Finnigan™ ConFlo III

Universal Interface

Operating Manual

Revision B
111 0042

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ConFlo III Universal Interface Operating Manual — *Revision B 111 0042*

	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.)

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

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Please fill in completely

Absender:
Dispatcher:

Geräte-Type:
Instrument Type:

Service-Nr.:
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**

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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. The superconducting magnet is still charged even if the instrument is turned off.
11. 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.

Contents

Read This First

Welcome to the Thermo Electron, Finnigan ConFlo III Universal Interface!

This **Finnigan ConFlo III Operating Manual** describes how to setup and use the Finnigan ConFlo III Universal Interface.

This manual includes the following chapters:

Chapter 1: Prior to Measurementdescribes precaution measures and important steps to be performed in the beginning before a measurement is started.

Chapter 2: The ConFlo III Interface deals with the open split principle and treats the front and rear panel of ConFlo III respectively.

Chapter 3: ConFlo III Interface and Elemental Analyzer describes how to install an autosampler, how to operate ConFlo III manually and how to get started with ConFlo III plus Elemental Analyzer.

Chapter 4: Nitrogen Measurement describes zero enrichment (standard on/off test) and linearity test of nitrogen.

> **Note.** This chapter treats the principles of blank measurement, reference measurement and amount percent determination. As they can be generalized for nitrogen, carbon and sulfur, the chapter already contains the remarks valid for any of the three elements.

Chapter 5: Carbon Measurement describes zero enrichment (standard on/off test) and linearity test of carbon.

Chapter 6: Dual Measurementdescribes the procedure and how to perform a jump calibration.

Chapter 7: Sulfur Measurement describes the sulfur measurement kit and how to prepare the system for a sulfur measurement. Besides, this chapter treats zero enrichment (standard on/off test) and linearity test of sulfur.

Chapter 8: Technical Information describes mechanics, capillaries, ferrules, the function schematic and connections for compressed air.

Changes to the Manual and Online Help

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

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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
ASCII	American Standard Code for Information Interchange
b	bit
B	byte (8 b)
baud rate	data transmission speed in events per second
°C	degrees Celsius
CD	compact disc
CD-ROM	compact disc read-only memory
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)
CRC	cyclic redundancy check
CRM	consecutive reaction monitoring
<Ctrl>	control key on the terminal keyboard
d	depth
Da	dalton
DAC	digital-to-analog converter
dc	direct current
DDS	direct digital synthesizer
DEP™	direct exposure probe
DS	data system
DSP	digital signal processor

EI	electron ionization
EMBL	European Molecular Biology Laboratory
<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
FTP	file transfer protocol
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™
ID	inside diameter
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)
KEGG	Kyoto Encyclopedia of Genes and Genomes
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
µ	micro (10^{-6})
m	meter
m	milli (10^{-3})
M	mega (10^{6})
M+	molecular ion
MB	Megabyte (1048576 bytes)
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)
OD	outside diameter
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
SSQ®	single stage quadrupole
TCP/IP	transmission control protocol / Internet protocol
TIC	total ion current
Torr	torr
TSQ®	triple stage quadrupole
u	atomic mass unit
URL	uniform resource locator
V	volt
V ac	volts alternating current
V dc	volts direct current
vol	volume
w	width
W	watt
WWW	World Wide Web

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 San Jose 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

Reply Cards

Thermo Electron San Jose 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 San Jose 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
Prior to Measurement

1.1 Precautions

Warning. Ensure that ConFlo III is not exposed to direct sunlight!

Warning. Keep the system away from heat, e.g. radiators, to avoid damage to the internal circuits and the external surface!

Warning. Avoid using aerosol sprays near the system as this could cause sudden ignition of the spray!

Warning. Do not clean ConFlo III using paint thinner, alcohol or other organic solvents!

Warning. Do not put any objects, especially liquids, upon ConFlo III!

Warning. Place the system on a flat and solid surface that can carry at least 100 kg!

1.2 About this Manual

This manual intends to be a user guide for working with the combined system of Elemental Analyzer (EA), ConFlo III and Isotope Ratio Mass Spectrometer (IRMS). As the system consists of three separate instruments with the Elemental Analyzer and IRMS having manuals of their own, this manual will mark on specific information about the ConFlo III system. It starts with an overview about Elemental Analyzers of different types followed by basic information about ConFlo III. Finally, isotope ratio determinations of C, N, S, H and O using ConFlo III are explained in turn. It is assumed that the user is not only familiar with clean operating procedures and sample preparation but also has already some working experience with IRMS and the software Isodat 2.0.

1.3 Introduction

The Thermo Electron Continuous Flow Interface (ConFlo III) provides an efficient and flexible way to couple an Elemental Analyzer (EA) to a stable isotope ratio mass spectrometer (IRMS) on-line. This combination has become standard for determining the isotope ratios of carbon (C), nitrogen (N), sulfur (S), hydrogen (H) and oxygen (O) of combustible organic materials in bulk samples. ConFlo III and Elemental Analyzer form an integrated and optimized sample preparation inlet system, which can be connected to any current Thermo Electron IRMS equipped for on-line isotope ratio analysis. As a particularity of gas isotope mass spectrometers, any samples must be converted into simple gases prior to introducing them into the ion source. This is achieved by the Elemental Analyzer via oxidation and reduction furnaces (Elemental Analyzer) or by High Temperature Conversion (TC/EA). Passing through a gaschromatographic column the produced gases are separated and consecutively enter ConFlo III.

ConFlo III can be connected to any current Thermo Electron Elemental Analyzer and Thermo Electron IRMS equipped for Continuous Flow application.

Chapter 2
The ConFlo III Interface

2.1 Introduction - The Open Split Principle

The ConFlo III Interface provides the means for coupling the EA to the IRMS. The connection of an Elemental Analyzer with an isotope ratio mass spectrometer via an open split arrangement has become standard for on-line isotope ratio analysis. The IRMS works with a helium flow of approximately 0.3 ml/min, whereas the EA is operated with a helium flow of 80 to 120 ml/min. Reducing this gas flow is the central idea of the open split arrangement.

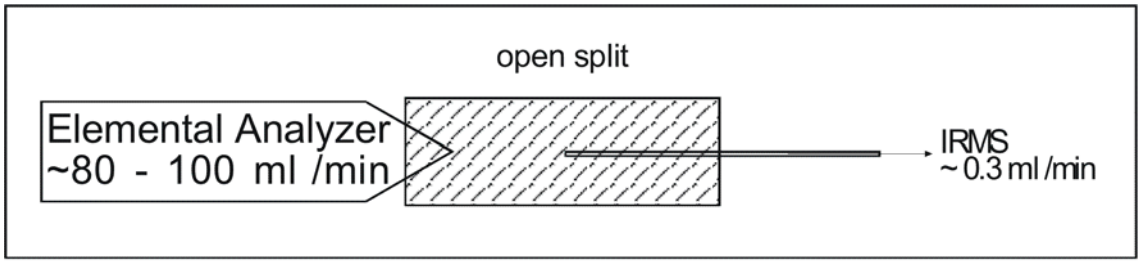


Figure 2-1. The Open Split Principle

The size of the IRMS capillary determines a gas flow of approximately 0.3 ml/min to the mass spectrometer (Figure 2-1). This IRMS capillary continuously "sniffs" the carrier gas (He) flowing through the cell. Hence, it may be referred to as the "sniffer" capillary. The carrier gas flushes the open split cell continuously with helium and passes the gaseous sample so transporting the produced compounds consecutively through the cell.

Procedure

The ConFlo III Interface forms an innovation in coupling an Elemental Analyzer to an IRMS in the way that two separate open split cells replace the "tube in tube" sample gas inlet performed in the predecessor model ConFlo II.

Through the separation of the sample stream and the reference inlet, ConFlo III forms a dynamic and flexible way of an EA/IRMS connection. These two open split cells, in the following titled as "sample section" and "reference section", allow a higher precision of on-line determination as well as a temporal independent sample/reference gas introduction to the IRMS (Figure 2-2).

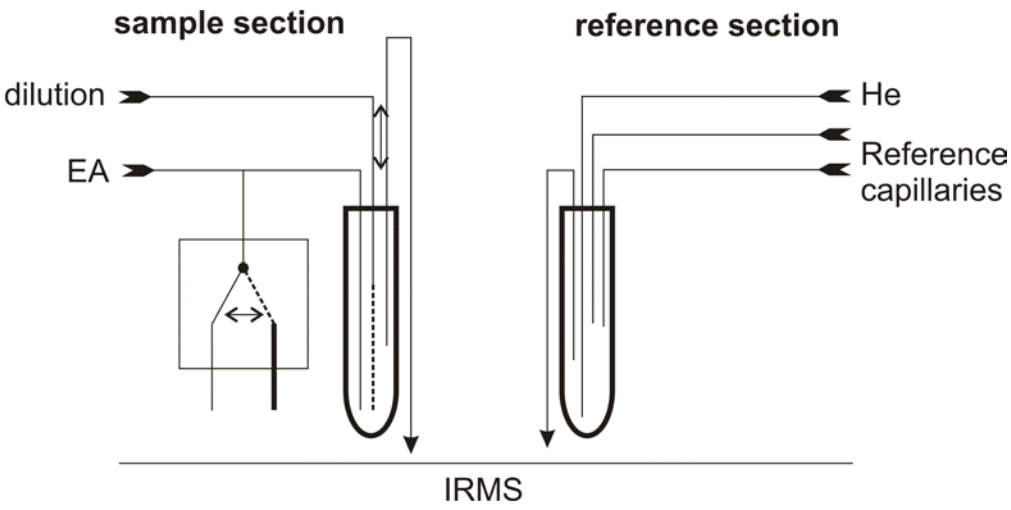


Figure 2-2. Sample Section and Reference Section

The Sample Section

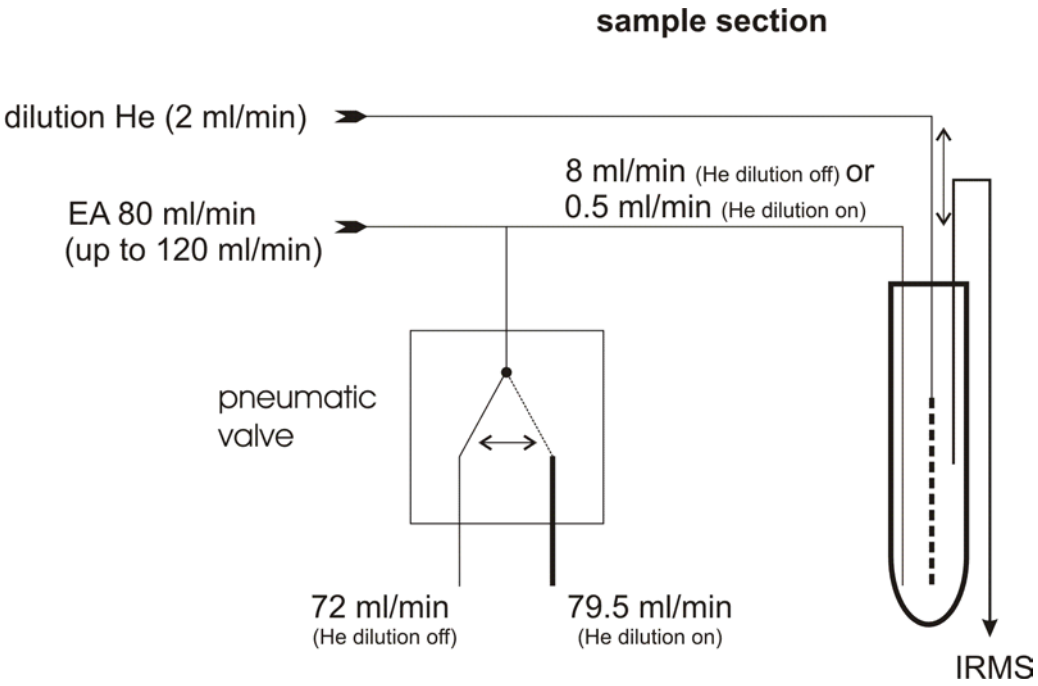


Figure 2-3. Sample Section

The open split arrangement of the sample section consists of three capillaries (Figure 2-3). The active, piston regulated dilution capillary, the fixed sample (delivering) capillary (which is located upstream the sniffer point) and the fixed "sniffer" (IRMS) capillary.

The effluent stream from the Elemental Analyzer is passed through a pneumatic valve before it enters the open split cell of the sample section. In case of dilution, the pneumatic valve is opened and the sample flow of the Elemental Analyzer is split in fixed relation (79.5 ml/min). Therefore, only 0.5 ml/min are delivered to the open split cell of the sample section. Simultaneously to the valve operation the piston with the dilution capillary moves down beyond the "sniffer" point. The He dilution flow can be varied manually between 5 and 50 ml/min resulting in an effect between 10 and 100-fold. The "sniffer" (IRMS) capillary with an inner diameter of 100 µm allows a flow of approximately 0.3 ml/min into the ion source. The dilution capillary is parked outside the “sniffed area“, if dilution of the sample is not necessary.

The Reference Section

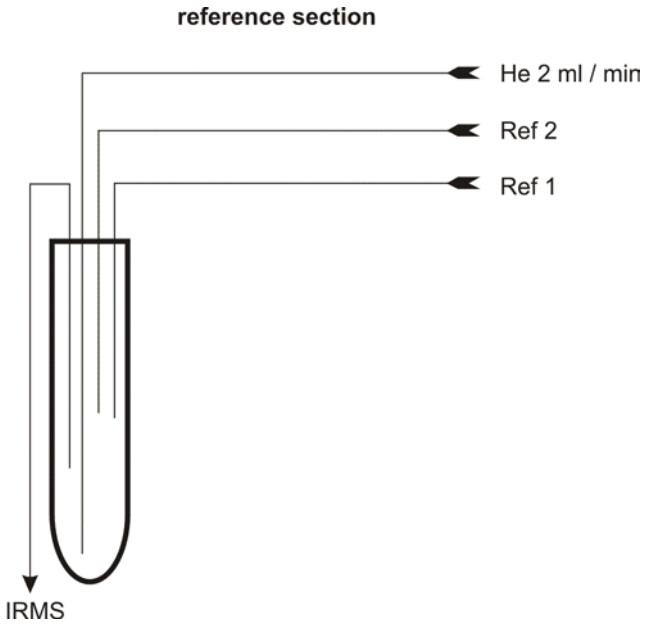


Figure 2-4. Reference Section

The open split cell of the reference section consists of four capillaries (Figure 2-4). The He carrier gas capillary as well as the IRMS capillary are fixed while a permanent stream of helium prevents a dry out of the IRMS capillary. In addition, two piston operated reference capillaries are located in the open split cell. Each reference capillary carries a continuous flow of gas

e.g. nitrogen and carbon dioxide. When a standard is required during an acquisition, the piston with the appropriate reference capillary moves down beyond the "sniffing" position and the standard is sniffed via the IRMS capillary to the mass spectrometer. Because the standard gas is injected directly into the open split cell, the mass spectrometer response is almost instantaneous, delayed only by the time the gas needs to pass through the capillary.

