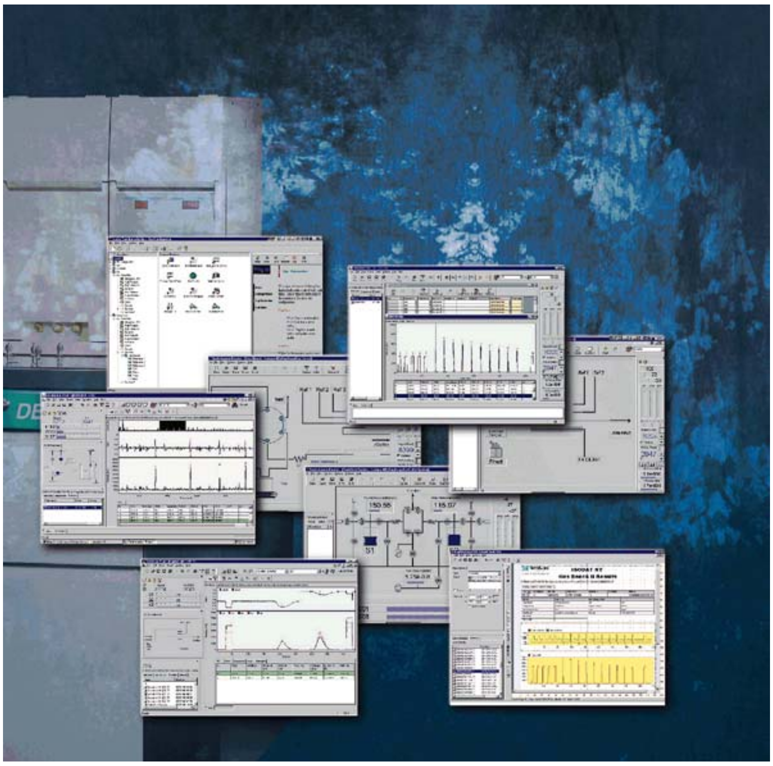
ISODAT NT



OPERATING MANUAL

Issue 06/2002

Ident. No. 109 24 81

Thermo Finnigan

Thermo Finnigan

Thermo Finnigan MAT GmbH
Postfach 1401 62
28088 Bremen
Germany

Reparatur-Begleitkarte*)
Repair-Covering Letter

Absender:
Despachter:

Geräte-Type:
Instrument Type:

Service-Nr.:
Service No ☐

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

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

Bitte detaillierte Angaben machen / Please specify in detail

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

Ja/Yes ☐ Nein/No ☐

Die Anlage ist außer Funktion
The system is out of function

Ja/Yes ☐ Nein/No ☐

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

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

Datum / date

Unterschrift / signature

---

*) Bitte vollständig ausfüllen / Please fill in completely

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1 Installation

2 Configurator

3 Instrument Control

4 Dual Inlet Measurements

5 Continuous Flow Measurements

6 Result Handling

7 Diagnosis

ISODAT NT

OPERATING MANUAL

1

INSTALLATION

Issue 06/2002

Thermo Finnigan

ISODAT NT INSTALLATION 1

1.1 INTRODUCTION

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

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

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

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

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

1.2 INSTALLATION OF BOARD DRIVERS

The following pages contain descriptions for the installation of drivers for the various extension boards of the computer.

1.2.1 "SERIAL SOLUTIONS" COM PORT EXTENSION BOARD DRIVER

The PCI Dual RS 232 Card is already part of your computer. Its driver by "Serial Solutions Products" makes available two additional COM Ports. Thus, install it only, if your computer is equipped with merely one COM Port.

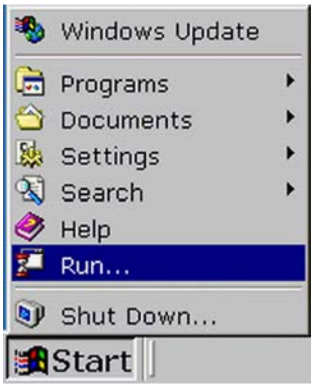
Install it once only, namely when you newly install your operating system.

⚠ ***Close all other applications! Otherwise, an error message might occur.***

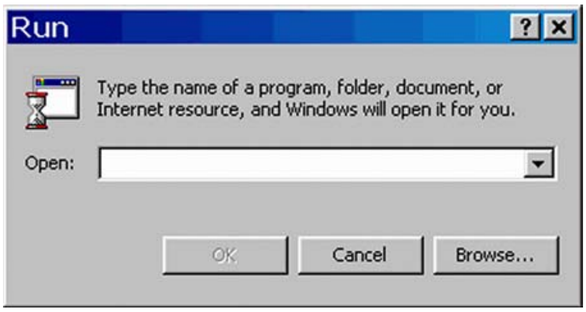
⚠ ***If your computer hangs up during either installation or reboot, shut it down and try to install again.***

If your system hangs up again, plug the PCI Dual RS 232 Card to another slot. Then only start your system without reinstallation.

- To install the driver, insert the "Serial Solutions" CD into your drive and close it. The CD is shipped with your system.
- If Auto Run is deactivated on your computer, press the ***Start*** button and then ***Run***.



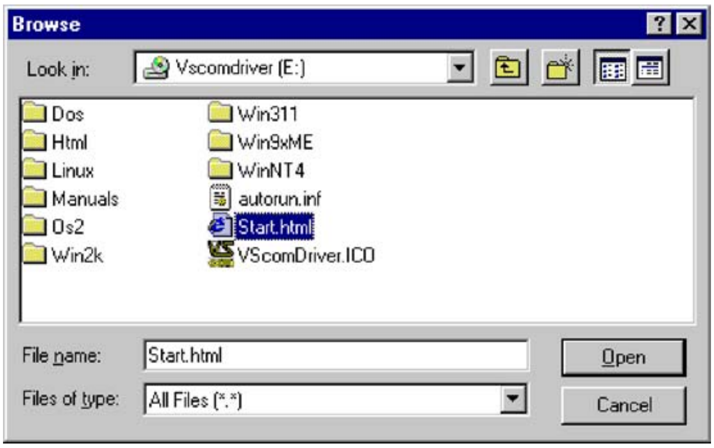
- Click ***Browse***.



Type the name of a program, folder, document, or Internet resource, and Windows will open it for you.

Open:

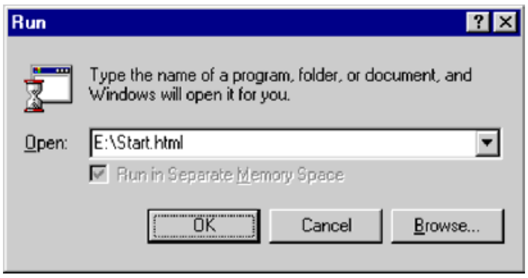
- Select your CD drive e.g. ***E:\Vscomdriver***. From the ***Files of type*** pulldown menu choose ***All Files (*.*)***. Click the file ***Start.html*** and then ***Open*** (or double-click ***Start.html***).



File name: Start.html

Files of type: All Files (*.*)

- Press ***OK***.

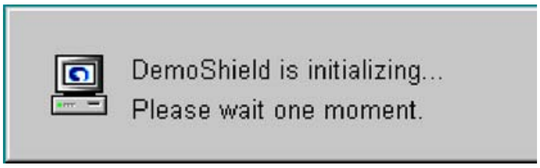


Type the name of a program, folder, or document, and Windows will open it for you.

Open: E:\Start.html

☑ Run in Separate Memory Space

- Wait, while the DemoShield is initializing.

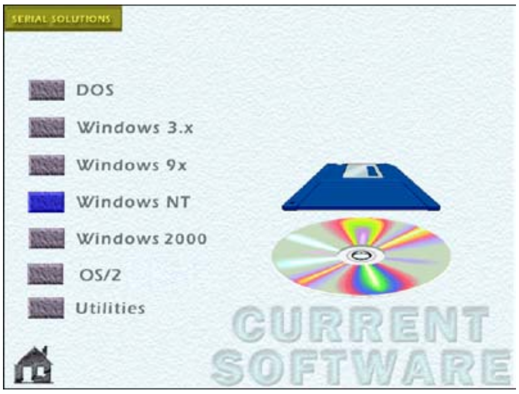


DemoShield is initializing...
Please wait one moment.

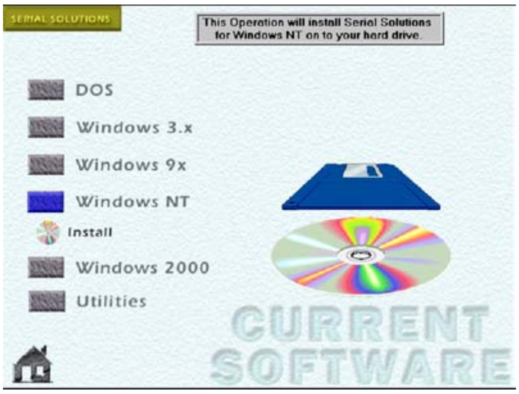
- On the Installation Screen, click ***Software***.

- ➢ Press ***Current***.

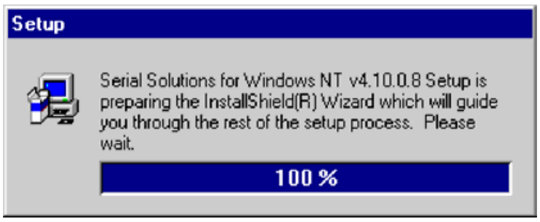
- ➢ Click ***Windows NT***.



- ➢ Press ***Install***.


This Operation will install Serial Solutions for Windows NT on to your hard drive.

- Wait, while Setup is preparing the InstallShield® Wizard.

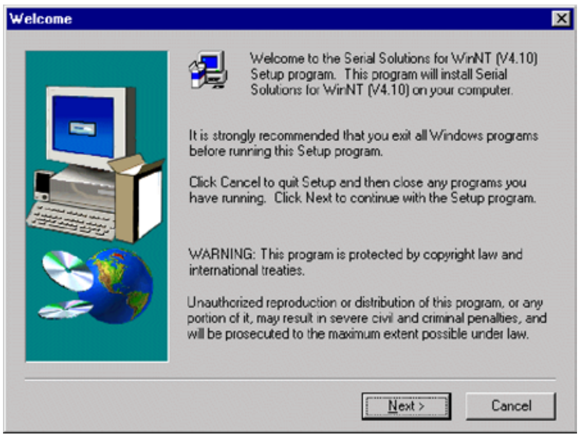


Setup

Serial Solutions for Windows NT v4.10.0.8 Setup is preparing the InstallShield(R) Wizard which will guide you through the rest of the setup process. Please wait.

100 %

- Carefully read the instructions about closing all applications and copyright law. Click ***Next*** to continue.
  (If Setup has found a previously installed version, continue as described below).



Welcome

Welcome to the Serial Solutions for WinNT (V4.10) Setup program. This program will install Serial Solutions for WinNT (V4.10) on your computer.

It is strongly recommended that you exit all Windows programs before running this Setup program.

Click Cancel to quit Setup and then close any programs you have running. Click Next to continue with the Setup program.

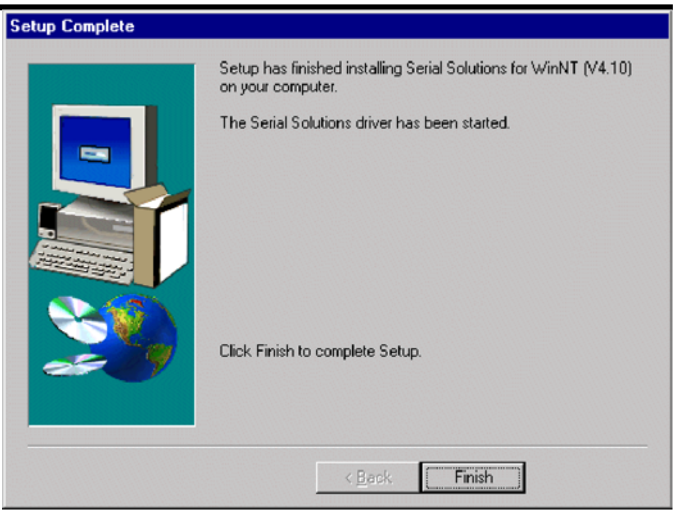
WARNING: This program is protected by copyright law and international treaties.

Unauthorized reproduction or distribution of this program, or any portion of it, may result in severe civil and criminal penalties, and will be prosecuted to the maximum extent possible under law.

- Wait, while Setup is copying the files.

Drivers

100 %

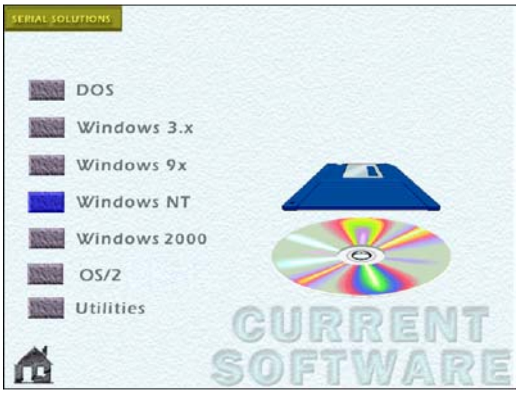


Setup has finished installing Serial Solutions for WinNT (V4.10) on your computer.

The Serial Solutions driver has been started.

Click Finish to complete Setup.

- ➢ Serial Solutions for Win NT has been installed on your computer.
- ➢ Click ***Finish*** to complete Setup.

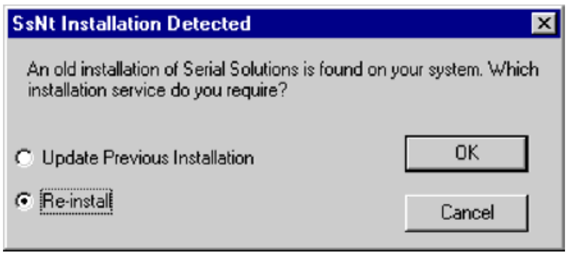


- ➢ Press the ***House*** button.

- ➢ Click ***Quit***. Then reboot your system.

1.2.1.1 REINSTALLING A PREVIOUSLY INSTALLED SERIAL SOLUTIONS DRIVER

- "Serial Solutions" Setup has detected a previously installed version.
- Choose ***Re-install***.

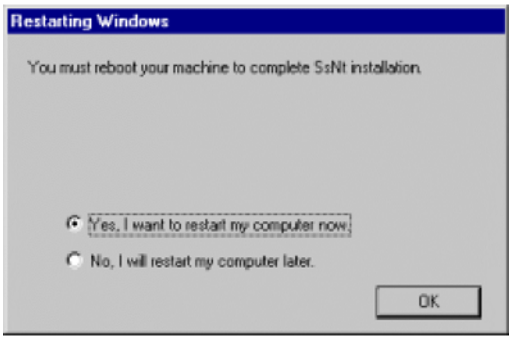


SsNt Installation Detected

An old installation of Serial Solutions is found on your system. Which installation service do you require?

○ Update Previous Installation

● Re-install



Restarting Windows

You must reboot your machine to complete SsNt installation.

● Yes, I want to restart my computer now.

○ No, I will restart my computer later.

- Select ***Yes, I want to restart my computer now*** and press ***OK*** to reboot.

NOTE: ***If your computer hangs up during either installation or reboot, shut it down and try to install again.***

If your system hangs up again, plug the PCI Dual RS 232 Card to another slot. Then restart your system without reinstallation

1.2.2 INSTALLING IEEE INTERFACE BOARD DRIVERS

Some peripherals (e.g. equilibration unit, carbonate device) require the installation of an IEEE board in the computer. The installation of the driver for this board is described on the following pages.

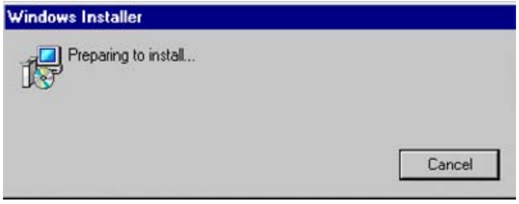
1.2.2.1 "NATIONAL INSTRUMENTS" GPIB PCI IEEE BOARD - INSTALLATION

Install the driver from the ***"National Instruments" NI 488.2 CD*** shipped with your system.

If ***Adobe Acrobat Reader*** is not installed on your computer, it can be installed later. It will be needed to read "National Instruments" Help files.

- ***Close all other applications!***

  Insert the CD into your CD drive and close it. The box shown above appears.
- Click ***Install NI-488.2 Software for Windows*** (turning into blue).



Preparing to install...

- Wait, while Installation is being prepared.

Welcome to the NI-488.2 for Windows Installation Wizard

Searching for installed applications...

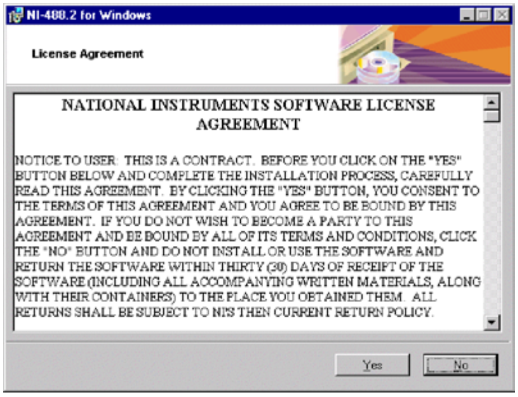
- Wait, while the NI-488.2 Installation Wizard appears and searches for previously installed applications.

- Click ***Next***.

Welcome to the NI-488.2 1.70 Installation Wizard

Click Next to continue with the setup program or Cancel to quit.

- Carefully read the "National Instruments" Software License Agreement. To continue press ***Yes***.

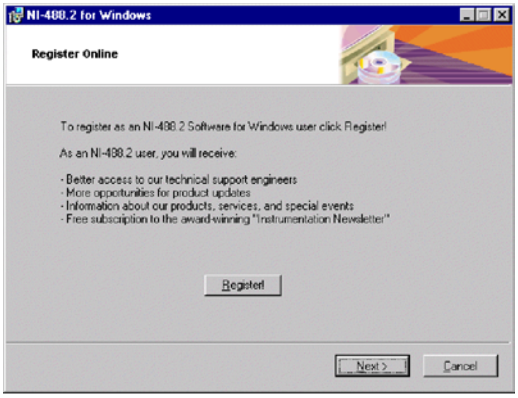


License Agreement

NATIONAL INSTRUMENTS SOFTWARE LICENSE AGREEMENT

NOTICE TO USER: THIS IS A CONTRACT. BEFORE YOU CLICK ON THE "YES" BUTTON BELOW AND COMPLETE THE INSTALLATION PROCESS, CAREFULLY READ THIS AGREEMENT. BY CLICKING THE "YES" BUTTON, YOU CONSENT TO THE TERMS OF THIS AGREEMENT AND YOU AGREE TO BE BOUND BY THIS AGREEMENT. IF YOU DO NOT WISH TO BECOME A PARTY TO THIS AGREEMENT AND BE BOUND BY ALL OF ITS TERMS AND CONDITIONS, CLICK THE "NO" BUTTON AND DO NOT INSTALL OR USE THE SOFTWARE AND RETURN THE SOFTWARE WITHIN THIRTY (30) DAYS OF RECEIPT OF THE SOFTWARE (INCLUDING ALL ACCOMPANYING WRITTEN MATERIALS, ALONG WITH THEIR CONTAINERS) TO THE PLACE YOU OBTAINED THEM. ALL RETURNS SHALL BE SUBJECT TO NI'S THEN CURRENT RETURN POLICY.

- Take your registration decision. Then press ***Next***.



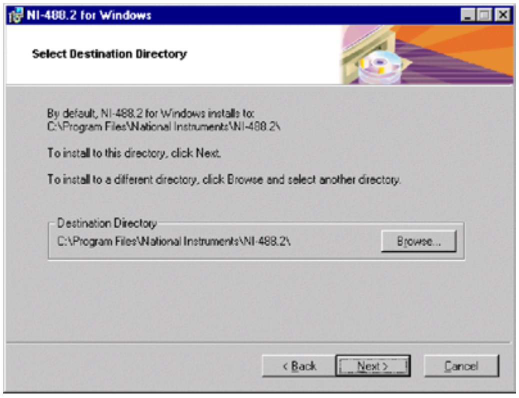
Register Online

To register as an NI-488.2 Software for Windows user click Register!

As an NI-488.2 user, you will receive:

- Better access to our technical support engineers
- More opportunities for product updates
- Information about our products, services, and special events
- Free subscription to the award-winning "Instrumentation Newsletter"

- To select the destination directory, either accept the proposed path and press ***Next*** or select another one by the ***Browse*** button.



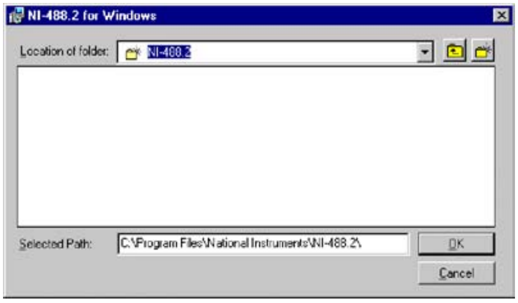
By default, NI-488.2 for Windows installs to:
C:\Program Files\National Instruments\NI-488.2\

To install to this directory, click Next.

To install to a different directory, click Browse and select another directory.

Destination Directory: C:\Program Files\National Instruments\NI-488.2\

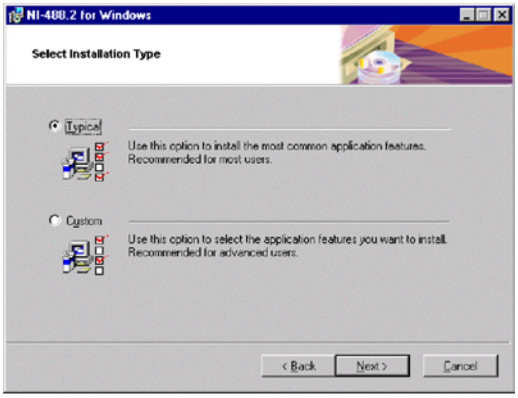
- If you pressed the ***Browse*** button, a box appears. From the ***Location of folder*** pulldown menu, choose the alternative destination folder. Note the according entry in the ***Selected Path*** box. Finally, press ***OK***.



Location of folder: NI-488.2

Selected Path: C:\Program Files\National Instruments\NI-488.2\

- Select the Installation type.
  Typical is recommended for most users, while ***Custom*** is recommended for advanced users. Finally, press ***Next***.



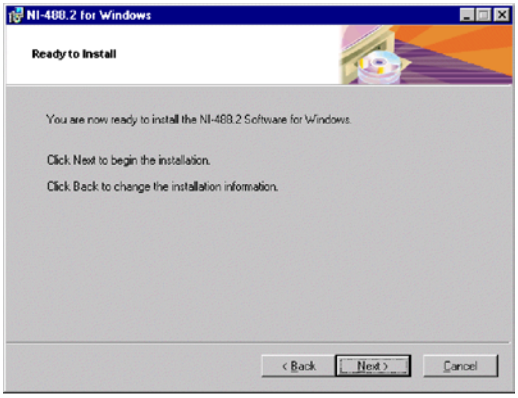
● Typical

Use this option to install the most common application features. Recommended for most users.

○ Custom

Use this option to select the application features you want to install. Recommended for advanced users.

- The NI-488.2 software is now ready to be installed. To begin the Installation press ***Next***.

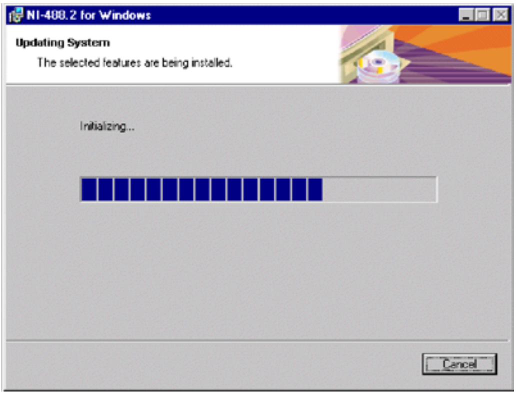


You are now ready to install the NI-488.2 Software for Windows.

Click Next to begin the installation.

Click Back to change the installation information.

- ➢ Wait, while Initialization begins.

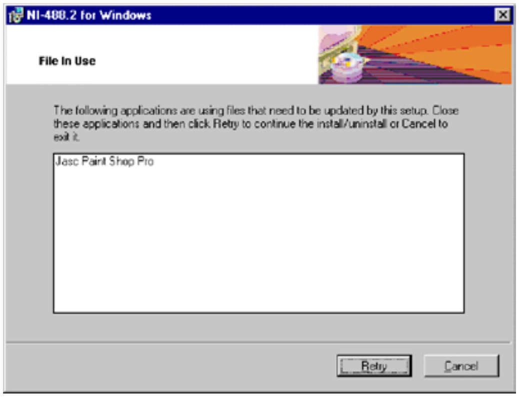


Updating System

The selected features are being installed.

Initializing...

- ➢ If you did not close all other applications yet (as was recommended, you are reminded to do it now:
  Click on the open applications' entries right to the ***Start*** button and close all of them.
  Finally, press ***Retry***.

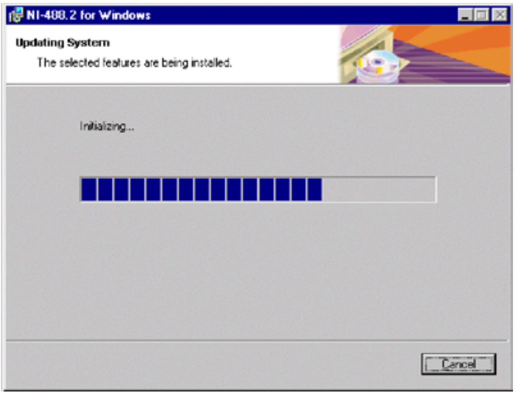


File In Use

The following applications are using files that need to be updated by this setup. Close these applications and then click Retry to continue the install/uninstall or Cancel to exit it.

Jasc Paint Shop Pro

- ➢ Wait while the Initialization is terminated.

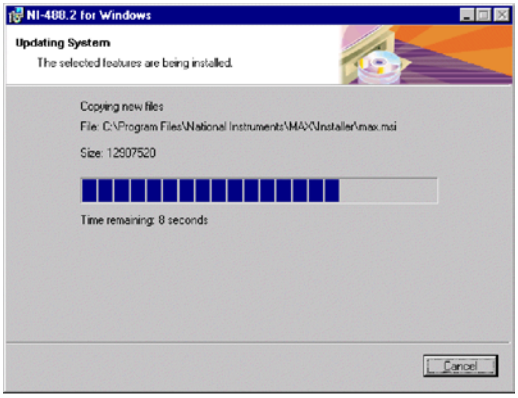


Updating System

The selected features are being installed.

Initializing...

- Wait, while the files are copied.



The selected features are being installed.

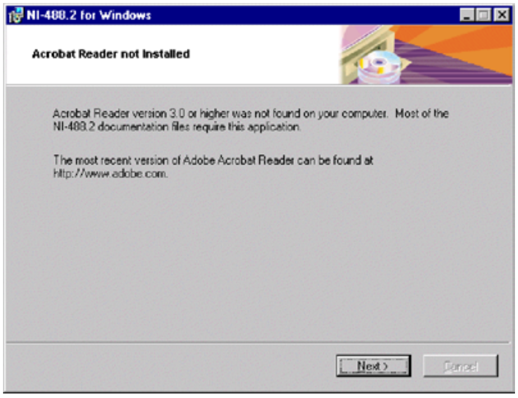
Copying new files

File: C:\Program Files\National Instruments\MAX\Installer\max.msi

Size: 12907520

Time remaining: 8 seconds

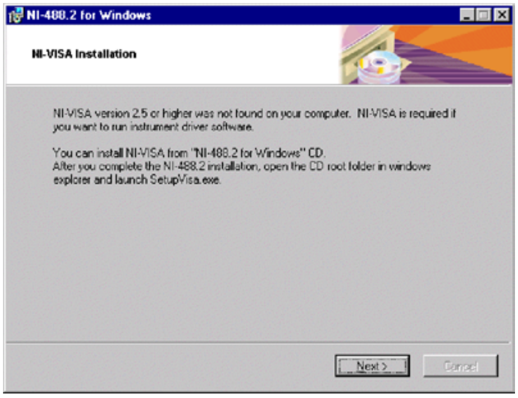
- Possibly, you might be reminded that Acrobat Reader 3.0 (or higher) was not found on your system.
  It is necessary to read "National Instruments" documentation files, but you can install them later.
  Press ***Next***.



Acrobat Reader version 3.0 or higher was not found on your computer. Most of the NI-488.2 documentation files require this application.

The most recent version of Adobe Acrobat Reader can be found at http://www.adobe.com.

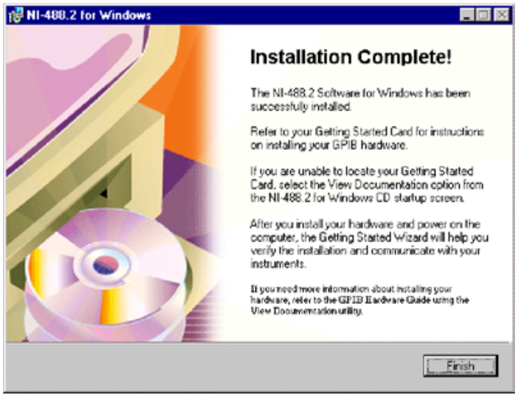
- This box informs about the separate NI-VISA Installation. This will be done after this installation is finished (see Chapter 1.2.2.4).
  Click ***Next***.



NI-VISA version 2.5 or higher was not found on your computer. NI-VISA is required if you want to run instrument driver software.

You can install NI-VISA from "NI-488.2 for Windows" CD.
After you complete the NI-488.2 installation, open the CD root folder in windows explorer and launch SetupVisa.exe.

- The NI-488.2 Software has been successfully installed. Click ***Finish***.



Installation Complete!

The NI-488.2 Software for Windows has been successfully installed.

Refer to your Getting Started Card for instructions on installing your GPIB hardware.

If you are unable to locate your Getting Started Card, select the View Documentation option from the NI-488.2 for Windows CD startup screen.

After you install your hardware and power on the computer, the Getting Started Wizard will help you verify the installation and communicate with your instruments.

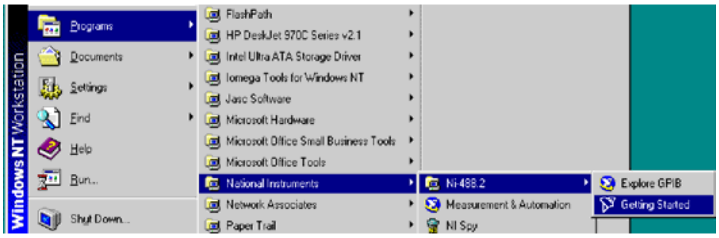
If you need more information about installing your hardware, refer to the GPIB Hardware Guide using the View Documentation utility.

- On your desktop, note the new NI-Icon.

1.2.2.2 "NATIONAL INSTRUMENTS" GPIB PCI IEEE BOARD - CONFIGURATION

After having installed the GPIB PCI driver, ***configure*** it now as shown below.

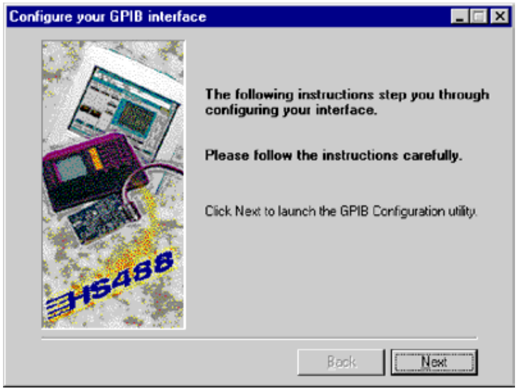
- To configure it press ***Start > Programs > National Instruments > NI 488.2 > Getting Started***.



- Point at ***Configure your GPIB Interface*** with your mouse and click.

Click on the following steps to use the NI-488.2 Software for Windows with your GPIB instruments:

- On the next window, click on the ***Next*** button to begin the Configuration.

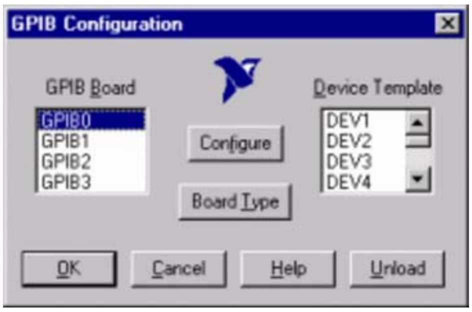


The following instructions step you through configuring your interface.

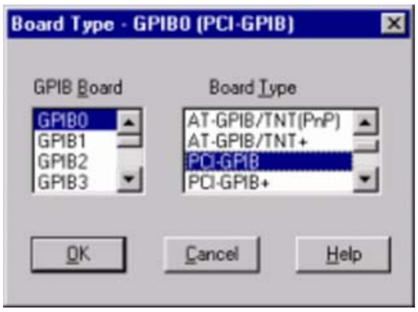
Please follow the instructions carefully.

Click Next to launch the GPIB Configuration utility.

- On the following window, press the ***Board Type*** button.



- From the ***Board Type*** pulldown list on the right, select ***PCI-GPIB***. Press ***OK***.

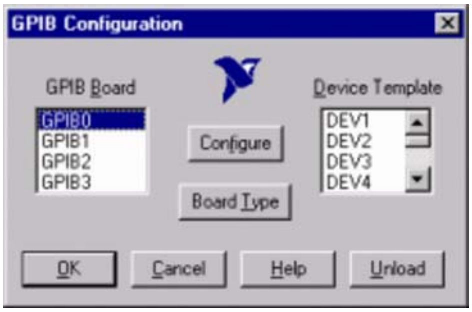


Board Type - GPIB0 (PCI-GPIB)

GPIB Board: GPIB0

Board Type: PCI-GPIB

- Press ***Configure***.



GPIB Configuration

- Press ***Software >>***.

Configure - GPIB0 (PCI-GPIB)

Hardware Settings

00b33bf9 Serial Number

500nsec Bus Timing

Disabled Cable Length for High-Speed

- On the next window, tick the checkboxes as shown in the figure right. Then click the ***OK*** button and confirm by ***OK*** on the following window (see above).

Configure - GPIB0 (PCI-GPIB)

Hardware Settings

00be0207 Serial Number

500nsec Bus Timing

Disabled Cable Length for High-Speed

GPIB Address

Primary: 0

Secondary: None

Advanced Items

☑ System Controller

☐ Assert REN when SC

☑ Enable Auto Polling

☑ Enable CIC Protocol

10sec I/O Timeout

Default Parallel Poll Duration

Termination

☐ Terminate Read on EOS

☐ Set EOI with EOS on Write

☐ 8-bit EOS Compare

☑ Send EOI at end of Write

0 EOS Byte

The configuration is finished now and the system starts the verification of the board driver (see Chapter 1.2.2.4).

Under some circumstances, the following window may appear.

- ➢ The error statement shown on the message box in the figure right indicates that the software cannot be started. Press ***OK*** to close the window.
  Check whether your Interface Card is properly inserted and then repeat the configuration of the GPIB-PCI driver.

Error starting NI-488.2 software

The NI-488.2 Configuration utility is unable to start the NI-488.2 software.

You need to examine the events logged by the NI-488.2 software to the NT Event Viewer for a complete description of the reason why the software failed to start properly.

The NI-488.2 Troubleshooting Wizard can help you use the Windows NT Event Viewer.

Run the NI-488.2 Troubleshooting wizard from Measurement and Automation Explorer, which is started from Start>>Programs>>National Instruments NI-488.2>>Explore GPIB. Start troubleshooting by selecting Help>>Troubleshooting>>NI-488.2 Troubleshooting Wizard.

1.2.2.3 "NATIONAL INSTRUMENTS" GPIB PCI IEEE BOARD - VERIFICATION

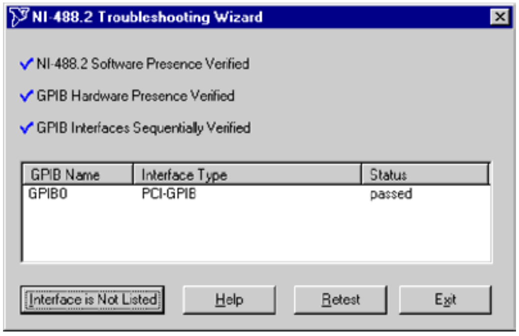
After installing and configuring your GPIB-PCI driver, ***verify*** it now as shown below.

- ➢ Point at ***Verify your hardware and software installation*** and click.

NI-488.2 Getting Started Wizard

Click on the following steps to use the NI-488.2 Software for Windows with your GPIB instruments:

- The NI-488.2 Software, GPIB Hardware and GPIB Interface(s) are successively being verified. A blue mark (✓) indicates successful verification. A red mark (✗) indicates that verification failed. If the red mark indicates that verification of your GPIB interface failed, press ***Help*** and finally ***Retest***.



✓ NI-488.2 Software Presence Verified
✓ GPIB Hardware Presence Verified
✓ GPIB Interfaces Sequentially Verified

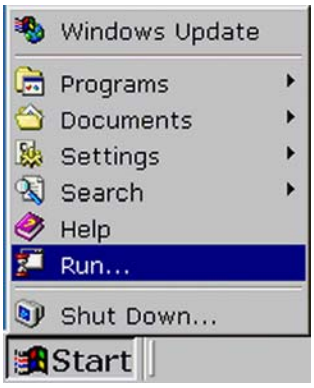
GPIB Name	Interface Type	Status
GPIB0	PCI-GPIB	passed

1.2.2.4 "NATIONAL INSTRUMENTS" GPIB PCI IEEE BOARD - VISA INSTALLATION

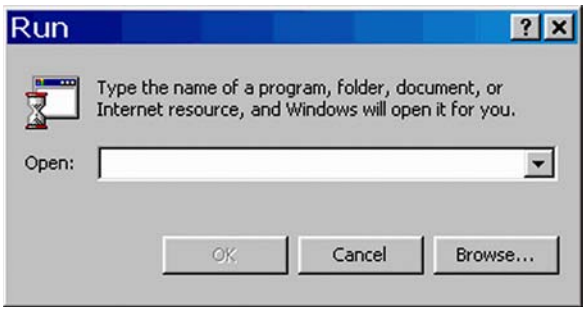
After having installed the NI-488.2 Software, the program "NI-Visa" will now be installed.

Close all applications!

- Press the ***Start*** button on the taskbar.

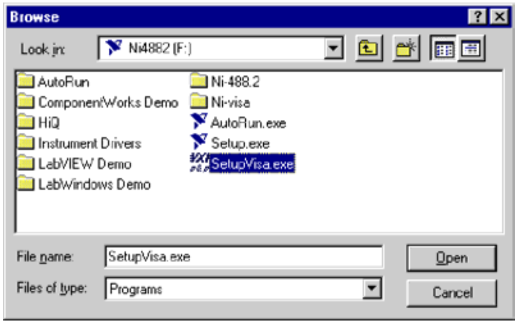


- Press ***Browse***.



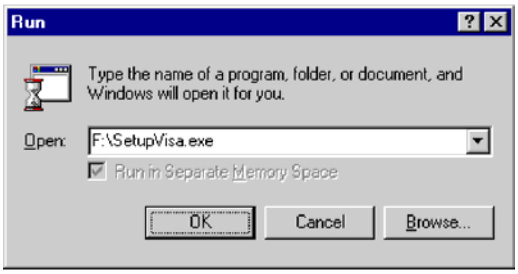
Type the name of a program, folder, document, or Internet resource, and Windows will open it for you.

- At the ***Look in*** pulldown menu, select your CD drive [e.g. Ni4882 (F:)].
- Select ***SetupVisa.exe*** and press ***Open*** (or double-click ***SetupVisa.exe***).



File name: SetupVisa.exe

- The path to ***SetupVisa.exe*** appears. Click ***OK***.



Type the name of a program, folder, or document, and Windows will open it for you.

Open: F:\SetupVisa.exe

- Wait, while the Installation program is being prepared.

Welcome to the NI-VISA 2.5 Installer

Please, wait while the Wise Installation Wizard® prepares the Windows installer.

- You are reminded to close all applications.
  To do so, if necessary, see how to exit Visa Setup.
  Then press ***Next***.

Welcome to the NI-VISA 2.5 Installer

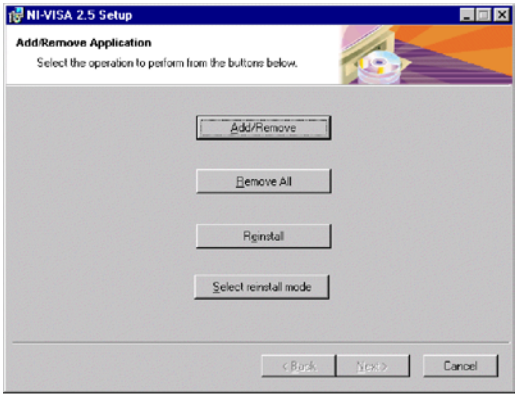
It is strongly recommended that you exit all Windows programs before running this Setup Program.

Click Cancel to quit Setup and close any programs you have running. Click Next to continue with the Setup program .

WARNING: This program is protected by copyright law and international treaties.

Unauthorized reproduction or distribution of this program, or any portion of it, may result in severe civil and criminal penalties, and will be prosecuted to the maximum extent possible under law.

- ➢ If the Setup program detects previously installed NI-Visa components, either add / remove some of them or choose reinstall / reinstall mode.

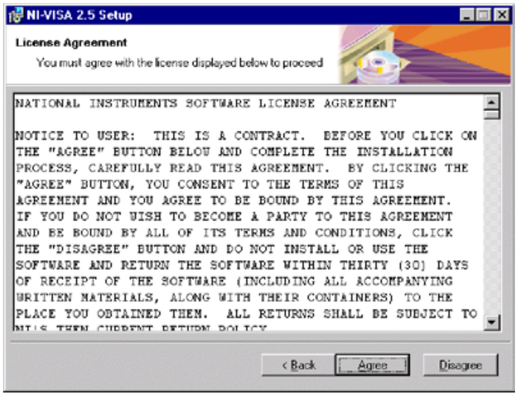


Add/Remove Application

Select the operation to perform from the buttons below.

- ➢ Read the License Agreement carefully and click ***Agree*** to proceed.

  Disagree would lead to exit Visa Setup.



License Agreement

You must agree with the license displayed below to proceed

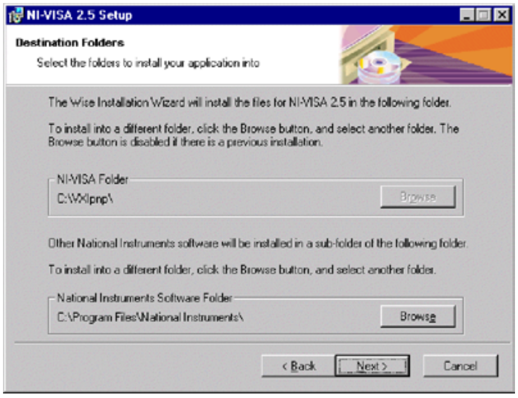
NATIONAL INSTRUMENTS SOFTWARE LICENSE AGREEMENT

NOTICE TO USER: THIS IS A CONTRACT. BEFORE YOU CLICK ON THE "AGREE" BUTTON BELOW AND COMPLETE THE INSTALLATION PROCESS, CAREFULLY READ THIS AGREEMENT. BY CLICKING THE "AGREE" BUTTON, YOU CONSENT TO THE TERMS OF THIS AGREEMENT AND YOU AGREE TO BE BOUND BY THIS AGREEMENT. IF YOU DO NOT WISH TO BECOME A PARTY TO THIS AGREEMENT AND BE BOUND BY ALL OF ITS TERMS AND CONDITIONS, CLICK THE "DISAGREE" BUTTON AND DO NOT INSTALL OR USE THE SOFTWARE AND RETURN THE SOFTWARE WITHIN THIRTY (30) DAYS OF RECEIPT OF THE SOFTWARE (INCLUDING ALL ACCOMPANYING WRITTEN MATERIALS, ALONG WITH THEIR CONTAINERS) TO THE PLACE YOU OBTAINED THEM. ALL RETURNS SHALL BE SUBJECT TO

- ➢ The Installation program proposes a destination folder for the ***NI-Visa*** Software (e.g. C:\VXIpnp\). To choose another folder, press the upper ***Browse*** button.

  In case of a previous NI-Visa installation, this button is disabled and the NI-Visa Software will be installed in the proposed folder.
- ➢ Beneath it, another folder is proposed for the installation of ***other NI*** Software (e.g. C:\Program Files\National Instruments\).

  To choose another folder use the lower ***Browse*** button. Finally, click ***Next***.



Destination Folders

Select the folders to install your application into

The Wise Installation Wizard will install the files for NI-VISA 2.5 in the following folder.

To install into a different folder, click the Browse button, and select another folder. The Browse button is disabled if there is a previous installation.

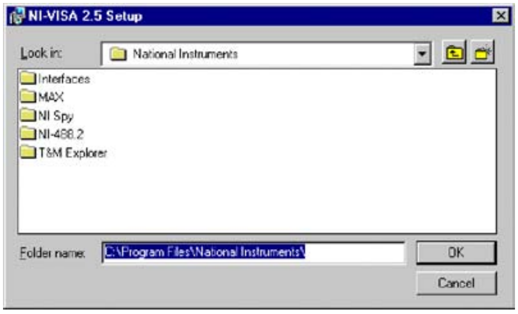
NI-VISA Folder: C:\VXIpnp\

Other National Instruments software will be installed in a sub-folder of the following folder.

To install into a different folder, click the Browse button, and select another folder.

National Instruments Software Folder: C:\Program Files\National Instruments\

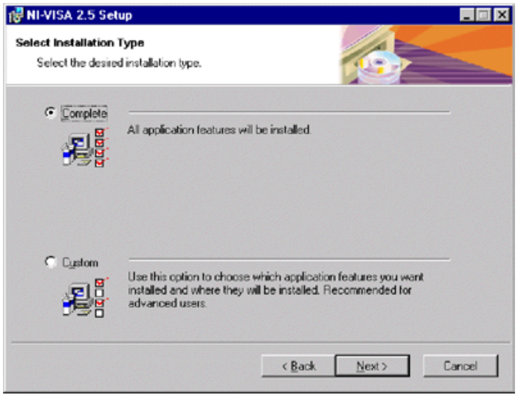
- Choose a folder (for the ***other*** NI Software). In the ***Folder Name*** box, the respective path is displayed. Press ***OK***.



NI-VISA 2.5 Setup

Folder name: C:\Program Files\National Instruments\

- To install all features select ***Complete*** (recommended usually) and press ***Next*** or use ***Custom*** (for advanced users) to install selected features and press ***Next***.



NI-VISA 2.5 Setup

Select Installation Type

Select the desired installation type.

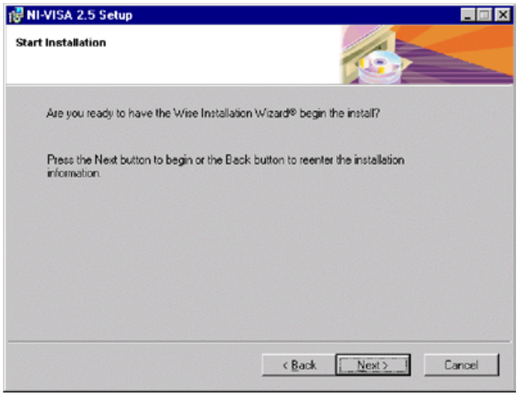
● Complete

All application features will be installed.

○ Custom

Use this option to choose which application features you want installed and where they will be installed. Recommended for advanced users.

- To start installing the files press ***Next***.



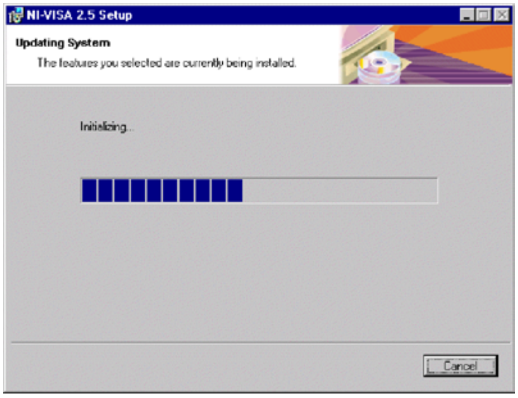
NI-VISA 2.5 Setup

Start Installation

Are you ready to have the Wise Installation Wizard® begin the install?

Press the Next button to begin or the Back button to reenter the installation information.

- Wait, while initializing takes place.



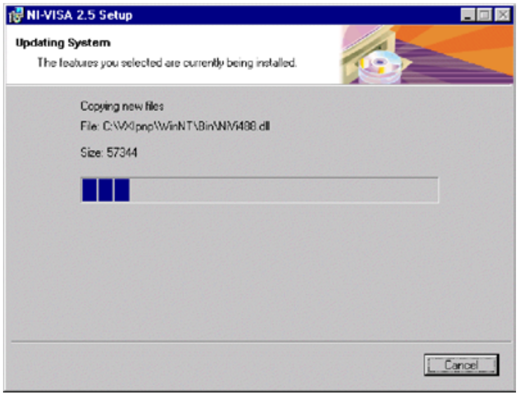
NI-VISA 2.5 Setup

Updating System

The features you selected are currently being installed.

Initializing...

- Wait, while the files are being copied.



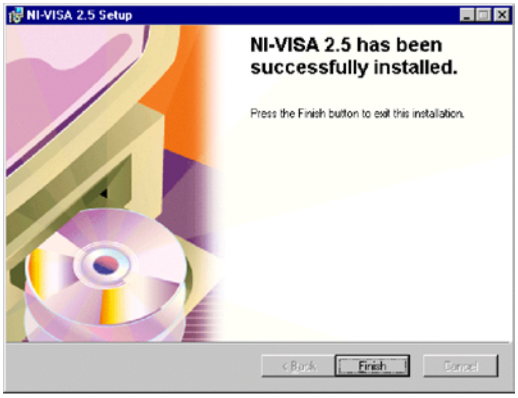
The features you selected are currently being installed.

Copying new files

File: C:\VXIpnp\WinNT\Bin\NiVi488.dll

Size: 57344

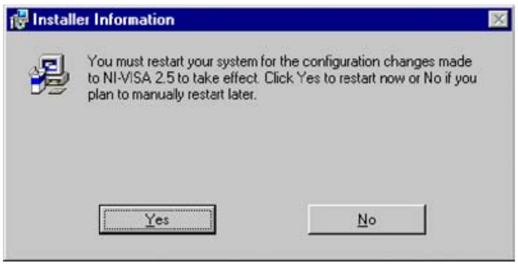
- After NI-Visa has been successfully installed press ***Finish***.



NI-VISA 2.5 has been successfully installed.

Press the Finish button to exit this installation.

- Click ***Yes*** to reboot your system now.



You must restart your system for the configuration changes made to NI-VISA 2.5 to take effect. Click Yes to restart now or No if you plan to manually restart later.

1.2.3 INSTALLATION OF THE SOUND CARD DRIVER

The installation of a sound card is required when a GC application is used as peripheral. The GC communicates with the IRMS via the joystick port of the sound card.

- Insert the sound card into slot #5.
- For the installation of the respective sound card driver, follow the instructions in the manual of the sound card manufacturer.

1.2.4 INSTALLATION OF THE "ANALOG JOYSTICK" DRIVER

The following GC applications additionally require the ***"Analog Joystick"*** driver. The GC communicates with the IRMS via the joystick port of the sound card.

- HP 6890 GC
- Trace GC
- any other "generic" GC

NOTE: ***The successful installation of a sound card with the appropriate driver is a prerequisite for the installation of the joystick driver.***

For Joystick driver handling follow the steps below:

To install it insert the ***"Operating System Backup CD"*** and follow the steps below.

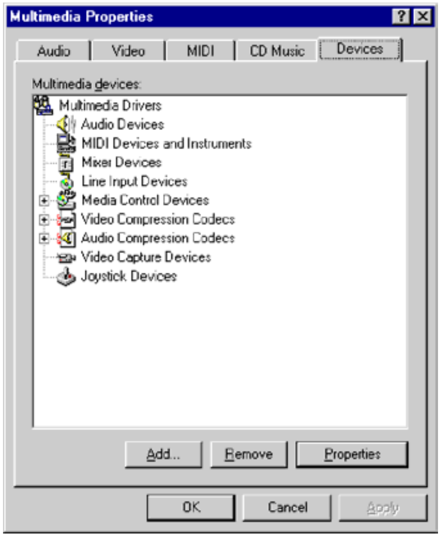
- Close the appearing box via the button (☒) at top right corner.

- Press ***Start > Settings > Control Panel***.

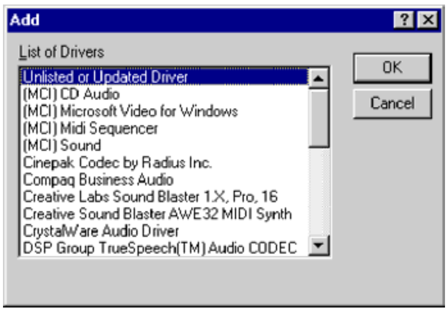
- ➢ Double-click the ***Multimedia*** Icon.

Changes multimedia device settings.

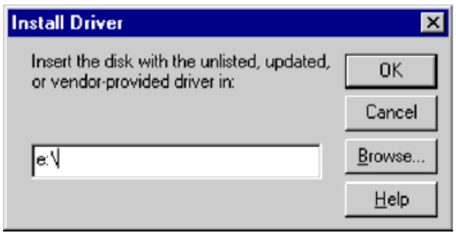
- ➢ Click the ***Devices*** tab.



- ➢ Press the ***Add*** button.



- ➢ Select ***Unlisted or updated driver*** and click ***OK***.



Insert the disk with the unlisted, updated, or vendor-provided driver in:

e:\

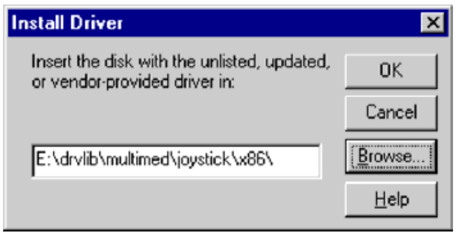
- Insert the ***"Operating System Backup CD"*** containing the driver - as was already recommended in Step 1. Type the letter of your CD drive (e.g. e:\) and click ***Browse***. From the ***Drives*** pulldown list select your CD drive (e.g. e: NTWKS 40A). In the ***Directories*** pulldown list double-click the folders, which successively lead to the Joystick drivers (e.g. e:\drvlib\multimed\joystick\x86).
- Click ***OK***.

Insert the disk with the unlisted, updated, or vendor-provided driver in:

Directories: e:\drvlib\multimed\joystick\x86

Drives: e: GATEWAY.VNT

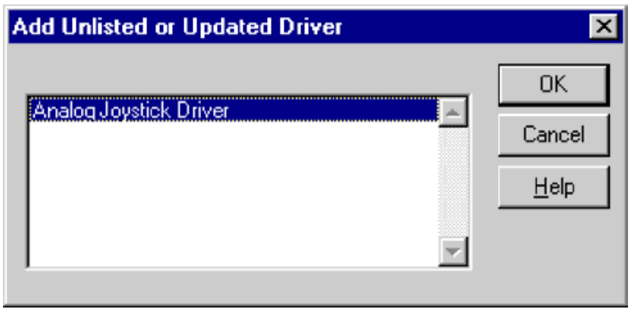
- Click ***OK***.



Insert the disk with the unlisted, updated, or vendor-provided driver in:

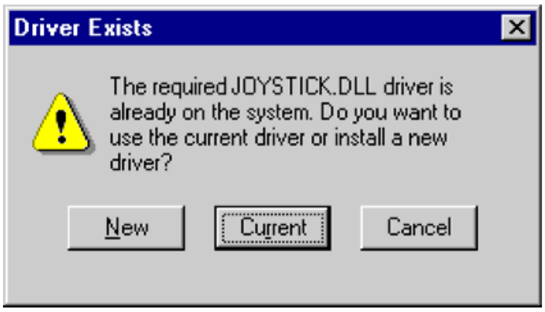
E:\drvlib\multimed\joystick\x86\

- Select ***Analog Joystick Driver*** and click ***OK***.



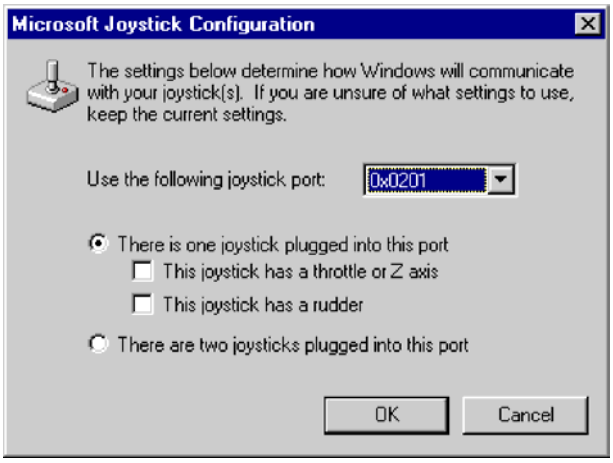
Analog Joystick Driver

- If the required joystick.dll driver is already installed:
  press ***New*** to install a new driver, or press ***Current*** to use the current driver.



The required JOYSTICK.DLL driver is already on the system. Do you want to use the current driver or install a new driver?

- In both cases, the box above appears.
  The different settings determine the communication of Windows with the joystick(s).
  If you are unsure of which settings to use, keep the default settings.
  Then click ***OK***.



Microsoft Joystick Configuration

The settings below determine how Windows will communicate with your joystick(s). If you are unsure of what settings to use, keep the current settings.

Use the following joystick port: 0x0201

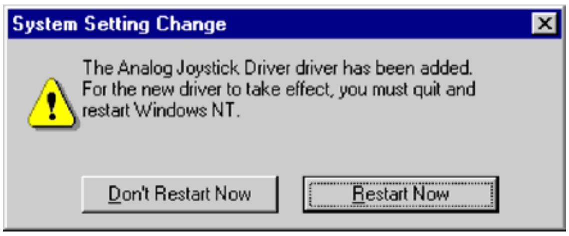
● There is one joystick plugged into this port

☐ This joystick has a throttle or Z axis

☐ This joystick has a rudder

○ There are two joysticks plugged into this port

- Click ***Restart Now***.



System Setting Change

The Analog Joystick Driver driver has been added. For the new driver to take effect, you must quit and restart Windows NT.

1.2.4.1 CALIBRATING THE JOYSTICK DRIVER

After the installation of the Joystick driver, it must now be calibrated and finally tested.

To calibrate it, follow the steps below.

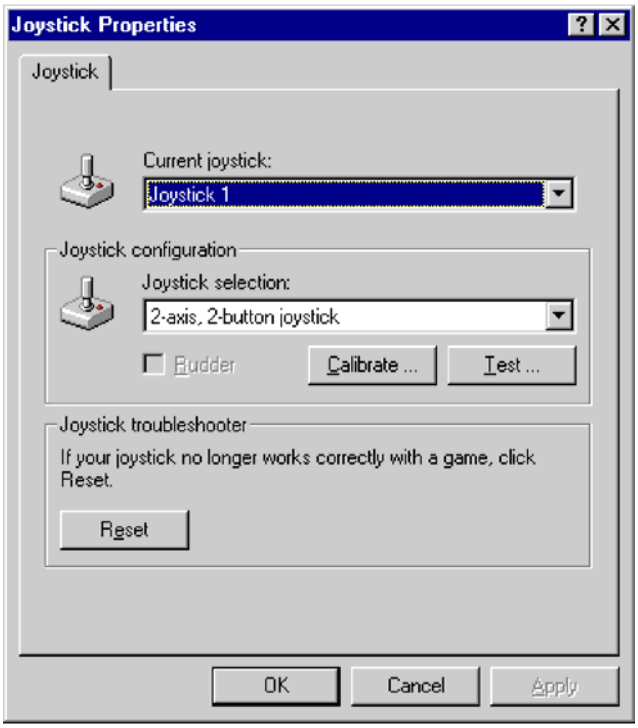
- Press ***Start > Settings > Control Panel***.

- Double-click the ***Joystick*** Icon.

Changes settings for joystick devices.

- At ***Joystick Configuration***, select the ***Calibrate*** button.

  An error message occurs, if the joystick is not correctly connected.



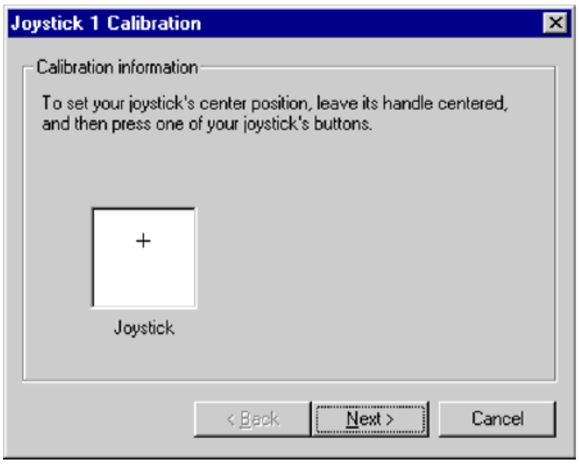
Current joystick: Joystick 1

Joystick selection: 2-axis, 2-button joystick

☐ Rudder

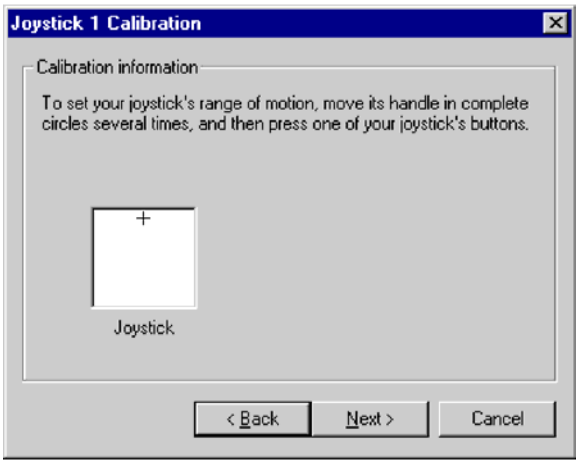
If your joystick no longer works correctly with a game, click Reset.

- To set the joystick's position, leave its handle centered.
  Then press one of the joystick's buttons.
  Click ***Next***.



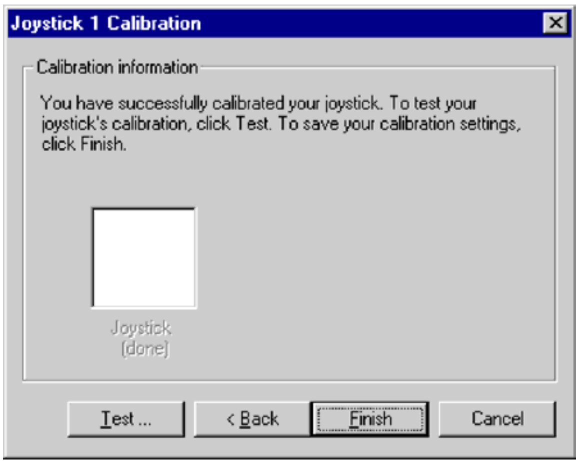
To set your joystick's center position, leave its handle centered, and then press one of your joystick's buttons.

- To set the joystick's motion range, move the handle in complete circles several times.
  Then press one of the joystick's buttons.



To set your joystick's range of motion, move its handle in complete circles several times, and then press one of your joystick's buttons.

- The joystick has been calibrated successfully.
  Click ***Finish*** to save your calibration settings.



You have successfully calibrated your joystick. To test your joystick's calibration, click Test. To save your calibration settings, click Finish.

- Test the calibration (see below).

1.2.4.2 TESTING THE JOYSTICK'S CALIBRATION

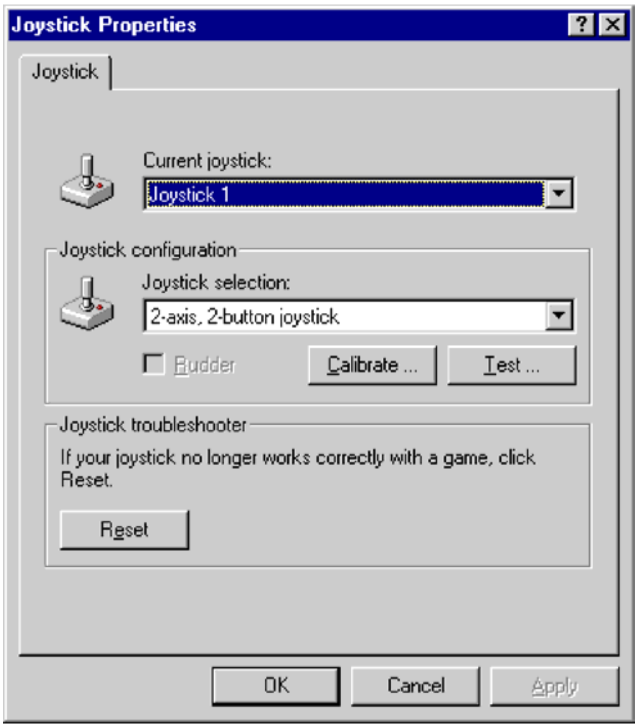
- ➢ Press ***Start* > *Settings* > *Control Panel***.

- ➢ Double-click the ***Joystick*** Icon.

Changes settings for joystick devices.

- At ***Joystick Configuration***, select the ***Test*** button.

  An error message occurs, if the joystick is not correctly connected.



Current joystick: Joystick 1

Joystick selection: 2-axis, 2-button joystick

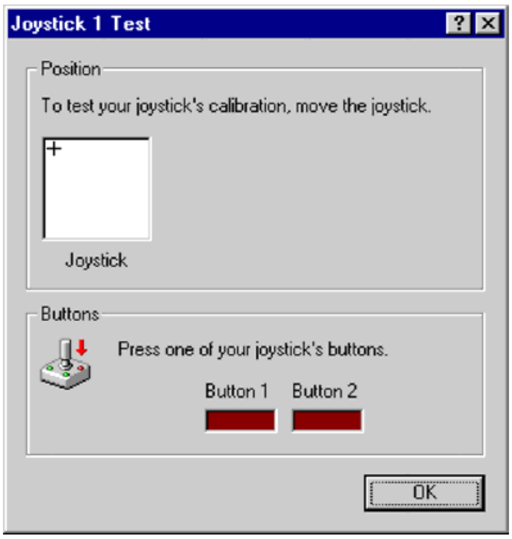
☐ Rudder

If your joystick no longer works correctly with a game, click Reset.

- To test the Joystick's calibration, move the Joystick.

  Click ***OK***.

  Press one of the Joystick's buttons.

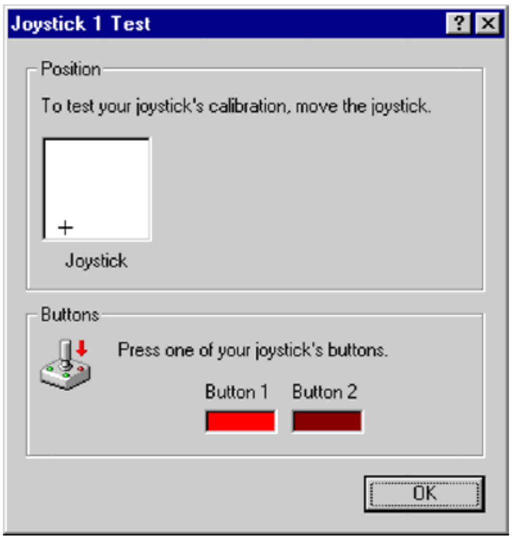


To test your joystick's calibration, move the joystick.

Press one of your joystick's buttons.

- One of the fields is lighted red (e.g. "Button 1").

  Press the other button.



Joystick 1 Test

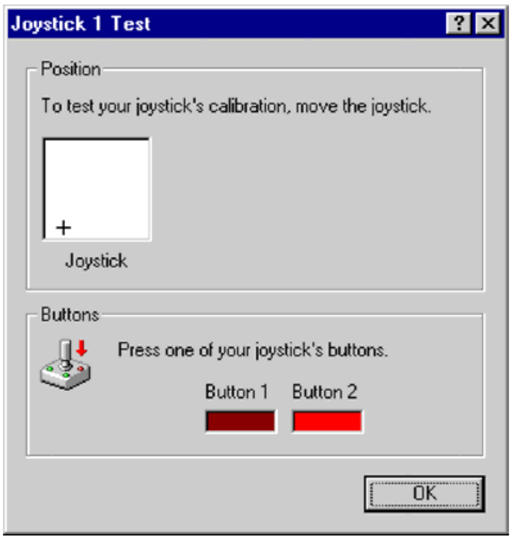
Position

To test your joystick's calibration, move the joystick.

Press one of your joystick's buttons.

- The other field is lighted red (e.g. "Button 2").

  Press ***OK***.



Joystick 1 Test

Position

To test your joystick's calibration, move the joystick.

Press one of your joystick's buttons.

1.2.5 GC AND AUTOSAMPLER REQUIREMENTS

1.2.5.1 HP 6890 GC

- Gameport Interface (in the PC)
- Gameport cable (ATT: modified version, two resistors)
- Serial cable from PC to HP 6890 (Null Modem 2X3) - Default: COM 2

1.2.5.2 TRACE GC

- Gameport Interface (in the PC)
- Gameport cable from PC to Trace GC
- Serial cable from PC to Trace GC (Null Modem 2X3) - Default: COM 2

1.2.5.3 ANY OTHER "GENERIC" GC

- Gameport Interface (in the PC)
- Gameport cable from PC to any other "generic" GC (only Trigger Mode)

1.2.5.4 A200S OR PAL AUTOSAMPLER

- Serial cable from PC to A200S Autosampler - Default: COM 1
- Cable for "GC ready" status from Autosampler to GC

1.3 INSTALLATION OF ISODAT NT

1.3.1 SYSTEM REQUIREMENTS

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

- Software requirements and
- Hardware requirements.

1.3.1.1 SOFTWARE REQUIREMENTS

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

Software requirements (minimal)	***Software requirements (recommended)***
Windows NT 4.0 (Intel) operating system	Windows NT 4.0 (Intel) operating system
Pentium class computer (233 MHz)	Pentium class computer (400 MHz or higher)
96 MB RAM	128 MB RAM
100 MB free disk space (only for ISODAT NT, without Backups and Result files)	500 MB free disk space (or higher)
Super VGA monitor (resolution 768 * 1280 pixels)	Super VGA monitor (resolution1280 * 1024 pixels)
	Windows NT 4.0 supported printer

1.3.1.2 HARDWARE REQUIREMENTS

- COM Port Extension Board
- IEEE Interface Board (for some peripherals)
- Sound Card (when a GC application is used as peripheral)
- Joystick requirements (when a GC application is used as peripheral)
- GC and Autosampler requirements

For the installation of the drivers for the above listed hardware, refer to Chapter 1.2.

1.3.2 INSTALLATION PROCEDURE

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

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

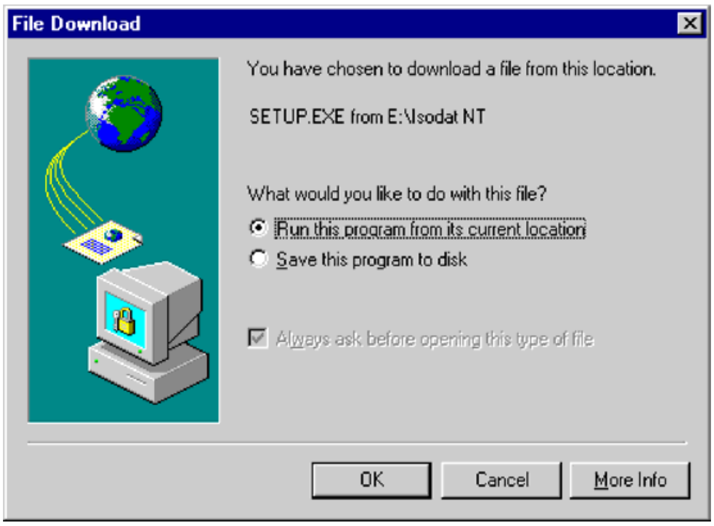
Otherwise, a message will indicate that Setup of ISODAT NT is not possible while older versions of ISODAT NT are running (see: how to shut down the system).

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

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

- From the gray Startup Screen, select ***Install ISODAT NT***.

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



You have chosen to download a file from this location.

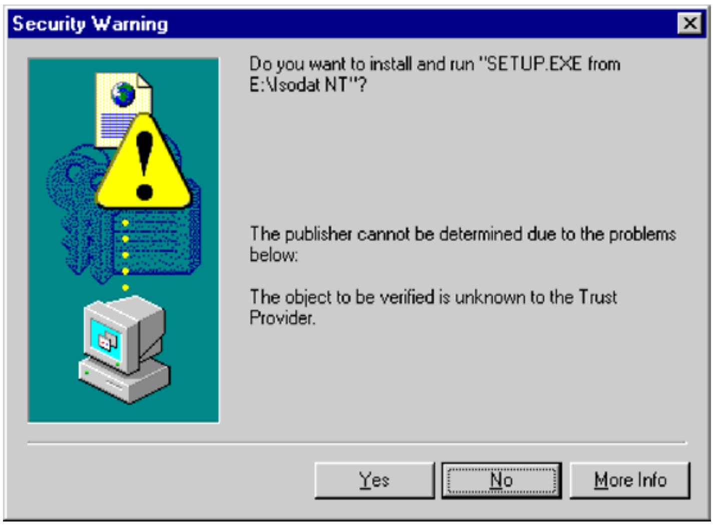
SETUP.EXE from E:\Isodat NT

What would you like to do with this file?

● Run this program from its current location
○ Save this program to disk

☑ Always ask before opening this type of file

- Click ***Yes***.

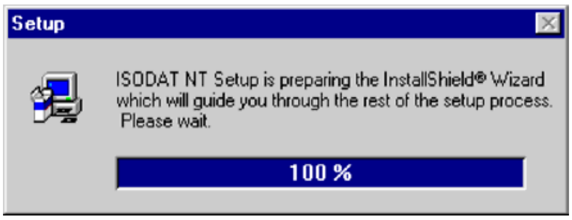


Do you want to install and run "SETUP.EXE from E:\Isodat NT"?

The publisher cannot be determined due to the problems below:

The object to be verified is unknown to the Trust Provider.

- Wait while Setup is preparing the InstallShield® Wizard.

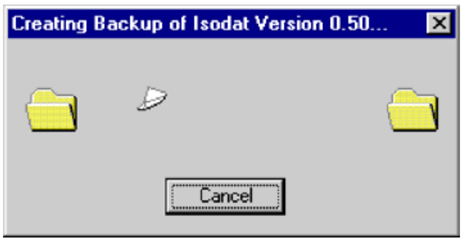


ISODAT NT Setup is preparing the InstallShield® Wizard which will guide you through the rest of the setup process. Please wait.

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

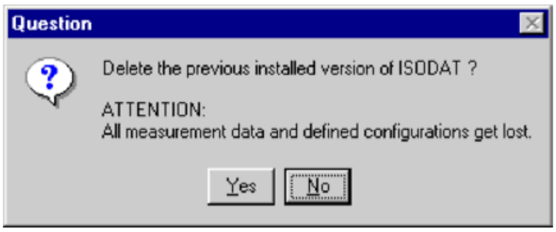
Backup Current Version?

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



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

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

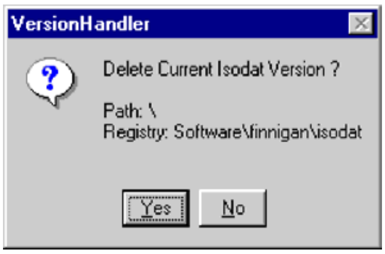


Delete the previous installed version of ISODAT ?

ATTENTION:
All measurement data and defined configurations get lost.

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

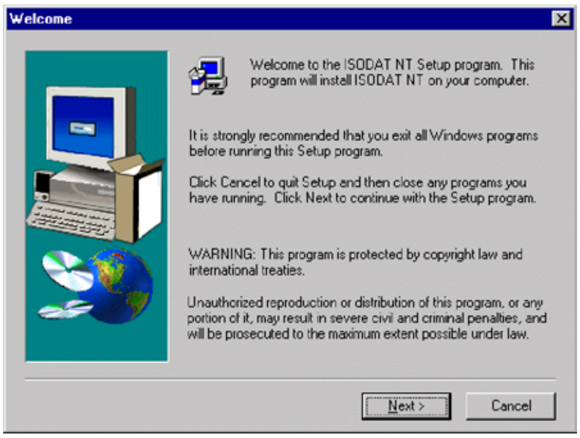
- ***No*** will overwrite the previous version. Data, Configurations and Mass Calibrations will remain untouched. Even though, it is recommended to delete old versions until unequivocal compatibility between the old and the new version is ensured.
- Confirm again with ***Yes*** to delete.



Delete Current Isodat Version ?

Path: \
Registry: Software\finnigan\isodat

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



Welcome to the ISODAT NT Setup program. This program will install ISODAT NT on your computer.

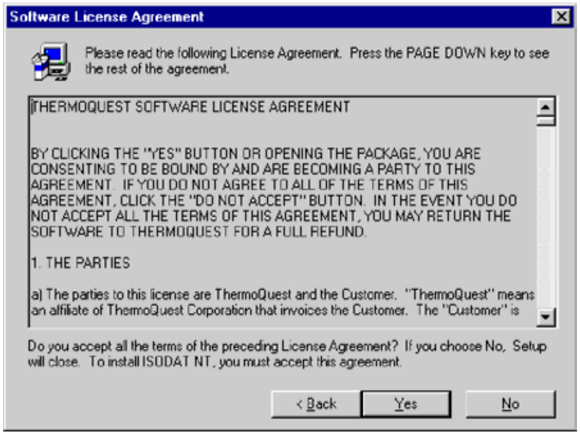
It is strongly recommended that you exit all Windows programs before running this Setup program.

Click Cancel to quit Setup and then close any programs you have running. Click Next to continue with the Setup program.

WARNING: This program is protected by copyright law and international treaties.

Unauthorized reproduction or distribution of this program, or any portion of it, may result in severe civil and criminal penalties, and will be prosecuted to the maximum extent possible under law.

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



Please read the following License Agreement. Press the PAGE DOWN key to see the rest of the agreement.

THERMOQUEST SOFTWARE LICENSE AGREEMENT

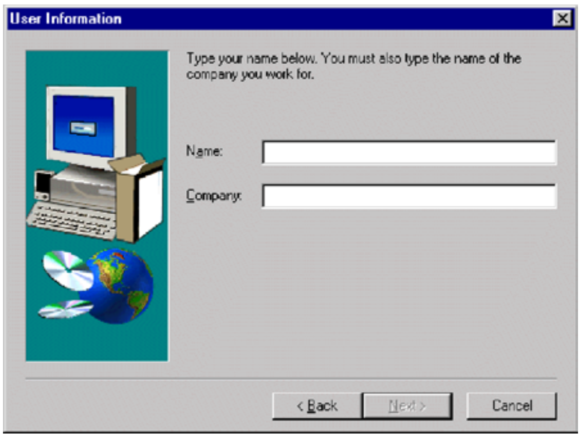
BY CLICKING THE "YES" BUTTON OR OPENING THE PACKAGE, YOU ARE CONSENTING TO BE BOUND BY AND ARE BECOMING A PARTY TO THIS AGREEMENT. IF YOU DO NOT AGREE TO ALL OF THE TERMS OF THIS AGREEMENT, CLICK THE "DO NOT ACCEPT" BUTTON. IN THE EVENT YOU DO NOT ACCEPT ALL THE TERMS OF THIS AGREEMENT, YOU MAY RETURN THE SOFTWARE TO THERMOQUEST FOR A FULL REFUND.

1. THE PARTIES

a) The parties to this license are ThermoQuest and the Customer. "ThermoQuest" means an affiliate of ThermoQuest Corporation that invoices the Customer. The "Customer" is

Do you accept all the terms of the preceding License Agreement? If you choose No, Setup will close. To install ISODAT NT, you must accept this agreement.

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

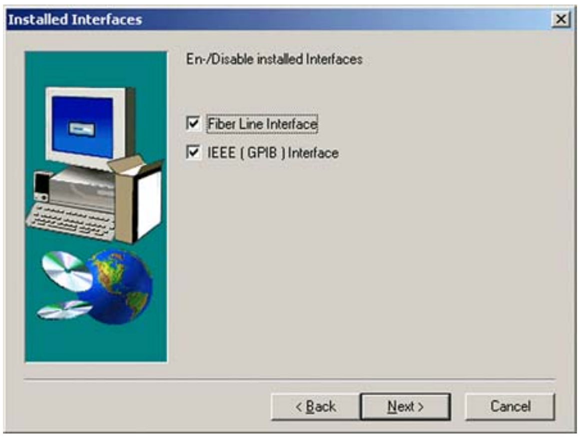


Type your name below. You must also type the name of the company you work for.

Name:

Company:

- On the next window, you have to indicate whether you have interfaces installed.
  As default, both checkboxes are selected. Select the checkbox(es) that correspond to your equipment, and then press ***Next***.
  - When you have ticked the checkbox ***Fiber Line Interface***, proceed with Page 1 – 39.
  - When you have ticked the checkbox ***IEEE (GBIB) Interface***, proceed with Page 1 – 38. This is also the case, when you have ticked both checkboxes.

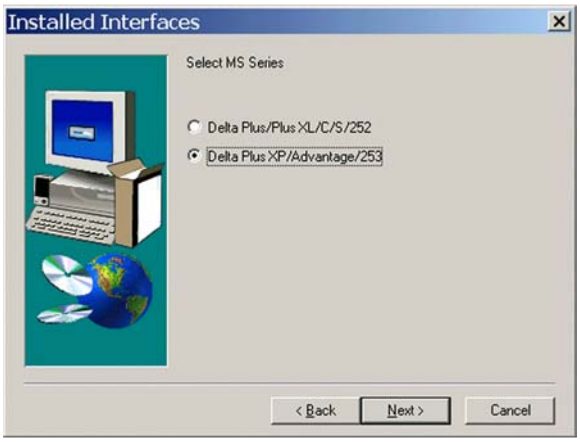


Installed Interfaces

En-/Disable installed Interfaces

☑ Fiber Line Interface
☑ IEEE (GPIB) Interface

- When you have deselected both checkboxes, the following window appears. Select the radio button ***DeltaPlusXP/Advantage/253*** and press ***Next***.
  This will install the *Fake Mode*, which can be used for offline computer systems and software testing purposes. Proceed with Page 1 – 39.

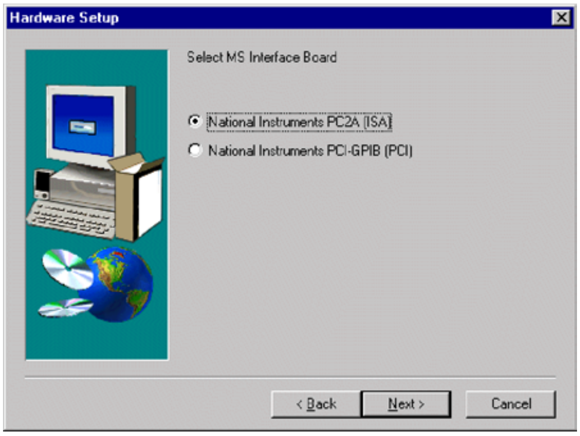


Installed Interfaces

Select MS Series

○ Delta Plus/Plus XL/C/S/252
● Delta Plus XP/Advantage/253

- Select your MS Interface Board for IEEE connection to the IRMS (on the rear side of your computer). Click ***Next***.
  - The "National Instruments" PC IIA (ISA) driver will automatically be installed.
  - The "National Instruments" PCI-GPIB (PCI) driver must be installed separately later.



Hardware Setup

Select MS Interface Board

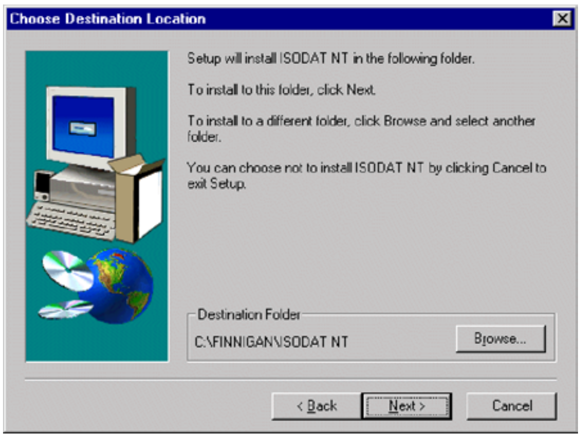
● National Instruments PC2A (ISA)
○ National Instruments PCI-GPIB (PCI)

- Having chosen PCI-GPIB (PCI), you are reminded that the "National Instruments" PCI-GPIB driver must be installed separately later using the "National Instruments" CD.

  Ensure that the appropriate IEEE Interface board driver has been installed. The required cables are part of the delivered equipment.

PCI-GPIB Driver from National Instrument has to be installed seperately to run ISODAT NT.

- Press ***OK***.



Setup will install ISODAT NT in the following folder.

To install to this folder, click Next.

To install to a different folder, click Browse and select another folder.

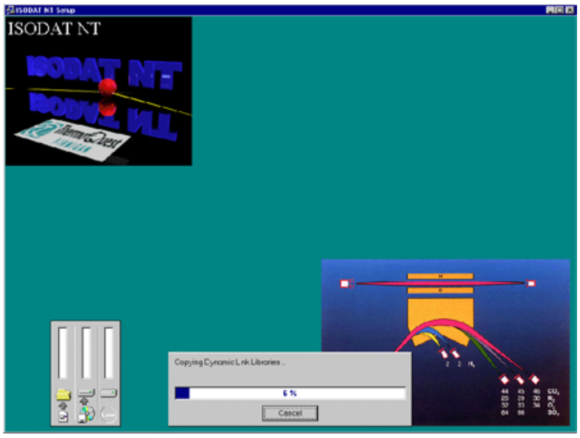
You can choose not to install ISODAT NT by clicking Cancel to exit Setup.

Destination Folder: C:\FINNIGAN\ISODAT NT

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

Updating Registry

- Wait while Setup is updating the registry.

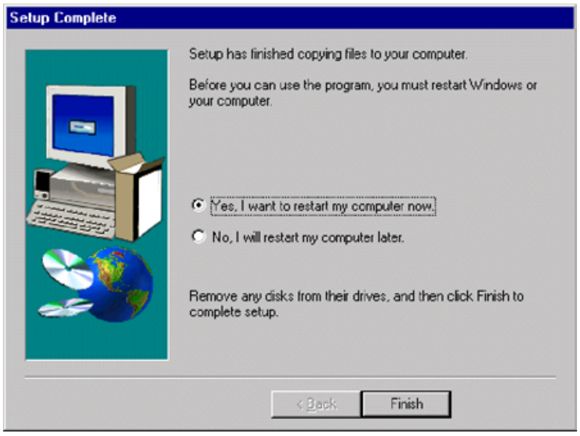


Copying Dynamic Link Libraries ...

6 %

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

- Choose ***Yes, I want to restart my computer now***.
- Remove the CD.
- Click ***Finish*** to reboot. ISODAT NT is now installed on your computer.



Setup Complete

Setup has finished copying files to your computer.

Before you can use the program, you must restart Windows or your computer.

● Yes, I want to restart my computer now.

○ No, I will restart my computer later.

Remove any disks from their drives, and then click Finish to complete setup.

ISODAT NT

OPERATING MANUAL

2

CONFIGURATOR

Issue 06/2002

Thermo Finnigan

2.1 PRINCIPLE

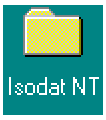
The **Configurator** creates an image of the hardware of your isotope ratio mass spectrometer (IRMS) resulting in a Configuration in the Configurations tree structure (i.e. Hardware Configuration). A Configuration always contains the IRMS and selected peripherals, representing all components necessary for a specific application. See an example.

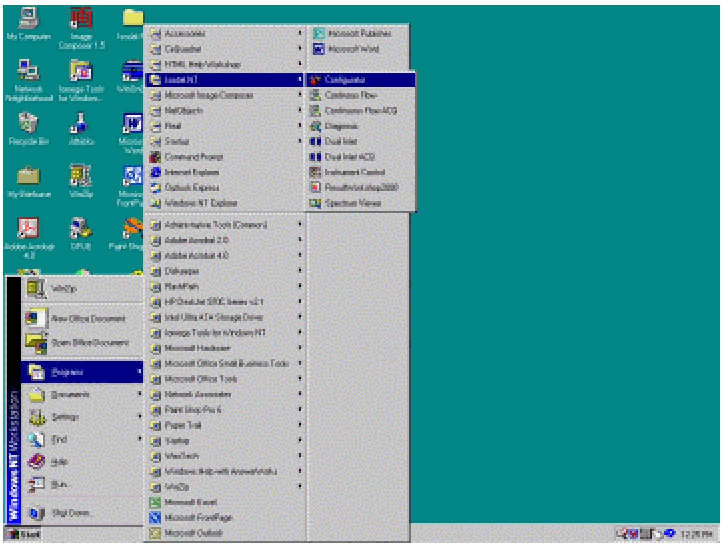
Configuration procedure

1. Defining ***hardware components*** of the IRMS
   - Select Mass Spectrometer
   - Edit Cup Settings
   - Edit Gas Configurations
2. Defining particular ***Configurations***

2.2 STARTING THE CONFIGURATOR

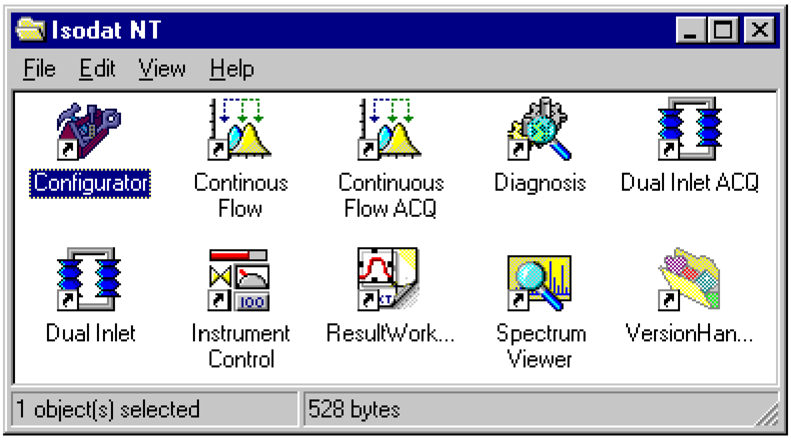
- On the desktop, click the ***Isodat NT*** button twice.





Alternative:

- Press ***Start*** > ***Programs*** > ***Isodat NT*** > ***Configurator***.



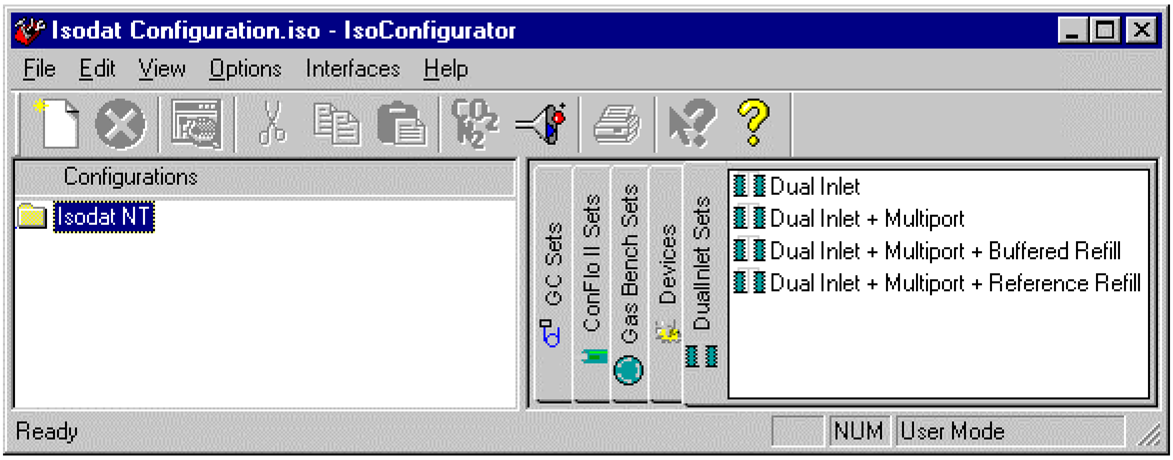
1 object(s) selected

528 bytes

- Double-click the ***Configurator*** Icon.

If ISODAT NT is already running, the Configurator can not be started.

An error message is displayed: Shut down the system, then start the Configurator again.

