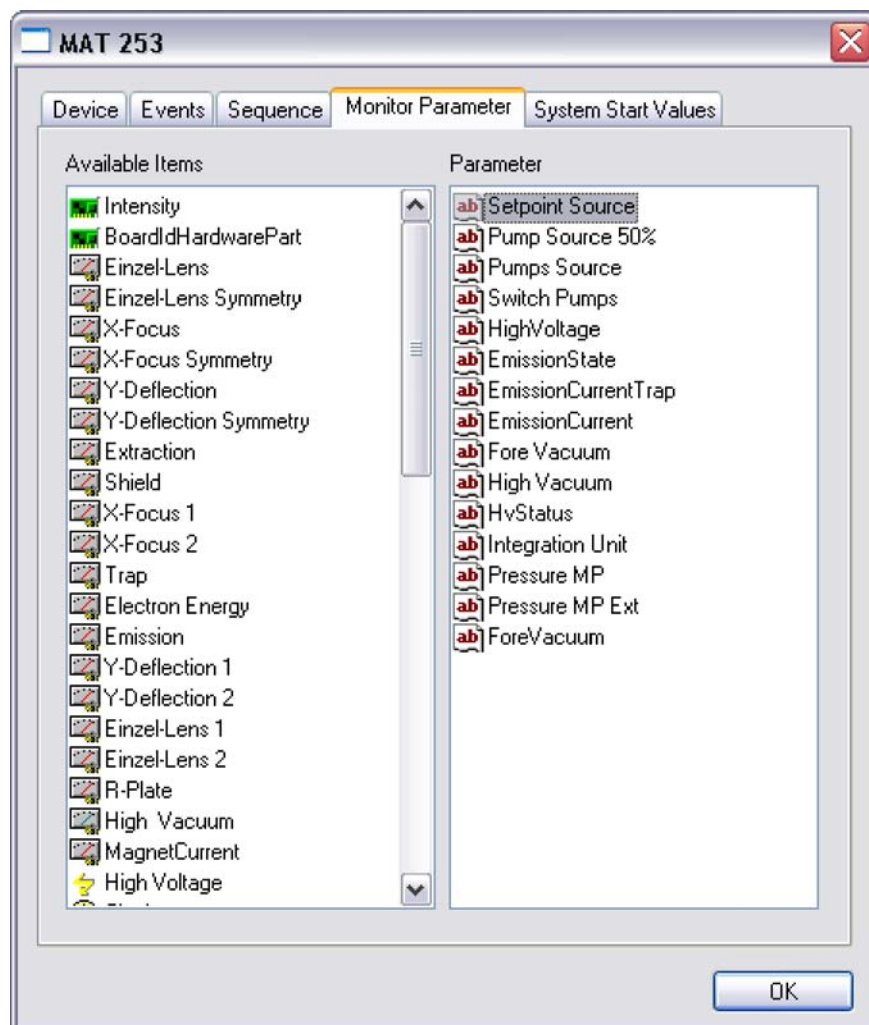
Thus, basic parameters are set only once and kept constant for the following samples. This ensures, for example, working with a constant flow rate during the entire sequence. The same is true for performing a peak center.

## Measurements

Editing the Base Instrument via Advanced Mode

## Monitor Parameter Tab

The right pane of the **Monitor Parameter** tab in [Figure 6-122](#) shows the parameters that are constantly read out and monitored, for example, the status of the acid pump (blocked or not) or the status of the six-port valve.



**Figure 6-122.** Monitor Parameter tab

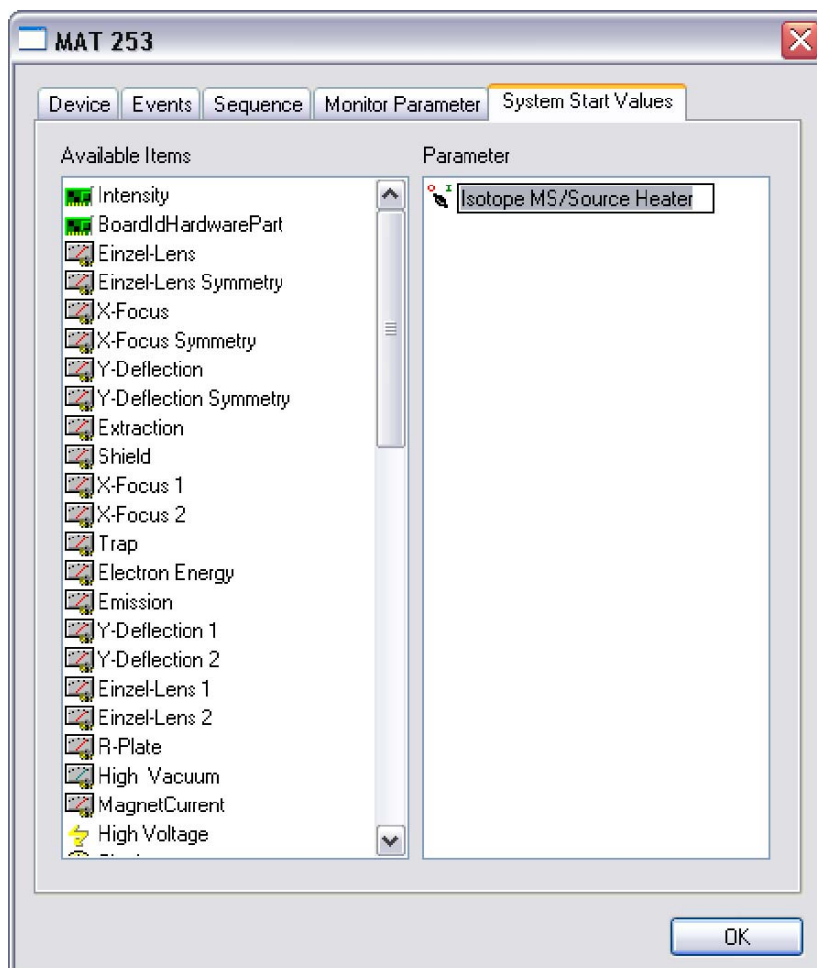
## System Start Values Tab

The right pane of the **System Start Values** tab in [Figure 6-123](#) lists parameters that are set to a certain value (the system start values) during start of Isodat. Individual components of the interface can be switched into an intended status (for example, switching the acid pump off) during start of Isodat.

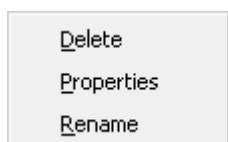
**Note** The shutdown parameters of the system can be edited in the Isodat Generic Editor. ▲


❖ **To set system start values**

1. In the Configurator, open the Advanced mode and open the Edit window of the Device MAT 253 as described in “[Editing the Base Instrument via Advanced Mode](#)” on page 6-78.
2. In the System Start Values tab, drag and drop **Isotope MS/Source Heater** from the left pane to the right pane, see [Figure 6-123](#).



**Figure 6-123.** System Start Values tab

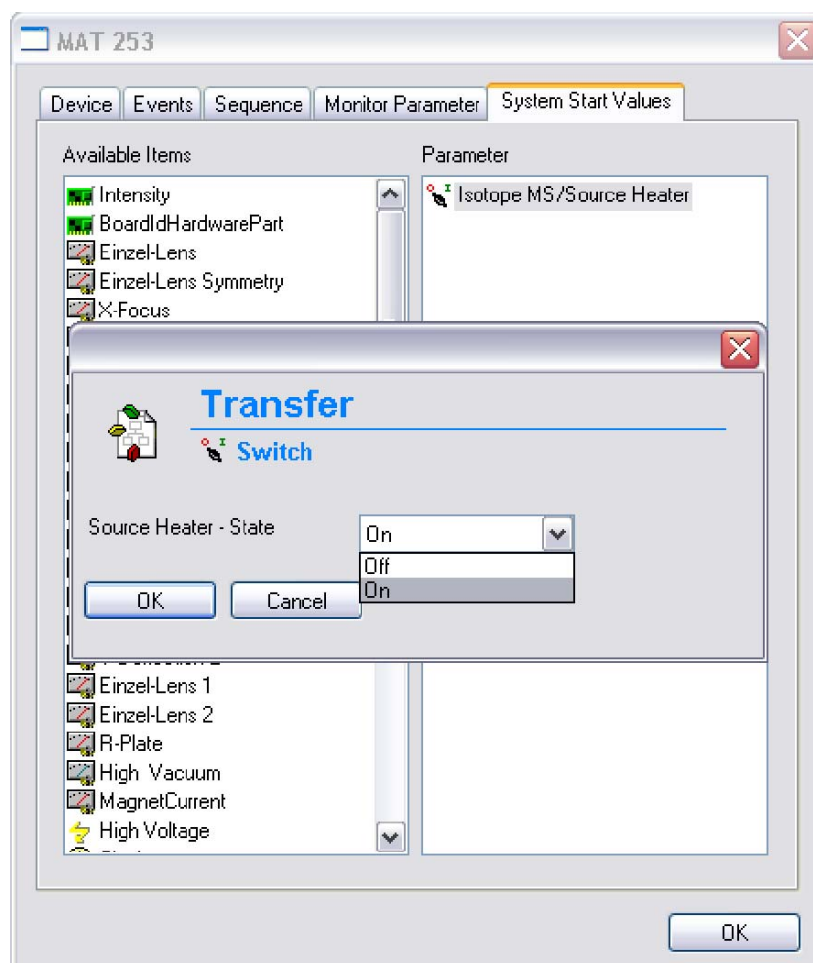


3. Right-click **Isotope MS/Source Heater** in the right pane and select **Properties** from the context menu.
4. Click  to open the drop-down list **Isotope MS/Source Heater**.

## Measurements

Editing the Base Instrument via Advanced Mode

5. Select **On**, see Figure 6-124.



**Figure 6-124.** Properties of Source Heater

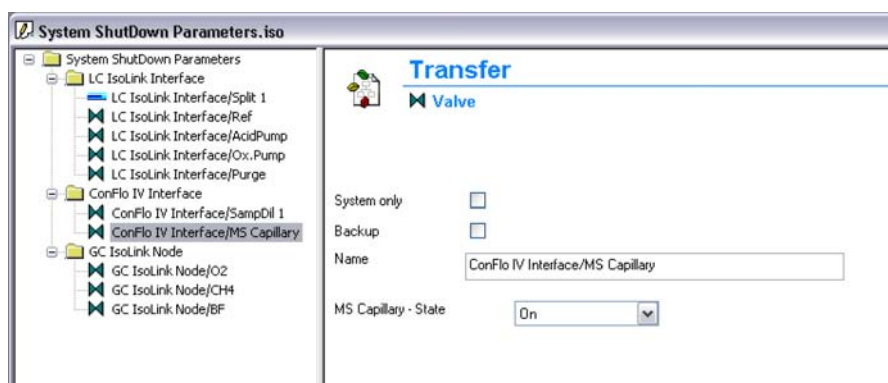
Now every time the Acquisition or Instrument Control starts the Source Heater is switched on.

**Note** If the Source Heater has been On before starting the Isodat software, nothing changes and the Source Heater stays On. ▲

### ❖ To open the Isodat Generic Editor

1. Double-click the file **IsodatEditor.exe** in the Thermo Fisher Scientific Isodat folder ...\\Global\\bin.
2. Select **File > Open**.
3. In the **Open** dialog, select the file **System ShutDown Parameters.iso** in the folder ...\\Global\\Databases.
4. Click **Open**.

Figure 6-125 shows the Isodat Generic Editor:



**Figure 6-125.** System shutdown parameters in Isodat Generic Editor

5. Edit the shutdown parameters of system components (acid pump, oxidation reagent pump, purge) as needed.


**Note** After altering shutdown parameters, a system restart of Isodat is necessary for the parameters to take effect. This feature is used for security. Some parameters are set in a way not to damage interface or IRMS during system startup and shutdown. ▲

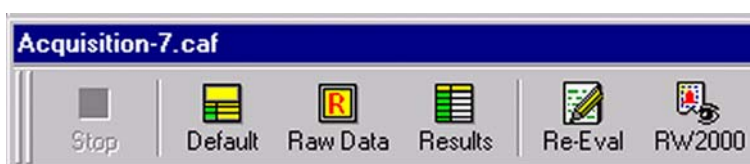
## Viewing Results in the Acquisition Window

Results of measurement are shown in the Acquisition window. This chapter describes the toolbar commands and pages of the Acquisition window.

### Toolbar Commands







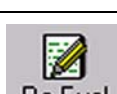

After the measurement has been started by clicking  in the Sequence window of the Acquisition tool, an acquisition file \*.caf is generated (for example Acquisition-7.caf). The Acquisition window opens, see [Figure 6-126](#):



**Figure 6-126.** Acquisition window – toolbar

The commands of the toolbar are explained in [Table 6-1](#):

**Table 6-1.** Toolbar commands description

Command	Description
	A running Dual Inlet data acquisition can be stopped.
	The data are shown both in a data grid and graphically (that is both in medium size).
	The graphical representation of the raw data is shown in maximum size (that is without data grid).
	The result grid is shown in maximum size (that is 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 (for example for preparing a printout).

## Sequence Line

A particular line of the chosen Sequence is shown. It denotes the currently measured sample. See [Figure 6-127](#).

Line					Fill + EQ	Unit 1 Port	Unit 1 Bank	Identifier 1	Method
47					No	5	3	water	CO2_EQ.met

**Figure 6-127.** Sequence line

## Pages of the Acquisition Window

The Acquisition window offers the following pages for data interpretation:

- [Raw Page](#)
- [Evaluated Page](#)
- [Errors Page](#)
- [Extended Page](#)
- [Method Page](#)

### Raw Page

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 of [Figure 6-128](#), the sample number is displayed (for example “Pre” and “1” to “8”; compare the lines of the selected Sequence):

Raw<CO2>	Evaluated<CO2>	Errors	Extended	Method						
	Int. 44 Sample [mV]	Int. 45 Sample [mV]	Int. 44 Standard [mV]	Int. 45 Standard [mV]	rR 45CO2/44CO2	rDelta 45CO2/44CO2 [%] vs. CO2_zero	rDelta 46CO2/44CO2 [%] vs. CO2_zero	Delta 13C/12C [%] vs. PDB	Delta 18O/16O [%] vs. V-SMOW	AT% 13C [%]
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

**Figure 6-128.** Raw page

The raw data grid are detailed in [Table 6-2](#):

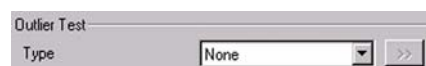
**Table 6-2.** Columns of the raw data display


Column	Description
Int Sample	Sample intensity of a particular mass in mV (for example mass 44). Each sample mass has a column of its own.
Int. Standard	Standard intensity of a particular mass in mV (for example mass 44). Each standard mass has a column of its own.
rR	Raw ratio of two masses, that is ratio of the intensities of these two masses (for example rR 45/44).
rDelta	Raw Delta of two molecular masses (in ‰) vs. a secondary standard - that is vs. a user standard. for example: rDelta [45 CO <sub>2</sub> /44 CO <sub>2</sub> ] vs. CO <sub>2</sub> _zero. These values are molecule deltas and are calculated from the raw ratios.
Delta	Delta value of two element isotopes (in ‰) vs. a primary standard. for example: 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.
AT %	Atomic percentage value of an isotope, for example: At % [13C] or At % [18O]. It is calculated from the element delta and the corresponding absolute element ratio of the standard.

### Representation of Data in the Raw Data Grid

5382.165

15.362



- A red value indicates that during integration the timeout criterion was reached. Red numbers only occur in case of intensities, that is in the columns Int. Sample and Int. Standard.
- A black value on a pink background is an outlier relating to the particulars given in the Outlier Test area of the Evaluation page.
- If **Sigma** was selected and specified by , Outliers may occur and are marked. They only occur in case of Delta values, for example 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 (that is the case of **Sigma**). If you then decide to reevaluate these data using a changed Method (via the Method page) selecting **None** in the Outlier Test area of the Evaluation page, no more outliers are calculated. Thus, the values will turn into normal black ones. See [Figure 6-129](#).

15.362 → 15.362

**Figure 6-129.** Values changing color

20.454

- A black value on a green background is a Normal value falling into the k-fold of  $\sigma$  (that is no outlier). They only occur in case of Delta values, for example Delta [13C/12C] vs. V-PDB or Delta [18O/16O] vs. V-SMOW.

## Evaluated Page

[Figure 6-130](#) shows the **Evaluated<CO2>** tab.

Raw<CO2>	Evaluated<CO2>	Errors	Extended	Method			
			$\delta$ Mean	$\delta$ Std.Dev.	$\delta$ ST. Error	Atom%	Outlier
			-2.7050	0.0095	0.0036	1.1083	1
			-4.2056	0.0234	0.0095	0.1993	2

**Figure 6-130.** Evaluated page

The Delta values (that is  $\delta$  13C/12C vs. V-PDB and  $\delta$  18O/16O vs. V-SMOW) marked pink or green in columns 5 of the raw data grid undergo a statistical analysis here, see [Table 6-3](#):

**Table 6-3.** Columns of the Evaluated<CO> tab

Column	Description
Mean	Mean of the Delta values noted in column 5 (for example Mean of all Delta 18O/16O values in column 5).
Std. Dev.	Standard deviation of the Delta values noted in column 5. (for example Standard deviation of all Delta 18O/16O values in column 5).
St. Error	Standard error of the Delta values noted in column 5 (for example Standard error of all Delta 18O/16O values in column 5).
Atom-%	Atomic percentage value of the heavier isotope. It is calculated from the $\delta$ Mean value (for example Atom-% of $^{13}\text{C}$ is calculated from $\delta$ [13C/12C]).
Outlier	Number of outliers in column 5 entering into calculation of $\delta$ Mean, $\delta$ Std. Dev and $\delta$ St. Error (for example 2).

## Errors Page

Table 6-131 shows the **Errors** tab.

Raw<CO2>	Evaluated<CO2>	Errors	Extended	Method
Number	Status	Object	Information	Script

**Figure 6-131.** Errors page

The Errors page contains detailed information about errors that occurred during data acquisition, see [Table 6-4](#):

**Table 6-4.** Columns of the Errors tab

Column	Description
Number	The consecutive number of the error (that is 1; 2; 3 ...)
Status	The quality or severity of the error (for example 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 Page

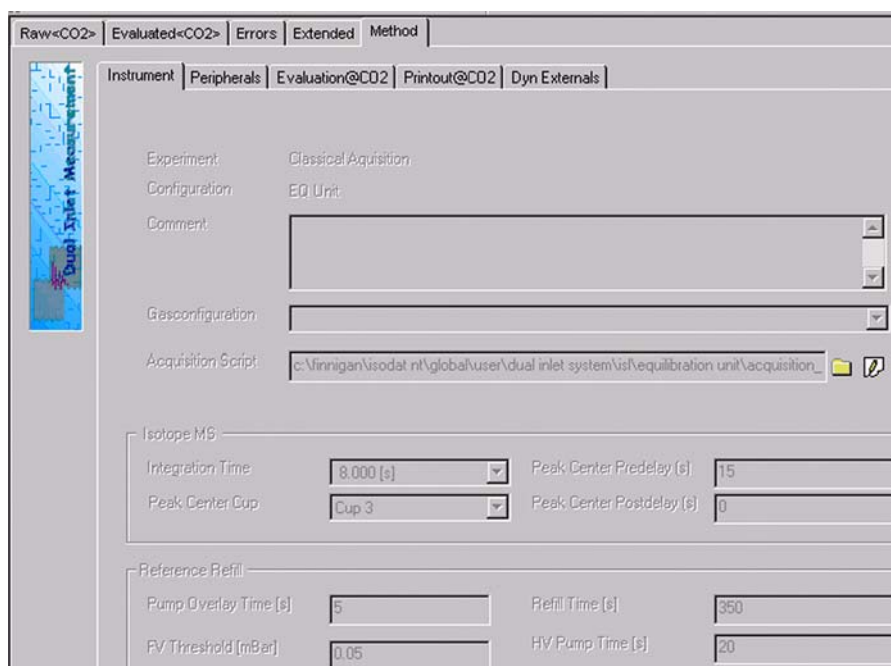
On the Extended page, a measurement protocol is presented. Detailed information is given about particular results during measurement progression (for example Peak Center results or Pressure Adjustment results). See [Figure 6-132](#). The kind of information depends on the selected configuration. For example, different information in case of **Dual Inlet + Microvolume** compared to **Dual Inlet + H-Device**.

Raw<CO2>	Evaluated<CO2>	Errors	Extended	Method
Information				
Sample Pressure:[mBar] 34.2				
PeakCenter 1981				
Pressure Adjustment: Left: 5517.0 Right: 5483.6				

**Figure 6-132.** Extended page

## Method Page

Figure 6-133 shows the **Method** tab.

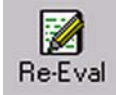


**Figure 6-133.** Method page

For Data Reevaluation purposes, some parameters of the Method selected prior to measurement start can be modified, mainly on the Peripherals page and the Evaluation page.

### ❖ To start reevaluation

1. Click the tabs with the changeable parameters, for example Peripherals page and Evaluation page.
2. Type in the new corrected values.  
After the parameters of interest have been modified, reevaluation of the data can be started.

3. To start reevaluation, click the  button.

## Excel Export of Results

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 (that is 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 (that is 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 of Excel Export

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 (for example multigas measurement).

Single parts of a measurement can be exported to an export file of their own (for example only the Method parameters).

Some types of data can be specified further using query tabs, where sophisticated export properties are defined (for example peak relevant data).

## Creating an Export Template

### ❖ To create an Excel export template

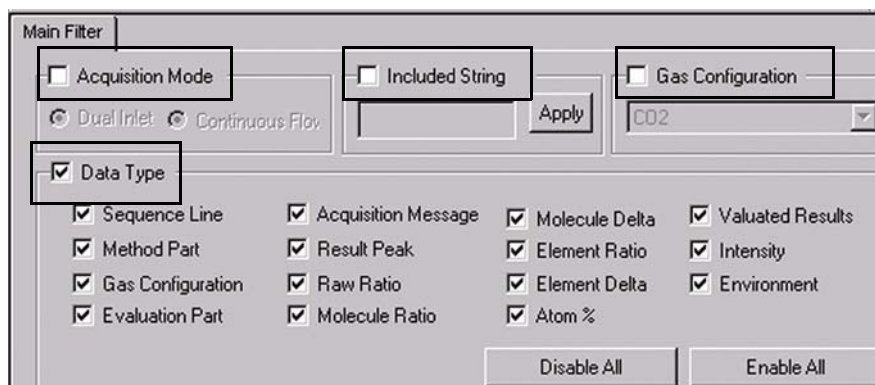
1. On the toolbar of the Dual Inlet window, click the  **New** button.

2. Click **Data Export** and confirm by **OK**. Thus, a new export template can be edited. See [Figure 6-134](#).



**Figure 6-134.** Creating a new export template

Four selector types can be selected. See [Figure 6-135](#).



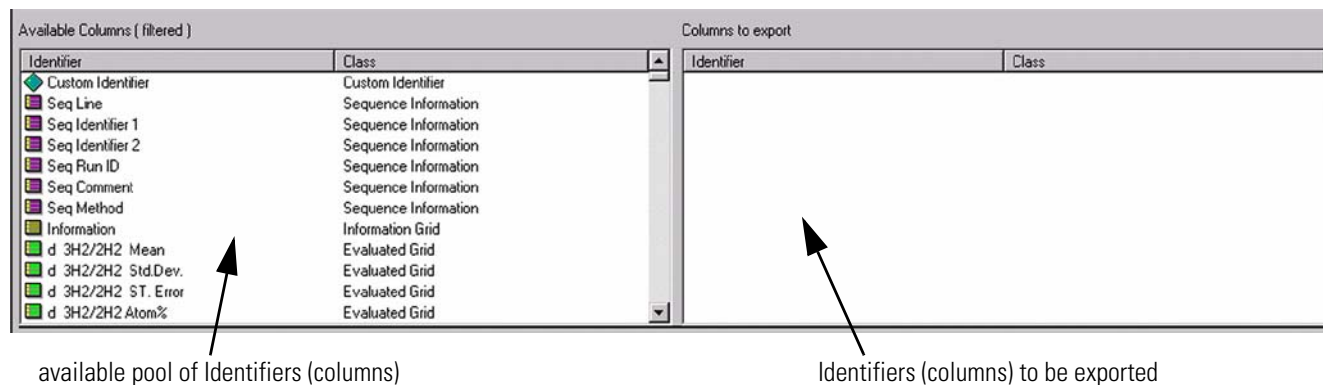
**Figure 6-135.** Main filter page

## Measurements

Excel Export of Results

### 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 of [Figure 6-136](#)). Without selectors, all exportable data are visible.



**Figure 6-136.** Selector types for template creation

**Note** Selectors are always combined by a conjunction (that is by an AND relation). ▲

**Note** (De-)activation of Selectors can be changed during template creation. The export columns will not be affected. ▲

### Acquisition Mode

If **Acquisition Mode** is selected, choose via click between parameters that are only relevant for Dual Inlet Acquisition and those only relevant for Continuous Flow Acquisition. As default, both parameter types are selected, that is no selector. See [Figure 6-137](#).



**Figure 6-137.** Acquisition Mode

### Included String

If **Included String** is selected and you type a string (for example **Delta**), the entries in the left pane (pool) will be selected according to that string after pressing **Apply**. See [Figure 6-138](#). The entries that match the string will be displayed in the right pane.



**Figure 6-138.** Included String

### Gas Configuration

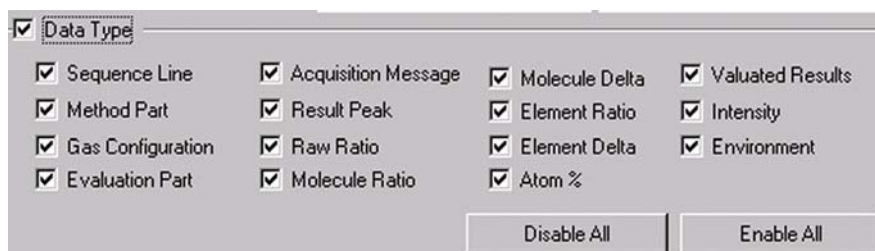
If **Gas Configuration** is selected, choose a gas configuration from the list (for example **CO2**). See [Figure 6-139](#). All relevant data for this gas configuration will be shown in the right pane.



**Figure 6-139.** Gas Configuration

### Data Type

If **Data Type** is selected, you can select one or more criteria according to data type (for example if only **Raw Ratio** is selected, merely entries concerning raw ratios will be displayed in the right pane). See [Figure 6-140](#). As default, all data types are selected. Click **Disable All** or **Enable All** to ease your work with the Data Type selector.



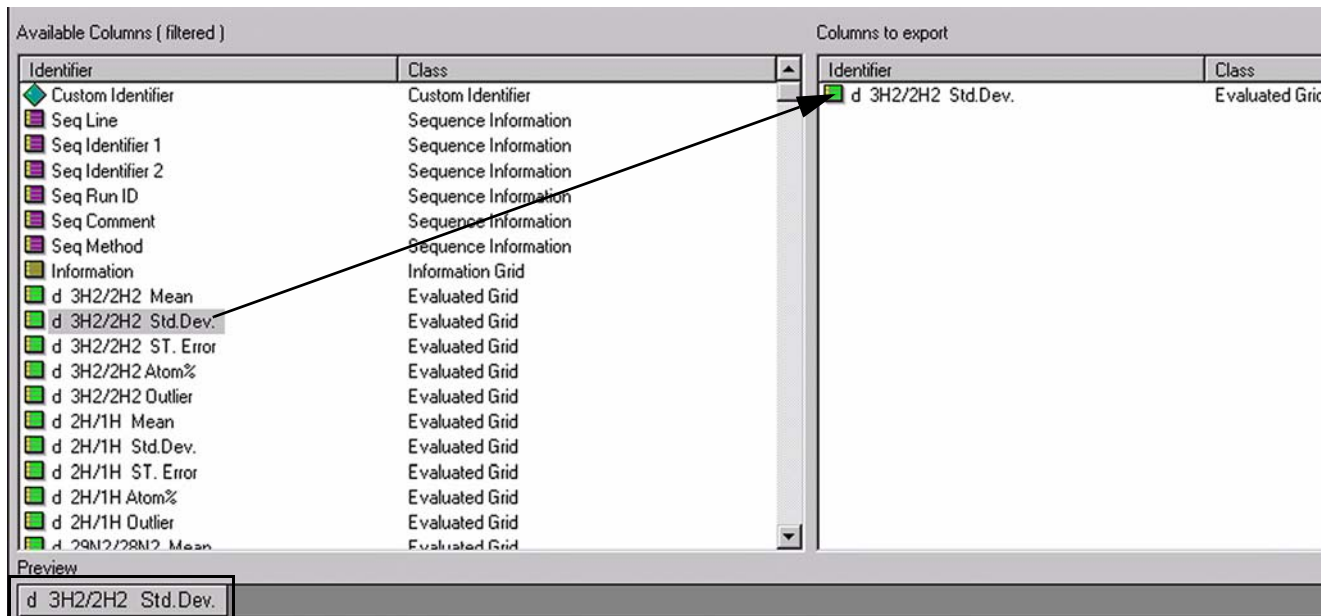
**Figure 6-140.** Data Type

## Measurements

Excel Export of Results

### Inserting Individual Identifiers

In addition to selectors, drag and drop individual Identifiers to the right pane to export them. Every individual Identifier will appear below the panes as a column. See [Figure 6-141](#).



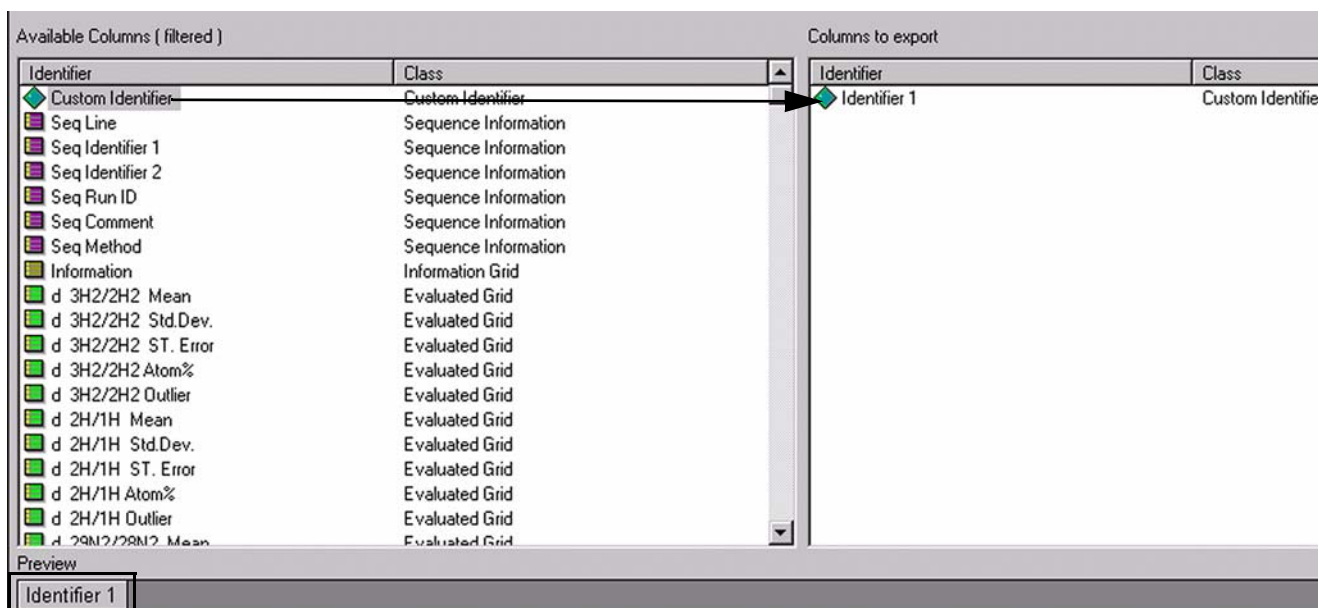
**Figure 6-141.** Individual Identifiers

### Custom Identifier

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.

❖ **Inserting the Custom Identifier**

1. Drag and drop the **Custom Identifier** from the left to the right pane. See [Figure 6-142](#).



**Figure 6-142.** Inserting the Custom Identifier

The following message box shown, see [Figure 6-143](#).



**Figure 6-143.** Insert Identifier dialog box

2. Enter a name for the custom identifier, for example **Identifier 1**.
3. Click **OK**.

The Custom Identifier also appears as a column in the wk1 Excel sheet. To create additional empty columns, proceed with the next Custom Identifiers) by repeating the steps.