2.2 Front and Rear Panel of ConFlo III

Front Panel

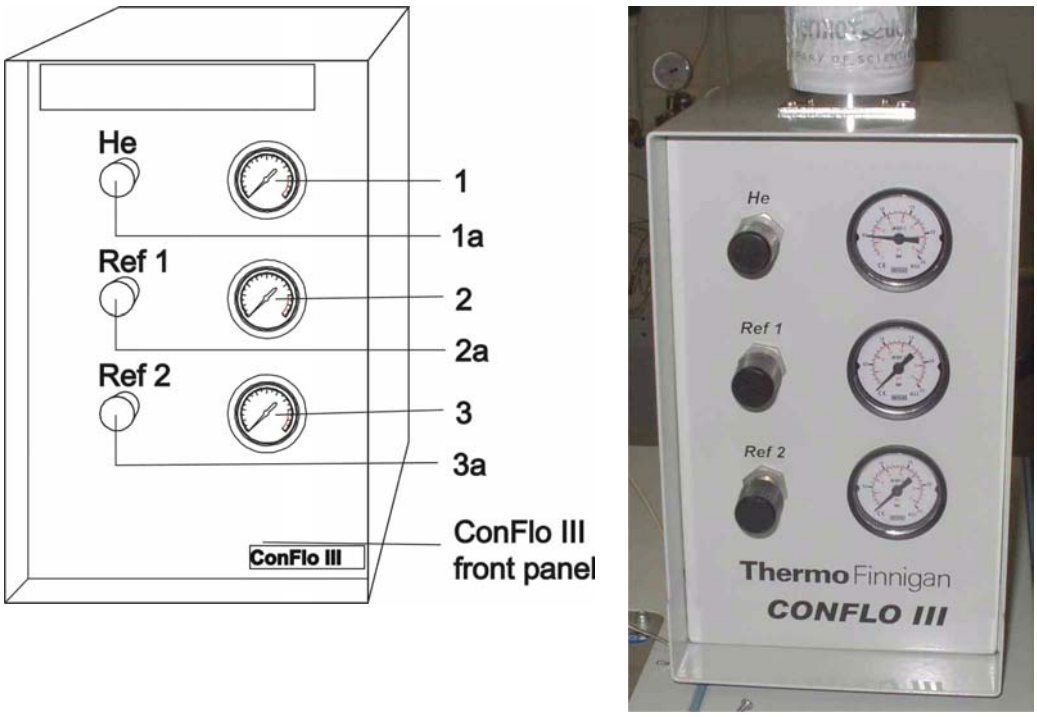


Figure 2-5. Front Panel of ConFlo III

1) Helium pressure gauge
1a) Helium pressure regulator
2) Reference gas 1 pressure gauge
2a) Reference gas 1 pressure regulator
3) Reference gas 2 pressure gauge
3a) Reference gas 2 pressure regulator

Side Views

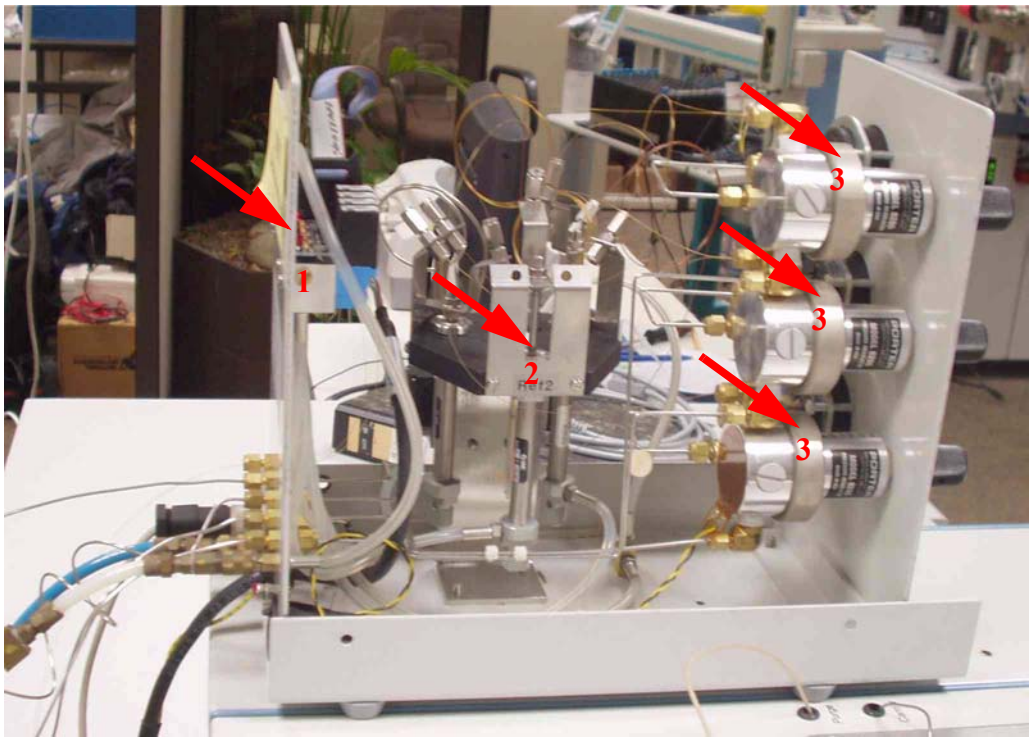


Figure 2-6. ConFlo III - Left Side View

1 manifold card

2 combi inlet with piston-operated actuators

3 pressure gauge

For technical information see also **Chapter 8: Technical Information**.

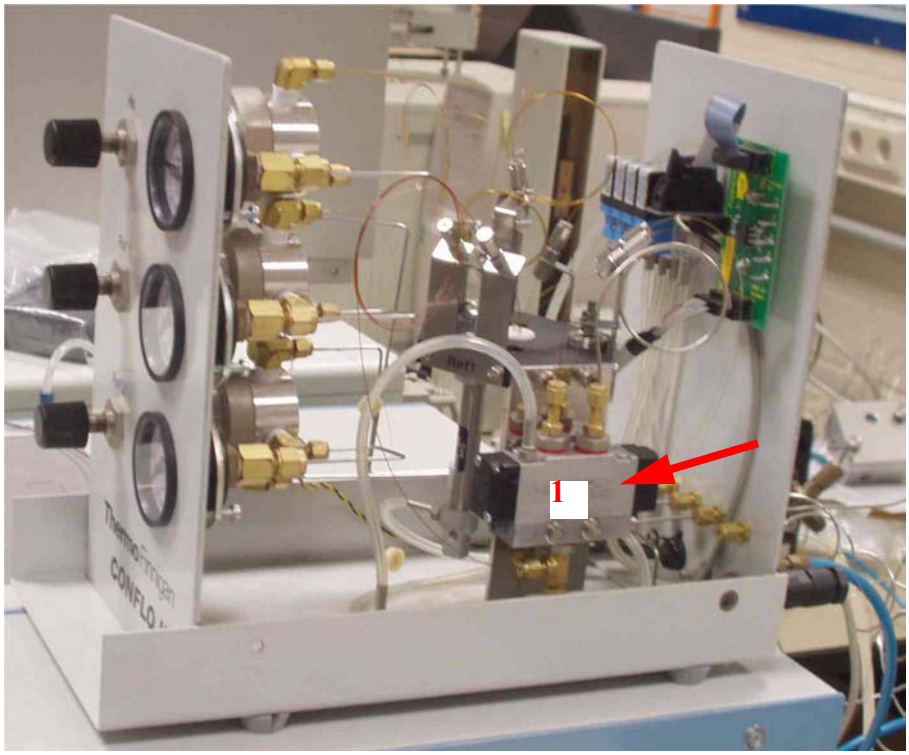


Figure 2-7. ConFlo III - Right Side View

1 two port valve (prediluter); see also Figure 8-1.

Rear Panel and Gas Connections

External gas connections are located at the rear panel of ConFlo III (Figure 2-8).

1. Fused silica port for the IRMS capillary
2. Remote Elemental Analyzer start cable
3. Compressed air outlet (time controlled)
4. Elemental Analyzer inlet
5. Reference gas 1 inlet
6. Reference gas 2 inlet
7. Helium inlet (carrier gas)
8. Compressed air inlet (permanent)
9. IRMS connection cable

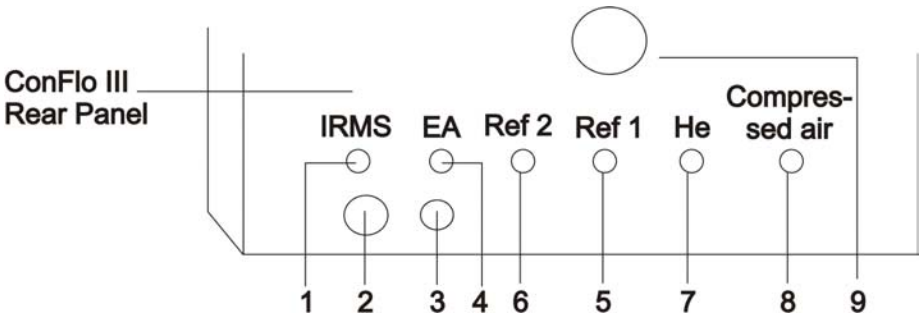


Figure 2-8. Rear Panel

Warning. For detailed information, refer to the gas flow diagram (Figure 2-9). Wrong handling of the toxic gases CO and SO_2 is perilous! Inform yourself how to handle these gases by reading the directions for use and asking your local gas tank seller. Thermo Electron does not take any responsibility if the standard gas (CO) or other gases, e.g. SO_2, are used incorrectly.

Gas Flow Diagram

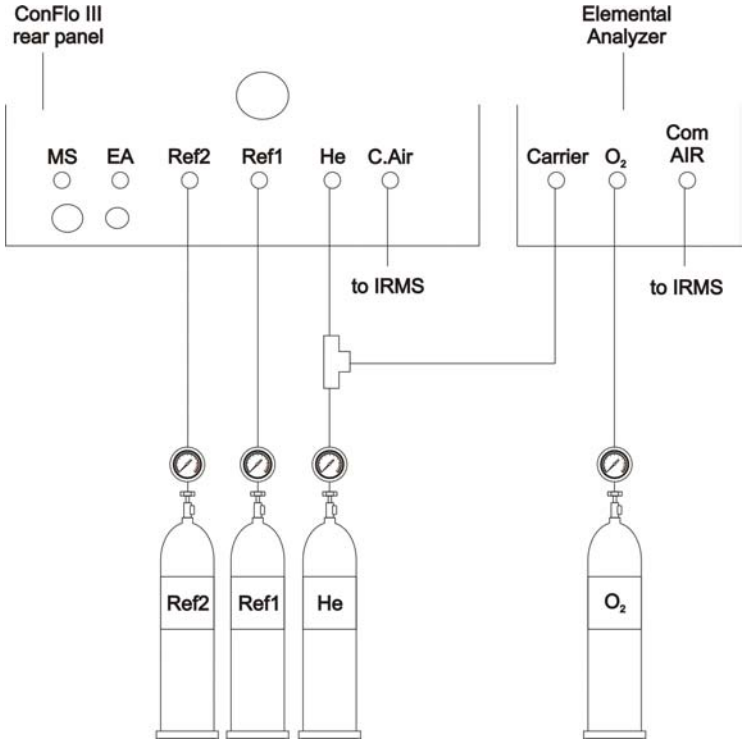


Figure 2-9. Gas Flow Diagram

Spare Parts of Reference and Sample Sections

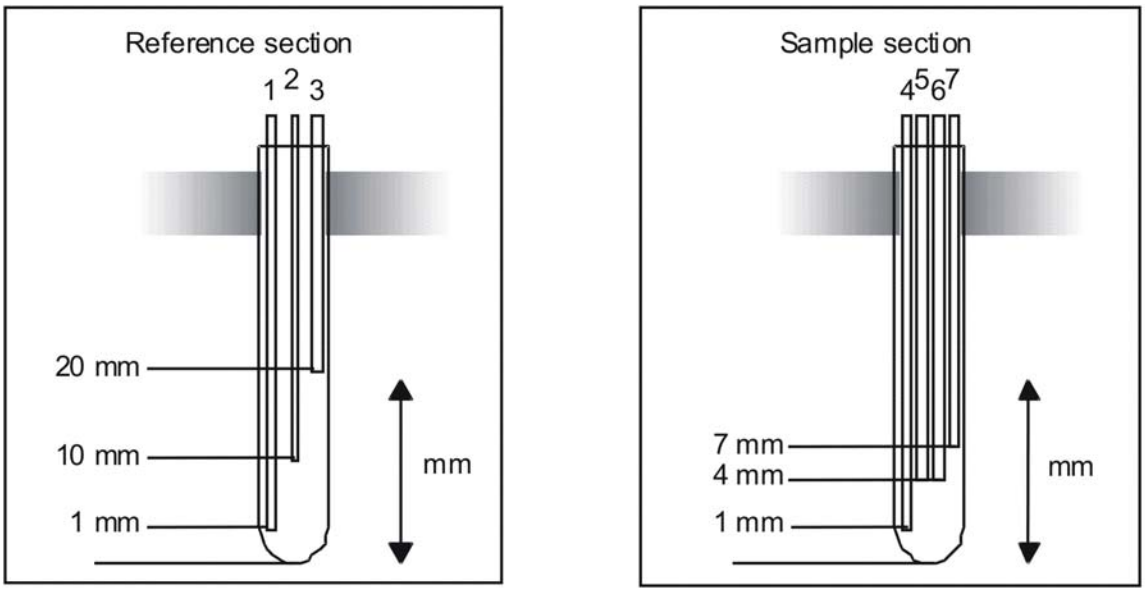


Figure 2-10. Reference Section and Sample Section

Table 2-1. Spare Parts of Reference and Sample Sections - Part 1

	Capillary	Length	Inner diameter	Part No.
1	Fused silica from EA (T-piece)	1000 mm	0.32 mm	100 4640
2	Fused silica helium dilution	2000 mm	0.18 mm	054 2910
		(or 1000 mm	0.18 mm	054 2910)
3	Fused silica to IRMS	2000 mm	0.10 mm	104 8990
4	Fused silica helium purge	250 mm	0.10 mm	104 8990
		(or 750 mm	0.18 mm	054 2910)
5	Fused silica Ref. 1	310 mm	0.05 mm	054 3380
		(or 2000 mm	0.075 mm	104 5480)
	Vent	200 mm	0.10 mm	104 8990
6	Fused silica Ref. 2	340 mm	0.05 mm	054 3380
		(or 2000 mm	0.075 mm	104 5480)
	Vent	200 mm	0.10 mm	104 8990
7	Fused silica to IRMS	1500 mm	0.05 mm	054 3380

Table 2-2. Spare Parts of Reference and Sample Sections - Part 2

	Capillary	Inner diameter	Ferrule	Part No.
1	Fused silica	0.32 mm	single hole	056 6390
2	Fused silica	0.18 mm	single hole	100 4850
3	Fused silica	0.10 mm	single hole	100 4850
4	Fused silica	0.05 mm	single hole	100 4850