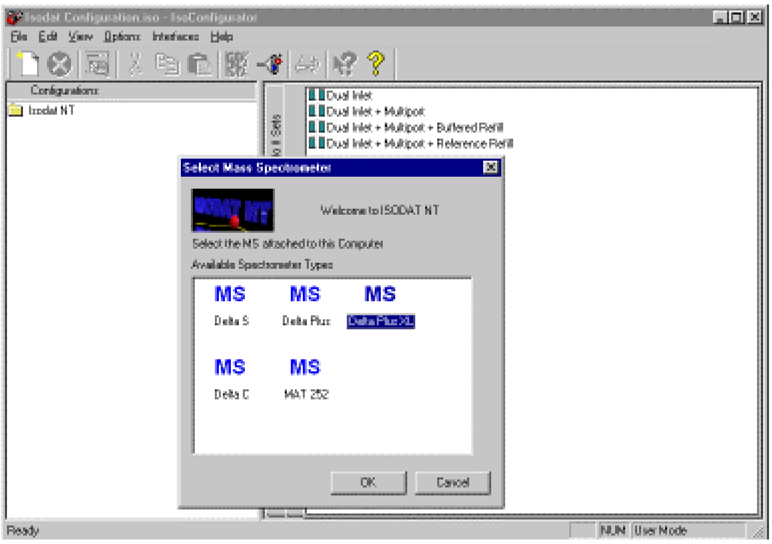


- The Configurator opens.

Left pane:	lists all defined Configurations beneath the "Isodat NT" folder. If you have never before defined one, it is the only one to be displayed. It has been assigned the preliminary name "New Configuration" which can be changed easily.
Middle pane:	five "Sets" tabs leading to the *generally* possible hardware combinations.
Right pane:	select those predefined hardware combinations which fit to your instrument.

2.3 SELECT MASS SPECTROMETER

Selecting the IRMS type defines mass spectrometer specific parameters (e.g. magnetic field, high voltage, pressure meters).



Welcome to ISODAT NT

Select the MS attached to this Computer

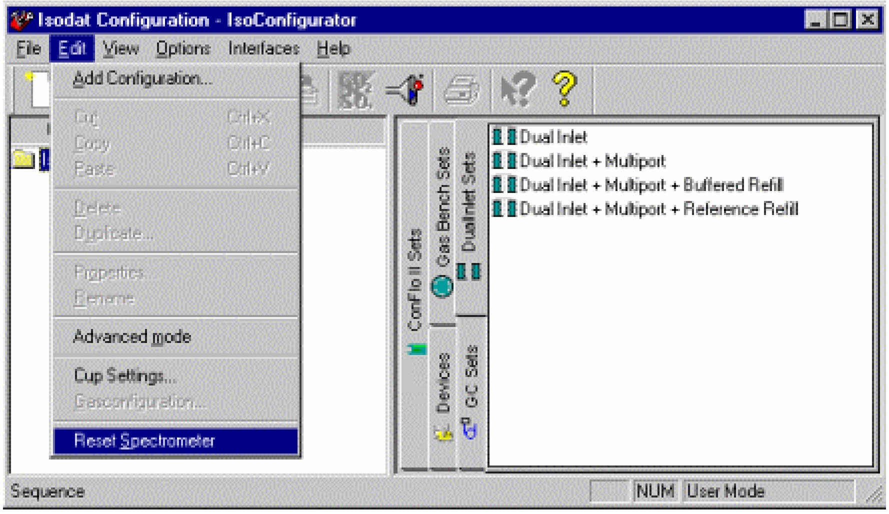
Available Spectrometer Types

The above box is shown only when starting the Configuration procedure the first time.

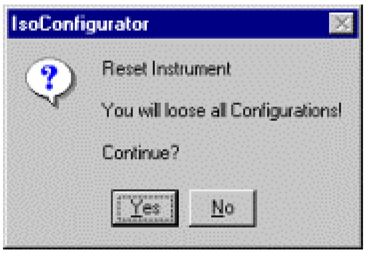
In the background window's left pane the "Isodat NT" folder is visible. All Configurations added later will be listed beneath it. Hitherto, no Hardware Configuration is defined.

- Select your IRMS among the available mass spectrometer types.

2.3.1 RESET MASS SPECTROMETER



- ➢ To select another IRMS click ***Reset Spectrometer*** in the ***Edit*** menu.



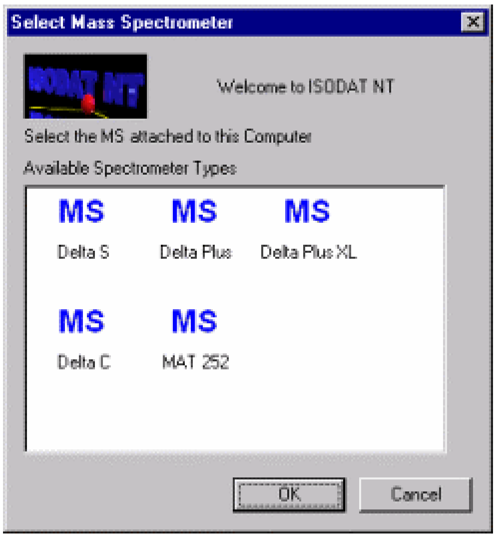
Reset Instrument

You will loose all Configurations!

Continue?

All existing Configurations will be deleted answering "Yes"!

- ➢ Proceed with ***Yes***, while ***No*** cancels the process.



Welcome to ISODAT NT

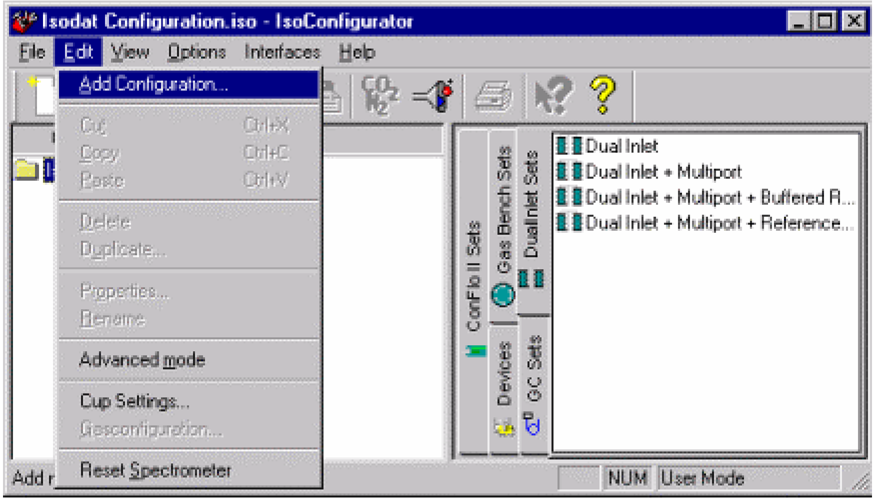
Select the MS attached to this Computer

Available Spectrometer Types

- ➢ Select your IRMS among the available Mass Spectrometer types.

2.4 ADDING NEW CONFIGURATIONS

After defining the IRMS a ***Configuration*** (i.e. a Hardware Configuration) must be established. To create one, your ***peripherals*** must be attached to the inlet port of the ***mass spectrometer***.



- ➢ In the ***Edit*** menu select ***Add Configuration***.

Alternative:

- Click the ***Add new Configuration*** Icon on the toolbar.

Definition of a Configuration by attaching peripherals will be explained by examples:

Example 1: Attaching a ***Dual Inlet*** peripheral to the IRMS

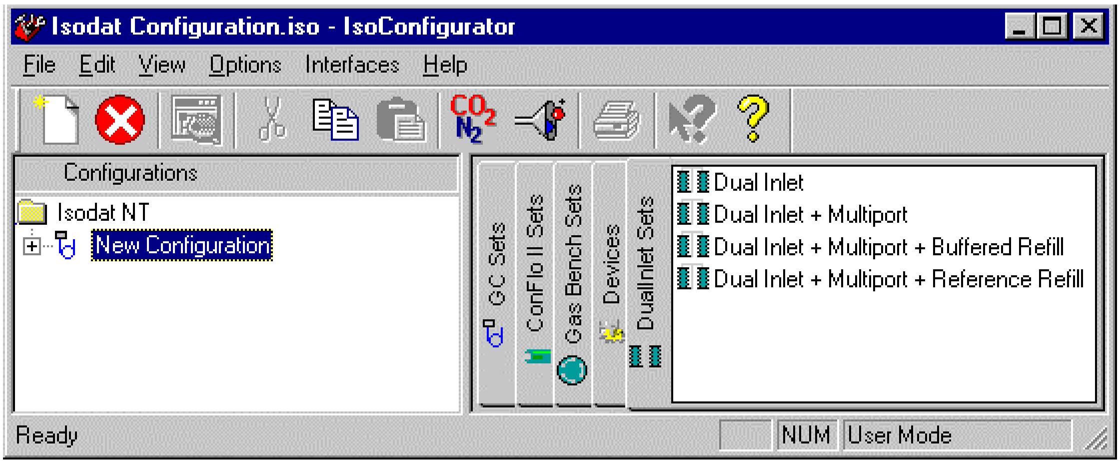
Example 2: Attaching a ***GC*** peripheral to the IRMS

Example 3: Attaching a ***ConFlo*** peripheral to the IRMS

Example 4: Attaching a ***GasBench*** peripheral to the IRMS

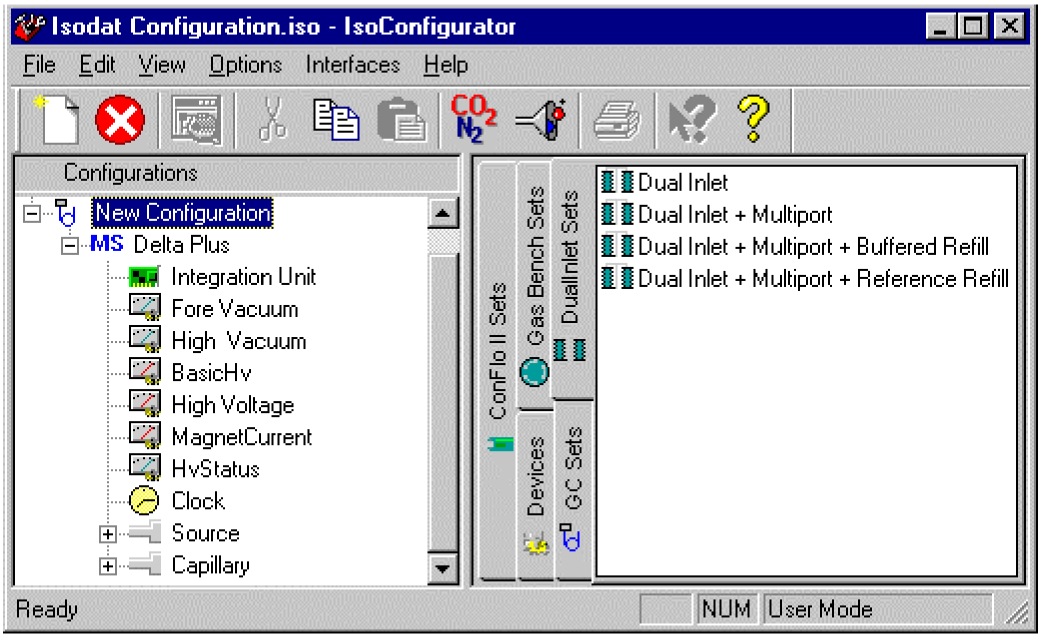
2.4.1 ATTACHING A DUAL INLET PERIPHERAL TO THE IRMS

A "Dual Inlet + Multiport" peripheral will be connected to a Delta^{Plus} Mass Spectrometer.

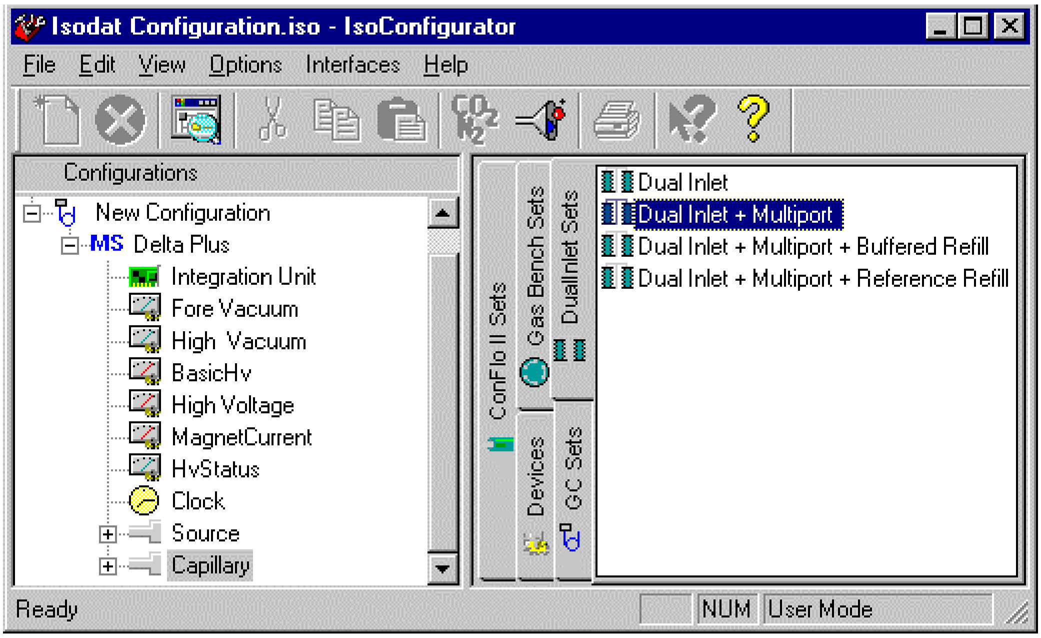


- In the Configurator's left pane beneath the "Isodat NT" folder, the Configuration being created occurs ("New Configuration"). Hitherto, a capillary is displayed close to it serving as default symbol.

- Beneath the "New Configuration" entry the mass spectrometer you chose can be seen (e.g. a Delta^{Plus}). Pressing its + sign shows all IRMS parts in the tree structure.

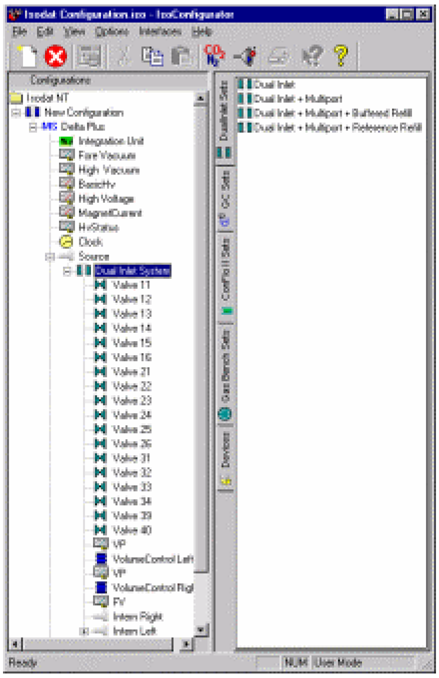


- ➢ Press the ***Dual Inlet Sets tab*** in the middle pane.
- ➢ Select the peripheral to be attached to the IRMS by a click on it in the right pane (e.g. Dual Inlet + Multiport).



- Drag and drop the selected entry (e.g. "Dual Inlet + Multiport") to the ***Source*** symbol of the MS Delta^{Plus}, at bottom of left pane.

Source

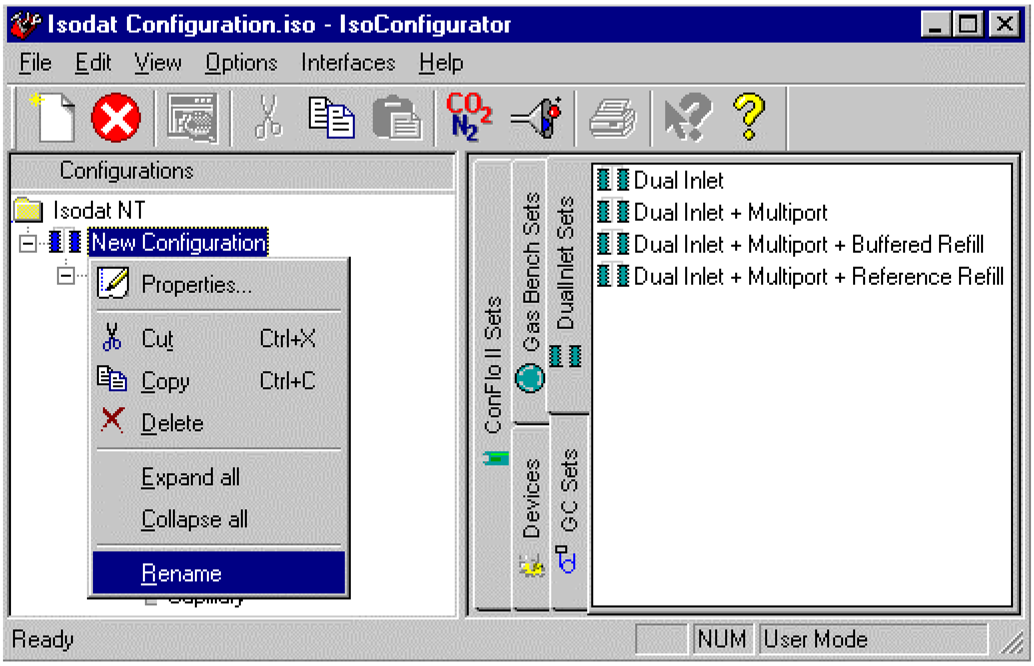


- The Dual Inlet periphery has been inserted beneath the Source symbol (beginning at the "Dual Inlet System" entry).

The capillary Icon on the left side of "New Configuration" has <u>changed</u> to a bellows Icon. ISODAT NT automatically detects that a Dual Inlet periphery is being attached and indicates this by the bellows Icon.

- Rename the new Configuration hitherto called "New Configuration". This could also already be done when the entry "New Configuration" appears the first time.

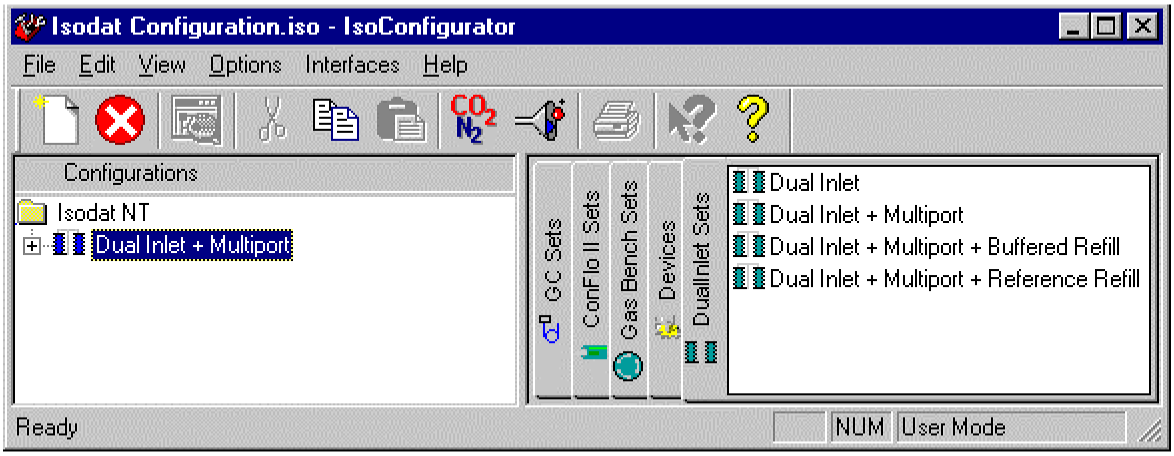
- Choose a significant name to identify it easily if creating more than one Configuration (e.g. "Dual Inlet + Multiport").



- ➢ Right-click on ***New Configuration***.
- ➢ In the popup menu select ***Rename***.

Alternative:

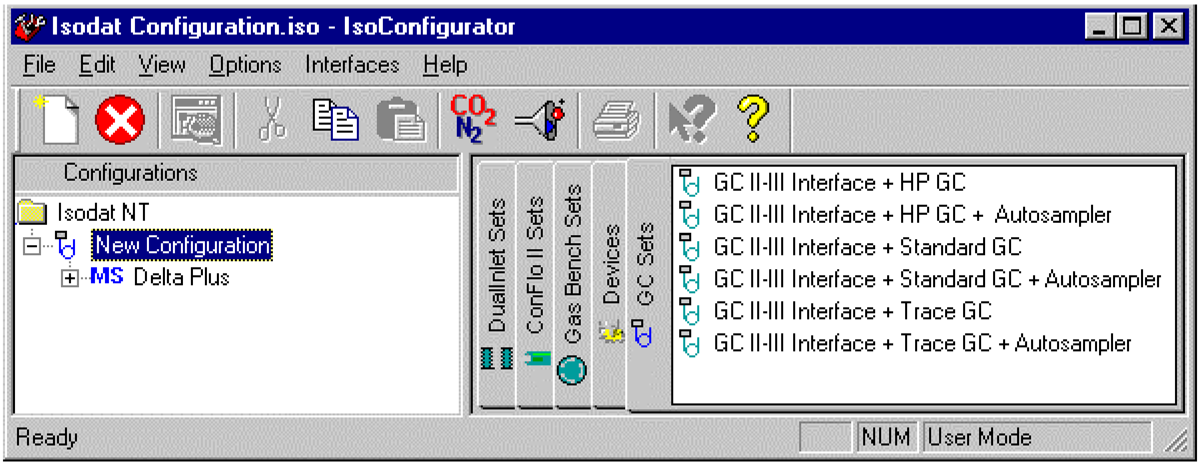
- ➢ From the ***Edit*** menu select ***Rename***.



- Type the name of the new Configuration (e.g. "Dual Inlet + Multiport").
- Click somewhere on the screen to finish renaming.

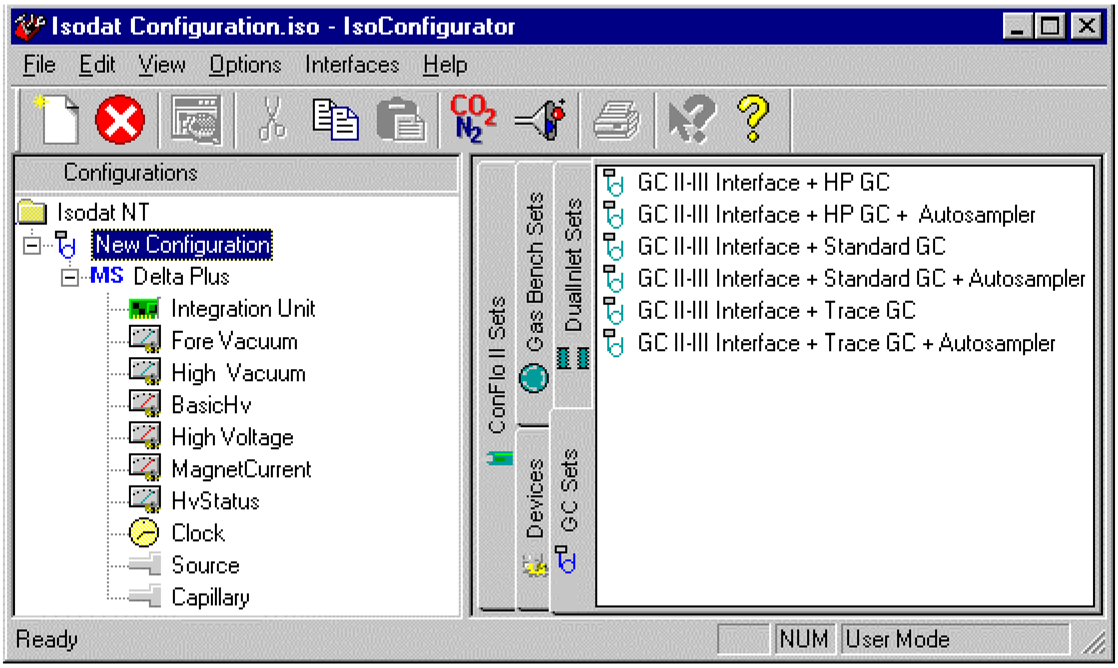
2.4.2 ATTACHING A GC PERIPHERAL TO THE IRMS

A "GC II-III Interface + Standard GC" periphery will be connected to a Delta^{Plus} Mass Spectrometer.

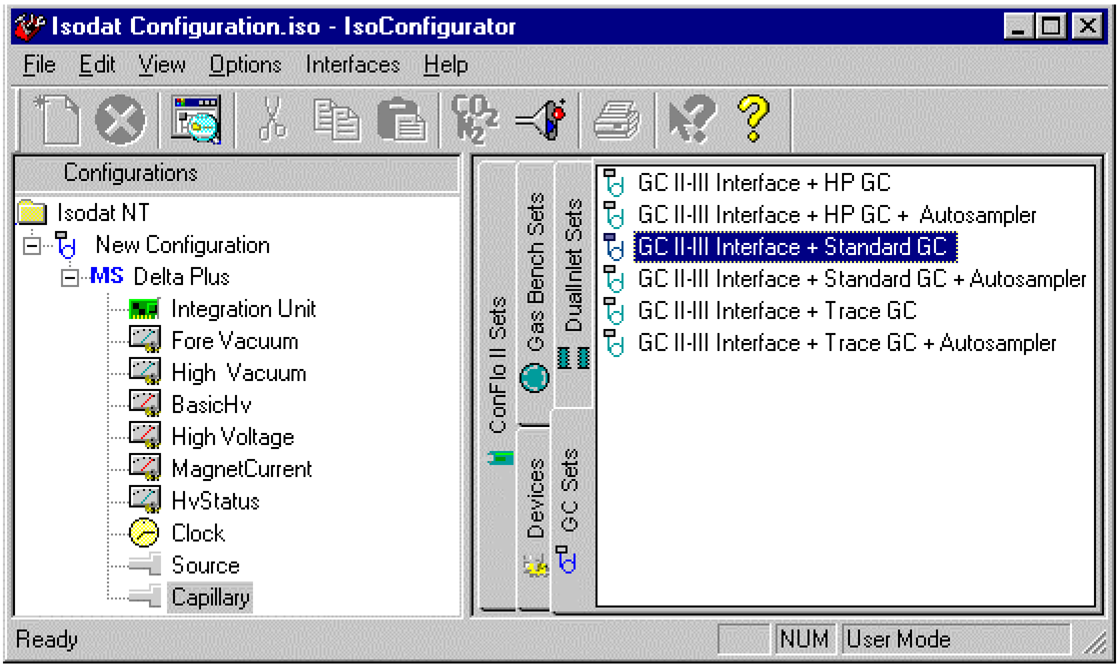


- In the Configurator's left pane beneath the "Isodat NT" folder, the Configuration being created occurs ("New Configuration"). Hitherto, a capillary is displayed close to it serving as default symbol.

- Beneath the "New Configuration" entry the mass spectrometer you chose can be seen (e.g. a Delta^{Plus}). Pressing its + sign shows all IRMS parts in the tree structure.

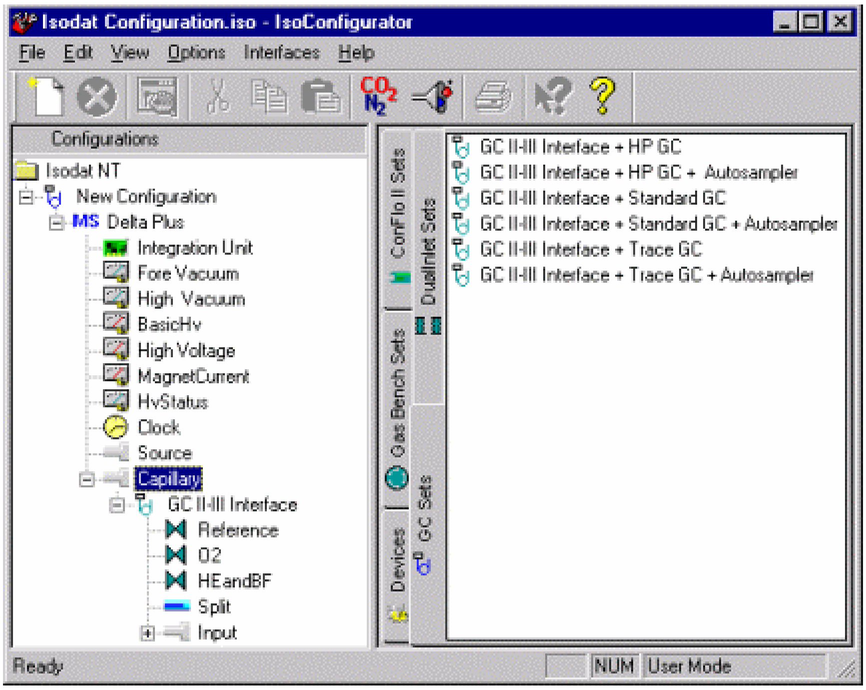


- ➢ Press the ***GC Sets tab*** in the middle pane.
- ➢ Select the peripheral to be attached to the IRMS by a click on it in the right pane (e.g. GC II-III Interface + Standard GC).



- Drag and drop the selected entry (e.g. "GC II-III Interface + Standard GC") to the "Capillary" symbol of the MS Delta^{Plus}, at bottom of left pane.

Capillary



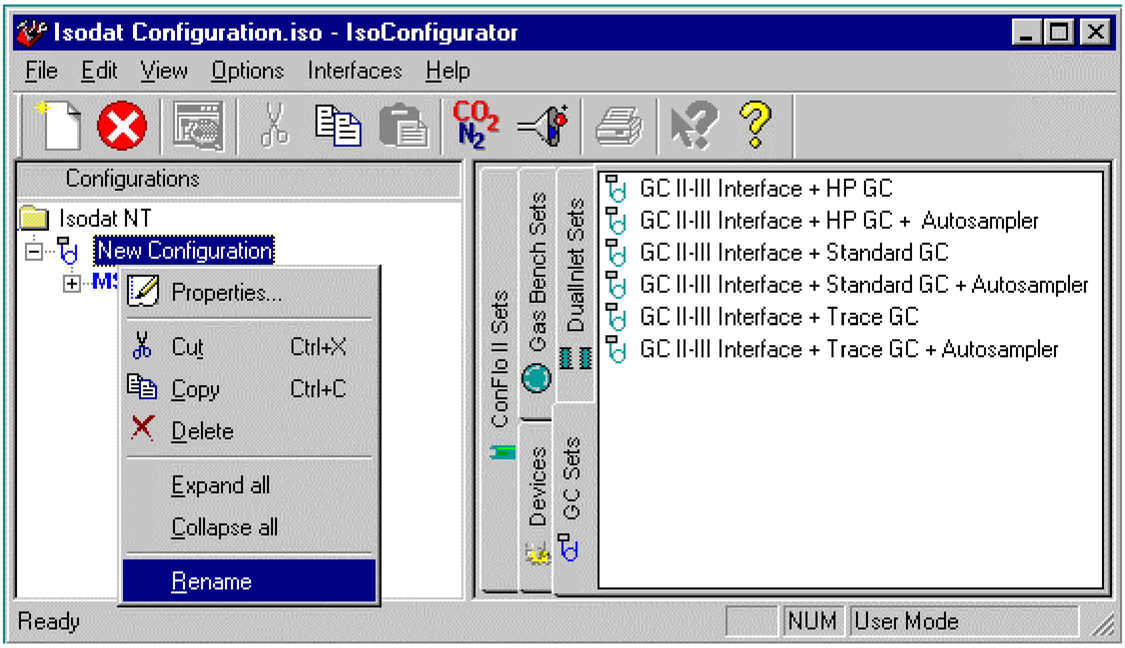
- The GC periphery has been inserted beneath the "Capillary" symbol (beginning at the "GC II-III Interface" entry).

The capillary Icon on the left side of "New Configuration" has not changed.

ISODAT NT automatically detects that a GC periphery is being attached and indicates this by the capillary Icon.

- Rename the new Configuration hitherto called "New Configuration". This could also already be done when the entry "New Configuration" appears the first time.

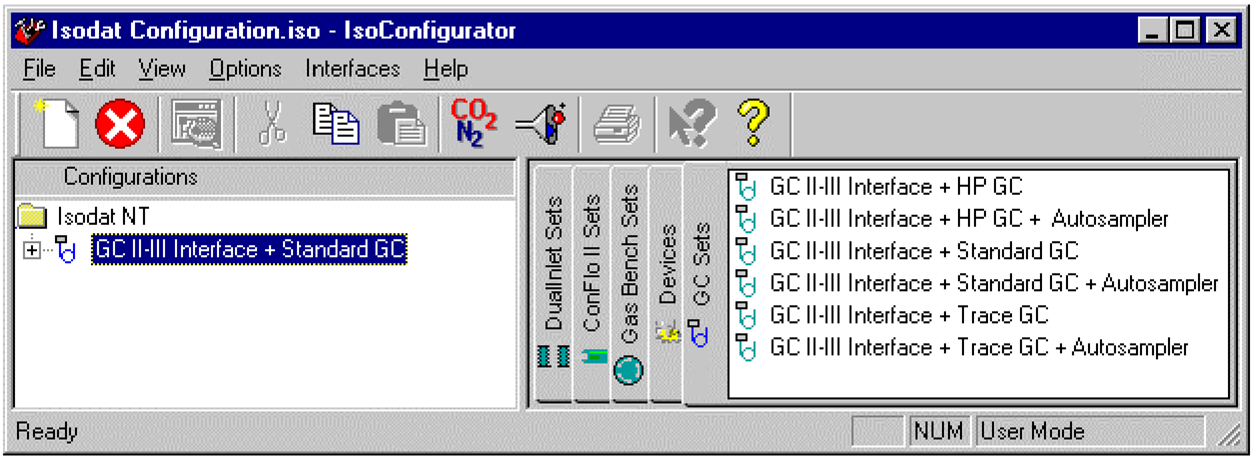
- Choose a significant name to identify it easily if creating more than one Configuration (e.g. "GC II-III Interface + Standard GC").



- ➢ Right-click on ***New Configuration***.
- ➢ In the popup menu select ***Rename***.

Alternative:

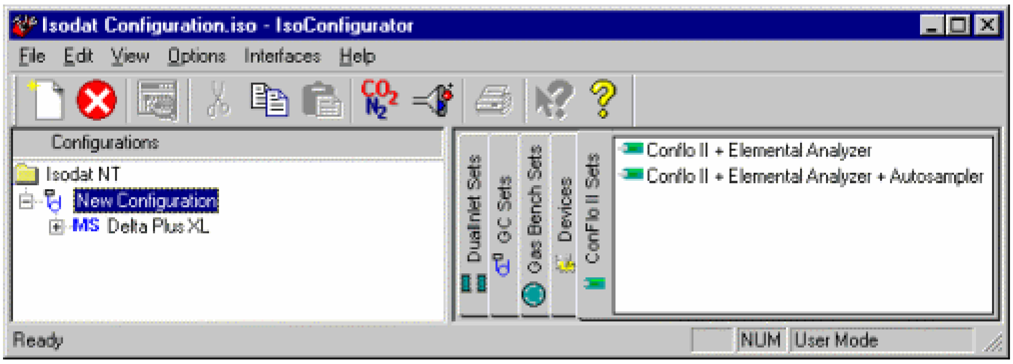
- ➢ From the ***Edit*** menu select ***Rename***.



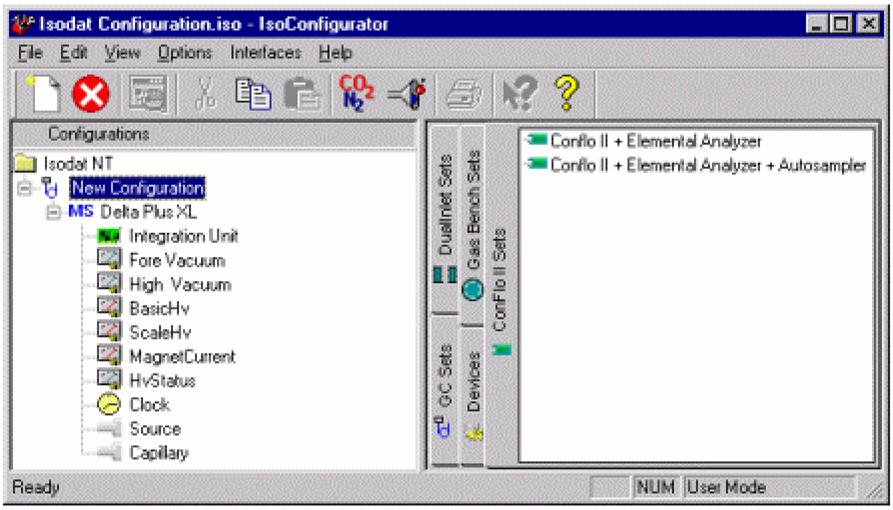
- Type the name of the new Configuration (e.g. "GC II-III Interface + Standard GC").
- Click somewhere on the screen to finish renaming.

2.4.3 ATTACHING A CON FLO PERIPHERAL TO THE IRMS

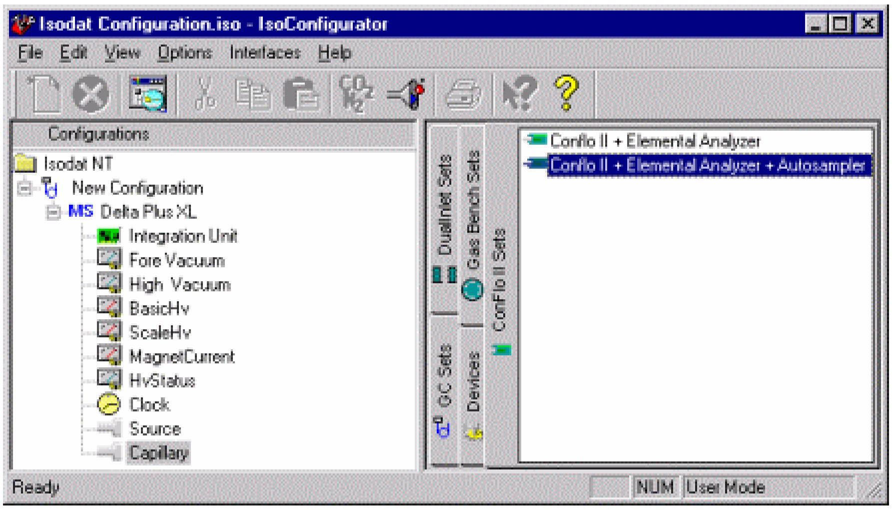
A "ConFloII + Elemental Analyzer + Autosampler" peripheral will be connected to a Delta^{Plus} XL Mass Spectrometer.



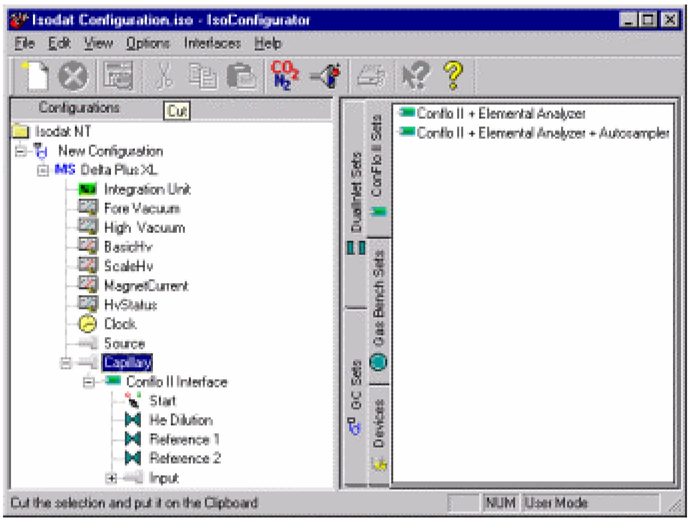
- In the Configurator's left pane beneath the "Isodat NT" folder, the Configuration being created occurs ("New Configuration"). Hitherto, a capillary is displayed close to it serving as default symbol.
- Beneath the "New Configuration" entry the mass spectrometer you chose can be seen (e.g. a DeltaPlus XL). Pressing its + sign shows all IRMS parts in the tree structure.



- Press the ***ConFloII Sets tab*** in the middle pane.
- Select the peripheral to be attached to the IRMS by clicking on it in the right pane (e.g. ConFloII + Elemental Analyzer + Autosampler).



- Drag and drop the selected entry (e.g. "ConFloII + Elemental Analyzer + Autosampler") to the "Capillary" symbol of the MS Delta^{Plus} XL, at bottom of left pane.



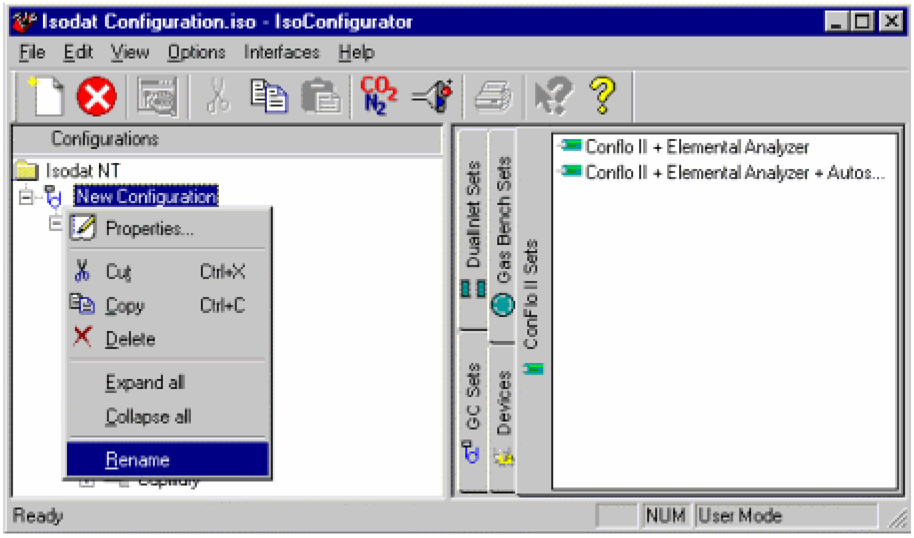
- The ConFlo periphery has been inserted beneath the "Capillary" symbol (beginning at the "ConFloII Interface" entry).

The capillary Icon on the left side of "New Configuration" has not changed.

ISODAT NT automatically detects that a ConFloII periphery is being attached and indicates this by the capillary Icon.

- Rename the new Configuration hitherto called "New Configuration". This could also already be done when the entry "New Configuration" appears the first time.

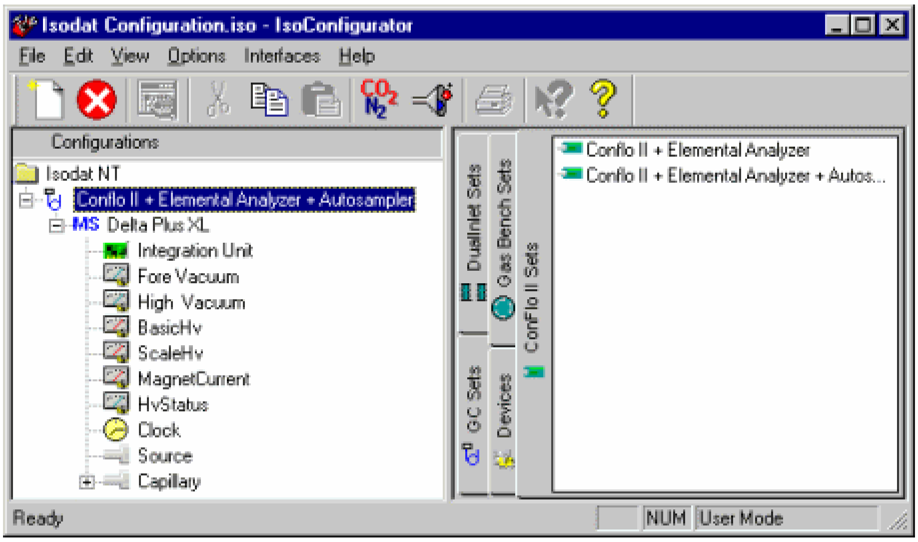
- Choose a significant name to identify it easily if creating more than one Configuration (e.g. "ConFloII Interface + Elemental Analyzer + Autosampler").



- Right-click on ***New Configuration***.
- In the popup menu select ***Rename***.

Alternative:

- From the ***Edit*** menu select ***Rename***.

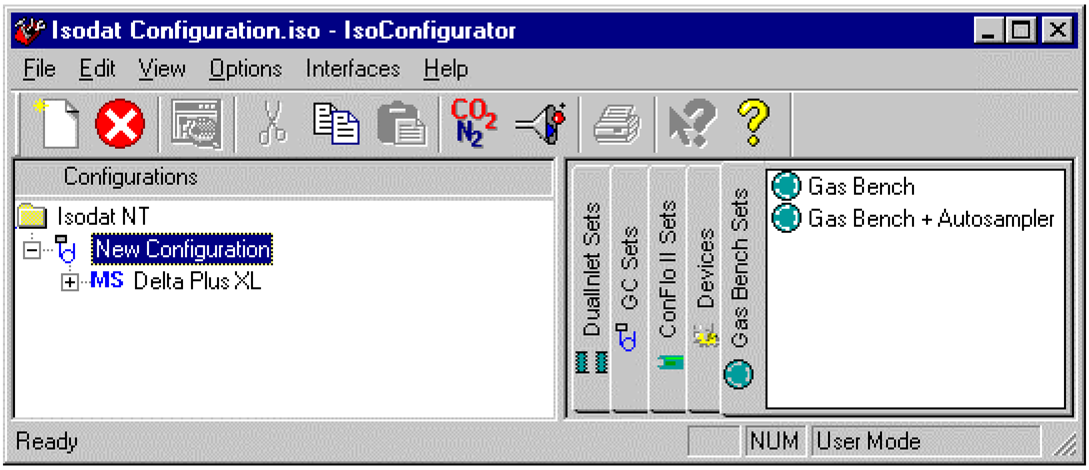


- ➢ Type the name of the new Configuration (e.g. "ConFloII Interface + Elemental Analyzer + Autosampler").

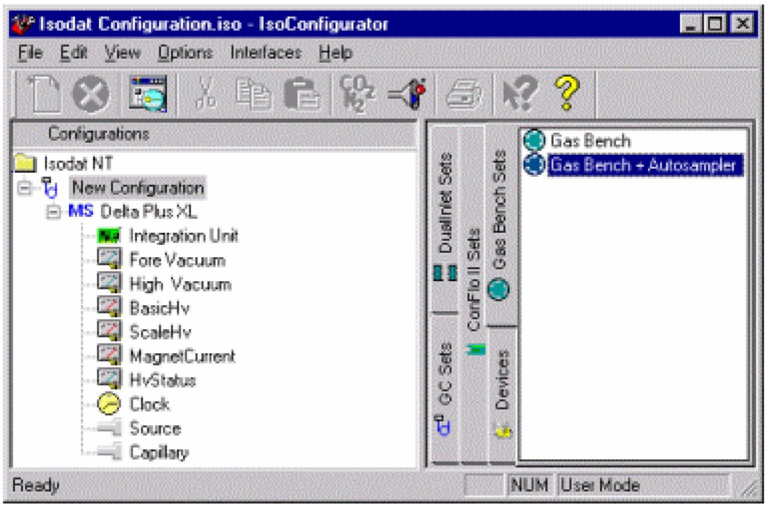
- ➢ Click somewhere on the screen to finish renaming.

2.4.4 ATTACHING A GASBENCH PERIPHERAL TO THE IRMS

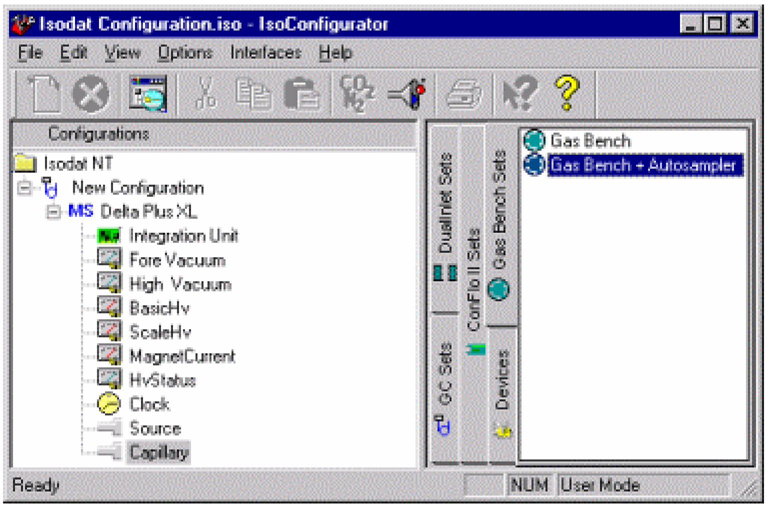
A "Gasbench + Autosampler" peripheral will be connected to a Delta$^{\text{Plus}}$ XL Mass spectrometer.



- In the Configurator's left pane beneath the "Isodat NT" folder, the Configuration being created occurs ("New Configuration"). Hitherto, a capillary is displayed close to it serving as default symbol.
- Beneath the "New Configuration" entry the mass spectrometer you chose can be seen (e.g. a Delta^{Plus} XL). Pressing its + sign shows all IRMS parts in the tree structure.

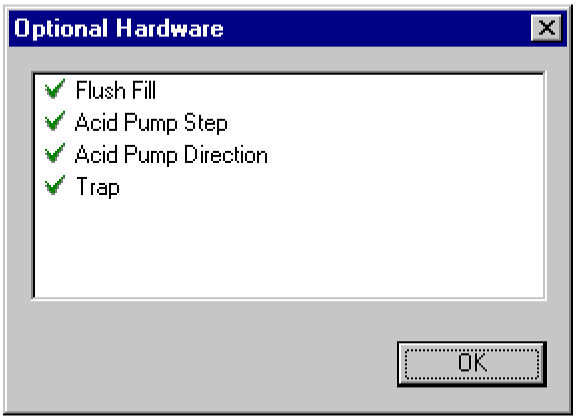


- Press the ***"Gasbench Sets" tab*** in the middle pane.
- Select the peripheral to be attached to the IRMS by a click on it in the right pane (e.g. Gasbench + Autosampler).



- Drag and drop the selected entry (e.g. "Gasbench + Autosampler") to the "Capillary" symbol of the MS Delta^{Plus} XL, at bottom of left pane.

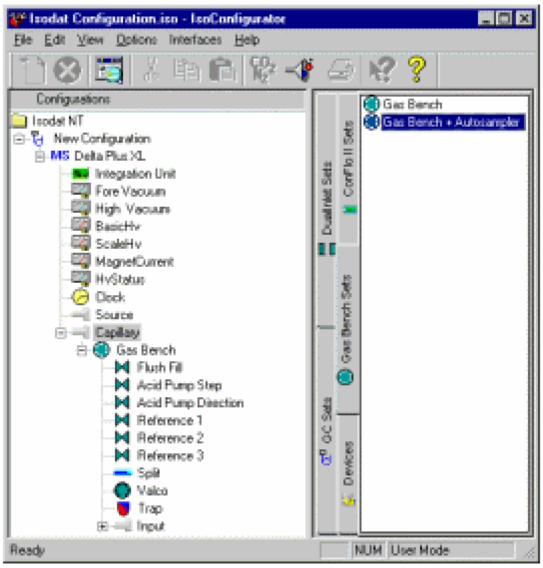
Capillary



✓ Flush Fill
✓ Acid Pump Step
✓ Acid Pump Direction
✓ Trap

Particularity of Gasbench peripherals:

- Deselect or select optional Hardware by marking or not marking the items.

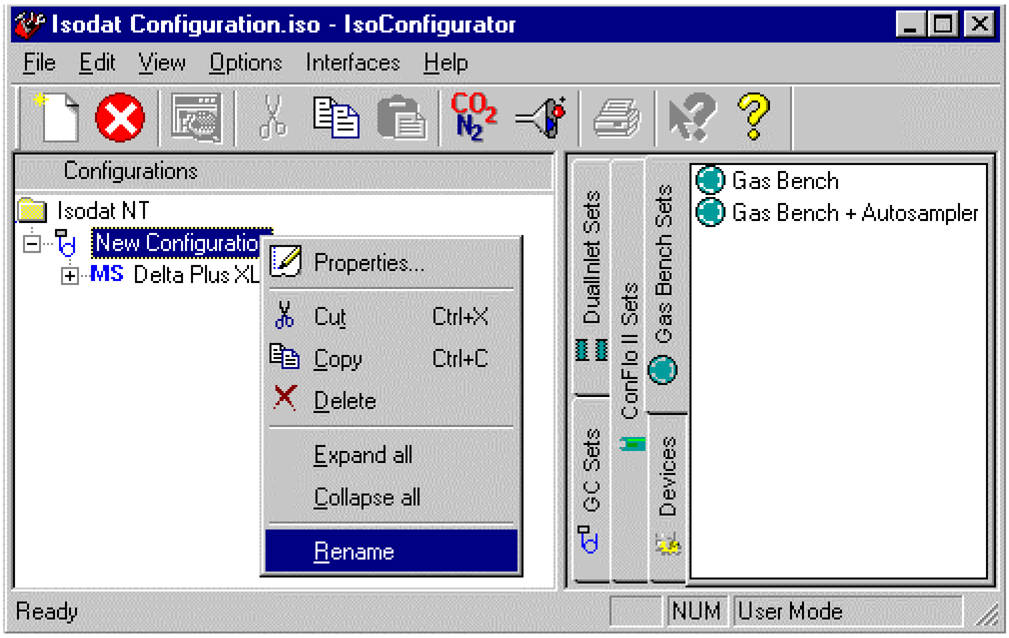


- The GasBench peripheral has been inserted beneath the "Capillary" symbol (beginning at the "GasBench" entry).

The capillary Icon on the left side of "New Configuration" has not changed. ISODAT NT automatically detects that a Gasbench peripheral is being attached and indicates this by the capillary Icon.

- Rename the new Configuration hitherto called "New Configuration". This could also already be done when the entry "New Configuration" appears the first time.

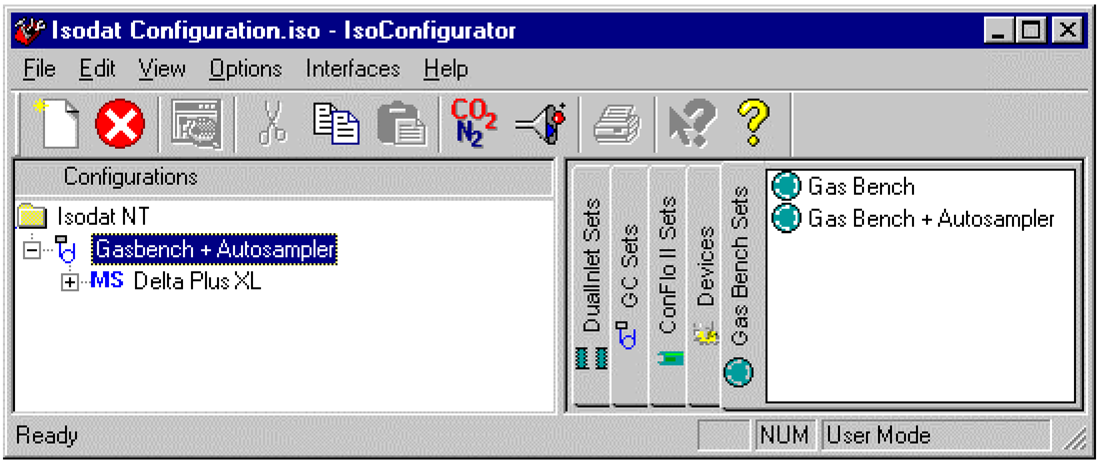
- Choose a significant name to identify it easily if creating more than one Configuration (e.g. "Gasbench + Autosampler").



- Right-click on ***New Configuration***.
- In the popup menu select ***Rename***.

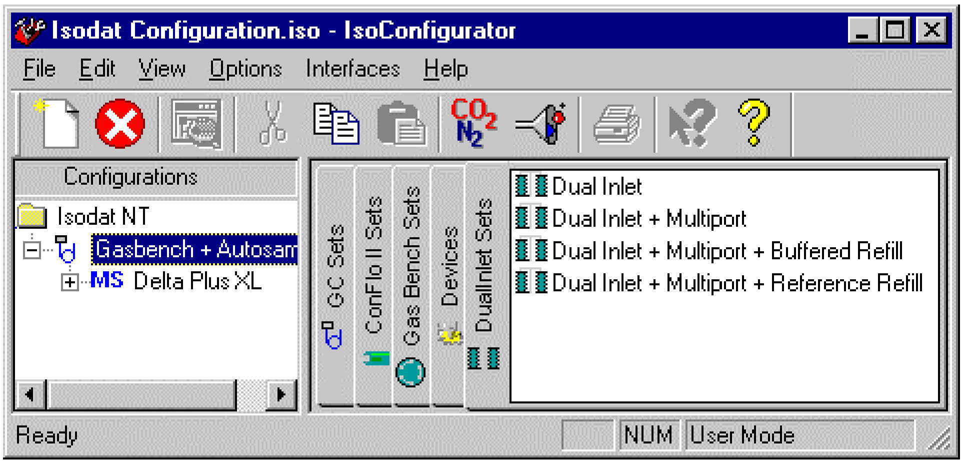
Alternative:

- From the ***Edit*** menu select ***Rename***.



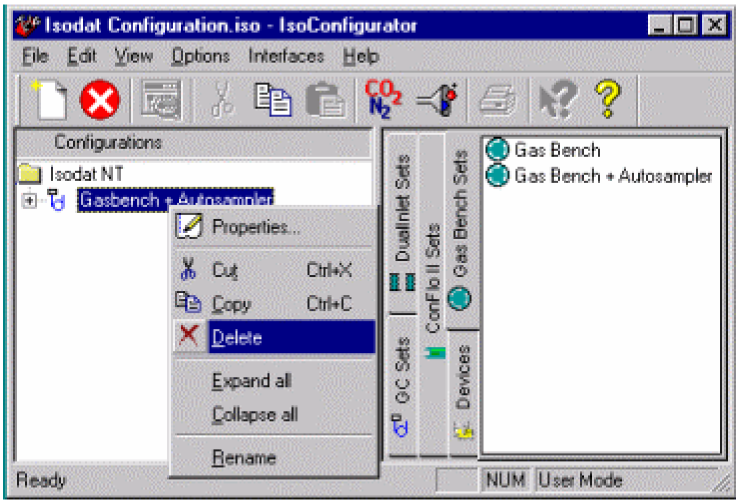
- ➢ Type the name of the new Configuration (e.g. "Gasbench + Autosampler").
- ➢ Click somewhere on the screen to finish renaming.

2.4.5 DELETING CONFIGURATIONS



- ➢ In the Configurator's left pane, click on the Configuration (e.g. "Gasbench + Autosampler").

- On the toolbar, press the **Delete** button.



Alternative:

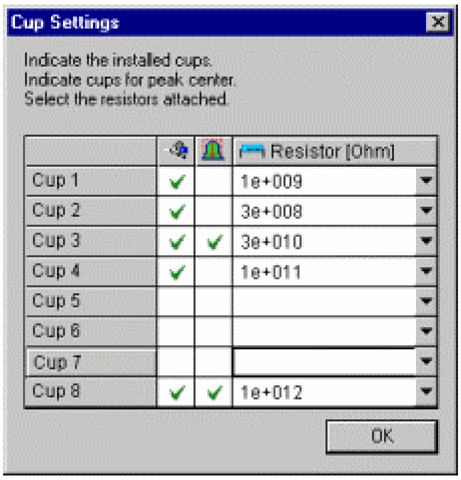
- Right-click the Configuration and select **Delete** from the popup menu.

Are you shure to delete <Gasbench + Autosampler?>

- Confirm by ***Yes***. ***No*** cancels the process.

2.5 EDITING CUP SETTINGS

The box below automatically appears after selecting the IRMS type.



Cup Settings

Indicate the installed cups.
Indicate cups for peak center.
Select the resistors attached.

			Resistor [Ohm]
Cup 1	✓		1e+009
Cup 2	✓		3e+008
Cup 3	✓	✓	3e+010
Cup 4	✓		1e+011
Cup 5			
Cup 6			
Cup 7			
Cup 8	✓	✓	1e+012

- Installed cups are marked in the left column. Only marked cups will be available in "Gas Configurations".
- Cups available for Peak Center are marked in the middle column. Peak Center is only recommended on narrow cups.
- The resistor values of the cups are shown and edited in the right column.

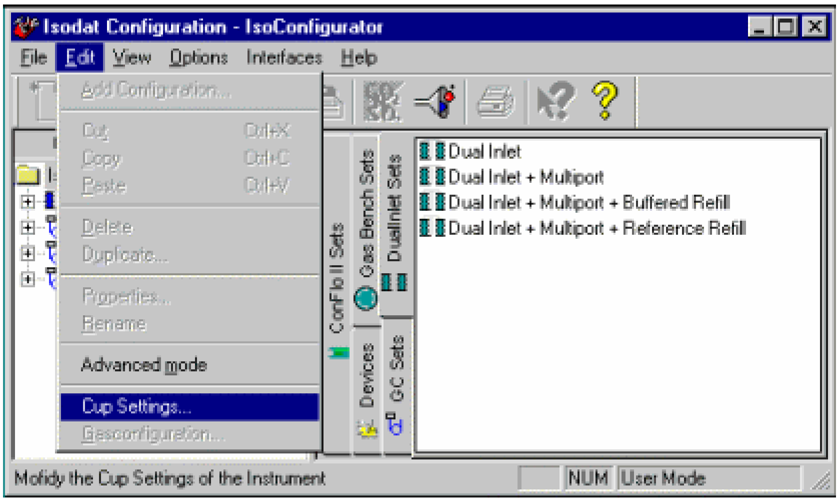
Ensure the Cup Settings are matching your IRMS configuration.

For specific Cup Settings consult the system's technical information.

- Select the particular fields, type in the correct numbers or select a resistor value from the pulldown list.

Resistor values influence:

- Continuous Flow area determinations
- Diagnosis results
- H3-factor determination



Mofidy the Cup Settings of the Instrument

- ➢ To change Cup Settings at ***later*** times select ***Cup Settings*** from the Configurator's ***Edit*** Menu.

Alternative:

- ➢ Click the ***Modify Cup Settings*** Icon on the toolbar.

2.5.1 EXAMPLES OF CUP SETTINGS - 1. DELTA$^{\text{PLUS}}$ XL

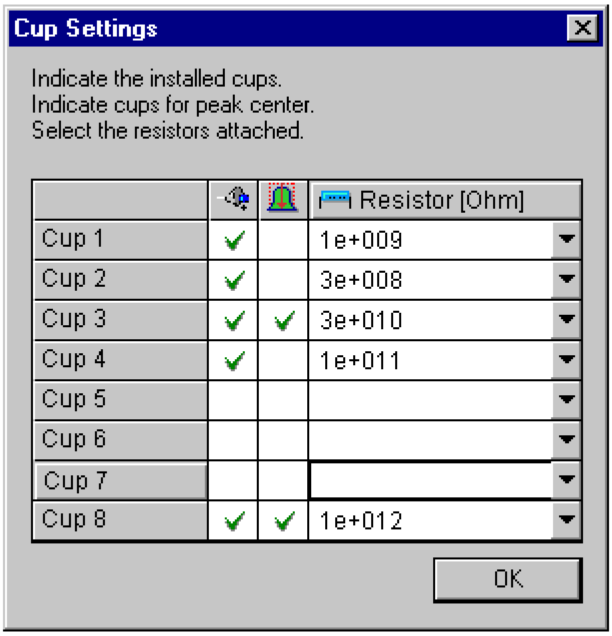
- ➢ Cups **2 - 4**:

  N_2-Cups and

  CO_2-Cups

- ➢ Cups **1 + 8**:

  H_2-Cups



Indicate the installed cups.
Indicate cups for peak center.
Select the resistors attached.

			Resistor [Ohm]
Cup 1	✓		1e+009
Cup 2	✓		3e+008
Cup 3	✓	✓	3e+010
Cup 4	✓		1e+011
Cup 5			
Cup 6			
Cup 7			
Cup 8	✓	✓	1e+012

Universal Triple Detector: Cup **2** - Cup **4**

Peak Centering can only be performed on Cup **3** (narrow cup).

Measurements of CO_2, N_2 are performed on the *same* Cups (Cup 2, 3 and 4) and with the *same* resistor values.

HD Detector: Cup **1** + Cup **8**

Peak Centering for H_2 is performed on Cup **8** (narrow cup).

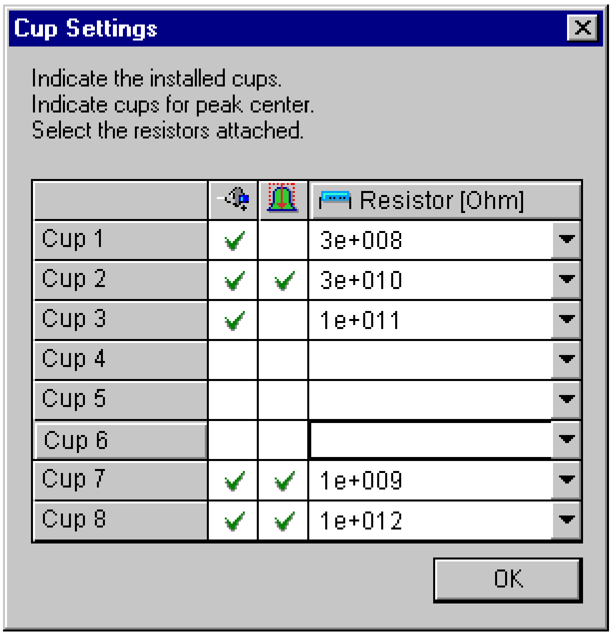
2.5.2 EXAMPLES OF CUP SETTINGS - 2. DELTA^{PLUS}

- Cups **1 - 3**:

  N_2-Cups and CO_2-Cups

- Cups **7 - 8**:

  H_2-Cups



Cup Settings

Indicate the installed cups.
Indicate cups for peak center.
Select the resistors attached.

			Resistor [Ohm]
Cup 1	✓		3e+008
Cup 2	✓	✓	3e+010
Cup 3	✓		1e+011
Cup 4			
Cup 5			
Cup 6			
Cup 7	✓	✓	1e+009
Cup 8	✓	✓	1e+012

NOTE: ***The Delta Plus Cup Settings are identical with Delta C, but additionally a H_2-option exists.***

Universal Triple Detector: Cup **1** - Cup **3**

Peak Centering can only be performed on Cup **2** (narrow cup).

Measurements of CO_2, N_2 are performed on the *same* Cups (Cup1, 2 and 3) and with the *same* resistor values.

HD Detector: Cup **7** - Cup **8**

Peak Centering is possible on both cups.

2.5.3 EXAMPLES OF CUP SETTINGS - 3. DELTA S AND MAT 252

- Cups 1 - 3:

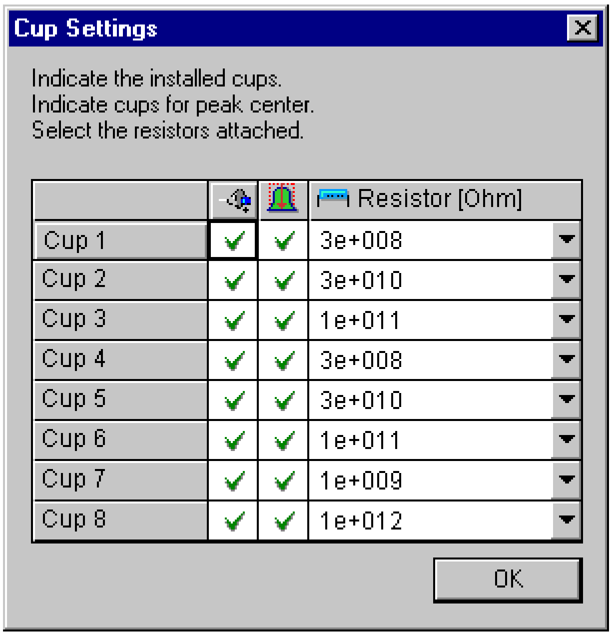
  N_2-Cups

- Cups 4 - 6:

  CO_2-Cups

- Cups 7 - 8:

  H_2-Cups



Cup Settings

Indicate the installed cups.
Indicate cups for peak center.
Select the resistors attached.

			Resistor [Ohm]
Cup 1	✓	✓	3e+008
Cup 2	✓	✓	3e+010
Cup 3	✓	✓	1e+011
Cup 4	✓	✓	3e+008
Cup 5	✓	✓	3e+010
Cup 6	✓	✓	1e+011
Cup 7	✓	✓	1e+009
Cup 8	✓	✓	1e+012

MEMCO Detector: Cup **1** - Cup **6**

Peak Centering is possible on ***all*** cups, because all cups are narrow.

HD Detector: Cup **7** - Cup **8**

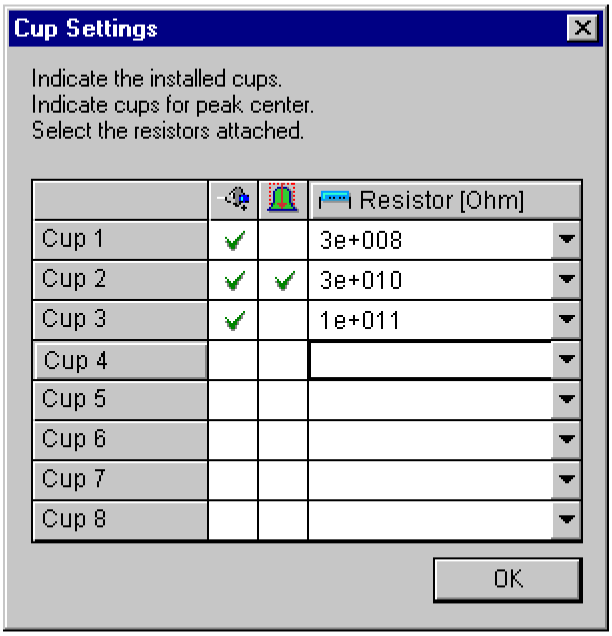
Peak Centering is possible on ***both*** cups.

2.5.4 EXAMPLES OF CUP SETTINGS - 4. DELTA C

- Cups **1 - 3**:

  N_2-Cups and

  CO_2-Cups



Cup Settings

Indicate the installed cups.
Indicate cups for peak center.
Select the resistors attached.

	☐	☐	Resistor [Ohm]
Cup 1	✓		3e+008
Cup 2	✓	✓	3e+010
Cup 3	✓		1e+011
Cup 4			
Cup 5			
Cup 6			
Cup 7			
Cup 8			

Universal Triple Detector: Cup **1** - Cup **3**

Peak Centering can only be performed on Cup 2 (narrow cup).

Measurements of CO_2 and N_2 are performed on the ***same*** Cups (Cup1, 2 and 3) and with the ***same*** resistor values.

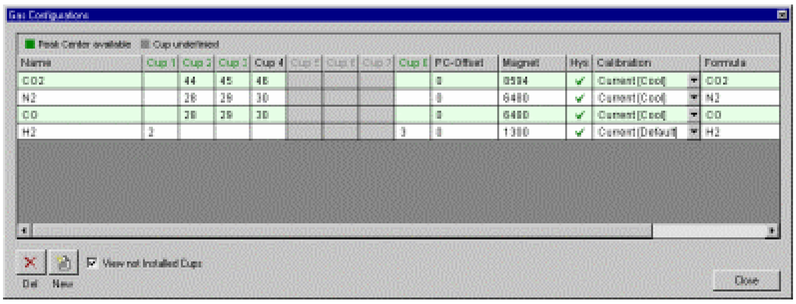
2.6 EDITING GAS CONFIGURATIONS

A Gas Configuration is the information pattern about the gases to be analyzed, the isotope masses of interest and their corresponding cups. Furthermore, data about PC-Offset, magnet information and Calibrations are given in the "Gas Configurations" window.

Green marked cups indicate peak centering availability. ***White*** marked ones are installed, but not available for peak centering. ***Grey*** marked ones are not installed (undefined).

Type the chemical formulas of the gases to be analyzed in the "Formula" column!

Typing it only in the "Name" column is not sufficient!

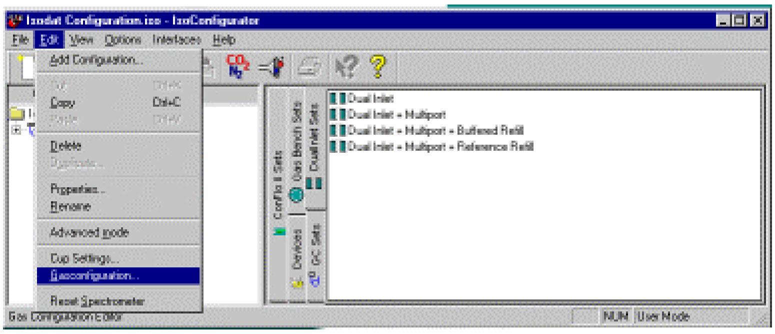


Name	Cup 1	Cup 2	Cup 3	Cup 4	Cup 5	Cup 6	Cup 7	Cup 8	PC-Offset	Magnet	Hys	Calibration	Formula
CO2		44	45	46					0	8594	✓	Current[Cool]	CO2
N2		28	29	30					0	6480	✓	Current[Cool]	N2
CO		28	29	30					0	6480	✓	Current[Cool]	CO
H2	2							3	0	1380	✓	Current[Default]	H2

Ensure that the Gas Configurations are matching your IRMS configuration.

- ➢ Select the particular fields, type in the correct numbers or choose them from the pulldown list.
- ➢ When all changes are made press ***Close***.

2.6.1 CHANGING GAS CONFIGURATIONS

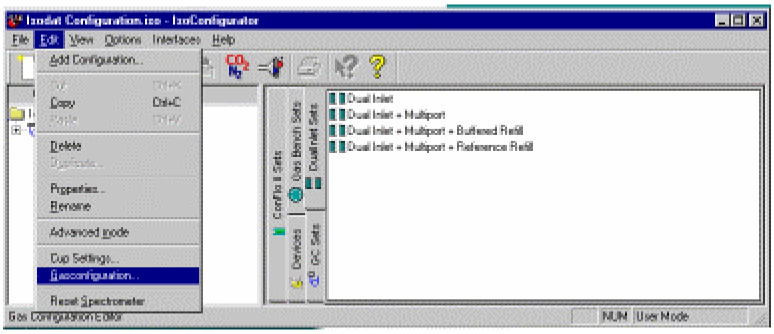


Alternative:

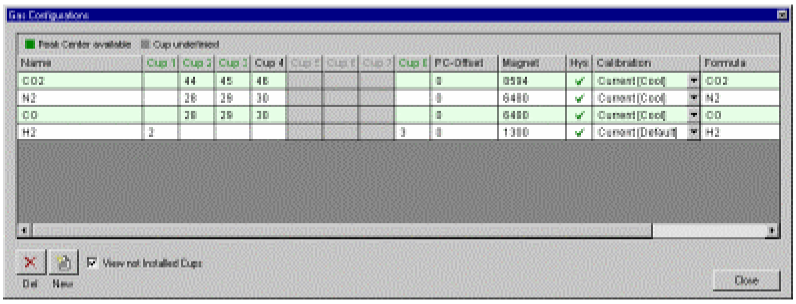
Click the ***Gas Configuration*** button on the toolbar.

Proceed as shown on Page 2 – 27.

2.6.2 ADDING GAS CONFIGURATIONS

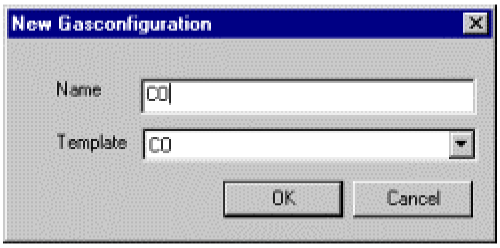


- ➢ From the Configurator's ***Edit*** menu select ***Gasconfiguration***.



Name	Cup 1	Cup 2	Cup 3	Cup 4	Cup 5	Cup 6	Cup 7	Cup 8	PC-Offset	Magnet	Hys	Calibration	Formula
CO2		44	45	46					0	8594	✓	Current[Cool]	CO2
N2		28	29	30					0	6480	✓	Current[Cool]	N2
CO		28	29	30					0	6480	✓	Current[Cool]	CO
H2	2							3	0	1300	✓	Current[Default]	H2

- ➢ Press the ***New*** button.



Name: CO

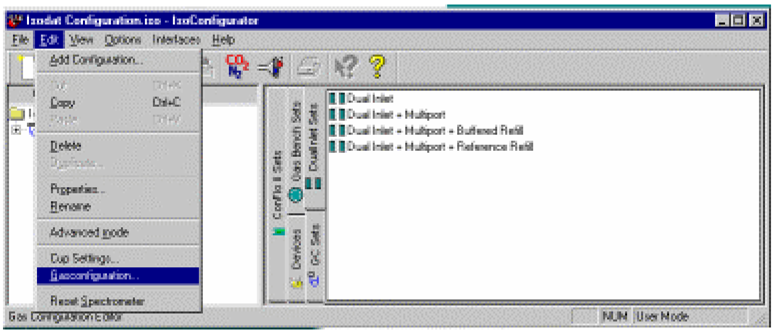
Template: CO

- ➢ In the ***Name*** edit box, type the Gas Configuration's name, e.g. "CO".

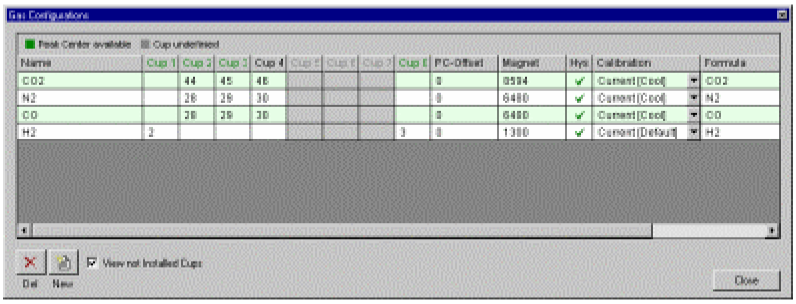
  Choose it as you like. It must not necessarily be a gas name, but is recommended.

- ➢ From the ***Template*** pulldown menu, select the gas.

2.6.3 DELETING GAS CONFIGURATIONS



- ➢ From the Configurator's ***Edit*** menu select ***Gasconfiguration***.



Name	Cup 1	Cup 2	Cup 3	Cup 4	Cup 5	Cup 6	Cup 7	Cup 8	PC-Offset	Magnet	Hys	Calibration	Formula
CO2		44	45	46					0	8594	✓	Current[Cool]	CO2
N2		28	29	30					0	6480	✓	Current[Cool]	N2
CO		28	29	30					0	6480	✓	Current[Cool]	CO
H2	2							3	0	1300	✓	Current[Default]	H2

☑ View not Installed Cups

- ➢ Select one or more Gas Configurations to delete.
- ➢ Press the ***Del*** button.

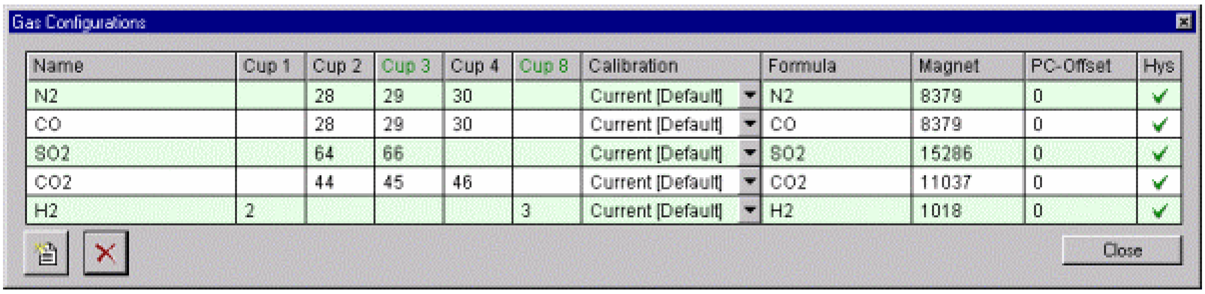
Delete Selected Gasconfigurations ?

N2
CO

- ➢ Confirm by ***Yes***.
- ➢ Close the Configurator.

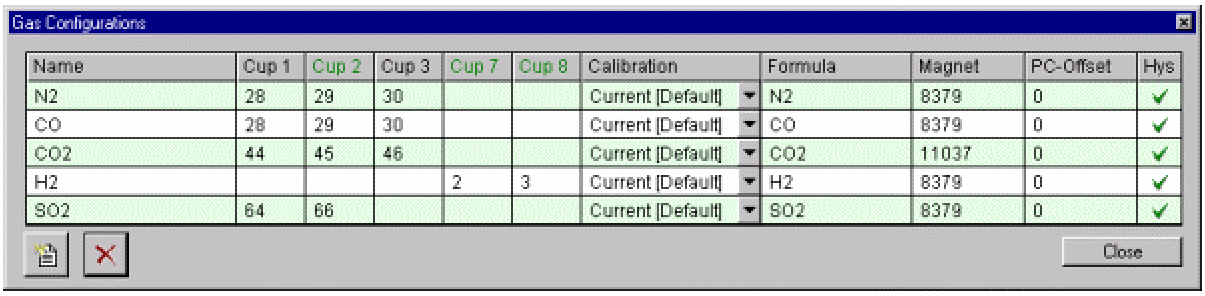
2.6.4 EXAMPLES FOR GAS CONFIGURATIONS

1. Delta^{Plus} XL



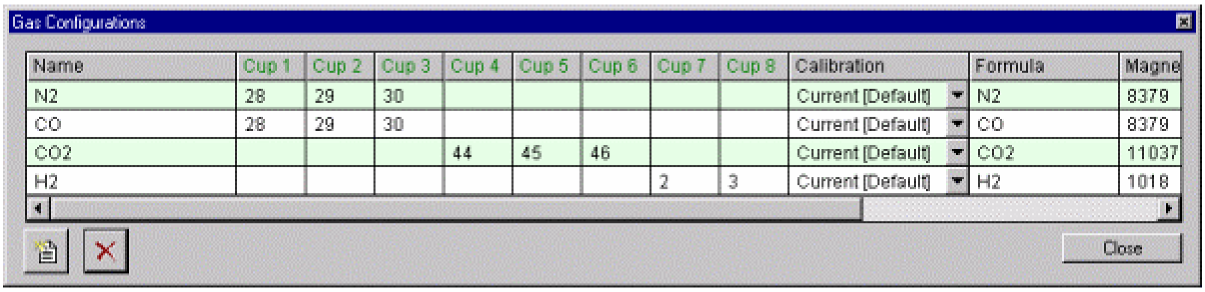
Name	Cup 1	Cup 2	Cup 3	Cup 4	Cup 8	Calibration	Formula	Magnet	PC-Offset	Hys
N2		28	29	30		Current [Default]	N2	8379	0	✓
CO		28	29	30		Current [Default]	CO	8379	0	✓
SO2		64	66			Current [Default]	SO2	15286	0	✓
CO2		44	45	46		Current [Default]	CO2	11037	0	✓
H2	2				3	Current [Default]	H2	1018	0	✓

2. Delta^{Plus}



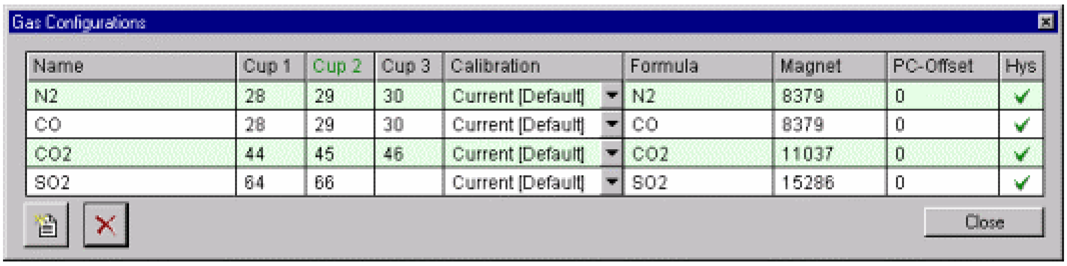
Name	Cup 1	Cup 2	Cup 3	Cup 7	Cup 8	Calibration	Formula	Magnet	PC-Offset	Hys
N2	28	29	30			Current [Default]	N2	8379	0	✓
CO	28	29	30			Current [Default]	CO	8379	0	✓
CO2	44	45	46			Current [Default]	CO2	11037	0	✓
H2				2	3	Current [Default]	H2	8379	0	✓
SO2	64	66				Current [Default]	SO2	8379	0	✓

3. Delta S and MAT 252



Name	Cup 1	Cup 2	Cup 3	Cup 4	Cup 5	Cup 6	Cup 7	Cup 8	Calibration	Formula	Magne
N2	28	29	30						Current [Default]	N2	8379
CO	28	29	30						Current [Default]	CO	8379
CO2				44	45	46			Current [Default]	CO2	11037
H2							2	3	Current [Default]	H2	1018

4. Delta C



Name	Cup 1	Cup 2	Cup 3	Calibration	Formula	Magnet	PC-Offset	Hys
N2	28	29	30	Current [Default]	N2	8379	0	✓
CO	28	29	30	Current [Default]	CO	8379	0	✓
CO2	44	45	46	Current [Default]	CO2	11037	0	✓
SO2	64	66		Current [Default]	SO2	15286	0	✓

ISODAT NT

OPERATING MANUAL

3

INSTRUMENT CONTROL

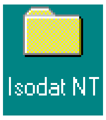
Issue 06/2002

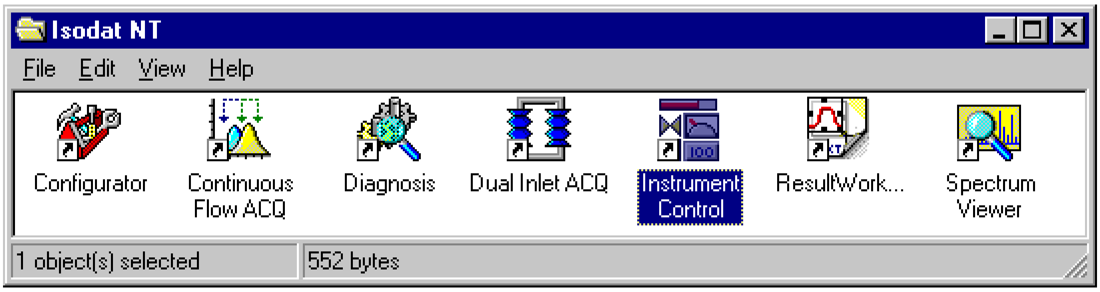
Thermo Finnigan

3.1 MASS CALIBRATION

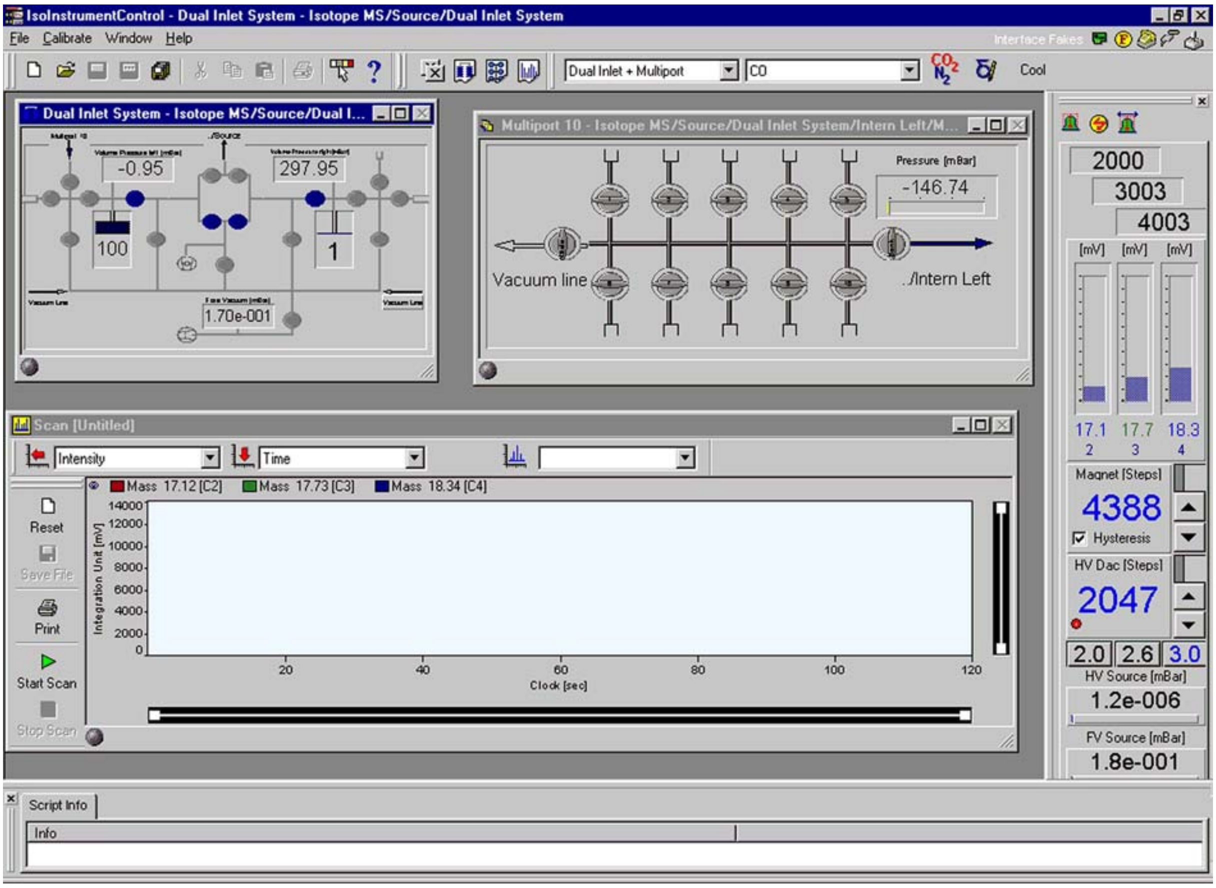
3.1.1 OPEN INSTRUMENT CONTROL

- Open the ***Isodat NT*** window on the desktop.





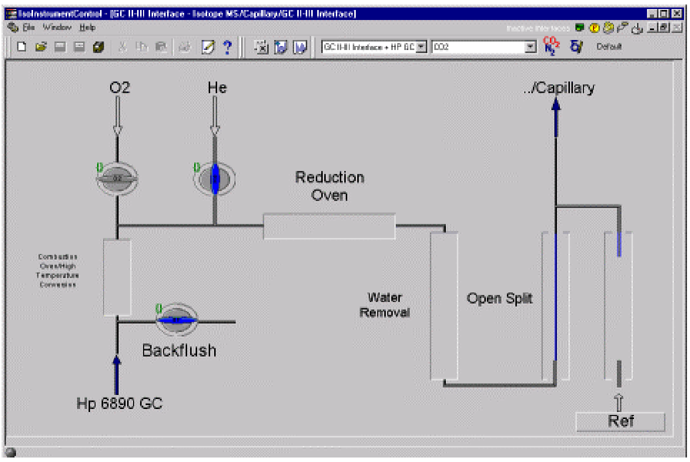
- Start Instrument Control by a double-click on the ***Instrument Control*** Icon.



- The ***Instrument Control window*** appears.

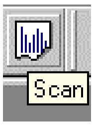
3.1.2 PREPARE CALIBRATION

- Select a ***Gas Configuration*** (e.g. "CO_2").
- Select a ***Configuration*** (e.g. "Dual Inlet + Multiport").
- Ensure that a ***Reference Gas container*** (e.g. CO_2) is attached to the hardware of the selected Configuration.

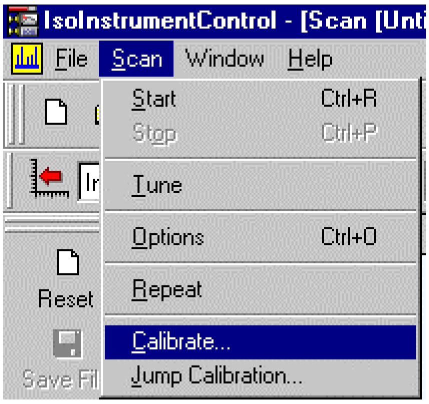


- Click to ***activate Reference Gas*** (e.g. GC II-III Interface).

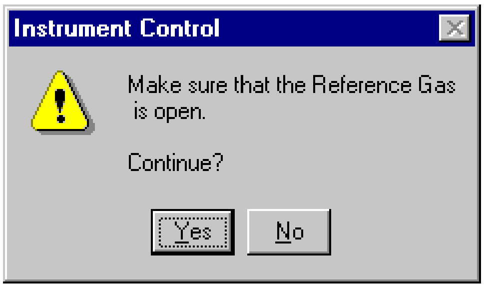
3.1.3 START CALIBRATION



- ➢ Activate the ***Scan Window*** by a click on the Icon.
- ➢ Thus, the ***Scan*** menu becomes available.