**Removing Identifiers**

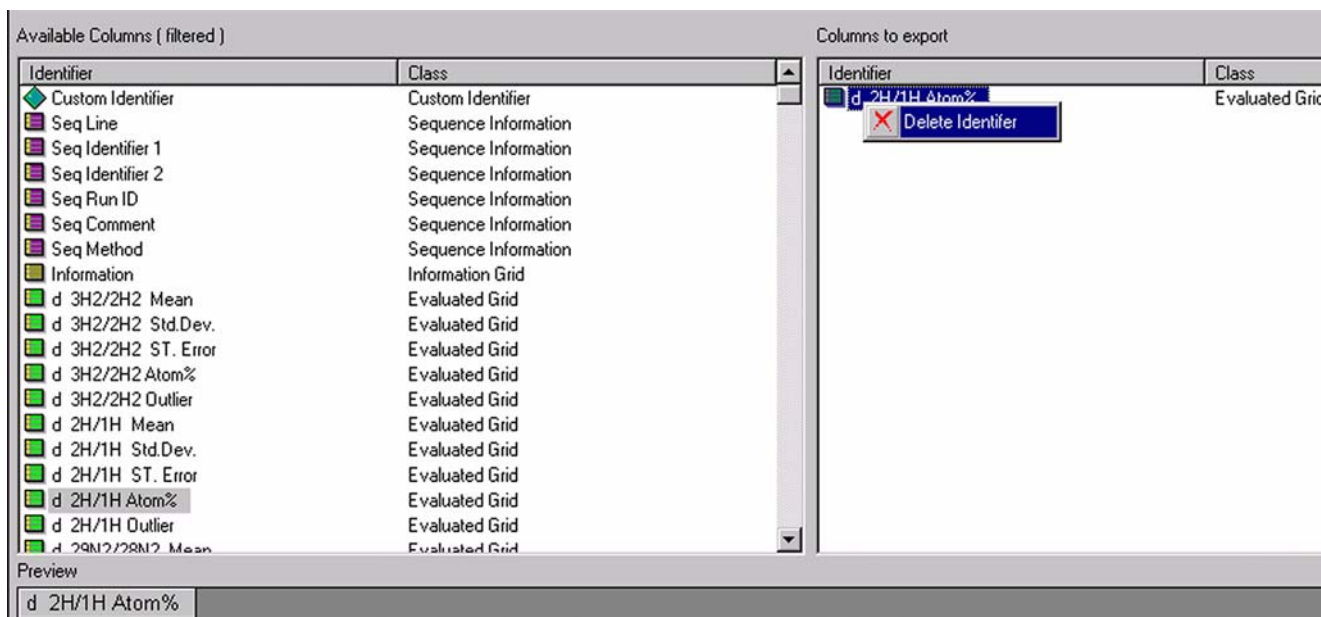
❖ **To remove Identifiers**

1. To remove one or more Identifiers (Custom Identifiers or other ones) from the right pane, mark them there. Then right-click them.

## Measurements

### Excel Export of Results

2. In the shortcut menu, choose **Delete Identifier**. See [Figure 6-144](#).



**Figure 6-144.** Deleting Identifiers

3. To remove the Identifiers), confirm by **Yes**. See [Figure 6-145](#).



**Figure 6-145.** Confirming the deletion of Identifiers

The selected Identifiers) will be removed from the right pane together with the provided empty columns in the Excel sheet.

## Saving an Export Template

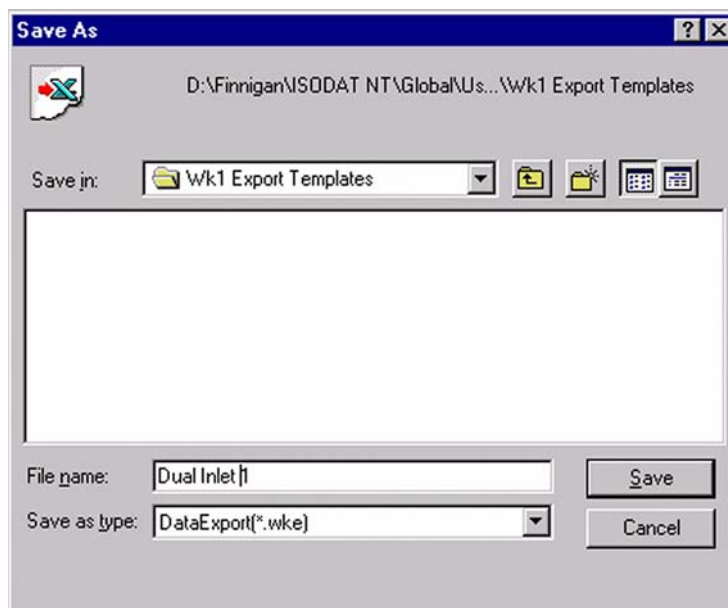
After creating an export template (that is a wke file), it must be saved.

### ❖ To save an export template

1. On the toolbar of the Dual Inlet window, click the **Save** or **Save As** button.



2. Save the export template in the folder Wk1 Export Templates! See [Figure 6-146](#).




**Figure 6-146.** Saving an Export Template

3. In the field **File Name**, enter a name for the export template, for example **Dual Inlet 1**.
4. In the Save as type field, accept the extension **.wke** and click **Save**.

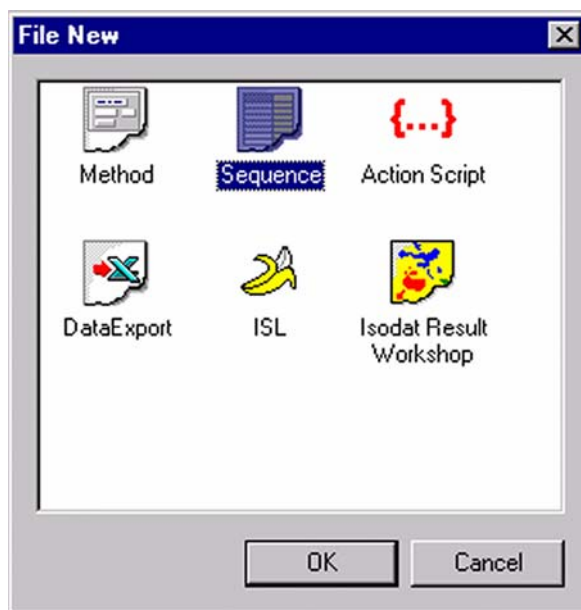
## Applying an Export Template

Several samples can be bundled into a single export file.

### ❖ To apply an export template

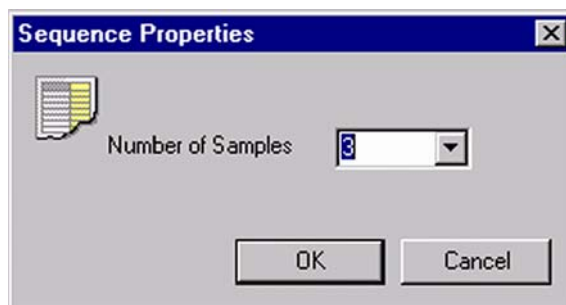
1. Open a new Sequence via the  **New** button.

2. Then click the Sequence icon. See [Figure 6-147](#).



**Figure 6-147.** Creating a new sequence

3. Confirm by **OK**.
4. Type your number of samples (for example **3**). See [Figure 6-148](#).



**Figure 6-148.** Entering the number of samples

5. Confirm by **OK**.
6. The Sequence of your choice is displayed. See [Figure 6-149](#).

Line				Identifier 1	Method
1	✓	✓	✓		CO2.met
2	✓	✓	✓		CO2.met
3	✓	✓	✓		CO2.met

**Figure 6-149.** Sequence List for export template

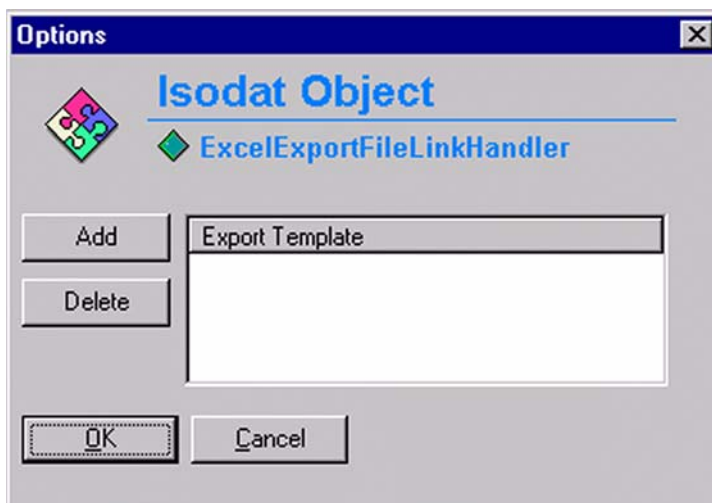


- Click the **Start** button.  
Because no export templates have been selected yet, the **Export WK1. File** check box is inactive. See [Figure 6-150](#).



**Figure 6-150.** Export WK1 File box is inactive

- Click the **>>** button.  
Because no export templates have been selected yet, the Export Template list is empty. See [Figure 6-151](#).



**Figure 6-151.** Export Template list is empty

- Click the **Add** button.

## Measurements

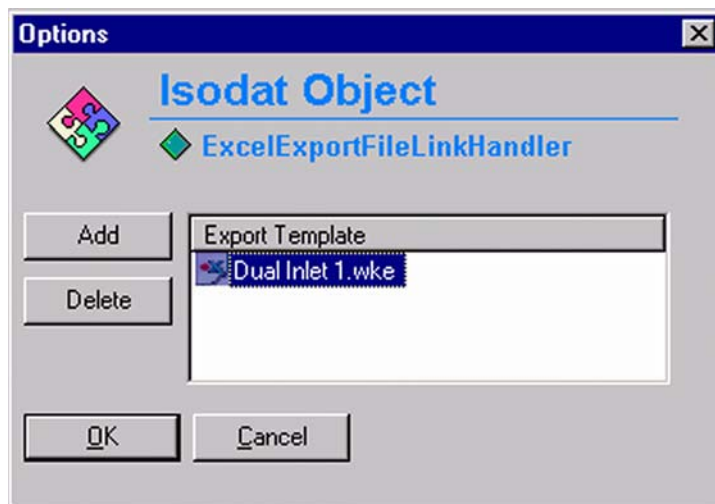
### Excel Export of Results

10. In the folder WK1 Export Templates, select your Export Template, for example **Dual Inlet 1.wke**. See [Figure 6-152](#).



**Figure 6-152.** Folder WK1 Export Templates

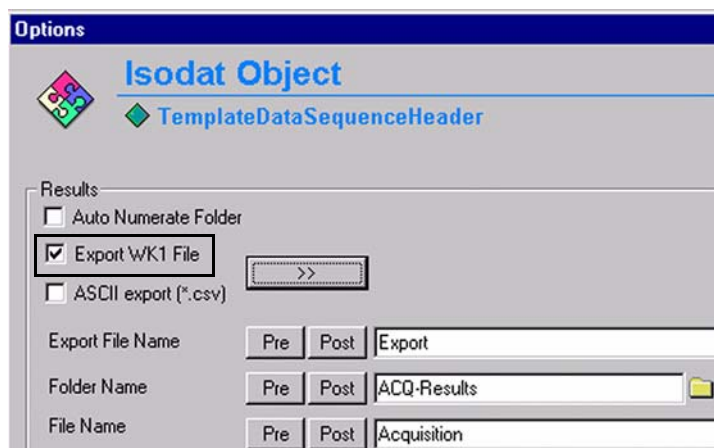
11. Click **Open**.
12. Select your Export Template (for example **Dual Inlet 1.wke**). See [Figure 6-153](#).



**Figure 6-153.** Selecting the export template

13. Confirm by **OK**.

The Export WK1 File box has now become active. See [Figure 6-154](#).



**Figure 6-154.** Export WK1 File check box has become active

14. Confirm by **OK**.

The options specified in the Sequence are now set and Data Acquisition starts.

**Note** The Excel Export will be performed online, that is during the measurement. This is different from a Re-evaluation, where the Excel Export takes place offline, that is after a measurement. ▲

## Measurements

Excel Export of Results

## Chapter 7 Diagnosis

This chapter gives instructions about performing various types of diagnosis. It contains the following topics:

- “Checking Performance Data” on page 7-2
- “How to Start Diagnosis” on page 7-3
- “Absolute Sensitivity” on page 7-4
- “Abundance” on page 7-11
- “Amplifier Test” on page 7-16
- “Compression Factor” on page 7-21
- “Linearity” on page 7-26
- “Peak Flatness” on page 7-30
- “Relative Sensitivity” on page 7-37
- “Resolution” on page 7-41
- “Signal Stability” on page 7-48
- “System Stability” on page 7-53
- “Conversion to Other Gases” on page 7-59
- “Heating Inlet Capillaries” on page 7-60
- “Replacing an Inlet Capillary” on page 7-62

## Checking Performance Data

Thermo Fisher Scientific has developed several test routines to check the performance data of the MAT 253 isotope ratio mass spectrometer.

For user's convenience, the Diagnosis program covering these test routines is included in the supplied version of the Isodat software. 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 (refer to “[Focusing](#)” on [page 1-56](#) about how to focus the ion source).

The Diagnosis program contains the following test routines:

- Absolute Sensitivity
- Abundance
- Amplifier Test
- Compression Factor
- Linearity
- Peak Flatness
- Relative Sensitivity
- Resolution
- Signal Stability
- System Stability

**Note** Reference gas for all performance data is CO<sub>2</sub>. Make sure to have properly filled CO<sub>2</sub> reservoirs attached to the inlet system before starting the Diagnosis program. ▲

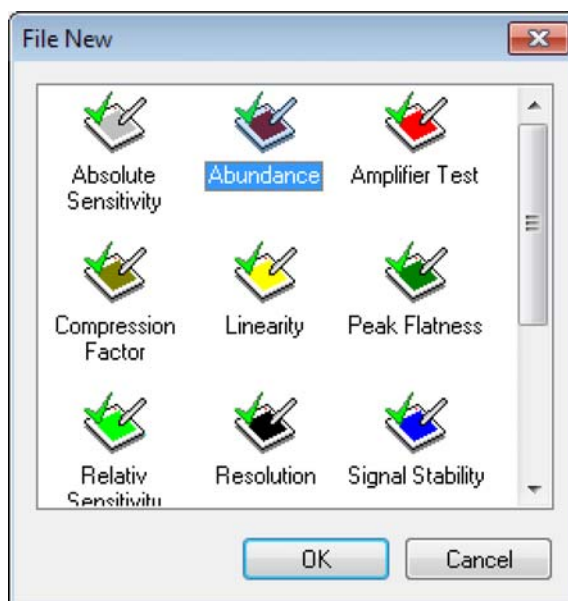
## How to Start Diagnosis

### ❖ To start Diagnosis

1. In the Isodat folder, double-click  to open the **Diagnosis** tool.

2. Click  to create a diagnosis test.

3. In the **File New** window, click the test routine to be performed, for example, **Abundance**, see [Figure 7-1](#).



**Figure 7-1.** Starting a diagnosis

4. Click **OK**.

## Absolute Sensitivity

**Note** Testing Absolute Sensitivity requires a Dual Inlet system. ▲

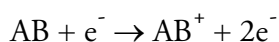
Absolute Sensitivity is defined as the number of molecules needed to generate an ion, which is then registered at the collector (for example one ion of mass 44 at the corresponding collector cup). It is thus dimensionless and measured in molecules per ion.

Based on a defined volume, the ion current is determined during a defined time period as a function of sample consumption (that is 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:

Ionization



Ionization and Dissociation



Definition of Absolute Sensitivity AS

$$AS = \frac{\Delta s}{n_{ion}} (\text{molecules/ion})$$

where:

$\Delta s$  Sample gas consumption

$n_{ion}$  Number of detected ions

The number of consumed sample molecules  $n$  is calculated via the ideal gas law:

$$pV = nRT$$

where:

$R$  Universal gas constant

$T$	Temperature
$p$	Pressure
$n$	Number of sample molecules
$V$	Volume (here 250 $\mu\text{L}$ )

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

## Test of Parameter

- Measurement starts with a determination of pressure and intensity.
- 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.
- The ion current is measured for the preset time.

**Note** Absolute Sensitivity should be about 800 molecules  $\text{CO}_2$  per mass 44 at the collector cup. ▲

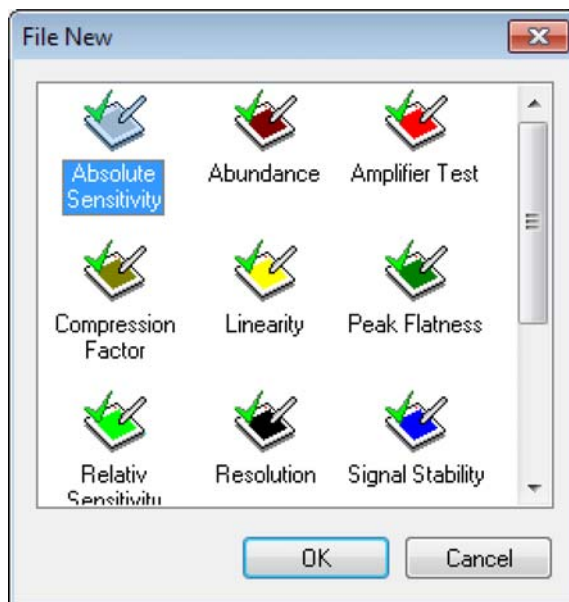
## Testing Absolute Sensitivity

### ❖ To test Absolute Sensitivity

1. In the Isodat folder, double-click  to open the **Diagnosis** tool.

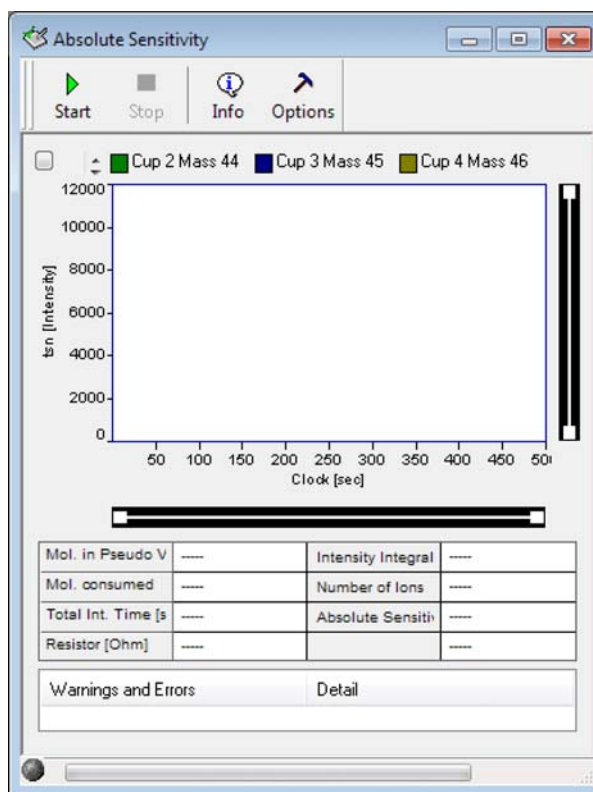
2. Click  to create a diagnosis test.

3. In the **File New** window, click **Absolute Sensitivity** and click **OK**. See [Figure 7-2](#).

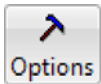


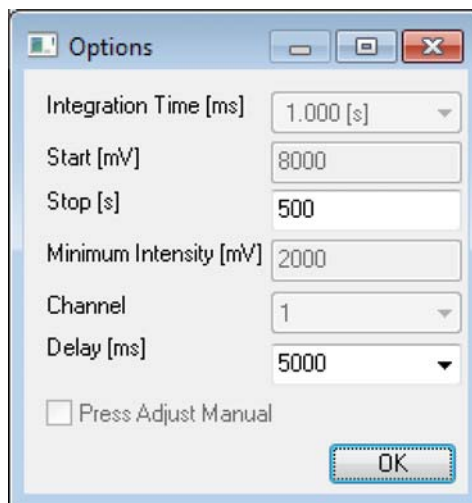
**Figure 7-2.** Opening Absolute Sensitivity

The Absolute Sensitivity window opens, see [Figure 7-3](#):



**Figure 7-3.** Absolute Sensitivity window


4. Click .
5. Enter a value for **Stop [s]**.
6. Select from the drop-down list a value for **Delay [ms]** between 100 and 20000 ms. See [Figure 7-4](#).



**Figure 7-4.** Options for Absolute Sensitivity

In Advanced Mode, all values can be edited. A recording **Channel** consists of an amplifier, a voltage-frequency (VF) converter and a counter. If the checkbox **Adjust Manual** is activated, pressure and peak center can be adjusted manually.

7. Click **OK**.

8. Click  .  
Testing starts with a Peak Center on the selected Cup, for example mass 45, Cup3, see [Figure 7-5](#).

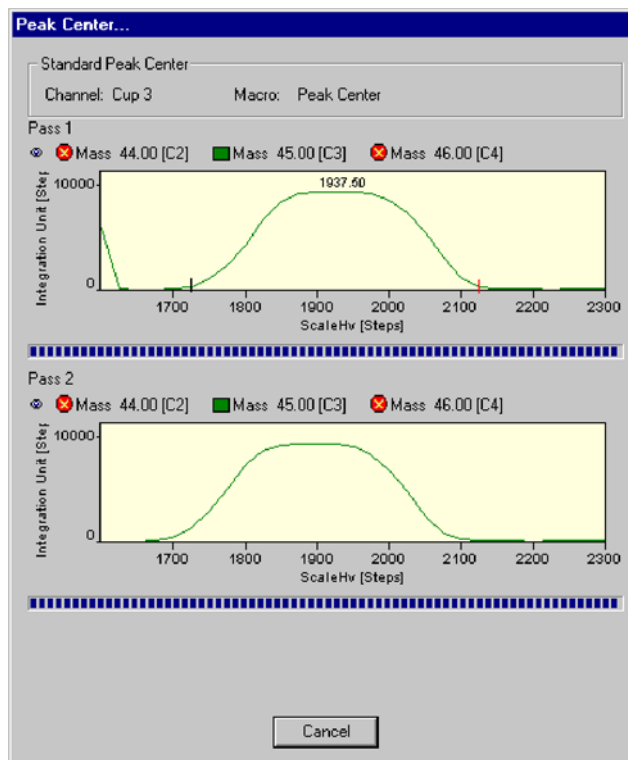
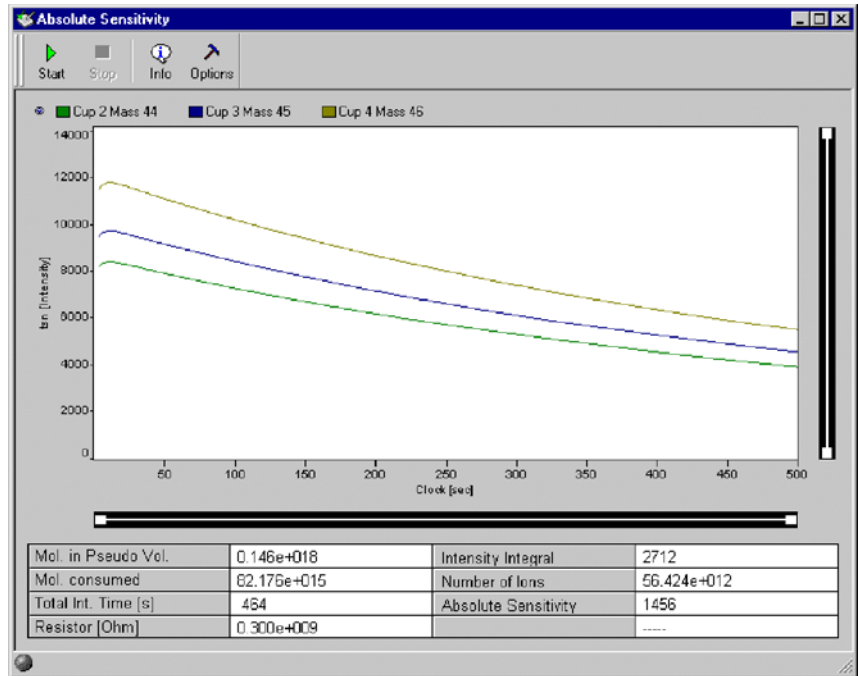



Figure 7-5. Peak Center

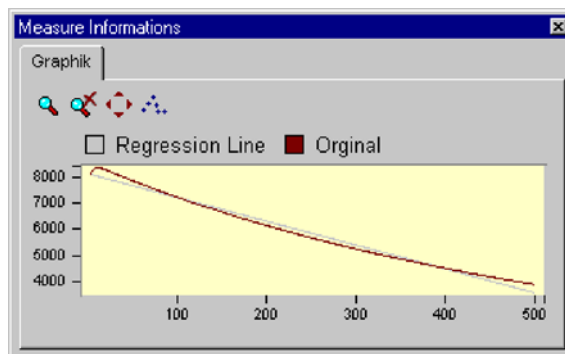
The bellows is reduced stepwise until the start level intensity, for example, 8000 mV, is reached. Then, intensity and pressure are measured for the preset time, for example 500 s. See Absolute Sensitivity window, [Figure 7-6](#).



**Figure 7-6.** Absolute Sensitivity window

Finally, Absolute Sensitivity and other parameters are calculated.

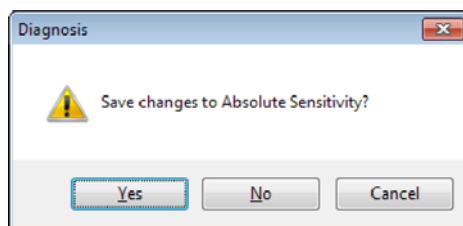
9. Click  .  
Original measurement and regression line are shown. See [Figure 7-7](#).



**Figure 7-7.** Measurement Information

10. Close the **Information** window and the **Absolute Sensitivity** window.

11. Confirm the message window [Figure 7-8](#) as appropriate.



**Figure 7-8.** Saving changes to Absolute Sensitivity

## Abundance

Abundance denotes the contribution of a mass to a neighbor mass (for example the amount of ions for mass 44 falling into cup 45). Thus, the intensity of a mass (for example mass 44) is compared to the intensity of the neighboring peak (for example mass 45).

In case of CO<sub>2</sub>, 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  $2 \times 10^{-6}$  for a MAT 253 with Dual Inlet system. The Abundance test is performed with CO<sub>2</sub> and the device must be calibrated. Peak center is performed on Channel 2 (that is narrow cup) before the measurement starts.

## Test of Parameter

- Signal height (that is intensity) on mass 44 is determined in the appropriate collector cup (for example Cup 3).
- The background signal on mass 45 is measured (that is electronic noise with no gas) from mass 44 to mass 45.5.
- With CO<sub>2</sub> 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 (for example Cup 4), with bigger resistor value in order to keep the signal in the detection range).
- The abundance (of mass 44 onto mass 45) from the signal to the left and to the right of mass 45 peak is extrapolated.


## Sources of Error

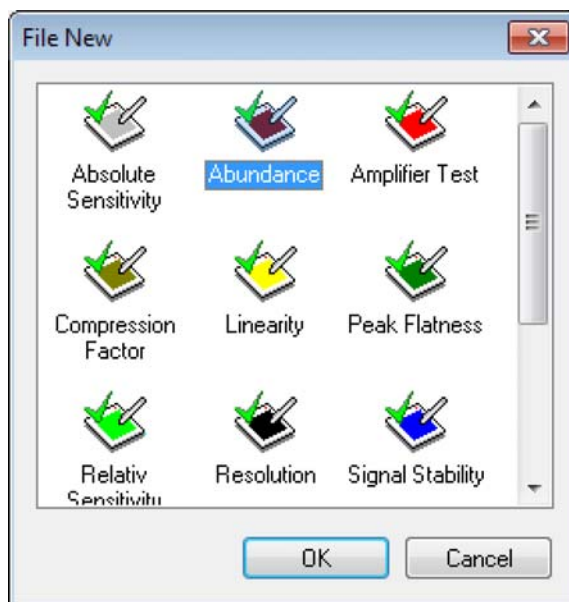
- Resistor values are not configured correctly.
- Due to electrons on the left and on the right side of the peak, a negative signal may result. This problem can be overcome by manual adjustment.

## Testing Abundance

### ❖ To test Abundance

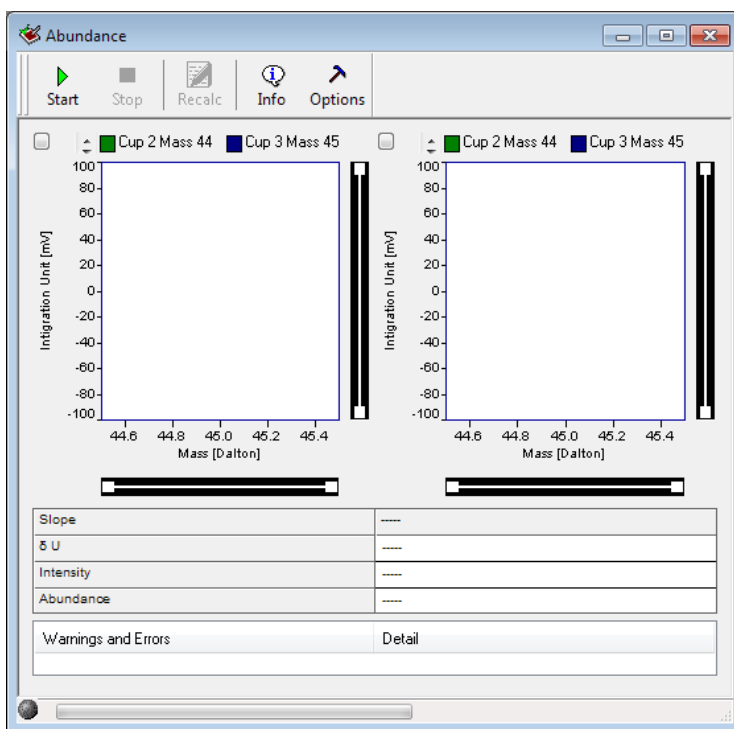
1. In the Isodat folder, double-click  to open the **Diagnosis** tool.

2. Click  to create a diagnosis test.
3. In the **File New** window, click **Abundance** and click **OK**. See [Figure 7-9](#).

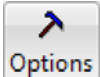


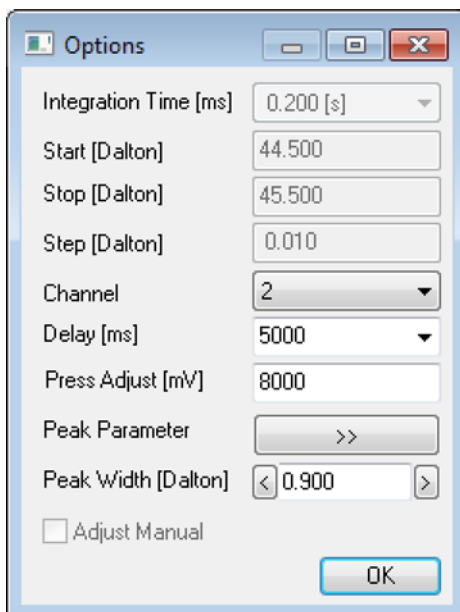
**Figure 7-9.** Opening Abundance

The **Abundance** window opens, see [Figure 7-10](#). The left window detects mass 44 ions in the correct cup. The right window detects mass 44 ions in the neighbor cup (that is cup of mass 45).



**Figure 7-10.** Abundance window

4. Click  **Options**.
- The **Options** window opens, See [Figure 7-11](#).



**Figure 7-11.** Options for Abundance

5. 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 (for example channel 2 from the pulldown list).

