Finnigan™
DELTA plus XP

Operating Manual

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Customer Registration... Register now and receive all the privileges associated with being a Thermo Electron, Finnigan product user, including application reports and technical reports.

Name

Title

Company

Address

City/State/Postal Code

Country

Telephone _____ **Ext.** _____

Tell us more... Let us know more about how you use this product:

My Organization Is: (Check one only)
- ❑ Commercial (for profit) lab
- ❑ Government lab
- ❑ Hospital / Clinic
- ❑ Research Institute
- ❑ University / College
- ❑ Veterinary
- ❑ Other _____

Job Function: (Check one only)
- ❑ Administration
- ❑ Lab Management
- ❑ Operator
- ❑ Other _____

My Primary Application Is: (Check one only)
- ❑ Analytical
- ❑ Biomedical
- ❑ Clinical / Toxicology
- ❑ Energy
- ❑ Food / Agriculture
- ❑ Forensic / Toxicology
- ❑ Pharmaceutical
- ❑ Research / Education
- ❑ Other _____

Reader Survey... Help us to improve the quality of our documentation by answering a few questions:

Finnigan DELTA^{plus}XP
Operating Manual

Revision A
114 9081

	Strongly Agree	Agree	Disagree	Strongly Disagree
The manual is well organized.	1	2	3	4
The manual is clearly written.	1	2	3	4
The manual contains all of the information I need.	1	2	3	4
The instructions are easy to follow.	1	2	3	4
The instructions are complete.	1	2	3	4
The technical information is easy to understand.	1	2	3	4
The figures are helpful.	1	2	3	4

Additional Comments: (Attach additional sheets if necessary.)

Tear this sheet from the manual, fold it closed, stamp it, and drop it in the mail.

From _____

_____

_____

Thermo Electron (Bremen) GmbH
Finnigan Advanced Mass Spectrometry
Product Marketing
Barkhausenstr. 2

D-28197 Bremen
Germany

Reparatur - Begleitkarte   # Repair-Covering Letter

Bitte vollständig ausfüllen   ### Please fill in completely

Absender: **Geräte-Type:**   **Service-Nr.:**
Dispatcher: **Instrument Type:**   **Service-No.:**

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

- ☐ **DOA**

- ☐ **Teil wurde nicht benutzt / part not used**

- ☐ **Teil wurde nur für Testzwecke verwendet / used for test purposes only**

- ☐ **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 gesundheits - schädlichen Stoffen sind, bzw. vor Ihrer Einsen - dung an Thermo Electron Bremen dekontami - niert 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 Electron Bremen.

-------------------------------   --
Datum / Date   **Unterschrift / Signature**

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

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

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

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

Delta plus XP
Basic Instrument

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

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

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

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

Ergänzende Informationen:
complementary information:
informazioni complementari:

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

Bremen, Germany, 21 March 2002

ThermoFinnigan

Der Betriebsleiter:
Operations Manager:
Direttore fabrizione:

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Notice on Lifting and Handling of
Thermo Electron Bremen Instruments

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

Notice on the Proper Use of
Thermo Electron Bremen Instruments

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

Safety and EMC Information

In accordance with our commitment to customer service and safety, these instruments have satisfied the requirements for the European CE Mark including the Low Voltage Directive.

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

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

Identifying Safety Information

This reference manual contains precautionary statements that can prevent personal injury, instrument damage, and loss of data if properly followed. All statements of this nature are called to your attention through the use of bold type and the following icons:

Warning!	Cold Surface!	Hot Surface	Strong Magnetic Field!	High Voltage!

Every instrument has specific hazards, so be sure to read and comply with the following precautions. They will help ensure the safe, long-term use of your system.

1. Before plugging in any of the instrument modules or turning on the power, always make sure that the voltage and fuses are set appropriately for your local line voltage.

2. Only use fuses of the type and current rating specified. Do not use repaired fuses and do not short-circuit the fuse holder.

3. The supplied power cord must be inserted into a power outlet with a protective earth contact (ground). When using an extension cord, make sure that the cord also has an earth contact.

4. Do not change the external or internal grounding connections. Tampering with or disconnecting these connections could endanger you and/or damage the system.

> **Caution.** The instrument is properly grounded in accordance with regulations when shipped. You do not need to make any changes to the electrical connections or to the instrument's chassis to ensure safe operation.

5. Never run the system without the housing on. Permanent damage can occur.

6. Do not turn the instrument on if you suspect that it has incurred any kind of electrical damage. Instead, disconnect the power cord and contact a Service Representative for a product evaluation. Do not attempt to use the instrument until it has been evaluated. (Electrical damage may have occurred if the system shows visible signs of damage, or has been transported under severe stress.)

7. Damage can also result if the instrument is stored for prolonged periods under unfavorable conditions (e.g., subjected to heat, water, etc.).

8. Always disconnect the power cord before attempting any type of maintenance.

9. Capacitors inside the instrument may still be charged even if the instrument is turned off.

10. Never try to repair or replace any component of the system that is not described in this manual without the assistance of your service representative.

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Finnigan DELTA^{plus}XP _____

Contents

Thermo
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Read This First

Welcome to the Thermo Electron, Finnigan™ DELTA^{plus}XP system! The DELTA^{plus}XP is a member of the Finnigan family of mass spectrometer (MS) detectors.

This Finnigan DELTA^{plus}XP Operating Manual contains a description of the modes of operation and principle hardware components of your DELTA^{plus}XP instrument. In addition, this manual provides step-by-step instructions for cleaning and maintaining your instrument.

This manual includes the following chapters:

Chapter 1: Getting Started gives instructions for setting up the instrument and the software.

Chapter 2: Analyzer gives an overview of the components of the analyzing system.

Chapter 3: Vacuum System gives an overview of the components of the vacuum system.

Chapter 4: Electronic Devices gives an overview of the electronic boards and their functions.

Chapter 5: Dual Inlet System gives an overview of the parts of the Dual Inlet System.

Chapter 6: Measurements gives instructions about performing various types of measurements.

Chapter 7: Diagnosis gives instructions about performing various types of diagnosis.

Chapter 8: Technical Information outlines the maintenance procedures that you should perform on a regular basis to maintain optimum MS detector performance. It also lists the replaceable parts for the MS detector and data system.

Changes to the Manual and Online Help

To suggest changes to this manual or the online Help, please send your comments to:

Editors, Technical Documentation
Thermo Electron (Bremen) GmbH
Finnigan Advanced Mass Spectrometry
Barkhausenstr. 2

D-28197 Bremen

Germany

e-mail: documentation@thermo-bremen.com

You are encouraged to report errors or omissions in the text or index.

Thank you.

Abbreviations

The following abbreviations are used in this and other manuals and in the online Help.

A	ampere
ac	alternating current
ADC	analog-to-digital converter
AP	acquisition processor
APCI	atmospheric pressure chemical ionization
API	atmospheric pressure ionization
APPI	atmospheric pressure photo ionization
b	bit
B	byte (8 b)
°C	degrees Celsius
CAN	Controller Area Network
cfm	cubic feet per minute
CI	chemical ionization
CIP	carriage and insurance paid to
cm	centimeter
cm^3	cubic centimeter
CPU	central processing unit (of a computer)
CRM	consecutive reaction monitoring
<Ctrl>	control key on the terminal keyboard
d	depth
Da	dalton
DAC	digital-to-analog converter
dc	direct current
DEP™	direct exposure probe
DSP	digital signal processor
EI	electron ionization
EMI	electromagnetic interference
<Enter>	enter key on the terminal keyboard
ESD	electrostatic discharge
ESI	electrospray ionization

eV	electron volt
f	femto (10^{-15})
°F	degrees Fahrenheit
.fasta	file extension of a SEQUEST search database file
FOB	free on board
ft.	foot; feet
FTP	file transfer protocol
FWHM	Full Width at Half Maximum
g	gram
G	giga (10^9)
GC	gas chromatograph; gas chromatography
GC/MS	gas chromatograph / mass spectrometer
GND	electrical ground
GPIB	general-purpose interface bus
GUI	graphical user interface
h	hour
h	height
HPLC	high-performance liquid chromatograph
HV	high voltage
Hz	hertz (cycles per second)
ICIS™	Interactive Chemical Information System
ICL™	Instrument Control Language.
IEC	International Electrotechnical Commission
IEEE	Institute of Electrical and Electronics Engineers
in.	inch
I/O	input/output
k	kilo (10^3, 1000)
K	kilo (2^{10}, 1024)
kg	kilogram
l	length
L	liter
LAN	local area network

lb.	pound
LC	liquid chromatograph; liquid chromatography
LC/MS	liquid chromatograph / mass spectrometer
LED	light-emitting diode
LHe	liquid helium
LN2	liquid nitrogen
μ	micro (10^{-6})
m	meter
m	milli (10^{-3})
M	mega (10^{6})
M^+	molecular ion
MH^+	protonated molecular ion
min	minute
mL	milliliter
mm	millimeter
MS	mass spectrometer; mass spectrometry
MS	MS^n power: where n = 1
MS/MS	MS^n power: where n = 2
MS^n	MS^n power: where n = 1 through 10
m/z	mass-to-charge ratio
n	nano (10^{-9})
NCBI	National Center for Biotechnology Information (USA)
NIST	National Institute of Standards and Technology (USA)
Ω	ohm
p	pico (10^{-12})
Pa	pascal
PCB	printed circuit board
PID	proportional / integral / differential
P/N	part number
P/P	peak-to-peak voltage
ppm	parts per million
psig	pounds per square inch, gauge

RAM	random access memory
RF	radio frequency
RMS	root mean square
ROM	read-only memory
RS-232	industry standard for serial communications
s	second
SIM	selected ion monitoring
solids probe	direct insertion probe
SRM	selected reaction monitoring
TCP/IP	transmission control protocol / Internet protocol
TIC	total ion current
TSQ™	triple stage quadrupole
TWA	Time Weighted Average
u	atomic mass unit
V	volt
V ac	volts alternating current
V dc	volts direct current
vol	volume
w	width
W	watt

> **Note.** Exponents are written as superscripts. In the corresponding online Help, exponents are sometimes written with a caret (^) or with *e* notation because of design constraints in the online Help. For example:
>
> MS^n (in this manual)   Ms^n (in the online Help)
>
> 10^5 (in this manual)   10^5 (in the online Help)

Typographical Conventions

Typographical conventions have been established for Thermo Electron Bremen manuals for the following:

• Data input

• Boxed information

• Topic headings

Data Input

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

• Messages displayed on the screen are represented by capitalizing the initial letter of each word and by italicizing each word.

• Input that you enter by keyboard is represented in **bold face letters**. (Titles of topics, chapters, and manuals also appear in bold face letters.)

• For brevity, expressions such as "choose **File > Directories**" are used rather than "pull down the File menu and choose Directories."

• Any command enclosed in angle brackets < > represents a single keystroke. For example, "press <F1>" means press the key labeled *F1*.

• Any command that requires pressing two or more keys simultaneously is shown with a plus sign connecting the keys. For example, "press **<Shift> + <F1>**" means press and hold the <Shift> key and then press the <F1> key.

• Any button that you click on the screen is represented in bold face letters and a different font. For example, "click on **Close**".

Boxed Information

Information that is important, but not part of the main flow of text, is displayed in a box such as the one below.

> **Note**. Boxes such as this are used to display information.

Boxed information can be of the following types:

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

• **Caution** – information necessary to protect your instrument from damage.

• **Warning** – hazards to human beings. Each Warning is accompanied by a Warning symbol.

Topic Headings

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

Chapter 1
Chapter Name

1.2 Second Level Topics

Third Level Topics

Fourth Level Topics

Fifth Level Topics

Safety Precautions

Observe the following safety precautions when you operate or perform service on the MS detector.

Do Not Perform Any Servicing Other Than That Contained in the Finnigan DELTA^{plus}XP Operating Manual.

To avoid personal injury or damage to the instrument, do not perform any servicing other than that contained in the **Finnigan DELTA^{plus}XP Operating Manual** or related manuals unless you are qualified to do so.

Shut Down the MS Detector and Disconnect It From Line Power Before You Service It.

High voltages capable of causing personal injury are used in the instrument. Some maintenance procedures require that the mass spectrometer be shut down and disconnected from line power before service is performed. Do not operate the mass spectrometer with the top or side covers off. Do not remove protective covers from PCBs.

Use Care When Changing Vacuum Pump Oil.

Treat drained vacuum pump oil and pump oil reservoirs with care. Hazardous compounds introduced into the system might have become dissolved in the pump oil. Always use approved containers and procedures for disposing of waste oil. Whenever a pump that has been operating on a system used for the analysis of toxic, carcinogenic, mutagenic, or corrosive/irritant chemicals, the pump must be decontaminated by the user. It must be certified to be free of contamination before repairs or adjustments are made by a Thermo Electron Customer Support Engineer or before it is sent back to the factory for service.

Service Philosophy

Servicing the DELTA^{plus}XP system consists of performing procedures required to maintain system performance standards, to prevent system failure, and/or to restore the system to an operating condition. Routine and preventive maintenance procedures are documented in this manual.

Routine and preventive maintenance are the responsibility of the user during and after the warranty period. Regular maintenance will increase the life of the system, maximize the up-time of your system, and allow you to achieve optimum system performance.

Service not described in this manual should be performed only by a Thermo Electron Customer Support Engineer or similarly trained and qualified technical personnel.

Level of Repair

Thermo Electron's service philosophy for the DELTA^{plus}XP system calls for troubleshooting to the lowest part, assembly, PCB, or module listed in the **Technical Information** chapter of this manual.

For mechanical failures: A mechanical assembly typically is to be repaired to the level of the smallest item listed in the **Technical Information** chapter of this manual.

For electronic failures: PCBs are not repaired to the component level except in certain cases of fuses, relays, etc. When these exceptions occur, the components can be found in the **Technical Information** chapter.

Repair-Covering Letter

This manual contains a Repair-Covering Letter, which is located at the front. Further forms can be ordered from the Field Service.

If you return repair/exchange parts, please add our Repair-Covering Letter, complete filled out. Additionally you confirm by signing, that vacuum parts are not contaminated.

Training

To reach a high level of performance with the DELTA^{plus}XP, we recommend making use of the operator courses provided by us at our facilities in Bremen, and/or onsite.

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

Thermo Electron (Bremen) GmbH
Finnigan Advanced Mass Spectrometry
Barkhausenstr. 2
D-28197 Bremen

Germany

Phone: +49-421-5493-0

Fax: +49-421-5493-396

Internet:www.thermo.com

Reply Cards

Thermo Electron Bremen manuals contain one or two reply cards. All manuals contain a Customer Registration / Reader Survey card and some contain a Change of Location card. These cards are located at the front of each manual.

The Customer Registration / Reader Survey card has two functions. First, when you return the card, you are placed on the Thermo Electron Bremen mailing list. As a member of this list, you receive application reports and technical reports in your area of interest, and you are notified of events of interest, such as user meetings. Second, it allows you to tell us what you like and do not like about the manual.

The Change of Location card allows us to track the whereabouts of the instrument. Fill out and return the card if you move the instrument to another site within your company or if you sell the instrument. Occasionally, we need to notify owners of our products about safety or other issues.

Chapter 1
Getting Started

1.1   Introduction

This manual describes the functions and the fundamental measuring procedures of your Finnigan DELTA^{plus}XP mass spectrometer. In addition, specific manuals for the purchased peripheral equipment are supplied.

To obtain a good understanding of the complete system, it is necessary to study the operating manual and the online-help of the software before starting up your instrument. A basic knowledge of handling computers and of the ISODAT NT software is assumed for proper operation of the DELTA^{plus}XP.

To reach a high level of performance with the DELTA^{plus}XP, we recommend making use of the operator courses provided by us at our facilities in Bremen, and/or onsite.

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

> Thermo Electron (Bremen) GmbH
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> Finnigan Advanced Mass Spectrometry
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> Barkhausenstr. 2
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> 28197 Bremen, Germany
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> Phone: +49-421-5493-0
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> Fax: +49-421-5493-396
>
> Internet: www.thermo.com

Basic Instrument

With the DELTA^{plus}XP, you can measure gas isotope ratio of:

H/D, $^{13}C/^{12}C$, $^{15}N/^{14}N$, $^{18}O/^{16}O$, $^{32}S/^{34}S$, and $^{37}Cl/^{35}Cl$.

The DELTA^{plus}XP provides a flexible and open platform for the connection of inlet systems and preparation devices. Thermo Electron-supplied inlet systems are automatically recognized by a "plug and measure" concept. In addition, the system is open for easy connection and control of custom inlet/preparation systems.

For classical applications, the basic instrument can be equipped with a dual inlet system. It is of modular design for the adaptation of different inlet modules. This enables a configuration of the instrument tailored to the requirements of the user.

On the sample side can be connected:

- a secondary inlet system for up to 20 samples, i.e. a multiport with or without automatic tube crackers.

- a microvolume for very small samples,

- external multisample inlets for separating and purifying samples or,

- installations for "on-line" coupling to gas chromatographs, elemental analyzer or other peripherals.

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

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

Figure 1-1.   Front View of DELTA^plus^XP, with TC/EA and ConFlo III

Isodat NT Software

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

System Control

All aspects of the mass spectrometer are controlled by software, including ion generation, mass separation and ion detection. Control of the ion source allows manual tuning, auto tuning, as well as storage and retrieval of ion source parameters.

Different configurations representing different analytical setups can be stored and retrieved. Up to eight simultaneous data acquisition streams are supported.

Automation

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

Open Architecture

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

Data Evaluation and Display

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

1.2   Getting Started: Hardware

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

First Steps

1. Unpack your IRMS and arrange it at the desired place in your laboratory.

2. Connect the end of the hose for compressed air (that is a thin transparent tube) that leaves the IRMS at its rear panel to your wall outlet or a compressor for compressed air. You need at least 5 bar.

to pressure regulator
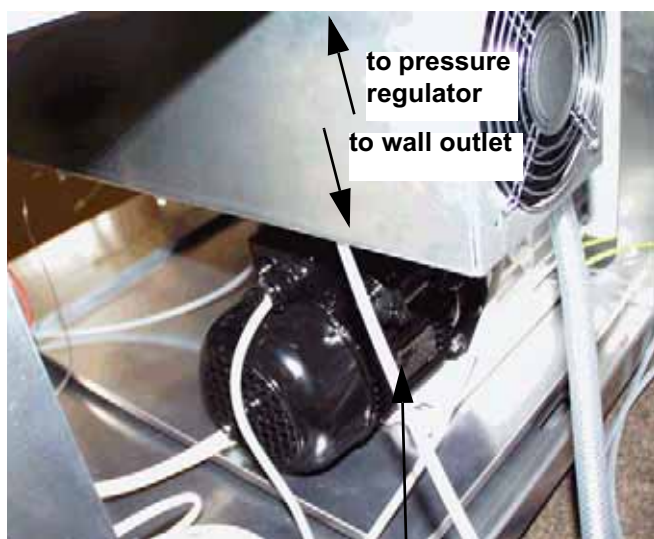
to wall outlet

tube for compressed air

- leaves IRMS at its rear panel

**- leads to wall outlet for
    compressed air and manometer**

**wall outlet for
compressed air**

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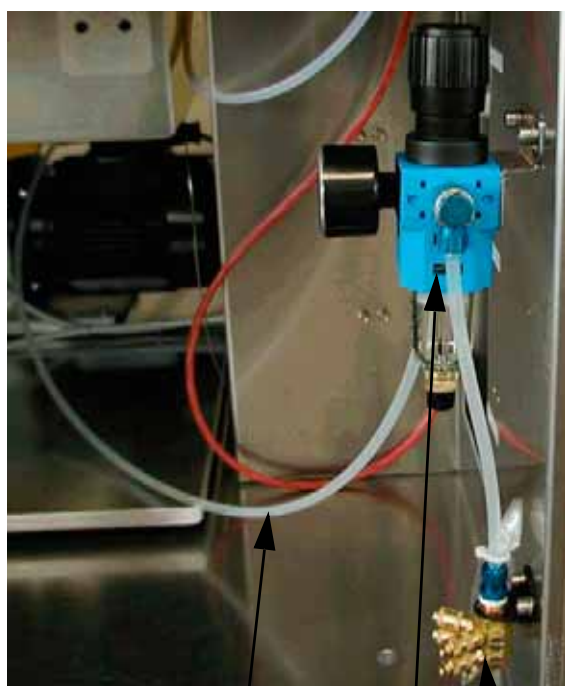
compressed air tube

pressure regulator

distributor with ports

pressure regulating screw

pressure regulator

compressed air tube

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

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

distributor with ports

4. Lay the waste gas tube (at the output of the vacuum pumps) outdoors in order to prevent accumulation of oil mist and perilous gases (e.g. CO, H_2). If available, connect to exhaust vent of your laboratory.

to Fore Vacuum Pump

outward

to Fore Vacuum Pump

outward
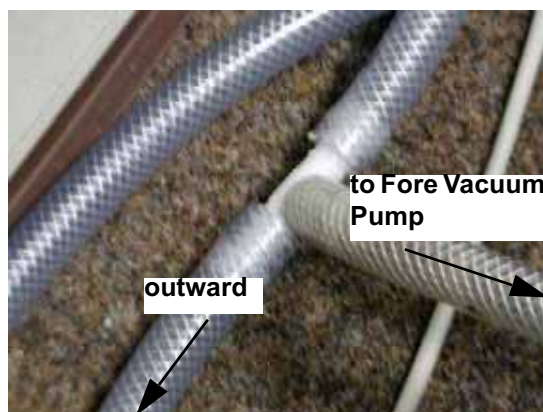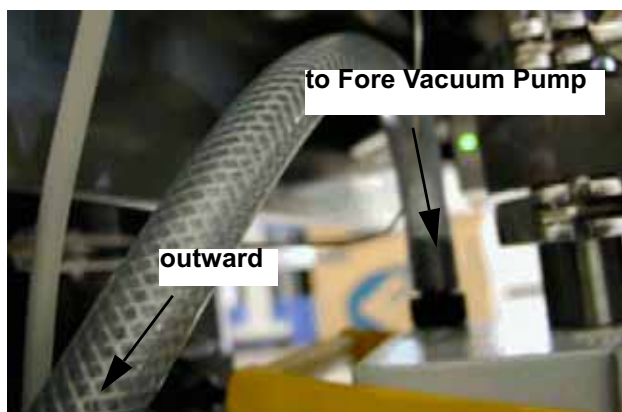
Power Supply

1. Connect your IRMS to the power supply.

to IRMS

to power supply

2. At the instrument's rear panel are sockets to connect up to four peripherals, if available. For first tests, the IRMS is checked without any peripheral connected to it. Later, they are connected one at a time.
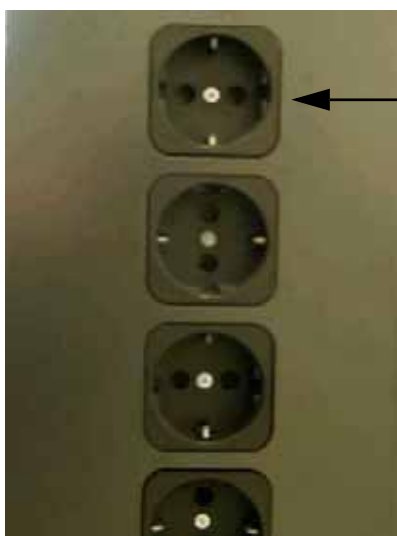
sockets for connecting peripherals

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

Pumping System

Three Fore Vacuum Pumps are used:

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

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

Before starting the pumping system, it is assumed that:

•   the Fore Vacuum pumps are filled with oil,

•   they are connected to the power supply, and

•   their gas ballast is shut.

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

The gas ballast is shut by turning the switch to position 0. In case of Pfeiffer "Duo 5", i.e. (1) the switch is located on top of the pump. In case of Pfeiffer "Duo 2.5", i.e. (2, 3) the switch is located sidewise (refer to the Pfeiffer Operating Instructions of your pumps).
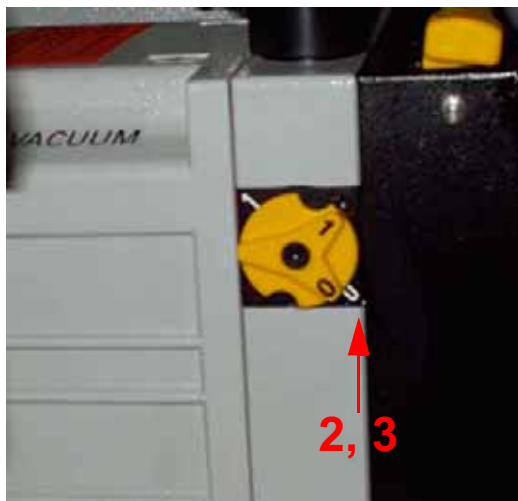
The Control Panel at the front panel of the electronic cabinet shows switches for operating the pumps. The corresponding LEDs are described below.
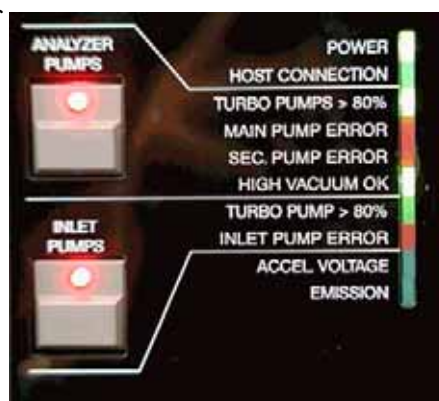
1.   Turn on the pumps by pressing the **Analyzer Pumps** switch.

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The **Inlet Pumps** switch must only be additionally pressed in case of a Dual Inlet System. In case of no Dual Inlet system, this switch has no function.

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

- After 15 min, the green LEDs "Analyzer Pumps: Turbo Pumps > 80%" and "Inlet Pumps: Turbo Pump > 80 %" must be on.

- If one of the turbo pumps does not reach 80 % of the rotation speed after a specific period of time, the pumping system will shut down automatically.

- After 20 min, the security threshold should be reached. The source can then be switched on.

- The red LEDs "Main Pump Error", "Sec. Pump Error" and "Inlet Pump Error" indicate errors concerning the turbo pumps (e.g. after about 15 min, the security threshold of $\approx 3*10^{-5}$ mbar has not been reached).

> **Note.** If properly functioning, the red LEDs must not be on!

Source Heater

When the source heater (the halogen lamps within the ion source) is turned on, both LEDs must be on. Otherwise, one of the heaters might be defective.
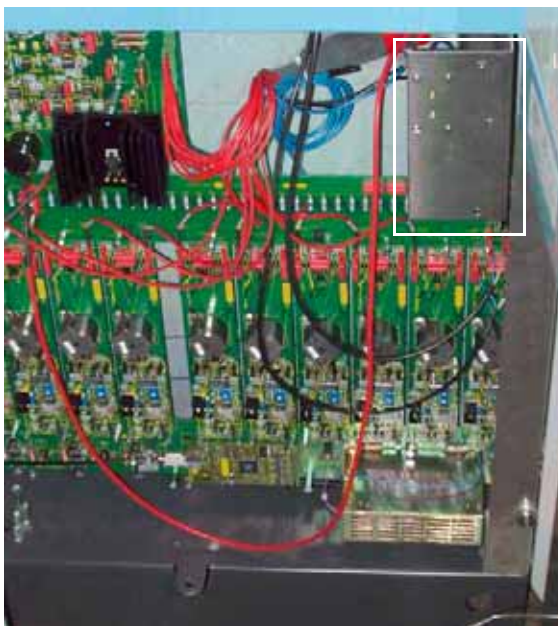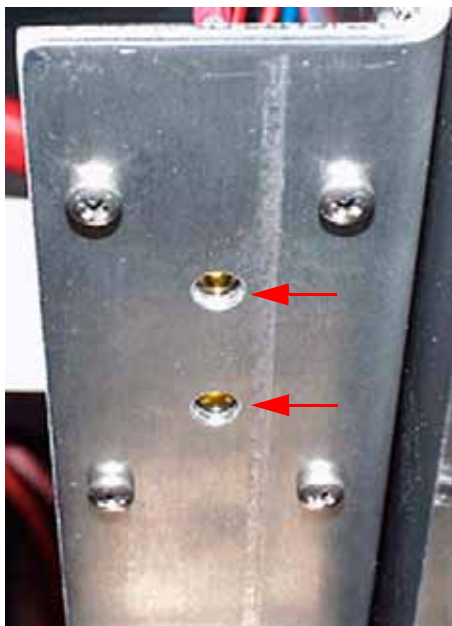
Heater of Changeover Valve and/or Needle Valve

Switch on heater of changeover valve and/or heater of needle valve (in case of Continuous Flow applications). This helps to prevent water condensation.
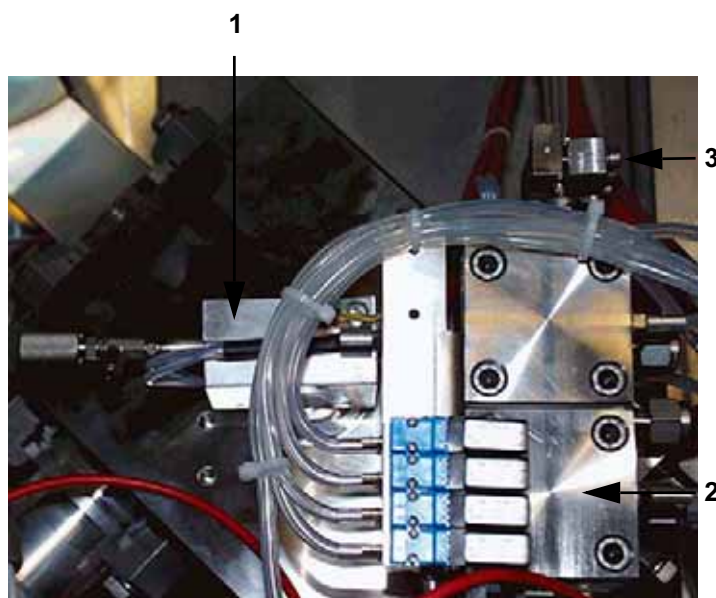
1. needle valve including needle valve heater

2. Changeover valve (COV) including changeover valve heater

3. device for crimp adjustment
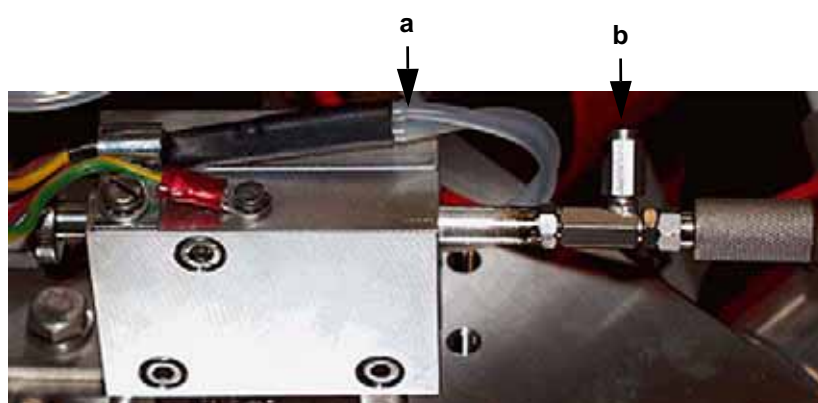
a. to needle valve heater

b. port for capillary

Figure 1-2.   needle valve including needle valve heater (side view)

device for crimp adjustment

needle valve
(frontal view)

to Changeover valve heater
(inside metal block)

to needle valve heater
(inside metal block)
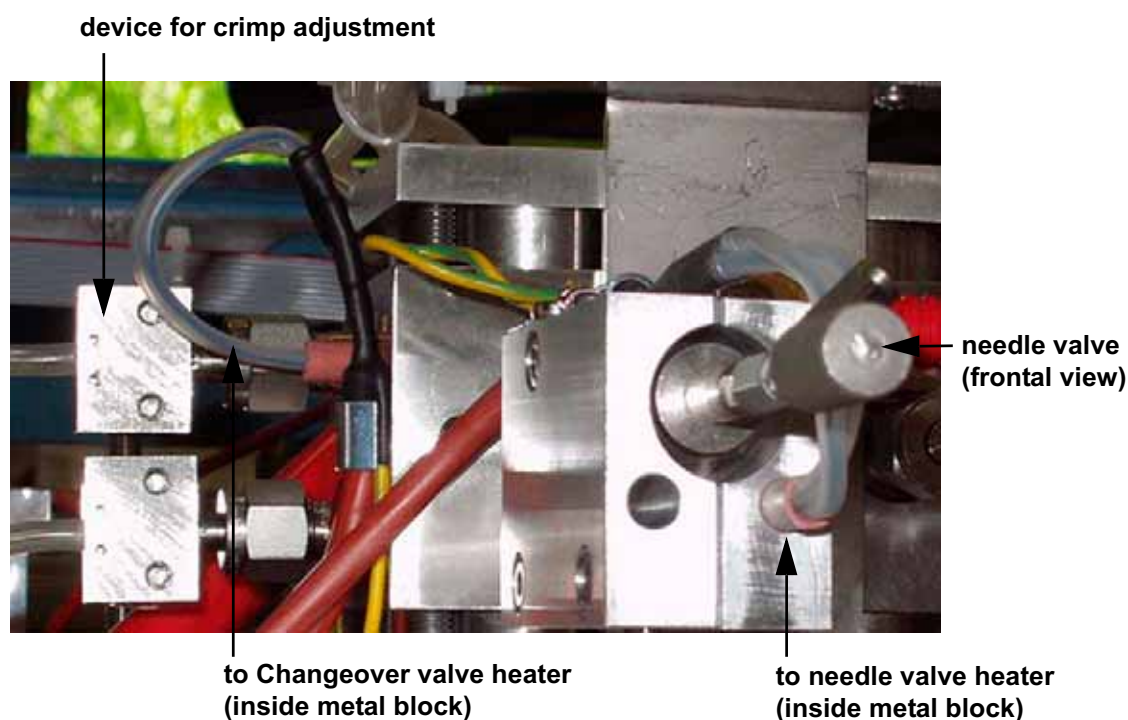
IRMS - Computer Connection

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

fiber line interface

fiber line
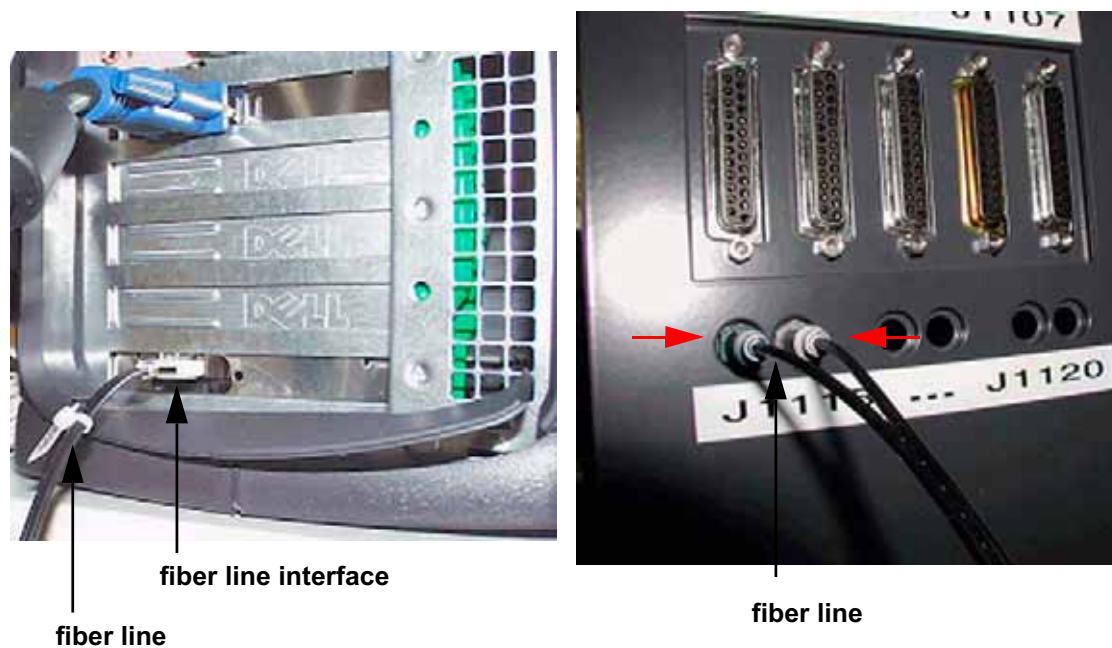
fiber line

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

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

   For detailed information, refer to the ISODAT NT Help System.

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

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

IRMS-Peripherals Connection

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

2. Peripherals are identified automatically by a plug and measure concept (see topic **The Plug and Measure Concept** on page 4-8).

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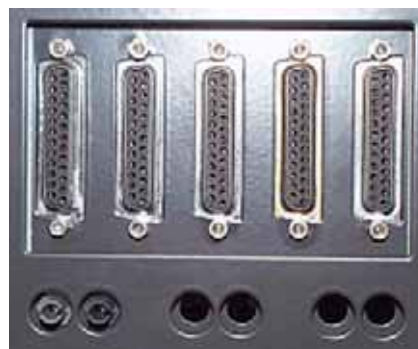
Starting Configurator

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

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

   When starting the Configurator the very first time (or after reset of the IRMS via **Reset Isotope MS** command in the Configurator's **Edit** menu), your type of IRMS is required. Then press **OK**.

2. Select your IRMS type.

3. Press **OK** to confirm.
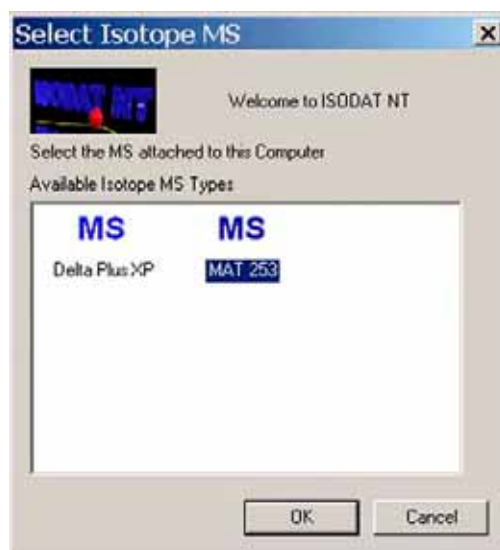
4. Check, whether the correct cups are installed.

5. Check, whether the correct cups are available for peak center.

6. Check, whether the resistor values are correct.

7. Confirm by **OK**.

Note. Up to eight channels can be used simultaneously!

---
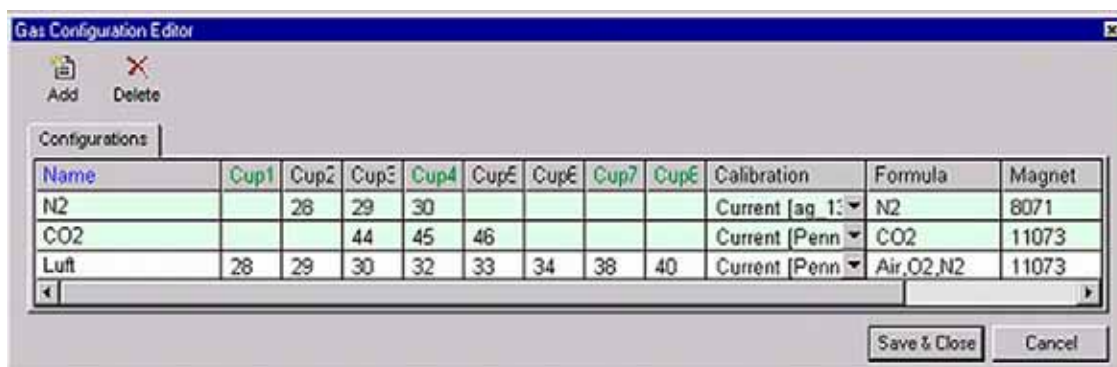
8. Check the available Gas Configurations. You can edit them by clicking on the fields (for detailed information refer to the ISODAT NT Help System).

9. Finally, press **Save & Close**.

10. Select one of the Configurations preset by Thermo Electron or add a new Configuration using the Add Configuration button.

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

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

   c. On the right pane, select Dual Inlet.

   d. Drag it to the Source port of the new Configuration.
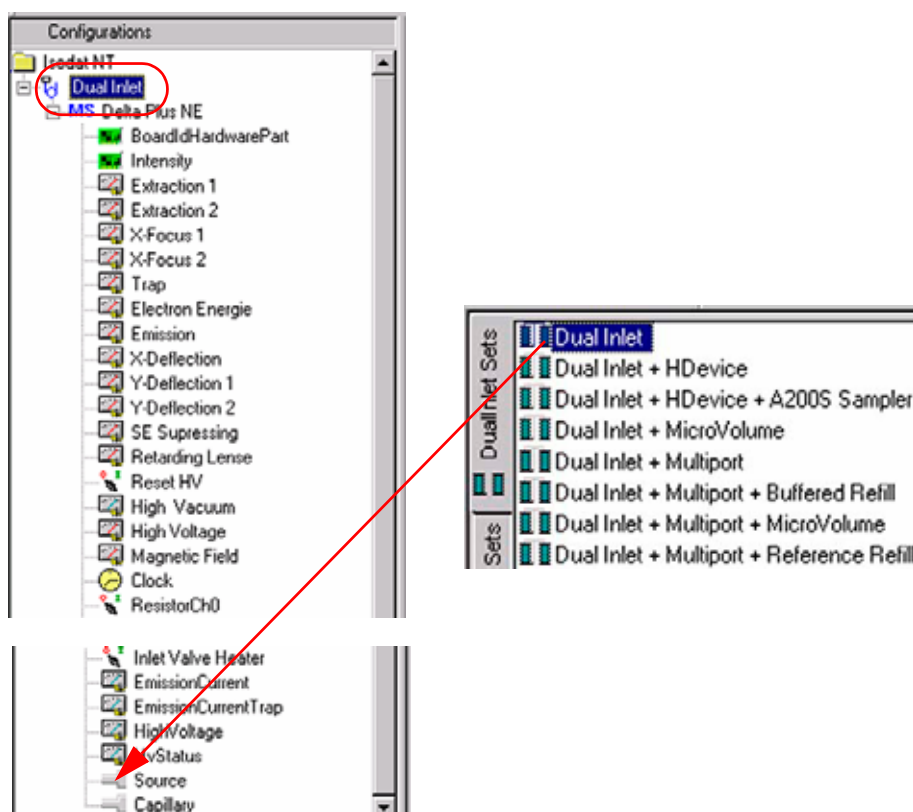
   e. A ⊞ sign appears:

   The Dual Inlet device has been attached to the Source.

f. Close the Configurator window.

All settings will be saved automatically.
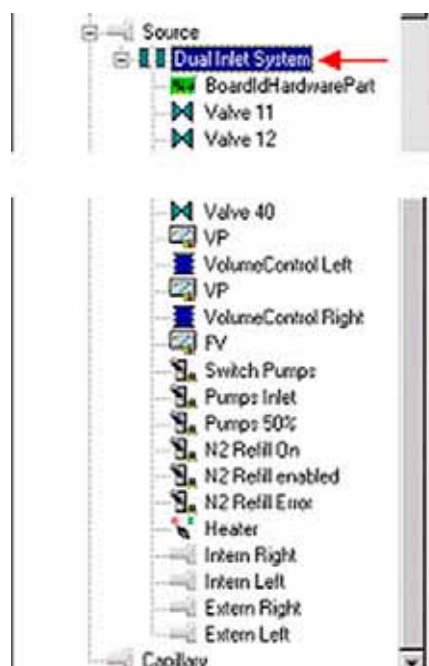
Starting Instrument Control

Instrument Control

1. Start Instrument Control by double-clicking the **Instrument Control** Icon. The Scan window appears.
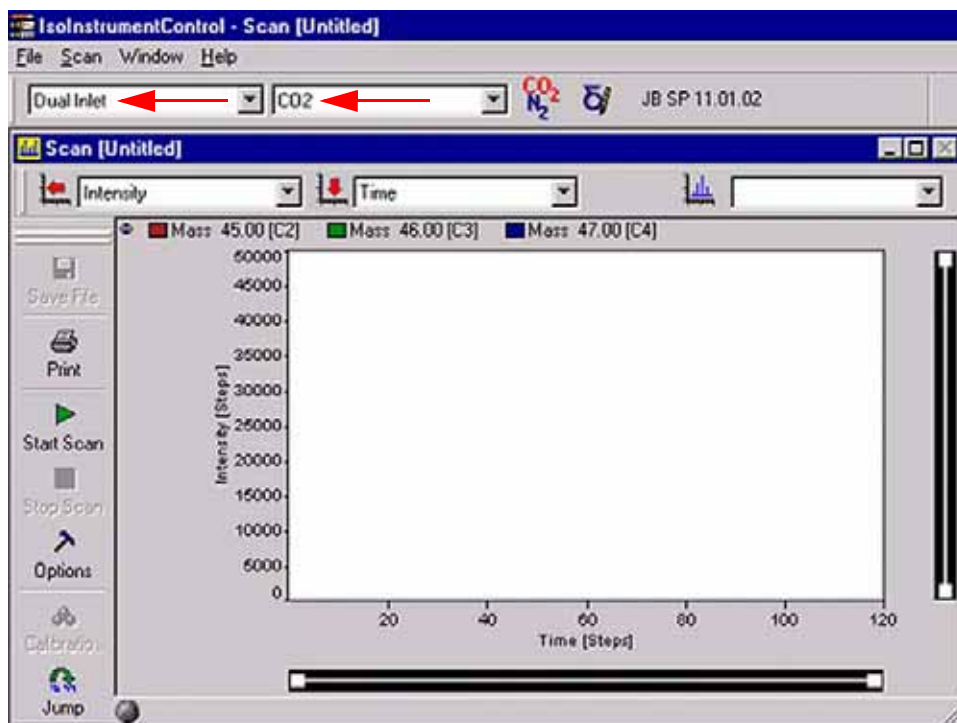
2. Select your Configuration, e.g. *Dual Inlet*.

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

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

5. In the Properties box, activate Configuration, Smart Isotope MS and
    Source Vacuum.
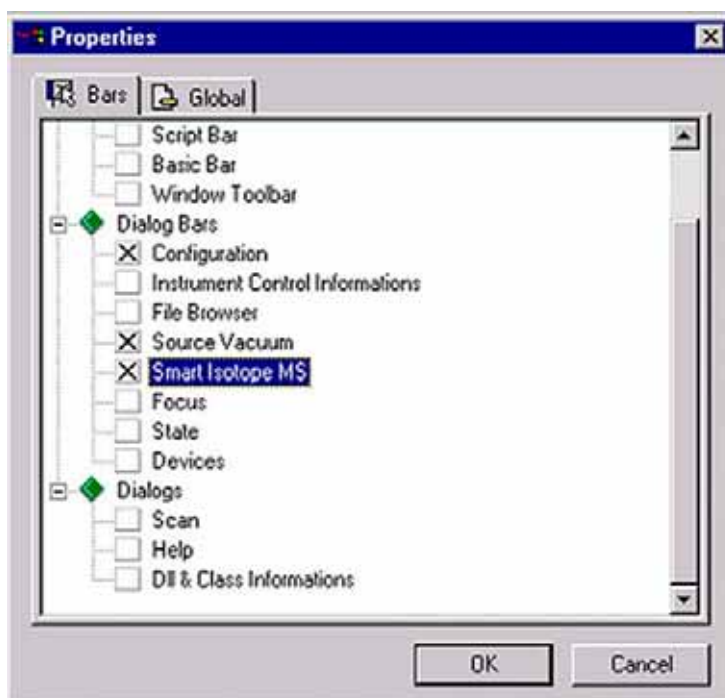
6. Confirm by **OK**.

7. Check the quality of the vacuum being established: the HV Source
    display reveals the current source vacuum in mbar.
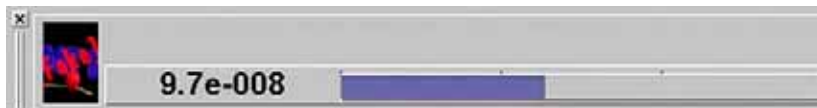
> **Note.** Before starting a measurement, after pumping the entire system for 15 min, a pressure between the lower 10^{-6} mbar and the upper 10^{-7} mbar range should be reached in the source.
>
> This pressure should even drop between the lower 10^{-7} mbar and the upper 10^{-8} mbar range as pumping continues for a longer time (if all valves are closed).
>
> If after 15 min of pumping a pressure of $1*10^{-5}$ mbar cannot be reached, probably a leak exists. As a safety measure, the cathode turns off at pressures above $3*10^{-5}$ mbar.

Evacuating the Dual Inlet System

Activate the Dual Inlet System window by clicking on **Window > Dual Inlet System**.
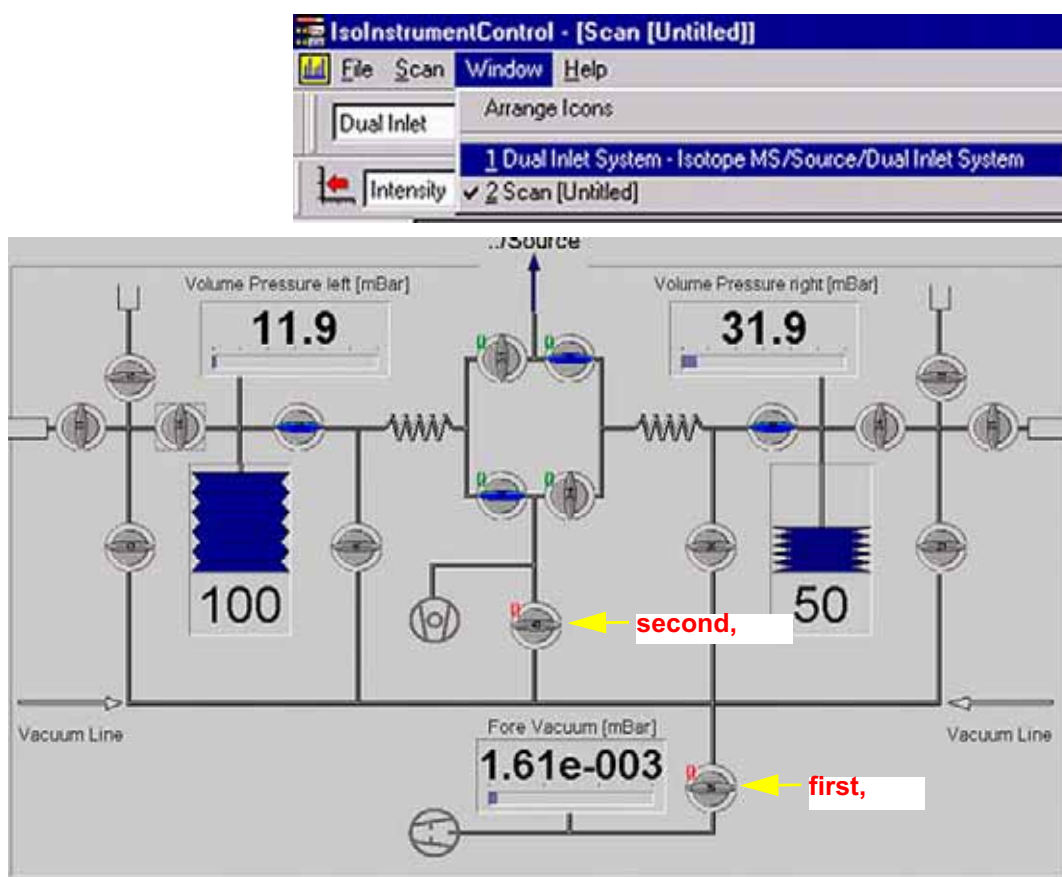
Figure 1-3.   Valves for Evacuation of Feed Lines

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

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

2. Open the respective valves and evacuate the feed lines between gas reservoir and IRMS (e.g. capillaries), which are still at atmospheric pressure. After evacuation close them again. See valves # 39 and # 40 in Figure 1-3.

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

4. Turn on the ion source via the icon at ISODAT NT's Smart Isotope MS bar
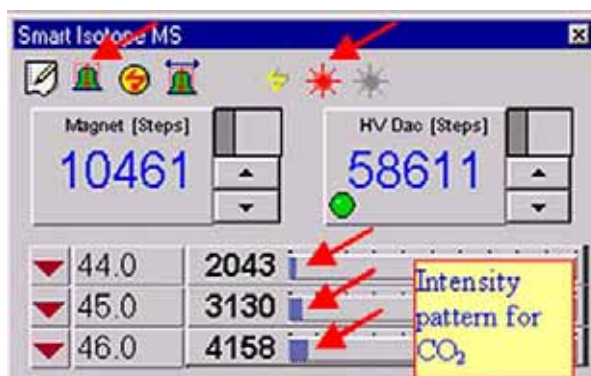
5. In case of a Dual Inlet System, open it at one side.

6. The intensity pattern characteristic for CO_2 is displayed at the Smart Isotope MS bar: the intensity for m/z 44 is smaller than the one on m/z 45, which in turn is smaller than the one on m/z 46.

7. If you got a signal on all cups, perform a peak center by clicking on the

    icon.

    Thus, the signal intensities should yield the pattern type shown above.

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

Principle of Peak Center

The magnet jumps to a BDAC value specific for the desired masses, that is m/z 44, m/z 45 and m/z 46 in case of CO_2. These values are stored and taken from the Gas Configuration, e.g. 10461 for CO_2 in the example shown above.

Peak center uses high voltage for fine adjustment to gain highest intensities in all cups.

> **Note.** If you run ISODAT NT for the first time, that is after installation, the software uses default values which may not correspond exactly to your system.

It is thus recommended that you perform a Mass Calibration. Peak Center parameters can be set by a right-click on the specific configuration in the Gas Configuration.

Mass Calibration

1. In Instrument Control's Scan Window select your (Hardware) Configuration, e.g. *Dual Inlet* in case of a Dual Inlet System.

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

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

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

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

   If not, press **No**. Let the reference gas flow into the source and start again.

   If the magnet is improperly positioned, the BDAC value is wrong (no intensity information).

5. At the Scan Basic box, select 2000 steps and 12000 steps as a range for the Magnet Scan.
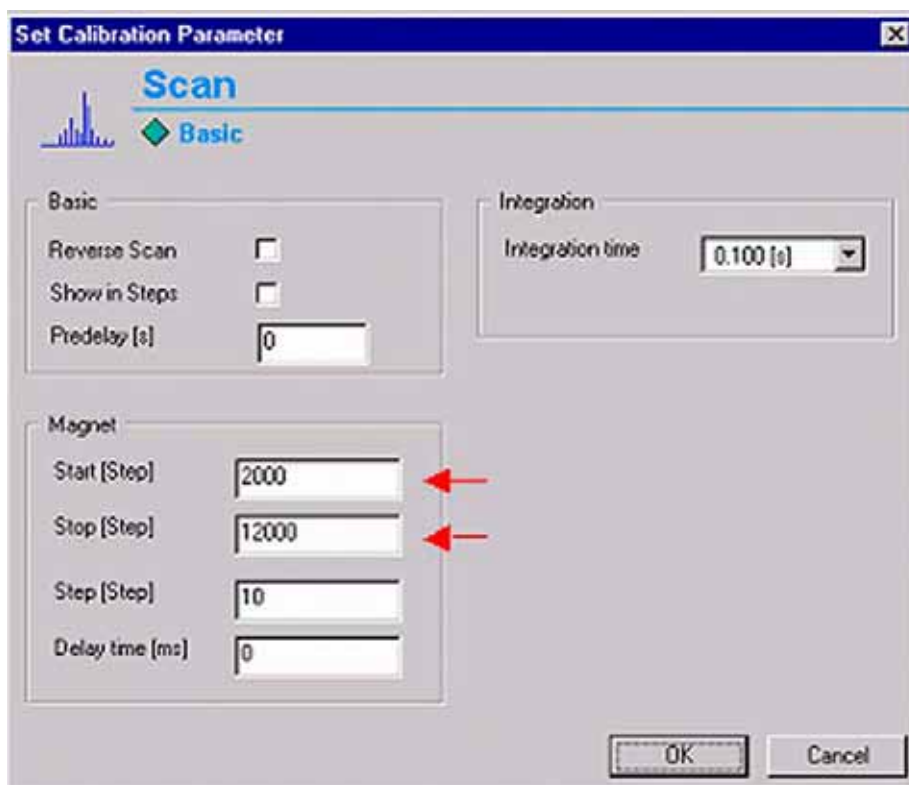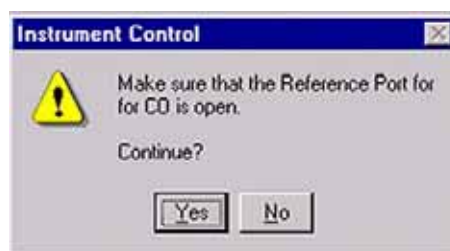
6. Accept the other default values.

7. Finally, press **OK**.

Principle: The m/z 12, m/z 28 and m/z 44 will be searched. A proposal about their position is then offered. On demand, additional masses can be inserted into the proposal list. If the positions don't match satisfactorily, they are slightly moved later.
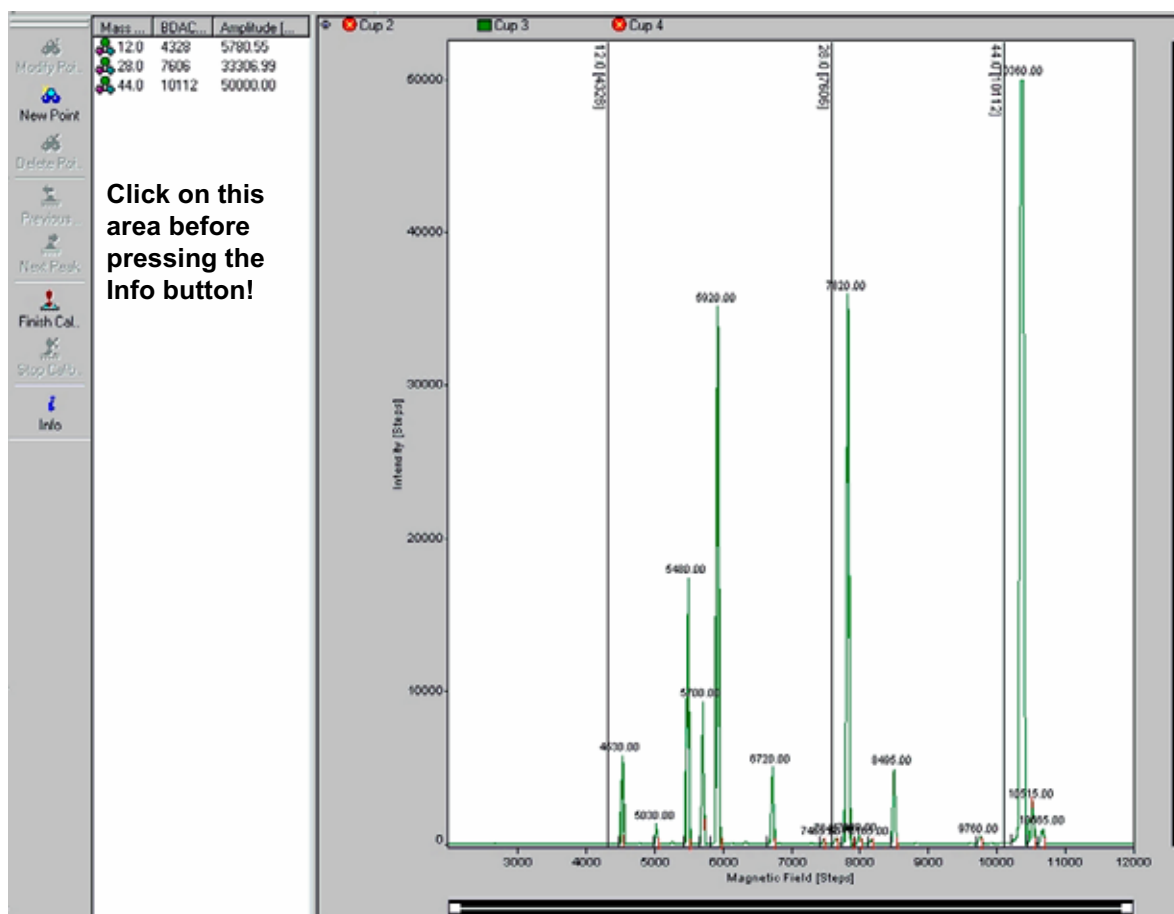
Sometimes, the proposals for the mass peak positions in the left pane are matching and sometimes they do not. During the very first Calibration, the proposals usually must be corrected: assign the calibration masses to their peaks by drag and drop of the vertical lines using your mouse, snapping the lines to the mass peaks.

During later Calibrations, the proposals mostly do not need to be corrected via drag and drop.

8.   Activate the area containing the mass proposals by clicking on it!

9.   Control whether you chose the correct masses using the **Info** button.
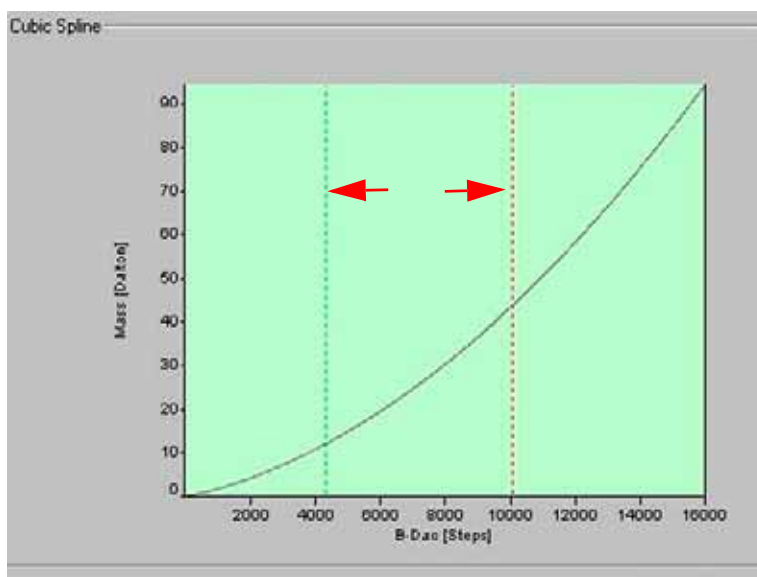
The curve should be approximately linear within the interval.

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

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

11. A peak center follows.

Crimp Adjustment

1. Open the entire Inlet System.

2. Let 50 mbar of CO_2 flow into each bellow ("equilibration").

3. Adjust the crimps for each inlet port until this bellow pressure leads to a signal of 5 V on both sides, that is standard side and sample side. About 10 mbar correspond to 1 V in case of CO_2.

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

Bellow Calibration

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

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

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

The Scan window appears.

Instrument
Control
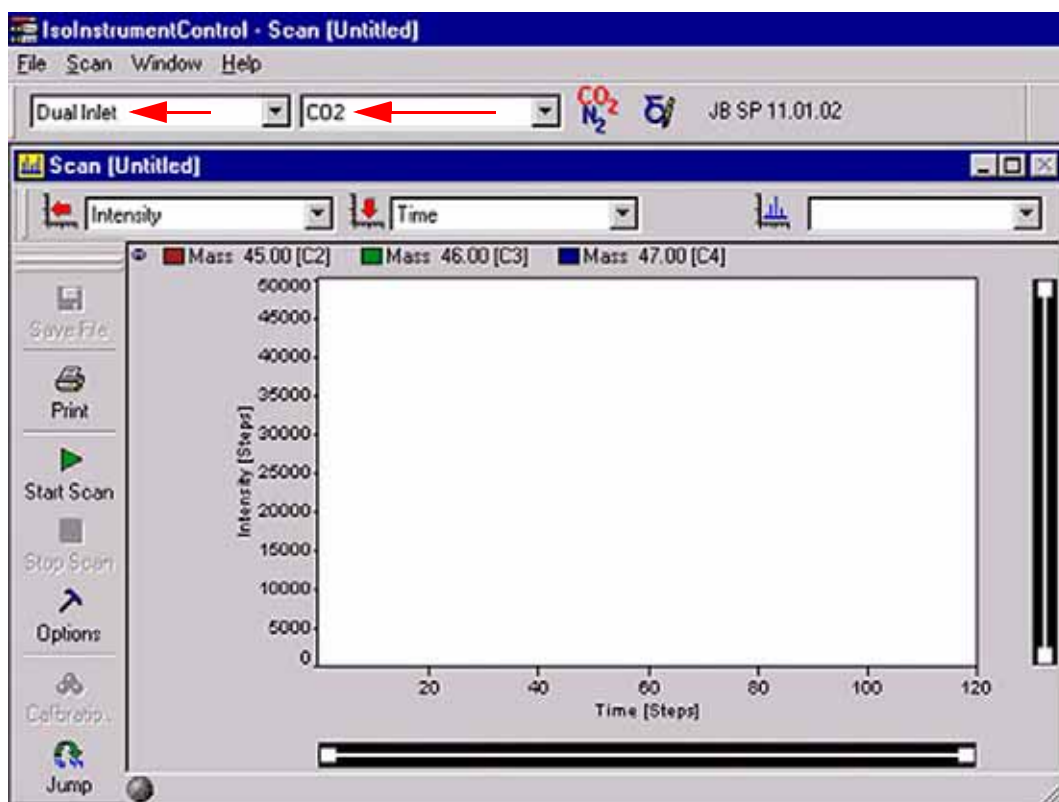
3. Select your Configuration, e.g. *Dual Inlet*.

4. Select your Gas Configuration, e.g. *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.
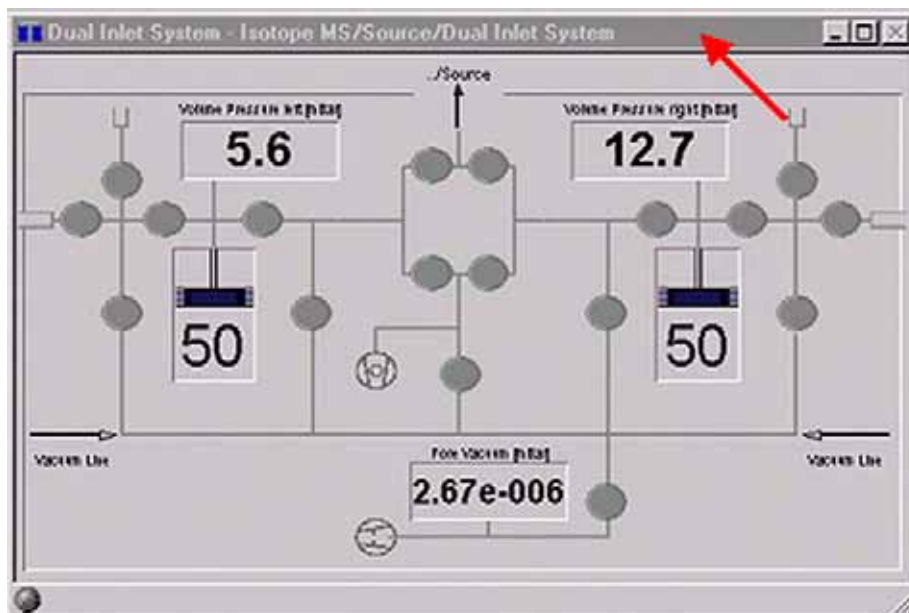
5. From the **Calibrate** Menu, select **Bellows**.

Bellow Calibration must be performed in two steps:

Hardware Calibration of Bellows

i. In Hardware Calibration, the bellows are opened to maximum, that is 100 % and closed again, that is 0 %.

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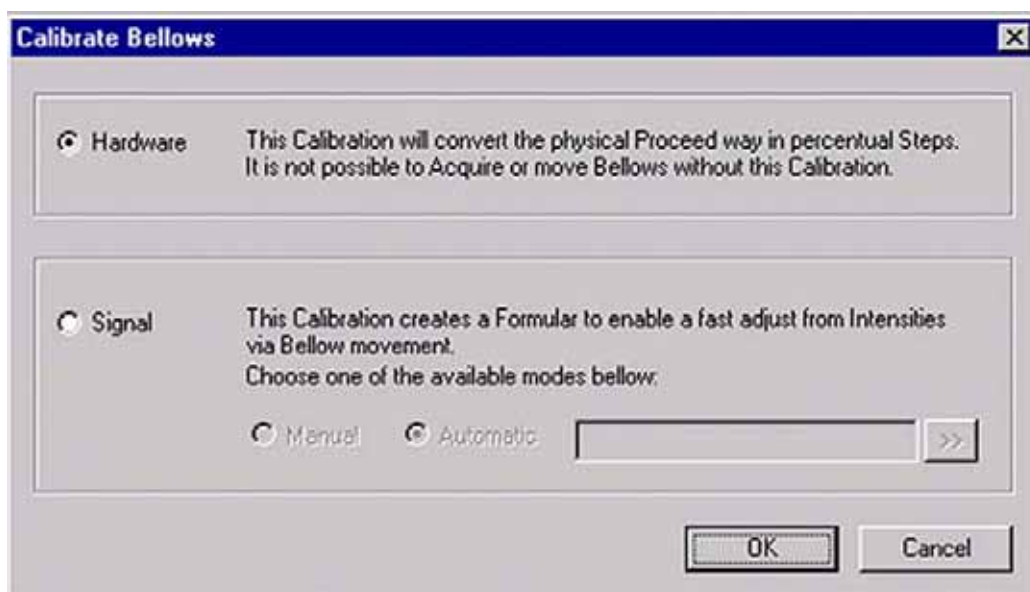
ii. Enable the Hardware radio button.

iii. Press **OK**.

> **Note.** A Hardware Calibration deletes a Signal Calibration. Therefore, Hardware Calibration must always be performed prior to Signal Calibration!
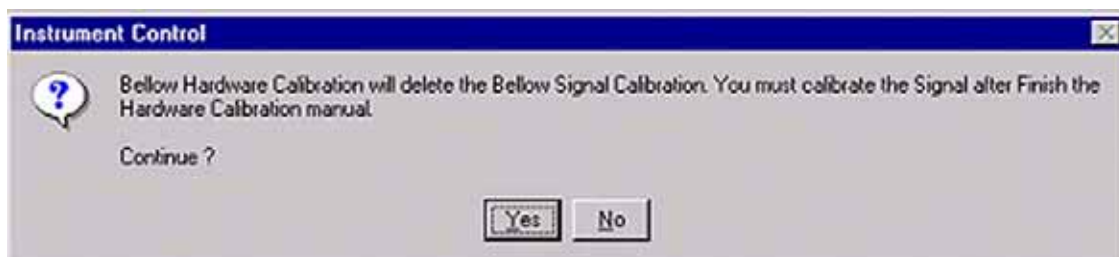
iv. Press **Yes** to continue.

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

Signal Calibration of Bellows

> **Note.** Signal Calibration requires a signal intensity of at least 3 V!

> **Note.** 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.

   vi. Activate the Dual Inlet System window again by a click on its title bar.

   vii. 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 10–20 mbar on each side. This gas amount is necessary to guarantee a certain signal intensity. If the gas amount should be either too small (e.g.< 10 mbar) or too large, warning messages will occur.
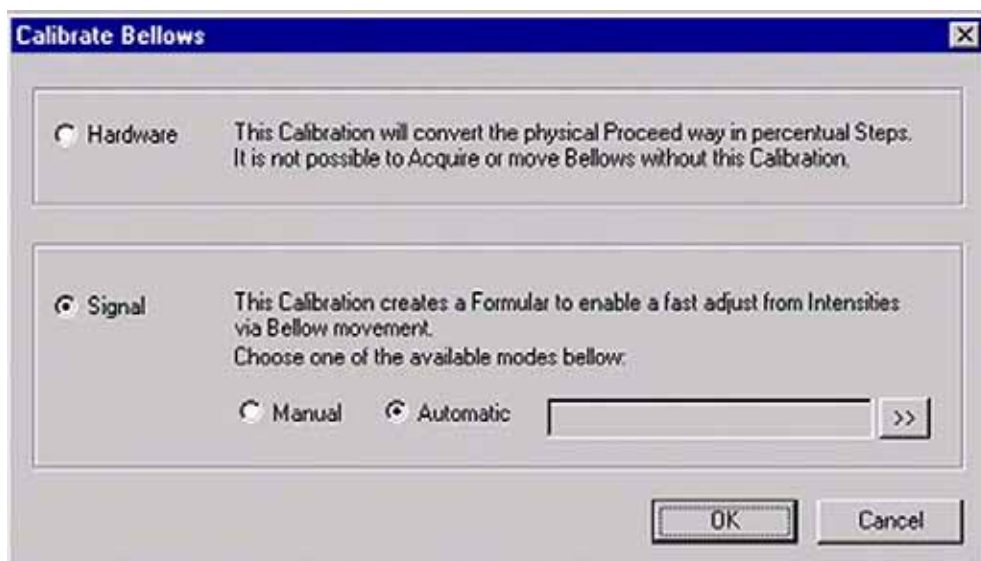
   viii. Enable the Signal radio button.

   ix. Select Automatic and press **OK**.

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

Focusing

The ion beam must now be focused (see **Principle of Focusing** on
page 1-43). Thermo Electron has already performed a focusing procedure
before your instrument was delivered. The obtained parameters, which can be
seen on Focus toolbar, have been assigned to the resp. Gas Configuration.
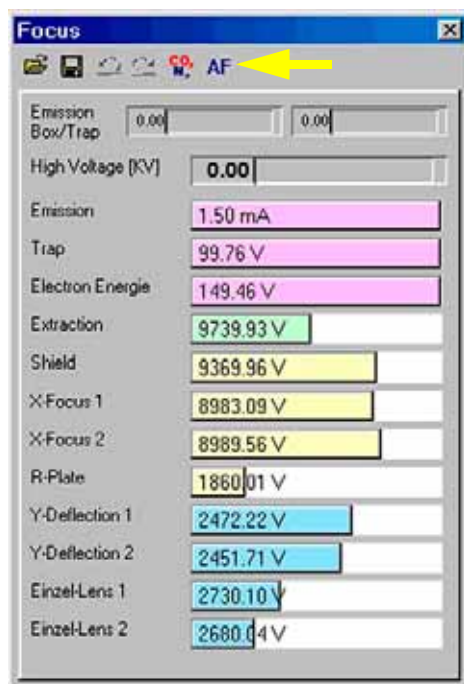
On top of the Focus window, notice
the **Focus toolbar** shown below.

It contains buttons which will be
described below.

Figure 1-4.   Focus window

Figure 1-5.   Focus toolbar

	Load	opens a "Focus Settings" document (*.fcs).
	Save	saves the actual Focus Settings as "Focus Settings" document (*.fcs).
	Undo	the last command is undone.
	Redo	the last command is redone.
	Pass to GC	attaches the current Focus Setting to the current Gas Configuration.