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



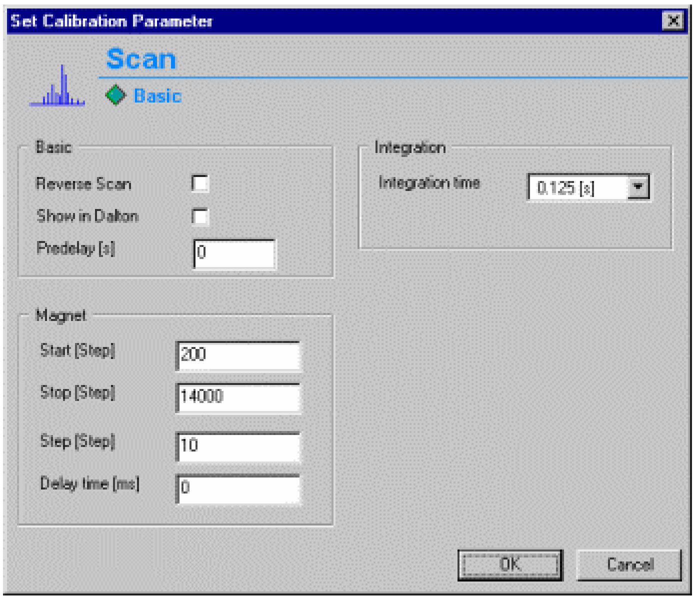
Make sure that the Reference Gas is open.

Continue?

- ➢ If the Reference Gas is already activated continue with ***Yes***.
- ➢ Otherwise, press ***No*** and refer to Chapter 3.1.2 to activate the Reference Gas.

3.1.4 SET CALIBRATION PARAMETERS

NOTE: ***Depending on the IRMS type, the Start values, Stop values and Calibration Scans can be different.***


Set Calibration Parameter

Scan

Basic

Basic

Reverse Scan ☐

Show in Dalton ☐

Predelay [s] 0

Magnet

Start [Step] 200

Stop [Step] 14000

Step [Step] 10

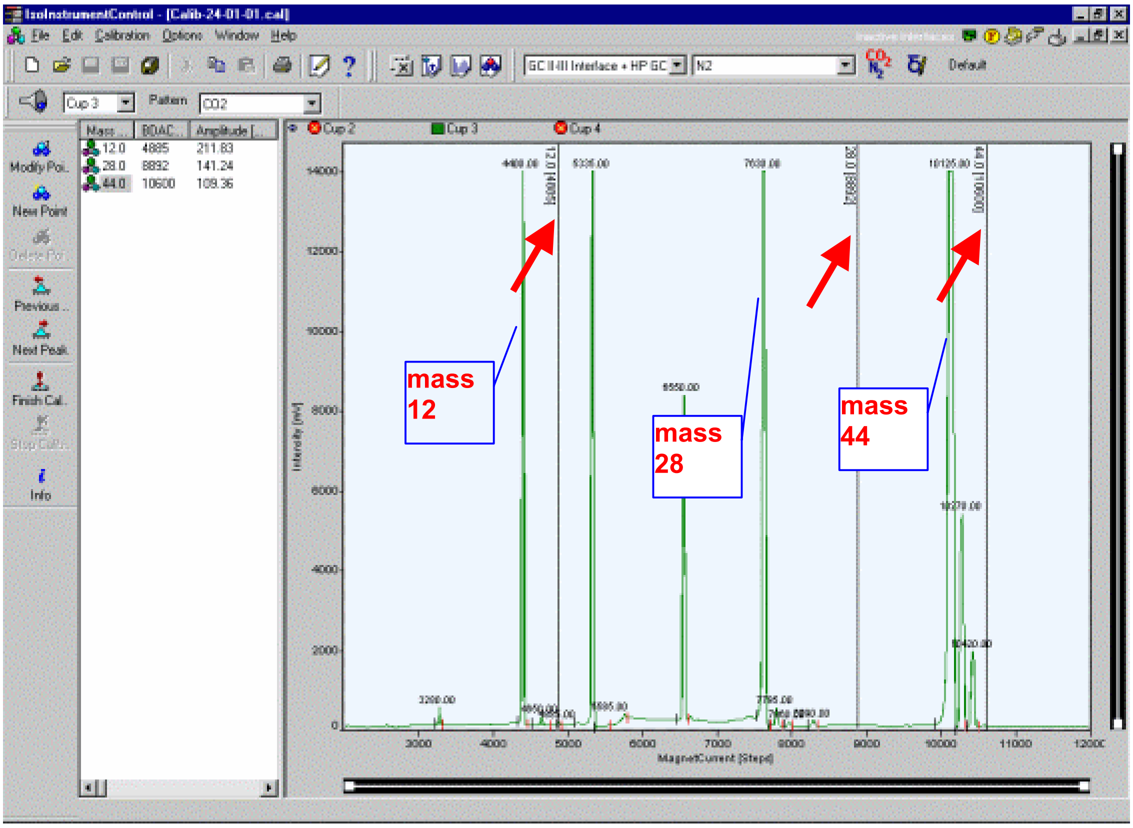
Delay time [ms] 0

Integration

Integration time 0.125 [s]

- For a first Scan, keep the default values.
- Confirm by ***OK***.

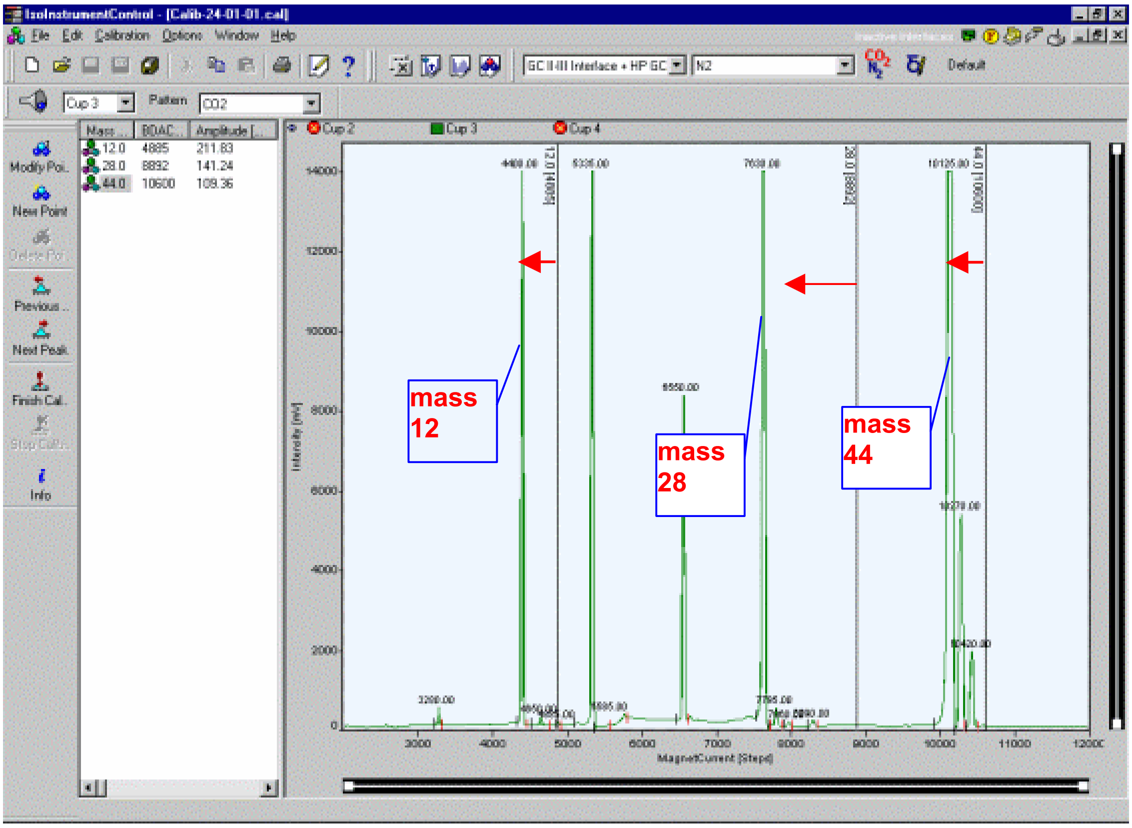
3.1.5 CALIBRATION SCAN



Mass ...	BDAC...	Amplitude [...
12.0	4885	211.83
28.0	8892	141.24
44.0	10600	109.36

- The Calibration Scan is finished.
- Vertical lines appear (see the arrows) for assigning the correct position of the calibration masses.

3.1.6 ASSIGN CALIBRATION MASSES



Mass ...	BDAC...	Amplitude [...
12.0	4885	211.83
28.0	8892	141.24
44.0	10600	109.36

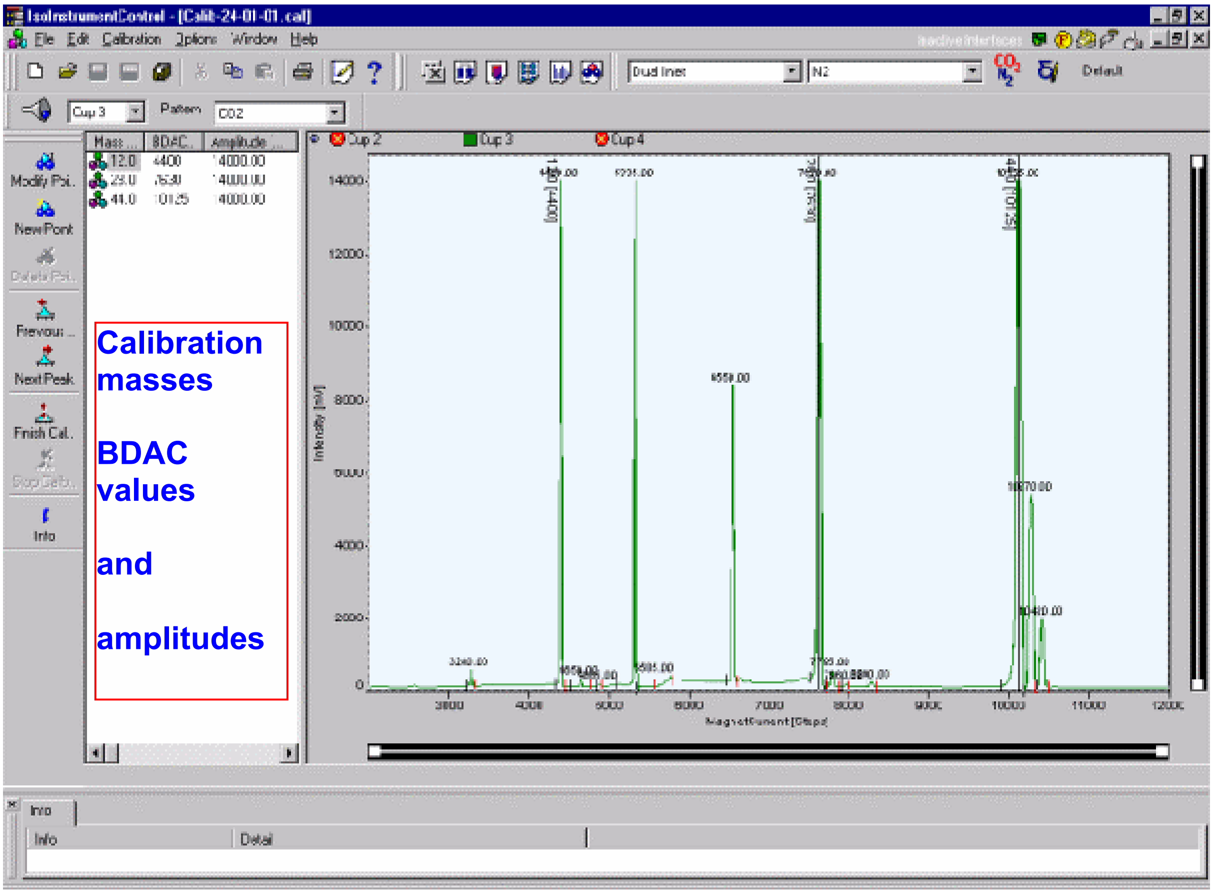
- ➢ Assign the calibration masses to their peaks by drag&drop of the vertical lines with your mouse, snapping the lines to the mass peaks.

Alternative:

- ➢ Click the ***Next Peak*** or ***Previous Peak*** Icon on the left toolbar to move the lines.

Next Peak

Previous ..



Mass ...	BDAC...	Amplitude ...
12.0	4400	4000.00
28.0	7630	4000.00
44.0	10125	4000.00

Calibration masses

BDAC values

and

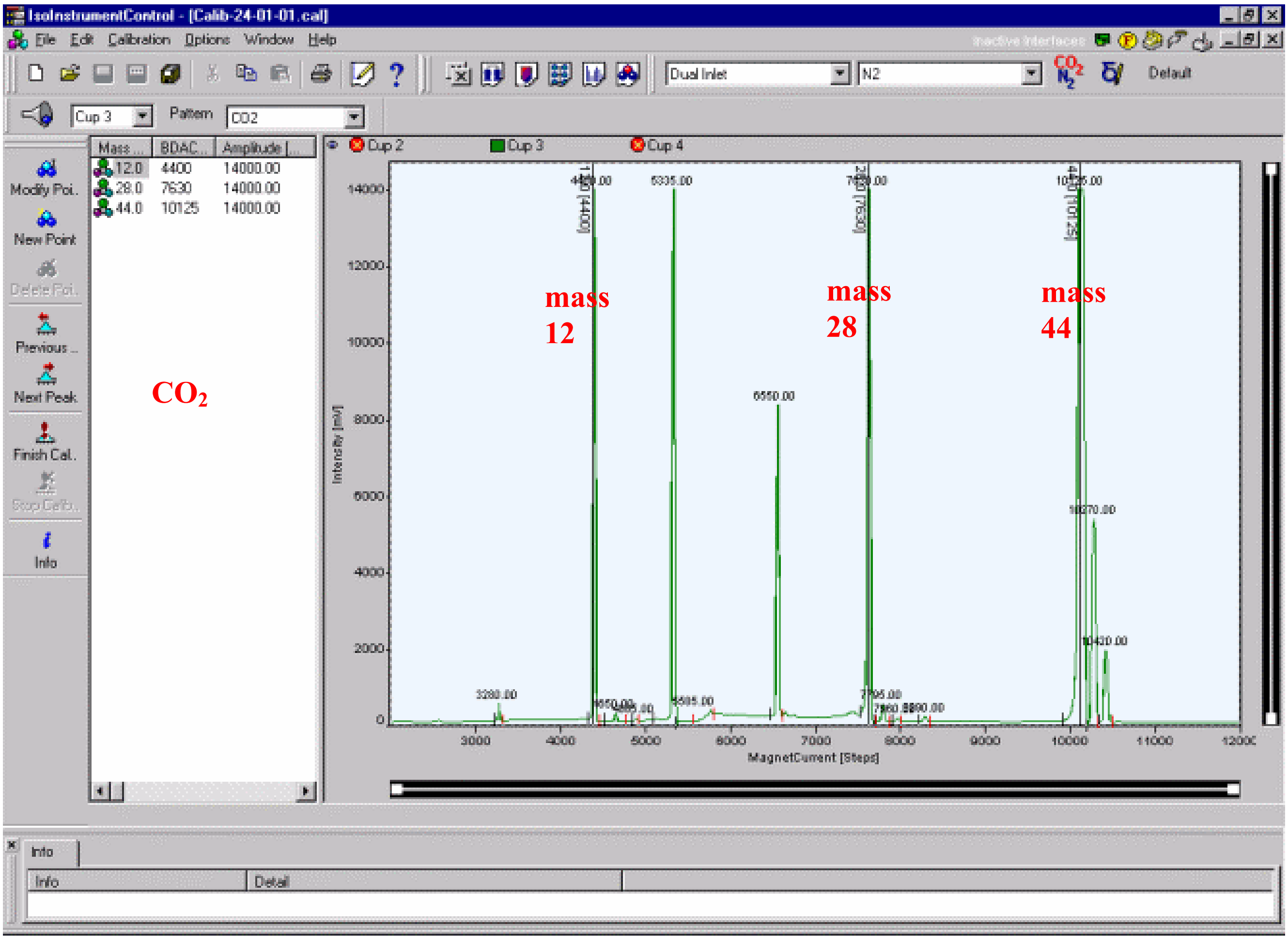
amplitudes

- In the left window, the calibration masses of the Calibration Scan are now listed together with their corresponding BDAC-values and amplitudes.

NOTE: ***Depending on the IRMS type and the calibration gases, the Calibration Scans are different.***

3.1.6.1 EXAMPLES FOR CALIBRATION SCANS

CO_2 Calibration Scan on a Delta Plus XL



IsoInstrumentControl - [Calib-24-01-01.cal]

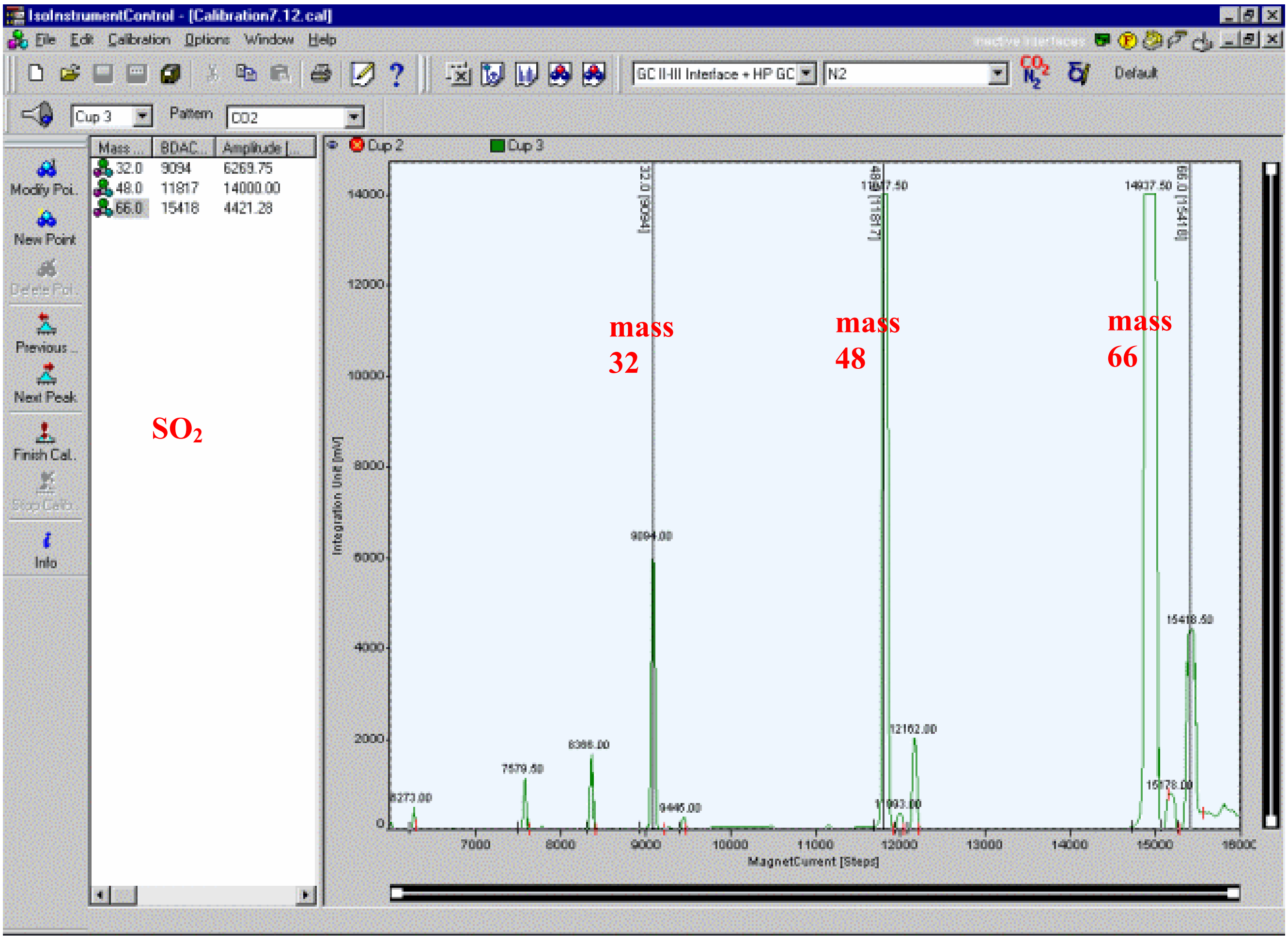
Dual Inlet N2 Default

Cup 3 Pattern CO2

Mass	BDAC	Amplitude
12.0	4400	14000.00
28.0	7630	14000.00
44.0	10125	14000.00

- ➤ Assign the calibration masses to their peaks by drag & drop of the vertical lines using your mouse, snapping the lines to the mass peaks.

SO_2 Calibration Scan on a Delta Plus XL



IsoInstrumentControl - [Calibration7.12.cal]

GC II-III Interface + HP GC · N2 · Default

Cup 3 · Pattern: CO2

Mass ...	BDAC...	Amplitude [...
32.0	9094	6269.75
48.0	11817	14000.00
66.0	15418	4421.28

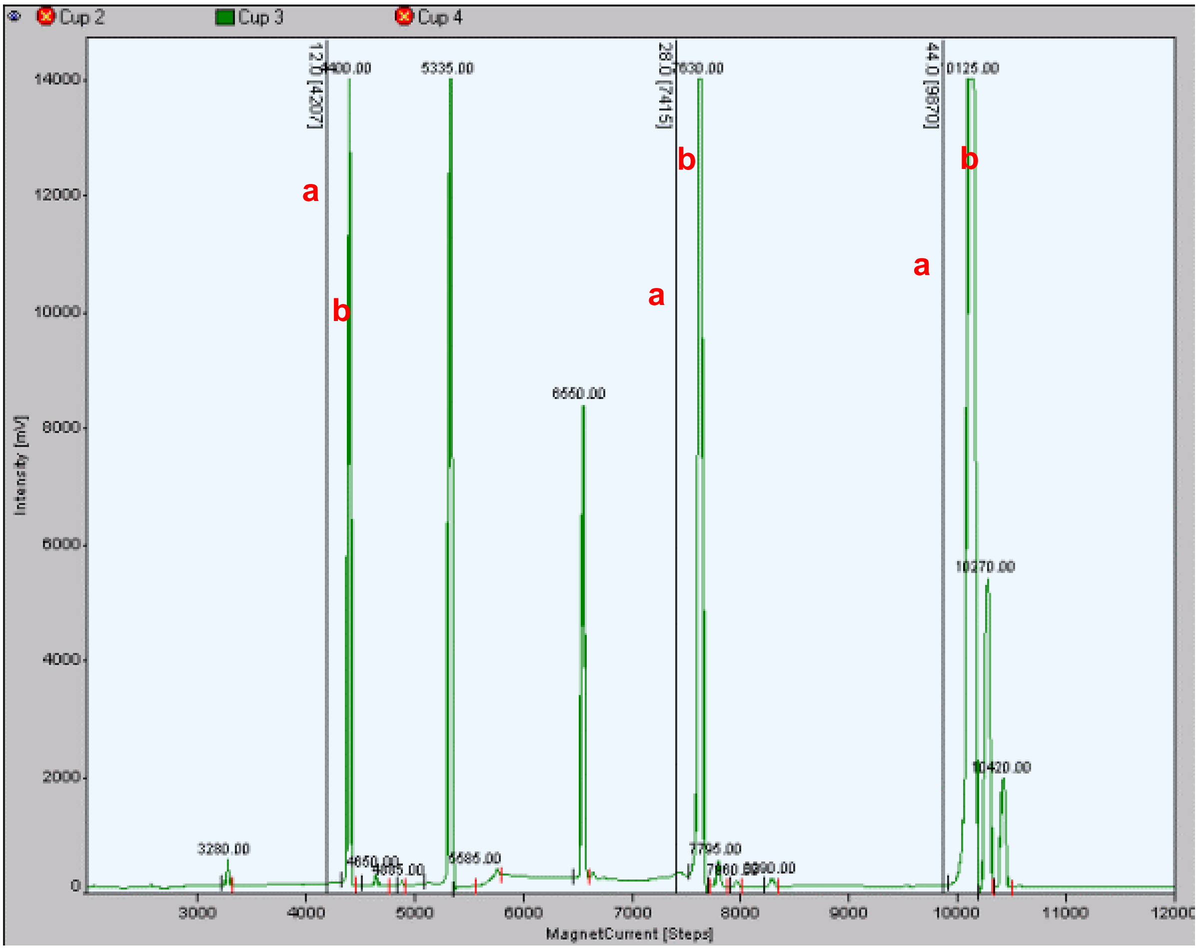
- ➢ Assign the calibration masses to their peaks by drag & drop of the vertical lines using your mouse, snapping the lines to the mass peaks.

3.1.7 FINISH CALIBRATION

- From the toolbar on the left select the ***Finish Calibration*** Icon.

- To finish Calibration, ISODAT NT jumps to the individual calibration masses and optimizes the BDAC value by a Peak Center procedure.

Finish Cal..



a optimized BDAC-value for mass peak after Mass Calibration

b mass peak by Magnet Scan

- The Calibration performed is optimized for jumping correctly at masses overcoming the

  magnet's hysteresis.

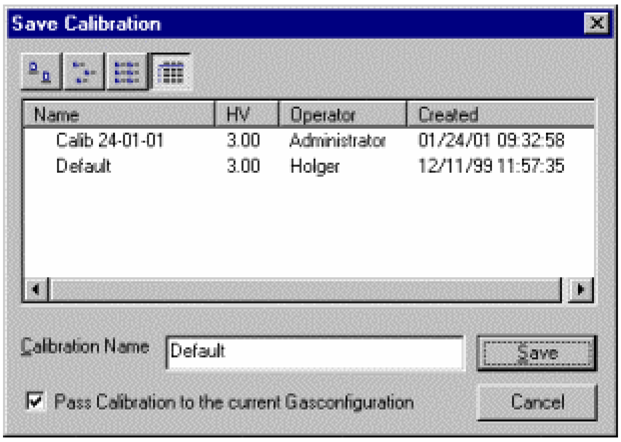
  In opposite to the previously performed Mass Scan, the correct BDAC-values always

  differ by several BDAC-steps to lower values, due to the magnet's hysteresis.

  BDAC-steps are transferred into masses (and vice versa).

3.1.8 SAVE CALIBRATION

When Calibration is finished, it must finally be saved.



Name	HV	Operator	Created
Calib 24-01-01	3.00	Administrator	01/24/01 09:32:58
Default	3.00	Holger	12/11/99 11:57:35

Calibration Name: Default

☑ Pass Calibration to the current Gasconfiguration

- Type a Calibration name and click ***Save***.

- Using ***Save As*** in the ***File*** menu, the Calibration Scan can be saved for documentation only.

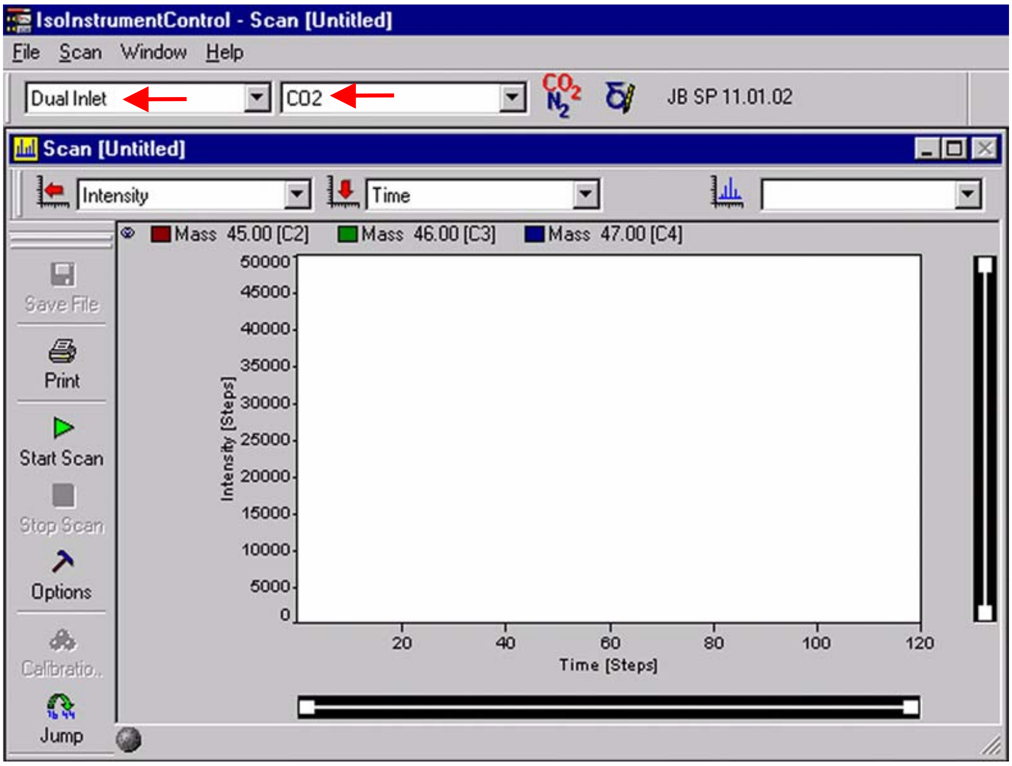
Do not use old Calibration Scans for Recalibration!

3.2 BELLOW CALIBRATION

Prior to a Dual Inlet Acquisition, a Bellow Calibration must be performed as follows:

It is presumed, that the source has been switched on and focusing has been performed.

- Start Instrument Control by double-clicking the ***Instrument Control*** Icon.
- The ***Scan*** window appears.



IsoInstrumentControl - Scan [Untitled]

Dual Inlet

CO2

JB SP 11.01.02

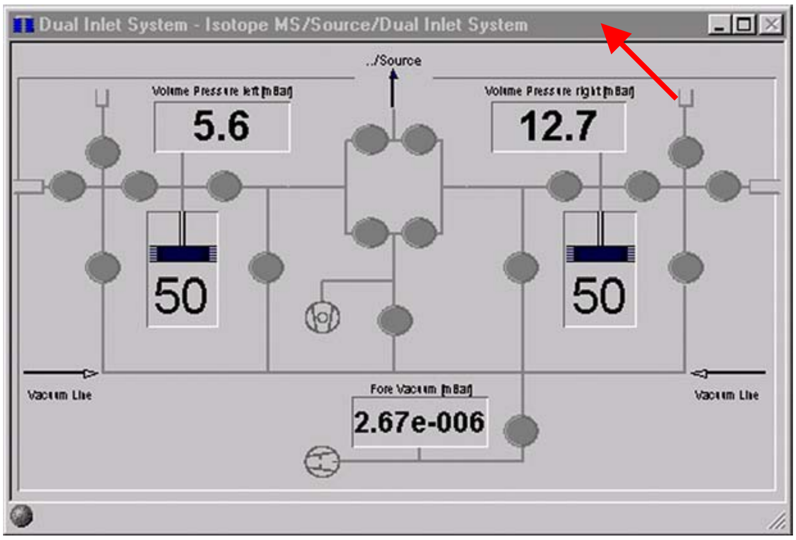
Scan [Untitled]

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

Dual Inlet

- Select your Gas Configuration, e.g. ***CO$_2$***.

CO2



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

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

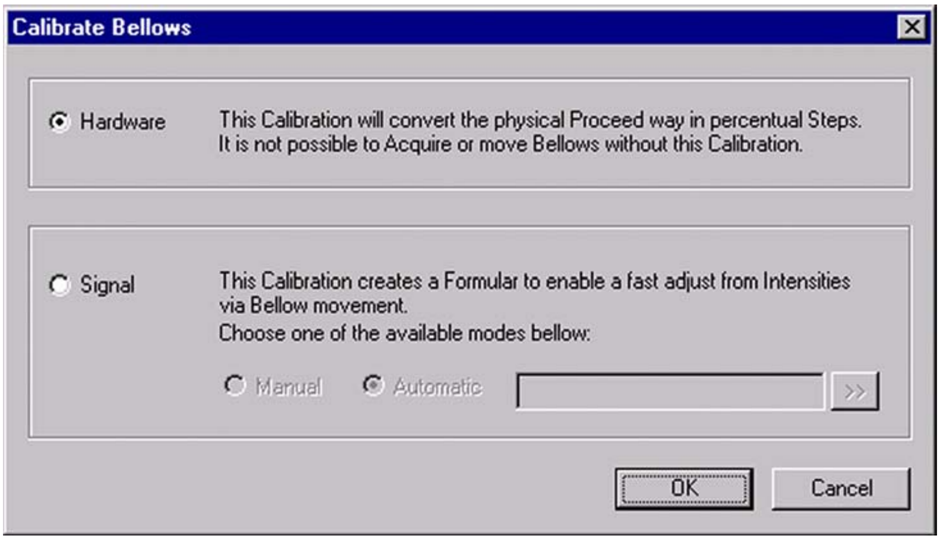
Access to the Calibrate Menu won't be possible while this window is inactive.

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

  Bellow Calibration must be performed in two steps:

Step 1 ***Hardware Calibration of the bellows***

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



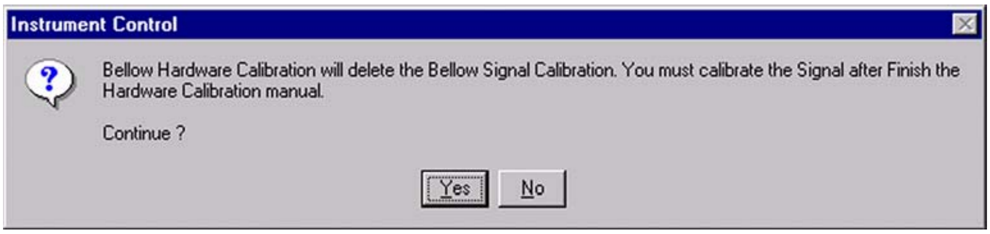
Calibrate Bellows

● Hardware — This Calibration will convert the physical Proceed way in percentual Steps. It is not possible to Acquire or move Bellows without this Calibration.

○ Signal — This Calibration creates a Formular to enable a fast adjust from Intensities via Bellow movement. Choose one of the available modes bellow:

○ Manual ● Automatic

- Enable the ***Hardware*** radio button.
- Press ***OK***.



Instrument Control

Bellow Hardware Calibration will delete the Bellow Signal Calibration. You must calibrate the Signal after Finish the Hardware Calibration manual.

Continue ?

- ➢ Press ***Yes*** to continue.

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

Instrument Control

Bellow Calibration complete !

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

Step 2 Signal Calibration of the bellows

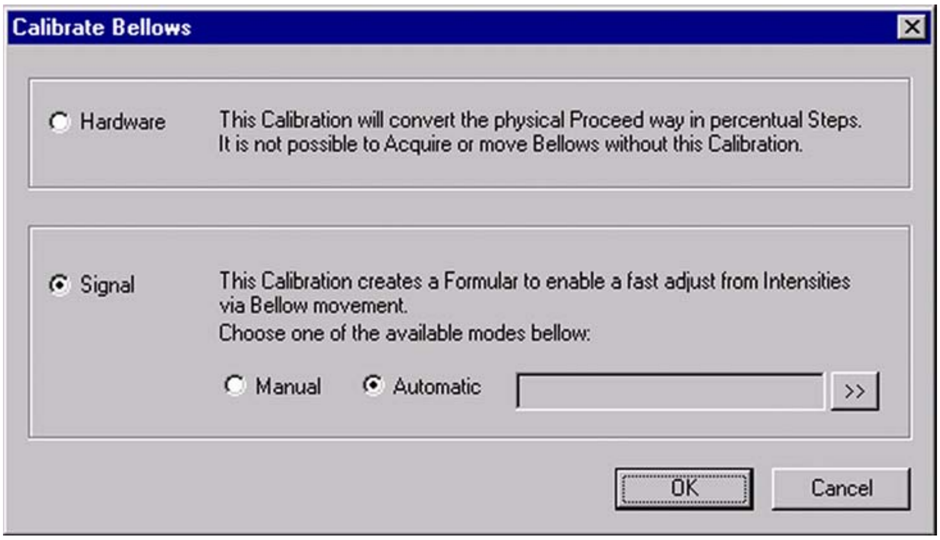
NOTE: ***Signal Calibration requires a signal intensity of at least 3 V!***

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

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

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

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



Calibrate Bellows

○ Hardware — This Calibration will convert the physical Proceed way in percentual Steps. It is not possible to Acquire or move Bellows without this Calibration.

● Signal — This Calibration creates a Formular to enable a fast adjust from Intensities via Bellow movement. Choose one of the available modes bellow:

○ Manual ● Automatic

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

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

ISODAT NT

OPERATING MANUAL

4

DUAL INLET MEASUREMENTS

Issue 06/2002

Thermo Finnigan

4.1 GENERAL REMARKS

4.1.1 PROCEDURE BEFORE STARTING ANY MEASUREMENTS

NOTE: ***Before starting any acquisition make sure, the following procedure has been performed:***

1. Electronic offset has been measured and saved.
2. All peripherals have been configured.
3. The Mass Calibration for all gases to be measured has been performed.
4. In case of a Dual Inlet application, the Bellow Calibration has been performed.
5. In case of hydrogen collectors, the H3 factor for Continuous Flow and Dual Inlet application has been determined.
6. In case of Elemental Analyzer application for C/N or H/O analysis, the Jump Calibration has been performed and saved.
7. In case of a Liquid Autosampler, the COM port and the other parameters are set.

NOTE: ***In case of any measurement, three steps must always be performed:***

1. Define a (Hardware-) ***Configuration***
2. Define a ***Method***
3. Define a ***Sequence***

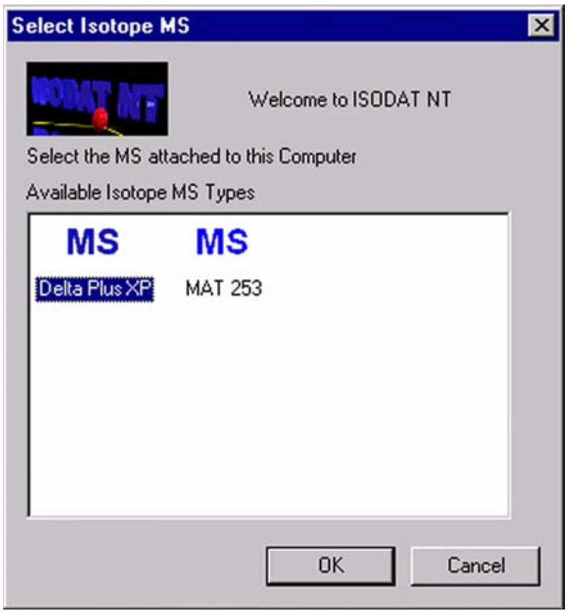
These three basic steps will be described chronologically for several peripherals in this chapter.

4.1.2 IF NO CONFIGURATION IS AVAILABLE

Configurator

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

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



Welcome to ISODAT NT

Select the MS attached to this Computer

Available Isotope MS Types

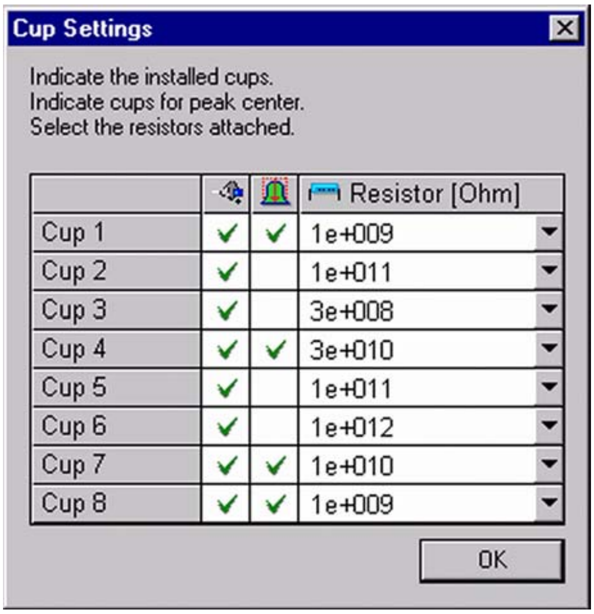
Delta Plus XP

MAT 253

- ➢ Your type of IRMS is required.

  Select your Mass Spectrometer (e.g. “Delta Plus XL”).

- ➢ Press ***OK***.



Indicate the installed cups.
Indicate cups for peak center.
Select the resistors attached.

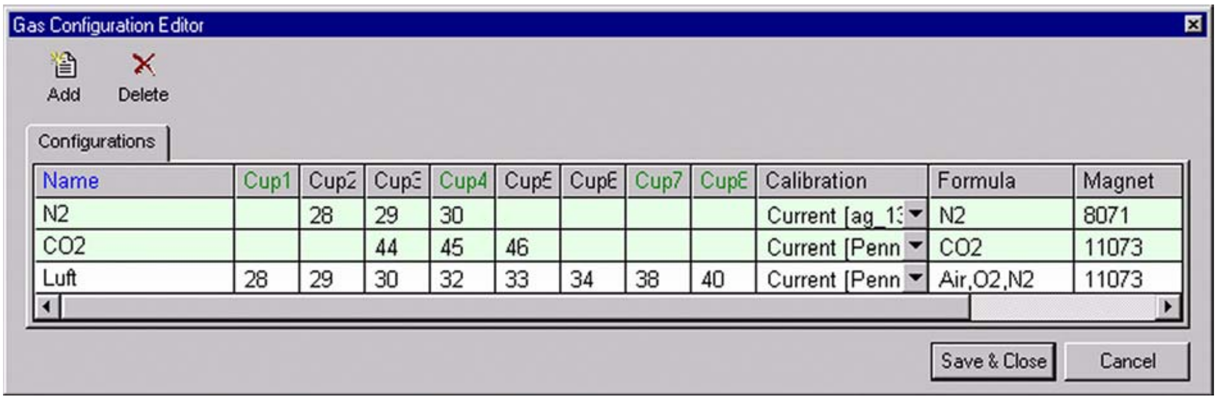
			Resistor [Ohm]
Cup 1	✓	✓	1e+009
Cup 2	✓		1e+011
Cup 3	✓		3e+008
Cup 4	✓	✓	3e+010
Cup 5	✓		1e+011
Cup 6	✓		1e+012
Cup 7	✓	✓	1e+010
Cup 8	✓	✓	1e+009

- ➢ Check, whether the correct cups are installed.

- ➢ Check, whether the correct cups are available for Peak Center.

- ➢ Check, whether the resistor values are correct.

- ➢ Confirm with ***OK***.



Name	Cup1	Cup2	Cup3	Cup4	Cup5	Cup6	Cup7	Cup8	Calibration	Formula	Magnet
N2		28	29	30					Current [ag_1	N2	8071
CO2			44	45	46				Current [Penn	CO2	11073
Luft	28	29	30	32	33	34	38	40	Current [Penn	Air,O2,N2	11073

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

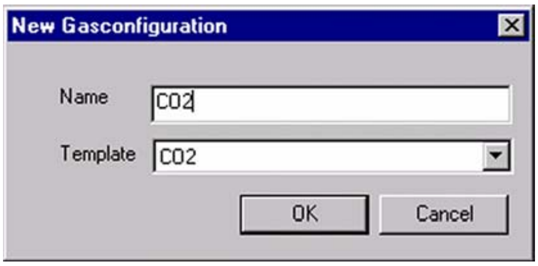
You can now proceed creating a ***new Configuration*** using the ***Configurator***.

4.1.3 HOW TO CREATE A NEW GAS CONFIGURATION

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

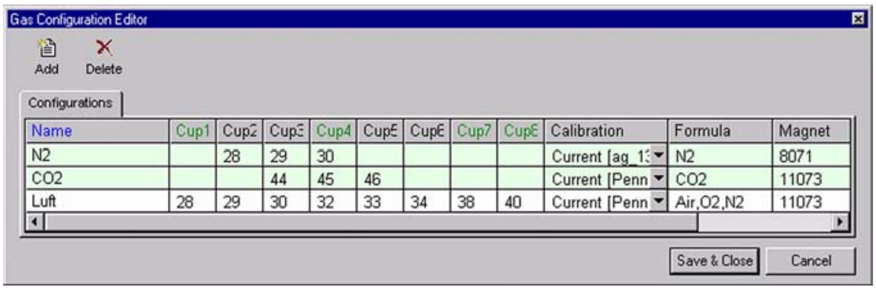
For a measurement, a new Gas Configuration has to be created for the masses 44 ($^{12}C^{16}O^{16}O$), 45 ($^{13}C^{16}O^{16}O$) and 46 ($^{12}C^{16}O^{18}O$).

- ➢ To create a new Gas Configuration, open the ***Dual Inlet*** module.
- ➢ Then open the ***Gas Configuration Editor***.
- ➢ ***Add*** a Gas Configuration.



Name: CO2
Template: CO2

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



Name	Cup1	Cup2	Cup3	Cup4	Cup5	Cup6	Cup7	Cup8	Calibration	Formula	Magnet
N2		28	29	30					Current [ag_1	N2	8071
CO2			44	45	46				Current [Penn	CO2	11073
Luft	28	29	30	32	33	34	38	40	Current [Penn	Air,O2,N2	11073

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

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

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

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

4.1.4 HOW TO CREATE A NEW METHOD

- ➢ Open the ***Dual Inlet*** module.

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

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

- ➢ Press the ***New*** button.

- ➢ Press the ***Method*** Icon.

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

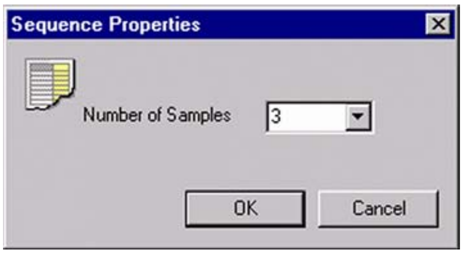
4.1.5 HOW TO CREATE A NEW SEQUENCE

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

- ➢ Press the ***New*** button.

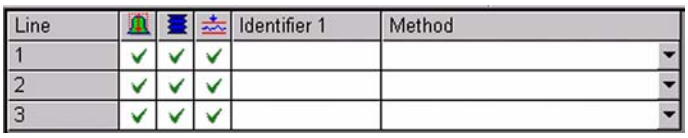
- ➢ Press the ***Sequence*** Icon.

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



Number of Samples: 3

- ➢ Edit the Sequence list.



Line				Identifier 1	Method
1	✓	✓	✓		
2	✓	✓	✓		
3	✓	✓	✓		

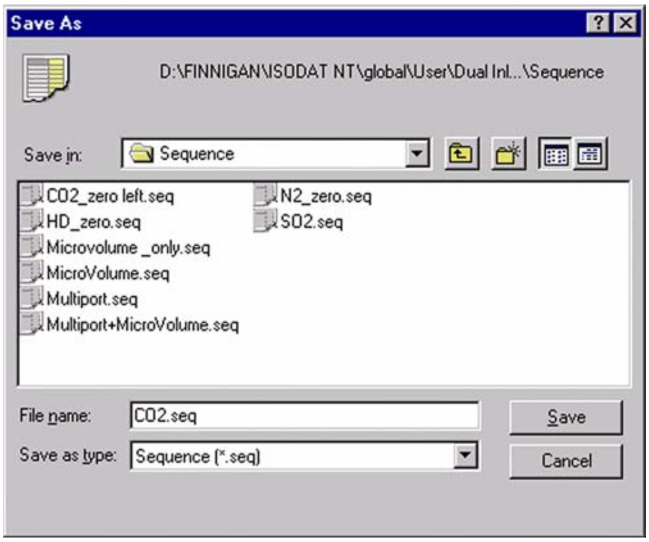
Peak Center	Enable ✓	to perform a Peak Center prior to measurement.
Press Adjust	Enable ✓	to perform a pressure adjust.
Background	Enable ✓	to perform a background measurement.

Identifier 1	Edit text to identify sample.
Method	Select IRMS Method (for each sample, e.g. “CO2.met”).

- ➢ Press the ***Start*** button.

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

- ➢ Press ***Save***.



D:\FINNIGAN\ISODAT NT\global\User\Dual Inl...\Sequence

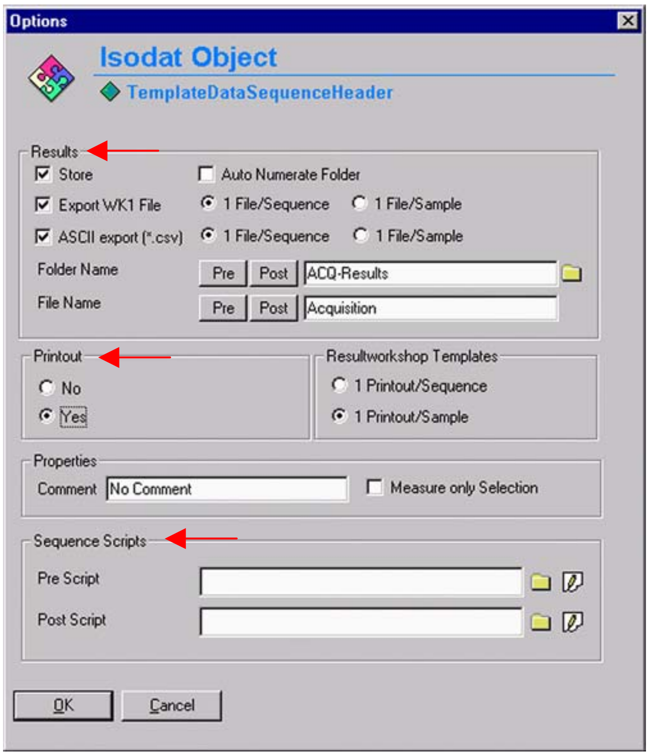
Save in: Sequence

CO2_zero left.seq
HD_zero.seq
Microvolume _only.seq
MicroVolume.seq
Multiport.seq
Multiport+MicroVolume.seq
N2_zero.seq
SO2.seq

File name: CO2.seq

Save as type: Sequence (*.seq)

- Define parameters for:
  - Results Export
  - Printout
  - Sequence Scripts
- Press ***OK*** to start Sequence Acquisition.



Options

Isodat Object

TemplateDataSequenceHeader

Results

☑ Store ☐ Auto Numerate Folder

☑ Export WK1 File ● 1 File/Sequence ○ 1 File/Sample

☑ ASCII export (*.csv) ● 1 File/Sequence ○ 1 File/Sample

Folder Name: ACQ-Results

File Name: Acquisition

Printout

○ No

● Yes

Resultworkshop Templates

○ 1 Printout/Sequence

● 1 Printout/Sample

Properties

Comment: No Comment

☐ Measure only Selection

Sequence Scripts

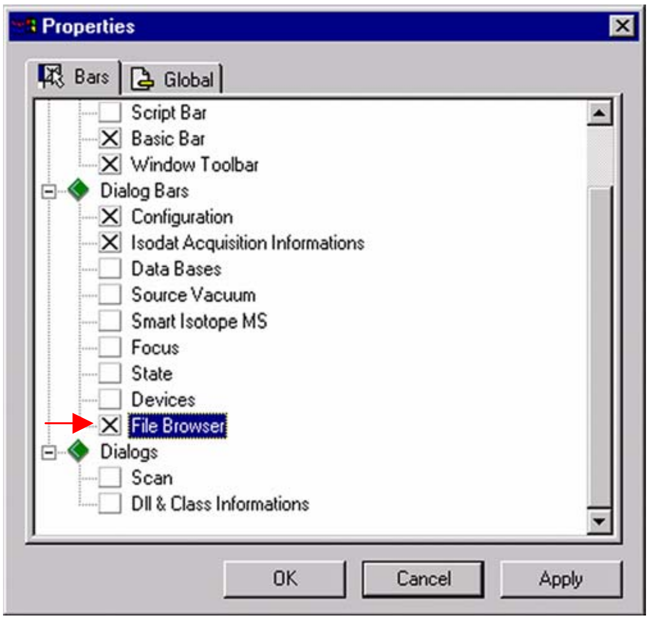
Pre Script:

Post Script:

4.2 ZERO ENRICHMENT (STANDARD ON / OFF TEST)

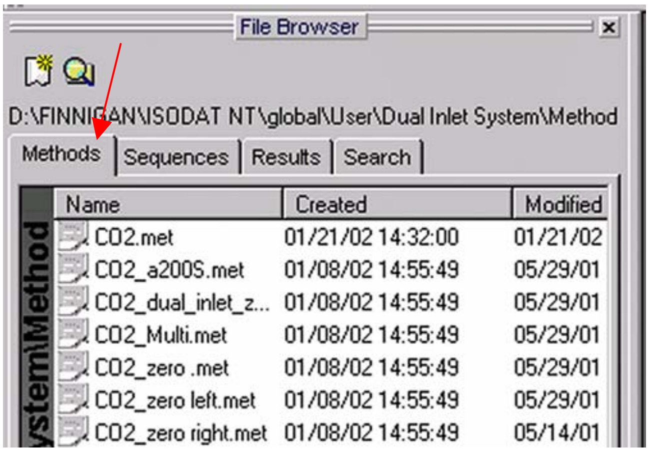
We assume, that the user already has working experience with the Dual Inlet System, the Dual Inlet devices and with IRMS. It is recommended to perform a simple check in order to test the analytical condition of the complete system before measuring any samples. Proceed as follows for zero enrichment:

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



☐ Script Bar
☒ Basic Bar
☒ Window Toolbar
Dialog Bars
☒ Configuration
☒ Isodat Acquisition Informations
☐ Data Bases
☐ Source Vacuum
☐ Smart Isotope MS
☐ Focus
☐ State
☐ Devices
☒ File Browser
Dialogs
☐ Scan
☐ Dll & Class Informations

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

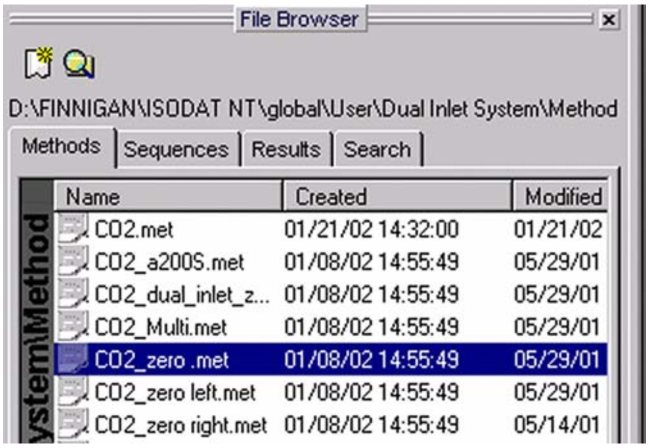


D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01

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

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



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01

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

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

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

The three Methods only differ from the Script.

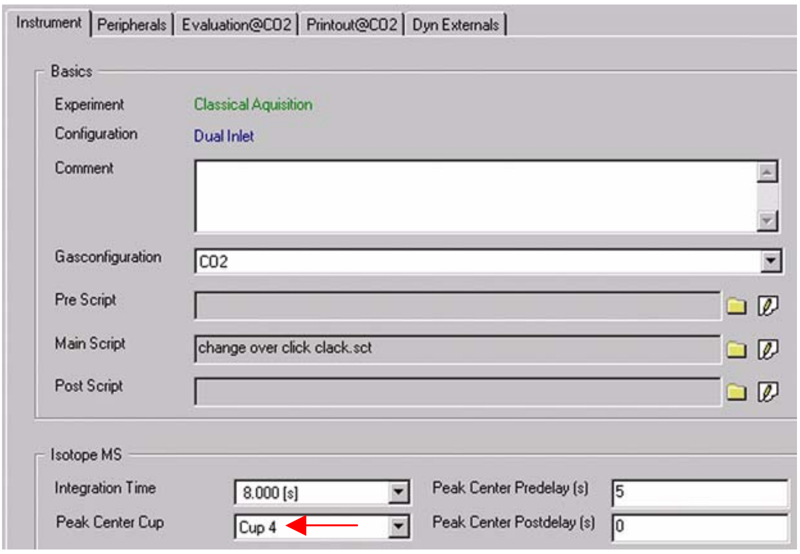
- If the Standards in the Method to be opened are older than the actual ones in the Standard Database, or if the user has changed the Method, the warning beside appears.

Standard values different to Standard Database!

Actualize now to new values!

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

- The ***Instrument tab*** of the Method “CO2_zero.met” appears.
- For details see Chapter 4.3.2.

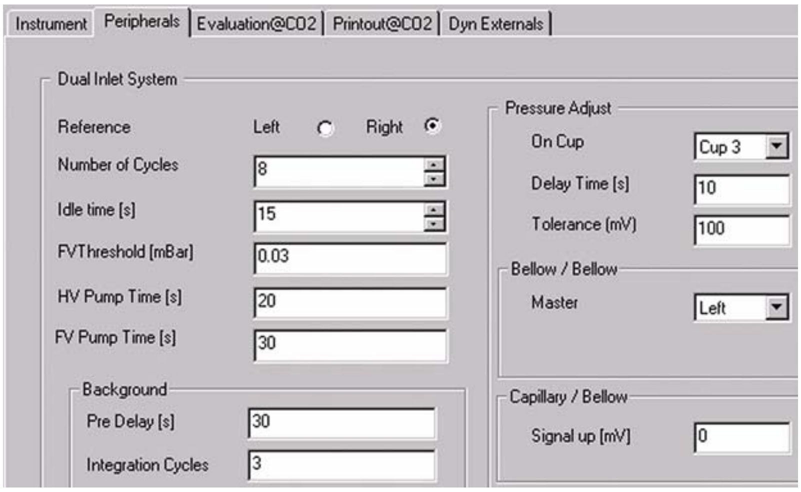


Experiment: Classical Aquisition
Configuration: Dual Inlet
Comment:
Gasconfiguration: CO2
Pre Script:
Main Script: change over click clack.sct
Post Script:
Integration Time: 8.000 [s]
Peak Center Cup: Cup 4
Peak Center Predelay [s]: 5
Peak Center Postdelay [s]: 0

NOTE: ***Ensure, that the proper peak center cup is selected.***

If necessary, correct the default cup (e. g. Cup 4)!

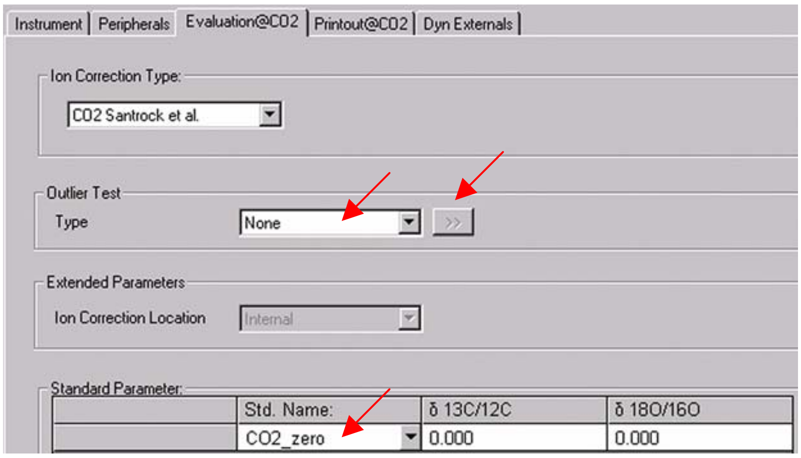
- At ***Peripherals tab***, see Chapter 4.3.2 for details.



Reference: Left ○ Right ●
Number of Cycles: 8
Idle time [s]: 15
FVThreshold [mBar]: 0.03
HV Pump Time [s]: 20
FV Pump Time [s]: 30
Pre Delay [s]: 30
Integration Cycles: 3
On Cup: Cup 3
Delay Time [s]: 10
Tolerance (mV): 100
Master: Left
Signal up [mV]: 0

- At ***Evaluation CO2 tab***, see Chapter 4.3.2 for details.
- Select ***None*** or ***Sigma*** as Outlier test.

  In case of ***Sigma***, specify the k-fold of the standard deviation using >> .



Ion Correction Type: CO2 Santrock et al.

Outlier Test
Type: None

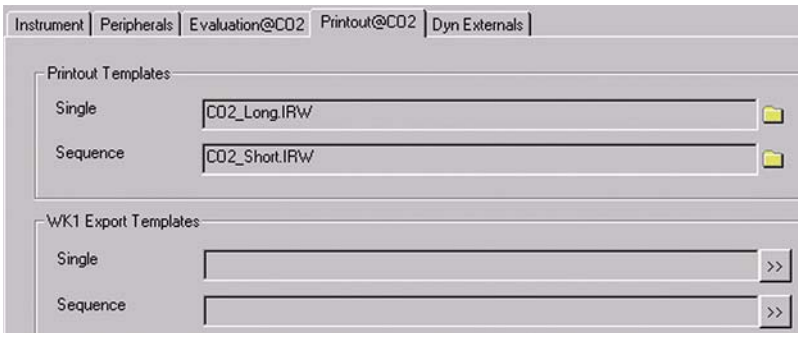
Extended Parameters
Ion Correction Location: Internal

Standard Parameter:

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

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

- At ***Printout tab***, see Chapter 4.3.2 for details.



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

WK1 Export Templates
Single:
Sequence:

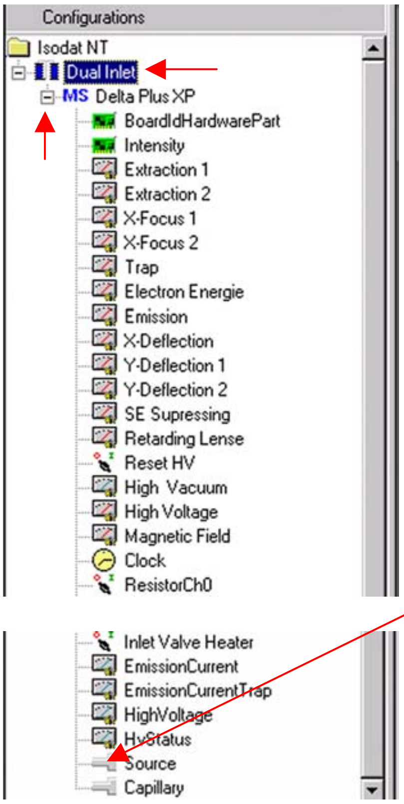
4.3 SIMPLE DUAL INLET MEASUREMENT

4.3.1 DEFINING A CONFIGURATION

Configurator

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

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



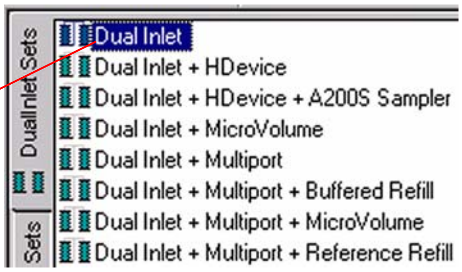
Configurations
Isodat NT
Dual Inlet
MS Delta Plus XP
BoardIdHardwarePart
Intensity
Extraction 1
Extraction 2
X-Focus 1
X-Focus 2
Trap
Electron Energie
Emission
X-Deflection
Y-Deflection 1
Y-Deflection 2
SE Supressing
Retarding Lense
Reset HV
High Vacuum
High Voltage
Magnetic Field
Clock
ResistorCh0
Inlet Valve Heater
EmissionCurrent
EmissionCurrentTrap
HighVoltage
HvStatus
Source
Capillary

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

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

- ➢ On the right pane,

  select ***Dual Inlet***.

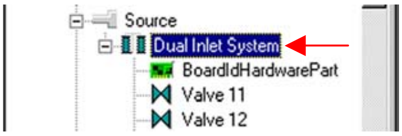


Dual Inlet
Dual Inlet + HDevice
Dual Inlet + HDevice + A200S Sampler
Dual Inlet + MicroVolume
Dual Inlet + Multiport
Dual Inlet + Multiport + Buffered Refill
Dual Inlet + Multiport + MicroVolume
Dual Inlet + Multiport + Reference Refill

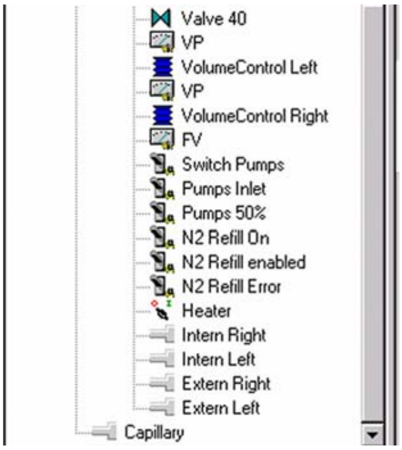
- ➢ Drag it to the Source port of the new Configuration.

  A ⊞ sign appears:

HvStatus
Source
Capillary



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

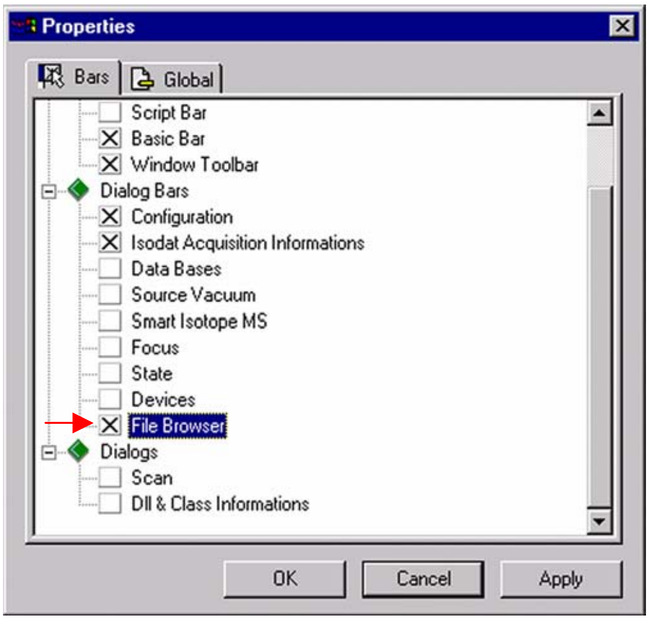


- Close the Configurator window.

All settings will be saved automatically.

4.3.2 USING A PREDEFINED METHOD

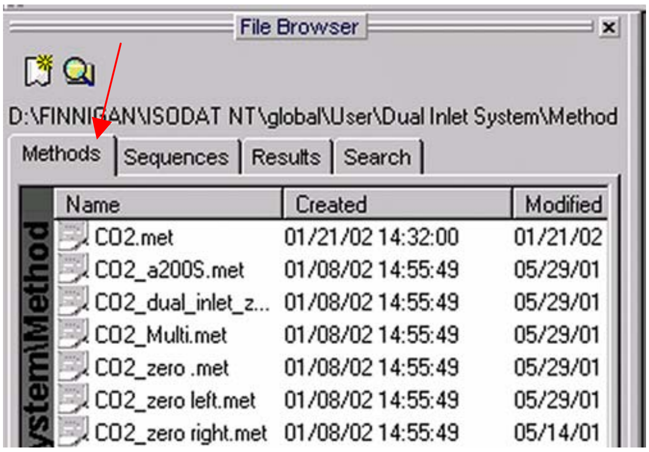
- If the File Browser cannot be seen, press the ***Options*** button.



- ☐ Script Bar
- ☒ Basic Bar
- ☒ Window Toolbar
- Dialog Bars
  - ☒ Configuration
  - ☒ Isodat Acquisition Informations
  - ☐ Data Bases
  - ☐ Source Vacuum
  - ☐ Smart Isotope MS
  - ☐ Focus
  - ☐ State
  - ☐ Devices
  - ☒ File Browser
- Dialogs
  - ☐ Scan
  - ☐ Dll & Class Informations

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

- Click ***OK***.

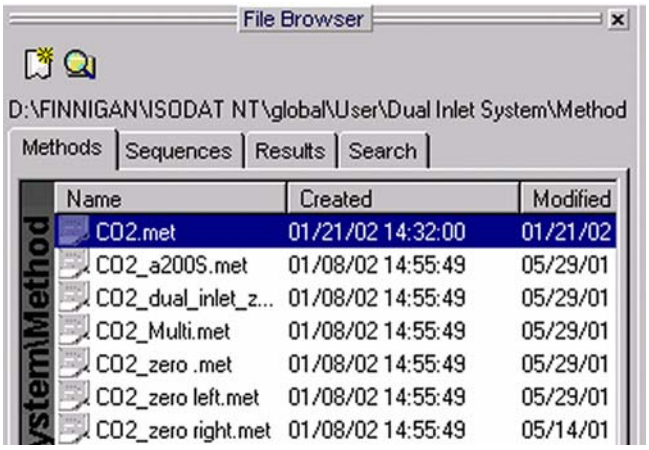


D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01

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

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



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01

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

Standard values different to Standard Database!

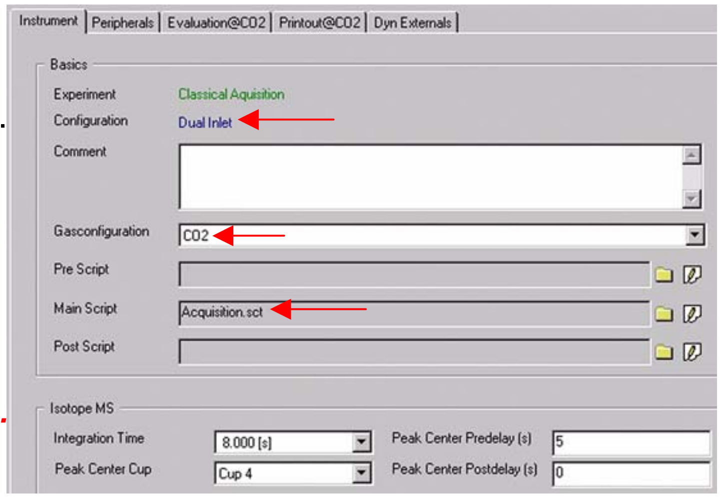
Actualize now to new values!

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

Instrument

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

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



Experiment: Classical Aquisition

Configuration: Dual Inlet

Comment:

Gasconfiguration: CO2

Pre Script:

Main Script: Acquisition.sct

Post Script:

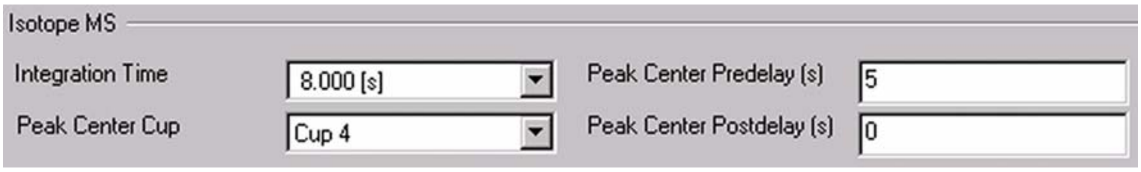
Integration Time: 8.000 [s]

Peak Center Cup: Cup 4

Peak Center Predelay (s): 5

Peak Center Postdelay (s): 0

Isotope MS

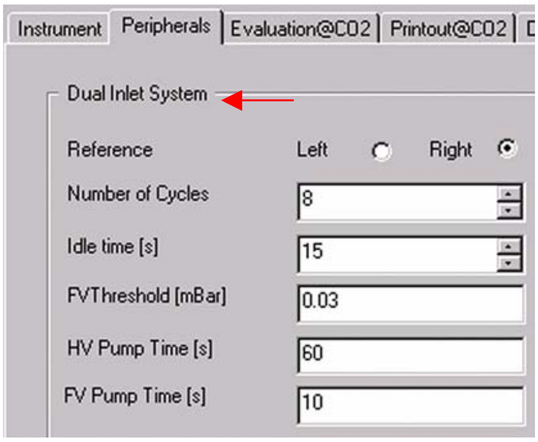


Integration Time: 8.000 [s]
Peak Center Cup: Cup 4
Peak Center Predelay [s]: 5
Peak Center Postdelay [s]: 0

- Select the ***Peak Center Cup***, e.g. **Cup 4** for a universal triple collector on a Delta^{Plus} XP (narrow cup for m/z 45).
- ***Peak Center Predelay*** is the time the system waits between activation of the reference gas and start of the peak center cycle (e.g. **5** s).
- ***Peak Center Postdelay*** is the time the system waits between the end of the peak center cycle and the start of the data acquisition (e.g. **0** s).
- ***Integration time*** is the time integrated to form a data point triplet (e.g. **8** s). It is needed to measure each individual ion intensity of the masses 44, 45 and 46.

Peripherals

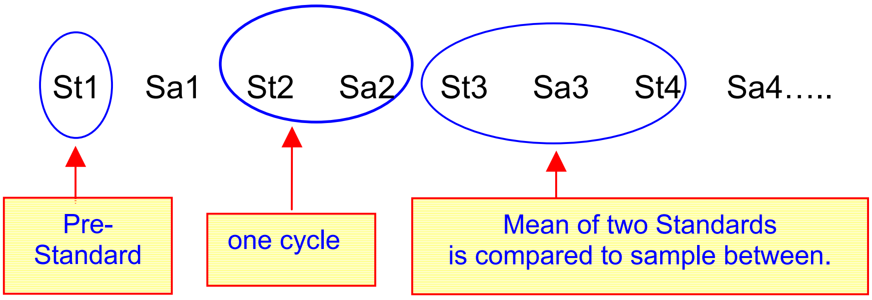
Dual Inlet System



Reference: Left ○ Right ●
Number of Cycles: 8
Idle time [s]: 15
FVThreshold [mBar]: 0.03
HV Pump Time [s]: 60
FV Pump Time [s]: 10

- ***Reference*** denotes, which bellow will contain the standard gas (default: right bellow). The other bellow will contain the sample.
- ***Number of cycles*** indicates, how often a sample will be measured successively (e.g. 8 cycles means, that eight times the standard and eight times the sample is measured).

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



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

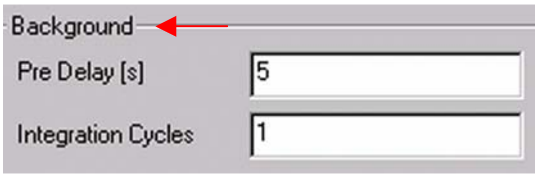
- ***Idle time*** is the waiting time after having switched the changeover from sample to standard side (or vice versa) before the ion intensities of the masses 44, 45 and 46 will be integrated. The gas needs a certain time to flow away, before the measurement can be started.

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

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

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

Background

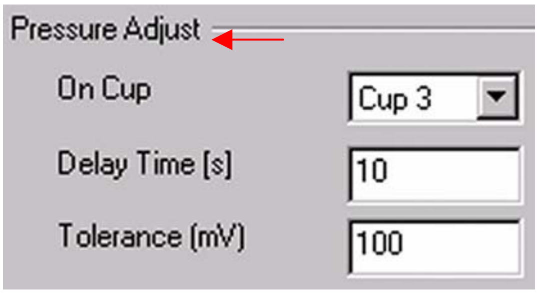

Background
Pre Delay [s] 5
Integration Cycles 1

- ***Background Predelay*** is the waiting time after closing the changeover valves until the background measurement begins.

- ***Background Integration Cycles*** denotes, how long the background measurement will be performed (default: 1 cycle). k integration cycles specify that background measurement spans the k-fold integration time.

Pressure Adjust

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


Pressure Adjust
On Cup: Cup 3
Delay Time [s] 10
Tolerance (mV) 100

- ***on cup***: cup on which the pressure adjust will be performed. Normally, it is the cup, where mass 44 is measured.

- ***Delay time*** is the waiting time after changing from sample to standard gas (or vice versa) before matching the standard bellow to sample ion intensity.

- ***Tolerance*** denotes the precision of the pressure adjust. It is the maximally acceptable deviation of the ion intensities between the sample signal and the standard signal after matching. If the deviation is less than or equals the value (e.g. 100 mV, this means ± 50 mV), the press adjust is finished successfully.

Bellow/Bellow

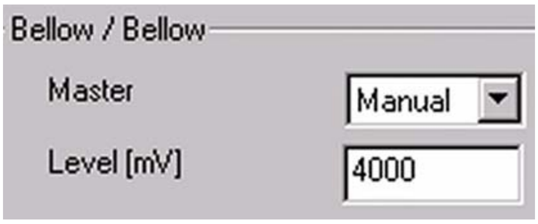

Bellow / Bellow

Master: Left

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

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

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


Bellow / Bellow

Master: Manual

Level [mV]: 4000

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

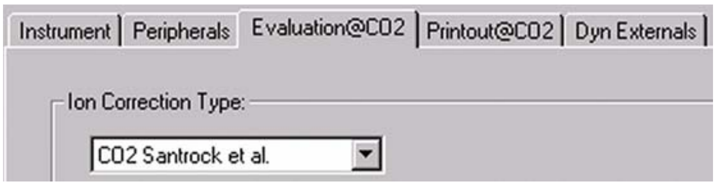
Capillary/Bellow


Capillary / Bellow

Signal up [mV]: 0

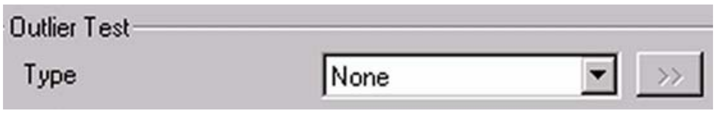
- ***Signal up***: match the ion intensity of the standard gas less than the given value before closing valve 25 and starting data acquisition.

Evaluation



Ion Correction Type: CO2 Santrock et al.

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

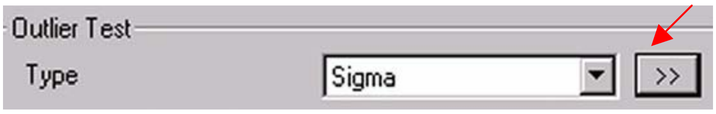


Outlier Test
Type: None

- Choose the type of ***Outlier Test***. (Rejection criterion for possible outliers: “None” or “Sigma”).

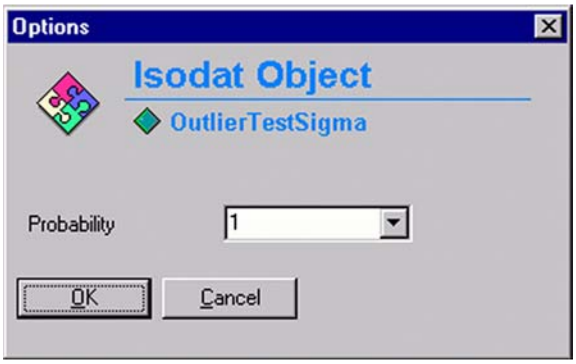
- If you select ***None***, only mean and standard deviation of the obtained delta values will be calculated. Outliers will not be identified.

- If you select ***Sigma***, those delta values beyond k times of the standard deviation will be identified and rejected.



Outlier Test
Type: Sigma

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



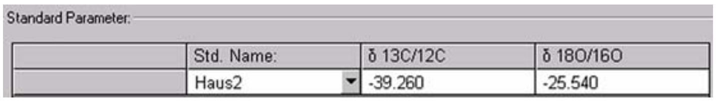
Probability: 1

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

Extended Parameters

Ion Correction Location: Internal

- **_Extended Parameters_**: this function has not been implemented yet. Soon, you can choose an editable Script here, which contains the Ion Correction (i.e. type of Ion Correction and its location).



Standard Parameter:

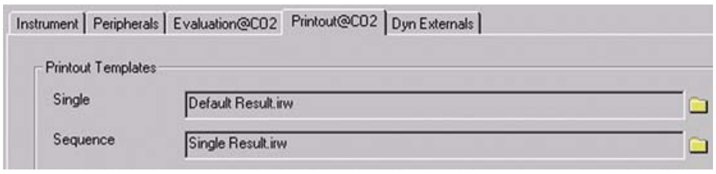
	Std. Name:	δ 13C/12C	δ 18O/16O
	Haus2	-39.260	-25.540

- **_Std. Name_**: Select a Standard Name or edit the related delta values (user defined).

The standards are predefined in the standard table. These values are isotope ratios of carbon and oxygen of your standard gas.

Printout

Printout Templates



Printout Templates

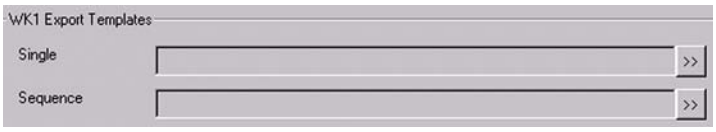
Single: Default Result.irw

Sequence: Single Result.irw

- **_Single Print_** selects a print template from the Result Workshop for an individual printout per sample.
- **_Sequence Print_** selects a print template from the Result Workshop for a reduced print per sample within a sequence summary.

WK1 Export Templates

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



- **_Single Print_** selects an Excel-Export Template from the Result Workshop for an individual printout per sample.
- **_Sequence Print_** selects an Excel-Export Template from the Result Workshop for a reduced print per sample within a sequence summary.

Dyn. Externals: will follow soon.

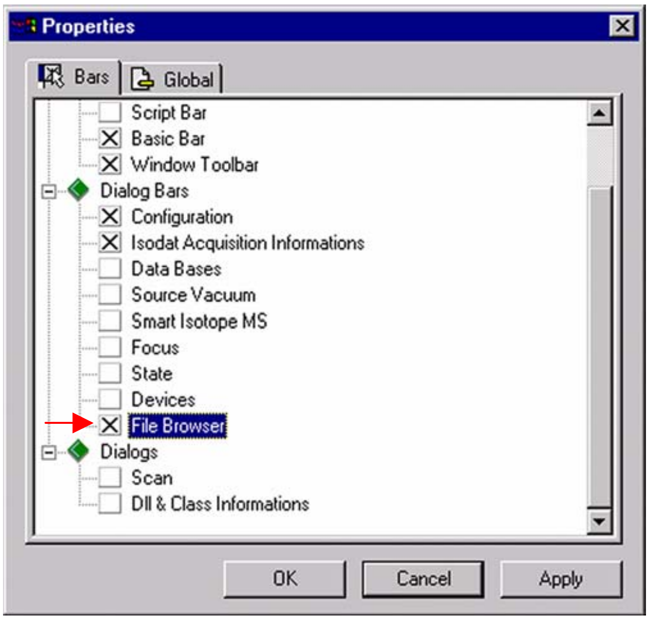
The **_Method_** for measurements using a simple Dual Inlet has now been established. As next step, either use a **_predefined Sequence_** (refer to Chapter 4.3.3) or create a **_new Sequence_** (refer to Chapter 4.1.5).

4.3.3 USING A PREDEFINED SEQUENCE

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

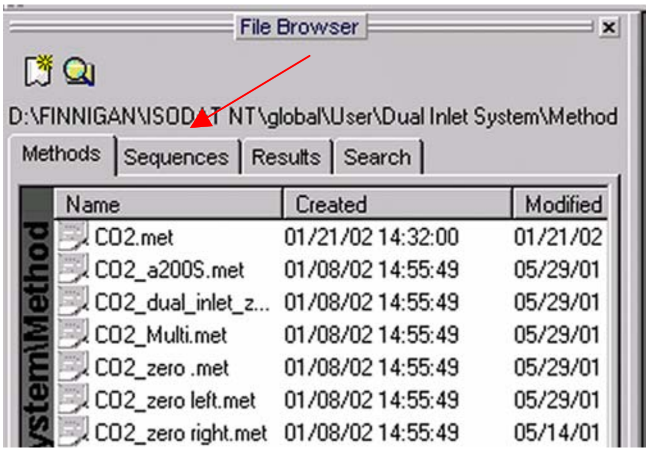
Options

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



☐ Script Bar
☒ Basic Bar
☒ Window Toolbar
Dialog Bars
☒ Configuration
☒ Isodat Acquisition Informations
☐ Data Bases
☐ Source Vacuum
☐ Smart Isotope MS
☐ Focus
☐ State
☐ Devices
☒ File Browser
Dialogs
☐ Scan
☐ Dll & Class Informations

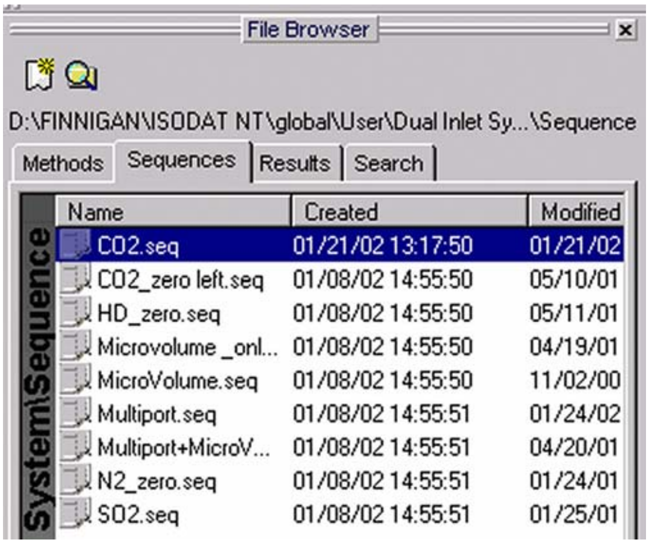
- On the File Browser, select the ***Sequences tab*** (default: Methods tab).



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01

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



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet Sy...\Sequence

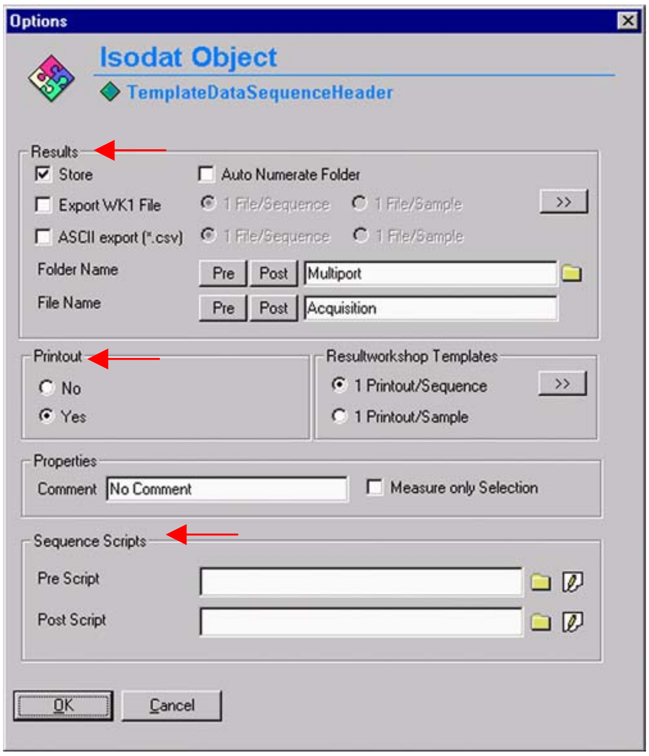
Name	Created	Modified
CO2.seq	01/21/02 13:17:50	01/21/02
CO2_zero left.seq	01/08/02 14:55:50	05/10/01
HD_zero.seq	01/08/02 14:55:50	05/11/01
Microvolume _onl...	01/08/02 14:55:50	04/19/01
MicroVolume.seq	01/08/02 14:55:50	11/02/00
Multiport.seq	01/08/02 14:55:51	01/24/02
Multiport+MicroV...	01/08/02 14:55:51	04/20/01
N2_zero.seq	01/08/02 14:55:51	01/24/01
SO2.seq	01/08/02 14:55:51	01/25/01

- In the "Method" column, the Method chosen in Chapter 4.3.2 occurs as default ("CO2.met").

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

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

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



Results

- ☑ Store
- ☐ Auto Numerate Folder
- ☐ Export WK1 File
- ☐ ASCII export (*.csv)

Folder Name: Multiport

File Name: Acquisition

Printout

- ○ No
- ● Yes

Resultworkshop Templates

- ● 1 Printout/Sequence
- ○ 1 Printout/Sample

Properties

Comment: No Comment

- ☐ Measure only Selection

Sequence Scripts

Pre Script:

Post Script:

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

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

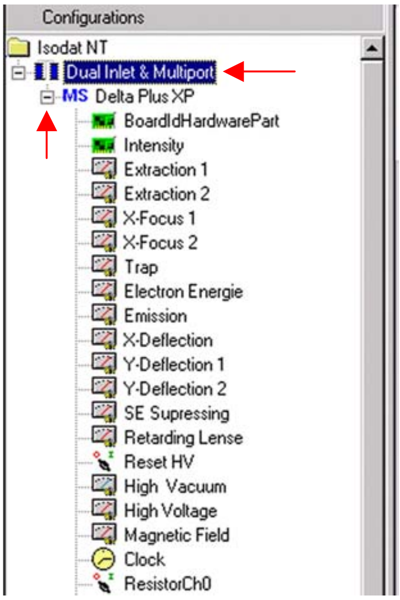
4.4 DUAL INLET MEASUREMENT INCLUDING MULTIPORT

4.4.1 DEFINING A CONFIGURATION

Configurator

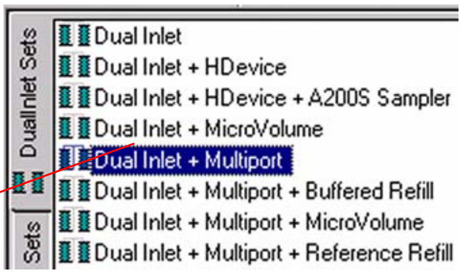
- ➢ Before operating, a ***Configuration*** containing the Dual Inlet System and the Multiport must be created in the ***Configurator*** as follows.

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



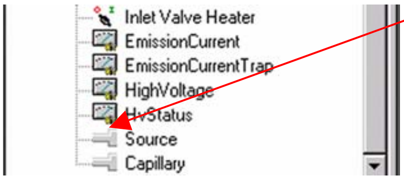
- ➢ Give it a significant name, e.g. ***“Dual Inlet & Multiport”***.

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



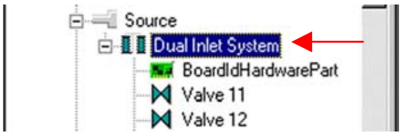
- ➢ On the right pane, select

  Dual Inlet + Multiport.

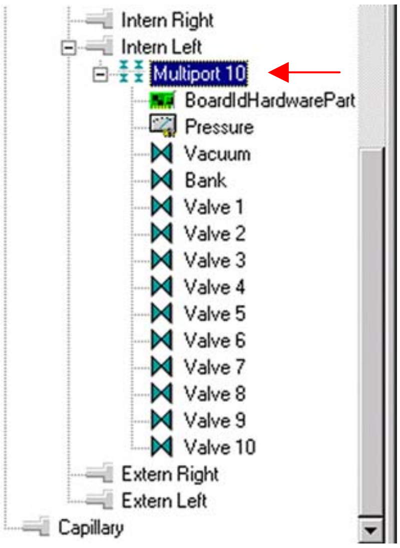


- ➢ Drag it to the Source port of the new Configuration.

  A ⊞ sign appears:



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



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

- ➢ Close the Configurator window.

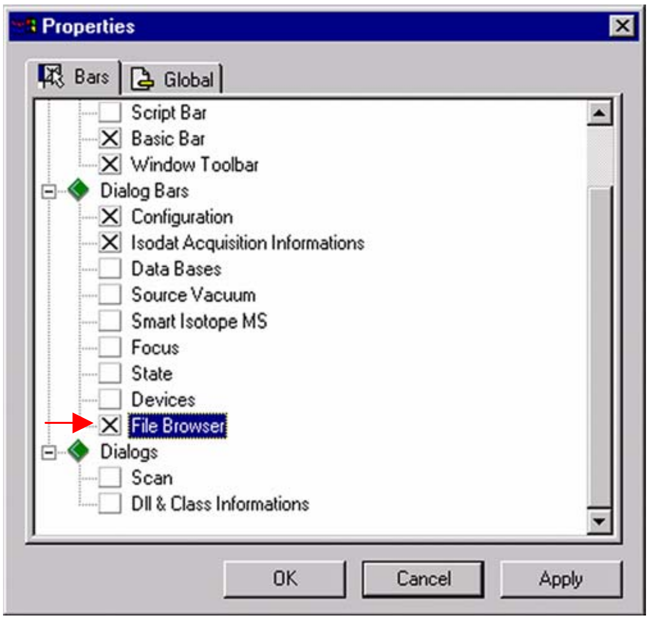
  All settings will be saved automatically.

4.4.2 USING A PREDEFINED METHOD

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

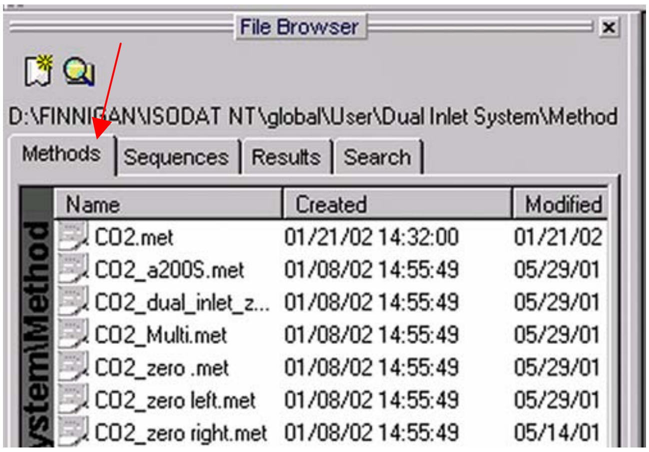
- Activate the ***File Browser*** check box.