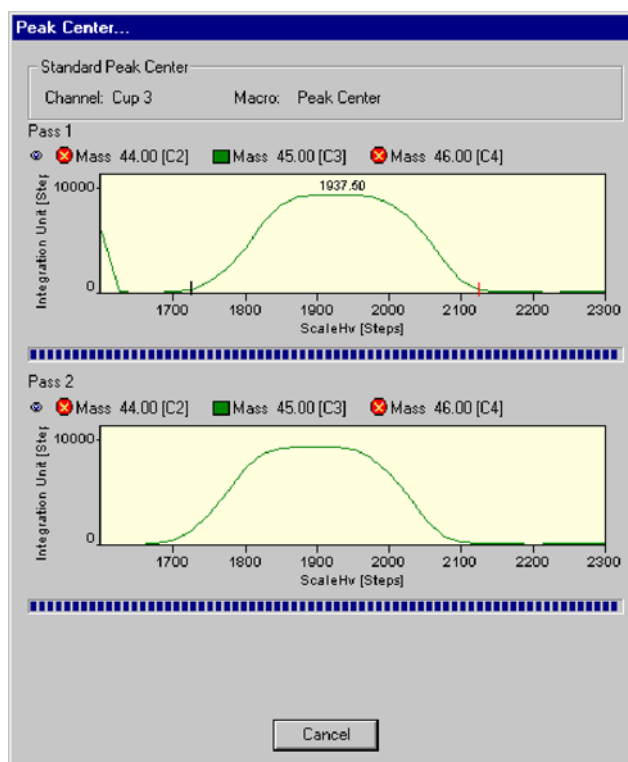
6. Choose a **Delay [ms]** value between 100 ms and 20000 ms to define measurement start delay.

In Standard Mode, the inactive variables cannot be edited. The Advanced Mode allows experienced users to set the start mass for the magnet field scan (**Start [Dalton]**), the end mass, where the magnet stops scanning (**Stop [Dalton]**), and the step width for the magnet scan (**Step [Dalton]**). If the checkbox **Adjust Manual** is activated, signal intensity and peak center must be performed manually.

7. Click **OK**.



8. Click **Start** to start the Abundance test.  
Testing starts with a Peak Center on the selected cup (for example mass 45, cup 3), see [Figure 7-12](#):

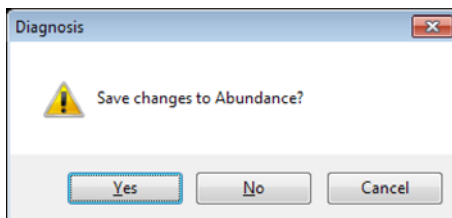


**Figure 7-12.** Peak Center for Abundance

9. It might be helpful to activate the Advanced Mode. Click the **Options** button and expand the interval between Start and Stop

values, that is decrease **Start [Dalton]** and increase **Stop [Dalton]** values). Values for Abundance, Intensity,  $\delta U$  and slope are displayed.

10. Close the **Abundance** window.
11. Confirm the message window [Figure 7-13](#) as appropriate.



**Figure 7-13.** Saving changes to Abundance

## Amplifier Test

The Amplifier Test (formerly called UFC-Test) checks the ion detection performance of the IRMS with no ions present. It informs about the background noise of electronic devices.

The amplifier baseline must be determined without an interfering signal. The ion source is switched off before measurement starts (that is 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.

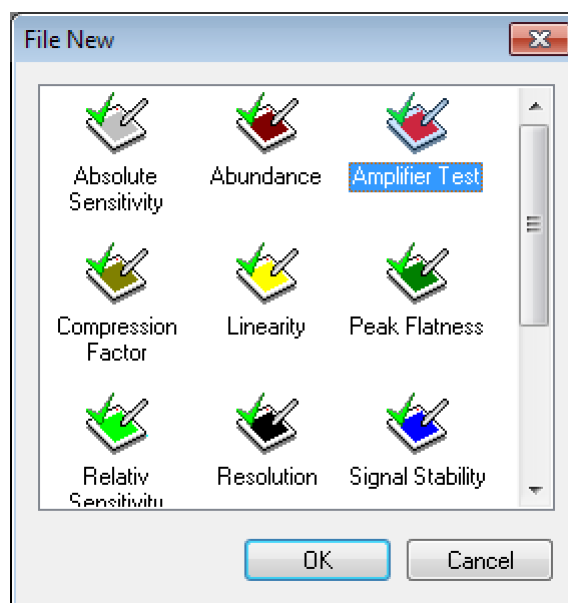
## Testing Amplifiers

### ❖ To test the amplifiers

1. In the Isodat folder, double-click  to open the **Diagnosis** tool.

2. Click  to create a diagnosis test.

3. In the **File New** window, click **Amplifier Test** and click **OK**. See [Figure 7-14](#).



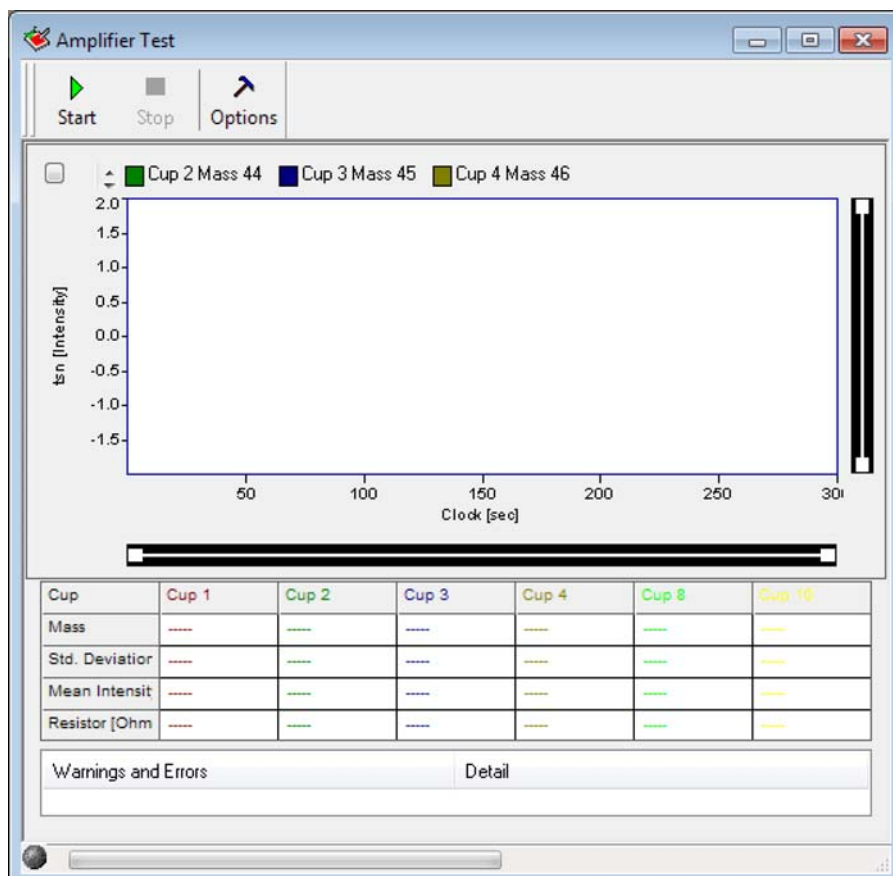
**Figure 7-14.** Opening Amplifier Test

4. Wait, while the available gas configurations are scanned., see [Figure 7-15](#).

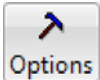


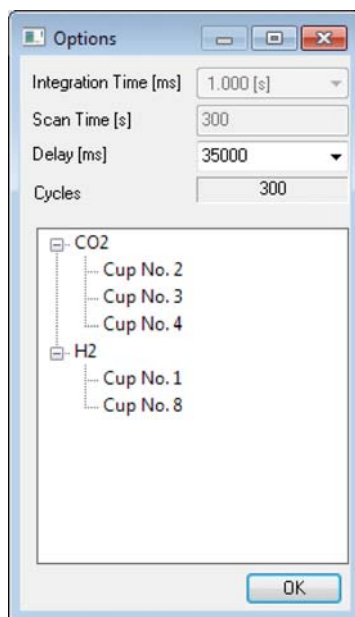
**Figure 7-15.** Scanning available gas configurations

The Amplifier Test window opens, see [Figure 7-16](#):



**Figure 7-16.** Amplifier Test window

5. Click  **Options** ..  
The Options window opens, see [Figure 7-17](#).



**Figure 7-17.** Options for Amplifier Test

With the exception of **Delay**, all parameters are preset and cannot be changed in Standard Mode. The Advanced Mode allows experienced users to modify all parameters, for example, accept the default value of **Integration Time [ms]**. The **Scan Time [s]** sets the duration of the scan. The measurement start is delayed by the displayed default value of **Delay [ms]** (value between 100 and 35 000 ms). **Cycles** defines the number of measurements. The intensities of the channel are measured for indicated cycles with a definite cycle time.

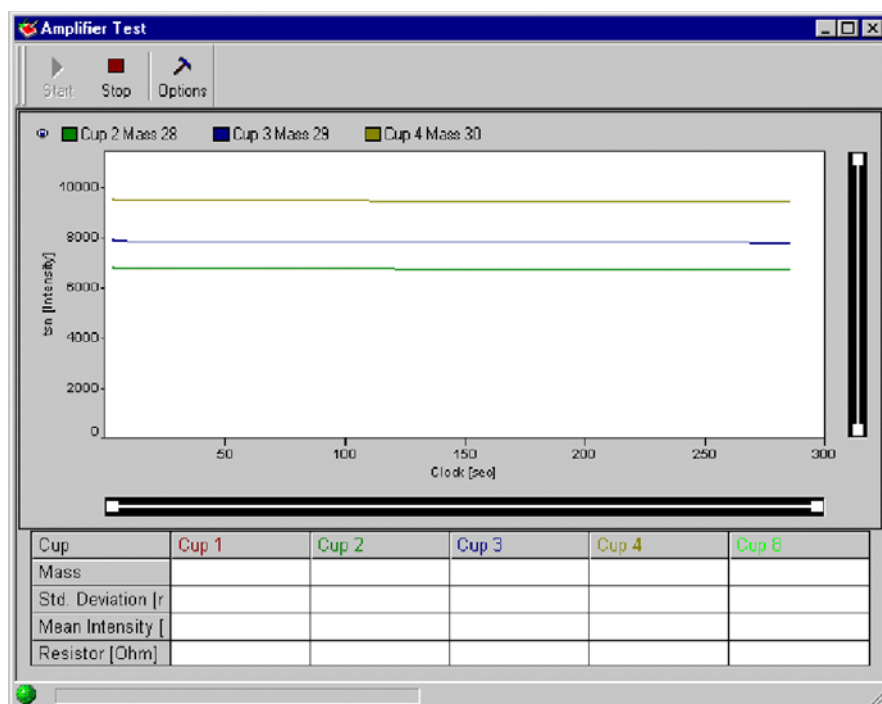
6. Click **OK**.

7. Click .



8. Switch off High Voltage.

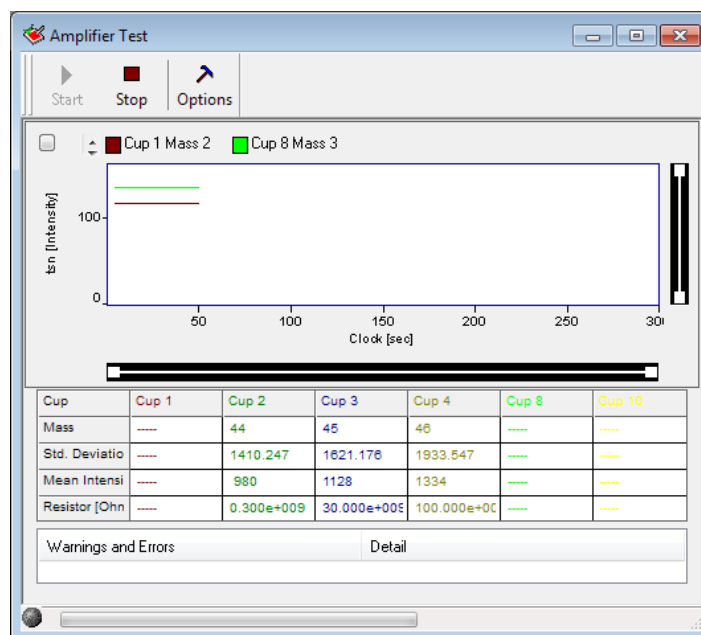
The correspondent intensity vs. time diagram is shown, see [Figure 7-18.](#):



**Figure 7-18.** Amplifier Test window intensity vs. time diagram

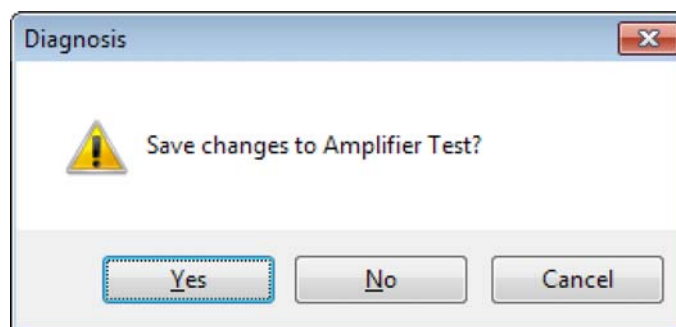
The baselines of the cups are shown with no ions present. Standard deviation [mV], mean [mV] and resistance [Ohm] of each selected cup are displayed.

The procedure is repeated for all amplifiers present in the system, see [Figure 7-19](#):



**Figure 7-19.** Amplifier Test window repetition for other masses

9. Close the **Amplifier Test** window.
10. Confirm the message window [Figure 7-20](#) as appropriate.



**Figure 7-20.** Saving changes to Amplifier Test

## 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]:

$$\text{Comp} = \frac{\text{Int}_{\text{end}}}{\text{Int}_{\text{start}}}$$

where:

*Int<sub>end</sub>* Intensity at the end of measurement

*Int<sub>start</sub>* Intensity at measurement start

The Compression Factor determines the dynamic range of the two bellows informing about their tightness and linearity. The ion signal (that is intensity) is measured at different bellows compressions: an intensity vs. volume diagram results. The standard deviation around the mean value of the signal is calculated.

**Note** The bellows must be calibrated before performing the test. ▲

To test this parameter, the peak intensity for a mass (for example 44) is measured starting at the maximum (that is 100%) down to the minimum (that is 0%). A minimum death volume of about 3 mL is still remaining at 0% volume. A certain level (for example 200 mV) serves as starting point of the measurement. The signal for the bellows 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. ▲

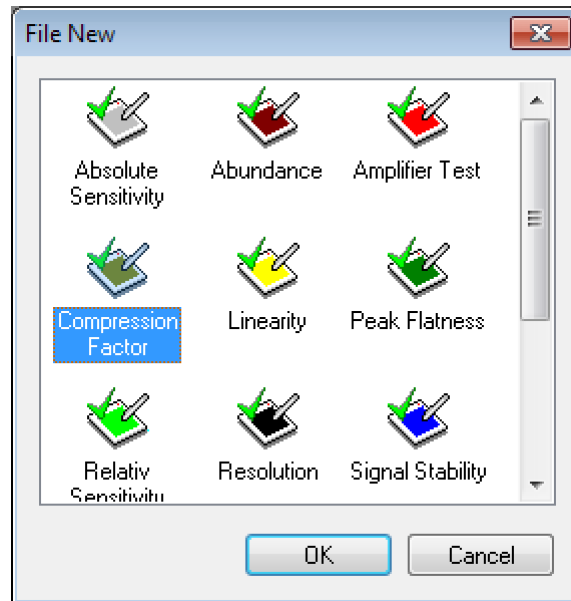
## Testing the Compression Factor

❖ **To test the Compression Factor**

1. In the Isodat folder, double-click  to open the **Diagnosis** tool.

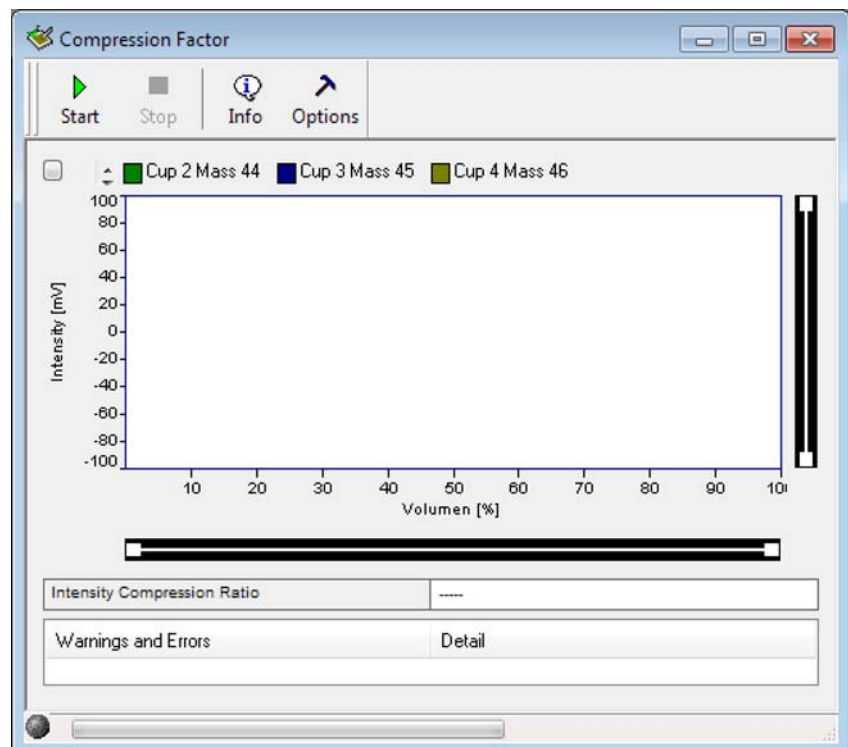
2. Click  to create a diagnosis test.

3. In the **File New** window, click **Compression Factor** and click **OK**. See [Figure 7-14](#).



**Figure 7-21.** Opening Compression Factor

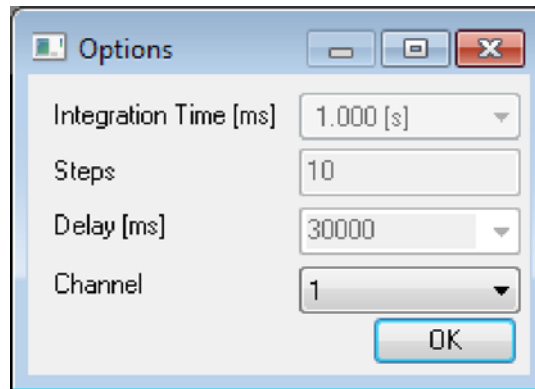
The Compression Factor window opens, see [Figure 7-22](#):



**Figure 7-22.** Compression Factor window

4. Click  **Options**.

In Standard Mode, you are only able to select a channel from the pull-down menu. See [Figure 7-23](#).



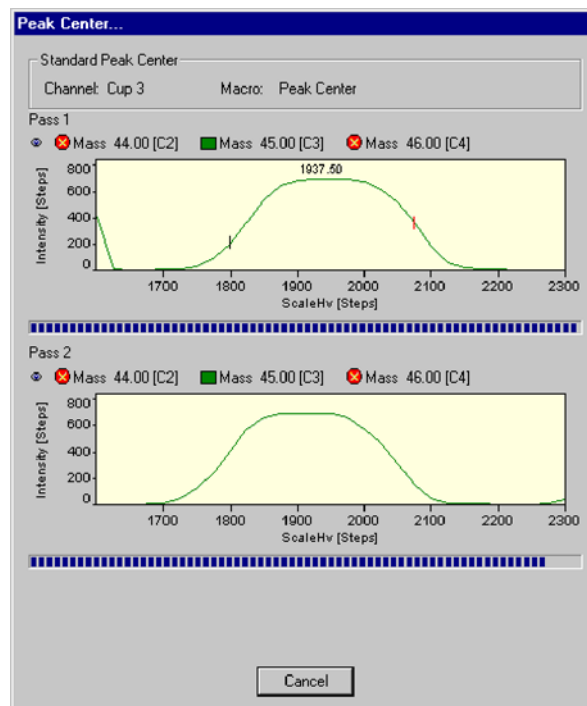
**Figure 7-23.** Options for Compression Factor

In Advanced Mode, the other variables can also be edited. The volume of the bellows is varied stepwise, for example in 10 **Steps**. The measurement start is delayed by the displayed value for **Delay [ms]**.

5. Click **OK**.

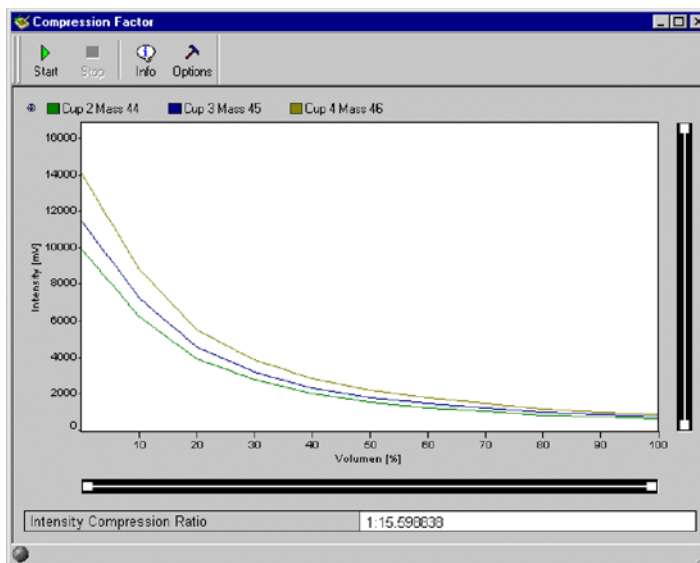


6. Click **Start**.  
Testing starts with a Peak Center for the first bellows, for example, Sample, see [Figure 7-24](#).



**Figure 7-24.** Peak Center for Compression Factor

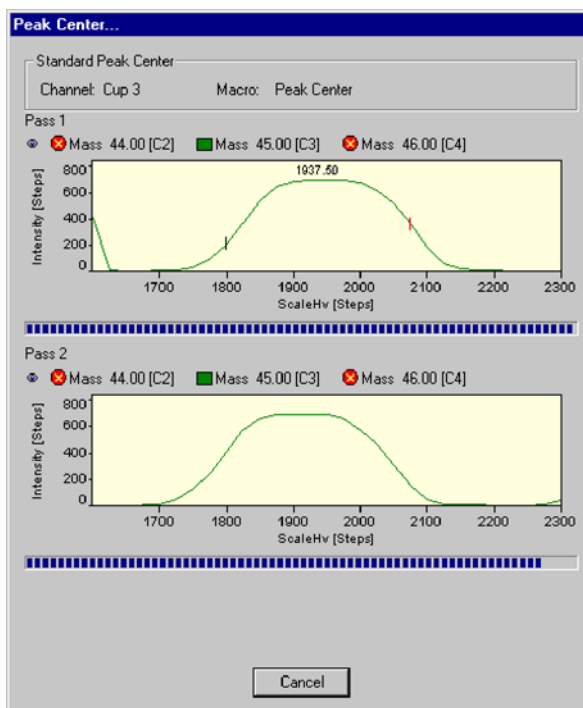
When the Peak Center is finished, signal intensity is shown during the volume change of the bellows for the different masses, see [Figure 7-25](#):



**Figure 7-25.** Compression Factor window

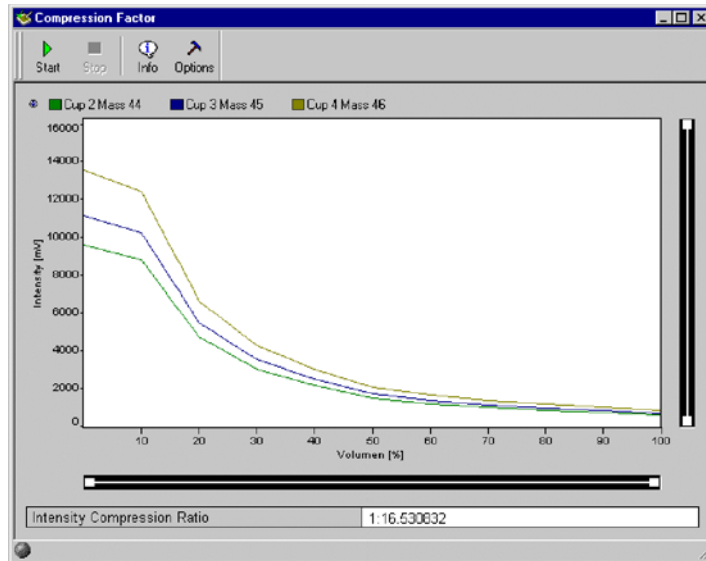
Finally, the Intensity-Compression ratio is calculated for the first bellows, for example, Sample.

The procedure is repeated for the second bellows, for example Standard, and a Peak Center is performed, see [Figure 7-26](#):



**Figure 7-26.** Peak Center

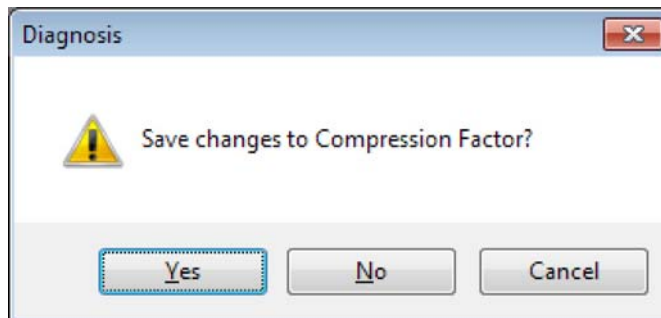
When the Peak Center is finished, again signal intensity is shown during the volume change of the bellows for the different masses, see [Figure 7-27](#):



**Figure 7-27.** Compression Factor window

The Intensity-Compression ratio is calculated for the second bellows (for example Standard).

7. Close the **Compression Factor** window.
8. Confirm the message window [Figure 7-20](#) as appropriate.



**Figure 7-28.** Saving changes to Compression Factor

## Linearity

At Linearity test, synonymously called Ratio Linearity, over a range of varying signals, signal linearity is checked vs. beam intensity (that is 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.

**Note** Source of error: Resistor values must be configured correctly. ▲

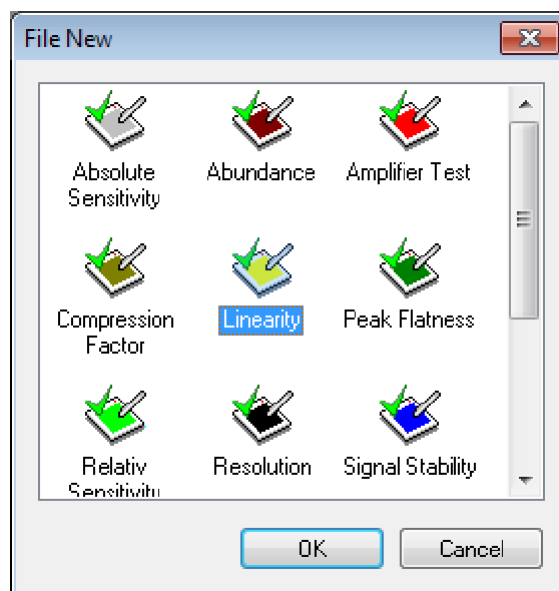
## Testing Linearity

### ❖ To test Linearity

1. In the Isodat folder, double-click  to open the **Diagnosis** tool.

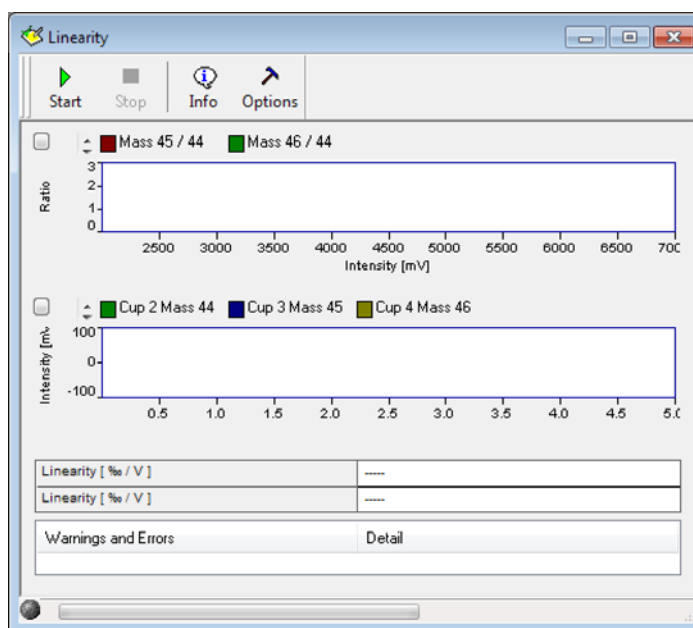
2. Click  to create a diagnosis test.

3. In the **File New** window, click **Linearity** and click **OK**. See [Figure 7-29](#).

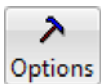


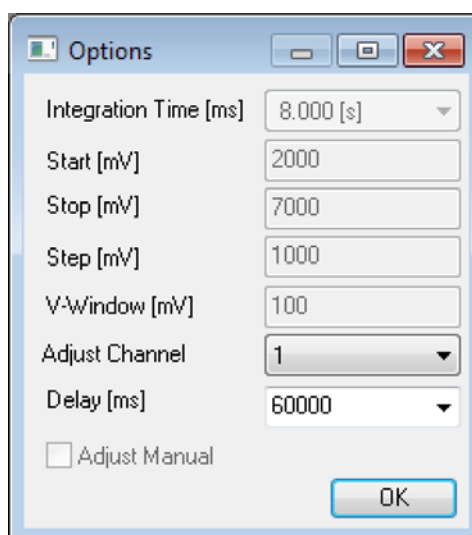
**Figure 7-29.** Opening Linearity

The Linearity window opens, see [Figure 7-30](#):



**Figure 7-30.** Linearity window

4. Click  **Options**.
5. From the drop-down lists, select a channel for **Adjust Channel** and a value between 100 ms and 20000 ms for **Delay [ms]**, see [Figure 7-31](#).



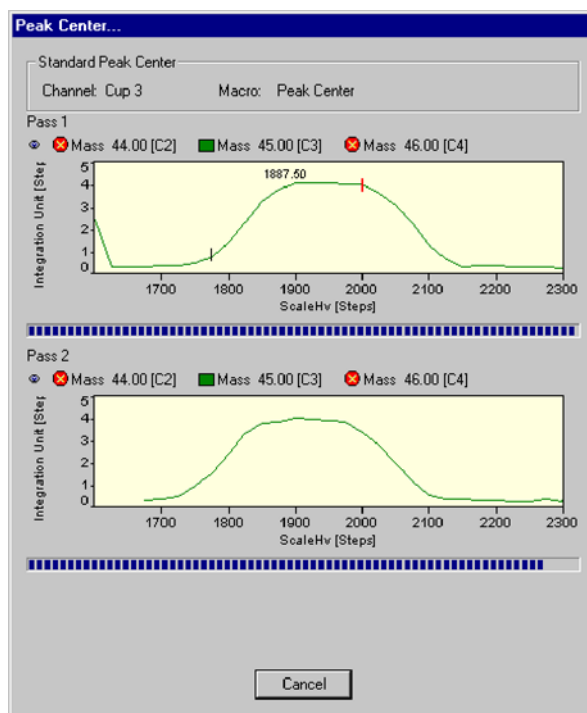
**Figure 7-31.** Options for Linearity

The other variables are preset in Standard Mode (to be accepted), but editable in Advanced Mode. If the intensities (voltage steps) between 2 V and 8 V are to be set manually, **Adjust Manual** must be selected.

6. Click **OK**.

7. Click  .

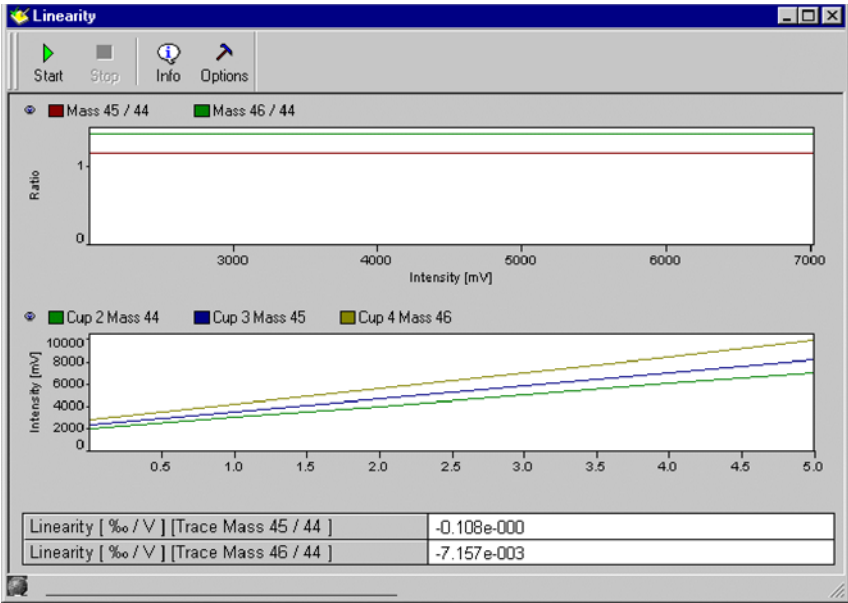
Testing starts with a Peak Center on cup 3 as the narrow cup, see [Figure 7-32](#).