Reset HV switches on high voltage.

Source On turns on the entire source electronics.

Source Off turns off the entire source electronics.

Autofocus calls up an Autofocus Script.

If you changed parameters at the Focus window, or after you finished your

own focusings, you can assign them to the active Gas Configuration by .

You can save new parameters by to load them again later by .

> **Note.** For each Gas Configuration, a specific focus can be set and saved.

Focusing can now be started:

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

2. To optimize the focusing settings by yourself, start a Tune Scan, which
   continues during the whole focusing process.
   A Tune Scan reveals the course of intensity over time while changing the
   parameters in the Focus window: At the Scan window, select *Tune Scan*.
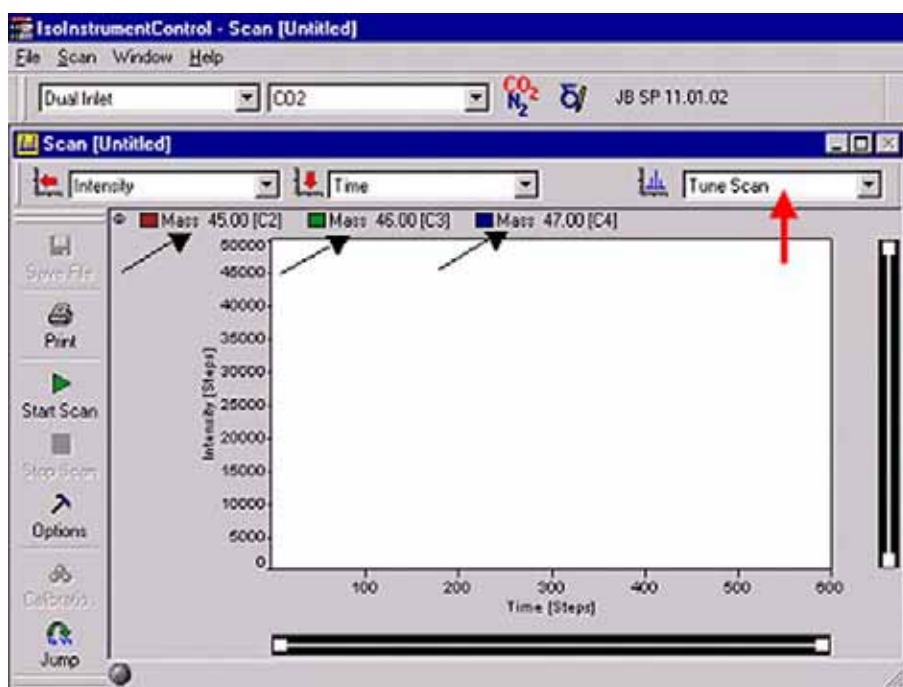
Figure 1-6. **Performing a Tune Scan**

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

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

Linearity Focusing

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

Sensitivity Focusing

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

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

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

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

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

Diagnosis

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

Starting a Measurement

After performing Diagnosis tests, you can start a measurement, that is a Dual Inlet Acquisition. For details, see **Chapter 6: Measurements**.

1.3   Getting Started: Software

This section contains the following topics:

• System Requirements

• Installing ISODAT NT

System Requirements

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

• Software requirements, and

• Hardware requirements.

Software Requirements

It is advantageous if your system meets the recommended requirements.

Table 1-1.   Software Requirements

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

Hardware Requirements

• COM Port Extension Board

• IEEE Interface Board

• Sound Card

• Joystick requirements

• GC and Autosampler requirements

For the installation of the drivers for the above listed hardware, refer to **Installation of Board Drivers** on page 8-5.

Installing ISODAT NT

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

> **Note.** Close all applications (especially older versions of ISODAT NT if already installed and running)!
>
> Otherwise, a message will indicate that Setup of ISODAT NT is not possible while older versions of ISODAT NT are running (see: how to shut down the system).

1. Open your CD drive and insert the ISODAT NT CD.

2. Close it.

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

Figure 1-7.   ISODAT NT startup screen

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

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

5. Click **Yes**.
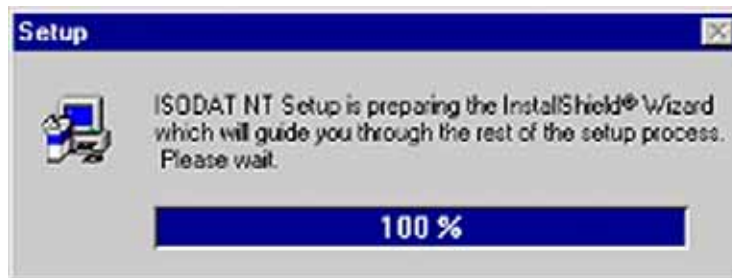
6. Wait while Setup is preparing the InstallShield® Wizard.

7. Respond with **Yes** (**No** will not create a backup).

    Make sure that you have enough free disk space for the backup that includes your Result files.

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

    • Each backup includes your Result files and is stored in a subdirectory of its own in the folder C:\Isodat_Backups.

    • Backups can be created and restored by using the Version Handler in the folder C:\Isodat_Backups.
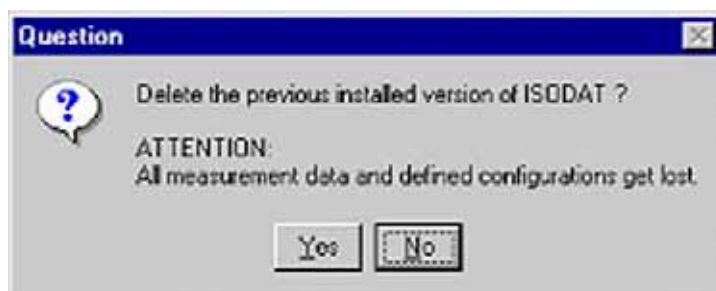
9. Respond with **Yes** to delete a previous version.

Yes leads to loss of Configurations and measurement data.

No will overwrite the previous version. Data, Configurations and Mass Calibrations will remain untouched.

Even though, it is recommended to delete old versions until unequivocal compatibility between the old and the new version is ensured.

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

Confirm again with **Yes** to delete.

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

12. Read the software license agreement carefully and click **Yes**.
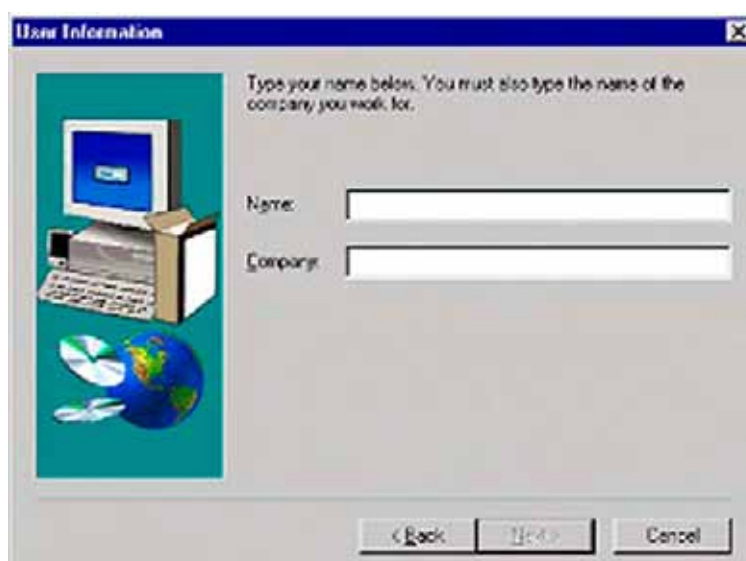
13. Fill in your name and company. Then click **Next**.

14. On the next window, you have to indicate whether you have interfaces installed.

    As default, both checkboxes are selected. Select the checkbox(es) that correspond to your equipment, and then press **Next**.

    • When you have ticked the checkbox Fiber Line Interface, proceed with step 18.

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

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

    Proceed with step 18.
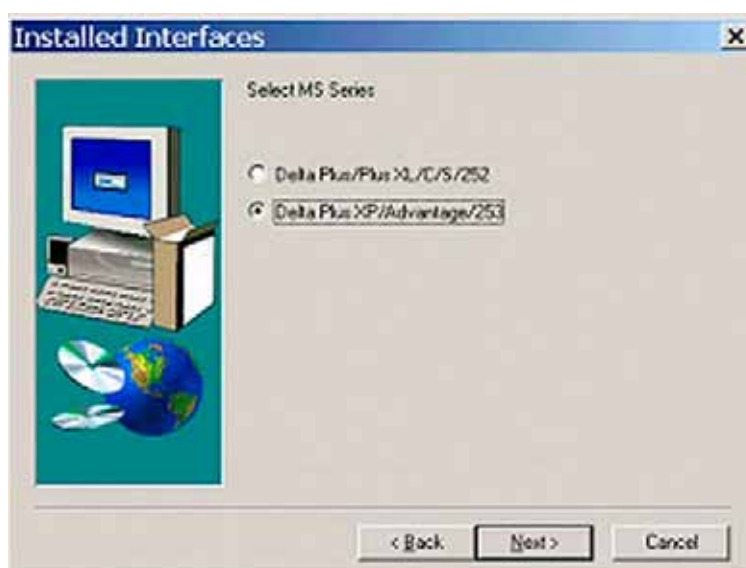
16. Select your MS Interface Board for IEEE connection to the IRMS (on the rear side of your computer). Click on **Next**.

    a. The "National Instruments" PC IIA (ISA) driver will automatically be installed.

    b. The "National Instruments" PCI-GPIB (PCI) driver must be installed separately later.

17. Having chosen
    PCI-GPIB (PCI) in
    step 16, you are
    reminded that the
    "National
    Instruments"
    PCI-GPIB driver
    must be installed
    separately later using the "National Instruments" CD.

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

    Press **OK**.

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

19. Wait while Setup is
    updating the registry.
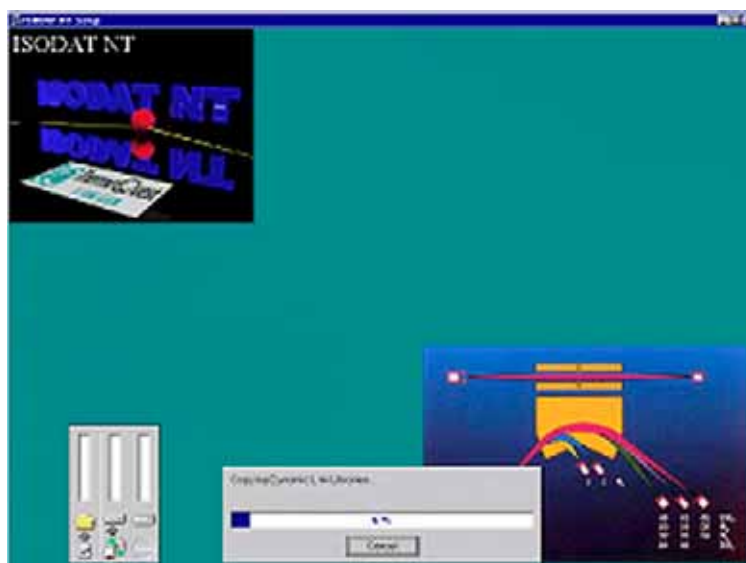
20. Wait while Setup is copying ISODAT NT onto your harddisk.

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

    Remove the CD.

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

1.4   Principle of Focusing

Reasons to Perform Focusing

Focusing needs to be performed

- to overcome the tiny remaining mechanical tolerances

- to overcome the gradual degradation of the ion source (e.g. due to metal deposition by sputtering)

- to overcome the different ion-optical properties of various gases (e.g. for H_2, a slightly different focusing compared to CO_2 yields better results).

- to optimize the system for various applications (e.g. reduce or increase sensitivity, optimize linearity)

How to Perform Focusing - (a) Autofocusing

Depending on your choice, all or only selected potentials might be varied.

For detailed information, refer to **Focusing of the Mass Spectrometer** on page 2-13.

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

    - Trap to maximum

    - Electron energy to maximum

    - Emission to maximum or lower

    - Extraction to about 2700 V for linearity focusing

2. Start Autofocusing by pressing the **AF** button.

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

How to Perform Focusing - (b) Manual Focusing

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

Use the **Undo** and **Redo** buttons on the Focus toolbar to correct inadvertently false edited parameters of the Focus window. As a parameter value increases, the colored part of its field grows.

The five following equivalent possibilities are available for data editing:

1. Click on the field to be edited. Then use the right or left cursor button to increase or decrease the value.
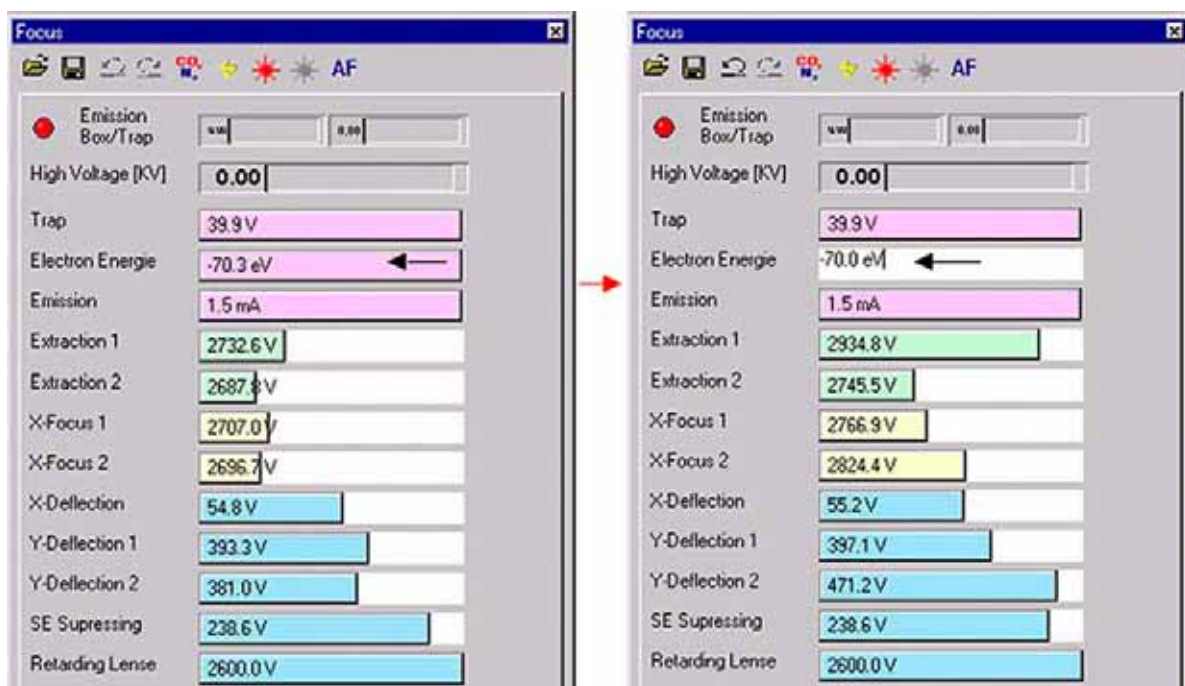
Figure 1-8.   Data editing

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

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

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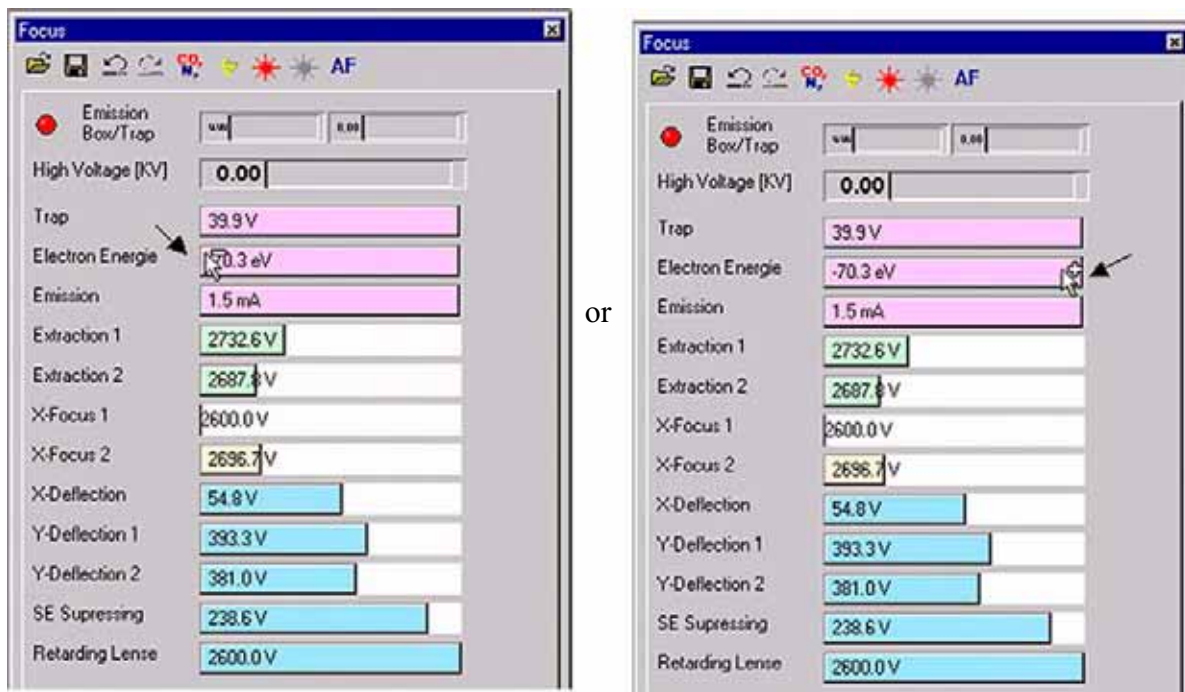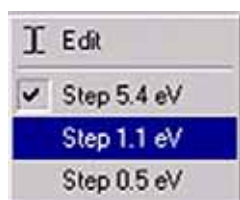
Clicking the right mouse button then allows selecting a step width.

The actual step width is marked.

4. Point to the field to be edited. A symbol consisting of two arrows occurs (one bottom-up and one top down). Click into the field. Then move your mouse to change the value. This procedure allows subtle corrections.
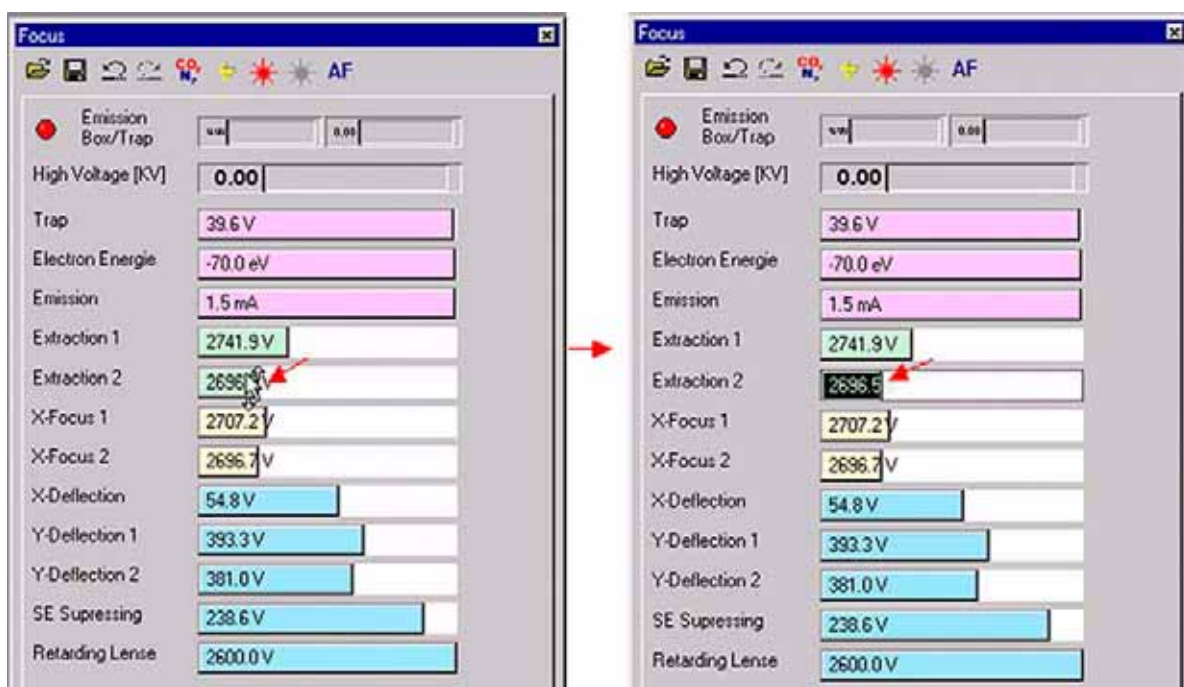
5. Point to the separation line between colored area and uncolored area of a field. An arrow occurs. Drag it to the right to increase or to the left to decrease the value. This procedure allows rough corrections.
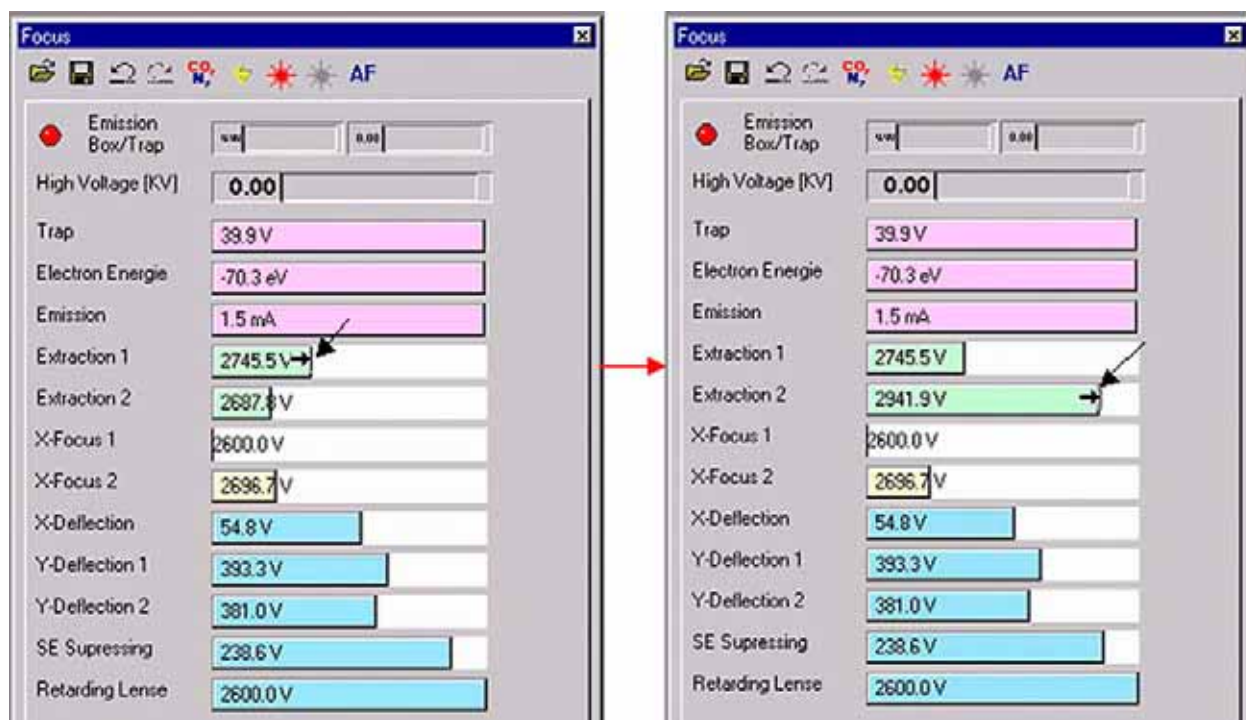
> **Note.** The two undermost parameters **Secondary Electron Suppressing** and **Retarding Lense** can only be edited via Manual Focusing (that is, not via Autofocusing).

Chapter 2
Analyzer

2.1   General

The gaseous sample to be analyzed is fed into the ion source via the inlet system. In the ion source, ions are generated in a high vacuum by the impact of electrons. The ions are then accelerated to energies of up to 3 keV and focused by electrostatic lenses to form a beam (See Figure 2-3 on page 2-6).

1 ion source housing
2 ion source flange
3 turbomolecular pump
4 high vacuum gauge
5 differential turbo pump
6 collector system housing
  (HD collector, triple collector)
7 amplifier housing
8 flight tube
9 electromagnet
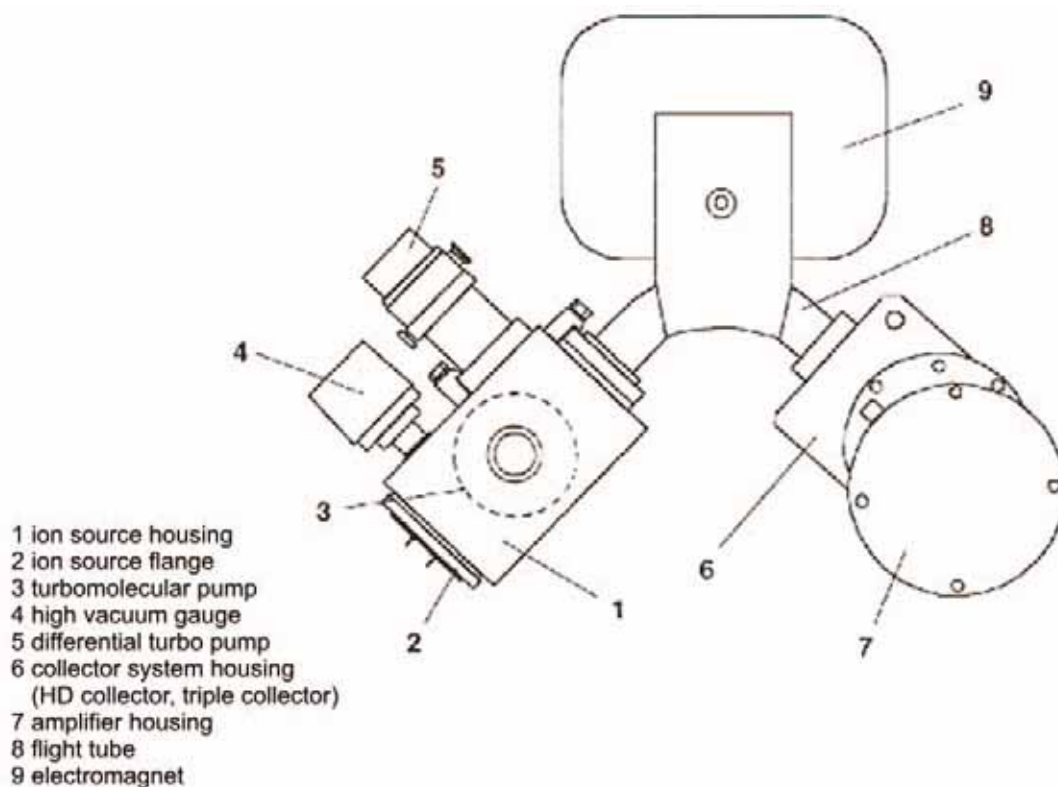
Figure 2-1.   Partial view of the instrument showing the arrangement of the analyzer

The ion beam exits the ion source into the magnetic field through a slit with a fixed width of 0.2 mm. It enters the magnetic field boundary at an angle of 26.5° and traverses the 90° magnetic sector field. Part of the ion beam exits at the same angle of 26.5° (see also Figure 2-2 on page 2-3).

Due to shaping and dimensions of the magnet, not only a focusing in X-direction but also in Y-direction is achieved. The refraction power (X-direction) is half as much as compared to the same magnet without shaping it. The focal length decreases and its value is the same for X- and Y-direction. Consequently, mass dispersion increases compared to the conventional arrangement, where the beam enters and exits the field normal to the boundaries. Thus, the 9 cm radius system has the same mass dispersion as the conventional 18 cm arrangement (where the beam enters and exits the field normal to the boundaries with a sector radius of 18 cm).
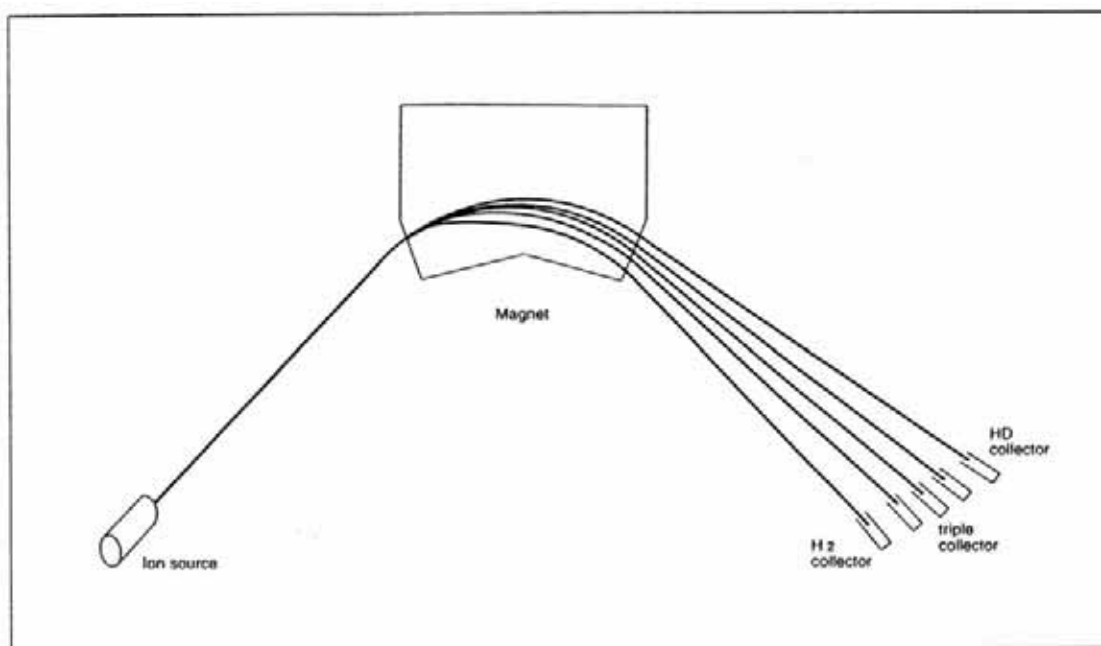
Figure 2-2. **Schematic of the ion path**

The magnetic sector field is generated by an electromagnet with a maximum field strength of 0.75 T. It covers a mass range up to m/z = 70 at full accelerating voltage.

The mass setting is achieved by variation of the magnetic field strength and/or of the accelerating voltage.

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

where z: elementary charge

$$k_M = \frac{r^2}{2 \times U}$$

r: nominal radius of ion path (r = 9 cm = const.)

U: accelerating voltage (not constant)

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

Example. For the special case of $U = 3$ kV, $k_M = 1.35*10^{-6}$ m^2/V results.

Usually, universal triple detectors are used (that is, standard case). The HD collectors are positioned in the housing of the collector systems. Their exit slit width is 2 mm. For the middle cup (that is, the narrow one), the resolution below results:

$$\frac{m}{\Delta m} = 95 \text{ (10\% valley)}$$

In the special case of C, N, O and S-collectors (in short CNOS collectors), MEMCO detectors are applied. Their collector slit width is 1.4 mm. Details on HD and CNOS collectors are given in topic **Ion Detection - Collector System** on page 2-17.

The electronic units supply the analyzer system operating voltages and control the analysis. They are described in detail in topic **Ion Source Control Unit** on page 2-8.

2.2 Ion Source

The ion source of the DELTA^{plus}XP is designed for high sensitivity (due to high gas density) and linearity. To ensure high sensitivity, the ion source is of gas tight design. The sample gas enters the ionization chamber through the changeover valve or the manually operated needle valve. It leaves it only via small apertures, which are required as a passage for the electron beam and the ions exiting into the analyzer.

The conductivity of these openings is much lower than the pumping speed of the vacuum pumps. Thus, the pressure within the ionization chamber is about 100 times higher than outside, which leads to high ion yields. The ions are generated in the source by electron impact ionization. The ionizing electrons are emitted by a thermionic cathode. The emission current is held constant by the emission regulator unit (see topic **Ion Source Control Unit** on page 2-8).

Two small permanent magnets are mounted to the ionization chamber (see Figure 2-3 on page 2-6), generating a magnetic field parallel to the electron beam. Due to the magnetic field, the electrons are moving along spiral trajectories, which increases their probability in the ionization volume. Thus, the probability to generate an ion with the aid of one single electron increases.

The energy of the ionizing electrons is determined by the potential difference between cathode and ionization chamber. It has a range between 70 and 124 eV. The electron beam leaves the ionization chamber via a small opening opposite to the cathode. It is collected in the electron trap, which is held on a positive potential relative to the ionization chamber.

Extraction plates accelerate ions out of the ionization chamber. The following lens system of different lenses focuses the ion beam onto the source slit. Mechanical tolerances might cause a slight out-of-axis deflection of the ion beam. Some of the system's lenses are half sections, which are insulated from each other. This construction allows for compensation by applying different potentials to the halves of the lenses. Figure 2-3 on page 2-6 shows schematically the lens arrangement and the potentials applied to them with an ion accelerating voltage set to 3 kV for example.
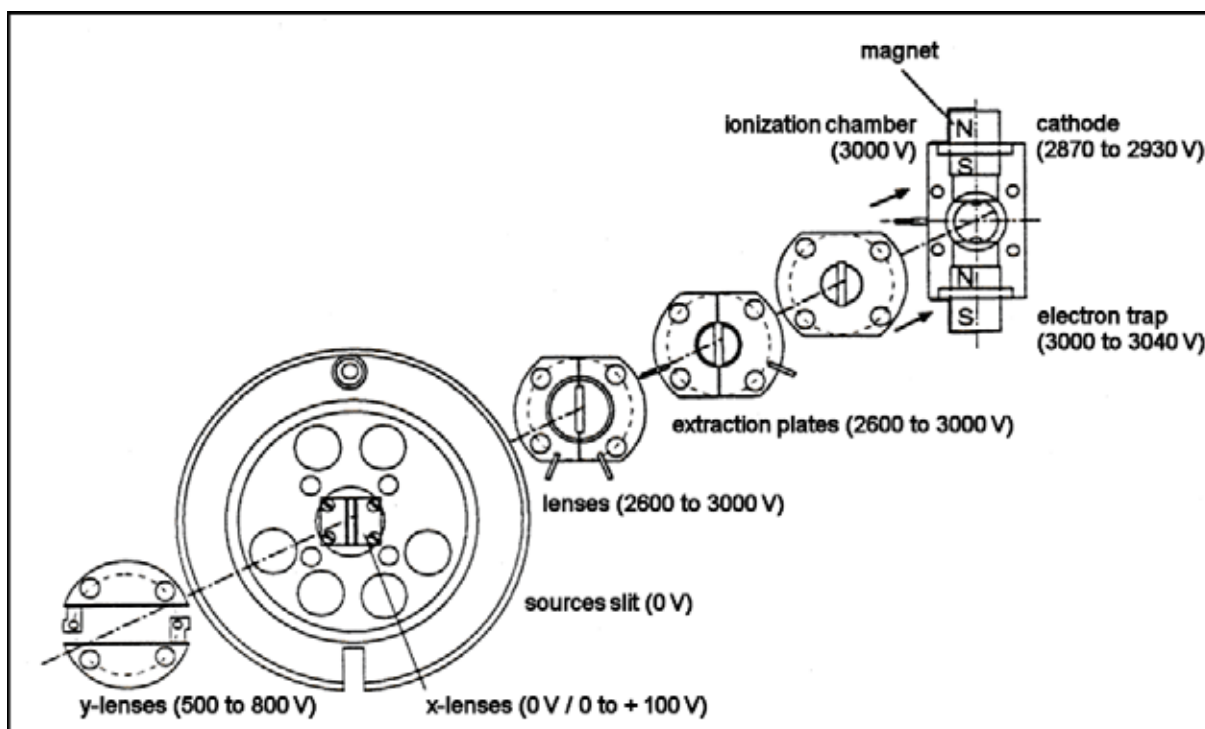
Figure 2-3.   Potentials (vs. ground) of the ion source at 3 kV ion acceleration voltage

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

Setting the accelerating voltage to lower values results in an enhancement of the mass range beyond 70. For example: a setting to

2.0 kV results in a mass range up to 105,

2.3 kV results in a mass range up to 90.

The ion source is mounted on the front flange for easy maintenance. A correct alignment of the ion source relative to the analyzer tube is achieved by a mating surface with the analyzer head. Details of the ion source, e.g. feedthroughs and correlation of lenses, are given in **Chapter 8: Technical Information**.
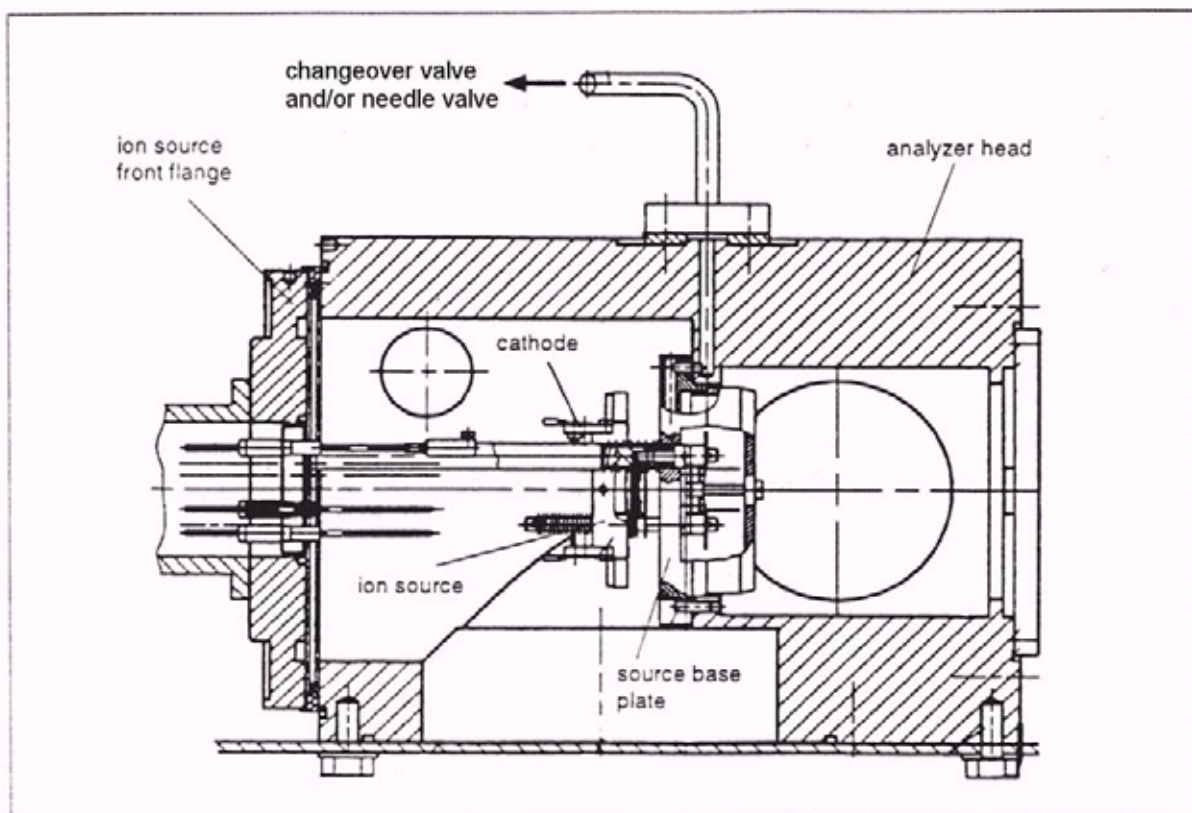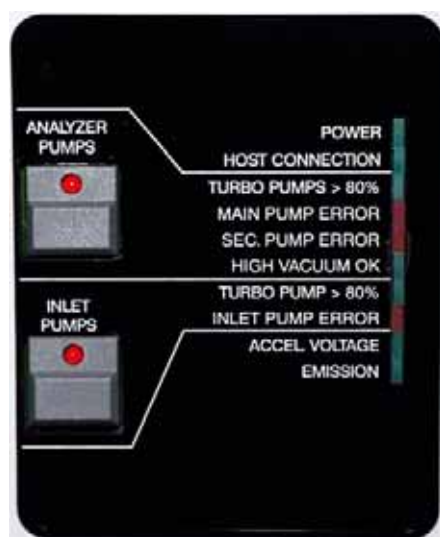
Figure 2-4.   Coupling of the inlet system to the ion source

2.3   Ion Source Control Unit

CAUTION!

> **Caution.** Opening the electronics cabinet is only allowed for maintenance purposes by qualified personnel.

Figure 2-5.   Control panel and main switch of the DELTA^{plus}XP

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

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

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Sec. Pump Error	turns on, if	an error at the differential pump has occurred.
High Vacuum OK	turns on, if	a pressure below 10^{-4} mbar has been reached.
		The penning can only start vacuum measurement below this pressure.

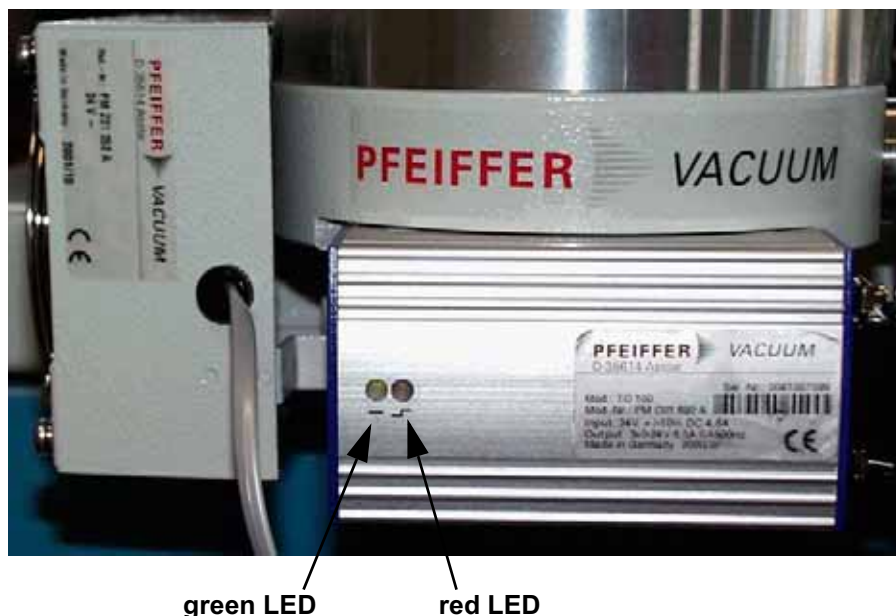green LED   red LED

Figure 2-6.   Main pump (i.e. source pump)

Figure 2-7.   Secondary pump (i.e. differential pump)

Inlet Pumps		Turbo pump of the Inlet System. It is connected to two fore vacuum pumps (e.g. #2 and #3).
Turbo Pump > 80 %	turns on, if	the turbo pump has reached 80% of its rotational speed.
Inlet Pump Error	turns on, if	an error at the inlet pumps has occurred.
Accel Voltage	turns on, if	the acceleration voltage for the ion source has been switched on.
Emission	turns on, if	the ion source has been switched on for emission.

Turbo Pump of the Inlet System →

The turbo pump of the Inlet System (only available in case of Dual Inlet) is located in the lower section of the cabinet's front panel under the bellows block.
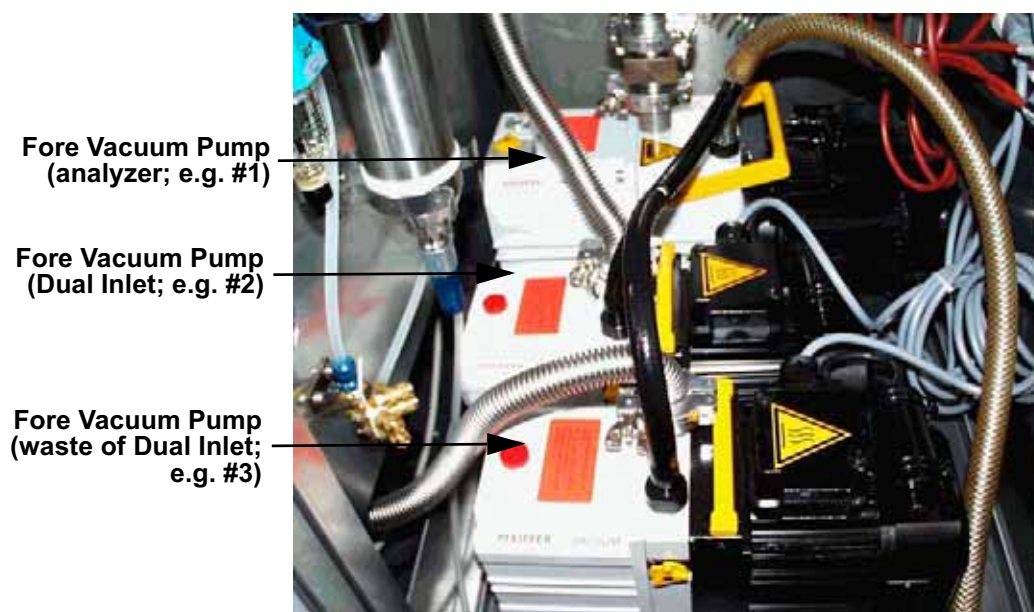
**Fore Vacuum Pump
(analyzer; e.g. #1)**

**Fore Vacuum Pump
(Dual Inlet; e.g. #2)**

**Fore Vacuum Pump
(waste of Dual Inlet;
e.g. #3)**

Figure 2-8.   Forevacuum pumps

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

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

In case of all three turbo pumps (that is turbo pump of the source, differential turbo pump, turbo pump of Inlet System; see LEDs in Figure 2-6 on page 2-9) notice:

- If a pump is off, the **green** LED blinks.

- If a pump is on, the **green** LED is turned on permanently. No information is revealed about the rotational speed of the pump.

- If a pump did not start up, the **red** LED is turned on permanently.

Emission Regulator

The emission regulator controls the current, which heats the cathode of the ion source and is switched on via ISODAT NT. The ionizing electron current, i.e. emission current, can be selected via ISODAT NT (maximum value: 1.5 mA). The cathode heating current is about 3.5 A. If the emission regulator is activated, the LED Emission on the front panel of the instrument turns on.

High Voltage Supply

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

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

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

- switched on the ion source (button ![laser icon] on the Focus bar in ISODAT NT),

- switched on Tune Scan (menu **Scan > Tune**) in ISODAT NT

- admitted gas into the inlet system and a sufficient amount of gas to the ion source. (See also **Chapter 5: Dual Inlet System**).
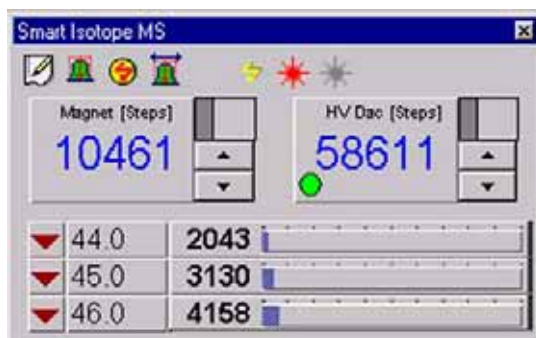

Figure 2-9.   Smart Isotope MS bar

Focusing of the ion beam is performed via ISODAT NT.

Focusing of the Mass Spectrometer

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

> **Note.** If you are not experienced with focusing we recommend using the autofocus.

## Detailed Description of Manual Focusing

A first type of manual focusing, intensity focusing (also called peak shape focusing or sensitivity focusing) is seldom performed. Sensitivity and peak shape are both improved, when the extraction voltage (i.e. Extraction 1 and Extraction 2) is decreased.

In the vast majority of cases however, the so-called linearity focusing is performed as one particular type of manual focusing. It requires relatively high extraction voltages (that is high values of Extraction 1 and Extraction 2) in order to extract the ions out of the ionization housing. It will be described below.

1. Basic Adjustment of Parameters

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

    a. Set Trap to 40 V.

    b. Set Electron Energy to the maximum value.

    c. Set Emission to 1 mA (i.e. to 50% below the maximum value of 1.5 mA).

    d. Set Extraction to a middle position (e.g. to 2600 V).

    e. Set X-Focus 1 and X-Focus 2 to a value smaller than middle position.

    f. Set X-Deflection to a middle position.

    g. Set Y-Deflection 1 and Y-Deflection 2 to a middle position.
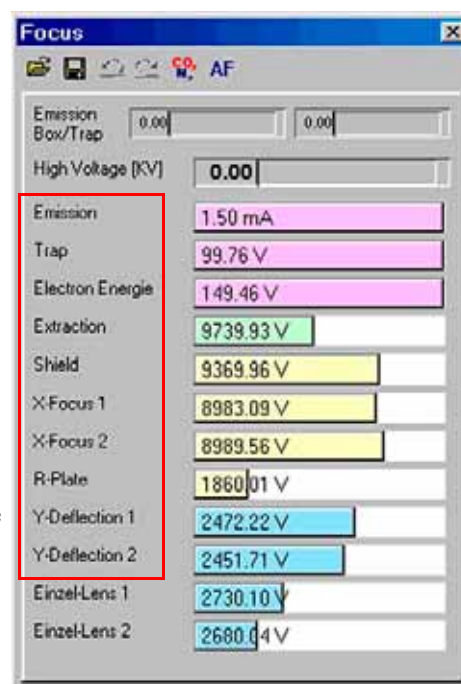
> **Note.** Do not change the parameters SE Suppressing and Retarding Lense!
> They are both preset: SE Suppressing is preset to about 240 V, whereas
> Retarding Lense is preset to about 2550 V.

> **Note.** In case of doubt, it is recommended to accept the default parameter
> values preset by Thermo Electron's Final Test group, if you perform manual
> focusing!

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

   a. Be sure that gas is flowing into the source (e.g. CO_2)

   b. If the mass scale is already calibrated, jump to mass (i.e. 45 on middle cup of universal triple cup). Perform a Peak center. If signal is too low to proceed (below 50 mV), jump to other mass (i.e. 44 on middle cup of universal triple collector cup)

   c. If no Mass Calibration is available, perform a Mass Scan. Then seek a peak for ion source optimization. Afterwards, perform a Mass Scan again. Finally, perform a Mass Calibration.

   d. Switch on Tune Scan in ISODAT NT's Instrument Control Mode.

3. Focusing

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

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

   - If the signal exceeds 50 V, either

     - decrease the gas flow into the IRMS or

     - use another mass (e.g. m/z 44 instead of m/z 45).

a **Intensity** focusing:

1. Set Extraction voltage to 50 V below acceleration voltage.

2. Perform a peak center.

3. Maximize signal using Trap Voltage and Electron Energy.

Linearity focusing:

1. Set Extraction voltage to 300 V below acceleration voltage.

2. Perform a peak center.

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

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

> **Note.** In the lens system, proceed outwards starting from the inside (according to Figure 2-3 on page 2-6).

b Maximize the signal using extraction lens and X-focusing voltage.

c Adjust X-Focus lenses to maximum signal intensity.

d Maximize signal using Trap voltage and Electron Energy.

e Adjust Y-Focus lenses to maximum signal intensity.

f Adjust X-deflection to maximum signal intensity.

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

h Try to increase the intensity further by slightly adjusting the extraction voltage

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

2.4　　Ion Deflection

Electromagnet

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

Magnet Current Regulator

The magnet current regulator provides the current required to generate the electromagnetic field. For further information (e.g. the schematics of the signal path regulating the magnet current), refer to **Chapter 4: Electronic Devices**. The relationship between magnet current and mass number is determined and stored by means of the mass calibration procedure. For details, see ISODAT NT Operating Manual.

2.5   Ion Detection - Collector System

General

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

The collector system is installed in the collector system housing (see Figure 2-1 on page 2-2, pos. 6). For HD isotope analysis, an optional collector system with two Faraday collector cups and amplifiers is available. The HD collector system is also installed in the collector system housing. Each collector cup has its own amplifier and the feedback resistor of the amplifier can be matched to the abundance of the isotope to be collected in this cup (see Table 2-1 on page 2-19). Each collector cup and its amplifier are connected to a voltage-to-frequency converter (VFC). The amplifier and the VFC are located on the preamplifier board. There are three or six VFCs for the MEMCO or universal triple collector system and two VFCs for the HD collector system available. They are allotted to one of the eight counters, so forming a measuring channel as shown in Figure 2-10.
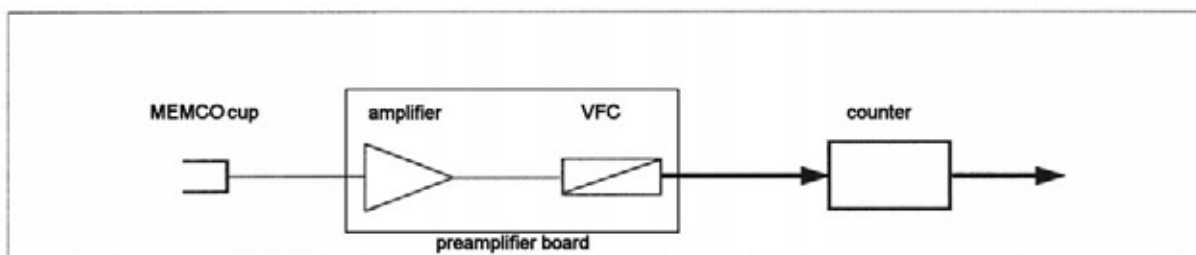

Figure 2-10.   Components forming a measuring channel

The converters transform the analog ion current signals into pulses. These pulses are fed to counters for a preselected integration time. At the end of each integration interval, the computer reads the number of counts and calculates the ion current ratios, see Figure 2-11.
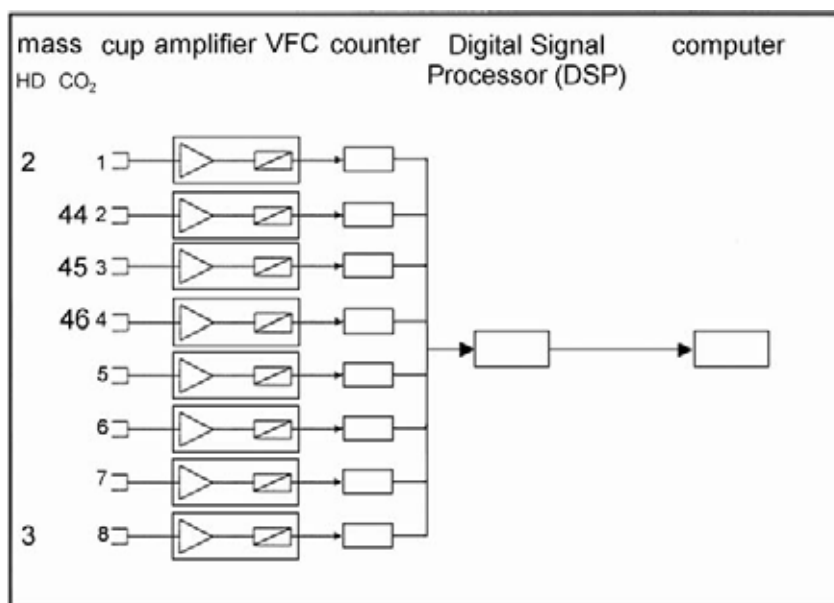
Figure 2-11.   Function schematic of the ion detection system

Amplifier

The DC amplifiers have 100 % inverse feedback. Their output voltage (50 V maximum) is the product of the input current and a feedback resistor. The feedback resistor has to match the abundance of the isotope to be collected in the respective collector cup. Table 2-1 on page 2-19 shows the resistance values to be used for the isotopes of the different gases.

> **Note.** Use ISODAT NT's Smart Isotope MS bar shown below to switch between resistors: a click on ▼ (that is, low resistance) changes the symbol to ▲ (that is, high resistance) and vice versa.
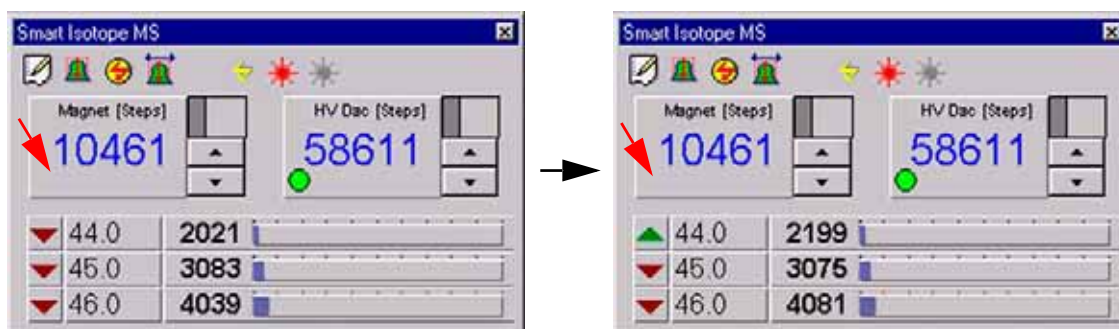
Figure 2-12.   Switching between resistors

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

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

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

CAUTION!

Caution. Never touch the surface of the high ohmic switchable resistors! Already a slight touch of a fingertip would contaminate the resistors resulting in signal instability. If you nevertheless accidentally once touched them, clean them using alcohol.

Collector Systems

The collector systems cover the mass range from 10 to 70 m/z at 3 kV accelerating voltage, allowing a resolution of m/Δm = 95 (10 % valley). Owing to high dispersion of the analyzer system, distance between collectors is extremely large (e.g. approximately 4 mm between m/z 44 and m/z 45). It was possible to design the Faraday collectors as deep, shielded buckets with integrated secondary electron suppression shields. See Figure 2-13. As a result, effects are eliminated that might degrade the ion current measurement.
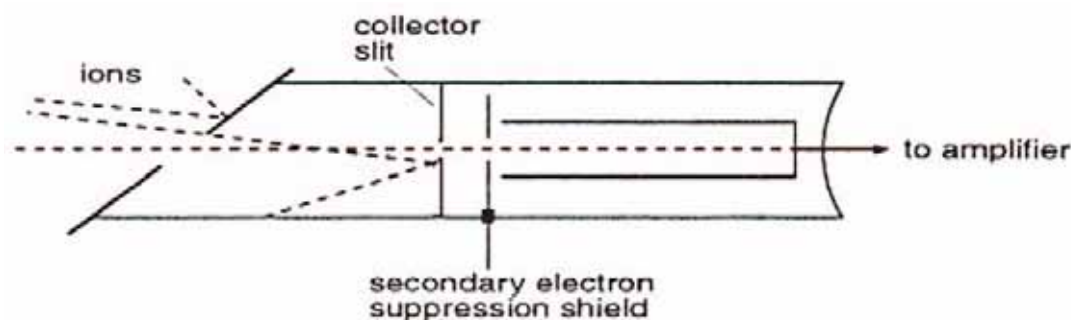
Figure 2-13.   Design of a Faraday collector cup

In most cases, universal triple collectors consisting of one narrow and two wide cups are used. On demand, the MEMCO collector system is delivered.

Universal Triple Collector System

The universal triple Faraday cup collector system is suitable for N_2, O_2, CO_2, and SO_2 measurement. This collector (see Figure 2-14 on page 2-20) consists of one small and two wide, individually shielded deep Faraday cups. The universal triple collector system can be accessed together with the optional HD collector system during one experiment (e.g. for background checks).
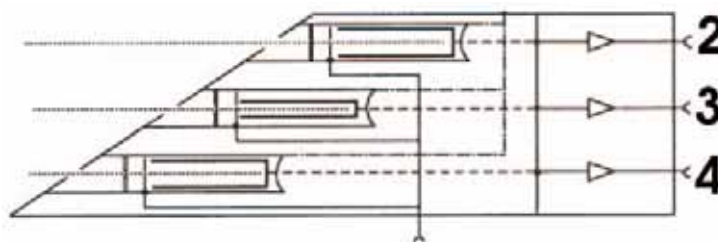
Figure 2-14.   Schematic of the universal triple system

> **Note.** Note the considerable advantage of the DELTA^{plus}XP: it offers switchable amplifiers.
>
> In case of switching from one gas to another, the resistors are changed and adjusted (including high voltage) via ISODAT NT. Refer to Table 2-2.

Table 2-2.   Cup assignment of the universal triple collector during measurements

	Cup 2	Cup 3	Cup 4
N_2	28	29	30
O_2	32	33	34

Table 2-2. **Cup assignment of the universal triple collector during measurements**

CO_2	44	45	46
SO_2	64	66	

MEMCO Collector System

The MEMCO collector system comprises three to six identical cups. The 3-cup version allows simultaneous measurement of two isotope ratios from the same sample, e.g. $^{13}C/^{12}C$ and $^{18}O/^{16}O$ of CO_2.

The 6-cup version permits the cup configuration to be preset for the consecutive measurement of different gases. As with the 3-cup version, simultaneous measurement of two isotope ratios of the same gas can be performed.

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

HD Collector System

The HD collector system is a dual Faraday collector assembly for hydrogen isotope measurement on a same ion path, operating in parallel to the MEMCO or universal triplet systems. The HD collector is located in the common collector housing. The collector cups are designed like those of the MEMCO system.

Chapter 3
Vacuum System

3.1 Pumping System

3.2 Vacuum System Control Unit

3.3 Turbomolecular Pump

3.1 Pumping System

Finnigan DELTA^{plus}XP is supplied with two optional pumping systems. The instrument is equipped either with or without a differential pumping system.

- The standard version comes with a turbomolecular pump to evacuate the analyzer system at a rate of 210 l/s (type: TMH 262, manufacturer: Pfeiffer). The required forevacuum is provided by a rotary pump rated at 5 m^3/h (type: DUO 5, manufacturer: Pfeiffer).

- The optional differential pumping system with an additional turbomolecular pump improves the vacuum in the analyzer system of the instrument. This improvement is required to reduce the high portion of He carrier gas of peripherals such as an elemental analyzer or a GC. The results are better abundance sensitivity, better peak shape and improved signal to background ratio at high ion source pressures.

  A vacuum lock or flow restriction separates the ion source section from the analyzer region.

  The additional turbomolecular pump evacuates the analyzer region at a rate of 60 l/s (type: TMH 071 P, manufacturer: Pfeiffer).

  The required forevacuum for both pumps is provided by a rotary pump rated at 5 m^3/h (type: DUO 5, manufacturer: Pfeiffer).

The inlet system is pre-evacuated by a forevacuum pump rated at 1.3 m^3/h (type: DUO 2.5, manufacturer: Pfeiffer).

The wasteline turbomolecular pump (type: TMH 071P) backed by a forevacuum pump (type: DUO 2.5) is used to provide high vacuum conditions in the inlet system.

For details see also topic **Turbomolecular Pump** on page 3-6.

3.2   Vacuum System Control Unit

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

Vacuum in Amplifier Housing

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

1. Turn control knob anti-clockwise from **0** to **FV**.

2. After approximately 30 min turn control knob clockwise from **FV** to **HV**. The housing is evacuated.

3. Before exchanging resistors on the amplifier, turn control knob clockwise from **HV** to **Vent**. After venting the housing, the cover can be removed.

4. In case of pump breakdown, restart turbo pump: press pump switch twice.
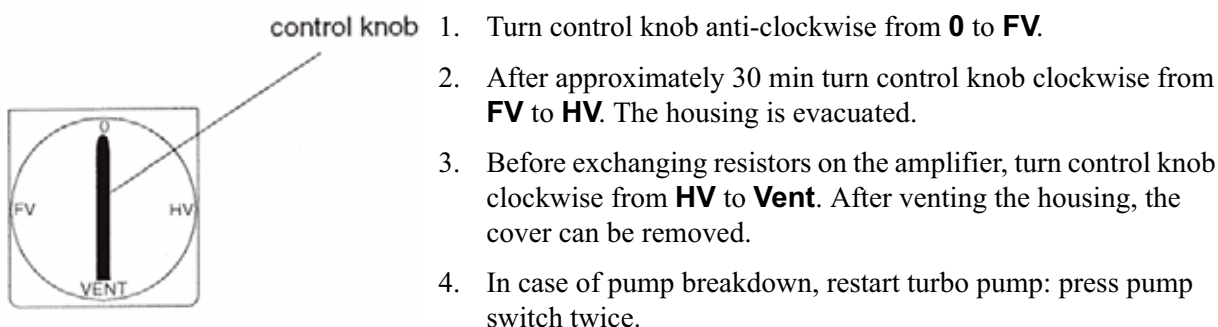
Figure 3-1.   3-port valve
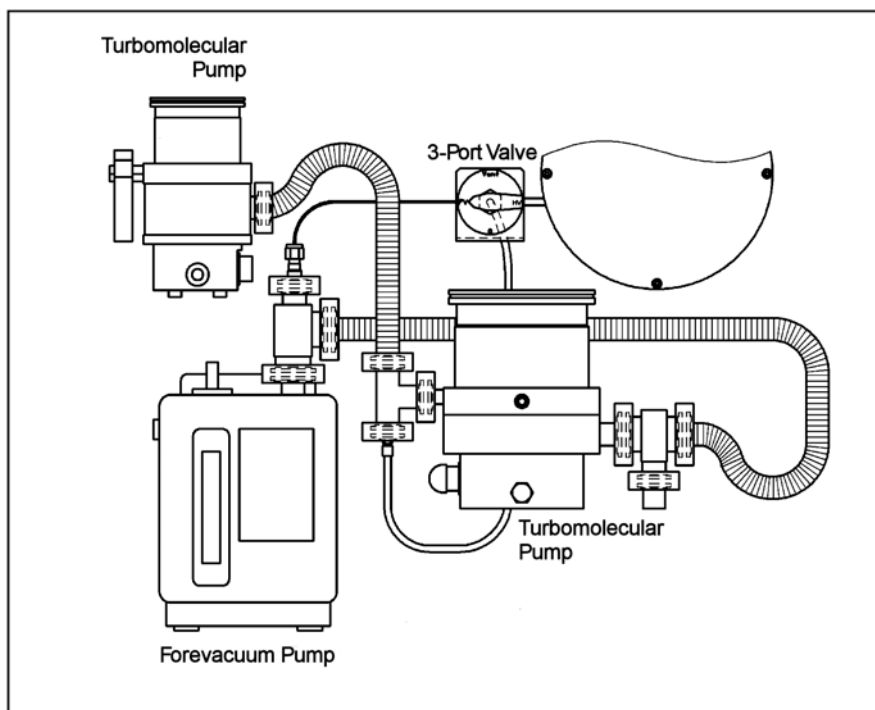
Figure 3-2.   Schematic view of the differential pumping system
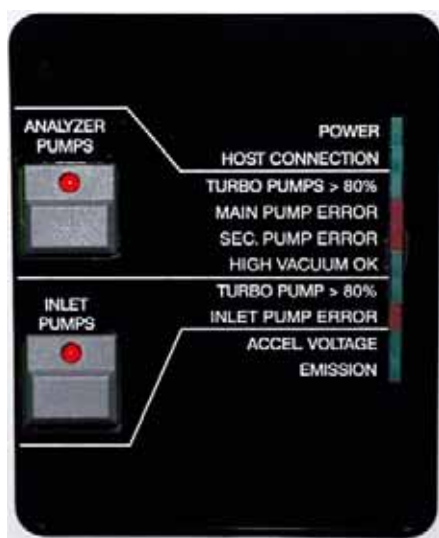
The row of LEDs is on when the turbomolecular pumps reach 50% of their nominal rotational speed.

The green LEDS indicate proper function of the respective system part.

The red LEDs indicate an error in the respective system part.

Figure 3-3.   Control Panel

Pushbuttons Switches

ANALYZER PUMPS   starts pumping system (turbomolecular pumps and forevacuum pump for analyzer system).

INLET PUMPS   starts the turbomolecular pump and the forevacuum pump for the wasteline and the inlet system.

> **Note.** In case of problems, e.g. a high vacuum $> 10^{-4}$ mbar, the source is cut off automatically.

LEDs

POWER   indicates the supply of main voltage

HOST CONNECTION   indicates that the DSP program located on the DSP/PCI card on the computer is started.

> **Note**. If the LED is not on with ISODAT NT started, no connection between the IRMS and the computer has been established.

ANALYZER PUMPS

TURBO PUMPS > 80%   indicates that the turbomolecular pumps of the analyzer have reached more than 80 % of their nominal rotational speed.

MAIN PUMP ERROR   indicates an error of the analyzer main pump.

SEC PUMP ERROR   indicates an error of the secondary analyzer turbomolecular pump.

> **Note.** In case the instrument is not equipped with a differential pumping system, this LED is not functional.

HIGH VACUUM OK   indicates that the vacuum has fallen below the set point of the Penning gauge.

> **Note**. The ion source can only be switched on when this LED is on.

INLET PUMPS

TURBO PUMP > 80%   indicates that the turbomolecular pump of the inlet system has reached more than 80 % of its nominal rotational speed.

INLET PUMP ERROR   indicates an error of the inlet turbomolecular pump.

ACCEL VOLTAGE   on when high voltage is provided to ion source.

EMISSION   only on when filament of the ion source operates.

> **Note.** If the vacuum system works properly the red LEDs are not on!

Guidance for Troubleshooting

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

HIGH VACUUM OK LED off:

(or too much background for m/z 28/40 is measured in the IRMS)

- There might be a leak in the system.
- There might be a malfunction of the Penning gauge.

ACCEL VOLTAGE LED on:

EMISSION LED off:

The cathode might be burned out. Checking and removal of the filament is described in detail in **Chapter 8: Technical Information**.

ACCEL VOLTAGE LED off:

EMISSION LED on:

High voltage is tripped when current exceeds 0.2 mA. This may be caused either by sparking or by a short circuit in the source or the connections to the source.

ACCEL VOLTAGE LED off:

EMISSION LED off:

The ion source is not switched on. Reset and switch on the ion source via software. When LED "HIGH VACUUM OK" is not on, see above.

3.3 Turbomolecular Pump

General

The turbomolecular pump works completely mechanically by rotor disks imparting momentum to the gas molecules. Baffles or cryogenic traps are not necessary for retention of pump fluid vapors.

The vacuum system is roughed by rotary pumps through the turbomolecular pumps. Furthermore, the turbomolecular pumps are started at atmospheric pressure. Hence, this arrangement also obviates the need for a high vacuum pump valve. Thus, the rated capacity of pump speed is available without restriction at the connecting flange of the source housing.

Because the molecular pump principle works in the molecular flow region only, the turbomolecular pumps require a forevacuum pump. This pump is roughing the vacuum system through the turbomolecular pump down to the upper limit of the turbomolecular pump operating range.

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

For details regarding function and design of the turbomolecular pumps, refer to the Operating Manual of the manufacturers.

Control Electronics and Power Supply

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

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Chapter 4
Electronic Devices

4.1 Overview of the Electronic Devices

Various electronic installations are required to carry out the procedures of Finnigan DELTA^{plus}XP. These comprise the following boards:

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

The electronics of DELTA^{plus}XP mass spectrometers contain complicated and numerous circuits. Therefore, only qualified and skilled electronics engineers should perform servicing.

It is recommended to call for the Thermo Electron Service if servicing is required. It is further recommended to use Thermo Electron spare parts only, since many parts are specially selected. When replacing fuses, only use the correct type.

Before calling the Thermo Electron Service, try to localize the defect. A precise description of the defect will ease the repair and reduce the costs.

> **Warning.** Be careful when removing the protective covers from plugs, cables and other parts.
>
> **DANGER! HIGH VOLTAGE!**

> **Warning.** To assure trouble-free operation of the IRMS, the maximum allowable voltage between neutral and ground must be less than 1 V!

Most of the electronic boards listed above are parts of the electronics cabinet that is located on the right side of the IRMS. This chapter describes the basic structure of the electronic equipment.

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Figure 4-1 shows the electronics cabinet of DELTA^{plus}XP.
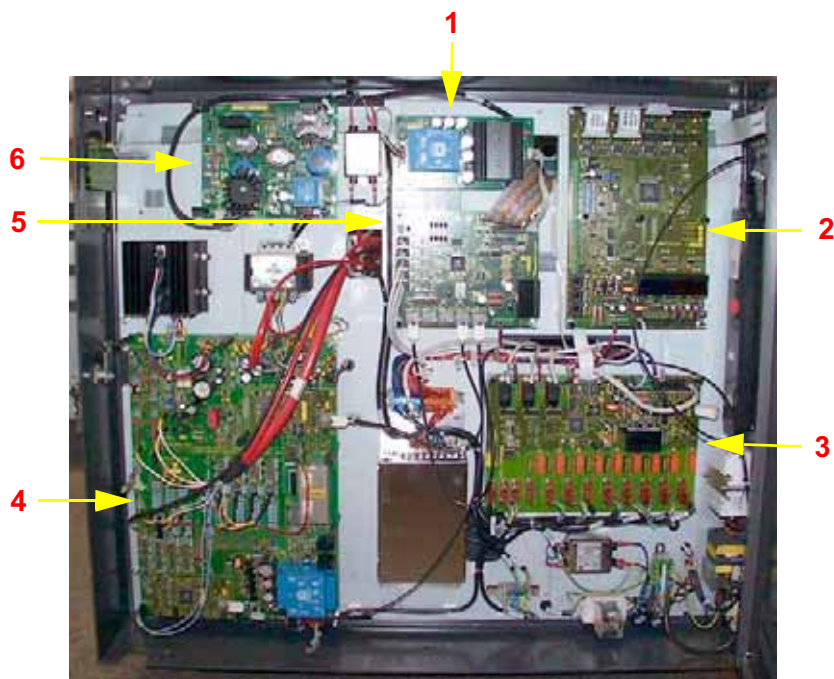
Figure 4-1.   Electronics cabinet

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

1   Power supply 55 V

2   Internal Inlet Controller

3   Power Distribution

4   Ion Source Controller

5   Data Logger

6   Magnet Current Regulator

> **Warning.** Opening the electronics cabinet is only allowed for maintenance purposes by qualified personal.

On the following pages are described selected boards that are located in the electronics cabinet as well as in other parts of DELTA^{plus}XP.