- Click ***OK***.



☐ Script Bar
☒ Basic Bar
☒ Window Toolbar
Dialog Bars
  ☒ Configuration
  ☒ Isodat Acquisition Informations
  ☐ Data Bases
  ☐ Source Vacuum
  ☐ Smart Isotope MS
  ☐ Focus
  ☐ State
  ☐ Devices
  ☒ File Browser
Dialogs
  ☐ Scan
  ☐ Dll & Class Informations

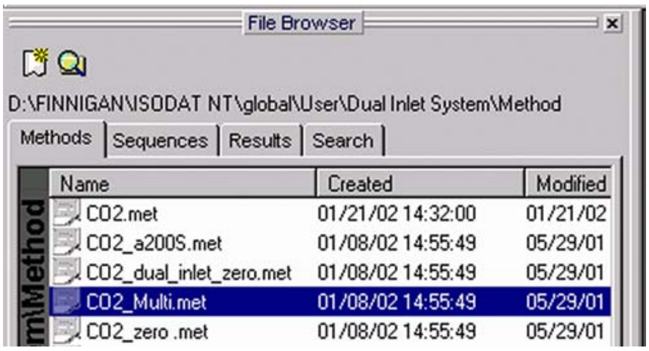
- On the File Browser, select the ***Methods tab*** (default).



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01

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



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_zero.met	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01

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

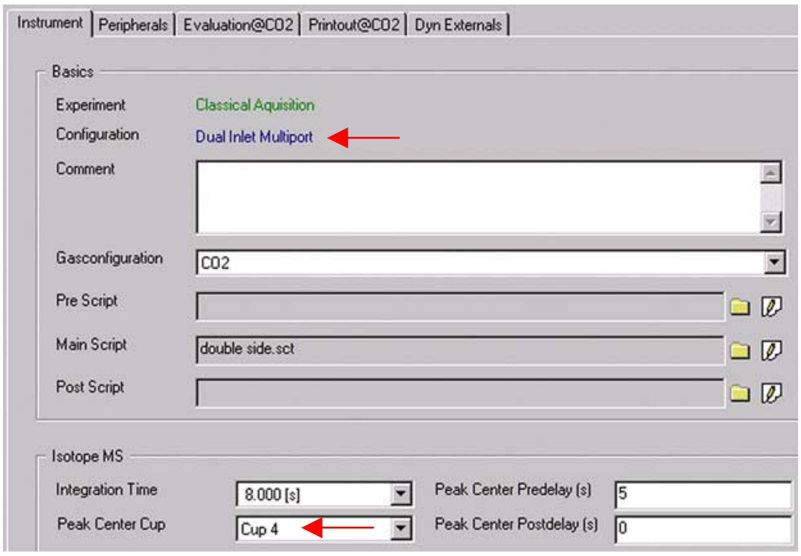
Standard values different to Standard Database!

Actualize now to new values!

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

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

  For details see Chapter 4.3.2.

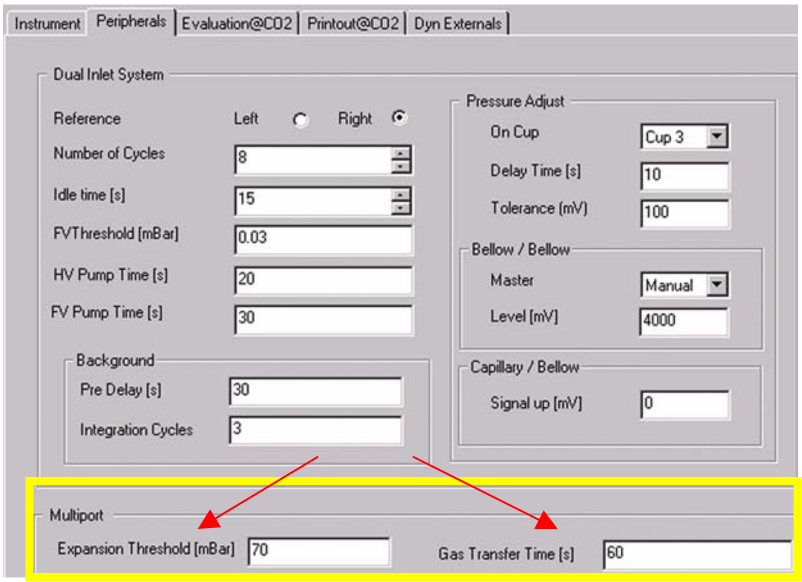


Experiment: Classical Aquisition
Configuration: Dual Inlet Multiport
Comment:
Gasconfiguration: CO2
Pre Script:
Main Script: double side.sct
Post Script:

Integration Time: 8.000 [s]
Peak Center Cup: Cup 4
Peak Center Predelay [s]: 5
Peak Center Postdelay [s]: 0

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

- See Chapter 4.3.2 for details at ***Peripherals tab***.



Dual Inlet System

Reference: Left ○ Right ●

Number of Cycles: 8

Idle time [s]: 15

FVThreshold [mBar]: 0.03

HV Pump Time [s]: 20

FV Pump Time [s]: 30

Background

Pre Delay [s]: 30

Integration Cycles: 3

Pressure Adjust

On Cup: Cup 3

Delay Time [s]: 10

Tolerance (mV): 100

Bellow / Bellow

Master: Manual

Level [mV]: 4000

Capillary / Bellow

Signal up [mV]: 0

Multiport

Expansion Threshold [mBar]: 70

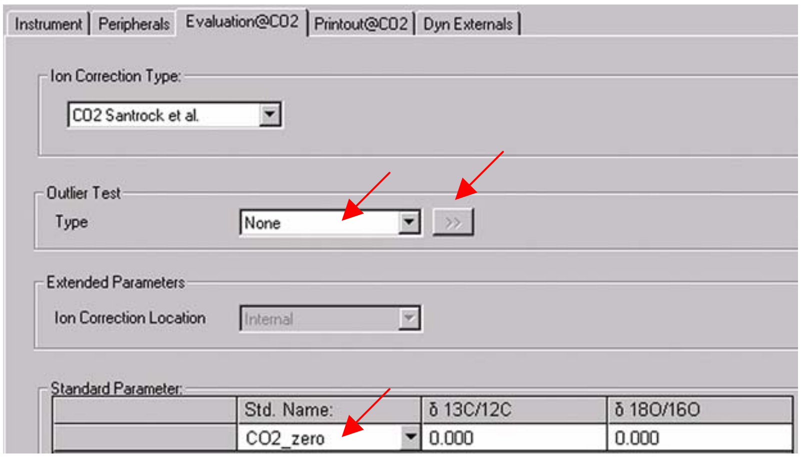
Gas Transfer Time [s]: 60

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

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

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

- At ***Evaluation CO2 tab***, see Chapter 4.3.2 for details.
- Select “None” or “Sigma” as Outlier test. In case of “Sigma”, specify the k-fold of the standard deviation using >>.



Ion Correction Type: CO2 Santrock et al.

Outlier Test
Type: None >>

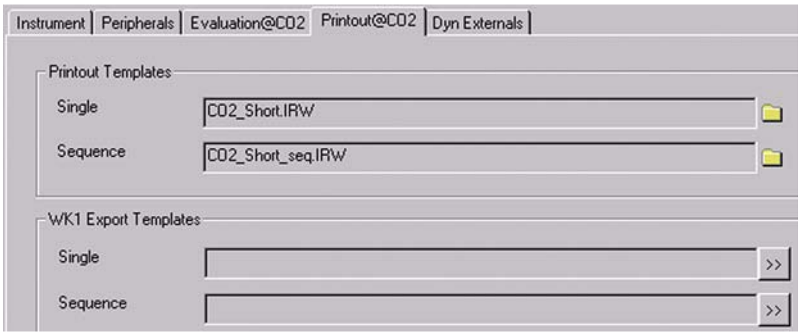
Extended Parameters
Ion Correction Location: Internal

Standard Parameter:

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

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

- At ***Printout tab***, see Chapter 4.3.2 for details.



Printout Templates
Single: CO2_Short.IRW
Sequence: CO2_Short_seq.IRW

WK1 Export Templates
Single:
Sequence:

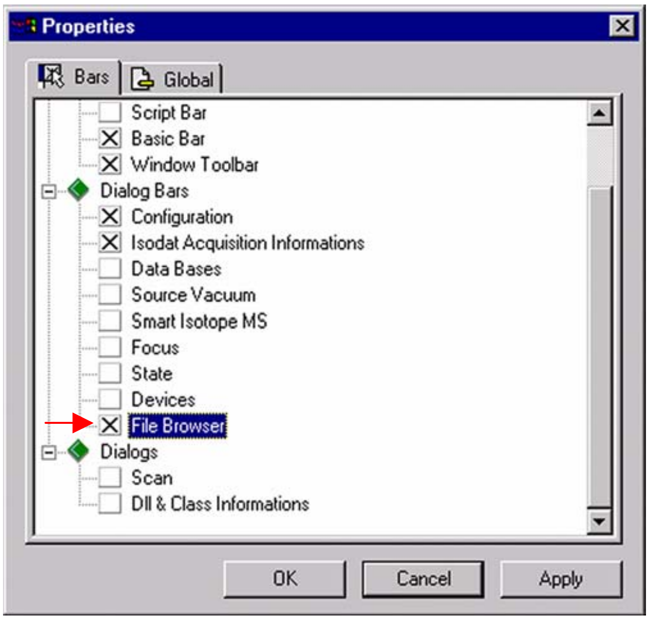
The ***Method*** for measurements using Dual Inlet in combination with a Multiport has now been established. As next step, either use a ***predefined Sequence*** (refer to Chapter 4.4.3) or create a ***new Sequence*** (refer to Chapter 4.1.5).

4.4.3 USING A PREDEFINED SEQUENCE

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

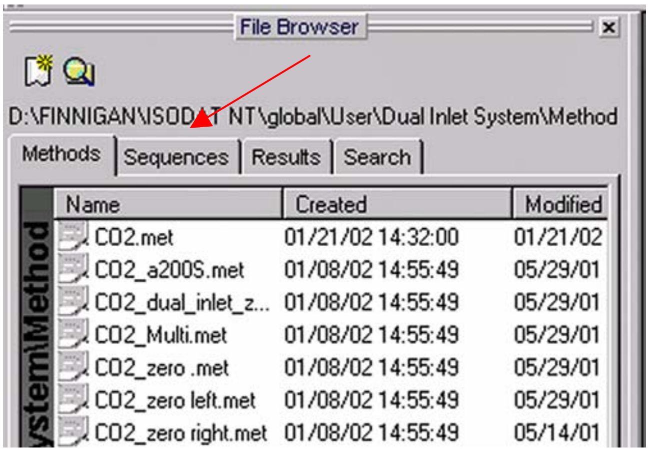
- ➢ Activate the ***File Browser*** check box.

- ➢ Click ***OK***.



- ☐ Script Bar
- ☒ Basic Bar
- ☒ Window Toolbar
- Dialog Bars
  - ☒ Configuration
  - ☒ Isodat Acquisition Informations
  - ☐ Data Bases
  - ☐ Source Vacuum
  - ☐ Smart Isotope MS
  - ☐ Focus
  - ☐ State
  - ☐ Devices
  - ☒ File Browser
- Dialogs
  - ☐ Scan
  - ☐ Dll & Class Informations

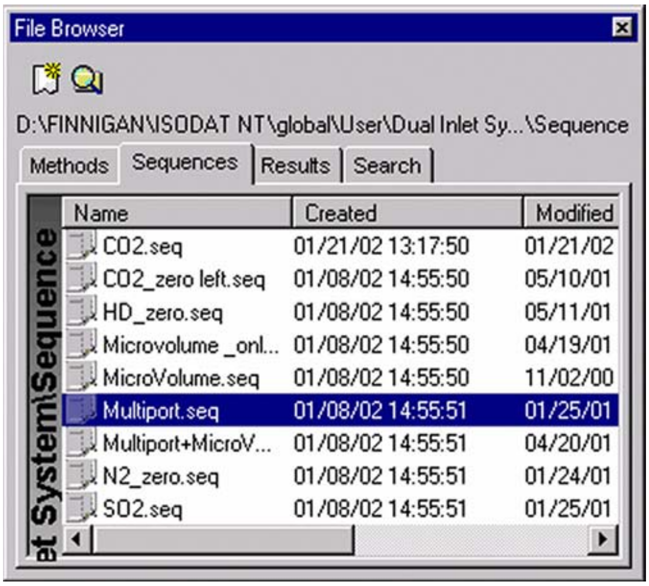
- ➢ On the File Browser, select the ***Sequences tab*** (default: Methods tab).



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01

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

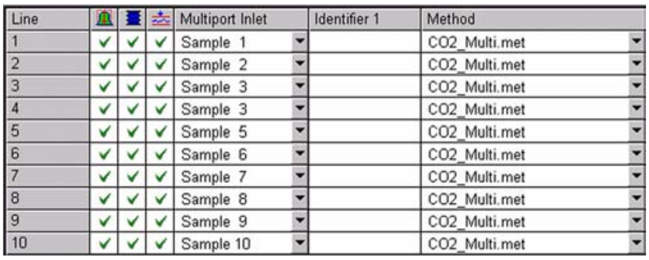


File Browser

D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet Sy...\Sequence

Name	Created	Modified
CO2.seq	01/21/02 13:17:50	01/21/02
CO2_zero left.seq	01/08/02 14:55:50	05/10/01
HD_zero.seq	01/08/02 14:55:50	05/11/01
Microvolume _onl...	01/08/02 14:55:50	04/19/01
MicroVolume.seq	01/08/02 14:55:50	11/02/00
Multiport.seq	01/08/02 14:55:51	01/25/01
Multiport+MicroV...	01/08/02 14:55:51	04/20/01
N2_zero.seq	01/08/02 14:55:51	01/24/01
SO2.seq	01/08/02 14:55:51	01/25/01

- In the "Method" column, the Method chosen in Chapter 4.4.2 occurs as default ("CO2_Multi.met").



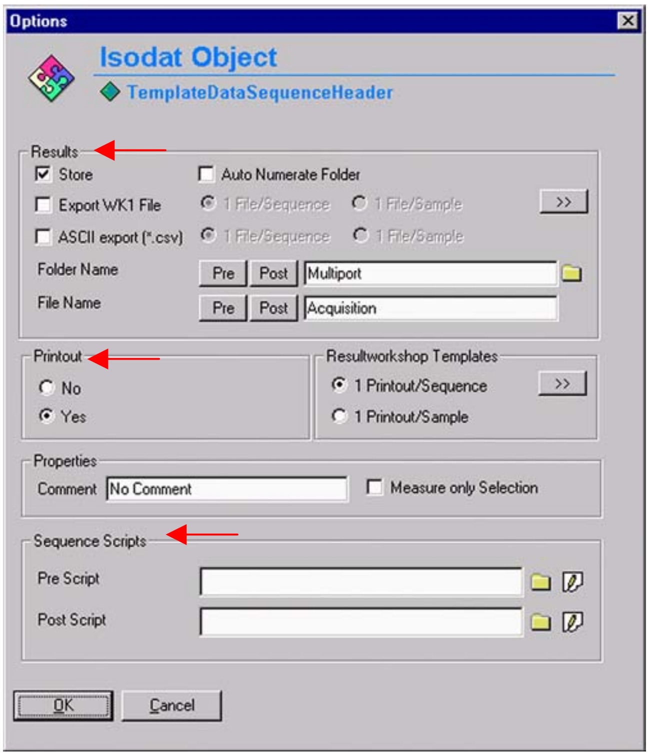
Line				Multiport Inlet	Identifier 1	Method
1	✓	✓	✓	Sample 1		CO2_Multi.met
2	✓	✓	✓	Sample 2		CO2_Multi.met
3	✓	✓	✓	Sample 3		CO2_Multi.met
4	✓	✓	✓	Sample 3		CO2_Multi.met
5	✓	✓	✓	Sample 5		CO2_Multi.met
6	✓	✓	✓	Sample 6		CO2_Multi.met
7	✓	✓	✓	Sample 7		CO2_Multi.met
8	✓	✓	✓	Sample 8		CO2_Multi.met
9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

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

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

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

- ➢ Define parameters for:
  - ❑ Results Export
  - ❑ Printout
  - ❑ Sequence Scripts
- ➢ Press ***OK*** to start Sequence Acquisition.



Isodat Object

TemplateDataSequenceHeader

Results

☑ Store ☐ Auto Numerate Folder

☐ Export WK1 File ● 1 File/Sequence ○ 1 File/Sample

☐ ASCII export (*.csv) ● 1 File/Sequence ○ 1 File/Sample

Folder Name: Multiport

File Name: Acquisition

Printout

○ No
● Yes

Resultworkshop Templates

● 1 Printout/Sequence
○ 1 Printout/Sample

Properties

Comment: No Comment ☐ Measure only Selection

Sequence Scripts

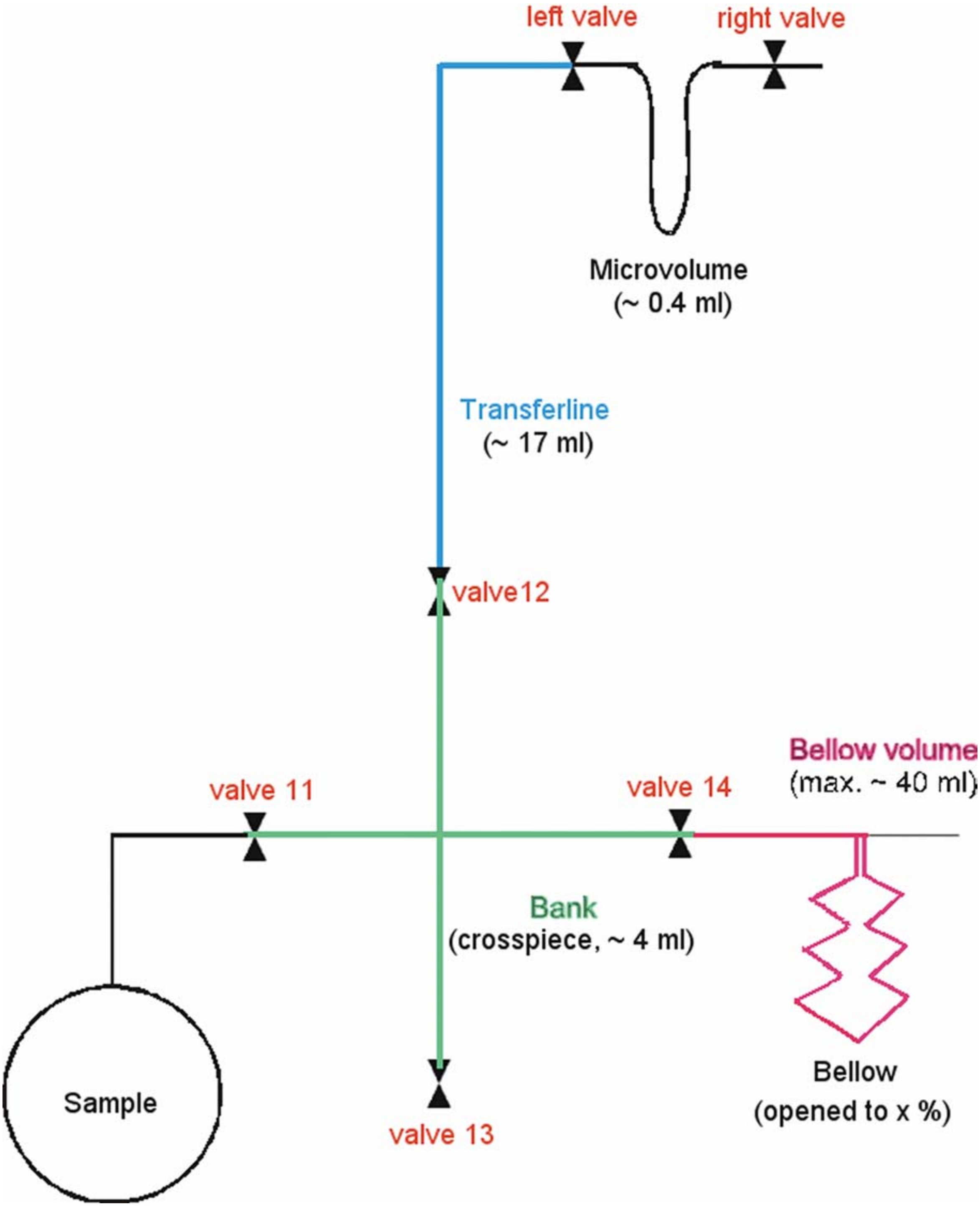
Pre Script:

Post Script:

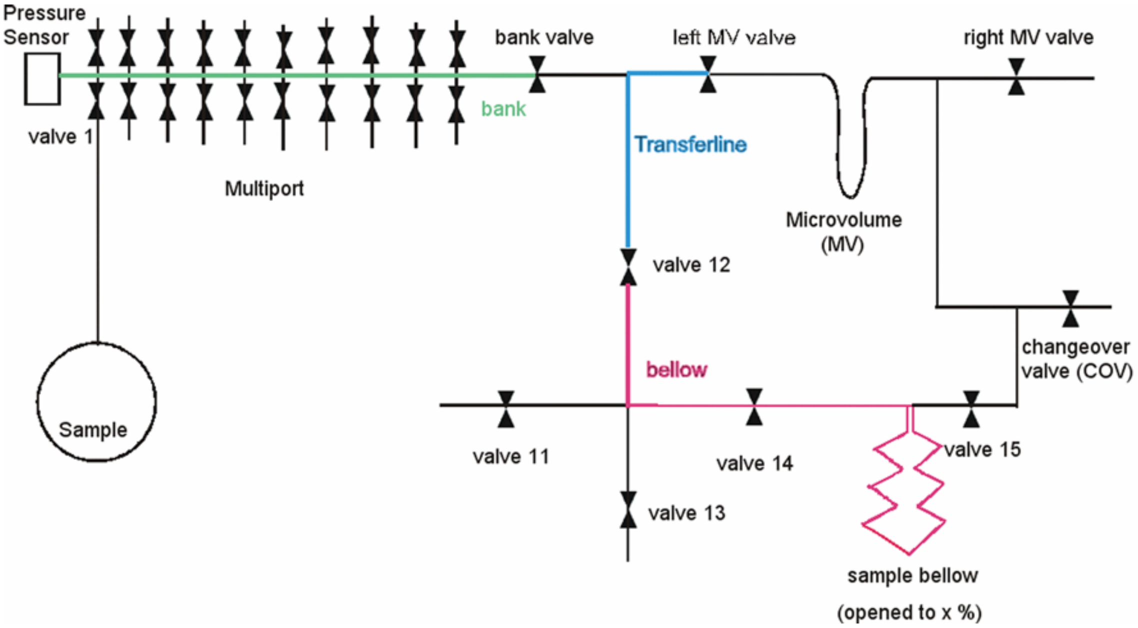
4.5 DUAL INLET MEASUREMENT INCLUDING MICROVOLUME

4.5.1 MICROVOLUME - MEASUREMENT PRINCIPLE

4.5.1.1 MICROVOLUME WITHOUT MULTIPORT - MEASUREMENT PRINCIPLE



4.5.1.2 MICROVOLUME VIA MULTIPORT - MEASUREMENT PRINCIPLE



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

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


Micro Volume

● Expansion ○ Freeze Direct

4.5.1.3 EXPANSION MODE – GENERAL REMARKS

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

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


Expansion Mode

○ Bank ○ Bank and Transferline ● Bank, Transferline and Bellow 50 Bellow Pos [%]

4.5.1.4 EXPANSION MODE – BEFORE MEASUREMENT

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

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

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

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

5 Start the Acquisition.

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

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

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

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

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

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

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

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

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

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

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

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

4.5.1.5 EXPANSION MODE – MEASUREMENT WITH BANK

Micro Volume

● Expansion ○ Freeze Direct

Expansion Mode

● Bank ○ Bank and Transferline ○ Bank, Transferline and Bellow 50 Bellow Pos [%]

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

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

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

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

4 The gas in the bank volume is pumped off.

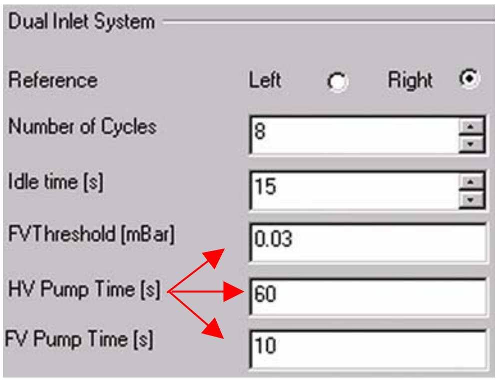
5 The sample valve, e.g. valve 1, is opened (of course, all other sample valves remain closed). The sample gas expands into the bank volume.

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

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

7 The pumping time depends on the three parameters

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



Dual Inlet System

Reference Left ○ Right ●

Number of Cycles	8
Idle time [s]	15
FVThreshold [mBar]	0.03
HV Pump Time [s]	60
FV Pump Time [s]	10

8. After pumping the sample valve (e.g. valve 1) is opened. Again, the gas expands into the Bank volume. Again, it is waited, until the Sample Equilibration Time (e.g. 60 s) has elapsed.
9. The decreased pressure p is measured again and compared to the Freeze Bank Threshold.

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

Sample Equilibration Time [s]: 60
Pump Out Temperature [°C]: 50
Initial Transfer Temperature [°C]: -50
Freeze Temperature [°C]: -175
Measure Temperature [°C]: 50
Bellow DirectThreshold [mBar]: 20
Freeze Bank Threshold [mBar]: 6
Freeze All Threshold [mBar]: 2
Freeze Time [s]: 60
Delay Before Measure [s]: 5

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

4.5.1.6 EXPANSION MODE – MEASUREMENT WITH BANK AND TRANSFERLINE

Micro Volume: ● Expansion ○ Freeze Direct
Expansion Mode: ○ Bank ● Bank and Transferline ○ Bank, Transferline and Bellow 50 Bellow Pos [%]

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

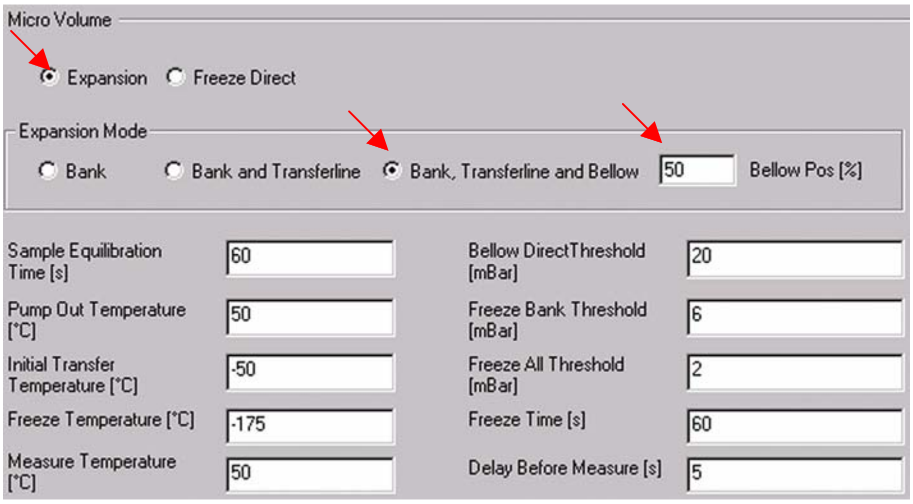
Example

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

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

4.5.1.7 EXPANSION MODE – MEASUREMENT WITH BANK, TRANSFERLINE AND BELLOW

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



Micro Volume

● Expansion ○ Freeze Direct

Expansion Mode

○ Bank ○ Bank and Transferline ● Bank, Transferline and Bellow 50 Bellow Pos [%]

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

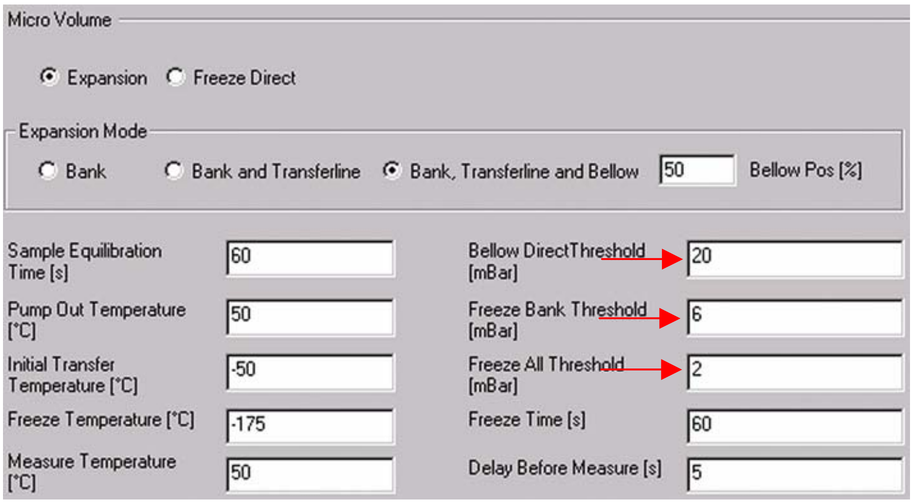
1 The bellow is opened to the specified percentage (e.g. 50 %).

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

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

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

5 The pressure p is measured.



Micro Volume

● Expansion ○ Freeze Direct

Expansion Mode

○ Bank ○ Bank and Transferline ● Bank, Transferline and Bellow 50 Bellow Pos [%]

Sample Equilibration Time [s]: 60

Pump Out Temperature [°C]: 50

Initial Transfer Temperature [°C]: -50

Freeze Temperature [°C]: -175

Measure Temperature [°C]: 50

Bellow DirectThreshold [mBar]: 20

Freeze Bank Threshold [mBar]: 6

Freeze All Threshold [mBar]: 2

Freeze Time [s]: 60

Delay Before Measure [s]: 5

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

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

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

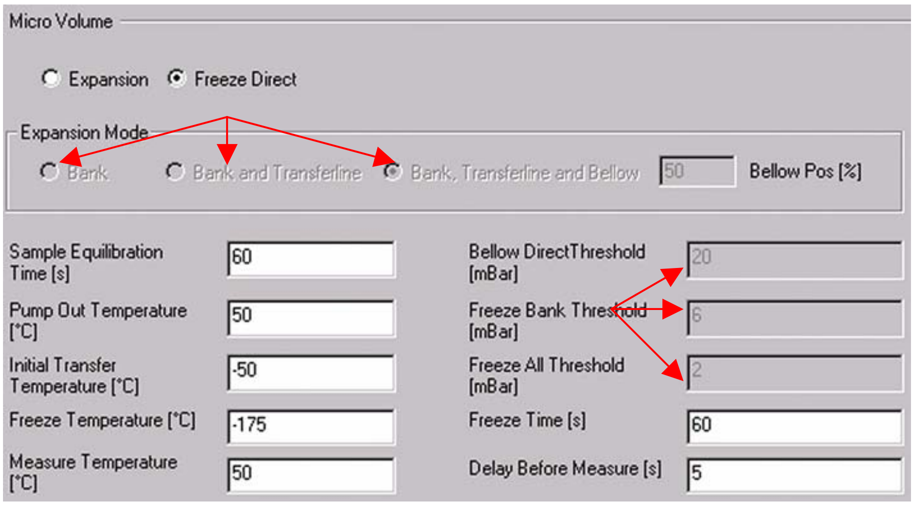
Case 4: If ***p < Freeze All Threshold***, only few gas is available. Therefore, the whole gas amount will be frozen.

4.5.1.8 FREEZE DIRECT MODE

Freeze direct mode is used

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

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



Micro Volume

○ Expansion ● Freeze Direct

Expansion Mode

○ Bank ○ Bank and Transferline ● Bank, Transferline and Bellow 50 Bellow Pos [%]

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

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

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

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

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

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

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

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

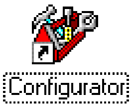
8 Then, bank valve and left valve of Microvolume are closed.

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

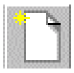
10 The system waits until Delay Before Measure has elapsed.

11 Measurement starts.

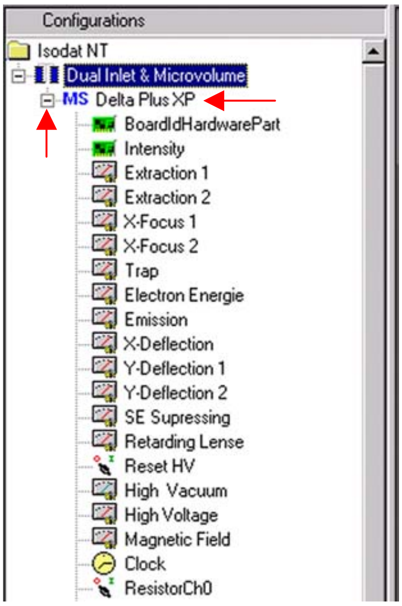
4.5.2 DEFINING A CONFIGURATION



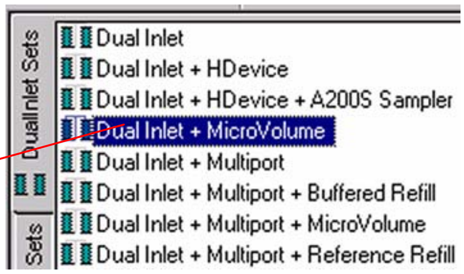
- ➢ Before operating, a ***Configuration*** containing the Dual Inlet System and the Microvolume must be created in the ***Configurator*** as follows.



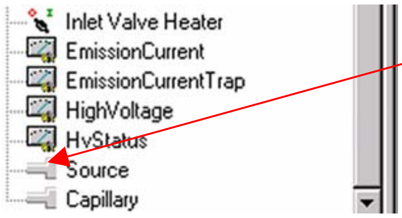
- ➢ Add a new Configuration using the ***Add Configuration*** button.



- ➢ Give it a significant name, e.g. ***“Dual Inlet & Microvolume”***.
- ➢ Open the tree structure by a click on ⊞ at ⊞ MS Delta Plus XP

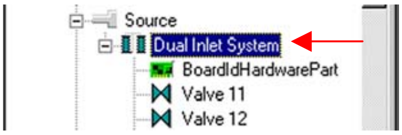


- ➢ On the right pane, select ***“Dual Inlet + Microvolume”***.



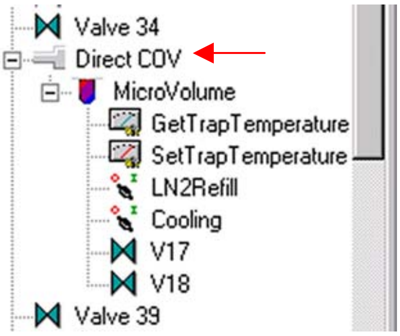
- ➢ Drag it to the Source port of the new Configuration.

A ⊞ sign appears:



Source
Dual Inlet System
BoardIdHardwarePart
Valve 11
Valve 12

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



Valve 34
Direct COV
MicroVolume
GetTrapTemperature
SetTrapTemperature
LN2Refill
Cooling
V17
V18
Valve 39

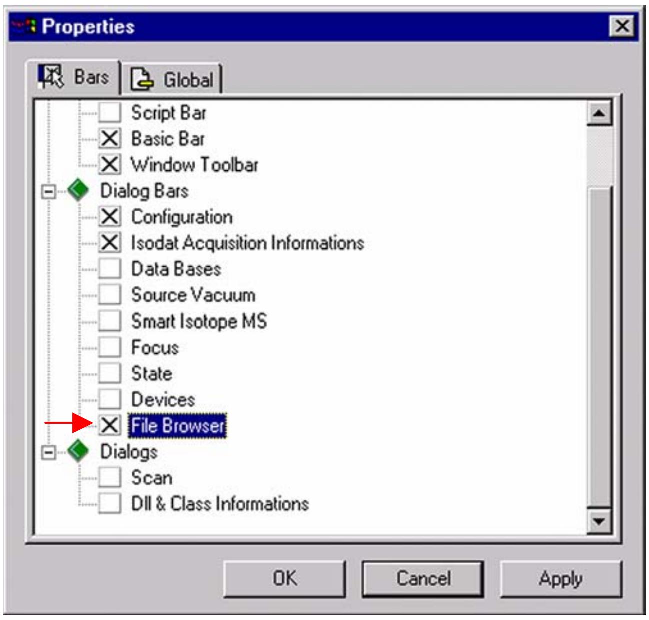
- ➢ The ***Microvolume*** has been attached to the ***Direct COV*** Port.

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

4.5.3 USING A PREDEFINED METHOD

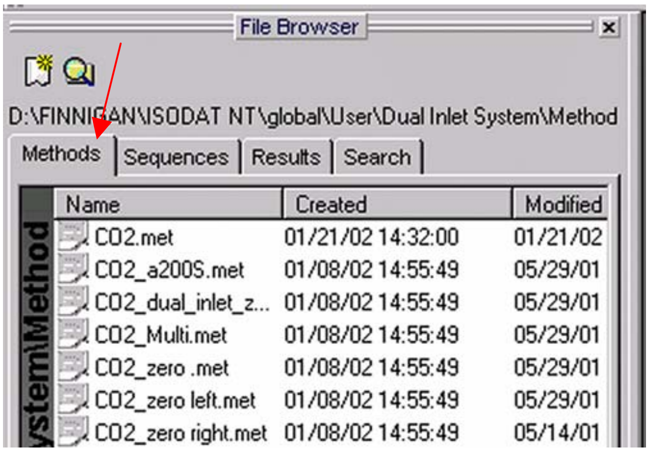
- If the File Browser cannot be seen, press the ***Options*** button.

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



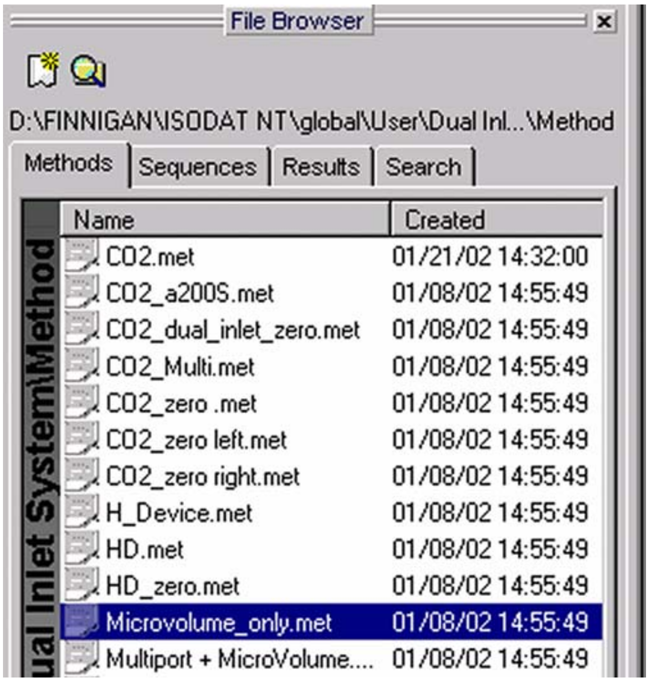
☐ Script Bar
☒ Basic Bar
☒ Window Toolbar
Dialog Bars
☒ Configuration
☒ Isodat Acquisition Informations
☐ Data Bases
☐ Source Vacuum
☐ Smart Isotope MS
☐ Focus
☐ State
☐ Devices
☒ File Browser
Dialogs
☐ Scan
☐ Dll & Class Informations

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



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01



D:\FINNIGAN\ISODAT NT\global\User\Dual Inl...\Method

Name	Created
CO2.met	01/21/02 14:32:00
CO2_a200S.met	01/08/02 14:55:49
CO2_dual_inlet_zero.met	01/08/02 14:55:49
CO2_Multi.met	01/08/02 14:55:49
CO2_zero .met	01/08/02 14:55:49
CO2_zero left.met	01/08/02 14:55:49
CO2_zero right.met	01/08/02 14:55:49
H_Device.met	01/08/02 14:55:49
HD.met	01/08/02 14:55:49
HD_zero.met	01/08/02 14:55:49
Microvolume_only.met	01/08/02 14:55:49
Multiport + MicroVolume....	01/08/02 14:55:49

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

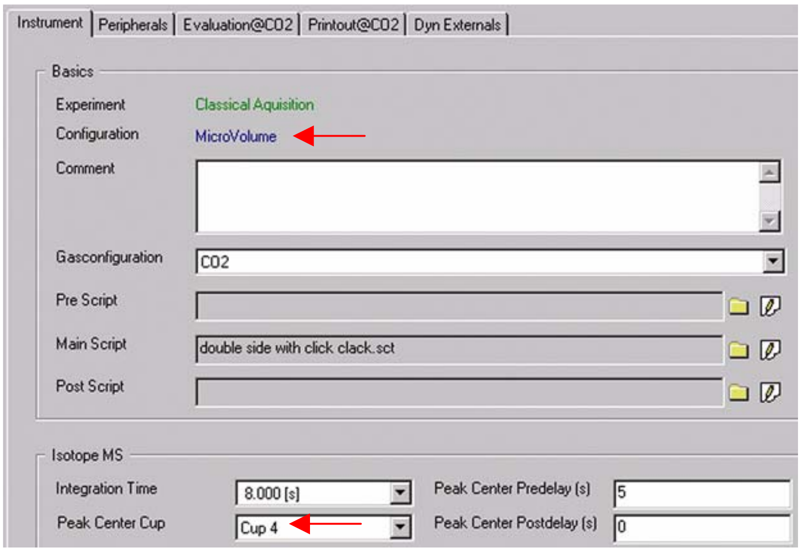
- If the Standards in the Method to be opened are older than the actual ones in the Standard Database, or if the user has changed the Method, the warning beside appears.

Standard values different to Standard Database!

Actualize now to new values!

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

- The ***Instrument tab*** of the Method “Microvolume_only.met” appears.
- For details see Chapter 4.3.2.



Basics

Experiment: Classical Aquisition

Configuration: MicroVolume

Comment:

Gasconfiguration: CO2

Pre Script:

Main Script: double side with click clack.sct

Post Script:

Isotope MS

Integration Time: 8.000 [s]

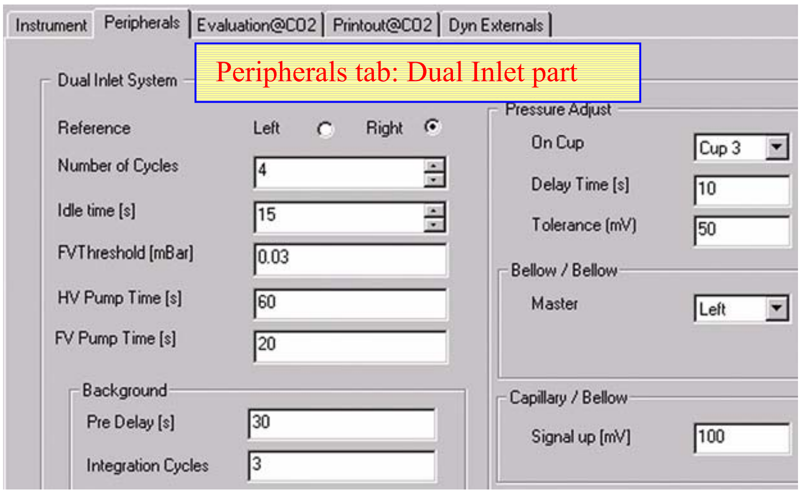
Peak Center Predelay [s]: 5

Peak Center Cup: Cup 4

Peak Center Postdelay [s]: 0

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

- For details at the Peripherals tab’s ***Dual Inlet*** part see Chapter 4.3.2.



Peripherals tab: Dual Inlet part

Dual Inlet System

Reference: Left ○ Right ●

Number of Cycles: 4

Idle time [s]: 15

FVThreshold [mBar]: 0.03

HV Pump Time [s]: 60

FV Pump Time [s]: 20

Background

Pre Delay [s]: 30

Integration Cycles: 3

Pressure Adjust

On Cup: Cup 3

Delay Time [s]: 10

Tolerance (mV): 50

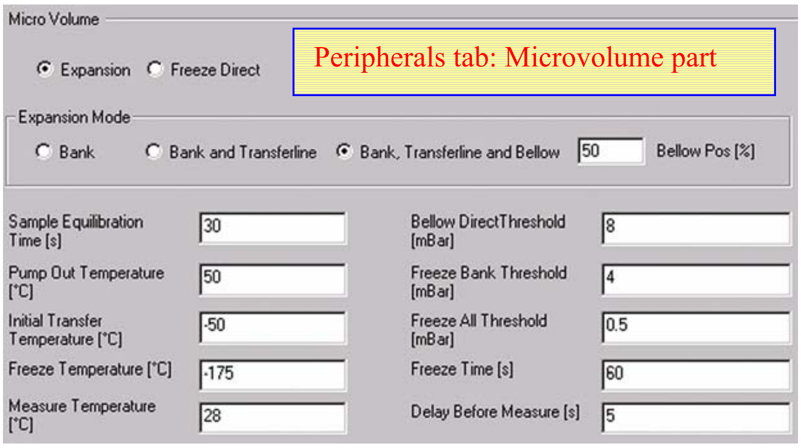
Bellow / Bellow

Master: Left

Capillary / Bellow

Signal up [mV]: 100

- For details about the Peripherals tab’s ***Microvolume*** part see Chapter 4.3.2.



Micro Volume

● Expansion ○ Freeze Direct

Peripherals tab: Microvolume part

Expansion Mode

○ Bank ○ Bank and Transferline ● Bank, Transferline and Bellow 50 Bellow Pos [%]

Sample Equilibration Time [s]: 30

Pump Out Temperature [°C]: 50

Initial Transfer Temperature [°C]: -50

Freeze Temperature [°C]: -175

Measure Temperature [°C]: 28

Bellow DirectThreshold [mBar]: 8

Freeze Bank Threshold [mBar]: 4

Freeze All Threshold [mBar]: 0.5

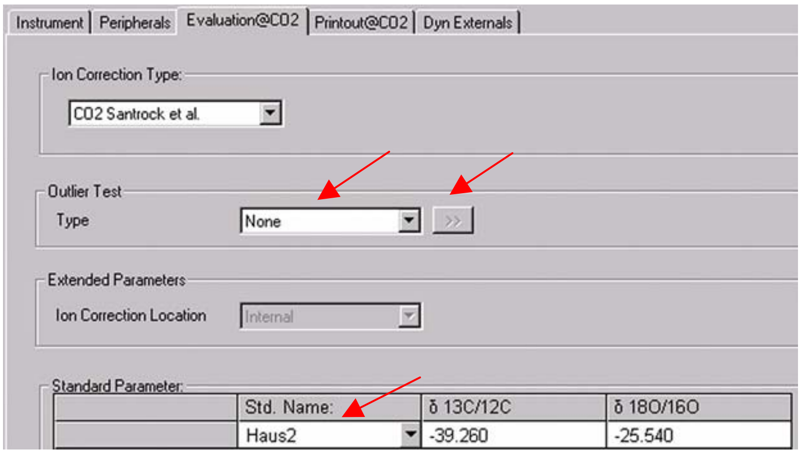
Freeze Time [s]: 60

Delay Before Measure [s]: 5

- At ***Evaluation CO2 tab***, select an Ion Correction type (e.g. “CO2 Santrock et al.”).

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

For details see Chapter 4.3.2.



Ion Correction Type: CO2 Santrock et al.

Outlier Test
Type: None

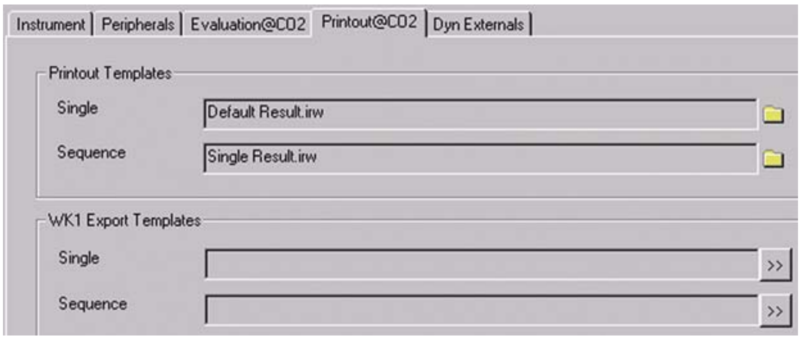
Extended Parameters
Ion Correction Location: Internal

Standard Parameter:

Std. Name:	δ 13C/12C	δ 18O/16O
Haus2	-39.260	-25.540

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

- For details at the ***Printout tab***, see Chapter 4.3.2.



Printout Templates
Single: Default Result.irw
Sequence: Single Result.irw

WK1 Export Templates
Single:
Sequence:

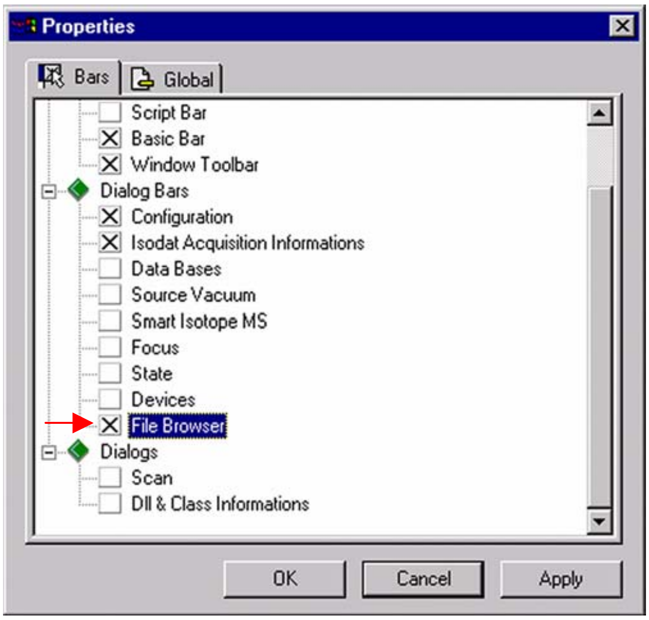
The ***Method*** for measurements using Dual Inlet in combination with a Microvolume has now been established. As next step, either use a ***predefined Sequence*** (refer to Chapter 4.5.4) or create a ***new Sequence*** (refer to Chapter 4.1.5).

4.5.4 USING A PREDEFINED SEQUENCE

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

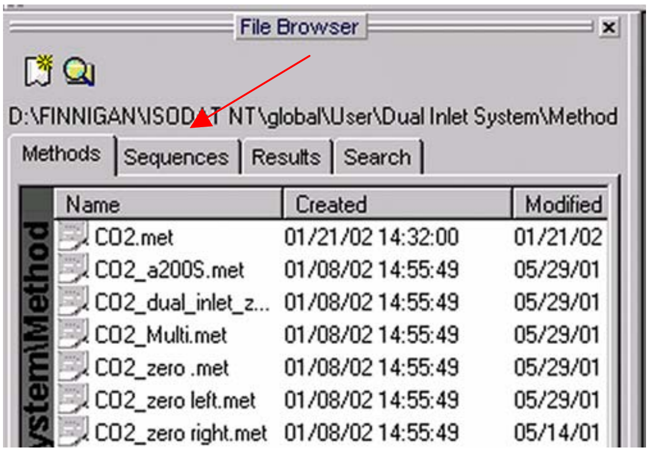
- Activate the ***File Browser*** check box.

- Click ***OK***.



☐ Script Bar
☒ Basic Bar
☒ Window Toolbar
Dialog Bars
☒ Configuration
☒ Isodat Acquisition Informations
☐ Data Bases
☐ Source Vacuum
☐ Smart Isotope MS
☐ Focus
☐ State
☐ Devices
☒ File Browser
Dialogs
☐ Scan
☐ Dll & Class Informations

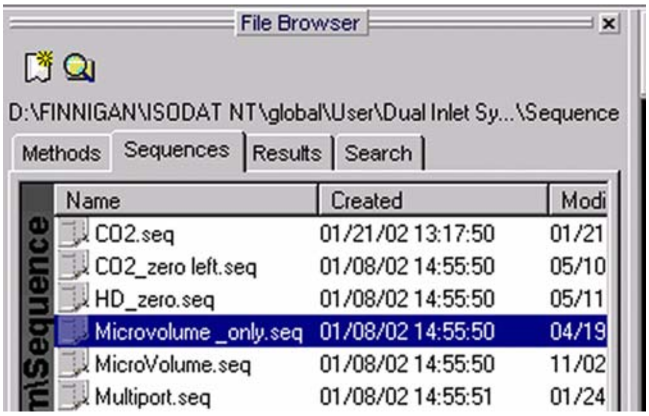
- On the File Browser, select the ***Sequences tab*** (default: Methods tab).



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

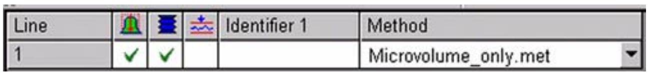
Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01

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



Name	Created	Modi
CO2.seq	01/21/02 13:17:50	01/21
CO2_zero left.seq	01/08/02 14:55:50	05/10
HD_zero.seq	01/08/02 14:55:50	05/11
Microvolume_only.seq	01/08/02 14:55:50	04/19
MicroVolume.seq	01/08/02 14:55:50	11/02
Multiport.seq	01/08/02 14:55:51	01/24

- In the "Method" column, the Method chosen in Chapter 4.5.3 occurs as default ("CO2_Multi.met").

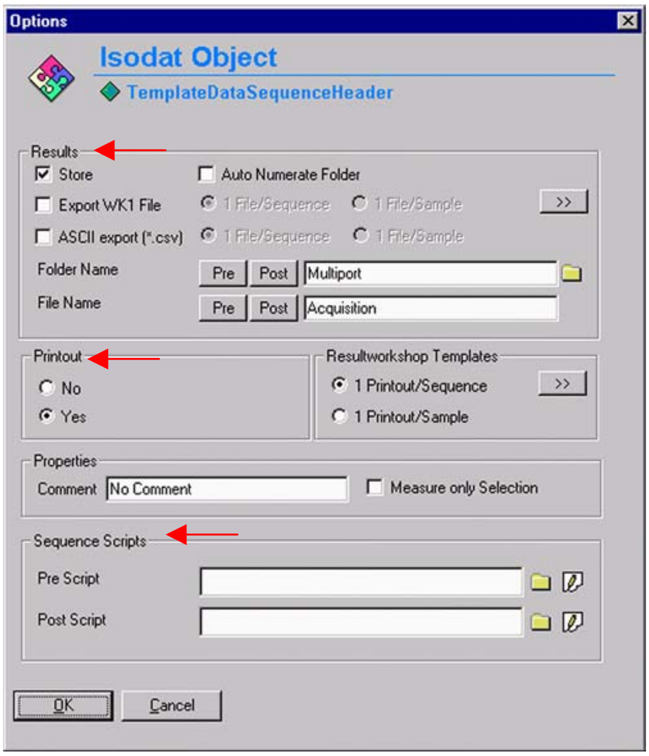


Line				Identifier 1	Method
1	✓	✓			Microvolume_only.met

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

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

- ➢ Define parameters for:
  - ❑ Results Export
  - ❑ Printout
  - ❑ Sequence Scripts
- ➢ Press ***OK*** to start Sequence Acquisition.



Results
☑ Store
☐ Auto Numerate Folder
☐ Export WK1 File ● 1 File/Sequence ○ 1 File/Sample
☐ ASCII export (*.csv) ● 1 File/Sequence ○ 1 File/Sample
Folder Name: Multiport
File Name: Acquisition

Printout
○ No
● Yes

Resultworkshop Templates
● 1 Printout/Sequence
○ 1 Printout/Sample

Properties
Comment: No Comment
☐ Measure only Selection

Sequence Scripts
Pre Script:
Post Script:

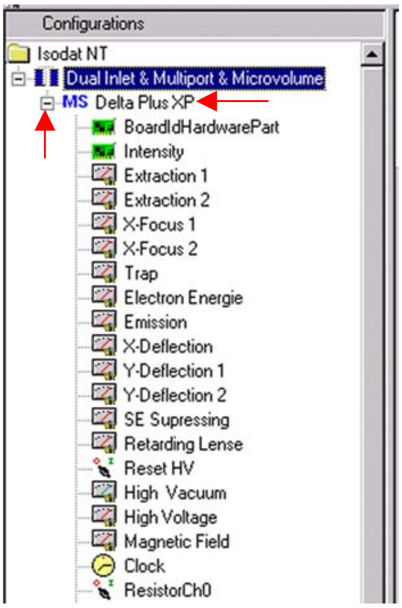
4.6 DUAL INLET MEASUREMENT INCLUDING MULTIPORT AND MICROVOLUME

4.6.1 DEFINING A CONFIGURATION

Configurator

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

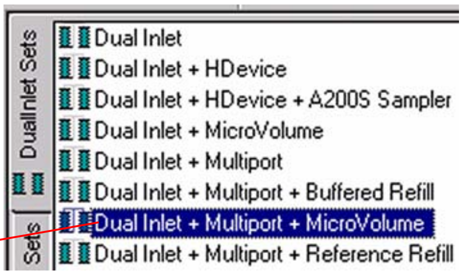
- Add a new Configuration using the ***Add Configuration*** button.

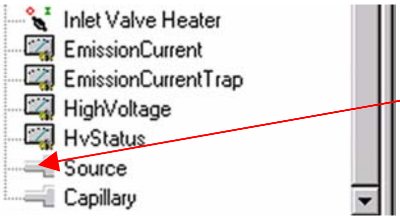


- Give it a significant name, e.g. ***"Dual Inlet & Multiport & Microvolume"***.

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

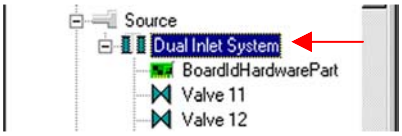
- On the right pane, select ***Dual Inlet +Multiport + Microvolume***.



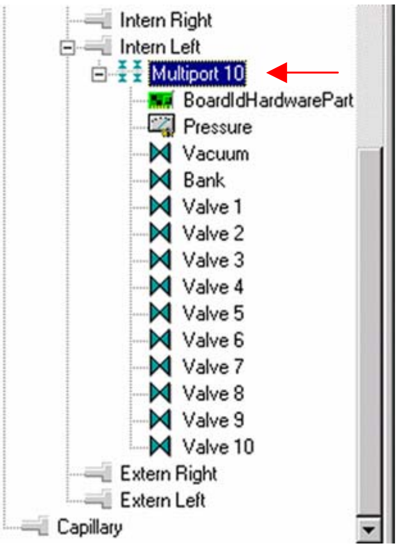


- Drag it to the Source port of the new Configuration.

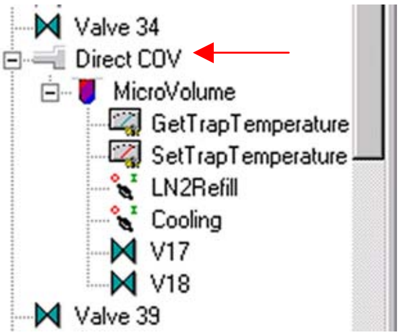
A ⊞ sign appears:



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



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



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

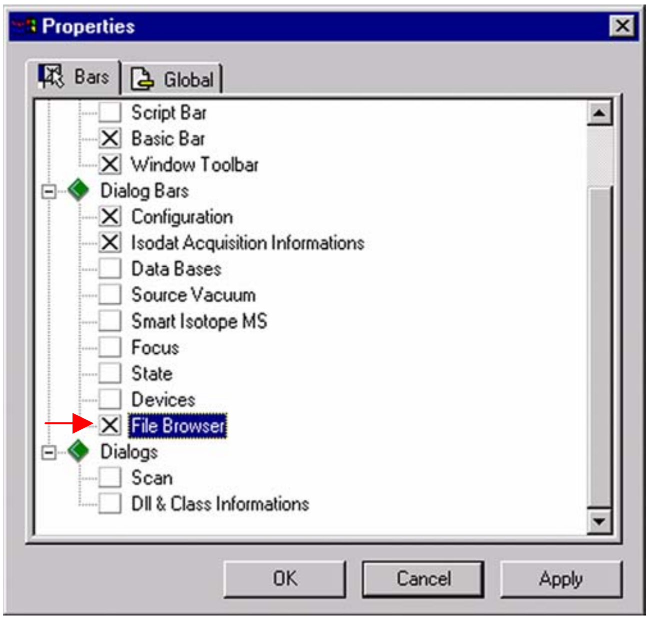
- Close the Configurator window.

  All settings will be saved automatically.

4.6.2 USING A PREDEFINED METHOD

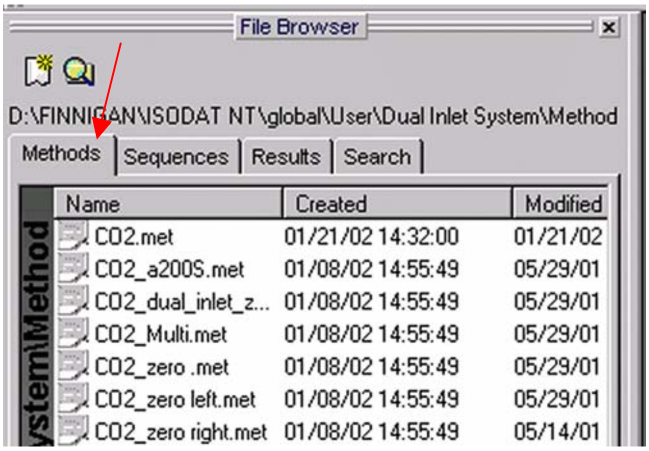
- If the File Browser cannot be seen, press the ***Options*** button.

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



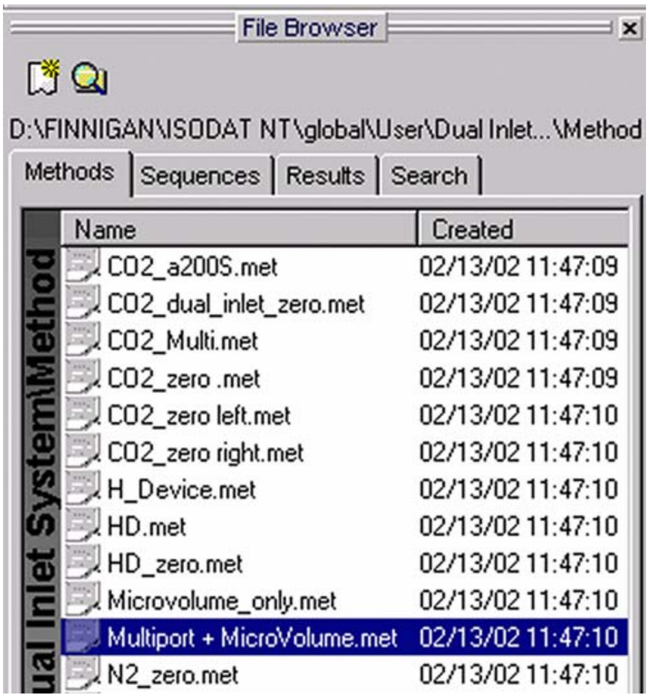
☐ Script Bar
☒ Basic Bar
☒ Window Toolbar
Dialog Bars
  ☒ Configuration
  ☒ Isodat Acquisition Informations
  ☐ Data Bases
  ☐ Source Vacuum
  ☐ Smart Isotope MS
  ☐ Focus
  ☐ State
  ☐ Devices
  ☒ File Browser
Dialogs
  ☐ Scan
  ☐ Dll & Class Informations

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



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01



Name	Created
CO2_a200S.met	02/13/02 11:47:09
CO2_dual_inlet_zero.met	02/13/02 11:47:09
CO2_Multi.met	02/13/02 11:47:09
CO2_zero .met	02/13/02 11:47:09
CO2_zero left.met	02/13/02 11:47:10
CO2_zero right.met	02/13/02 11:47:10
H_Device.met	02/13/02 11:47:10
HD.met	02/13/02 11:47:10
HD_zero.met	02/13/02 11:47:10
Microvolume_only.met	02/13/02 11:47:10
Multiport + MicroVolume.met	02/13/02 11:47:10
N2_zero.met	02/13/02 11:47:10

- ➢ From the predefined Methods choose ***“Multiport + Microvolume.met”*** by double-click.

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

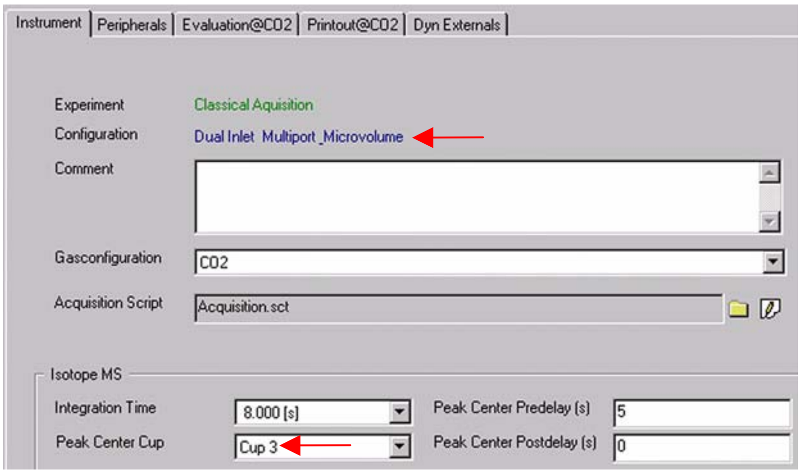
Standard values different to Standard Database!

Actualize now to new values!

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

- The ***Instrument tab*** of the Method “Multiport + Microvolume.met” appears.

- For details see Chapter 4.3.2.



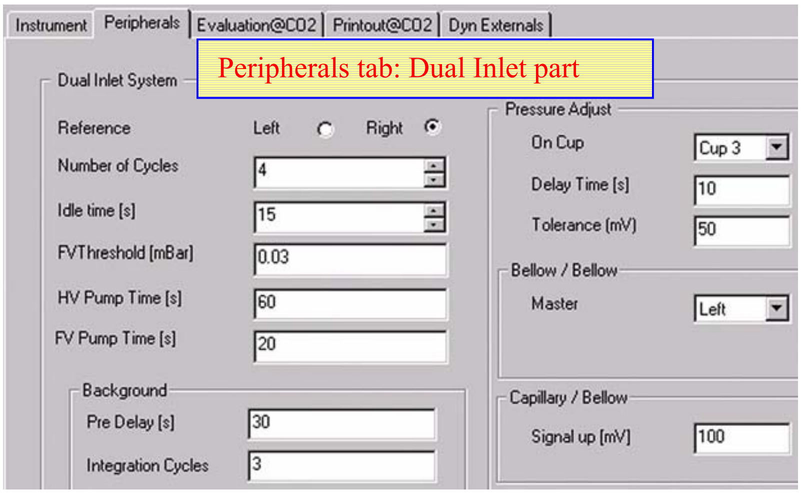
Experiment	Classical Aquisition
Configuration	Dual Inlet Multiport Microvolume
Comment	
Gasconfiguration	CO2
Acquisition Script	Acquisition.sct

Isotope MS

Integration Time	8.000 [s]	Peak Center Predelay [s]	5
Peak Center Cup	Cup 3	Peak Center Postdelay [s]	0

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

- For details at the Peripherals tab’s ***Dual Inlet*** part, see Chapter 4.3.2.



Peripherals tab: Dual Inlet part

Dual Inlet System

Reference	Left ○ Right ●
Number of Cycles	4
Idle time [s]	15
FVThreshold [mBar]	0.03
HV Pump Time [s]	60
FV Pump Time [s]	20

Background

Pre Delay [s]	30
Integration Cycles	3

Pressure Adjust

On Cup	Cup 3
Delay Time [s]	10
Tolerance (mV)	50

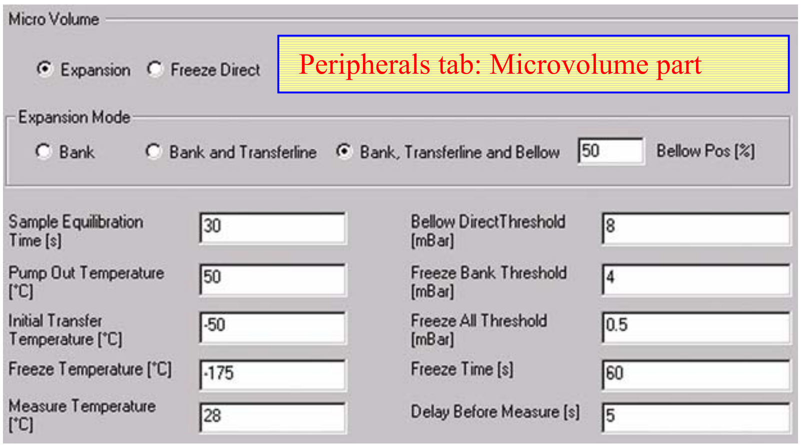
Bellow / Bellow

Master	Left

Capillary / Bellow

Signal up [mV]	100

- For details at the Peripherals tab’s ***Microvolume*** part, see Chapter 4.5.3.


Peripherals tab: Microvolume part

Micro Volume

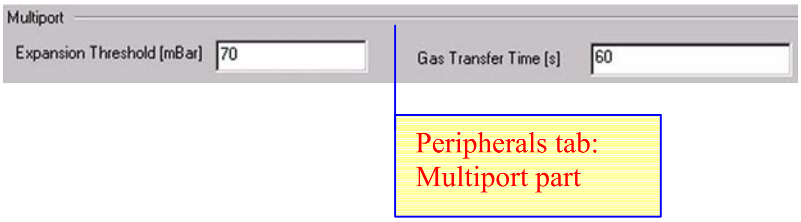
● Expansion ○ Freeze Direct

Expansion Mode

○ Bank ○ Bank and Transferline ● Bank, Transferline and Bellow 50 Bellow Pos [%]

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

- For details at the Peripherals tab’s ***Multiport*** part, see Chapter 4.4.2.



Expansion Threshold [mBar] 70

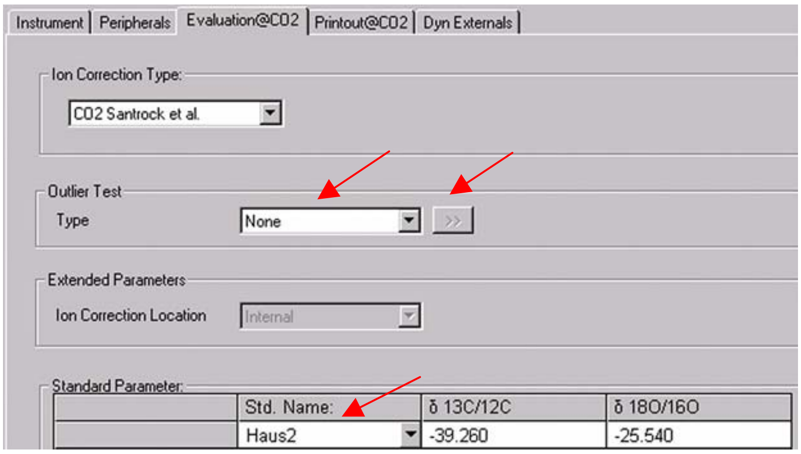
Gas Transfer Time [s] 60

Peripherals tab:
Multiport part

- At ***Evaluation CO2 tab***, Select an Ion Correct type, e.g. “CO2 Santrock et al.”.

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

For details see Chapter 4.3.2.



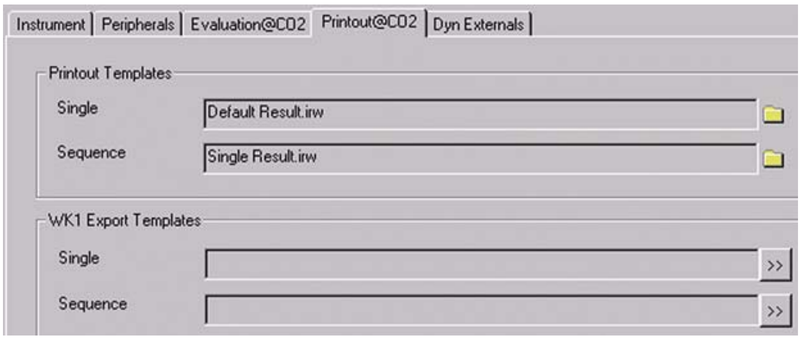
Ion Correction Type: CO2 Santrock et al.

Type None

Ion Correction Location Internal

Std. Name:	δ 13C/12C	δ 18O/16O
Haus2	-39.260	-25.540

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



Single Default Result.irw

Sequence Single Result.irw

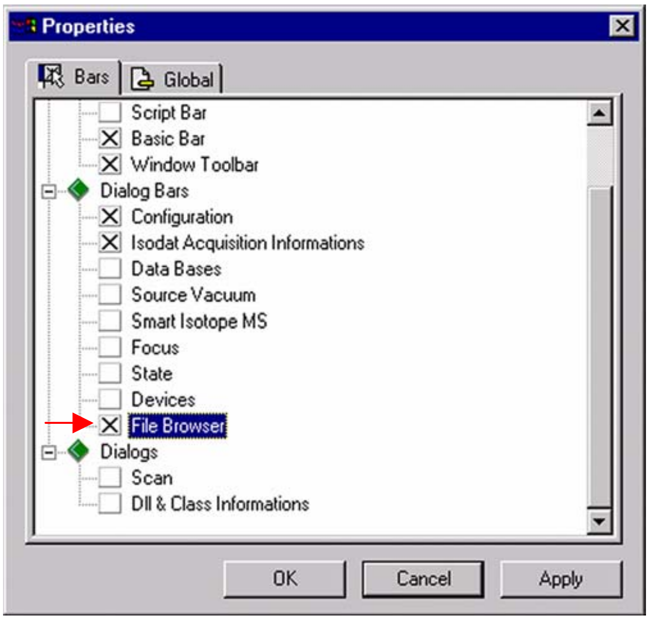
- For details at ***Printout tab***, see Chapter 4.3.2.

The ***Method*** for measurements using Dual Inlet in combination with a Multiport and a Microvolume has now been established. As next step, either use a ***predefined Sequence*** (refer to Chapter 4.6.3) or create a ***new Sequence*** (refer to Chapter 4.1.5).

4.6.3 USING A PREDEFINED SEQUENCE

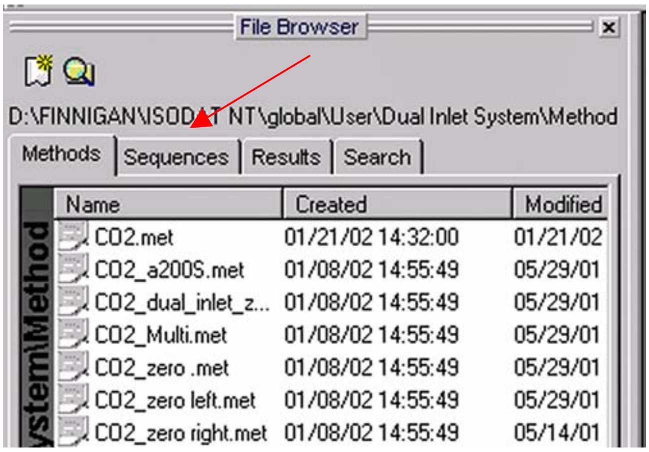
- If the File Browser cannot be seen, press the ***Options*** button.

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



☐ Script Bar
☒ Basic Bar
☒ Window Toolbar
Dialog Bars
☒ Configuration
☒ Isodat Acquisition Informations
☐ Data Bases
☐ Source Vacuum
☐ Smart Isotope MS
☐ Focus
☐ State
☐ Devices
☒ File Browser
Dialogs
☐ Scan
☐ Dll & Class Informations

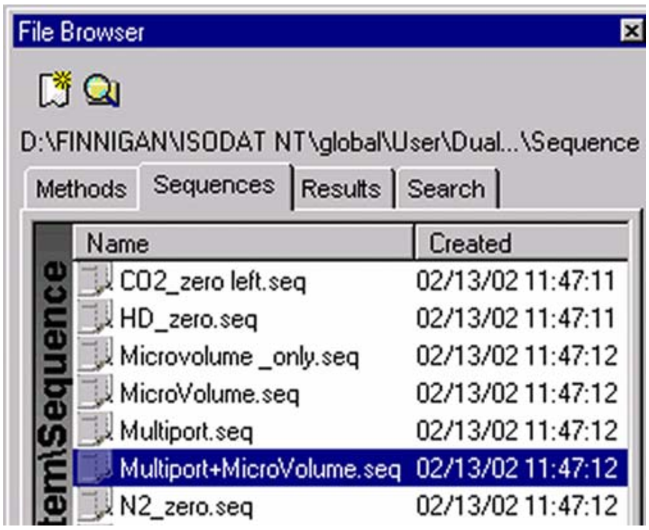
- On the File Browser, select the ***Sequences tab*** (default: Methods tab).



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01

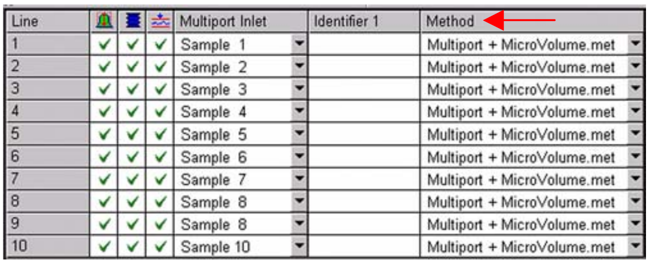
- From the predefined Sequences choose ***"Multiport + Microvolume.seq"*** by double-click.



D:\FINNIGAN\ISODAT NT\global\User\Dual...\Sequence

Name	Created
CO2_zero left.seq	02/13/02 11:47:11
HD_zero.seq	02/13/02 11:47:11
Microvolume _only.seq	02/13/02 11:47:12
MicroVolume.seq	02/13/02 11:47:12
Multiport.seq	02/13/02 11:47:12
Multiport+MicroVolume.seq	02/13/02 11:47:12
N2_zero.seq	02/13/02 11:47:12

- In the "Method" column, the Method chosen in Chapter 4.6.2 occurs as default ("Multiport + Microvolume.met").

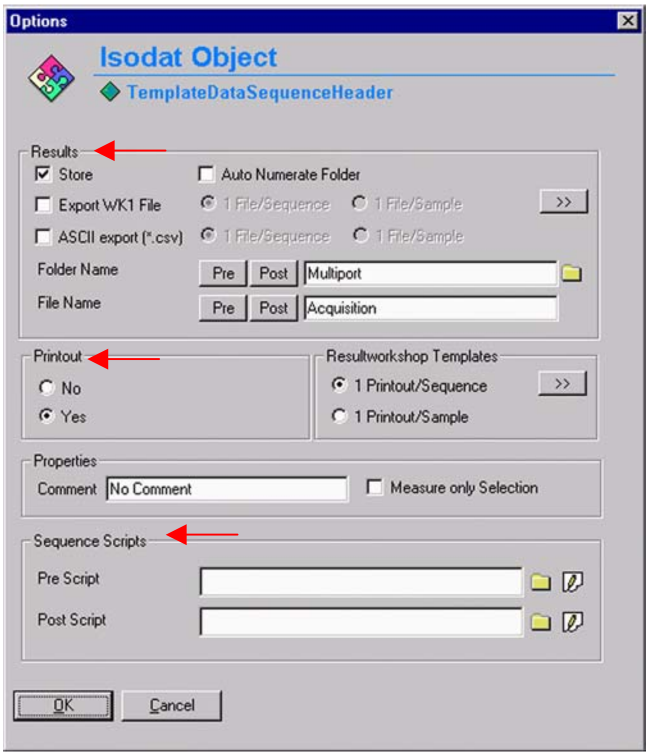


Line				Multiport Inlet	Identifier 1	Method
1	✓	✓	✓	Sample 1		Multiport + MicroVolume.met
2	✓	✓	✓	Sample 2		Multiport + MicroVolume.met
3	✓	✓	✓	Sample 3		Multiport + MicroVolume.met
4	✓	✓	✓	Sample 4		Multiport + MicroVolume.met
5	✓	✓	✓	Sample 5		Multiport + MicroVolume.met
6	✓	✓	✓	Sample 6		Multiport + 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

Normally, the Sequence List needs not to be edited further. It is possible, however, e.g. to select another Method from the "Method" column or to modify the "Multiport Inlet" column. For details, see Chapter 4.3.3.

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

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



Options

Isodat Object

TemplateDataSequenceHeader

Results
☑ Store
☐ Auto Numerate Folder
☐ Export WK1 File ● 1 File/Sequence ○ 1 File/Sample
☐ ASCII export (*.csv) ● 1 File/Sequence ○ 1 File/Sample
Folder Name: Multiport
File Name: Acquisition

Printout
○ No
● Yes

Resultworkshop Templates
● 1 Printout/Sequence
○ 1 Printout/Sample

Properties
Comment: No Comment
☐ Measure only Selection

Sequence Scripts
Pre Script:
Post Script:

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

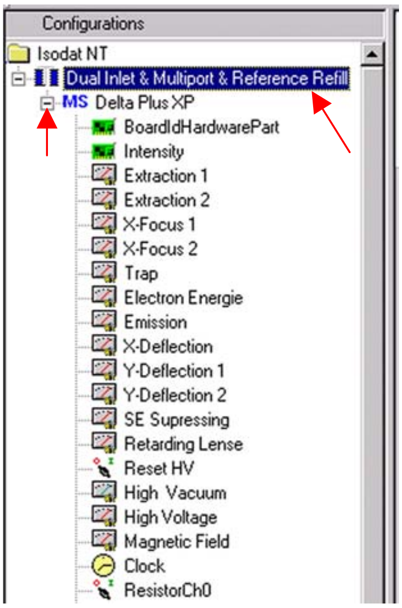
4.7 DUAL INLET MEASUREMENT INCLUDING MULTIPORT AND REFERENCE REFILL

4.7.1 DEFINING A CONFIGURATION

Configurator

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

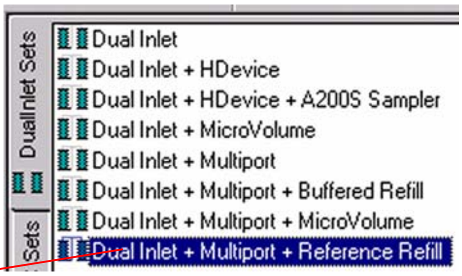
- ➢ Add a new Configuration using the ***Add Configuration*** button.



Configurations
Isodat NT
Dual Inlet & Multiport & Reference Refill
MS Delta Plus XP
BoardIdHardwarePart
Intensity
Extraction 1
Extraction 2
X-Focus 1
X-Focus 2
Trap
Electron Energie
Emission
X-Deflection
Y-Deflection 1
Y-Deflection 2
SE Supressing
Retarding Lense
Reset HV
High Vacuum
High Voltage
Magnetic Field
Clock
ResistorCh0

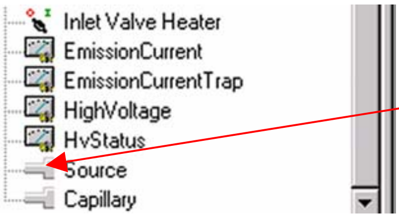
- ➢ Give it a significant name, e.g. ***“Dual Inlet & Multiport & Reference Refill”***.

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



Dual Inlet
Dual Inlet + HDevice
Dual Inlet + HDevice + A200S Sampler
Dual Inlet + MicroVolume
Dual Inlet + Multiport
Dual Inlet + Multiport + Buffered Refill
Dual Inlet + Multiport + MicroVolume
Dual Inlet + Multiport + Reference Refill

- ➢ On the right pane, select ***Dual Inlet + Multiport + Reference Refill***.

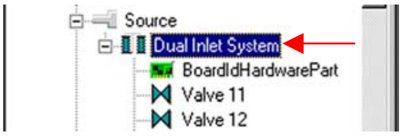


Inlet Valve Heater
EmissionCurrent
EmissionCurrentTrap
HighVoltage
HvStatus
Source
Capillary

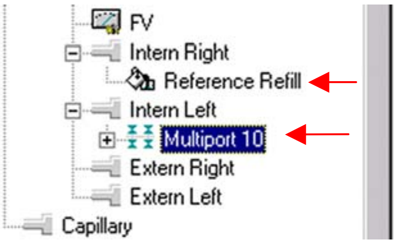
- ➢ Drag it to the Source port of the new Configuration.

  A ⊞ sign appears:

HvStatus
Source
Capillary



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



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

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

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

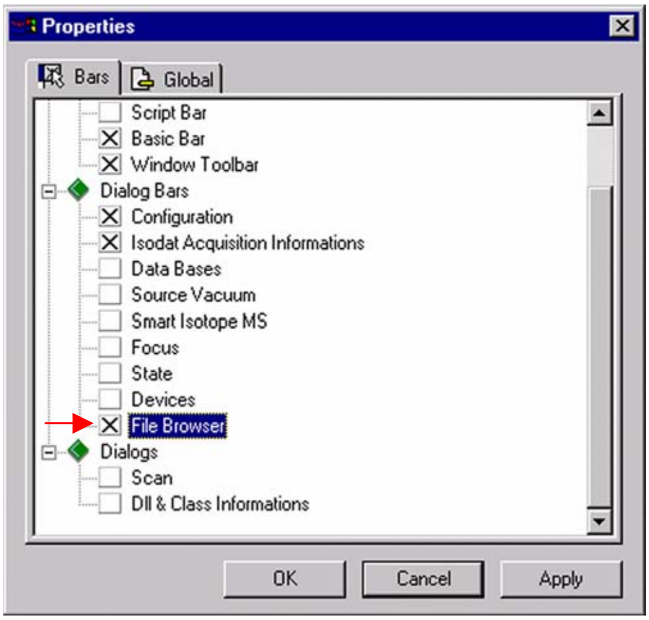
- ➢ Close the Configurator window.

  All settings will be saved automatically.

4.7.2 USING A PREDEFINED METHOD

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

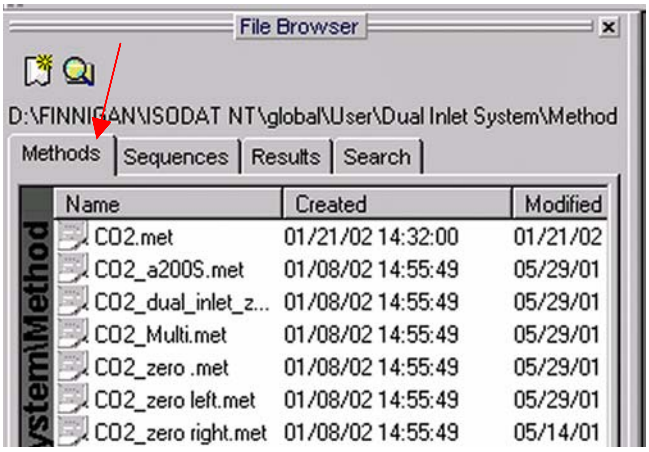
- ➢ Activate the ***File Browser*** check box.



☐ Script Bar
☒ Basic Bar
☒ Window Toolbar
Dialog Bars
☒ Configuration
☒ Isodat Acquisition Informations
☐ Data Bases
☐ Source Vacuum
☐ Smart Isotope MS
☐ Focus
☐ State
☐ Devices
☒ File Browser
Dialogs
☐ Scan
☐ Dll & Class Informations

- ➢ Click ***OK***.

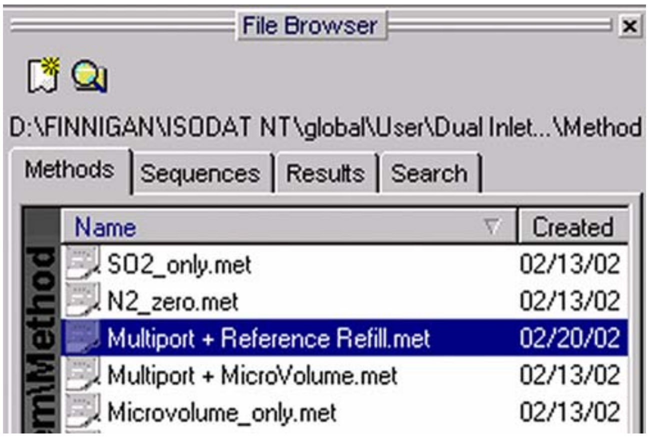
- ➢ On the File Browser, select the ***Methods tab*** (default).



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01

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



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet...\Method

Name	Created
SO2_only.met	02/13/02
N2_zero.met	02/13/02
Multiport + Reference Refill.met	02/20/02
Multiport + MicroVolume.met	02/13/02
Microvolume_only.met	02/13/02

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

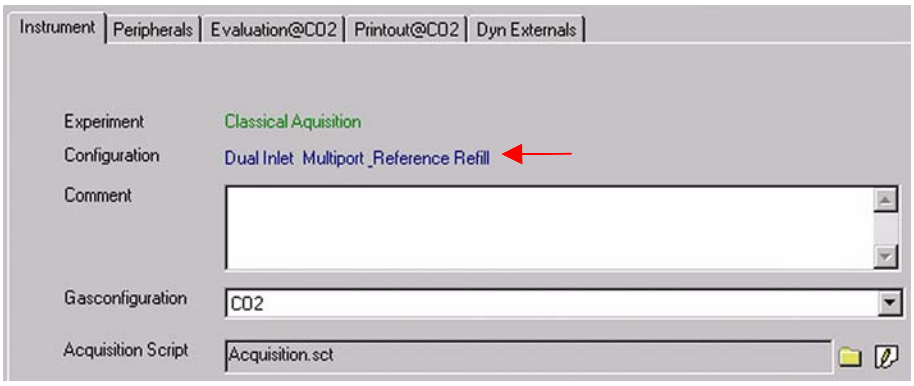
Standard values different to Standard Database!

Actualize now to new values!

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

Instrument tab

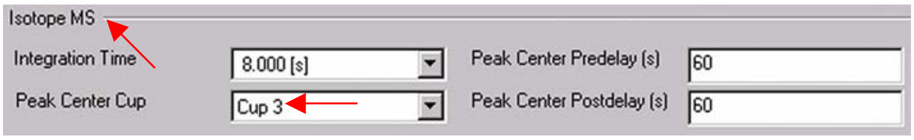
The ***Instrument tab*** of the Method “Multiport + Reference Refill.met” appears.



Experiment Classical Aquisition
Configuration Dual Inlet Multiport_Reference Refill
Comment
Gasconfiguration CO2
Acquisition Script Acquisition.sct

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

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



Isotope MS

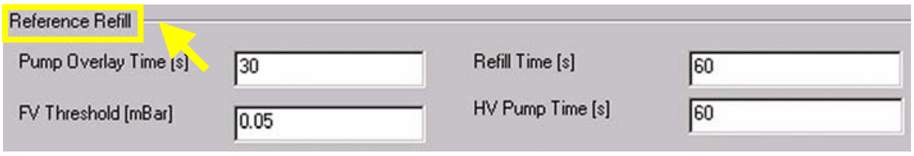
Integration Time: 8.000 [s]

Peak Center Cup: Cup 3

Peak Center Predelay [s]: 60

Peak Center Postdelay [s]: 60

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



Reference Refill

Pump Overlay Time [s]: 30

FV Threshold [mBar]: 0.05

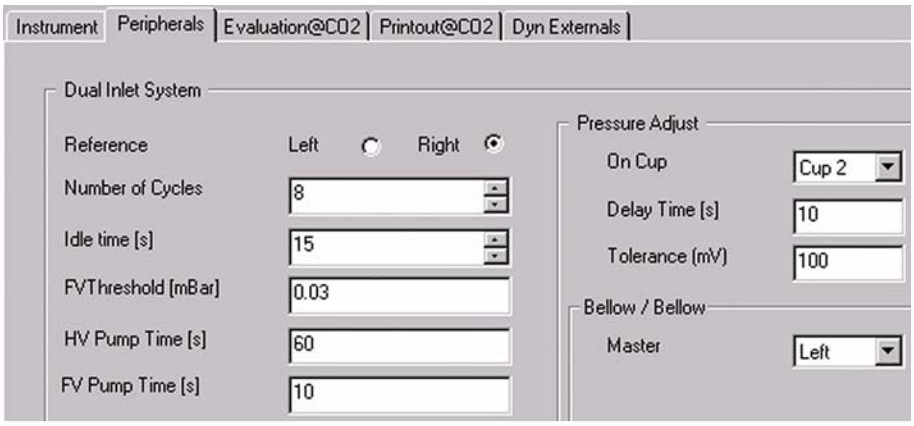
Refill Time [s]: 60

HV Pump Time [s]: 60

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

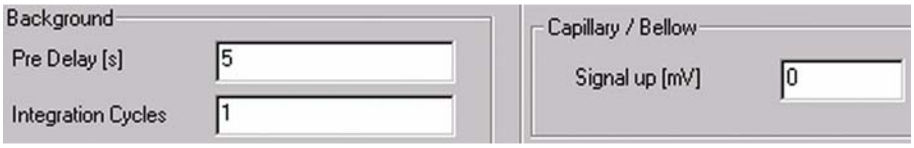
➢ ***Pump Overlay Time***:	Capillary pump out time of Reference Refill tank.
➢ ***FV Threshold***:	Minimum pressure of standard bellow including the valves and tubes evacuated using fore vacuum pump before continuing pumping using turbo molecular pump.
➢ ***Refill Time***:	Gas flow time from Reference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

Peripherals tab



Reference Left ○ Right ●
Number of Cycles 8
Idle time [s] 15
FVThreshold [mBar] 0.03
HV Pump Time [s] 60
FV Pump Time [s] 10
On Cup Cup 2
Delay Time [s] 10
Tolerance (mV) 100
Master Left

For details see Chapter 4.3.2.