**Figure 7-32.** Peak Center


If no peak could be found, the measurement is terminated.

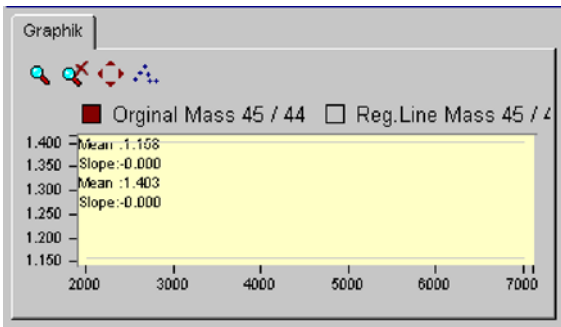
Two linearity values [%/V] are calculated, one for mass 45/44 and one for mass 46/44., see [Figure 7-33](#):



**Figure 7-33.** Linearity window

**Note** Linearity must be less than 0.06 %/V! ▲

8. Click . Mean and slope values are displayed, see [Figure 7-34](#).



**Figure 7-34.** Info window Linearity

## 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 (that is 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 (that is at a parameterized mass range around the center) is measured. Peak Flatness can be determined for different gases and different collector cups (for example for a CO<sub>2</sub>-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]

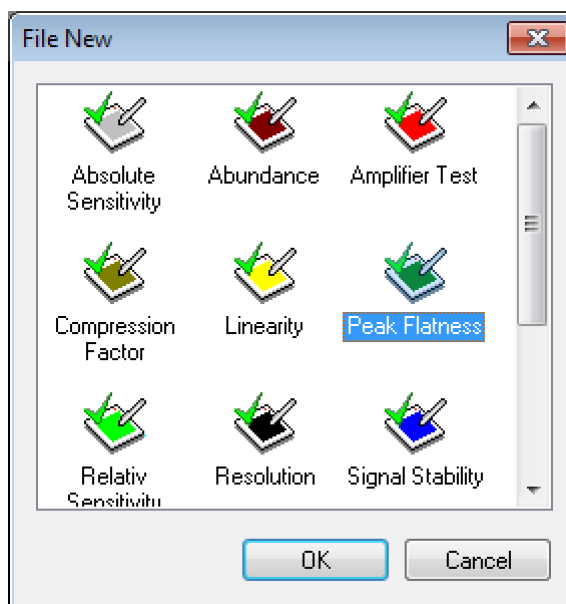
## Testing Peak Flatness

### ❖ To test Peak Flatness

1. In the Isodat folder, double-click  to open the **Diagnosis** tool.

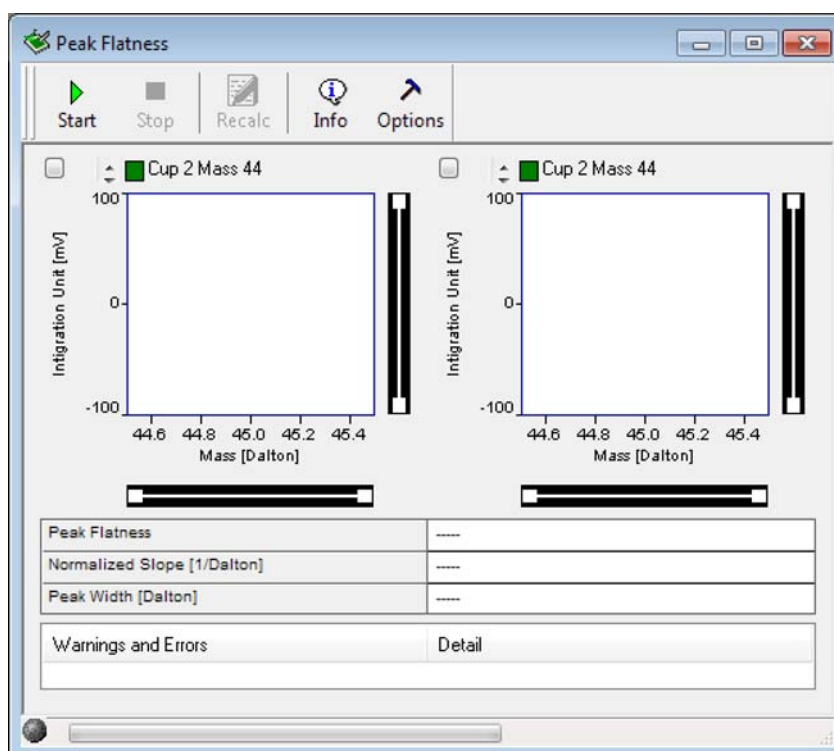
2. Click  to create a diagnosis test.

- In the **File New** window, click **Linearity** and click **OK**. See [Figure 7-35](#).



**Figure 7-35.** Opening Peak Flatness

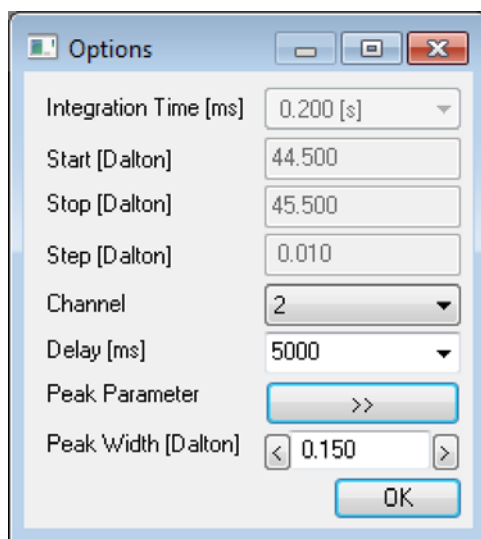
The Peak Flatness window opens, see [Figure 7-36](#):




**Figure 7-36.** Peak Flatness window

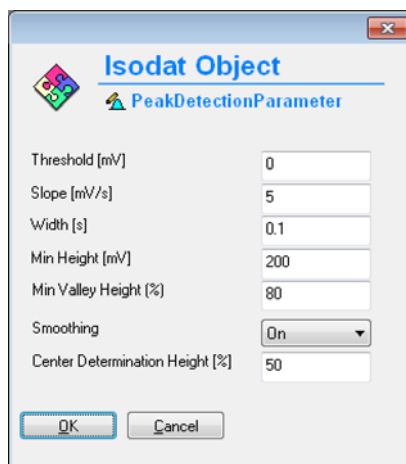
- Click  **Options**.

5. Select a **Channel** and a **Delay [ms]** value between 100 ms and 20000 ms. Select an appropriate value for **Peak Width [Dalton]**, see [Figure 7-37](#).



**Figure 7-37.** Options for Peak Flatness

6. Click  next to **Peak Parameter** to open the object window, see [Figure 7-38](#).



**Figure 7-38.** Peak detection parameters

7. Change the values according to your needs.
8. Click **OK**.

9. Click  .

Testing starts with a Peak Center determination on the selected cup (for example mass 45, cup 3), see [Figure 7-39](#).

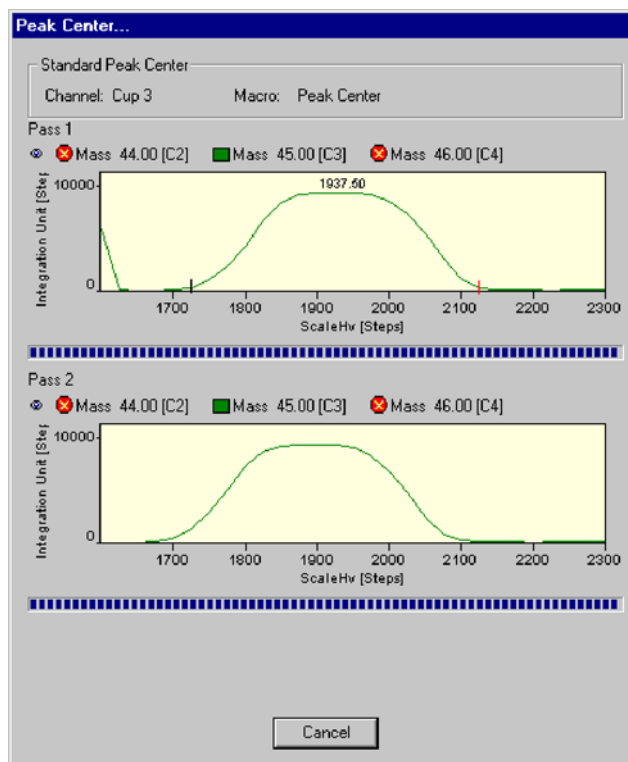


Figure 7-39. Peak Center

With the aid of the magnet field, the peak is scanned and the peak width is determined.

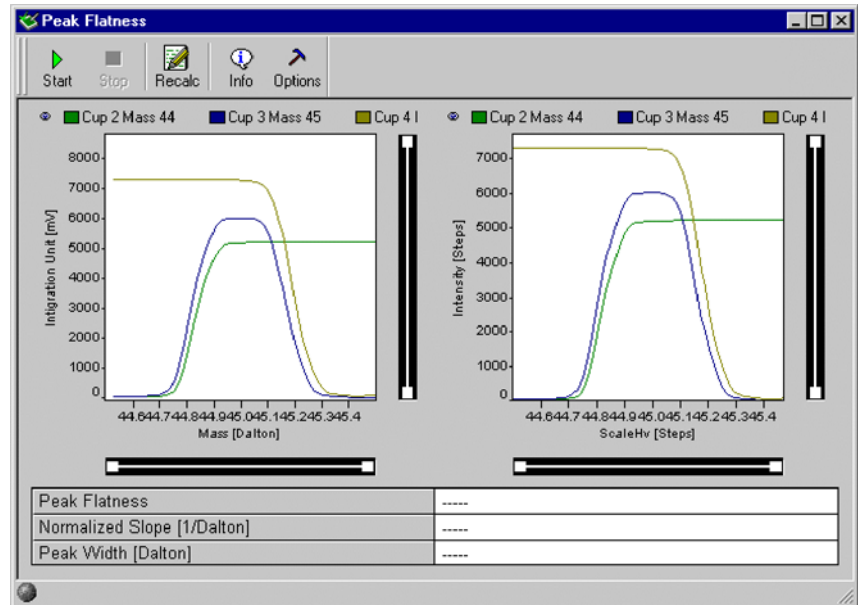


Figure 7-40. Peak Flatness window

A Peak Center determination for Energy Correction is performed, see Figure 7-41.

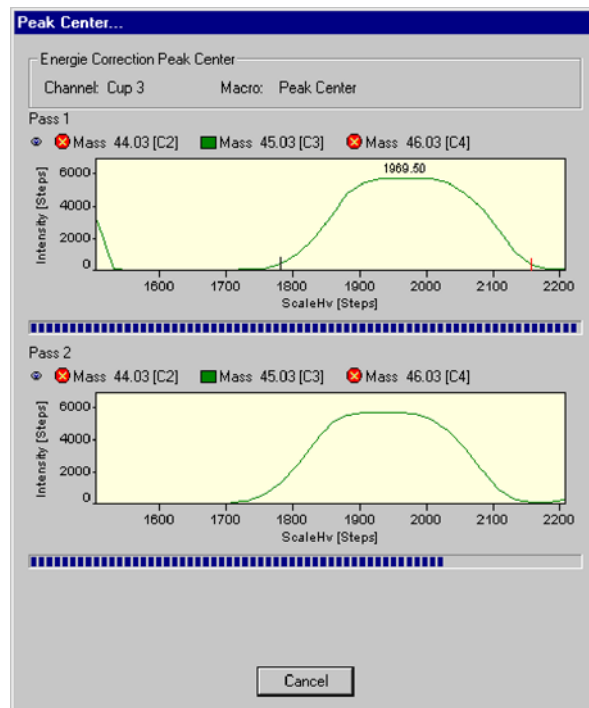


Figure 7-41. Peak Center for Energy Correction

10. If no Peak Center can be found, the measurement is stopped. In this case, click **Cancel** to proceed.

Values for Peak Flatness, normalized slope [ $\text{Da}^{-1}$ ] and Peak Width [Da] are calculated, see Figure 7-42:

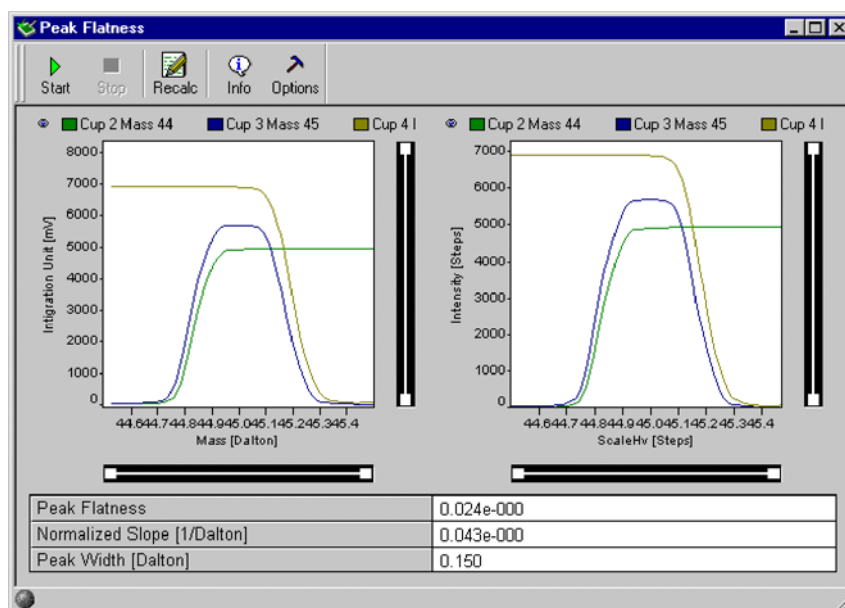


Figure 7-42. Peak Flatness window

11. Click .

The first tab of the Information window opens, see Figure 7-43.

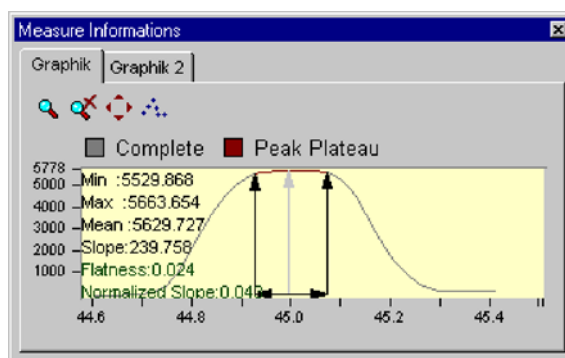
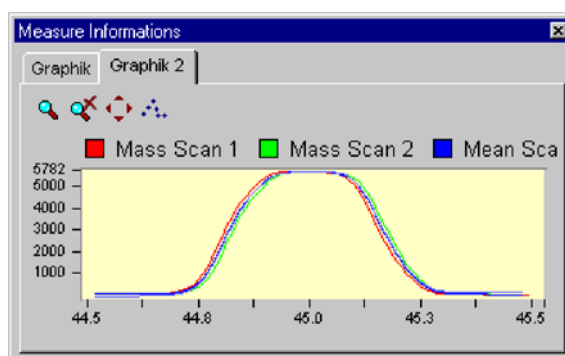


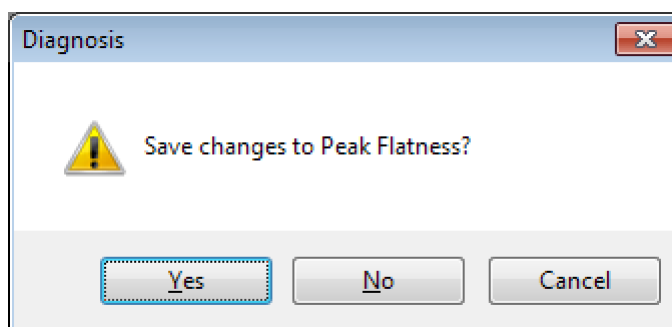
Figure 7-43. Information page, first tab

The second tab of Information window is shown in [Figure 7-44](#).



**Figure 7-44.** Information page, second tab

12. Close the **Peak Flatness** window.
13. Confirm the message window [Figure 7-45](#) as appropriate.



**Figure 7-45.** Saving changes to Peak Flatness

## Relative Sensitivity

Relative Sensitivity ( $S_{rel}$ ) describes the dependency of signal intensity (that is 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 (for example Cup 3, mass 44)
- $R$  Resistor value (for example  $3 \times 10^8 \Omega$  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.
- $\Delta p$  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 (that is 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 for example 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 (that is 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, which is limited.

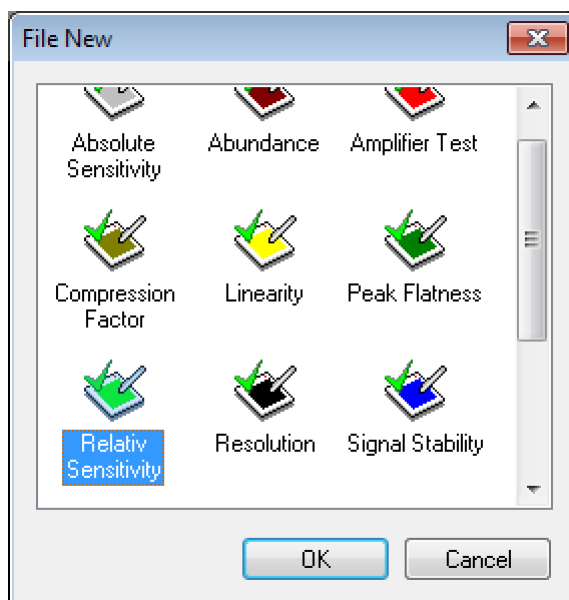
## Testing Relative Sensitivity

### ❖ To test Relative Sensitivity

1. In the Isodat folder, double-click  to open the **Diagnosis** tool.

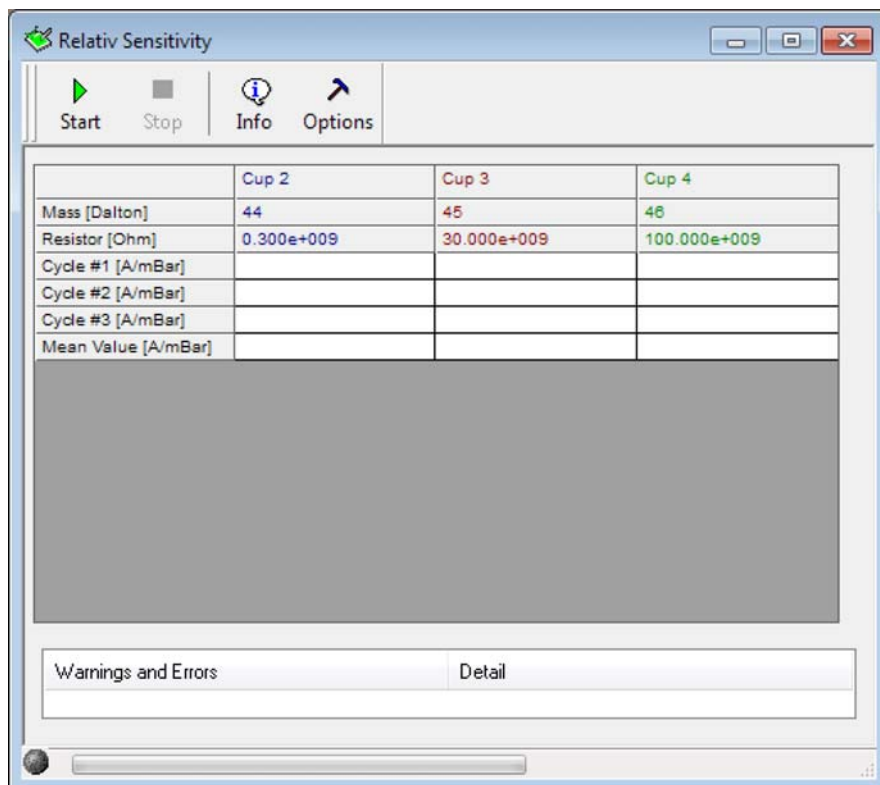
2. Click  to create a diagnosis test.

3. In the **File New** window, click **Relative Sensitivity** and click **OK**. See [Figure 7-46](#).

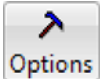


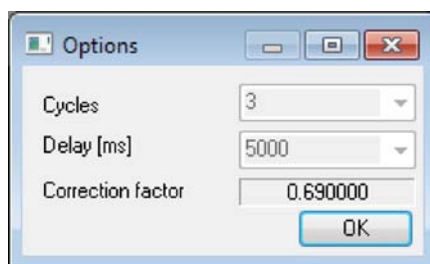
**Figure 7-46.** Opening Relative Sensitivity

The cups with the corresponding masses and resistor values are shown. See [Figure 7-47](#).




**Figure 7-47.** Relative Sensitivity window

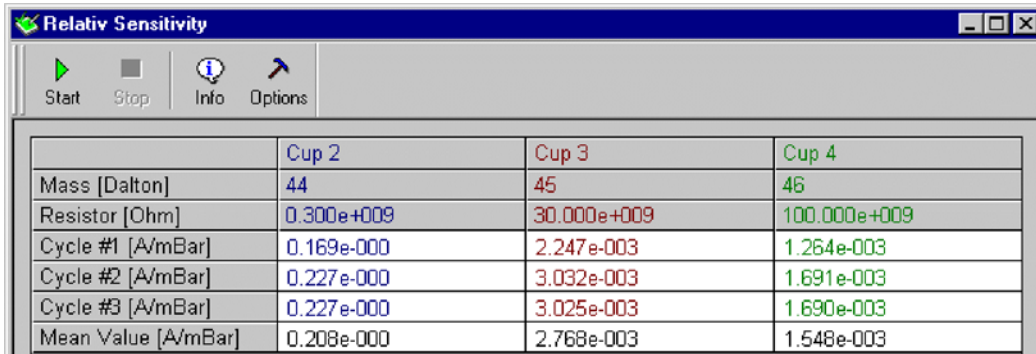
4. Click  **Options**.
5. Click **OK**.  
The Options window opens, see [Figure 7-48](#).



**Figure 7-48.** Options for Relative Sensitivity

Experienced users can change the values via the Advanced Mode.

6. Click  .  
For each cup, the values of three cycles and their mean are displayed, see [Figure 7-49](#).

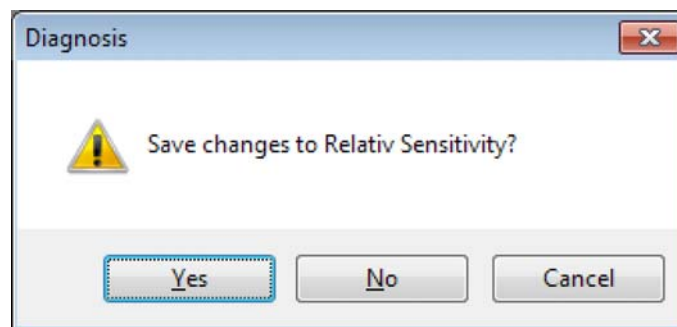


The screenshot shows a window titled "Relative Sensitivity" with a toolbar containing "Start", "Stop", "Info", and "Options" buttons. Below the toolbar is a table with the following data:

	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

**Figure 7-49.** Relative Sensitivity window

7. Close the **Relativ Sensitivity** window.  
8. Confirm the message window [Figure 7-50](#) as appropriate.

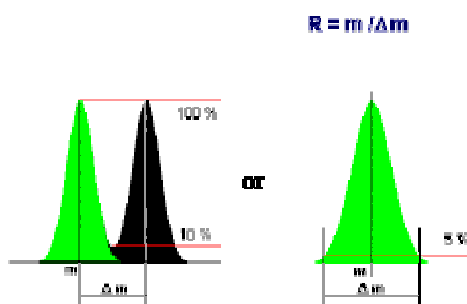


**Figure 7-50.** Saving changes to Relativ Sensitivity

## Resolution

Resolution describes the masses, which can be separated from each other (that is the minimal relative distance between two masses, which can be resolved). Different definitions of resolution are used in mass spectrometry.

The 10% valley definition, which is commonly used for double focusing sector field mass spectrometers, means:



**Figure 7-51.** 10% valley definition

Resolution can be defined as:

- Mass divided by the mass difference of two neighboring peaks, if the valley between the peaks drops to 10% of the peak height.
- Or mass divided by the peak width (in Dalton) at 5% of peak height.

According to the 10% valley definition, it is dimensionless (given in  $m/\Delta m$ ):

$$R = \frac{m}{\Delta m}$$

where:

- $R$  Resolution  
 $m$  Mass of the interesting isotope  
 $\Delta m$  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):

$$R = \frac{m \cdot a}{\Delta m \cdot b}$$

where:

- a* Distance between peak centers
- b* Peak width of the isotope of interest

Resolution is determined using the narrowest cup (that is 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<sub>2</sub>, 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.

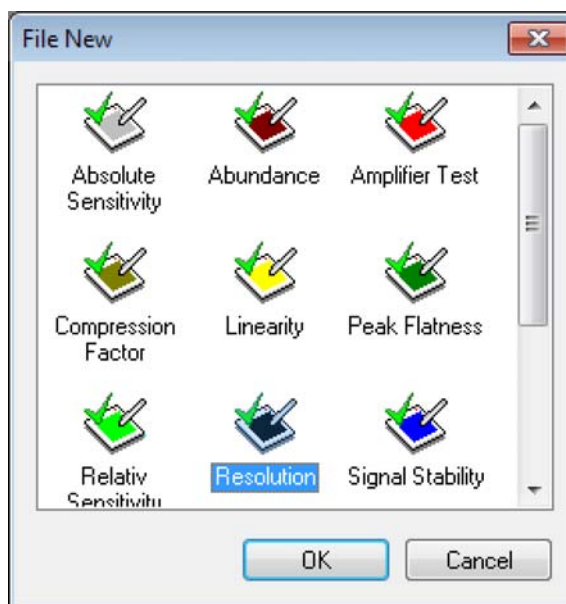
## Testing Resolution

### ❖ To test the Resolution

1. In the Isodat folder, double-click  to open the **Diagnosis** tool.

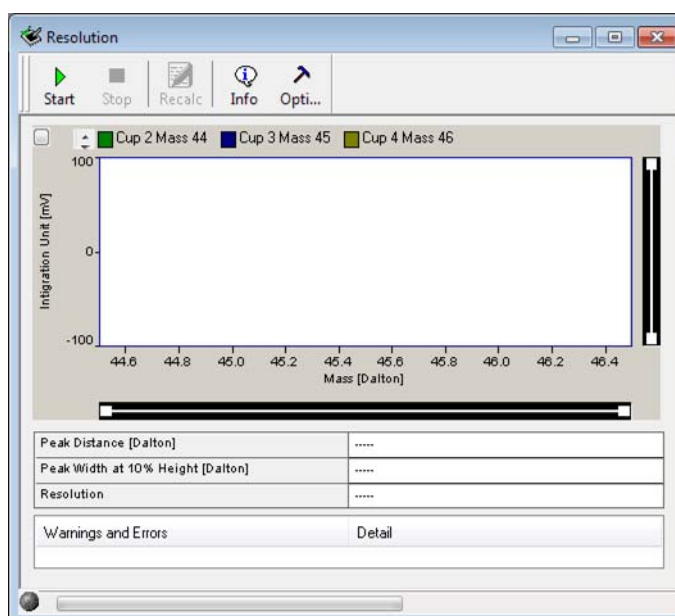
2. Click  to create a diagnosis test.

3. In the **File New** window, click **Resolution** and click **OK**. See [Figure 7-52](#).



**Figure 7-52.** Opening Resolution

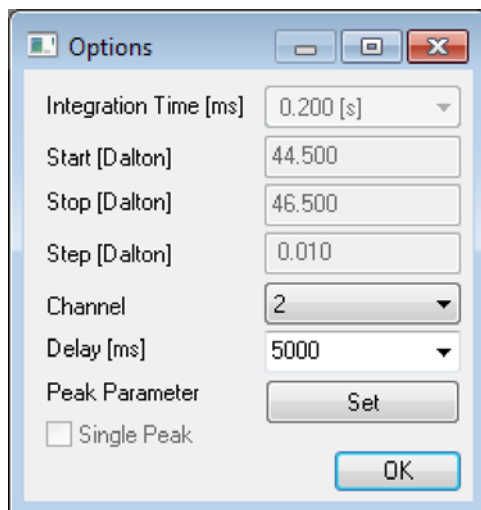
The **Resolution** window opens, see [Figure 7-53](#):



**Figure 7-53.** Resolution window

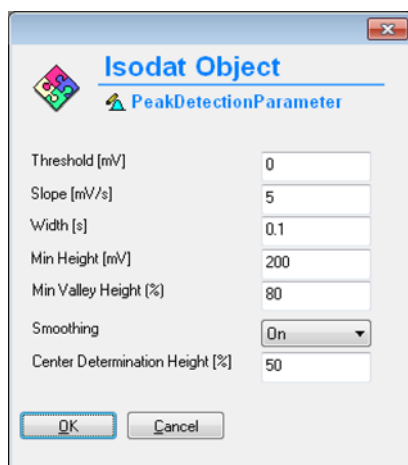
4. Click  **Options**.

5. Select a **Channel** and a **Delay [ms]** value between 100 ms and 20000 ms, see [Figure 7-54](#).



**Figure 7-54.** Options for Resolution

6. Click **Set** next to **Peak Parameter** to open the object window, see [Figure 7-55](#).

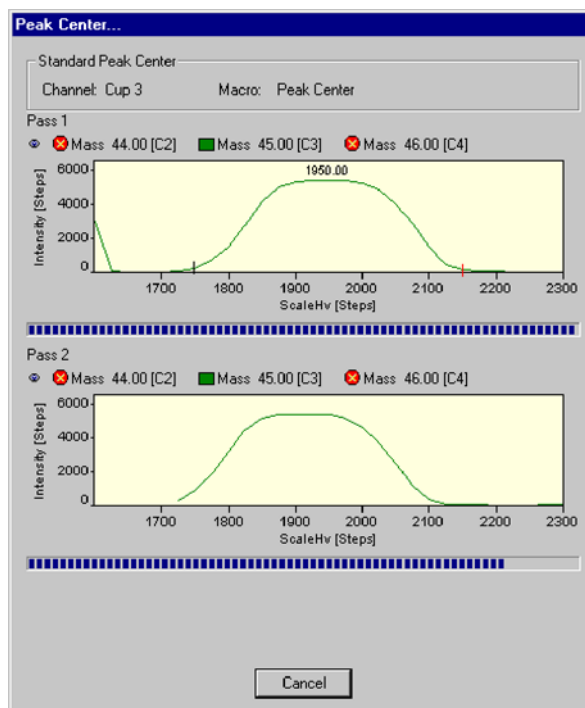


**Figure 7-55.** Peak detection parameters

7. Change the values according to your needs.
8. Click **OK** to close the Isodat Object window.
9. Click **OK** to close the Options window.

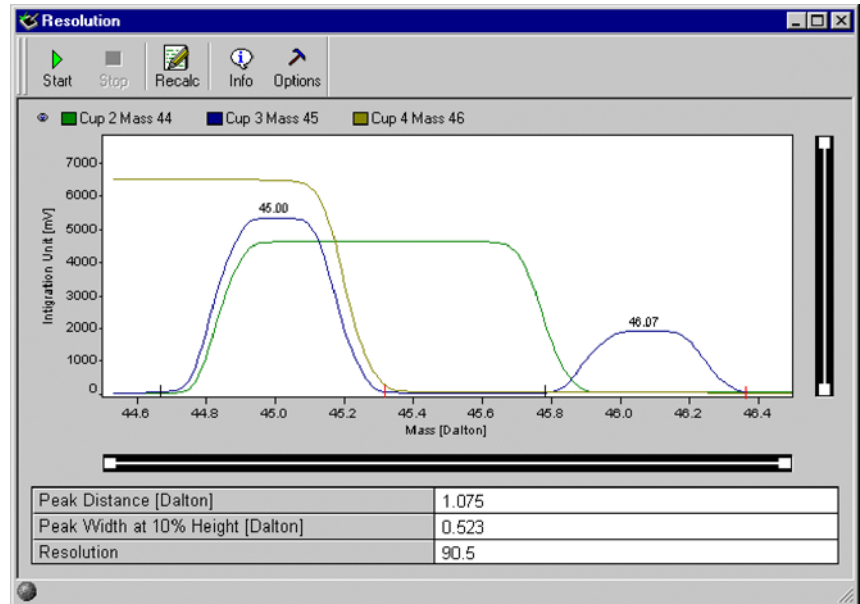
10. Click  .