Manometer

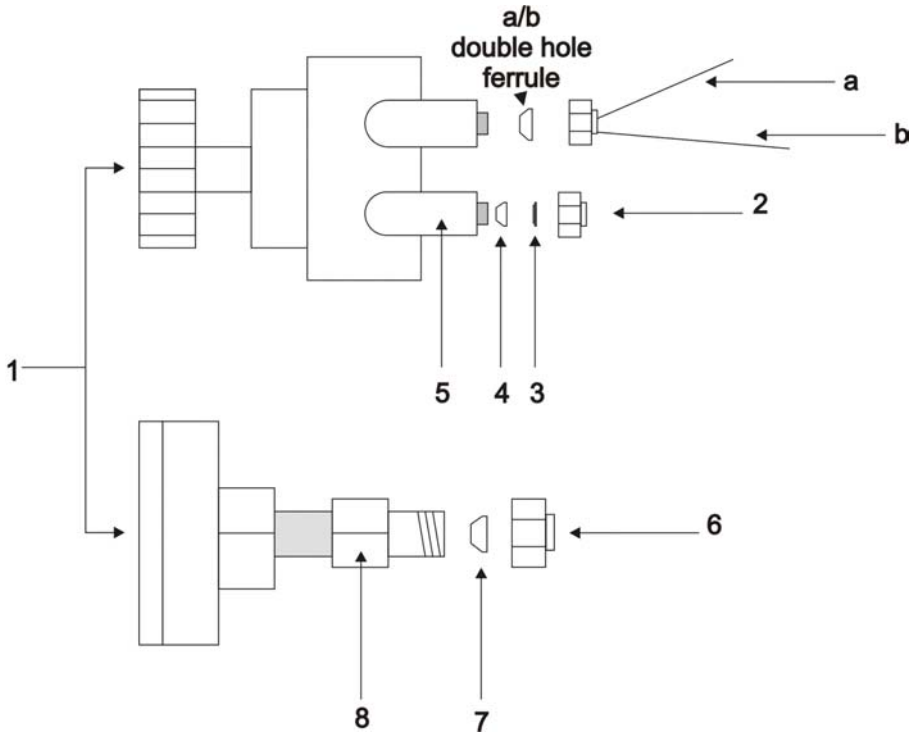


Figure 2-11. Manometer

Table 2-3. Manometer Parts

Pos.	Designation	Part No.
a)	He dilution	a / b double hole ferrule: 106 0170
b)	He purge	
a)	Reference gas	a / b double hole ferrule: 100 4780
b)	Reference purge	
1	Manometer 0 - 2.5 bar	052 43 91
2	Cap nut 1/16´´	052 3610
3	Back ferrule 1/16´´	052 3210
4	Front ferrule 1/16´´	052 3440
5	Elbow	049 3190
6	Cap nut 1/8´´	052 1500
7	Ferrule; Teflon 1/8´´	067 4790
8	Coupling 1/8´´	074 3360

Chapter 3
ConFlo III Interface and Elemental Analyzer

3.1 ConFlo III Software in Old Isodat

The ConFlo III application software allows fully automated determination of the isotope ratios of carbon, nitrogen, oxygen, hydrogen and sulfur from combustible organic matter.

> **Note.** The ConFlo III software is used within the old Isodat software. Therefore, it is necessary to have the old Isodat software properly installed.

The ConFlo III software is installed as follows:

- Stop computer from ***Isodat*** mode by holding the ***Alt*** key while typing ***Quit***.
- Press ***Ctrl*** and ***4*** simultaneously. The computer shows: c:\>ISOTST.
- Type: c:\>ISOTST>***install***.

> **Note.** If the message **Not enough memory** appears:
>
> Type: **BANK=ON**.
>
> Press **ENTER**.

- Press ***ENTER***. The ***<Isodat> Installation Menu*** appears:

1. GEM Installation Menu
2. …
3. …
6. GC/ConFlo III Installation Menu
7. …

- Press ***6***, then ***ENTER***.
- The ***GC/ConFlo Installation Menu*** appears:

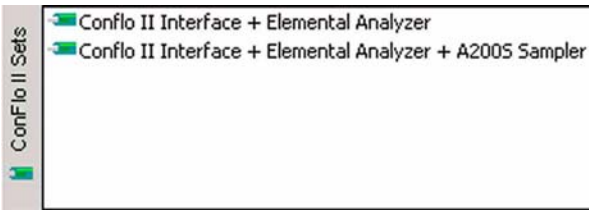
1. …
2. ConFlo III Installation
3. …

- Press ***2***, then ***ENTER*** and continue the procedure.
- After finishing boot the system by pressing ***Ctrl+Alt+Del*** simultaneously.

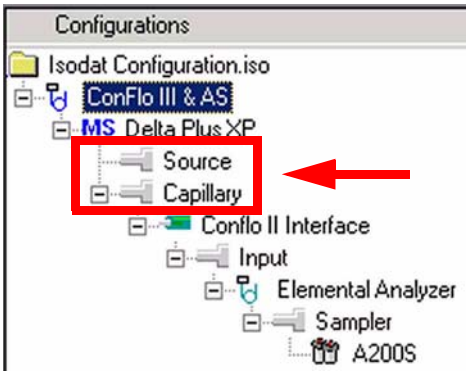
3.2 ConFlo III Configuration in Isodat 2.0

- Before operating, a ***Configuration*** containing the ConFlo II/III Interface needs to be created in the ***Configurator***.

- Press the ***Add Configuration*** button to add a new Configuration.
- Give it a name, e.g. “ConFlo III & AS”.



- Select one of the ConFlo II sets (also for ConFlo III) and drag it either to the ***Source*** port or to the ***Capillary*** port in the new Configuration.
- In case of a ConFlo Interface and an autosampler with a RS 232 interface to the computer select ***ConFlo Interface*** + ***Elemental Analyzer*** + ***A200S Sampler***.



- Open the complete tree structure of the ConFlo Interface to check for the hardware attached (Elemental Analyzer, autosampler).
- In case of a ConFlo II/III and a liquid autosampler with a RS 232 interface to the computer, the COM Port settings can be checked in the ***Advanced Mode***.

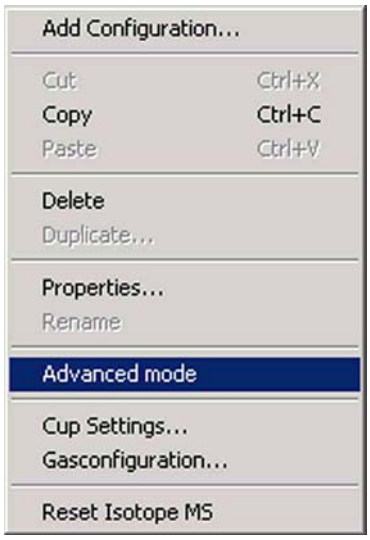
  See **Installation of the A200S and GC PAL Autosampler** on page 3-4.

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

3.3 Installation of the A200S and GC PAL Autosampler

A200S and GC PAL autosampler require operating signals from the software via RS 232 cable (COM Port). Special scripts for using these autosamplers together with the software Isodat 2.0 are installed automatically by the selection of a ConFlo III set with liquid autosampler in the Isodat 2.0 ***Configurator***.

The COM Port settings for the autosampler can be viewed using the ***Advanced Mode*** (i.e. ***Edit > Advanced Mode***):

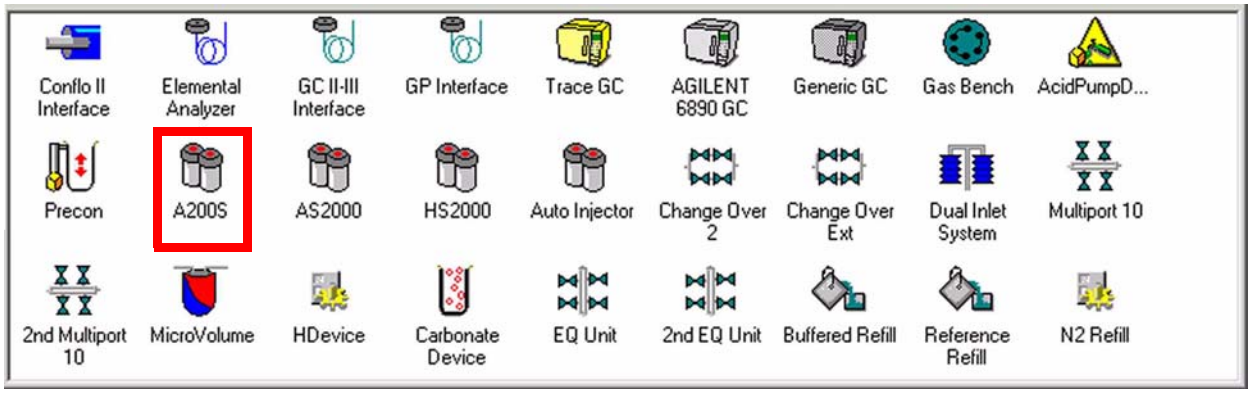


- In the ***Edit*** Menu, activate the ***Advanced Mode***.

- Confirm by ***OK***.

Note. The COM Port settings are essential for proper communication with the autosampler. Incorrect settings will lead to inactive liquid autosampler control.

- Press the ***Devices*** tab.

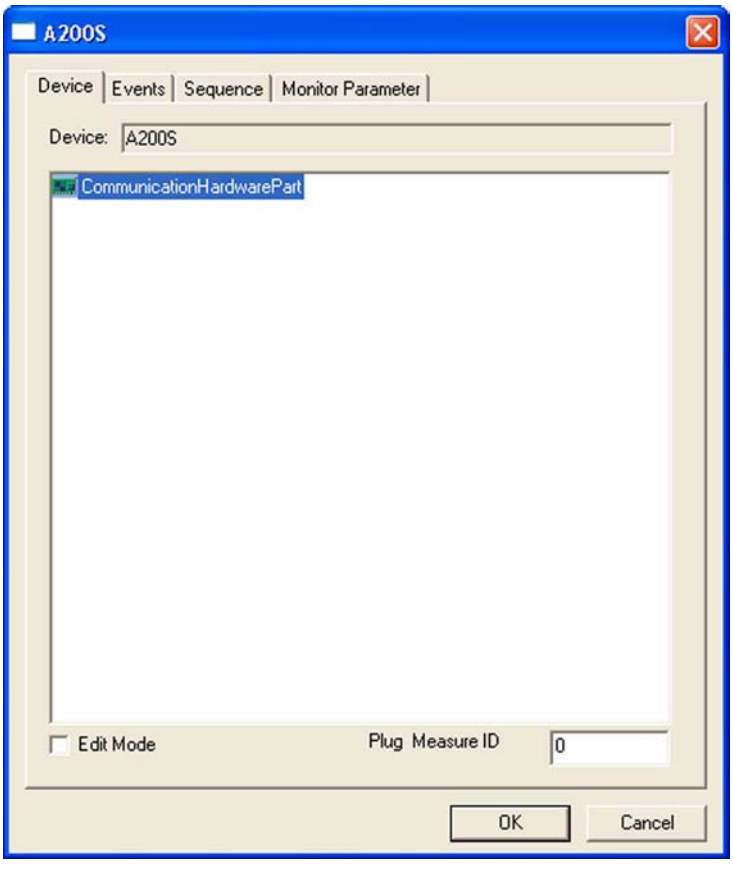


- The pool of available devices is shown.

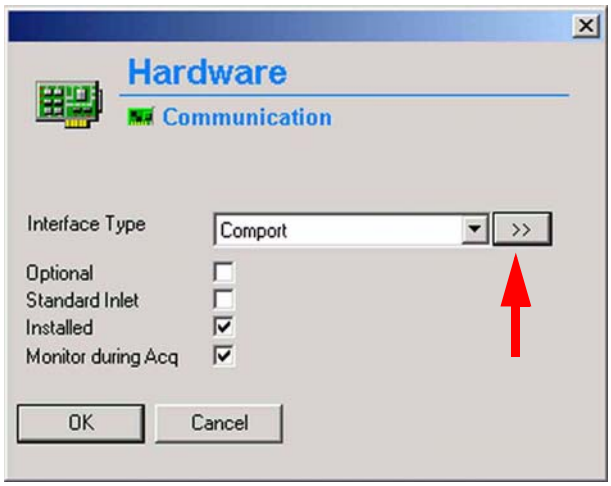
- ***Right-click*** on your autosampler (e.g. A200S). For GC PAL autosampler also right-click A200S.
- Select ***Properties***, if you only want to check the COM Port your autosampler is connected to or
- Select ***Edit...*** to edit the default communication parameters.

- If you selected ***Properties*** above, choose the COM Port your autosampler is connected to, e.g. COM 1.
- Confirm by ***OK***.

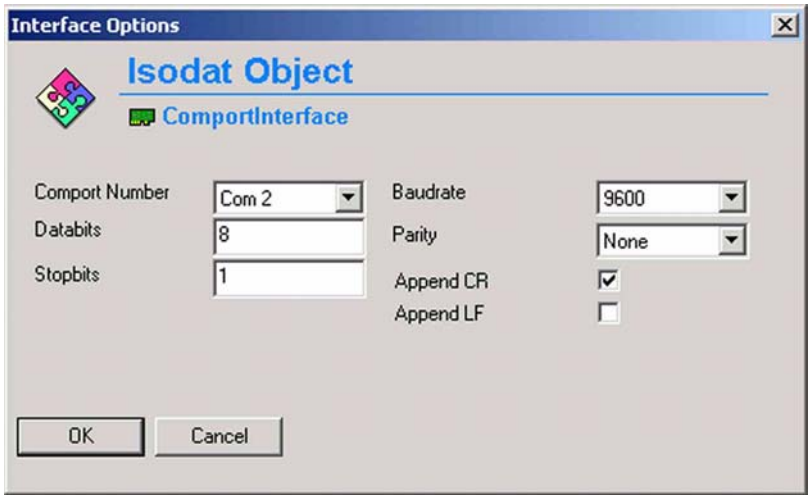
> **Note.** The **Properties** command is available also in **Normal** Mode, i.e. you don't need to be in **Advanced** Mode to only check the COM Port your autosampler is connected to.
>
> The **Edit...** command, however, is available only in **Advanced** Mode.



- If you selected ***Edit...*** above, double-click on ***Communication Hardware Part***. Alternatively, right-click on it and select ***Properties***.



- The default settings are shown above. Usually, they need not to be edited.
- Choose your interface type ***(Comport)***.
- To specify its properties, press the >> button.

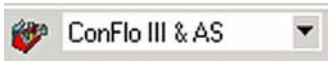


- The default settings are shown above (usually kept default).
- Check, whether the COM Port corresponds to the COM Port your autosampler is connected to. Confirm by ***OK***.

> **Note.** The COM Port settings are essential for proper communication. Incorrect settings will lead to inactive hardware control.

3.4 Manual Operation of ConFlo III

- Open the ***Instrument Control*** module.



- Select a ***Configuration*** that contains the ConFlo III interface.