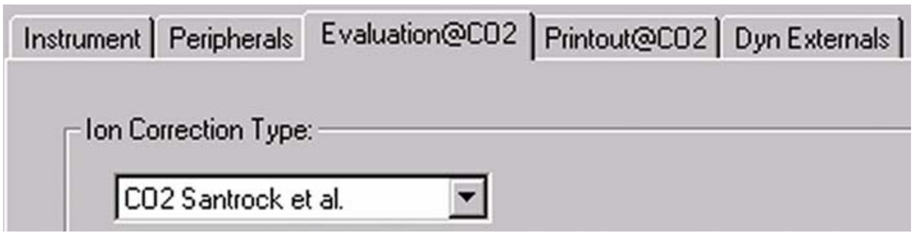
Pre Delay [s] 5
Integration Cycles 1
Signal up [mV] 0

For details see Chapter 4.3.2.

Expansion Threshold [mBar] 70
Gas Transfer Time [s] 60

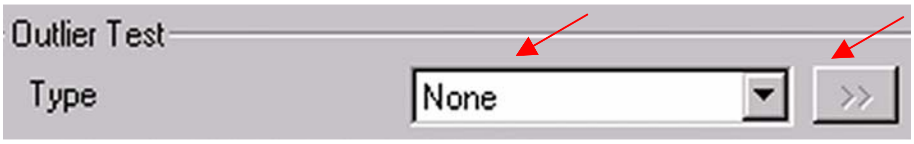
For details see Chapter 4.3.2.

Evaluation tab



Ion Correction Type: CO2 Santrock et al.

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



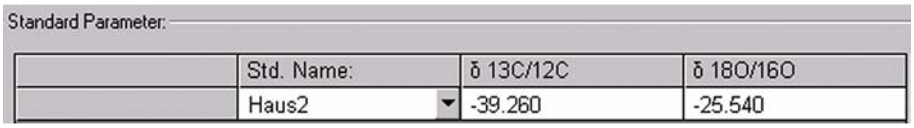
Outlier Test

Type: None

- As ***Outlier test*** select “None” or “Sigma”. In case of “Sigma”, specify the k-fold of the standard deviation using >>. For details see Chapter 4.3.2.

Extended Parameters

Ion Correction Location: Internal

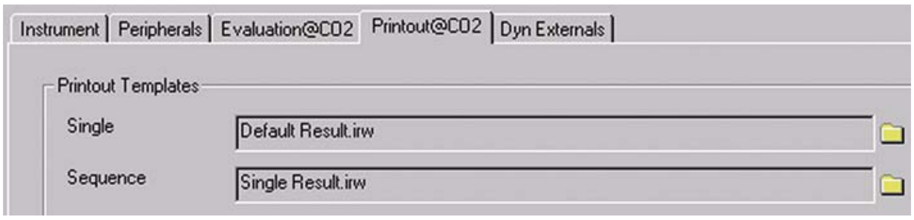


Standard Parameter:

	Std. Name:	δ 13C/12C	δ 18O/16O
	Haus2	-39.260	-25.540

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

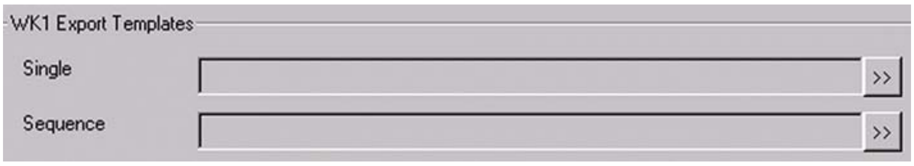
Printout tab



Single: Default Result.irw

Sequence: Single Result.irw

For details see Chapter 4.3.2.



For details see Chapter 4.3.2.

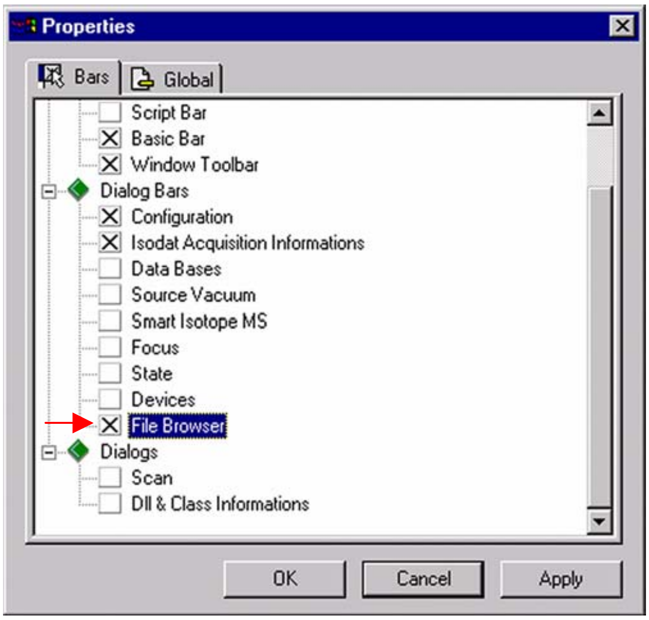
The ***Method*** for measurements using Dual Inlet in combination with a Multiport and a Reference Refill has now been established. As next step, either use a ***predefined Sequence*** (refer to Chapter 4.7.3) or create a ***new Sequence*** (refer to Chapter 4.1.5).

4.7.3 USING A PREDEFINED SEQUENCE

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

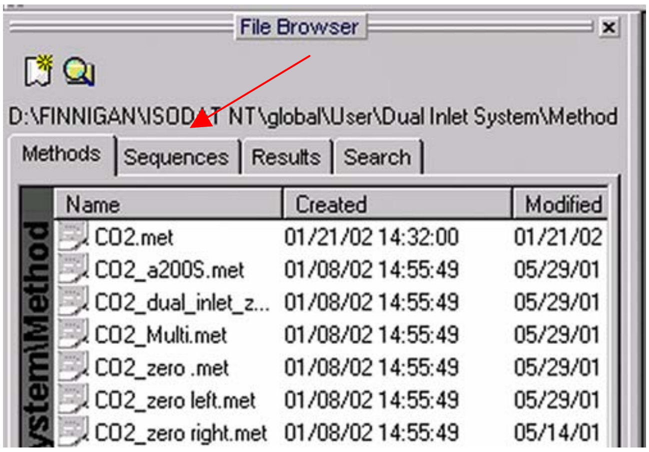
- Activate the ***File Browser*** check box.

- Click ***OK***.



☐ Script Bar
☒ Basic Bar
☒ Window Toolbar
Dialog Bars
☒ Configuration
☒ Isodat Acquisition Informations
☐ Data Bases
☐ Source Vacuum
☐ Smart Isotope MS
☐ Focus
☐ State
☐ Devices
☒ File Browser
Dialogs
☐ Scan
☐ Dll & Class Informations

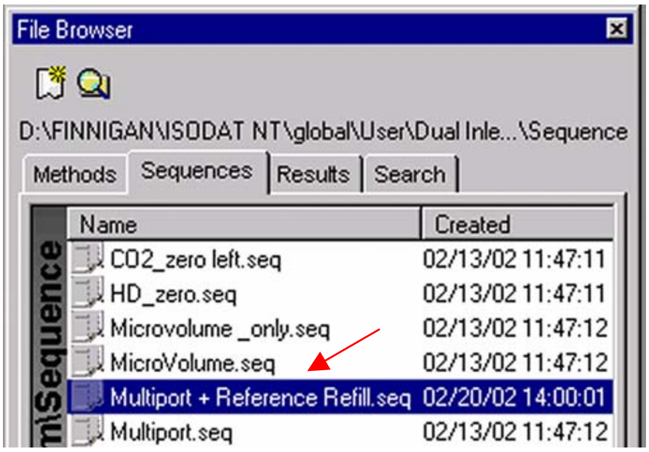
- On the File Browser, select the ***Sequences tab*** (default: Methods tab).



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

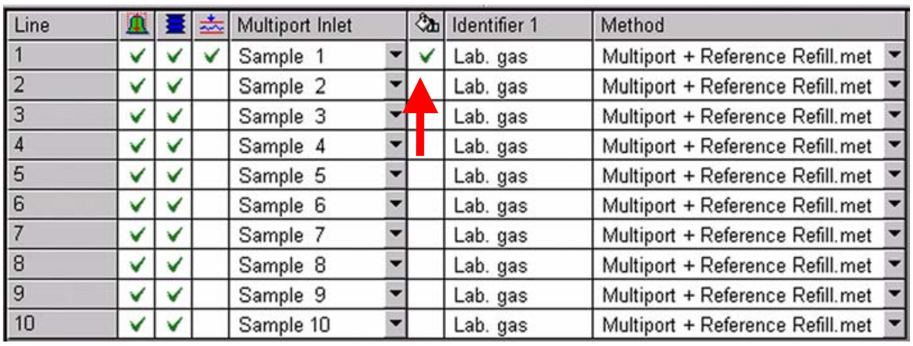
Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01

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



D:\FINNIGAN\ISODAT NT\global\User\Dual Inle...\Sequence

Name	Created
CO2_zero left.seq	02/13/02 11:47:11
HD_zero.seq	02/13/02 11:47:11
Microvolume _only.seq	02/13/02 11:47:12
MicroVolume.seq	02/13/02 11:47:12
Multiport + Reference Refill.seq	02/20/02 14:00:01
Multiport.seq	02/13/02 11:47:12

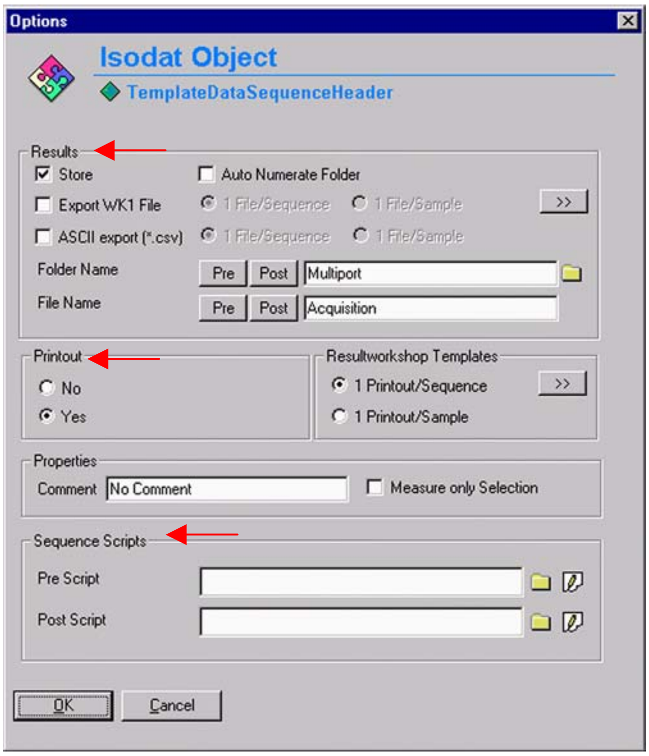


Line				Multiport Inlet		Identifier 1	Method
1	✓	✓	✓	Sample 1	✓	Lab. gas	Multiport + Reference Refill.met
2	✓	✓		Sample 2		Lab. gas	Multiport + Reference Refill.met
3	✓	✓		Sample 3		Lab. gas	Multiport + Reference Refill.met
4	✓	✓		Sample 4		Lab. gas	Multiport + Reference Refill.met
5	✓	✓		Sample 5		Lab.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

- In the ***Method*** column, the Method chosen in Chapter 4.7.2 occurs as default ("Multiport + Reference Refill.met").
- Normally, the Sequence List needs not to be edited further. It is possible, however, e.g. to select another Method from the "Method" column or to modify the "Multiport Inlet" column.
- The ***Multiport Inlet*** column defines the Multiport Inlet Port from which a particular sample is taken (e.g. Sample 2 enters via Multiport Inlet Port 2).
  If enough sample is available, it is possible to measure repeatedly out of the same port.

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

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



Options

Isodat Object

TemplateDataSequenceHeader

Results

☑ Store ☐ Auto Numerate Folder

☐ Export WK1 File ● 1 File/Sequence ○ 1 File/Sample >>

☐ ASCII export (*.csv) ● 1 File/Sequence ○ 1 File/Sample

Folder Name Pre Post Multiport

File Name Pre Post Acquisition

Printout ○ No ● Yes

Resultworkshop Templates ● 1 Printout/Sequence ○ 1 Printout/Sample >>

Properties

Comment No Comment ☐ Measure only Selection

Sequence Scripts

Pre Script

Post Script

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

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

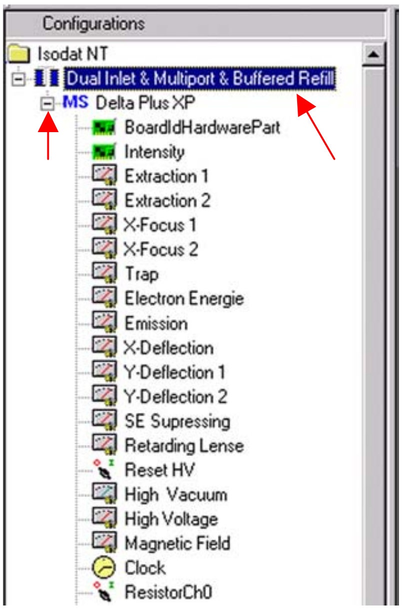
4.8 DUAL INLET MEASUREMENT INCLUDING MULTIPORT AND BUFFERED REFILL

4.8.1 DEFINING A CONFIGURATION

Configurator

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

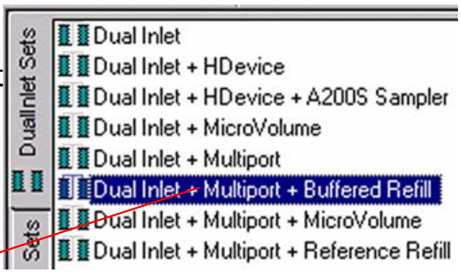
- Add a new Configuration using the ***Add Configuration*** button.

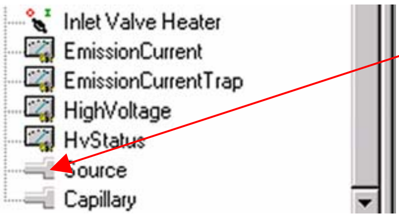


- Give it a significant name, e.g. ***“Dual Inlet & Multiport & Buffered Refill”***.

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

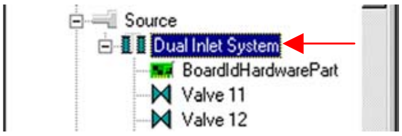
- On the right pane, select ***Dual Inlet + Multiport + Buffered Refill***.



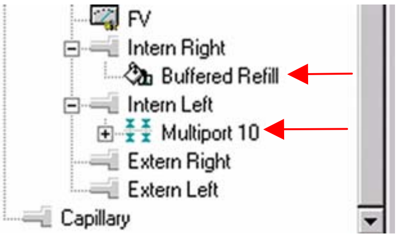


- Drag it to the Source port of the new Configuration.

A ⊞ sign appears:



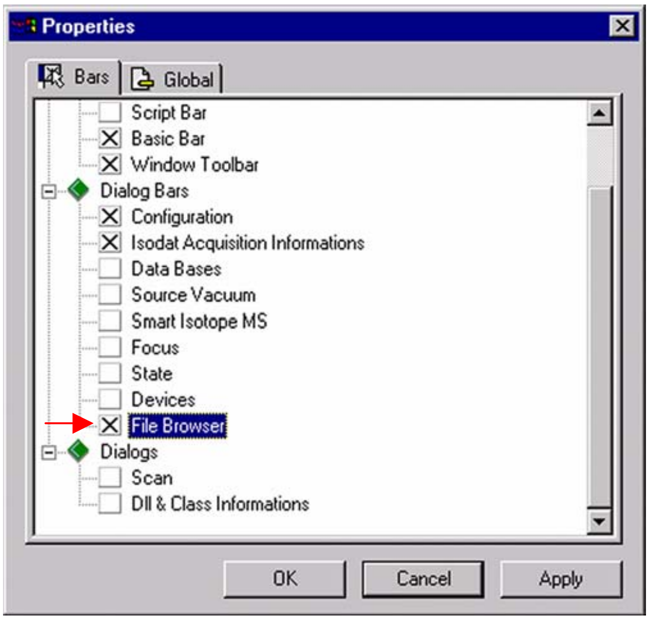
- ➢ The ***Dual Inlet*** device has been attached to the ***Source***.



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

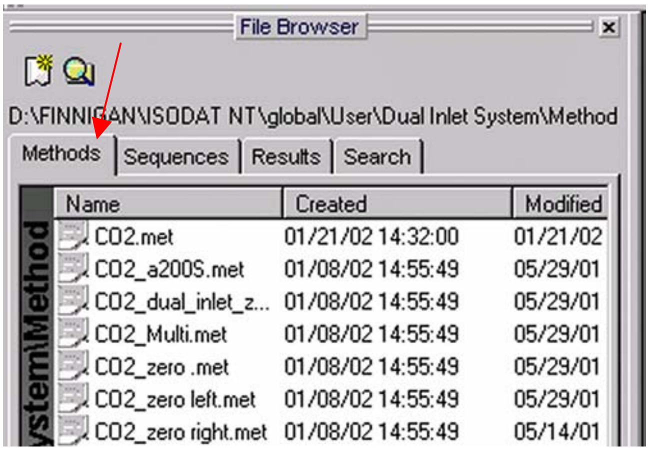
4.8.2 USING A PREDEFINED METHOD

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



☐ Script Bar
☒ Basic Bar
☒ Window Toolbar
Dialog Bars
  ☒ Configuration
  ☒ Isodat Acquisition Informations
  ☐ Data Bases
  ☐ Source Vacuum
  ☐ Smart Isotope MS
  ☐ Focus
  ☐ State
  ☐ Devices
  ☒ File Browser
Dialogs
  ☐ Scan
  ☐ Dll & Class Informations

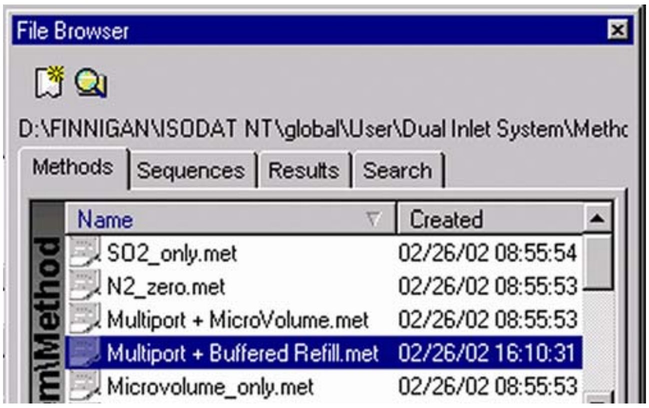
- Activate the ***File Browser*** check box.
- Click ***OK***.
- On the File Browser, select the ***Methods tab*** (default).



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01

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



File Browser

D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Methc

Name	Created
SO2_only.met	02/26/02 08:55:54
N2_zero.met	02/26/02 08:55:53
Multiport + MicroVolume.met	02/26/02 08:55:53
Multiport + Buffered Refill.met	02/26/02 16:10:31
Microvolume_only.met	02/26/02 08:55:53

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

Isodat Acquisition

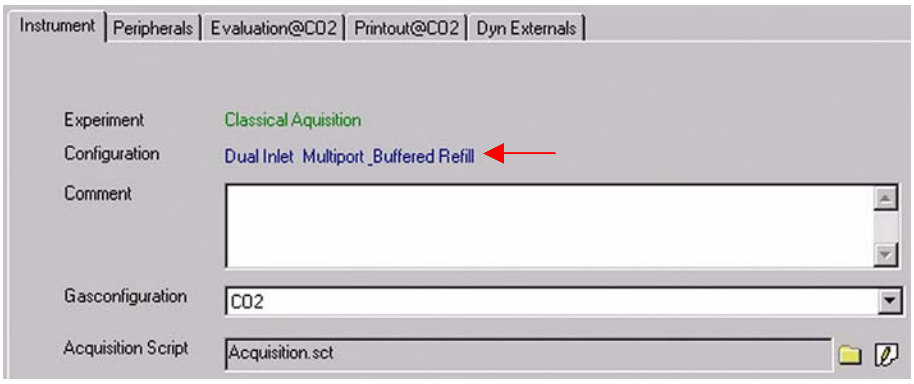
Standard values different to Standard Database!

Actualize now to new values!

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

Instrument tab

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



Experiment Classical Aquisition

Configuration Dual Inlet Multiport _Buffered Refill

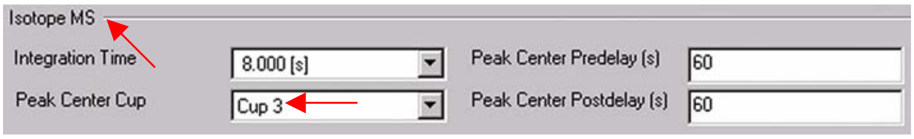
Comment

Gasconfiguration CO2

Acquisition Script Acquisition.sct

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

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


Isotope MS

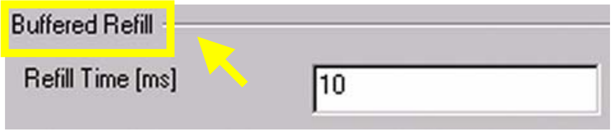
Integration Time 8.000 [s]

Peak Center Cup Cup 3

Peak Center Predelay [s] 60

Peak Center Postdelay [s] 60

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

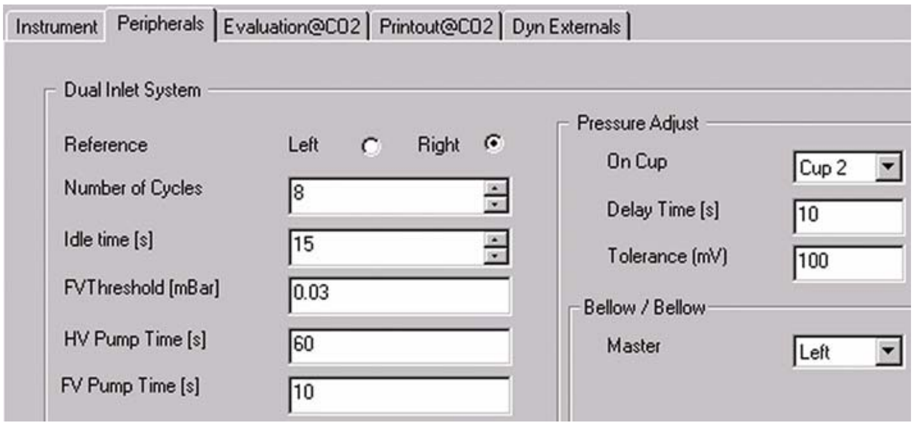

Buffered Refill

Refill Time [ms] 10

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

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

Peripherals tab



Dual Inlet System

Reference Left ○ Right ●

Number of Cycles 8

Idle time [s] 15

FVThreshold [mBar] 0.03

HV Pump Time [s] 60

FV Pump Time [s] 10

Pressure Adjust

On Cup: Cup 2

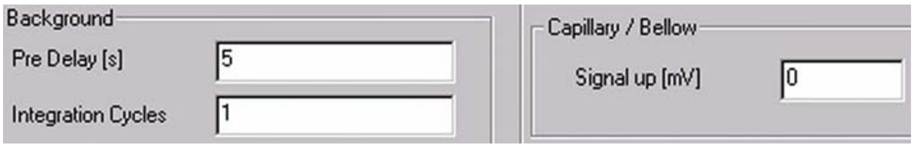
Delay Time [s] 10

Tolerance (mV) 100

Bellow / Bellow

Master: Left

For details see Chapter 4.3.2.



Background

Pre Delay [s] 5

Integration Cycles 1

Capillary / Bellow

Signal up [mV] 0

For details see Chapter 4.3.2.

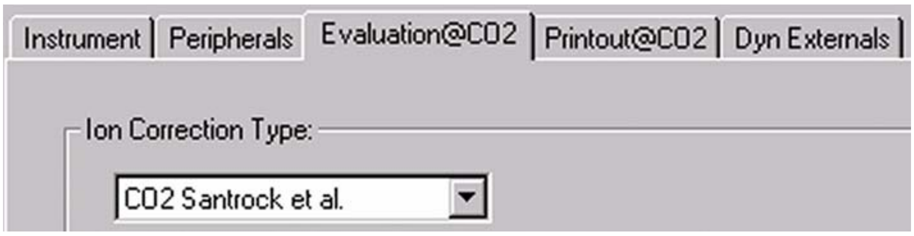
Multiport

Expansion Threshold [mBar] 70

Gas Transfer Time [s] 60

For details see Chapter 4.3.2.

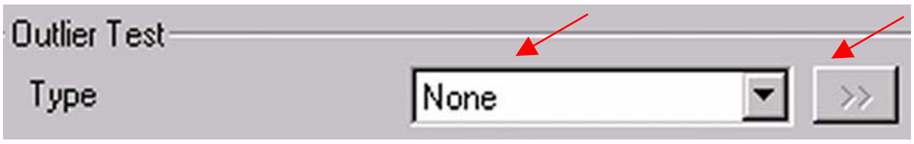
Evaluation tab



Ion Correction Type:

CO2 Santrock et al.

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



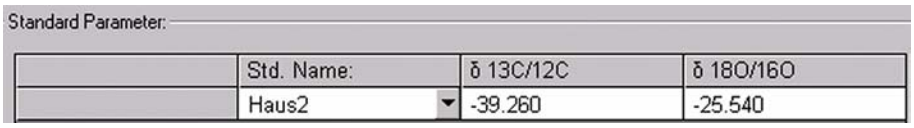
Outlier Test

Type None

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

Extended Parameters

Ion Correction Location Internal

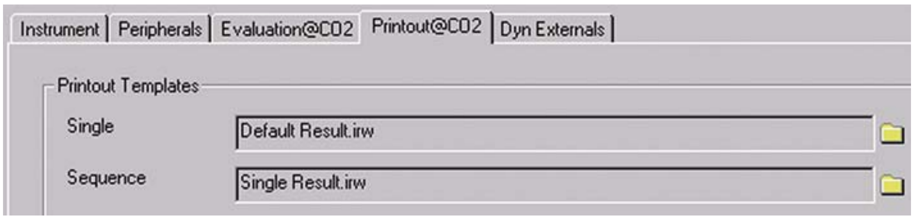


Standard Parameter:

	Std. Name:	δ 13C/12C	δ 18O/16O
	Haus2	-39.260	-25.540

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

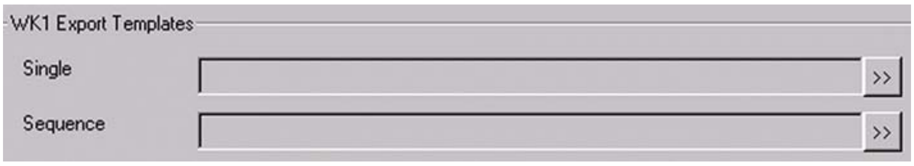
Printout tab



Single: Default Result.irw

Sequence: Single Result.irw

For details see Chapter 4.3.2.



For details see Chapter 4.3.2.

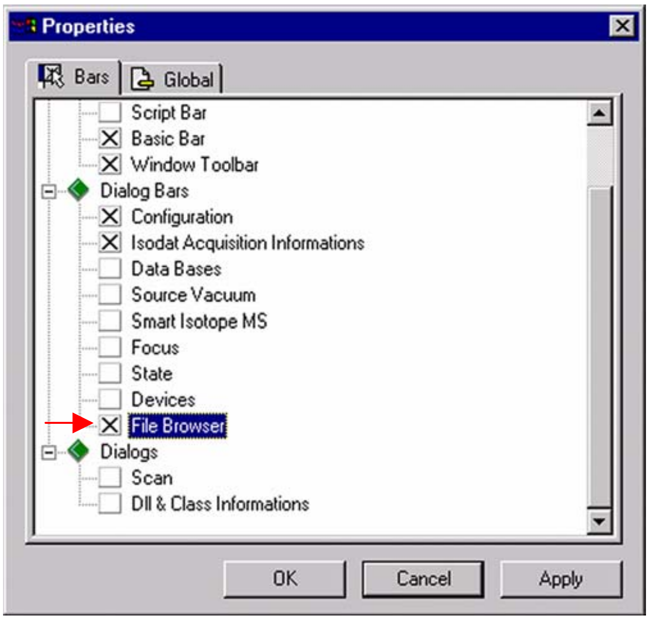
The ***Method*** for measurements using Dual Inlet in combination with a Multiport and a Buffered Refill has now been established. As next step, either use a ***predefined Sequence*** (refer to Chapter 4.8.3) or create a ***new Sequence*** (refer to Chapter 4.1.5).

4.8.3 USING A PREDEFINED SEQUENCE

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

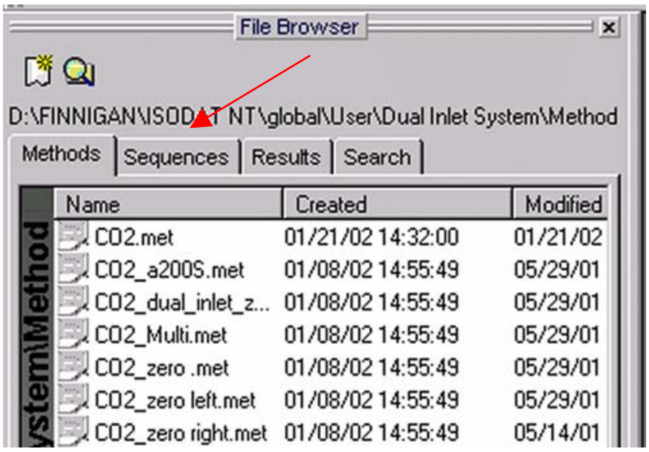
- Activate the ***File Browser*** check box.

- Click ***OK***.



☐ Script Bar
☒ Basic Bar
☒ Window Toolbar
Dialog Bars
☒ Configuration
☒ Isodat Acquisition Informations
☐ Data Bases
☐ Source Vacuum
☐ Smart Isotope MS
☐ Focus
☐ State
☐ Devices
☒ File Browser
Dialogs
☐ Scan
☐ Dll & Class Informations

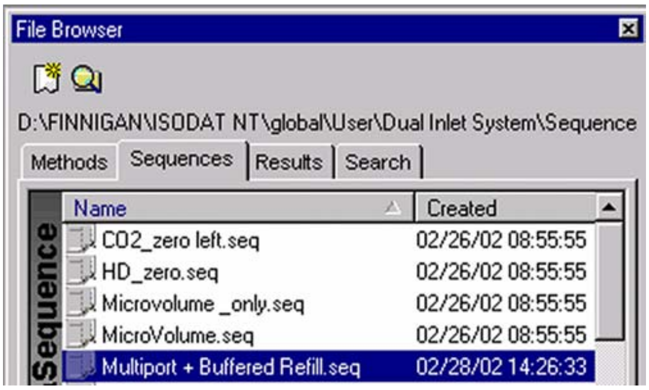
- On the File Browser, select the ***Sequences tab*** (default: Methods tab).



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01

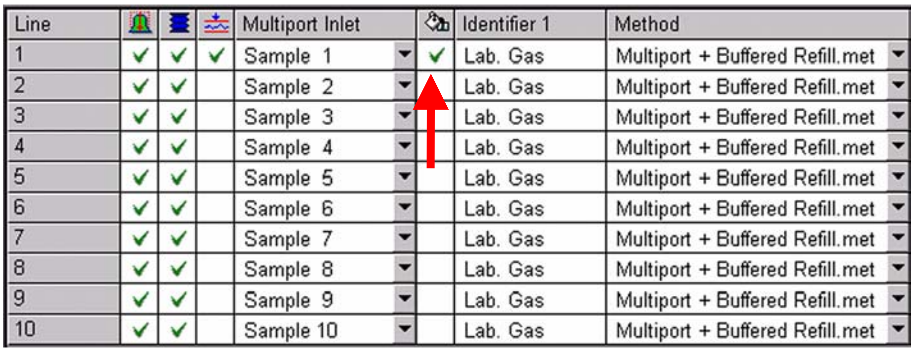
- From the predefined Sequences choose ***Multiport + Buffered Refill.seq*** by double-click.



File Browser

D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Sequence

Name	Created
CO2_zero left.seq	02/26/02 08:55:55
HD_zero.seq	02/26/02 08:55:55
Microvolume _only.seq	02/26/02 08:55:55
MicroVolume.seq	02/26/02 08:55:55
Multiport + Buffered Refill.seq	02/28/02 14:26:33



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

- In the ***Method*** column, the Method chosen in Chapter 4.8.2 occurs as default ("Multiport + Buffered Refill.met").

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

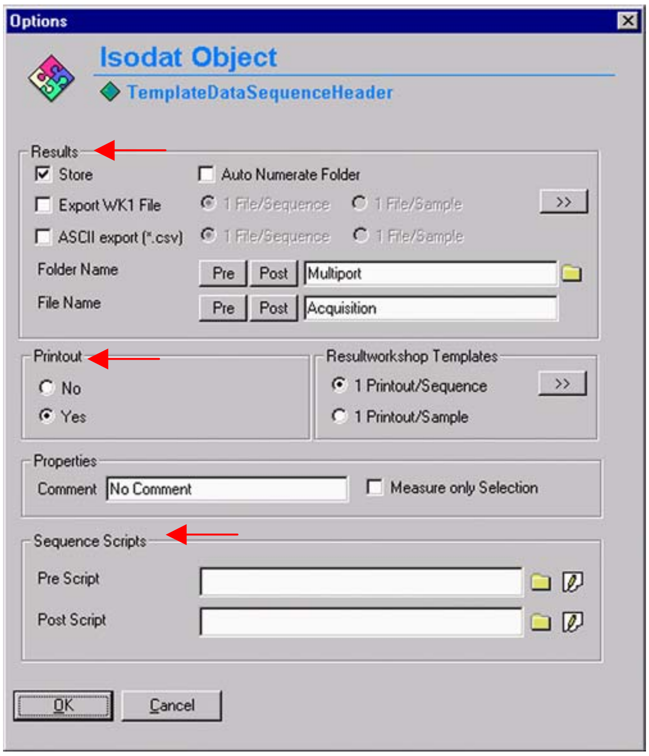
- The ***Multiport Inlet*** column defines the Multiport Inlet Port from which a particular sample is taken (e.g. Sample 2 enters via Multiport Inlet Port 2).
  If enough sample is available, it is possible to measure repeatedly out of the same port.

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

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

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

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



Options

Isodat Object

TemplateDataSequenceHeader

Results
- ☑ Store
- ☐ Auto Numerate Folder
- ☐ Export WK1 File — ● 1 File/Sequence ○ 1 File/Sample
- ☐ ASCII export (*.csv) — ● 1 File/Sequence ○ 1 File/Sample
- Folder Name: Multiport
- File Name: Acquisition

Printout
- ○ No
- ● Yes

Resultworkshop Templates
- ● 1 Printout/Sequence
- ○ 1 Printout/Sample

Properties
- Comment: No Comment
- ☐ Measure only Selection

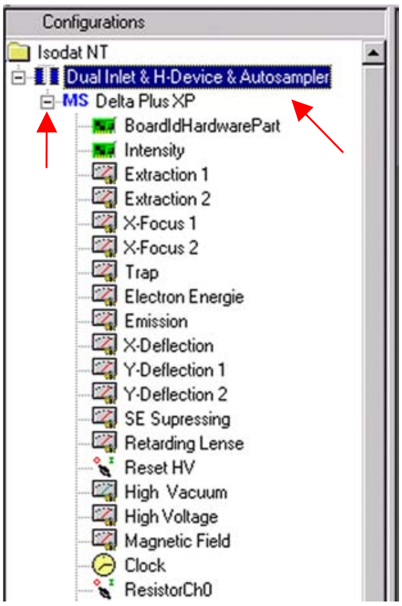
Sequence Scripts
- Pre Script:
- Post Script:

4.9 DUAL INLET MEASUREMENT INCLUDING H-DEVICE AND AUTOSAMPLER A200S

4.9.1 DEFINING A CONFIGURATION

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

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

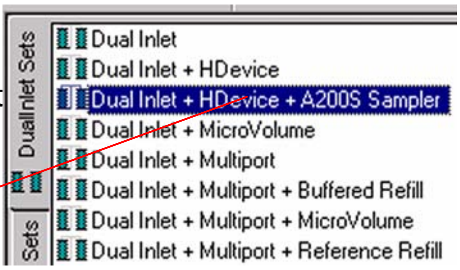


Configurations
Isodat NT
Dual Inlet & H-Device & Autosampler
MS Delta Plus XP
BoardIdHardwarePart
Intensity
Extraction 1
Extraction 2
X-Focus 1
X-Focus 2
Trap
Electron Energie
Emission
X-Deflection
Y-Deflection 1
Y-Deflection 2
SE Supressing
Retarding Lense
Reset HV
High Vacuum
High Voltage
Magnetic Field
Clock
ResistorCh0

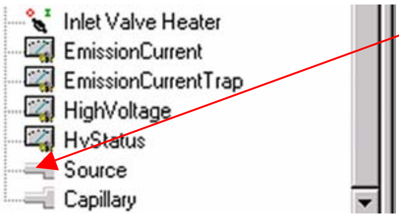
- Give it a significant name, e.g. ***“Dual Inlet & HDevice & Autosampler”***.

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

- On the right pane, select ***Dual Inlet + HDevice + A200S Sampler***.



Dual Inlet
Dual Inlet + HDevice
Dual Inlet + HDevice + A200S Sampler
Dual Inlet + MicroVolume
Dual Inlet + Multiport
Dual Inlet + Multiport + Buffered Refill
Dual Inlet + Multiport + MicroVolume
Dual Inlet + Multiport + Reference Refill

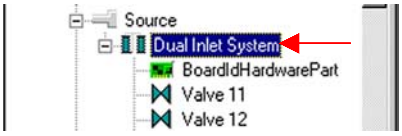


Inlet Valve Heater
EmissionCurrent
EmissionCurrentTrap
HighVoltage
HvStatus
Source
Capillary

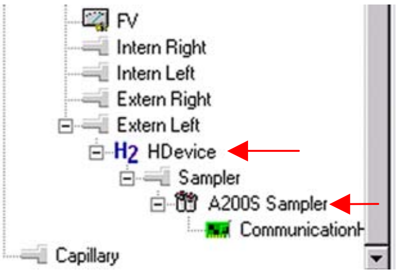
- Drag it to the Source port of the new Configuration.

A ⊞ sign appears:

HvStatus
Source
Capillary



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



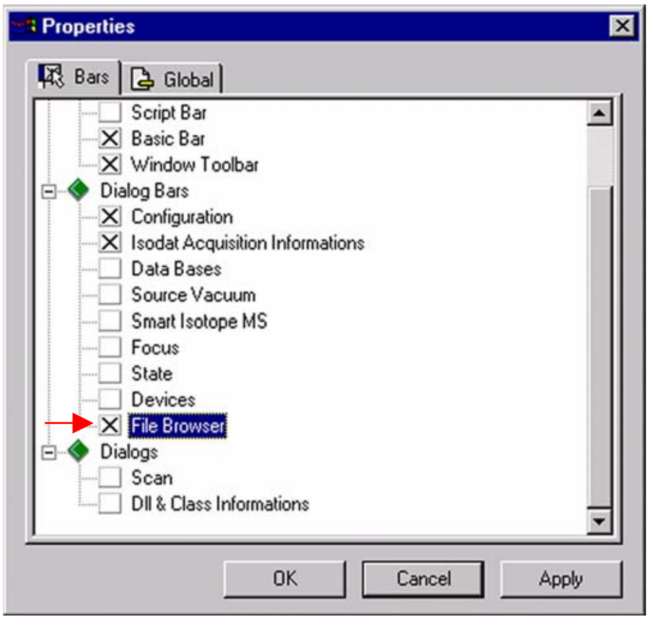
- The ***H-Device*** has been attached to the ***Extern Left*** Port.
- The ***A200S Autosampler*** has been attached to the ***Sampler*** Port.
- Close the Configurator window.

  All settings will be saved automatically.

4.9.2 USING A PREDEFINED METHOD

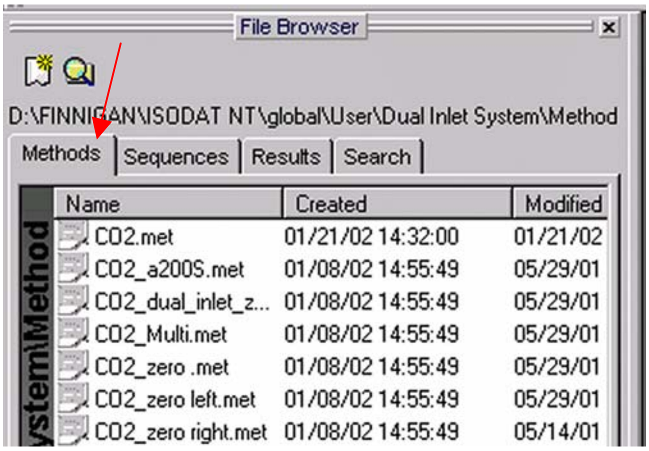
- ➢ If the File Browser cannot be seen, press the ***Options*** button.

- ➢ Activate the ***File Browser*** check box.
- ➢ Click ***OK***.



☐ Script Bar
☒ Basic Bar
☒ Window Toolbar
Dialog Bars
☒ Configuration
☒ Isodat Acquisition Informations
☐ Data Bases
☐ Source Vacuum
☐ Smart Isotope MS
☐ Focus
☐ State
☐ Devices
☒ File Browser
Dialogs
☐ Scan
☐ Dll & Class Informations

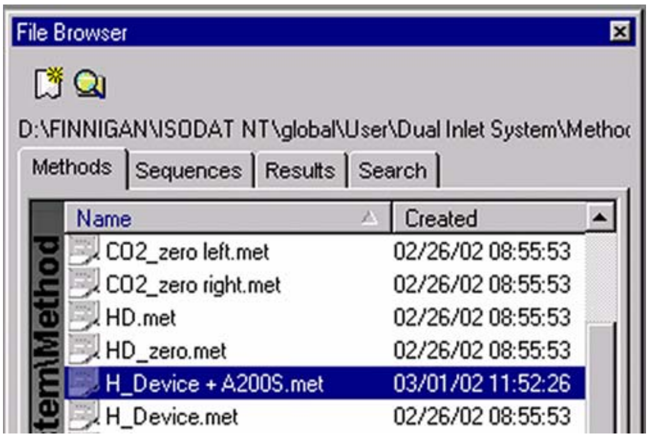
- ➢ On the File Browser, select the ***Methods tab*** (default).



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01

- From the predefined Methods choose a suitable Method (e.g. ***"H_Device + A200S.met"***) by double-click.



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Metho

Name	Created
CO2_zero left.met	02/26/02 08:55:53
CO2_zero right.met	02/26/02 08:55:53
HD.met	02/26/02 08:55:53
HD_zero.met	02/26/02 08:55:53
H_Device + A200S.met	03/01/02 11:52:26
H_Device.met	02/26/02 08:55:53

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

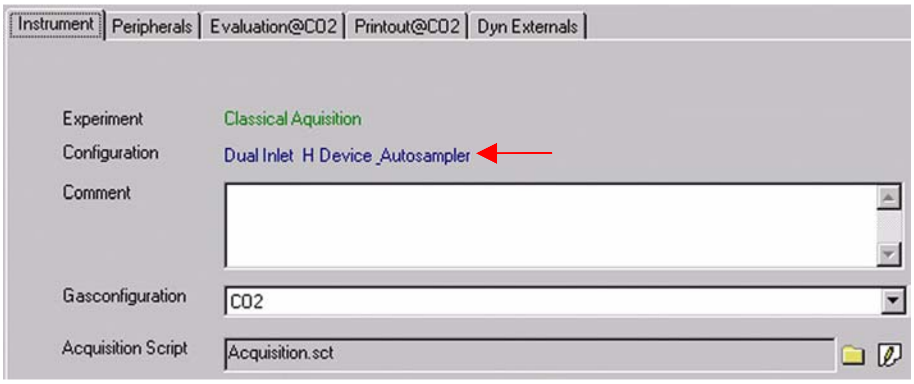
Standard values different to Standard Database!

Actualize now to new values!

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

Instrument tab

The ***Instrument tab*** of the Method "H_Device + A200S.met" appears.



Experiment Classical Aquisition

Configuration Dual Inlet H Device Autosampler

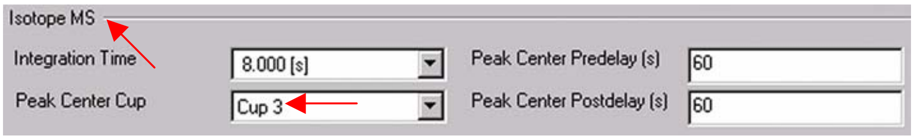
Comment

Gasconfiguration CO2

Acquisition Script Acquisition.sct

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

<u>NOTE:</u> *It should only be edited by users trained on script editing.*



Isotope MS

Integration Time: 8.000 [s]

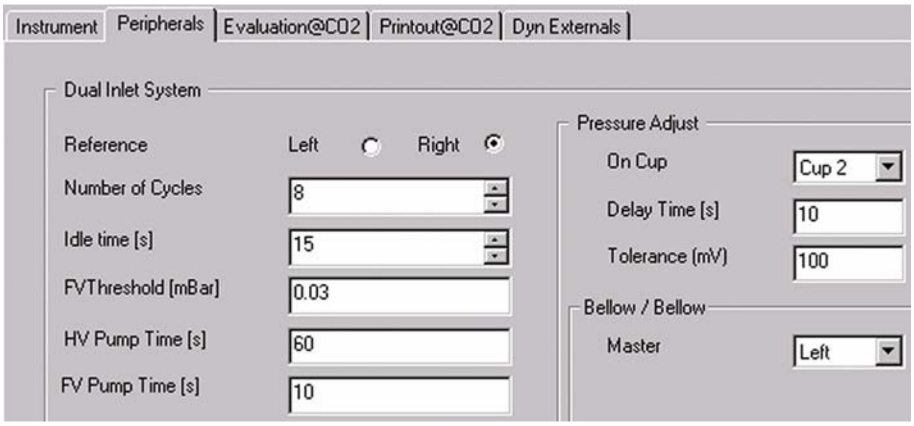
Peak Center Cup: Cup 3

Peak Center Predelay [s]: 60

Peak Center Postdelay [s]: 60

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

Peripherals tab



Dual Inlet System

Reference: Left ○ Right ●

Number of Cycles: 8

Idle time [s]: 15

FVThreshold [mBar]: 0.03

HV Pump Time [s]: 60

FV Pump Time [s]: 10

Pressure Adjust

On Cup: Cup 2

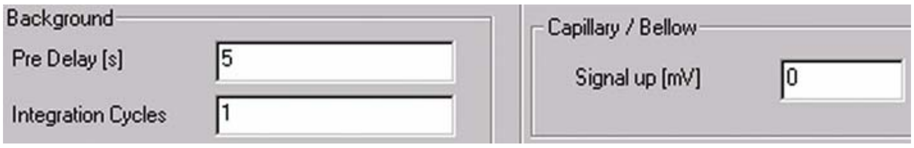
Delay Time [s]: 10

Tolerance (mV): 100

Bellow / Bellow

Master: Left

For details see Chapter 4.3.2.



Background

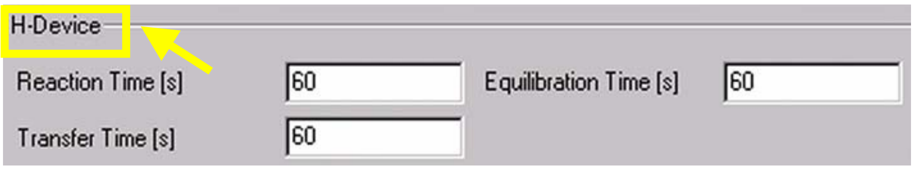
Pre Delay [s]: 5

Integration Cycles: 1

Capillary / Bellow

Signal up [mV]: 0

For details see Chapter 4.3.2.



H-Device

Reaction Time [s]: 60

Equilibration Time [s]: 60

Transfer Time [s]: 60

NOTE: ***The above information is specific for H-Device applications.***

➢ ***Reaction Time***:	Time for the injected substance to react with chromium (e.g. 60 s). Reaction time differs between substances. Water only needs 20 to 60 s to react, whereas organic compounds need longer times, which must be determined empirically.

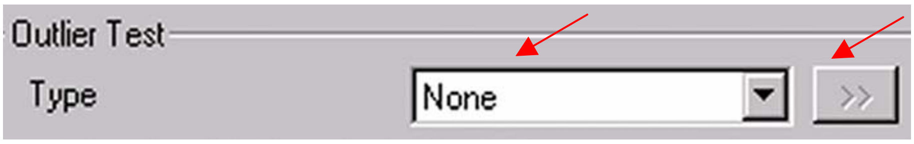
⚠ **NOTE:** ***Valve 11 must be closed!***

➢ ***Transfer Time***:	Time for the gas to expand from the expansion volume into the bellow (e.g. 60 s).
➢ ***Equilibration Time***:	Valve 11 is opened and the gas mixture is allowed to expand into the intermediate or expansion volume (connecting metal hose plus valve body). An equilibration time of 30 s is recommended for 800 ºC to 850 ºC. For other temperatures, this needs to be tested empirically.

Evaluation tab

Ion Correction Type: CO2 Santrock et al.

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



Outlier Test

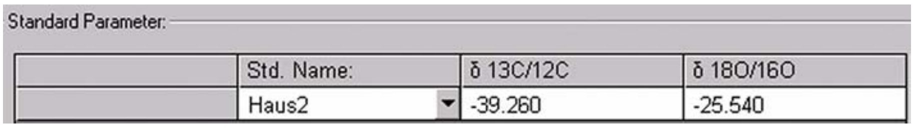
Type: None

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

Extended Parameters

Ion Correction Location: Internal

For details see Chapter 4.3.2.

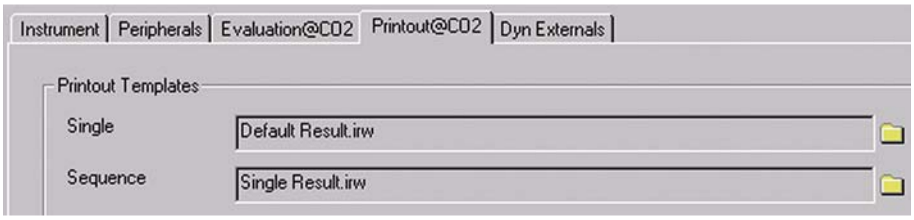


Standard Parameter:

	Std. Name:	δ 13C/12C	δ 18O/16O
	Haus2	-39.260	-25.540

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

Printout tab

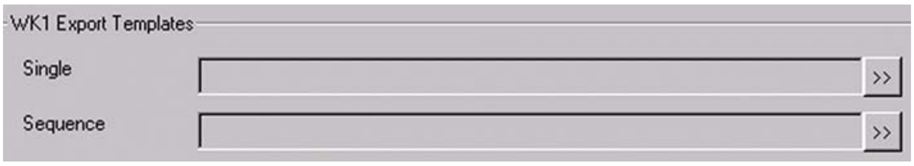


Printout Templates

Single: Default Result.irw

Sequence: Single Result.irw

For details see Chapter 4.3.2.



For details see Chapter 4.3.2.

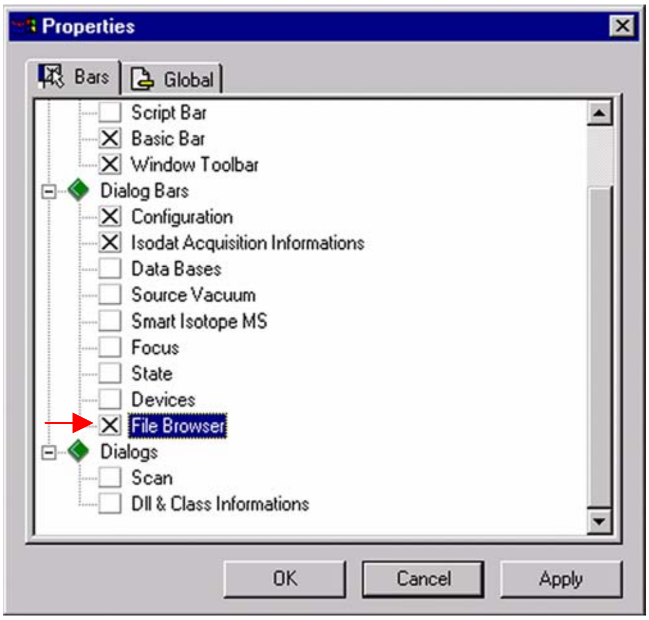
The ***Method*** for measurements using Dual Inlet in combination with a H-Device and an A200S Autosampler has now been established. As next step, either use a ***predefined Sequence*** (refer to Chapter 4.9.3) or create a ***new Sequence*** (refer to Chapter 4.1.5).

4.9.3 USING A PREDEFINED SEQUENCE

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

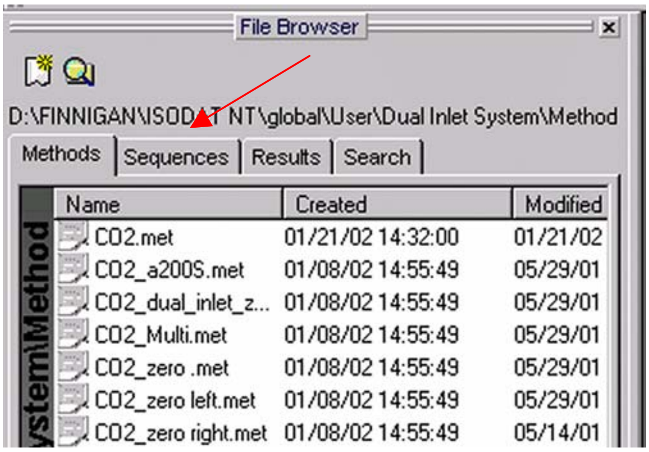
- Activate the ***File Browser*** check box.

- Click ***OK***.



☐ Script Bar
☒ Basic Bar
☒ Window Toolbar
Dialog Bars
  ☒ Configuration
  ☒ Isodat Acquisition Informations
  ☐ Data Bases
  ☐ Source Vacuum
  ☐ Smart Isotope MS
  ☐ Focus
  ☐ State
  ☐ Devices
  ☒ File Browser
Dialogs
  ☐ Scan
  ☐ Dll & Class Informations

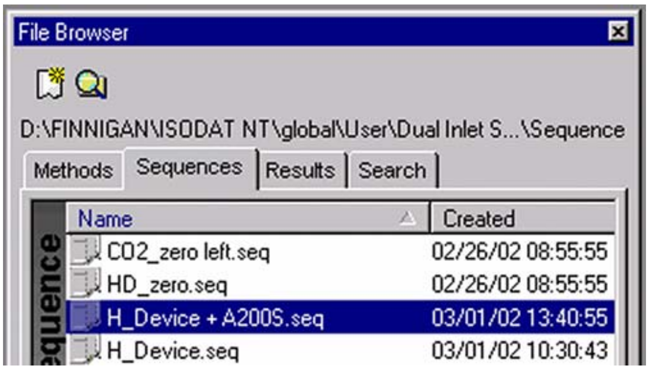
- On the File Browser, select the ***Sequences tab*** (default: Methods tab).



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet System\Method

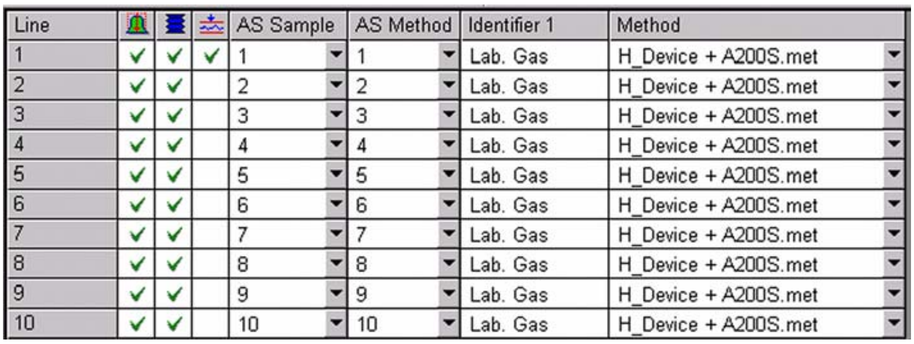
Name	Created	Modified
CO2.met	01/21/02 14:32:00	01/21/02
CO2_a200S.met	01/08/02 14:55:49	05/29/01
CO2_dual_inlet_z...	01/08/02 14:55:49	05/29/01
CO2_Multi.met	01/08/02 14:55:49	05/29/01
CO2_zero .met	01/08/02 14:55:49	05/29/01
CO2_zero left.met	01/08/02 14:55:49	05/29/01
CO2_zero right.met	01/08/02 14:55:49	05/14/01

- From the predefined Sequences choose a suitable one (e.g. ***H_Device + A200S.seq***) by double-click.



D:\FINNIGAN\ISODAT NT\global\User\Dual Inlet S...\Sequence

Name	Created
C02_zero left.seq	02/26/02 08:55:55
HD_zero.seq	02/26/02 08:55:55
H_Device + A200S.seq	03/01/02 13:40:55
H_Device.seq	03/01/02 10:30:43



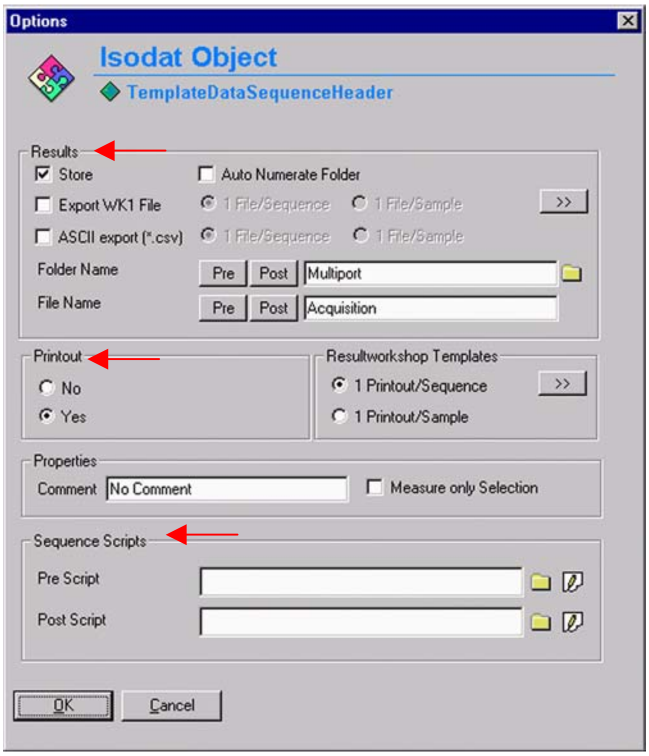
Line				AS Sample	AS Method	Identifier 1	Method
1	✓	✓	✓	1	1	Lab. Gas	H_Device + A200S.met
2	✓	✓		2	2	Lab. Gas	H_Device + A200S.met
3	✓	✓		3	3	Lab. Gas	H_Device + A200S.met
4	✓	✓		4	4	Lab. Gas	H_Device + A200S.met
5	✓	✓		5	5	Lab. Gas	H_Device + A200S.met
6	✓	✓		6	6	Lab. Gas	H_Device + A200S.met
7	✓	✓		7	7	Lab. Gas	H_Device + A200S.met
8	✓	✓		8	8	Lab. Gas	H_Device + A200S.met
9	✓	✓		9	9	Lab. Gas	H_Device + A200S.met
10	✓	✓		10	10	Lab. Gas	H_Device + A200S.met

- Normally, the Sequence List needs not to be edited further. It is possible, however, e.g. to select another Method from the “Method” pulldown menu.

- ***AS Sample***: Type in the sample position in the Autosampler tray.
- ***AS Method***: Select Autosampler Method.
- ***Identifier 1***: Edit text to identify the sample.
- ***Method***: Select IRMS Method. The Method chosen in Chapter 4.9.2 occurs as default (“H_Device + A200S.met”).

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

- Define parameters for:
  - Results Export
  - Printout
  - Sequence Scripts
- Press ***OK*** to start Sequence Acquisition.



Results
☑ Store
☐ Auto Numerate Folder
☐ Export WK1 File ● 1 File/Sequence ○ 1 File/Sample
☐ ASCII export (*.csv) ● 1 File/Sequence ○ 1 File/Sample
Folder Name: Multiport
File Name: Acquisition

Printout
○ No
● Yes

Resultworkshop Templates
● 1 Printout/Sequence
○ 1 Printout/Sample

Properties
Comment: No Comment
☐ Measure only Selection

Sequence Scripts
Pre Script:
Post Script:

ISODAT NT

OPERATING MANUAL

5

CONTINUOUS FLOW MEASUREMENTS

Issue 06/2002

Thermo Finnigan

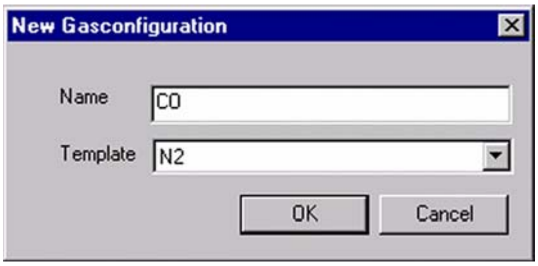
5.1 OXYGEN MEASUREMENT

5.1.1 DEFINING A GAS CONFIGURATION

Prior to defining this Gas Configuration ensure the connected IRMS has the cups set for the simultaneous detection of masses 28, 29 and 30 and Mass Calibration for these cups has already been performed.

For an ^{18}O measurement, a new Gasconfiguration has to be created for the masses 28 ($^{12}C^{16}O$), 29 ($^{13}C^{16}O$) and 30 ($^{12}C^{18}O$).

- Open the ***Continuous Flow*** module.
- Open the ***Gas Configuration Editor***.
- ***Add*** a Gas Configuration.

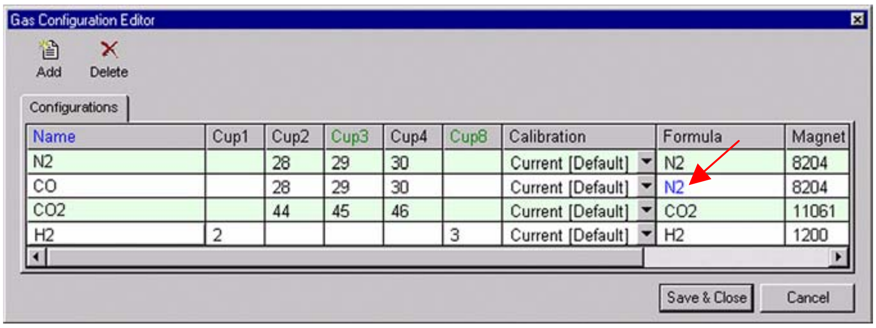


New Gasconfiguration

Name: CO

Template: N2

- Select Gas Configuration ***N2*** as ***Template***.
- Type ***CO*** for the Name.
- Confirm by ***OK***.



Gas Configuration Editor

Name	Cup1	Cup2	Cup3	Cup4	Cup8	Calibration	Formula	Magnet
N2		28	29	30		Current [Default]	N2	8204
CO		28	29	30		Current [Default]	N2	8204
CO2		44	45	46		Current [Default]	CO2	11061
H2	2				3	Current [Default]	H2	1200

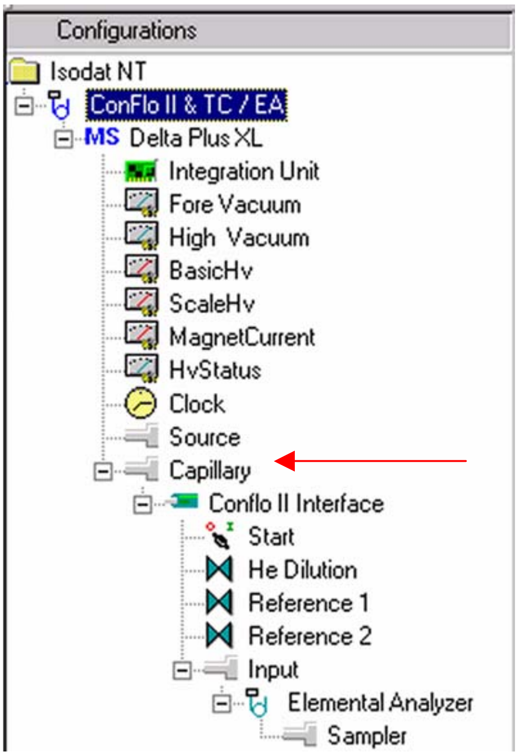
- Select a ***Calibration*** valid for the selected cups.
- Change the ***Formula*** from ***N2*** to ***CO***.
- Press ***Save & Close***.

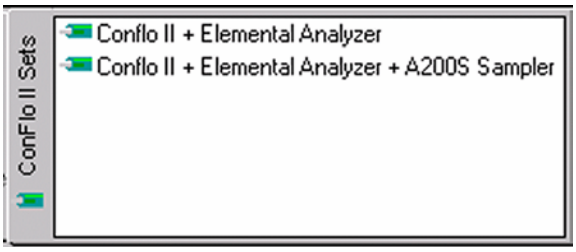
5.1.2 DEFINING A CONFIGURATION

Configurator

Before operating, a Configuration must be created in the ***Configurator***.

- Add a new Configuration with the ***“Add Configuration”*** button.
- Give it a name, e.g. “ConFlo II & TC / EA”.





- Select e.g. one of the “ConFlo II Sets” (also for ConFlo III) and drag it to the Source port or Capillary port in the new Configuration.
- Open the complete tree structure of e.g. the ConFlo II Interface to check for the attached hardware.
- Activate the ***Advanced Mode*** in the ***Edit*** menu.
- Click with the right mouse button on the ***Communication Hardware*** object to open the ***Properties*** window.
- Close the Configurator window. All settings will be saved automatically.

5.1.3 DEFINING A NEW METHOD

The Continuous Flow application software allows fully automated isotope ratio determination of oxygen of bulk samples. All parameters relevant for data acquisition of a sample are stored in a ***Method***. The following steps are needed to define a new Method.

- Open the ***Continuous Flow*** module.


ConFlo II & TC / EA

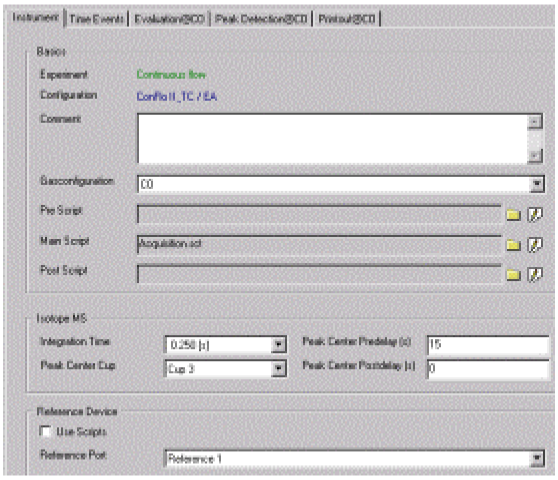
- Select the ***Configuration*** (e.g. “ConFlo II & TC/EA”).


CO

- Select the ***Gas Configuration*** for ^{18}O determination.
- Create a new Method.

The new Method is structured in Tab pages: Instrument, Time Events, Evaluation, Peak Detection, and Printout. The following Method explains the parameters of an ^{18}O Method.

Instrument



Basics
Experiment: Continuous flow
Configuration: ConFlo II_TC / EA
Comment:
Gasconfiguration: CO
Pre Script:
Main Script: Acquisition.scl
Post Script:

Isotope MS
Integration Time: 0.250 [s]
Peak Center Cup: Cup 3
Peak Center Predelay [s]: 15
Peak Center Postdelay [s]: 0

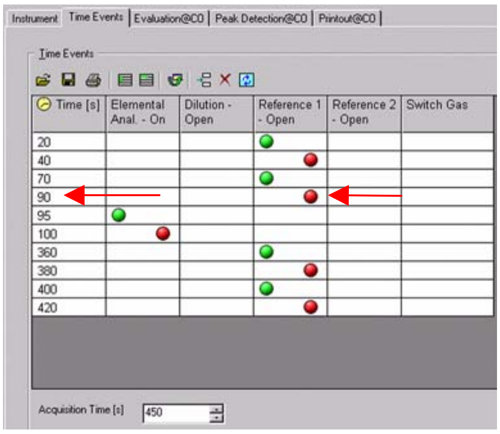
Reference Device
☐ Use Scripts
Reference Port: Reference 1

- Select ***Gas Configuration*** for ^{18}O determination (e.g. ***“CO”***).
- ***Main Script*** controls the acquisition cycle.

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

- Select the ***Peak Center Cup***, e.g. **Cup 3** for a triple collector on a Delta^{Plus} XL (narrow center cup for m/z 29).
- ***Peak Center Predelay*** is the time the system waits between activation of the reference gas and start of the peak center cycle (e.g. > **15** s).
- ***Peak Center Postdelay*** is the time the system waits between the end of the peak center cycle and the start of the data acquisition (e.g. **0** s).
- ***Integration time*** is the time integrated to form a data point triplet (e.g. **0.25** s).
- Select the ***Reference Port*** to be used for peak center (e.g. Reference 1).

Time Events



Time [s]	Elemental Anal. - On	Dilution - Open	Reference 1 - Open	Reference 2 - Open	Switch Gas
20			🟢		
40			🔴		
70			🟢		
90			🔴		
95	🟢				
100	🔴				
360			🟢		
380			🔴		
400			🟢		
420			🔴		

Acquisition Time [s] 450

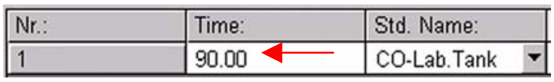
The “Time Events” list controls all valves of the selected Configuration during data acquisition.

- ***Insert lines*** using right mouse button or click on .
- Edit the ***Time*** at which the event will happen and ***double-click*** the field of a valve to set or toggle its status to active 🟢 or inactive 🔴.
- Edit the ***Acquisition Time***. The acquisition time is the end time of data acquisition. After the acquisition time, no further actions will be executed from the time events list.

Evaluation

Ion Correction Type: CO Santrock et al.

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



Nr.:	Time:	Std. Name:
1	90.00	CO-Lab.Tank

- ***Add a standard*** using the right mouse button. Enter the retention time of the standard peak(s) defined in "Time Events" which are used for calculating the corresponding delta value(s).

  If the assigned time for Standard peak detection falls in between the Peak Start and Peak Stop marks of a peak, this peak will be used for delta value calculation.

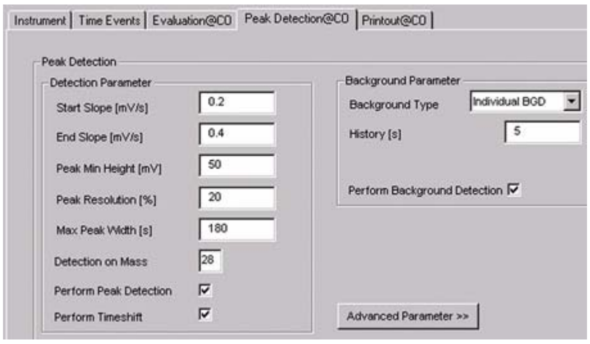
- Select a ***Std. Name*** or edit the related delta values (user defined).

The retention time must be set to the “Reference Out” value of the second CO Reference Gas pulse (in “Time Events”). This accommodates for the delay associated with the gas passage through the capillary to the IRMS.

Reference/Blank
Significant Peak Start [s] 150.000000
Significant Peak Stop [s] 220.000000
Weight Percent [%] 26.220000

- ***Reference / Blank:*** For a blank determination, a time window needs to be defined by significant peak start and stop in which the corresponding CO peak will appear.
- For ***weight percent*** determination type in the appropriate oxygen weight percent of the reference compound used for Calibration.

Peak Detection



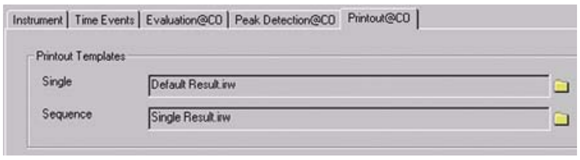
Peak Detection

Detection Parameter
Start Slope [mV/s] 0.2
End Slope [mV/s] 0.4
Peak Min Height [mV] 50
Peak Resolution [%] 20
Max Peak Width [s] 180
Detection on Mass 28
Perform Peak Detection ☑
Perform Timeshift ☑

Background Parameter
Background Type Individual BGD
History [s] 5
Perform Background Detection ☑

- Edit ***Start Slope***, ***End Slope***, and ***Peak Min. Height***. All other parameters can be kept default.
- A five point average slope of **0.2** mV/s (Start Slope) and of **0.4** mV/s (End Slope) is recommended for safe recognition of the peak boundaries. The best slope values depend on the nature of the chromatogram.
- ***Peak Min. Height*** limits the number of reported peaks. Default is **50** mV.
- ***Individual BGD*** is the default ***Background Type***.

Printout



Printout Templates
Single Default Result.irw
Sequence Single Result.irw

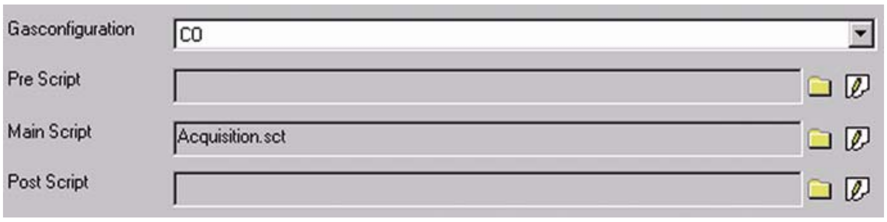
- ***Single Print*** selects a print template from the Result Workshop for an individual printout per sample.
- ***Sequence Print*** selects a print template from the Result Workshop for a reduced print per sample within a sequence summary.

5.1.4 ZERO ENRICHMENT (ST ON / OFF TEST)

We assume that the user already has working experience with the complete system. It is recommended to perform a simple check in order to test its analytical condition before measuring any samples.

1 Use the following default ***Method*** (“CO_zero.met”) as a guideline.

Instrument



Gasconfiguration: CO

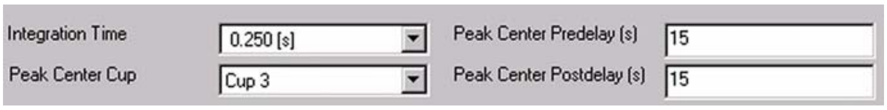
Pre Script:

Main Script: Acquisition.sct

Post Script:

Reference Port: Reference 1

Isotope MS



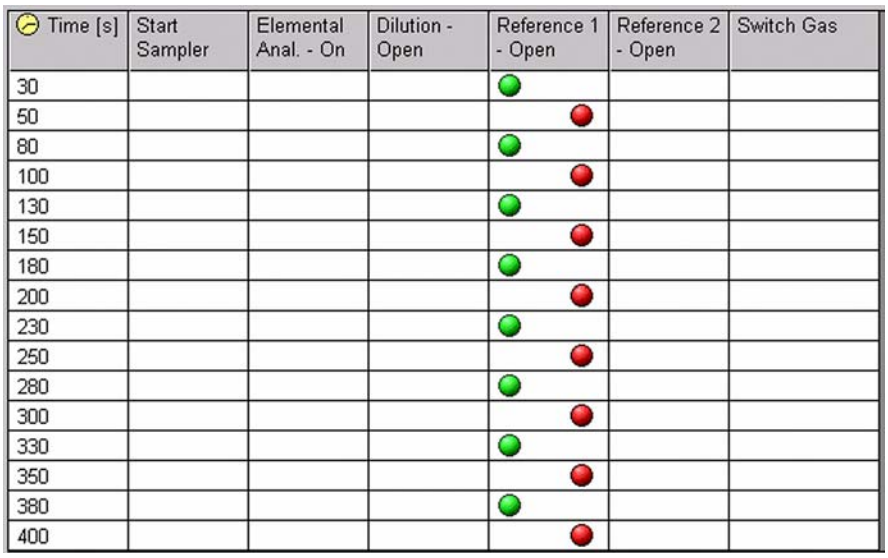
Integration Time: 0.250 [s]

Peak Center Cup: Cup 3

Peak Center Predelay [s]: 15

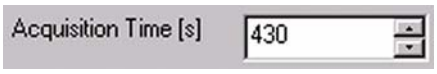
Peak Center Postdelay [s]: 15

Time Events



Time [s]	Start Sampler	Elemental Anal. - On	Dilution - Open	Reference 1 - Open	Reference 2 - Open	Switch Gas
30				🟢		
50				🔴		
80				🟢		
100				🔴		
130				🟢		
150				🔴		
180				🟢		
200				🔴		
230				🟢		
250				🔴		
280				🟢		
300				🔴		
330				🟢		
350				🔴		
380				🟢		
400				🔴		

Acq. End Time

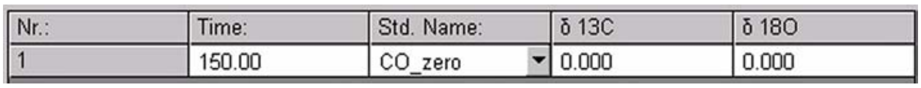


Acquisition Time [s]: 430

Evaluation

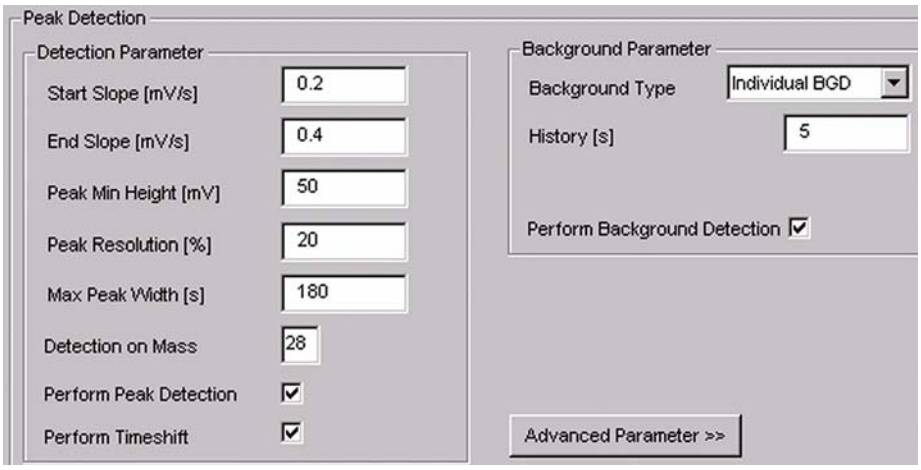
Ion Correction Type:

CO Santrock et al.



Nr.:	Time:	Std. Name:	δ 13C	δ 18O
1	150.00	CO_zero	0.000	0.000

Peak Detection



Peak Detection

Detection Parameter

Start Slope [mV/s] 0.2

End Slope [mV/s] 0.4

Peak Min Height [mV] 50

Peak Resolution [%] 20

Max Peak Width [s] 180

Detection on Mass 28

Perform Peak Detection ☑

Perform Timeshift ☑

Background Parameter

Background Type Individual BGD

History [s] 5

Perform Background Detection ☑

Printout

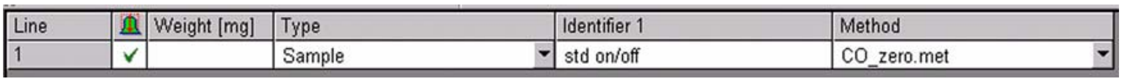
Printout Templates

Single Default Result.irw

Sequence Default Sequence Result.IRW

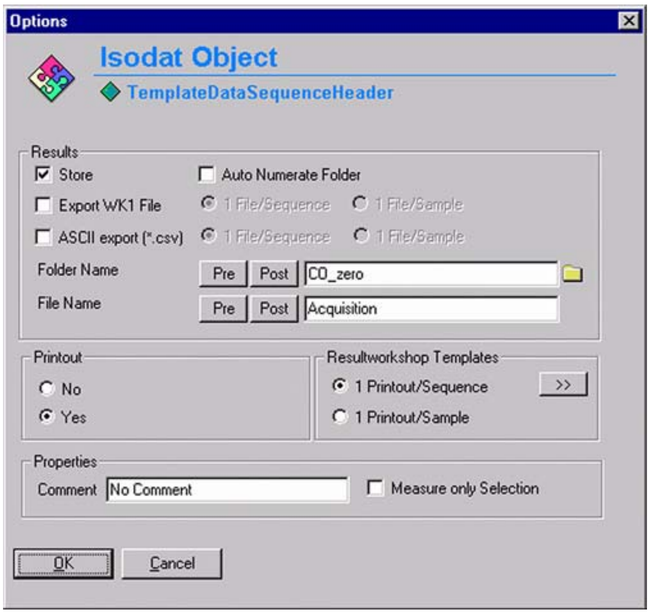
2 Set the ion intensity of mass 28 ($^{12}C^{16}O$) to approximately 3 – 4 V by opening Reference 1 and adjusting the pressure to the desired intensity.

3 Create a new ***Sequence*** or select a predefined Sequence from the File Browser.



Line	✓	Weight [mg]	Type	Identifier 1	Method
1	✓		Sample	std on/off	CO_zero.met

4 Press the Start button.



Results

☑ Store ☐ Auto Numerate Folder

☐ Export WK1 File ● 1 File/Sequence ○ 1 File/Sample

☐ ASCII export (*.csv) ● 1 File/Sequence ○ 1 File/Sample

Folder Name Pre Post CO_zero

File Name Pre Post Acquisition

Printout

○ No

● Yes

Resultworkshop Templates

● 1 Printout/Sequence >>

○ 1 Printout/Sample

Properties

Comment No Comment ☐ Measure only Selection

5 Select your destination folder.

Make your Printout decision (***Yes*** / ***No***).

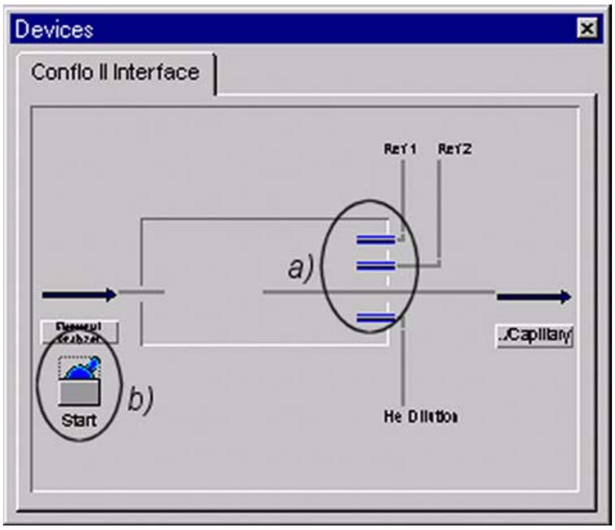
Finally, confirm by ***OK***.

5.1.5 SOLID-AUTOSAMPLER LEAK TEST

While monitoring mass 28, start the solid-autosampler manually. The intensity of mass 28 should not increase more than 20 mV. If the measured value is 1.5 times higher than expected, increase the He purge and repeat the test.

NOTE: ***a)*** Neither Reference 1 nor Reference 2 nor He dilution should be open during the test.

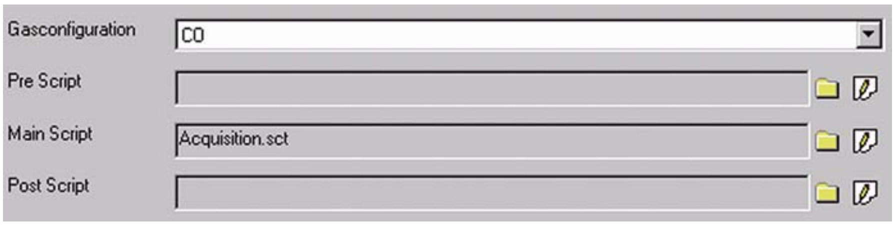
b) To activate the solid-autosampler manually, switch the symbol to the position "Start" by a mouse click.



5.1.6 HOW TO START AN OXYGEN MEASUREMENT

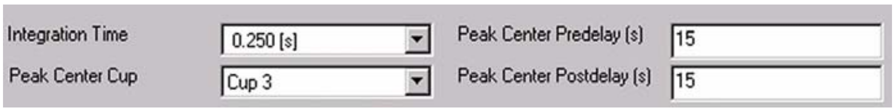
1 Define a Method

Instrument



Gasconfiguration: CO
Pre Script:
Main Script: Acquisition.sct
Post Script:

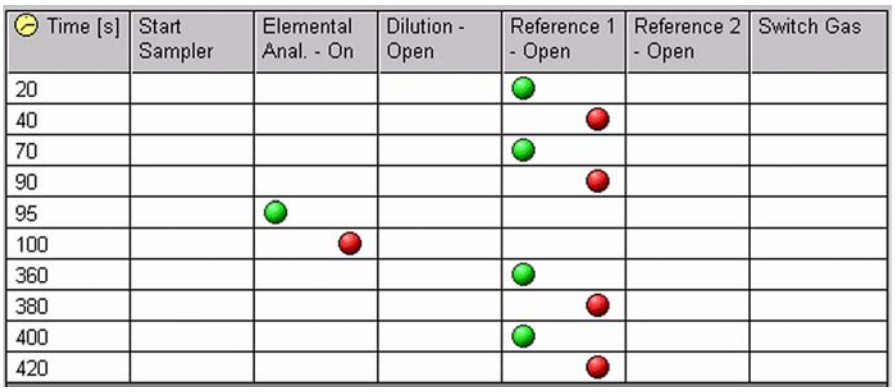
Isotope MS



Integration Time: 0.250 [s]
Peak Center Cup: Cup 3
Peak Center Predelay [s]: 15
Peak Center Postdelay [s]: 15

Reference Port: Reference 1

Time Events

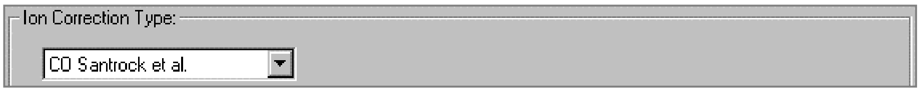


Time [s]	Start Sampler	Elemental Anal. - On	Dilution - Open	Reference 1 - Open	Reference 2 - Open	Switch Gas
20				🟢		
40				🔴		
70				🟢		
90				🔴		
95		🟢				
100		🔴				
360				🟢		
380				🔴		
400				🟢		
420				🔴		

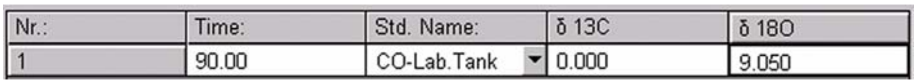
Acq. End Time

Acquisition Time [s]: 450

Evaluation



Ion Correction Type: CO Santrock et al.



Nr.:	Time:	Std. Name:	δ 13C	δ 18O
1	90.00	CO-Lab.Tank	0.000	9.050

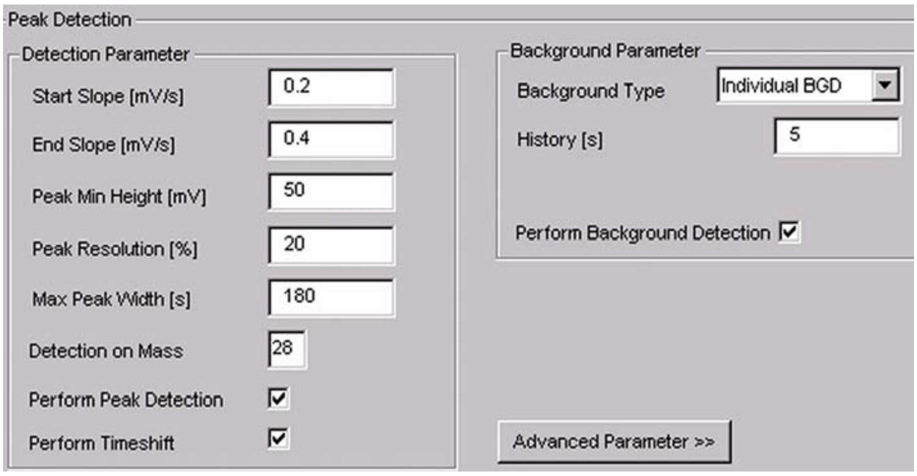
Reference/Blank

Significant Peak Start [s]: 150.000000

Significant Peak Stop [s]: 220.000000

Weight Percent [%]: 26.220000

Peak Detection



Peak Detection

Detection Parameter

Start Slope [mV/s]: 0.2

End Slope [mV/s]: 0.4

Peak Min Height [mV]: 50

Peak Resolution [%]: 20

Max Peak Width [s]: 180

Detection on Mass: 28

Perform Peak Detection ☑

Perform Timeshift ☑

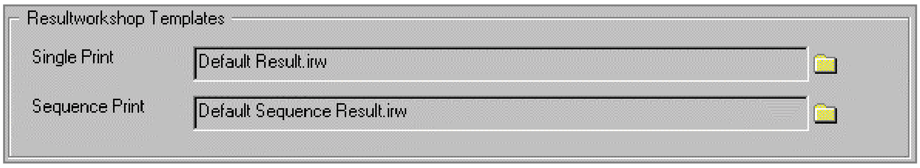
Background Parameter

Background Type: Individual BGD

History [s]: 5

Perform Background Detection ☑

Printout



Resultworkshop Templates

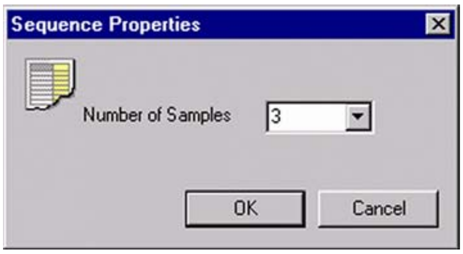
Single Print: Default Result.irw

Sequence Print: Default Sequence Result.irw

3 Place a sample (e.g. 0.285 mg of benzoic acid) in the solid-autosampler.

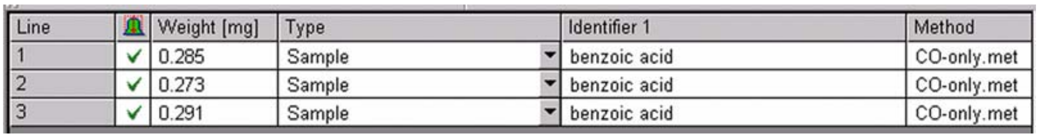
4 ***Create a new Sequence***.

5 Define the number of samples (e.g. 3).



Number of Samples: 3

6 Edit the Sequence list.

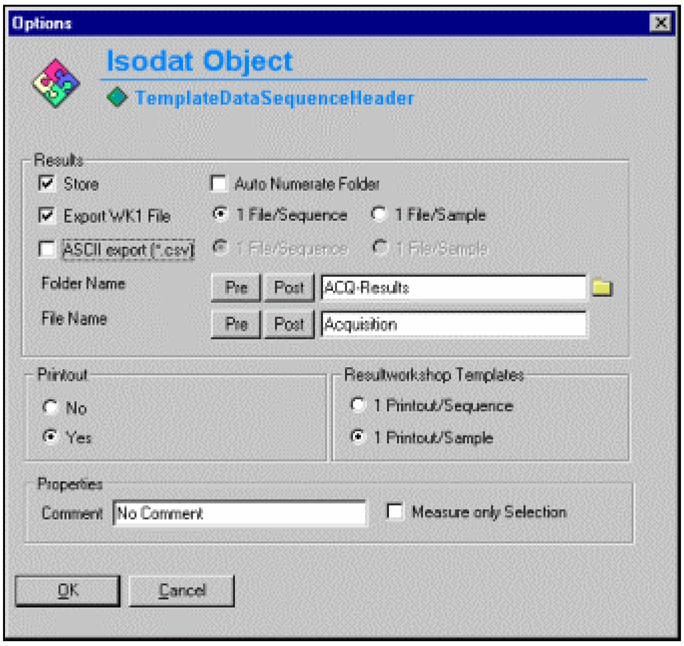


Line	✓	Weight [mg]	Type	Identifier 1	Method
1	✓	0.285	Sample	benzoic acid	CO-only.met
2	✓	0.273	Sample	benzoic acid	CO-only.met
3	✓	0.291	Sample	benzoic acid	CO-only.met

Peak Center	Enable ☑ to perform a Peak Center prior to measurement.
Identifier 1	Edit text to identify sample.
Method	Select IRMS Method.