Testing starts with a Peak Center on the selected cup (for example mass 45, cup 3), see [Figure 7-56](#).



**Figure 7-56.** Peak Center

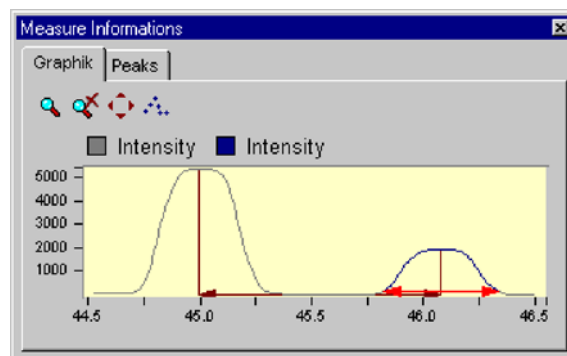
If no peak could be detected, the measurement is stopped. If not enough peaks could be detected, an error message occurs. Finally, the values of Peak Distance [Da], Peak Width at 10% Height [Da] and Resolution are displayed, see [Figure 7-57](#).



**Figure 7-57.** Resolution window

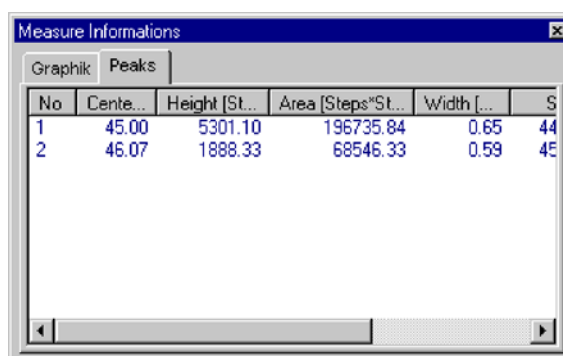
11. Click  .

The Information window opens, see [Figure 7-58](#):



**Figure 7-58.** Resolution Information page

12. Click the **Peaks** tab, see [Figure 7-59](#).

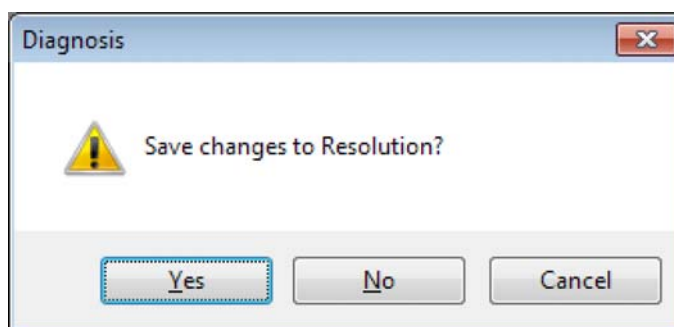


No	Centr...	Height [St...	Area [Steps*St...	Width [...	S
1	45.00	5301.10	196735.84	0.65	44
2	46.07	1888.33	68546.33	0.59	45

**Figure 7-59.** Peaks page

13. Close the **Resolution** window.

14. Confirm the message window [Figure 7-60](#) as appropriate.



**Figure 7-60.** Saving changes to Resolution

## Signal Stability

Signal Stability describes the stability of the intensity (that is peak height). The intensity on top of the peak is measured for a limited period of time (for example 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  $2 \times 10^{-4}$  (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)

**Note** 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.

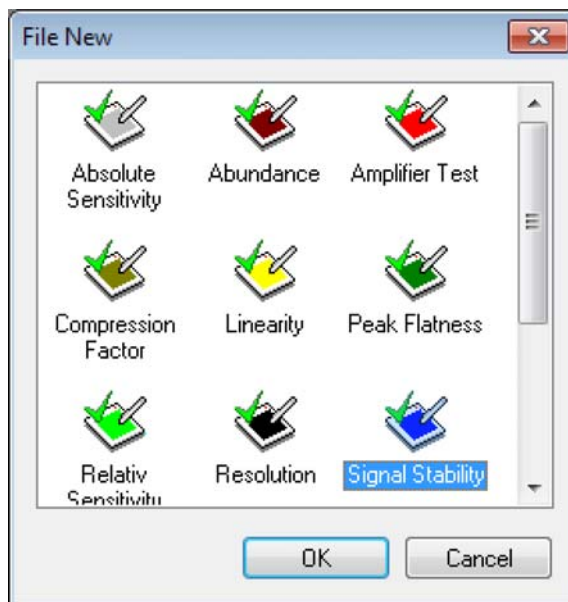
## Testing Signal Stability

### ❖ To test Signal Stability

1. In the Isodat folder, double-click  to open the **Diagnosis** tool.

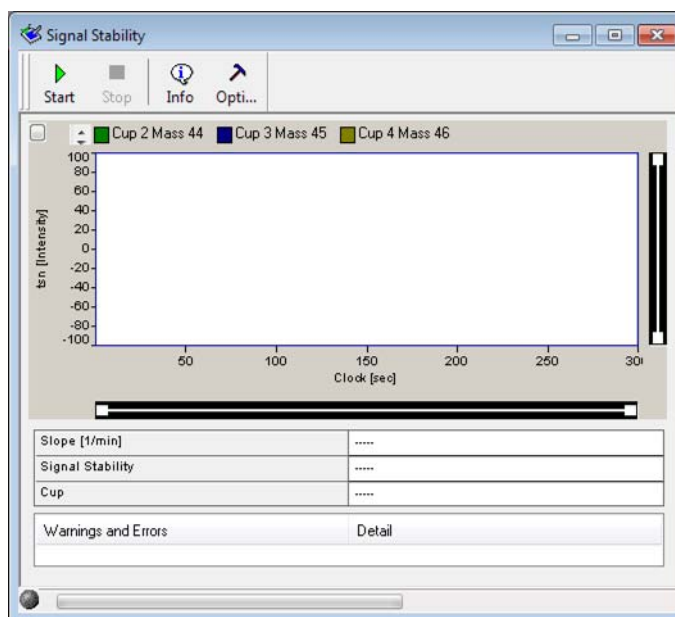
2. Click  to create a diagnosis test.

3. In the **File New** window, click **Signal Stability** and click **OK**. See [Figure 7-61](#).




**Figure 7-61.** Opening Signal Stability

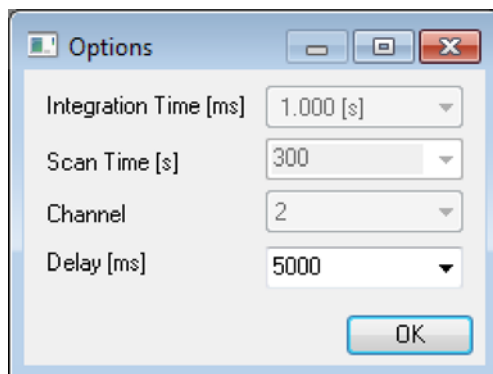
The **Signal Stability** window opens, see [Figure 7-62](#):



**Figure 7-62.** Signal Stability window

4. Click  **Options**.

- From the drop-down list, select a **Delay [ms]** value between 100 ms and 20000 ms, see [Figure 7-63](#).

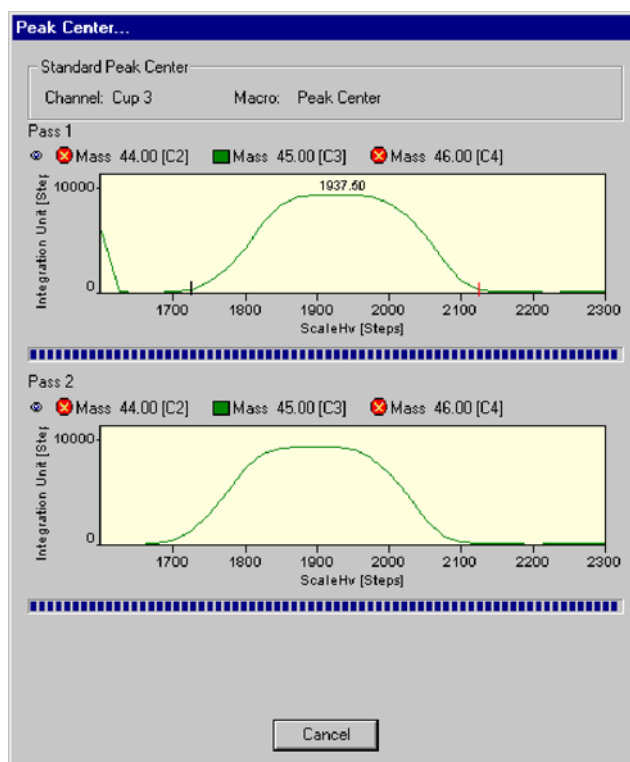


**Figure 7-63.** Options for Signal Stability

- Click **OK**.

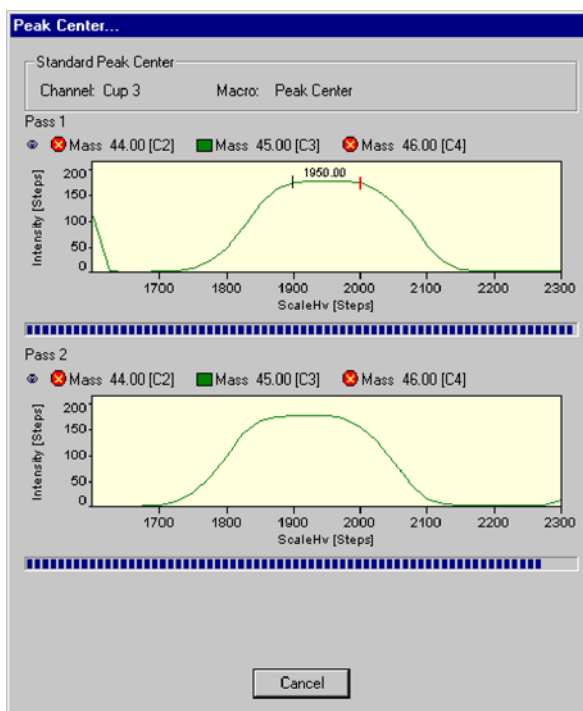


- Click **Start**.  
Testing starts with a Peak Center on the selected Cup (for example mass 45, Cup 3), see [Figure 7-64](#).



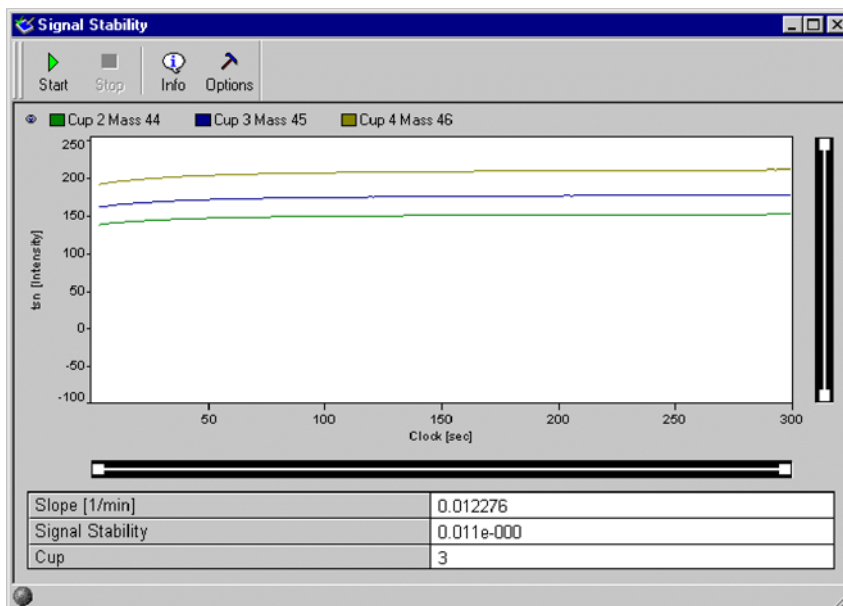
**Figure 7-64.** Peak Center

Another Peak Center is performed, see [Figure 7-65](#).



**Figure 7-65.** Peak Center

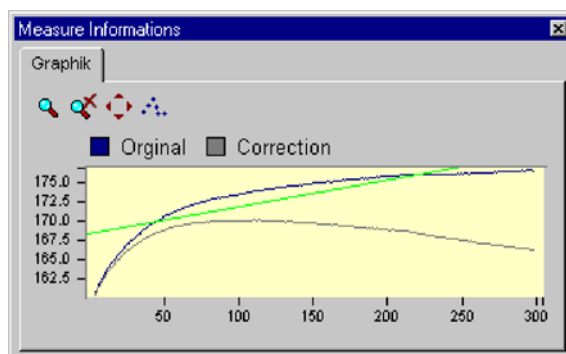
Finally, values of slope [1/min], Signal Stability and the Cup number are displayed, see [Figure 7-66](#).



**Figure 7-66.** Signal Stability window

8. Click  .

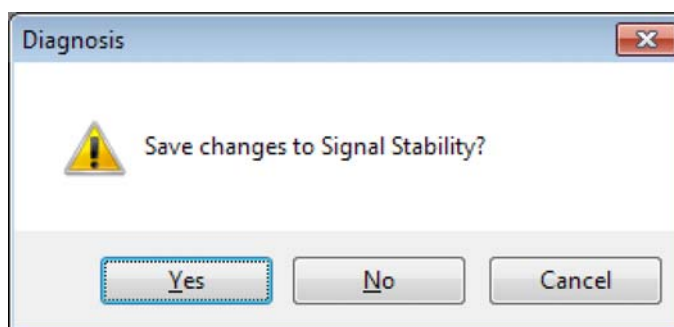
9. Click the **Graphik** tab, see [Figure 7-67](#).



**Figure 7-67.** Graphik page

10. Close the **Signal Stability** window.

11. Confirm the message window [Figure 7-68](#) as appropriate.



**Figure 7-68.** Saving changes to signal Stability

## 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
- Setting 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 (for example 15 min).
- New peak center procedure
- Calculation of System Stability [ $\text{min}^{-1}$ ] and Relative Mass Drift [ $\text{min}^{-1}$ ] (either electronic or to magnetic drift) using the slope of the peak flank.

**Note** Testing System Stability requires a signal of 3 V or more! ▲

Two results are obtained:


- Slope of relative mass drift vs. time (time drift)
- Standard deviation of this slope (scatter of the mass)

A value of  $5 \times 10^{-4}$  measured for a period of 15 min reflects a good System Stability.

## Testing System Stability

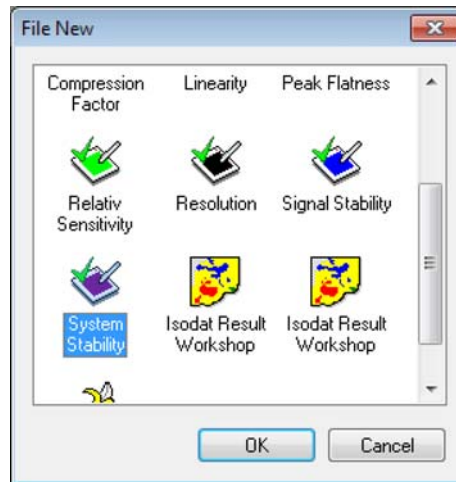
### ❖ To test System Stability

1. In the Isodat folder, double-click  to open the **Diagnosis** tool.

2. Click  to create a diagnosis test.

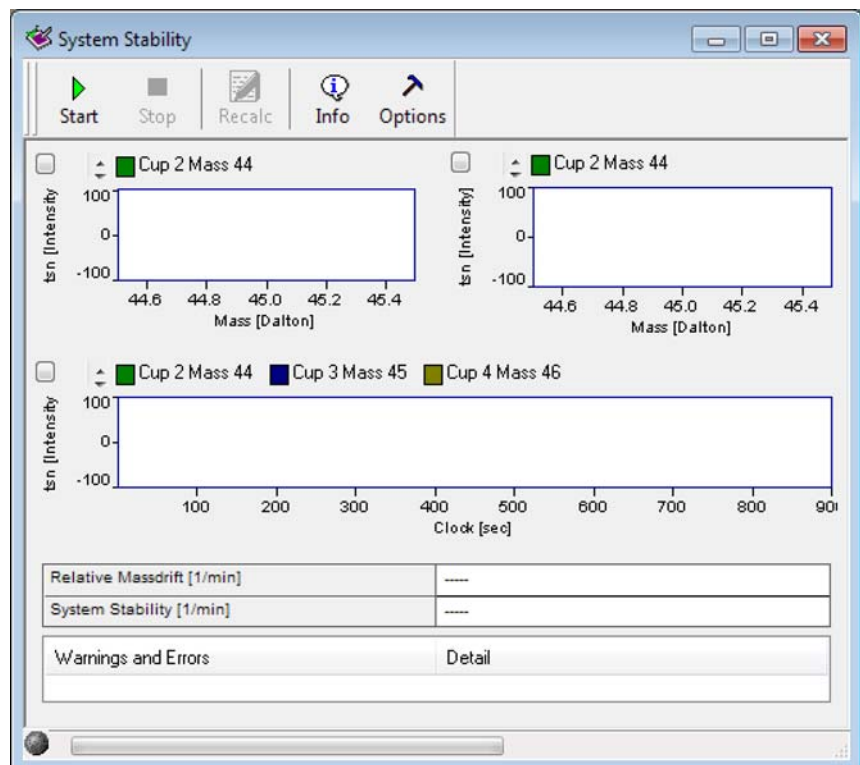
1. Click the **System Stability** icon.

2. In the **File New** window, click **System Stability** and click **OK**. See [Figure 7-69](#).



**Figure 7-69.** Testing System Stability

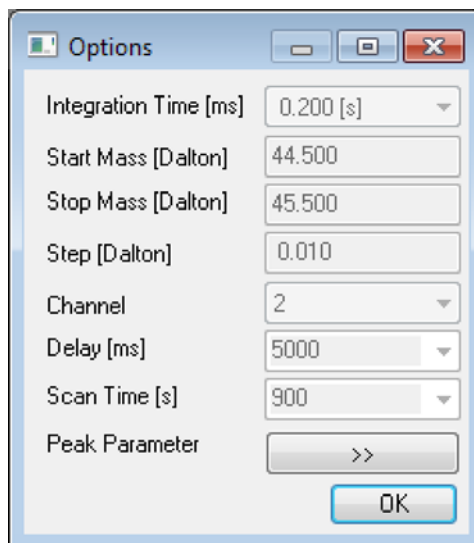
The **System Stability** window opens, see [Figure 7-70](#):



**Figure 7-70.** System Stability window

3. Click  **Options** .

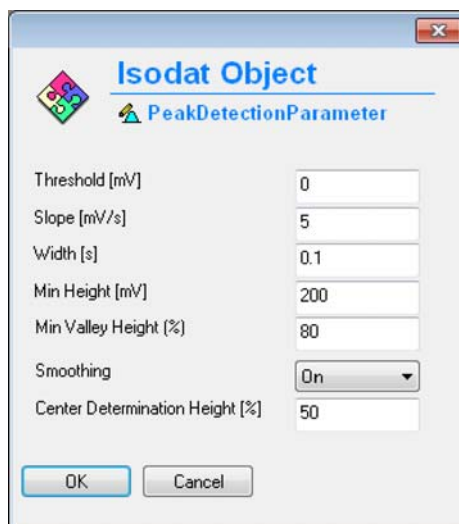
- If you simply want to accept the Peak parameters, see [Figure 7-71](#), click **OK**.



**Figure 7-71.** Options for System Stability


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.

- Click  next to **Peak Parameter** to open the object window, see [Figure 7-72](#).



**Figure 7-72.** Peak Detection Parameters

- Make your changes or accept the defaults.
- Then click **OK**.

8. Click  .  
Testing starts with a Peak Center on the selected Cup, for example mass 45, Cup 3, see [Figure 7-73](#).

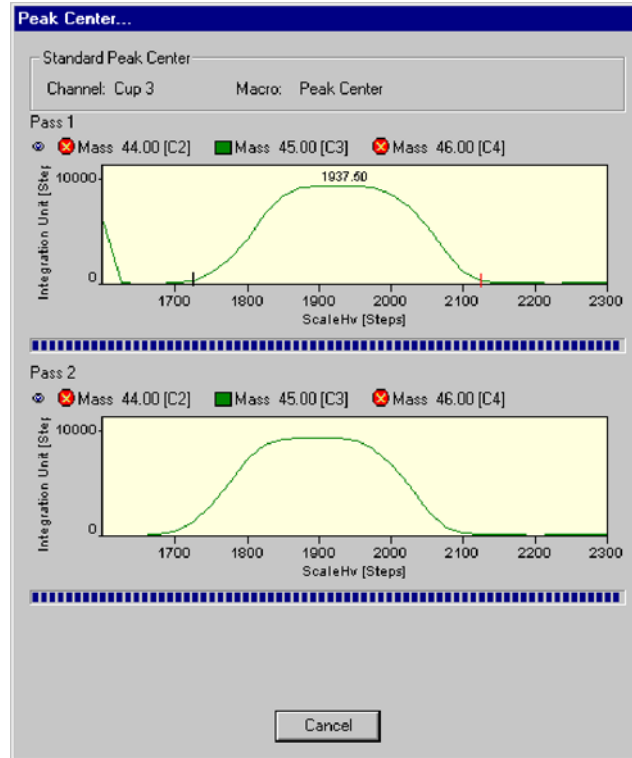


Figure 7-73. Peak Center

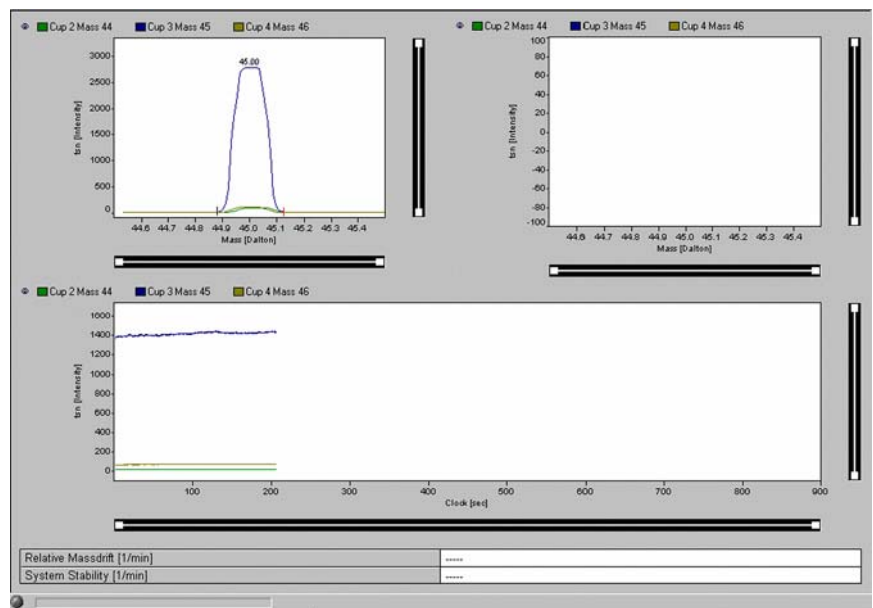


Figure 7-74. Continuing Peak Center

Testing continues with another Peak Center on the selected Cup, see Figure 7-75.

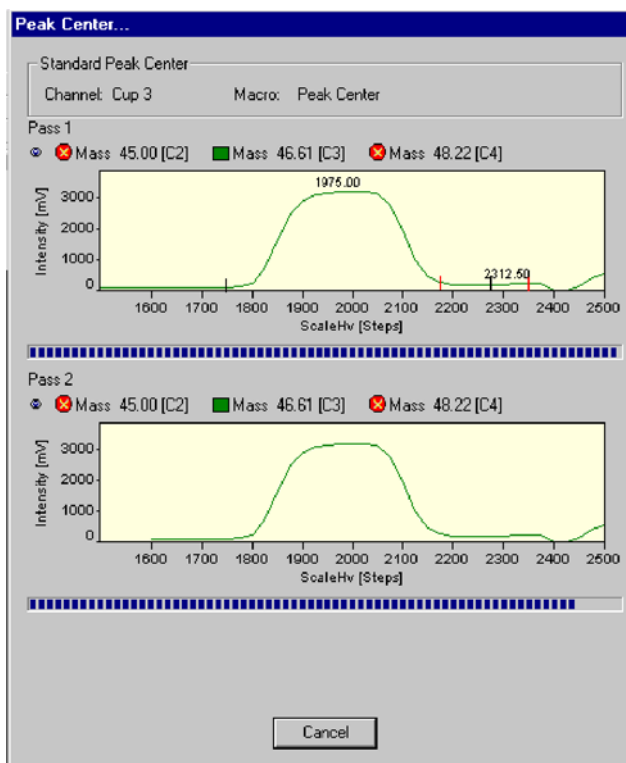


Figure 7-75. Peak Center

Finally, values for System Stability [ $\text{min}^{-1}$ ] and Relative Mass Drift [ $\text{min}^{-1}$ ] are displayed, see Figure 7-76.

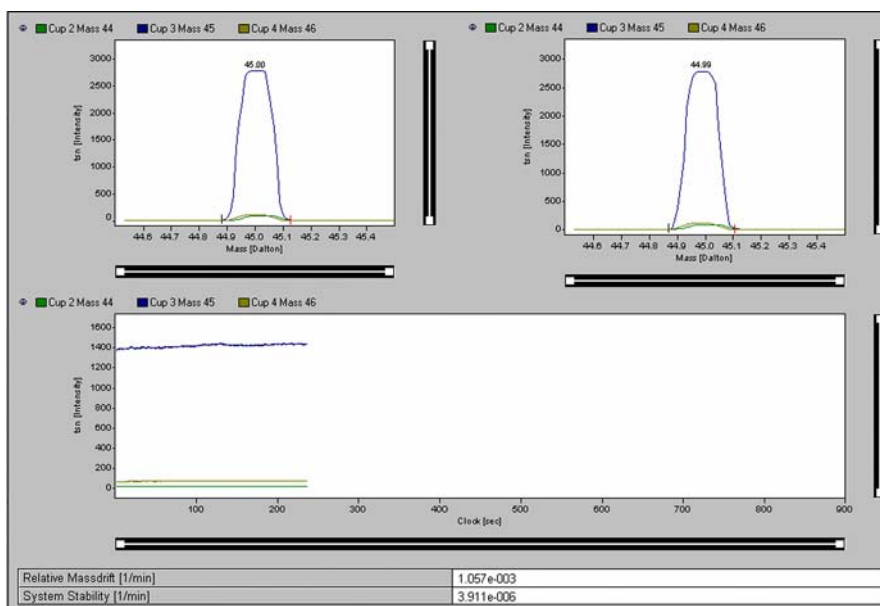
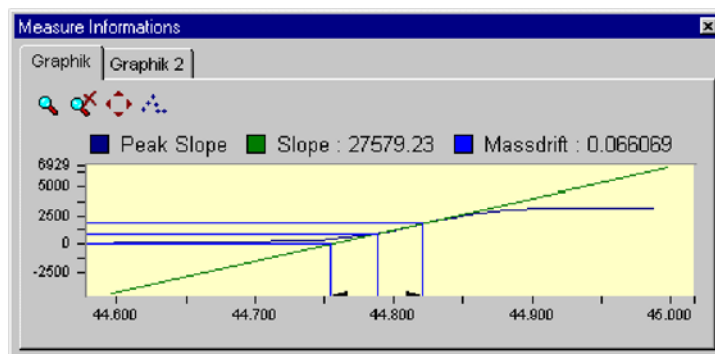


Figure 7-76. Values for System Stability and Relative Mass Drift

9. Click  .

10. Click the **Graphik** tab.

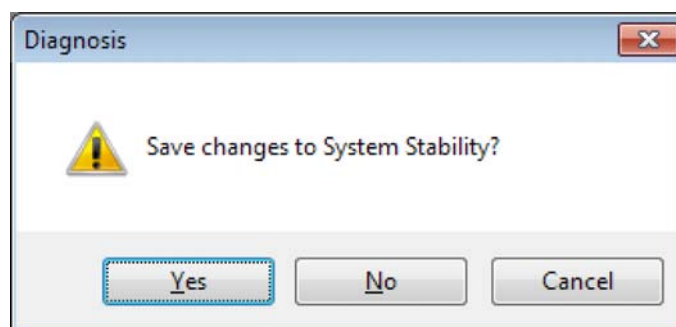
The diagram for calculating Slope and Mass Drift is shown together with the respective values., see [Figure 7-77](#).



**Figure 7-77.** Graphik page

11. Close the **System Stability** window.

12. Confirm the message window [Figure 7-78](#) as appropriate.



**Figure 7-78.** Saving changes to system Stability

## 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 (that is MEMCO 6) is given in [Table 7-1](#).

**Table 7-1.** Example: Assignment of a collector with six cups

Measuring channel		1	2	3	4	5	6
<b>VF-converter</b>		1	2	3	4	5	6
<b>Element/</b>	N <sub>2</sub>	28	29	30			
<b>Mass</b>	CO <sub>2</sub>				44	45	46
	SO <sub>2</sub>			64	66		

With this combination, there is no need to switch the feedback resistors when changing from CO<sub>2</sub> analysis to N<sub>2</sub> analysis. The switching is done automatically by the Isodat software changing the cup configuration.

**Note** For SO<sub>2</sub> operation the VISC must be opened. ▲

Only in case of switching to SO<sub>2</sub> analysis, it is necessary to switch the amplification factor (done automatically by switching the gas configuration).

## Heating Inlet Capillaries

**Note** The heating system can be regulated via the Isodat software. ▲

**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 (90 VA / 220 V / 7.7 V / 12 A) is supplied for heating purposes. To heat all capillaries simultaneously, it is advisable to acquire additional heating transformers.

### ❖ To heat inlet capillaries

1. Before heating the capillary itself, heat the surrounding of the capillary to approximately 80 °C for about 30 to 60 minutes, that is heat the valves of the inlet system (including 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.

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.
3. Plug the third cable to the second socket and connect the clip to the middle of the capillary.

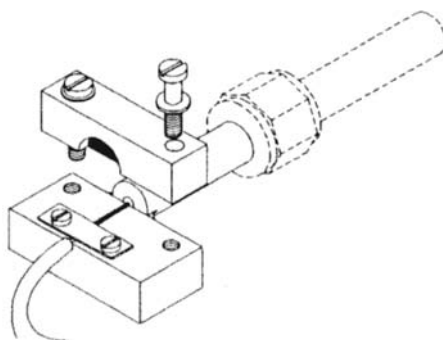
Capillaries supplied by Thermo Fisher Scientific have a metal sleeve attached in the middle providing better electrical contact.

**Caution** After the capillary has been wired, make sure that 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. ▲

4. Connect the transformer to the mains supply and switch it on. The heating phase should be controlled by monitoring the signal intensity of H<sub>2</sub>O (mass 18, measured on the channel for mass 45).

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. ▲



**Figure 7-79.** Crimping device at the end piece of a capillary

5. When fitting the crimp block again, make sure the capillary is exactly placed in the groove 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 (refer to [“Replacing an Inlet Capillary”](#) on [page 7-62](#)).


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, that is measuring the same gas on sample and standard side.

## Replacing an Inlet Capillary

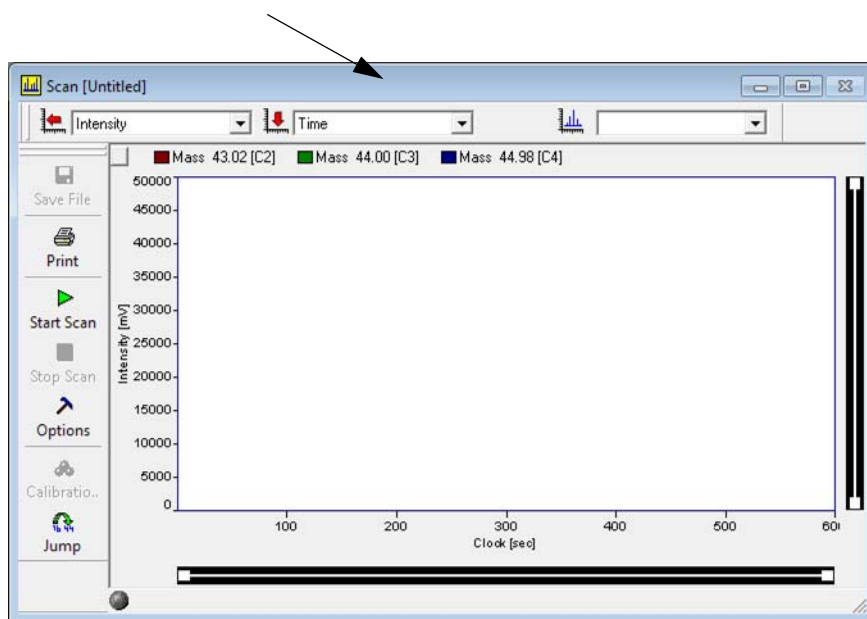
Replacement of an inlet capillary may become necessary in case of contamination or mechanical damage. After replacement, the flow rate of the new capillary has to be set by crimping. The crimping device consists of two metal blocks. The base is attached to the end piece of the new capillary, which has 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.