4.2   Power Distribution

The power distribution board (Part No. 204 1280) is located on the right side of the electronics cabinet.
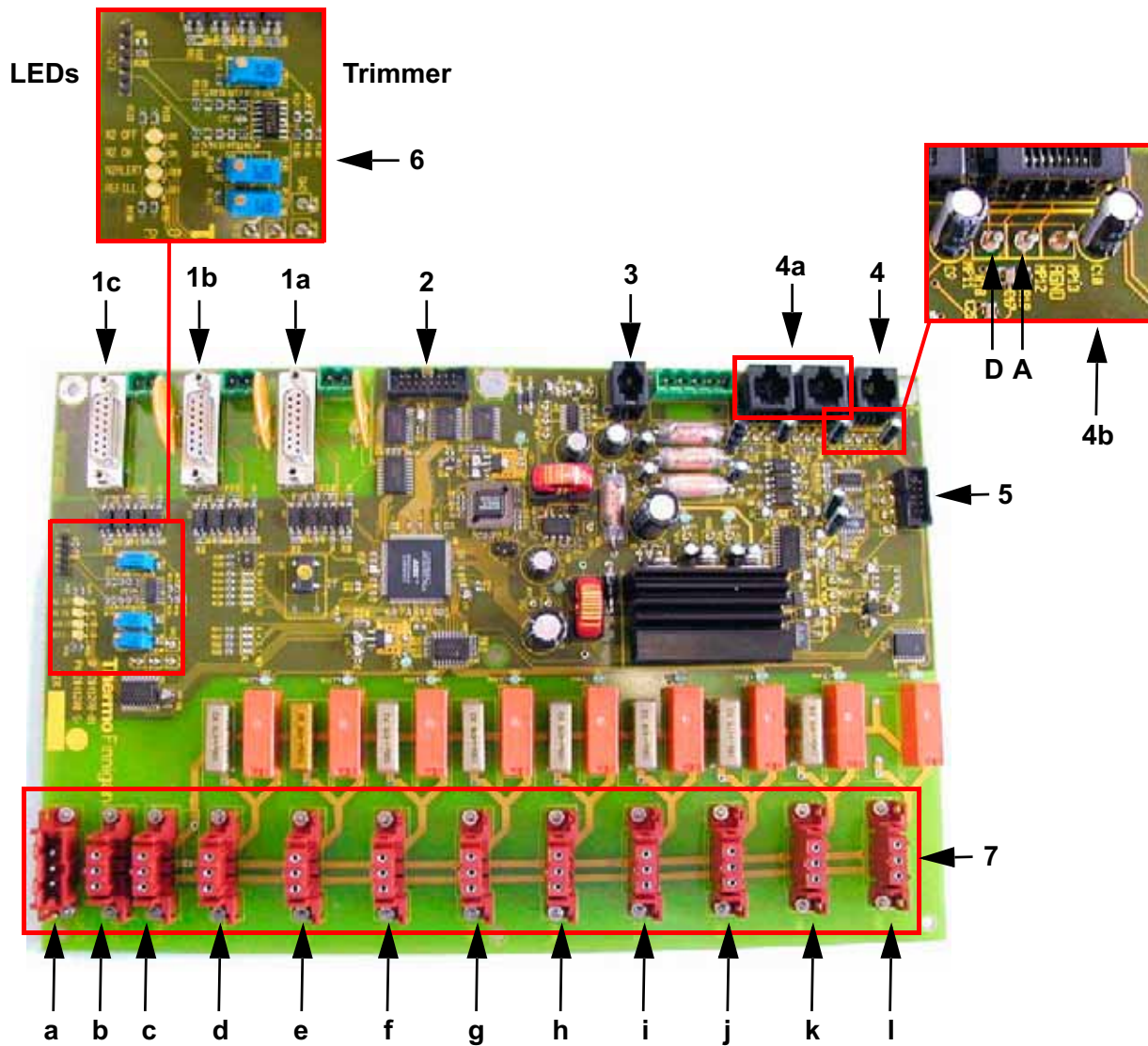
Figure 4-2.   Power distribution

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

1a   Connection to turbo pump analyzer

1b   Connection to turbo pump ion source

1c   Connection to turbo pump inlet system

2   Connection to front panel

3 Connection to serial interface

4 Connection to penning gauge

The penning gauge for the high vacuum measurement is connected here. This is important for the proper function of the high vacuum security.

4a Vacuum sensors

Additional vacuum gauges can be connected here. Anyway, the vacuum security is not affected.

4b Tapping points for penning gauge

The threshold (D) and actual (A) current values of the penning gauge can be remeasured here.

5 Connection to magnet current regulator

6 Controls for refill equipment

The settings of the trimmers determine the trigger thresholds.
The LED "N2 OFF" is on when the liquid nitrogen has reached the maximum level.
The LED "N2 ON" is on when the liquid nitrogen has fallen below the minimum level.
The LED "N2ALERT" is on when the liquid nitrogen has reached a critical level.
The LED "REFILL" is on when the refill is switched on.

7 Connections to all 230 V consumers

a Current input

b Power supply 24 V

c Power supply 55 V $\rightarrow$Socket J503

d Inlet valve heater $\rightarrow$Socket J1112

e Ion source controller $\rightarrow$Socket J322

f Refill equipment

g Ion source heater $\rightarrow$Socket J1114

h Source heating

i Inlet system heater $\rightarrow$Socket J1110/J1111

j Inlet pump $\rightarrow$Socket J1109

k Pump source & analyzer $\rightarrow$Socket J1108

l MS heater $\rightarrow$Socket J1113

4.3   Inlet Controller

The IRMS is provided with two variants of the inlet controller board (Part No. 204 1300). The internal board is located at the top right side of the electronics cabinet. The external board is located on the left side of the IRMS.
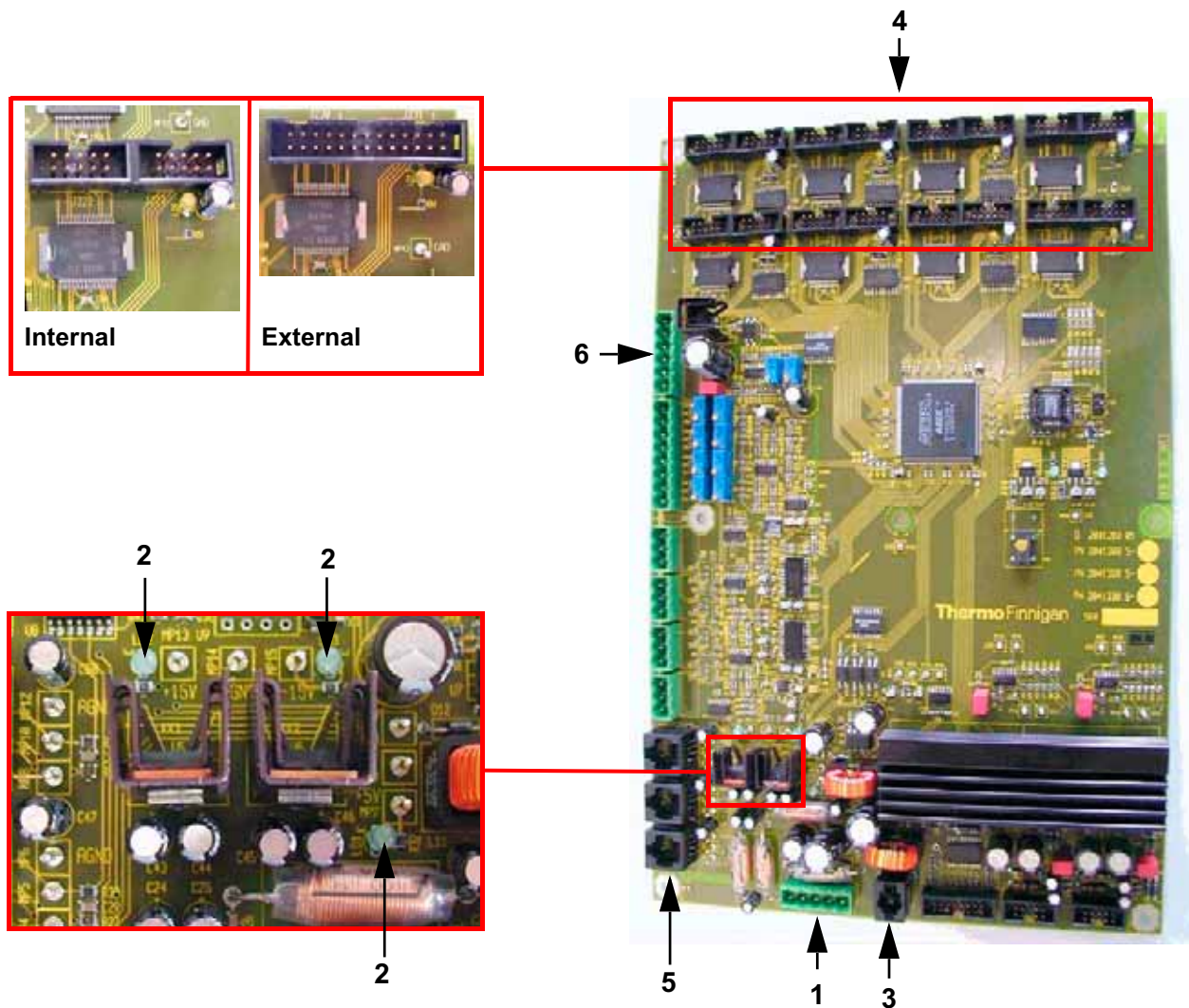
Figure 4-3.   Inlet Controller

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

1 Power supply 24 V

2 These LEDs are lit when the supply current is available.

3 Connection to serial data link

4 Control of the internal/external valve banks

5 Connection to optional pressure sensors

6 Connection to microvolume

The inlet controller board has the following functions:

- Control of the valve banks

- Connection for three additional vacuum gauges

- Connection for the operation device for the cooling trap

- Motion control of the bellows

4.4   The Plug and Measure Concept

Each peripheral device has its own plug and measure code. This code is encoded:

- either in the cable to the device

- or in a plug and measure adapter

This is also used for downward compatibility using an old peripheral device in combination with the new DELTA^{plus}XP electronics.

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

The Plug and Measure Adapter
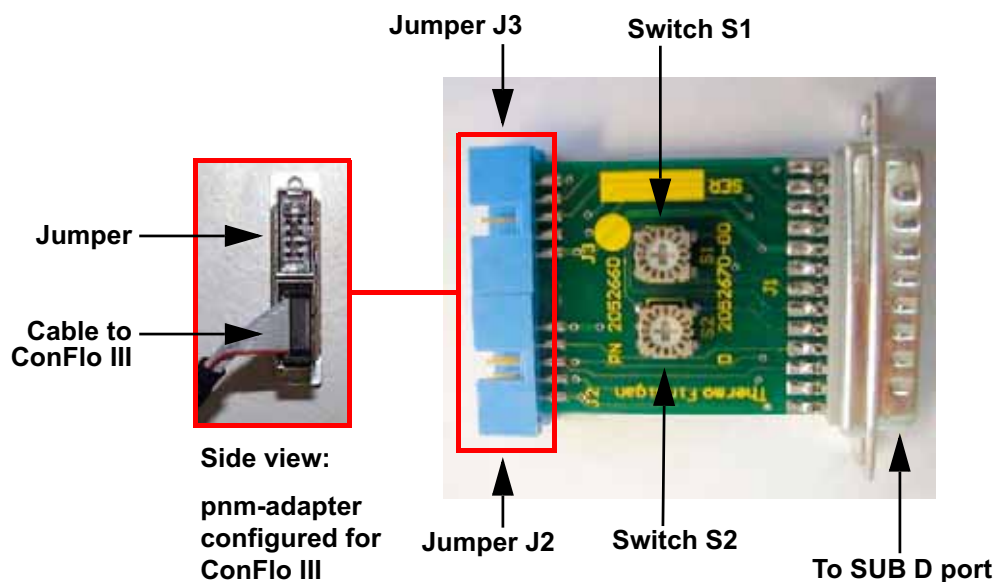
Figure 4-4.   PNM-Adapter

The plug & measure adapter (pnm-adapter) is used for the connection of the peripherals. It is pre-configured at the Thermo Electron factory for a defined option (e.g. ConFlo). A supplementary reconfiguration by the user is not recommended. The plug & measure adapter is connected to one of the five identical SUB D ports at the rear panel of the IRMS. The peripheral is then connected to the IRMS via the bottom port of the pnm-adapter.

The peripherals are identified by the settings of the turn switches and the jumpers. The turn switches are used to specify the pnm-ID for the peripheral (e.g. for ConFlo, set S1 to 2). The jumpers are also used to identify the kind of

peripheral that is connected to the IRMS). If indicated, the lowest two contacts of the plug socket at the pnm-adapter, that is either socket J3 or J2, are cut short (from the outside of the pnm-adapter).

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

Grounding Cable for Peripherals

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

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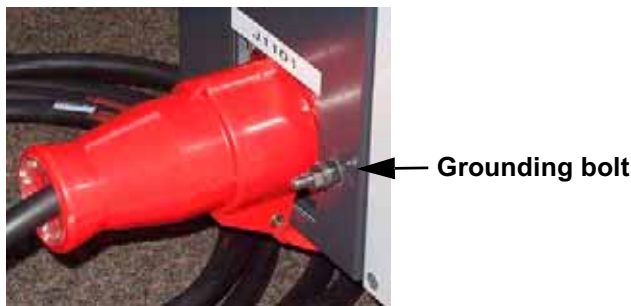
←— **Grounding bolt**

Figure 4-5. **Main switch with grounding bolt**

Configuring the Plug and Measure Devices

Internal options: Each option has a dedicated plug socket (with the exception of the tube cracker second bank). The software recognizes whether an option is connected to that socket or not.

External options: Five SUB-D ports are located at the instrument's rear side. The option is recognized, because it is encoded with a pnm-ID. The electronics recognizes:

• that there is an option connected to a certain port

• which kind of option it is

Table 4-1.   Settings for Plug and Measure Device

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

pnm-ID:	by switches inside the pnm-adapter or by shortcuts inside the cable
2nd ID:	if two instruments of the same type are installed
Jumper pin 9/10:	if indicated, the lowest two contacts of the plug socket at the pnm-adapter, i.e. either socket J3 or J2, are cut short (from the outside of the pnm-adapter).
New cable:	No pnm-adapter. Instead, an exchange of the cable is necessary.

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

Using the Options GasBench, PreCon and TubeCracker with another IRMS

The options GasBench, PreCon or TubeCracker are available in two versions:

- One for the DELTA^{plus}XP, DELTA^{plus}Advantage, or MAT 253 and

- one for the DELTA^{plus}, DELTA^{plus}XL, or MAT 252.

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

1. Applying an older peripheral device to a newer generation IRMS

    Use a plug and measure device, in case of PreCon and GP-Interface use a new connection cable.

    a. Each option delivered after June 2002 is delivered with the necessary hardware.

    b. For older devices, order a plug and measure device (Part No. 205 2660) or a connection cable.

2. Applying a peripheral device ordered with or for a newer generation mass spectrometer to an older generation IRMS

    Connect the connection cable to the driver board (see Figure 4-6 on page 4-12).

    a. For most peripheral devices the driver board supplied with the older generation IRMS can be used directly

    b. For devices like GasBench, PreCon or TubeCracker it is recommended to order a dedicated driver board (Part No. 202 5001).

        Alternatively, a new address can be assigned to the driver board delivered with the older generation IRMS. See Figure 4-6.

CAUTION!

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

3. Applying an option using an IEEE interface (Carbonate device / HDO device) delivered with an older generation IRMS to a newer generation IRMS

    Install an additional IEEE interface (Part No. 079 9130).
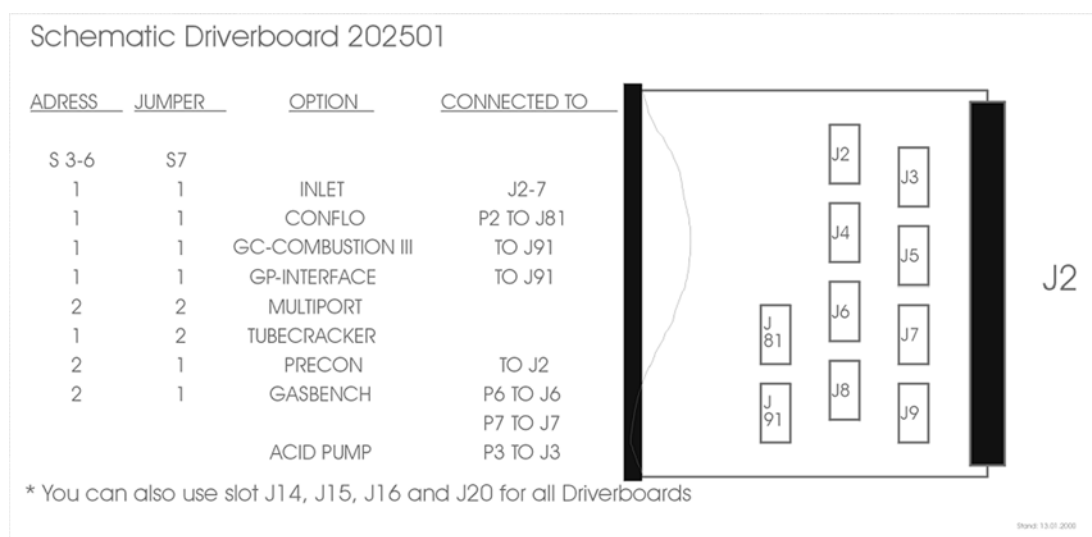
Figure 4-6.   Driver board

4.5   Data Acquisition

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

Data Logger

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

Connection to preamplifier housing in DELTA XL →

Connection to triple amplifiers in DELTA^{plus} →

Connection to HD-preamplifier →
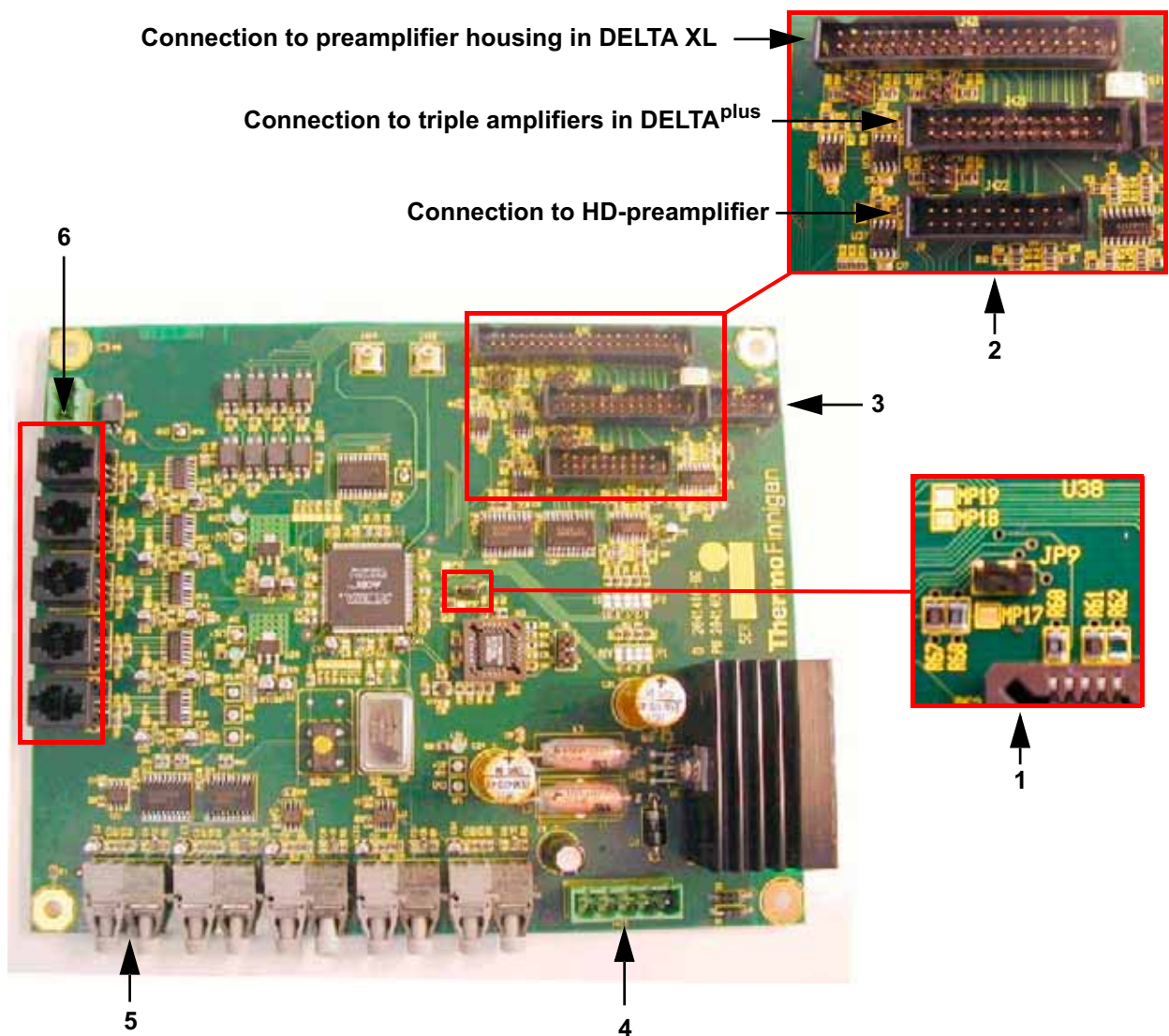
Figure 4-7.   Data logger

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

1   This jumper (JP9) is used to switch on the power supply of the amplifiers when the amplifier housing is open.

> **Caution.** The amplifiers can be damaged when they are exchanged while the power supply is switched on.

2   Connections to other boards

3   Connection to 55 V power supply

4   Connection to 24 V supply

5   Connection from serial interface to PC (DEL-PCI controller) via optical fiber

6   Serial data link to all other boards (e.g. power distributor, inlet control)

Groundplane

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

Port for preamplifier board (one of

Photo diode

Figure 4-8.   Groundplane

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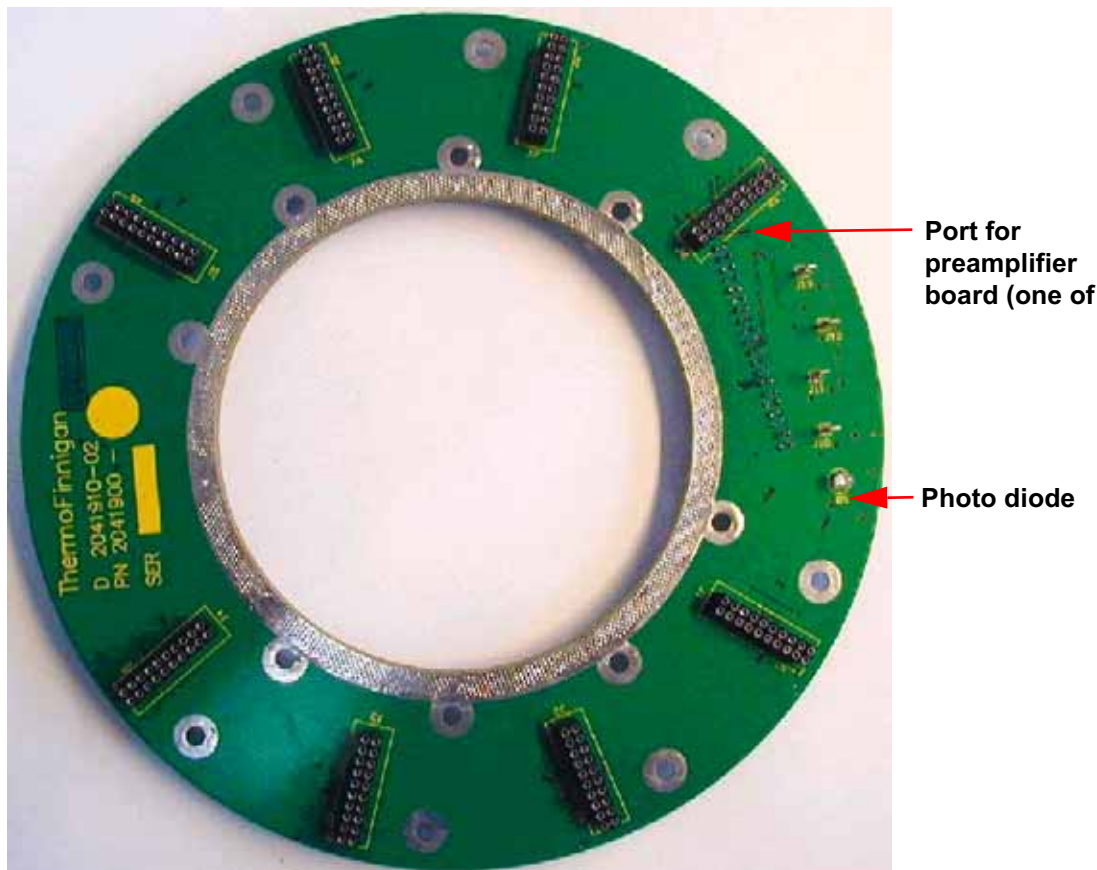
Amplifier & VFC

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

R1 at J7

C1 at J6

R2 at J8

C2 at J9
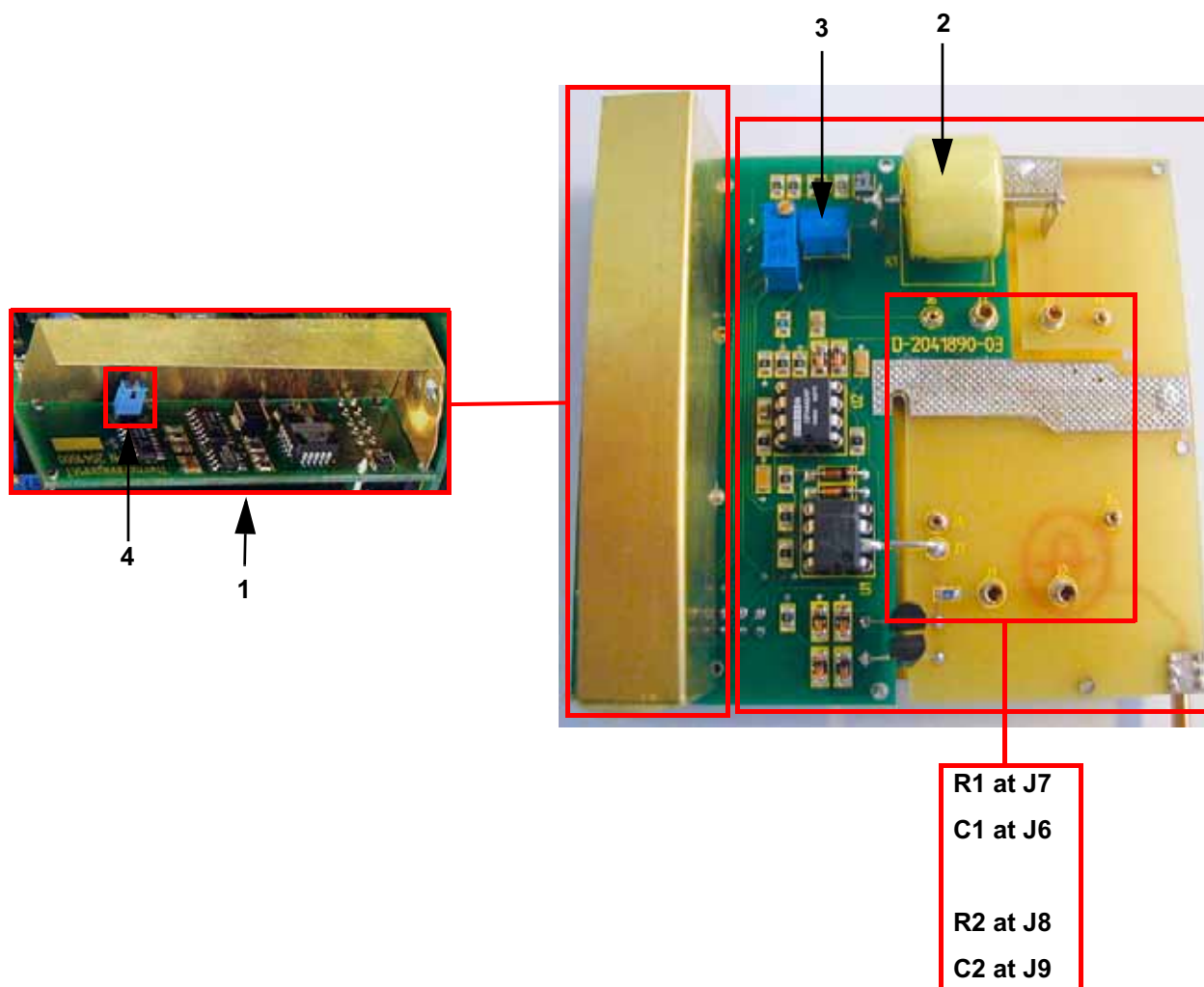
Figure 4-9.   Preamplifier board

For a description of selected board parts, see the following explanations:

1   VFC

2   Amplifier

3   Setting of the time constant

4   Jumper to disconnect amplifier and VFC (for service purposes)

Changing the Amplifier

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

Amplifier Lid
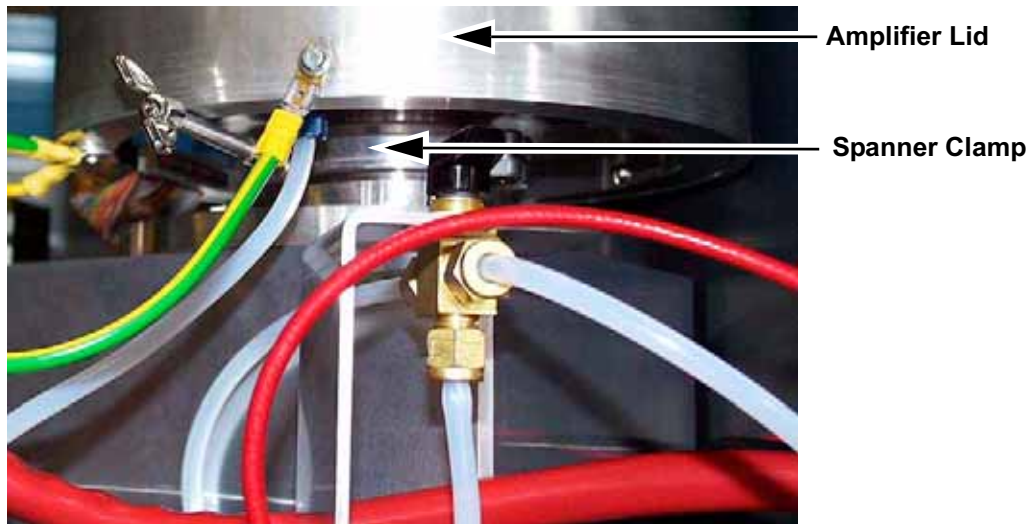
Spanner Clamp

Figure 4-10.   Bottom side of amplifier housing

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

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

Working with the Collector - Removing the Amplifier Housing

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

1. Control, whether the spanner clamp fits correctly.

2. Remove the amplifier lid.

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

4. Unclip the spanner clamp by tearing or pressing slightly.

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

Working with the Collector - Mounting the Amplifier again

To mount the amplifier again, proceed as follows:

1. Position the open spanner clamp on the flange.

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

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

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

CAUTION!

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

5. Secure the preamplifier boards by screws.

6. Put on the amplifier lid again.

Power Supply 55 V

The 55 V power supply (Part No. 204 1920) is located at the top middle of the electronics cabinet. It provides the voltages (-55 V, +15 V, -15 V) that are required by the amplifiers. The four LEDs on the right side of the board are all on when the supply voltages are available.
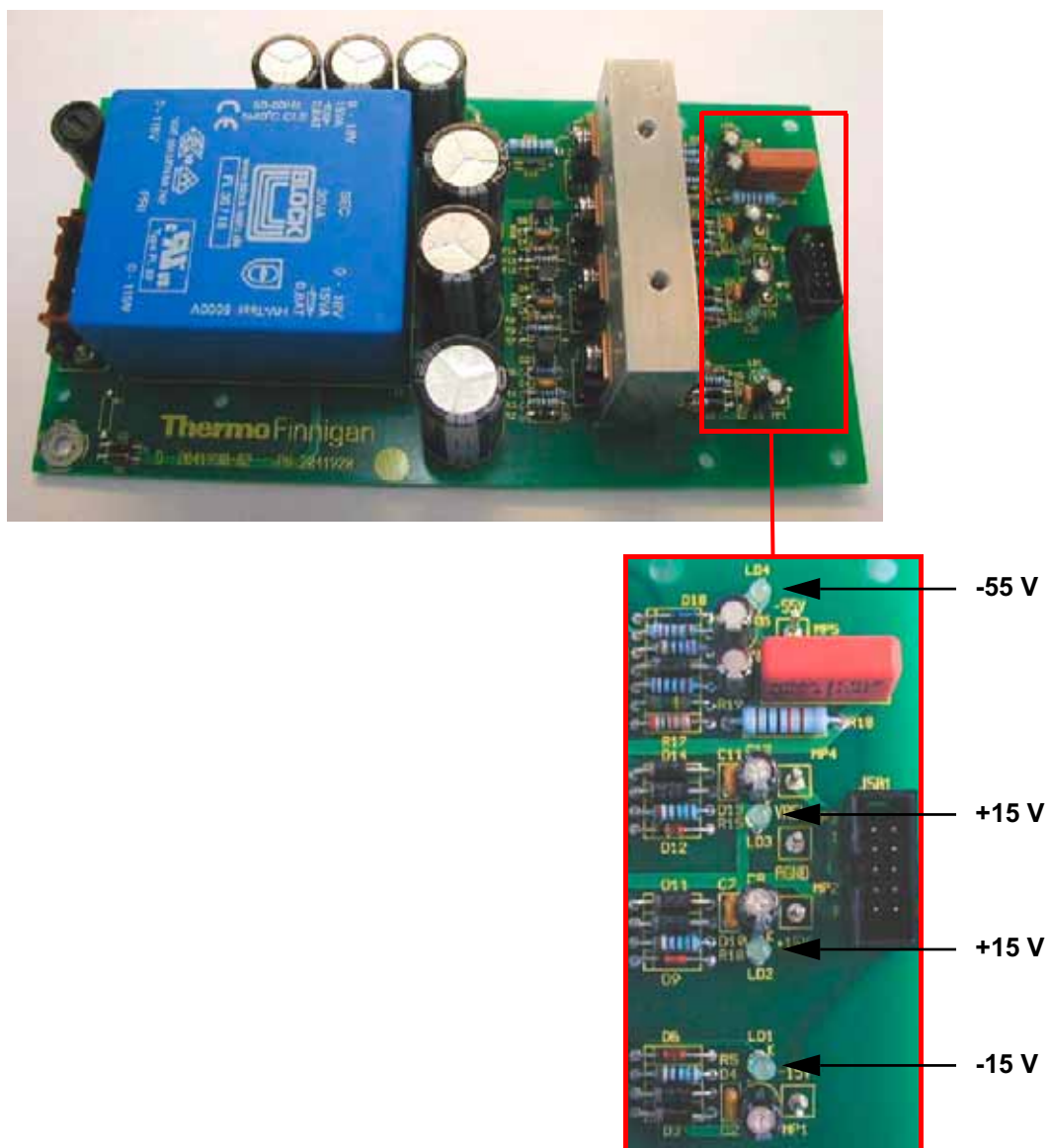
Figure 4-11.   Power supply 55 V

4.6   Ion Source Controller

The regulation results from the sum of the box and the trap current.

The regulation only results from the trap current.
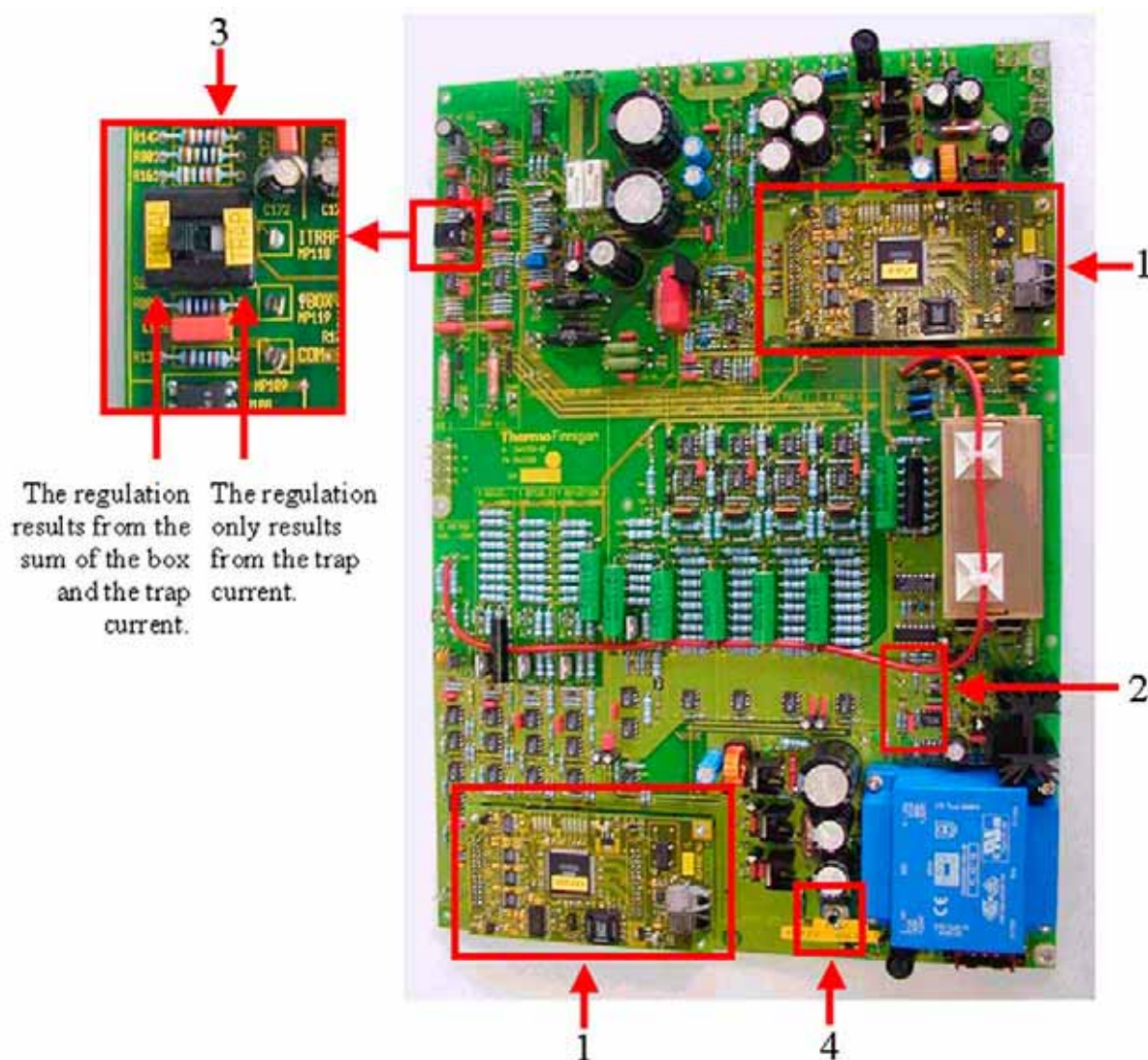
Figure 4-12.   Ion Source Controller

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

1. Two bus controller boards control the ion source.

2. These LEDs indicate the proper function of the ion source.

3. The setting of this switch determines whether only the trap current or also the box current is used for the emission regulation.

4. On-off switch for high voltage

The ion source controller (Part No. 204 13 40) is located at the bottom left of the electronics cabinet. It has the following functions:

- creation of high voltage

- emission control

- creation of all required potentials (as X-focus, deflection, etc.)

Bus Controller

The bus controller is a universal control unit. Two boards of this type (see table) are located on the ion source controller.

Table 4-2.   Bus Controller

Designation	Part No.
Bus controller, ground	205 5000
Bus controller, high voltage	204 1450
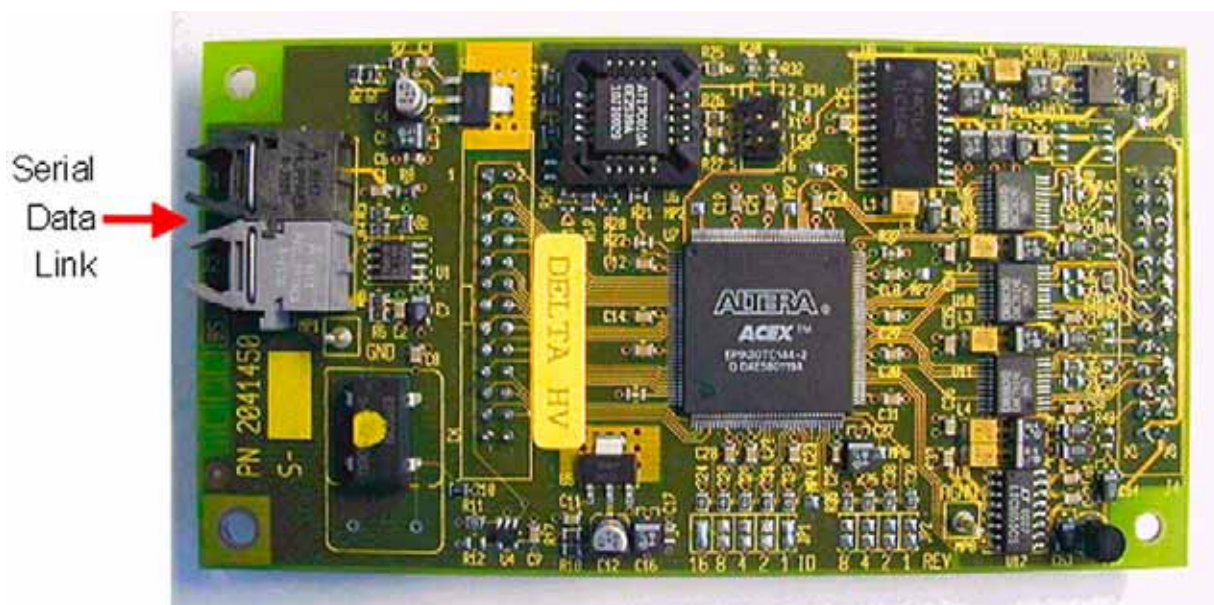
Figure 4-13.   Bus Controller

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

4.7   Magnet Current Regulator

The magnet current regulator (Part No. 024 33 12) is located at the top left of the electronics cabinet. It provides the current required to generate the electromagnetic field. The current is computer-controlled via the DAC printed circuit board (digital-to-analog converter).

The computer feeds the information of the specified mass number via the optical bus and the data logger to the microprocessor, which controls the DAC on the power distribution. Here, this information is converted into an output voltage, which controls the power supply of the magnet current regulator.
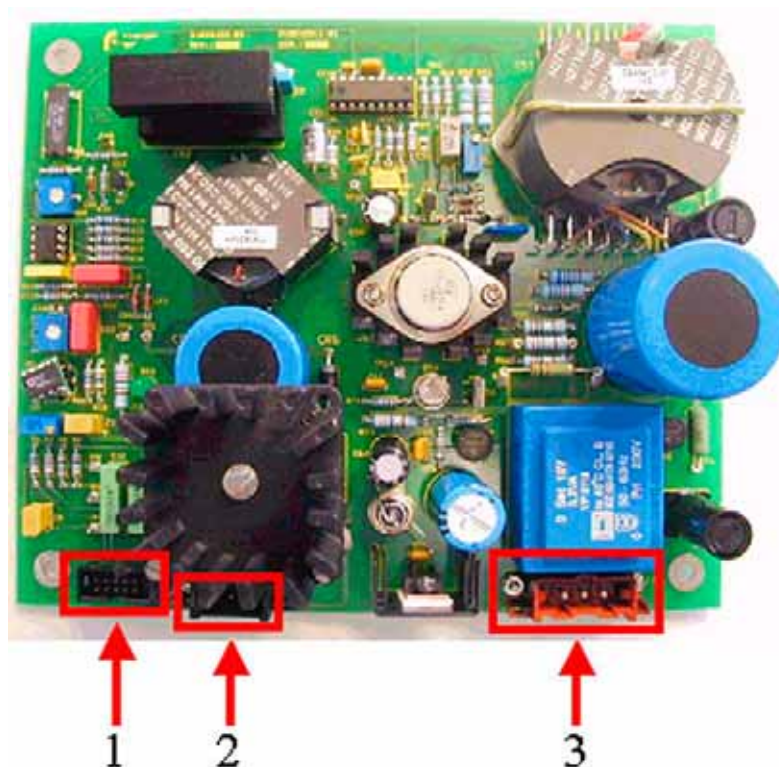
Figure 4-14.   Magnet Current Regulator

1.   Connection to power distribution

2.   Connection to magnet

3.   Connection to line power supply 230 V

DANGER!

Danger. Parts of the board are at 230 V voltage. Never touch this board without safety measures!

4.8   DEL-PCI Controller

The DEL-PCI controller (Part No. 205 25 80) is plugged in a PCI slot of the computer that is delivered with the IRMS. It is connected to the bus controllers in the IRMS via optical fibers. This board is the instrument controller and contains the front-end processor.
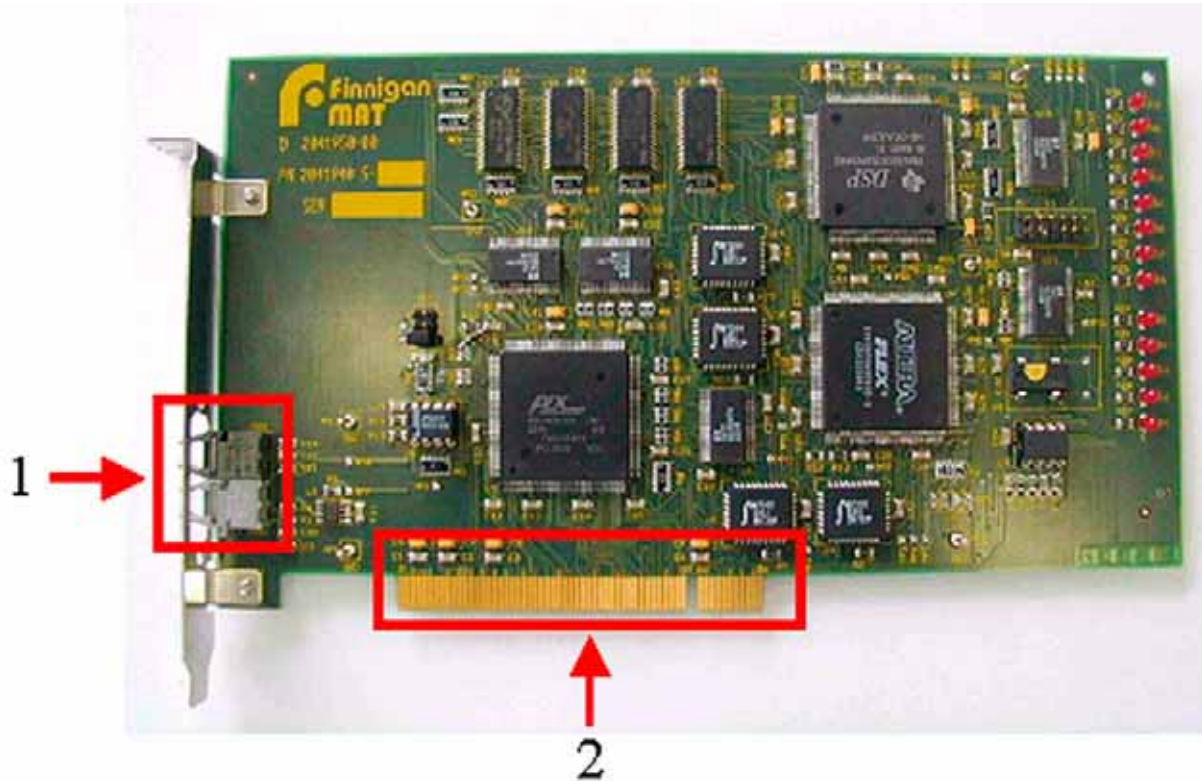
Figure 4-15.   DEL-PCI Controller

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

1.   Serial data link to IRMS via optical fibers

2.   Connection to PCI slot of computer

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Chapter 5

Dual Inlet System

5.1 General Description of the Dual Inlet System

The Dual Inlet system of the Finnigan DELTA^{plus}XP isotope ratio mass spectrometer has a symmetric design to allow alternating measurements of a sample and a standard gas.

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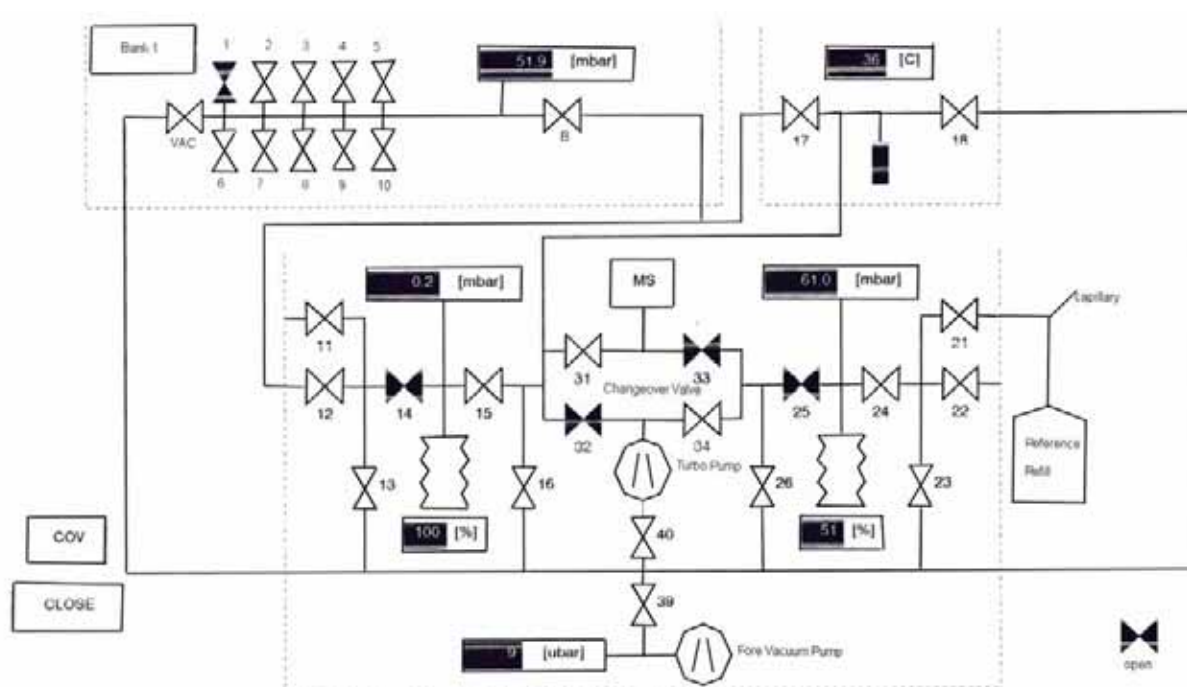
Figure 5-1.   Dual Inlet System

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

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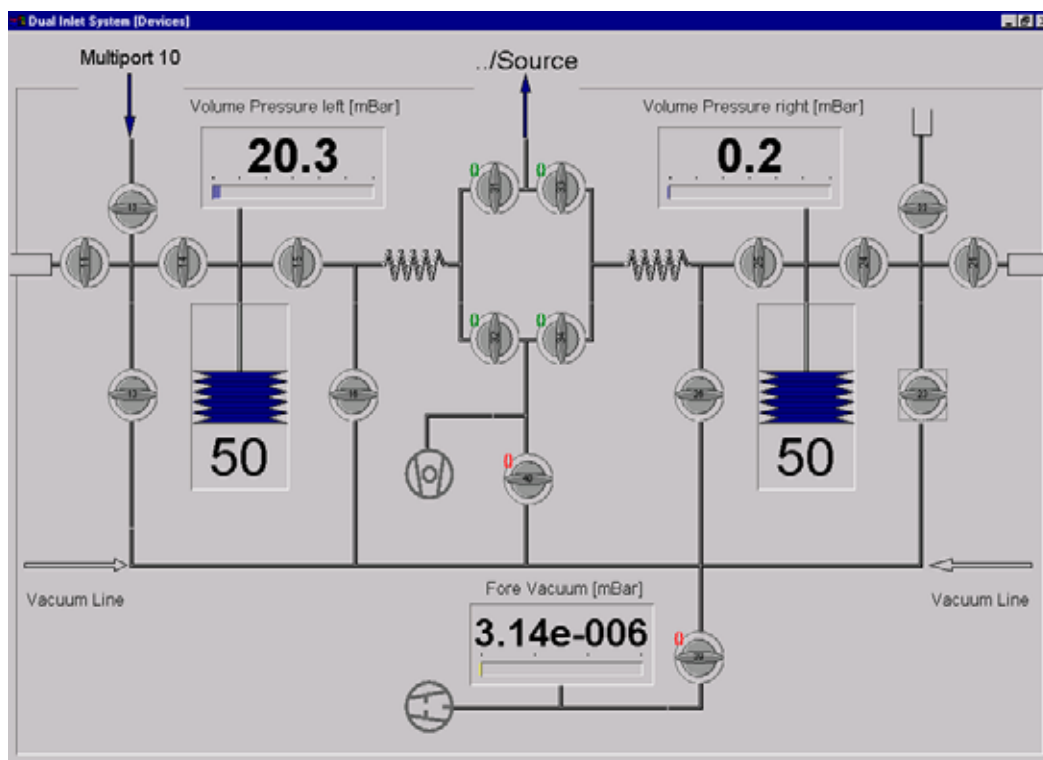
Figure 5-2.   Schematic of the Dual Inlet System

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

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

The crimps of the capillaries are factory-set, but must be set new when a capillary is replaced. How to crimp a capillary to a specific flow resistance is described in topic **Heating Inlet Capillaries** on page 7-51 and topic **Replacing Inlet Capillaries** on page 7-53.

The variable volumes adjust the pressure for larger samples (> 50 barµl). Very small samples, as low as 5 barµl, can be analyzed using the optional Microvolume inlet. For more details, refer to topic **Microvolume** on page 5-8.

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

5.2   Valve System

The inlet system is operated by pneumatic valves with a nominal closing pressure of 4 bar. The compressed air is either supplied by an optional compressor attached to the mass spectrometer or by a user supplied pressure air line. The metal valves are equipped with gold gaskets and gold seats acting on knife-edges. Up to six valves are machined into one monoblock, thus considerably reducing the volume in plumbing as well as possible leakage of the installation. This type of valve blocks is used throughout all inlet modules. For plumbing the valve blocks are fitted with 1/4" Swagelok connectors. Compressed air is fed to the pneumatically operated valves by solenoid valves. These are controlled by dedicated electronics linked to the computer via a data bus.
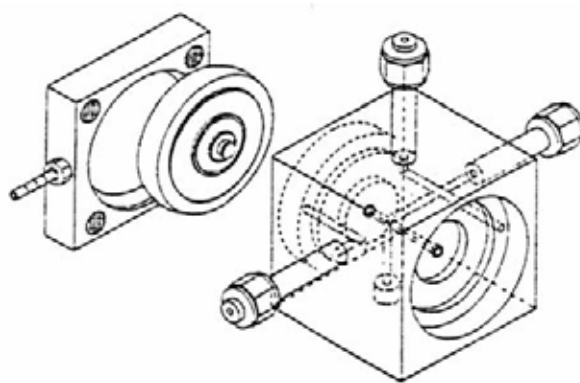
Figure 5-3.   Schematic view of a double valve block

Four of the solenoid three-way valves are located on a "Manifold" block. The solenoid valves are operated with a voltage of 24 V. The voltage is supplied by one of the inlet boards. The solenoid valves are normally open and the working condition is signaled by a red LED located on the printed board.
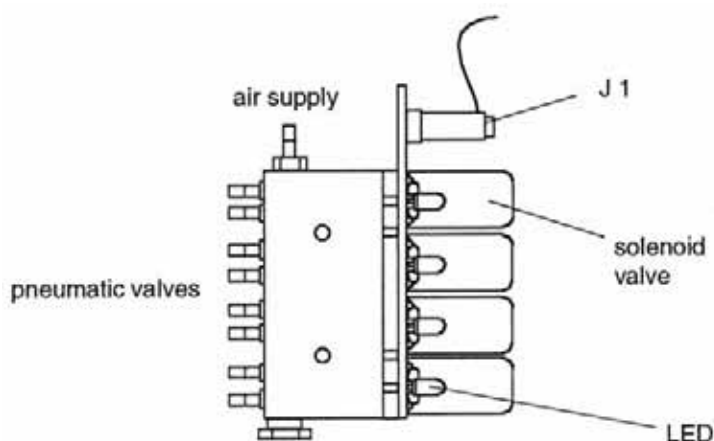
Figure 5-4.   "Manifold" block with 4 solenoid valves

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

Figure 5-5.   Driver board with distributor function

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

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

5.3   Changeover Valve

Basic Module

The basic module of a changeover valve consists of a single block attached to the analyzer housing and accepts the coupling of capillaries for sample and standard gas. The changeover valve is operated automatically by ISODAT NT or manually via the monitor display of the inlet schematic.
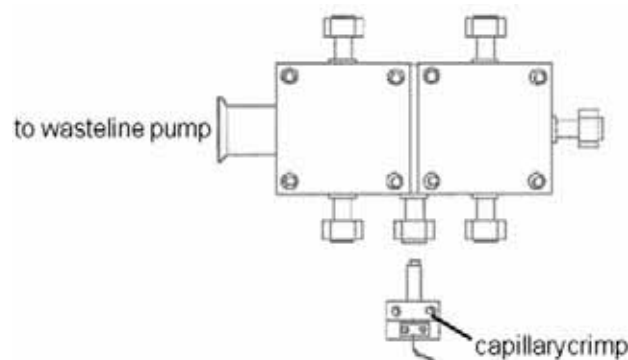
Figure 5-6.   **Basic module of changeover valve block**

Extension Module

An extension module may be added to the changeover valve as an option. The extension module is flanged to the changeover valve by ¼" Swagelok connectors. It provides two additional inlet ports allowing coupling of further inlet system options, e.g. a carbonate system. To monitor and operate the extended changeover valve, activate the respective ISODAT NT routine.
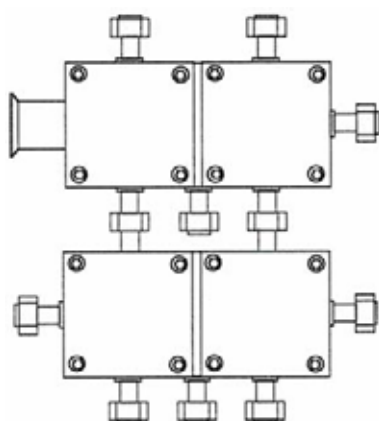
Figure 5-7.   **Extension module attached to the basic module of the changeover valve**

5.4   Multiport

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

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

To monitor and to operate the Multiport valve system on screen the respective ISODAT NT routine has to be activated.
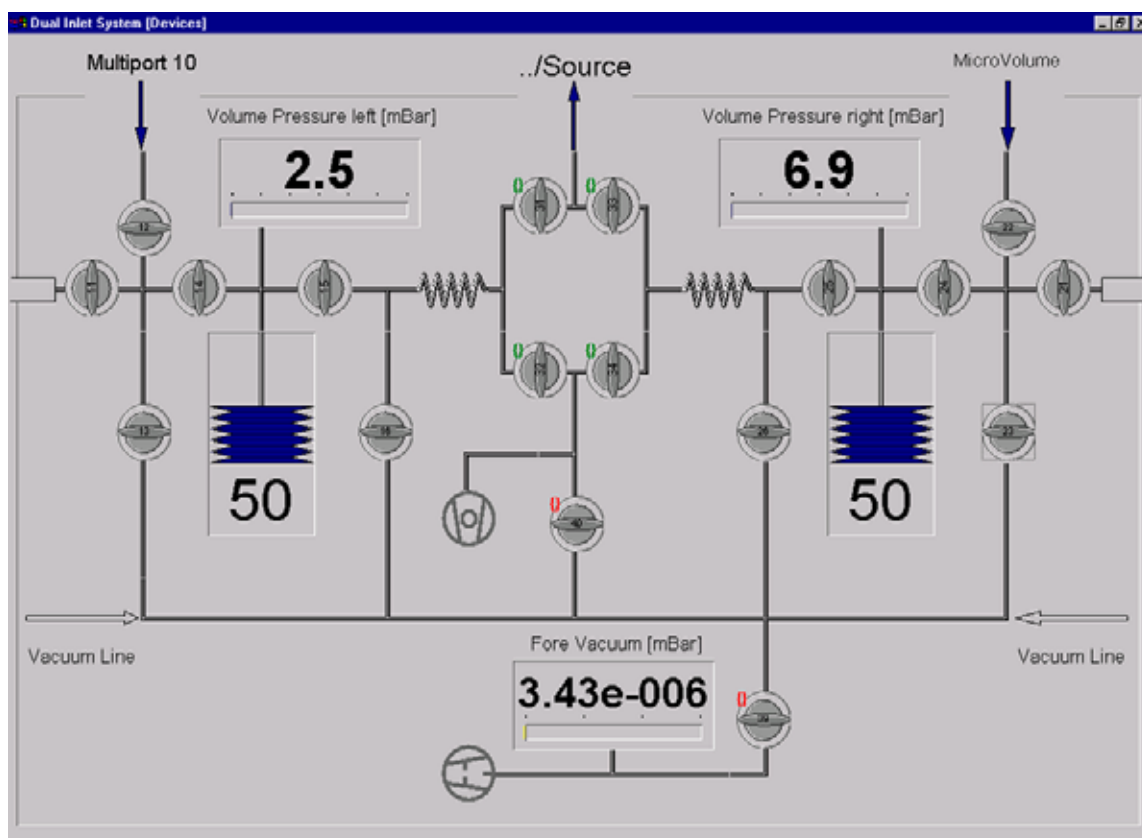
Figure 5-8.   Schematic of a Dual Inlet System with Multiport and Microvolume

Measurement using a Multiport as an Inlet System is described in topic **Dual Inlet Measurement Including Multiport** on page 6-25.

5.5 Microvolume

Microvolume or "cold finger" as optional Inlet module for very small samples may be installed in combination with a Dual Inlet system or a Multiport. In both cases, Microvolume is connected to the left port (valve # 12) of the Dual Inlet system (Figure 5-2 on page 5-3 and Figure 5-8 on page 5-7).
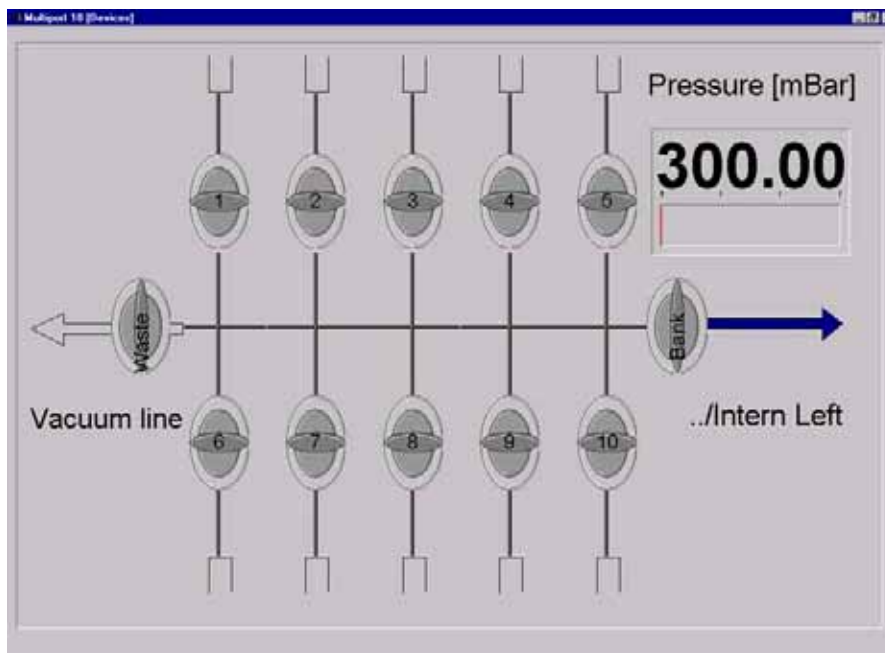
Figure 5-9.   Schematic of a Multiport
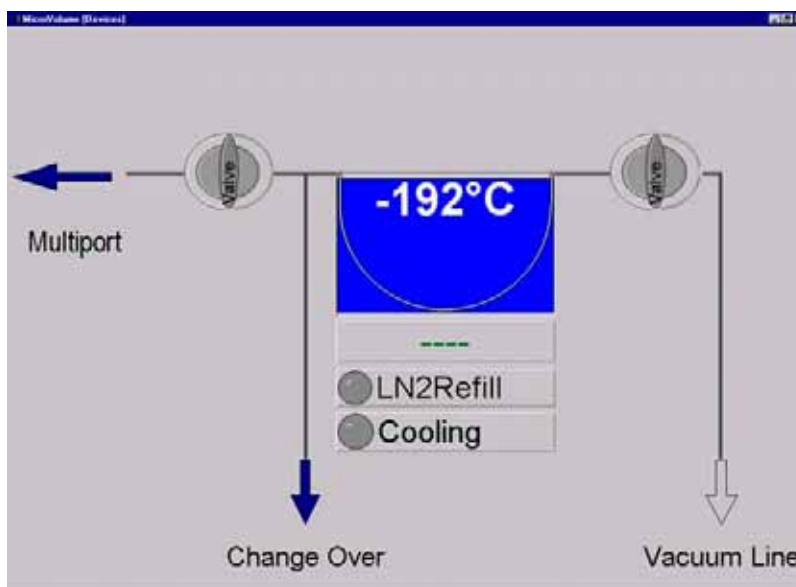
Figure 5-10.   Schematic of a Microvolume

1 "coldfinger"
2 fitting
3 copperrod
4 washer
5 gasket (gold)
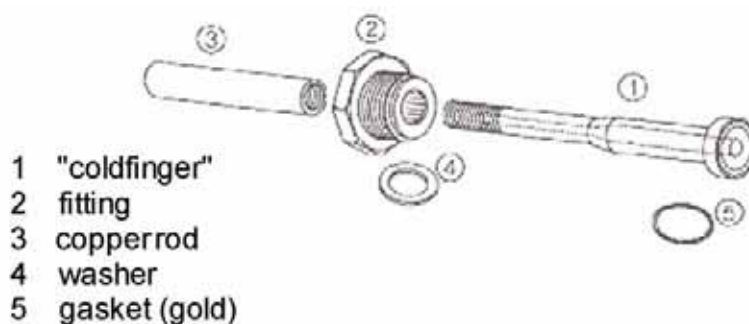
A Microvolume consists of a "cold finger", a valve block, an Autocool unit and a separate capillary.

This capillary leads directly to the changeover valve of the system.

Figure 5-11.   Parts of a Microvolume to be inserted into an Autocool unit

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

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

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

Autocool Unit

The temperature of the Autocool unit, which cools the "cold finger", can be set via ISODAT NT's Microvolume control (i.e. via the "Instrument" tab's "Microvolume" part; refer to topic **Dual Inlet Measurement Including Microvolume** on page 6-32). A temperature range between -180 °C and +155 °C can be covered.

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

-180 °C up to +50 °C is about 1 min.

The Microvolume fits into a thermal contact pipe attached to the lid of a Dewar. The Dewar contains liquid nitrogen. An electrical heater element, a temperature sensor and a cascade arrangement of three small bowls are fitted to the contact pipe (see Figure 5-12). All parts of the assembly are made of a material of high thermal conductivity and are placed in close thermal contact to each other. Thus, a quick changeover from one temperature to another is

provided. To heat the Microvolume to a defined temperature, the heater element is activated. The heating phase is controlled by the temperature sensor. The three thermistors **(7)** are Pt 100 resistors (that is 100 Ω).

1 thermal contact pipe
2 heater element
3 hood and standpipe
4 immersed heater element
5 temperature sensor
6 cascaded arrangement of bowls
7 level sensor with 3 thermistors
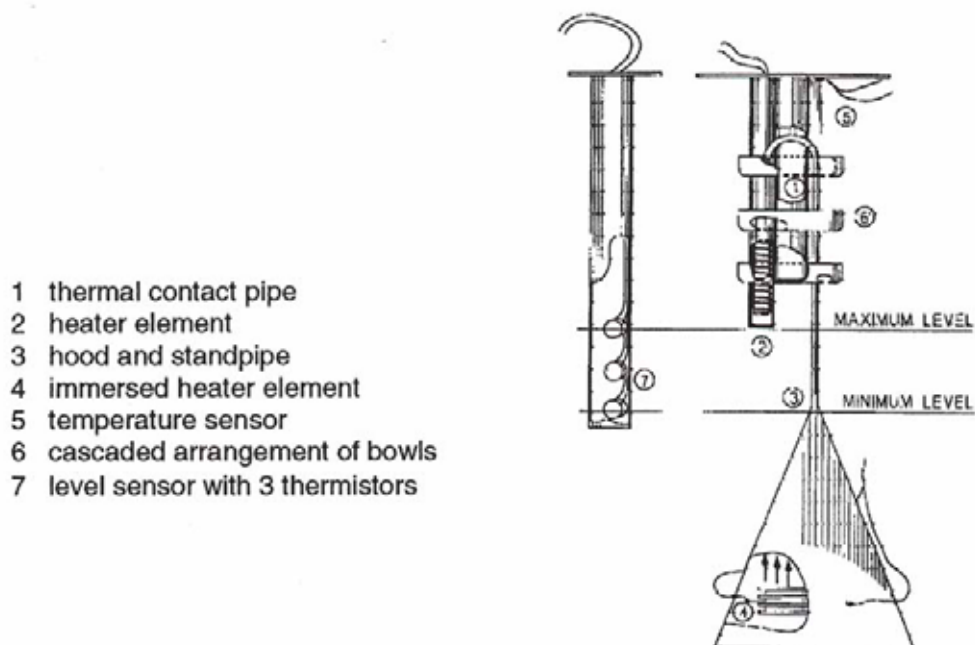
Figure 5-12. Schematic view of an Autocool unit

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

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

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

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

Autocool Refill Device

Safety Warnings

Warning. Keep in mind: Safety First!

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

Make sure that only authorized and fully trained operators use this equipment and they are fully conversant with these safety notes.

Warning. The refill device contains extremely cold liquid gas. Careless handling might cause severe personal injury including frostbite.

Only use the cryogenic liquid specified on the label on the refill device.

Warning. Wear protective clothing when operating this equipment, including protective gloves and face shield. Do not overfill or tilt the refill device! Prevent spills.

CAUTION!

Caution. Use the refill device only in well-ventilated areas. Poor ventilation might cause suffocation. Follow correct First Aid procedures. If gas was inhaled, remove victim to fresh air. If necessary give artificial respiration and seek medical assistance immediately.

General Remarks

Refill device provides a constant level of liquid N_2 in the Dewar of Autocool unit. It consists of a storage Dewar of 75 l or 25 l and is equipped with safety devices, valves and pressure gauges required for safely handling liquid N_2.
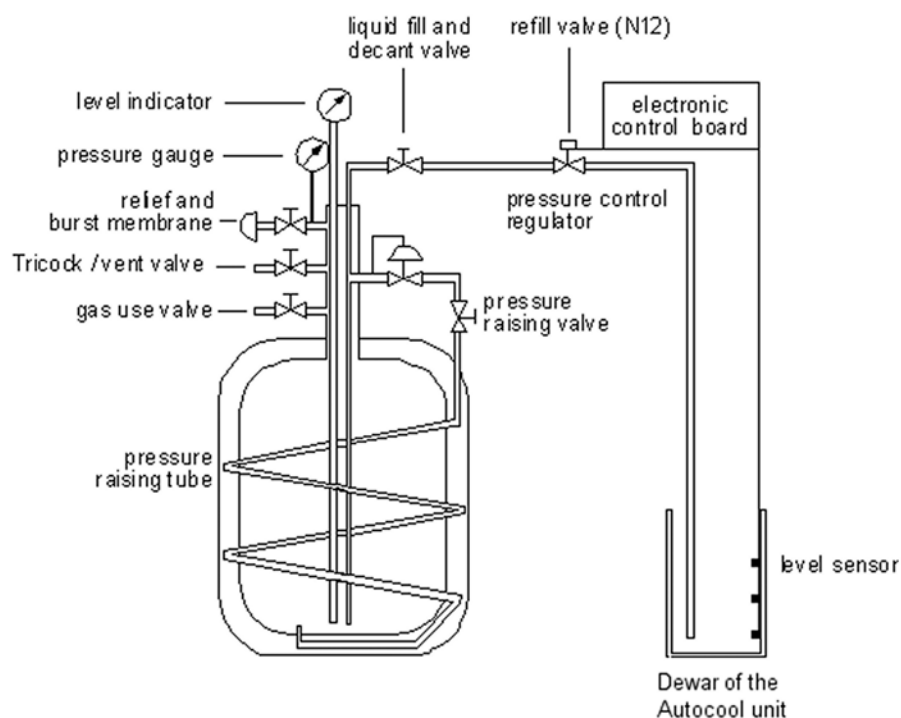
Figure 5-13.   Schematic view of a refill device

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

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

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

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

• yellow LED (upper position): high liquid N_2 level, indicates refill end.

• yellow LED (middle position): low liquid N_2 level, indicates refill start.