7 Press the ***Start*** button.

8 Define Results Export and Printout parameters.



Options

Isodat Object

TemplateDataSequenceHeader

Results

☑ Store ☐ Auto Numerate Folder

☑ Export WK1 File ● 1 File/Sequence ○ 1 File/Sample

☐ ASCII export (*.csv) ● 1 File/Sequence ○ 1 File/Sample

Folder Name: ACQ-Results

File Name: Acquisition

Printout: ○ No ● Yes

Resultworkshop Templates: ○ 1 Printout/Sequence ● 1 Printout/Sample

Properties

Comment: No Comment ☐ Measure only Selection

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

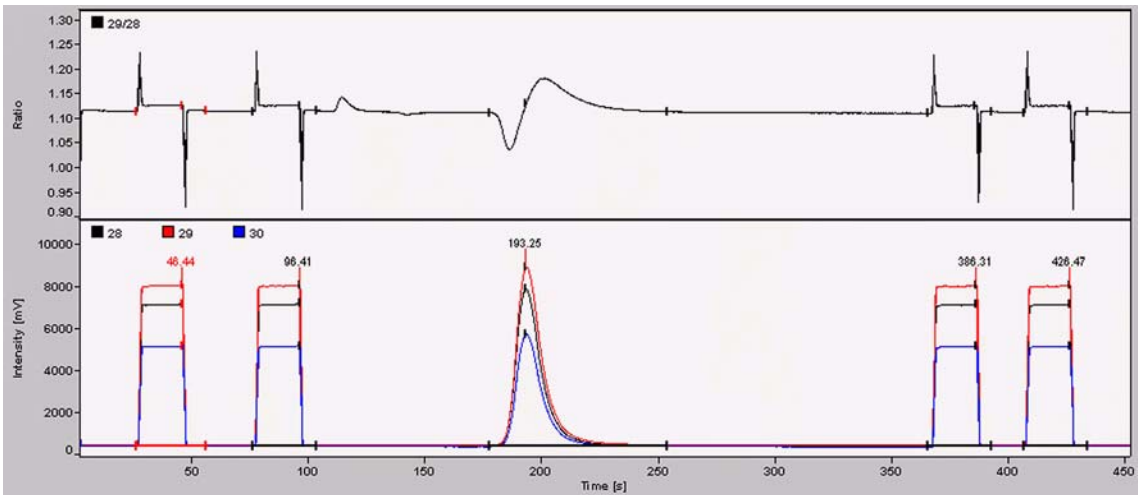
Events during Acquisition

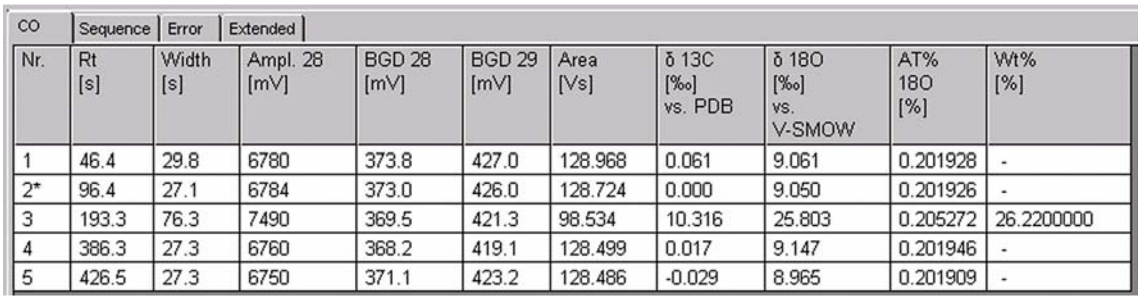
- Peak Center procedure
- First CO Reference Gas pulse activated at 20 s (duration: 20 s).
- Second CO Reference Gas pulse activated at 70 s (duration: 20 s).
  It is assigned as Standard pulse for delta value calculation.
  Refer to “Time” column in “Evaluation” tab.
- Sample is dropped into reactor.
- Sample peak appears approximately 100 s after start of reaction.
- Third CO Reference Gas pulse activated at 360 s (duration: 20 s).
- Forth CO Reference Gas pulse activated at 400 s (duration 20 s).
- Acquisition stops at 450 s.

After finishing data acquisition the printer creates a data output sheet as defined by the selected Results Workshop template (*.irw). The Results are also exported to a spreadsheet file.

C:\Finnigan\ISODAT NT\Global\User\Conflo II Interface\Results\Benzoic acid CO.CF

Line	🔔	Weight [mg]	Type	Identifier 1	Method
1	✓	0.285	Sample	benzoic acid	CO-only.met





CO | Sequence | Error | Extended

Nr.	Rt [s]	Width [s]	Ampl. 28 [mV]	BGD 28 [mV]	BGD 29 [mV]	Area [Vs]	δ 13C [‰] vs. PDB	δ 18O [‰] vs. V-SMOW	AT% 18O [%]	Wt% [%]
1	46.4	29.8	6780	373.8	427.0	128.968	0.061	9.061	0.201928	-
2*	96.4	27.1	6784	373.0	426.0	128.724	0.000	9.050	0.201926	-
3	193.3	76.3	7490	369.5	421.3	98.534	10.316	25.803	0.205272	26.2200000
4	386.3	27.3	6760	368.2	419.1	128.499	0.017	9.147	0.201946	-
5	426.5	27.3	6750	371.1	423.2	128.486	-0.029	8.965	0.201909	-

5.1.7 WEIGHT PERCENT DETERMINATION - GENERAL

1 *Identification of the significant peak's position*

In the Method's Evaluation tab a weight percent determination can be chosen for the significant Peak of ***References***. It cannot be performed in case of Blanks.

All calculations of weight percent determination refer to the so called ***significant Peak***. To first and foremost determine its position, in the Method's Evaluation tab an interval must be defined by a Significant Peak *Start* value and a Significant Peak *Stop* value.

Since this interval will not automatically be identified, a sample chromatogram must be recorded which reveals the position of the significant Peak. Consider that the position can vary slightly over time due to alteration processes (e.g. of glassy carbon). Dimensioning the interval sufficiently broad will prevent the significant peak from falling or migrating outside it.

2 *Calculation of weight percent*

Weight percent determination bases upon the relationships between peak area of the significant peak and sample weight.

First, a Reference (i.e. a well-known substance; not necessarily identical to the compound of the later sample) is measured to obtain the k-Factor k according to the formula:

$$k = \frac{w_{ref} * m_{ref}}{A_{ref}}$$

The Reference's weight percent w_{ref} and mass m_{ref} are given whereas its peak area A_{ref} emerges from the measurement. Possibly, A_{ref} can be used blank-corrected to enhance the precision of k-Factor calculation.

Using the k-Factor just calculated, now the sample itself can be measured. Its peak area A_{sample} results (possibly blank-corrected), whereas sample weight m_{sample} and k-Factor k are well known, so that the parameter of interest, the sample's weight percentage w_{sample}, can be easily obtained rearranging the above equation:

$$w_{sample} = \frac{k * A_{sample}}{m_{sample}}$$

5.1.8 WEIGHT PERCENT DETERMINATION - PROCEDURE FOR OXYGEN

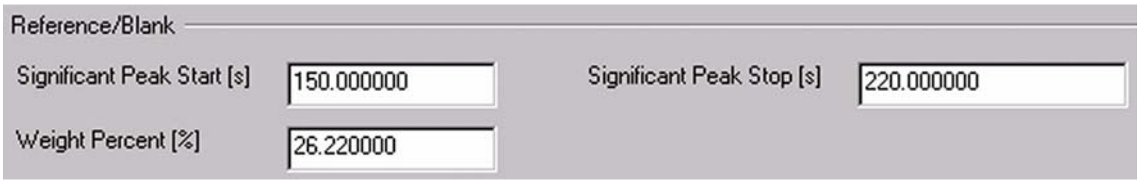
The system requires a Calibration procedure in order to cal-culate the amount of oxygen in the sample.

1 Weigh a reference compound, e.g. 0.285 mg of benzoic acid, in a silver capsule and wrap it carefully.

NOTE: ***The precision of weighing is directly related to the precision of weight percent determination.***

2 Place the capsule in the solid-autosampler.

3 ***Method (Evaluation tab)***



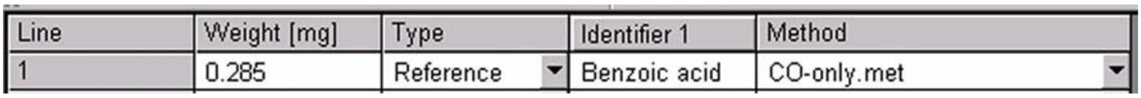
Reference/Blank

Significant Peak Start [s] 150.000000

Significant Peak Stop [s] 220.000000

Weight Percent [%] 26.220000

4 ***Sequence***



Line	Weight [mg]	Type	Identifier 1	Method
1	0.285	Reference	Benzoic acid	CO-only.met

The Method "CO-only.met" is described in detail in Chapter 5.1.3.

5 Start the Sequence. 

NOTE: In order to obtain reasonable results, this Calibration has to be performed daily and after changing any parameters of the system.

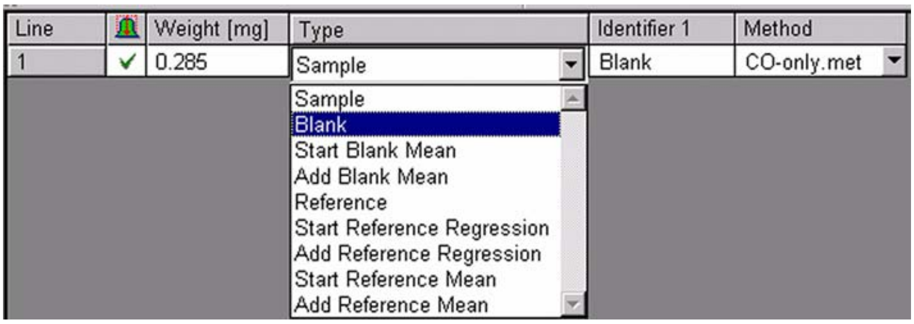
5.1.9 BLANK MEASUREMENT - GENERAL

Blank value determination can help to enhance precision. The resulting blank value is then subtracted from both reference and sample peaks. Thus, the chronological order to proceed normally is: 1. Blank, 2. Reference, 3. Sample.

Blank and Reference values are stored in separate databases. A further criterion of separate storage is the gas name. To measure samples *directly*, i.e. without Blank and Reference before, delete these databases via ***Quant*** Menu.

In the following, the different commands for Blank measurement will be explained.

Blank command

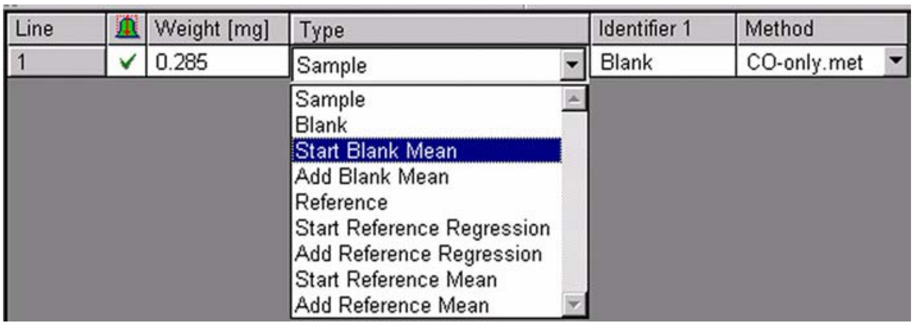


Line	✓	Weight [mg]	Type	Identifier 1	Method
1	✓	0.285	Sample	Blank	CO-only.met

- From the ***Type*** Menu select ***Blank***.

The previous Blank value will be deleted. Instead, the result of this current Blank measurement will be enlisted as new Blank value.

Start Blank Mean command

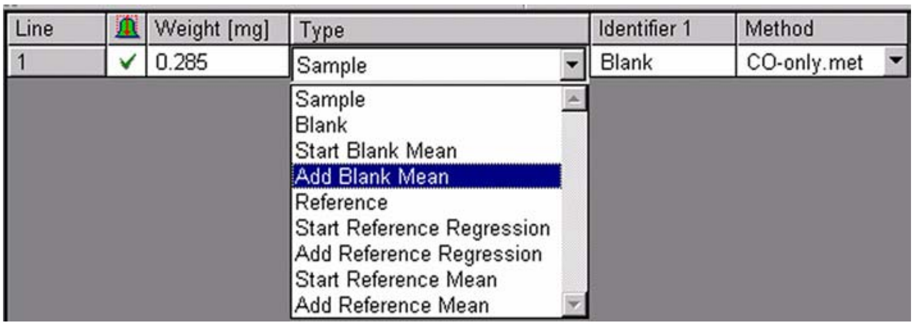


Line	✓	Weight [mg]	Type	Identifier 1	Method
1	✓	0.285	Sample	Blank	CO-only.met

- From the ***Type*** Menu select ***Start Blank Mean***.

The previous Blank value will be deleted. Instead, the result of this current Blank measurement will be enlisted as new Blank value. Further Blanks can be added to be taken into account determining the Blank Mean.

Add Blank Mean command



Line	✓	Weight [mg]	Type	Identifier 1	Method
1	✓	0.285	Sample	Blank	CO-only.met

- ➢ From the ***Type*** Menu select ***Add Blank Mean***.

The old Blank Mean will be corrected using the new Blank value to yield the new Blank Mean according to the formula:

$$m_b' = \frac{m_b * n_b + b}{n_b + 1}$$

where:

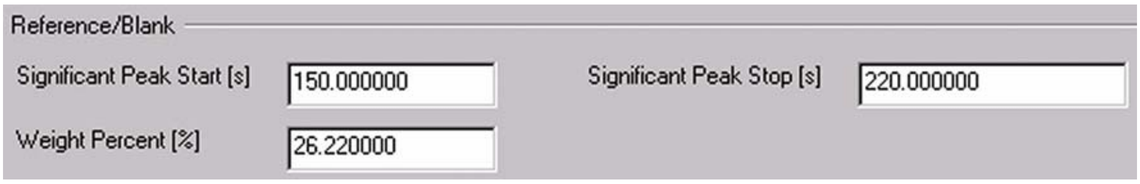
m_b' new Blank Mean
m_b old Blank Mean
n_b number of previously measured Blank values (since Blank Start)
b new Blank value

5.1.10 BLANK MEASUREMENT - PROCEDURE FOR OXYGEN

The tiny blank signal must be detected in the window.

1 Place an empty silver capsule into the solid-autosampler.

2 Method (Evaluation tab)



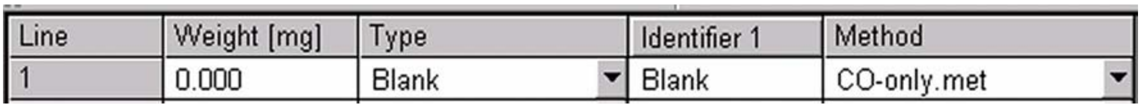
Reference/Blank

Significant Peak Start [s] 150.000000

Significant Peak Stop [s] 220.000000

Weight Percent [%] 26.220000

3 ***Sequence***



Line	Weight [mg]	Type	Identifier 1	Method
1	0.000	Blank	Blank	CO-only.met

The Method “CO-only.met” is described in detail in Chapter 5.1.3.

4 Start the Sequence.

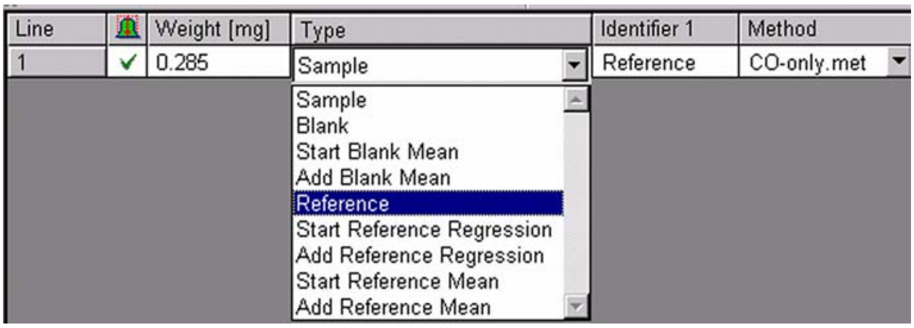
NOTE: In order to obtain reasonable results, this Calibration has to be performed daily and after changing any parameters of the system.

5.1.11 REFERENCE MEASUREMENT - GENERAL

Three procedures (i.e. commands) exist to perform Reference measurements:

- Reference
- Start Reference Mean
- Start Reference Regression

Reference command

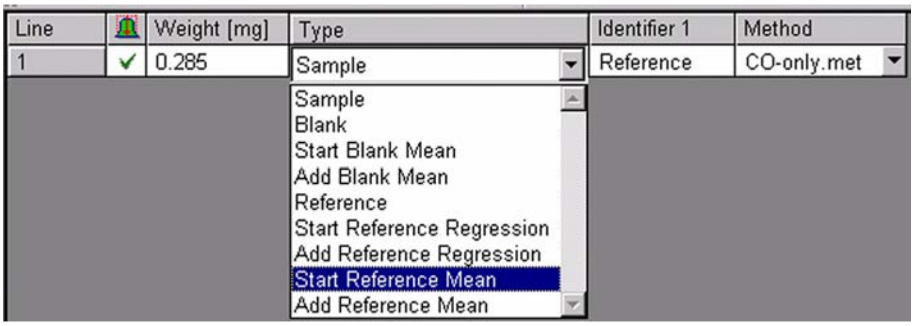


Line		Weight [mg]	Type	Identifier 1	Method
1	✓	0.285	Sample	Reference	CO-only.met

- From the ***Type*** Menu select ***Reference***.

The previous Reference value will be deleted. Instead, the result of this current Reference measurement will be enlisted as new Reference value.

Start Reference Mean command

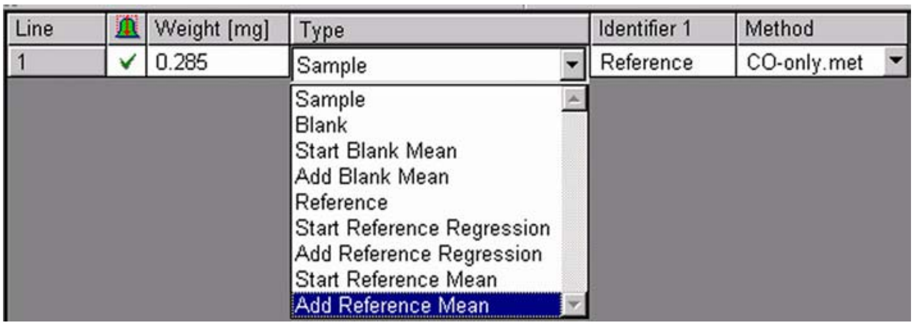


Line		Weight [mg]	Type	Identifier 1	Method
1	✓	0.285	Sample	Reference	CO-only.met

- From the ***Type*** Menu select ***Start Reference Mean***.

The previous Reference value will be deleted. Instead, the result of this current Reference measurement will be enlisted as new Reference value. Further References can be added to be taken into account determining the Reference Mean.

Add Reference Mean command



Line		Weight [mg]	Type	Identifier 1	Method
1	✓	0.285	Sample	Reference	CO-only.met

- From the ***Type*** Menu select ***Add Reference Mean***.

The old Reference Mean will be corrected using the new Reference value to yield the new Reference Mean according to the formula:

$$m_r' = \frac{m_r * n_r + r}{n_r + 1}$$

where:

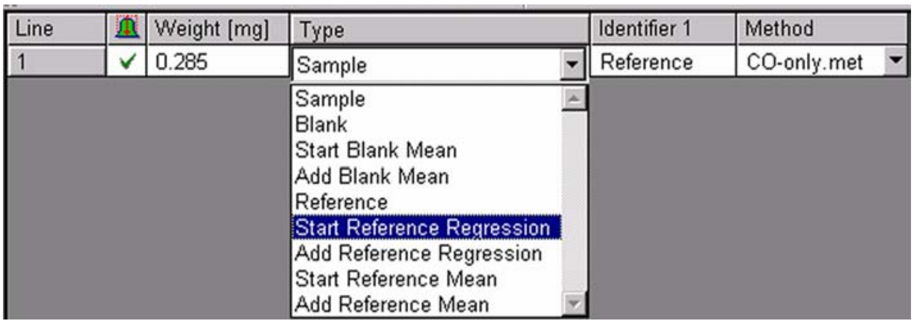
m_r'	new Reference Mean
m_r	old Reference Mean
n_r	number of previously measured Reference values (since Reference Start)
r	new Reference value

5.1.12 REFERENCE REGRESSION

In addition to determining a Reference ***Mean*** (see above) a Reference ***calibration curve*** can be generated: the k-Factor is plotted vs. the sample weight.

The Reference *Mean* is calculated in case of References of *very similar* weights. With References of *considerably differing* weights, it is advantageous to generate a Reference *calibration curve*. Measuring a sample afterwards, the k-Factor can be acquired using the calibration curve.

Start Reference Regression command

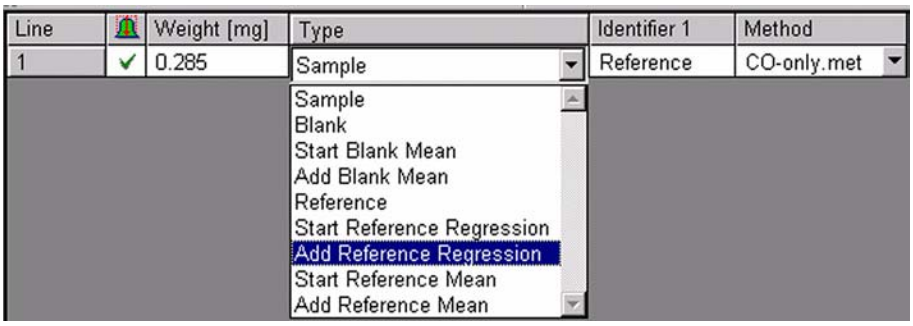


Line	✓	Weight [mg]	Type	Identifier 1	Method
1	✓	0.285	Sample	Reference	CO-only.met

- ➢ From the ***Type*** Menu select ***Start Reference Regression***.

This command corresponds to the *Start Reference* ***Mean*** command (see page 5 – 20). The previous Reference value will be deleted. Instead, the result of this current Reference measurement will be enlisted as new Reference value. Further References can be added to extend the Reference calibration curve.

Add Reference Regression command



Line	✓	Weight [mg]	Type	Identifier 1	Method
1	✓	0.285	Sample	Reference	CO-only.met

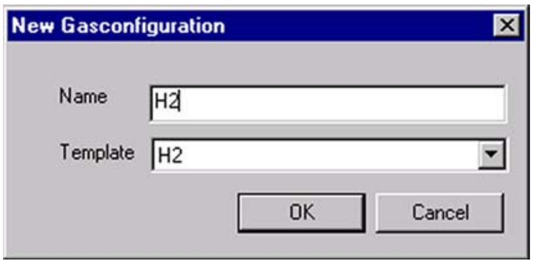
- ➢ From the ***Type*** Menu select ***Add Reference Regression***.

This command corresponds to the *Add Reference* ***Mean*** command (see page 5 – 21). A new point on the calibration curve is enlisted.

5.2 HYDROGEN MEASUREMENT

5.2.1 DEFINING A GAS CONFIGURATION

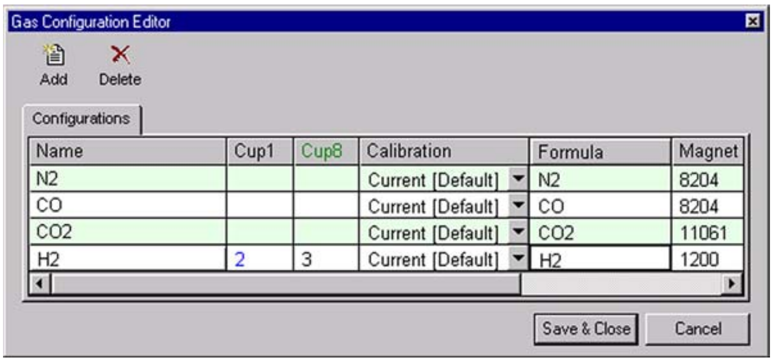
- Open the ***Continuous Flow*** module.
- Open the ***Gas Configuration Editor***.
- ***Add*** a Gas Configuration, if a H_2 Gas Configuration is not yet available.



Name: H2

Template: H2

- Select any Gas Configuration as ***Template*** or ***H2***, if already available.
- Type ***H2*** for the Name.
- Confirm by ***OK***.



Name	Cup1	Cup8	Calibration	Formula	Magnet
N2			Current [Default]	N2	8204
CO			Current [Default]	CO	8204
CO2			Current [Default]	CO2	11061
H2	2	3	Current [Default]	H2	1200

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

NOTE: ***Ensure that a suited Configuration is available. To set up a new Configuration refer to Chapter 5.1.2.***

NOTE: ***For detailed information refer to the ISODAT NT Help System (Configurator), which is delivered on your ISODAT NT CD.***

5.2.2 H_3-FACTOR DETERMINATION

Protonation reactions in the ion source result in H_3^+ ion production. The H_3^+ portion of the m/z ion beam is determined as H_3-Factor. The H_3-Factor is used to correct the H_3^+ contribution to the m/z 3 signal. A low and stable H_3-Factor is needed for a good DH/H_2 determination.

- It is assumed that ion source setting for hydrogen determination and Mass Calibration for hydrogen have already been performed.
- Standard gases (i.e. CO and H_2) are installed.

NOTE: ***During the first setup, H_3-Factor determination must be performed before any other procedure. Later on, at least once a day, a H_3-Factor determination is recommended.***

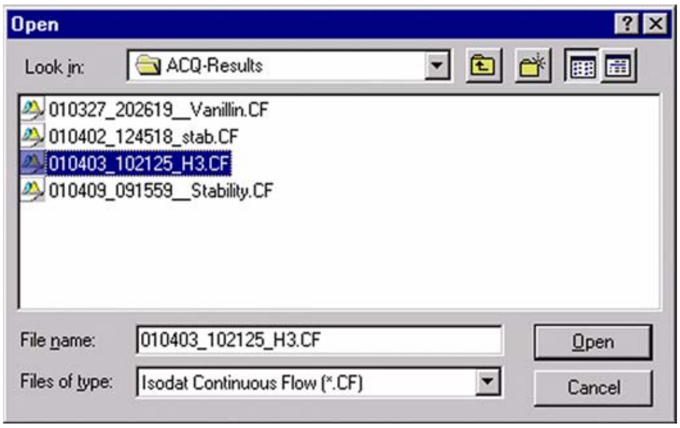
Continuous Flow ACQ

- Open the ***Continuous Flow*** module.

H3-Factor determination bases on peak area information of a standard gas on/off chromatogram (*.cf) with standard gas pulses of different amplitudes. Chromatograms are stored in result folders, e.g.: C:\Finnigan\ISODAT NT\Global\User\ConFloIIInterface\Results\ACQ-Results.

Open

- To load it, press the ***Open*** button.



Look in: ACQ-Results

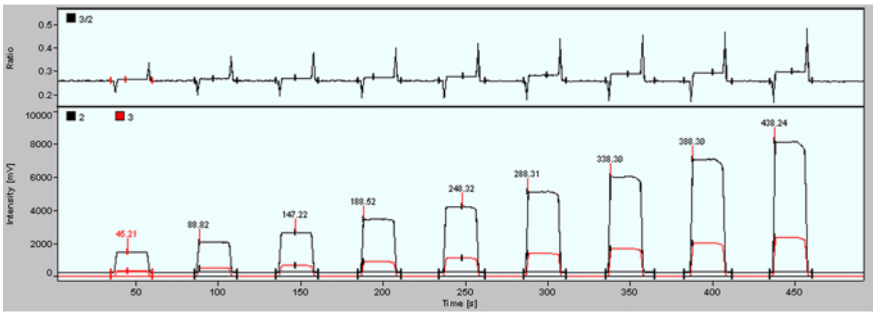
010327_202619__Vanillin.CF
010402_124518_stab.CF
010403_102125_H3.CF
010409_091559__Stability.CF

File name: 010403_102125_H3.CF

Files of type: Isodat Continuous Flow (*.CF)

- Localize your *.cf file and press ***Open***.

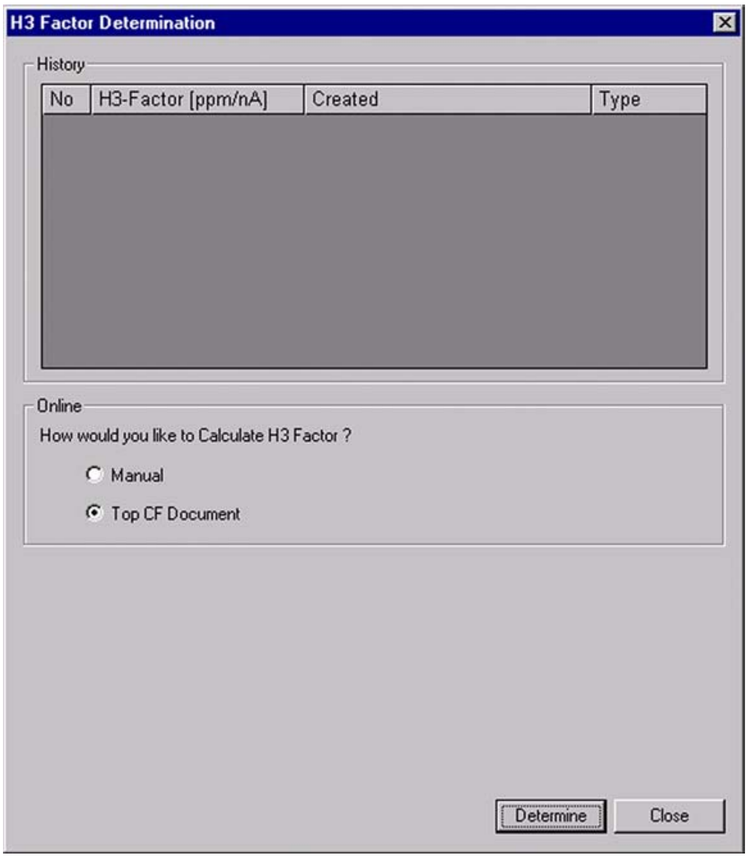
C:\Finnigan\ISODAT NT\Global\User\Conflo II Interface\Results\ACQ-Results\010403_102125_H3.CF



- ➢ The chromatogram will be loaded.

To see how such a chromatogram is obtained (i.e. via stepwise increases of the reference gas pressure using the Method “H2_zero.met”), refer to Chapter 5.2.3.

- ➢ Click the ***H3-Factor*** button (on Smart Isotope MS bar or on Isotope MS bar).



No	H3-Factor [ppm/nA]	Created	Type

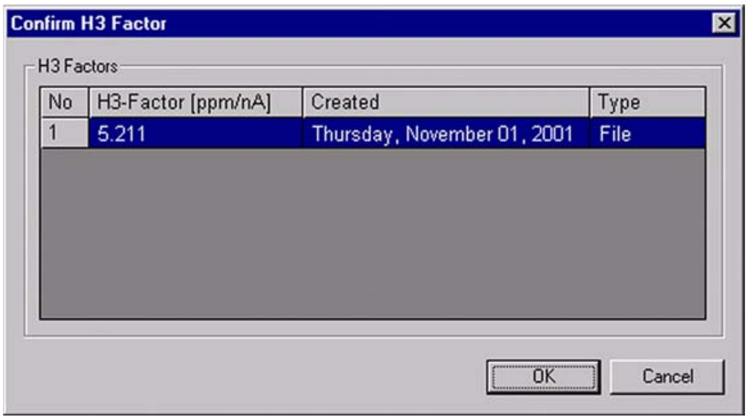
Online

How would you like to Calculate H3 Factor ?

○ Manual

● Top CF Document

- ➢ The H3-Factor is calculated on the basis of a chromatogram (*.cf).
- ➢ Select ***Top CF Document***.
- ➢ Press ***Determine***.



Confirm H3 Factor

H3 Factors

No	H3-Factor [ppm/nA]	Created	Type
1	5.211	Thursday, November 01, 2001	File

- ➢ Confirm the calculated H3-Factor by ***OK***.
- ➢ Upon ***Cancel***, a new H3-Factor can be determined.

The calculated H3-Factor is also displayed in the ***H3-Factor*** tab page beneath the chromatogram and will be used for further data acquisitions (see Chapter 6.1.2.2).

5.2.3 ZERO ENRICHMENT (ST ON / OFF TEST)

We assume that the user already has some working experience with the system, and that H_3-Factor determination has already been performed (refer to Chapter 5.2.2). It is recommended to perform a simple check in order to test the analytical condition of the IRMS before measuring any samples.

The following steps need to be performed to define a Method.

- ➢ Open the ***Continuous Flow*** module.

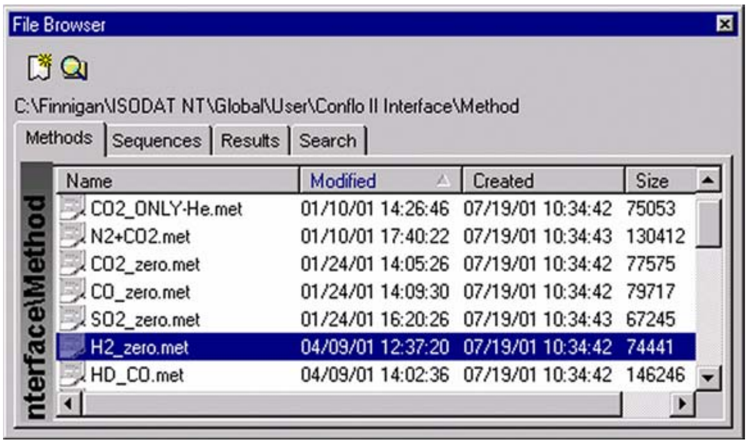
ConFlo II & TC / EA

- ➢ Select the ***Configuration*** (e.g. for ConFlo II/III application).

CO

- ➢ Select the ***Gas Configuration*** for ^{18}O determination.
- ➢ Create a new Method **or**

  Select a Method from the File Browser.



C:\Finnigan\ISODAT NT\Global\User\Conflo II Interface\Method

Name	Modified	Created	Size
CO2_ONLY-He.met	01/10/01 14:26:46	07/19/01 10:34:42	75053
N2+CO2.met	01/10/01 17:40:22	07/19/01 10:34:43	130412
CO2_zero.met	01/24/01 14:05:26	07/19/01 10:34:42	77575
CO_zero.met	01/24/01 14:09:30	07/19/01 10:34:42	79717
SO2_zero.met	01/24/01 16:20:26	07/19/01 10:34:43	67245
H2_zero.met	04/09/01 12:37:20	07/19/01 10:34:42	74441
HD_CO.met	04/09/01 14:02:36	07/19/01 10:34:42	146246

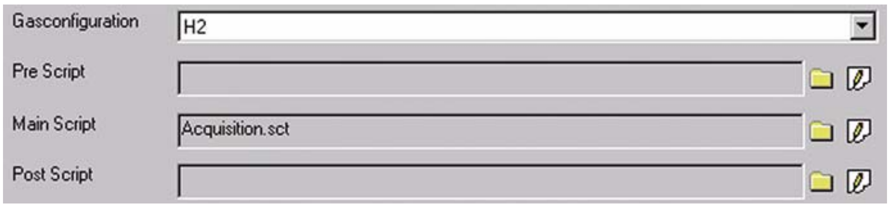
- On the File Browser, click the ***Methods*** tab.
- Double-click the Method ***H2_zero.met***.

The Method is structured in Tab-pages: Instrument, Time Events, Evaluation, Peak Detection, and Printout.

For a detailed explanation of the Method's structure and editing refer to Chapter 5.1.3.

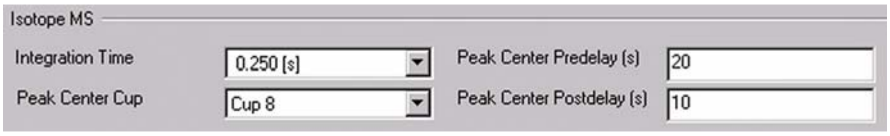
As a guideline, use the following Method (***"H2_zero.met"***).

Instrument



Gasconfiguration: H2
Pre Script:
Main Script: Acquisition.sct
Post Script:

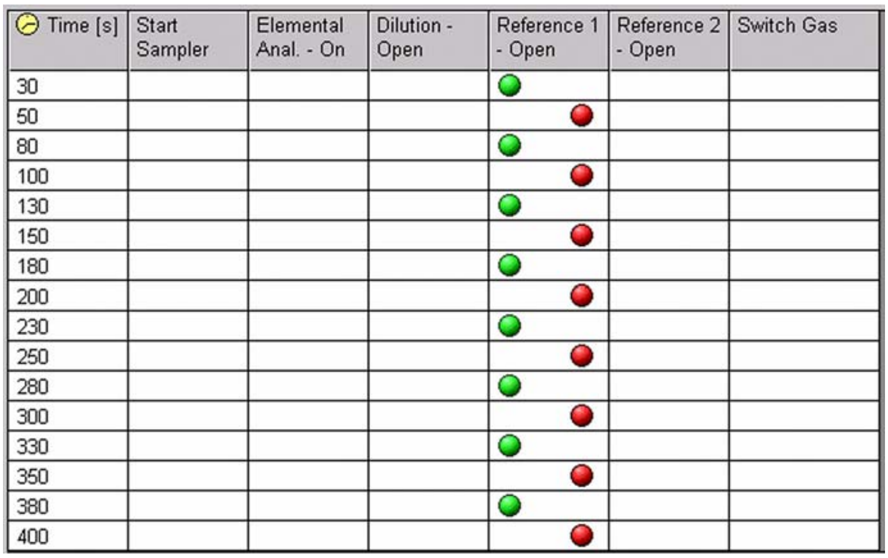
Isotope MS



Isotope MS
Integration Time: 0.250 [s]
Peak Center Cup: Cup 8
Peak Center Predelay [s]: 20
Peak Center Postdelay [s]: 10

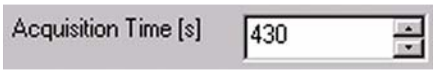
Reference Port: Reference 2

Time Events



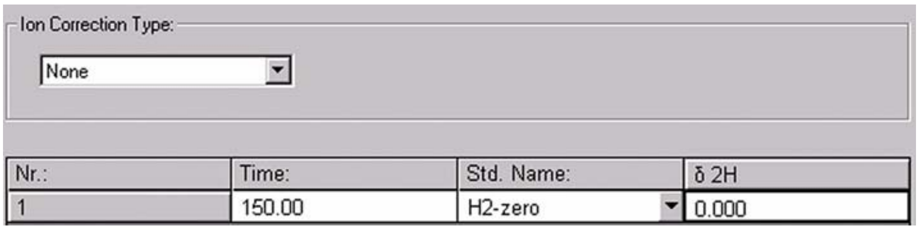
Time [s]	Start Sampler	Elemental Anal. - On	Dilution - Open	Reference 1 - Open	Reference 2 - Open	Switch Gas
30				🟢		
50				🔴		
80				🟢		
100				🔴		
130				🟢		
150				🔴		
180				🟢		
200				🔴		
230				🟢		
250				🔴		
280				🟢		
300				🔴		
330				🟢		
350				🔴		
380				🟢		
400				🔴		

Acq. End Time



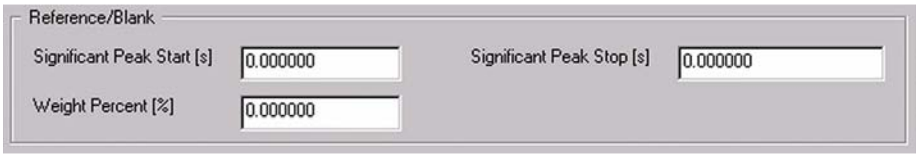
Acquisition Time [s] 430

Evaluation



Ion Correction Type: None

Nr.:	Time:	Std. Name:	δ 2H
1	150.00	H2-zero	0.000



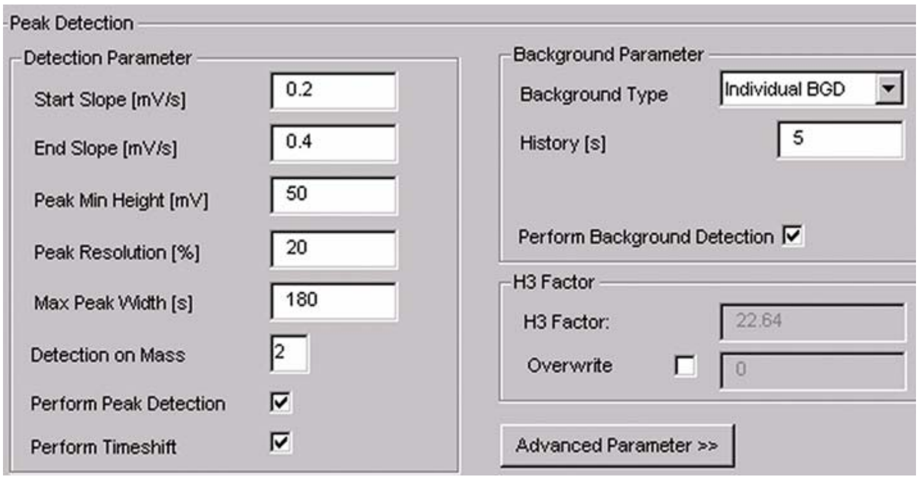
Reference/Blank

Significant Peak Start [s] 0.000000

Significant Peak Stop [s] 0.000000

Weight Percent [%] 0.000000

Peak Detection



Peak Detection

Detection Parameter

Start Slope [mV/s] 0.2

End Slope [mV/s] 0.4

Peak Min Height [mV] 50

Peak Resolution [%] 20

Max Peak Width [s] 180

Detection on Mass 2

Perform Peak Detection ☑

Perform Timeshift ☑

Background Parameter

Background Type Individual BGD

History [s] 5

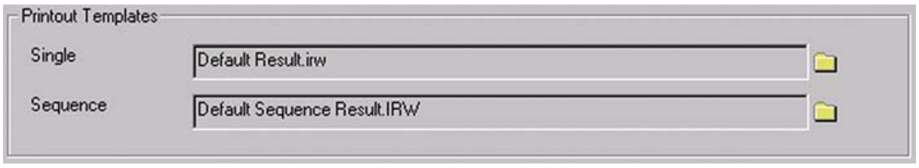
Perform Background Detection ☑

H3 Factor

H3 Factor: 22.64

Overwrite ☐ 0

Printout



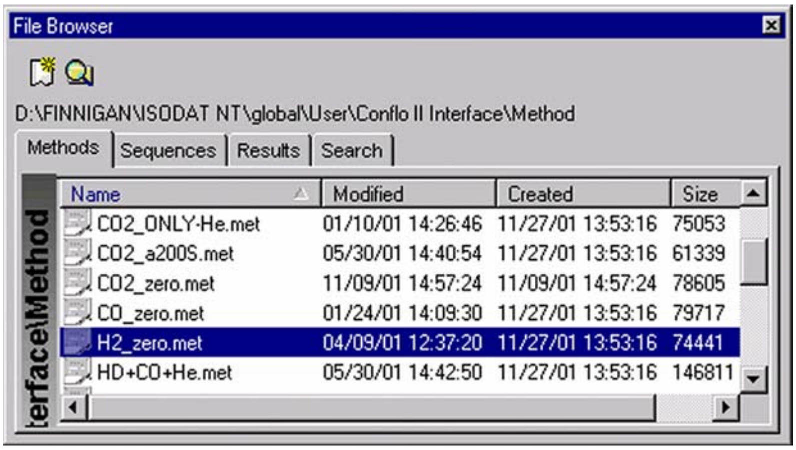
Printout Templates

Single Default Result.irw

Sequence Default Sequence Result.IRW

2 Set the ion intensity of mass 2 (H_2) to approximately ***4 V***.

3 Create a new Sequence or select a predefined Sequence from File Browser.

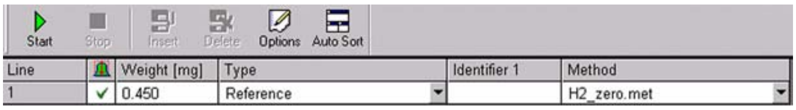


File Browser

D:\FINNIGAN\ISODAT NT\global\User\Conflo II Interface\Method

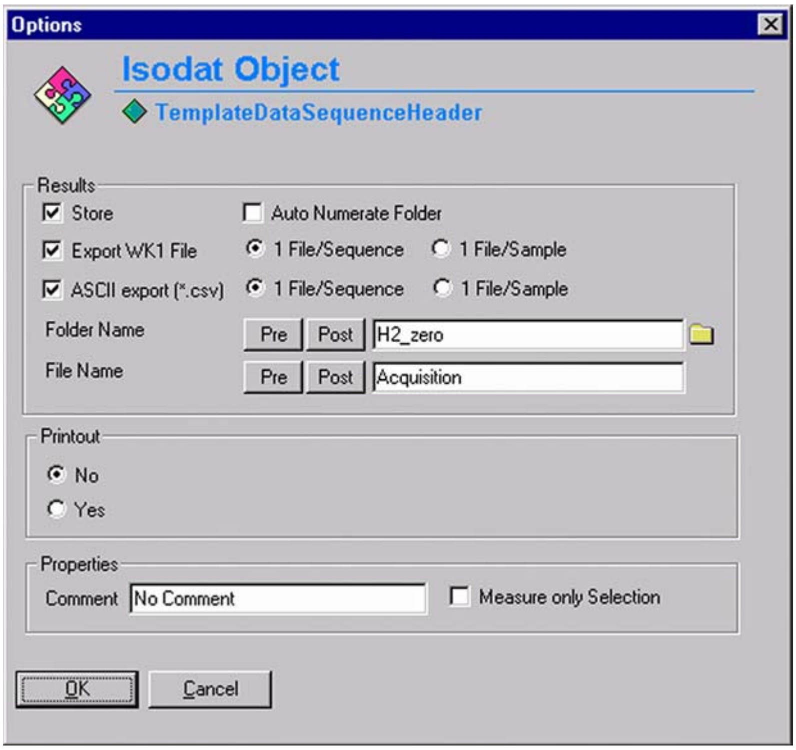
Name	Modified	Created	Size
CO2_ONLY-He.met	01/10/01 14:26:46	11/27/01 13:53:16	75053
CO2_a200S.met	05/30/01 14:40:54	11/27/01 13:53:16	61339
CO2_zero.met	11/09/01 14:57:24	11/09/01 14:57:24	78605
CO_zero.met	01/24/01 14:09:30	11/27/01 13:53:16	79717
H2_zero.met	04/09/01 12:37:20	11/27/01 13:53:16	74441
HD+CO+He.met	05/30/01 14:42:50	11/27/01 13:53:16	146811

- Click the ***Sequences*** tab.
- Doubleclick the Sequence file ***H2_zero.seq***.



Line		Weight [mg]	Type	Identifier 1	Method
1	✓	0.450	Reference		H2_zero.met

- ➢ Click the ***Start*** button.



Results
☑ Store ☐ Auto Numerate Folder
☑ Export WK1 File ● 1 File/Sequence ○ 1 File/Sample
☑ ASCII export (*.csv) ● 1 File/Sequence ○ 1 File/Sample
Folder Name Pre Post H2_zero
File Name Pre Post Acquisition

Printout
● No
○ Yes

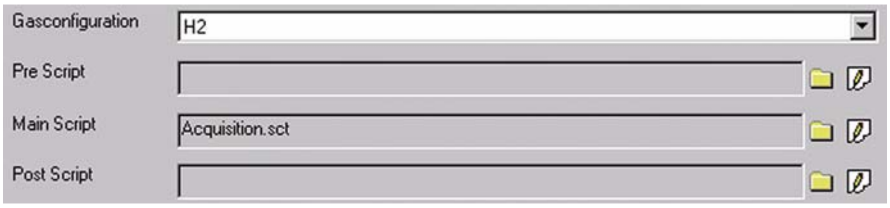
Properties
Comment No Comment ☐ Measure only Selection

- ➢ Edit how to export results.
- ➢ Name folder and file for data storage.
- ➢ Activate or deactivate ***Printout***.
- ➢ Confirm your decision with ***OK***.

5.2.4 HOW TO START A HYDROGEN MEASUREMENT

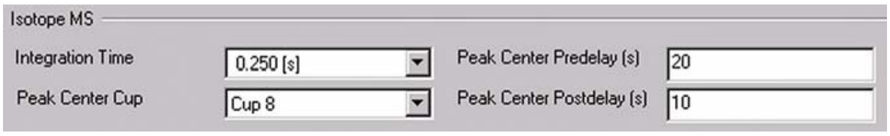
1 Define a Method (as shown in Chapter 5.1.3).
As a guideline, use the following Method ***(HD_only.met)***:

Instrument



Gasconfiguration: H2
Pre Script:
Main Script: Acquisition.sct
Post Script:

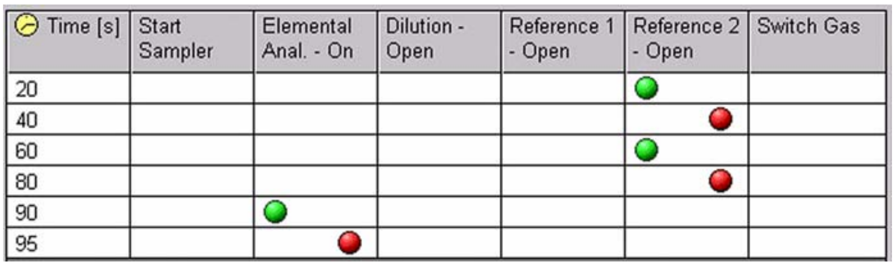
Isotope MS



Isotope MS
Integration Time: 0.250 [s]
Peak Center Cup: Cup 8
Peak Center Predelay [s]: 20
Peak Center Postdelay [s]: 10

Reference Port: Reference 2

Time Events

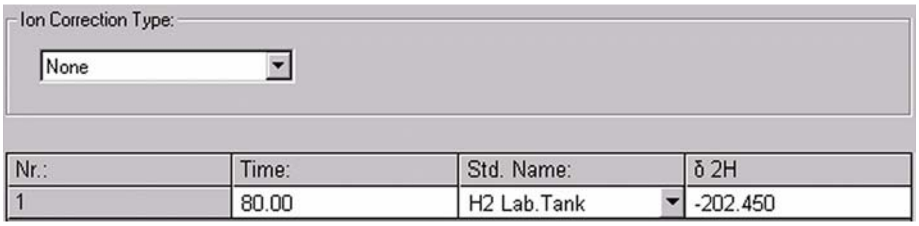


Time [s]	Start Sampler	Elemental Anal. - On	Dilution - Open	Reference 1 - Open	Reference 2 - Open	Switch Gas
20					🟢	
40					🔴	
60					🟢	
80					🔴	
90		🟢				
95		🔴				

Acq. End Time

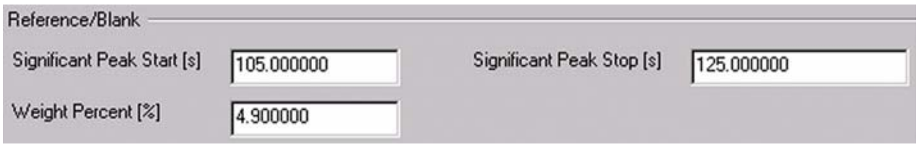
Acquisition Time [s] 160

Evaluation



Ion Correction Type: None

Nr.:	Time:	Std. Name:	δ 2H
1	80.00	H2 Lab.Tank	-202.450



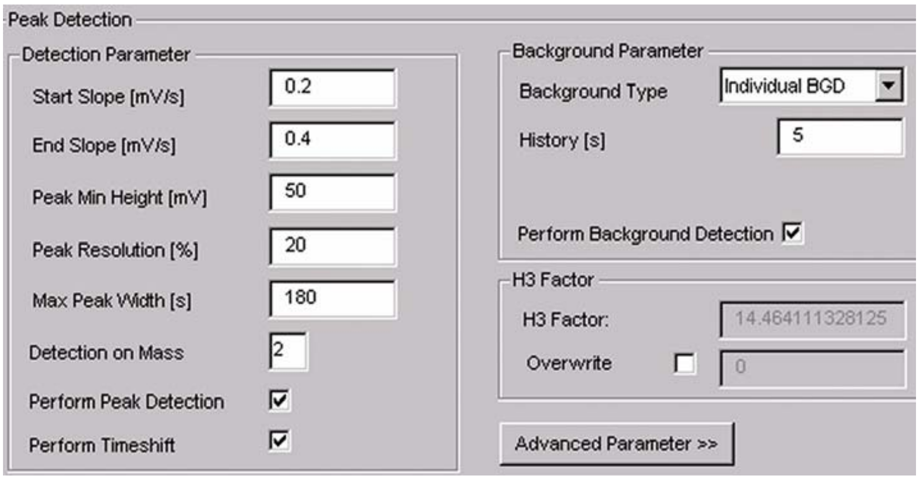
Reference/Blank

Significant Peak Start [s] 105.000000

Significant Peak Stop [s] 125.000000

Weight Percent [%] 4.900000

Peak Detection



Peak Detection

Detection Parameter

Start Slope [mV/s] 0.2

End Slope [mV/s] 0.4

Peak Min Height [mV] 50

Peak Resolution [%] 20

Max Peak Width [s] 180

Detection on Mass 2

Perform Peak Detection ☑

Perform Timeshift ☑

Background Parameter

Background Type Individual BGD

History [s] 5

Perform Background Detection ☑

H3 Factor

H3 Factor: 14.464111328125

Overwrite ☐ 0

Printout

Printout Templates

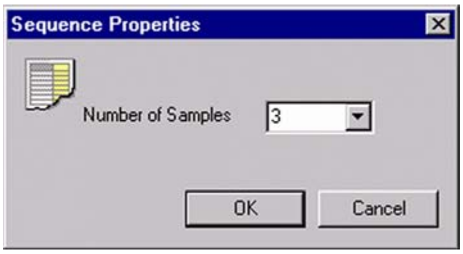
Single: HD_only.IRW

Sequence: Single Result.IRW

2 Place a sample (e.g. 0.281 mg of benzoic acid) in the solid-autosampler.

3 ***Create a new Sequence***.

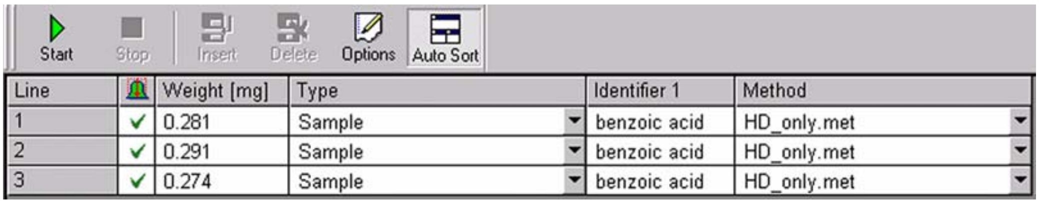
4 Define number of samples (e.g. 3).



Sequence Properties

Number of Samples: 3

5 Edit the Sequence list.

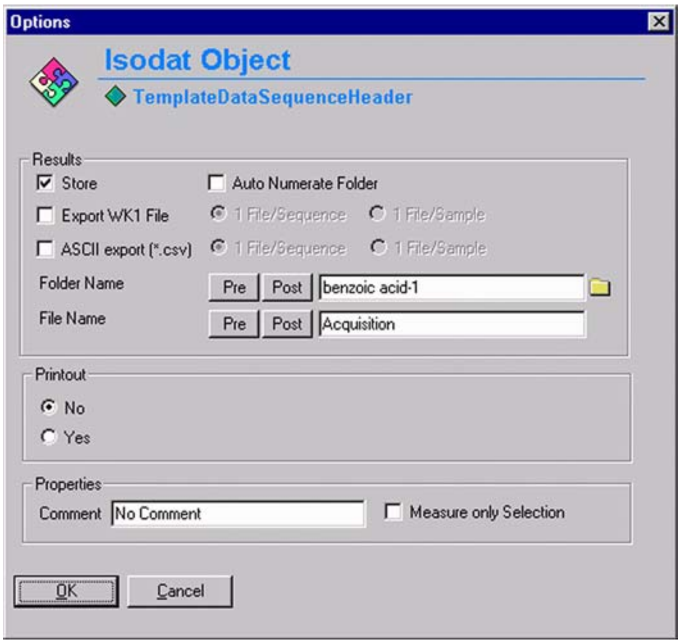


Line		Weight [mg]	Type	Identifier 1	Method
1	✓	0.281	Sample	benzoic acid	HD_only.met
2	✓	0.291	Sample	benzoic acid	HD_only.met
3	✓	0.274	Sample	benzoic acid	HD_only.met

Peak Center	Enable ☑ in order to perform a Peak Center prior to measurement.
Identifier 1	Edit text to identify sample.
Method	Select IRMS Method.

6 Press the ***Start*** button.

7 Define Results Storage, Export, and Printout parameters.



Options

Isodat Object

TemplateDataSequenceHeader

Results

☑ Store ☐ Auto Numerate Folder

☐ Export WK1 File ● 1 File/Sequence ○ 1 File/Sample

☐ ASCII export (*.csv) ● 1 File/Sequence ○ 1 File/Sample

Folder Name Pre Post benzoic acid-1

File Name Pre Post Acquisition

Printout

● No

○ Yes

Properties

Comment No Comment ☐ Measure only Selection

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

Events during Acquisition

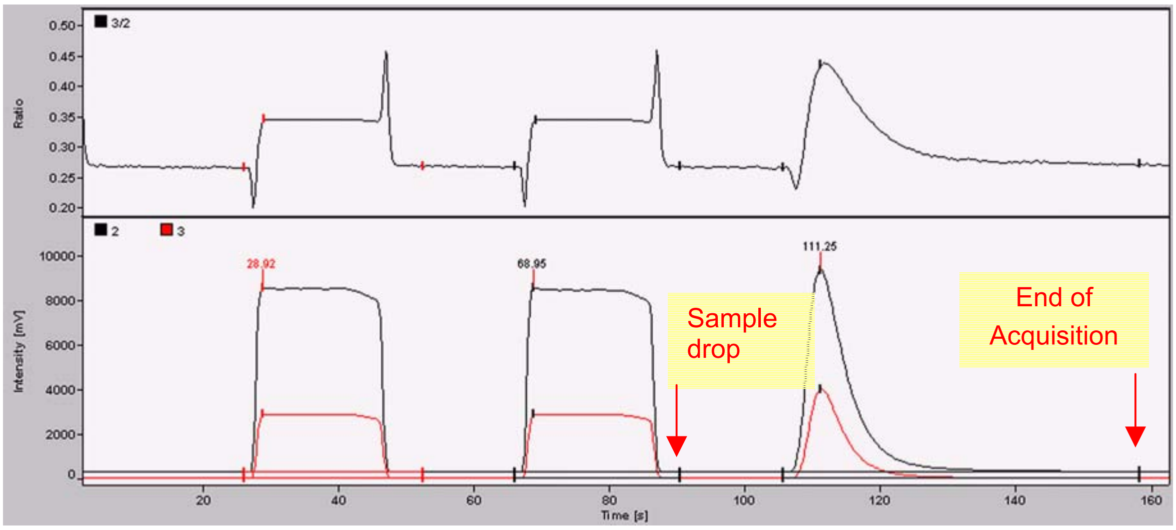
- Peak Center procedure
- First H_2 Reference Gas pulse activated at 20 s (duration: 20 s).
- Second H_2 Reference Gas pulse activated at 60 s (duration: 20 s).
- Sample is dropped into reactor at 90 s.
- H_2 sample peak appears approximately 15 s after start of reaction.
- Acquisition stops at 160 s.

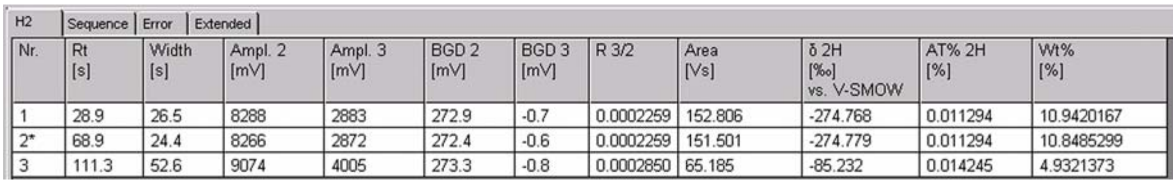
After finishing data acquisition the printer creates a data output sheet as defined by the selected Results Workshop template (*.irw).

The Results will also be exported to a spreadsheet file, if activated.

C:\FINNIGAN\ISODAT NT\Global\User\Conflo II Interface\Results\Benzoic acid HD.CF

Line		Weight [mg]	Type	Identifier 1	Method
1	✓	0.281	Sample	benzoic acid	HD_only.met





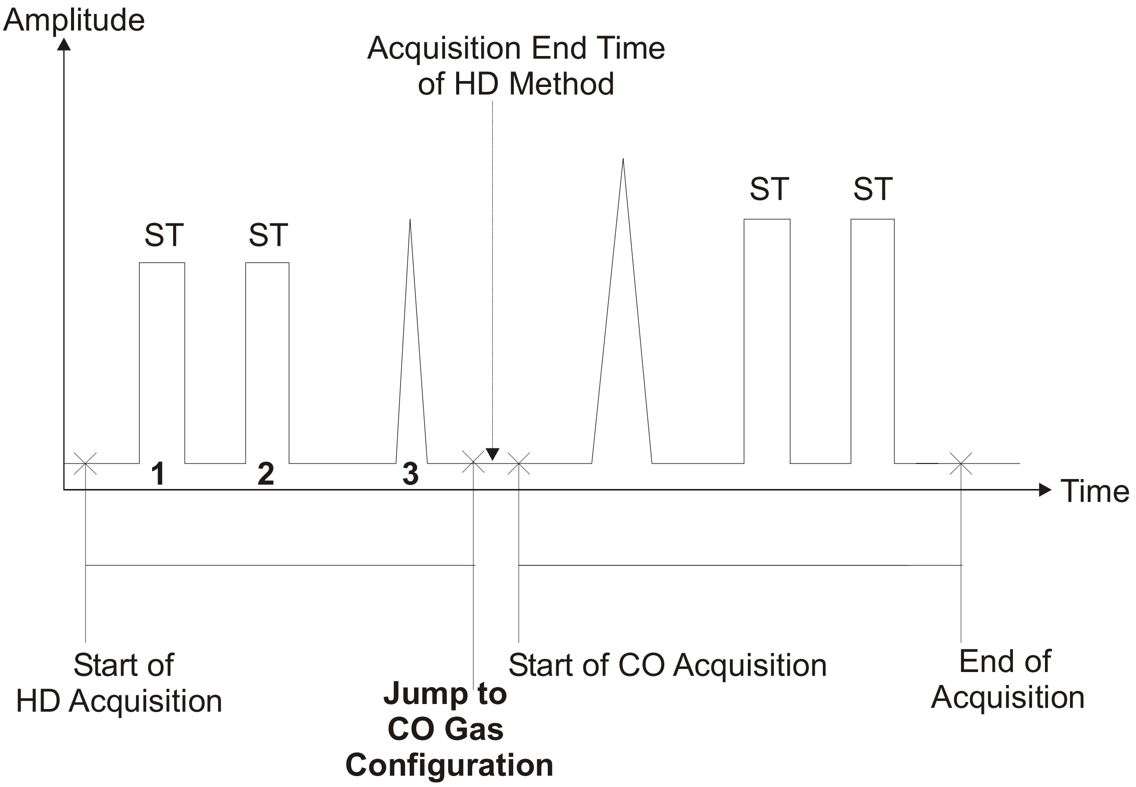
Nr.	Rt [s]	Width [s]	Ampl. 2 [mV]	Ampl. 3 [mV]	BGD 2 [mV]	BGD 3 [mV]	R 3/2	Area [Vs]	δ 2H [‰] vs. V-SMOW	AT% 2H [%]	Wt% [%]
1	28.9	26.5	8288	2883	272.9	-0.7	0.0002259	152.806	-274.768	0.011294	10.9420167
2*	68.9	24.4	8266	2872	272.4	-0.6	0.0002259	151.501	-274.779	0.011294	10.8485299
3	111.3	52.6	9074	4005	273.3	-0.8	0.0002850	65.185	-85.232	0.014245	4.9321373

5.3 DUAL MEASUREMENT

For the analysis of two isotopic species (hydrogen and oxygen) from a single sample, a Method, which comprises both of them must be defined. The acquisition can be completed after less than 7 min (400 s).

As soon as the hydrogen peak (3) has been identified, ISODAT NT stops the HD acquisition. The magnet jumps to the CO Configuration (see: "Switch Gas" column in the Time Events list on page 5 – 40).
If no hydrogen peak can be found, ISODAT NT waits a certain time (e.g. 25 s).
After each injection, immediately data acquisition follows.

Schematic chromatogram:



5.3.1 HOW TO PERFORM A JUMP CALIBRATION

After determining the H3-Factor (refer to Chapter 5. 2.2), a so-called ***Jump Calibration*** must be performed. A Jump Calibration calibrates for a fast variation of the magnetic field.

In order to determine the isotope ratios of different elements during the same run, switching to another Gas Configuration is necessary.

In contrast to a ***single*** element measurement, in which the magnetic field runs the gamut from high to low and after that to the pre-calculated magnetic field, there is not sufficient time in ***dual*** measurement to perform this procedure for the next gas configuration.

For this reason, a Jump Calibration from the first gas configuration to the next gas configuration is necessary.
After the Jump Calibration has been performed, the computer finds exactly the peak center even without performing any peak center procedure.

A Jump Calibration should be performed daily to stabilize the performance of the magnet.

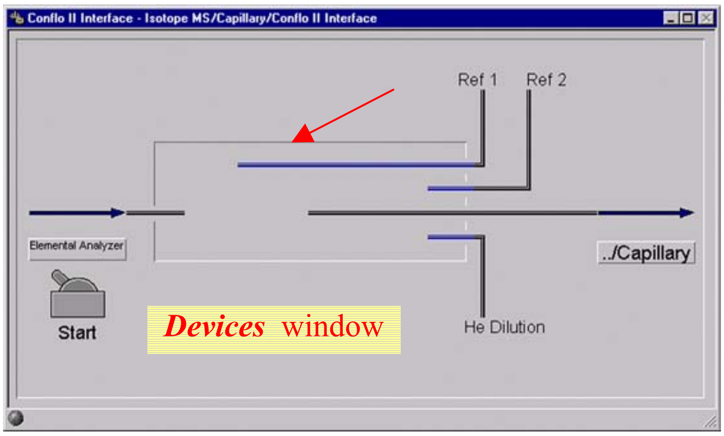
NOTE: ***Both Gas Configurations used (i.e. "H2" and "CO") need to be calibrated prior to Jump Calibration.***

Instrument Control

- ➢ Open Instrument Control.

ConFlo II & TC / EA & AS

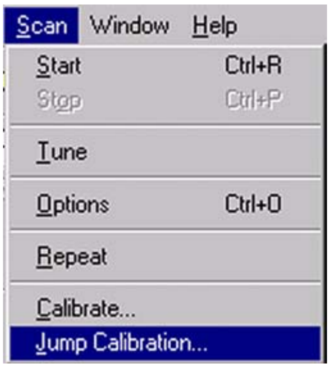
- ➢ Choose your ***Configuration*** (e.g. "ConFlo II & TC/EA & AS")
- ➢ Turn to the ***Devices*** window by pressing this Icon.



- ➢ Open Reference 1 by a click on ***Ref 1*** capillary.

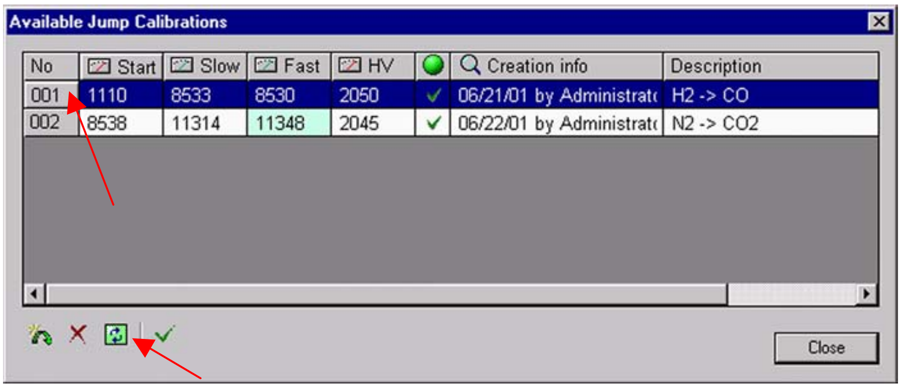
  Thus, the blue capillary symbol elongates and CO enters the system.

- ➢ Return to the Instrument Control window by this Icon.



- ➢ Select ***Scan*** > ***Jump Calibration*** or alternatively:

  on the toolbar next to the Instrument Control window press the ***Jump*** button.



No	Start	Slow	Fast	HV	●	Creation info	Description
001	1110	8533	8530	2050	✓	06/21/01 by Administrat	H2 -> CO
002	8538	11314	11348	2045	✓	06/22/01 by Administrat	N2 -> CO2

- ➢ In the list of available Jump Calibrations mark the one for H_2 → CO by clicking its No. (e.g. 001).

- ➢ Press ***Recalibrate*** Icon.

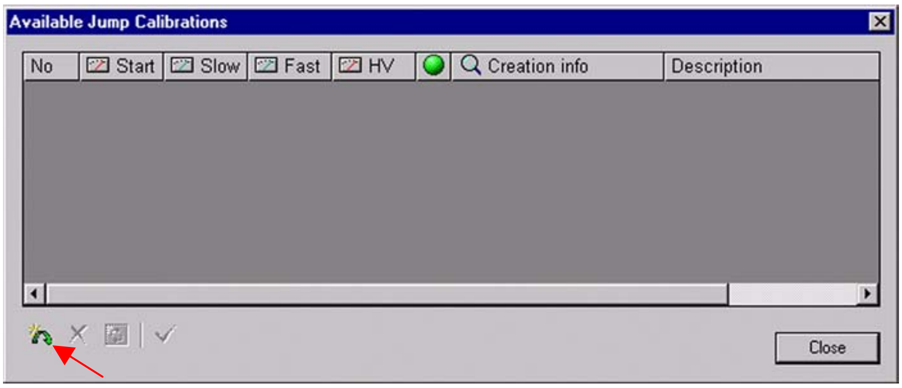
Slow: compensate hysteresis by Max/Min settings of the magnet.

Fast: magnet setting of Jump Calibration

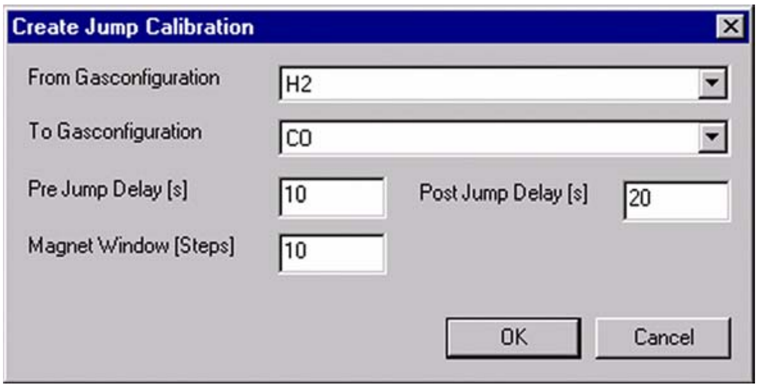
HV: high voltage setting of Jump Calibration

NOTE: ***If no Jump Calibration is available in the list, create a new one as shown below.***

Comment: How to create a ***new*** Jump Calibration



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



From Gasconfiguration: H2
To Gasconfiguration: CO
Pre Jump Delay [s]: 10
Post Jump Delay [s]: 20
Magnet Window [Steps]: 10

- *From Gasconfiguration* select ***H2***.
- *To Gasconfiguration* choose ***CO***.

  Accept the default values.

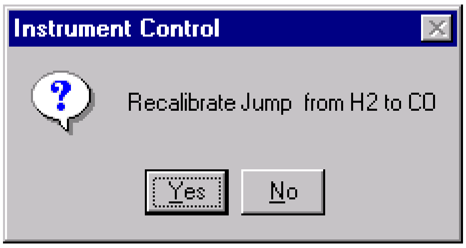
- Press ***OK***.

NOTE: ***Magnet jumps are always from a low mass to a high mass (e.g. 2 to 28 at $H_2 \rightarrow CO$ or 28 to 44 at $N_2 \rightarrow CO_2$).***

Make sure the Reference Port for CO is open.

Continue?

- Confirm by ***Yes***, because you have already opened the Reference Port.



Instrument Control

Recalibrate Jump from H2 to CO

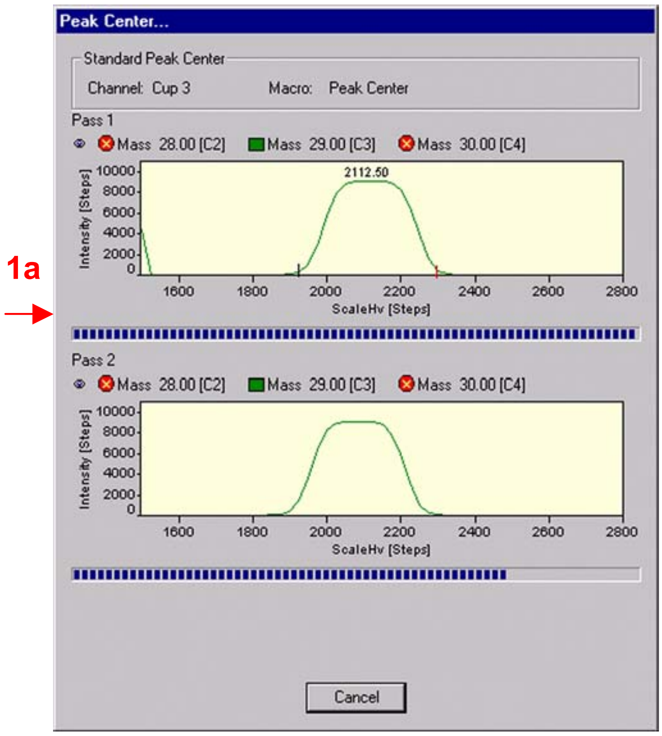
- Press ***Yes*** to start Jump Calibration.

Jump Calibration Procedure

1a Jump to CO (along hysteresis curve).
1b Perform a peak center for CO in order to get the signal height.

2 Jump to H_2 (along hysteresis curve; H_2 is origin).

3a Jump to CO (***not*** along hysteresis curve).
3b Perform a peak center for CO in order to catch the peak.



Peak Center...

Standard Peak Center

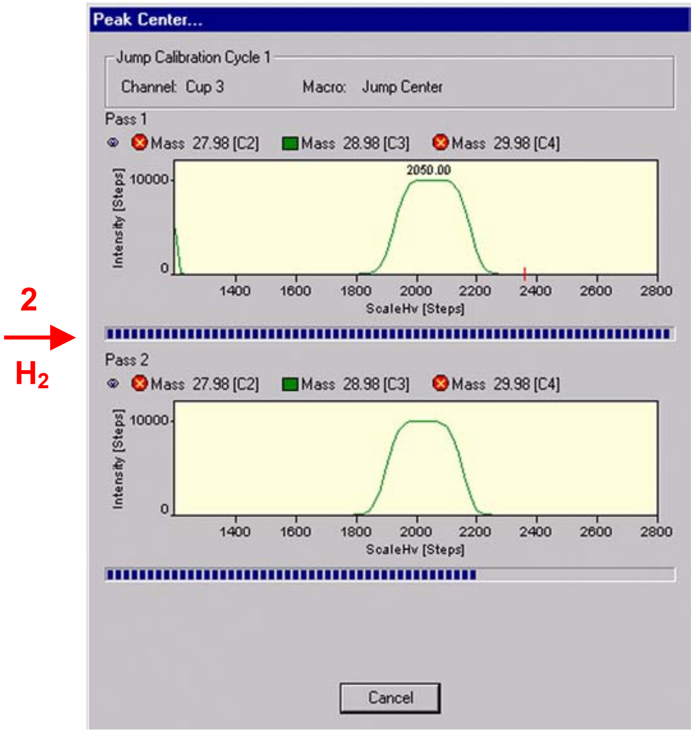
Channel: Cup 3 Macro: Peak Center

Pass 1

Mass 28.00 [C2] Mass 29.00 [C3] Mass 30.00 [C4]

Pass 2

Mass 28.00 [C2] Mass 29.00 [C3] Mass 30.00 [C4]



Peak Center...

Jump Calibration Cycle 1

Channel: Cup 3 Macro: Jump Center

Pass 1

Mass 27.98 [C2] Mass 28.98 [C3] Mass 29.98 [C4]

Pass 2

Mass 27.98 [C2] Mass 28.98 [C3] Mass 29.98 [C4]

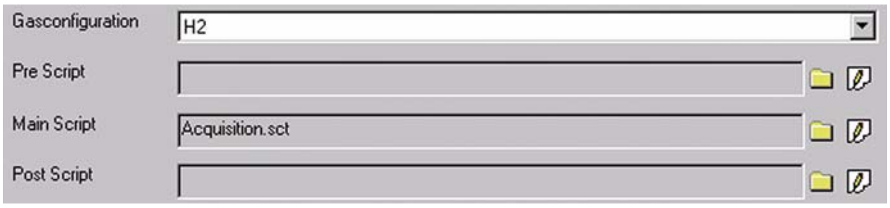
Repeat until magnet field setting is within the “magnet window” defined.

5.3.2 HOW TO START A DUAL MEASUREMENT

1 Determine a ***H3-Factor*** as described in Chapter 5.2.2.

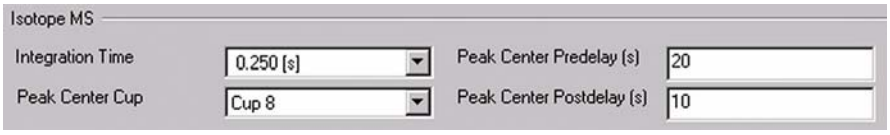
2 Define a ***Method*** (as shown in Chapter 5.1.3).
As a guideline, use the following Method ***("HD_CO.met")***:

Instrument



Gasconfiguration: H2
Pre Script:
Main Script: Acquisition.sct
Post Script:

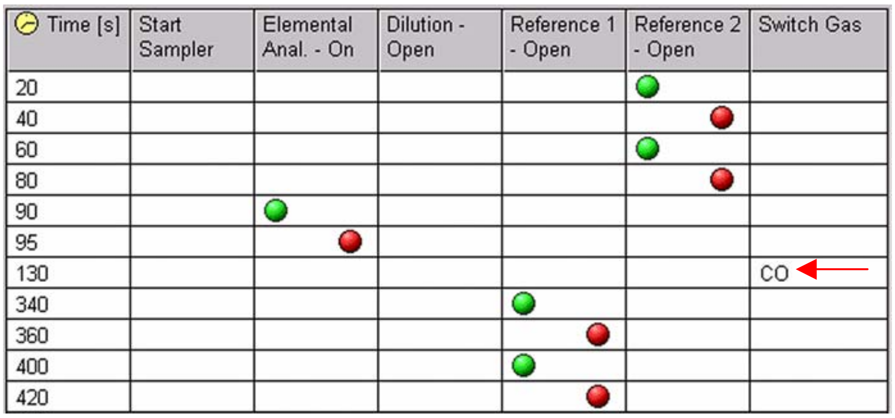
Isotope MS



Isotope MS
Integration Time: 0.250 [s]
Peak Center Cup: Cup 8
Peak Center Predelay [s]: 20
Peak Center Postdelay [s]: 10

Reference Port: Reference 2

Time Events



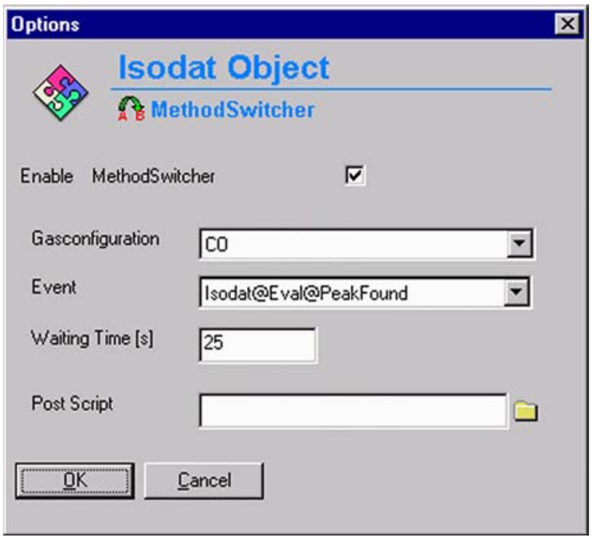
Time [s]	Start Sampler	Elemental Anal. - On	Dilution - Open	Reference 1 - Open	Reference 2 - Open	Switch Gas
20					🟢	
40					🔴	
60					🟢	
80					🔴	
90		🟢				
95		🔴				
130						CO
340				🟢		
360				🔴		
400				🟢		
420				🔴		

- Double-click on ***CO*** in the ***Switch Gas*** column to edit.

  (The Event Time is active since e.g. 130 s).

Acq. End Time

Acquisition Time [s]: 450



Enable MethodSwitcher ☑

Gasconfiguration: CO

Event: Isodat@Eval@PeakFound

Waiting Time [s]: 25

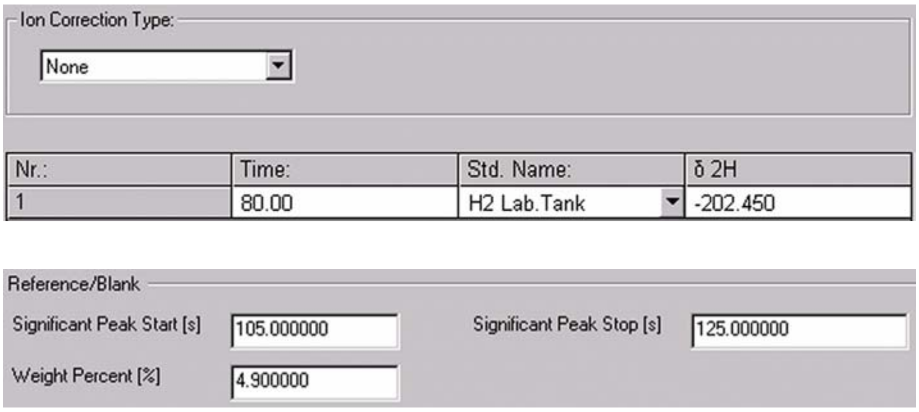
Post Script:

- The Gas Configuration to be switched to is selected in the ***Gasconfiguration*** menu (e.g. "CO").
- The switch will be performed when the peak has been found. This is defined in the ***Event*** menu (e.g. "Isodat@Eval@PeakFound").
- Type in the last jump time at ***Waiting Time***. At e.g. 130 s, waiting for the peak begins. If a peak is found, the jump takes place. If no peak is found, the waiting time elapses before the jump takes place.
- Select a ***Post Script***, which will be executed during the jump.

NOTE: ***For samples with a high O to H ratio, the activation of the dilution can be directly linked to the magnet jump to CO. In this case, use the He dilution Script.***

NOTE: ***For H_2 and CO <u>different</u> Evaluation tabs, Peak Detection tabs and Printout tabs appear (e.g. "Evaluation@H2", "Evaluation@CO2").***

<u>Evaluation@H2</u>



Ion Correction Type: None

Nr.:	Time:	Std. Name:	δ 2H
1	80.00	H2 Lab.Tank	-202.450

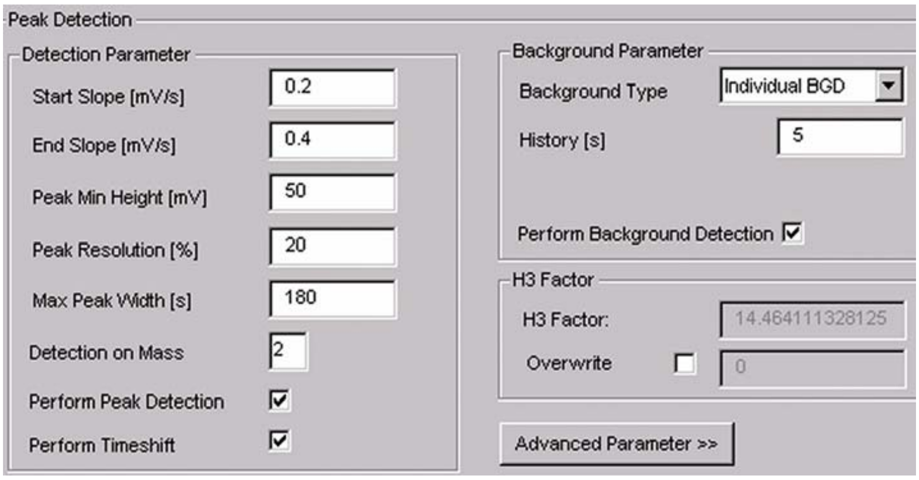
Reference/Blank

Significant Peak Start [s]: 105.000000

Significant Peak Stop [s]: 125.000000

Weight Percent [%]: 4.900000

Peak Detection@H2



Peak Detection

Detection Parameter

Start Slope [mV/s]	0.2
End Slope [mV/s]	0.4
Peak Min Height [mV]	50
Peak Resolution [%]	20
Max Peak Width [s]	180
Detection on Mass	2
Perform Peak Detection	☑
Perform Timeshift	☑

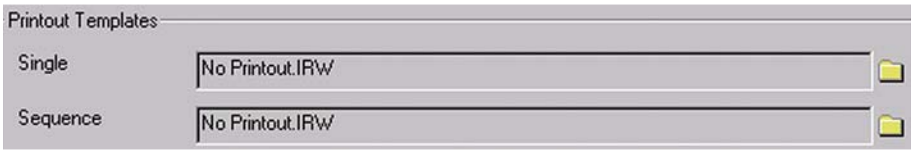
Background Parameter

Background Type	Individual BGD
History [s]	5
Perform Background Detection	☑

H3 Factor

H3 Factor:		14.464111328125
Overwrite	☐	0

Printout@H2

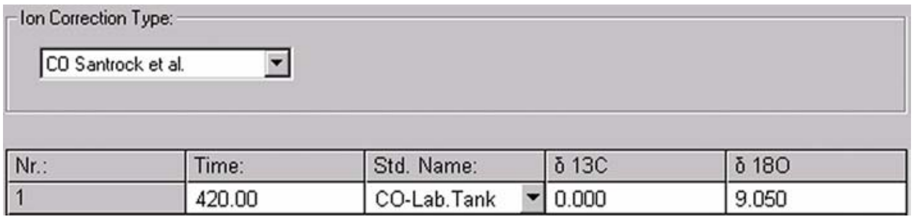


Printout Templates

Single	No Printout.IRW
Sequence	No Printout.IRW

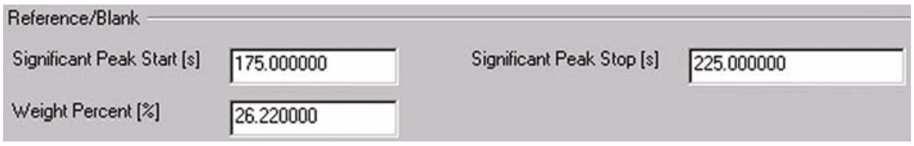
NOTE: ***Usually, the printout of results is performed not until the complete measurement has been finished. Therefore, it is recommended to choose "No Printout.irw" at "Printout@H2" tab, because at this moment, data acquisition is still running. Instead, printout options are defined at the end at "Printout@CO" tab.***

Evaluation@CO



Ion Correction Type: CO Santrock et al.

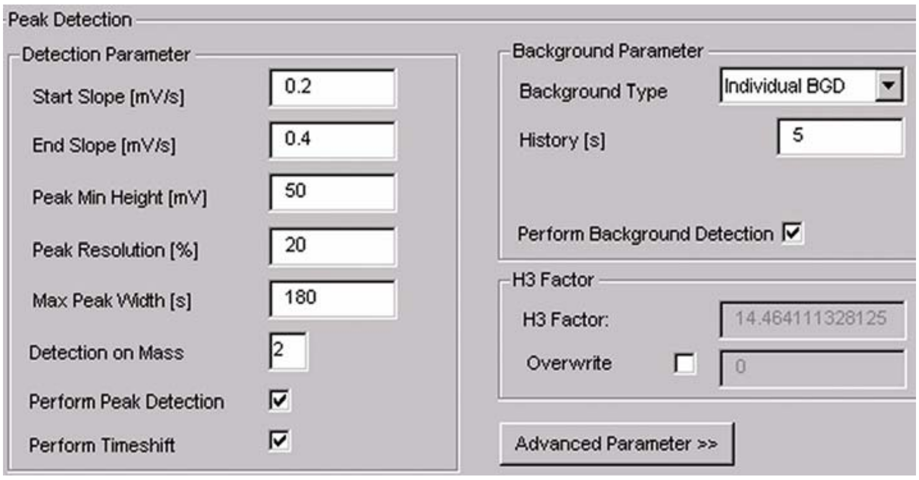
Nr.:	Time:	Std. Name:	δ 13C	δ 18O
1	420.00	CO-Lab.Tank	0.000	9.050



Reference/Blank

Significant Peak Start [s]	175.000000	Significant Peak Stop [s]	225.000000
Weight Percent [%]	26.220000		

Peak Detection@CO



Peak Detection

Detection Parameter

Start Slope [mV/s]: 0.2

End Slope [mV/s]: 0.4

Peak Min Height [mV]: 50

Peak Resolution [%]: 20

Max Peak Width [s]: 180

Detection on Mass: 2

Perform Peak Detection ☑

Perform Timeshift ☑

Background Parameter

Background Type: Individual BGD

History [s]: 5

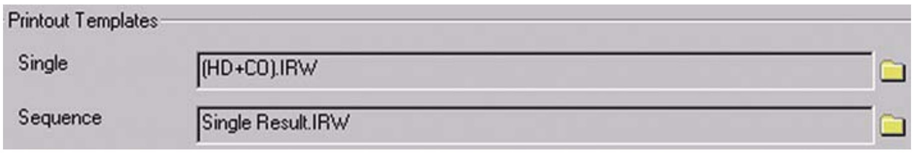
Perform Background Detection ☑

H3 Factor

H3 Factor: 14.464111328125

Overwrite ☐ 0

Printout @CO



Printout Templates

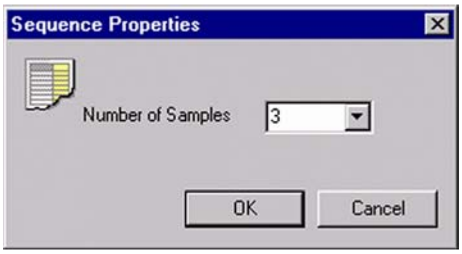
Single: (HD+CO).IRW

Sequence: Single Result.IRW

3 Place a sample (e.g. 0.281 mg of benzoic acid) in the solid-autosampler.

4 ***Create a new Sequence***

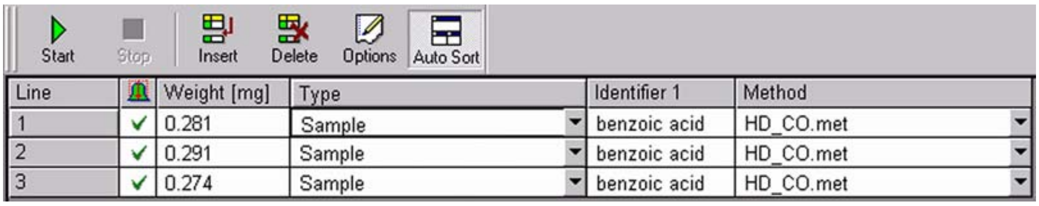
5 Define number of samples (e.g. 3).