**Note** Make sure that all valves are closed before venting the surrounding area of the capillary, which is to be exchanged. ▲

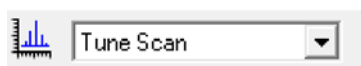
### ❖ To replace an inlet capillary

1. Vent the parts of the inlet system and the changeover valve, which are connected by the capillary.
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.
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.
4. Pump out the inlet system.
5. Admit a proper amount of CO<sub>2</sub> into the inlet system, so that the storage reservoir pressure is about 20 mbar on both sides of the capillary.
6. In the Isodat folder, double-click  to open the **Instrument Control** tool.

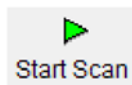
- Click the title bar of the Scan window to activate it, see [Figure 7-80](#).



**Figure 7-80.** Activating the Scan window



- Select **Tune Scan**.



- Click the **Start Scan** button.
- Tighten the screws of the crimp block carefully and squeeze the capillary until the output signal reaches 1 Volt per 10 mbar with CO<sub>2</sub> used for measurement.

After crimping, the capillary has to be heated (refer to [“Heating Inlet Capillaries”](#) on [page 7-60](#)).

## **Diagnosis**

### Replacing an Inlet Capillary

## Chapter 8 Technical Information

This chapter outlines the maintenance procedures that you should perform on a regular basis to maintain optimum MS detector performance. It also lists the replaceable parts for the MS detector and data system.

This chapter contains the following topics:

- “Introduction” on page 8-2
- “Maintenance Operations” on page 8-5
- “Valves of Dual Inlet System” on page 8-36
- “Changeover Valve” on page 8-41
- “Parts Lists” on page 8-45

## Introduction



**Warning Electric Current.** Danger of injury or death. 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! ▲

**Caution** Be careful when servicing the vacuum system. Abrupt opening to atmosphere might destroy the filament or damage the collector system and other expensive parts. ▲

This chapter contains information about requirements, maintenance and repair of the MAT 253 mass spectrometer. It also includes schematic drawings and spare parts lists.

The MAT 253 contains elaborate and expensive components. Only qualified and skilled personnel, therefore, should perform servicing. It is recommended that the Thermo Fisher Scientific service be called if there are any uncertainties or if difficulties arise. It is further recommended to use original Thermo Fisher Scientific 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.

Before starting maintenance and repair, read the appropriate chapters of the manuals.

Before calling the Thermo Fisher Scientific service, try to localize the defect! A precise description of the defect will ease the repair and reduce the costs.

When working with solvents and sample residuals, consider your regional safety instructions.

# Requirements

Your system must meet the following 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 hardware, refer to [“Maintenance Operations”](#) on [page 8-5](#).

Your computer system must meet the recommended requirements summarized in [Table 8-1](#).

**Table 8-1.** Software 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, 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

## Safety Rules for the Repair Service

Thermo Scientific 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 (Maximale Arbeitsplatzkonzentration, German for Maximum Exposure Limit (MEL) or Threshold Limit Value (TLV)) list 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.

Take care that vacuum pumps and all other parts which had been in contact to hazardous materials, will be properly decontaminated prior to returning to Thermo Fisher Scientific.

Parts contaminated by radioisotopes are not subject to return to Thermo Fisher Scientific neither under warranty nor under the exchange part program.

When returning parts to Thermo Fisher Scientific, the use of our repair-covering letters (<http://decon.thermo-bremen.com>) is obligatory.

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.

## Maintenance Operations

Regular maintenance including functional checks and service routines is required to maintain trouble free and continuous operation. These maintenance operations are listed in [Table 8-2](#).

**Table 8-2.** Maintenance operations

Part	Maintenance operation	Frequency
Rotary pump	Check oil level Exchange oil	Refer to manufacturer's instructions
Turbomolecular pump	Exchange oil reservoir	Refer to manufacturer's instructions
Compressed air service unit	Check oil level Drain condensate and clean filter	Monthly If required
Dual Inlet (option)	Refer to <a href="#">"Ion Source Maintenance"</a> on page 8-12	Once a year
Continuous Flow (option)	Refer to <a href="#">"Ion Source Maintenance"</a> on page 8-12	On demand (> two years)
Safety circuit	Check function	Every 6 months

## Basic Rules for Cleaning

**Caution** Ceramic parts which cannot be disconnected from metal parts must not be annealed. ▲

**Caution** When re-assembling, do not touch the washed parts with naked hands! Use non-fibrous gloves and clean tools! ▲



Wear protective gloves when handling the cleaning agent. ▲

**Note** Do not economize with the cleaning agent! Renew the cleaning bath frequently. ▲

❖ **To check highly volatile cleaning agents (cyclohexane, acetone)**

1. 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).
2. 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!
3. Rinse with a lot of water if using cleaning agents in aqueous solution! Use warm water, finally rinse with distilled (or de-ionized) water!
4. Dry the parts that 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.
5. 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.

6. 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.

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!

## Vacuum System Maintenance

**Caution** Never use silicon oils or silicon greases! Silicon layers lead to surface charges and are difficult to eliminate! ▲

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. For distinguishing whether elevated pressures are caused by leaks or by contamination, take a background spectrum.

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. Because 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 Fisher Scientific only.
- By introducing solvents that had not been removed carefully after cleaning.

### 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. ▲

## Flanges With Viton Gaskets

**Caution** Do not operate Viton at temperatures above 150 °C. ▲

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 Fisher Scientific 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.

## 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.

## Cleaning the Vacuum Components

Cleaning the parts of the high vacuum regions is described in [“Ion Source Maintenance”](#) on page 8-12.

The best cleaning is obtained when using a hot ultra sonic bath.

### ❖ To be observed when cleaning

1. Use pure solvents only.
2. When cleaning with solvents, use a hood.
3. Pay attention to your country's safety regulations.
4. After cleaning, dry the parts carefully.
5. Do not touch the cleaned parts with your fingers (use lint-free gloves).

## Leak Detection



**Warning Explosive Gases.** Danger of injury. Ethanol vapors are explosive. Do not smoke or handle with open fire during leak detection. Ventilate the laboratory carefully. ▲

Two methods are recommended for leak detection:

❖ **To perform mass spectrometric leak detection**

Mass spectrometric leak detection can be applied if the leak permits mass spectrometer operation (pressure  $\leq 6 \times 10^{-4}$  mbar).

1. Use a container with suitable leak detection gas (for example argon).
2. Connect the reducing valve to a PVC tube and the open end of the tube to a fine capillary (for example glass or metal tubing).
3. 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.

❖ **To perform leak detection in the fore vacuum section or if the pressure is too high for mass spectrometer operation**

1. Use a small washing bottle with ethanol and spray a fine ethanol beam onto those connections where a leak is suspected.
2. 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.

## Ion Gauge Maintenance

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 “[Basic Rules for Cleaning](#)” on [page 8-6](#).

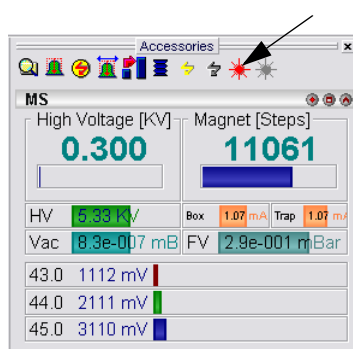
## Ion Source Maintenance

This 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.

### Removal of the Ion Source

❖ **To remove the ion source**

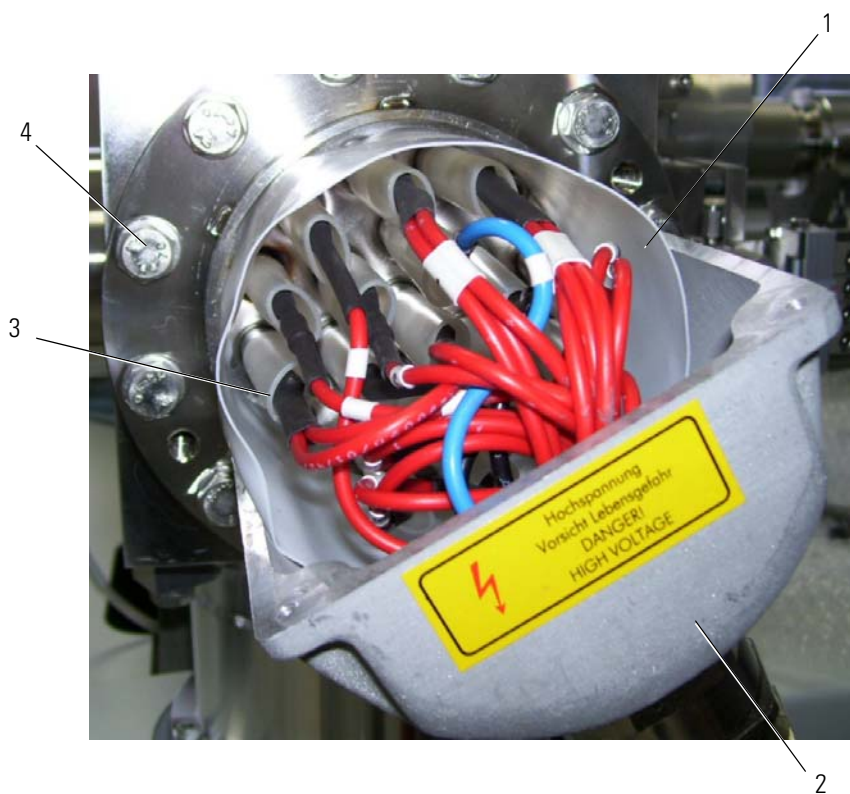
1. Click the  button to switch off high voltage (source off), see [Figure 8-1](#).



**Figure 8-1.** Switch off source

2. Switch off the ANALYZER PUMPS at the main panel (see [Figure 1-9](#)) and wait until MAT 253 vents.
3. Unscrew the four screws of the upper source flange cover and remove it.

Figure 8-2 shows the ion source with the upper cover removed:



Labeled Components: 1=Teflon shield, 2=bottom cover, 3=insulating sleeves, 4=bolts (twelve pieces)

**Figure 8-2.** Ion source (upper cover removed) showing assembly of isolations at source

4. Remove protective Teflon shield **1** in [Figure 8-2](#).
5. Pull back the twelve individual insulation sleeves (see **3** in [Figure 8-2](#)).
6. Unscrew all connections between cable and flange with a 1.5 mm Allen wrench.
7. Completely remove the bottom cover of the source flange (see **2** in [Figure 8-2](#)).
8. Completely open changeover actuator for the VISC valve. Refer to chapter “Gas Inlet” on [page 2-5](#) and **6** in [Figure 2-3](#) for details.
9. Unscrew the twelve bolts (see **4** in [Figure 8-2](#)) of the ion source flange and remove the ion source carefully without knocking against the ion source housing.
10. Place the ion source in a secure holder on a desk.

11. In case the ion source is removed for a longer period of time, for example for cleaning, close the opening of the analyzer head by suitable means in order to prevent foreign particles from entering the ion source chamber.

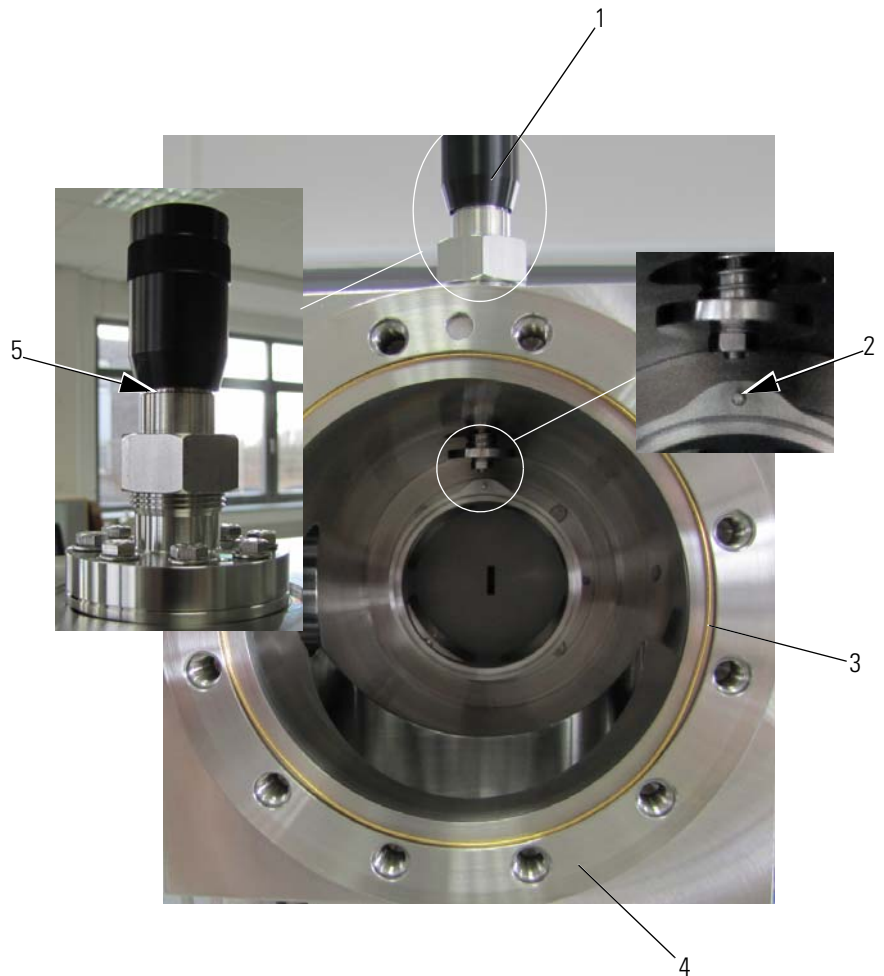
## **Reinstallation of the Ion Source**

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.

### **❖ To reinstall the ion source**

1. Inspect the gold gasket carefully for absence of scratches and lint.
2. Place the spacer ring (see **4** in [Figure 8-3](#)) on the flange of the analyzer head.
3. Place the gold gasket **3** in [Figure 8-3](#) inside the spacer ring as shown in [Figure 8-3](#).

4. Turn the changeover valve actuator for VISC **1** in [Figure 8-3](#) until the valve is completely open.  
The groove **5** in detail [Figure 8-3](#) must be visible.

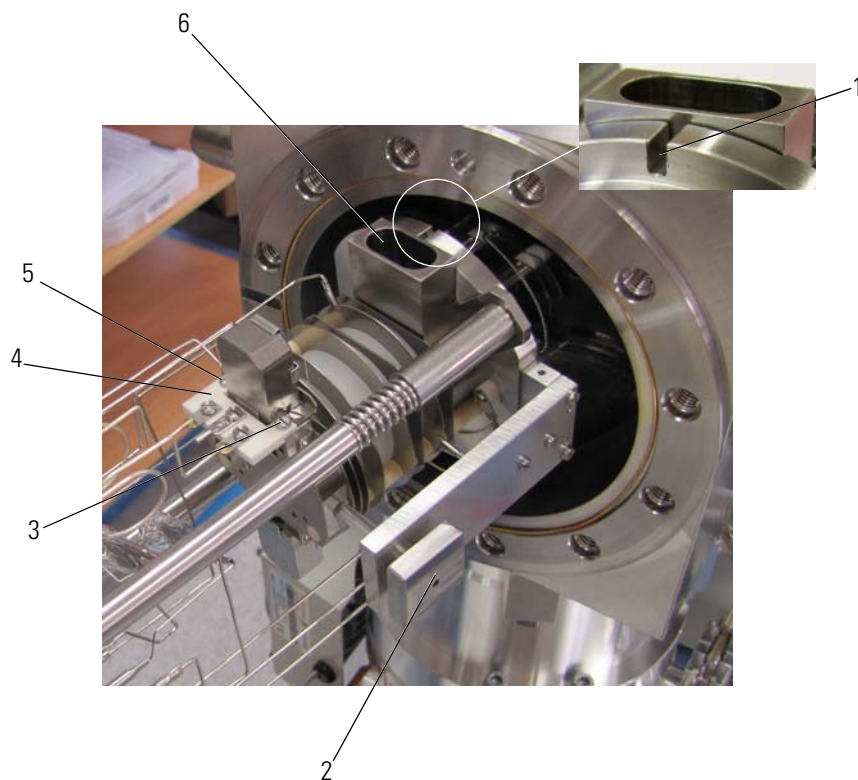


Labeled Components: 1=changeover valve actuator for VISC (valve open), 2=pin, 3=gasket (gold), 4=spacer ring, 5=groove visible if valve completely open

**Figure 8-3.** Analyzer head flange with spacer ring and gold gasket

5. With the opening of the VISC window **8** in [Figure 8-4](#) facing upwards, insert the ion source into the analyzer head.  
Both the cathode **4** in [Figure 8-4](#) and the mechanical connections (**3**

and **5** in Figure 8-4) can be seen from above. The gas inlet is positioned on the right, see **2** in Figure 8-4.



Labeled Components: 1=groove for pin alignment, 2=gas inlet, 3=mechanical connections, 4=cathode, 5=mechanical connections, 6=VISC window,

**Figure 8-4.** Placing the ion source in the analyzer head

6. Take care that the groove **1** in Figure 8-4 in the analyzing housing aligns with the pin **2** in Figure 8-3. When inserted, the ion source flange and the making flange should be aligned in parallel.

**Caution** Do not proceed tightening if the flanges touch the spacer ring! ▲

7. Press the ion source in the direction of the analyzer and tighten the screws crosswise, see [Figure 8-5](#).  
Fasten only finger tight.



**Figure 8-5.** Tightening the ion source in the analyzer head

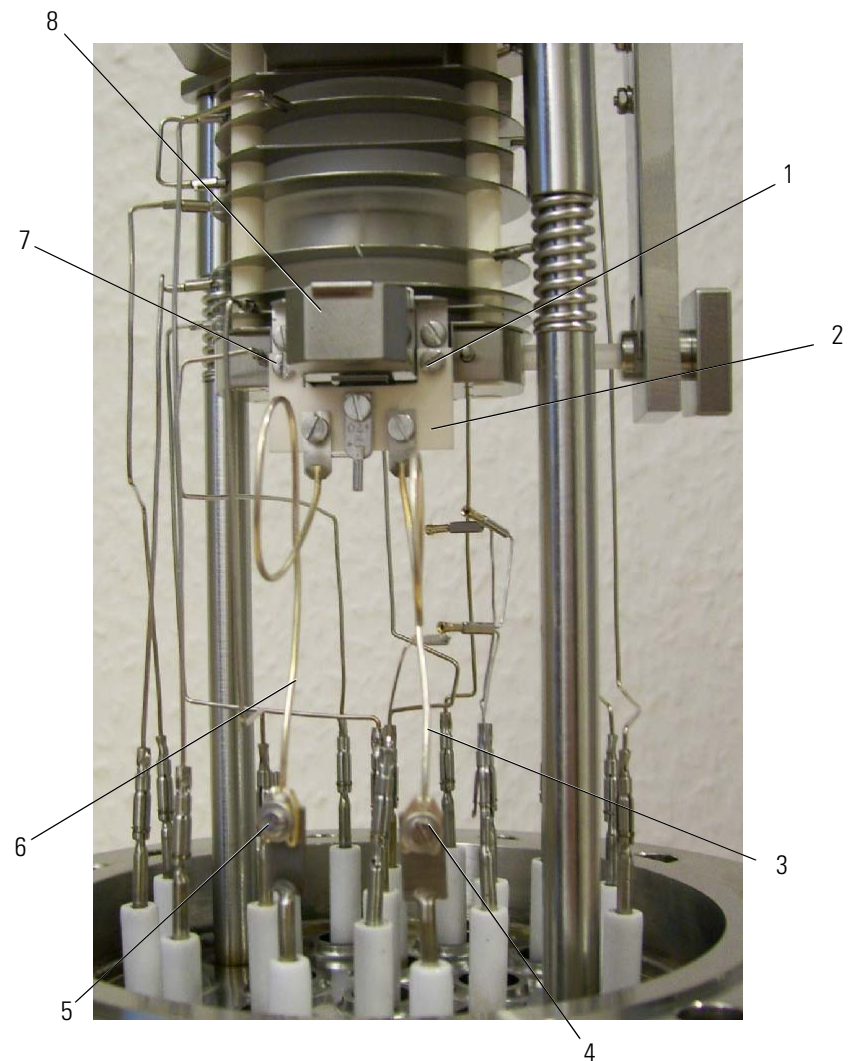
8. Check for short circuits.  
Use an ohmmeter to check every pin for short circuit against the analyzer housing, and check every pin for short circuit against every pin. For details refer to [“Checking the Ion Source”](#) on [page 8-30](#)
9. Check that flanges and spacer ring are equally spaced.  
Stop when flange and spacer ring touch.
10. Turn the changeover valve actuator for VISC **1** in [Figure 8-3](#) until the valve is completely closed.
11. Reconnect electrical connections, refer to [“Connections to Ion Source”](#) on [page 8-32](#).
12. Reinstall insulation sleeves, see **3** in [Figure 8-2](#).
13. Reinstall the Teflon shield, see **1** in [Figure 8-2](#).
14. Reinstall the upper source flange cover and tighten with four screws.
15. Switch on the ANALYZER PUMPS at the main panel (see [Figure 1-9](#)).
16. Wait until all pumps have reached rotational speed and the pressure falls below  $2 \times 10^{-6}$  mbar.

## Exchanging the Cathode Unit

**Caution** Use lint-free gloves and clean tools for the exchange. ▲

❖ **To exchange the cathode unit**

1. Remove the ion source as described in “Removal of the Ion Source” on page 8-12.
2. Unscrew the mechanical connections **1** and **7** in Figure 8-6.

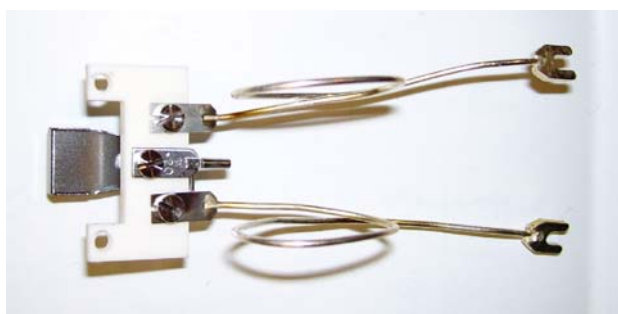


Labeled Components: 1=mechanical connection, 2=cathode (filament), 3=supply line, 4=supply connection, 5=supply connection, 6=supply line, 7=mechanical connection, 8=magnet

**Figure 8-6.** Ion source connections - side showing filament

3. Unscrew the supply connection **4** and **5** in Figure 8-6 and disconnect the supply lines **3** and **6** in Figure 8-6.

4. Insert a new cathode (see [Figure 8-7](#)).



**Figure 8-7.** Ion source cathode

5. Reconnect the supply lines **3** and **6** in with screws **4** and **5** in [Figure 8-6](#).
6. Tighten the screws for the mechanical connections **1** and **7** in [Figure 8-6](#).
7. Perform the source check as described in “[Checking the Ion Source](#)” on [page 8-30](#).
8. Reinstall the ion source as described in “[Reinstallation of the Ion Source](#)” on [page 8-14](#).

## Disassembling the Ion Source

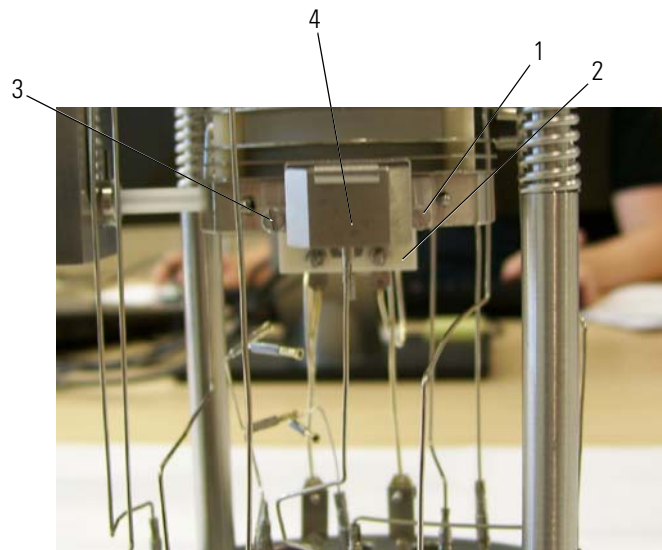
### ❖ To prepare the disassembly

1. Use a table with clean and smooth surface.
2. Put a white and clean paper onto this table.
3. Prepare receptacles for the small ion source parts.
4. Separate metal and ceramic parts.

### ❖ To disassemble the ion source

1. Remove the ion source as described in “[Removal of the Ion Source](#)” on [page 8-12](#).
2. Remove the cathode as described in “[Exchanging the Cathode Unit](#)” on [page 8-18](#).

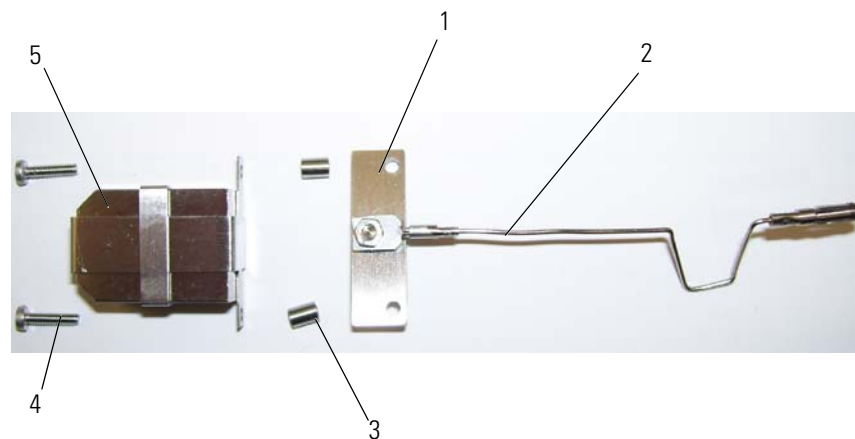
Figure 8-8 shows the other side of the ion source:



Labeled Components: 1=screw (magnet), 2=electron trap (collector), 3=screw (magnet), 4=magnet

**Figure 8-8.** Ion source connections - side showing electron trap

3. Unscrew the screws **1** and **3** in [Figure 8-8](#).
4. Remove the magnet **4** in [Figure 8-8](#) (**5** in [Figure 8-9](#)) and the electron trap **2** in [Figure 8-8](#) (**1** in [Figure 8-9](#)).



Labeled Components: 1= electron trap (collector), 2= electrical connection, 3=bushing (two pieces), 4=screw (two pieces), 5=magnet

**Figure 8-9.** Ion source: electron trap (collector) disassembled

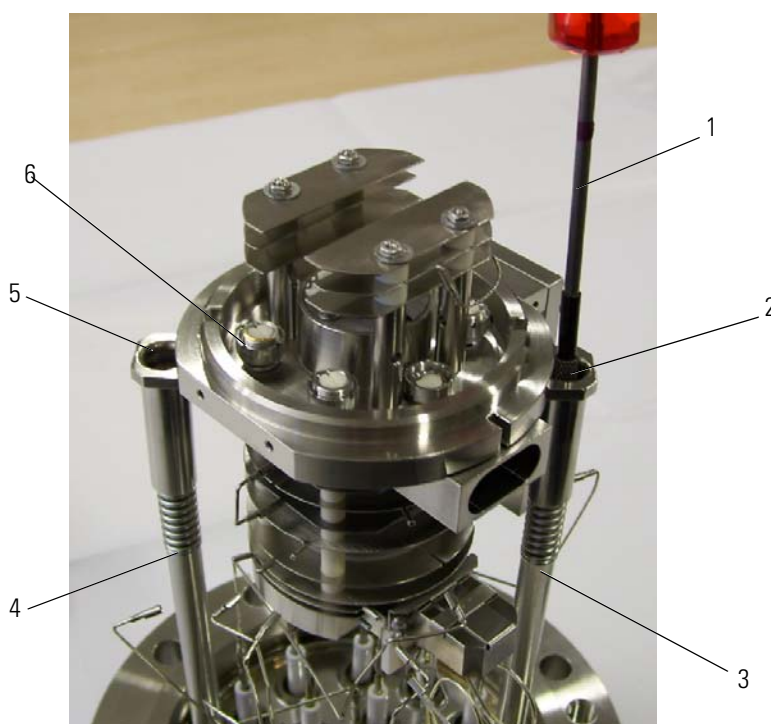
Magnet **5** in [Figure 8-9](#) and electron trap **1** in [Figure 8-9](#) are fastened with screws **1** and **3** in [Figure 8-8](#) only (see also **4** in [Figure 8-9](#)). The two small bushings **3** in [Figure 8-9](#) drop out when the screws are removed.

- Carefully pull off the electrical connection **2** in [Figure 8-9](#) from the electron trap **1** in [Figure 8-9](#) and bend the lead wires somewhat apart. Take care not to alter the curved shape of the lead wires as this facilitates correct reconnection.

If not necessary, do not pull off the lead wires from the feedthrough connectors of the flange.

This saves time and eases the assembly.

- Unscrew the cylinder head screws **2** and **5** in [Figure 8-10](#) and remove the ion source from its supporting rod **3** and **4** in [Figure 8-10](#).

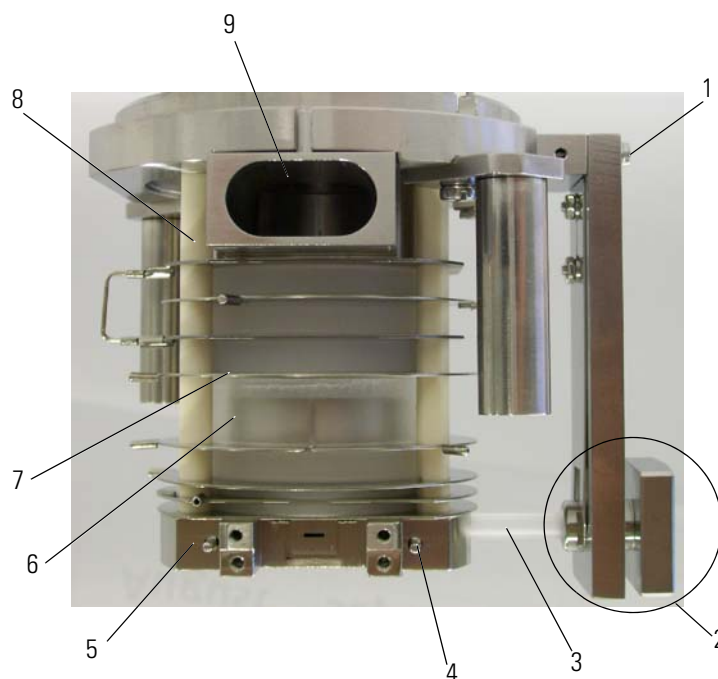


Labeled Components: 1=screw driver, 2=cylinder head screw, 3= supporting rods, 4=supporting rod, 5=cylinder head screw, 6=threaded sleeve (four pieces)

**Figure 8-10.** Ion source, on supporting rods, with einzel lens unit

- Unscrew the cylinder screws **1** in [Figure 8-11](#) and carefully take off the gas inlet assembly **2** in [Figure 8-11](#).
- Pull the quartz glass tube (see **3** in [Figure 8-11](#)) out of the gas inlet assembly.
- Loosen the four threaded sleeves (see **6** in [Figure 8-10](#) and **4** in [Figure 8-13](#)) using the source mounting tool **10** in [Figure 8-22](#).