• yellow LED: alarm level, no refill has been performed.
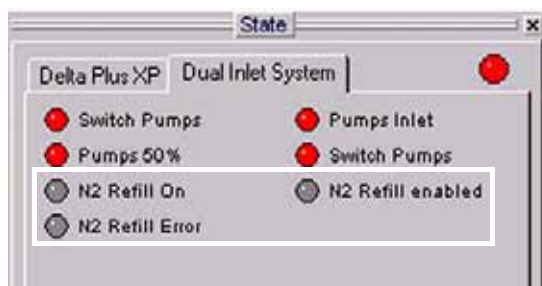
Figure 5-14.   State window, showing the Dual Inlet System tab

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

•   the yellow LED on the control board panel,

•   a warning message of the system on the monitor,

•   an interruption of the measurement

Control of the Refill Device
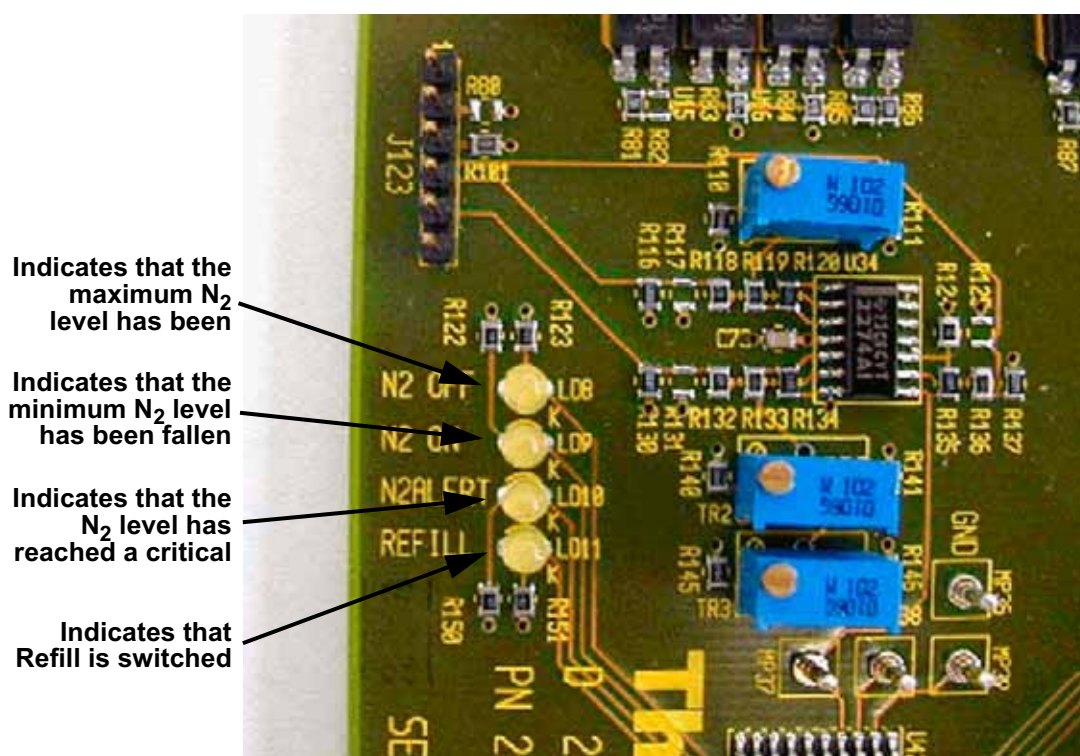
Figure 5-15.   Power Distribution board, showing the control LEDs of the refill device

Working Principle

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

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

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

The blow-off valve is set to a limit of about 1.5 bar. An additional burst membrane prevents from building up a dangerous pressure. The gas vent valve allows bleeding excessive pressure, if necessary. The function schematic of the liquid nitrogen refill device is shown in Figure 5-13 on page 5-12. The 75 l storage Dewar is equipped with a level indicator monitoring the liquid nitrogen content.

Maintenance

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

Occasionally, the pressure should be increased up to the relief valve setting to ensure satisfactory functioning of this safety device. Setting the pressure control regulator is performed as follows:

1. Loosen hexagon locking nut below adjusting screw.

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

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

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

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

Checking the Liquid Nitrogen Evaporation Rate

If you suspect that the evaporation rate of the refill device is excessive, note down the decrease of the liquid N_2 level for some time. To check the loss rate, close the pressure raising valve and open the Trycock/vent valve. After the contents are fully vented down to atmospheric pressure, measure the boil off rate using a simple flow meter. The normal boil-off rate for nitrogen is:

1 l/min for gas, that is about 2 l of liquid per day.

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

Operating Instructions

As a quick reference, the arrangement of valves for filling, dispensing and storage of liquid nitrogen is shown below (refer to Figure 5-13 on page 5-12):

Table 5-1.   Valve states during operation

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

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

1. Open the Trycock/vent valve and close the pressure-raising valve.

2. Close gas use valve. and fill via the opened liquid fill and decant valve.

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

1. Check the pipe connection leading to the Dewar of the Autocool unit.

2. Close the gas vent valve and open the liquid fill and decant valve.

3. Open the pressure-raising valve.

The refill device is now connected to the Dewar of the Microvolume via the opened liquid fill and decant valve.

The flow is controlled via the refill valve N 12. The pressure-building valve may be closed when the working pressure is reached, or it may remain open - provided the pressure regulator is set to a suitable working pressure.

Standard Refill (Reference Gas Refill)

When working with a Multiport Inlet System, a reference gas refill may be necessary in order to avoid running out of reference gas during measurements. Reference Refill provides the reference gas supply to the Inlet System. Reference Refill is a hardware option. It consists of a metal tank (its capacity is approximately 5 L) with a manual valve connected via a capillary to one of the Inlet ports on the standard side (see Figure 5-16). With the Reference Refill selected, the standard side of the inlet system is completely pumped out before it is filled again for the next measurement Sequence.
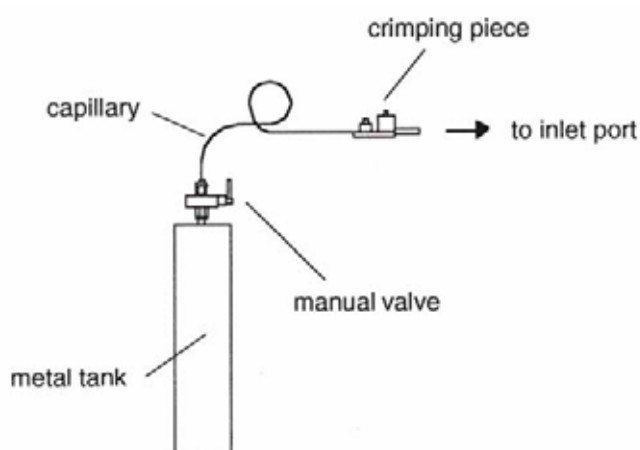
Figure 5-16.   Reference Gas Refill

1. To create a Configuration containing Reference Refill, at Dual Inlet Sets select the predefined Dual Inlet + Multiport + Reference Refill device and drag it to the Source.

    Thus, the Reference Refill will automatically be connected to the Internal Right port (at Internal Right port, two possibilities exist for connection. Either of them can be used).

    For detailed information about Reference Refill, refer to topic **Dual Inlet Measurement Including Multiport and Reference Refill** on page 6-54.

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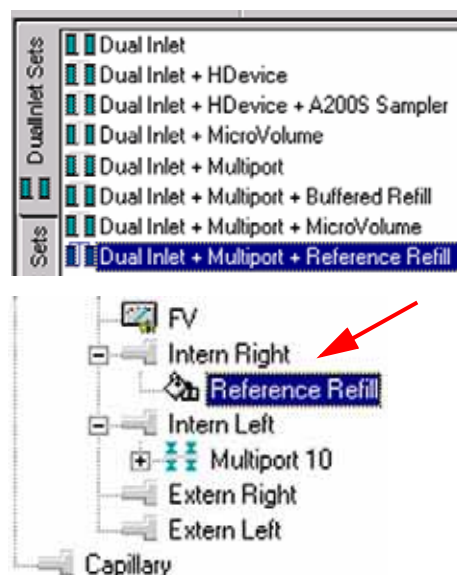
2. Creating a new Method or using a predefined one, note the Instrument tab, where some peculiar information concerning Reference Refill is summarized (for details, see **Using a Predefined Method** on page 6-55):

Figure 5-17.   Reference Refill group box

3. Creating a new Sequence, note the Sequence list.

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

Figure 5-18.   Activating Reference Refill

Chapter 6

Measurements

6.1   General Remarks

Procedure before Starting any Measurements

Before starting any measurements, three steps must be performed:

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

2. Define a **Method**

3. Define a **Sequence**

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

If no Configuration is Available

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

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

1. Your type of IRMS is required. Select **Delta Plus XP**.

2. Press **OK.**

Figure 6-1.   Selecting the instrument type

3. Check, whether the correct cups are installed.

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

5. Check, whether the resistor values are correct.

6. Confirm by **OK**.

Figure 6-2.   Checking the cup settings

7. Check the available Gas Configurations. You can edit them by clicking on the fields (refer to the ISODAT NT Help System).

8. Finally, press **Save & Close**.
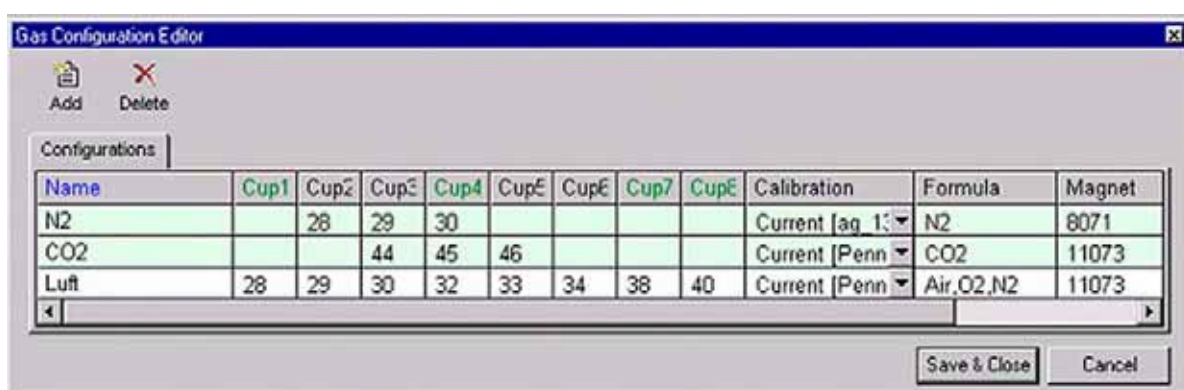
Figure 6-3.   Checking the Gas Configurations

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

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 ($^{12}C^{16}O^{18}O$).

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

2. Then open the Gas Configuration Editor.

3. Add a Gas Configuration.

Figure 6-4.   New Gas Configuration

4. Type CO2 for the Name.

5. Select Gas Configuration CO2 as Template.

6. Confirm with **OK**.
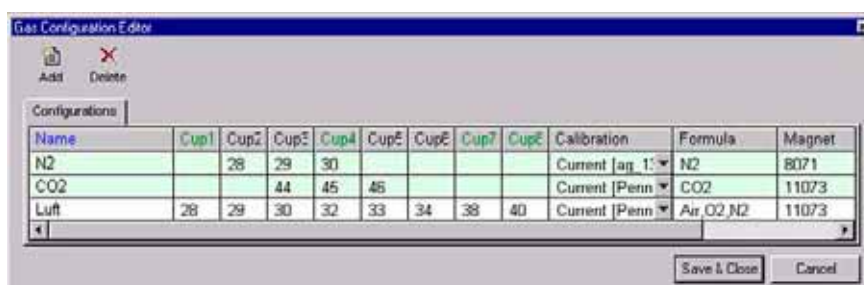
7. Select a Calibration valid for the selected cups.

8. 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:

How to Create a New Method

1. Open the Dual Inlet mode.

2. Select the **Configuration** for
   your application (e.g. *Dual Inlet*).

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

4. Press the **New** button.

5. 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.

How to Create a New Sequence

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

1. Press the **New** button.

2. Press the **Sequence** Icon.

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

Figure 6-5. **Defining the number of samples**

4. Edit the Sequence list.

Figure 6-6. **Editing the Sequence list**

		Peak Center	Enable ✓ to perform a Peak Center prior to measurement.
		Press Adjust	Enable ✓ to perform a pressure adjust.
		Background	Enable ✓ to perform a background measurement.
		Identifier 1	Edit text to identify sample.
		Method	Select IRMS Method (for each sample, e.g. "CO2.met").

5. Press the **Start** button.
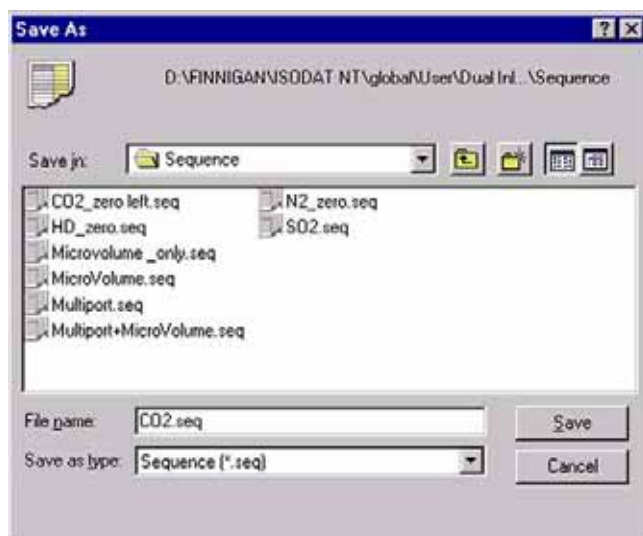
Figure 6-7.   Saving a new sequence

6.   Type a new significant File name retaining the extension .seq (e.g. "CO2.seq").

7.   Press **Save**.
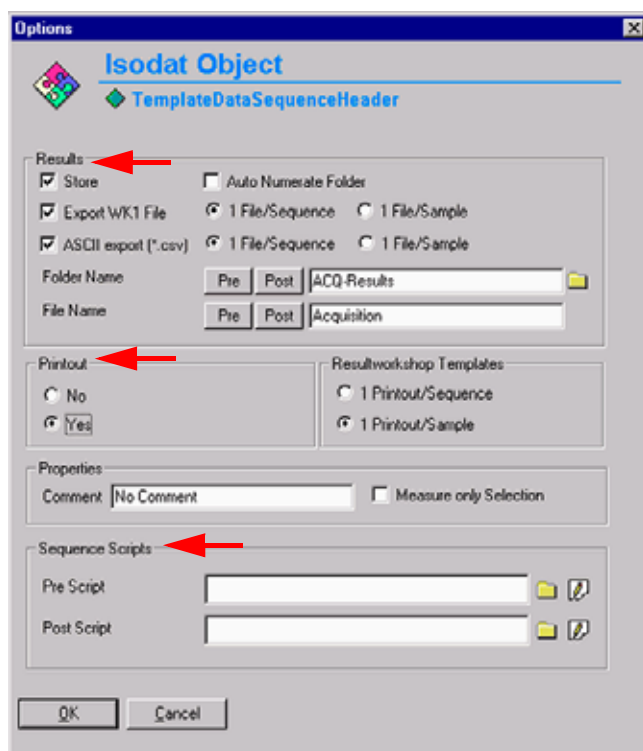
Figure 6-8.   Defining Sequence parameters

8. Define parameters for:

    •   Results Export

    •   Printout

    •   Sequence Scripts (See Figure 6-28 on page 6-24.)

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

6.2 Zero Enrichment (Standard On/Off Test)

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

Proceed as follows for zero enrichment:

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

2. Activate the File Browser check box.
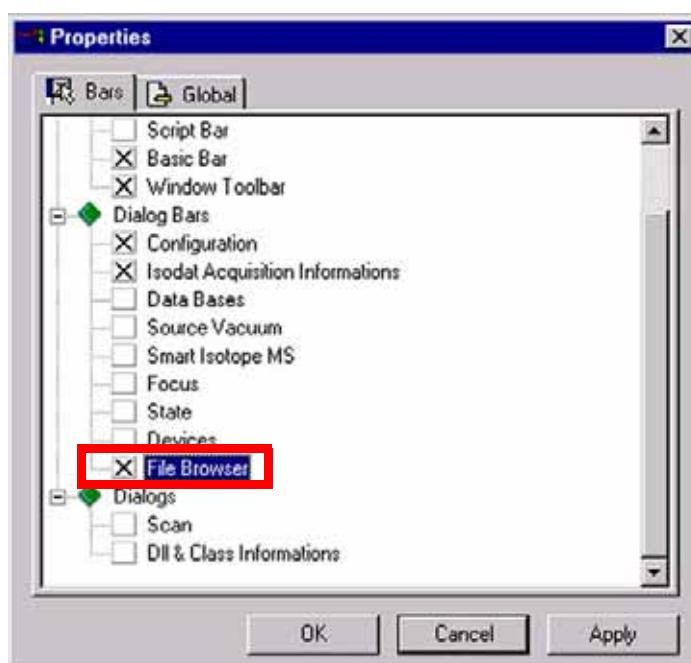
Figure 6-9.   Activating the File Browser

3. Click **OK**.
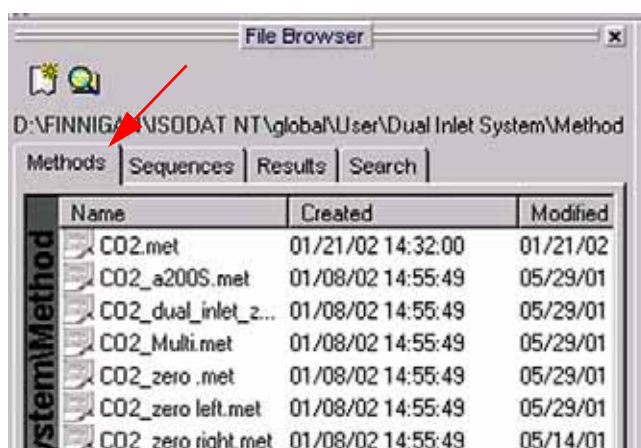
Figure 6-10.   Selecting the Method tab

4.   On the File Browser, select the Methods tab (default). See Figure 6-10.
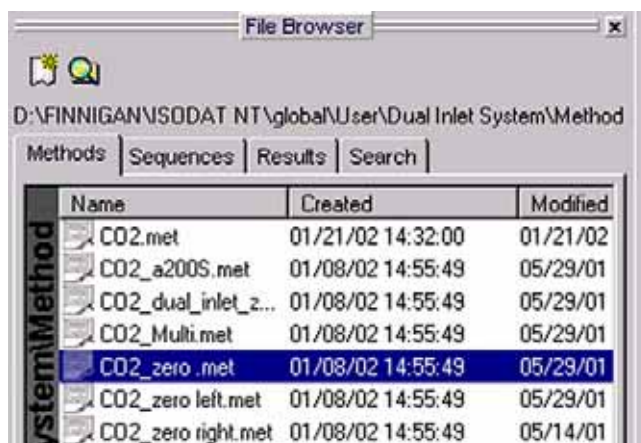
5.   From the predefined Methods, choose "CO2_zero.met" by double-click.

> **Note.** The Method "CO2_zero.met" for zero enrichment will be described here.
>
> It must be used, when both bellows are measured against each other.
>
> The Method "CO2_zero left.met" is used, when only the left bellow is measured (against itself).
>
> The Method "CO2_zero right.met" is used, when only the right bellow is measured (against itself).
>
> The three Methods only differ from the Script.

6.   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, a warning appears. See Figure 6-11 on page 6-11.

Figure 6-11.   Message box, informing of different standard values

7.   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.
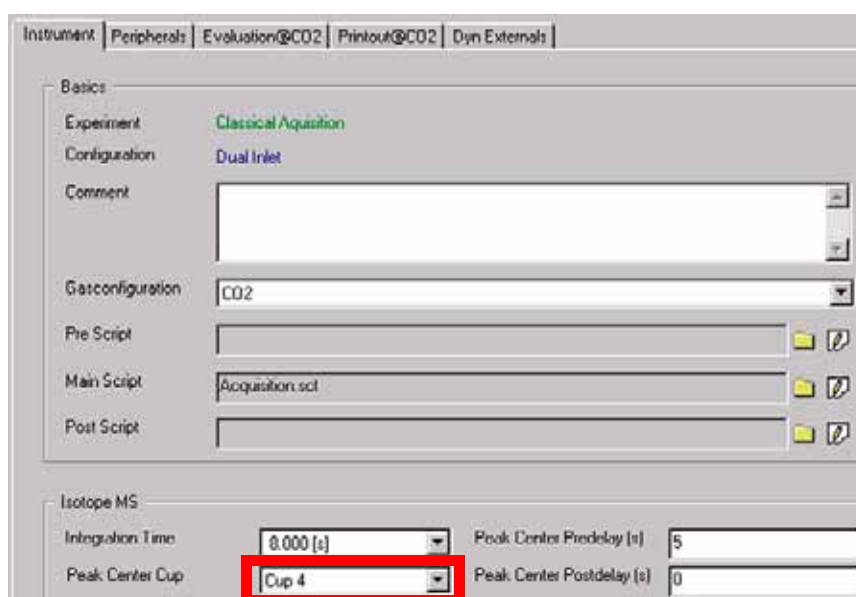
8.   The Instrument tab of the Method "CO2_zero.met" appears.

For details, see topic **Using a Predefined Method** on page 6-15.

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

At Peripherals tab, see topic **Using a Predefined Method** on page 6-15 for details.

At Evaluation CO2 tab, see topic **Using a Predefined Method** on page 6-15 for details.

9. Select **None** or **Sigma** as Outlier test.

In case of "Sigma", specify the k-fold of the standard deviation using [>>].

Note. At Std. Name, choose a suitable Standard!
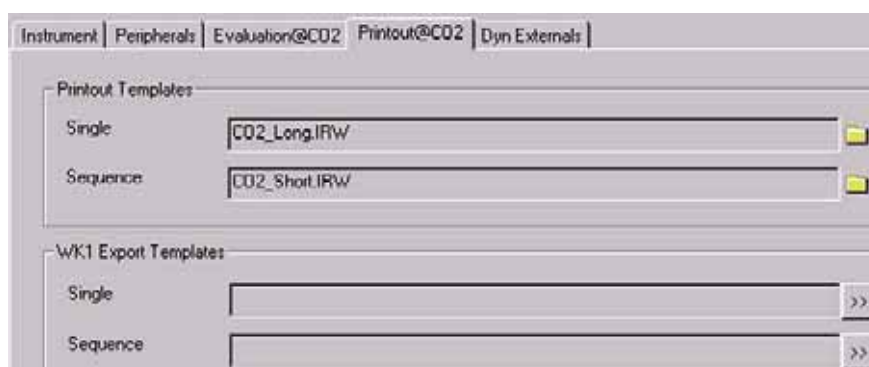
At Printout tab, see topic **Using a Predefined Method** on page 6-15 for details.

6.3 Simple Dual Inlet Measurement

Defining a Configuration

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

1. Add a new Configuration using the **Add Configuration** button.
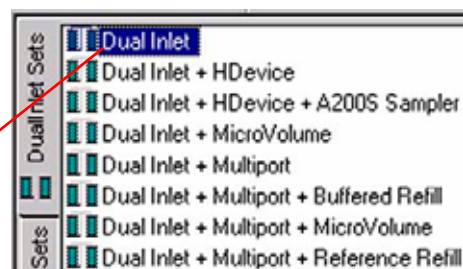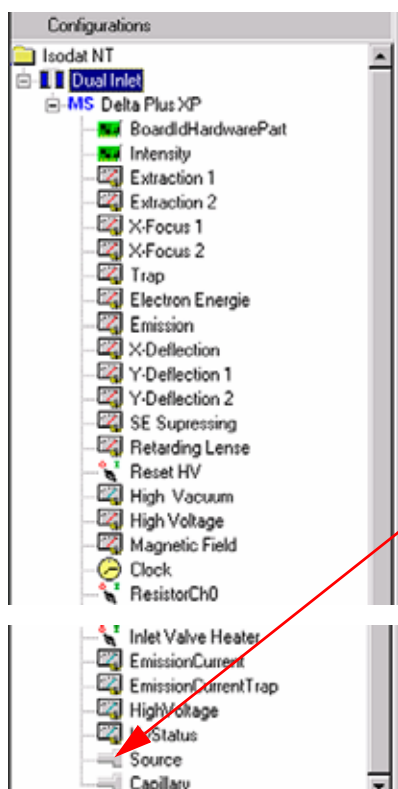
2. Give it a significant name, e.g. "Dual Inlet".

3. Open the tree structure by a click on ⊞ at ⊞ **MS** Delta Plus XP .

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

5. Drag it to the Source port of the new Configuration. A ⊞ sign appears:
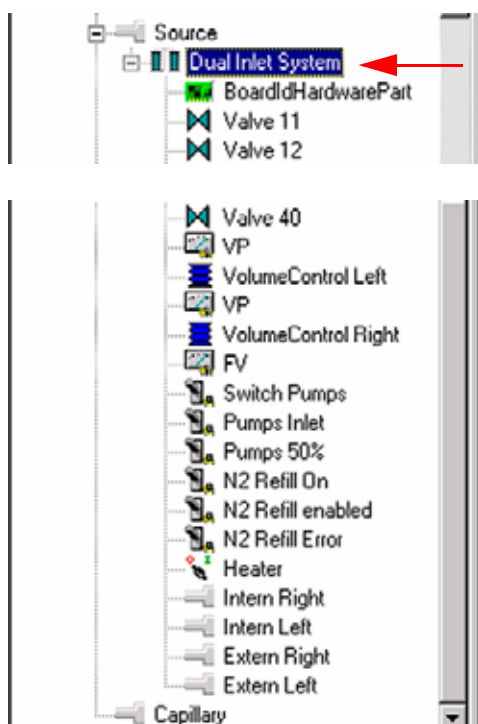
6. The Dual Inlet device has been attached to the Source.

7. Close the Configurator window.

   All settings will be saved automatically.

Using a Predefined Method

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

2. Activate the File Browser check box. See Figure 6-9 on page 6-9.

3. Click **OK**.

4. On the File Browser, select the **Methods** tab (default). See Figure 6-10
   on page 6-10.
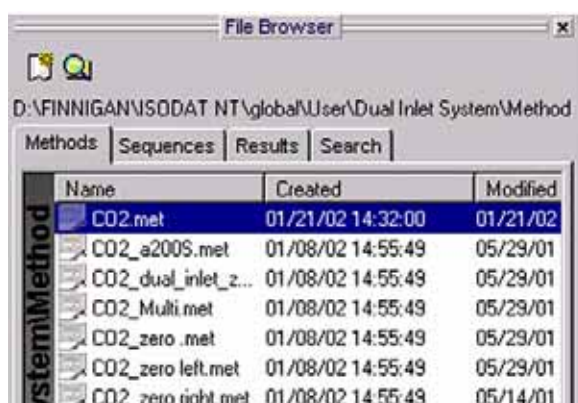
5. From the predefined Methods, choose a suitable one by double-click (e.g. "CO2.met").

6. 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. See Figure 6-11 on page 6-11.

7. 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

Figure 6-12. Instrument tab

1. Select the Gas Configuration for your determination type (e.g. "CO2").

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2. The Main Script controls the acquisition cycle.

> **Note.** It should only be edited by users trained on script editing.

Isotope MS
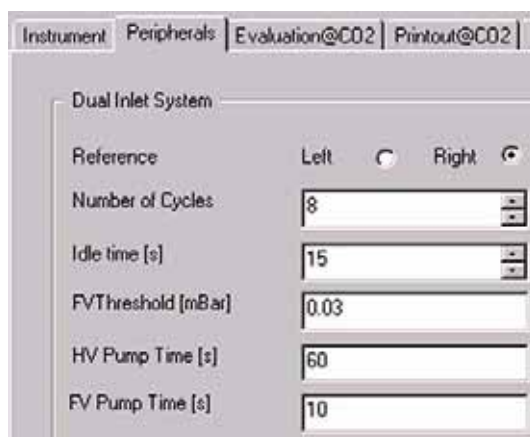
Figure 6-13. Instrument tab: Isotope MS group box

1. 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).

2. 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).

3. 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).

4. 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

Figure 6-14. Peripherals tab: Dual Inlet System group box

- 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:

St1   Sa1   St2   Sa2   St3   Sa3   St4   Sa4…..

Pre-Standard   **one cycle**   **Mean of two Standards is compared to sample between.**
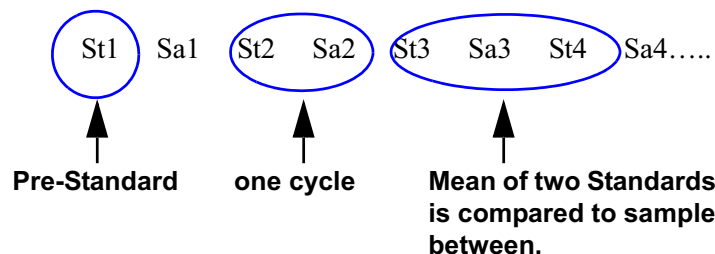
The first standard is called pre-standard (i.e. St1). The mean of two consecutive standards is calculated and compared to the flanked sample (e.g. mean of St1 and St2 is compared to Sa1).

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

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

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

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

Background

Background
Pre Delay [s]   5
Integration Cycles   1

Figure 6-15.   Peripherals tab: Background group box

- 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
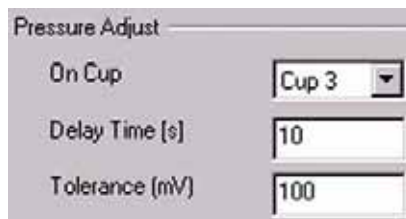
Figure 6-16.   Peripherals tab: Pressure Adjust group box

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

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

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

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

Bellow/Bellow

Figure 6-17.   Peripherals tab: Bellow / Bellow group box, with Left option selected

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

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

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

- If you select *Manual*, you can edit the pressure level (intensity) that must be reached in both bellows.
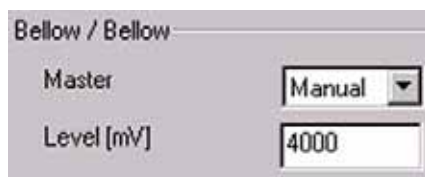
Figure 6-18.   Peripherals tab: Bellow / Bellow group box, with *Manual* option selected

Capillary/Bellow

Figure 6-19.   Peripherals tab: Capillary / Bellow group box

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

Evaluation
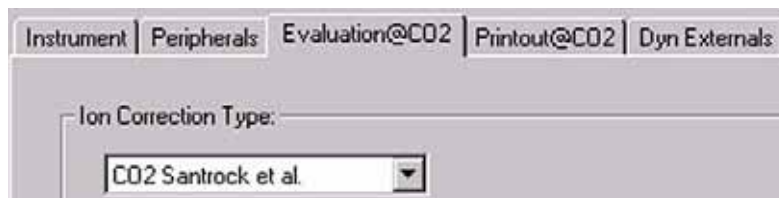
Figure 6-20.   Evaluation tab, with ion correction type *CO2 Santrock et al.* selected

1. Select an **Ion Correction** type (e.g. *CO2 Santrock et al.*).

Figure 6-21.   Evaluation tab: Outlier test group box, with option *None* selected

2. Choose the type of Outlier Test.

   (Rejection criterion for possible outliers: *None* or *Sigma*).

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• 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.

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

Figure 6-22.   Evaluation tab: Outlier test group box, with option *Sigma* selected

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

Figure 6-23.   Selecting the k-fold standard deviation

5. 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).

Figure 6-24.   Extended Parameters group box

6. Std. Name: Select a Standard Name or edit the related delta values (user defined).

Figure 6-25.   Selecting the Standard Name

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

Printout

Printout Templates

Figure 6-26.   Printout tab, showing the Printout Templates group box

*   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

Figure 6-27.   WK1 Export Templates group box

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

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 topic **Using a Predefined Sequence** on page 6-23) or create a new Sequence (refer to topic **How to Create a New Sequence** on page 6-5).

Using a Predefined Sequence

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

2. Activate the File Browser check box. See Figure 6-9 on page 6-9.

3. Click **OK**.

4. On the File Browser, select the Sequences tab (default: Methods tab). See Figure 6-10 on page 6-10.

5. From the predefined Sequences, choose a suitable one by double-click (e.g."CO2.seq").
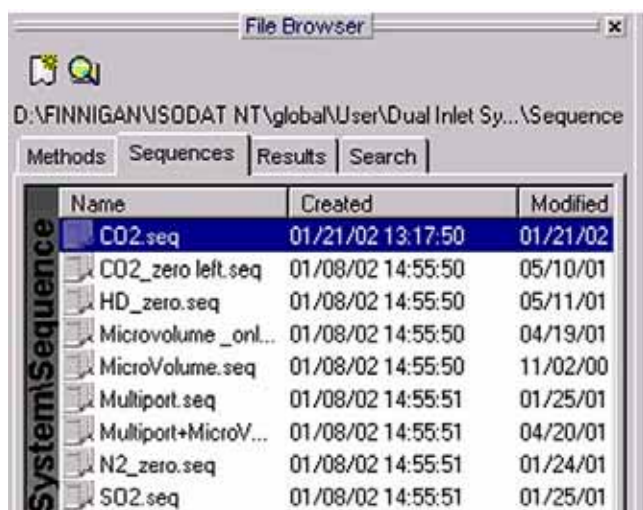
6. In the "Method" column, the Method chosen in topic **Using a Predefined Method** occurs as default (*CO2.met*).

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.

7. Press the **Start** button.
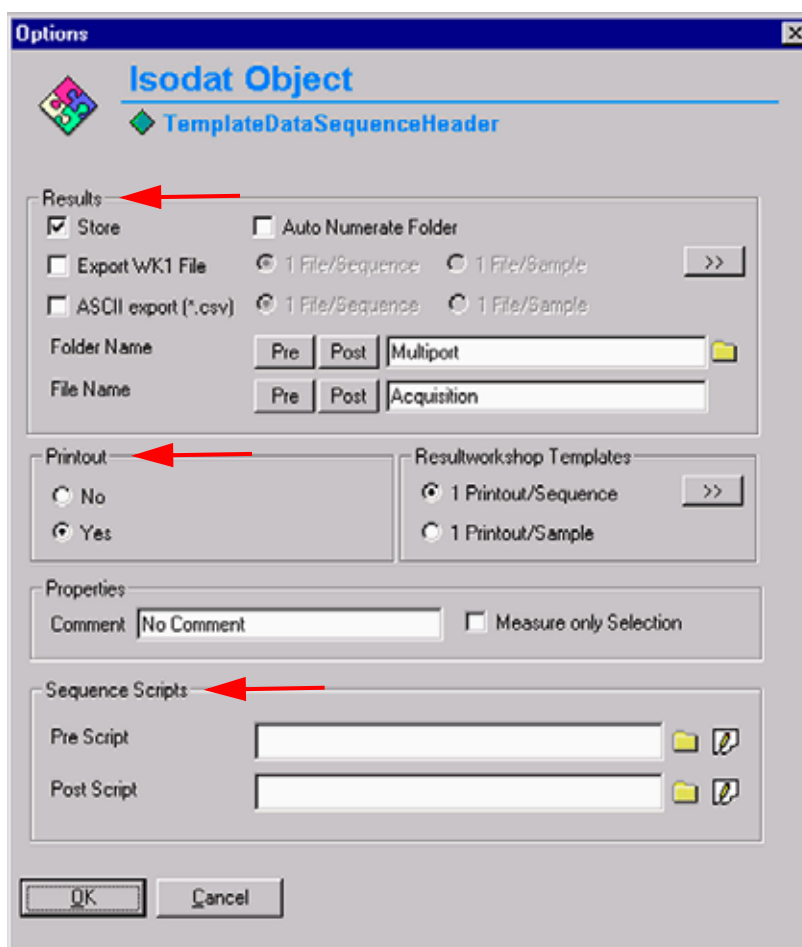
Figure 6-28.   Editing the Sequence Header

8. Define parameters for:

   - Results Export

   - Printout

   - Sequence Scripts (See Figure 6-28.)

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

6.4 Dual Inlet Measurement Including Multiport

Defining a Configuration

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

1. Add a new Configuration using the **Add Configuration** button.
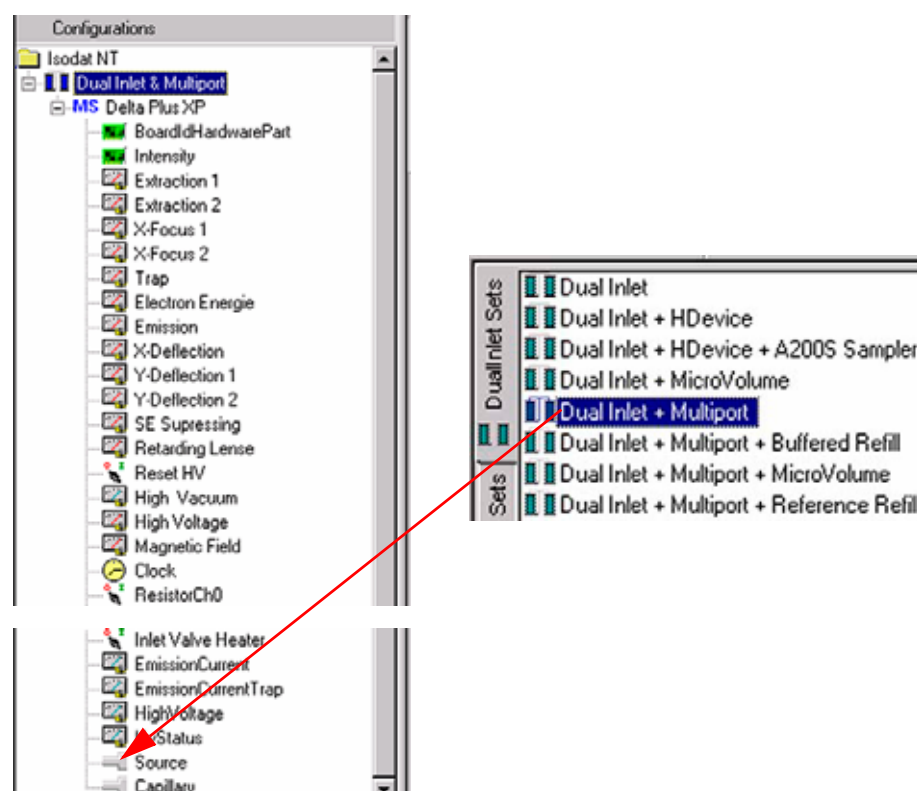
2. Give it a significant name, e.g. "Dual Inlet & Multiport".

3. Open the tree structure by a click on [+] at [+] **MS** Delta Plus XP.

4. On the right pane, select Dual Inlet + Multiport.

5. Drag it to the Source port   of the new
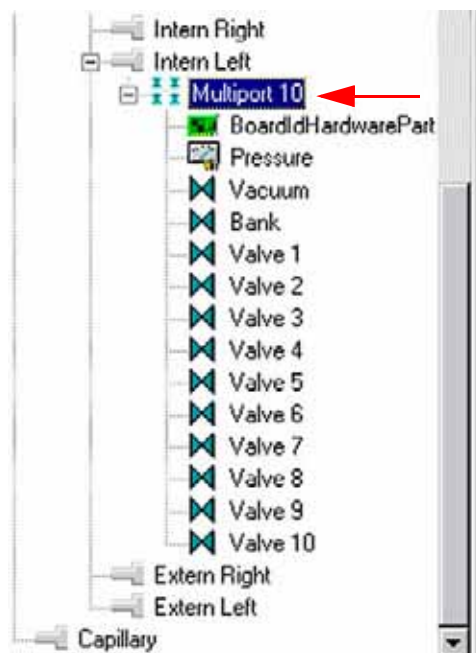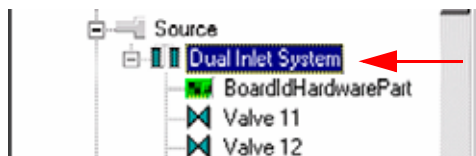
    Configuration. A ⊞ sign appears:

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

7. The Multiport has been attached to the Intern Left Port.

8. Close the Configurator window.

    All settings will be saved automatically.

Using a Predefined Method

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

2. Activate the File Browser check box. See Figure 6-9 on page 6-9.

3. Click **OK**.

4. On the File Browser, select the Methods tab (default). See Figure 6-10 on
    page 6-10.
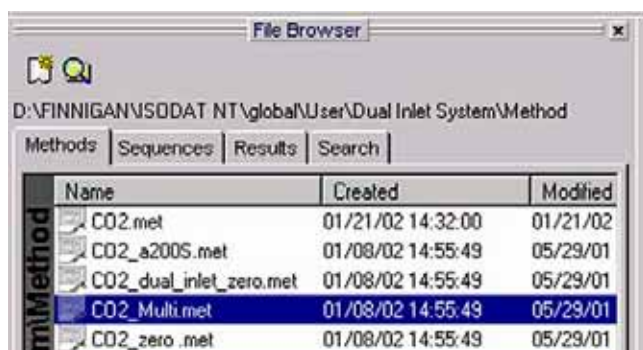
5. From the predefined Methods, choose "CO2_multi.met" by double-click.

6. 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, a warning appears. See Figure 6-11 on page 6-11.

7. 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.

8. The Instrument tab of the Method "CO2_multi.met" appears.
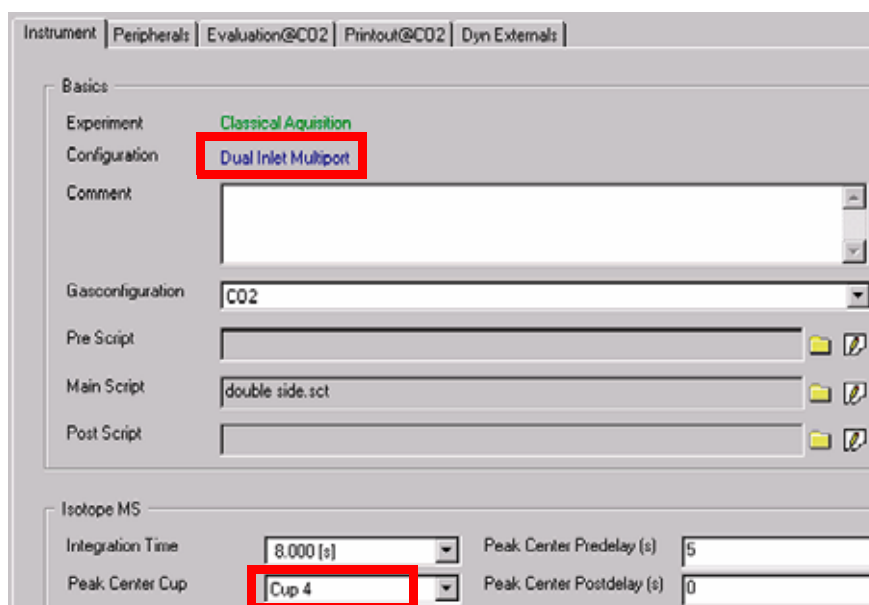
For details, see topic **Using a Predefined Method** on page 6-15.

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

9. See topic **Using a Predefined Method** on page 6-15 for details at Peripherals tab.

Figure 6-29.   Peripherals tab for measurements including Multiport

> **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.

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10. At Evaluation CO2 tab, see topic **Using a Predefined Method** on page 6-15 for details.

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

> **Note.** At Std. Name, choose a suitable Standard!

12. At Printout tab, see topic **Using a Predefined Method** on page 6-15 for details.

Figure 6-30. Printout tab with settings for measurements using Dual Inlet in combination with a Multiport

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 topic **Using a Predefined Sequence** below) or create a new Sequence (refer to topic **How to Create a New Sequence** on page 6-5).

Using a Predefined Sequence

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

2. Activate the File Browser check box. See Figure 6-9 on page 6-9.

3. Click **OK**.
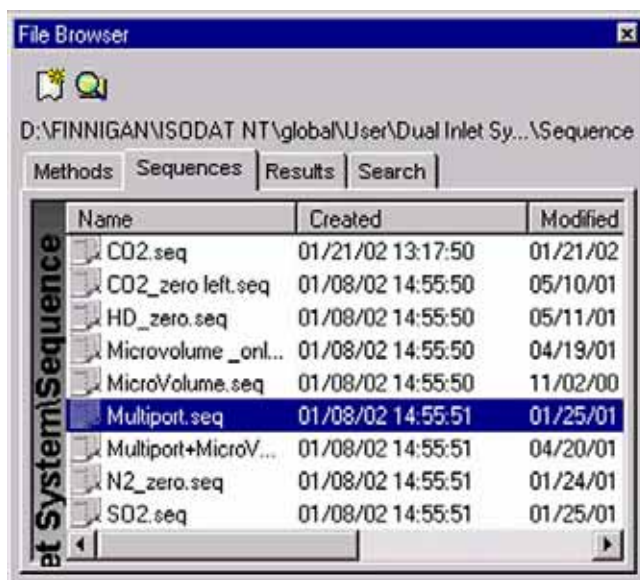
Figure 6-31. Sequences tab, with Multiport.seq selected

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

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

6. In the "Method" column, the Method chosen in topic **Using a Predefined Method** on page 6-26 occurs as default ("CO2_Multi.met").

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

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

7. Press the **Start** button.

8. Define parameters for:

   • Results Export

   • Printout

   • Sequence Scripts (See Figure 6-28 on page 6-24.)

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

6.5 Dual Inlet Measurement Including Microvolume

Microvolume - Measurement Principle

Microvolume without Multiport - Measurement Principle
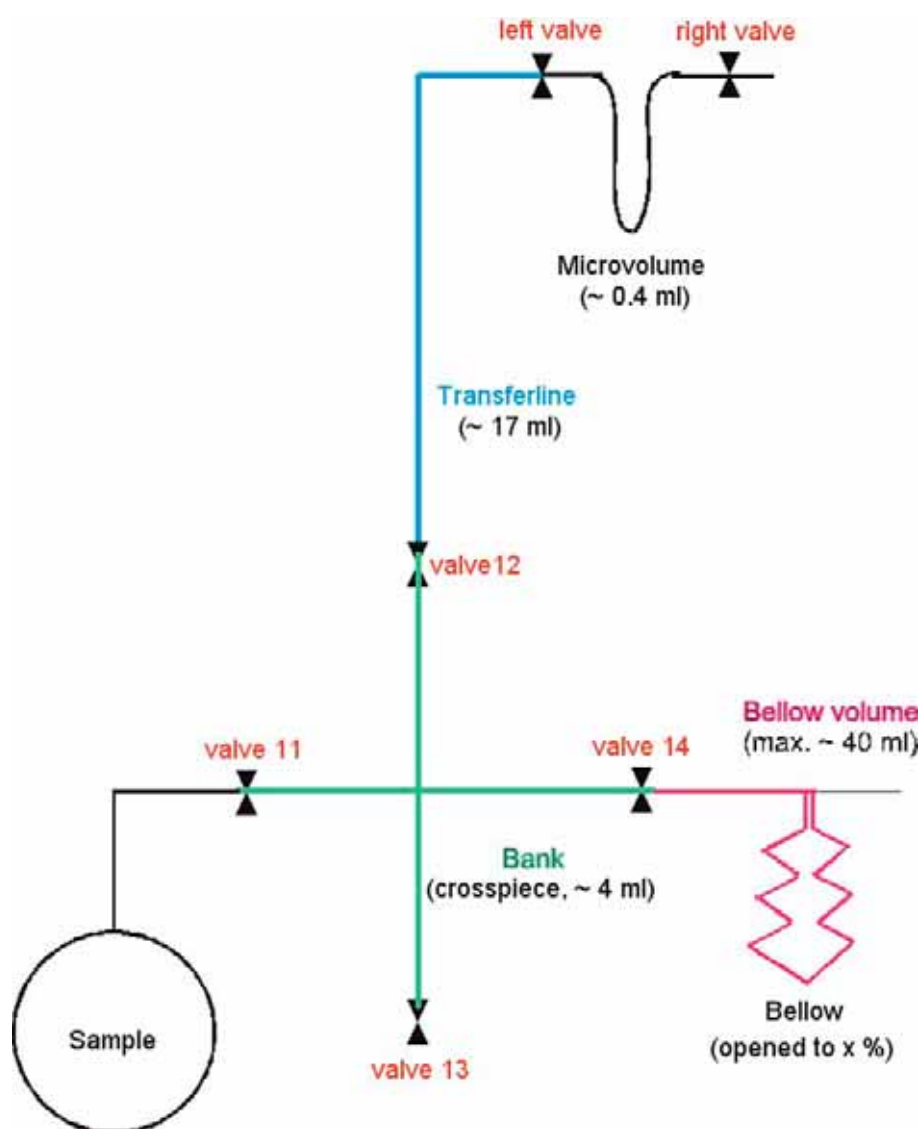
Figure 6-32.   Microvolume without Multiport - Measurement Principle

Microvolume via Multiport - Measurement Principle
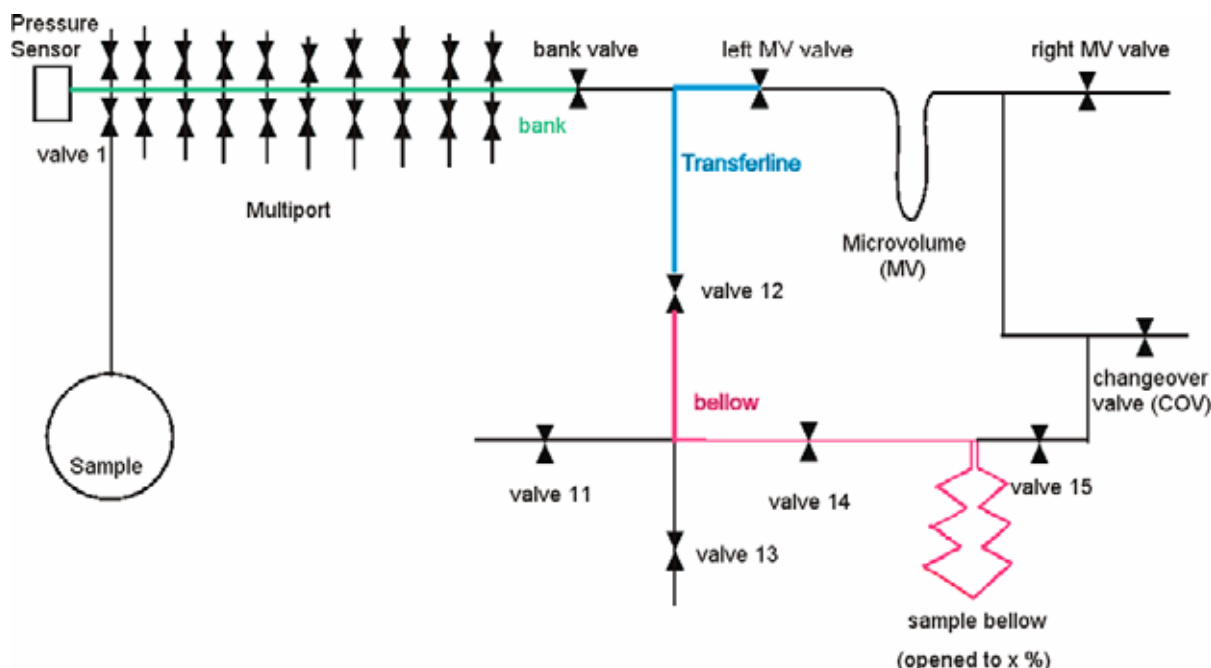
Figure 6-33.   Microvolume via Multiport - Measurement Principle

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

*   **Expansion** mode
*   **Freeze Direct** mode

Figure 6-34.   Measuring modes for Microvolume via Multiport

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% (that is Bellow Pos. [%]).

Figure 6-35. Options for Expansion mode

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 Acquisition.

6. After starting the Acquisition, the bellow of the Sample side (i.e. mostly left) will be opened fully, that is 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. 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 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 (that is, sensor).

Expansion Mode - Measurement with Bank

Figure 6-36.   Expansion Mode - Measurement with Bank

1. The pressure p is measured using a pressure-meter (that is, sensor).

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

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

4. The gas in the bank volume is pumped off.

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

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

7. The pumping time depends on the three parameters:

  • FV Threshold [mbar]

  • HV Pump Time [s]

  • FV Pump Time [s]

Figure 6-37. Parameters for pumping time

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

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

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

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

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Expansion Mode - Measurement with Bank and Transferline

Figure 6-38. Expansion Mode - Measurement with Bank and Transferline
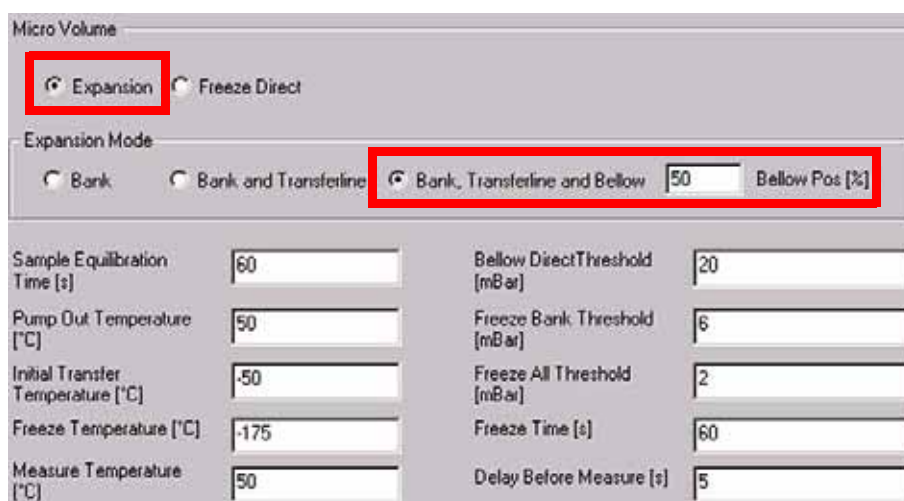
The working principle is the same as described at "Expansion Mode - Bank". Only the route of the sample gas is longer.

Example

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

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

Expansion Mode - Measurement with Bank, Transferline and Bellow

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

Figure 6-39. Expansion Mode - Measurement with Bank, Transferline and Bellow

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 (that is, the whole volume is filled with gas).

5. The pressure p is measured.

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

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

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

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

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, notice that the fields related to Expansion mode are inactive. See Figure 6-40.

Figure 6-40.   Settings for Freeze Direct Mode

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.

Defining a Configuration

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

1. Add a new Configuration using the **Add Configuration** button.
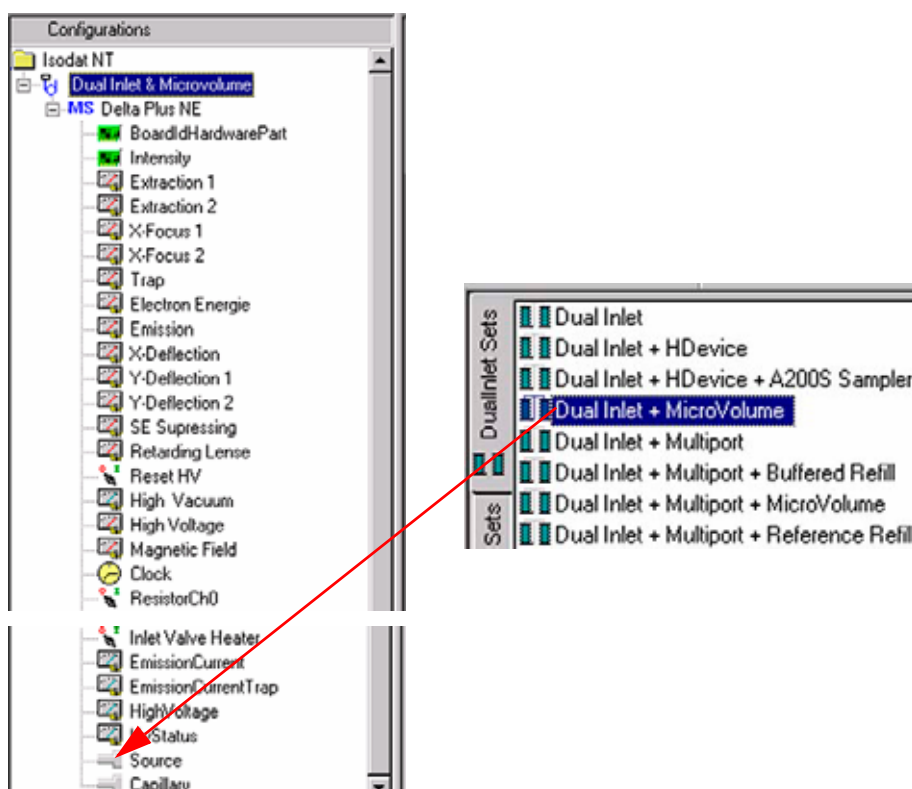
2. Give it a significant name, e.g. "Dual Inlet & Microvolume".

3. Open the tree structure by a click on at .

4. On the right pane, select "Dual Inlet + Microvolume".

5. Drag it to the Source port of the new Configuration. A sign appears:
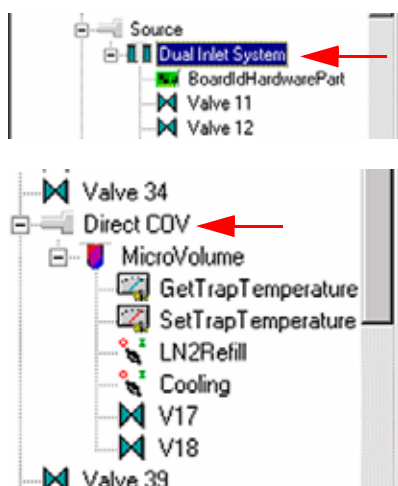
6. The Dual Inlet device has been attached to the Source.

7. The Microvolume has been attached to the Direct COV Port.

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

Using a Predefined Method

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

2. Activate File Browser check box. See Figure 6-9 on page 6-9. Click **OK**.

3. On File Browser, select **Methods** tab. See Figure 6-10 on page 6-10.
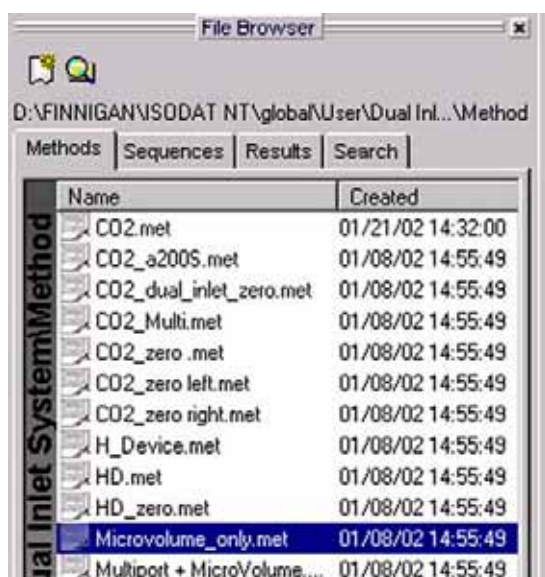
4. Among the predefined Methods, choose **Microvolume_only.met**.

5. 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, a warning appears. See Figure 6-11 on page 6-11.

6. 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.

7. The Instrument tab of the Method **Microvolume_only.met** appears.
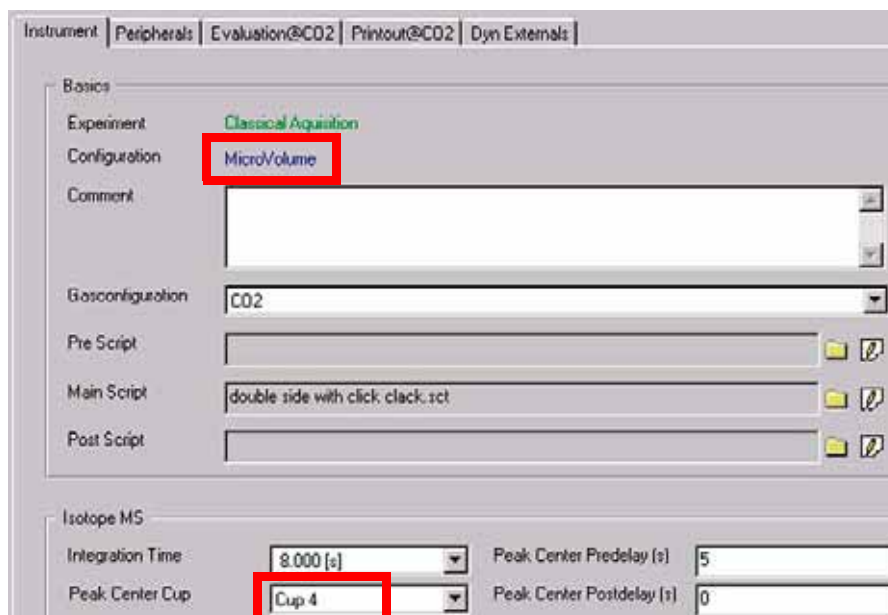
For details see topic **Using a Predefined Method** on page 6-26.

> **Note.** Ensure, that the proper peak center cup is selected. If necessary, correct the default cup (that is Cup 4)!

For details at the Peripherals tab's Dual Inlet part, see topic **Using a Predefined Method** on page 6-26.

Figure 6-41.   Peripherals tab: Dual Inlet part
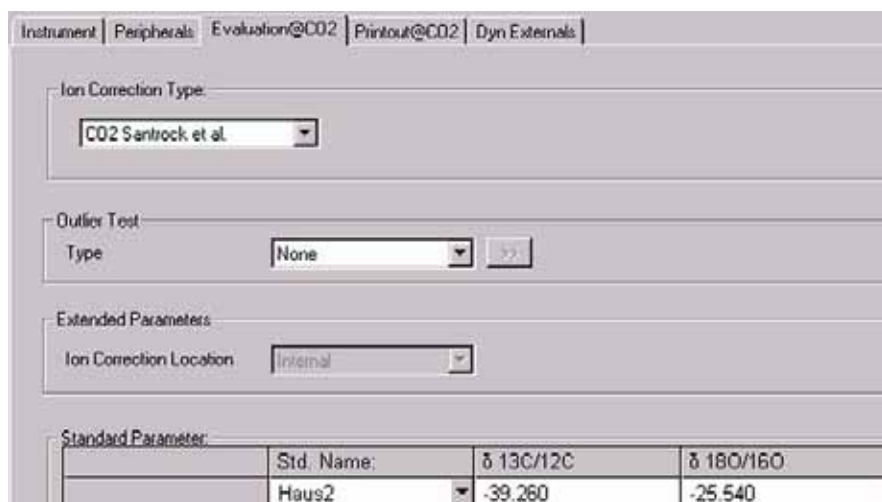
For details about the Peripherals tab's Microvolume part, see topic **Using a Predefined Method** on page 6-26.

Figure 6-42.   Peripherals tab: Microvolume part

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

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

   For details, see **Using a Predefined Method** on page 6-26.

   > **Note.** At Std. Name, choose a suitable Standard!

   For details at Printout tab, see **Using a Predefined Method** on page 6-26.
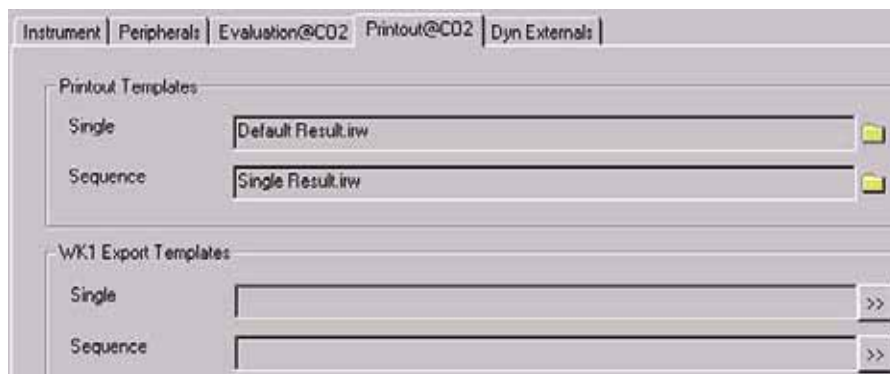
Figure 6-43.   Printout tab with settings for Microvolume

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 **Using a Predefined Sequence** on page 6-46) or create a new Sequence (refer to **How to Create a New Sequence** on page 6-5).

Using a Predefined Sequence

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

2. Activate the File Browser check box. See Figure 6-9 on page 6-9.

3. Click **OK**.

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

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

6. In the "Method" column, the Method chosen in topic **Using a Predefined Method** on page 6-42 occurs as default ("CO2_Multi.met").

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

7. Press the Start button.

8. Define parameters for:

    *   Results Export

    *   Printout

    *   Sequence Scripts (see Figure 6-28 on page 6-24.)

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

6.6   Dual Inlet Measurement Including Multiport and Microvolume

Defining a Configuration

Configurator

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

1. Add a new Configuration using the **Add Configuration** button.
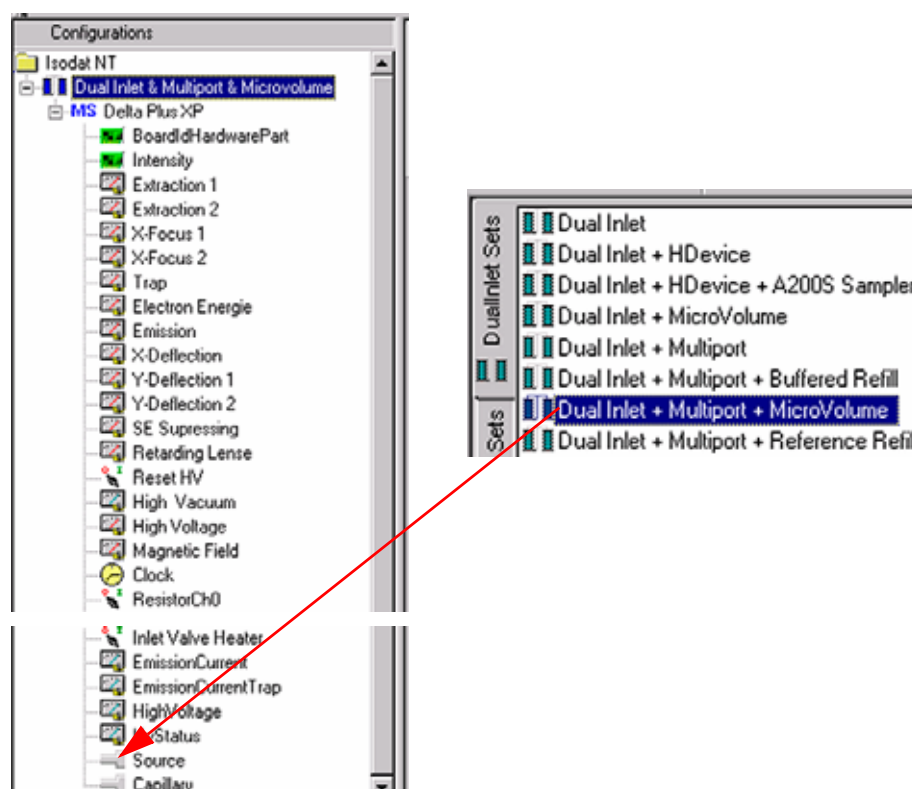
2. Give it a significant name, e.g. "Dual Inlet & Multiport & Microvolume".

3. Open the tree structure by a click on 🖃 at 🖃 **MS** Delta Plus XP .

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

5. Drag it to the Source port [image] of the new
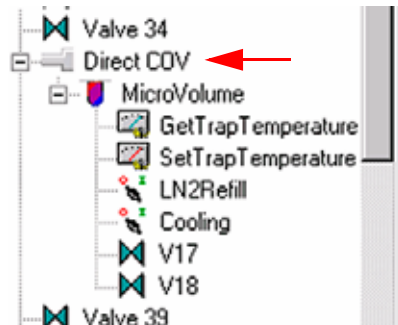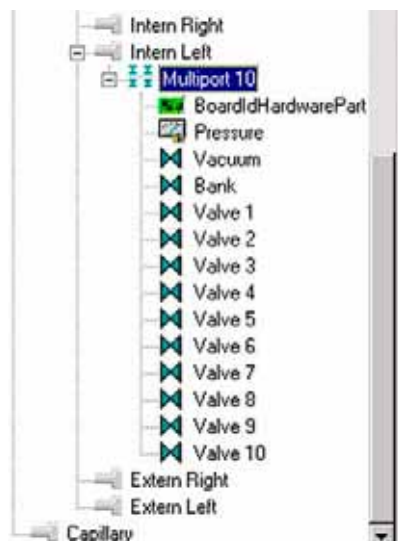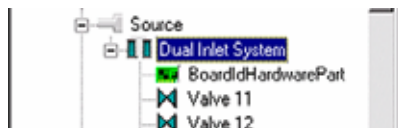
   Configuration. A [+] sign appears:

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

7. The Multiport has been attached to the Intern Left Port.

8. The Microvolume has been attached to the Direct COV Port.

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

Using a Predefined Method

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

2. Activate File Browser check box. See Figure 6-9 on page 6-9. Click **OK**.

3. On File Browser, select **Methods** tab. See Figure 6-10 on page 6-10.

4. From the predefined Methods, choose **Multiport + Microvolume.met**.

5. 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, a warning appears. See Figure 6-11 on page 6-11.

6. 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.

7. Instrument tab of the Method "Multiport + Microvolume.met" appears.

For details, see topic **Using a Predefined Method** on page 6-15.

> **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 topic **Using a Predefined Method** on page 6-26.

For details about the Peripherals tab's Microvolume part, see **Using a Predefined Method** on page 6-26.

For details at the Peripherals tab's Multiport part, see topic **Using a Predefined Method** on page 6-26.

Figure 6-44.   Peripherals tab: Multiport part

8.   At Evaluation CO2 tab, select an Ion Correct type, e.g. *CO2 Santrock et al.*

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

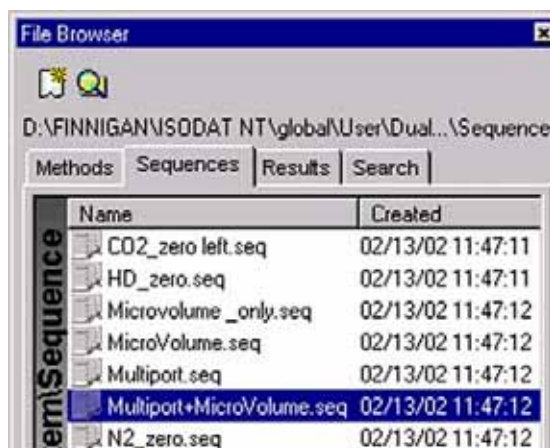For details see topic **Using a Predefined Method** on page 6-15.

> **Note.** At Std. Name, choose a suitable Standard!

For details at Printout tab, see **Using a Predefined Method** on page 6-15.

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 **Using a Predefined Sequence** on page 6-52) or create a new Sequence (refer to **How to Create a New Sequence** on page 6-5).

Using a Predefined Sequence

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

2. Activate File Browser check box. See Figure 6-9 on page 6-9. Click **OK**.

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

4. From the predefined Sequences; choose "Multiport + Microvolume.seq".

5. In **Method** column, the Method chosen in topic **Using a Predefined Method** on page 6-49 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 **Using a Predefined Sequence** on page 6-23.

6. Press the Start button.

7. Define parameters for:

   • Results Export

   • Printout

   • Sequence Scripts (See Figure 6-28 on page 6-24.)

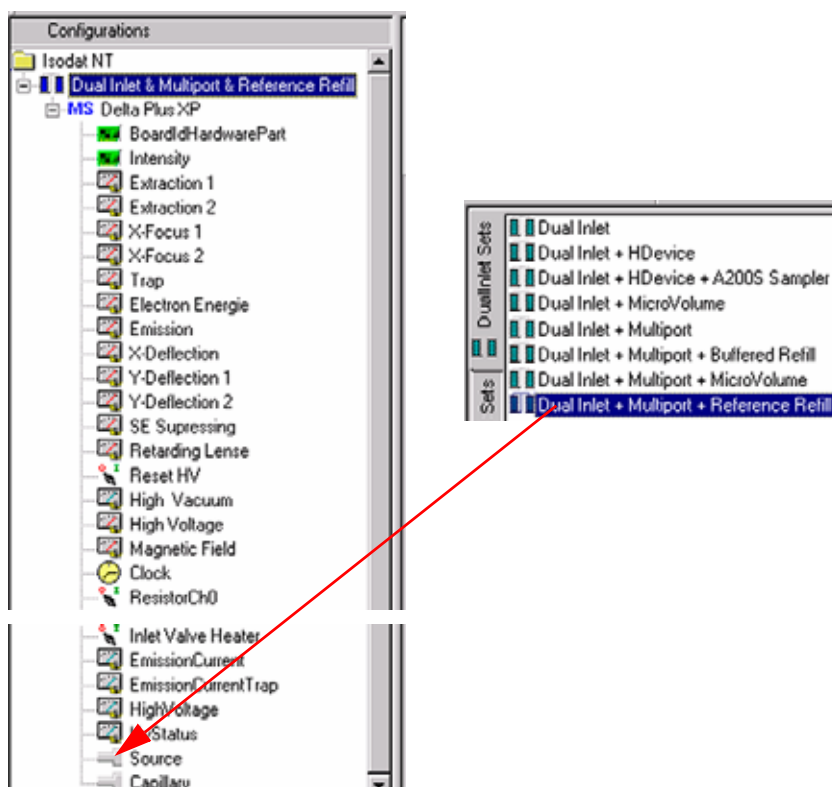8. Press **OK** to start Sequence Acquisition.

6.7 Dual Inlet Measurement Including Multiport and Reference Refill

Defining a Configuration

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

Configurator

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

2. Give it a name, e.g. "Dual Inlet & Multiport & Reference Refill".

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

4. On the right pane, select Dual Inlet + Multiport + Reference Refill.

5. Drag it to Source port of the new Configuration. A ⊞ appears:
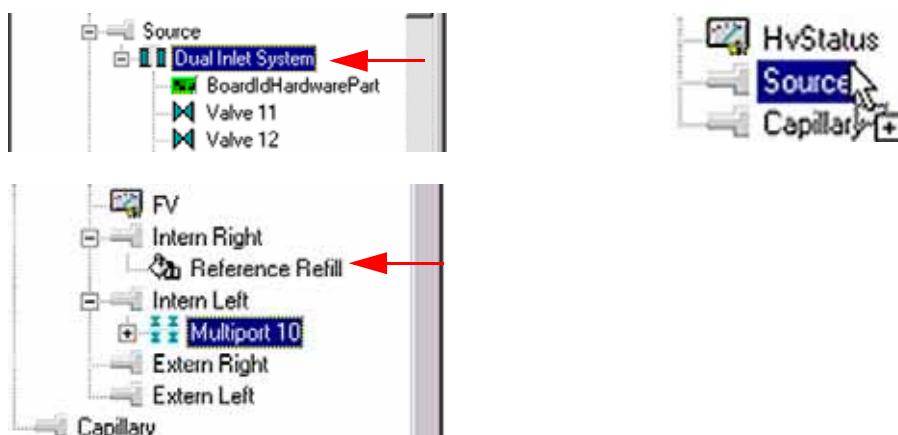
6. The Dual Inlet device has been attached to the Source.

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

   It is always connected to the right side (that is, the Standard side) - internal or external.

8. The Multiport has been attached to the Intern Left Port.

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

Using a Predefined Method

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

2. Activate File Browser check box. See Figure 6-9 on page 6-9. Click **OK**.

3. On File Browser, select **Methods** tab. See Figure 6-10 on page 6-10.

4. From the predefined Methods, choose a suitable Method (e.g. "Multiport + Reference Refill.met").
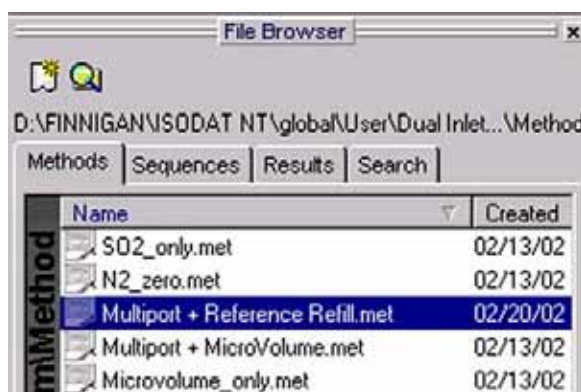
5. If the Standards in the Method to be opened are older than the actual ones in the Standard Database, or if the user has changed the Method, the warning beside appears. See Figure 6-11 on page 6-11.