Sequence Properties

Number of Samples: 3

6 Edit the Sequence list.

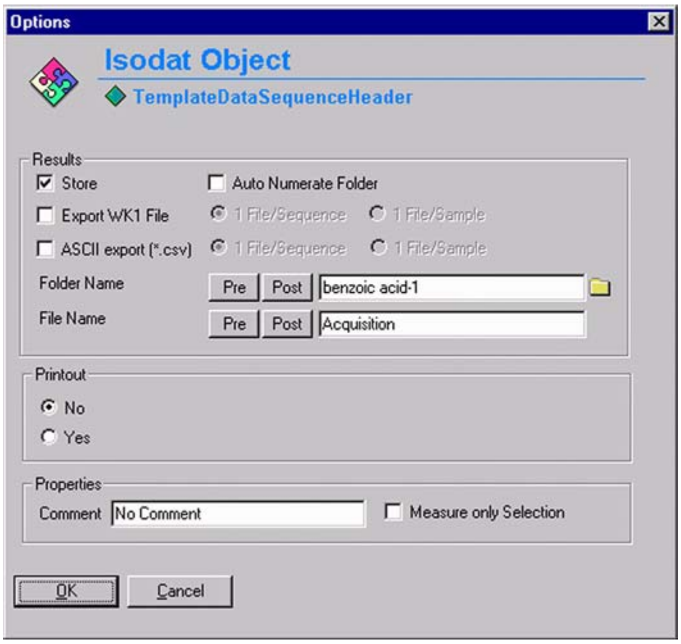


Line		Weight [mg]	Type	Identifier 1	Method
1	✓	0.281	Sample	benzoic acid	HD_CO.met
2	✓	0.291	Sample	benzoic acid	HD_CO.met
3	✓	0.274	Sample	benzoic acid	HD_CO.met

Peak Center	Enable ☑ to perform a Peak Center prior to measurement.
Weight	Type in sample mass [mg].
Type	Select kind of species (e.g. “Sample”).
Identifier 1	Edit text to identify sample.
Method	Select IRMS Method (e.g. “HD_CO.met”).

7 Press the ***Start*** button.

8 Define Results Storage, Export and Printout parameters.



Results

☑ Store ☐ Auto Numerate Folder

☐ Export WK1 File ● 1 File/Sequence ○ 1 File/Sample

☐ ASCII export (*.csv) ● 1 File/Sequence ○ 1 File/Sample

Folder Name: benzoic acid-1

File Name: Acquisition

Printout

● No

○ Yes

Properties

Comment: No Comment ☐ Measure only Selection

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

Events during Acquisition

- Peak Center procedure
- H_2 Reference Gas pulse activated at 20 s (duration: 20 s).
- H_2 Reference Gas pulse activated at 60 s (duration: 20 s).
- Sample is dropped into reactor.
- Sample peak (H_2) appears approximately 15 s after reaction start.
- Sample peak (CO) appears approximately 100 s after reaction start.
- CO Reference Gas pulse activated at 340 s (duration: 20 s).
- CO Reference Gas pulse activated at 400 s (duration: 20 s).
- Acquisition stops at 450 s.

ISODAT NT

OPERATING MANUAL

6

RESULT HANDLING

Issue 06/2002

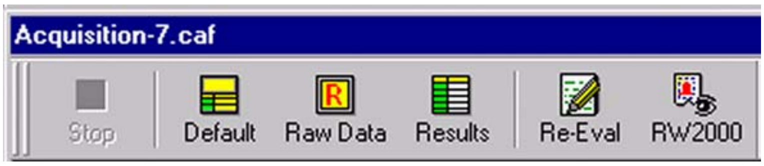
Thermo Finnigan

6.1 HANDLING OF RESULTS

6.1.1 DUAL INLET RESULTS

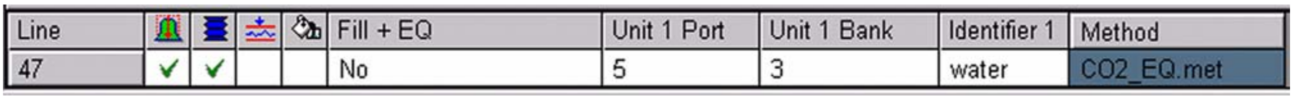
Toolbar Commands

After the measurement has been started by Start, a ***classical acquisition file***, i.e. ****.caf***, is generated (e.g. “Acquisition-7.caf”). The Acquisition window opens. It contains the toolbar shown below with the following Icons:



- A running Dual Inlet data acquisition can be stopped. Stop
- The data are shown ***both*** in a data grid and graphically (i.e. both in medium size). Default
- The ***graphical*** representation of the raw data is shown in maximum size (i.e. without data grid). Raw Data
- The ***result grid*** is shown in maximum size (i.e. without graphical representation). Results
- Some parameters of the Method can be changed (not the Method itself). Afterwards, the recorded data are reevaluated using the new parameter values. Re-Eval
- Opens a Result Workshop Template containing the data (e.g. for preparing a printout). RW2000

Sequence line



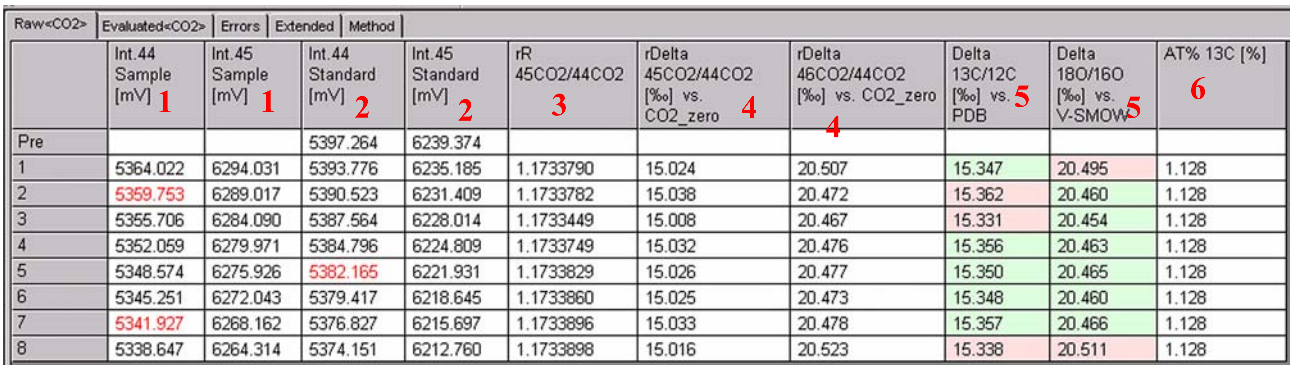
Line	🟩	☰	≋	✎	Fill + EQ	Unit 1 Port	Unit 1 Bank	Identifier 1	Method
47	✓	✓			No	5	3	water	CO2_EQ.met

A particular line of the chosen Sequence is shown. It denotes the currently measured sample.

The tabs of the Acquisition window

The Acquisition window offers the following tabs for data interpretation:

Raw tab



	Int.44 Sample [mV] 1	Int.45 Sample [mV] 1	Int.44 Standard [mV] 2	Int.45 Standard [mV] 2	rR 45CO2/44CO2 3	rDelta 45CO2/44CO2 [‰] vs. CO2_zero 4	rDelta 46CO2/44CO2 [‰] vs. CO2_zero 4	Delta 13C/12C [‰] vs. PDB 5	Delta 18O/16O [‰] vs. V-SMOW 5	AT% 13C [%] 6
Pre			5397.264	6239.374						
1	5364.022	6294.031	5393.776	6235.185	1.1733790	15.024	20.507	15.347	20.495	1.128
2	5359.753	6289.017	5390.523	6231.409	1.1733782	15.038	20.472	15.362	20.460	1.128
3	5355.706	6284.090	5387.564	6228.014	1.1733449	15.008	20.467	15.331	20.454	1.128
4	5352.059	6279.971	5384.796	6224.809	1.1733749	15.032	20.476	15.356	20.463	1.128
5	5348.574	6275.926	5382.165	6221.931	1.1733829	15.026	20.477	15.350	20.465	1.128
6	5345.251	6272.043	5379.417	6218.645	1.1733860	15.025	20.473	15.348	20.460	1.128
7	5341.927	6268.162	5376.827	6215.697	1.1733896	15.033	20.478	15.357	20.466	1.128
8	5338.647	6264.314	5374.151	6212.760	1.1733898	15.016	20.523	15.338	20.511	1.128

In case of ***Dual Inlet*** applications, ***intensity*** values are determined (while in case of ***Continuous Flow*** applications, ***peak areas*** are calculated).

In the leftmost column, the ***sample number*** is displayed (e.g. “Pre” and “1” to “8”; cf. the lines of the chosen Sequence). Besides, the raw data grid contains the following columns:

1	**Int Sample**	**Sample intensity** of a particular mass in mV (e.g. mass 44). Each sample mass has a column of its own.
2	**Int. Standard**	**Standard intensity** *of a particular mass in mV (e.g. mass 44).* Each standard mass has a column of its own.

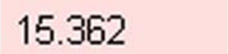
3	**rR**	**Raw ratio** of two masses, i.e. ratio of the intensities of these two masses (e.g. rR 45/44).
4	**rDelta**	**Raw Delta** of two molecular masses (in ‰) vs. a secondary standard - i.e. vs. a user standard. E.g.: rDelta [45 CO2/44 CO2] vs. CO2_zero. These values are **molecule** deltas and are calculated from the raw ratios.
5	**Delta**	**Delta** value of two element isotopes (in ‰) vs. a primary standard. E.g.: Delta [13C/12C] vs V-PDB or Delta [18O/16O] vs V-SMOW. These values are ***element*** deltas and are calculated from the true element sample ratios and the true element standard ratios. The true element standard ratios are read from primary standards defined in the primary standard database.
6	**At %**	**Atomic percentage** value of an isotope, e.g.: At % [13C] or At % [18O]. It is calculated from the element delta and the corresponding absolute element ratio of the standard.

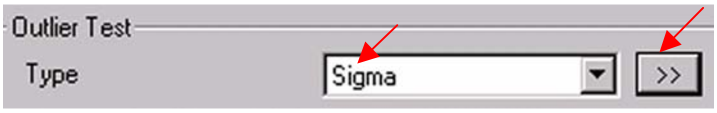
Kinds of data in the raw data grid

- A red value indicates that during integration the timeout criterion was reached. Red numbers only occur in case of intensities, i.e. in the columns ***Int. Sample*** and ***Int. Standard***.


5382.165

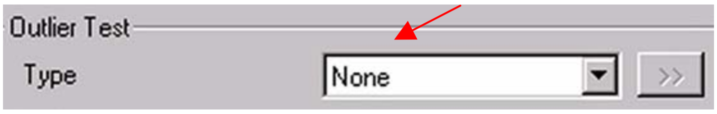
- A black value on a pink background is an outlier relating to the particulars given at the “Evaluation” tab’s “Outlier Test”:


15.362



Type Sigma

If ***Sigma*** was selected there and specified by >> , outliers may occur and are marked as shown above. They only occur in case of ***Delta*** values, e.g. Delta [13C/12C] vs. V-PDB

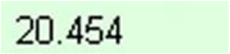


Type None

If ***None*** was selected there, no outliers are calculated at all. Therefore, no black values on a pink background will occur.

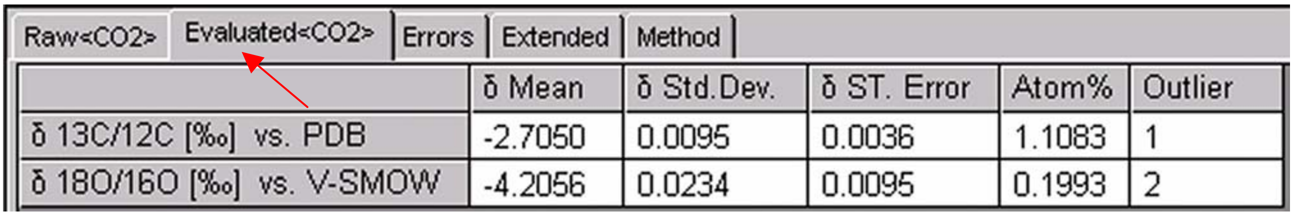
Suppose, that outliers have occurred (i.e. the case of ***Sigma***). If you then decide to reevaluate these data using a changed Method (yia the ***Method*** tab) selecting ***None*** at the “Evaluation” tab’s “Outlier Test”, no more outliers will be calculated. Thus, the values will turn into normal black ones:

- A black value on a green background is a Normal value falling into the k-fold of σ (i.e. no outlier). They only occur in case of ***Delta*** values, e.g. Delta [13C/12C] vs. V-PDB or Delta [18O/16O] vs. V-SMOW.



20.454

Evaluated tab



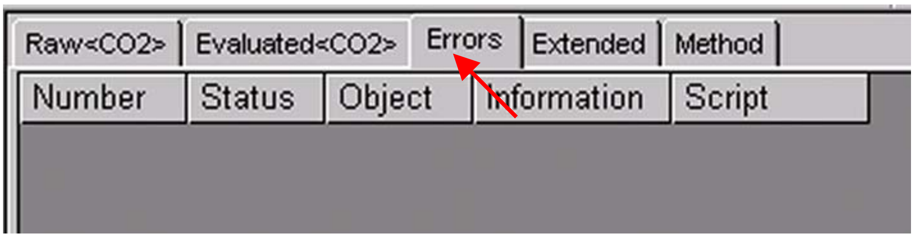
	δ Mean	δ Std.Dev.	δ ST. Error	Atom%	Outlier
δ 13C/12C [‰] vs. PDB	-2.7050	0.0095	0.0036	1.1083	1
δ 18O/16O [‰] vs. V-SMOW	-4.2056	0.0234	0.0095	0.1993	2

The ***Delta*** values (i.e. δ 13C/12C vs. V-PDB and δ 18O/16O vs. V-SMOW) marked pink or green in the raw data grid's columns **5** undergo a statistical analysis here:

δ Mean	Mean of the ***Delta*** values noted in column **5** (e.g. Mean of all Delta 18O/16O values in column **5**).
δ Std. Dev.	Standard deviation of the ***Delta*** values noted in column **5**. (e.g. Standard deviation of all Delta 18O/16O values in column **5**).
δ St. Error	Standard error of the ***Delta*** values noted in column **5** (e.g. Standard error of all Delta 18O/16O values in column **5**).
Atom-%	Atomic percentage value of the heavier isotope. It is calculated from the ***δ Mean*** value (e.g. Atom-% of ^{13}C is calculated from δ [13C/12C]).

➢ ***Outlier***	Number of outliers in column **5** entering into calculation of δ Mean, δ Std. Dev and δ St. Error (e.g. 2).

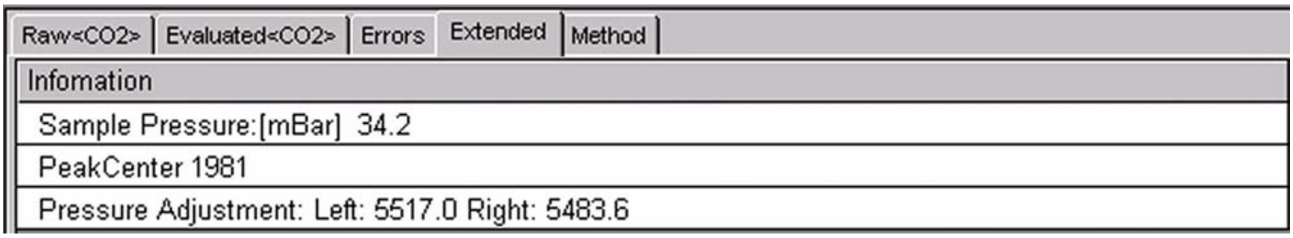
Errors tab



The “Errors” tab contains detailed information about errors that occurred during Data Acquisition:

➢ ***Number***	The consecutive number of the error (i.e. 1; 2; 3…)
➢ ***Status***	The quality or gravidity of the error (e.g. fatal; warning; error)
➢ ***Object***	Object in the Script that caused the particular error
➢ ***Information***	Explanation concerning the error
➢ ***Script***	Script that caused the error

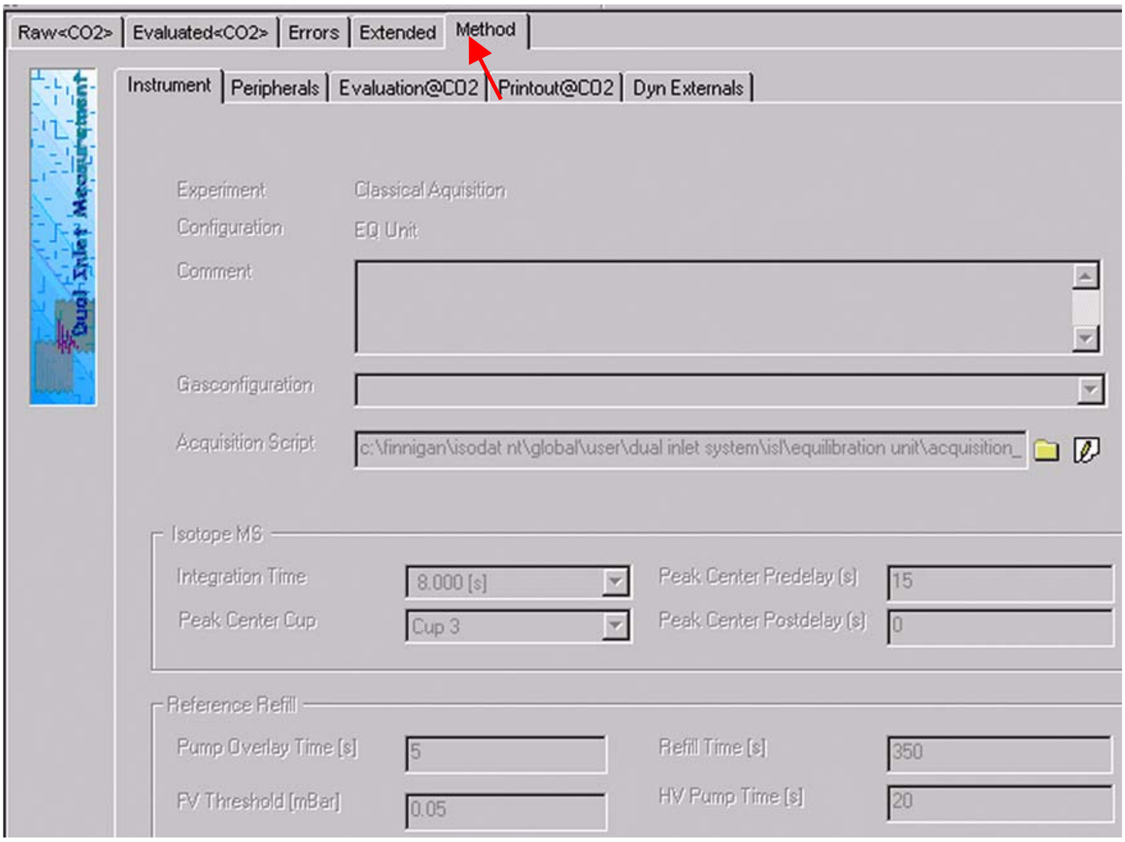
Extended tab



Infomation
Sample Pressure:[mBar] 34.2
PeakCenter 1981
Pressure Adjustment: Left: 5517.0 Right: 5483.6

In the “Extended” tab, a measurement protocol is presented: detailed information is given about particular results during measurement progression (e.g. Peak Center results or Pressure Adjustment results). The kind of information depends on the selected ***Configuration***; e.g. different information in case of “Dual Inlet + Microvolume” compared to “Dual Inlet + H-Device”.

Method tab



Experiment: Classical Aquisition
Configuration: EQ Unit
Comment:
Gasconfiguration:
Acquisition Script: c:\finnigan\isodat nt\global\user\dual inlet system\isl\equilibration unit\acquisition_

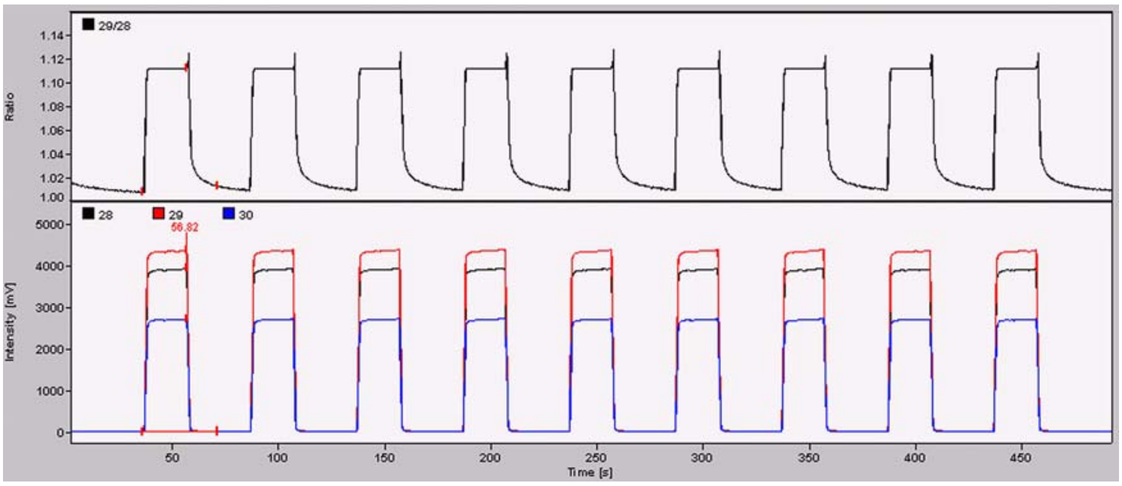
Isotope MS
Integration Time: 8.000 [s]
Peak Center Cup: Cup 3
Peak Center Predelay [s]: 15
Peak Center Postdelay [s]: 0

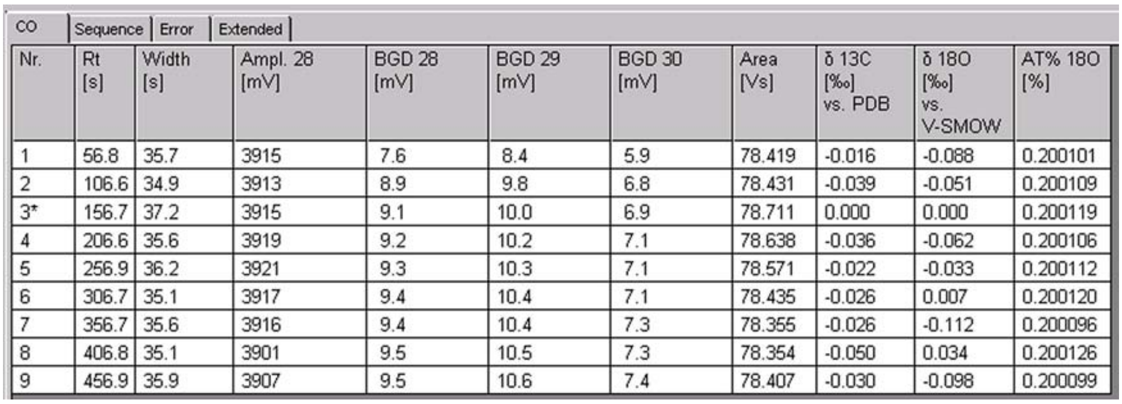
Reference Refill
Pump Overlay Time [s]: 5
Refill Time [s]: 350
PV Threshold [mBar]: 0.05
HV Pump Time [s]: 20

For Data Reevaluation purposes, some parameters of the Method selected prior to measurement start can be modified (mainly in the “Peripherals” tab and the “Evaluation” tab; click the tabs in order to determine the changeable parameters and then type in the new corrected values). After the parameters of interest have been modified, reevaluation of the data can be started. Therefore, press the ***Re-Eval*** button.

6.1.2 CONTINUOUS FLOW RESULTS

6.1.2.1 OXYGEN RESULTS





Nr.	Rt [s]	Width [s]	Ampl. 28 [mV]	BGD 28 [mV]	BGD 29 [mV]	BGD 30 [mV]	Area [Vs]	δ 13C [‰] vs. PDB	δ 18O [‰] vs. V-SMOW	AT% 18O [%]
1	56.8	35.7	3915	7.6	8.4	5.9	78.419	-0.016	-0.088	0.200101
2	106.6	34.9	3913	8.9	9.8	6.8	78.431	-0.039	-0.051	0.200109
3*	156.7	37.2	3915	9.1	10.0	6.9	78.711	0.000	0.000	0.200119
4	206.6	35.6	3919	9.2	10.2	7.1	78.638	-0.036	-0.062	0.200106
5	256.9	36.2	3921	9.3	10.3	7.1	78.571	-0.022	-0.033	0.200112
6	306.7	35.1	3917	9.4	10.4	7.1	78.435	-0.026	0.007	0.200120
7	356.7	35.6	3916	9.4	10.4	7.3	78.355	-0.026	-0.112	0.200096
8	406.8	35.1	3901	9.5	10.5	7.3	78.354	-0.050	0.034	0.200126
9	456.9	35.9	3907	9.5	10.6	7.4	78.407	-0.030	-0.098	0.200099

To calculate the standard deviation of ***delta 29 / 28*** ($^{13}C^{16}O$ / $^{12}C^{16}O$):

- Hold the Shift-key and mark the ***δ 13C vs. PDB*** column by a right-click.
- Then choose ***Calculate*** and finally look for the ***Std. Dev.*** value.

NOTE: ***The standard deviation of delta 29 / 28 ($^{13}C^{16}O$ / $^{12}C^{16}O$) should be < 0.08 ‰.***

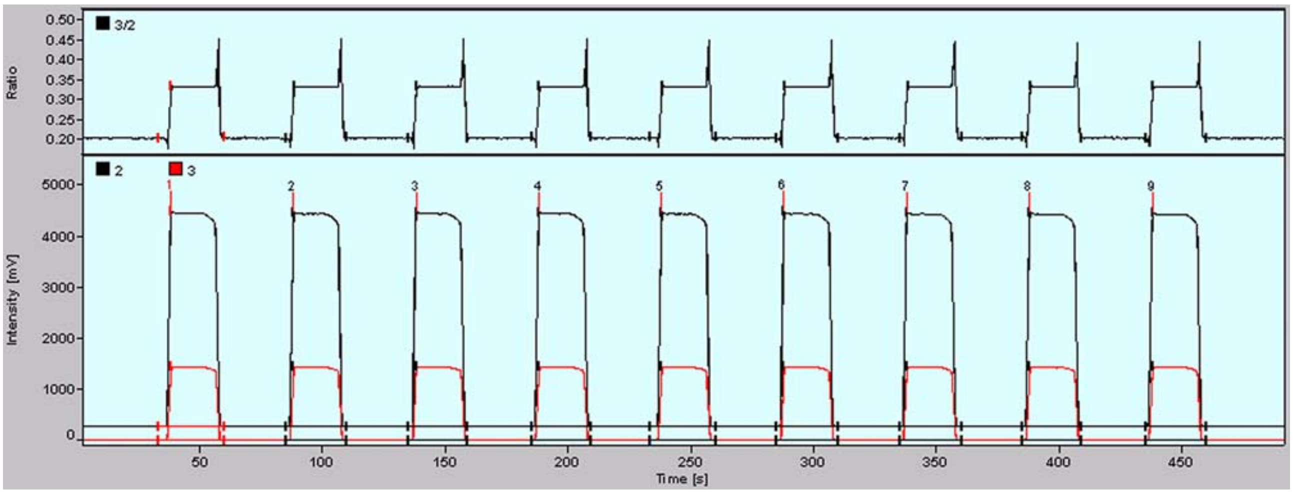
To calculate the standard deviation of ***delta 18 / 16*** ($^{12}C^{18}O$ / $^{12}C^{16}O$):

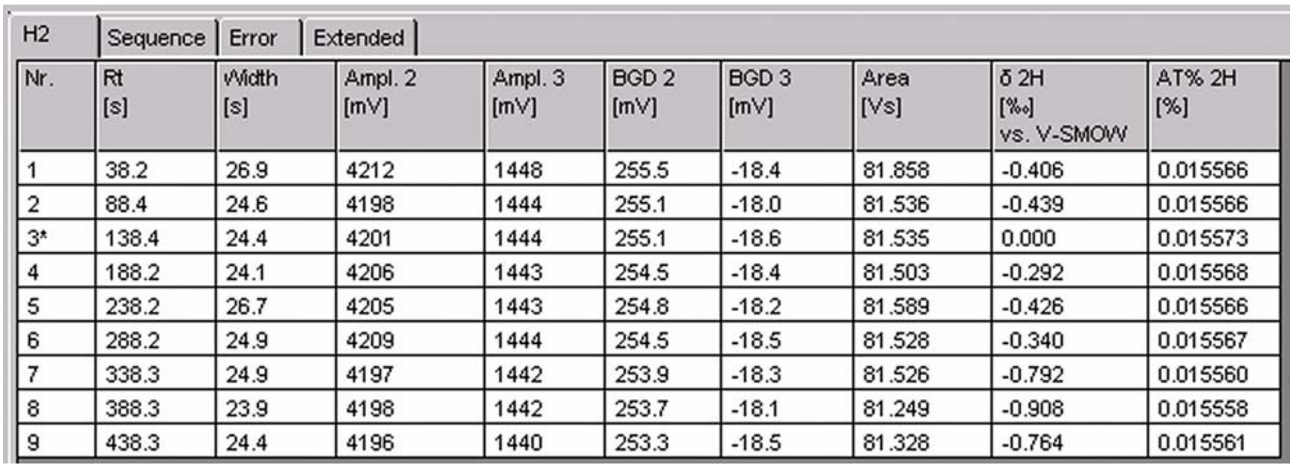
- Hold the Shift-key and mark the ***δ 18 O vs. V-SMOW*** column by a right-click.
- Then choose ***Calculate*** and finally look for the ***Std. Dev.*** value.

NOTE: ***The standard deviation of delta 18 / 16 ($^{12}C^{18}O$ / $^{12}C^{16}O$) should be < 0.1 ‰.***

6.1.2.2 HYDROGEN RESULTS

C:\Finnigan\ISODAT NT\Global\User\Conflo II Interface\Results\ACQ-Results\010402_124518_stab.CF





Nr.	Rt [s]	Width [s]	Ampl. 2 [mV]	Ampl. 3 [mV]	BGD 2 [mV]	BGD 3 [mV]	Area [Vs]	δ 2H [‰] vs. V-SMOW	AT% 2H [%]
1	38.2	26.9	4212	1448	255.5	-18.4	81.858	-0.406	0.015566
2	88.4	24.6	4198	1444	255.1	-18.0	81.536	-0.439	0.015566
3*	138.4	24.4	4201	1444	255.1	-18.6	81.535	0.000	0.015573
4	188.2	24.1	4206	1443	254.5	-18.4	81.503	-0.292	0.015568
5	238.2	26.7	4205	1443	254.8	-18.2	81.589	-0.426	0.015566
6	288.2	24.9	4209	1444	254.5	-18.5	81.528	-0.340	0.015567
7	338.3	24.9	4197	1442	253.9	-18.3	81.526	-0.792	0.015560
8	388.3	23.9	4198	1442	253.7	-18.1	81.249	-0.908	0.015558
9	438.3	24.4	4196	1440	253.3	-18.5	81.328	-0.764	0.015561

A chromatogram with equal peak heights results. Beneath the chromatogram, the corresponding parameter values are listed.

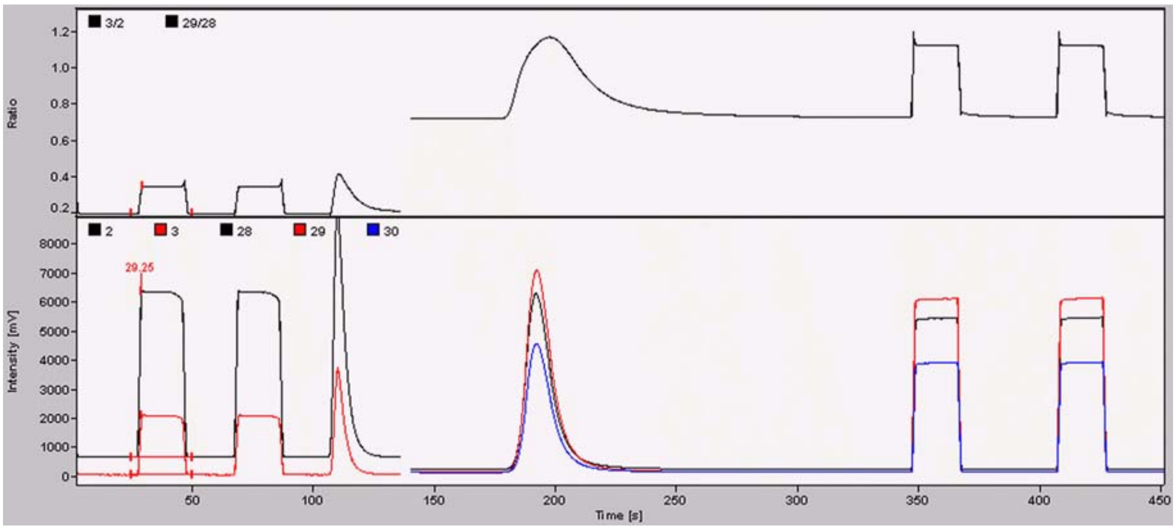
NOTE:* *The mean value of standard deviation of delta 3/2 should be < 1‰.

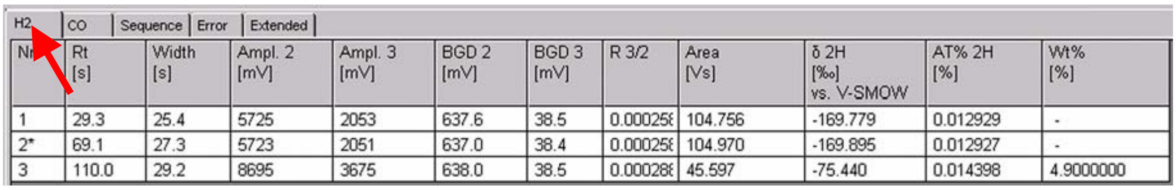
6.1.2.3 DUAL MEASUREMENT RESULTS

After finishing data acquisition the printer creates a data output sheet as defined by the selected Result Workshop template (*.irw). The Results will also be exported to a spreadsheet file, if activated.

C:\FINNIGAN\ISODAT NT\Global\User\Conflo II Interface\Results\Benzoic acid HD-CO.CF

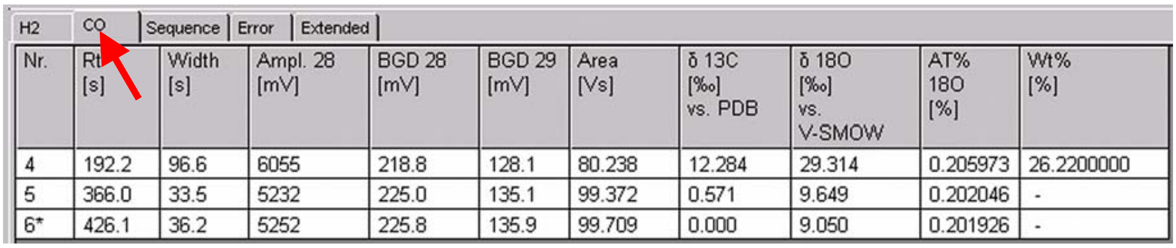
Line		Weight [mg]	Type	Identifier 1	Method
1	✓	0.281	Sample	benzoic acid	HD_CO.met





Nr	Rt [s]	Width [s]	Ampl. 2 [mV]	Ampl. 3 [mV]	BGD 2 [mV]	BGD 3 [mV]	R 3/2	Area [Vs]	δ 2H [‰] vs. V-SMOW	AT% 2H [%]	Wt% [%]
1	29.3	25.4	5725	2053	637.6	38.5	0.00025	104.756	-169.779	0.012929	-
2*	69.1	27.3	5723	2051	637.0	38.4	0.00025	104.970	-169.895	0.012927	-
3	110.0	29.2	8695	3675	638.0	38.5	0.00028	45.597	-75.440	0.014398	4.9000000

The ***H2*** tab reveals information about the hydrogen related peaks.



Nr.	Rt [s]	Width [s]	Ampl. 28 [mV]	BGD 28 [mV]	BGD 29 [mV]	Area [Vs]	δ 13C [‰] vs. PDB	δ 18O [‰] vs. V-SMOW	AT% 18O [%]	Wt% [%]
4	192.2	96.6	6055	218.8	128.1	80.238	12.284	29.314	0.205973	26.2200000
5	366.0	33.5	5232	225.0	135.1	99.372	0.571	9.649	0.202046	-
6*	426.1	36.2	5252	225.8	135.9	99.709	0.000	9.050	0.201926	-

The ***CO*** tab reveals information about the oxygen related peaks.

6.2 EXCEL EXPORT OF RESULTS

6.2.1 PRINCIPLE OF EXCEL EXPORT

Principle: Measurement parameters and results are exportable as an Excel file. The Excel export can easily be tailored to your individual needs. Therefore, rules must be established to determine how to perform the export. These export rules are described in an export ***template*** (i.e. a ***wke*** file).

Various ***selectors***, which group Identifiers, are used to create an export template. For an export, more than one template can be selected. Multiple templates can be used simultaneously. Every single template (i.e. every wke file) finally leads to an export file of its own, its ***wk1*** file. Every wk1 file is created according to the rules of the wke file.

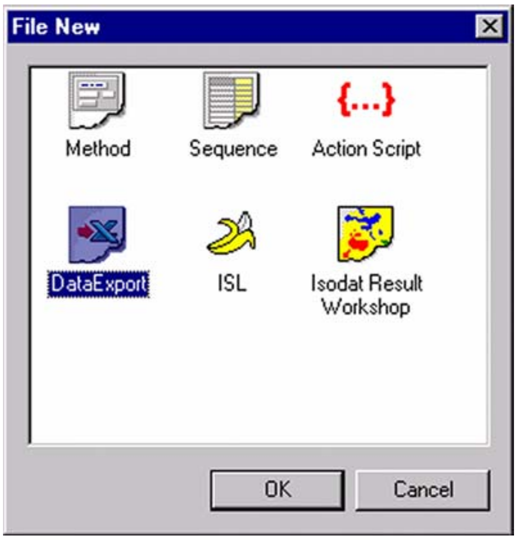
Advantages: Although measurement data are stored in one result file, the data concerning ***every single gas type*** can be exported separately to an export file of their own (e.g. multigas measurement).

Single ***parts*** of a measurement can be exported to an export file of their own (e.g. only the Method parameters).

Some types of data can be specified further using ***query tabs***, where sophisticated export properties are defined (e.g. peak relevant data).

6.2.2 CREATING AN EXPORT TEMPLATE

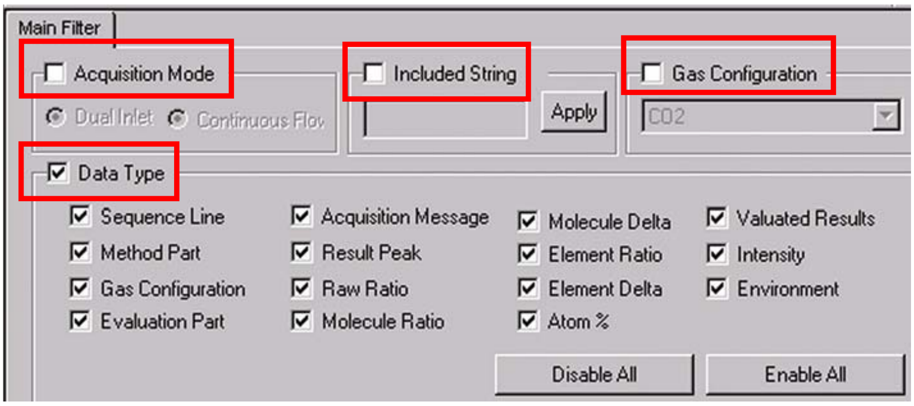
- On the Dual Inlet window's toolbar, press the ***New*** button.



- Click ***Data Export*** and confirm by ***OK***.

Thus, a new export template can be edited.

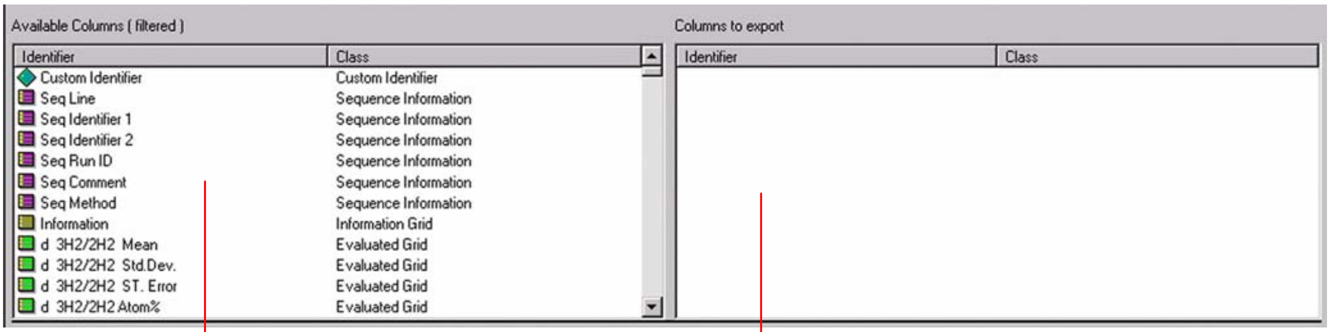
- Four selector types can be selected.



☐ Acquisition Mode
○ Dual Inlet ○ Continuous Flow
☐ Included String
☐ Gas Configuration
CO2
☑ Data Type
☑ Sequence Line
☑ Method Part
☑ Gas Configuration
☑ Evaluation Part
☑ Acquisition Message
☑ Result Peak
☑ Raw Ratio
☑ Molecule Ratio
☑ Molecule Delta
☑ Element Ratio
☑ Element Delta
☑ Atom %
☑ Valuated Results
☑ Intensity
☑ Environment

The four selector types for template creation

Selectors facilitate the Excel-Export: the list of exportable data can be shortened (see the Identifiers in the left pane). Without selectors, all exportable data are visible.



Available Columns (filtered)

Identifier	Class
Custom Identifier	Custom Identifier
Seq Line	Sequence Information
Seq Identifier 1	Sequence Information
Seq Identifier 2	Sequence Information
Seq Run ID	Sequence Information
Seq Comment	Sequence Information
Seq Method	Sequence Information
Information	Information Grid
d 3H2/2H2 Mean	Evaluated Grid
d 3H2/2H2 Std.Dev.	Evaluated Grid
d 3H2/2H2 ST. Error	Evaluated Grid
d 3H2/2H2 Atom%	Evaluated Grid

Columns to export

Identifier	Class

available pool of Identifers (columns)

Identifiers (columns) to be exported

NOTE: ***Selectors are always combined by a conjunction (i.e. by an AND relation).***

NOTE: ***(De-)activation of Selectors can be changed during template creation.***

The export columns will not be affected.

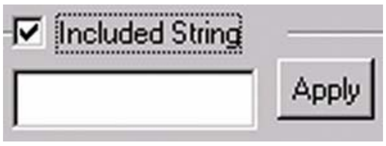
1 *Acquisition Mode*

☑ Acquisition Mode

● Dual Inlet ● Continuous Flow

If Acquisition Mode is activated, choose via click between parameters, which are only relevant for Dual Inlet Acquisition and those only relevant for Continuous Flow Acquisition. As default, both parameter types are selected, i.e. no selector.

2 *Included String*



☑ Included String

If Included String is activated and you type a string (e.g. “Delta”), the entries in the left pane (“pool”) will be selected according to that string after pressing ***Apply***. The entries that match the string will be displayed in the right pane.

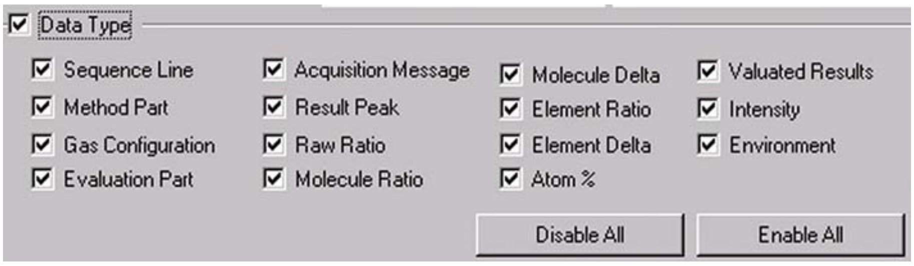
3 *Gas Configuration*

☑ Gas Configuration

CO2

If Gas Configuration is activated, choose a Gas Configuration from the list (e.g. "CO2"). All relevant data for this Gas Configuration will be shown in the right pane.

4 *Data Type*



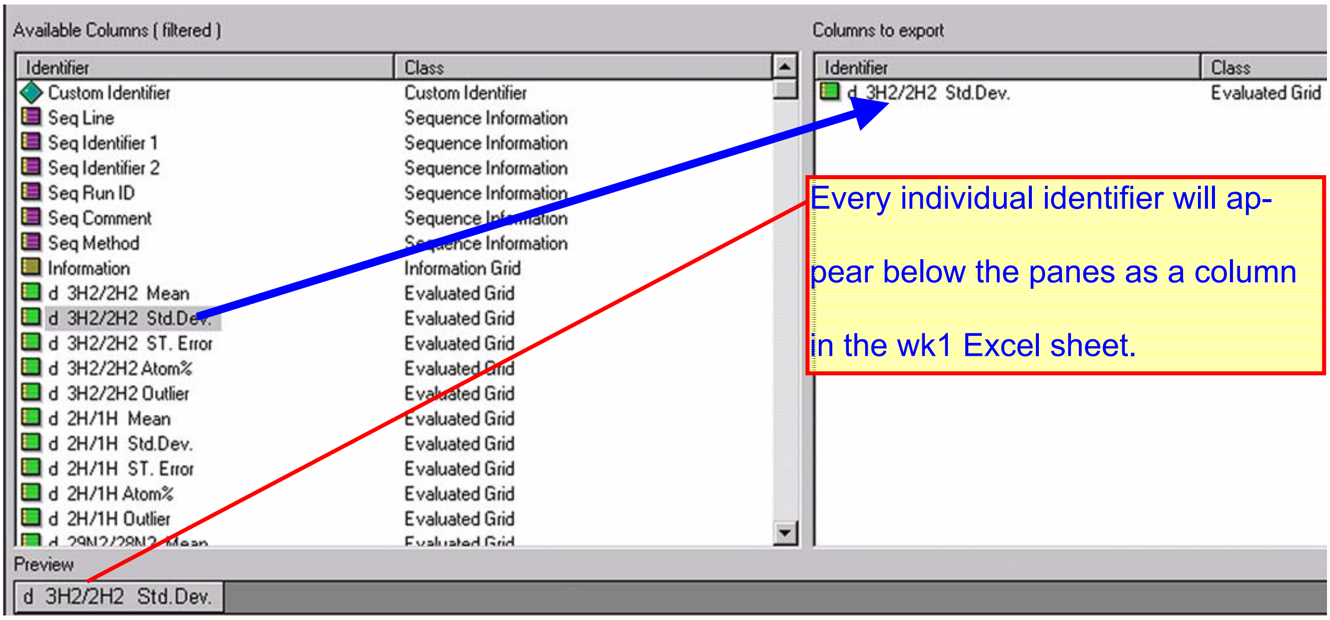
☑ Data Type

☑ Sequence Line
☑ Method Part
☑ Gas Configuration
☑ Evaluation Part
☑ Acquisition Message
☑ Result Peak
☑ Raw Ratio
☑ Molecule Ratio
☑ Molecule Delta
☑ Element Ratio
☑ Element Delta
☑ Atom %
☑ Valuated Results
☑ Intensity
☑ Environment

If activated, you can select according to data type. Activate one or more selection criteria (e.g. if only "Raw Ratio" is selected, merely entries concerning raw ratios will be displayed in the right pane). As default, all data types are activated. Use ***Disable All*** or ***Enable All*** to ease your work with the Data Type selector.

Individual Identifiers

In addition to selectors drag and drop ***individual*** Identifiers to the right pane to export them:



Available Columns (filtered)

Identifier	Class
Custom Identifier	Custom Identifier
Seq Line	Sequence Information
Seq Identifier 1	Sequence Information
Seq Identifier 2	Sequence Information
Seq Run ID	Sequence Information
Seq Comment	Sequence Information
Seq Method	Sequence Information
Information	Information Grid
d 3H2/2H2 Mean	Evaluated Grid
d 3H2/2H2 Std.Dev.	Evaluated Grid
d 3H2/2H2 ST. Error	Evaluated Grid
d 3H2/2H2 Atom%	Evaluated Grid
d 3H2/2H2 Outlier	Evaluated Grid
d 2H/1H Mean	Evaluated Grid
d 2H/1H Std.Dev.	Evaluated Grid
d 2H/1H ST. Error	Evaluated Grid
d 2H/1H Atom%	Evaluated Grid
d 2H/1H Outlier	Evaluated Grid
d 29N2/28N2 Mean	Evaluated Grid

Columns to export

Identifier	Class
d 3H2/2H2 Std.Dev.	Evaluated Grid

Preview

d 3H2/2H2 Std.Dev.

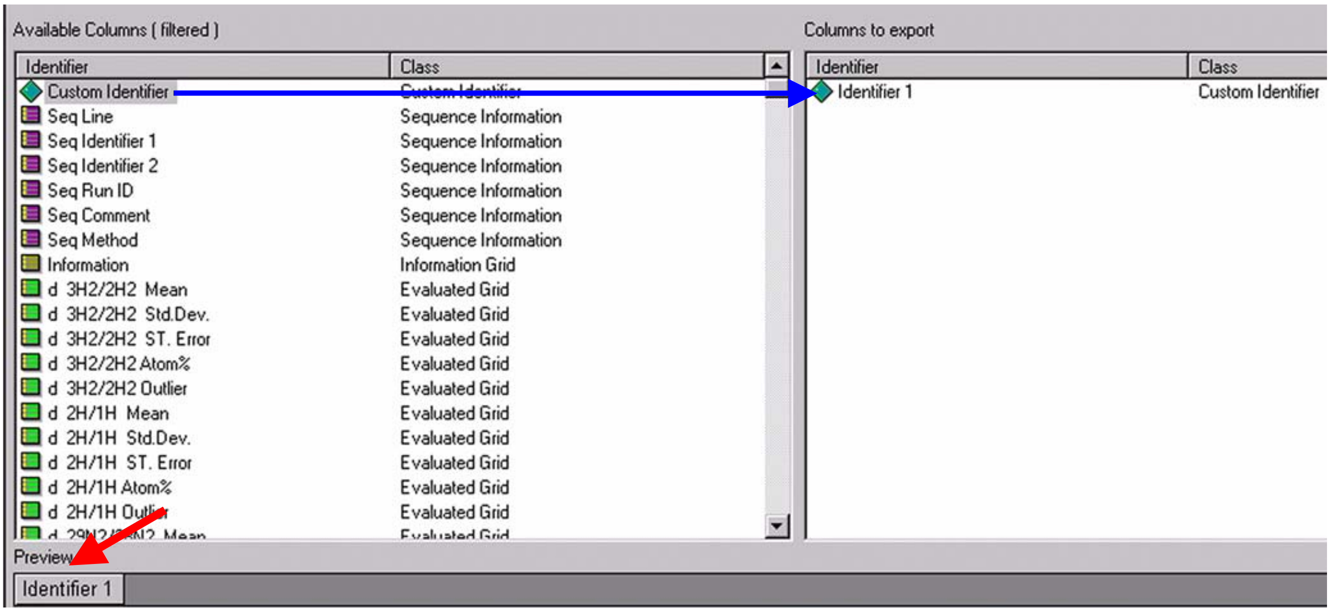
Every individual identifier will appear below the panes as a column in the wk1 Excel sheet.

Drag and drop of the ***Custom Identifier*** allows inserting a column that contains no data. You can give it a name of your own choice. Later on, you can, for example, import data into this column. During drag and drop of the Custom Identifier from the left to the right pane, the box shown below appears.


Insert Identifier

Custom Identifier for Export: Identifier 1

- ➢ Type a significant name for the custom identifier (e.g. “Identifier 1”).
- ➢ Press ***OK***.


Available Columns (filtered)

Identifier	Class
Custom Identifier	Custom Identifier
Seq Line	Sequence Information
Seq Identifier 1	Sequence Information
Seq Identifier 2	Sequence Information
Seq Run ID	Sequence Information
Seq Comment	Sequence Information
Seq Method	Sequence Information
Information	Information Grid
d 3H2/2H2 Mean	Evaluated Grid
d 3H2/2H2 Std.Dev.	Evaluated Grid
d 3H2/2H2 ST. Error	Evaluated Grid
d 3H2/2H2 Atom%	Evaluated Grid
d 3H2/2H2 Outlier	Evaluated Grid
d 2H/1H Mean	Evaluated Grid
d 2H/1H Std.Dev.	Evaluated Grid
d 2H/1H ST. Error	Evaluated Grid
d 2H/1H Atom%	Evaluated Grid
d 2H/1H Outlier	Evaluated Grid

Columns to export

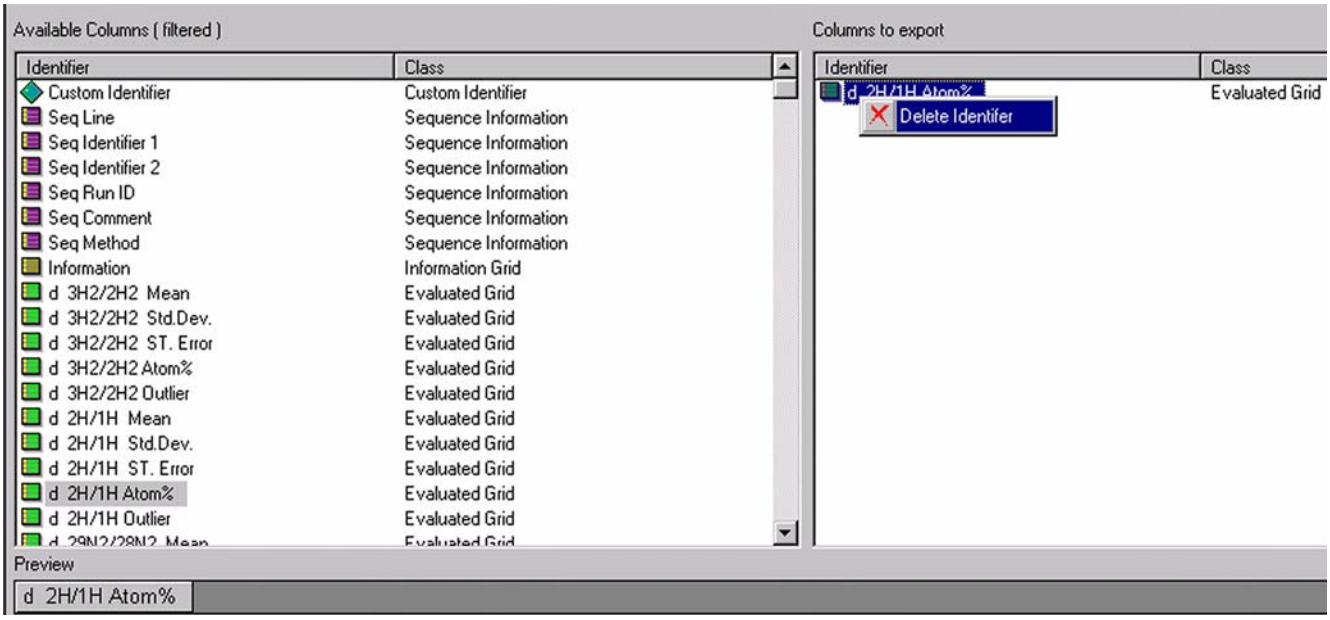
Identifier	Class
Identifier 1	Custom Identifier

Preview

Identifier 1

The Custom Identifier also appears as a column in the wk1 Excel sheet. To create additional empty columns, proceed with the next Custom Identifier(s) by repeating the steps above.

To remove one or more Identifiers (Custom Identifiers or other ones) from the right pane, mark them there. Then right-click them.



Identifier	Class
Custom Identifier	Custom Identifier
Seq Line	Sequence Information
Seq Identifier 1	Sequence Information
Seq Identifier 2	Sequence Information
Seq Run ID	Sequence Information
Seq Comment	Sequence Information
Seq Method	Sequence Information
Information	Information Grid
d 3H2/2H2 Mean	Evaluated Grid
d 3H2/2H2 Std.Dev.	Evaluated Grid
d 3H2/2H2 ST. Error	Evaluated Grid
d 3H2/2H2 Atom%	Evaluated Grid
d 3H2/2H2 Outlier	Evaluated Grid
d 2H/1H Mean	Evaluated Grid
d 2H/1H Std.Dev.	Evaluated Grid
d 2H/1H ST. Error	Evaluated Grid
d 2H/1H Atom%	Evaluated Grid
d 2H/1H Outlier	Evaluated Grid
d 29N2/28N2 Mean	Evaluated Grid

Identifier	Class
d 2H/1H Atom%	Evaluated Grid

d 2H/1H Atom%

Select ***Delete Identifier***.

Delete selected Objects ?

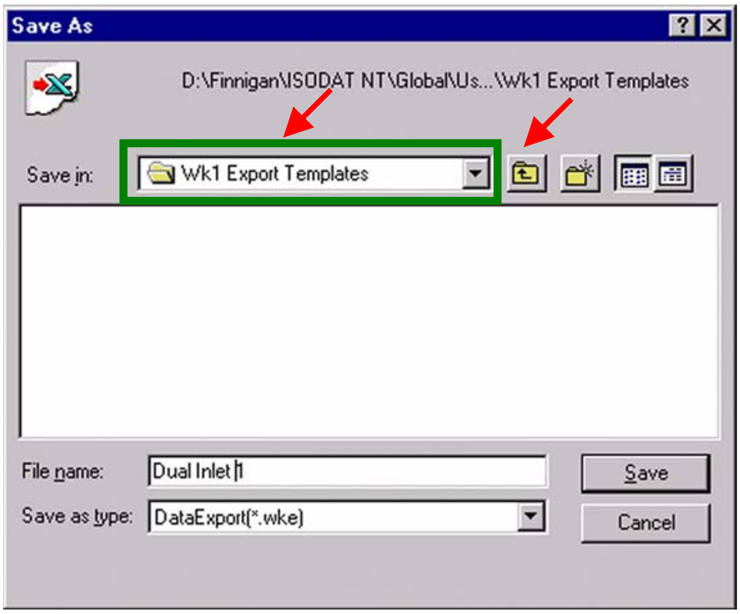
- To remove the Identifier(s) confirm by ***Yes***.

The selected Identifier(s) will be removed from the right pane together with the provided empty columns in the Excel sheet.

6.2.3 SAVING AN EXPORT TEMPLATE

After creating an export template (i.e. a wke file) it must be saved. Therefore, on the Dual Inlet window's toolbar press the ***Save*** or ***Save As*** button

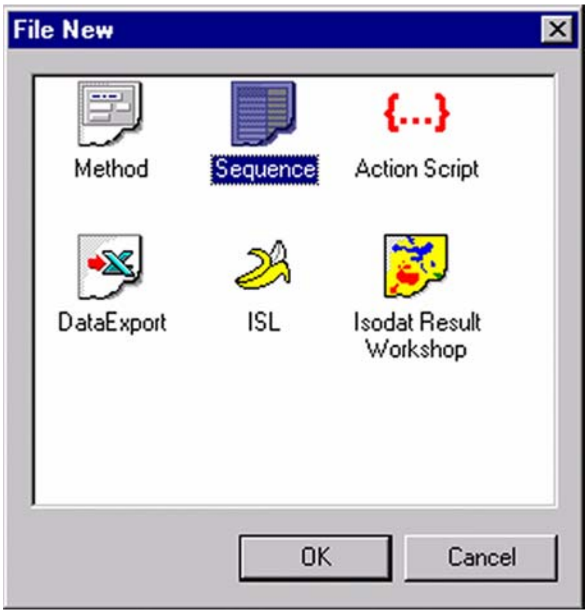
.



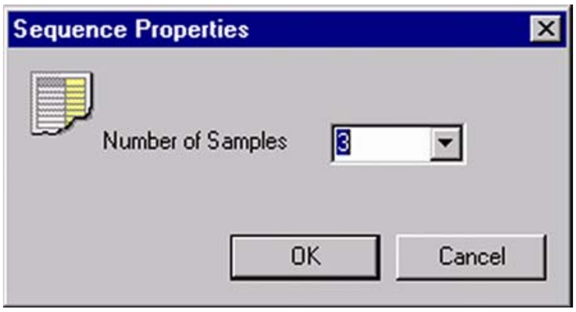
- ***Save the export template in the folder <u>Wk1 Export Templates!</u>***
- In the ***File Name*** field, type a significant name for the export template (e.g. Dual Inlet 1).
- In the ***Save as type*** field, accept the extension .wke and press ***Save***.

6.2.4 <u>APPLYING AN EXPORT TEMPLATE</u>

A Sequence is always exported as several samples can thus be bundled into a single export file. To apply an export template, open a new Sequence via the ***New*** button New .



- Then click the ***Sequence*** Icon.
- Confirm by ***OK***.

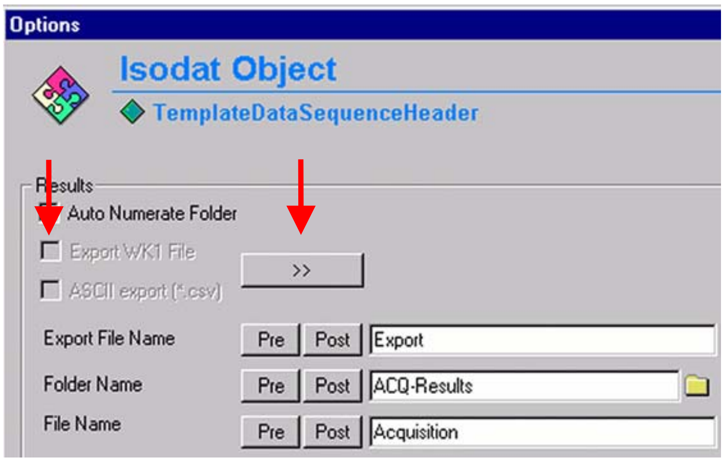


Number of Samples: 3

- ➢ Type your number of samples (e.g. 3).
- ➢ Confirm by ***OK***.

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

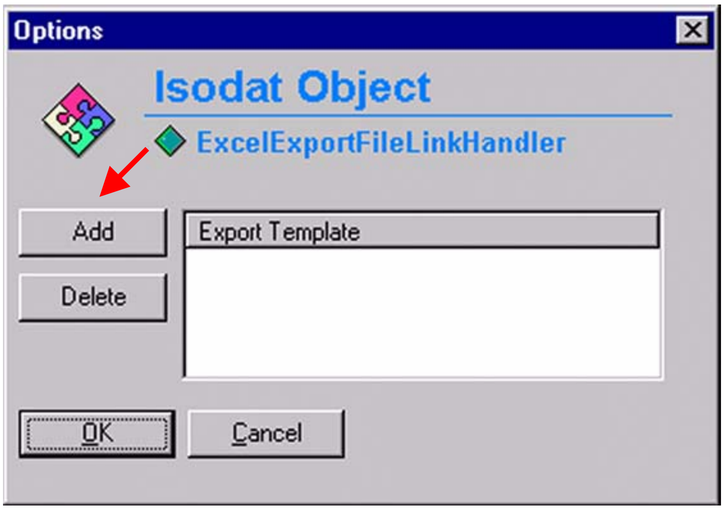
- ➢ The Sequence of your choice is displayed.
- ➢ Press the ***Start*** button Start .



Auto Numerate Folder
☐ Export WK1 File
☐ ASCII export (*.csv)

Export File Name: Export
Folder Name: ACQ-Results
File Name: Acquisition

- ➢ Since no export templates have been selected yet, the ***Export WK1. File*** box is inactive.
- ➢ Press the >> button.



- ➢ Since no export templates have been selected yet, the Export Template list is empty.
- ➢ Press the Add button.

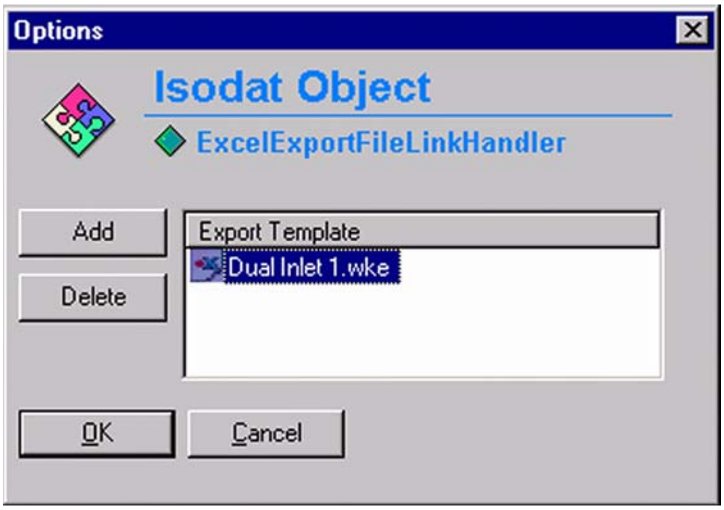
Look in: Wk1 Export Templates

Dual Inlet 1.wke

File name: Dual Inlet 1.wke

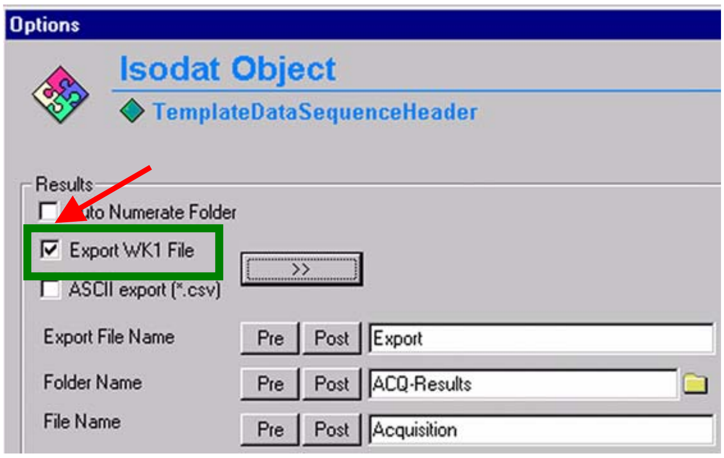
Files of type: Wk1 Export Template (*.wke)

- In the folder ***WK1 Export Templates***, select your Export Template (e.g. Dual Inlet 1.wke).
- Press ***Open***.



Export Template: Dual Inlet 1.wke

- Select your Export Template (e.g. Dual Inlet 1.wke).
- Confirm by ***OK***.



Results

☐ Auto Numerate Folder

☑ Export WK1 File

☐ ASCII export (*.csv)

Export File Name: Export

Folder Name: ACQ-Results

File Name: Acquisition

- Note that the ***Export WK1 File*** box has now become active.
- Confirm by ***OK***.

The options specified in the Sequence are now set and Data Acquisition starts.

NOTE: ***The Excel Export will be performed online, i.e. during the measurement.***

This is different from a Re-evaluation, where the Excel Export takes place offline, i.e. after a measurement.

ISODAT NT

OPERATING MANUAL

7

DIAGNOSIS

Issue 06/2002

Thermo Finnigan

7.1 CHECKING PERFORMANCE DATA

Thermo Finnigan MAT has developed several test routines to check the performance data of isotope ratio mass spectrometers.
For user's convenience, the program ***Diagnosis*** covering these test routines is included in the supplied version of ISODAT NT. It must be noted that operating some of the test routines requires technical knowledge of the instrument's internals. In addition, successful execution of some of the tests depends upon instrument preconditions.

When running the test routines, a highly sensitive focusing of your instrument will lead to the best specifications results.

The program ***Diagnosis*** contains the following test routines:

1 Absolute Sensitivity

2 Abundance

3 Amplifier Test

4 Compression Factor

5 Linearity

6 Peak Flatness

7 Relative Sensitivity

8 Resolution

9 Signal Stability

10 System Stability

NOTE: ***Reference gas for all performance data is CO_2.***
Make sure to have properly filled CO_2 reservoirs attached to the inlet system before starting the "Diagnosis" program.

7.2 HOW TO START DIAGNOSIS

- Start ISODAT NT by double-clicking the Icon on your desktop.

Isodat NT

- Double-click the ***Diagnosis*** Icon.

Diagnosis

- Mark the test routine to be performed (e.g. “Abundance”).
- Press ***OK***.

7.2.1 ABSOLUTE SENSITIVITY

NOTE: ***Testing Absolute Sensitivity requires a Dual Inlet system***

Absolute Sensitivity is defined as the number of molecules needed to generate an ion, which is then registered at the collector (e.g. one ion of mass 44 at the corresponding collector cup). It is thus dimensionless and measured in molecules per ion.

Based on a defined volume, the ion current is determined during a defined time period as a function of sample consumption (i.e. sample loss). The small, defined volume is located between valve 25 of the inlet system and the inlet capillary. It amounts to approximately 250 µl.
Integrating the ion current over time yields the number of ions. From the signal drop during measurement the number of molecules necessary to generate this ion amount is calculated. To obtain Absolute Sensitivity, the number of molecules is divided by the number of ions.

Positively charged ions are produced in the ion source by electron bombardment. This electron impact (EI) ionization is described by:

$$AB + e^- \rightarrow AB^+ + 2e^- \qquad \text{(ionization)}$$

$$AB + e^- \rightarrow A^+ + B + 2e^- \qquad \text{(ionization and dissociation)}$$

Definition of Absolute Sensitivity AS:

$$AS = \frac{\Delta s}{n_{ion}} \ (molecules \,/\, ion)$$

where Δs: sample gas consumption
n_{ion}: number of detected ions

The number of consumed sample molecules n is calculated via the ideal gas law:

$$p V = n R T$$

where:
R: universal gas constant
T: temperature
p: pressure
n: number of sample molecules
V: volume (here 250 µl)

The amount of detected ions n_{ion} in the collector cup can be calculated via the electrons needed to neutralize the positive ions:

$$Q = \int_{t_1}^{t_2} I \, dt$$

where:
Q: charge
I: intensity

To test this parameter

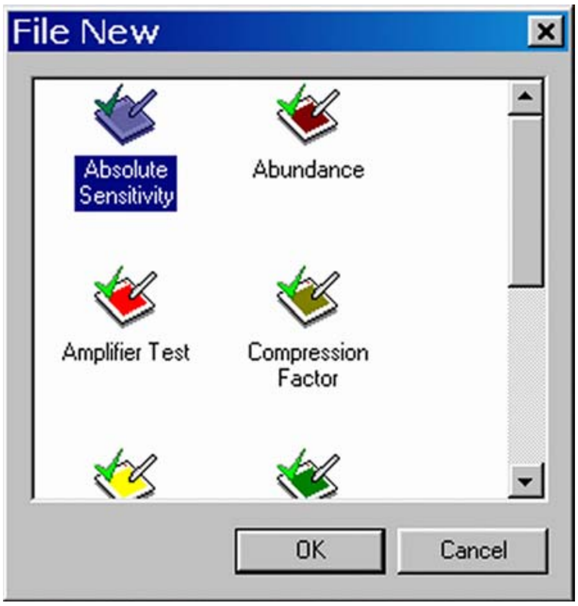
1 Measurement starts with a determination of pressure and intensity.

2 The volume is reduced to the defined volume by closing valve 25 of the inlet system. The system is in a waiting position until the start pressure and intensity are reached.

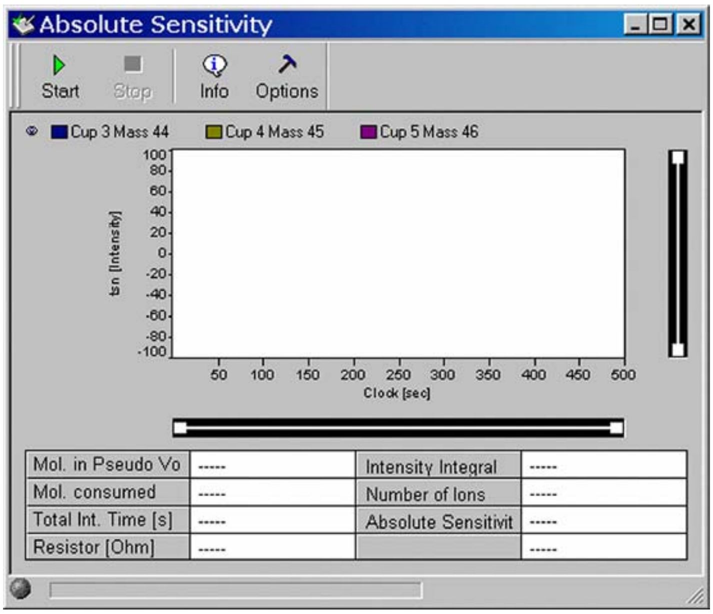
3 The ion current is measured for the preset time.

NOTE: ***Absolute Sensitivity should be about 1500 molecules CO_2 per mass 44 at the collector cup.***

Testing Absolute Sensitivity



- Click on the ***Absolute Sensitivity Icon*** and press ***OK*** (or double-click on the Icon).

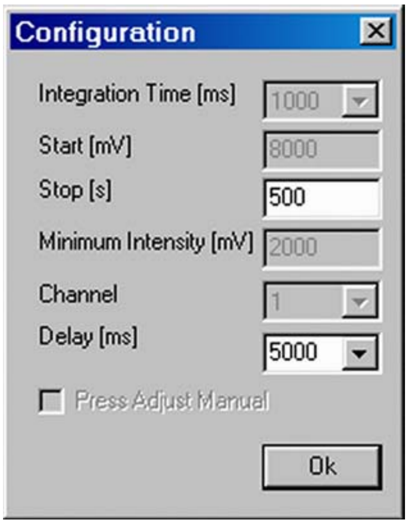


Mol. in Pseudo Vo	-----	Intensity Integral	-----
Mol. consumed	-----	Number of Ions	-----
Total Int. Time [s]	-----	Absolute Sensitivit	-----
Resistor [Ohm]	-----		-----

- On the "Window" toolbar, the ***Absolute Sensitivity Icon*** becomes visible.

- Press the ***Options*** button.

- Accept the defaults or from the pulldown menus, enter a value for ***Stop*** [s] and choose a value for ***Delay*** between 100 ms and 20000 ms from the list.



Integration Time [ms] 1000
Start [mV] 8000
Stop [s] 500
Minimum Intensity [mV] 2000
Channel 1
Delay [ms] 5000
☐ Press Adjust Manual

- In ***Advanced Mode***, all values can be edited:

  Integration time in ms

  Start Intensity value ("Start [mV]")

  Minimum Intensity [mV]

  Channel: a recording channel consists of an amplifier, a voltage-frequency (VF) converter and a counter.

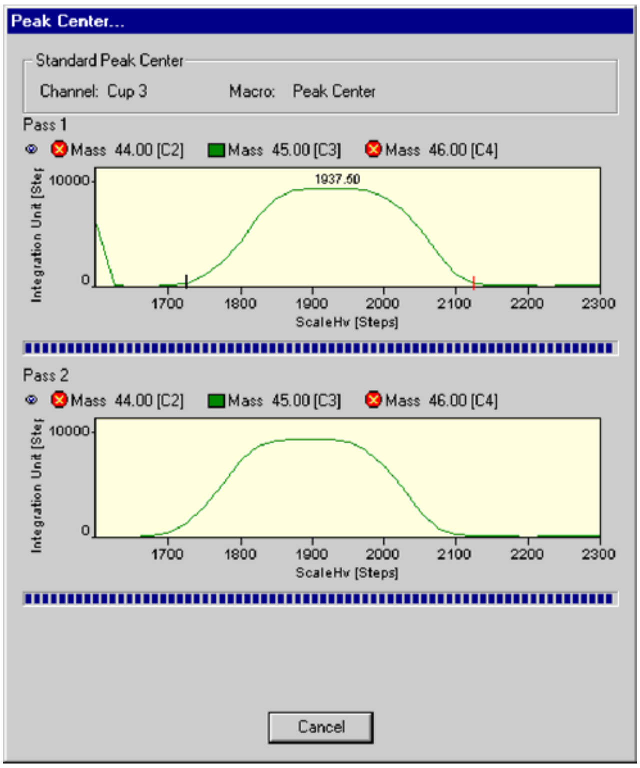
  Press Adjust Manual: activate it, if you wish to adjust pressure and peak center manually (if it is activated, you are requested to adjust to e.g. 8000 mV manually).

- Finally, click ***OK***.

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

- Testing starts with a Peak Center on the selected Cup, e.g. mass 45, Cup3.

The bellow is reduced stepwise until the start level intensity (e.g. 8000 mV) is reached. Then, intensity and pressure are measured for the preset time (e.g. 500 s).

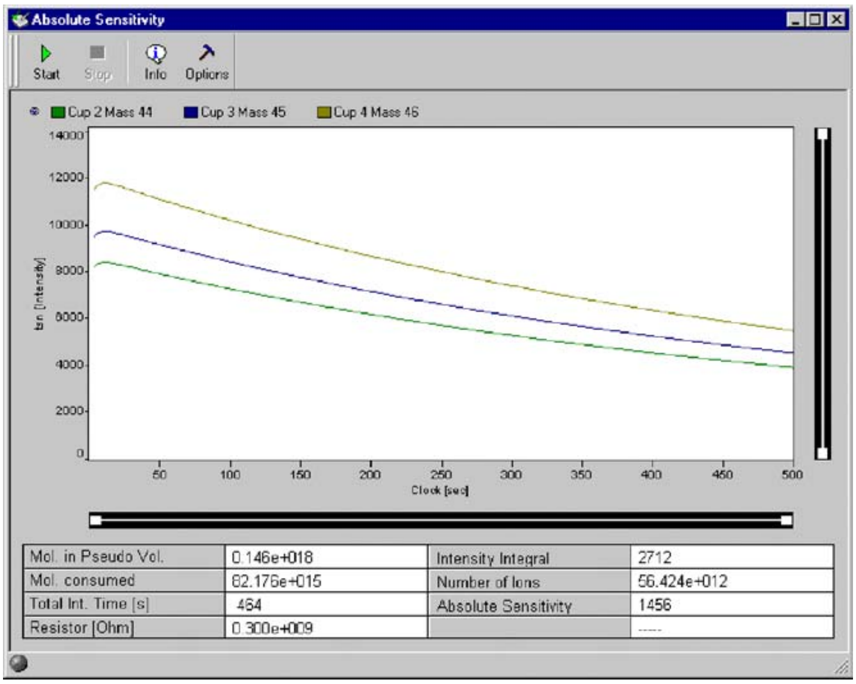


Peak Center...

Standard Peak Center

Channel: Cup 3 Macro: Peak Center

- Finally, Absolute Sensitivity and other parameters are calculated.

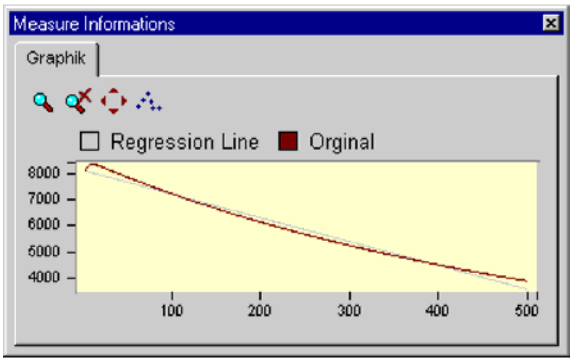


Absolute Sensitivity

Mol. in Pseudo Vol.	0.146e+018	Intensity Integral	2712
Mol. consumed	82.176e+015	Number of Ions	56.424e+012
Total Int. Time [s]	464	Absolute Sensitivity	1456
Resistor [Ohm]	0.300e+009		-----

- Press the ***Info*** button.

- Original measurement and regression line are shown.



- When closing the "Absolute Sensitivity" window you are asked, whether to save the changes.


Diagnosis

Save changes to Absolute Sensitivity?

7.2.2 ABUNDANCE

Abundance denotes the contribution of a mass to a neighbor mass (e.g. the amount of ions for mass 44 falling into cup 45). Thus, the intensity of a mass (e.g. mass 44) is compared to the intensity of the neighboring peak (e.g. mass 45).

In case of CO_2, divide the amount of ion current of mass 44 falling into the mass 45 cup by the ion current of mass 44 into the mass 44 cup.

Measured as ratio of two ion currents it is dimensionless and quoted in % or ppm. The Abundance test is performed with CO_2, and the device must be calibrated. Peak center is performed on Channel 2 (i.e. narrow cup) before the measurement starts.

To test this parameter

1. Signal height (i.e. intensity) on mass 44 is determined in the appropriate collector cup (e.g. Cup 3).

2. The background signal on mass 45 is measured (i.e. electronic noise with no gas) from mass 44 to mass 45.5.

3. With CO2 as sample gas the magnetic field is scanned from mass 44 to mass 45.5, and the intensities are measured on the neighboring Cup of mass 45 (e.g. Cup 4, with bigger resistor value in order to keep the signal in the detection range).

4. Extrapolate the abundance (of mass 44 onto mass 45) from the signal to the left and to the right of mass 45 peak.

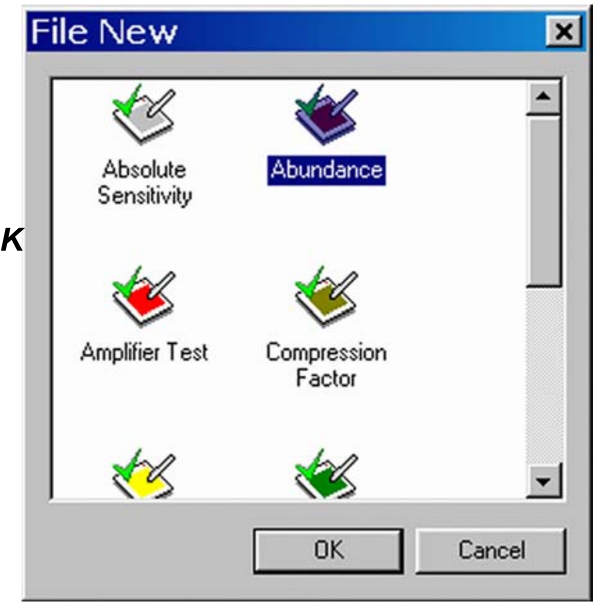
5. Calculate the abundance as described above.

Sources of error

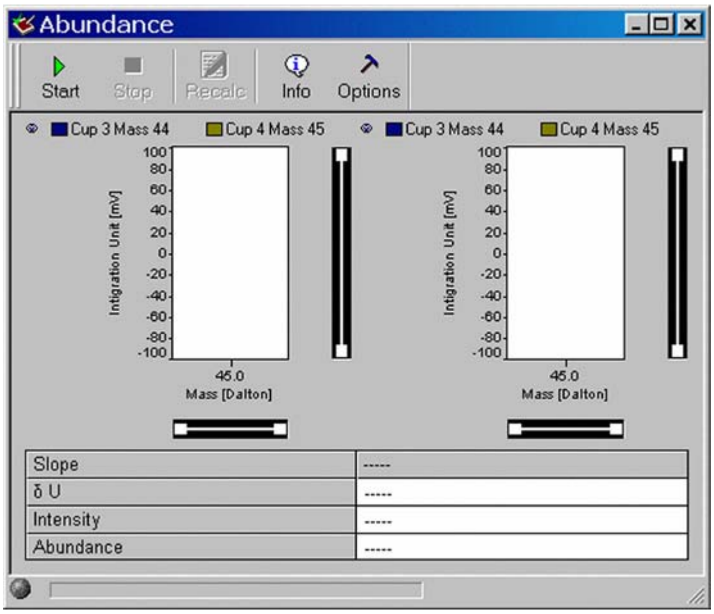
- Resistor values are not configured correctly.
- Due to electrons on the left and on the right side of the peak, a negative signal may result. This problem can be overcome by manual adjustment.

Testing Abundance

- Click on the ***Abundance Icon*** and press ***OK*** (or double-click the Icon).



- The left window detects mass 44 ions in the correct cup. The right window detects mass 44 ions in the neighbor cup (i.e. cup of mass 45).



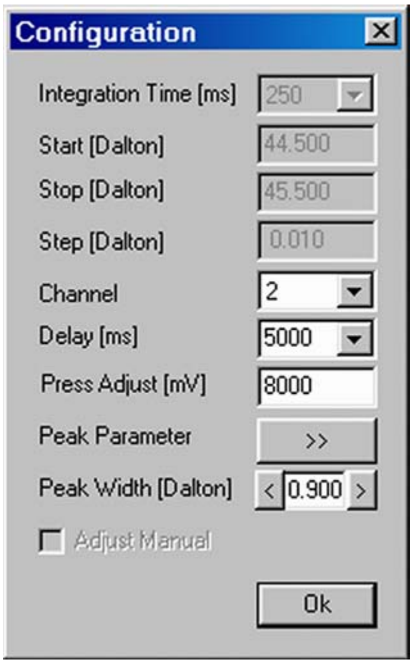
Slope	-----
δ U	-----
Intensity	-----
Abundance	-----

- ➢ On the "Window" toolbar, the ***Abundance Icon*** becomes visible.

- ➢ Press the ***Options*** button.

- ❑ Accept the defaults, or type appropriate values for "Press Adjust [mV]" and "Peak Width [Dalton]".
  The selected cup for the Abundance test is connected with the indicated recording channel (e.g. choose channel 2 from the pulldown list). Choose a "Delay [ms]" value between 100 ms and 20000 ms. The measurement start will be delayed by it.

  In ***Standard Mode***, the inactive, i.e. gray, variables cannot be edited. The ***Advanced Mode*** allows experienced users to set them all:

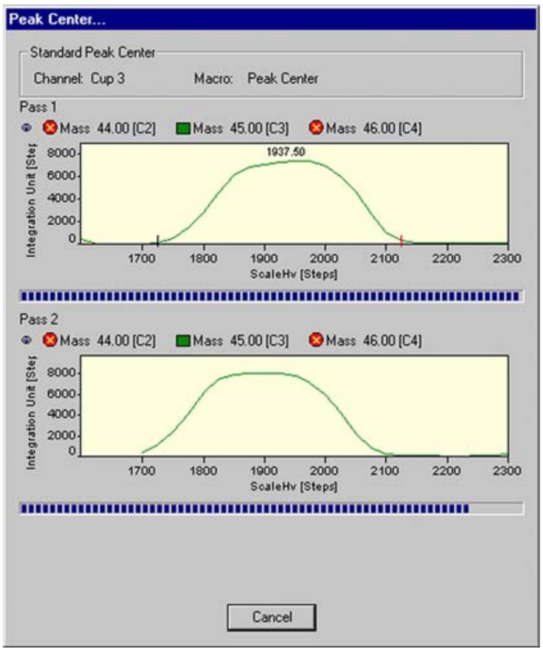


Integration Time [ms] 250
Start [Dalton] 44.500
Stop [Dalton] 45.500
Step [Dalton] 0.010
Channel 2
Delay [ms] 5000
Press Adjust [mV] 8000
Peak Width [Dalton] 0.900
☐ Adjust Manual

  - ❑ the start mass for the magnet field scan ("Start [Dalton]"),
  - ❑ the end mass, where the magnet stops scanning ("Stop [Dalton]")
  - ❑ the step width for the magnet scan ("Step [Dalton]")
  - ❑ the "Adjust Manual" check box - if it is activated, signal intensity and peak center must be performed manually.

- ➢ Finally, click ***OK***.
- ➢ Start the Abundance test by the ***Start*** button.

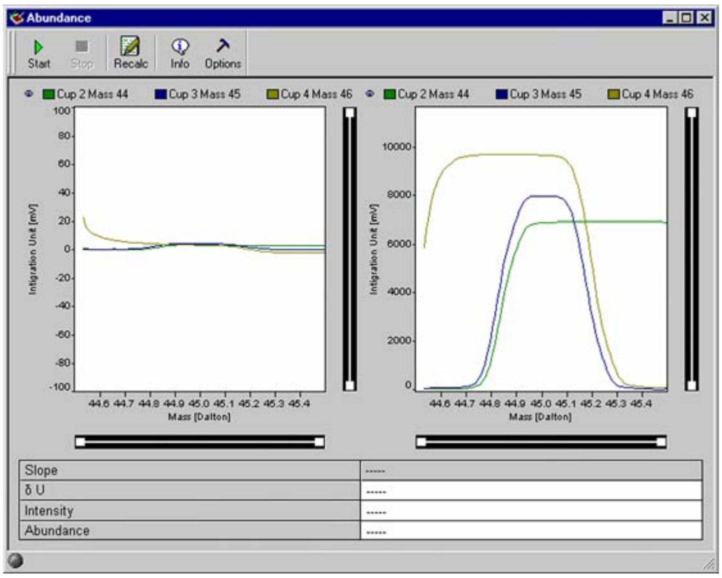
- Testing starts with a Peak Center on the selected cup (e.g. mass 45, cup 3).


Peak Center...

Standard Peak Center

Channel: Cup 3 Macro: Peak Center

- It might be helpful to activate the ***Advanced Mode***, press the ***Options*** button and enhance the interval between "Start" and "Stop" values (i.e. decrease "Start" and increase "Stop" values).



Slope	-----
δ U	-----
Intensity	-----
Abundance	-----

- Values for Abundance, Intensity, δU and slope are displayed.

- When closing the "Abundance" window you are asked whether to save the changes.


Diagnosis

Save changes to Abundance?

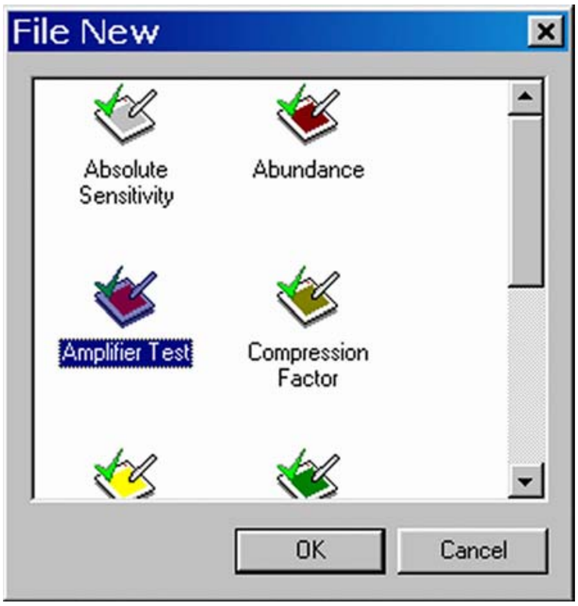
7.2.3 AMPLIFIER TEST

The Amplifier Test (formerly called UFC-Test) checks the ion detection performance of the IRMS with no ions present. It thus informs about the background noise of electronic devices.

The amplifier baseline must be determined without an interfering signal. Thus, the ion source is switched off before measurement starts (i.e. ion current equals zero). The signal intensity of every cup is individually measured at least 200 times for a defined integration time. Finally, mean and standard deviation are calculated.

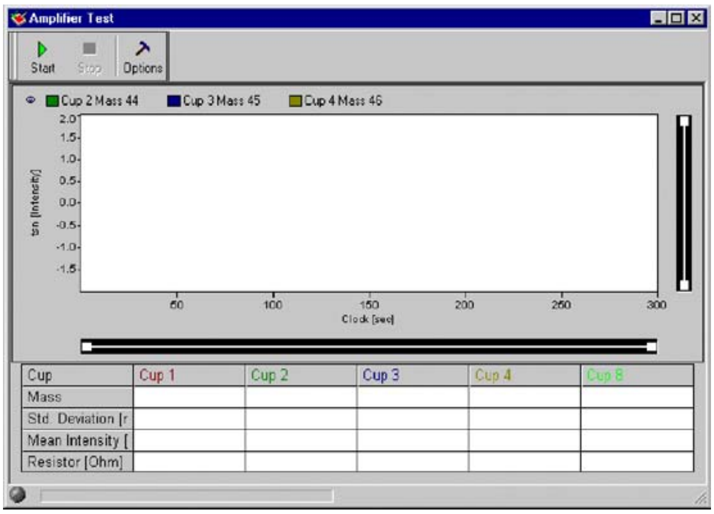
Testing Amplifiers

- Click the ***Amplifier Test Icon*** and press ***OK*** (or double-click the Icon).



- Wait, while the available Gas Configurations are scanned.


Scan available Gas Configurations...

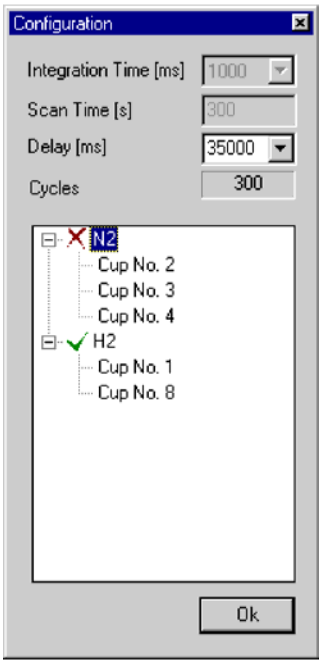


Cup	Cup 1	Cup 2	Cup 3	Cup 4	Cup 8
Mass					
Std. Deviation [r					
Mean Intensity [					
Resistor [Ohm]					

- On the "Window" toolbar, the ***Amplifier Icon*** becomes visible.