10. Pull out the four cylinder pins **4** in [Figure 8-11](#) which fix the other end of the ceramic rods **8** in [Figure 8-11](#) to the ionization housing **5** in [Figure 8-11](#).



Labeled Components: 1=cylinder screws (two pieces), 2=gas inlet assembly, 3=quartz glass tube, 4=cylinder pin (four pieces), 5=ionization housing, 6=spacer ring (for details, see [Figure 8-22](#)), 7=plates (for details, see [Figure 8-22](#)), 8=ceramic rod (four pieces), 9=VISC opening

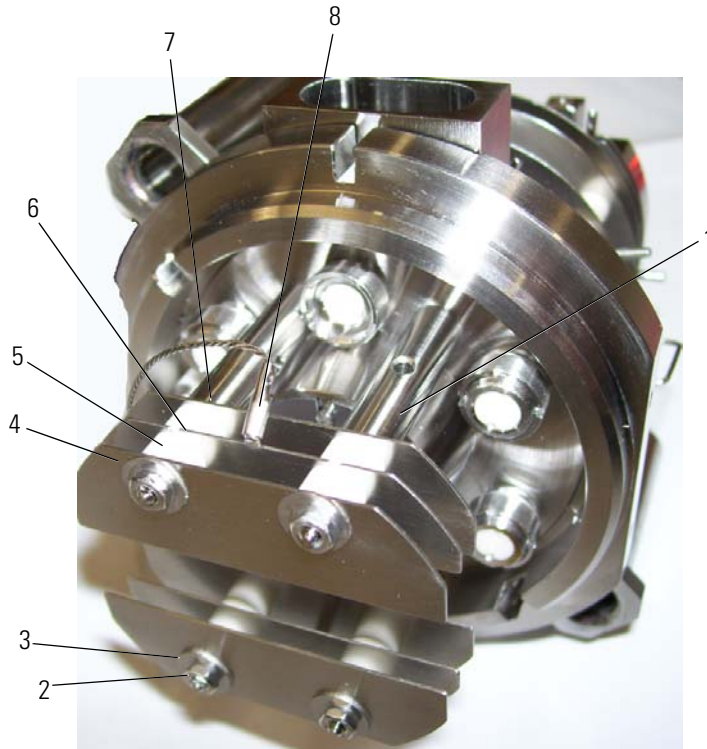
**Figure 8-11.** Ion source lower part

11. Remove the ionization housing **5** in [Figure 8-11](#) from the ceramic rods.

**Caution** 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. ▲

12. Carefully remove all spacer rings **6** and plates **7** in [Figure 8-11](#) from the ceramic rods and remember the correct order.  
Best is to place all plates and spacer rings on a clean surface in the order shown in [Figure 8-22](#).

13. To disassemble the einzel lens unit, unscrew the nuts **2** in [Figure 8-12](#) and remove the washers **3** in [Figure 8-12](#).

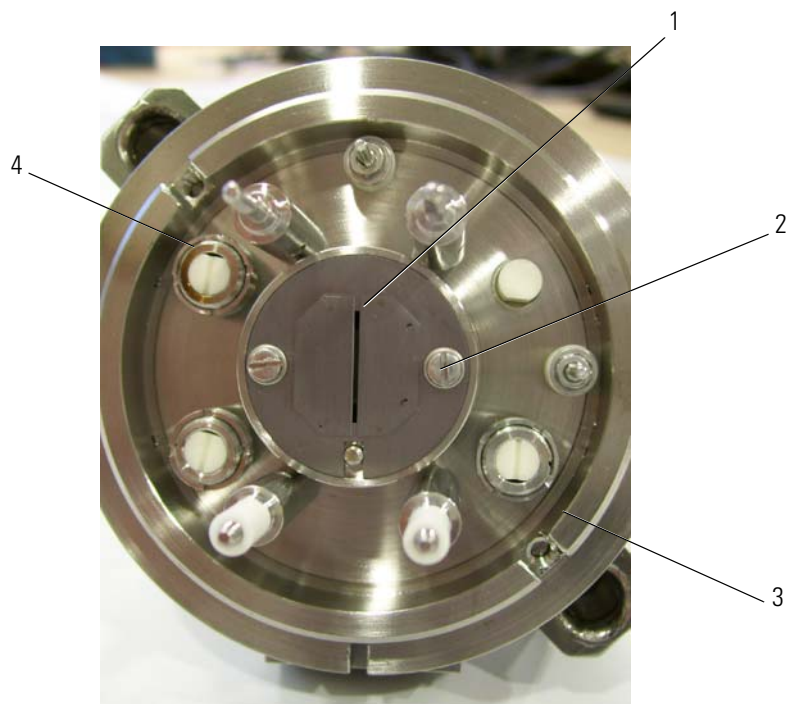


Labeled Components: 1=threaded rods (four pieces, stainless steel), 2=nuts (four pieces), 3=washer (four pieces), 4=grounding plate (two pairs), 5=ceramic spacer tube (eight pieces), 6=one pair of einzel lens, 7=ceramic spacer tube, not visible (four pieces), 8=electrical connection

**Figure 8-12.** Einzel lens unit - disassemble

14. Remove plates and spacer rings **4**, **5**, **6**, and **7** in [Figure 8-12](#) from the threaded rods **1** in [Figure 8-12](#).
15. Remove the electrical connections of the einzel lens (**8** in [Figure 8-12](#)).
16. Unscrew the cylinder screws **2** in [Figure 8-13](#) and remove the screws and the washers.

17. Remove the slit plate **1** in [Figure 8-13](#) from the ion source base **3** in [Figure 8-13](#).

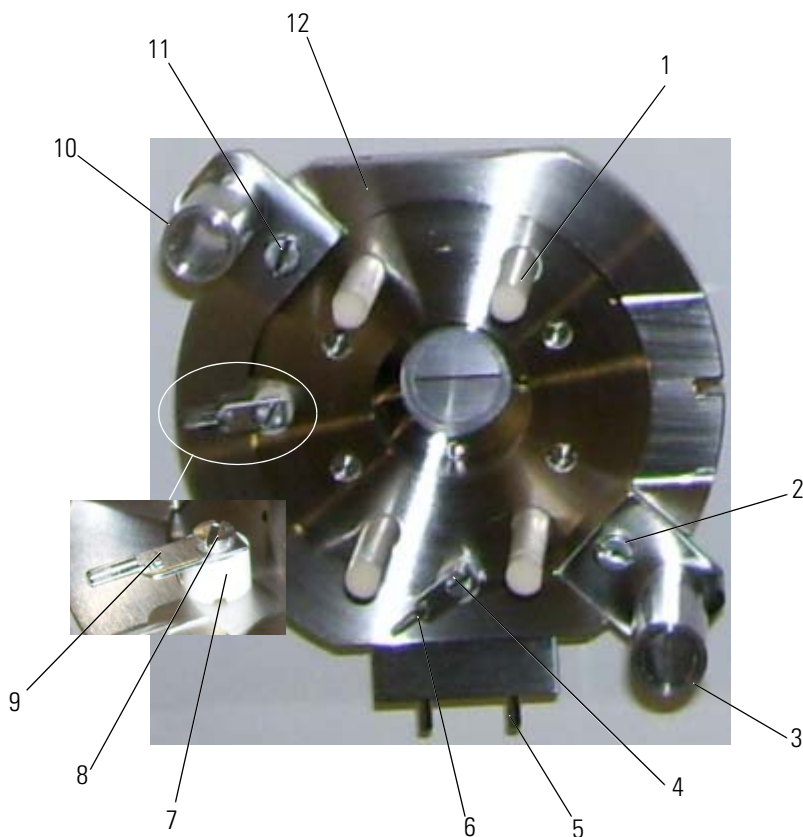


Labeled Components: 1=slit plate, 2=cylinder screw (two, with washers), 3=ion source base, 4=threaded sleeves

**Figure 8-13.** Ion source base without einzel lens

18. Turn the ion source base, see [Figure 8-14](#).

19. Unscrew the cylinder screws **2** and **11** in [Figure 8-14](#) and remove the supporting tubes **3** and **10** in [Figure 8-14](#).



Labeled Components: 1=ceramic rods (four pieces), 2=cylinder screw, 3=supporting tube, 4=contact screw (and ceramic spacer tube), 5=cylinder pin (two pieces) and holding block, 6=connection lug with pin, 7=ceramic spacer tube, 8=contact screw, 9=connection lug with pin, 10=supporting tube, 11=cylinder screw, 12=ion source base

**Figure 8-14.** Ion source base without plates and spacer rings

20. Remove the two cylinder pins **5** and the holding block in [Figure 8-14](#).
21. Unscrew the contact screws **4** and **8** in [Figure 8-14](#) and remove the connection lugs **6** and **9** with the ceramic spacer tubes (**7** in [Figure 8-14](#)).
22. Remove the four ceramic rods **1** in [Figure 8-14](#).

The ion source and base are completely disassembled.

## Cleaning the Ion Source Parts

The ion source parts include the metallic parts and the ceramic insulators.

## Cleaning the Metallic Parts

**Caution** Do not use ultrasonic when cleaning the ion source magnets! ▲

**Caution** Parts which are not made of stainless steel and the ion source magnets must not be cleaned in RBS. Clean these parts with cyclohexane. ▲

Grind all contaminated and discolored metal parts with fine emery paper (use 360 grain or finer).

The chemical cleaning must follow the mechanical cleaning.

For chemical cleaning, distinguish between the different types of metallic parts: All parts must be cleaned within a grease-dissolving bath (for example cyclohexane). Use a bath temperature of about 60 °C. An ultrasonic bath will improve and accelerate the cleaning procedure.

The basic rules for cleaning are described in “[Basic Rules for Cleaning](#)” on [page 8-6](#). 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 to 70 °C. Additionally, refer to the basic rules of cleaning in “[Basic Rules for Cleaning](#)” on [page 8-6](#).

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.

## Cleaning the Ceramic Insulators

**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! ▲

### ❖ To clean the ceramic insulators

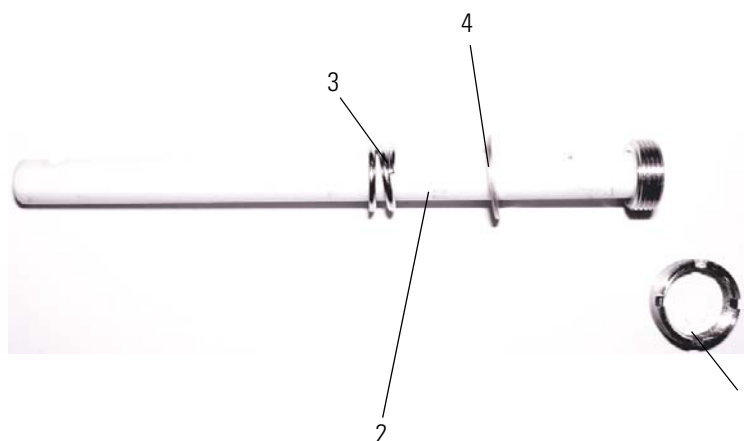
1. Use a clean diamond file for eliminating metal abrasion and evaporated films.  
Use this method only for cleaning the ceramic insulator of the cathode and the electron collector. Heavily contaminated ceramic parts may be cleaned later in RBS solution (do not use RBS for the cathode and the electron collector).

2. Anneal the ceramic spacer rings to soft red heat. Use a propane/oxygen flame or a muffle furnace for annealing (maximum temperature: 800 to 1000 °C).

## Assembling the Ion Source

**Caution** Touch the ion source parts with clean, lint-free gloves only. Use clean tools only! ▲

1. Before assembling the ion source, arrange the parts and the tools in a clear manner on white clean paper, see for example [Figure 8-22](#).
2. Assemble the ion source base (see [Figure 8-13](#) for assembled base).
3. Fit the slit plate **1** in [Figure 8-13](#) to the ion source base **3** in [Figure 8-13](#).
4. Install the cylinder screws and the washers **2** in [Figure 8-13](#) and tighten the screws.
5. Reinstall the connection lugs **6** and **9** and the ceramic spacer tubes (**7** in [Figure 8-14](#)) and tighten the contact screws **4** and **8** in [Figure 8-14](#).
6. Loosely screw the threaded sleeves **1** in [Figure 8-15](#) on to the ceramic rods (see **2** in [Figure 8-15](#) or **1** in [Figure 8-14](#)).
7. Slide a spring **3** in [Figure 8-15](#) and a washer **4** in [Figure 8-15](#) each on ceramic rods.



Labeled Components: 1=threaded sleeve, 2=ceramic rod, 3=spring, 4=washer

**Figure 8-15.** Threaded sleeves with ceramic rods

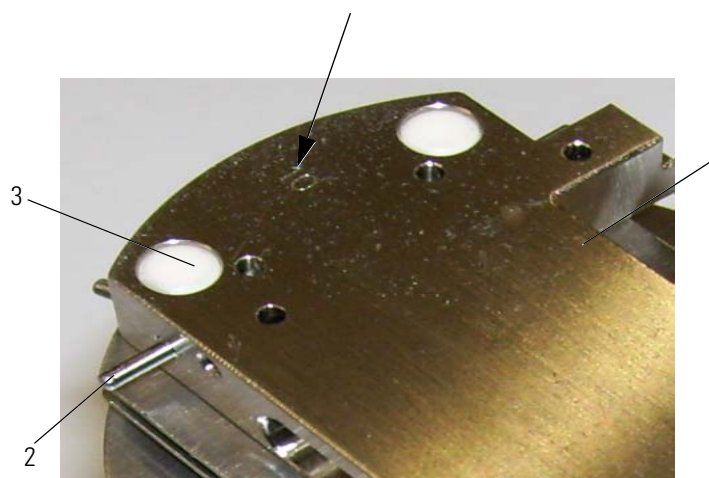
8. Pass the four ceramic rods **1** in [Figure 8-14](#) through the holes of the ion source base (see **12** in [Figure 8-14](#)) such that the punched numbers are visible from above.

The ion source base, the plates and the ionization housing are numbered, for example, see arrow [Figure 8-16](#). The numbers must be arranged with ascending sequence and in the same direction.

**Note** Spacer rings and plates must fit tightly together. ▲

**Caution** Reinstall 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. ▲

- Carefully install the spacer rings (**6** in [Figure 8-11](#)) and ion source plates (**7** in [Figure 8-11](#)) on the ceramic rods in the order shown in [Figure 8-22](#).
- Fit the ionization housing **1** in [Figure 8-16](#) to the ceramic rods **3** in [Figure 8-16](#) carefully and avoid metallic abrasion.
- Turn the four ceramic rods such that the slot grinded into their upper end is aligned with the boreholes (see **3** in [Figure 8-16](#)) in the ionization housing (see **1** in [Figure 8-16](#)), allowing the four cylinder pins (see **2** in [Figure 8-16](#)) to be pushed in smoothly.

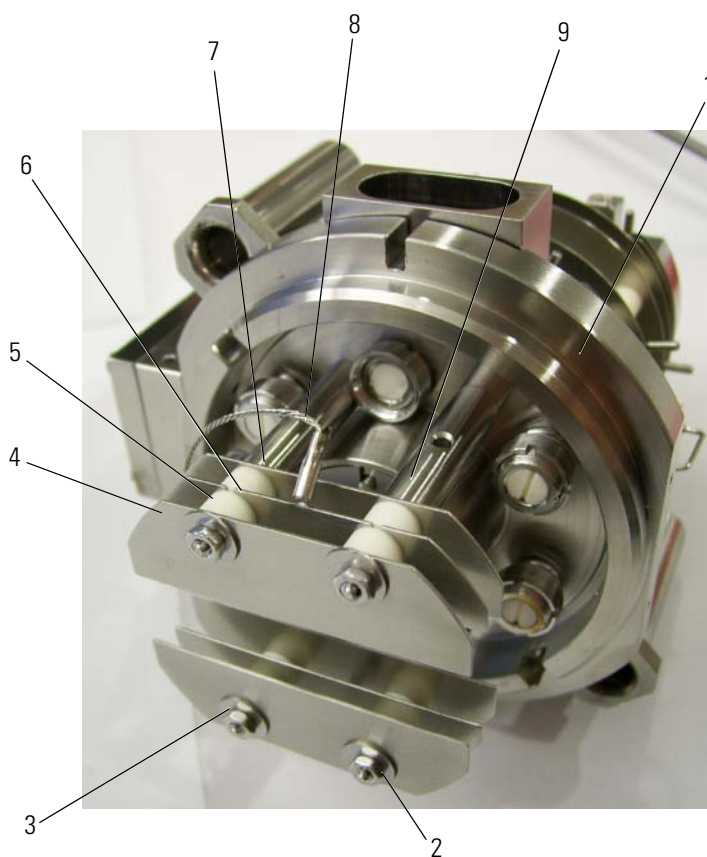


Labeled Components: 1= ionization housing, 2=cylinder pin, 3=ceramic rod

**Figure 8-16.** Ionization housing with punched number visible

- Tighten the four threaded sleeves **4** in [Figure 8-13](#) carefully with the source mounting tool **10** in [Figure 8-22](#) (part of the instrument tool kit), but as fast as it is necessary to have the parts of the ion source clamped firmly together.
- Fit the quartz glass tube **3** in [Figure 8-11](#) onto the gas inlet assembly **2** in [Figure 8-11](#).

14. Mount the gas inlet assembly **2** in [Figure 8-11](#) with the cylinder screw (see **1** in [Figure 8-11](#)) to the ion source base (see **3** in [Figure 8-13](#)).
15. Assemble the parts of the electron trap (collector, see [Figure 8-9](#)).
16. Paying attention to the correct polarity, attach the rod magnets (see **8** in [Figure 8-6](#) and **3** [Figure 8-8](#)) together with the electron trap (collector, **1** in [Figure 8-9](#)) and the cathode (filament, see [Figure 8-7](#) and **2** in [Figure 8-6](#)) to the ionization housing.
17. Tighten the cylinder screw **1** and **7** in [Figure 8-6](#) and **1** and **2** in [Figure 8-8](#).
18. Screw the four threaded rods (see **9** in [Figure 8-17](#)) into the ion source base **1** in [Figure 8-17](#).



Labeled Components: 1=ion source base, 2=nuts (four pieces), 3=washer (four pieces), 4=grounding plate (two pairs), 5=ceramic spacer tube (eight pieces), 6=one pair of einzel lens, 7=ceramic spacer tube, not visible (four pieces), 8=electrical connection, 9=threaded rods (four pieces, stainless steel)

**Figure 8-17.** Einzel lens unit

**Caution** Do not tilt the plates when reinstalling. They must also be fitted exactly without leaving space. ▲

19. Slip the parts **4**, **5**, **6** and **7** onto the threaded rods, as shown in [Figure 8-17](#).
20. Slip on the washers (**3** in [Figure 8-17](#)) and screw on the nuts **2** in [Figure 8-17](#).
21. Make sure that all plates and spacer rings are aligned in parallel and no gaps are left in between, see also [Figure 8-11](#).

**Caution** The feedthroughs contain delicate ceramics. Do not break them! ▲

22. Connect the ion source connection leads to the pins of the source and to the feedthroughs of the ion source flange as shown in “[Connections to Ion Source](#)” on [page 8-32s](#).  
The plugs must fit tightly onto the pins.

**Note** Hold the pins of the feedthroughs with small pliers while inserting the plugs. ▲

## Checking the Ion Source

The position of the connection wires must be carefully checked. 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.

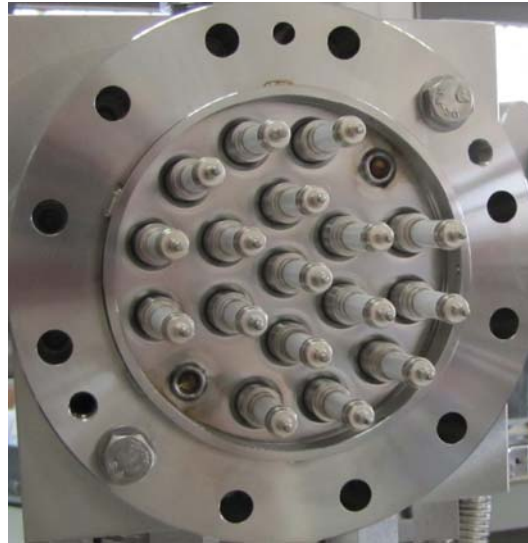
### ❖ To check the ion source

1. Use an ohmmeter and check the connection between the pins of the ion source socket and the ion source electrodes. See “[Connections to Ion Source](#)” on [page 8-32](#) for details.
2. 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<sup>12</sup> Ω (unless these are interconnected internally).
3. Check the gold gasket and the sealing surfaces of the ion source flange for radial scratches before inserting the ion source into the analyzer head.

**Note** 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.

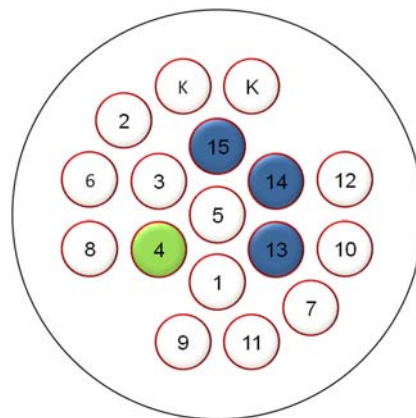
## Connections to Ion Source

Figure 8-18 shows the source feedthroughs as viewed from the outside.



**Figure 8-18.** Ion source feedthroughs

Figure 8-19 shows a schematic of the source feedthroughs as viewed from the outside.



Labeled Components: 1=electron trap (collector), 2=ionization housing, 3=extraction, 4=unused, 5=shield, 6=x-focus left, 7=x-focus right, 8=R-plate, 9=y-deflection top, 10=y-deflection bottom, 11=einzel lens bottom, 12=einzel lens top, 13=light bulb connection top, 14=light bulb connection center, 15=light bulb connection bottom, Cathode (filament) connections

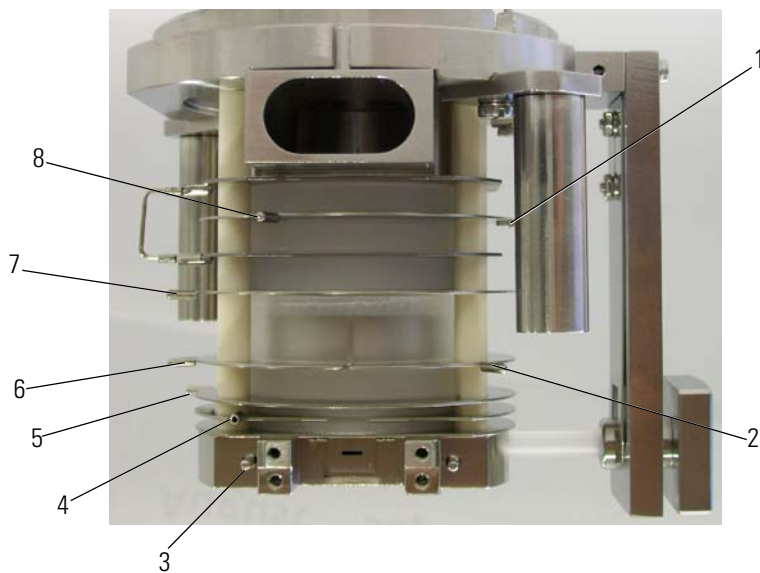
**Figure 8-19.** Source feedthroughs - schematic

Each feedthrough needs to be connected to the respective plate. See [Table 8-3](#) for voltage and specified position:

**Table 8-3.** Ion source connections

Plate name	Operating high voltage	Connects to	Plate voltage (high voltage disabled)
Electron trap (collector)	10000 to 10100	<b>2</b> in <a href="#">Figure 8-9</a>	0 to 100
Ionization housing	10000	<b>3</b> in <a href="#">Figure 8-20</a>	0
Cathode (filament)	9850 to 9950	<b>2</b> in <a href="#">Figure 8-6</a>	-150 to -50
Extraction electrode plate	9400 to 9900	<b>4</b> in <a href="#">Figure 8-20</a>	-600 to -100
Shield	9000 to 9500	<b>5</b> in <a href="#">Figure 8-20</a>	-500 to 0
X-focus left	8800 to 9050	<b>6</b> in <a href="#">Figure 8-20</a>	-250 to 0
X-focus right	8800 to 9050	<b>2</b> in <a href="#">Figure 8-20</a>	-250 to 0
R-plate	1800 to 2200	<b>7</b> in <a href="#">Figure 8-20</a>	-500 to 0
Y-deflection top	2150 to 2650	<b>8</b> in <a href="#">Figure 8-20</a>	-500 to 0
Y-deflection bottom	2150 to 2650	<b>1</b> in <a href="#">Figure 8-20</a>	-500 to 0
Einzel lens bottom	2150 to 2650	<b>1</b> in <a href="#">Figure 8-21</a>	-500 to 0
Einzel lens top	2150 to 2650	<b>4</b> in <a href="#">Figure 8-21</a>	-500 to 0

[Figure 8-20](#) shows the ion source connections to the individual plates:



Labeled Components: 1=y-deflection bottom, 2=x-focus right, 3=ionization housing, 4=extraction electrode plate, 5=shield, 6=x-focus left, 7=R-plate, 8=y-deflection top

**Figure 8-20.** Ion source lower part - plates and connections to ion source

Figure 8-21 shows the ion source connections to the einzel lens plates:

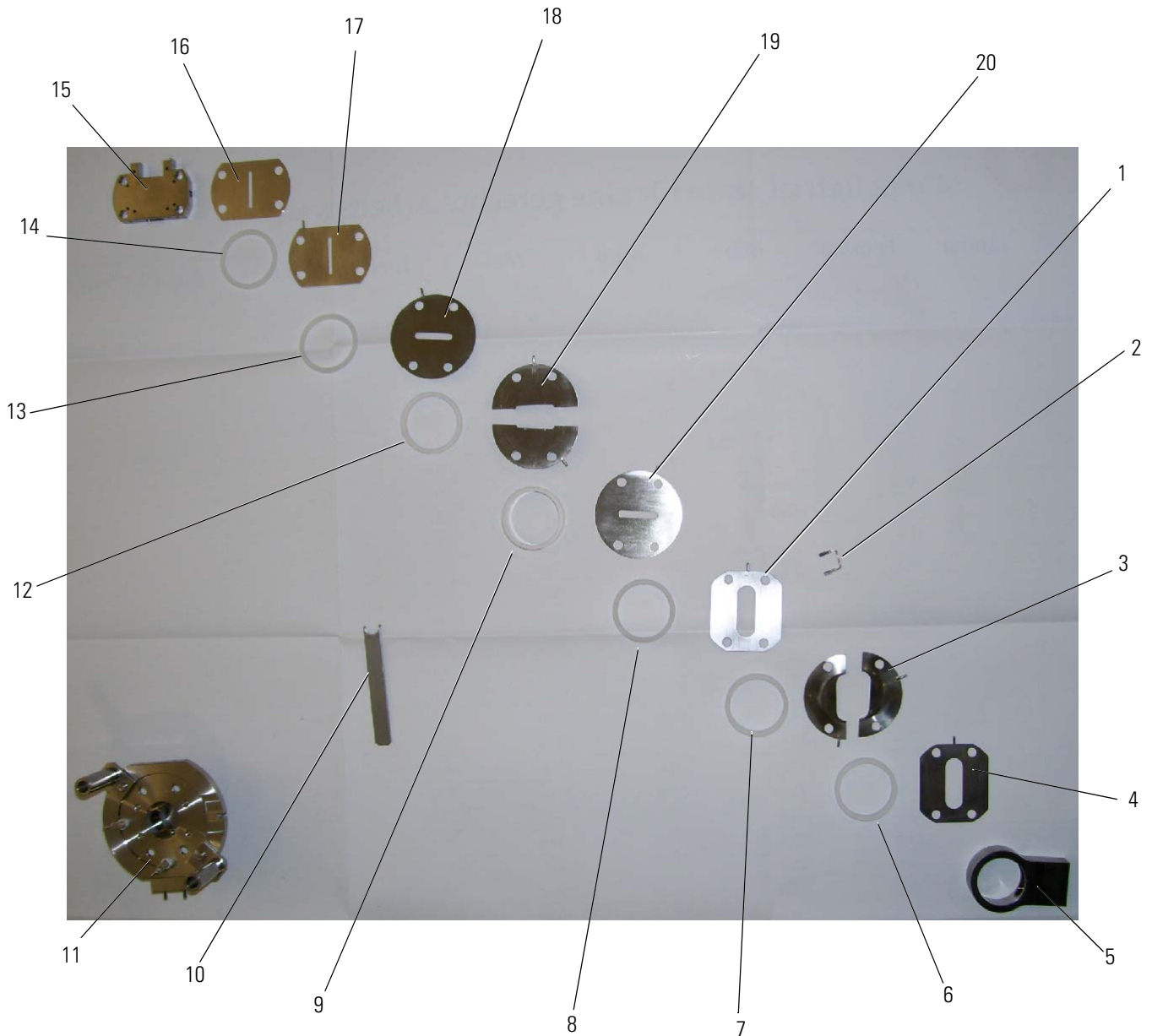


Labeled Components: 1=einzel lens bottom connection, 2=VISC opening, 3=groove for pin alignment, 4=einzel lens top connection

**Figure 8-21.** Einzel lens - connections to feedthroughs

## Ion Source Lower Part Details

Figure 8-22 shows in an exploded view, the individual plates of the lower part of the ion source:



Labeled Components: 1=grounding plate, 2=electrical connection for grounding plates, 3=y-deflection plates, 4=grounding plate, 5=distance ring for VISC, 6=spacer tube 5 mm (glass quartz), 7=spacer tube 5 mm (glass quartz), 8=spacer tube 5 mm (glass quartz), 9=spacer tube 10 mm (glass quartz), 10=source mounting tool, 11=ion source base, 12=spacer tube 5 mm (glass quartz), 13=spacer tube 2 mm (glass quartz), 14=spacer tube 2 mm (glass quartz), 15=ionization housing, 16=cover plate, 17=extraction plate, 18=shield, 19=x-focus lenses, 20=R-plate

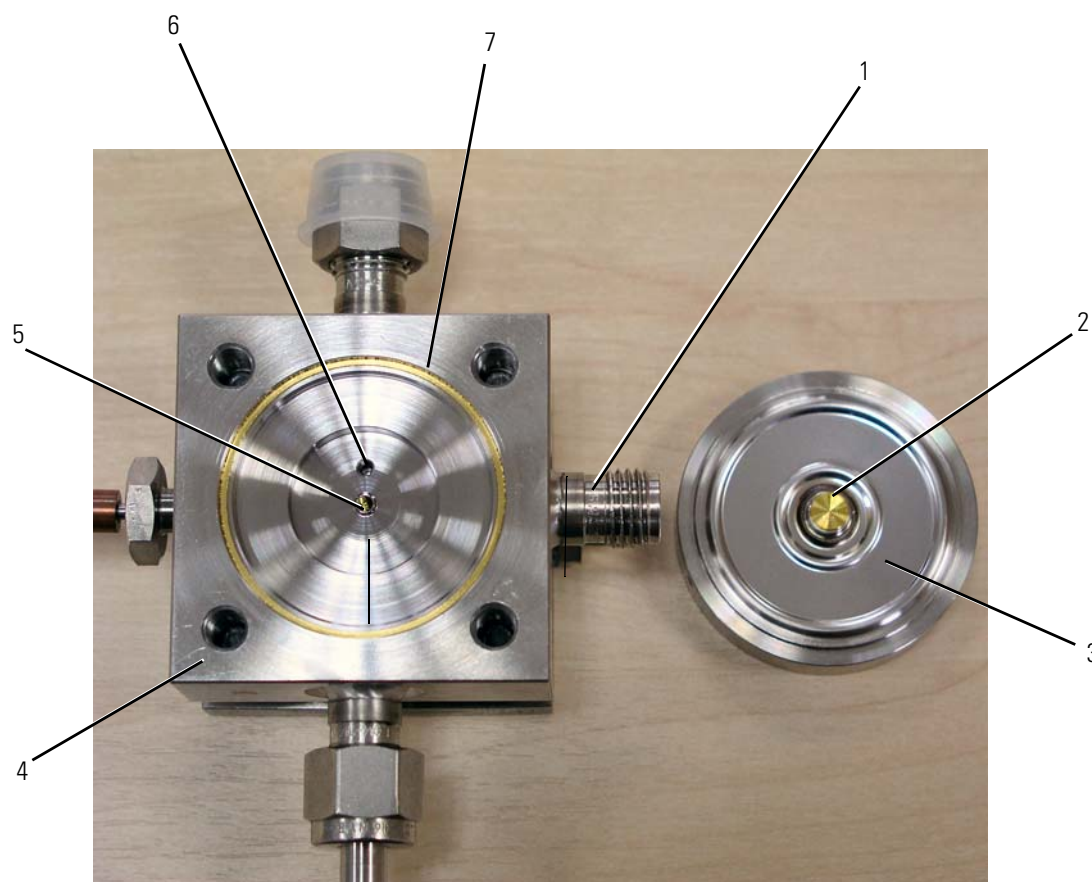
**Figure 8-22.** Ion source lower part - exploded view of plates

## Valves of Dual Inlet System

The Dual Inlet system is operated by pneumatic valves with a nominal closing pressure of 4 bar. Even though they are all made of stainless steel, after long-term operation they might be worn nevertheless. All the valves base upon the same construction principle. [Figure 8-23](#) shows such a valve with its high-vacuum side opened.