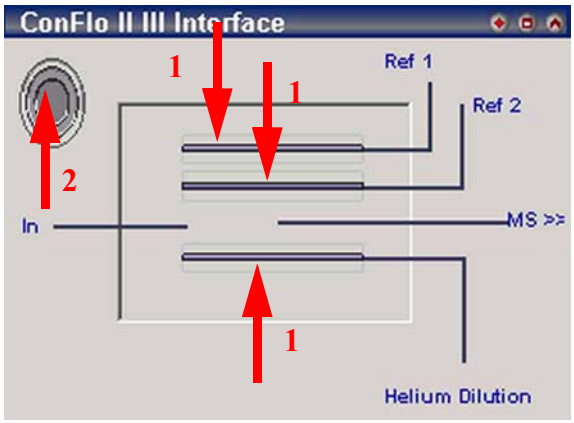


Figure 3-1. ConFlo II III Interface Window

The ***ConFlo II III Interface window*** allows:

reference gas inlet and/or dilution (**1**)

autosampler/Elemental Analyzer start signal (**2**)

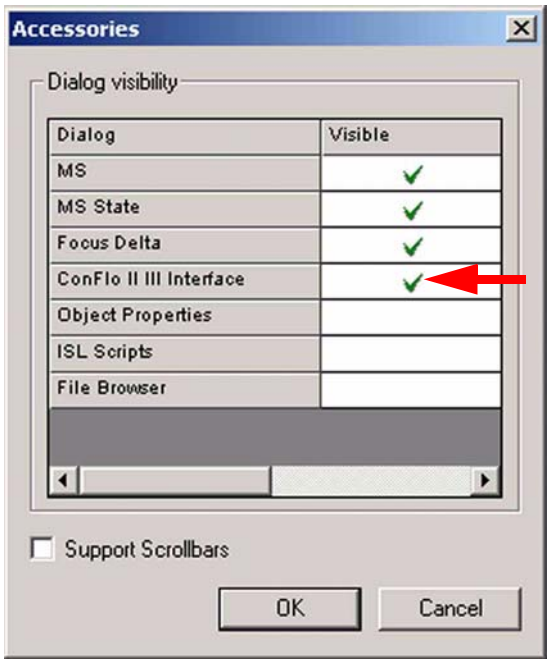
Capillaries can be moved/activated by mouse clicks.

- Open ***Reference 1*** by a ***click*** on ***Ref 1*** capillary. If CO_2 is connected to ***Reference 2***, click ***Ref 2*** capillary.

  Thus, the blue capillary symbol elongates and CO_2 enters the system.

Comment: If the ConFlo II III Interface Window is not Visible

- ***Right-click*** somewhere into the ***Accessories*** toolbar.
- Click on the ***Administrative Panels...*** button.



The ***Accessories*** window allows to mark dialogs (i.e. windows) by ✓ . If you mark a dialog, the respective window will be shown at the ***Accessories*** toolbar.

- Mark ***ConFlo II III Interface***.

- Confirm by ***OK***.

Thus, the ConFlo II III Interface window will be shown at the ***Accessories*** toolbar.

Figure 3-2. Visible Windows at the Accessories Toolbar

Comment: If only the Title Bar of the ConFlo II III Interface Window is Visible

- Click on the ***Unshrink*** button at the right corner of the Title bar.
  Thus, ConFlo II III Interface window will appear.

3.5 How to Get Started with ConFlo III and Elemental Analyzer

1. Connect the Elemental Analyzer to ConFlo III for the transfer of the start signal.
2. In case you have Teflon tubing, replace it with stainless steel at your Elemental Analyzer, because atmospheric N_2 passes Teflon and interferes the quality of the determination.
3. Connect the reducing valves to the gas supplies (He, O_2, N_2, CO_2).
4. Connect the gas tank of O_2 and He to the Elemental Analyzer.
5. Connect He, N_2 (as Ref 2) and CO_2 (as Ref 1) to ConFlo III.
6. Open the main valves on the gas tanks and adjust the pressure.
7. Leak-check all connections and valves outside ConFlo III.
8. Make sure that the Elemental Analyzer is set up (i.e. oxidation and reduction reactors are packed and installed, water trap is installed, autosampler is fixed etc. Also refer to the Elemental Analyzer's manual).
9. Connect the Elemental Analyzer to the computer, if the Elemental Analyzer is run by separate software, i.e. Eager software.
10. Connect the Elemental Analyzer to the 220 V main power supply.
11. Connect compressed air to ConFlo III.
12. Connect compressed air to Elemental Analyzer if necessary (depending on the autosampler).
13. Connect ConFlo III to the IRMS (rear panel).
14. Ensure that the IRMS connection cable is connected to the driver board of the computer.
15. Make sure that the ConFlo III configuration has been installed during Isodat 2.0 setup.
    See **ConFlo III Configuration in Isodat 2.0** on page 3-3.
16. By using the ConFlo III device window in Isodat 2.0, make sure that the respective pistons of ConFlo III are movable (click on them using your mouse) and watch them moving in the combi inlet of ConFlo III.
17. Connect the fused silica capillaries of ConFlo III to the needle valve of the IRMS.
18. Switch on the Elemental Analyzer.
19. Adjust the gas pressures of the Elemental Analyzer and ConFlo III. Take the following settings as a guideline:

Table 3-1. Adjustment of Gas Pressures

He	Elemental Analyzer	100 kPa
O_2	Elemental Analyzer	120 kPa
Purge with	He	110 - 200 ml/min depending on your autosampler:
		• AS 200 and AS 128: 110 ml/min
		• MAS 200: 200 ml/min
He	ConFlo III	0.8 bar
N_2	ConFlo III	1.3 bar
CO_2	ConFlo III	0.9 bar

Caution. To prevent atmospheric air from entering the open split cell, never set the He flow on ConFlo III to less than 0.5 bar.

20. Switch off the ion source.
21. Inject the He dilution capillary to ensure only He entering the source.
22. Open the needle valve slowly.
23. Make sure the ion source high vacuum is between $3 * 10^{-6}$ mbar and $1.5 * 10^{-6}$ mbar.
24. Switch the ion source on. The expected background after approximately 30 min should be as described below:

Table 3-2. Expected Background After Approximately 30 min

mass	measured in cup for mass	resistor [Ω]	expected background
28	28	$3 * 10^8$	< 20 mV
29	29	$3 * 10^{10}$	< 20 mV
18	29	$3 * 10^{10}$	< 10 mV
40	29	$3 * 10^{10}$	< 10 mV

Note. Background values may vary depending on sensitivity and focus settings. They are given here as a general guideline.

25. Leak-check the needle valve.

> **Note.** Leak check can be performed using a triggling stream of Ar while monitoring mass 40 on the IRMS.

26. Switch the needle valve heater ***ON***.
27. Switch the ion source heater ***ON***.
28. Leak-check all connections of the Elemental Analyzer following the procedure described in the respective Elemental Analyzer manual.
29. Heat up the following parts of the Elemental Analyzer in three steps.

> **Note.** The Elemental Analyzer should not be connected to ConFlo III yet!

Table 3-3. Heating Up Elemental Analyzer Parts in Three Steps

	step 1	step 2	step 3
oxidation furnace	400 °C	750 °C	1020 °C
reduction furnace	400 °C	500 °C	650 °C
column	40 °C	75 °C	120 °C

30. Leak-check after each step and wait for two hours after reaching the final temperature.
31. Keep the system overnight in the last status to remove organic material and water and for column conditioning.
32. Also leave ConFlo III under above conditions overnight to remove all organic material from the fused silica capillaries, connection lines, valves, manometers etc.
33. Cool down the GC column to 40 °C for measurements.
34. Before connecting the Elemental Analyzer to ConFlo III, measure the He flow at the end of the stainless steel capillary.
    Adjust the pressure at the Elemental Analyzer to set the flow to 80 ml/min - 100 ml/min depending on sample and application to be performed.
35. Connect the Elemental Analyzer to ConFlo III.
36. Make sure that no standard gas is injected, He dilution is ***off*** and all other valves are closed.

The expected background is:

Table 3-4. Expected Background

mass	measured in cup for mass	resistor [Ω]	expected background
28	28	$3 * 10^8$	< 20 mV
29	29	$3 * 10^{10}$	< 20 mV
18	29	$3 * 10^{10}$	< 2 mV
40	29	$3 * 10^{10}$	< 10 mV

Note. Background values may vary depending on sensitivity and focus settings. They are given here as a general guideline.

Note. The above ion intensity depends on the sensitivity of the IRMS. If the ion intensity is higher than expected and no leak is present, the background must decrease after two or three days and stabilize.

Chapter 4
Nitrogen Measurement

4.1 Zero Enrichment of Nitrogen (Standard On/Off Test)

We assume that the user already has working experience with the ConFlo III Interface and IRMS. It is recommended to perform a simple check in order to test the analytical condition of ConFlo III and IRMS before measuring any samples.The most important checks to test the analytical condition are ***zero enrichment*** and ***linearity test***.

1. Only as a guideline use the following method from the File Browser's ***Examples*** folder (***N2_zero.met***):

Instrument

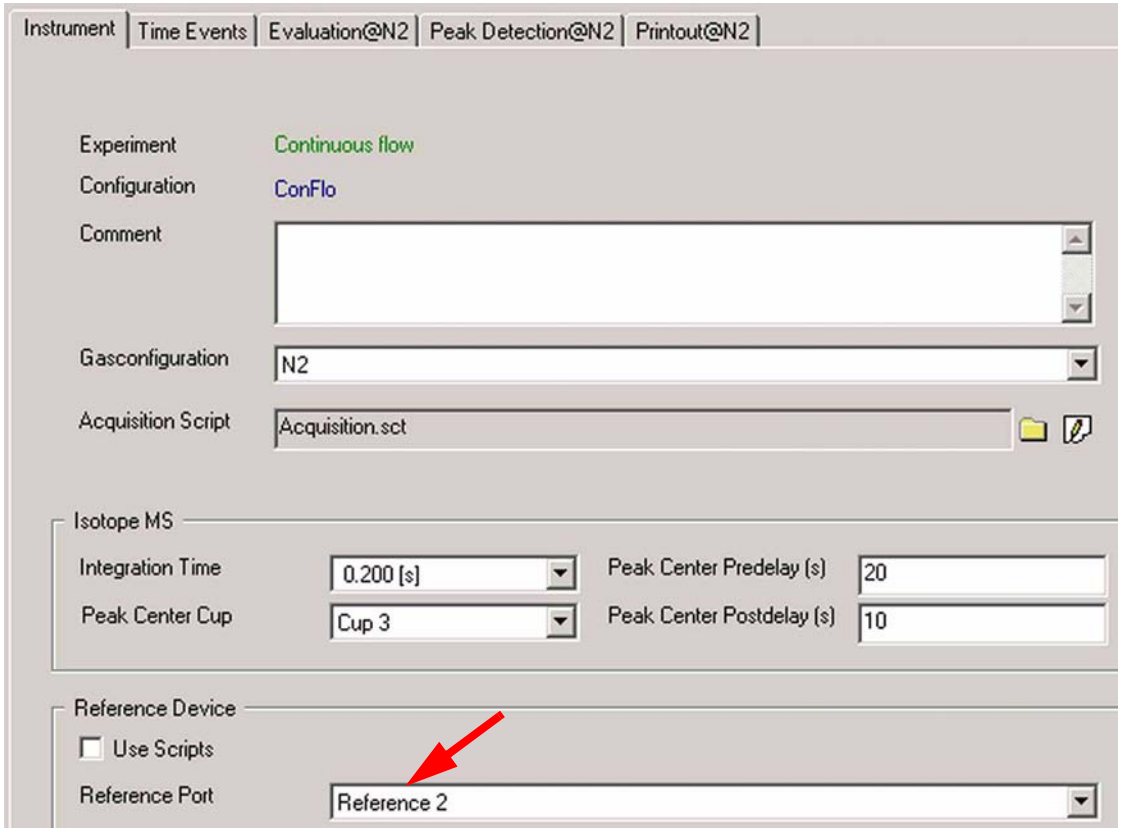


Figure 4-1. Zero Enrichment of Nitrogen - Instrument Tab

- Select the reference port your reference gas is connected to at the ConFlo interface (e.g. Reference 2).
- Adapt the reference gas port to the respective column of the Time Events list: e.g. if you choose Reference 2, the on-off entries must occur in the Reference 2-On column of the Time Events list (see Figure 4-2).

Time Events

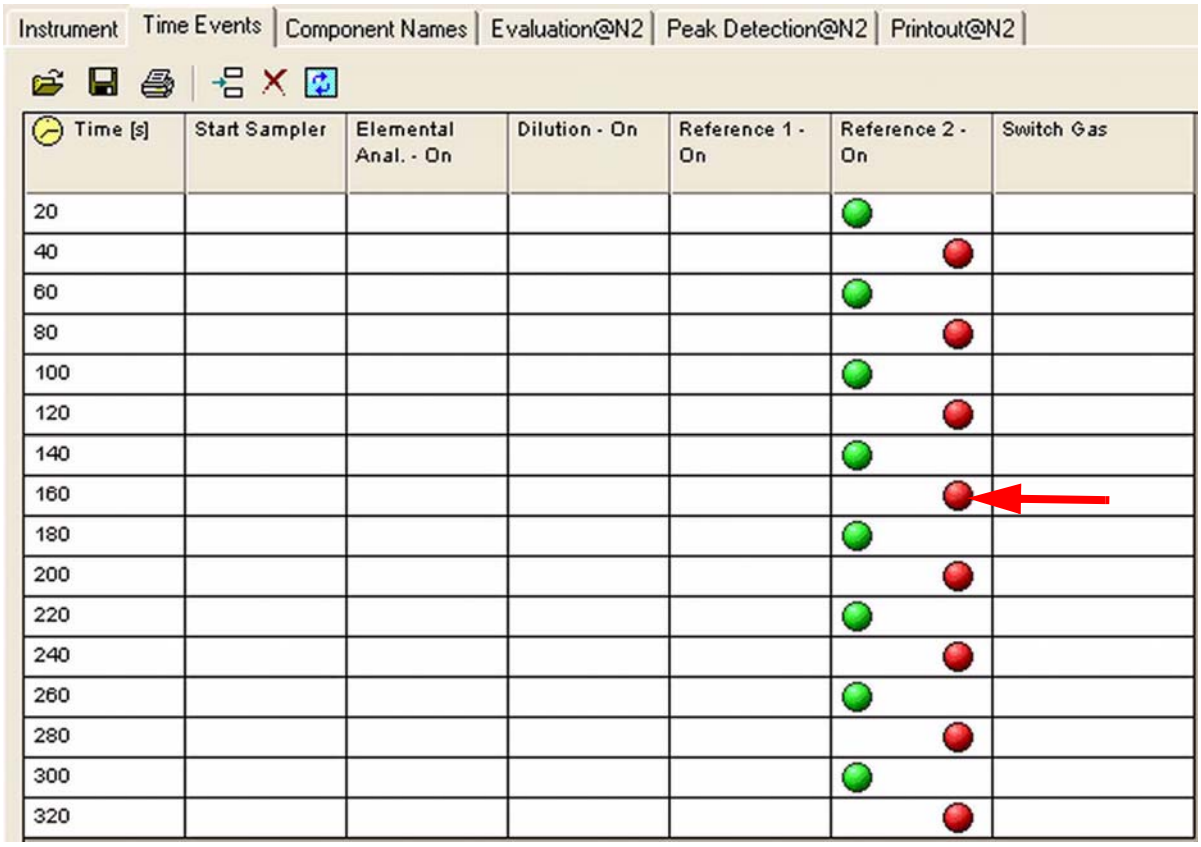


Figure 4-2. Zero Enrichment of Nitrogen - Time Events Tab

- Recognize the eight on-off pulses shown above in the expected chromatogram (Figure 4-8).
- Note that the off-time of the e.g. forth reference gas pulse is 160 s (Figure 4-4).