- Press the ***Options*** button.

- With the exception of ***Delay***, all parameters are preset and not changeable in Standard Mode.
    - The Advanced Mode allows experienced users to modify all parameters.
    - Accept the default value of ***Integration Time*** (in ms).
    - ***Scan Time*** (in s) denotes the duration of the scan.
    - The measurement start is delayed by the displayed default value of ***Delay*** time (in ms). Accept it, or from the pulldown menu, select a suitable value between 100 ms and 35000 ms.



Integration Time [ms] 1000
Scan Time [s] 300
Delay [ms] 35000
Cycles 300

- N2
    - Cup No. 2
    - Cup No. 3
    - Cup No. 4
- H2
    - Cup No. 1
    - Cup No. 8

NOTE: ***In one of the next versions, the process will be simplified: all amplifiers can then be measured using one single Scan.***

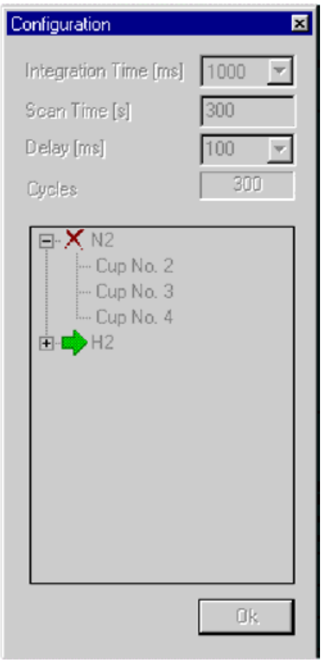
- ❑ ***Cycles*** denotes the number of measurements. The intensities of the channel are measured for indicated cycles with a definite cycle time.

- ➢ Finally, press ***OK***.
- ➢ Press the ***Start*** button.

- ➢ ***Switch*** off High Voltage (on the ***Focus*** bar).

  Then press ***OK***.

High Voltage should be off !



Integration Time [ms] 1000

Scan Time [s] 300

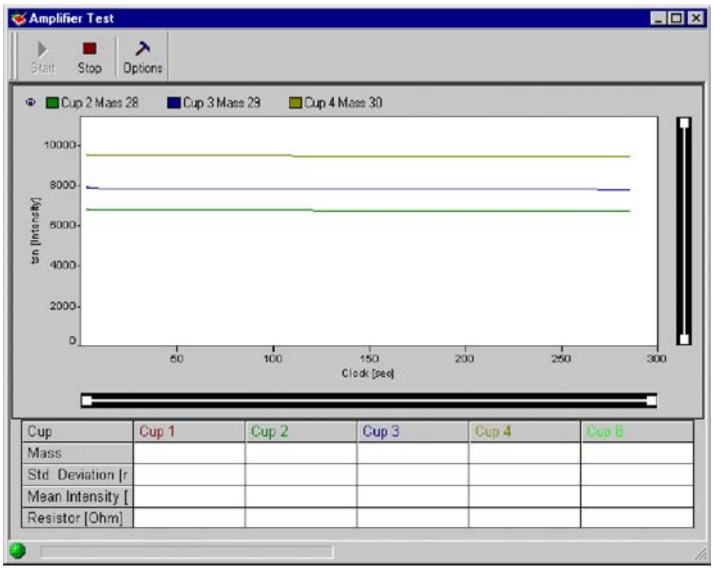
Delay [ms] 100

Cycles 300

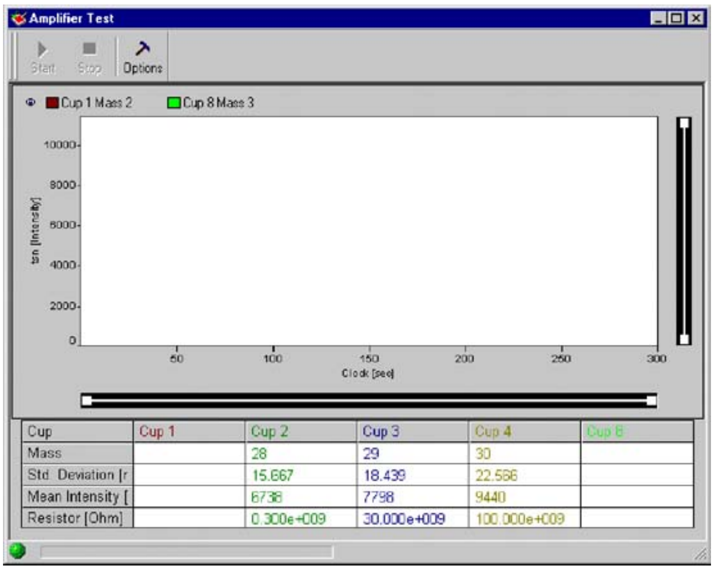
- ➢ The amplifiers of the cups activated in step 5 are being tested, e.g. cup 1, mass 2, brown and cup 8, mass 3, green.

- ➢ If you press the ***Options*** button during measurement, a green arrow informs you about what is currently done.

- The correspondent intensity vs. time diagram is shown.



Cup	Cup 1	Cup 2	Cup 3	Cup 4	Cup 8
Mass					
Std. Deviation [r					
Mean Intensity [					
Resistor [Ohm]					



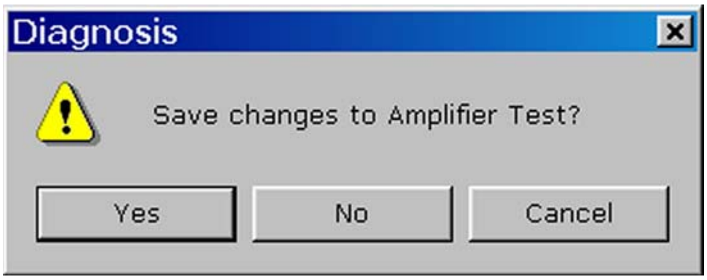
Cup	Cup 1	Cup 2	Cup 3	Cup 4	Cup 8
Mass		28	29	30	
Std. Deviation [r		15.667	18.439	22.566	
Mean Intensity [		6738	7798	9440	
Resistor [Ohm]		0.300e+009	30.000e+009	100.000e+009	

- The baselines of the cups are shown with no ions present.

Standard deviation [mV], mean [mV] and resistance [Ohm] of each selected cup are displayed.

- The procedure is repeated for the other masses.

- When closing the "Amplifier Test" window you are asked, whether to save the changes.



Save changes to Amplifier Test?

7.2.4 COMPRESSION FACTOR

NOTE: ***Testing Compression Factor requires a Dual Inlet system.***

The Compression Factor, formerly called ***Pressure Ratio***, is defined as intensity ratio and is thus dimensionless [mV/mV]:

$$Comp = \frac{Int_{end}}{Int_{start}}$$

where:

Int_{end}: Intensity at the end of measurement

Int_{start}: Intensity at measurement start

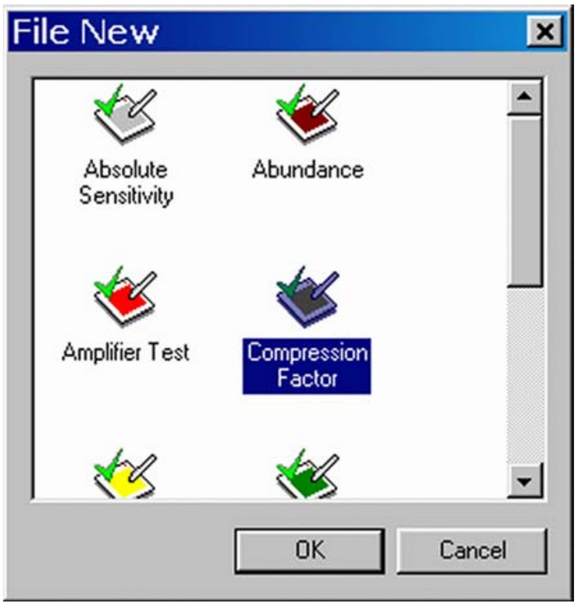
The Compression Factor determines the dynamic range of the two bellows informing about their tightness and linearity. The ion signal (i.e. intensity) is measured at different bellow compressions: an intensity vs. volume diagram results. The standard deviation around the signal's mean is calculated.

NOTE: ***The bellows must be calibrated before performing the test.***

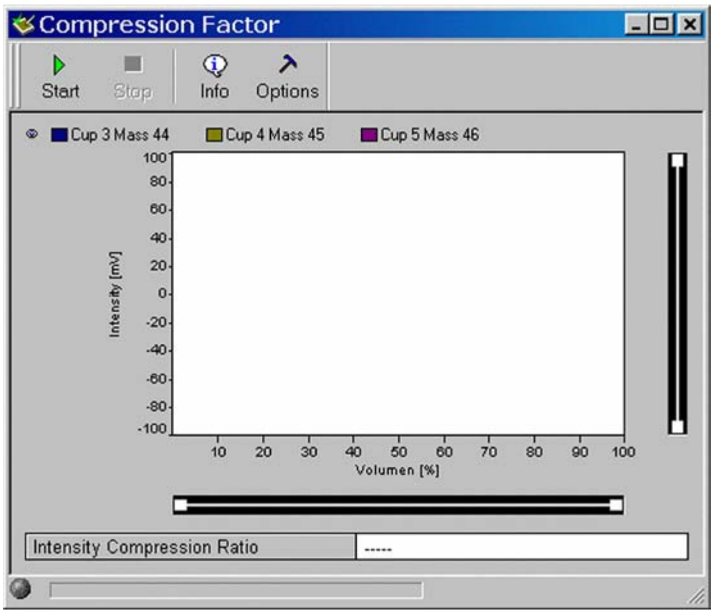
To test this parameter, the peak intensity for a mass (e.g. 44) is measured starting at the maximum (i.e. 100 %) down to the minimum (i.e. 0 %). A minimum death volume of about 3 ml is still remaining at 0 % volume. A certain level (e.g. 200 mV) serves as starting point of the measurement. The signal for the bellow expanded to maximum should be at this level. If it is not the case, the inlet system is expanded and pumped automatically until the reference level is reached.

NOTE: ***The Compression Factors should be about the same for both bellows: at least 1:10 or higher.***

Testing the Compression Factor



- Click on the ***Compression Factor Icon*** and press ***OK*** (or double-click the Icon).



Intensity Compression Ratio -----

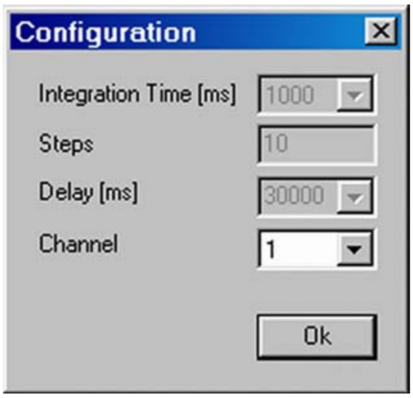
- On the "Window" toolbar, the ***Compression Factor Icon*** becomes visible.

- Press the ***Options*** button.

- In ***Standard Mode***, you are only able to select a channel from the pulldown menu.

  In ***Advanced Mode***, also the other variables can be edited:

  - Integration time [ms],
  - Steps: the volume of the bellows is varied stepwise, e.g. in 10 steps,
  - Delay [ms]: the measurement start is delayed by the displayed value.



Configuration

Integration Time [ms] 1000

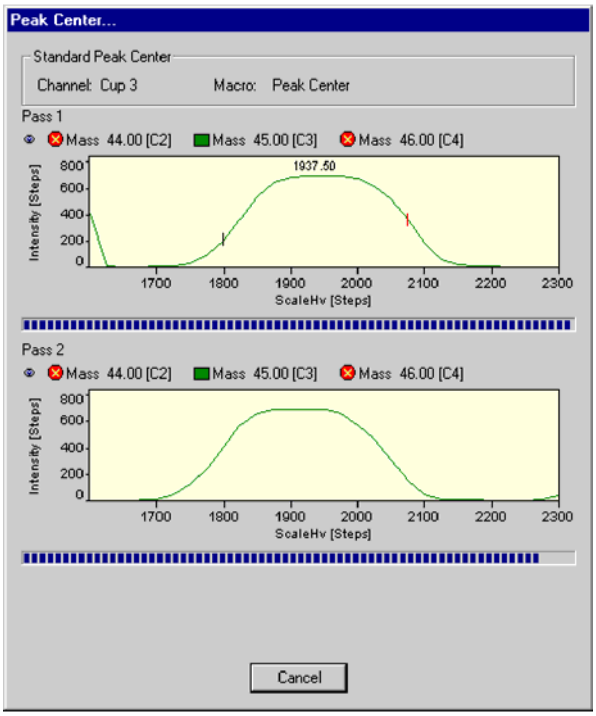
Steps 10

Delay [ms] 30000

Channel 1

- Finally, press ***OK***.
- Press the ***Start*** button.

- Testing starts with a Peak Center for the first bellow (e.g. Sample).



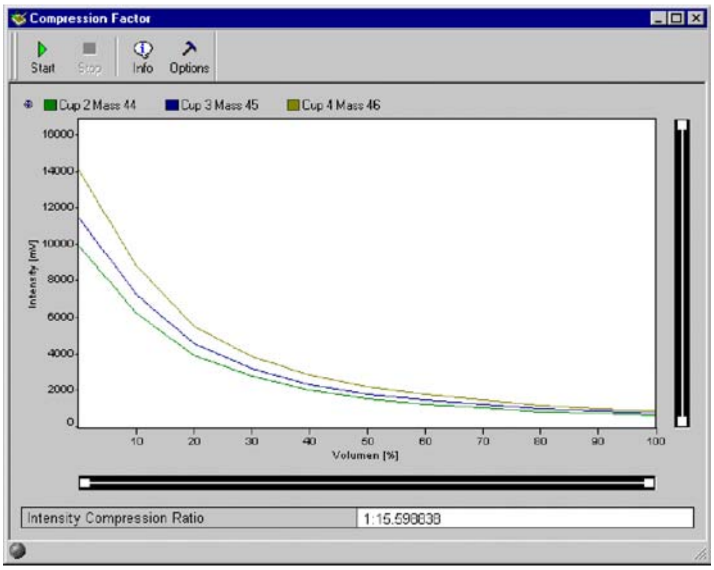
Peak Center...

Standard Peak Center

Channel: Cup 3 Macro: Peak Center

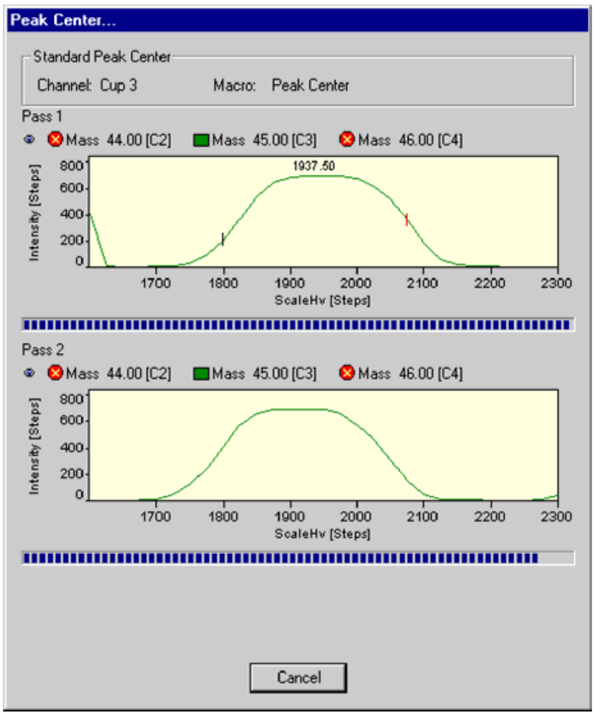
- ➢ When the Peak Center is finished, signal intensity is shown during the volume change of the bellows (for the different masses).

  Finally, the Intensity-Compression ratio is calculated for the first bellow, e.g. Sample.



Intensity Compression Ratio 1:15.598838

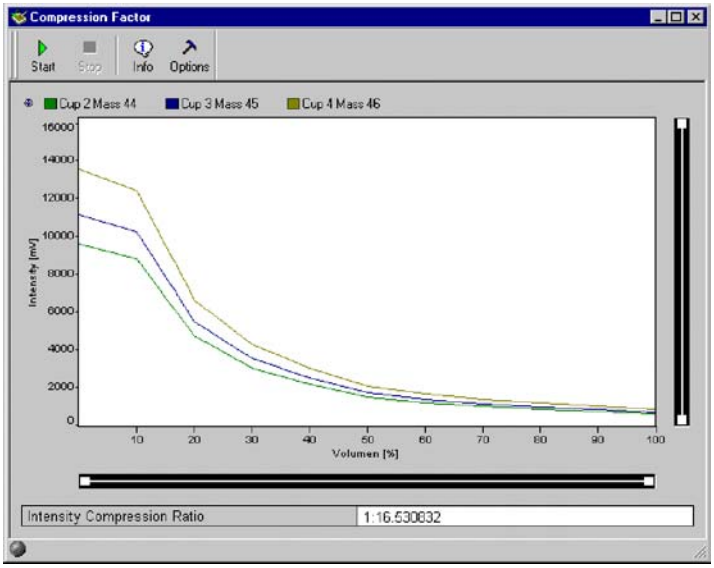
- ➢ The procedure is repeated for the second bellow (e.g. Standard): a Peak Center is performed.



Channel: Cup 3 Macro: Peak Center

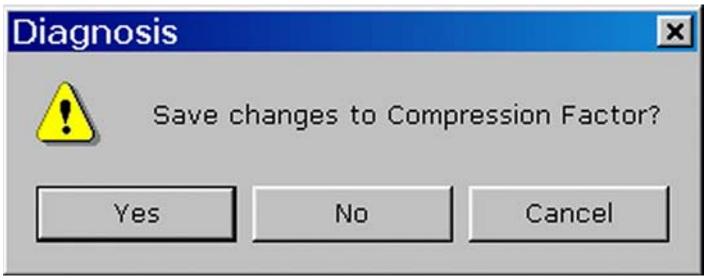
- The procedure is repeated for the second bellow (e.g. Standard).

  When the Peak Center is finished, an Intensity vs. Volume diagram for the different masses is shown. Finally, the Intensity Compression ratio is calculated for the second bellow (e.g. Standard).



Intensity Compression Ratio 1:16.530832

- When closing the "Compression Factor" window you are asked, whether to save the changes.



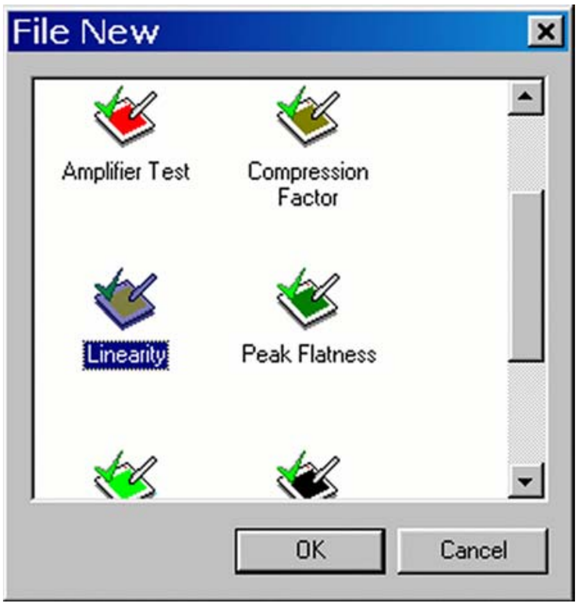
Save changes to Compression Factor?

7.2.5 LINEARITY

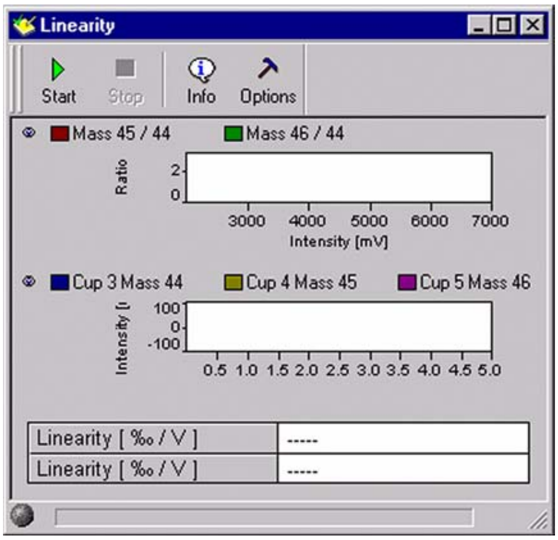
At Linearity test, synonymously called ***Ratio Linearity***, over a range of varying signals, signal linearity is checked vs. beam intensity (i.e. intensity of the main ion current). The signal intensity is measured, and the isotope ratios are displayed vs. beam intensity. Linearity is calculated as slope of the regression line [‰/V]. The ratios are monitored between 2 V and 8 V in 1 V steps. For each data point, the background is subtracted.

Source of error: Resistor values must be configured correctly.

Testing Linearity



- Click the ***Linearity*** Icon and press ***OK*** (or double-click the Icon).

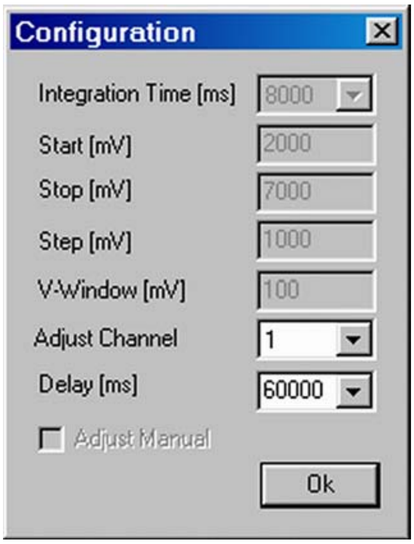


Linearity [‰ / V]	-----
Linearity [‰ / V]	-----

- On the "Window" toolbar, the ***Linearity Icon*** becomes visible.

- Press the ***Options*** button.

- From the pulldown menus select:



Integration Time [ms] 8000
Start [mV] 2000
Stop [mV] 7000
Step [mV] 1000
V-Window [mV] 100
Adjust Channel 1
Delay [ms] 60000
☐ Adjust Manual

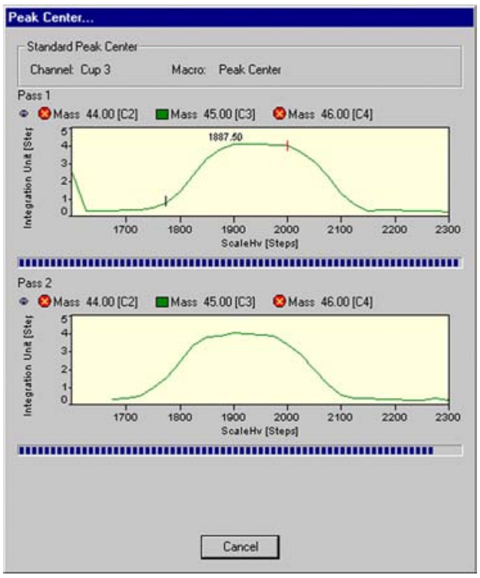
  - a channel and a value between 100 ms and 20000 ms for "Delay [ms]".

    The other variables are preset in Standard Mode (to be accepted), but editable in Advanced Mode:

  - Integration time [ms], Start [mV], Stop [mV] Step width [mV], V-Window [mV]
  - Adjust Manual: activate it, if the intensities (i.e. voltage steps) between 2 V and 8 V are to be set manually.

- Finally, press ***OK***.

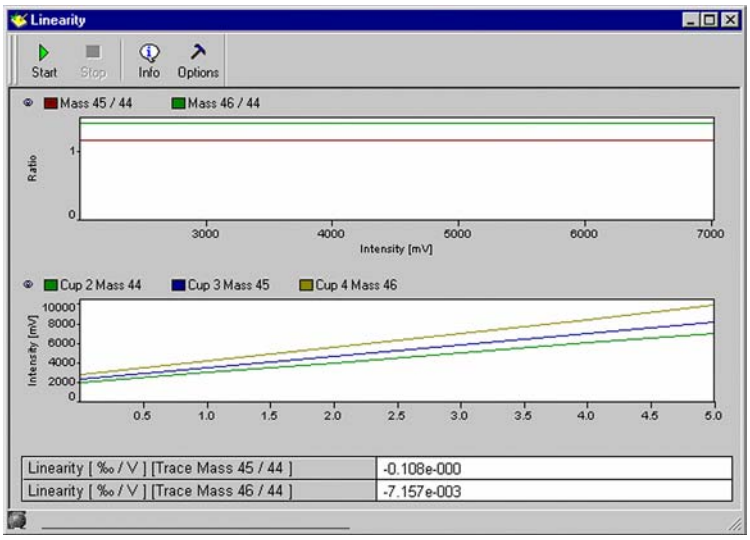
- Press the ***Start*** button.



Standard Peak Center
Channel: Cup 3
Macro: Peak Center

- ➢ Testing starts with a Peak Center on cup 3 as the narrow cup.

  If no peak could be found, the measurement will be terminated.



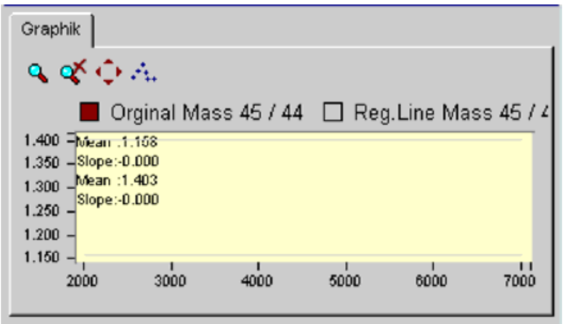
Linearity [‰ / V] [Trace Mass 45 / 44]	-0.108e-000
Linearity [‰ / V] [Trace Mass 46 / 44]	-7.157e-003

- ➢ Two linearity values [‰/V] are calculated: one for mass 45/44 and one for mass 46/44.

NOTE: ***Linearity must be less than 0.06 ‰/V!***

- ➢ Press the ***Info*** button.

- ➢ Mean and slope values are displayed.



Mean :1.158
Slope:-0.000
Mean :1.403
Slope:-0.000

7.2.6 PEAK FLATNESS

As slope of the peak plateau, Peak Flatness reflects the quality of the ion stream. A correction is necessary to eliminate effects of descending peak plateau with increasing high voltage. This is done by measuring the peak twice - first with increasing and then with decreasing high voltage. The resulting peak represents the mean values of both runs. The measured intensity is a function of the acceleration voltage (i.e. ion energy). Therefore, during a high voltage scan the intensity is slightly affected by this effect. To overcome this an "energy correction" is performed. The ion intensity on top of the peak (i.e. at a parameterized mass range around the center) is measured. Peak Flat-ness can be determined for different gases and different collector cups (e.g. for a CO_2-peak at mass 45 at Cup 2).

Two results are obtained:

- Maximal intensity deviation divided by the intensity
- Slope of the regression line [1/Da]

Differences to ISODAT Old

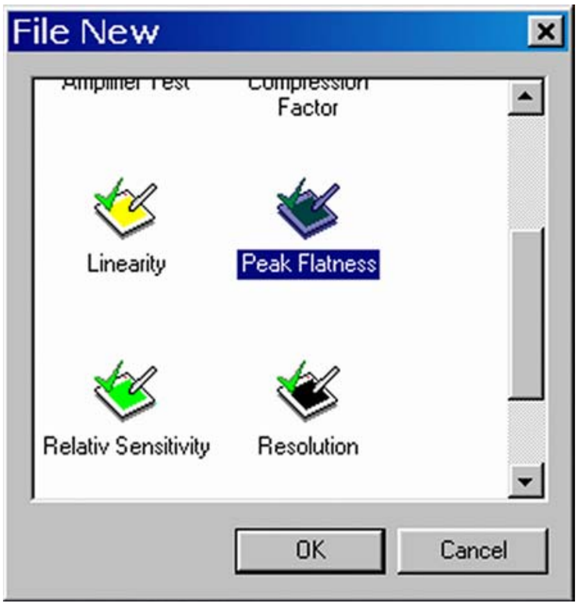
- ***Slope of the regression line as additional result***

  Dividing System Stability by this slope yields a measure for Signal Stability with respect to high voltage or magnet current instabilities.
  It is complementary to the Diagnosis parameter Signal Stability which is also sensitive to emission fluctuations.

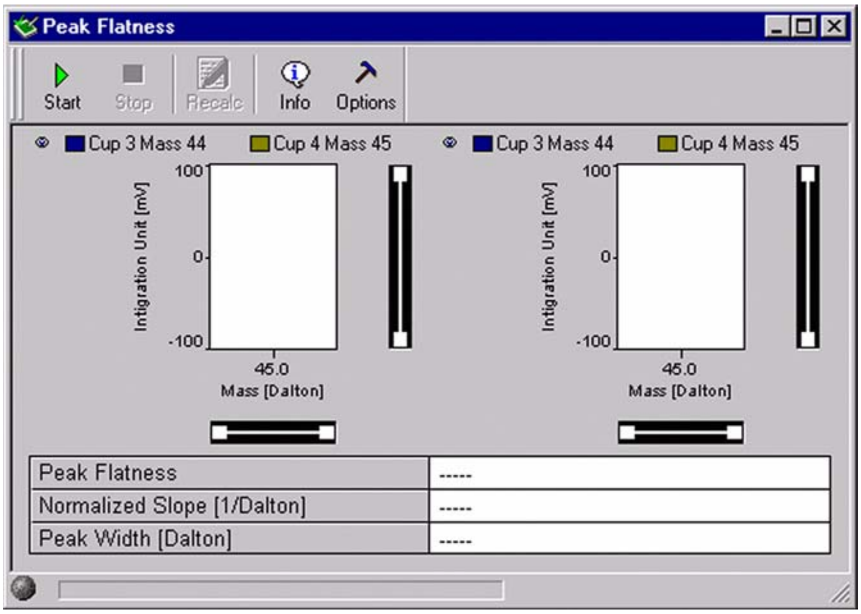
- ***Parameterized mass range***

  In ISODAT Old, a certain proportion of the peak top (30%) is used instead of a parameterized mass range. However, to know how much the measurement is affected by a certain system drift, remember that this is a proportion of mass scale rather than of cup width. The default mass range is set in such a way that the final result is comparable to the one of ISODAT Old using a typical cup width.

Testing Peak Flatness



- Click on the ***Peak Flatness Icon*** and press ***OK*** (or double-click on the Icon).



Peak Flatness	-----
Normalized Slope [1/Dalton]	-----
Peak Width [Dalton]	-----

- On the "Window" toolbar, the ***Peak Flatness Icon*** becomes visible.

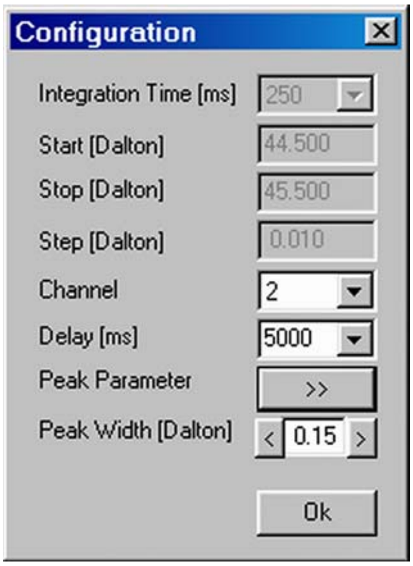
- Press the ***Options*** button.

- Accept the defaults or from the pulldown menus, select a Channel and a Delay value between 100 ms and 20000 ms.

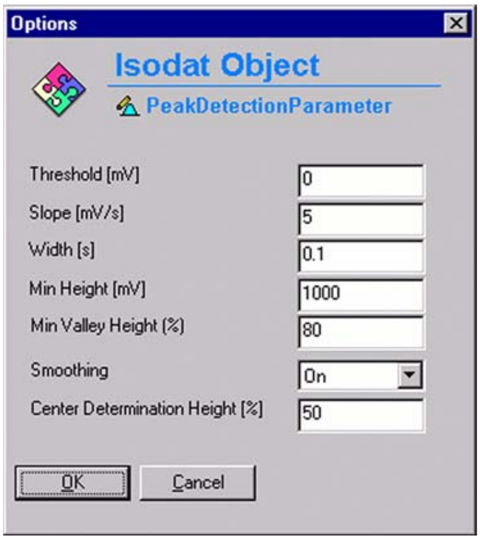
- Accept the default, or choose an appropriate Peak Width value by the < or > buttons.

- If you simply want to accept the Peak Detection parameters without a glance, do **not** press the ***Peak Parameter*** button. Instead, click ***OK***.

- However, as a sophisticated user, you may want to look at the Peak Detection parameters. To do so, press the ***Peak Parameter*** button.


Configuration

Integration Time [ms]: 250
Start [Dalton]: 44.500
Stop [Dalton]: 45.500
Step [Dalton]: 0.010
Channel: 2
Delay [ms]: 5000
Peak Parameter: >>
Peak Width [Dalton]: 0.15


Options

Isodat Object
PeakDetectionParameter

Threshold [mV]: 0
Slope [mV/s]: 5
Width [s]: 0.1
Min Height [mV]: 1000
Min Valley Height [%]: 80
Smoothing: On
Center Determination Height [%]: 50

- Change the values according to your needs.

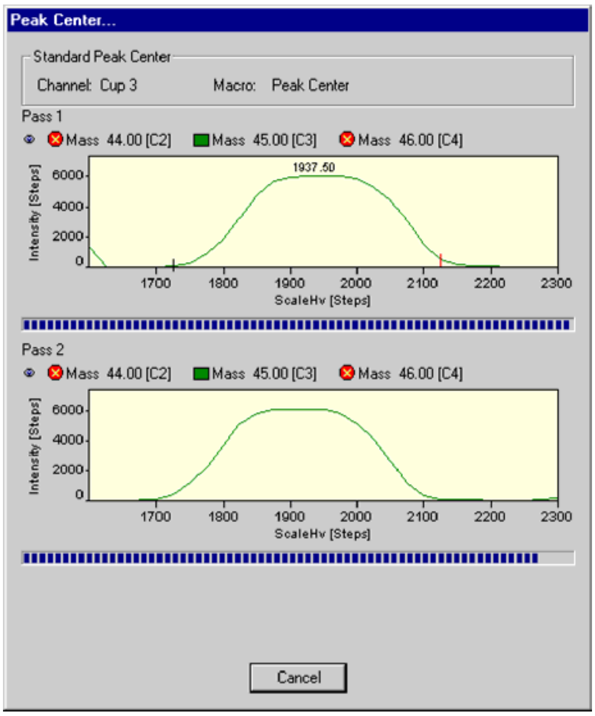
- Then click ***OK***.

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

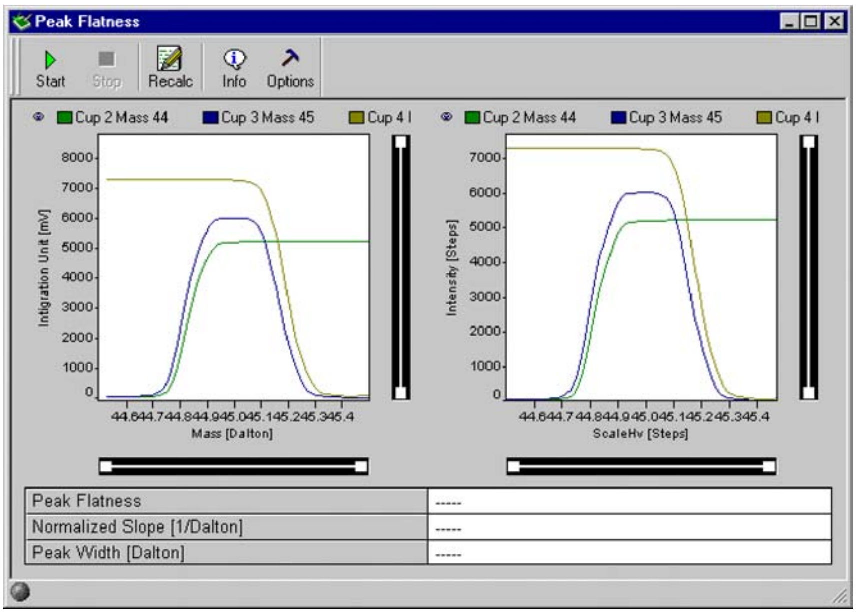
- Testing starts with a Peak Center on the selected cup (e.g. mass 45, cup 3).

  If no peak can be found, the measurement will be stopped.

In this case, press ***Cancel*** to proceed.



Channel: Cup 3
Macro: Peak Center

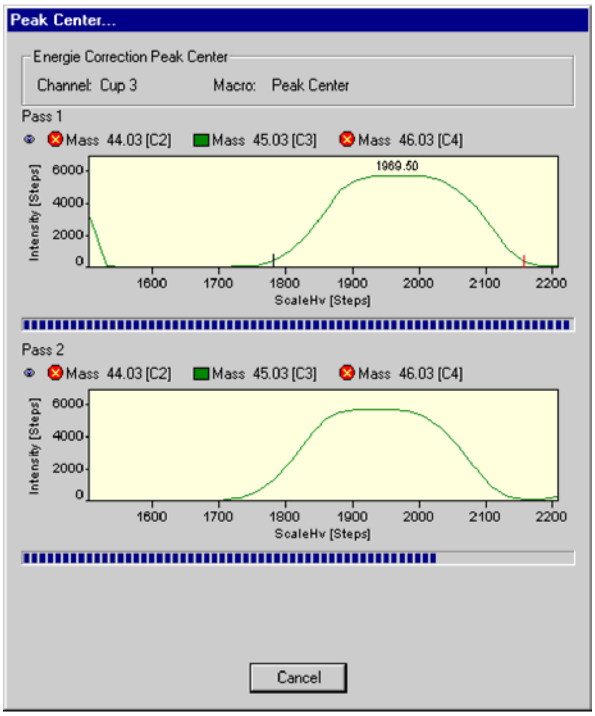


Peak Flatness	-----
Normalized Slope [1/Dalton]	-----
Peak Width [Dalton]	-----

- A Peak Center for Energy Correction is performed.

  If no Peak Center can be found, the measurement will be stopped.

  In this case, press ***Cancel*** to proceed.



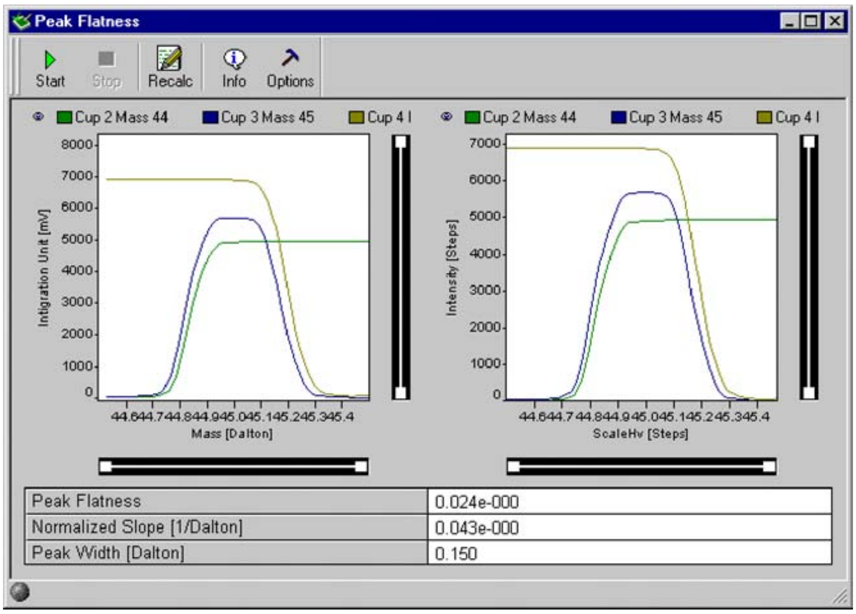
Peak Center...

Energie Correction Peak Center

Channel: Cup 3

Macro: Peak Center

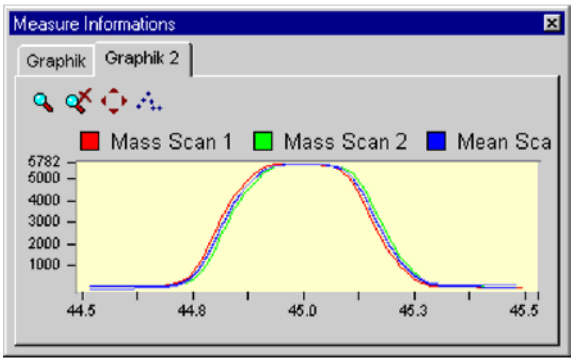
- Values for Peak Flatness, normalized slope [Da^{-1}] and Peak Width [Da] are calculated.



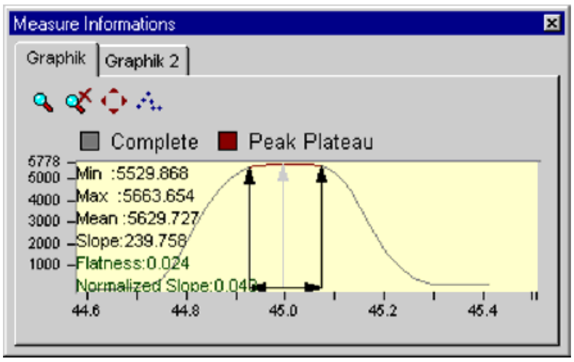
Peak Flatness

Peak Flatness	0.024e-000
Normalized Slope [1/Dalton]	0.043e-000
Peak Width [Dalton]	0.150

- Press the ***Graphic*** tab to view relevant data.



- Press the ***Graphic 2*** tab to view relevant data.



Min :5529.868
Max :5663.654
Mean :5629.727
Slope:239.758
Flatness:0.024
Normalized Slope:0.04

- When closing the "Peak Flatness" window you are asked whether to save the changes.

Save changes to Peak Flatness?

7.2.7 RELATIVE SENSITIVITY

Relative Sensitivity (S_{rel}) describes the dependency of signal intensity (i.e. ion current) on the ion source pressure and is thus given in A/mbar:

$$S_{rel} = \frac{1}{0.69} \frac{1}{p} \frac{U}{R}$$

where:

U: voltage measured at the amplifier of the collector cup (e.g. Cup 3, mass 44)

R: resistor value (e.g. $3 * 10^{8}\ \Omega$ 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.

Δ p: pressure difference between a measurement with and without reference gas.

0.69: correction factor for CO_2. The ion gauge is calibrated with N_2, however, which has a different ionizing probability. The correction factor takes this into account. To calculate it, intensities (i.e. ion currents) and pressures are measured with and without reference gas.

NOTE: ***Two different Relative Sensitivity values exist depending on whether the instrument is equipped with a differential pumping system or not. The difference is due to different pressure readings at the same flow. The Absolute Sensitivity (given in molecules/ion) however, is the same.***

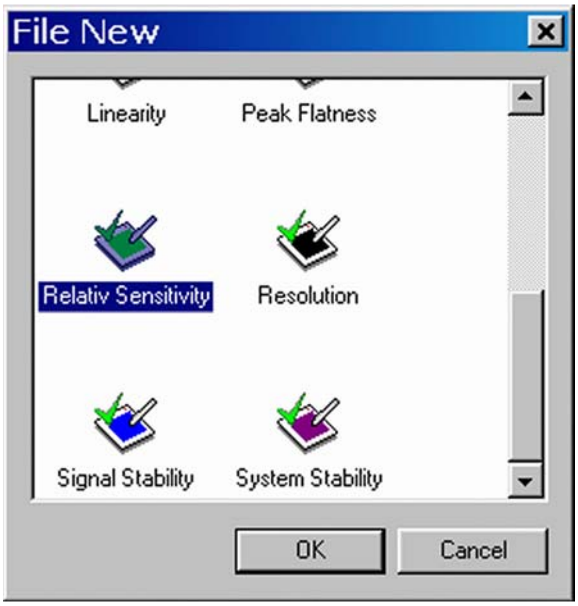
After a cycle of e.g. three measurements and calculations of Relative Sensitivity a mean value is displayed. This value should be about 0.2 A/mbar for a standard system and about 0.5 A/mbar in case of a differentially pumped system. They depend on the pumping capacity of the turbomolecular pumps. Each cup (i.e. each mass) is characterized by a Relative Sensitivity value of its own.

Different values of Relative Sensitivities can only be compared, if pumping speed, conductance, location of the ion gauge etc. are identical. Therefore, it is senseless to compare different types of instruments.

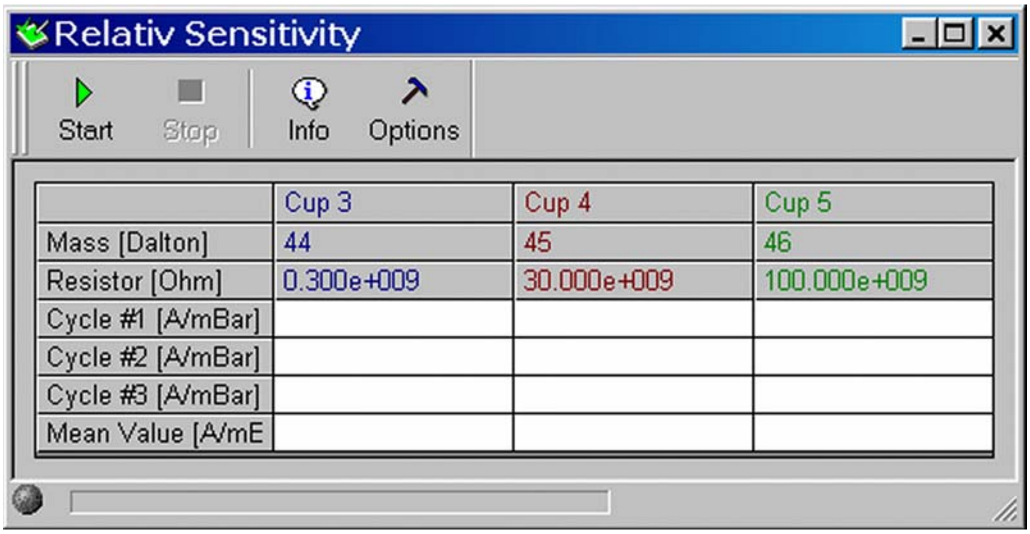
Sources of error

- Resistor values must be configured correctly.
- Relative Sensitivity depends on the accuracy of the high vacuum pressure gauge's accuracy, which is limited.

Testing Relative Sensitivity



- Click the ***Relative Sensitivity Icon*** and press ***OK*** (or double-click the Icon).



	Cup 3	Cup 4	Cup 5
Mass [Dalton]	44	45	46
Resistor [Ohm]	0.300e+009	30.000e+009	100.000e+009
Cycle #1 [A/mBar]			
Cycle #2 [A/mBar]			
Cycle #3 [A/mBar]			
Mean Value [A/mE			

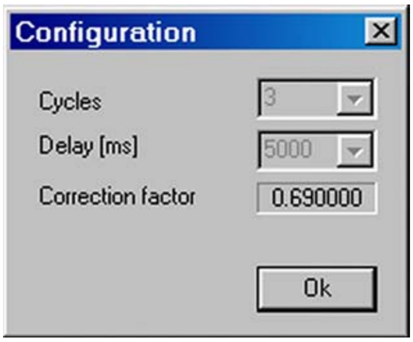
- The cups with the corresponding masses and resistor values are shown.

- On the "Window" toolbar, the ***Relative Sensitivity Icon*** becomes visible.

- Press the ***Options*** button.

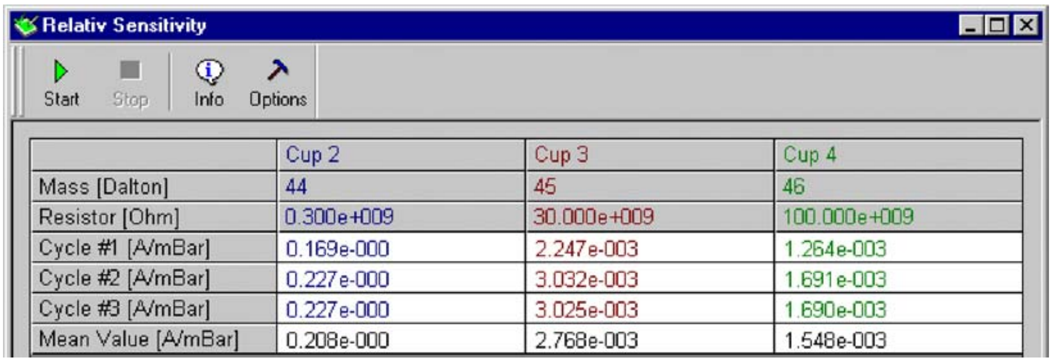
- Press ***OK***.

  Experienced users can change the values via the ***Advanced Mode***.



Cycles: 3
Delay [ms]: 5000
Correction factor: 0.690000

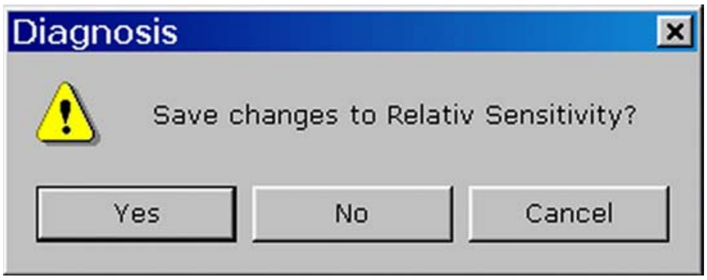
- Click the ***Start*** button.



	Cup 2	Cup 3	Cup 4
Mass [Dalton]	44	45	46
Resistor [Ohm]	0.300e+009	30.000e+009	100.000e+009
Cycle #1 [A/mBar]	0.169e-000	2.247e-003	1.264e-003
Cycle #2 [A/mBar]	0.227e-000	3.032e-003	1.691e-003
Cycle #3 [A/mBar]	0.227e-000	3.025e-003	1.690e-003
Mean Value [A/mBar]	0.208e-000	2.768e-003	1.548e-003

- Finally, for each cup, the values of three cycles and their mean are displayed.

- When closing the "Relative Sensitivity" window you are asked, whether to save the changes.

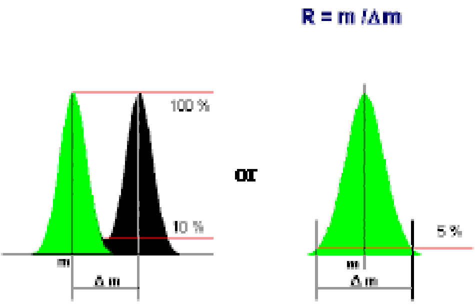


Save changes to Relativ Sensitivity?

7.2.8 RESOLUTION

Resolution describes the masses, which can be separated from each other (i.e. the minimal relative distance between two masses, which can be resolved). Different definitions of resolution are used in mass spectrometry.

- The **10%** valley definition, commonly used for double focusing sector field mass spectrometers, means:



Resolution can be defined as:

1. Mass divided by the mass difference of two neighboring peaks, if the valley between the peaks drops to 10% of the peak height, **or** as

2. Mass divided by the peak width (in Dalton) at 5% of peak height.

According to the 10% valley definition, it is dimensionless (given in m/Δm):

$$R = \frac{m}{\Delta m}$$

where:

R:	Resolution
m:	Mass of the interesting isotope
Δm:	Mass difference between neighboring peaks

In ***Dual Peak mode***, the distance between Peak Centers of two neighboring peaks is measured [Da]. The peak width of one peak is measured at 5% peak height.

In ***Single Peak mode***, the distance between neighboring peaks is set to 1 Da. In both modes, Resolution can be calculated as follows (the mass difference is usually 1 Da):

$$R = \frac{m}{\Delta m}\,\frac{a}{b}$$

where:

a: distance between peak centers

b: peak width of the isotope of interest

Resolution is determined using the narrowest cup (i.e. usually Cup 2). Mass 45 is used.

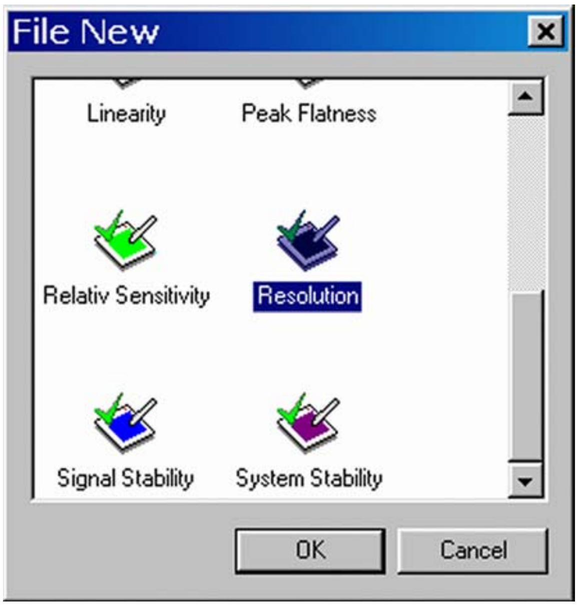
Example

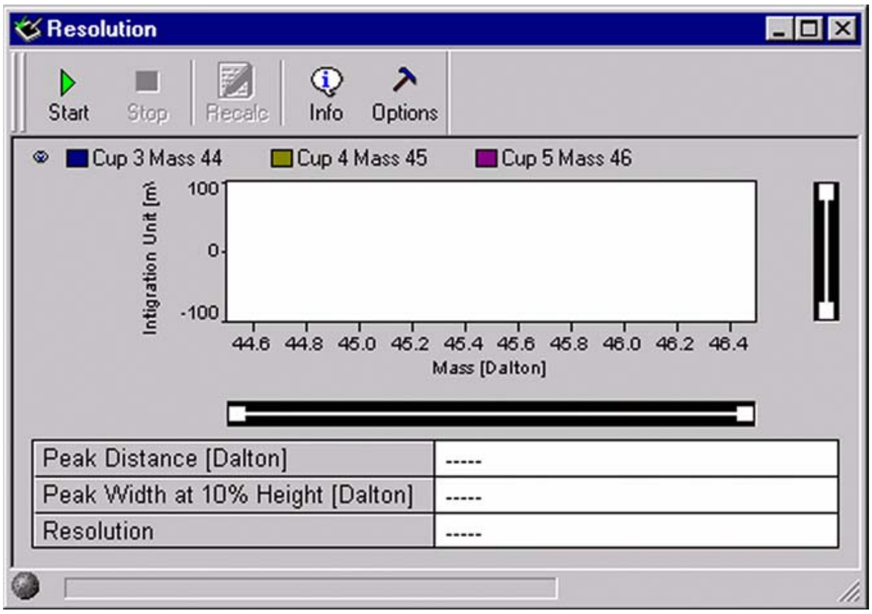
For a resolution of 88 and mass 44, a peak with a distance of (44/88) Da = 0.5 Da could be resolved using the 10% criterion.

Start mass and end mass of the magnetic field scan can be edited. In case of CO_2, the mass ranges from about 43 to 45.5. The BDAC values referring to the masses 44 and 45 are determined and the Resolution is calculated.

Testing Resolution

- Click on the ***Resolution Icon*** and press ***OK*** (or double-click the Icon).

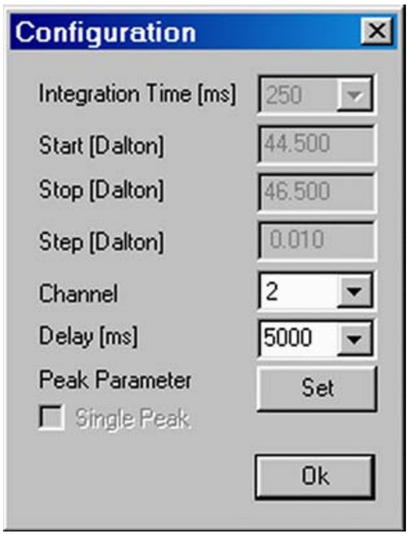




Peak Distance [Dalton]	-----
Peak Width at 10% Height [Dalton]	-----
Resolution	-----

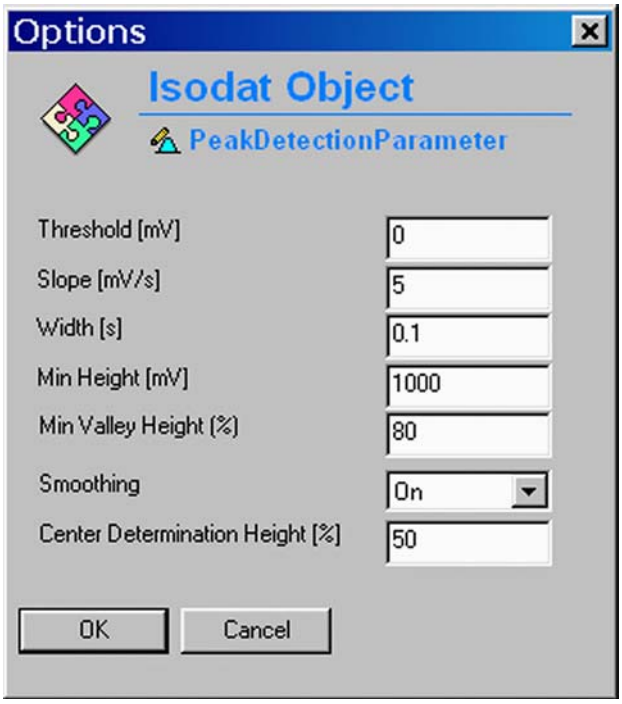
- ➤ On the "Window" toolbar, the ***Resolution Icon*** becomes visible.

- ➤ Press the ***Options*** button.



Integration Time [ms] 250
Start [Dalton] 44.500
Stop [Dalton] 46.500
Step [Dalton] 0.010
Channel 2
Delay [ms] 5000
Peak Parameter
☐ Single Peak

- ➤ Accept the defaults or from the pulldown menus, select a Channel and a Delay value between 100 ms and 20000 ms.
- ➤ If you simply want to accept the Peak Detection parameters without a glance, do not press the ***Set*** button. Instead, click ***OK***.
- ➤ However, as a sophisticated user, you may want to look at the Peak Detection parameters. To do so, press the ***Set*** button.



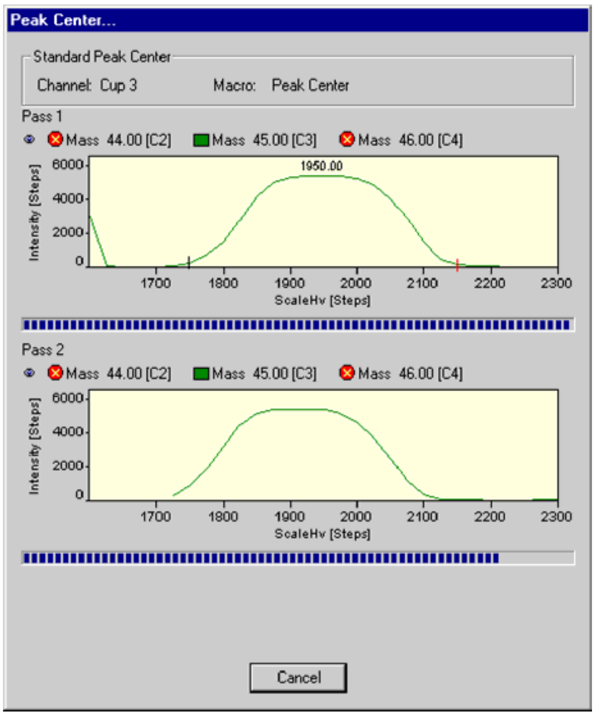
Options

Isodat Object

PeakDetectionParameter

Threshold [mV]	0
Slope [mV/s]	5
Width [s]	0.1
Min Height [mV]	1000
Min Valley Height [%]	80
Smoothing	On
Center Determination Height [%]	50

- ➤ Change the values or accept the defaults.
- ➤ Then click ***OK***.
- ➤ Press the ***Start*** button.



Peak Center...

Standard Peak Center

Channel: Cup 3 Macro: Peak Center

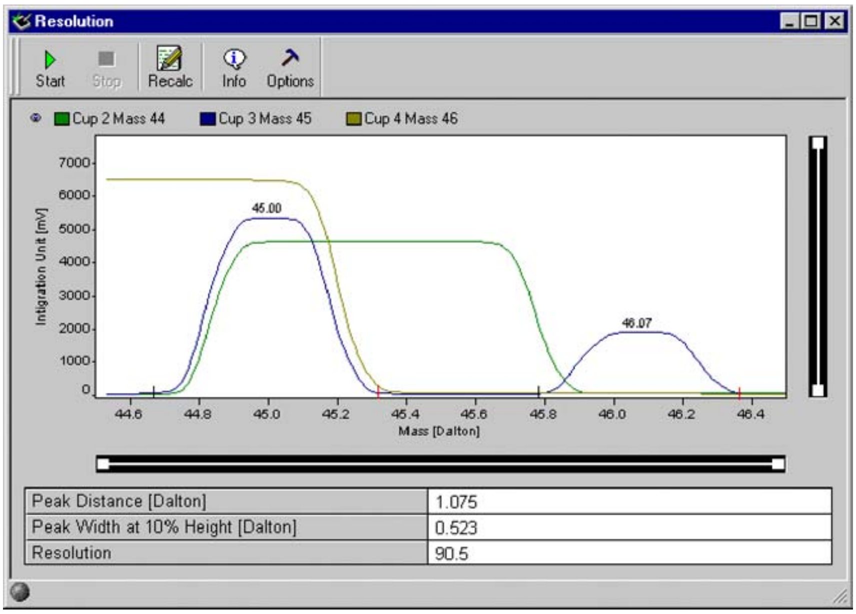
Pass 1

Mass 44.00 [C2] Mass 45.00 [C3] Mass 46.00 [C4]

Pass 2

Mass 44.00 [C2] Mass 45.00 [C3] Mass 46.00 [C4]

- ➤ Testing starts with a Peak Center on the selected cup (e.g. mass 45, cup 3).
  - ❑ If no peak could be detected, the measurement is stopped.
  - ❑ If not enough peaks could be detected, an error message occurs.

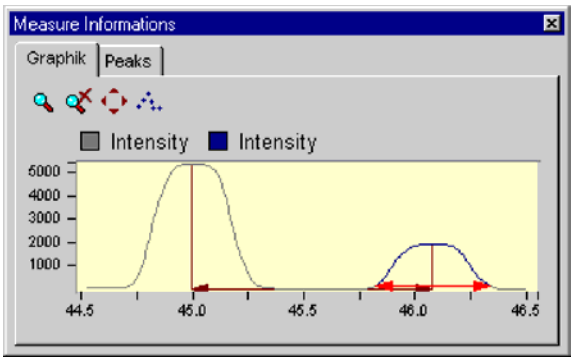


Peak Distance [Dalton]	1.075
Peak Width at 10% Height [Dalton]	0.523
Resolution	90.5

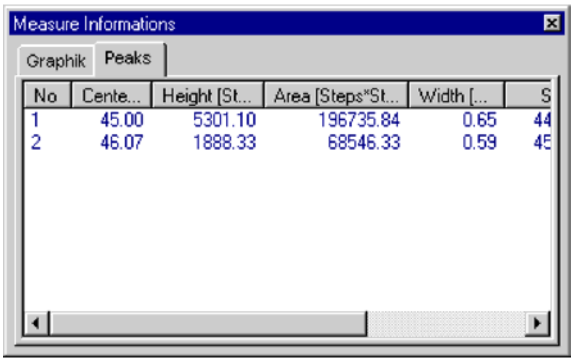
- ➢ Finally, the values of Peak Distance [Da], Peak Width at 10% Height [Da] and Resolution are displayed.

- ➢ Click the ***Info*** button.

- ➢ Press the ***Graphik*** tab.

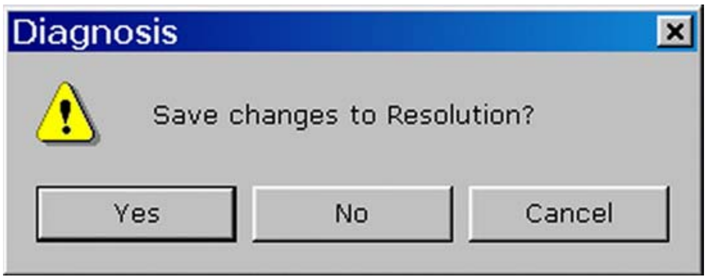


- Press the ***Peaks*** tab.



No	Cente...	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

- When closing the "Resolution" window you are asked, whether to save the changes.



Save changes to Resolution?

7.2.9 SIGNAL STABILITY

Signal Stability describes the stability of the intensity (i.e. peak height). The intensity on top of the peak is measured for a limited period of time (e.g. 5 min). Note the similarity to the System Stability, but here, the stability is not measured at the peak flank, but at the peak center. The value of Signal Stability should be about $2 * 10^{-4}$ (for 5 min).

Two results are obtained:

- Slope of the regression line (normalized by the intensity)
- Standard deviation of the regression line (normalized by the intensity)

Testing Signal Stability requires a signal of 3 V or more!

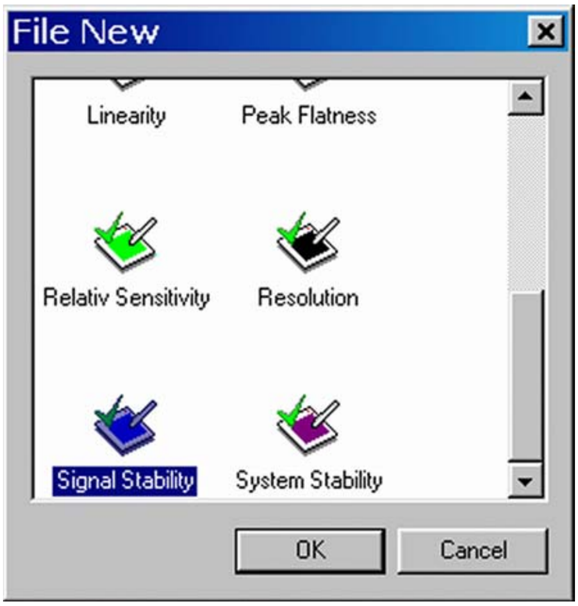
Sources of error

- The slope is usually due to gas consumption during measurement. However, it should be checked, if an unusual result is obtained.
- Instabilities of the emission may cause an unstable signal although a stable high voltage and magnetic field are given.
- Pressure fluctuations (check the oil of the forevacuum pumps!) or temperature fluctuations particularly at the crimps.

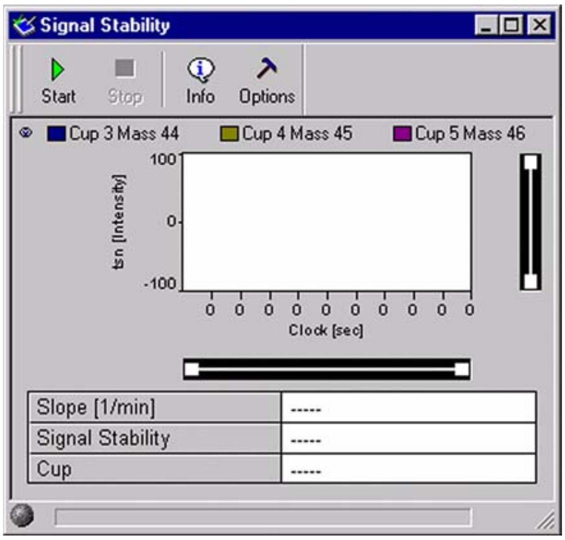
Differences to ISODAT Old

1. In ISODAT Old, maximum deviation is reported instead of standard deviation. However, for a statistical process standard deviation is more relevant as it is less sensitive to measuring time and outliers.
2. Conversion of results to the ISODAT Old scale: Multiplication by a factor of approximately four.

Testing Signal Stability



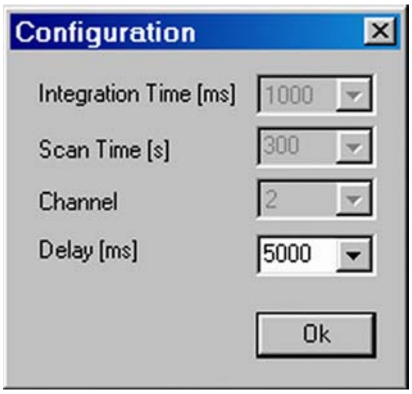
- Click the ***Signal Stability*** Icon and press ***OK*** (or double-click the Icon).



Slope [1/min]	-----
Signal Stability	-----
Cup	-----

- On "Window" toolbar, the ***Signal Stability Icon*** becomes visible.

- Press the ***Options*** button.



Configuration

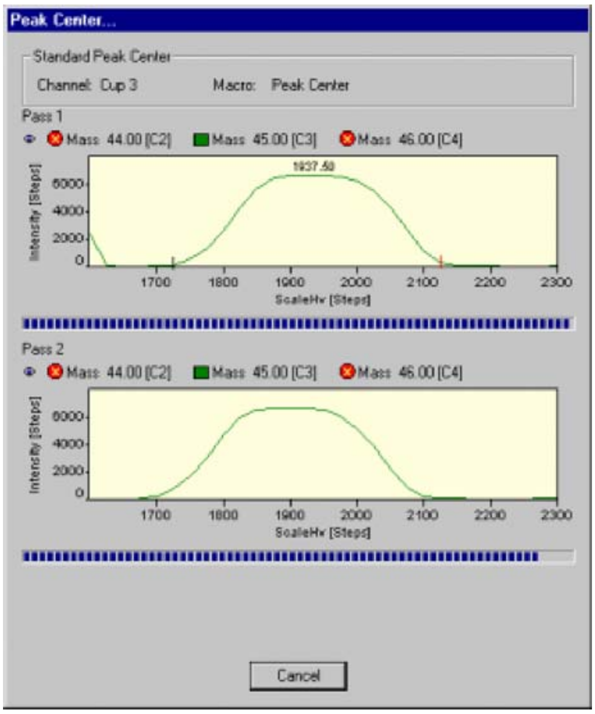
Integration Time [ms] 1000

Scan Time [s] 300

Channel 2

Delay [ms] 5000

- ➢ From the pulldown menu, select a delay value between 100 ms and 20000 ms.
- ➢ Click ***OK***.
- ➢ Press the ***Start*** button.

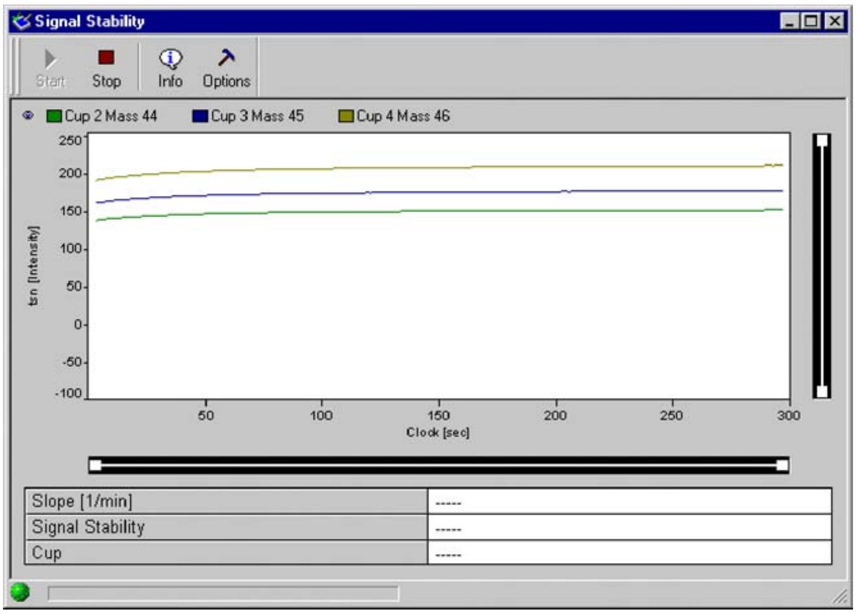


Peak Center...

Standard Peak Center

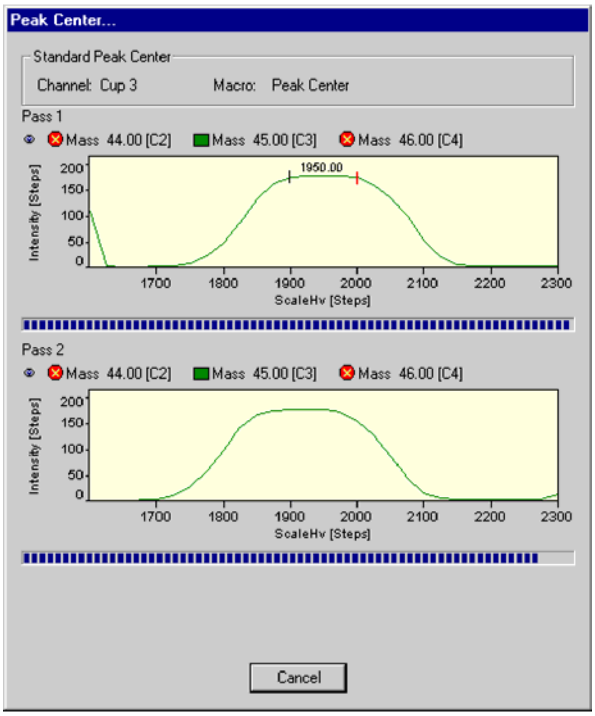
Channel: Cup 3 Macro: Peak Center

- ➢ Testing starts with a Peak Center on the selected Cup (e.g. mass 45, Cup 3).

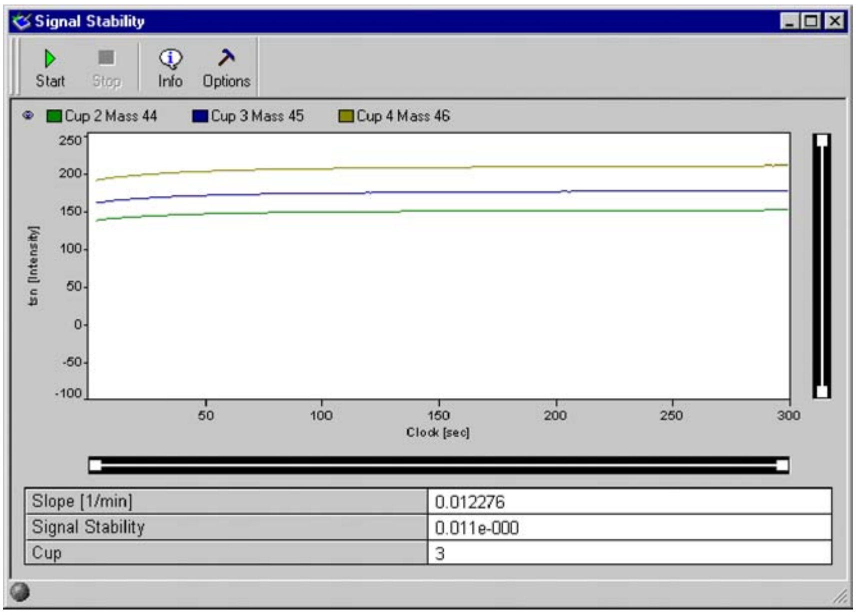


Slope [1/min]	-----
Signal Stability	-----
Cup	-----

- ➢ Another Peak Center is performed.

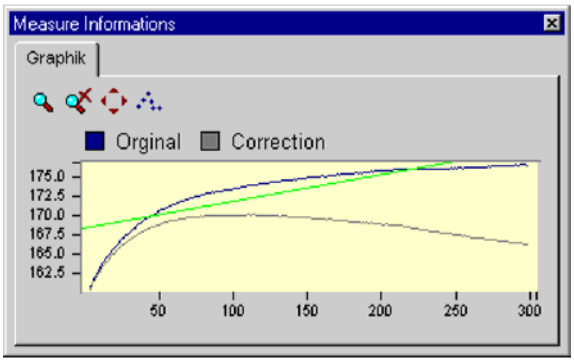


Channel: Cup 3
Macro: Peak Center

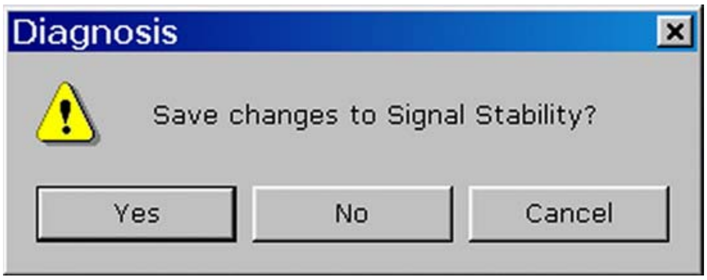


Slope [1/min]	0.012276
Signal Stability	0.011e-000
Cup	3

- ➢ Finally, values of slope [1/min], Signal Stability and the Cup number are displayed.
- ➢ Press the ***Info*** button.
- ➢ Press the ***Graphik*** tab.



- ➢ When closing the "Signal Stability" window you are asked whether to save the changes.



Save changes to Signal Stability?

7.2.10 SYSTEM STABILITY

System Stability informs about high voltage stability and thus magnetic field stability. Already small variations of high voltage or magnetic field dramatically influence signal intensity: They cause peak shifts. The fluctuations of high voltage or the magnetic field strength are measured at the peak flank, because they exert a much higher impact on peak intensity at the flank than on top.

The System Stability test comprises the following steps:

- Determination of peak center and peak flanks
- Set magnetic field to 50% of peak height (at the peak flank).
- Measurement of signal intensity (high voltage fluctuation) at the peak flank for a defined period of time (e.g. 15 min).
- New peak center procedure
- Calculation of System Stability [min^{-1}] and Relative Mass Drift [min^{-1}] (either electronic or to magnetic drift) using the slope of the peak flank.

⚠ ***Testing System Stability requires a signal of 3 V or more!***

Two results are obtained:

- Slope of relative mass drift vs. time (time drift)
- Standard deviation of this slope (scatter of the mass)

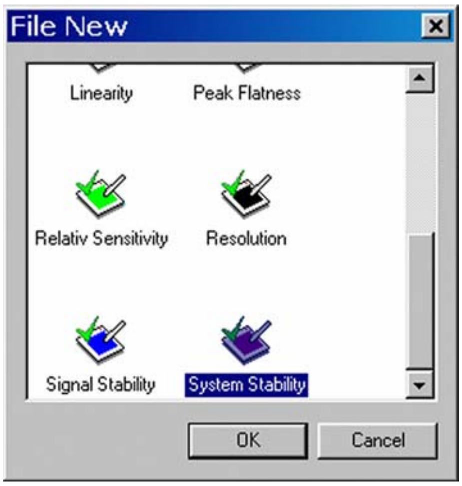
A value of $5*10^{-4}$ measured for a period of 15 min reflects a good System Stability.

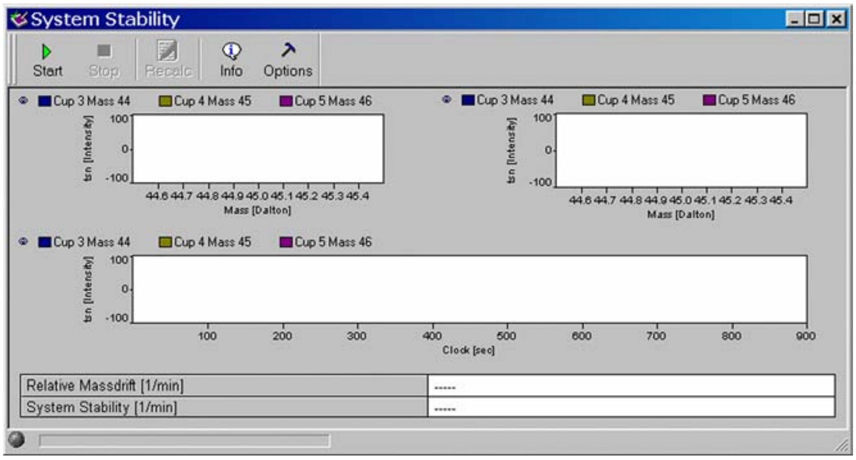
Differences to ISODAT Old

- The slope is a new, but important parameter, as it shows the effect of slow fluctuations in temperature, supplied voltage etc.
- In ISODAT Old, maximum deviation is reported instead of standard deviation. However, for a statistical process, standard deviation is more relevant as it is less sensitive to measuring time and outliers.
- Conversion of the Results to the ISODAT Old scale: multiplication by a factor.

Testing Signal Stability

- Click the ***System Stability Icon*** and press ***OK*** (or double-click the Icon).



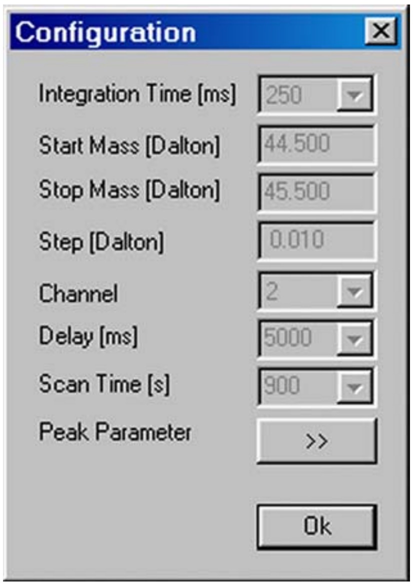


Relative Massdrift [1/min]	-----
System Stability [1/min]	-----

- On the "Window" toolbar, the ***System Stability Icon*** becomes visible.

- Press the ***Options*** button.

- If you simply want to accept the Peak parameters without glancing at them, do not press the ***Peak Parameter*** button. Instead, click ***OK***.
- However, as an experienced user, you may want to look at the Peak parameters. To do so, press the ***Peak Parameter*** button.



Configuration

Integration Time [ms] 250

Start Mass [Dalton] 44.500

Stop Mass [Dalton] 45.500

Step [Dalton] 0.010

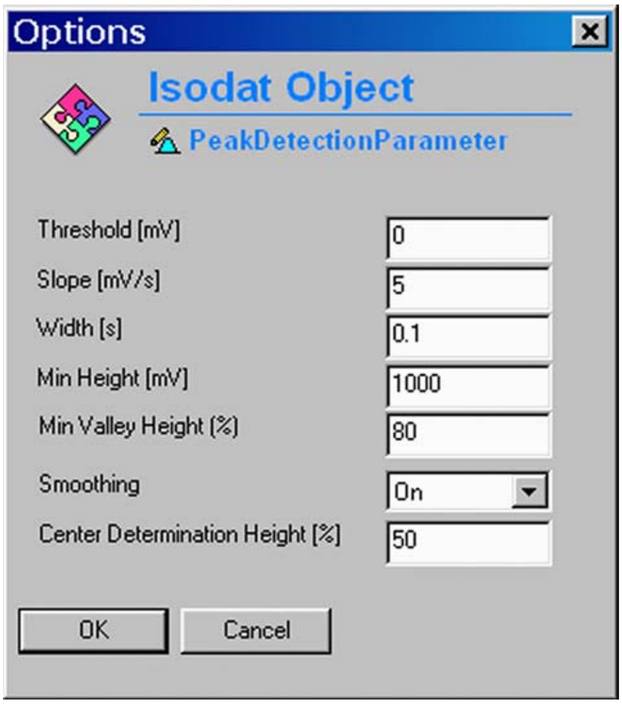
Channel 2

Delay [ms] 5000

Scan Time [s] 900

Peak Parameter >>

- Experienced users may further want to change the gray default values (from Integration Time to Scan Time). This can be achieved via ***Advanced Mode***.



Options

Isodat Object

PeakDetectionParameter

Threshold [mV] 0

Slope [mV/s] 5

Width [s] 0.1

Min Height [mV] 1000

Min Valley Height (%) 80

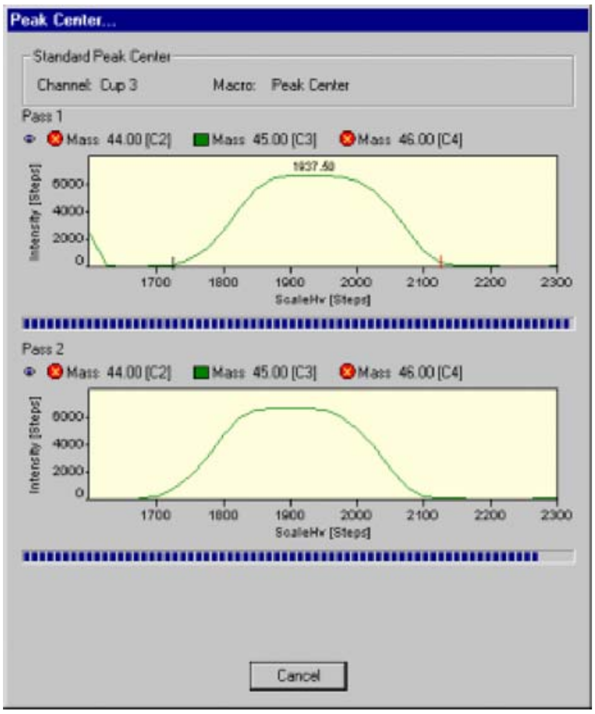
Smoothing On

Center Determination Height [%] 50

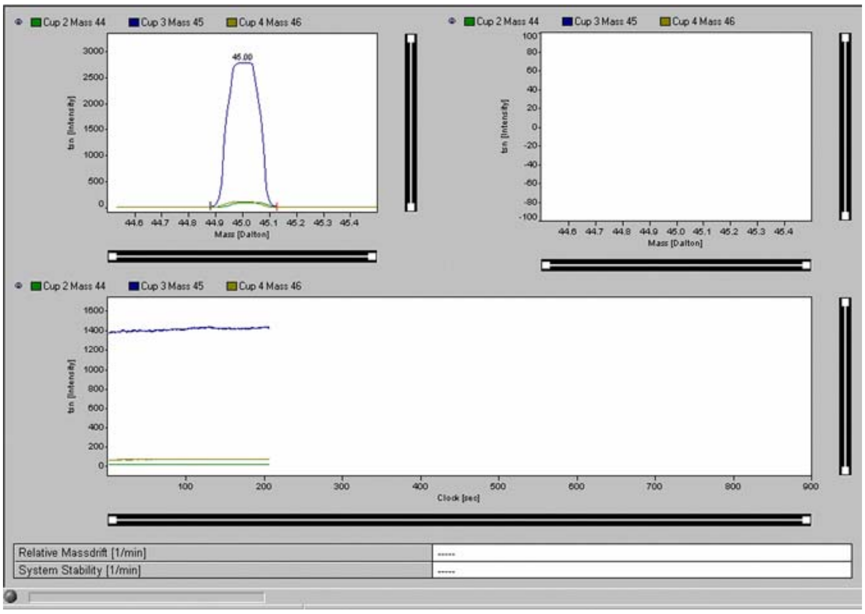
- Make your changes or accept the defaults.
- Then press ***OK***.

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

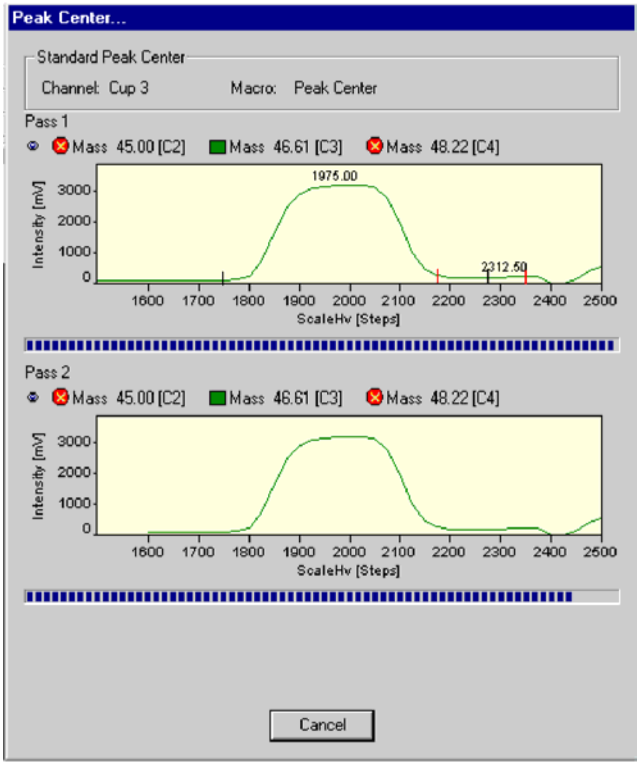
- Testing starts with a Peak Center on the selected Cup, e.g. mass 45, Cup 3.



Channel: Cup 3 Macro: Peak Center



Relative Massdrift [1/min]	-----
System Stability [1/min]	-----



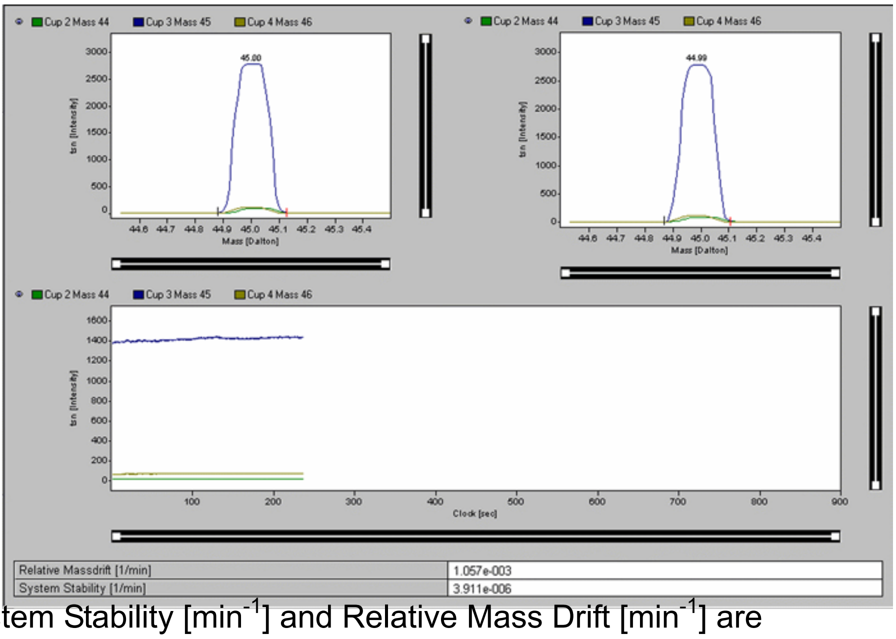
Peak Center...

Standard Peak Center

Channel: Cup 3

Macro: Peak Center

- ➢ Testing continues with another Peak Center on the selected Cup.

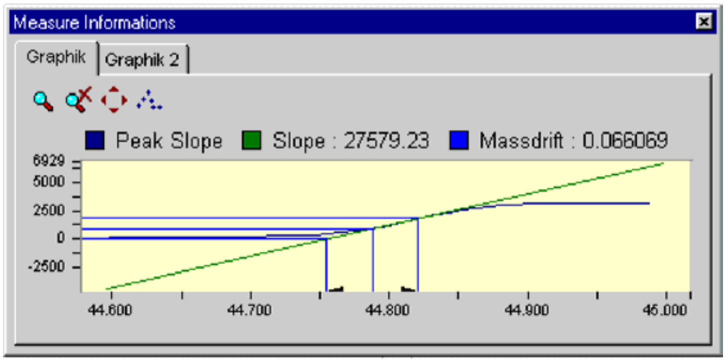


Relative Massdrift [1/min]	1.057e-003
System Stability [1/min]	3.911e-006

- ➢ Finally, values for System Stability [min^{-1}] and Relative Mass Drift [min^{-1}] are displayed.

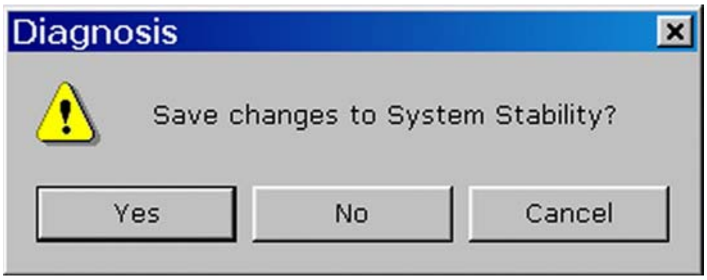
- Press the ***Info*** button.

- Pressing the ***Graphik*** tab, the diagram for calculating Slope and Mass Drift is shown together with the respective values.



■ Peak Slope ■ Slope : 27579.23 ■ Massdrift : 0.066069

- When closing the "System Stability" window you are asked whether to save the changes.



Save changes to System Stability?

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MANUAL HISTORY

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