6. 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.
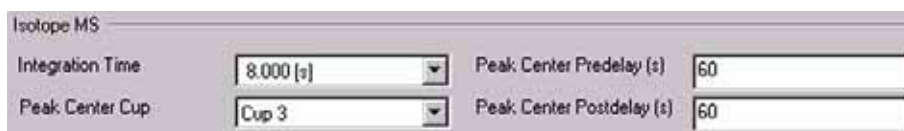
Instrument tab

The Instrument tab of the Method "Multiport + Reference Refill.met" appears.
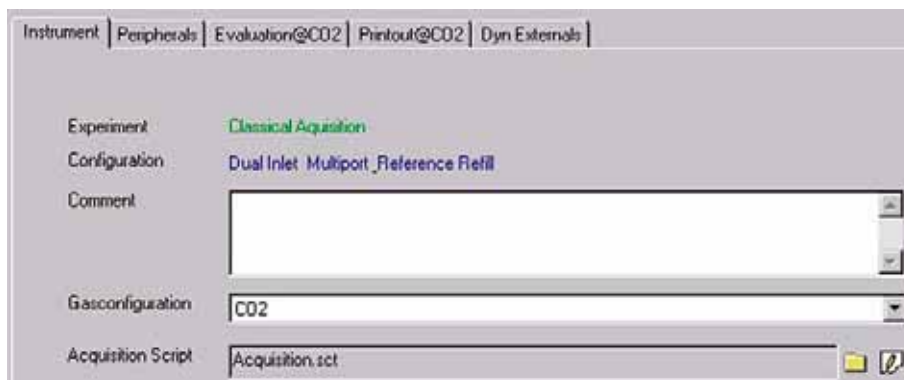
1. As Gasconfiguration, select *CO2*.

2. The Main Script controls the acquisition cycle.

> **Note.** It should only be edited by users trained on script editing.

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

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

Pump Overlay Time: Capillary pump out time of Reference Refill tank.

FV Threshold: Minimum pressure of standard bellow including the valves and tubes evacuated using fore vacuum pump before continuing pumping using turbo molecular pump.

Refill Time: Gas flow time from Reference Refill tank into inlet port of standard bellow.

HV Pump Time: Pumping time of bellow including the valves and tubes using turbo molecular pump.

Peripherals tab

For details, see topic **Using a Predefined Method** on page 6-15.

Evaluation tab

1. Select the Ion Correction Type (e.g. "CO2 Santrock et al.").

2. As Outlier test, select *None* or *Sigma*. In case of *Sigma*, specify the k-fold

    of the standard deviation using [>>]. For details, see topic **Using a
    Predefined Method** on page 6-15.

3. At Std. Name, choose a suitable Standard.

Printout tab

For details, see topic **Using a Predefined Method** on page 6-15.

The Method for measurements using Dual Inlet in combination with a Multiport and a Reference Refill has been established. As next step, either use a predefined Sequence (see **Using a Predefined Sequence** on page 6-59) or create a new Sequence (see **How to Create a New Sequence** on page 6-5).

Using a Predefined Sequence

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

2. Activate File Browser check box. See Figure 6-9 on page 6-9. Click **OK**.

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

4. From the predefined Sequences, choose "Multiport + Reference Refill.seq".

5. In **Method** column, the Method chosen in **Using a Predefined Method** on page 6-55 occurs as default ("Multiport + Reference Refill.met").

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	▼

6. 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.

7. 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.

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

9. Press the **Start** button.

10. Define parameters for:

   • Results Export

   • Printout

   • Sequence Scripts (See Figure 6-28 on page 6-24.)

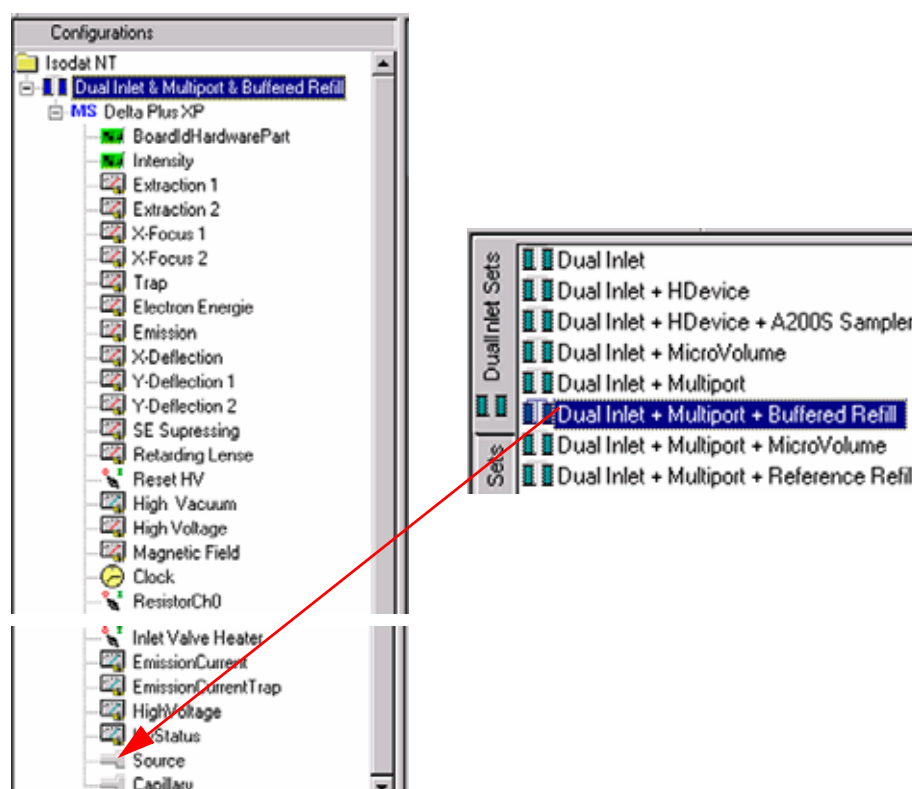11. Press **OK** to start Sequence Acquisition.

6.8   Dual Inlet Measurement Including Multiport and Buffered Refill

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.

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

2.   Give it a name, e.g. "Dual Inlet & Multiport & Buffered Refill".

3.   Open the tree structure by a click on ⊞ at ⊞·**MS** Delta Plus XP .

4.   On the right pane, select Dual Inlet + Multiport + Buffered Refill.

5. Drag it to the Source port [icon] of the new

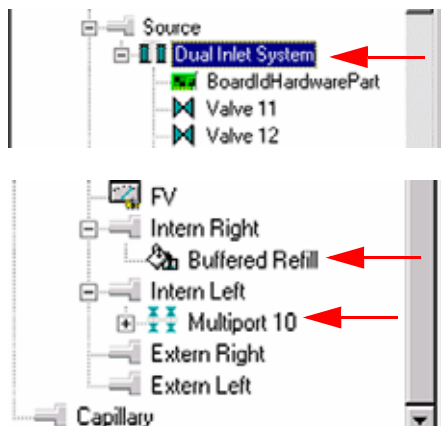   Configuration. A [+] sign appears:

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

7. The Buffered Refill device has been attached to the Intern Right Port.

8. The Multiport device has been attached to the Intern Left Port.

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

Using a Predefined Method

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

2. Activate File Browser check box. See Figure 6-9 on page 6-9. Click **OK**.

3. On File Browser, select Methods tab. See Figure 6-10 on page 6-10.

4. From the predefined Methods, choose a suitable Method (e.g. "Multiport + Buffered Refill.met").
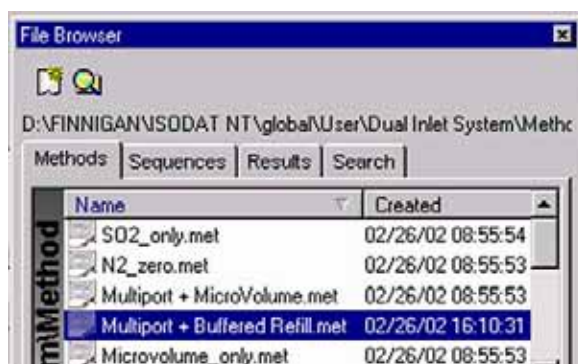
5. If the Standards in the Method to be opened are older than the actual ones in the Standard Database, or if the user has changed the Method, the warning beside appears. See Figure 6-11 on page 6-11.

6. 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.
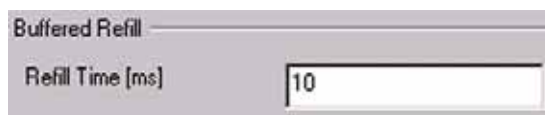
Instrument tab

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

1. As **Gasconfiguration**, select *CO2*.

2. The **Main Script** controls the acquisition cycle.

> **Note.** It should only be edited by users trained on script editing.

Integration time:	The time needed to measure each individual ion intensity of m/z 44, m/z 45 and m/z 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).
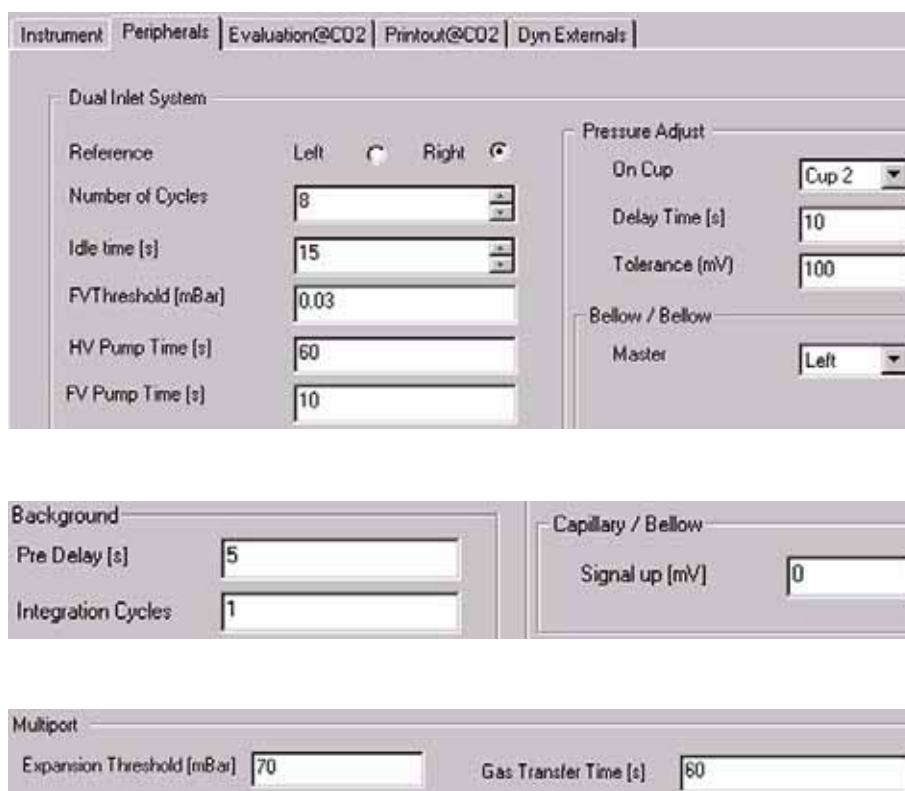
Figure 6-45.   Buffered Refill group box

> **Note.** The above information is specific for Buffered Refill applications.

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

Peripherals tab

For details, see topic **Using a Predefined Method** on page 6-15.

Evaluation tab

1. Select the Ion Correction Type (e.g. "CO2 Santrock et al.").

2. As Outlier test, select *None* or *Sigma*. In case of *Sigma*, specify the k-fold
    of the standard deviation using ⟩⟩ . For details, see topic **Using a
    Predefined Method** on page 6-15.

3. At Std. Name, choose a suitable Standard.

Printout tab

For details, see topic **Using a Predefined Method** on page 6-15.

The Method for measurements using Dual Inlet in combination with a Multiport and a Buffered Refill has been established. As next step, either use a predefined Sequence (see **Using a Predefined Sequence** on page 6-66) or create a new Sequence (see **How to Create a New Sequence** on page 6-5).

Using a Predefined Sequence

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

2. Activate File Browser check box. See Figure 6-9 on page 6-9. Click **OK**.

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

4. From the predefined Sequences, choose Multiport + Buffered Refill.seq.

5. In **Method** column, the Method chosen in **Using a Predefined Method** on page 6-62 occurs as default ("Multiport + Buffered Refill.met").

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	▼

6. 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.

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7. 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.

8. If a Buffered Refill is to be performed, the ⬛ column must be activated by ✓ for the particular sample (e.g. for sample 1).

9. Press the Start button.

10. Define parameters for:
    • Results Export
    • Printout
    • Sequence Scripts (see Figure 6-28 on page 6-24.)

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

6.9 Dual Inlet Measurement Including H-Device and Autosampler A200S

Defining a Configuration

Configurator

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

1. Add a new Configuration using the **Add Configuration** button.
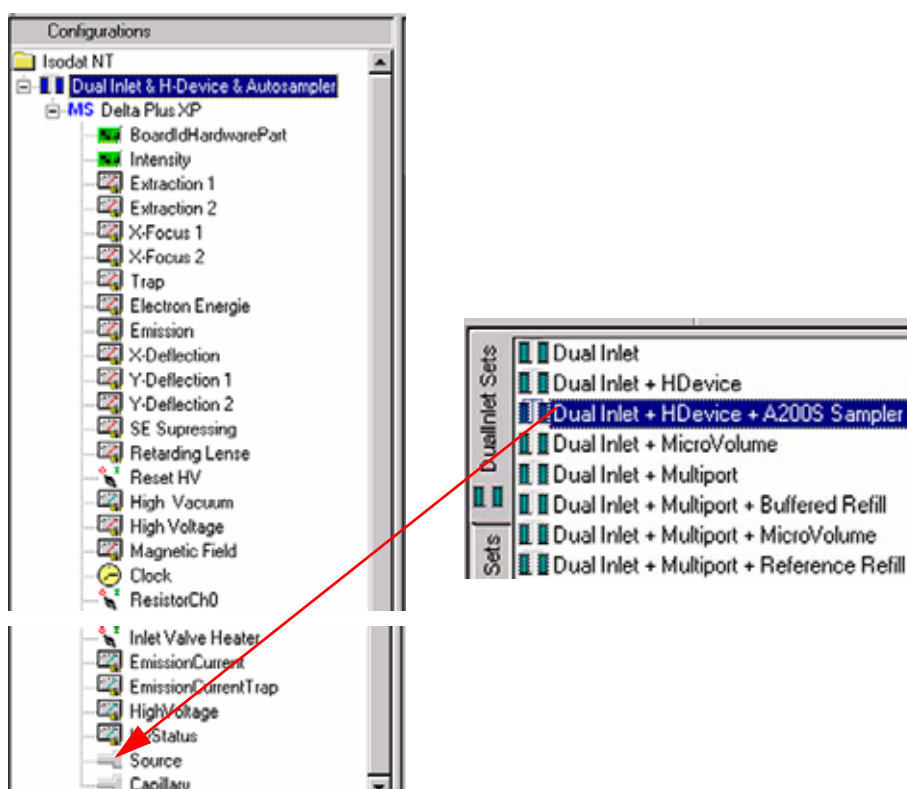
2. Give it a significant name, e.g. "Dual Inlet & HDevice & Autosampler".

3. Open the tree structure by a click on [+] at [+] MS Delta Plus XP .

4. On the right pane, select Dual Inlet + HDevice + A200S Sampler.

5. Drag it to the Source port   of the new

Configuration. A ⊞ sign appears:
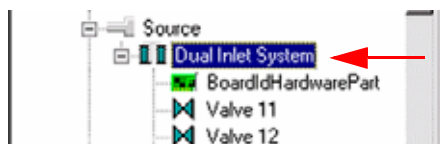
6. The Dual Inlet device has been attached to the Source.

7. The H-Device has been attached to the Extern Left Port.

8. The A200S Autosampler has been attached to the Sampler Port.

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

Using a Predefined Method

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

2. Activate File Browser check box. See Figure 6-9 on page 6-9. Click **OK**.

3. On the File Browser, select the **Methods** tab (default). From the predefined Methods, choose a suitable one, e.g. "H_Device + A200S.met".

4. 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. See Figure 6-11 on page 6-11.

5. 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.

1. As **Gasconfiguration**, select *CO2*.

2. The **Main Script** controls the acquisition cycle.

> **Note.** It should only be edited by users trained on script editing.

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

Peripherals tab

For details, see topic **Using a Predefined Method** on page 6-15.

> **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 expands into the intermediate or expansion volume (connecting metal tube plus valve body). An equilibration time of 30 s is recommended for 800 ºC to 850 ºC. For other temperatures, this must be tested empirically.

Evaluation tab
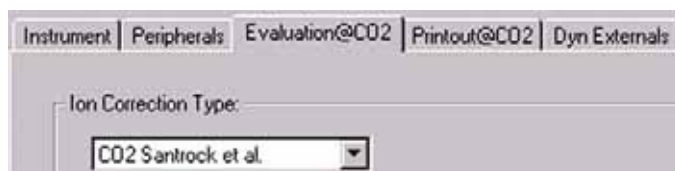
1. Select the Ion Correction Type (e.g. "CO2 Santrock et al.").
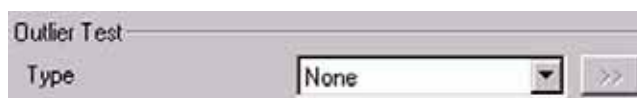
2. As Outlier test, select *None* or *Sigma*. If *Sigma*, specify k-fold of standard deviation using [>>]. See **Using a Predefined Method** on page 6-15.
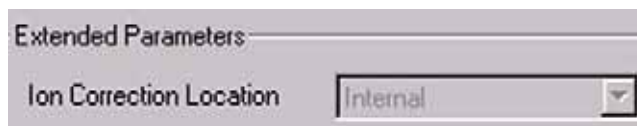
3. At Std. Name, choose a suitable Standard.

Printout tab

For details, see topic **Using a Predefined Method** on page 6-15.

The Method for measurements using Dual Inlet in combination with a H-Device and an A200S Autosampler has been established. Now, either use a predefined Sequence (see **Using a Predefined Sequence** on page 6-73) or create a new Sequence (see **How to Create a New Sequence** on page 6-5).

Using a Predefined Sequence

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

2. Activate File Browser check box. See Figure 6-9 on page 6-9. Click **OK**.

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

4. From the predefined Sequences, choose a suitable one (e.g. H_Device + A200S.seq) by double-click.

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

a. AS Sample: Type in the sample position in the Autosampler tray.

b. AS Method: Select Autosampler Method.

c. Identifier 1: Edit text to identify the sample.

d. Method: Select IRMS Method. The Method chosen in topic **Using a Predefined Method** on page 6-69 occurs as default ("H_Device + A200S.met").

5. Press the **Start** button.

6. Define parameters for:

   • Results Export

   • Printout

   • Sequence Scripts (See Figure 6-28 on page 6-24.)

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

6.10 Fundamental Procedures before any Measurement

Before starting any acquisition make sure, the following procedures have been performed:

1. Electronic offset has been measured and saved.

2. All peripherals have been configured.

3. The Mass Calibration for all gases to be measured has been performed.

4. In case of a Dual Inlet application, the Bellow Calibration has been performed.

5. In case of hydrogen collectors, the H3 factor for Continuous Flow and Dual Inlet application has been determined.

6. In case of Elemental Analyzer application for C/N or H/O analysis, the Jump Calibration has been performed and saved.

7. In case of a Liquid Autosampler, the COM port and the other parameters are set.

6.11   Handling of Results

Toolbar Commands

After the measurement has been started by [Start], a classical acquisition file, that is *.caf, is generated (e.g. "Acquisition-7.caf"). The Acquisition window opens. It contains the toolbar shown below (Figure 6-46) with the following icons:
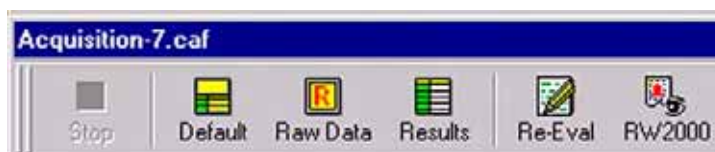
Figure 6-46.   Toolbar of the Acquisition window

- A running Dual Inlet data acquisition can be stopped.

- The data are shown both in a data grid and graphically (i.e. both in medium size).

- The graphical representation of the raw data is shown in maximum size (i.e. without data grid).

- The result grid is shown in maximum size (i.e. without graphical representation).

- Some parameters of the Method can be changed (not the Method itself). Afterwards, the recorded data are reevaluated using the new parameter values.

- Opens a Result Workshop Template containing the data (e.g. for preparing a printout).

Sequence Line

Line	🔔	🏳	⚖	🔗	Fill + EQ	Unit 1 Port	Unit 1 Bank	Identifier 1	Method
47	✓	✓			No	5	3	water	CO2_EQ.met

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A particular line of the chosen Sequence is shown. It denotes the currently measured sample.

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	20.483	1.128
5	5348.574	6275.926	5382.165	6221.931	1.1733829	15.026	20.477	15.350	20.465	1.128
6	5345.251	6272.043	5379.417	6218.645	1.1733860	15.025	20.473	15.348	20.460	1.128
7	5341.927	6268.162	5376.827	6215.697	1.1733896	15.033	20.478	15.357	20.466	1.128
8	5338.647	6264.314	5374.151	6212.760	1.1733898	15.016	20.523	15.338	20.511	1.128

Figure 6-47. Raw tab

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. m/z 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 calculated from true element sample ratios and 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":
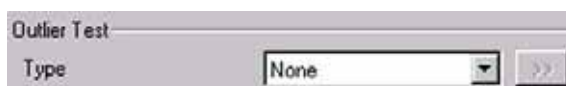
  15.362
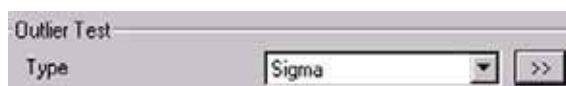
  If *Sigma* was selected there and specified by

  [>>] , outliers may occur and are marked as shown above. They only occur in case of Delta values, e.g. Delta [13C/12C] vs. V-PDB.

  If *None* was selected there, no outliers are calculated at all. Therefore, no black values on a pink background will occur.

  Suppose, that outliers have occurred (i.e.the case of Sigma). If you then decide to reevaluate these data using a changed Method (via the Method tab) selecting *None* at the "Evaluation" tab's "OutlierTest", no more outliers will be calculated.

  Thus, the values will turn into normal black ones: 15.362   →   15.362

• A black value on a green background is a Normal value falling into the k-fold of s (that is, 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.

Evaluated tab

Figure 6-48.   Evaluated tab

The Delta values (d 13C/12C vs. V-PDB and d 18O/16O vs. V-SMOW) marked pink or green in the columns **5** undergo statistical analysis:

• d Mean   Mean of the Delta values noted in column **5**

  (e.g. Mean of all Delta 18O/16O values in column **5**).

• d 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**).

• d 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 d Mean value (e.g. Atom-% of 13C is calculated from d [13C/12C]).

• Outlier   Number of outliers in column **5** entering into calculation of d Mean, d Std. Dev and d St. Error (e.g. 2).

Errors tab

Figure 6-49.   Errors tab

The Errors tab contains detailed information about errors that occurred during Data Acquisition:

- Number   consecutive number of the error (i.e. 1; 2; 3…)
- Status   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
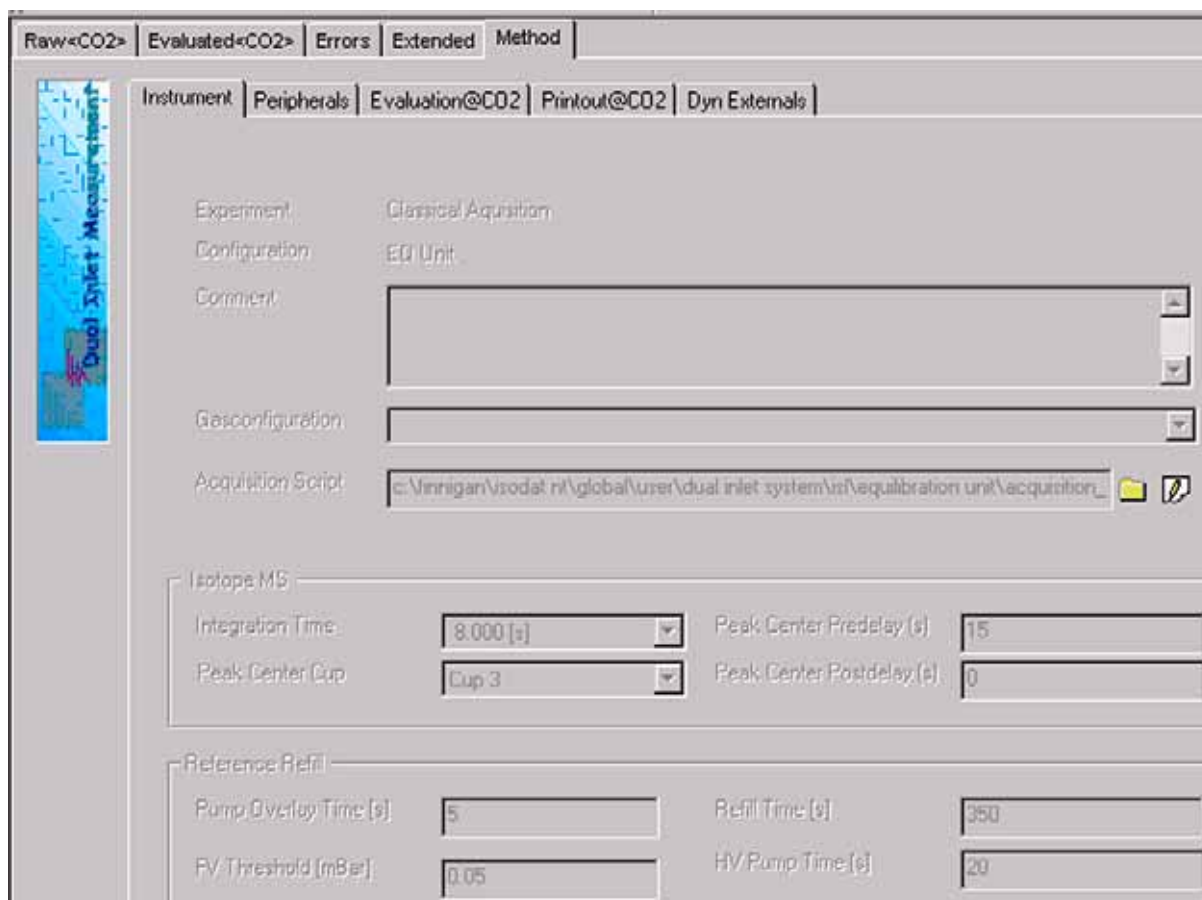- Script   script that caused the error

Extended tab

Figure 6-50.   Extended tab

A measurement protocol is presented containing details about particular results during measurement progression (e.g. results of Peak Center or Pressure Adjustment). 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

Figure 6-51. Method tab

For Data Reevaluation purposes, some parameters of the Method selected prior to measurement start can be modified (mainly in the "Peripherals" tab and the "Evaluation" tab; click the tabs in order to determine the changeable parameters and then type in the new corrected values). After the parameters of interest have been modified, reevaluation of the data can be started. Therefore, press the **Re-Eval** button.

6.12   Excel Export of Results

Principle of Excel Export

Measurement parameters and results are exportable as an Excel file. The Excel export can easily be tailored to your individual needs. Therefore, rules must be established to determine how to perform the export. These export rules are described in an export template (that is, a wke file).

Various selectors, which group Identifiers, are used to create an export template. For an export, more than one template can be selected. Multiple templates can be used simultaneously. Every single template (that is, every wke file) finally leads to an export file of its own, its wk1 file. Every wk1 file is created according to the rules of the wke file.

Advantages: Although measurement data are stored in one result file, the data of 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 export properties are defined (e.g. peak relevant data).

Creating an Export Template

1.   On the Dual Inlet window's toolbar, press the New button [ ] .

2.   Click **Data Export** and confirm by **OK**.

Thus, a new export template can be edited.

Figure 6-52.   Creating a new export template

3. Four selector types can be selected.

Figure 6-53.   Main Filter tab

The Four Selector Types for Template Creation

Selectors facilitate Excel-Export: the list of exportable data can be shortened (see the Identifiers in the left pane). Without selectors, all exportable data are visible.

**available pool of
Identifiers (columns)**

**Identifiers (columns)
to be exported**
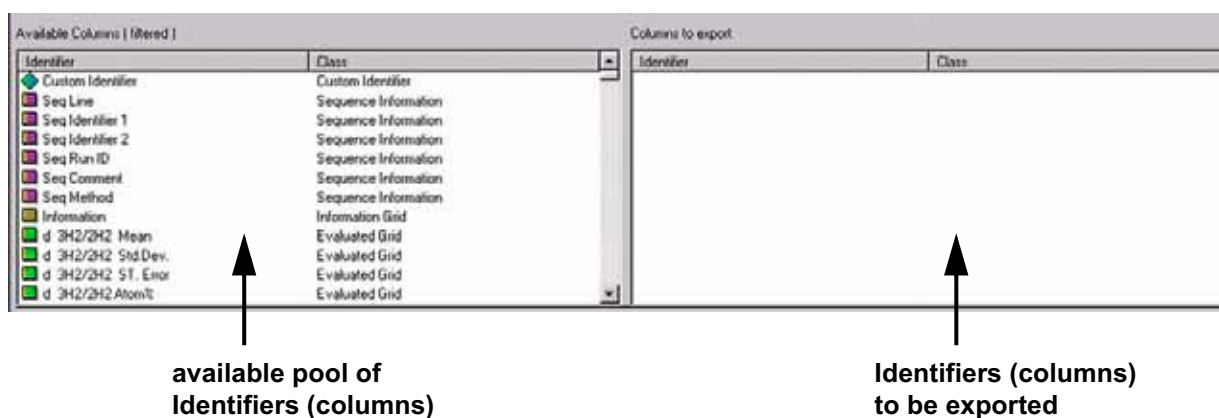
> **Note.** Selectors are combined by a conjunction (that is, by an AND relation).

> **Note.** (De-)activation of Selectors can be changed during template creation.
>
> The export columns will not be affected.

1 **Acquisition Mode**

If Acquisition Mode is activated, choose 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, that is, no selector.

2   Included String

If Included String is activated and you type a string (e.g. "Delta"), the entries in the left pane ("pool") will be selected according to that string after pressing **Apply**. The entries that match the string will be displayed in the right pane.

3   Gas Configuration

If Gas Configuration is activated, choose a Gas Configuration from the list (e.g. *CO2*). All relevant data for this Gas Configuration will be shown in the right pane.
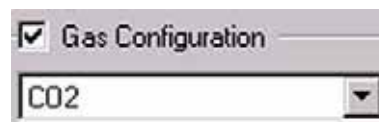
4   Data Type
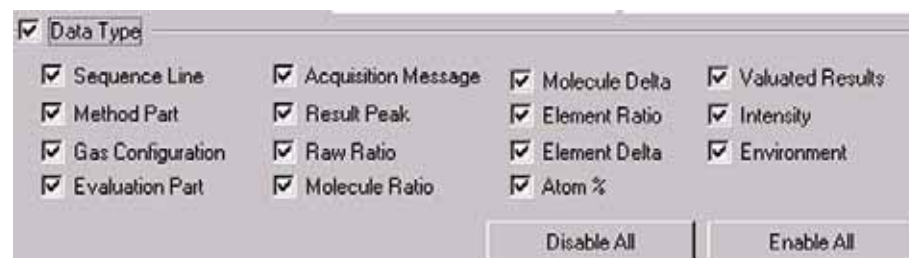
Figure 6-54.   Data Type group box

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:

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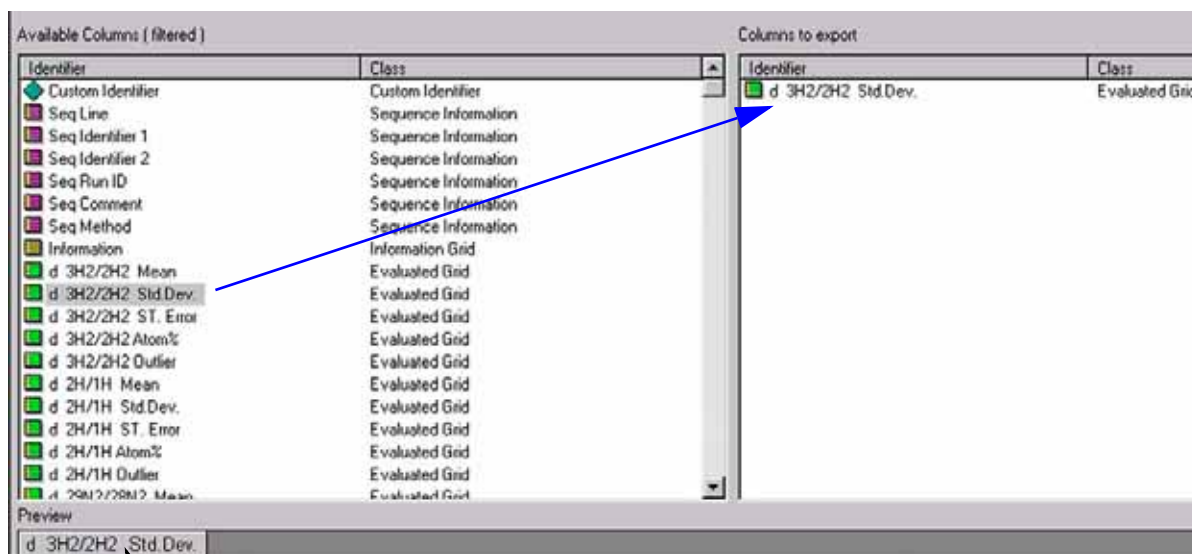
Every individual identifier will appear below the panes as a column in the wk1 Excel sheet.

Figure 6-55.   Exporting individual Identifiers

Drag and drop of the Custom Identifier allows inserting a column that contains no data. 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.

Figure 6-56.   Insert Identifier dialog box

1.   Type a name for the custom identifier (e.g. "Identifier 1"). Press **OK**.
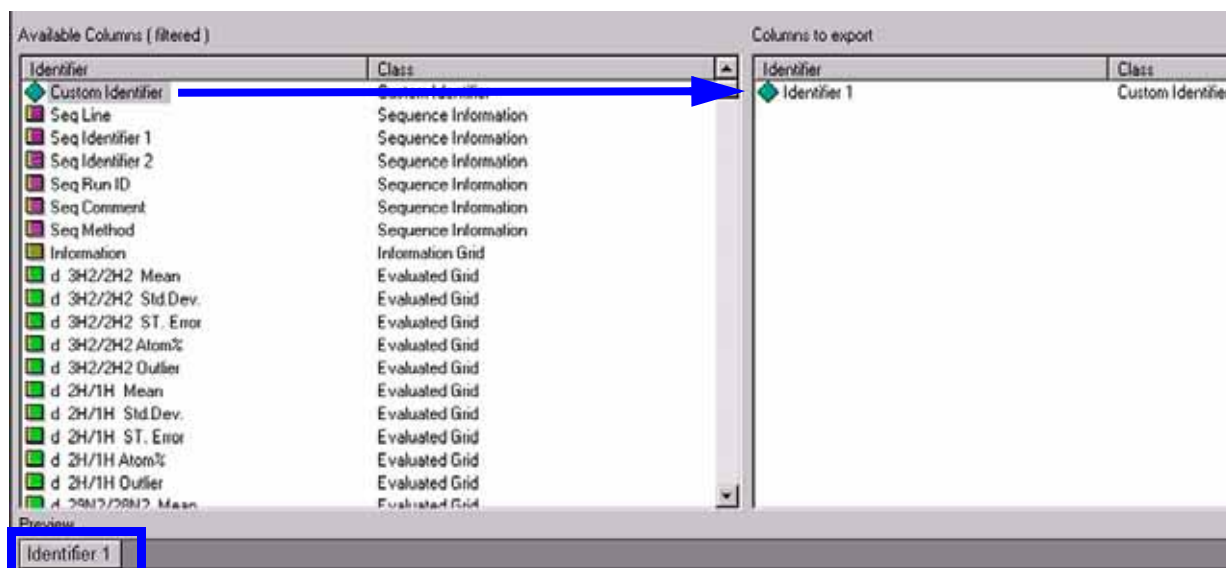
Figure 6-57. Exporting Custom Identifiers

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.

Figure 6-58. Deleting Identifiers

1. To remove one or more Identifiers (Custom Identifiers or other ones) from the right pane, mark them there. Then right-click them.

2. Select **Delete Identifier**. See Figure 6-58.

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3. 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.
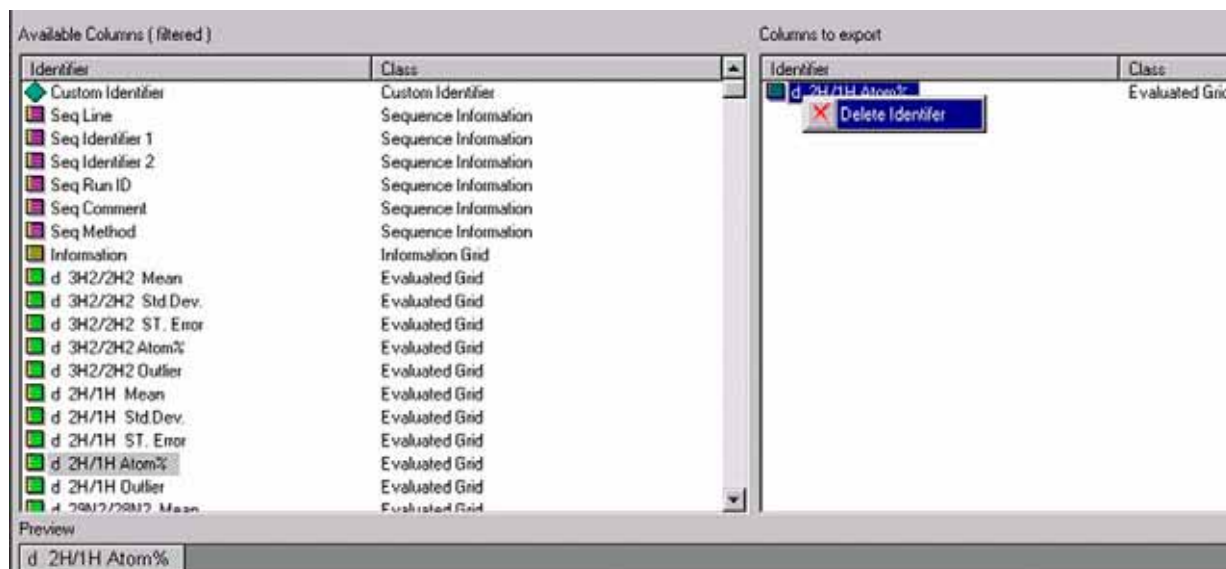
Saving an Export Template

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.
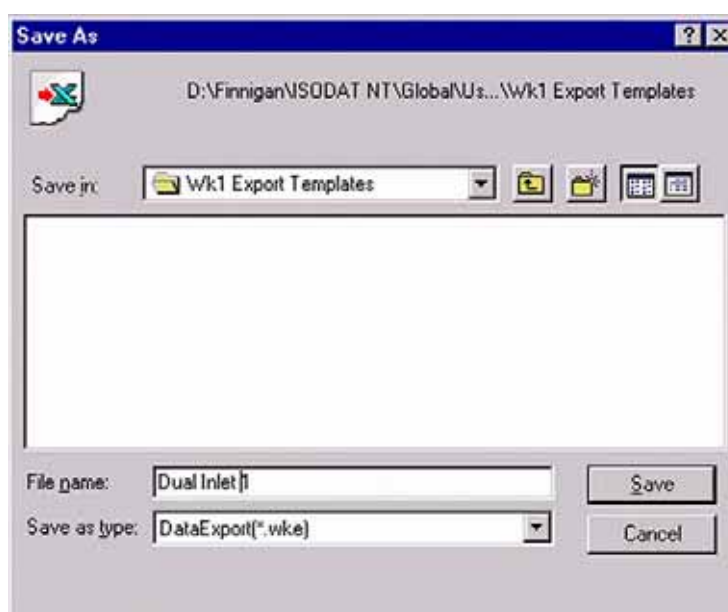
Figure 6-59.   Saving an export template

1. Save the export template in the folder Wk1 Export Templates!

2. In the File Name field, type a significant name for the export template (e.g. Dual Inlet 1).

3. In the Save as type field, accept the extension .wke and press **Save**.

Applying an Export Template

A Sequence is always exported as several samples can thus be bundled into a single export file.

1. To apply an export template, open a new Sequence via the **New** button.

Figure 6-60.   Creating a new sequence

2.   Then click the Sequence Icon. Confirm by **OK**.
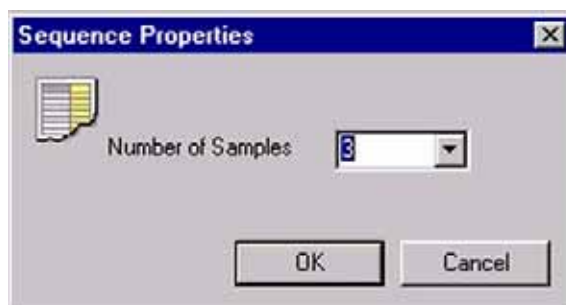
Figure 6-61.   Sequence Properties dialog box

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	▾

4.   The Sequence of your choice is displayed.

5.   Press the [▷ Start] button.

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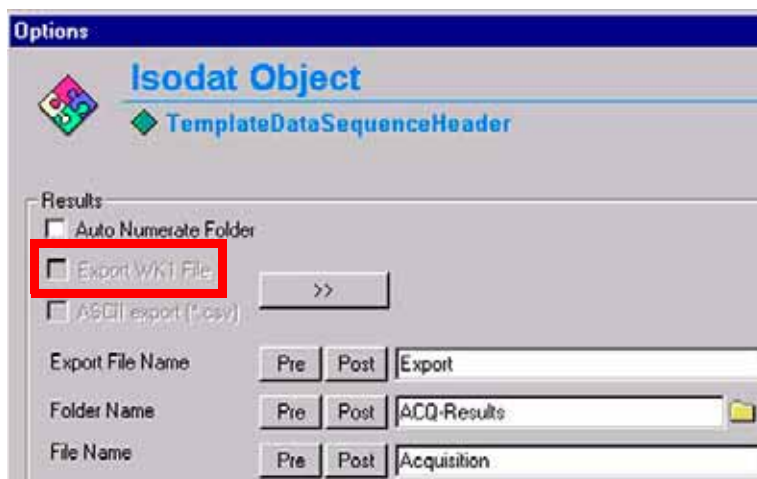
6. Since no export templates have been selected yet, the Export WK1. File box is inactive.

   Press the [>>] button.

7. Since no export templates have been selected yet, the Export Template list is empty.

   Press the [Add] button.

8. In the folder WK1 Export Templates, select your Export Template (e.g. Dual Inlet 1.wke).

   Press **Open**.

Figure 6-62.   Opening an Export Template
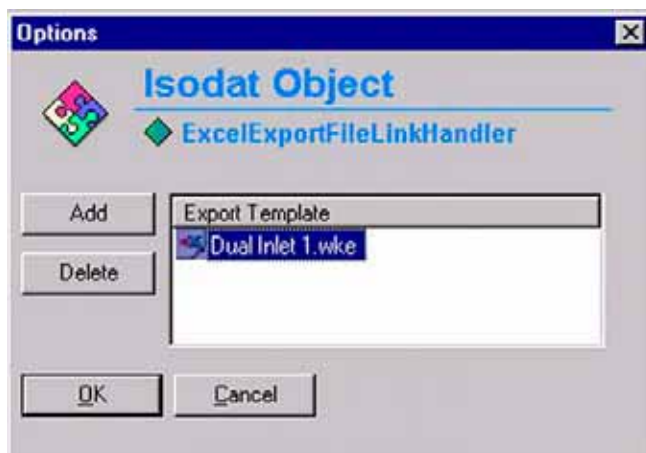
9. Select your Export Template
    (e.g. Dual Inlet 1.wke).

    Confirm by **OK**.
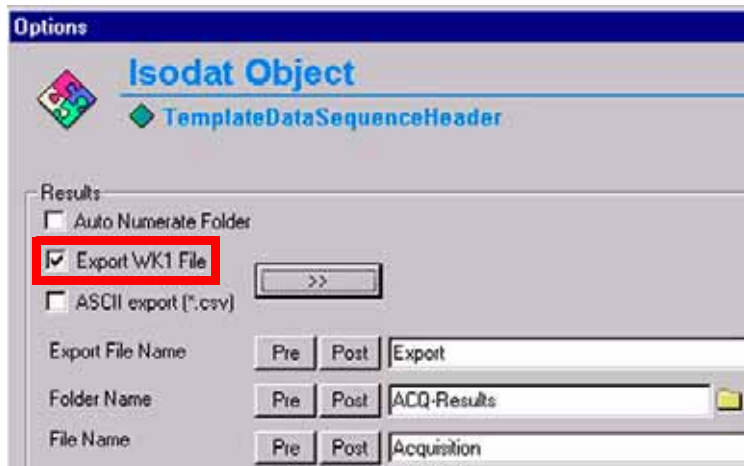
10. Notice that the Export WK1 File
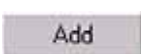   box has become active. Confirm by
    OK.

    The options specified in the Sequence
    are now set and Data Acquisition starts.

> **Note.** Excel Export will be performed online, that is during measurement.
>
> This is different from a Re-evaluation, where the Excel Export takes place offline, that is, after a measurement.

Chapter 7

Diagnosis

7.1   Checking Performance Data

Thermo Electron has developed several test routines to check the performance data of the Finnigan DELTA^{plus}XP isotope ratio mass spectrometer.

For user's convenience, the program Diagnosis covering these test routines is included in the supplied version of ISODAT NT. It must be noted that operating some of the test routines requires technical knowledge of the instrument's internals. In addition, successful execution of some of the tests depends upon instrument preconditions.

When running the test routines, a highly sensitive focusing of your instrument will lead to the best specifications results (see topic **Principle of Focusing** on page 1-43 about how to focus the Ion Source).

The program Diagnosis contains the following test routines:

1. Absolute Sensitivity

2. Abundance

3. Amplifier Test

4. Compression Factor

5. Linearity

6. Peak Flatness

7. Relative Sensitivity

8. Resolution

9. Signal Stability

10. System Stability

> **Note.** Reference gas for all performance data is CO_2.
>
> Make sure to have properly filled CO_2 reservoirs attached to the inlet system before starting the "Diagnosis" program.

7.2   How to Start Diagnosis

1. Start ISODAT NT by double-clicking the icon on your desktop.

2. Double-click the Diagnosis icon.

3. Mark the test routine to be performed (e.g. "Abundance").

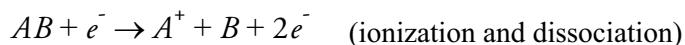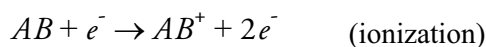4. Press **OK**.

Absolute Sensitivity

> **Note.** Testing Absolute Sensitivity requires a Dual Inlet system.

Absolute Sensitivity is defined as the number of molecules needed to generate an ion, which is then registered at the collector (e.g. one ion of m/z 44 at the corresponding collector cup). It is thus dimensionless and measured in molecules per ion.

Based on a defined volume, the ion current is determined during a defined time period as a function of sample consumption (that is, sample loss). The small, defined volume is located between valve 25 of the inlet system and the inlet capillary. It amounts to approximately 250 µl.

Integrating the ion current over time yields the number of ions. From the signal drop during measurement the number of molecules necessary to generate this ion amount is calculated. To obtain Absolute Sensitivity, the number of molecules is divided by the number of ions.

Positively charged ions are produced in the ion source by electron bombardment. This electron impact (EI) ionization is described by:

$$AB + e^- \rightarrow AB^+ + 2e^- \qquad \text{(ionization)}$$

$$AB + e^- \rightarrow A^+ + B + 2e^- \qquad \text{(ionization and dissociation)}$$

Definition of Absolute Sensitivity *AS*:

$$AS = \frac{\Delta s}{n_{ion}} \qquad \text{(molecules / ion)}$$

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:

$$p \times v = n \times R \times T$$

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
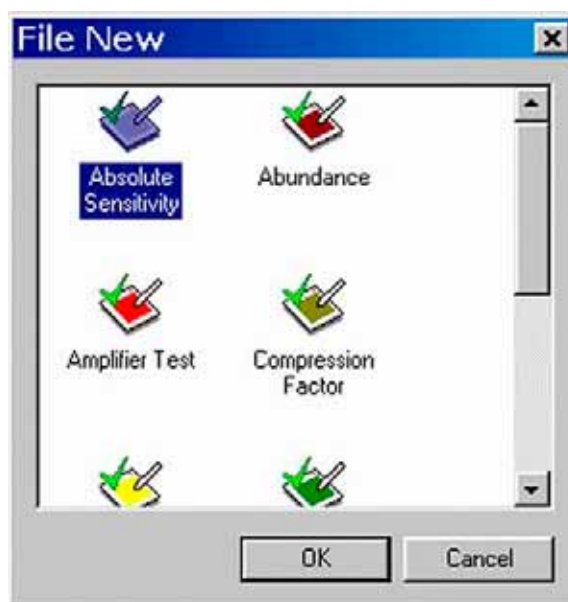
1. Click on the Absolute Sensitivity icon.
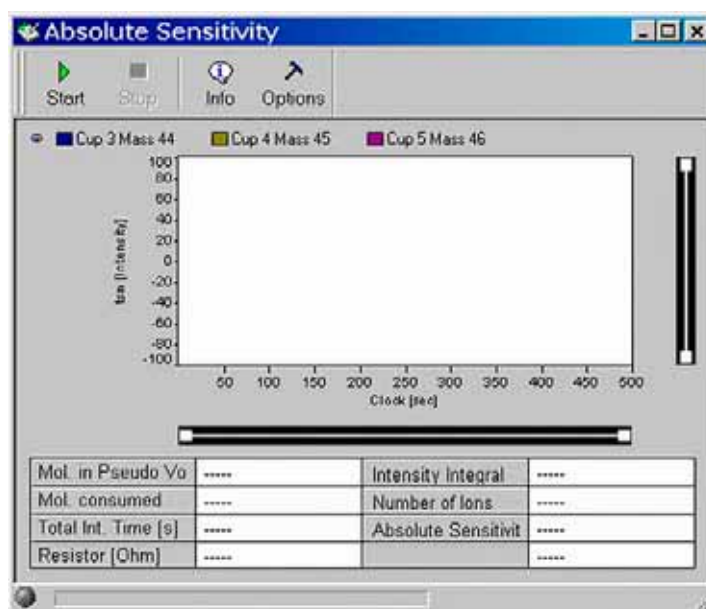
   Press **OK**.

Figure 7-1.   Absolute Sensitivity window

2. On "Window" toolbar, the Ab
   solute Sensitivity icon becomes
   visible.

3. Press the **Options** button.

4. 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.

   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).
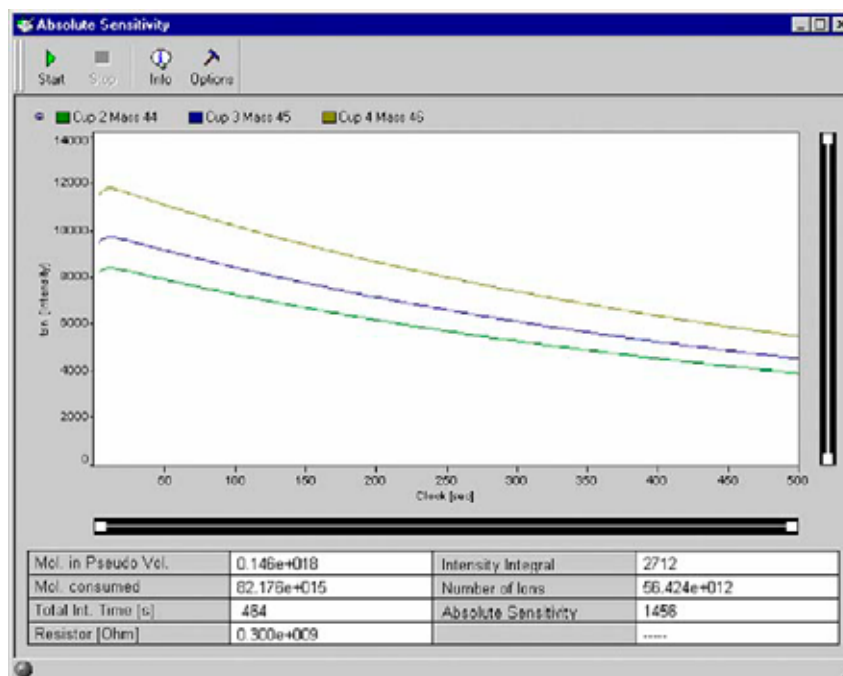
5. Finally, click **OK**.

6. Press the **Start** button.

7. Testing starts with a Peak Center on the selected cup, e.g. m/z 45, Cup 3.
   The bellow is reduced stepwise until the start level intensity
   (e.g. 8000 mV) is reached. Then, intensity and pressure are measured for
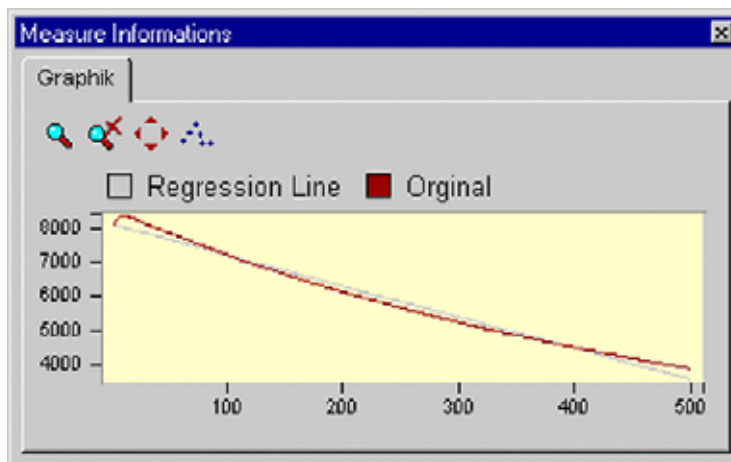   the preset time (e.g. 500 s).
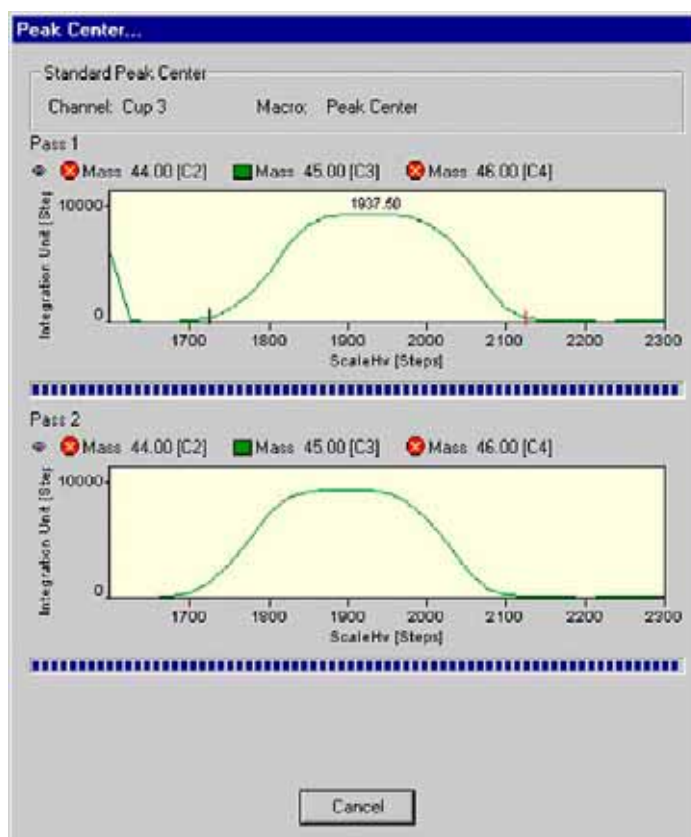
8. Finally, Absolute Sensitivity and other parameters are calculated.

9. Press the **Info** button.

10. Original measurement and regression line are shown.

11. When closing the "Absolute Sensitivity" window you are asked, whether to save the changes.

Figure 7-2.   Conformation dialog box for Absolute Sensitivity

Abundance

Abundance denotes the contribution of a mass to a neighbor mass (e.g. the amount of ions for m/z 44 falling into m/z 45 cup). Thus, the intensity of a mass (e.g. m/z 44) is compared to the intensity of the neighboring peak (e.g. m/z 45).

In case of CO_2, divide the amount of ion current of mass 44 falling into the m/z 45 cup by the ion current of m/z 44 into the m/z 44 cup.

Measured as ratio of two ion currents it is dimensionless and quoted in % or ppm. It should not exceed $2*10^{-6}$ for a DELTA^{plus}XP with Dual Inlet system. The Abundance test is performed with CO_2, and the device must be calibrated. Peak center is performed on Channel 2 (that is, narrow cup) before measurement starts.

To Test this Parameter

1. Signal height (that is, intensity) on m/z 44 is determined in the appropriate collector cup (e.g. Cup 3).

2. The background signal on m/z 45 is measured (that is, electronic noise with no gas) from m/z 44 to m/z 45.5.

3. With CO_2 as sample gas the magnetic field is scanned from m/z 44 to m/z 45.5, and the intensities are measured on the neighboring cup of m/z 45 (e.g. Cup 4, with bigger resistor value in order to keep the signal in the detection range).

4. Extrapolate the abundance (of m/z 44 onto m/z 45) from the signal to the left and to the right of m/z 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

1. Click on the Abundance Icon and press **OK** (or double-click the Icon).
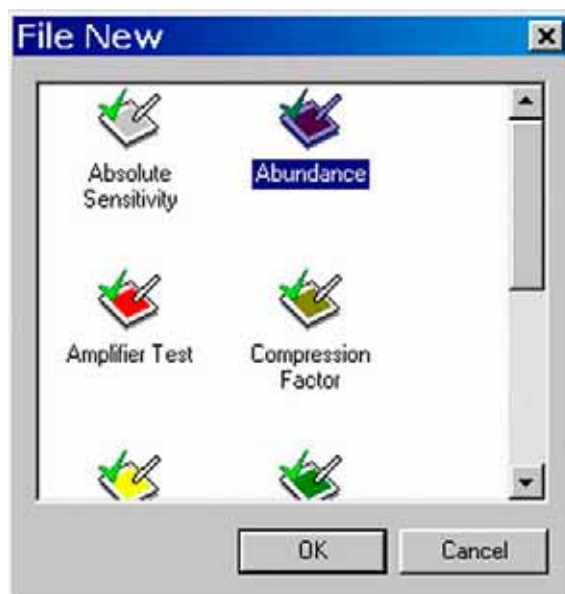
2. The left window detects mass 44 ions in the correct cup. The right window detects mass 44 ions in the neighbor cup (i.e. cup of mass 45).
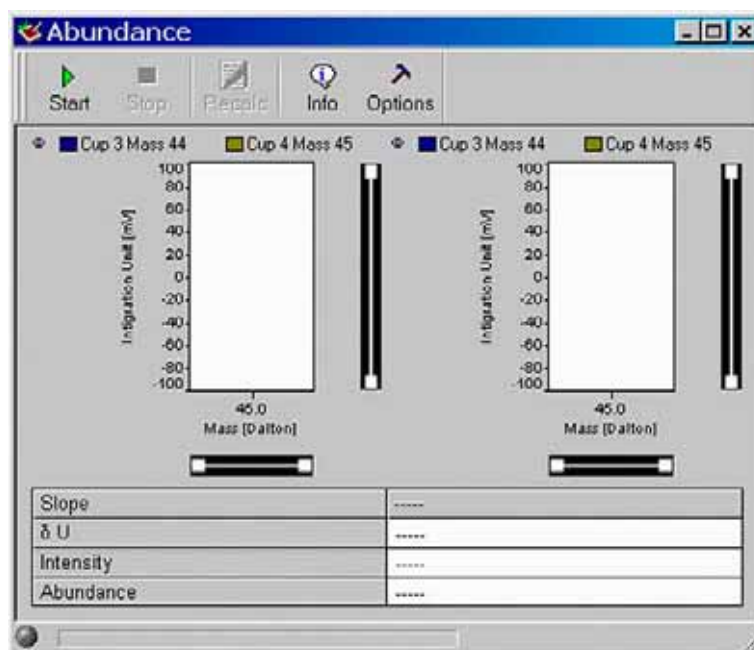
Figure 7-3.   Abundance window

3. On the "Window" toolbar, the
    Abundance Icon becomes visible.

4. Press the **Options** button.

5. Accept the defaults, or type
    appropriate values for "Press
    Adjust [mV]" and "Peak Width
    [Dalton]".

    The selected cup for the
    Abundance test is connected with
    the indicated recording channel
    (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, that is gray, variables cannot be edited. The Advanced Mode allows experienced users to set them all:

- the start mass for the magnet field scan ("Start [Dalton]"),

- the end mass, where the magnet stops scanning ("Stop [Dalton]")

- the step width for the magnet scan ("Step [Dalton]")

- the "Adjust Manual" check box - if it is activated, signal intensity and peak center must be performed manually.
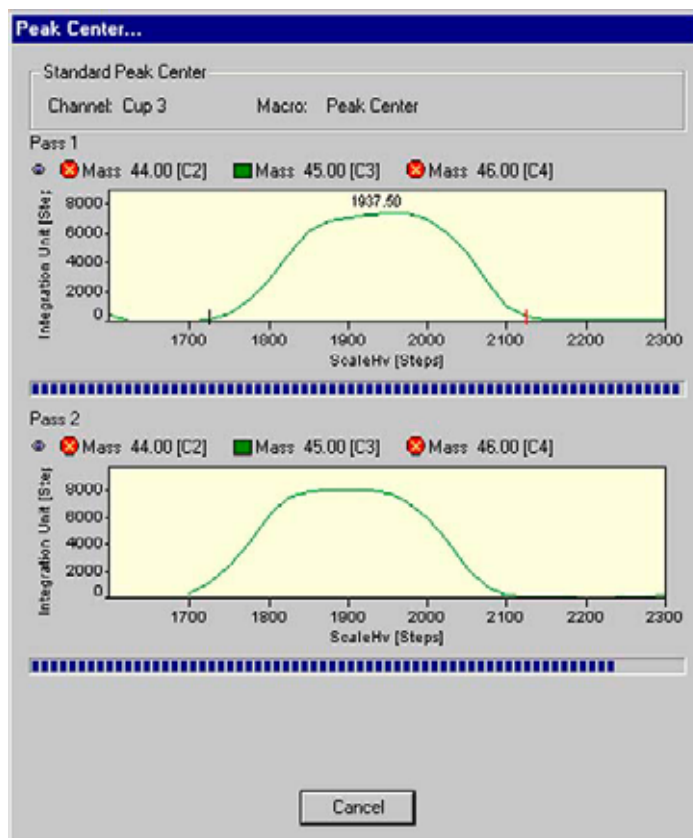
6. Finally, click **OK**.

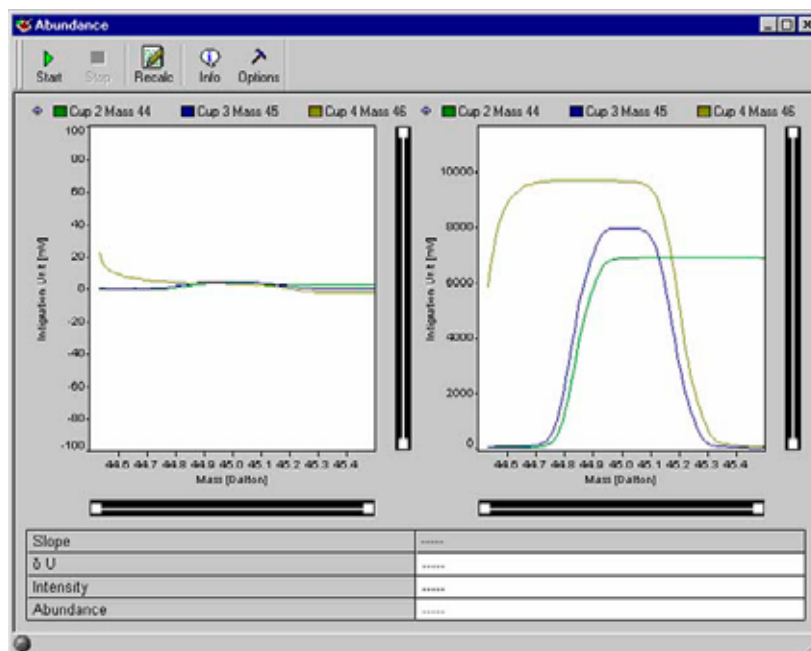7. Start the Abundance test by the **Start** button.

8. Testing starts with a Peak Center on the selected cup (e.g. mass 45, cup 3).

9. 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).

10. Values for Abundance, Intensity, dU and slope are displayed.

11. When closing the "Abundance" window you are asked whether to save the changes.

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

1. Click the Amplifier Test Icon and press **OK** (or double-click the Icon).

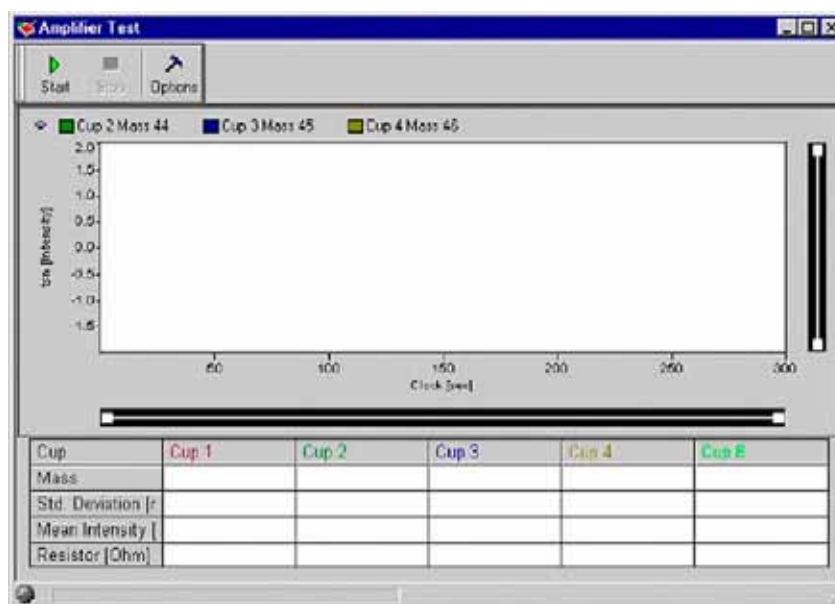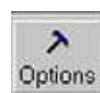2. Wait, while the available Gas Configurations are scanned.


Figure 7-4. Amplifier Test window

3. On the "Window" toolbar, the Amplifier button becomes visible.

4. Press the **Options** button.

5. With the exception of Delay, all parameters are preset and not changeable in Standard Mode.

   - The Advanced Mode allows experienced users to modify all parameters.

   - Accept the default value of Integration Time (in ms).

   - Scan Time (in s) denotes the duration of the scan.

   - The measurement start is delayed by the displayed default value of Delay time (in ms). Accept it, or from the pulldown menu, select a suitable value between 100 ms and 35000 ms.
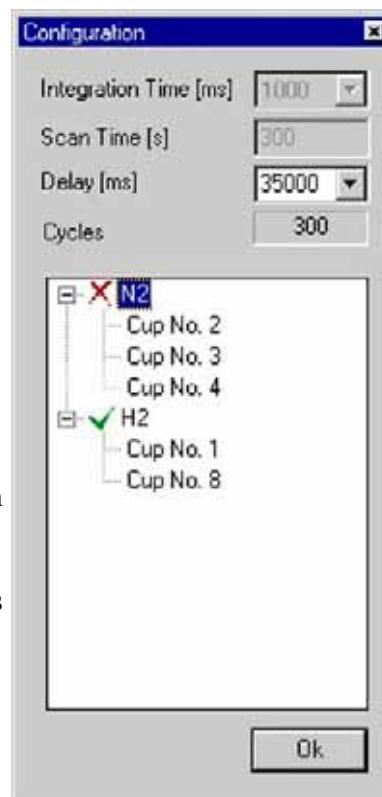
> **Note.** In one of the next versions, the process will be simplified: all amplifiers can then be measured using one single Scan.

Cycles denotes the number of measurements. The intensities of the channel are measured for indicated cycles with a definite cycle time.
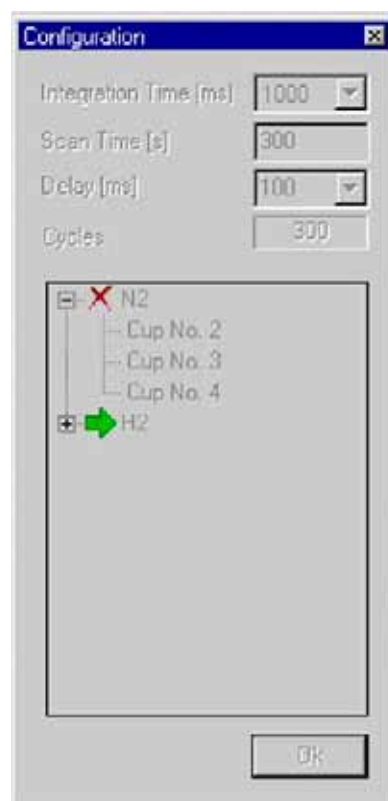
6. Finally, press **OK**.

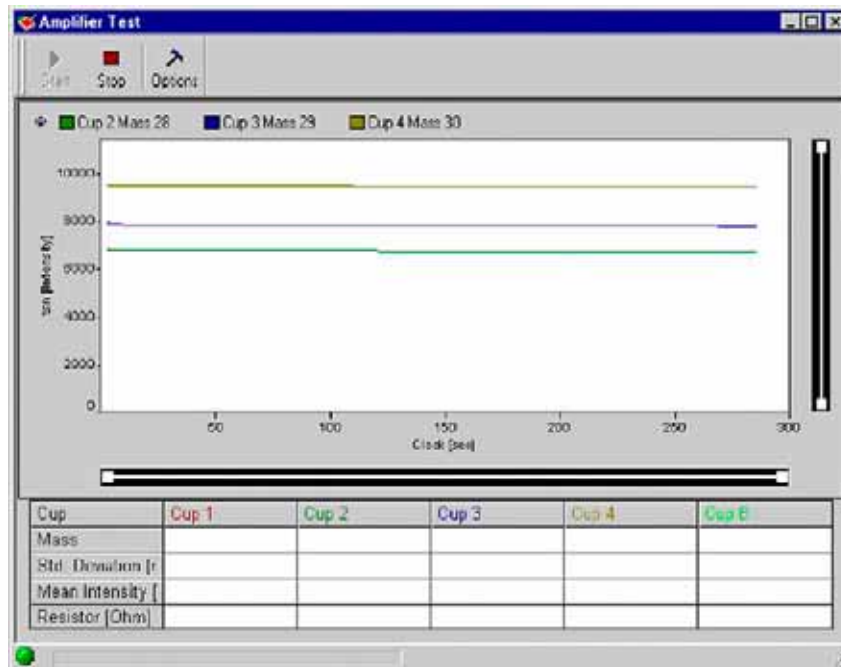7. Press the **Start** button.

8. Switch off High Voltage (on the Focus bar). Then press **OK**.

9. 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.

10. If you press the **Options** button during measurement, a green arrow informs you about what is currently done.
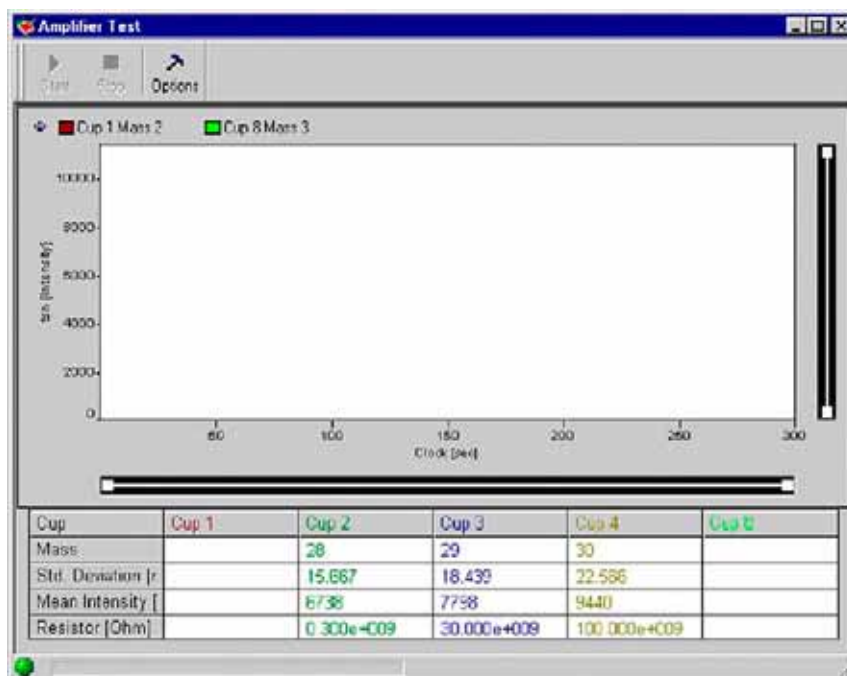

11. The correspondent intensity vs. time diagram is shown.

12. 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.