### Parts of a Valve

[Figure 8-23](#) shows a Dual Inlet system valve:

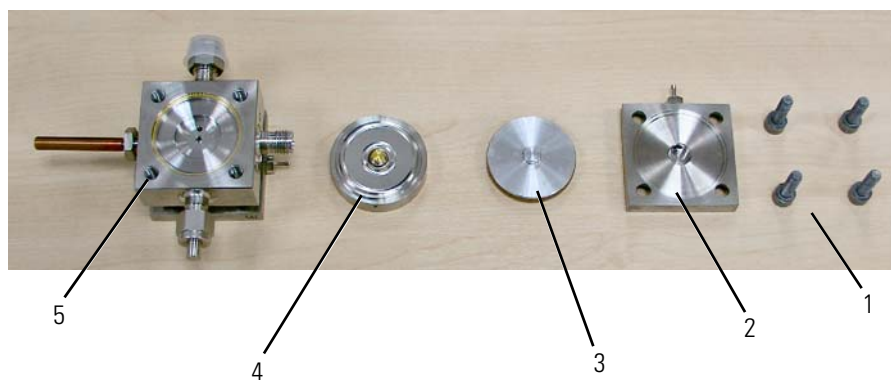


Labeled Components: 1=gas inlet (Swagelok connector) laterally welded on the valve block, 2=plunger (gold) fits exactly to the edge, 3=stainless steel membrane with valve plug that closes the valve, 4=valve block (stainless steel), 5=gas exhaust (knife), 6=gas inlet to the valve, 7=gasket (gold), seals valve block against stainless steel membrane

**Figure 8-23.** Dual Inlet system valve

The stainless steel membrane **3** is turned down by 180° and then laid onto the gold-made gasket **7**, with the plunger **2** oriented downwards.

The parts of a Dual Inlet system valve are shown in [Figure 8-24](#):



Labeled Components: 1=four screws to fasten covering cap, 2=covering cap, 3=actuator for compressed air (usually lying within rear side), 4=stainless steel membrane with valve plug that closes the valve (with sleeve attached to rear side), 5=valve block (stainless steel)

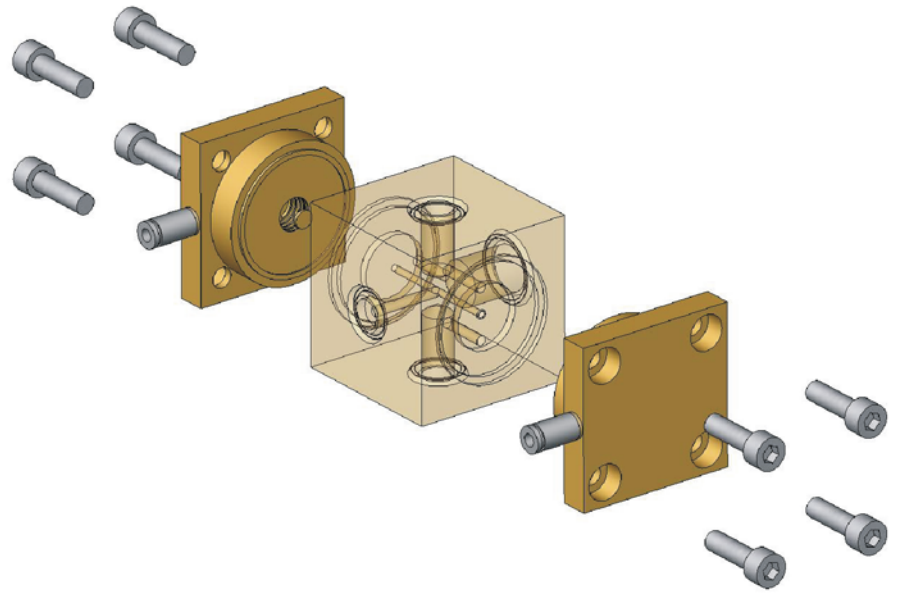
**Figure 8-24.** Details of Dual Inlet system valve

## Arrangement in Valve Blocks

Compressed air is either supplied by an optional compressor attached to the IRMS 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 block is used throughout all inlet modules. For plumbing the valve blocks are fitted with 1/4 in 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.

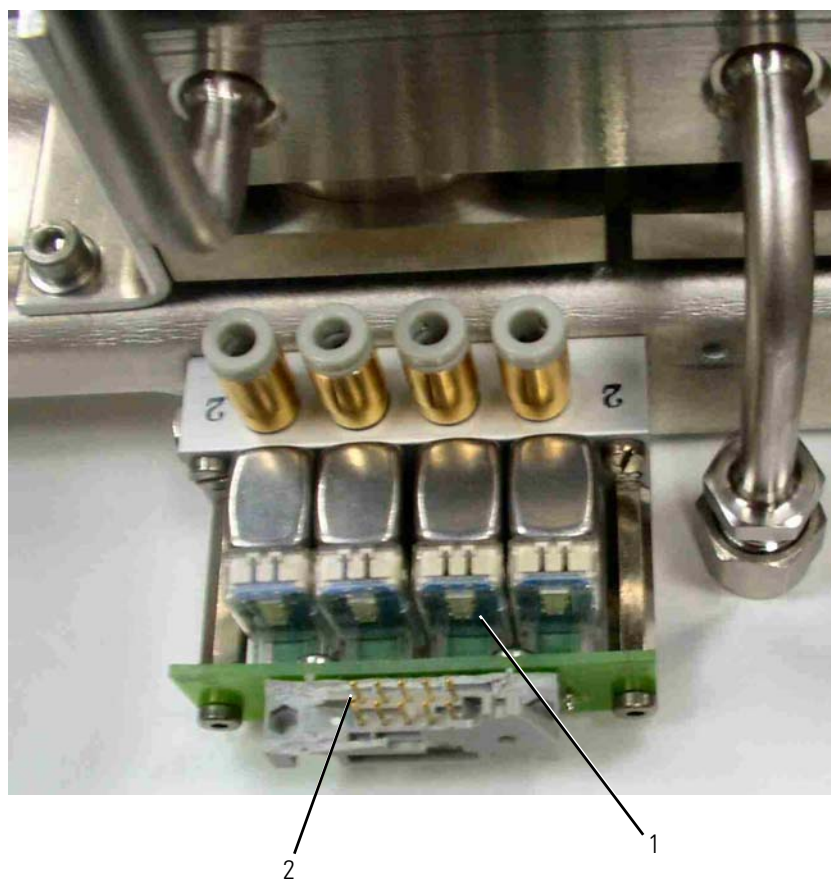
Figure 8-25 shows a double valve block:



**Figure 8-25.** Double valve block

## Manifold Block with Solenoid Valves

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 the Dual Inlet board. See [Figure 8-26](#) for manifold block.



Labeled Components: 1=four LEDs reveal individual switching status (on/off),  
2=connector to Dual Inlet board

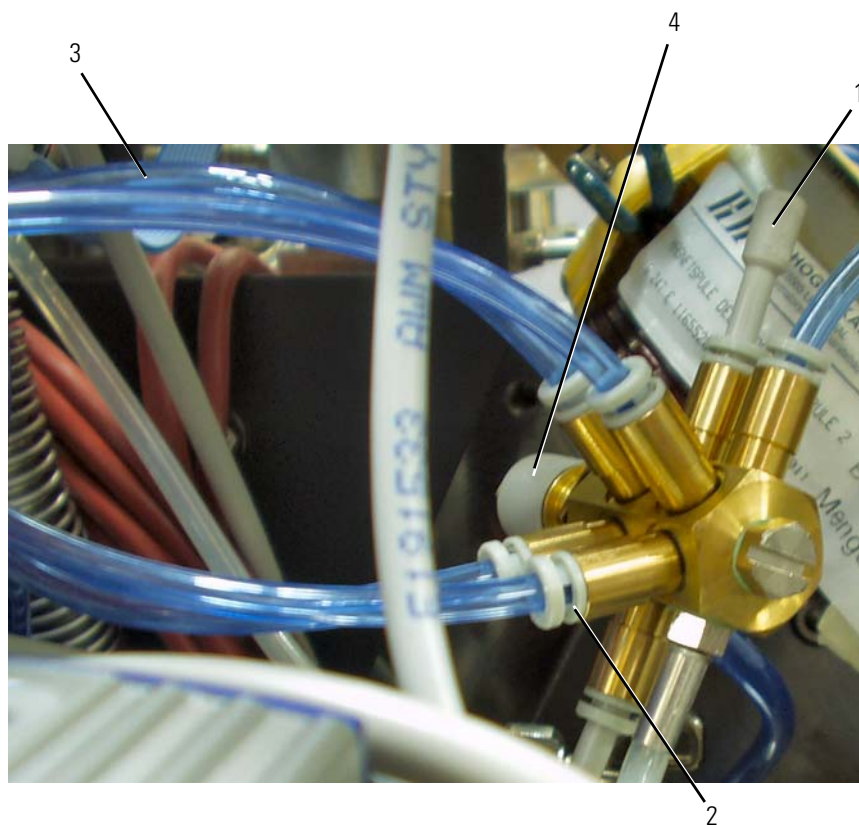
**Figure 8-26.** Manifold block with four solenoid valves

The solenoid valves are normally open (with the exception of TubeCracker). The working condition is signaled by a red LED located on the board. The actuators for compressed air transform a signal A into another signal B: they switch an electrical signal generated at the Dual Inlet board into a compressed air signal. Thereby, compressed air is provided which forcefully switches the actual valves.

**Note** In case of a power failure, all solenoid valves open automatically (exception: TubeCracker, where they close automatically). Thus, the pneumatic valves in the entire Dual Inlet system close avoiding its contamination. ▲

## Compressed Air Distributor

The compressed air distributor, [Figure 8-27](#), is part of the Dual Inlet system. Therefore, it is missing if no Dual Inlet system is available.



Labeled Components: 1=blind plug, 2=tubing connections, 3=tubings, 4=connection where compressed air enters

**Figure 8-27.** Compressed air distributor

The compressed air connections of the distributor are all equivalent ones. Compressed air enters at **4** in [Figure 8-27](#) and is then distributed to all compressed air valves.

The number of compressed air valves of the system depends on which particular options for the Dual Inlet system are available. If only few compressed air valves must be connected to the compressed air distributor, one or more blind plugs **1** in [Figure 8-27](#) allow to close the unused connections tightly.

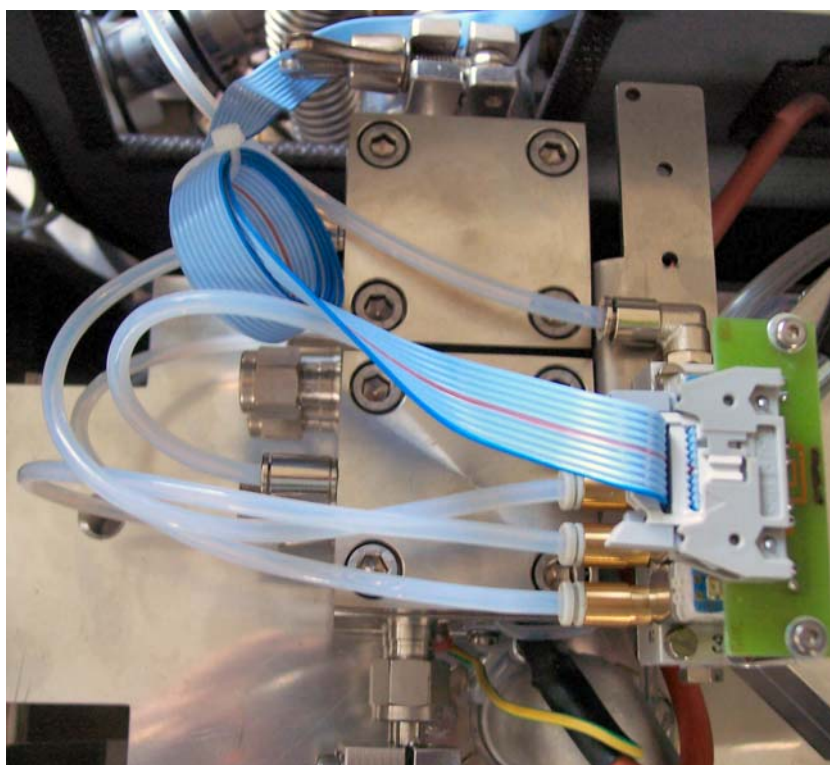
## Changeover Valve

The Changeover Valve allows to maintain a continuous, never interrupted flow of gas as it switches between reference gas flow and sample gas flow (in the range of some tens to some hundred  $\text{mbar} \times \text{L} \times \text{s}^{-1}$  each). Alternatingly, one gas flow is continuously led into the ion source (evacuated by analyzer turbo pump), whereas the other gas flow is continuously pumped off by the Dual Inlet system turbo pump.

## Layout

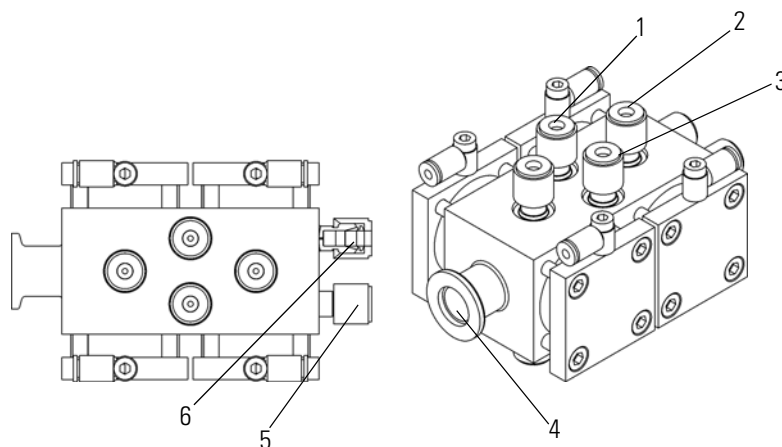
The Changeover valve consists of a single block attached to the analyzer housing and accepts the coupling of capillaries for sample and standard gas. It is operated automatically by Isodat or manually via the monitor display of the inlet schematic.

Figure 8-28 and Figure 8-29 show the layout and position of the Changeover valve in the analyzer. Made of one metallic block containing four individual valves, the Changeover valve allows to repeatedly alternate between sample gas and reference gas. Switching between *no gas into the ion source*, *gas from the right bellows into the ion source* and *gas from the left bellows into the ion source* is possible.



**Figure 8-28.** Changeover valve

The capillaries from the bellows of the Dual Inlet system – or from Kiel IV or a Microvolume – are connected at **1** and **3** in [Figure 8-29](#). **4** leads to Dual Inlet system turbo pump. **2** connects to the IRMS source inlet.



**Figure 8-29.** Schematic views of Changeover valve

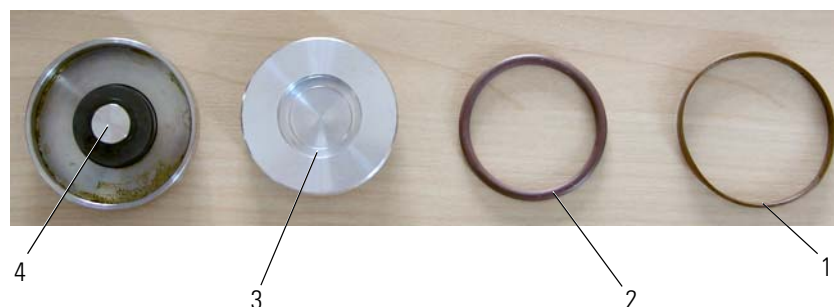
Alternatively to **1** in [Figure 8-29](#), the capillaries can be connected to **6**. Alternatively to **3**, the capillaries can be connected to **5** in [Figure 8-29](#).

## Pneumatic Valves

When a cylinder on top of the pneumatic valve is actuated by compressed air, the plunger (gold) presses a membrane underneath and thus blocks the valve. Gas transfer is not possible. Without compressed air, the cylinder is not actuated, the plunger does not press the membrane and thus does not block the valve. Gas transfer is possible.

### Parts of a Pneumatic Valve

[Figure 8-30](#) shows the individual parts of a pneumatic valve.



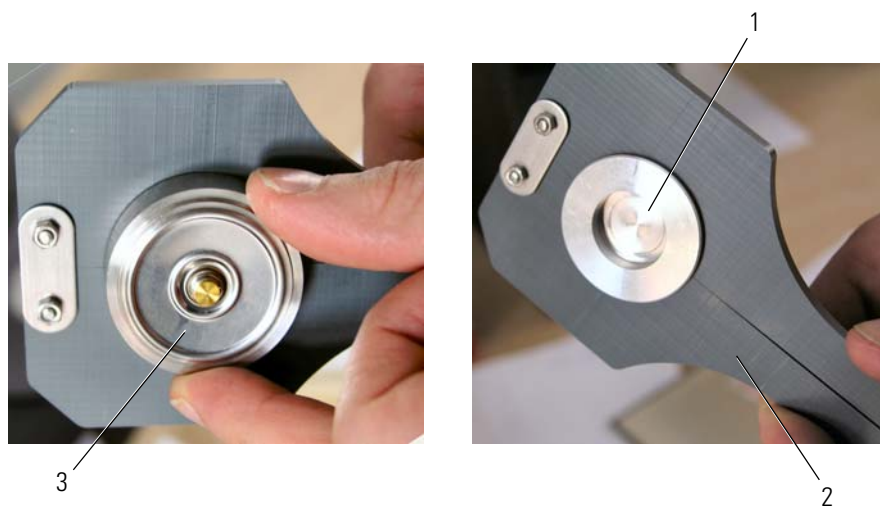
Labeled Components: 1=guide sleeve (Pertinax), 2=O-ring seal, 3=compressed air plunger, 4=sleeve (compressed air plunger moves within it)

**Figure 8-30.** Parts of a pneumatic valve

The compressed air plunger **3** moves within the sleeve **4**. The O-ring seal **2** seals the compressed air plunger against the sleeve. The guide sleeve **1** is arranged above the O-ring seal.

## Inserting a Pneumatic Valve

Figure 8-31 shows the pliers **2** to properly insert a pneumatic valve **3**.



Labeled Components: 1=compressed air plunger, 2=pliers, 3=pneumatic valve

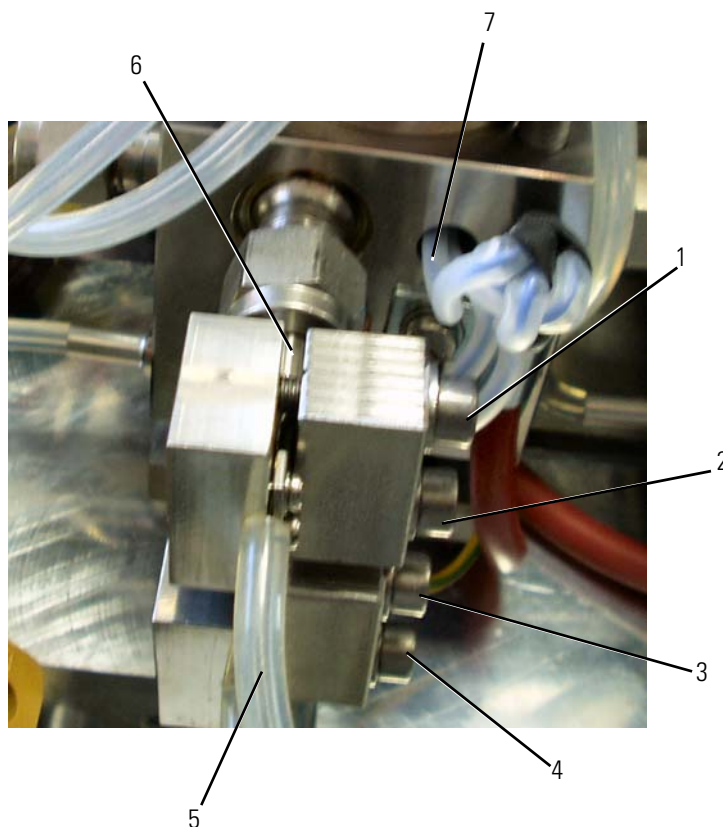
**Figure 8-31.** Pliers to insert a pneumatic valve

### ❖ To insert a pneumatic valve

1. Hold the pneumatic valve **3** tight within the pliers **2**, see left in [Figure 8-31](#).
2. Place the outer sleeve **4** in [Figure 8-30](#) above the pneumatic valve, see right in [Figure 8-31](#).

## Capillary Connections

Figure 8-32 depicts how a stainless steel capillary is connected to the Changeover valve.



Labeled Components: 1=crimping screw, 2=crimping screw, 3=crimping screw, 4=crimping screw, 5=stainless steel capillary, 6=crimping position, 7=position of heating cartridge

**Figure 8-32.** Capillary connections at Changeover valve

The stainless steel capillary **5** with the crimping position **6** is shown in Figure 8-32. **1** to **4** are the crimping screws.

A heating cartridge is positioned at **7** in Figure 8-32. Several cartridges are housed within the metal block. They serve to heat out the Changeover valve and usually need neither to be replaced nor maintained.

## Parts Lists

When ordering a part, always give the designation and the part number. Also provide the service number of the instrument in question. In this way, the processing of your order will be expedited.

Because of our modular instrument concept it might happen that parts are listed that are not supplied with your instrument. Note that listing a part does not imply that this part is necessary required for your particular instrument configuration.

This chapter list the spare parts for MAT 253.

## Basic Unit

Table 8-4 lists the parts of the basic unit:

**Table 8-4.** Parts list of basic unit (P/N 1136000)

P/N	Description
0545270	GASKET GOLD OD38XID36 5
0551600	GASK.GOLD OD117 5ID115 5
0693220	Centering Ring Aluminum DN 63 ISO-K
0569110	Edge sealing Aluminum DN 100
0439530	ROD CER. AD5 LG69 5
0783900	TUBE QUARTZ 4x2x23
0460060	Hex nut ISO4035-M2-A4
0783930	SPACER 35.6 X 30 X 2
0783920	Spacer 35.6 X 30 X 5
0783910	Spacer 35.6 X 30 X 10
0560570	SPACER 4 X 2 1 X 10
0560780	Spacer 6 X 4.2 X 4 Ceramic
0560760	SPACER 4 X 2 1 X 8
0430960	PLATE SPRING
0910450	Threaded Sleeve M8
0672460	Filament Tungsten
1147320	Lamp wiring MAT253 source
1220110	Halogenlamp 20W 12V - Osram
1050160	Fiber Reservoir TMH 260/262
1094301	OIL PREVAC. PUMP P 3 - 3 LTRS

## Dual Inlet (Option)

Table 8-5 lists the parts of the Dual Inlet system:

**Table 8-5.** Parts list optional Dual Inlet system (P/N 1082862)

P/N	Description
0703310	BELLOWS UNIT SMALL
0703330	MOTOR COMPLETE/Var.Vol.
0101660	HELICAL POT 100 KO 3 W
0653010	MEMBRAN COMPLETE
0653041	STAMP-AU-SEAL.TIP D=3 9
0553140	SET OF SEALING RINGS
0545270	GASKET GOLD OD 38 X ID 36 5
0534500	Metal Hose KF NW 16 X 500
0172350	FIBER RESERVOIR TPH/TMH62/64/71
1094301	OIL PREVAC. PUMP P 3 - 3 LTRS
1084890	Vent Valve 24V
0671182	CAPILLARY TUBE HEATABLE
0792800	FERRULE FR.1/4 in SWAGEL.
0792810	FERRULE R.1/4 in SWAGEL.
0521160	CUP Nut sst, 1/4 in

## Differential Pump Module (Option)

Table 8-6 lists the parts of the pump module:

**Table 8-6.** Parts list of optional differential Pump Module (P/N 1111121)

P/N	Description
0172350	FIBER RESERVOIR TPH/TMH62/64/71

# Glossary

This section lists and defines terms used in this manual. It also includes acronyms, metric prefixes, symbols, and abbreviations.

A B C D E F G H I J K L M N O P Q R S T U V W X Y Z

- A**
- A** ampere
- ac** alternating current
- ADC** analog-to-digital converter
- AP** acquisition processor
- API** atmospheric pressure ionization
- ASCII** American Standard Code for Information Interchange
- B**
- b** bit
- B** byte (8 b)
- baud rate** data transmission speed in events per second
- C**
- °C** degrees Celsius
- cfm** cubic feet per minute
- CI** chemical ionization
- CID** collision-induced dissociation
- cm** centimeter
- cm<sup>3</sup>** cubic centimeter
- Continuous-Flow (CF)** Automated preparation device and mass spectrometer in which sample analysis is conducted in a continuous stream of helium carrier gas.
- CPU** central processing unit (of a computer)
- CRC** cyclic redundancy check
- CRM** consecutive reaction monitoring
- <Ctrl>** control key on the terminal keyboard
- D**
- d** depth
- Da** dalton
- DAC** digital-to-analog converter
- dc** direct current
- driver** A device-specific control program that enables a computer to work with a particular device.
- DS** data system
- DSP** digital signal processor
- Dual Inlet (DI)** Inlet method in which a pure gas sample is admitted into an isotope ratio mass spectrometer (**IRMS**) by a variable volume bellows. A reference gas is admitted into the IRMS via a second variable volume bellows. The bellows are balanced to provide sample and reference signal responses of equal intensity.
- E**
- EI** electron ionization
- Elemental Analyzer (EA)** Automated sample preparation instrument in which samples are automatically converted into pure gases for isotope ratio analysis. An Elemental Analyzer contains the

following elements: (i) furnace for combustion, reduction or pyrolysis of sample material; (ii) chemical traps for analyte gas purification; (iii) gas chromatography for time separation of these analyte gases.

**EMBL** European Molecular Biology Laboratory

**<Enter>** Enter key on the terminal keyboard

**ESD** electrostatic discharge

**ESI** electrospray ionization

**eV** electron volt

## **F**

**f** femto ( $10^{-15}$ )

**°F** degrees Fahrenheit

**forepump** The pump that evacuates the foreline. A rotary-vane pump is a type of forepump.

**ft** foot

**FTP** file transfer protocol

**FWHM** Full Width at Half Maximum

## **G**

**g** gram

**G** Gauss; giga ( $10^9$ )

**GC** gas chromatograph; gas chromatography

**GC/MS** gas chromatograph / mass spectrometer

**GISP** Greenland Ice Sheet Precipitation. International reference standard for hydrogen and oxygen isotopes.

See also [SLAP](#) and [VSMOW](#).

**GUI** graphical user interface

## **H**

**h** hour

**b** height

**HPLC** High Performance Liquid Chromatography.

Standalone liquid chromatography system (or inlet for mass spectrometry detector).

**HV** high voltage

**Hz** hertz (cycles per second)

## **I**

**IAEA** International Atomic Energy Agency

**ICIS™** Interactive Chemical Information System

**ICL™** Instrument Control Language™

**ICP** inductively coupled plasma

**ICP-OES** inductively coupled plasma optical emission spectroscopy

**ID** inside diameter

**IEC** International Electrotechnical Commission

**IEEE** Institute of Electrical and Electronics Engineers

**in.** inch

**I/O** input/output

**ion optics** Focuses and transmits ions from the ion source to the mass analyzer.

**ion source** A device that converts samples to gas-phase ions.

**IRMS** isotope ratio mass spectrometer

## **K**

**k** kilo ( $10^3$ , 1000)

**K** kilo ( $2^{10}$ , 1024)

**KEGG** Kyoto Encyclopedia of Genes and Genomes

**kg** kilogram

## **L**

**l** length

**L** liter

**LAN** local area network

**lb** pound

**LC** Liquid chromatography. A process that separates a chemical mixture carried by liquid into components as a result of differential distribution of the solutes as they flow around or over a stationary or solid phase.

**LC/MS** liquid chromatograph / mass spectrometer

**LED** light-emitting diode

**log file** A text file, with a .log file extension, that is used to store lists of information.

**μ** micro ( $10^{-6}$ )

## M

**m** meter

**m** milli ( $10^{-3}$ )

**M** mega ( $10^6$ )

**M<sup>+</sup>** molecular ion

**MB** Megabyte (1048576 bytes)

**MH<sup>+</sup>** protonated molecular ion

**min** minute

**mL** milliliter

**mm** millimeter

**MS** mass spectrometer; mass spectrometry

**MS** MS<sup>n</sup> power: where n = 1

**MS/MS** MS<sup>n</sup> power: where n = 2

**MS<sup>n</sup>** MS<sup>n</sup> power: where n = 1 through 10

**m/z** Mass-to-charge ratio. An abbreviation used to denote the quantity formed by dividing the mass of an ion (in u) by the number of charges carried by the ion. For example, for the ion C<sub>7</sub>H<sub>7</sub><sup>2+</sup>, m/z=45.5.

## N

**n** nano ( $10^{-9}$ )

**Natural Abundance** The concentration of isotopes as found in nature.

**NCBI** National Center for Biotechnology Information (USA)

**NIST** National Institute of Standards and Technology (USA)

**noise** Any random disturbance that obscures the clarity of a signal.

## O

**OD** outside diameter

**Ω** ohm

**outlier** A calibration data point that does not appear to correlate to other calibration data points within experimental error.

## P

**p** pico ( $10^{-12}$ )

**Pa** pascal

**PCB** printed circuit board

**PE** protective earth

**PID** proportional / integral / differential

**P/N** part number

**P/P** peak-to-peak voltage

**ppm** parts per million

**psig** pounds per square inch, gauge

## R

**RAM** random access memory

**relative standard deviation** A measure of the dispersion of a group of measurements relative to the mean of the group. Relative standard deviation is expressed as a percentage of the average value. The percent relative standard deviation is calculated as:

$$\%RSD = 100 (S / \bar{X})$$

where S is the **standard deviation** and  $\bar{X}$  is the sample mean.

**RF** radio frequency

**RMS** root mean square

**ROM** read-only memory

**rotary-vane pump** A mechanical vacuum pump that establishes the vacuum necessary for the proper operation of the turbomolecular pump. (Also called a roughing pump or forepump.)

**RS-232** An accepted industry standard for serial communication connections. This Recommended Standard (RS) defines the specific lines and signal characteristics used by serial communications controllers to standardize the transmission of serial data between devices.

## S

**s** second

**serial port** An input/output location (channel) for serial data transmission.

**SIM** selected ion monitoring

**SLAP** Standard Light Antarctic Precipitation; international reference standard for hydrogen and oxygen isotopes.

See also [VSMOW](#).

**SRM** selected reaction monitoring

**standard deviation** In statistics, the standard deviation is a measure of the dispersion of a group of measurements. For example, masses, times, or intensities. Standard deviation is calculated as follows:

$$s = \sqrt{\text{Var}(x_1 \dots x_n)}$$

where Var (X<sub>1</sub>...X<sub>N</sub>) is the variance.

See also [relative standard deviation](#).

## T

**TCP/IP** transmission control protocol / Internet protocol

**TIC** total ion current

**Torr** A unit of pressure, equal to 1 mm of mercury and 133.32 Pa.

**turbomolecular pump** A vacuum pump that provides a high vacuum for the mass spectrometer and detector system.

## U

**u** atomic mass unit

**UHV** ultra high vacuum

## V

**V** volt

**V ac** volts alternating current

**V dc** volts direct current

**vol** volume

**VCDT** Vienna Canyon Diablo Troilite; international reference standard for sulfur isotopes.

**VPDB** Vienna Pee Dee Belemnite; international reference standard for carbon and oxygen isotopes.

**VSMOW** Vienna Standard Mean Ocean Water; international reference standard for hydrogen and oxygen isotopes.

See also [SLAP](#).

## W

**w** width

**W** watt

**WEEE** European Union Waste Electrical and Electronic Equipment Directive. Provides guidelines for disposal of electronic waste.

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