Figure 4-3. Zero Enrichment of Nitrogen - Acquisition Start and Acquisition End Time

Evaluation

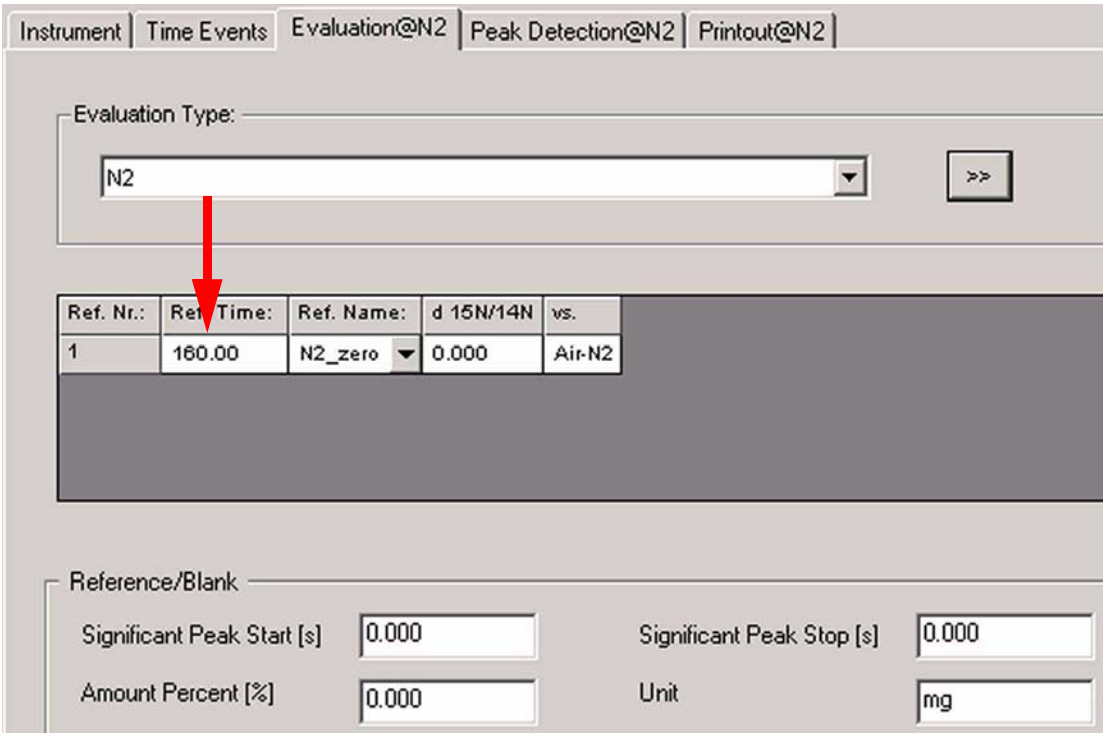


Figure 4-4. Zero Enrichment of Nitrogen - Evaluation Tab

> **Note.** At **Ref. Time**, the **off**-value of an arbitrary reference gas peak according to the Time Events list must be typed (e.g. 160 s as the **off**-value of the forth reference gas peak, see Figure 4-2).

Peak Detection

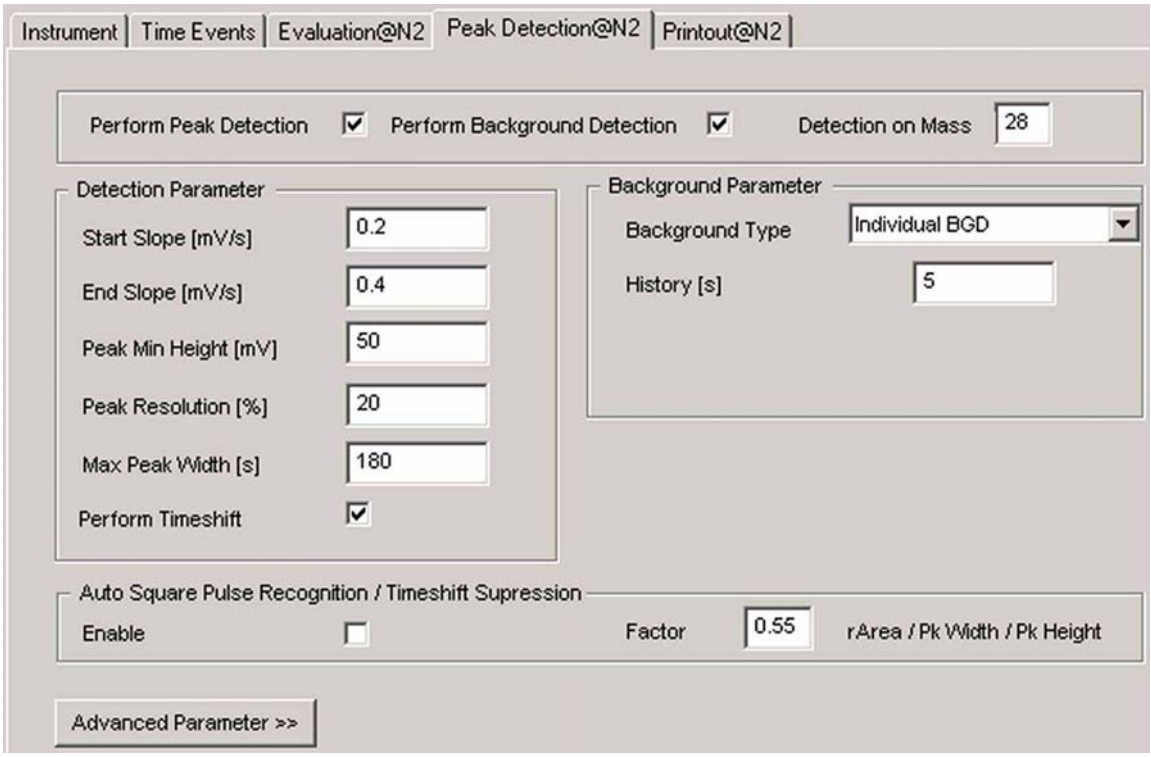


Figure 4-5. Zero Enrichment of Nitrogen - Peak Detection Tab

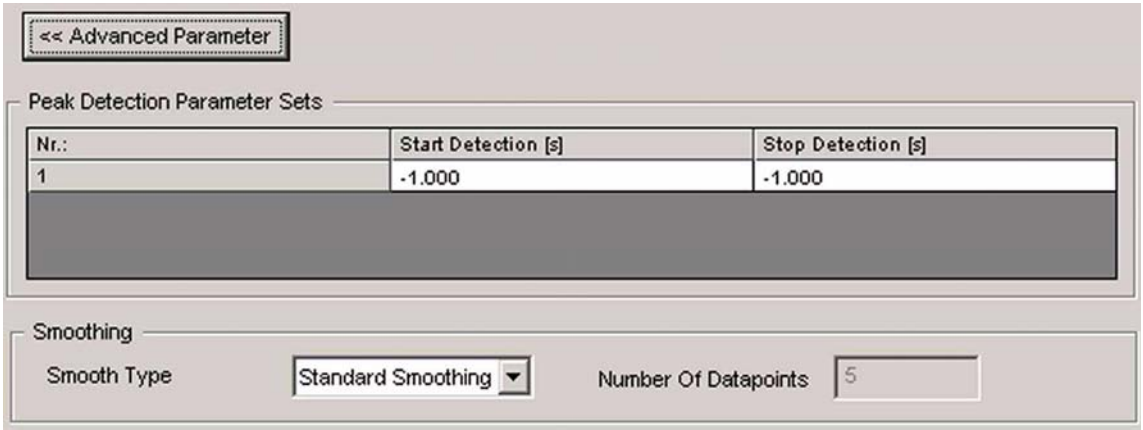


Figure 4-6. Zero Enrichment of Nitrogen - Advanced Parameters in Peak Detection Tab

> **Note.** A value of -1 denotes unlimited.

Printout

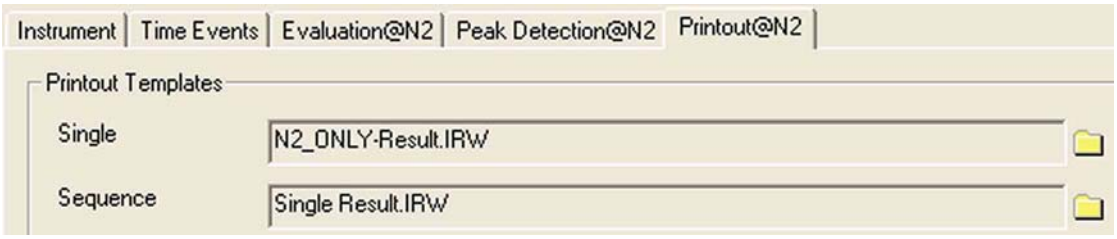


Figure 4-7. Zero Enrichment of Nitrogen - Printout Tab

2. At the corresponding pressure regulator of ConFlo III, set the ion intensity of mass 28 (i.e. $^{14}N^{14}N$) to 3 V - 4 V.
3. Create a new Sequence.
4. Press the ***Start*** button and confirm by ***OK***.
5. Expected data after three or four measurements:

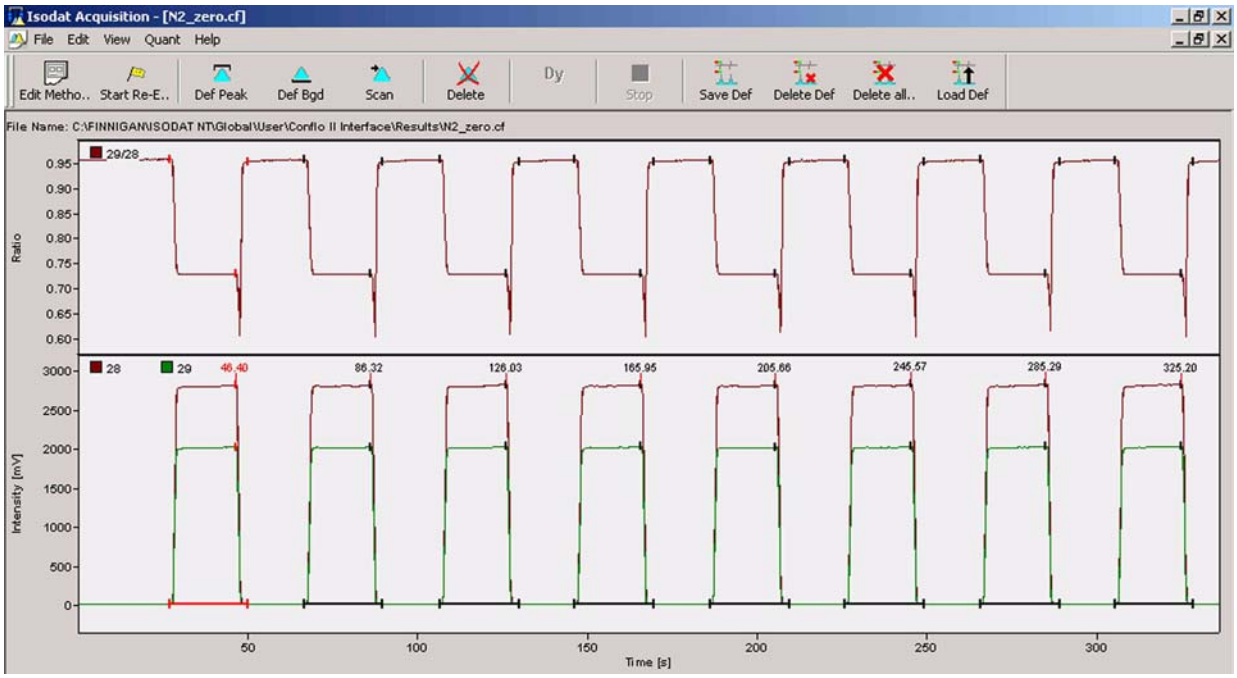
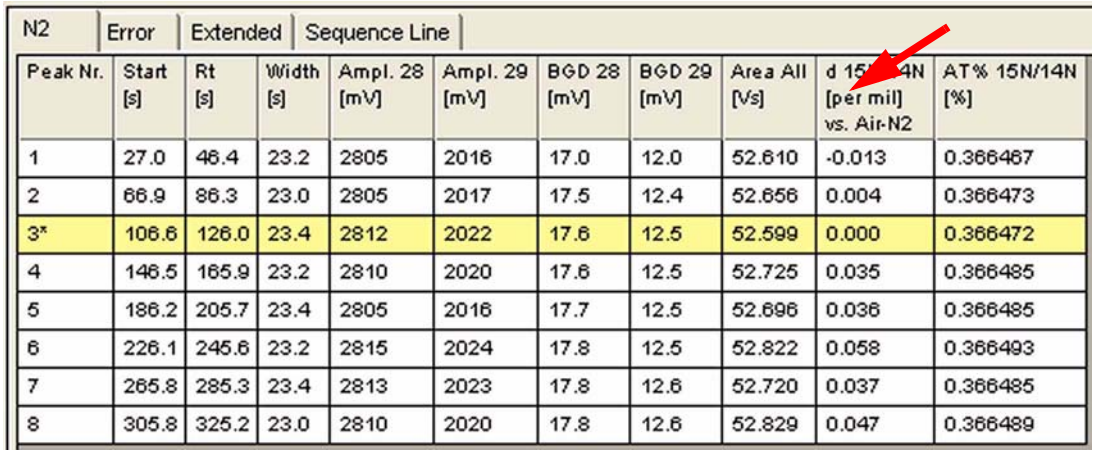


Figure 4-8. Zero Enrichment of Nitrogen - Chromatogram

Recognize the eight peaks shown above as the eight on-off pulses in the Time Events list (Figure 4-2).



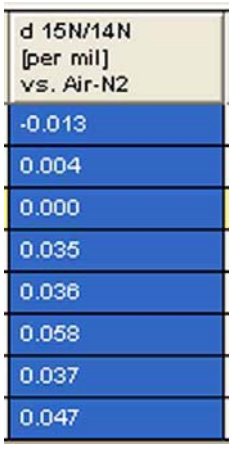
N2 | Error | Extended | Sequence Line

Peak Nr.	Start [s]	Rt [s]	Width [s]	Ampl. 28 [mV]	Ampl. 29 [mV]	BGD 28 [mV]	BGD 29 [mV]	Area All [Vs]	d 15/14N [per mil] vs. Air-N2	AT% 15N/14N [%]
1	27.0	46.4	23.2	2805	2016	17.0	12.0	52.610	-0.013	0.366467
2	66.9	86.3	23.0	2805	2017	17.5	12.4	52.656	0.004	0.366473
3*	106.6	126.0	23.4	2812	2022	17.6	12.5	52.599	0.000	0.366472
4	146.5	165.9	23.2	2810	2020	17.6	12.5	52.725	0.035	0.366485
5	186.2	205.7	23.4	2805	2016	17.7	12.5	52.696	0.036	0.366485
6	226.1	245.6	23.2	2815	2024	17.8	12.5	52.822	0.058	0.366493
7	265.8	285.3	23.4	2813	2023	17.8	12.6	52.720	0.037	0.366485
8	305.8	325.2	23.0	2810	2020	17.8	12.6	52.829	0.047	0.366489

Figure 4-9. Zero Enrichment of Nitrogen - Result Grid

> **Note.** The standard deviation of $\delta^{15}N$ ($^{15}N^{14}N/^{14}N^{14}N$) should be less than ***0.05 ‰***.