13. The procedure is repeated for the other masses.

14. When closing the "Amplifier Test" window you are asked, whether to save the changes.

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. m/z 44) is measured starting at the maximum (that is, 100 %) down to the minimum (that is, 0 %). A minimum death volume of about 3 ml is still remaining at 0 % volume. A certain level (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.
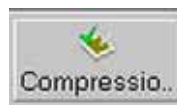
Testing the Compression Factor

1. Click on Compression Factor Icon and press **OK**

   (or double-click it).
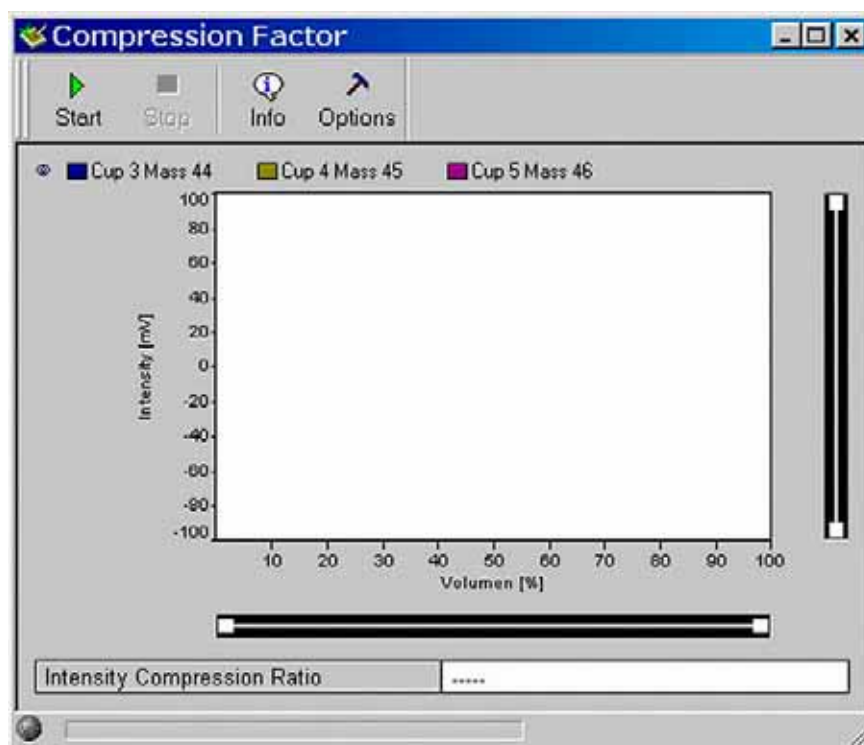
2. On the "Window" toolbar, the Compression Factor Icon becomes visible.

3. Press the **Options** button.

4. 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 step-wise, e.g. in 10 steps,

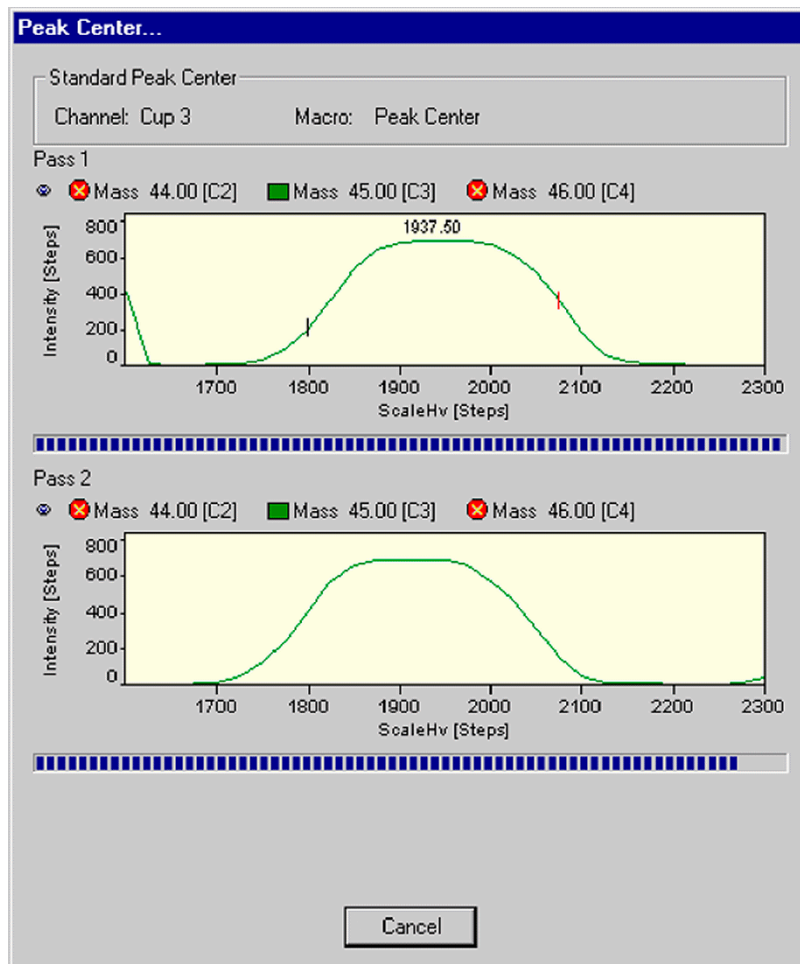   • Delay [ms]: the measurement start is delayed by the displayed value.
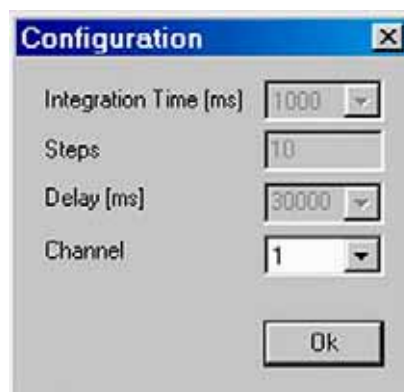
5. Finally, press **OK**.

6. Press the **Start** button.

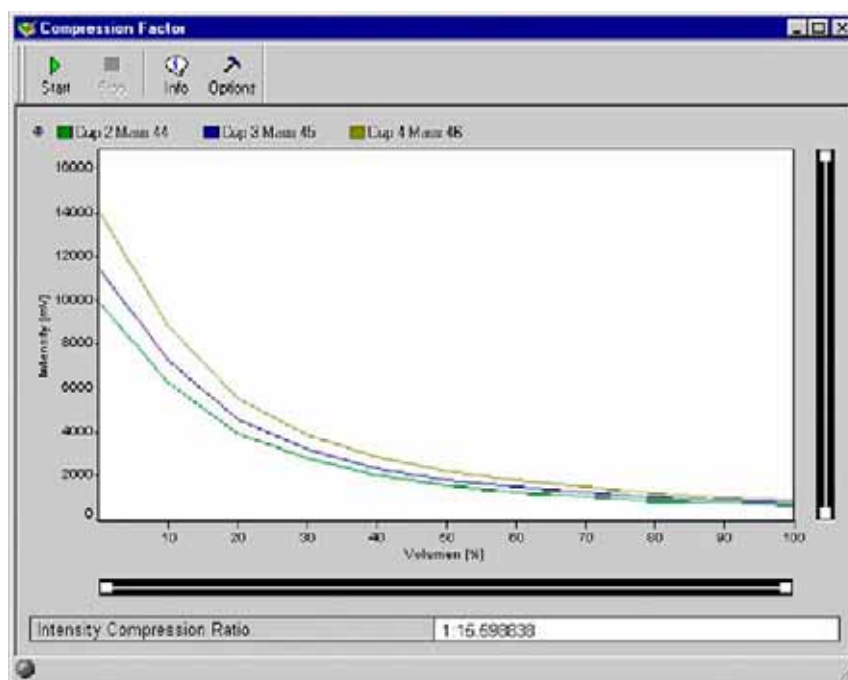7. Testing starts with a Peak Center for the first bellow (e.g. Sample).

8. 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.
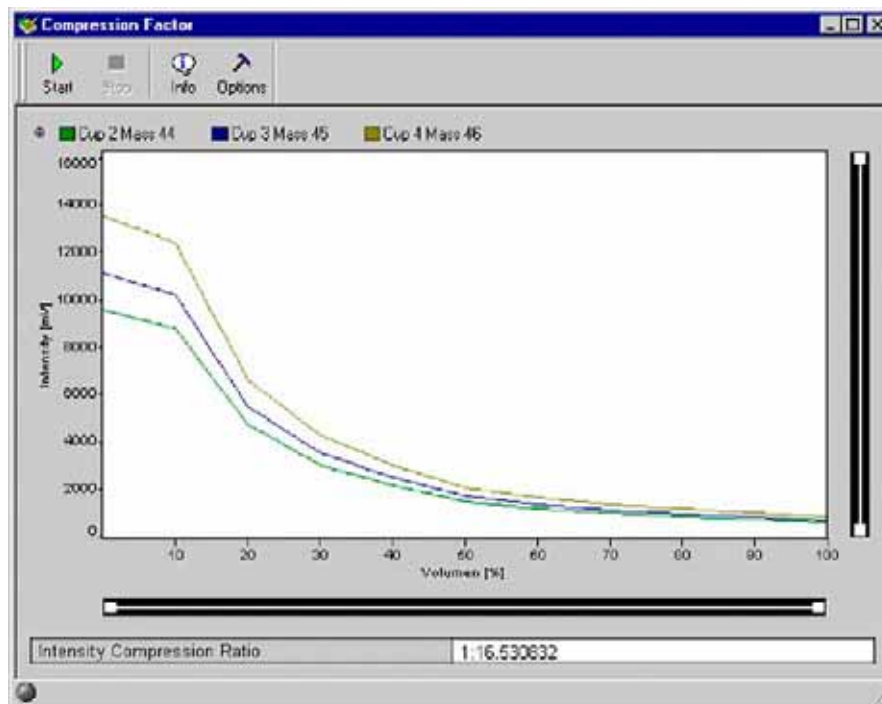
9. The procedure is repeated for the second bellow (e.g. Standard): a Peak Center is performed.
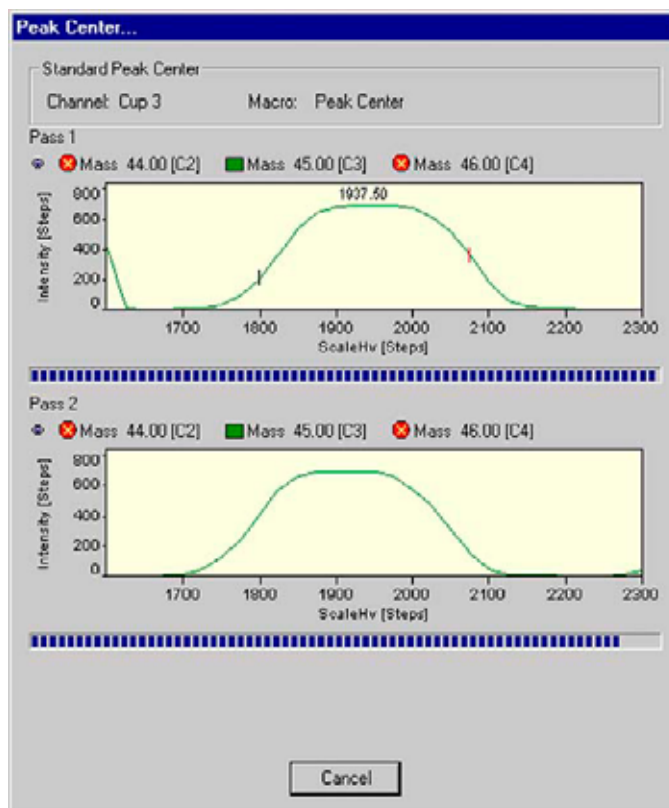
10. The procedure is repeated for the second bellow (e.g. Standard).

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).

11. When closing the "Compression Factor" window you are asked, whether to save the changes.

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

1. Click the Linearity Icon and press **OK**

   (or double-click it).
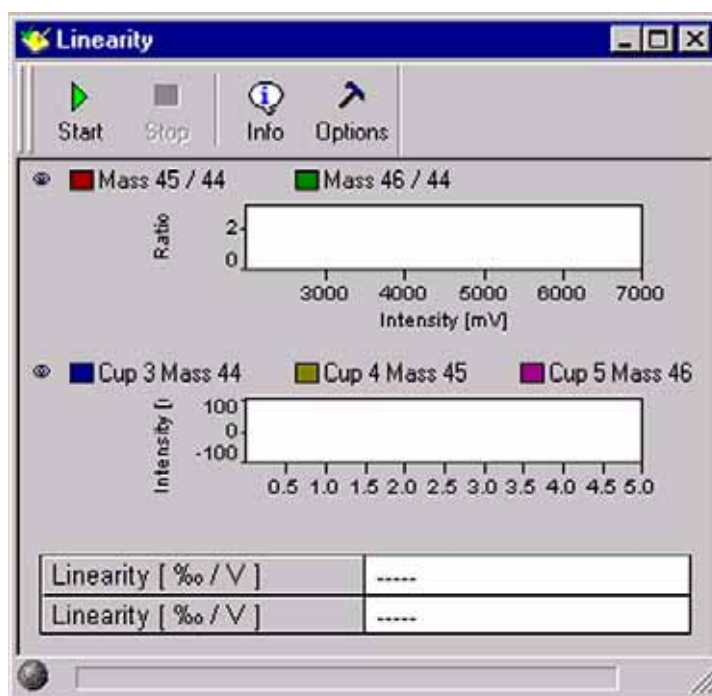
2. On the "Window" toolbar, the Linearity Icon becomes visible.
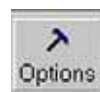

3. Press the **Options** button.

4. From the pulldown menus select:

    *   a channel and a value
        between 100 ms and
        20000 ms for "Delay [ms]".

    The other variables are preset in
    Standard Mode (to be accepted),
    but editable in Advanced Mode:

    *   Integration time [ms], Start
        [mV], Stop [mV] Step width
        [mV], V-Window [mV]

    *   Adjust Manual:
        activate it, if the intensities
        (that is, voltage steps)
        between 2 V and 8 V are to
        be set manually.

5. Press the **Start** button.

6. Testing starts with a Peak Center on cup 3 as the narrow cup. If no peak
    could be found, the measurement will be terminated.
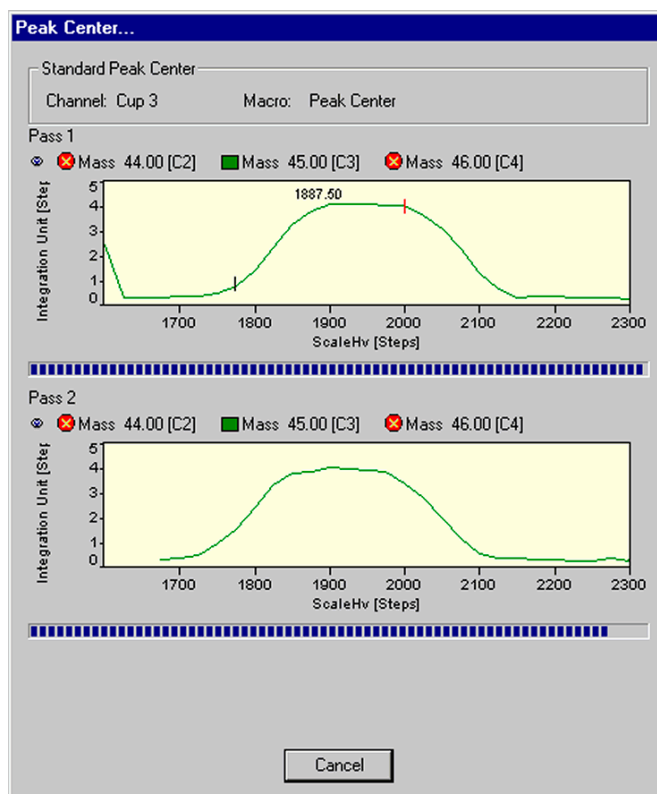
7. Two linearity values [‰/V] are calculated: one for mass 45/44 and one for mass 46/44.

> **Note.** Linearity must be less than 0.06 ‰/V!

8. Press the **Info** button.

9. Mean and slope values are displayed.

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 plat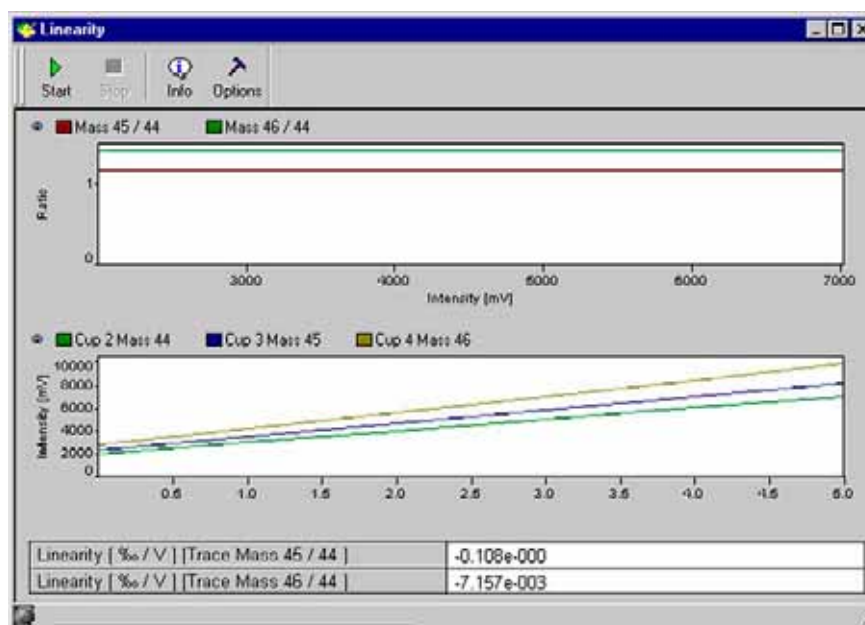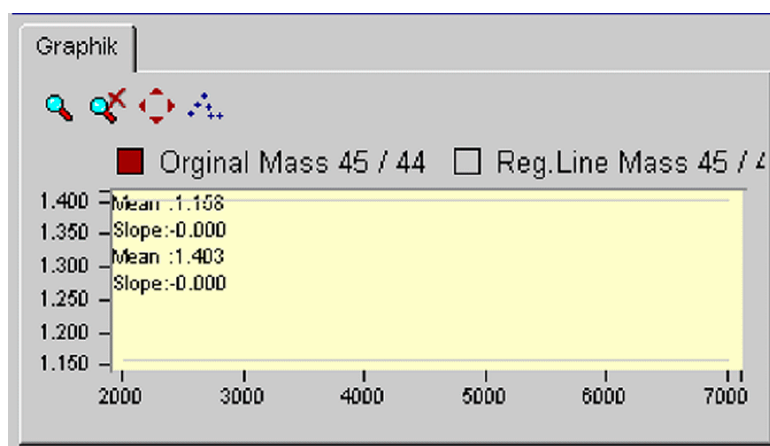eau with increasing high voltage. This is done by measuring the peak twice - first with increasing and then with decreasing high voltage. The resulting peak represents the mean values of both runs. The measured intensity is a function of the acceleration voltage (that is, ion energy). Therefore, during a high voltage scan the intensity is slightly affected by this effect. To overcome this an "energy correction" is performed. The ion intensity on top of the peak (that is, at a parameterized mass range around the center) is measured. Peak Flatness can be determined for different gases and different collector cups (e.g. for a CO_2-peak at m/z 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

1. Click on the Peak Flatness
   Icon and press **OK**
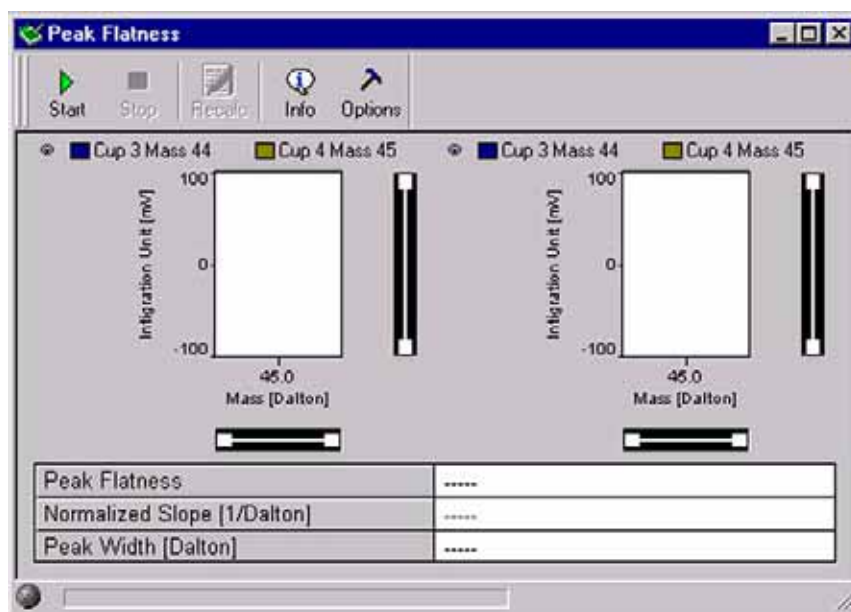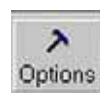
   (or double-click it).
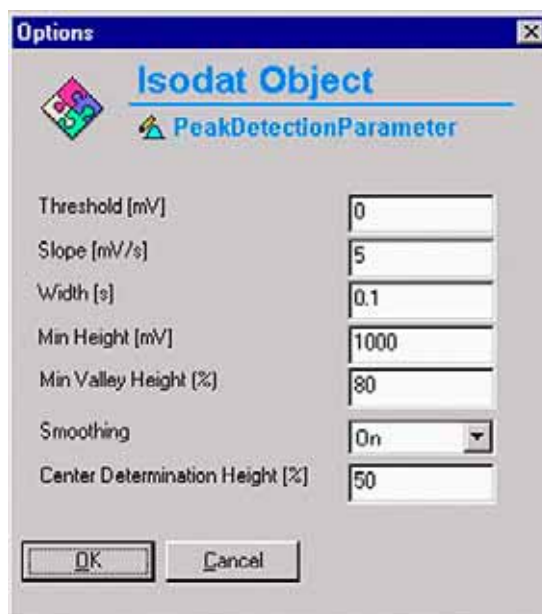

Figure 7-5.   Peak Flatness window

2. On the "Window" toolbar, the
   Peak Flatness Icon becomes
   visible.

3. Press the **Options** button.

4. Accept the defaults or from the pulldown menus, select a Channel and a Delay value between 100 ms and 20000 ms.

   a. Accept the default, or choose an appropriate Peak Width value by the **<** or **>** buttons.

   b. If you simply want to accept the Peak Detection parameters without a glance, do not press the **Peak Parameter** button. Instead, click **OK**.

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 >	
	Ok	

5. However, as a sophisticated user, you may want to look at the Peak Detection parameters. To do so, press the **Peak Parameter** 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

OK   Cancel

Change the values according to your needs.

Then click **OK**.

6. Press the **Start** button.

▷
Start

7. 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.
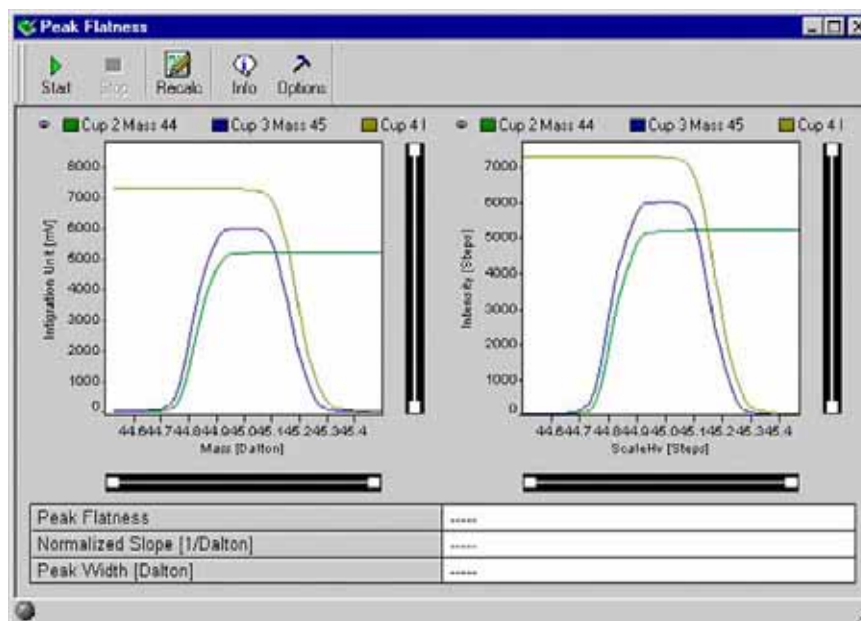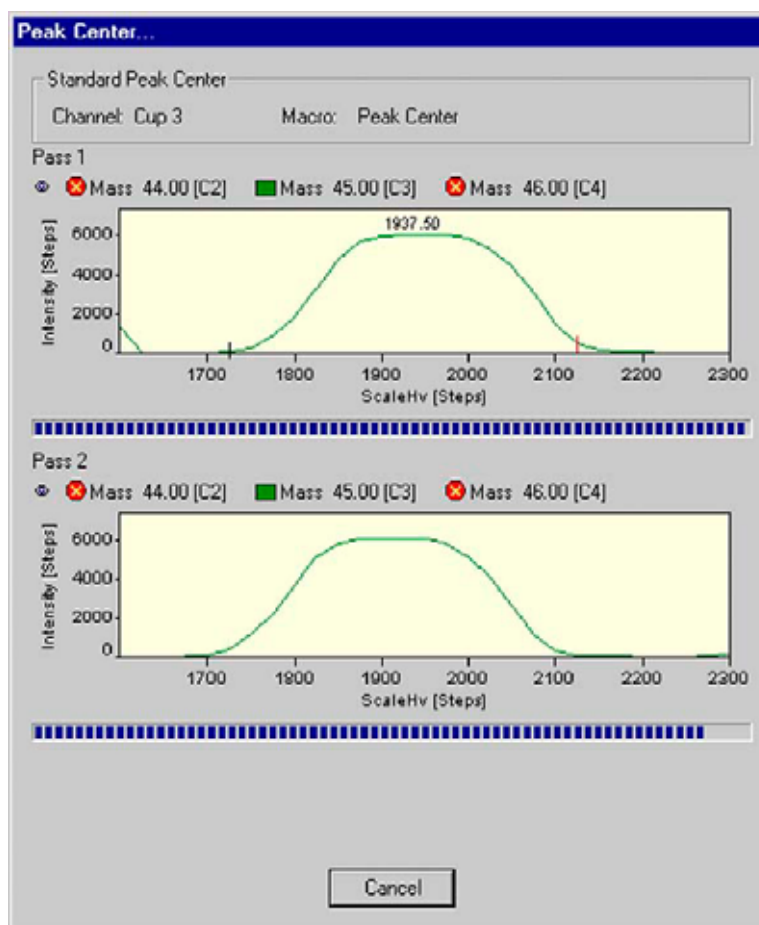
8. A Peak Center for Energy Correction is performed.

    If no Peak Center can be found, the measurement will be stopped.

    In this case, press **Cancel** to proceed.
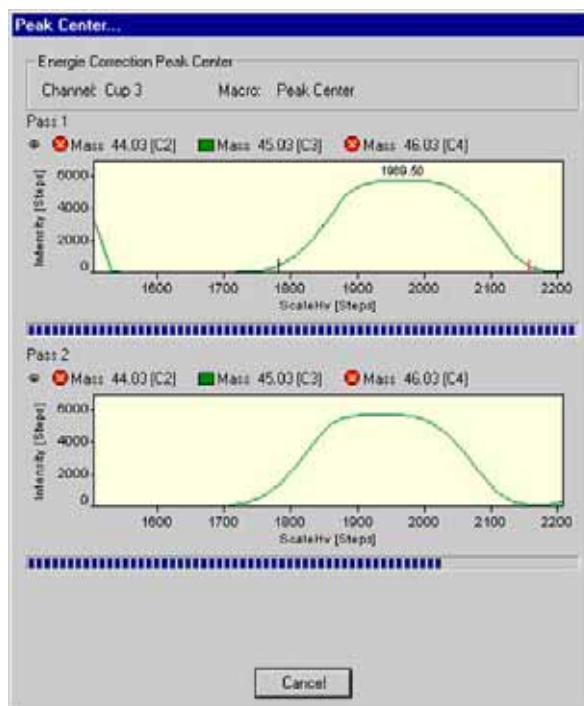
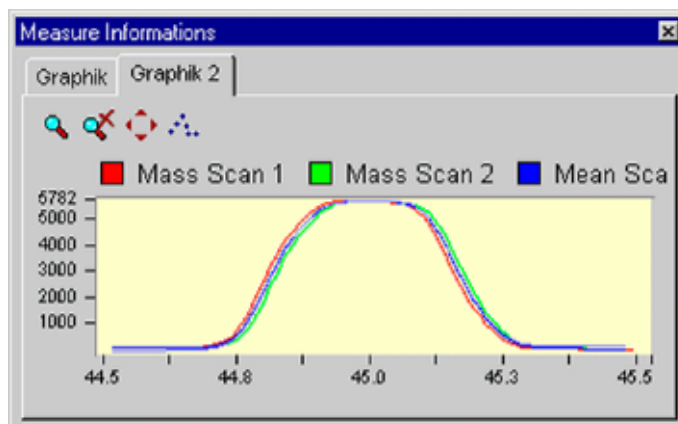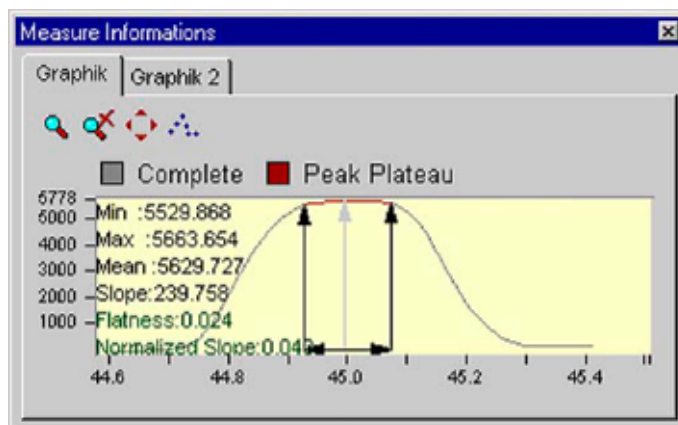9. Values for Peak Flatness, normalized slope $[Da^{-1}]$ and Peak Width $[Da]$ are calculated.
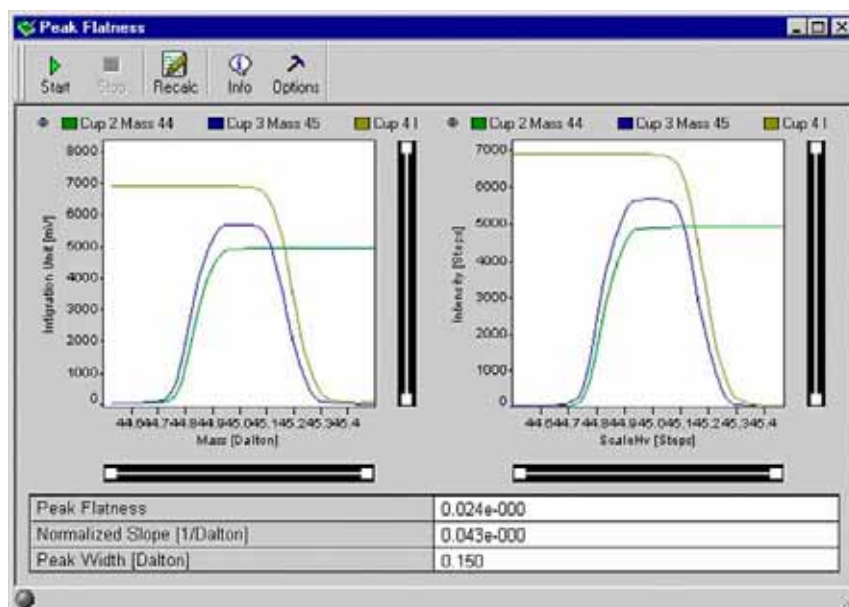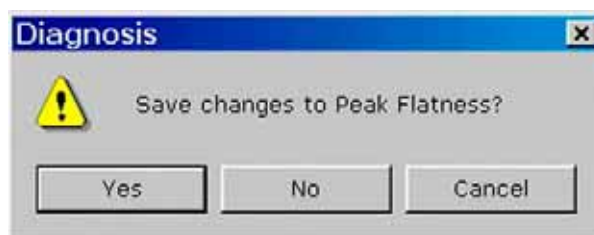
10. Press the Graphic tab to view relevant data.

11. Press the Graphic 2 tab to view relevant data.

12. When closing the "Peak Flatness" window you are asked whether to save the changes.

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:

$$S_{rel} = \frac{1}{0.69} \times \frac{1}{p} \times \frac{U}{R}$$

where:

U: voltage measured at amplifier of collector cup (e.g. Cup 3, m/z 44)

R: resistor value (e.g. $3*10^8$ Ω for m/z 44). This value is the same for N_2 and CO_2 as reference gases. It must be changed in special cases only.

Δp: 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

1. Click the Relative Sensitivity Icon and press **OK** (or double-click it).
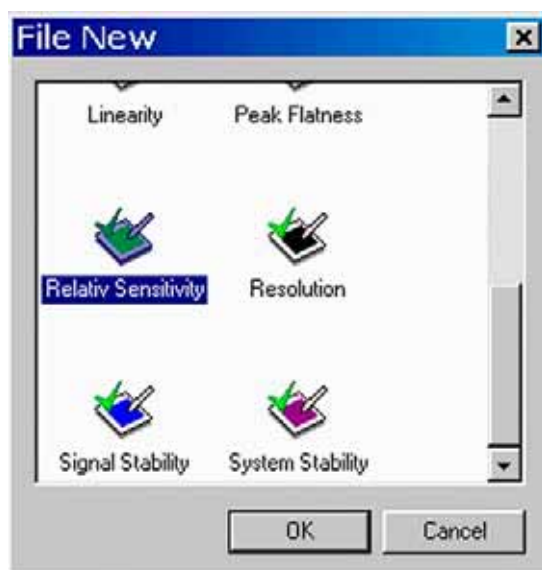
2. The cups with the corresponding masses and resistor values are shown.

Figure 7-6.   Relative Sensitive window
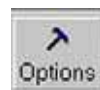
3. On the "Window" toolbar, the
   Relative Sensitivity Icon
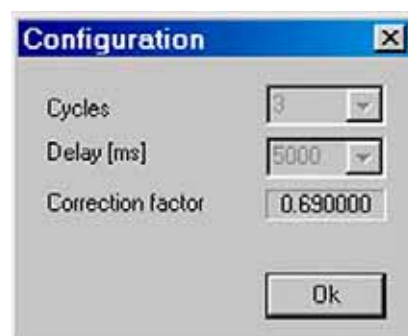   becomes visible.

4. Press the **Options** button.

5. Press **OK**.

   Experienced users can change
   the values via the Advanced
   Mode.

6. Click the **Start** button.

7. For each cup, the values of three cycles and their mean are displayed.

	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

8. When closing the "Relative Sensitivity" window, you are asked whether
   to save the changes.

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:
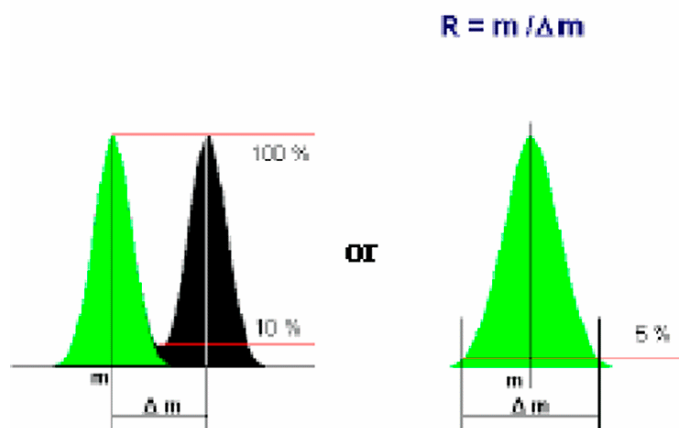
Resolution can be defined as:

- 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

- 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):

$$R = \frac{m}{\Delta m} \times \frac{a}{b}$$

where:

a: distance between peak centers

*b*:   peak width of the isotope of interest

Resolution is determined using the narrowest cup (that is, usually cup 2); m/z 45 is used.

Example

For a resolution of 88 and m/z 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
m/z 44 and m/z 45 are determined and Resolution is calculated.

Testing Resolution
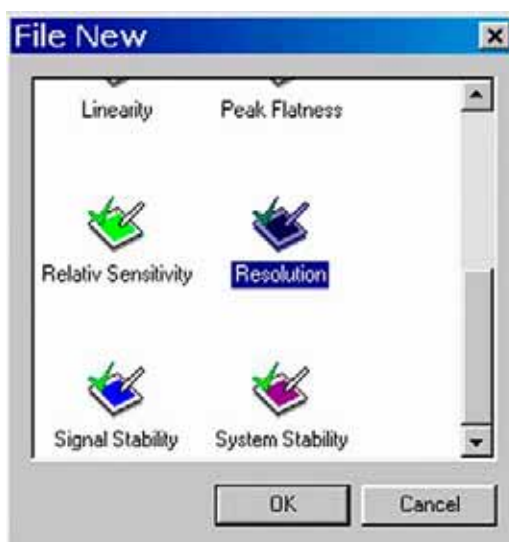
1. Click on the Resolution
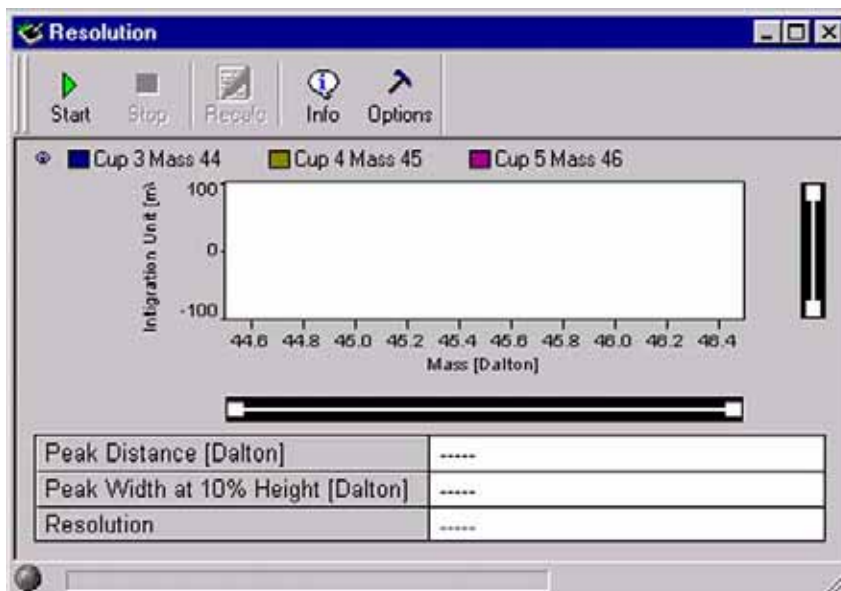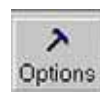   Icon and press **OK**

   (or double-click the Icon).

Figure 7-7.   Resolution window

2. On the "Window" toolbar, the
    Resolution Icon becomes visible.

3. Press the **Options** button.

4. Accept the defaults or from the
    pulldown menus, select a
    Channel and a Delay value
    between 100 ms and 20000 ms.

    a. If you simply want to accept
        the Peak Detection
        parameters without a glance,
        do not press the **Set** button.
        Instead, click **OK**.
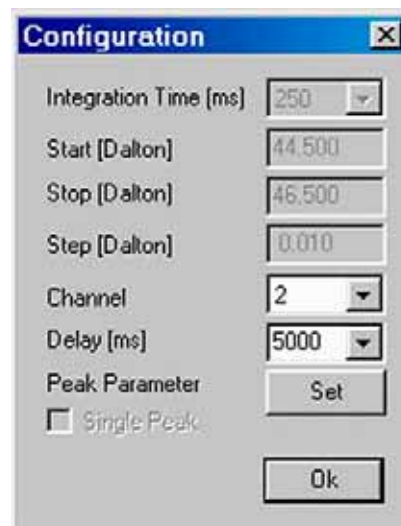
5. However, as a sophisticated user, you may want to look at the Peak
    Detection parameters. To do so, press the **Set** button.
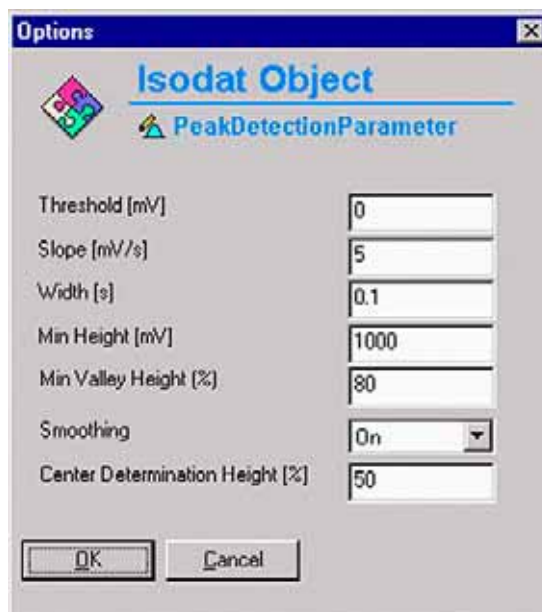
Change the values or
accept the defaults.

Then click **OK**

6. Press the **Start** button.

7. Testing starts with a Peak Center on the selected cup (e.g. m/z 45, cup 3).

    •   If no peak could be detected, the measurement is stopped.

    •   If not enough peaks could be detected, an error message occurs.
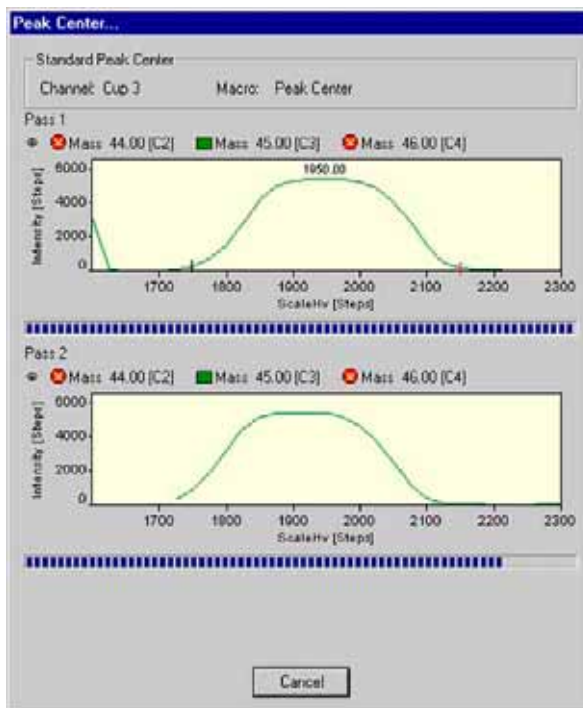
8. Finally, the values of Peak Distance [Da], Peak Width at 10% Height [Da] and Resolution are displayed.
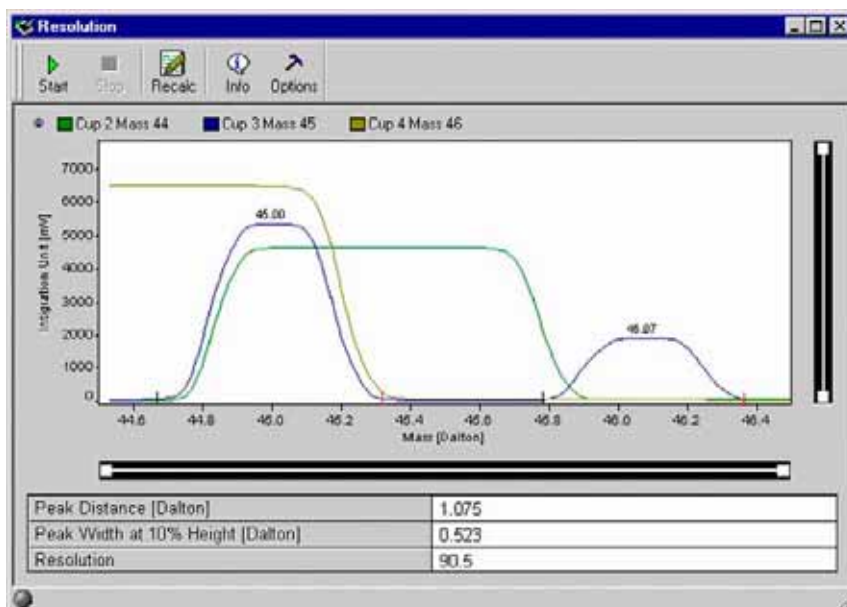
9. Click the **Info** button.

10. Press the Graphik tab.
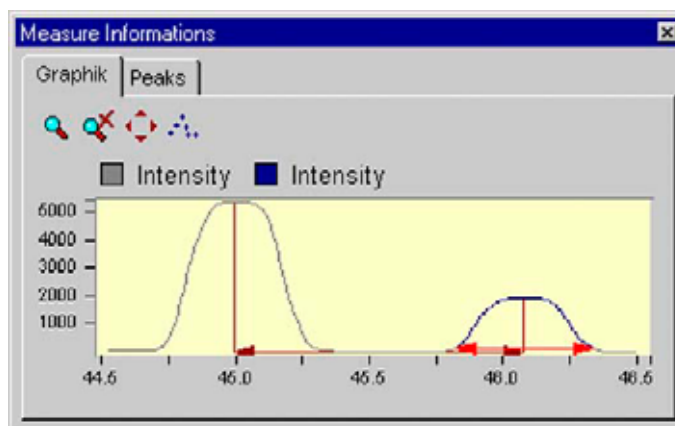
11. Press the Peaks tab.

12. When closing the "Resolution" window, you are asked whether to save the changes.
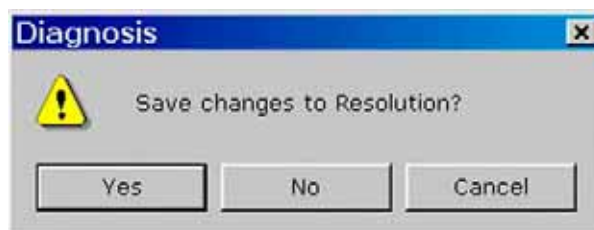
Signal Stability

Signal Stability describes intensity stability (peak height). Intensity on top of the peak is measured for a limited period (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. Signal Stability should be ~ $2*10^{-4}$ (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)

> **Note.** Testing Signal Stability requires a signal of 3 V or more!

Sources of Error

• The slope is usually due to gas consumption during measurement. However, it should be checked, if an unusual result is obtained.

• Instabilities of the emission may cause an unstable signal although a stable high voltage and magnetic field are given.

• Pressure fluctuations (check the oil of the forevacuum pumps!) or temperature fluctuations particularly at the crimps.

Differences to ISODAT Old

• 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 results to ISODAT Old scale: Multiplication by a factor of approximately four.

Testing Signal Stability

1. Click the Signal Stability Icon and press **OK**

   (or double-click the Icon).

2. Press **OK**.
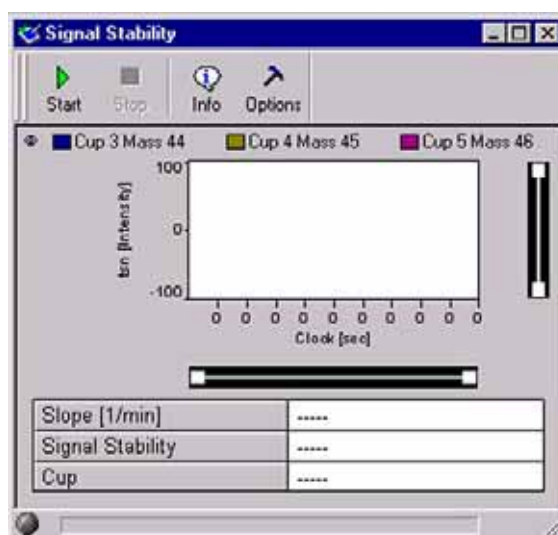
Figure 7-8.   Signal Stability window

3. On "Window" toolbar, the Signal Stability Icon becomes visible.

4. Press the **Options** button.
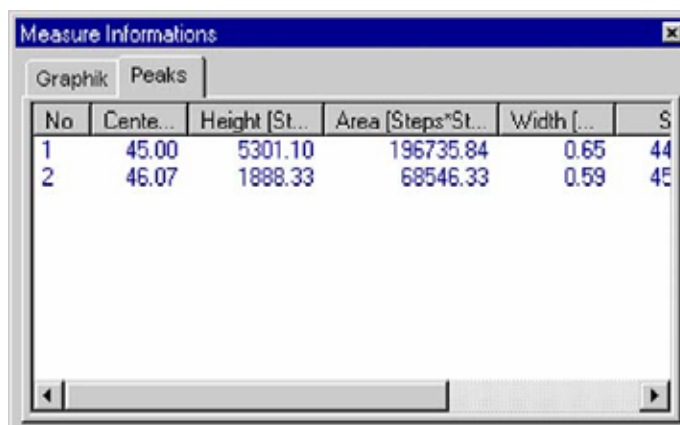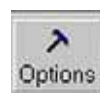
5. From the pulldown menu, select a delay value between 100 ms and 20000 ms.

6. Click **OK**.

7. Press the **Start** button.

8. Testing starts with a Peak Center on the selected cup (e.g. m/z 45, Cup 3).
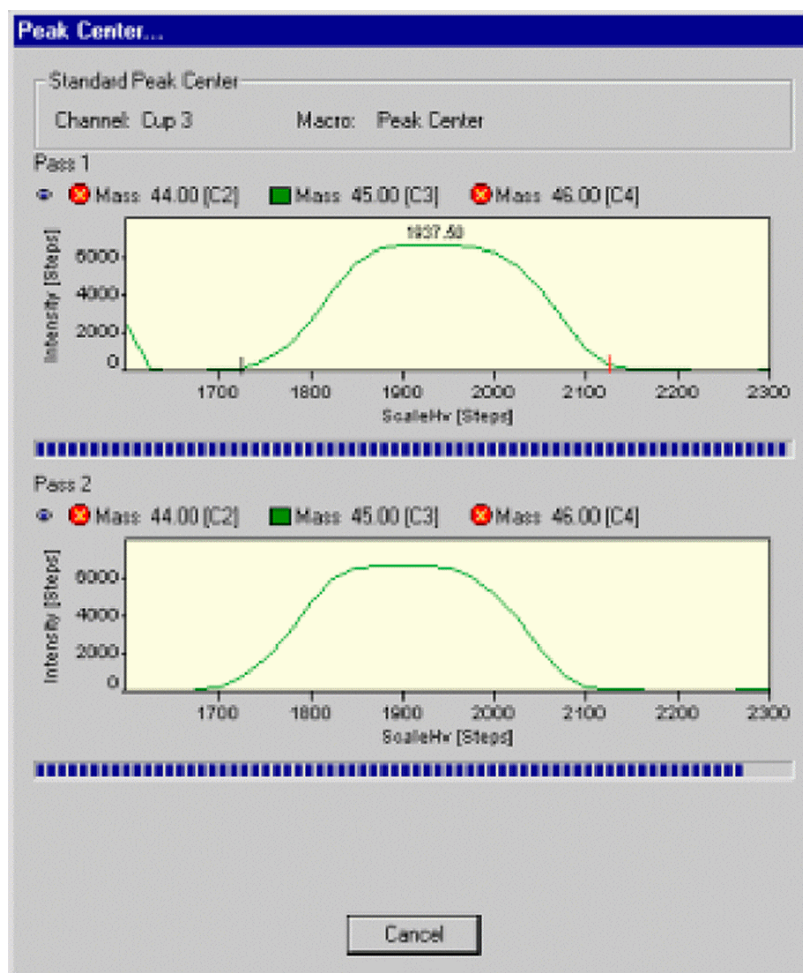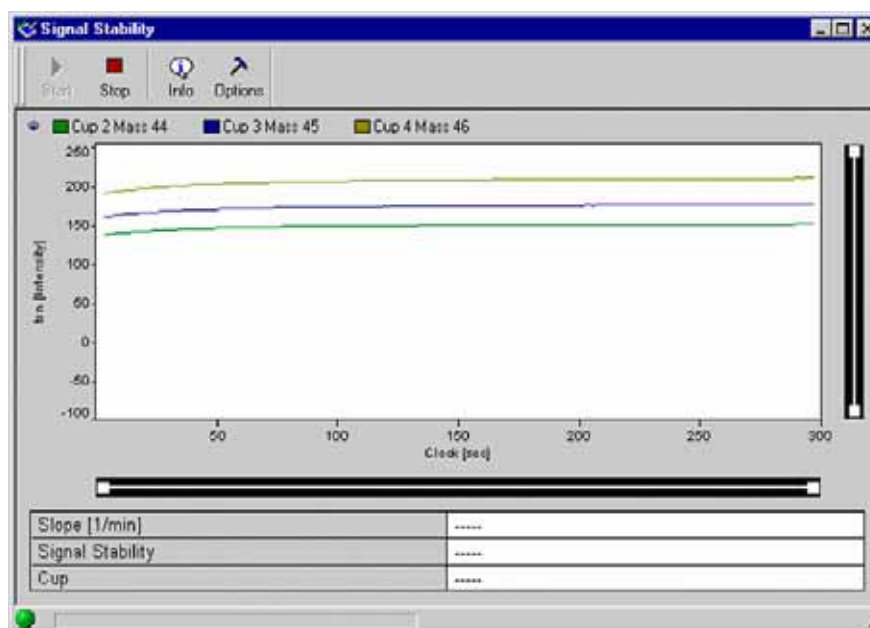
9. Another Peak Center is performed.
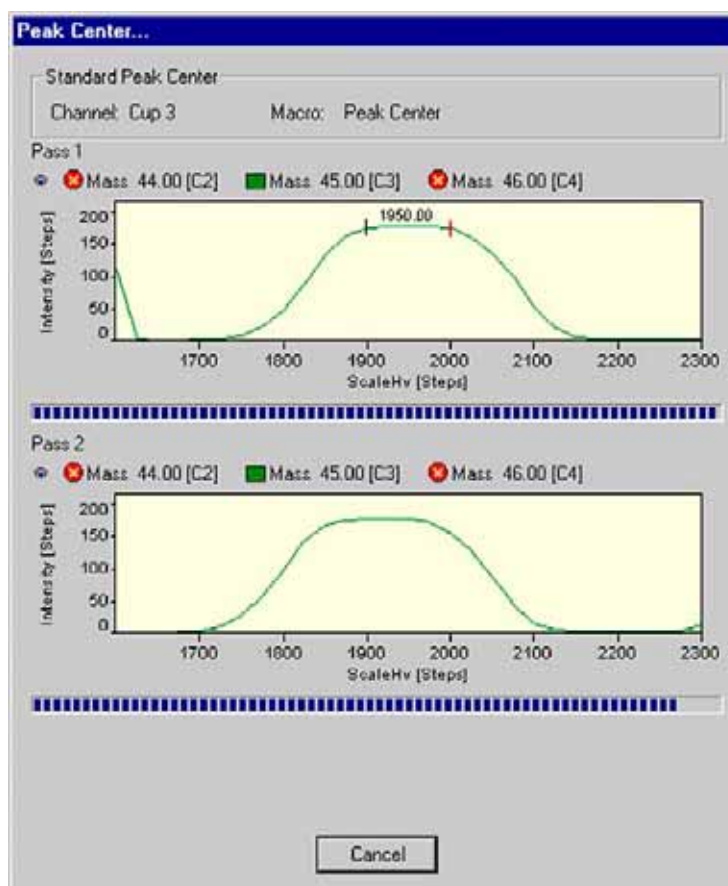
10. Values of slope [1/min], Signal Stability and cup number are displayed.
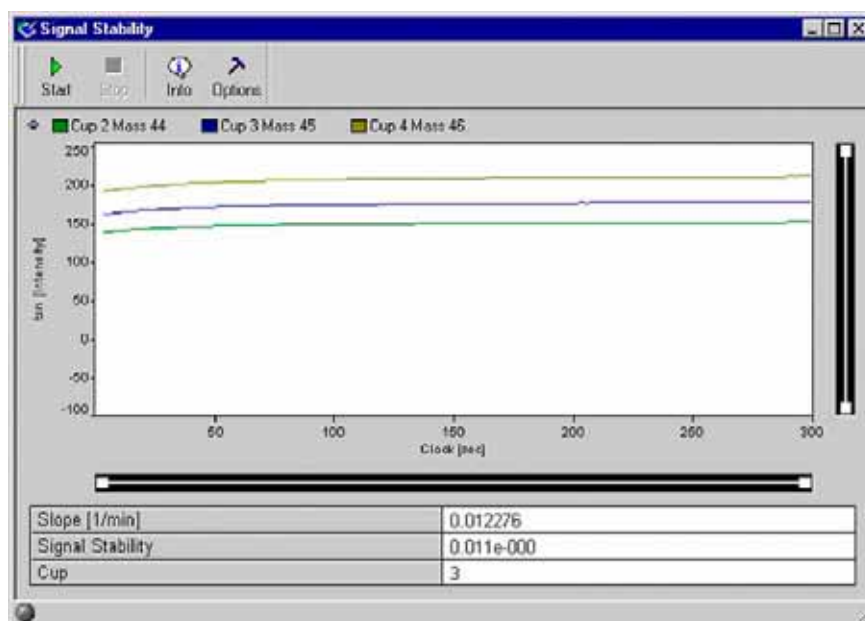
11. Press the **Info** button.

12. Press the Graphik tab.
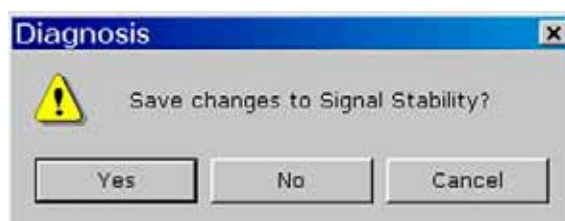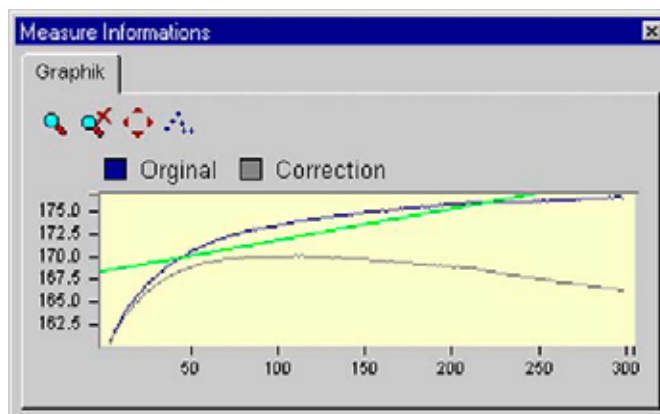
Closing the "Signal Stability" window, you are reminded to save changes.

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.

> **Note.** Testing System Stability requires a signal of 3 V or more!

Two results are obtained:

- Slope of relative mass drift vs. time (time drift)

- Standard deviation of this slope (scatter of the mass)

A value of $5*10^{-4}$ measured over 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 Results to ISODAT Old scale: multiplication by a factor.

Testing System Stability

1. Click the System Stability Icon and press **OK** (or double-click the Icon)
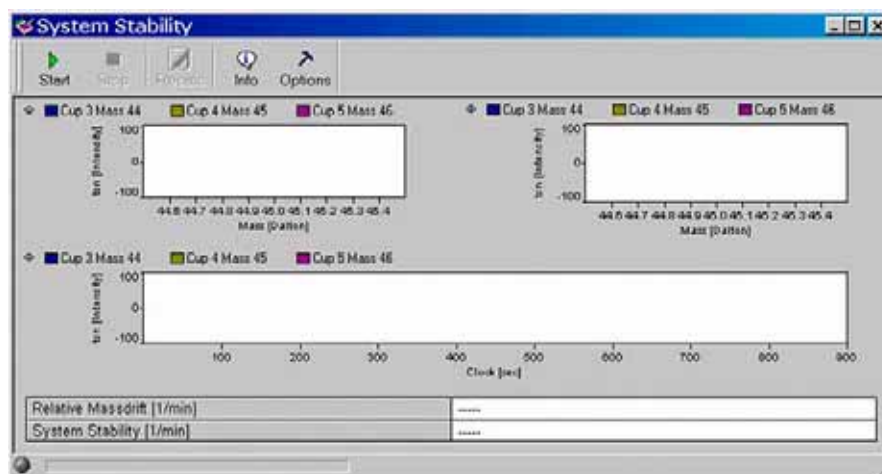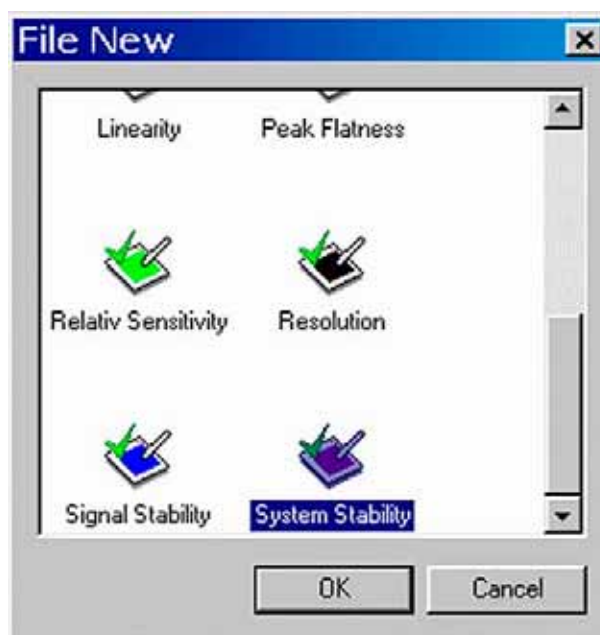
Figure 7-9.   System Stability window

2. On the "Window" toolbar, the System Stability Icon becomes visible.

3. Press the **Options** button.
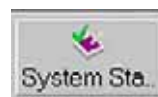
4. If you simply want to accept the Peak parameters without glancing at them, do not press the **Peak Parameter** button. Instead, click **OK**.

5. However, as an experienced user, you may want to look at the Peak parameters. To do so, press the **Peak Parameter** button.

Experienced users may further want to change the gray default values (from Integration Time to Scan Time). This can be achieved via the Advanced Mode.

6. Make your changes or accept the defaults. Then press **OK**.
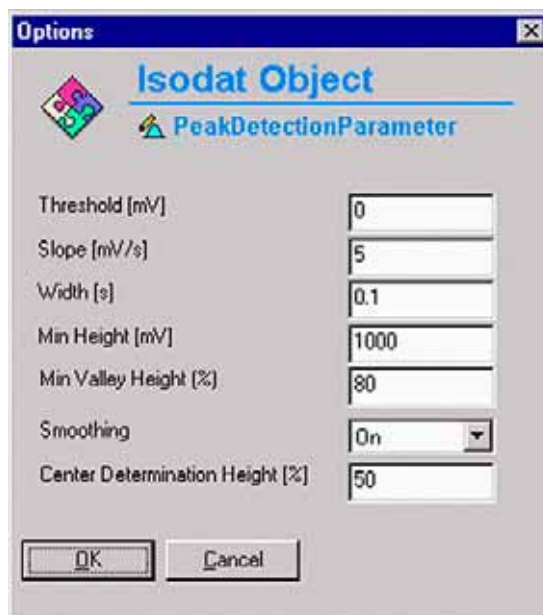
7. Press the **Start** button.

8. Testing starts with a Peak Center on the selected cup, e.g. m/z 45, Cup 3.
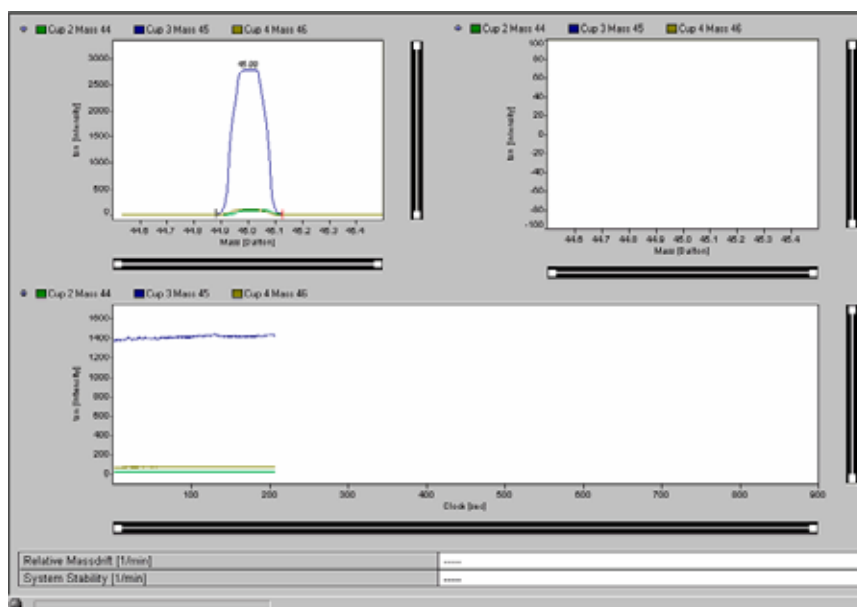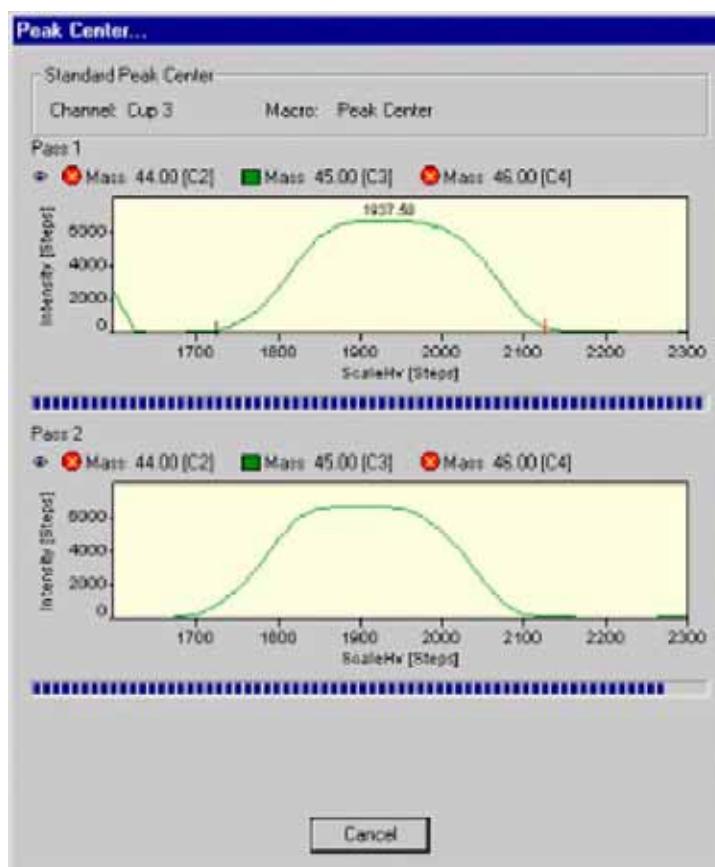
9. Testing continues with another Peak Center on the selected Cup.
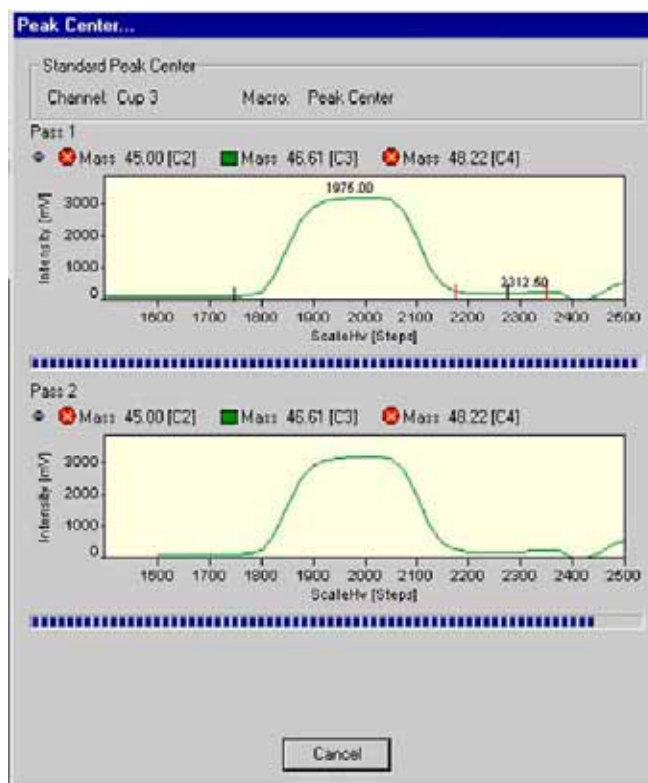
10. Values for System Stability [min^{-1}] and Relative Mass Drift [min^{-1}] are displayed.
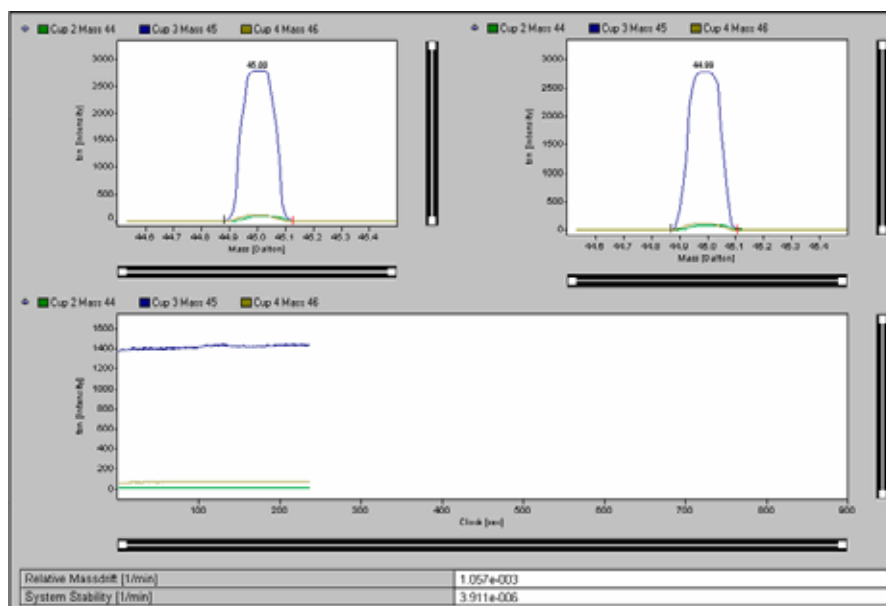
11. Press the **Info** button.

12. Pressing the Graphik tab, the diagram for calculating Slope and Mass Drift is shown together with the respective values.
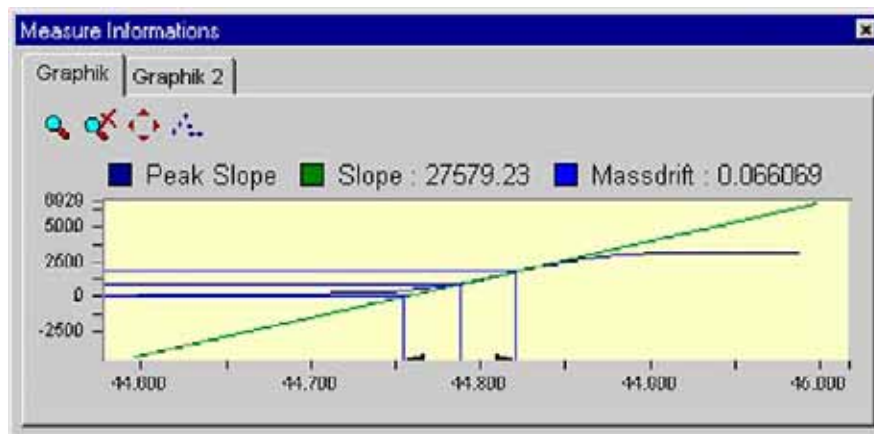
13. When closing the "System Stability" window, you are asked whether to save the changes.
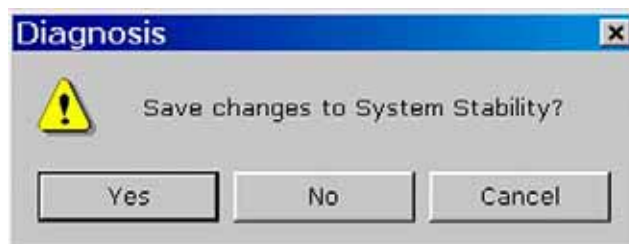
7.3   Conversion to Other Gases

Depending on the number of collector cups installed it may be necessary to switch the feedback resistor of an amplifier when intending to analyze different gases. This is done automatically. An example of a collector assignment with six cups is given below:

Example: Assignment of a Collector with Six Cups (that is, MEMCO 6)

Measuring Channel	1	2	3	4	5	6
VF-converter	1	2	3	4	5	6
element and mass						
N_2	28	29	30			
CO_2				44	45	46
SO_2			64	66		

With this combination, there is no need to switch the feedback resistors when changing from CO_2 analysis to N_2 analysis. The switching is done automatically by ISODAT NT changing the cup configuration. Only in case of switching to SO_2 analysis, it is necessary to switch the amplification factor (done automatically by switching the Gas Configuration).

7.4   Heating Inlet Capillaries

> **Note.** The heating system can be regulated via ISODAT NT.

> **Note.** ISL Scripts can be used to create time programs for the heating process.

If a contamination of the instrument is detected, also the inlet capillaries should be heated. Together with the instrument, a transformer (90 VA/220 V/ 7.7 V/12 A) is supplied for heating purposes. To heat all capillaries simultaneously, it is advisable to acquire additional heating transformers.

1. Before heating the capillary itself, the surrounding of the capillary has to be heated to approximately 80 °C for about 30-60 min, that is, the valves of the inlet system (incl. the valves of a Multiport, if in use), the changeover valve, the ion source and the analyzer housing.

   During the heating period, all valves must be open. Swagelok connectors should be heated separately for a short while using a flame or a heat gun.

2. Plug two cables parallel to one socket of the transformer and clip on the ends with the alligator clips to each end of the capillary to be heated or to the related Swagelok connector.

   Plug the third cable to the second socket and connect the clip to the middle of the capillary. Capillaries supplied by Thermo Electron have a metal sleeve attached in the middle providing better electrical contact.

> **Note.** After the capillary has been wired make sure, the capillary (with or without insulation) has no contact to any plastic surface of tubes, housings, cables etc. to avoid melting or smoldering caused by a hot capillary.

3. Connect the transformer to the mains and switch it on. The heating phase should be controlled by monitoring the signal intensity of H_2O (mass 18, measured on the channel for mass 45; see chapter **Instrument Control** in ISODAT NT Online Help).

   It is recommended to heat also the crimped part of the capillary separately for a few minutes using a flame or a heat gun.

> **Note.** Before removing the upper crimp block mark the parts of the crimping device to avoid mismatch when reassembling.
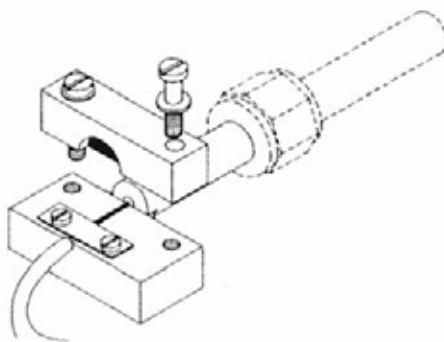
Figure 7-10.   Crimping device at the end piece of a capillary

4. When fitting the crimp block again, make sure the capillary is exactly placed in the grove of the base and the die of the upper block in the crimp of the capillary. After heating the capillary, the flow resistance of the crimp has to be checked and reset to 1 Volt per 10 mbar if required (see topic **Replacing Inlet Capillaries** on page 7-53).

During the start phase of the heating, the signal intensity increases but it decreases and stabilizes later. The best results of decontaminating capillaries are achieved by heating for approximately 6 to 8 hours. With stabilized signal intensity lower than the first signal, a successful decontamination can be assumed. The result can be checked by a zero-measurement, that is measuring the same gas on sample and standard side.

7.5   Replacing Inlet Capillaries

Replacement of an inlet capillary may become necessary in case of contamination or mechanical damage. After replacement, the flow rate of the new capillary has to be set by crimping. The crimping device consists of two metal blocks. The base is attached to the end piece of the new capillary, which has to be fitted to the changeover valve. The second block, to be bolted on top of the base block, holds a metal pin in a spacing, which will squeeze the capillary when bolting the two blocks together. To replace a capillary proceed as follows:

> **Note.** Make sure that all valves are closed before venting the surrounding area of the capillary, which is to be exchanged.

1. Vent the parts of the inlet system and the changeover valve, which are connected by the capillary.

2. Loosen the Swagelok fittings holding both end pieces of the capillary to be exchanged. New capillaries are delivered with close ends. Use a diamond file to cut a capillary end at opposite sides before breaking off the tip. Then smoothen the end of the capillary.

3. Fasten the end pieces of the new capillary with the Swagelok connectors. The end piece with the crimp block has to be connected to the changeover valve. Fasten the upper crimp block loosely onto the base with the capillary.

4. Pump out the inlet system.

5. Admit a proper amount of CO_2 into the inlet system, so that the storage reservoir pressure is about 20 mbar on both sides of the capillary.

6. Start ISODAT NT's Instrument Control.

    a. Activate the Scan window by a click on its frame.

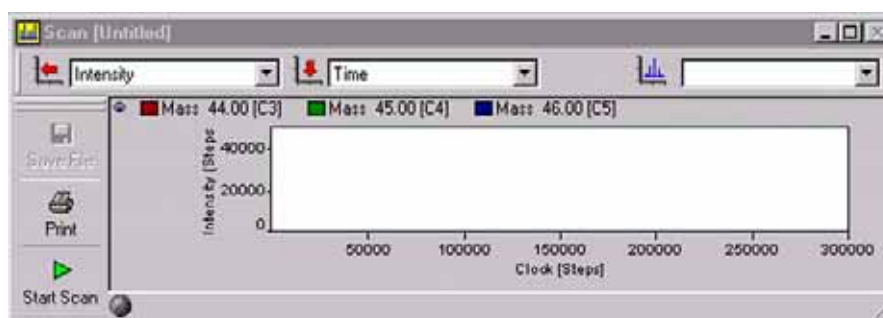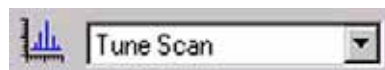**Figure 7-11.   Scan window**

b. Select *Tune Scan*.

c. Press the **Start Scan** button.

7. Tighten the screws of the crimp block carefully and squeeze the capillary until the output signal reaches 1 V per 10 mbar with CO_2 used for measurement.

8. After crimping the capillary needs to be heated (see **Heating Inlet Capillaries** on page 7-51).

Chapter 8

Technical Information

8.1 Introduction

This chapter contains information required for maintenance and repair of the Finnigan DELTA^{plus}XP mass spectrometer. It also includes schematic drawings and spare parts lists as well as instructions for the installation of various hardware drivers.

General Remarks

The DELTA^{plus}XP contains elaborate and expensive components. Only qualified and skilled personnel, therefore, should perform servicing. It is recommended that the Thermo Electron service by called if there are any uncertainties or if difficulties arise. It is further recommended to use original Thermo Electron spare parts only. Note that many adjustments can be made only by the use of special tools and instruments, which are not supplied with the system. (See for references within the manuals.)

Before starting maintenance and repair, please read the appropriate chapters of the manuals.

Before calling the Thermo Electron service, please try to localize the defect! A precise description of the defect will ease the repair and reduce the costs.

> **Warning.** Some parts of the DELTA^{plus}XP mass spectrometer are at dangerously high voltages! Therefore, opening the electronics cabinet is only allowed for maintenance purposes by qualified service personal.

CAUTION!

> **Caution.** When replacing fuses, only use the correct types!
>
> **Caution.** Be careful when servicing the vacuum system. Abrupt opening to atmosphere might destroy the filament or damage the collector system and other expensive parts.

When working with solvents might and sample residuals, please consider your regional safety instructions!

Maintenance

To maintain optimum mass spectrometer performance, the user must perform routine preventive maintenance. Please, see topic **Maintenance** on page 8-44.

Spare Parts Lists

In the diagrams attached to the spare parts lists, the individual spare parts are shown with item numbers. The parts are classified by these item numbers in the corresponding lists and provided with a designation and a part number.

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When ordering a part, always give the designation and the part number. Also provide the service number of the instrument in question. In this way, the processing of your order will be expedited.

Service Instructions

Wherever necessary, service instructions supplement the spare parts lists.

Repair Request Forms

If parts are to be returned to the factory for repair, please fill in the repair request form and enclose it with the shipment. In this way, you will help to avoid delays frequently caused by inquiries, which become necessary if parts are returned to us without reference to the instrument to which they belong or without any details about the faults observed.

Shipment Lists

Because of our modular instrument concept it might happen that parts are listed which are not supplied with your instrument. Please note that listing a part does not imply that this part is necessary required for your particular instrument configuration.

Technical Modifications

Continuous improvement of the performance of our products may result in technical modifications. We are always striving to have the spare parts lists up-to-date.

Safety Rules for the Repair Service

Thermo Electron, Finnigan mass spectrometers frequently are used for analyzing materials, which are insalubrious. In these cases, usually certain parts of the system will be contaminated.

To protect the health of our employees we ask you for some special precautions when returning those parts for exchange or repair.

Mass spectrometer parts, which have been contaminated by hazardous materials, we can accept only if they have been decontaminated prior to return. Hazardous materials are those materials listed up on the MAK list (Maximale Arbeitsplatzkonzentration) and on the EPA (Environmental Protection Agency) priority list.

Additionally such materials are enclosed which due to their structure and the applied concentration might be toxic or which in publication are reported to be toxic. Finally, such materials are concerned which in combination with other present materials will generate synergetic hazardous effects.

Take care that vacuum pumps and all other parts which had been in contact to hazardous materials, will be properly decontaminated prior to return to Thermo Electron Bremen.

Parts contaminated by radioisotopes are not subject to return to Thermo Electron Bremen neither under warranty nor under the exchange part program.

When returning parts to Thermo Electron Bremen, the use of our repair-covering letters is obligatory.

State by your signature on this repair-covering letter, that the returned part had been decontaminated and is free of hazardous materials.

We hope that the analysts of our customers will understand these safety regulations.

8.2 Installation of Board Drivers

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

"Serial Solutions" COM Port Extension Board Driver

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

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

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

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

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

1. To install the driver, insert the "Serial Solutions" CD into your drive and close it. The CD is shipped with your system.

2. If Auto Run is deactivated on your computer, press the **Start** button and then **Run**. If Auto Run is activated on your computer, proceed with step 3.
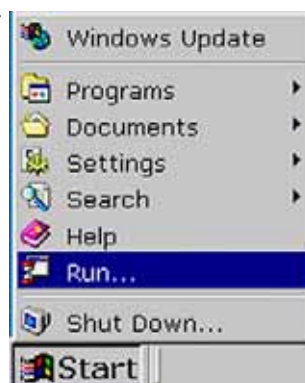
3. Click **Browse**.
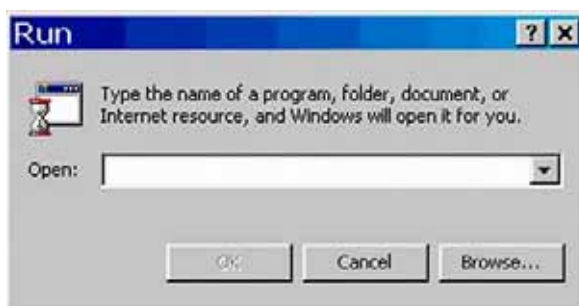
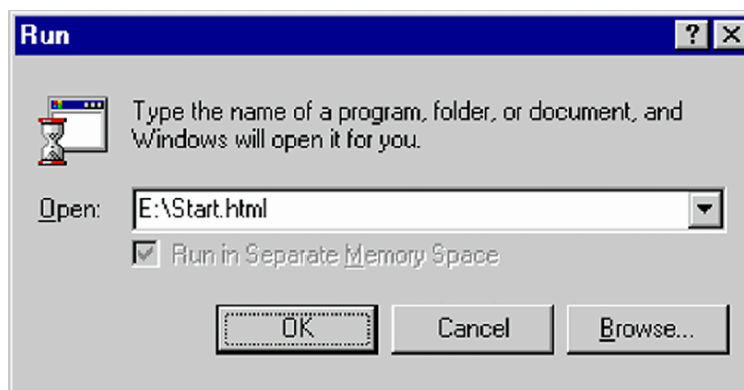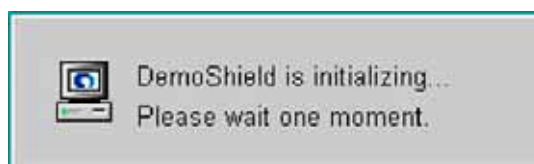4. Select your CD drive, e.g. "E:\Vscomdriver". From the Files of type pulldown menu, choose All Files (*.*). Click the file *Start.html* and then Open (or double-click *Start.html*).
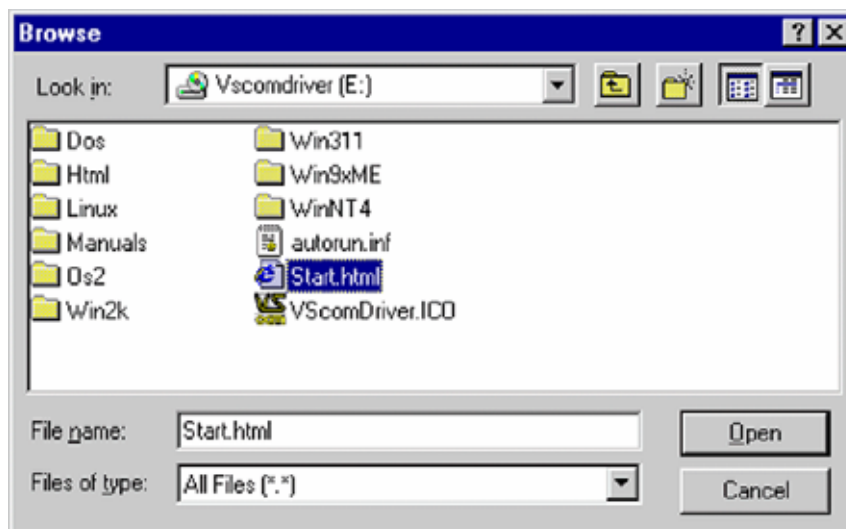
5. Press **OK**.

6. Wait, while the DemoShield is initializing.

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

8. Press **Current**.

9. Click Windows NT.

10. Press Install.

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

12. Carefully read the instructions about closing all applications and copyright law. Click **Next** to continue.

   If Setup found a previous version, continue as described below.
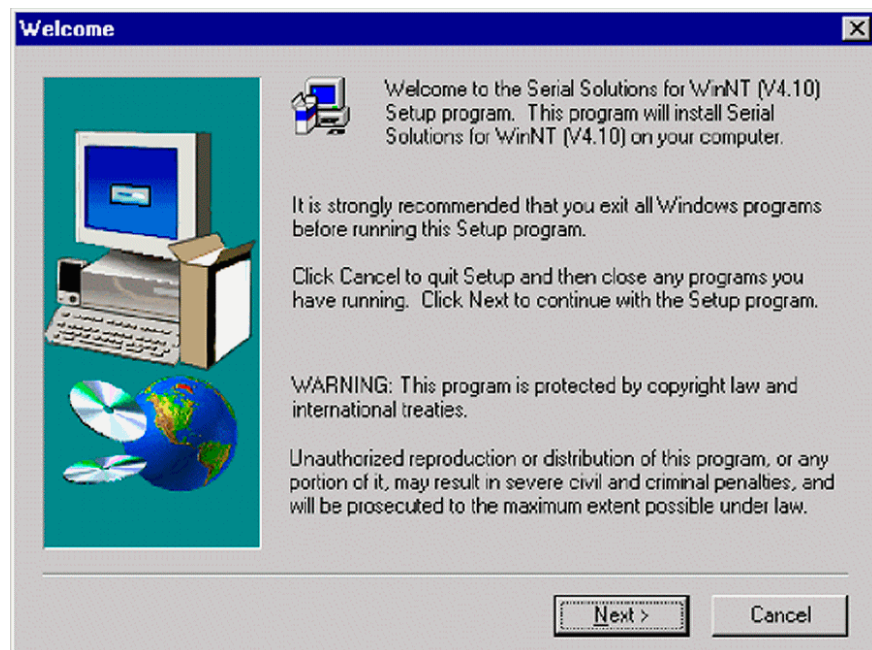
13. Wait, while Setup is copying the files.

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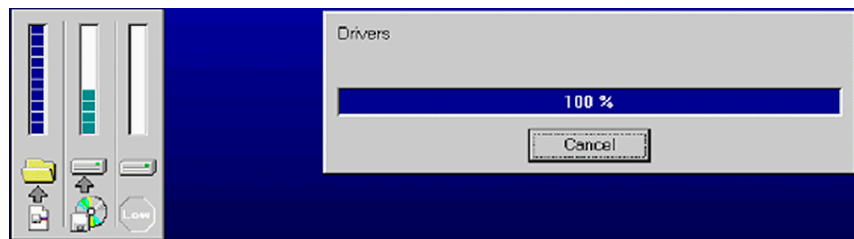
14. "Serial Solutions for Win NT" has been installed on your computer.

15. Click **Finish** to complete Setup.

16. Press the **House** button.

17. Click **Quit**. Then reboot your
    system.

Reinstalling a previously installed Serial Solutions Driver

1. "Serial Solutions" Setup has detected a previously installed version.

2. Choose Re-install.

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

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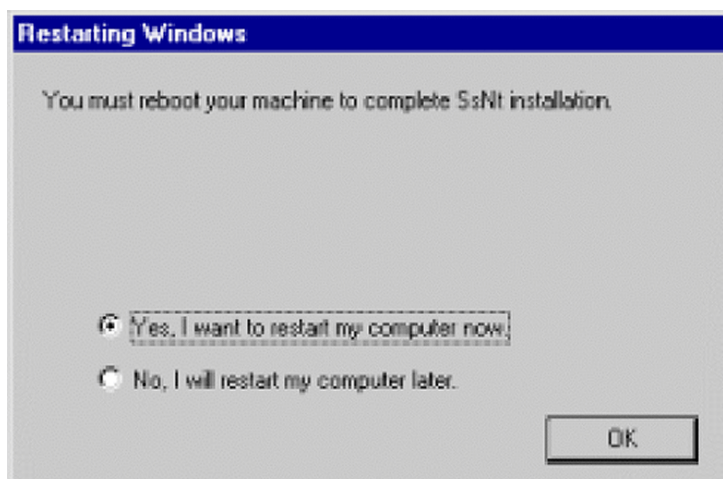> **Note.** If your computer hangs up during installation or reboot, shut it down and try to install again. If your system hangs up again, plug the PCI Dual RS 232 Card to another slot. Then restart your system without reinstallation.

Installing IEEE Interface Board Drives

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.

"National Instruments" GPIB PCI IEEE Board - Installation

Install driver from "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.

1. 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).

2. Wait, while Installation is being prepared.
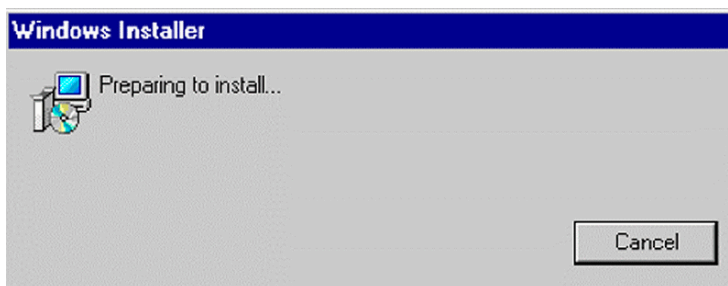
3. Wait, while the NI-488.2 Installation Wizard appears and searches for previously installed applications.
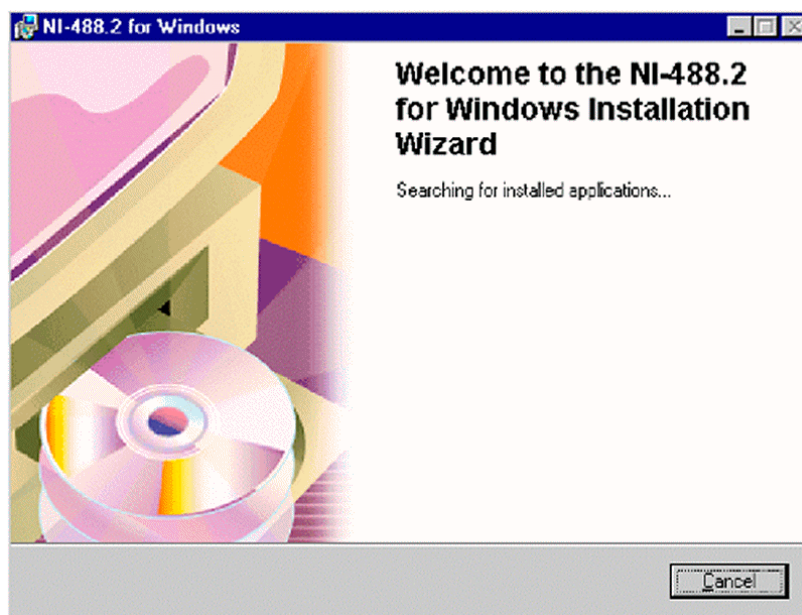
4. Click **Next**.
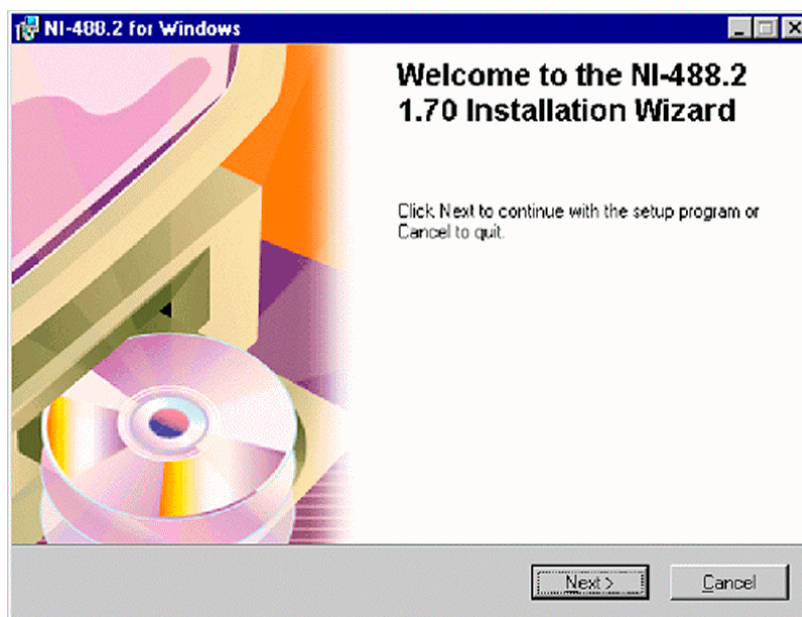
5. Carefully read the "National Instruments" Software License Agreement. To continue, press **Yes**.
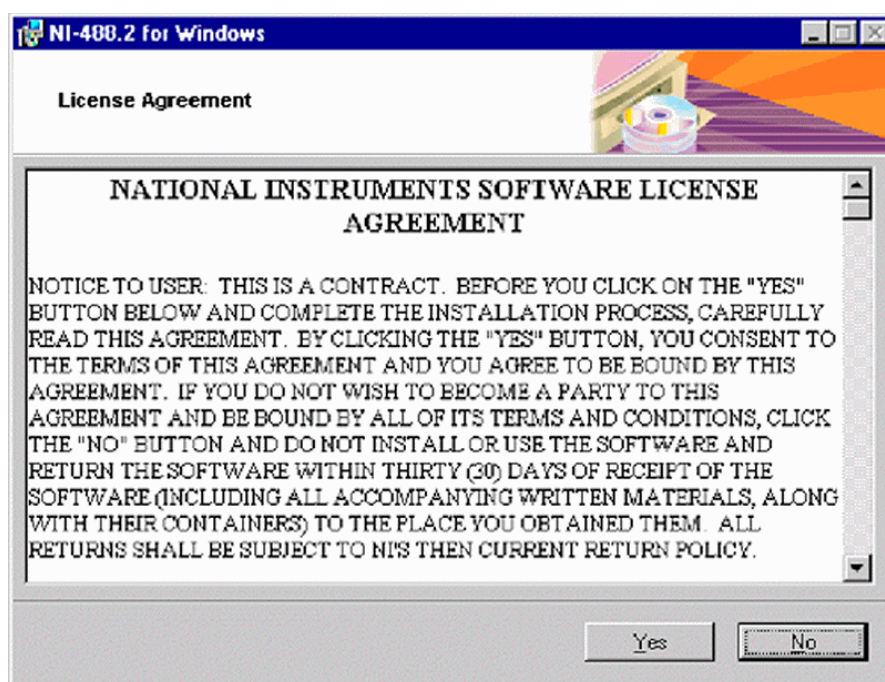
6. Take your registration decision. Then press **Next**.
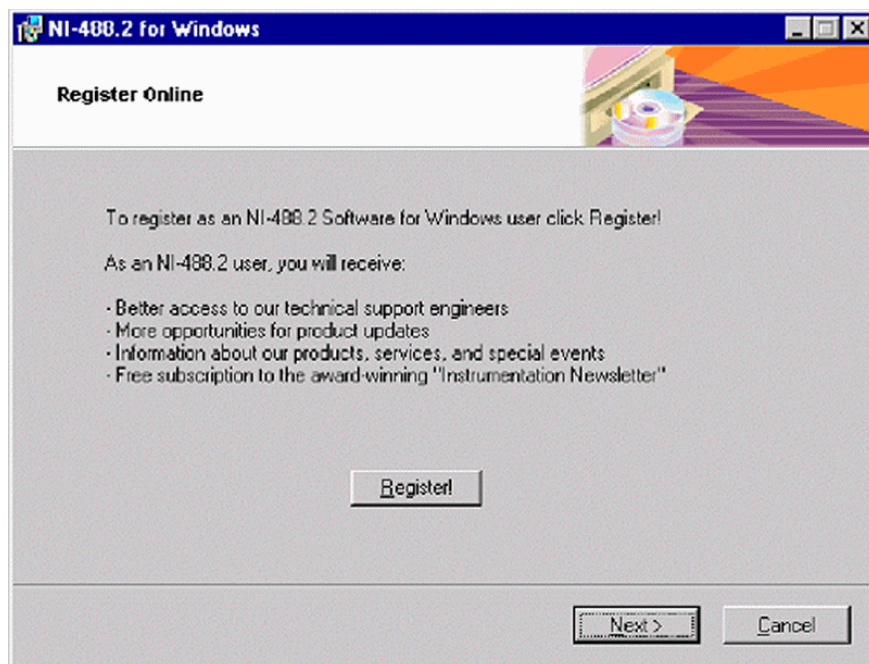
7. To select the destination directory, either accept the proposed path and press **Next**. Then continue with step 8.
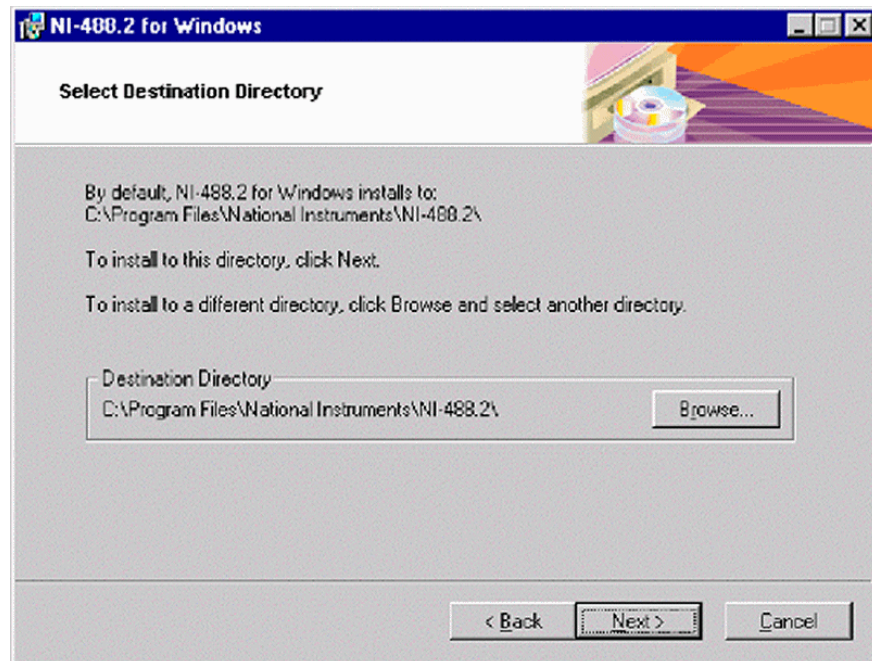
Alternatively, click on the **Browse** button to display a folder selection dialog box. From the Location of folder pulldown menu, choose the alternative destination folder. Note the according entry in the Selected Path box. Finally, press **OK**.
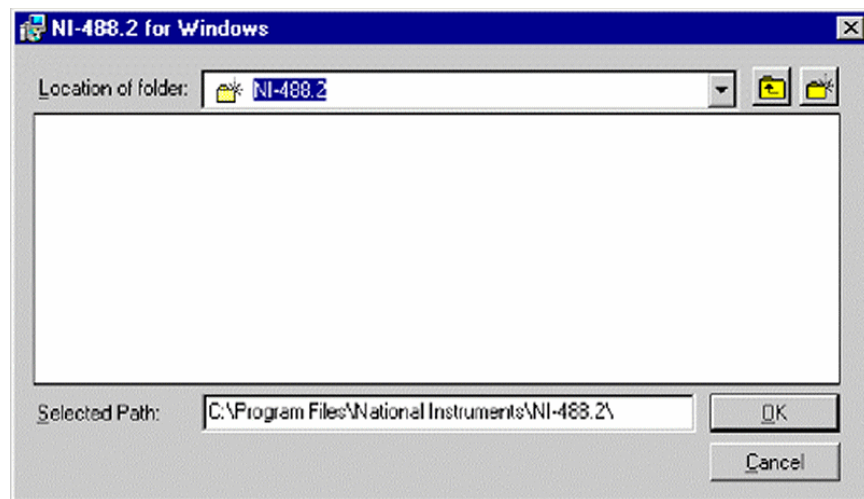
8. Select the Installation type. **Typical** is recommended for most users, while Custom is recommended for advanced users. Finally, press **Next**.

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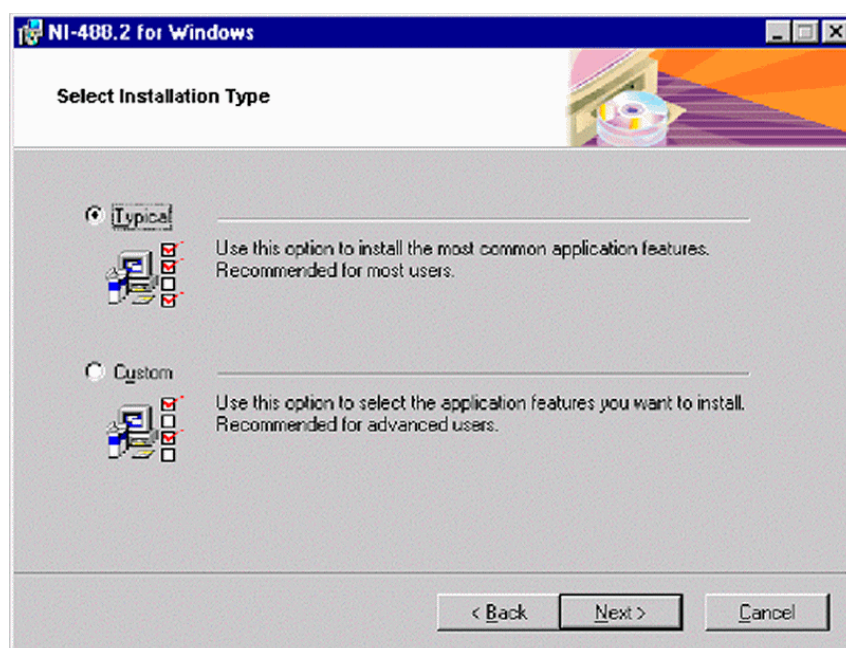
9. The NI-488.2 software is now ready to be installed.

    To begin the Installation, press **Next**.
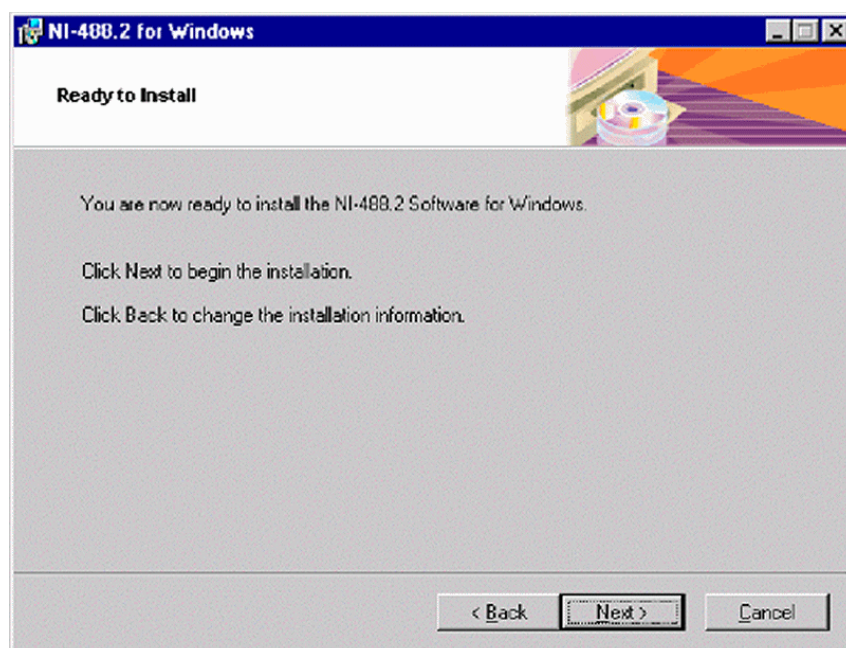
10. Wait, while Initialization begins.
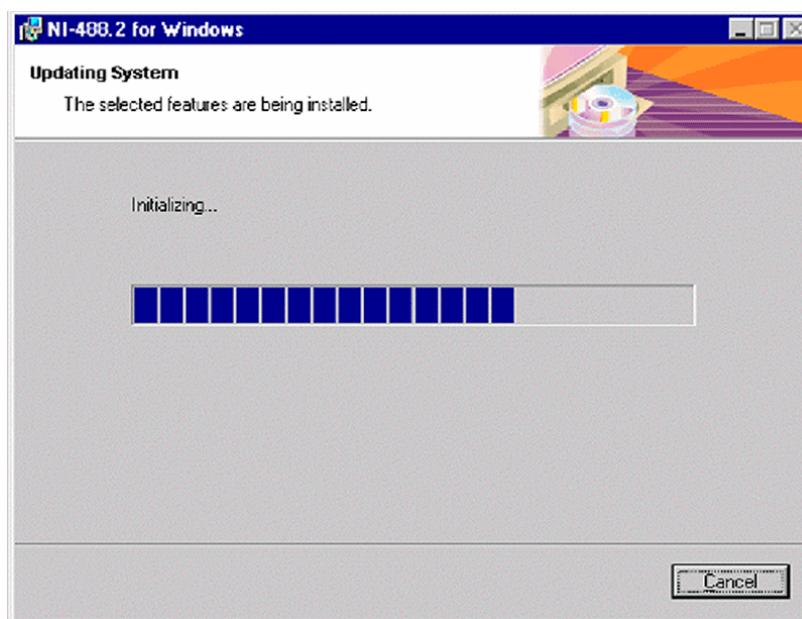
11. If you did not close all other applications yet (as was recommended in step 1), you are reminded to do it now:

    Click on the open applications' entries right to the **Start** button and close all of them. Finally, press **Retry**.
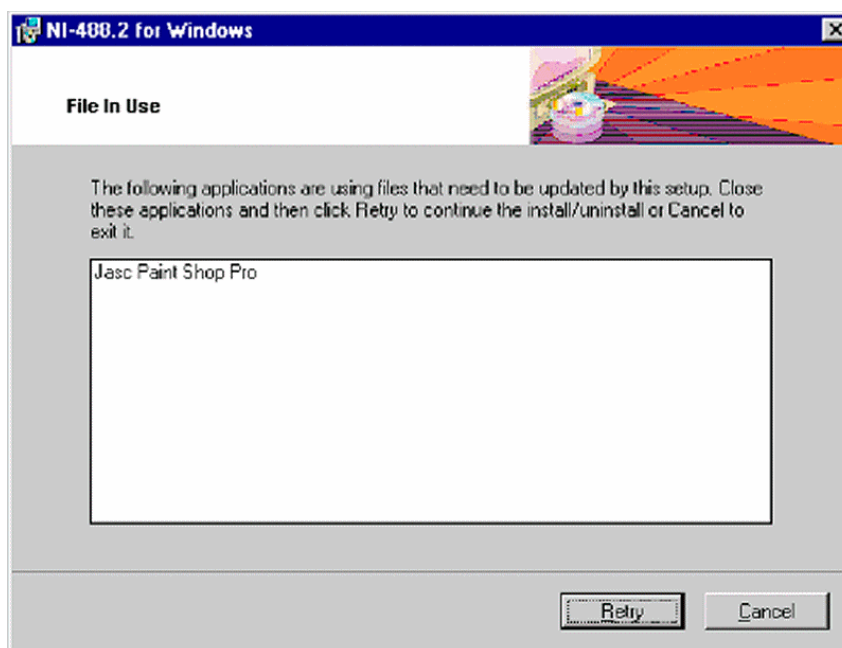
12. Wait while Initialization is terminated.
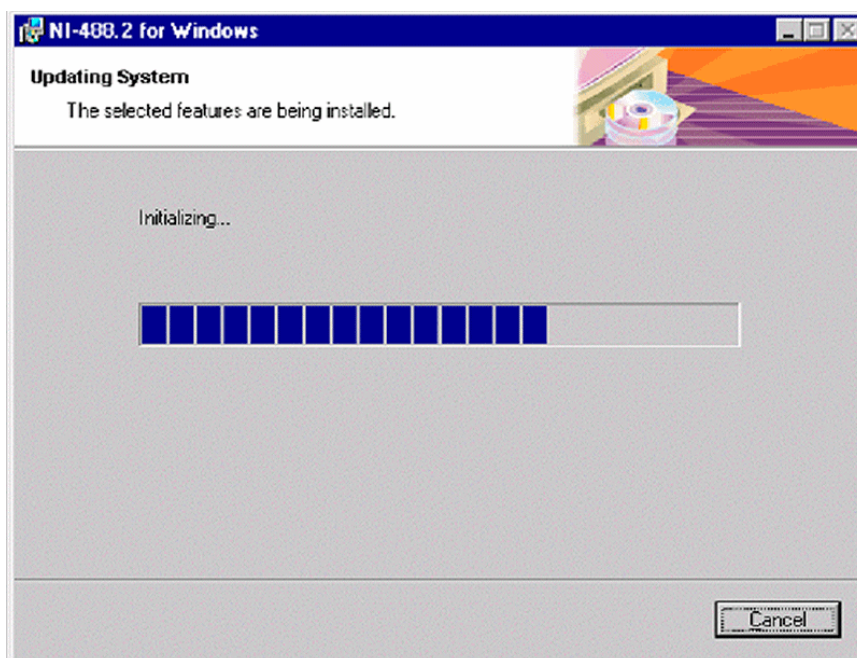
13. Wait, while the files are copied.
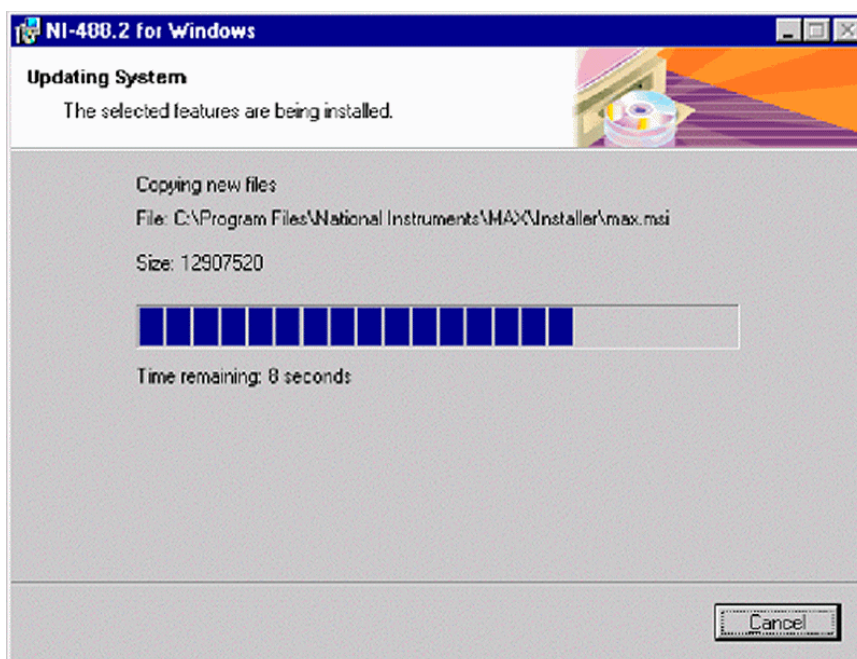
14. Possibly, you might be reminded that Acrobat Reader 3.0 (or higher) was not found on your system. It is necessary to read "National Instruments" documentation files, but you can install them later. Press **Next**.
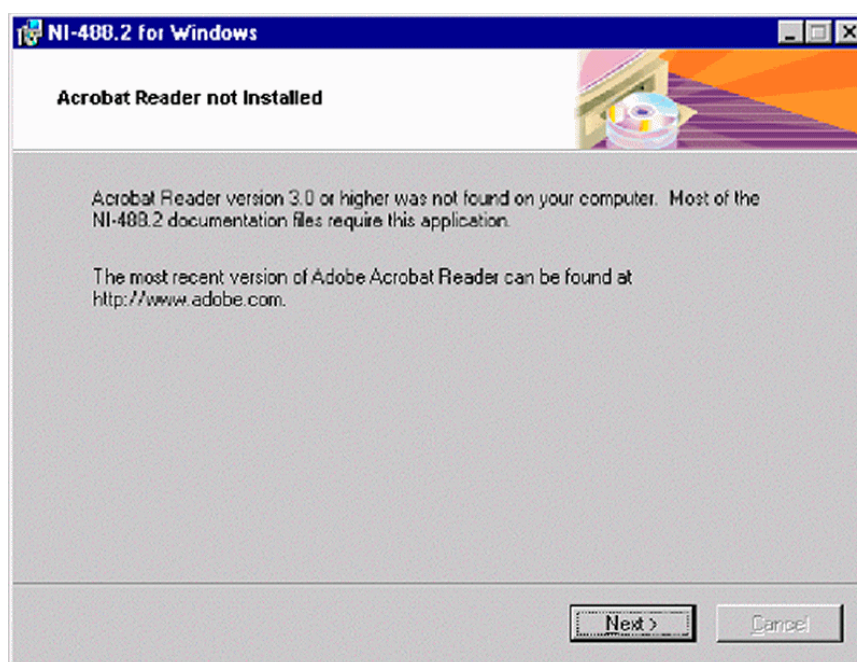
15. This box informs about separate NI-VISA Installation. This will be done after this installation is finished (see topic **"National Instruments" GPIB PCI IEEE Board - Visa Installation** on page 8-24). Click **Next**.
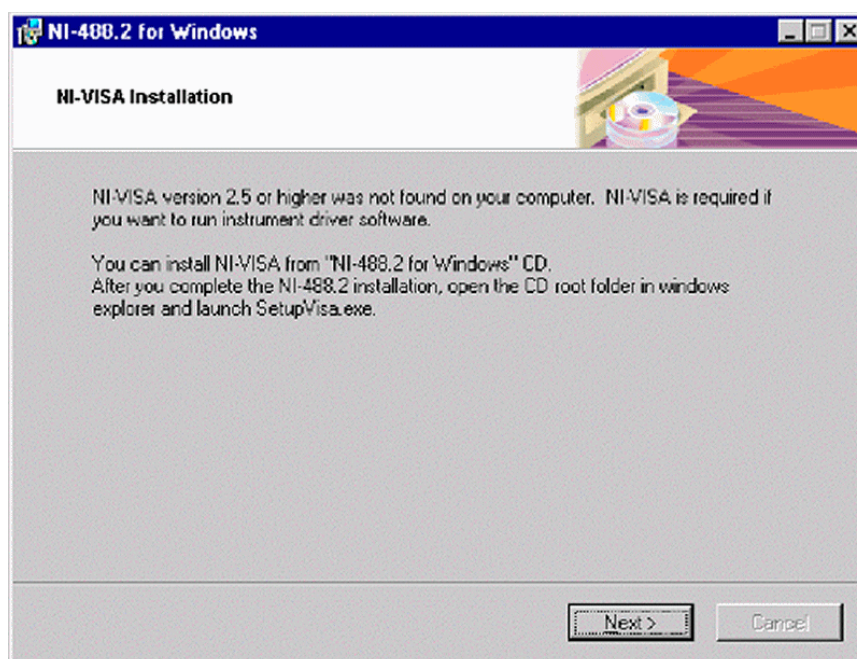
16. The NI-488.2 Software has been successfully installed. Click **Finish**.
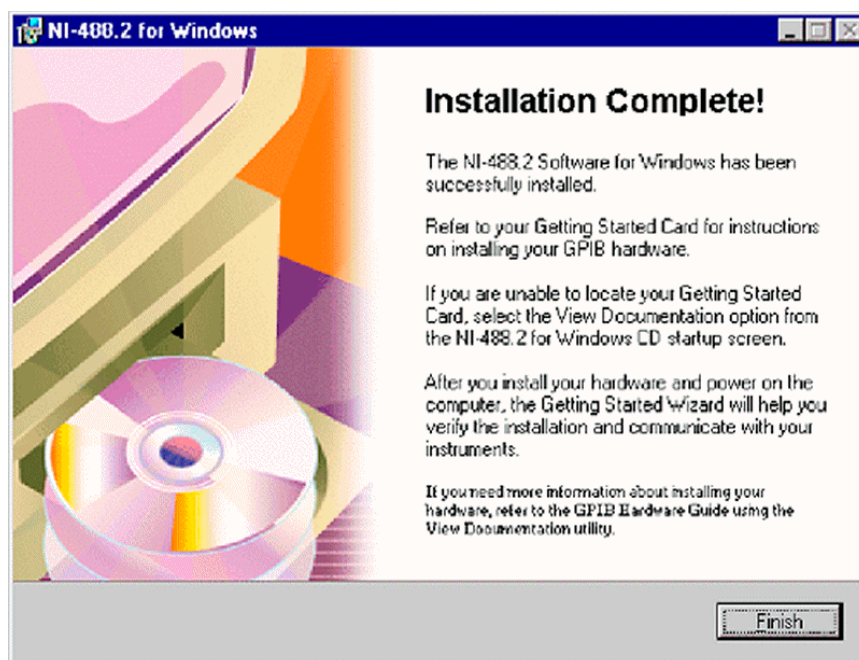
17. On your desktop, notice the new NI-Icon.

"National Instruments" GPIB PCI IEEE Board - Configuration

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

1. To configure it, press **Start > Programs > National Instruments > NI 488.2 > Getting Started**.
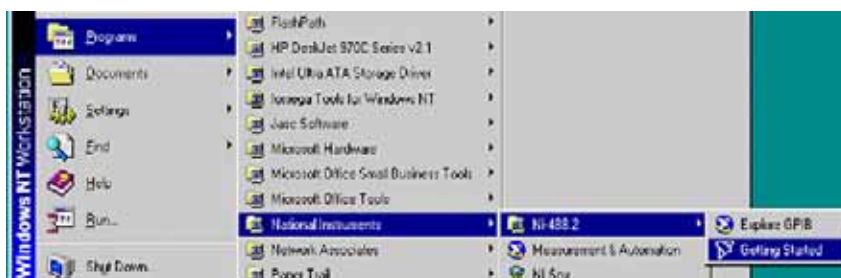
2. Point with the mouse at Configure your GPIB interface and click.

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

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

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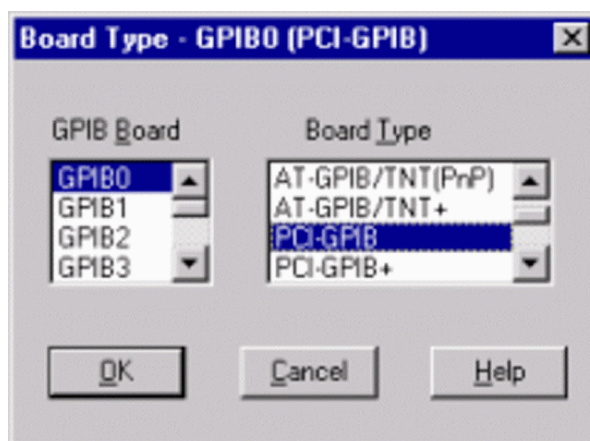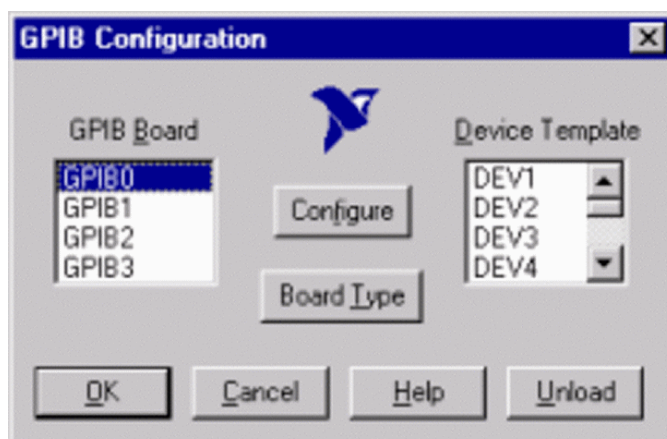
5. From Board Type pulldown list on the right, select *PCI-GPIB*. Press **OK**.

6. Press **Configure**.
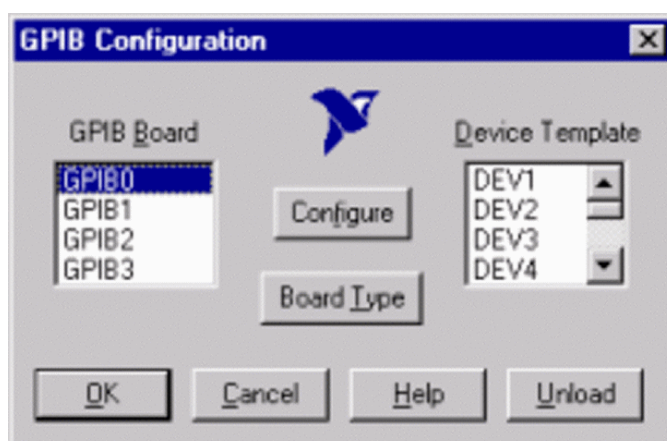
7. Press **Software >>**.
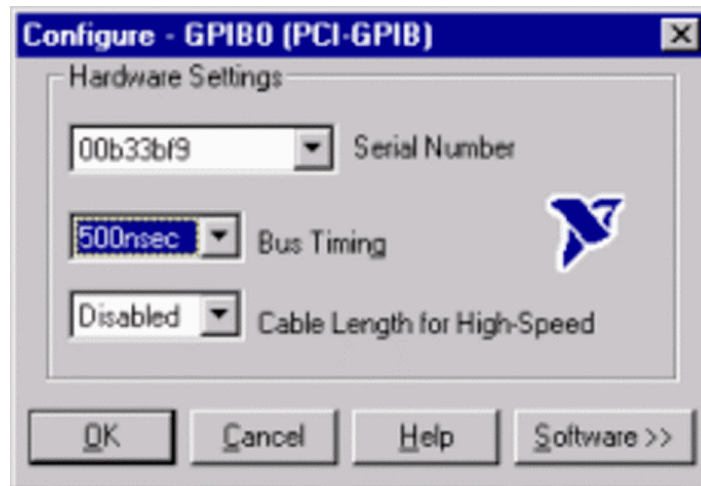
8. On the next window, tick the check boxes as shown in the figure below. Click **OK** and confirm with **OK** on the following window (see above).

The configuration is finished now and the system starts the verification of the board driver (see topic **"National Instruments" GPIB PCI IEEE Board - Verification** on page 8-23).

Under some circumstances, the following window may appear:

The error statement shown on the message box in the figure above 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.

"National Instruments" GPIB PCI IEEE Board - Verification

After installing and configuring your GPIB-PCI driver, verify it as follows.

1. Point at Verify your hardware and software installation and click.

2. 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**.
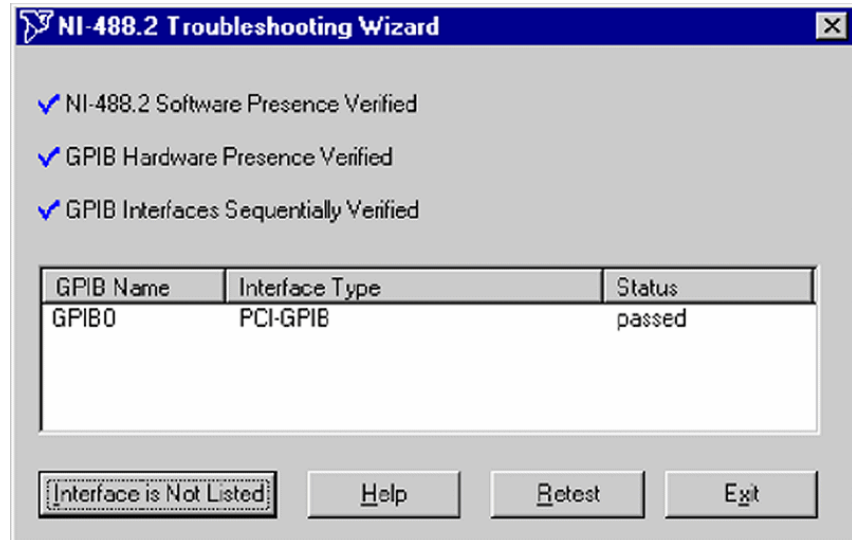
"National Instruments" GPIB PCI IEEE Board - Visa Installation

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

> **Note.** Close all applications!

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

Thermo
ELECTRON CORPORATION
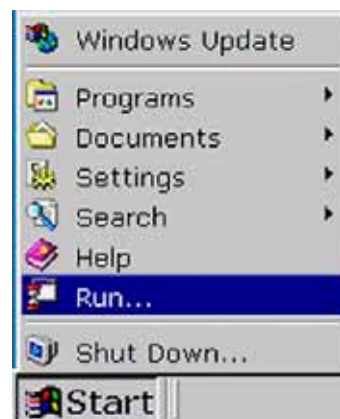
4. Press **Browse**.
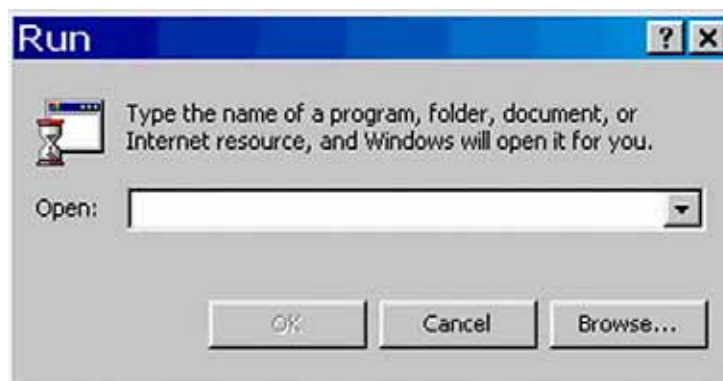
5. At the Look in pulldown menu, select your CD drive [e.g. Ni4882 (F:)].

   Select *SetupVisa.exe* and press **Open** (or double-click *SetupVisa.exe*).
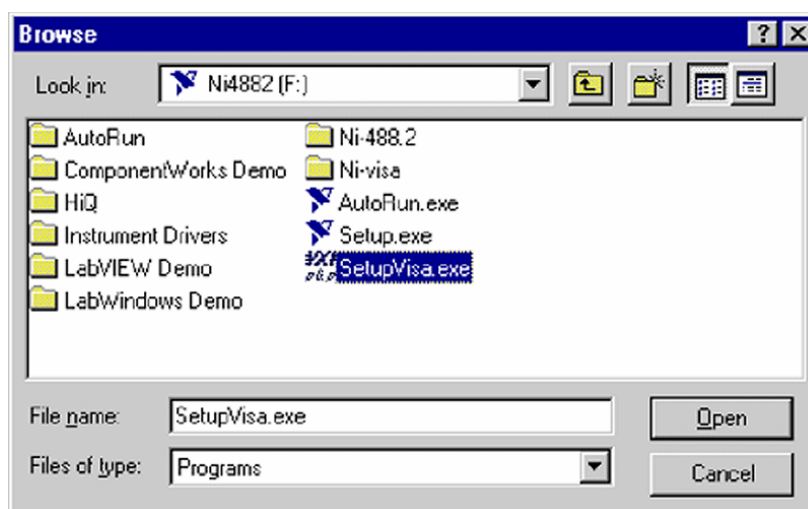
6. The path to "SetupVisa.exe" appears. Click OK.
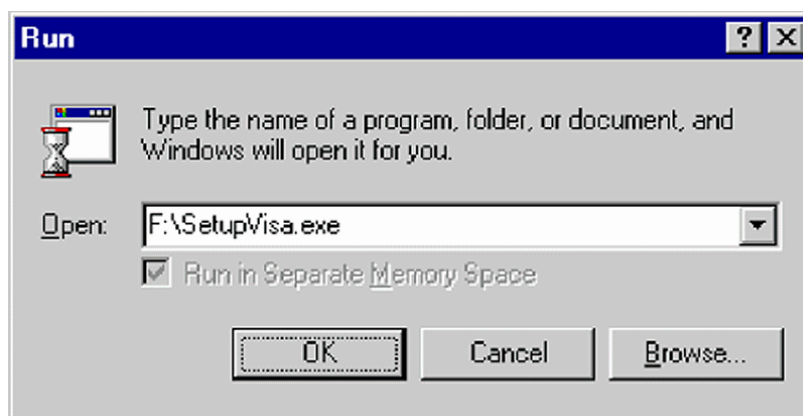
7. Wait, while the Installation program is being prepared.

8. You are reminded to close all applications. To do so, follow the instructions on the dialog box. Then press **Next**.

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

Thermo
ELECTRON CORPORATION
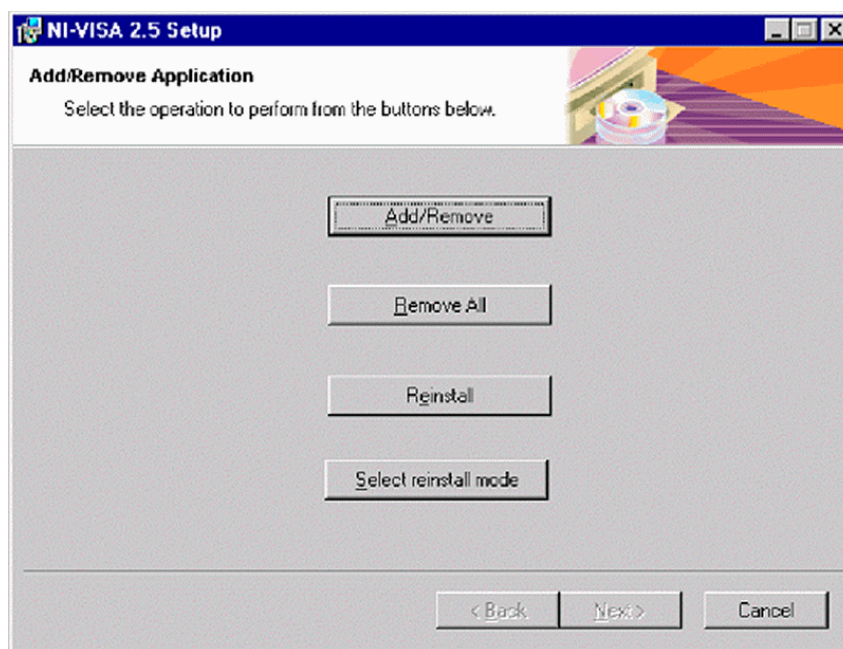
10. Read the License Agreement carefully and click **Agree** to proceed.
    Disagree would lead to exit Visa Setup.
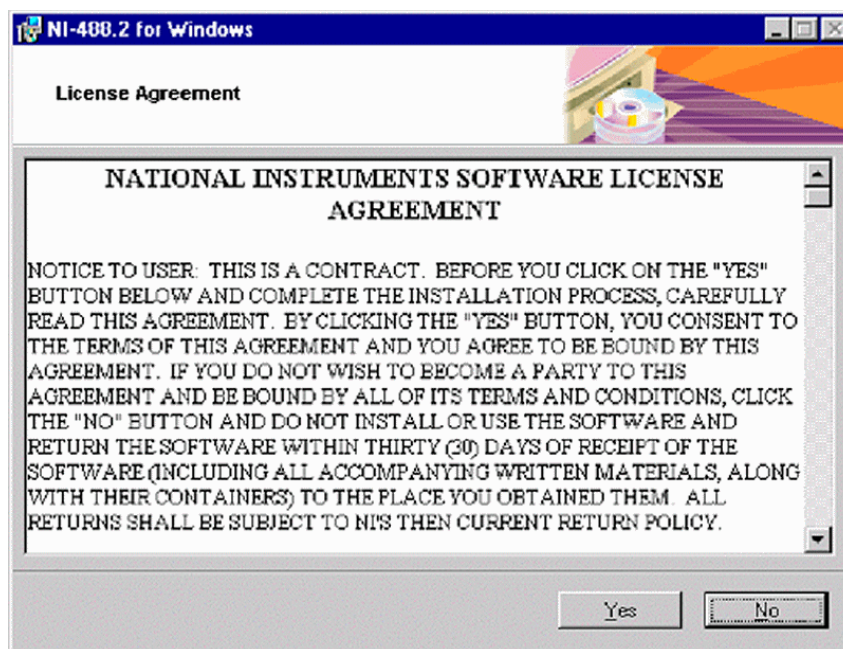
11. The Installation program proposes a destination folder for the NI-Visa
    Software (e.g. *C:\VXIpnp*). To choose another folder, press the upper
    Browse button.

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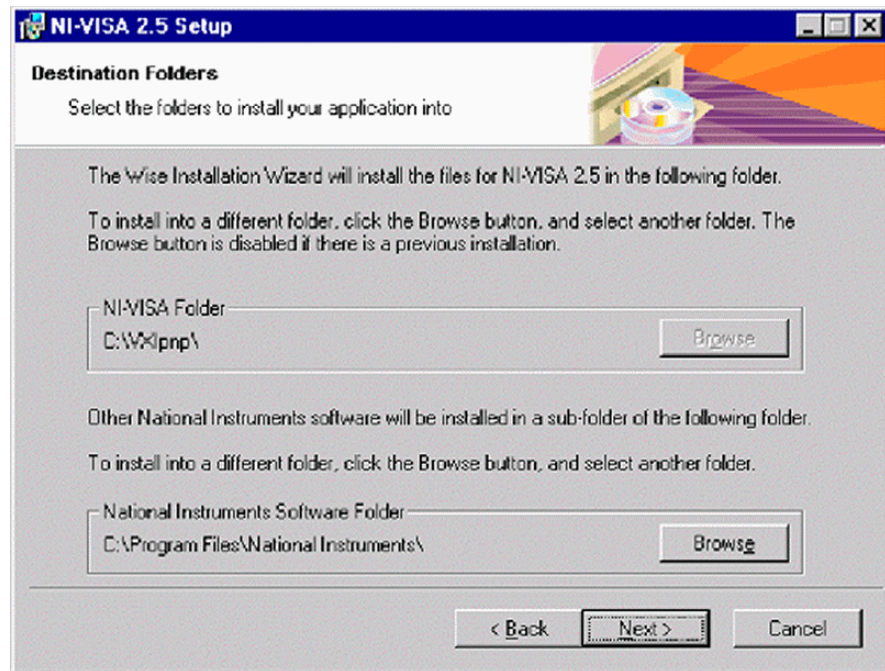
Beneath it, another folder is proposed for the installation of other NI Software (e.g. *C:\Program Files\National Instruments*).

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

12. Choose a folder (for the other NI Software). In the Folder Name box, the respective path is displayed. Press **OK**.
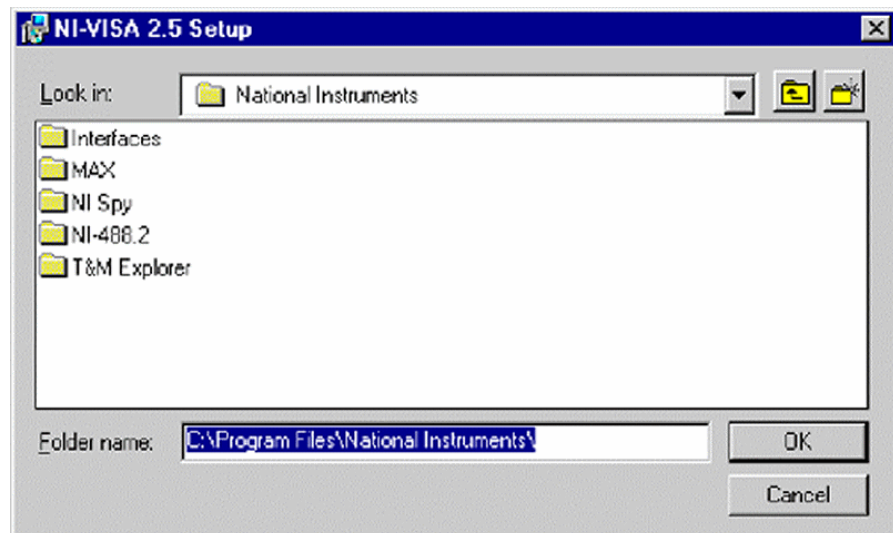
13. To install all features, select Complete (recommended) and press **Next**, or use Custom (advanced users) to install selected features and press Next.
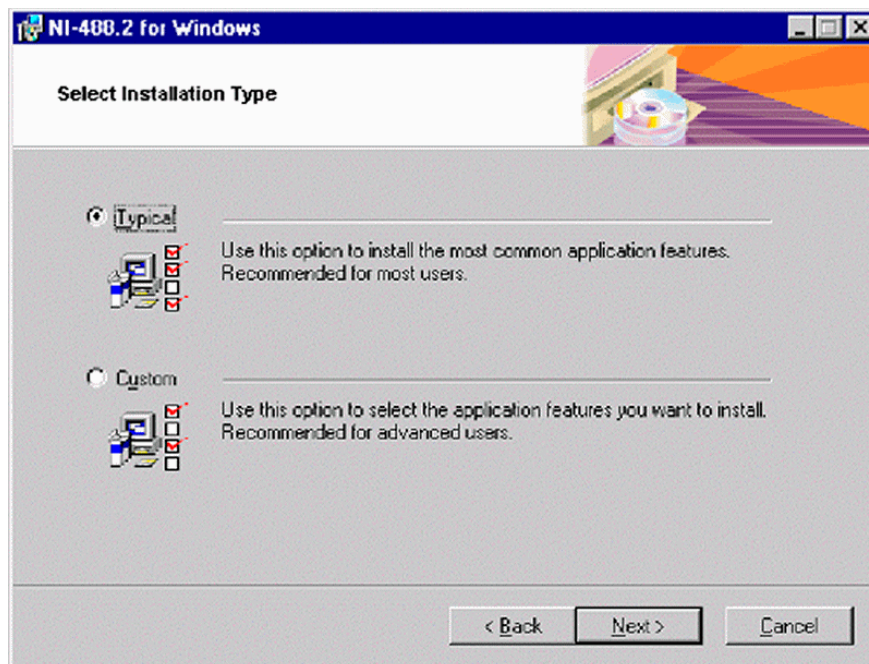
14. To start installing the files, press **Next**.
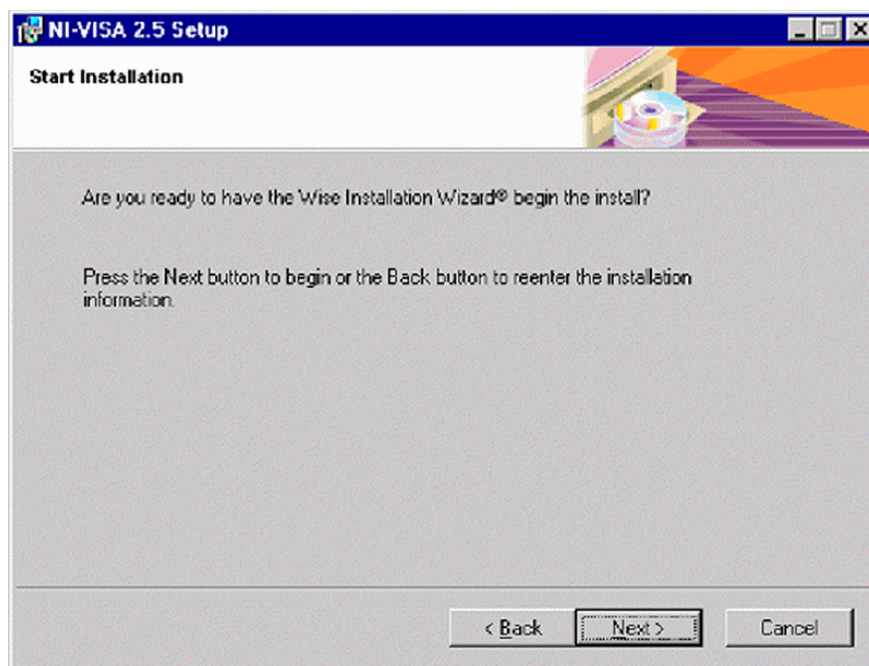
15. Wait, while the initializing takes place.
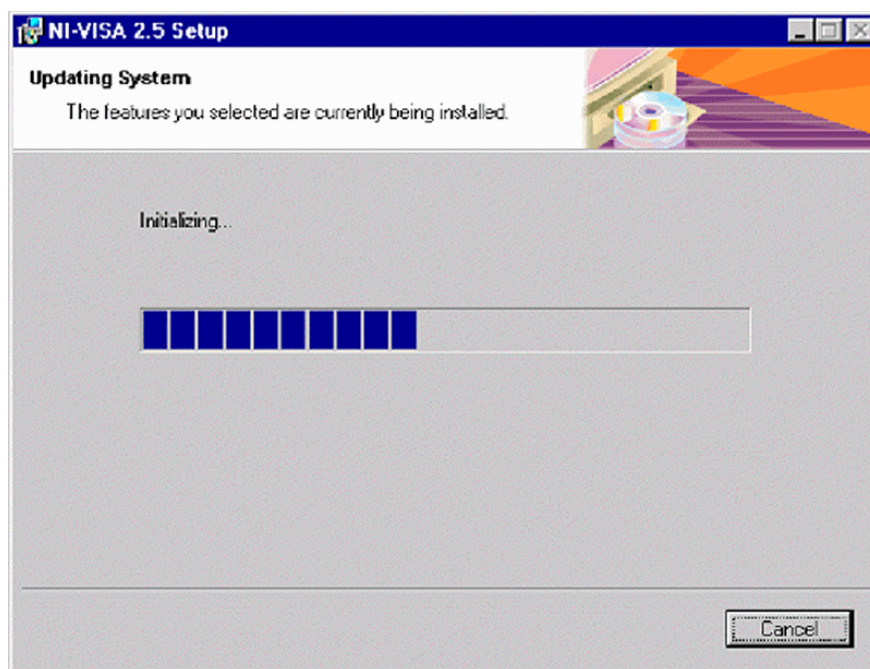
16. Wait, while the files are being copied.
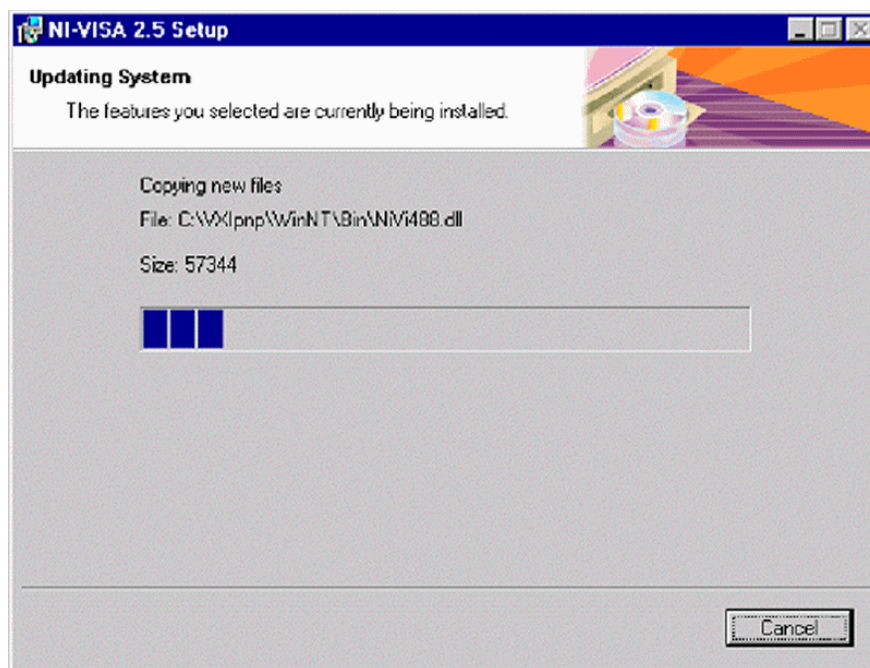
17. After NI-Visa has been successfully installed, press **Finish**.

Thermo
ELECTRON CORPORATION
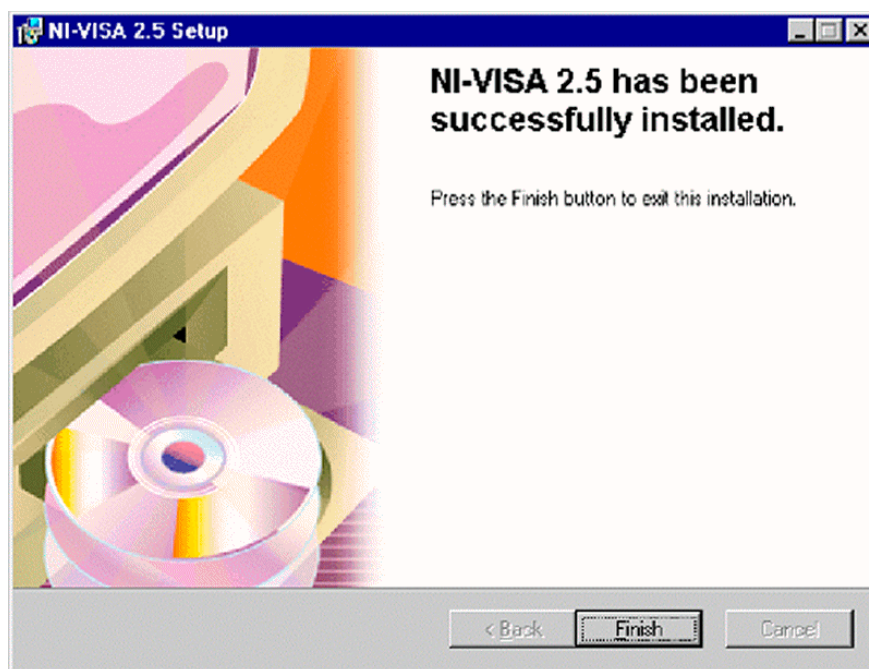
18. Click **Yes** to reboot your system now.
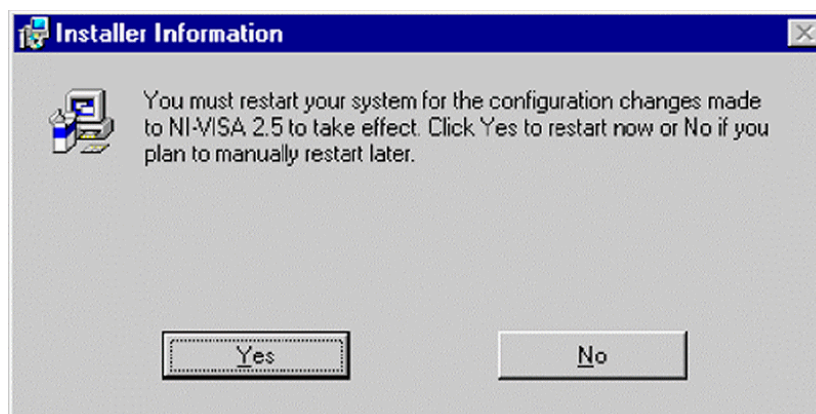
Installation of the Sound Card Driver

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.

1. Insert the sound card into slot #5.

2. For the installation of the respective sound card driver, follow the instructions in the manual of the sound card manufacturer.