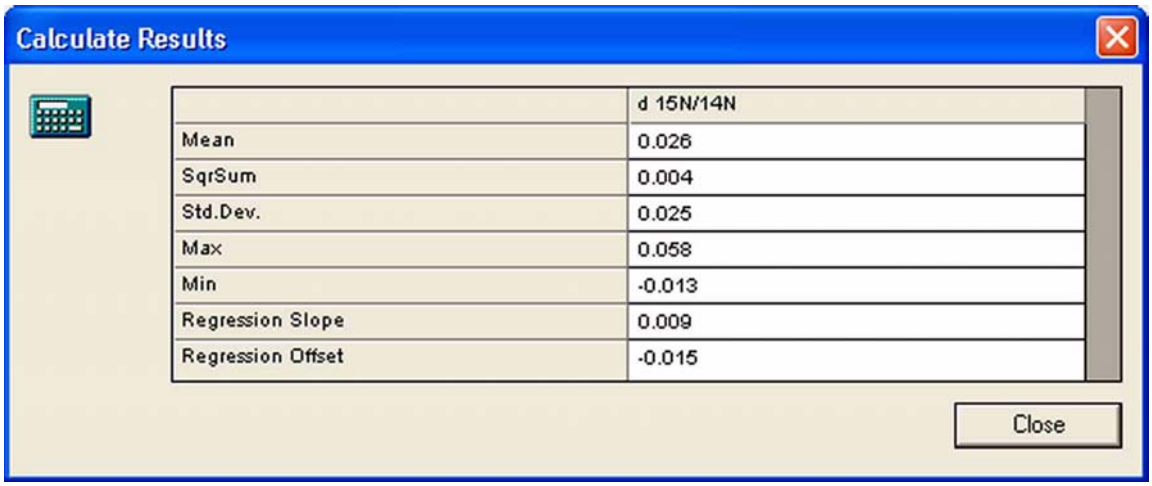
To obtain the standard deviation of all eight peaks perform as follows:



d 15N/14N [per mil] vs. Air-N2
-0.013
0.004
0.000
0.035
0.036
0.058
0.037
0.047

- Click on the column header of the ***d 15N/14N [per mil] vs. Air-N2*** column. It will be highlighted.

- Right-click on the column header.
- Choose ***Calculate***.



4.2 Linearity Test of Nitrogen

- Use the same method as defined for zero enrichment (see chapter **Zero Enrichment of Nitrogen (Standard On/Off Test)** on page 4-2).
- Start the acquisition as a single run.
- At each detection of a peak de- or increase the reference gas pressure at ConFlo III. Expected data after three or four measurements:

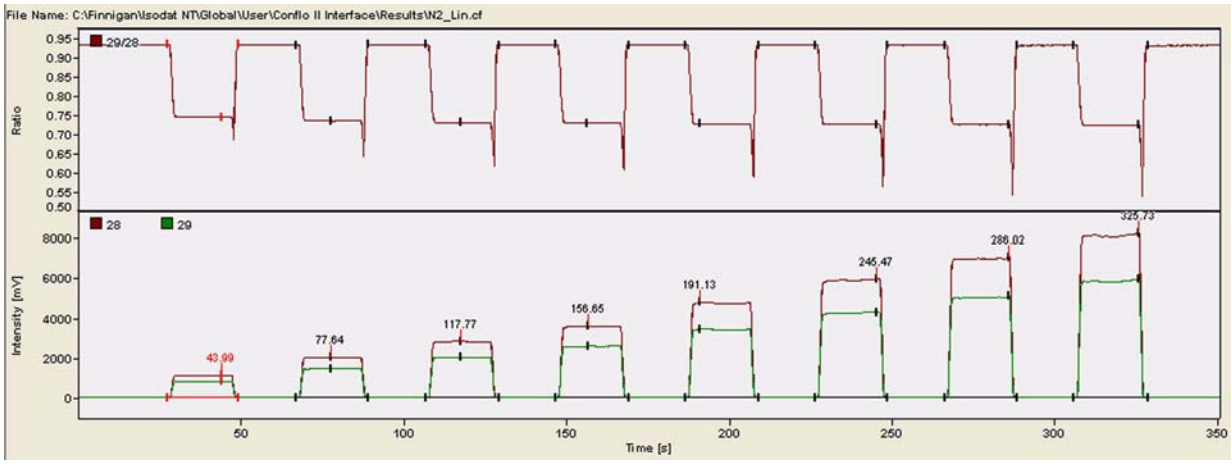


Figure 4-10. Linearity Test of Nitrogen - Chromatogram

N2 | Error | Extended | Sequence Line

Peak Nr.	Start [s]	Rt [s]	Width [s]	Ampl. 28 [mV]	Ampl. 29 [mV]	BGD 28 [mV]	BGD 29 [mV]	Area All [Vs]	d 15N/14N [per mil] vs. Air-N2	AT% 15N/14N [%]
1	27.5	44.0	21.9	1109	800	36.8	27.8	20.617	0.050	0.366490
2	67.2	77.6	22.2	2008	1449	36.7	27.7	37.623	0.101	0.366509
3*	106.9	117.8	22.4	2809	2026	36.8	27.7	52.679	0.000	0.366472
4	146.8	156.6	22.4	3577	2581	36.9	27.8	67.121	0.016	0.366478
5	186.5	191.1	22.6	4758	3433	37.0	27.9	89.271	0.046	0.366489
6	226.5	245.5	22.4	5932	4281	37.1	28.0	111.019	0.097	0.366507
7	266.4	286.0	22.4	7029	5072	37.3	28.1	131.249	0.113	0.366513
8	306.1	325.7	22.8	8194	5913	37.5	28.2	152.882	0.170	0.366534

Figure 4-11. Linearity Test of Nitrogen - Result Grid

> **Note.** The linear regression of the $\delta^{15}N/^{14}N$ values vs. the working standard should be less than ***0.06 ‰/V***.

Chapter 5
Carbon Measurement

5.1 Zero Enrichment of Carbon (Standard On/Off Test)

We assume that the user already has working experience with the ConFlo III Interface and IRMS. It is recommended to perform a simple check in order to test the analytical condition of ConFlo III and IRMS before measuring any samples.The most important checks to test the analytical condition are ***zero enrichment*** and ***linearity test***.

1. Only as a guideline use the following Method from the File Browser's ***Examples*** folder (***CO2_zero.met***):

Instrument

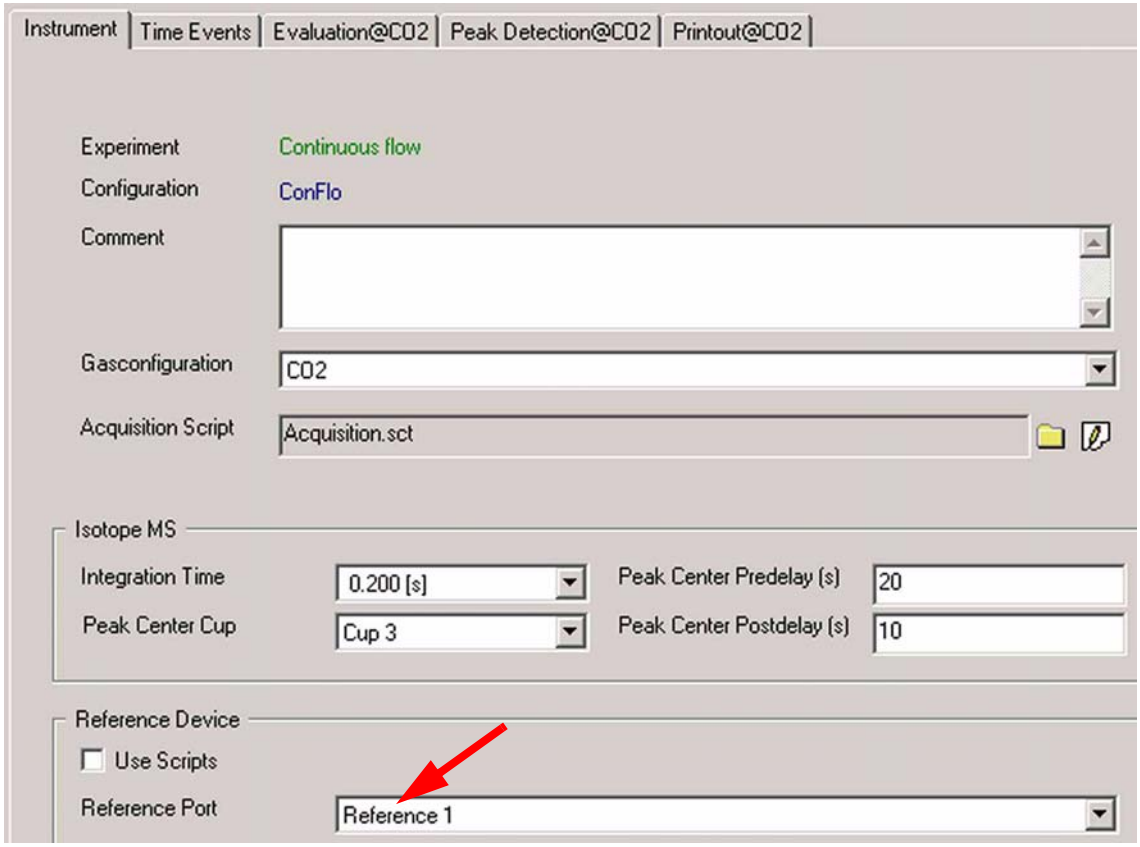


Figure 5-1. Zero Enrichment of Carbon - Instrument tab

- Select the reference port your reference gas is connected to at the ConFlo interface (e.g. Reference 1).
- Adapt the reference gas port to the respective column of the Time Events list: e.g. if you choose Reference 1, the on-off entries must occur in the Reference 1-On column of the Time Events list (see Figure 5-2).

Time Events

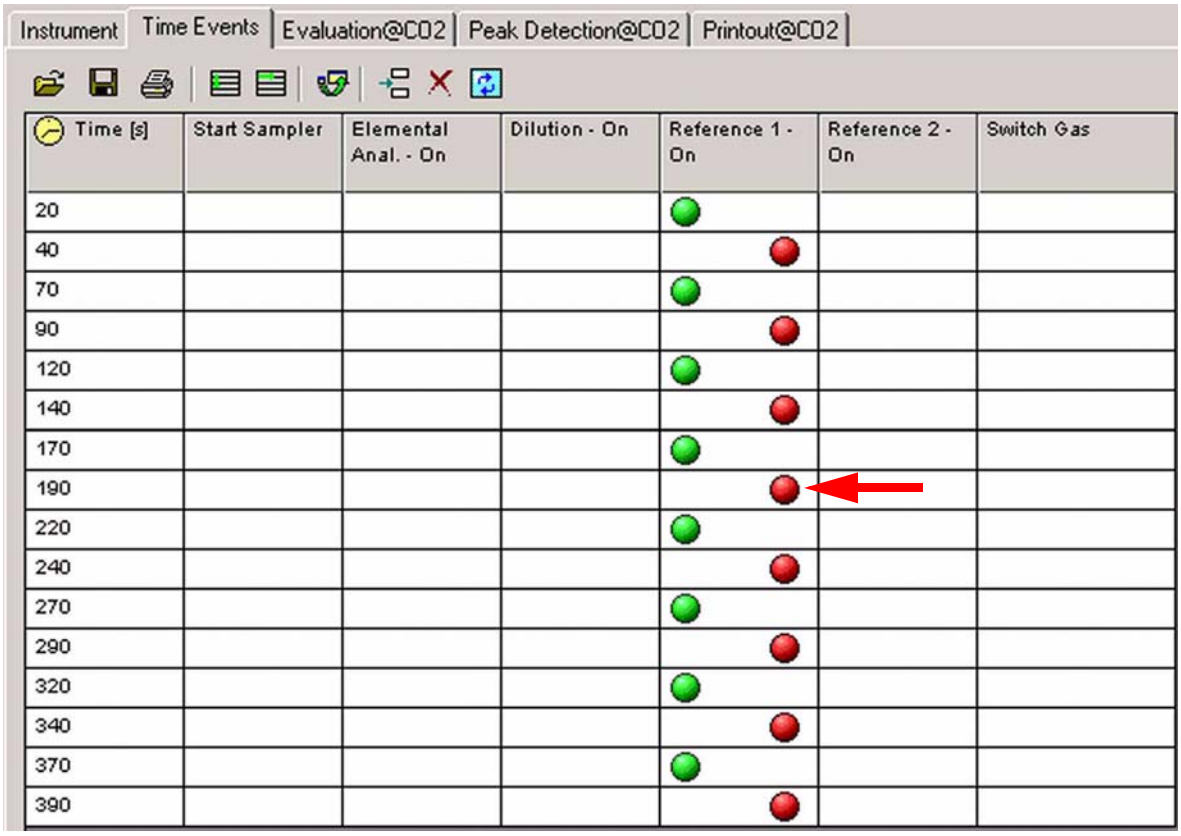


Figure 5-2. Zero Enrichment of Carbon - Time Events tab

- Recognize the eight on-off pulses shown above in the expected chromatogram (Figure 5-8).
- Note that the off-time of the e.g. forth reference gas pulse is 190 s (Figure 5-4).

Figure 5-3. Zero Enrichment of Carbon - Acquisition Start and Acquisition End Time

Evaluation

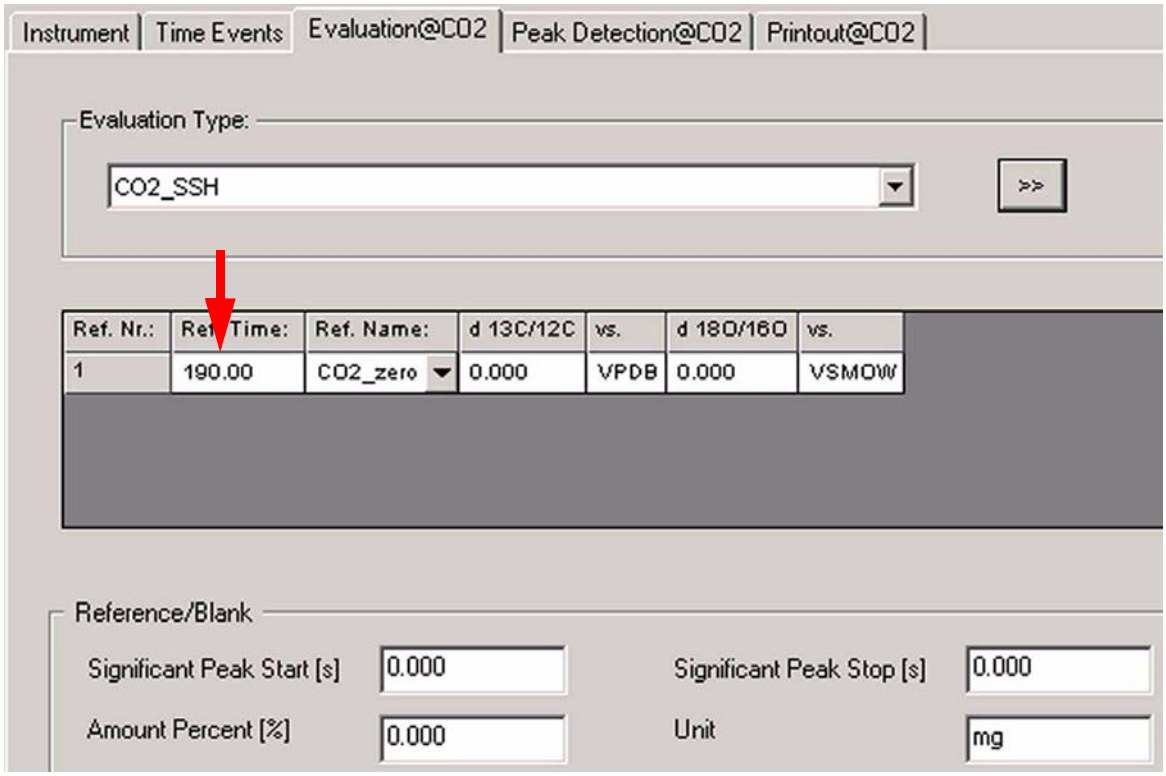


Figure 5-4. Zero Enrichment of Carbon - Evaluation tab

> **Note.** At **Ref. Time**, the **off**-value of an arbitrary reference gas peak according to the Time Events list must be typed (e.g. 190 s as the **off**-value of the forth reference gas peak, see Figure 5-2).

Peak Detection

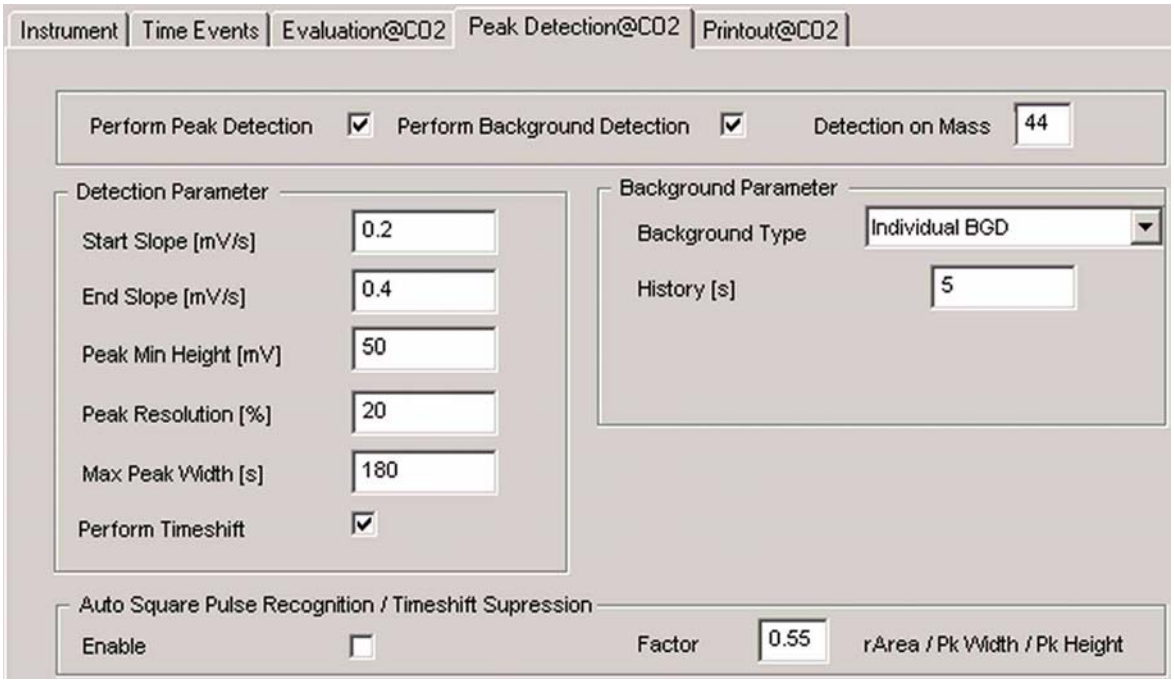


Figure 5-5. Zero Enrichment of Carbon - Peak Detection tab

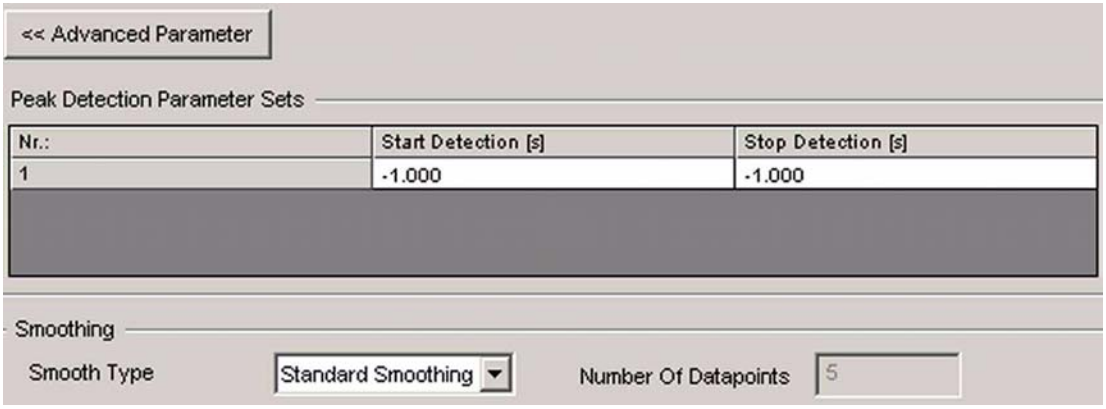


Figure 5-6. Zero Enrichment of Carbon - Advanced Parameters in Peak Detection tab

> **Note.** A value of -1 denotes unlimited.

Printout

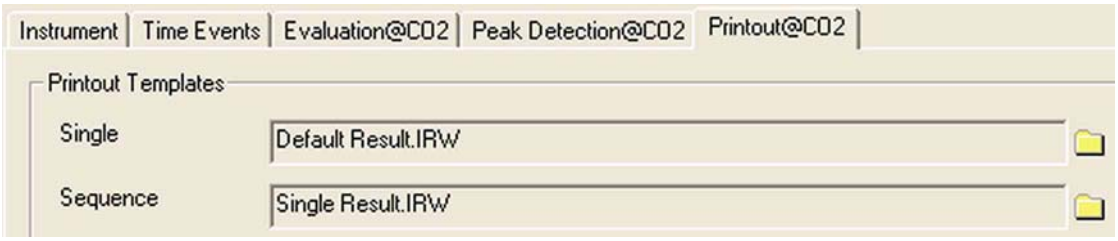


Figure 5-7. Zero Enrichment of Carbon - Printout tab

2. At the corresponding pressure regulator of ConFlo III, set the ion intensity of mass 44 (i.e. $^{12}C^{16}O^{16}O$) to 3 V - 4 V.
3. Create a new Sequence.
4. Press the ***Start*** button and confirm by ***OK***.
5. Expected data after three or four measurements:

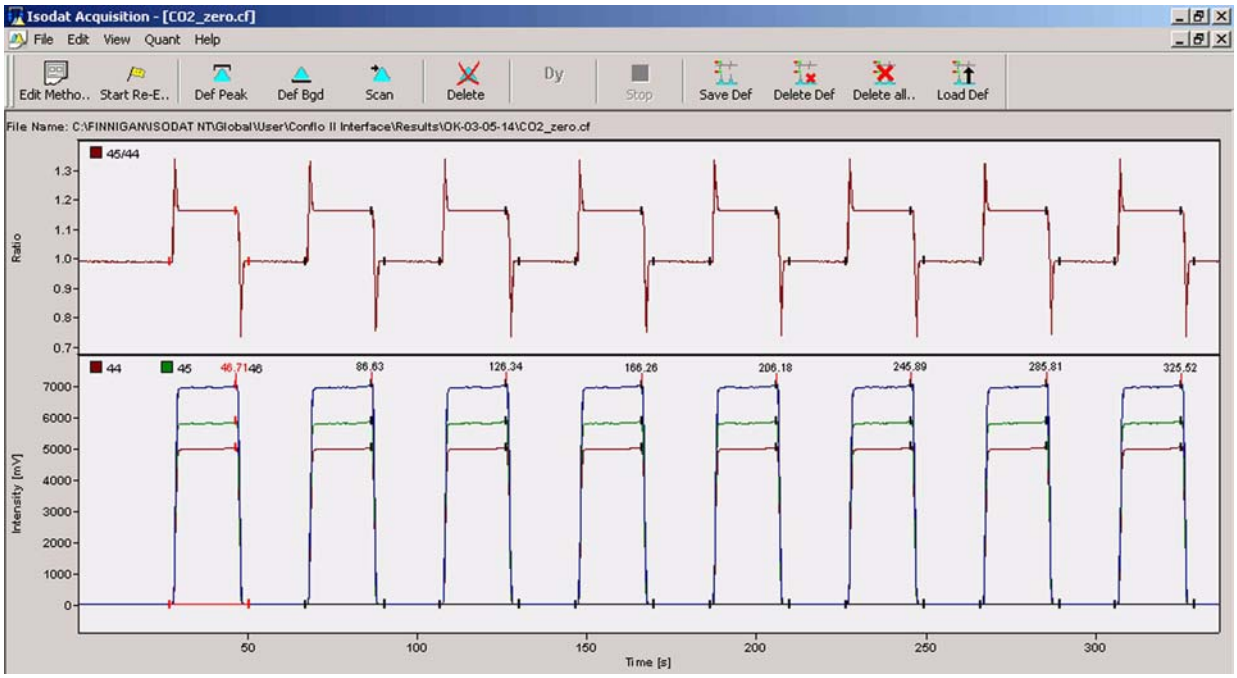
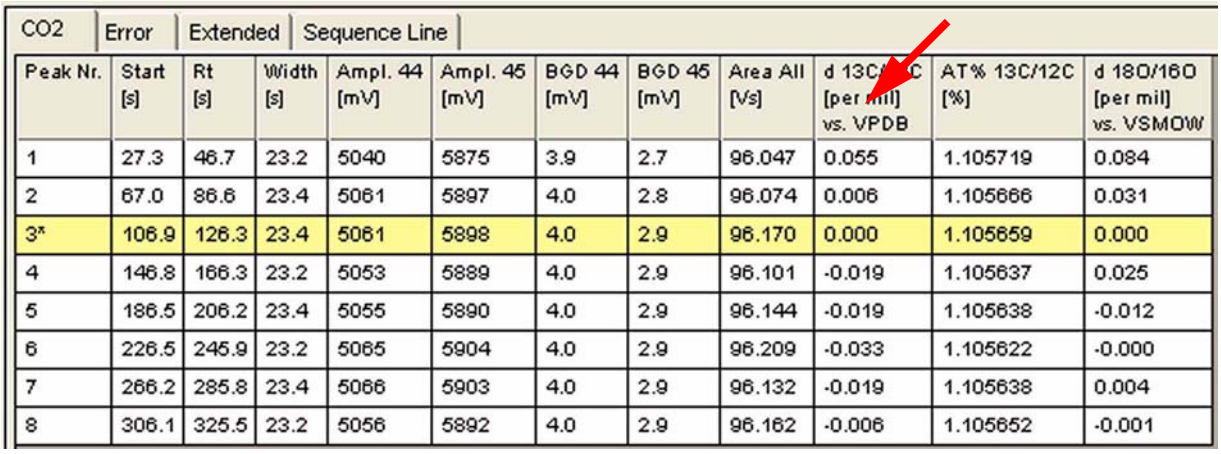


Figure 5-8. Zero Enrichment of Carbon - Chromatogram

Recognize the eight peaks shown above as the eight on-off pulses in the Time Events list (Figure 5-2).



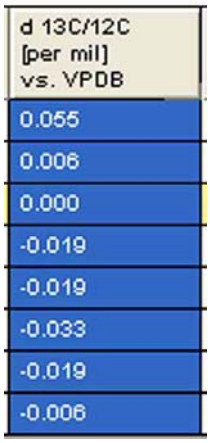
CO2 | Error | Extended | Sequence Line

Peak Nr.	Start [s]	Rt [s]	Width [s]	Ampl. 44 [mV]	Ampl. 45 [mV]	BGD 44 [mV]	BGD 45 [mV]	Area All [Vs]	d 13C/12C [per mil] vs. VPDB	AT% 13C/12C [%]	d 18O/16O [per mil] vs. VSMOW
1	27.3	46.7	23.2	5040	5875	3.9	2.7	96.047	0.055	1.105719	0.084
2	67.0	86.6	23.4	5061	5897	4.0	2.8	96.074	0.006	1.105666	0.031
3*	106.9	126.3	23.4	5061	5898	4.0	2.9	96.170	0.000	1.105659	0.000
4	146.8	166.3	23.2	5053	5889	4.0	2.9	96.101	-0.019	1.105637	0.025
5	186.5	206.2	23.4	5055	5890	4.0	2.9	96.144	-0.019	1.105638	-0.012
6	226.5	245.9	23.2	5065	5904	4.0	2.9	96.209	-0.033	1.105622	-0.000
7	266.2	285.8	23.4	5066	5903	4.0	2.9	96.132	-0.019	1.105638	0.004
8	306.1	325.5	23.2	5056	5892	4.0	2.9	96.162	-0.006	1.105652	-0.001

Figure 5-9. Zero Enrichment of Carbon - Result Grid

Note. The standard deviation of $\delta^{13}C/^{12}C$ should be ***0.05 ‰*** or less.

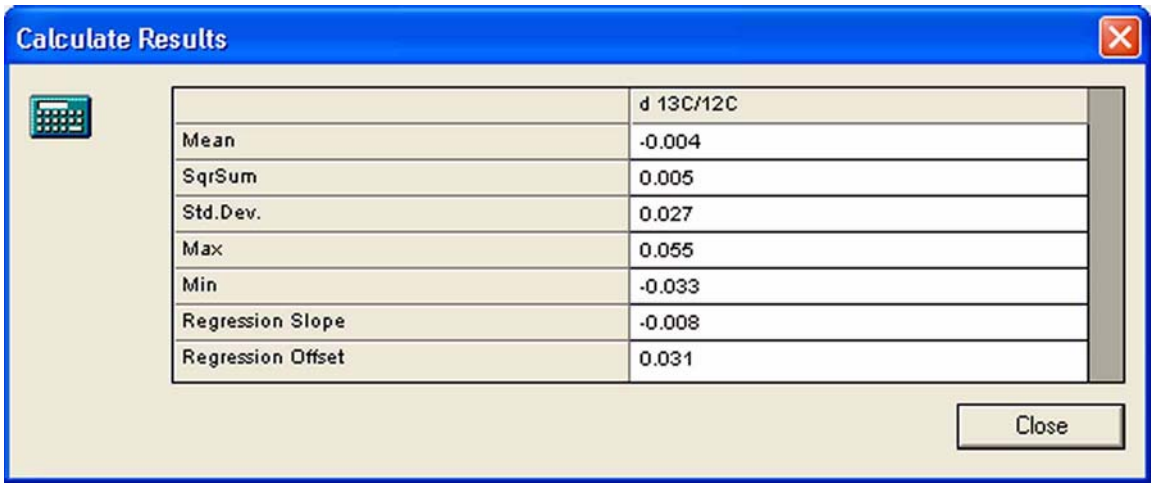
To obtain the standard deviation of all eight peaks perform as follows:



d 13C/12C [per mil] vs. VPDB
0.055
0.006
0.000
-0.019
-0.019
-0.033
-0.019
-0.006

- Click on the column header of the ***d 13C/12C [per mil] vs. VPDB*** column. It will be highlighted.

- Right-click on the column header.
- Choose ***Calculate***.