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:

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

2. Close the appearing box via the button ([×]) at top right corner.

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

Thermo
ELECTRON CORPORATION
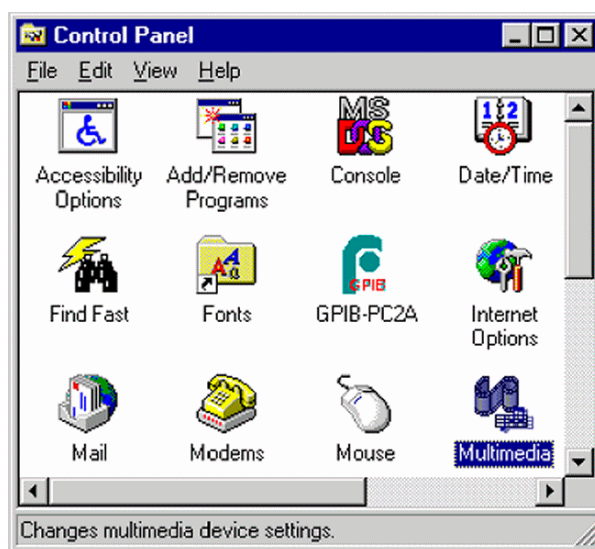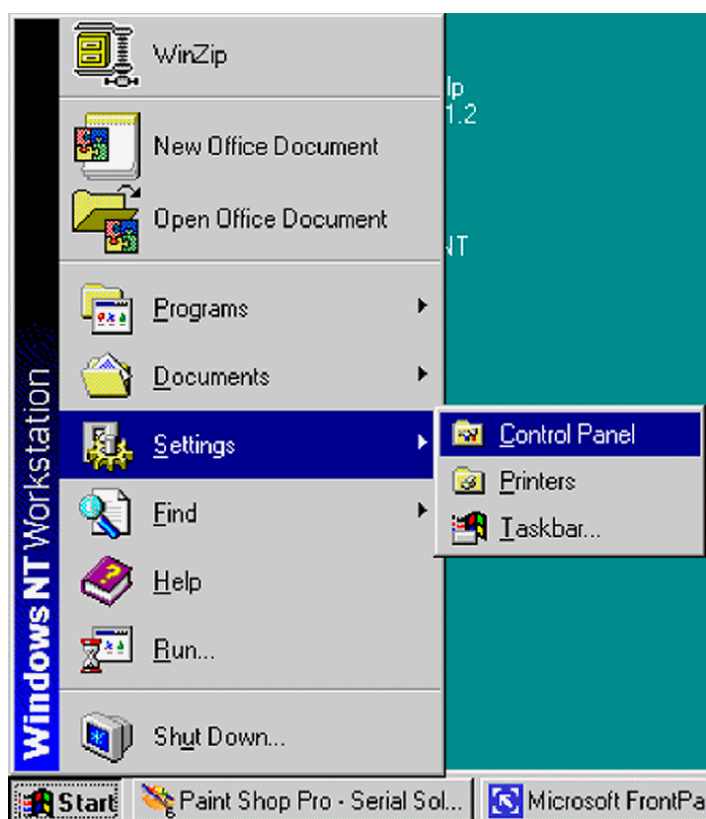
4. Double-click the
   Multimedia Icon.
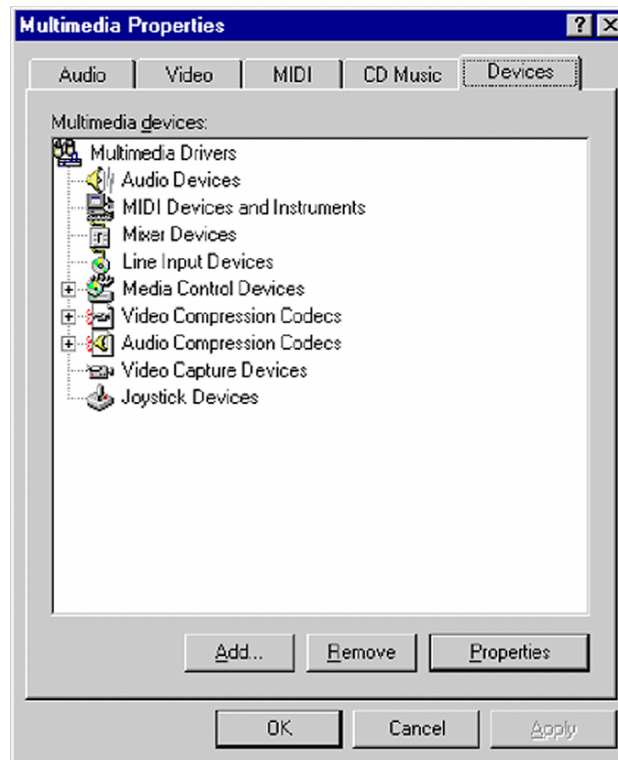
5. Click the **Devices** tab.

6. Press the **Add** button.
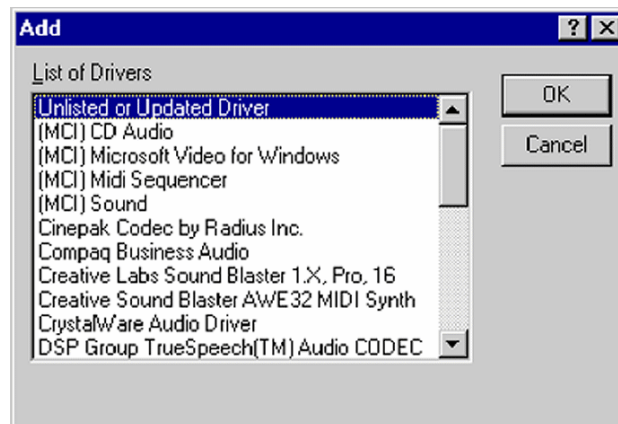
7. Select *Unlisted or updated driver* and click **OK**.

8. 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**.
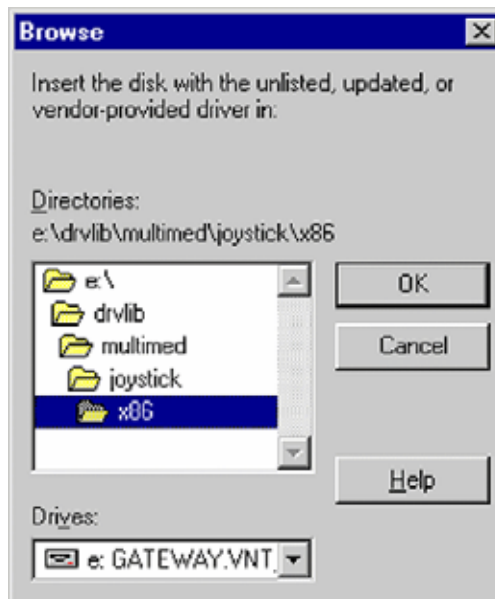
a. From the Drives pulldown list, select your CD drive (e.g. e: NTWKS 40A).

b. In the Directories pulldown list double-click the folders, which successively lead to the Joystick drivers (e.g. *e:\drvlib\multimed\joystick\x86*).

c. Click **OK**.

9. Click **OK**.
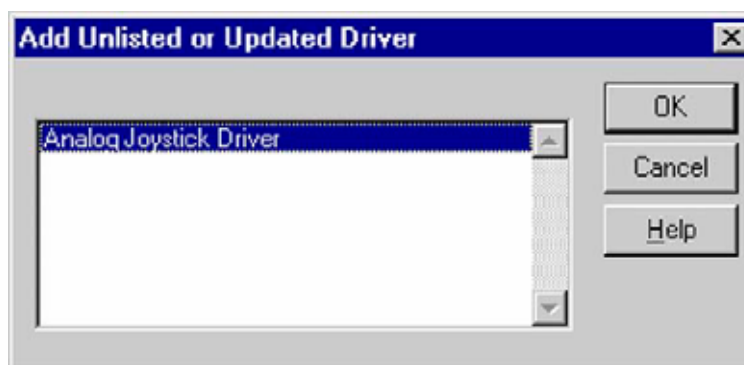
10. Select **Analog Joystick Driver** and click **OK**.

11. If the required joystick.dll driver is already installed:

Press **New** to install a new driver, or press **Current** to use the current driver.
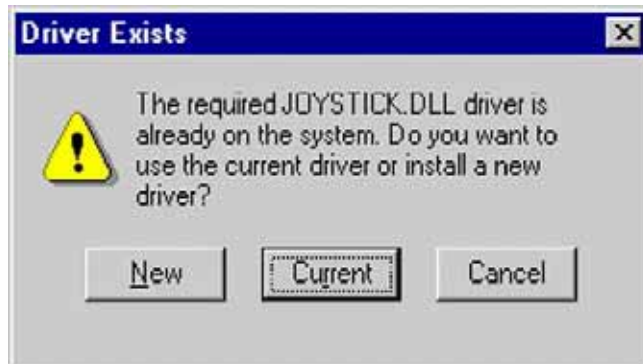
12. In both cases, the box below appears. The different settings determine the communication of Windows with the joystick(s). If you are unsure of which settings to use, keep the default settings. Then click **OK**.
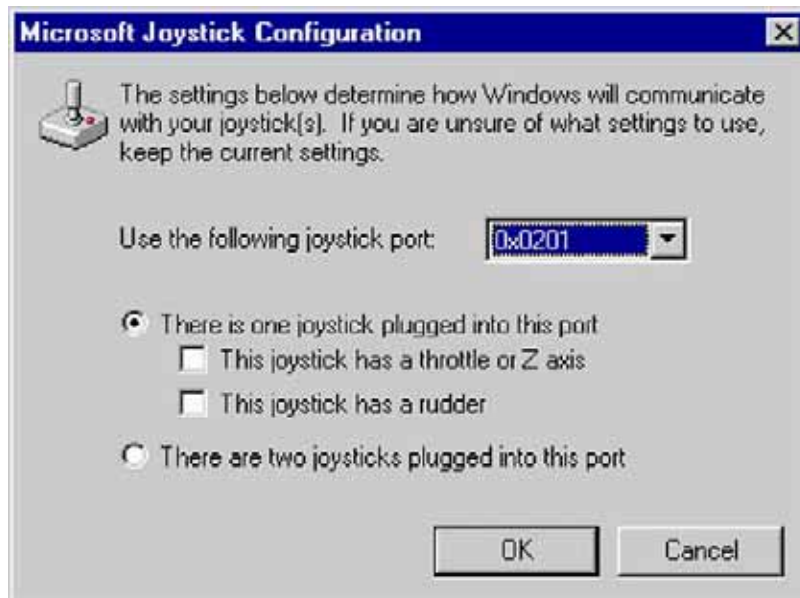
13. Click **Restart Now**.
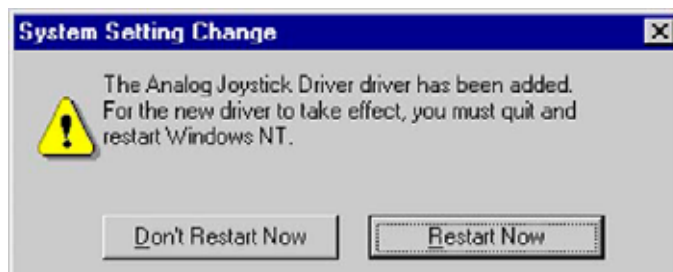
Calibrating the Joystick Driver

After the installation of the Joystick driver, it must now be calibrated and finally tested. To calibrate it, follow the steps below:

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

2. Double-click on the **Joystick** icon.

3. At **Joystick Configuration**, select the **Calibrate** button. An error message occurs, if the joystick is not correctly connected.
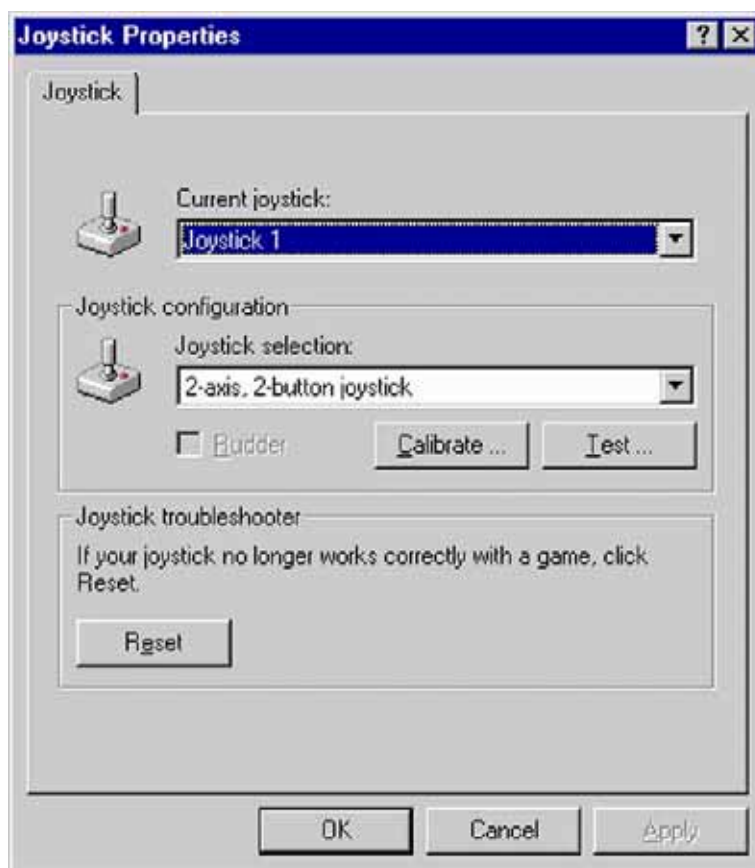
4. To set the joystick's position, leave its handle centered.

   Then press one of the joystick's buttons. Click **Next**.

Thermo
ELECTRON CORPORATION
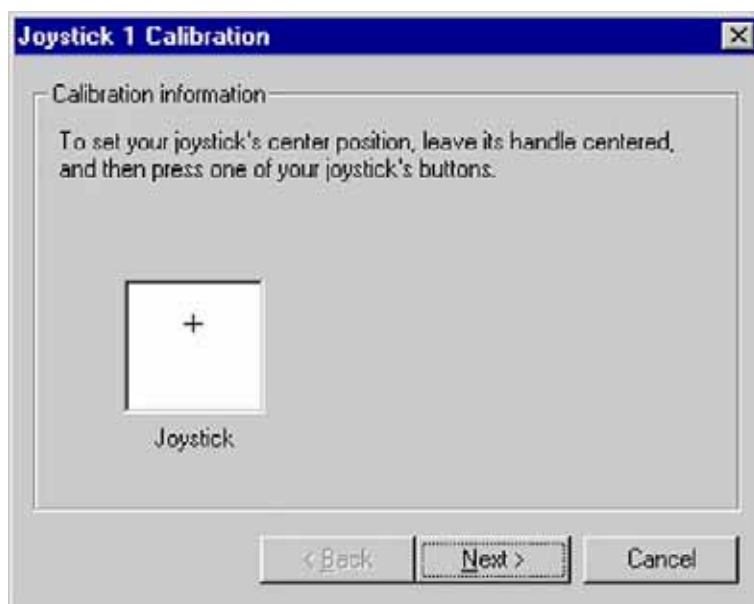
5. To set the joystick's motion range, move the handle in complete circles several times. Then press one of the joystick's buttons.
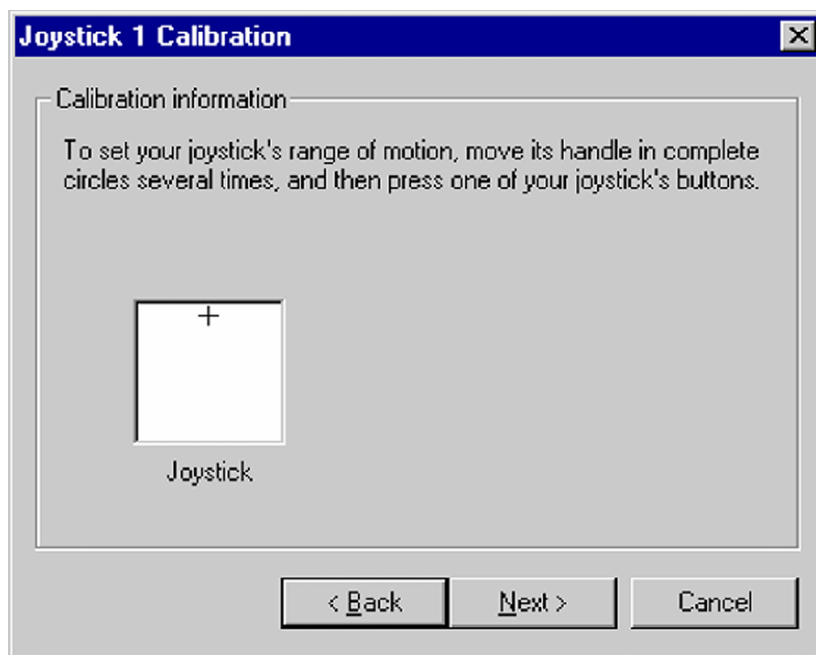
6. The joystick has been calibrated successfully.

   Click **Finish** to save your calibration settings.
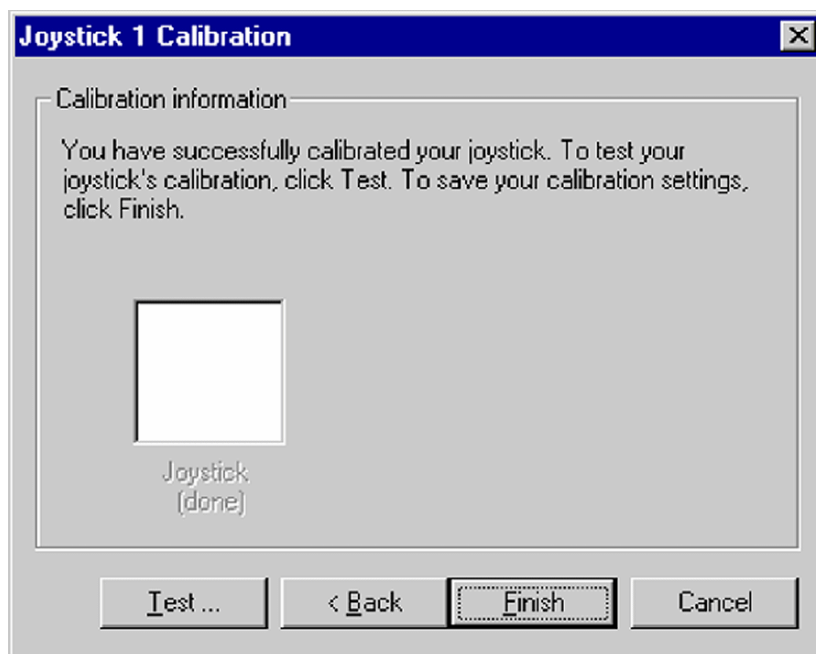
7. Test the calibration. See **Testing the Joystick's Calibration** below.

Testing the Joystick's Calibration

1. Press **Start > Settings > Control Panel.**
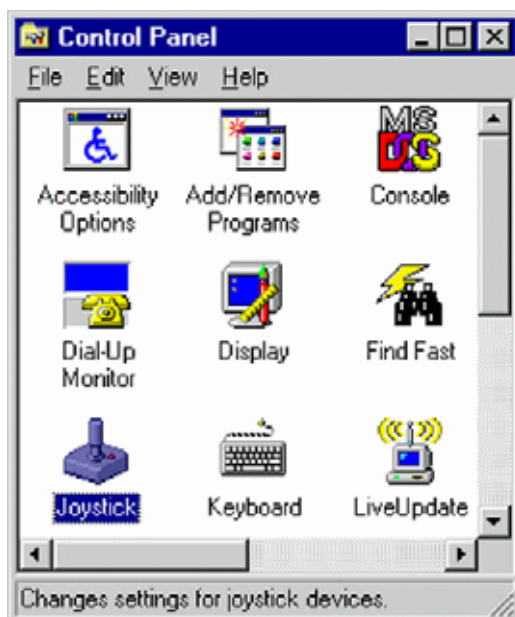
2. Double-click the **Joystick** icon.

3. At **Joystick Configuration**, select the **Test** button. An error message occurs, if the joystick is not correctly connected.
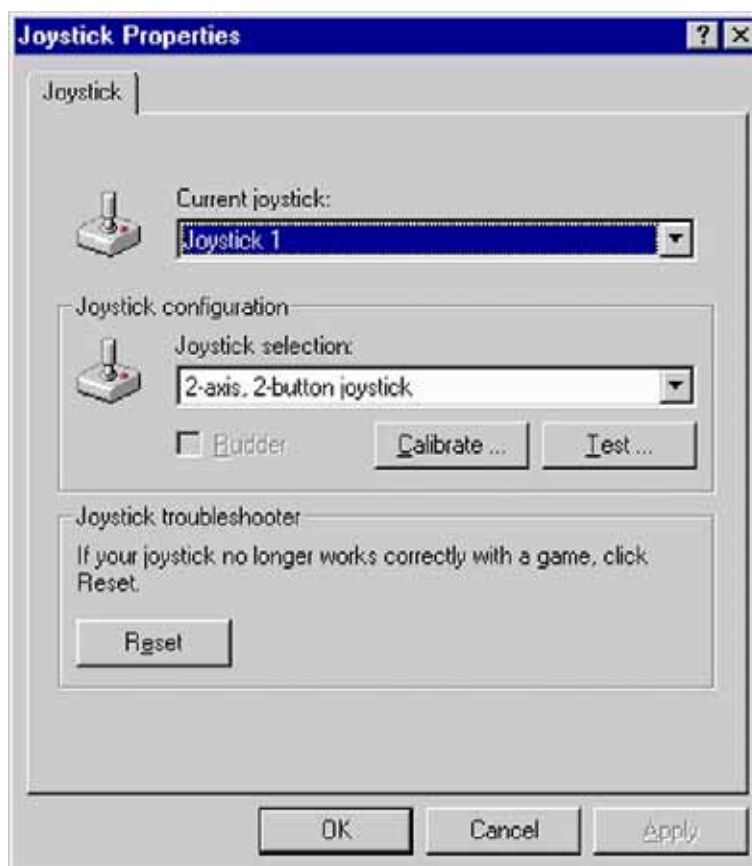
4. To test the Joystick's calibration:

   a. Move the Joystick.

   b. Click **OK**.

   c. Press one of the Joystick's buttons.
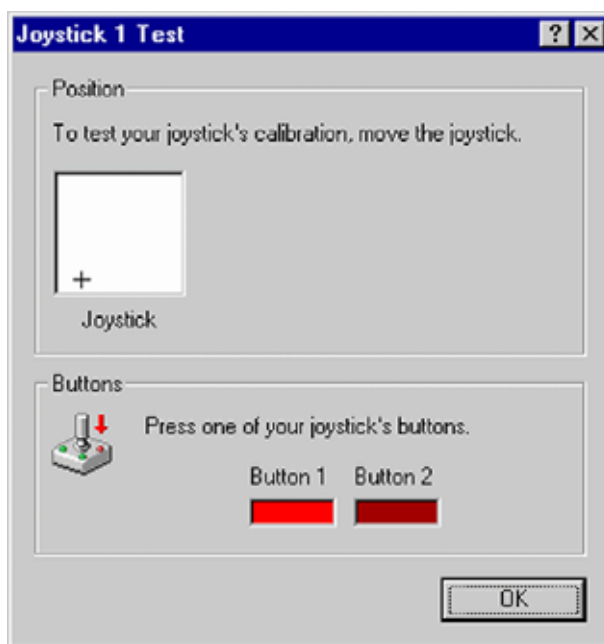
d. One of the fields is lighted red (e.g. "Button 1").

    Press the other button.
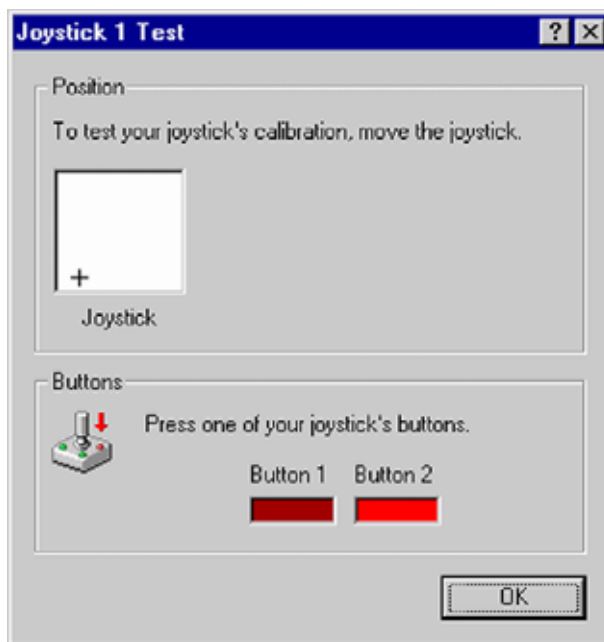
e. The other field is lighted red

    (e.g. "Button 2").

    Press **OK**.

GC and Autosampler Requirements

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

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

Any other "Generic" GC

- Gameport Interface (in the PC)

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

A200S or PAL Autosampler

- Serial cable from PC to A200S Autosampler - Default: COM 1

- Cable for "GC ready" status from Autosampler to GC

8.3   Maintenance

Regular maintenance including functional checks and service routines is required to maintain trouble free and continuous operation. These maintenance operations are listed in Table 8-1.

Table 8-1.   Maintenance Operation

Part	Maintenance Operation	Frequency
Rotary pumps	Check oil level Exchange oil Refer to manufacturer's instructions	Refer to manufacturer's instructions
Turbomolecular pumps	Exchange oil reservoir Refer to manufacturer's instructions	Refer to manufacturer's instructions
Compressed air service unit	Check oil level Drain condensate and clean filter	Monthly If required
Safety circuit	Check function	Every 6 months

Basic Rules for Cleaning

> **Note.** Do not economize with the cleaning agent! Renew the cleaning bath frequently.

Method of checking highly volatile cleaning agents (cyclohexane, acetone): Dip a polished metal plate into the bath and take out again so that as much liquid as possible remains behind on the plate (preferable bend the plate to a concave shape). Allow the bath liquid to evaporate. No residue should be left on the plate. Otherwise, change the bath liquid.

> **Note.** Use slightly contaminated bath liquids for preliminarily washing severely contaminated parts!
>
> Do not rinse with water if using volatile cleaning agents!
>
> Rinse with a lot of water if using cleaning agents in aqueous solution! Use warm water, finally rinse with distilled (or de-ionized) water!

Dry the parts, which have been rinsed in water, well in a dust free area, preferably in a vacuum drying oven! In the case of drying ovens with forced air circulation, the air must be free of dust. If necessary, wrap the parts loosely in tissue paper and place them in the oven.

Because contaminations on ceramic parts consist of evaporated-on metal layers, they cannot be removed with volatile cleaning agents. For this reason, use a high percentage aqueous solution of RBS 50 (Trade name of the firm Carl Roth, Karlsruhe).

Using an ultrasonic bath increases the probability of a successful removal of contaminations.

Even with RBS 50, a successful removal of contaminations from ceramic parts may not always be possible. In this case, replace these parts by new ones.

If new parts are not available, strongly adhering layers can be removed as follows:

- By filing down with a diamond file. After being filed, the parts must be washed. The diamond file can easily be cleaned again with an erasing rubber.

- By annealing at red heat in a propane-oxygen flame. Too high temperature may cause distortion of ceramic parts. Therefore, avoid the white heat range!

  Do not wash any parts after they have been annealed.

CAUTION!

Caution. Ceramic parts, which cannot be disconnected from metal parts, must not be annealed.

When re-assembling, do not touch the washed parts with naked hands! Use non-fibrous gloves and clean tools!

In the case of difficult assembly jobs, it may be necessary to work without gloves. Then thoroughly wash your hands and remove any fat or grease from the fingertips with a solvent. The parts should then be touched only with degreased fingertips.

In order to avoid any damage to the skin, rub the fingertips over with a fatty skin cream when work is completed!

8.4   Analyzer System
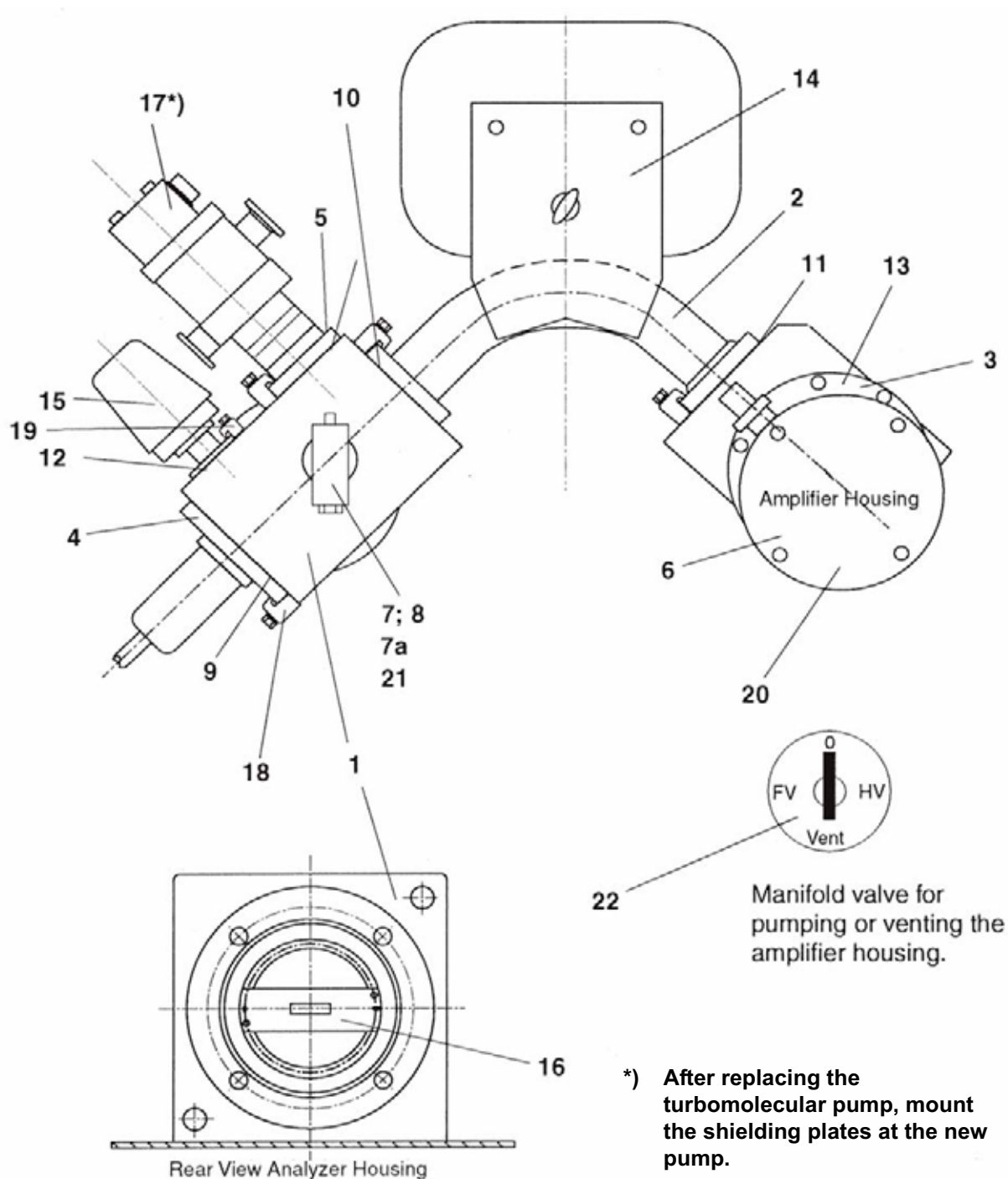
Figure 8-1.   Basic unit (Part No. 114 2900)

Amplifier Housing

Manifold valve for pumping or venting the amplifier housing.

Rear View Analyzer Housing

*)   **After replacing the turbomolecular pump, mount the shielding plates at the new pump.**

Pos. No.	Qty.	Designation	Part No.
1	1	Analyzer housing	106 9950
2	1	Flight tube	109 9100
3		Collector system	------------
4	1	Ion source with flange, see topic **Ion Source** on page 8-52	111 9460
5	1	Flange	065 2620
6	1	O ring; 185 x 2, Viton	111 6520
7	1	Gas inlet	114 7210
7a	1	Gasket; 11.5, gold	055 1240
8	1	Spacer ring; stainless steel, NW 6	095 1600
9	2	Edge sealing; DN 100, aluminum	056 9110
10	2	Edge sealing; DN 63, aluminum	069 3220
11	1	O ring; 76 x 2.5, Viton	109 0140
12	2	Sealing washer	055 2930
13	1	O ring; 160 x 2, Viton	111 6510
14	1	Magnet, compl.	109 9430
15	1	Penning gauge	111 8320
16		Aperture plate	112 2460
17	1	Turbomolecular pump TMH 071 P See topic **Vacuum System** on page 8-48	114 1500
18	28	Clamping screw	102 8380
19	3	Clamping screw	1027301
20	1	Amplifier housing	114 7060
21	1	Heater for gas inlet	114 7220
22	1	Valve, 3 port	112 2300

8.5   Vacuum System

The main reasons of troubles with the vacuum system are leaks and contaminations. **Leaks** may be caused:

- if flange connections and/or sealings are improperly treated during service operations.

- accidentally during cooling down periods after baking.

- if heavily treated sealing components are worn out.

As leak detection is rather time-consuming, make sure that flange connections are carefully and properly assembled during service. Additionally, watch the pressure during cooling down after baking. If necessary, retighten the flanges.

Contaminations may be caused:

- By water vapor stemming from the sample preparation devices.

- By using improper elastomer gaskets, especially at elevated temperatures. We recommend ordering spare gaskets from Thermo Electron only.

- By introducing solvents, which weren't removed carefully after cleaning.

The final pressure should be checked daily. About 24 h after start, without sample inflow, the pressure should be in the 10^{-8} mbar range.

CAUTION!

> **Caution.** Never use silicon greases or silicon oils! Silicon layers lead to surface charges and are difficult to eliminate!

Flanges with Metal Gaskets

Before mounting the flange connection, make sure that the sealing surfaces of the flanges are clean and that the gaskets are clean and intact. Afterwards put the spacer ring onto the protruding edge of the flange, place the metal gasket inside the spacer ring and insert the counter flange into the spacer ring. Make sure that the flanges are not canted, that flanges and spacer ring are centered in the right way and have equal distances. Then insert and manually tighten the screws. Afterwards use a wrench and tighten the screws crosswise at least two procedures. Check that flanges and spacer ring have equal distances. Stop tightening, when the flanges touch the spacer ring.

If using the metal gaskets for more than one time, do not take off the gasket from the spacer ring. Make sure that the gaskets are not bent or damaged. Note that the probability for leakage increases with increasing use.

> **Note.** Do not re-use a baked metal gasket.

Flanges with Viton Gaskets

When changing the Viton gasket, do not use other materials. The Viton used must be of good and preheated quality. For this reason, we recommend to order spare gaskets from Thermo Electron only. Before using the gasket, ensure that sealing surfaces and gasket are clean and that the gasket surfaces are not injured. For cleaning, wipe the gasket with clean paper.

> **Note.** Do not use vacuum grease, and do not clean Viton gaskets with solvents. Viton must not be operated at temperatures above 150 °C.

PVC Tubings of Fore Vacuum Lines

If the tubings are contaminated by forevacuum pump oil, replace them with new ones. Never clean PVC tubings with solvents. PVC tubings are sensitive to elevated temperatures. Thus, make sure that they do not contact hot parts.

Cleaning the Vacuum Components

Cleaning the parts of the high vacuum regions is described in **Ion Source** on page 8-52. For cleaning parts of the fore vacuum region, many different methods are known. We recommend using cyclohexane as solvent. The best cleaning is obtained by using a hot ultrasonic bath. Observe the following recommendations:

- Use pure solvents only.

- When cleaning with solvents, use a hood.

- Pay attention to your country's safety regulations.

- After cleaning, dry the parts carefully.

- Do not touch the cleaned parts with your fingers (use lint-free gloves).

Leak Detection

Two different methods are recommended:

- Mass spectrometric leak detection can be applied if the leak permits MS operation (p ~ $6*10^{-4}$ mbar). Use a container with a suitable test gas (e.g. argon). Connect the reducing valve to a PVC tube and the open end of the tube to a fine capillary (e.g. glass or metal tubing). Using a sensitive cup, set the IRMS to the Ar peak (m/z 40) and blow a fine argon beam onto those parts, where leaks are suspected.

• For leak detection in the forevacuum section, or if the pressure is too high for MS operation: use a small washing bottle with ethanol and spray a fine ethanol beam onto those connections where a leak is suspected. When hitting the leak, the pressure, after a short time of delay, will first decrease and afterwards increase rapidly. Use the ion gauge for pressure reading.

> **Warning.** Ethanol vapors are explosive. Do not smoke or handle with open fire. During leak detection ventilate the laboratory carefully.

Ion Gauge

When replacing the ion gauge tube, take care that the filament is placed in the downward direction. Otherwise, a short circuit may occur. It is recommended to use aluminum gaskets for sealing the ion gauge tube. When using a Viton gasket, make sure that the Viton ring is of good and preheated quality. The gasket is warmed up during operation.
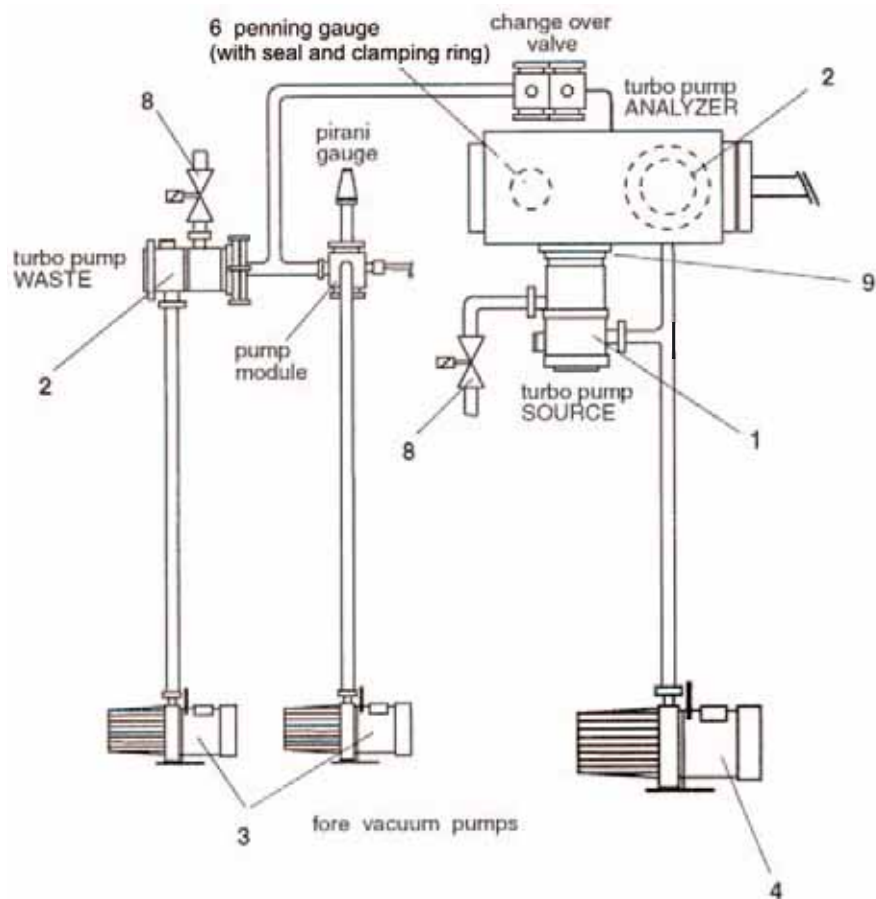
Figure 8-2.   Schematic View of the Vacuum System

Thermo
ELECTRON CORPORATION

Pos. No.	Qty.	Designation	Part No.
1	1	Turbomolecular pump TMH 262	114 1600
	1	Resource storage for TMH 262	105 0160
2	2	Turbomolecular pump TMH 071 P	114 1500
	1	Resource storage for TMH 071 P	017 2350
3	2	Rotary pump; DUO 2.5	109 5950
4	1	Rotary pump; DUO 005	109 7510
6	1	Penning gauge, for high vacuum	111 8320
8	1	Vent valve	108 4890
9	1	Edge sealing; NW 100	056 9110
		Accessories	
	1	Cross piece; DN 25/16	108 2410
	1	Blind flange; aluminum NW 16	052 2090
	2	Centering ring; NW 16, Viton	052 2140
	3	Centering ring; NW 25, Viton	052 2150
	1	Metal tube; NW 25 x 250	052 4270
	1	Metal tube; NW 16 x 500	053 4500
	3	Clamping ring; DN 10-16 KF	079 4480
	3	Clamping ring; DN 20-25 KF	052 1560
	20	Screw; stainless steel, M 8 x 35	045 4400
	20	Washer; stainless steel, I.D. 8.4	047 0070
	5 m	Exhaust tube; 9 x 3; PVC	104 9540
	1	Edge sealing; aluminum, DN 63	069 3220

8.6   Ion Source

The following section describes the ion source connections, the removal and the insertion of the ion source, the replacement of the filament, the disassembling of the ion source, the cleaning procedure and the reassembly of the ion source.

Removal of the Ion Source

1. Switch off ion source.

2. Close inlet system (close changeover valve)

3. Switch off the pump system

4. Vent the analyzer system with dry gas (Ar).

5. Unscrew the eight bolts of the ion source flange, and remove the ion source carefully

6. In case the ion source shall be removed for a longer period of time, e.g. for cleaning, close the opening of the analyzer head by suitable means in order to prevent foreign particles from entering the ion source chamber.

Reinstallation of the Ion Source

1. Inspect the edge sealing carefully for absence of scratches and lint.

2. Place the edge sealing on the flange of the analyzer head.

3. Insert the ion source into the analyzer head. When inserted, ion source flange and making flange should be aligned in parallel.

4. Press the ion source in the direction of the analyzer and tighten the screw hand tight. Check for short circuits. Afterwards use a wrench and tighten the screws crosswise within at last two procedures.

5. After exchanging the cathode unit and/or disassembly of the ion source, it is necessary to readjust the ion source potentials.

Exchange of the Cathode Unit

1. Remove the ion source as described in topic **Removal of the Ion Source** above and put the source onto a table with the flange downward.

2. Loosen the clamp of the source magnet at the cathode unit side, turn aside and remove the magnet.

3. Unscrew the cathode unit from the ionization housing and remove the cathode unit.

4. The new unit must fit to the edge of the housing. Afterwards tighten the screws.

5. Hold the feedthroughs with small pliers and screw on the connection wires.

6. Replace the source magnet and fasten with the clamp.

7. Perform the source check.

8. Reinstall the ion source.

> **Note.** Use lint-free gloves and clean tools for the exchange.

Cleaning the Ion Source Parts

This section contains the following topics:

- Cleaning the Metallic Parts

- Cleaning the Ceramic Insulators

Cleaning the Metallic Parts

1. Grind all contaminated and discolored metal parts with aluminum oxide powder (600 grain). The mechanical cleaning described above must be followed by a chemical cleaning.

2. All parts must be cleaned within a grease dissolving cyclohexane bath. An ultra sonic bath will improve and accelerate the cleaning procedure.

> **Note.** Do not use ultrasonic when cleaning the ion source magnets.

3. Afterwards all parts made of stainless steel should be cleaned in a 3% RBS 50 solution at 60 °C for about 1 or 2 h.

4. After cleaning in RBS 50 and in distilled water, the parts should be dried chemically within a Freon bath or a pure alcohol.

5. After chemical drying, the parts should be additionally dried by a fan or a drying oven to eliminate solvent residuals.

Cleaning the Ceramic Insulators

1. Use a clean diamond file for eliminating metal abrasion and evaporated films. Use this method only for cleaning the ceramic insulator of the cathode and the electron collector.

2. Anneal the ceramic spacer rings to red heat. Use a propane-oxygen flame or a muffle furnace for annealing (maximum temperature: 800–1000 °C).

Source Feedthroughs, see Figure 8-4

Source Wiring, see Figure 8-6

Pos. 26/26a are not shown
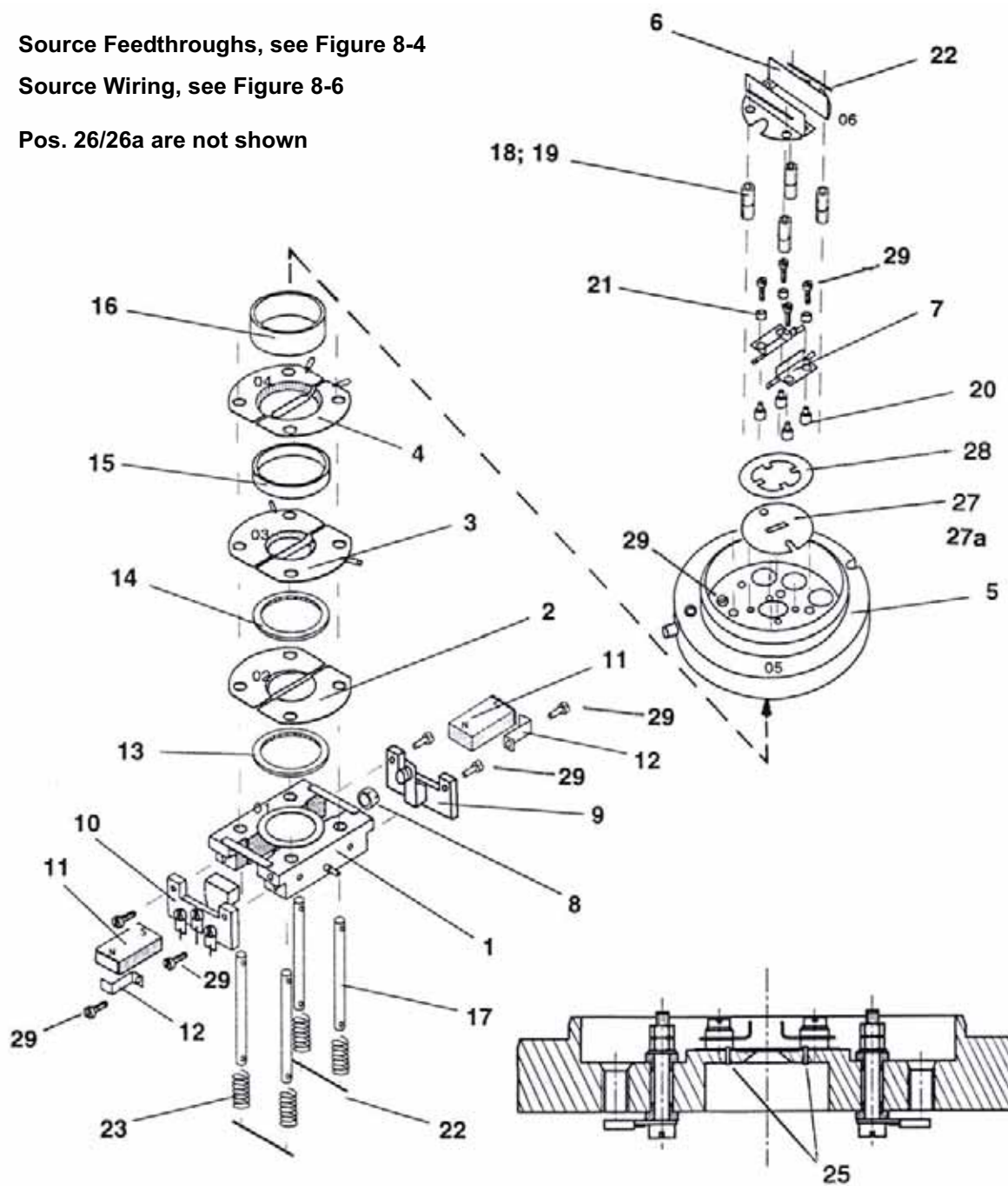

Figure 8-3.   Ion Source (Part No.: 111 9440)

Pos. No.	Qty.	Designation	Part No.
1	1	Ionization housing	111 9480
2	1	Extraction plate	102 8040
3	1	Extraction plate	058 2460

4	1	Lens	058 2440
5	1	Ion source base plate	108 2350
6	2	Z deflection	050 2040
7	2	R deflection	050 2060
8	1	Spacer ring; quartz glass, 6 x 4.1 x 4	106 7930
9	1	Collector (Trap)	074 2600
10*	1	Cathode (Filament)	102 7920
11	1	Magnet	057 9300
12	2	Spring	074 2380
13	1	Spacer ring; metal	102 8160
14*	1	Spacer ring; ceramic, 24.8 x 21 x 1	056 0010
15	1	Spacer ring; quartz glass, 24.8 x 21 x 2	106 7910
16	1	Spacer ring; quartz glass, 24.8 x 21 x 4	106 7920
17*	4	Rod; ceramic	102 7480
18*	4	Spacer tube; ceramic, 6 x 4.1 x 4	056 0710
19*	8	Spacer tube; ceramic, 6 x 4.2 x 3	056 0150
20*	10	Bush, ceramic	048 5280
21*	4	Spacer tube; ceramic, 5 x 3.2 x 1.5	056 1650
22	4	Pin, long	102 6730
23	4	Pressure spring; I.D. 4.2, LD 1	102 8920
24	3	Connection pin	081 0170
25	2	Pin; 1 x 3	111 9430
26	2	Halogen lamp; 12 V/10 W, see Figure 8-5	108 5040
26 a	1	Set heater connection, see Figure 8-5 (ground line, lines 12/13 and 2 halogen lamps)	900 0374
27	1	Source slit, tantalum	112 0940
27a	1	Source slit, gold	111 8050
28	1	Spring	111 8040
29	11	Screw; DIN 84, M 2 x 6	045 3650

> **Note.** The parts marked with an asterisk (*) frequently break. It is recommended to provide for replacements before the source is opened.
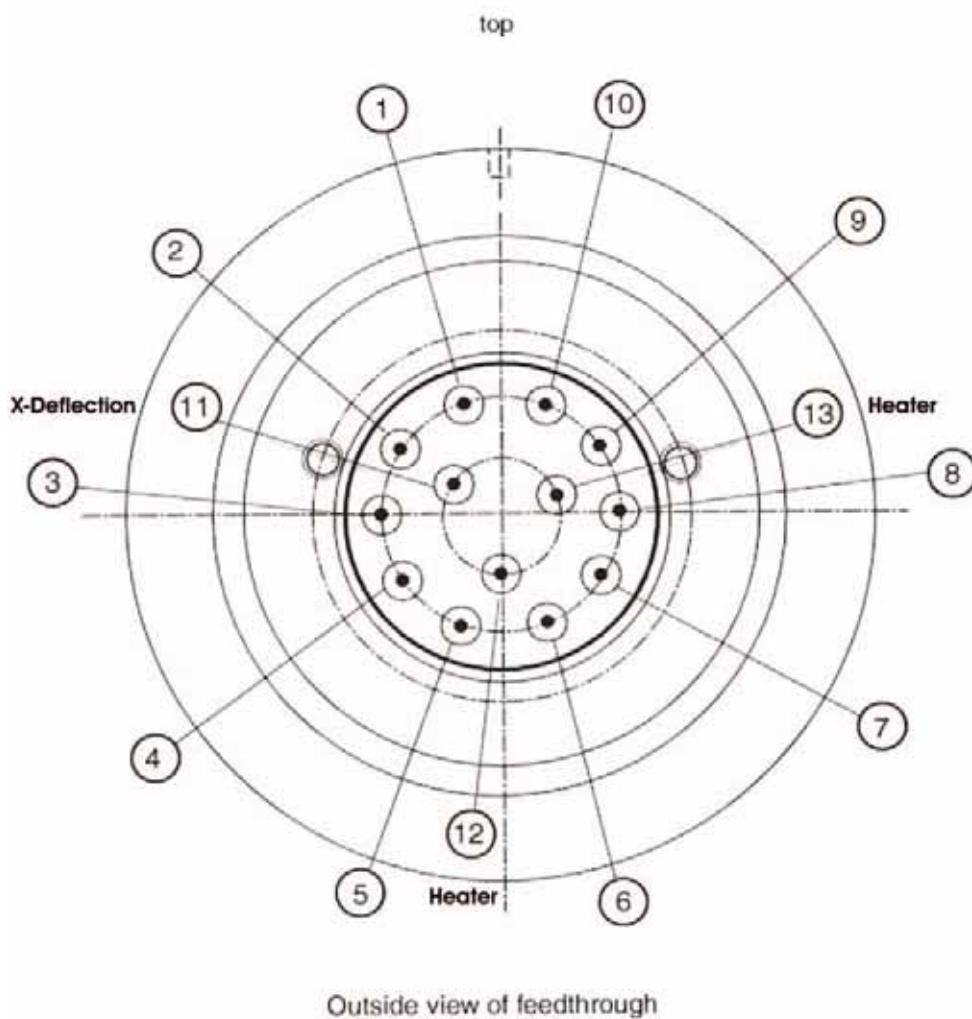
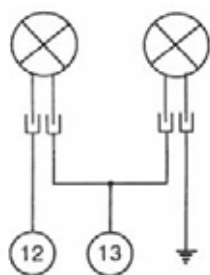**Figure 8-4.   Source Feedthroughs**

Figure 8-5.   Connection Scheme for Lamps, for part numbers, see page 8-55.
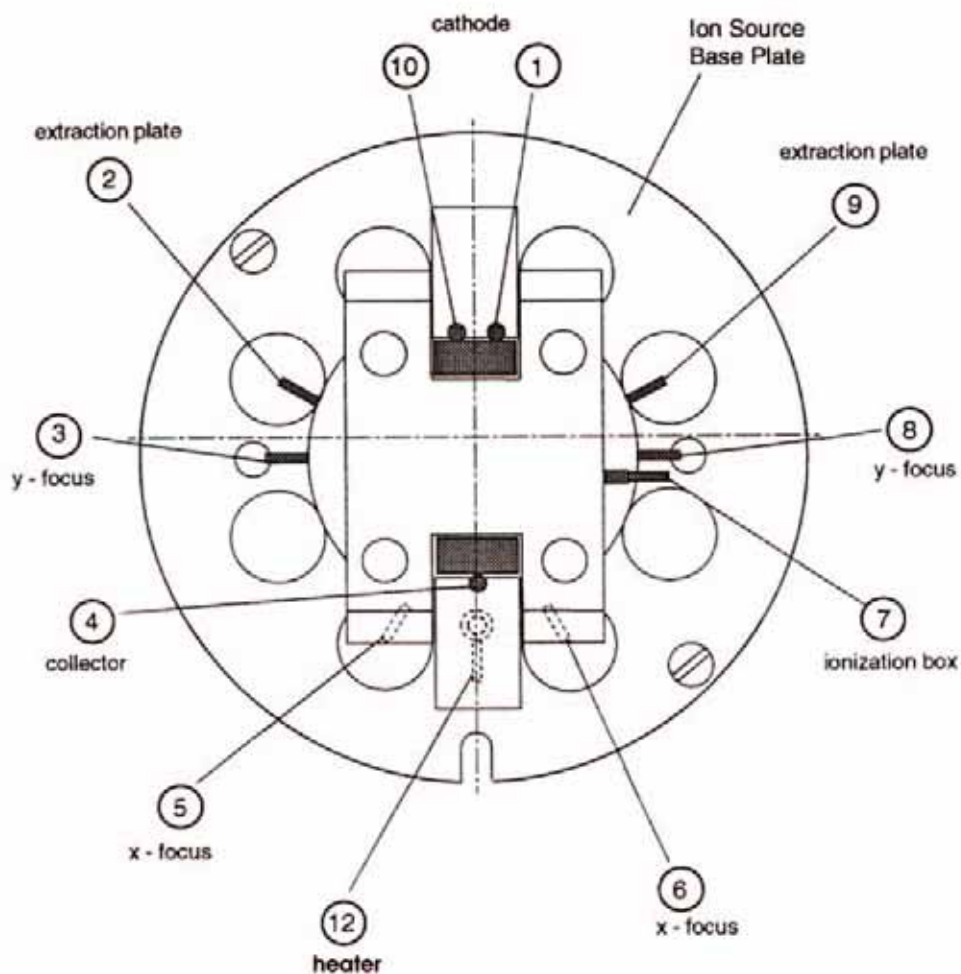
Figure 8-6.   Source Wiring

8.7   Inlet Systems

compressed air generation
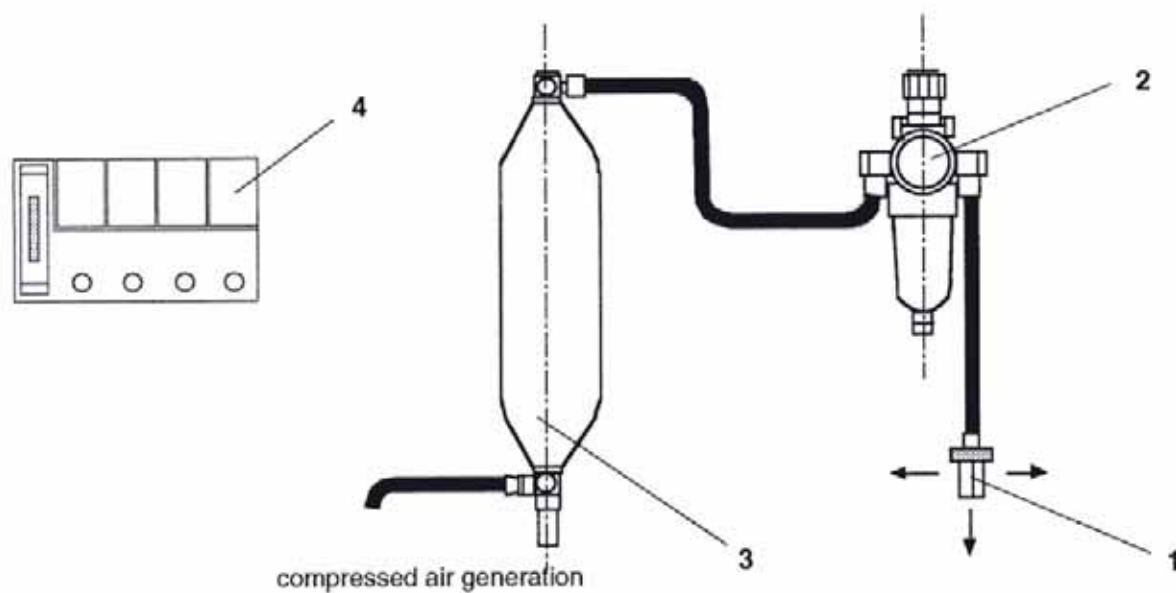
Figure 8-7.   Compressed Air Supply (Part No. 108 2520)

Pos. No.	Qty.	Designation	Part No.
1	1	Compressed air distributor	106 8410
2	1	Compressed air service unit	052 1630
3	1	Compressed air store	049 3830
4	5	Valve control unit manifold card	108 3241
		Accessories	
	2	Swivel joint	052 1730
	1	Quick connection	052 1620
	6 m	Tube	069 0740
	4	Screw	045 2130
	4	Washer	047 0050
	11	Sealing ring	050 5260
	5	Blind stopper	052 1950
	4	Coupling compl.	067 4652
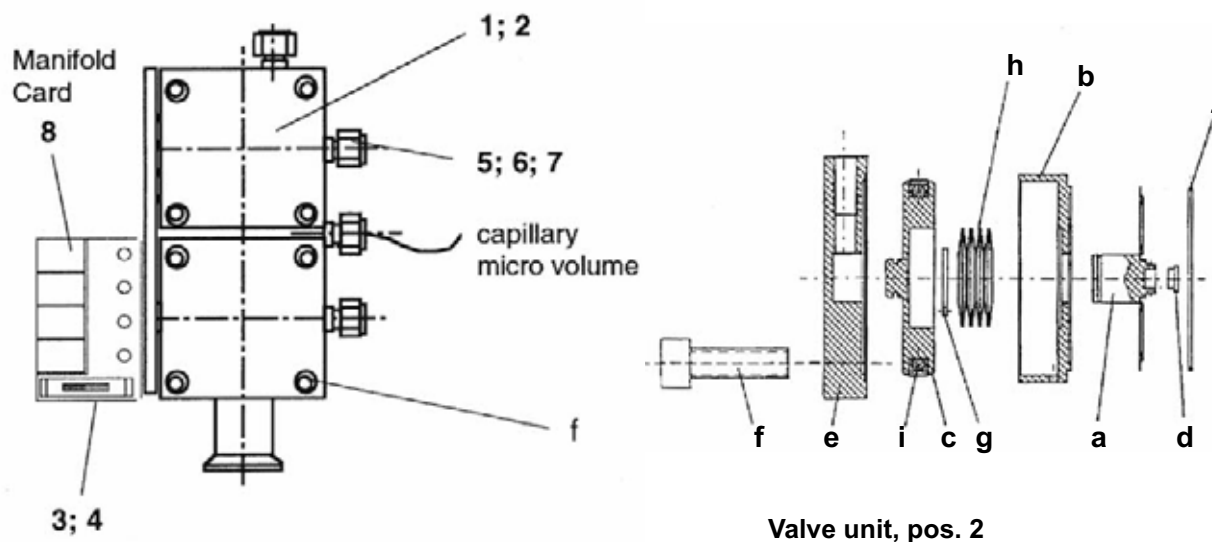
Valve unit, pos. 2

Figure 8-8.   Basic Module Changeover (Part No. 108 2630)

Pos. No.	Qty.	Designation	Part No.
1	1	Valve body	108 2620
2	4	Valve unit	065 3001
a	1	Membrane	065 3010
b	1	Pressure unit	065 3050
c	1	Piston	065 3030
d	1	Stamp, gold	065 3041
e	1	Cover	065 3060
f	4	Screw; M 6 x 20	045 3420
g	1	Guard ring; 10 x 1	047 3430
h	8	Plate spring; 20 x 10.2 x 0.4	043 1570
i	1	Jacket ring	055 3140
j	1	Gasket; gold, 38	054 5270
k	1	Lithium fat	079 1140
3	4	Connecting fitting	070 3780
4	4	Gasket	050 5260
5	8	Cap nut	052 1160
6	8	Front ferrule	079 2800
7	8	Back ferrule	079 2810
8	1	Manifold Card	108 3241
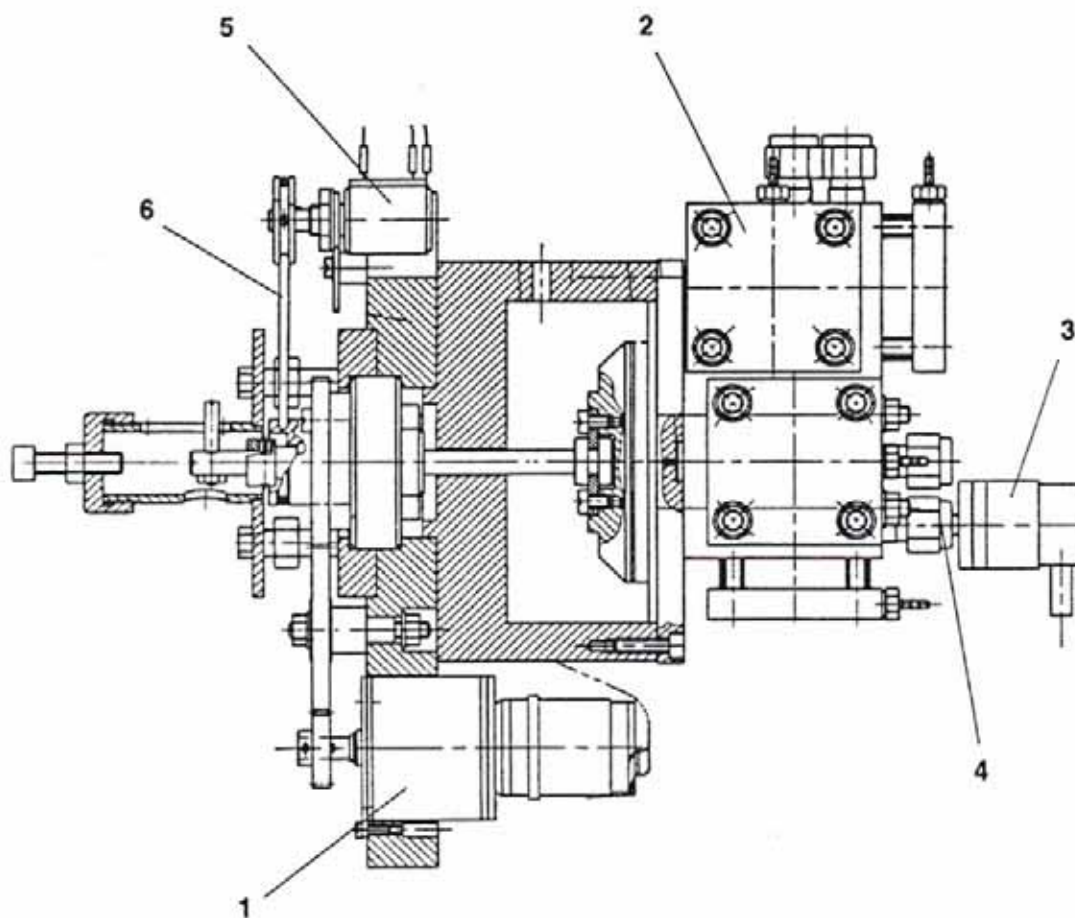
Figure 8-9.   Variable Volume (Part No. 108 2681)

Pos. No.	Qty.	Designation	Part No.
1	1	Motor, compl.	070 3330
2	1	Valve unit	108 2670
3	1	Manometer	101 6802
4	1	Capillary	067 1182
5	1	Potentiometer	070 3540
6	1	O ring	055 3180
7	1	Limit stop	070 3610
8	1	Bellows unit, small	070 3310

Technical Information
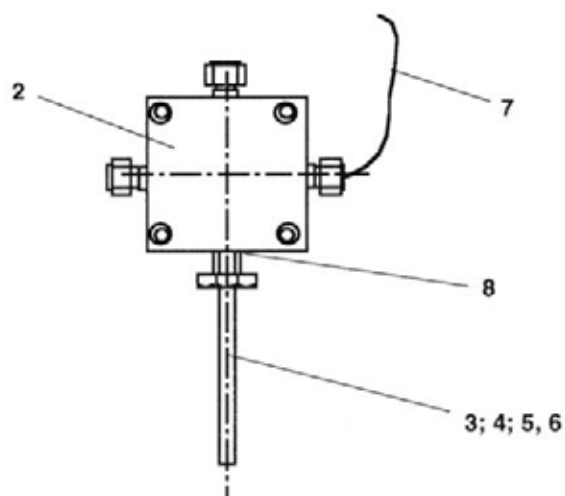
Figure 8-10.   Microvolume (Part No. 108 2900)

Pos. No.	Qty.	Designation	Part No.
2	1	Valve	106 8970
3	1	Cold finger; CO_2	078 3330
4	1	Cold finger; N_2	078 3340
5	1	Cooling plate; CO_2	041 2300
6	1	Cooling plate; N_2	058 3290
7	1	Capillary tube, heatable	067 1182
8	1	Gasket; gold, 6.3	055 1010
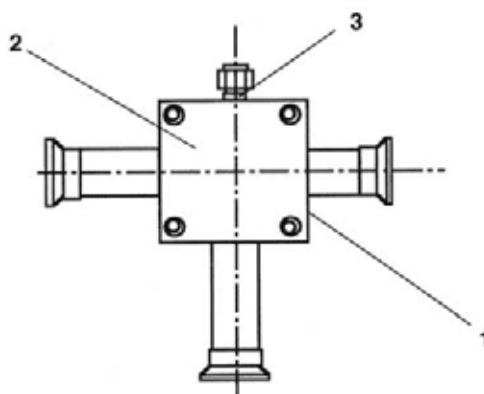
Figure 8-11.   Pumping Unit (Part No. 108 3300)

Pos. No.	Qty.	Designation	Part No.
1	1	Valve body	059 3410
2	2	Valve unit	065 3001
3	2	Gasket	050 5260

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Sample   **Pos. 2 and 7 are not shown**   Pump
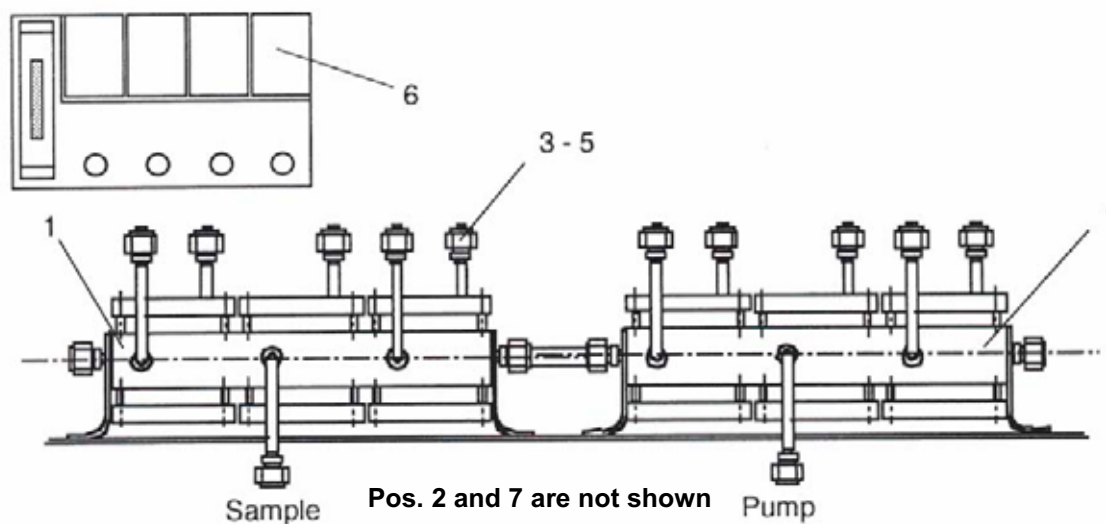
Figure 8-12.   Multiport Module (Part No. 108 3200)

Pos. No.	Qty.	Designation	Part No.
1	2	Valve bloc	106 8770
2	1	Manometer	101 6801
3	5	Nut	052 1160
4	5	Front ferrule	079 2800
5	5	Back ferrule	0792810
6	3	Manifold card	108 3241
7	5 m	Silicon hose	101 5830

Pos. 2 is not shown
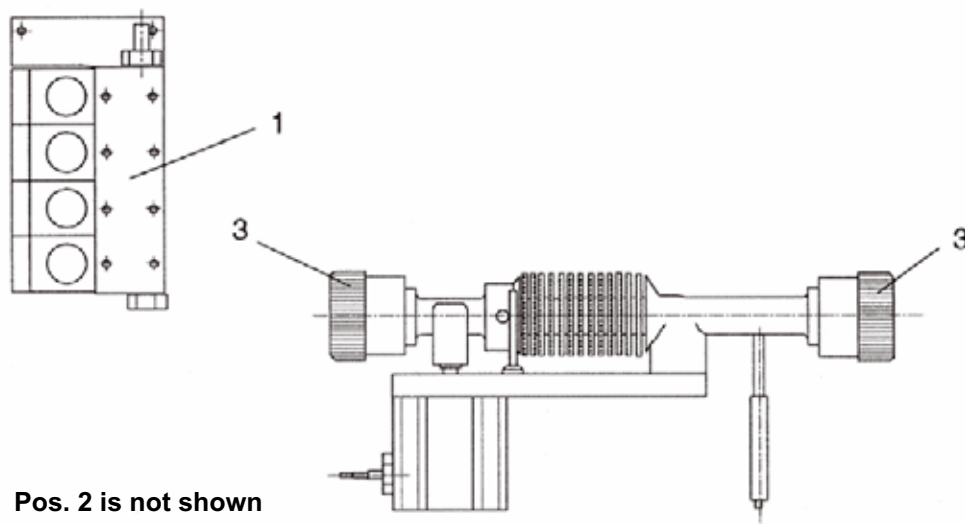
Figure 8-13.   Tube Cracker (Part No. 108 28 40)

Pos. No.	Qty.	Designation	Part No.
1	3	Manifold Card	109 7750
2	5 m	Silicon hose	101 5830
3	2	O ring	055 2180

Autocool

Pos. 16 is not shown

Figure 8-14. Autocool (Part No. 049 3661)

Pos. No.	Qty.	Designation	Part No.
1	1	Cooling unit	079 2400
2	1	Blank flange	059 7380
3	1	Cover	059 6530
4	1	Insulating case	054 2830

5	1	Lifting device	060 9260
6	1	Filler cap	112 8230
7	1	Sleeve	075 4850
8	1	Insert	075 4710
9	1	Refill device	039 5441
10	1	Magnet valve, for LN2	041 4130
11	1	Manifold, for LN2	048 2610
12	1	Hose for refill box	059 9610
13	1	Screw; M 4 x 10	045 088
14	4	Nut; M 4	046 0220
15	2	Screw; M 4 x 25	045 0500
16	1	Connection cable for trap	025 3791
17	1	Container 75 l, f. liquid N_2	079 4700

Accessories
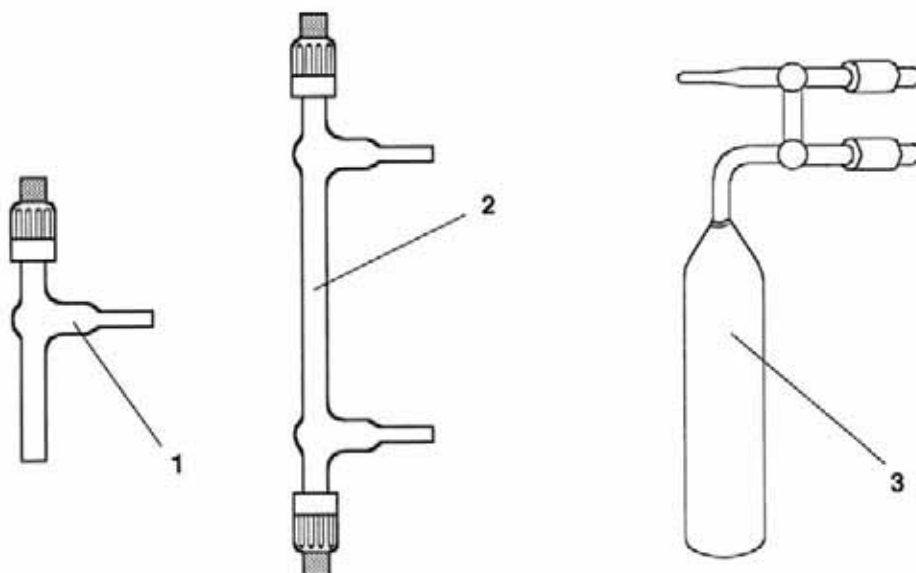
Figure 8-15.   Accessories

Pos. No.	Qty.	Designation	Part No.
1	1	Sample vial (5 ml)	100 3560
2	1	Sample vial (10 ml)	100 3840
3	1	Sample vial (200 ml)	025 4650

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