5.2 Linearity Test of Carbon

- Use the same method as defined for ***zero enrichment*** (i.e. ***CO2_zero.met***, see chapter **Zero Enrichment of Carbon (Standard On/Off Test)** on page 5-2).
- Start the acquisition as single run.
- At each detection of a peak de- or increase the reference gas pressure at ConFlo III. Expected data after three or four measurements:

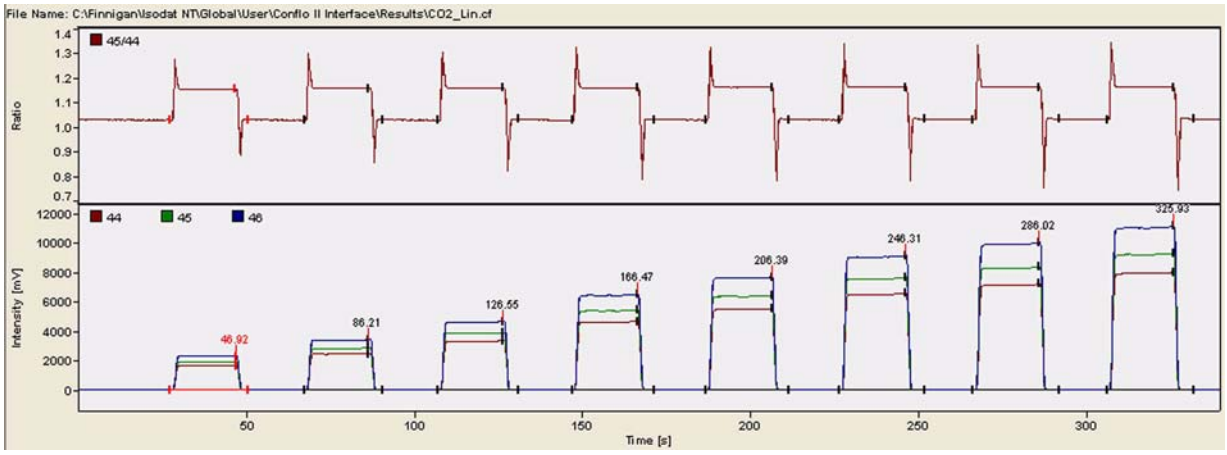


Figure 5-10. Linearity Test of Carbon - Chromatogram

CO2 | Error | Extended | Sequence Line

Peak Nr.	Start [s]	Rt [s]	Width [s]	Ampl. 44 [mV]	Ampl. 45 [mV]	BGD 44 [mV]	BGD 45 [mV]	Area All [Vs]	d 13C/12C [per mil] vs. VPDB	AT% 13C/12C [%]	d 18O/16O [per mil] vs. VSMOW
1	27.5	46.9	23.0	1643	1910	12.3	15.4	31.347	-0.020	1.105637	0.126
2	67.4	86.2	23.4	2450	2847	12.2	15.3	46.763	-0.002	1.105656	0.132
3*	107.1	126.5	23.8	3343	3886	12.2	15.3	63.796	0.000	1.105659	0.000
4	147.0	166.5	24.5	4660	5417	12.3	15.3	88.951	-0.035	1.105621	-0.123
5	186.7	206.4	24.9	5514	6410	12.4	15.5	105.362	-0.083	1.105567	-0.213
6	226.7	246.3	25.3	6583	7650	12.5	15.6	125.489	-0.123	1.105525	-0.349
7	266.4	286.0	25.7	7203	8373	12.7	15.9	137.413	-0.181	1.105460	-0.422
8	306.3	325.9	25.7	8011	9310	12.9	16.0	153.033	-0.223	1.105415	-0.580

Figure 5-11. Linearity Test of Carbon - Result Grid

> **Note.** The linear regression of the $\delta^{13}C/^{12}C$ values vs. the working standard should be less than ***0.06 ‰/V***.

Chapter 6
Dual Measurement

6.1 Introduction

It is possible to perform dual measurements of nitrogen and carbon from a single sample with the system (ConFlo II/III and IRMS) in less than eight minutes offering fast sample throughput and high productivity. The technique is suitable for both solid and liquid samples.

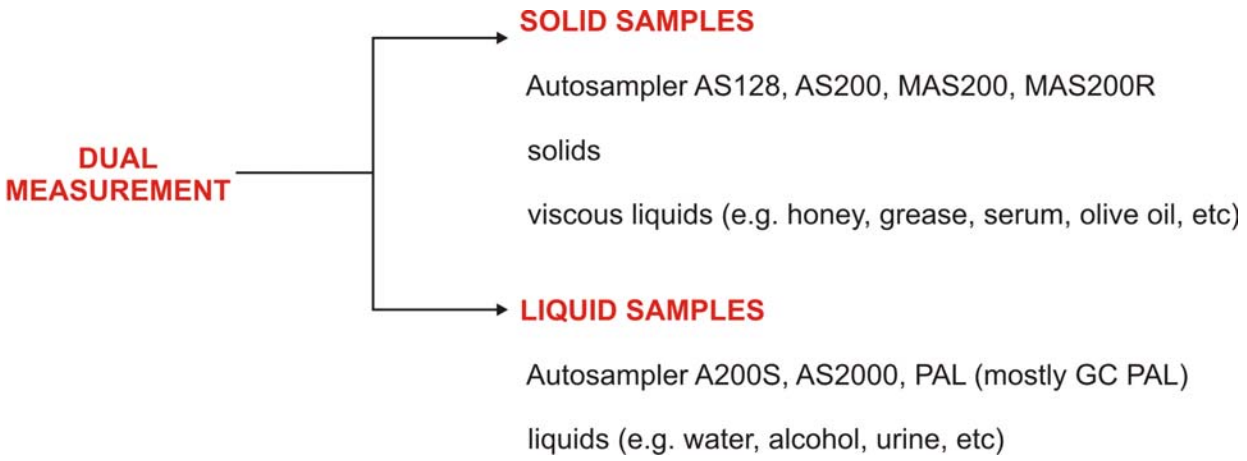


Figure 6-1. Schematic Diagram of Running Solid and Liquid Samples

As shown in Figure 6-1, different autosamplers are used to measure different sample types. Concerning the installation of the autosamplers refer to the manuals of the corresponding manufacturers.

6.2 Procedure

For the analysis of two isotopic species (nitrogen and carbon) from a single sample, a method, which comprises both gas configurations must be defined. The acquisition can be completed after less than eight minutes.

Due to the separation of the gases on a GC column, nitrogen appears first. As soon as the nitrogen peak (3) has been identified, Isodat 2.0 stops the nitrogen acquisition. The magnet "jumps" to the CO_2 configuration. A magnet jump means that the magnetic field is changed due to the entries from the mass calibration enabling the system to now collect masses 44, 45, 46 in the cups used for N_2 masses before.

If no nitrogen peak can be found, Isodat 2.0 waits a certain time, which has been set in the method, before automatically switching to CO_2 configuration.

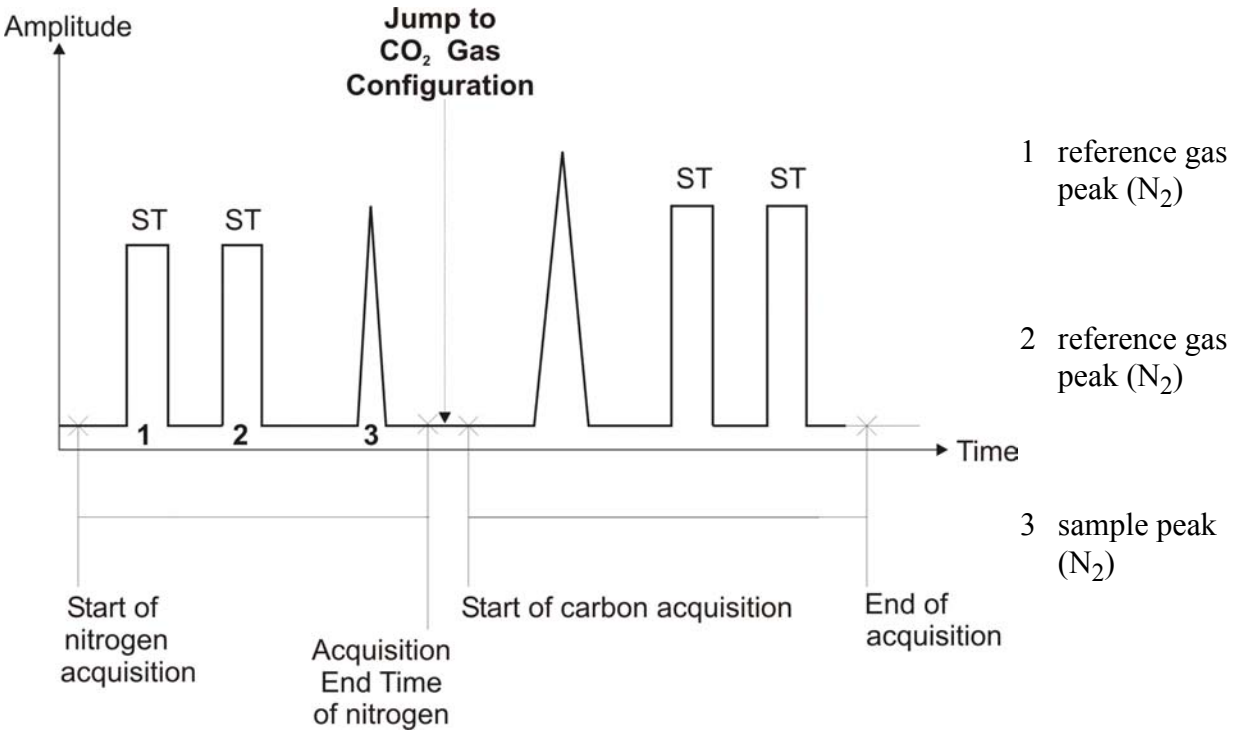


Figure 6-2. Dual Measurement - Schematic Chromatogram

6.3 How to Perform a Jump Calibration

To determine the isotope ratios of different elements during the same run, ***switching to another Gas Configuration*** is necessary.

In contrast to a single element measurement, in which the magnetic field runs the gamut from high to low and after that to the pre-calculated magnetic field, there is not sufficient time in Dual Measurement to perform this procedure for the next Gas Configuration.

For this reason, a ***Jump Calibration*** from the first Gas Configuration to the next Gas Configuration is necessary. It calibrates for a fast variation of the magnetic field.

After the Jump Calibration has been performed, the computer finds exactly the peak center even without performing any peak center procedure.

Temperature changes may influence the magnet performance on a daily basis. Therefore, a Jump Calibration should be performed daily.

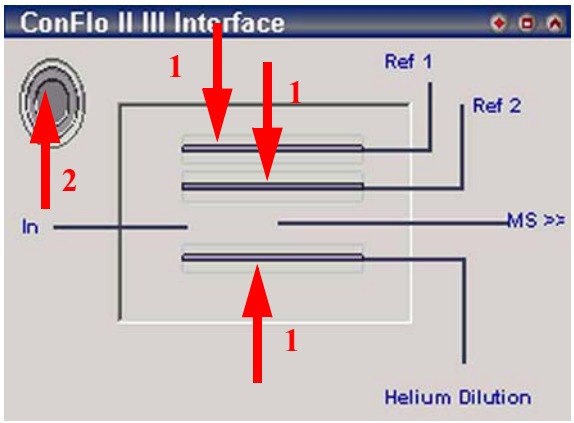
> **Note.** Both Gas Configurations used (i.e. **N2** and **CO2**) need to be configured prior to Jump Calibration.

> **Note.** A ConFlo configuration needs to be configured.

- Open ***Instrument Control***.

- Switch to ConFlo ***Configuration***.

  The following window must appear at the ***Accessories*** toolbar. If it does not appear, check, if it is not visible (refer to Figure 6-3).



The ***ConFlo II III Interface window*** allows:

reference gas inlet and/or dilution (**1**)

autosampler/Elemental Analyzer start signal (**2**)

Capillaries can be moved/activated by mouse clicks.

- Open ***Reference 1*** by a ***click*** on ***Ref 1*** capillary. If CO_2 is connected to ***Reference 2***, click ***Ref 2*** capillary.

  Thus, the blue capillary symbol elongates and CO_2 enters the system.

Figure 6-3. ConFlo II III Interface Window

Comment: If the ConFlo II III Interface Window is not Visible

- ***Right-click*** somewhere into the ***Accessories*** toolbar.
- Click on the ***Administrative Panels...*** button.

The ***Accessories*** window allows to mark dialogs (i.e. windows) by ✓. If you mark a dialog, the respective window will be shown at the ***Accessories*** toolbar.

- Mark ***ConFlo II III Interface***.

- Confirm by ***OK***.

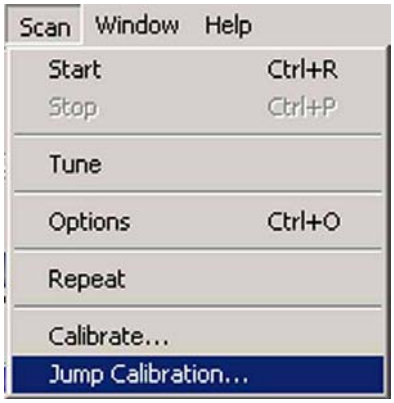
  Thus, the ConFlo II III Interface window will be shown at the ***Accessories*** toolbar.

Figure 6-4. Visible Windows at the Accessories Toolbar

Comment: If only the Title Bar of the ConFlo II III Interface Window is Visible

- Click on the ***Unshrink*** button at the right corner of the Title bar.
  Thus, ConFlo II III Interface window will appear.

Jump Calibration - Before Start



- Press ***Scan*** > ***Jump Calibration...*** or

- Alternatively, press the ***Jump*** button on the toolbar next to the Instrument Control window.

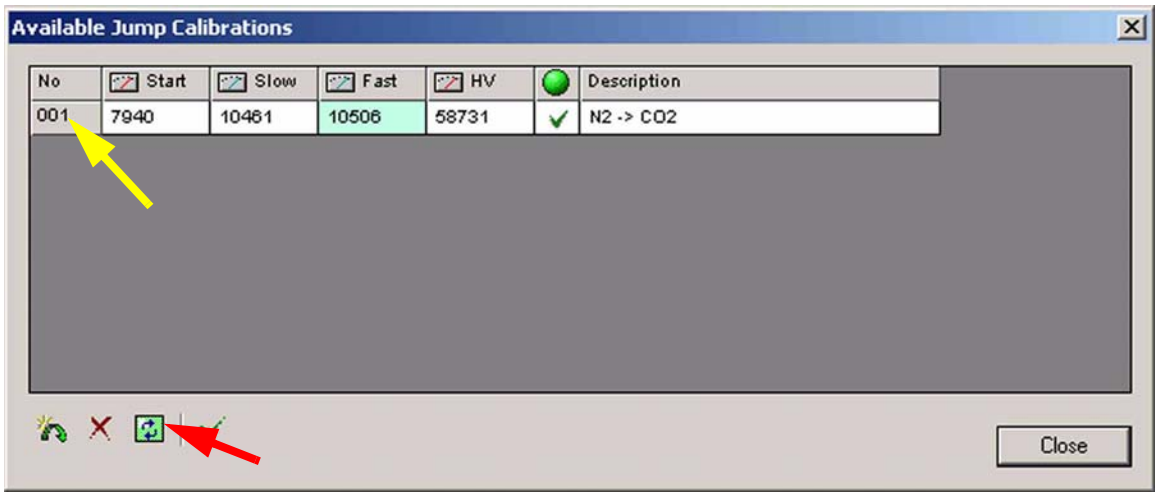


Figure 6-5. Available Jump Calibrations

- In the list of available Jump Calibrations mark the one for $N_2 \rightarrow CO_2$ by ***clicking its No.***, e.g. 001.
- ***Slow*** compensate hysteresis by Max/Min settings of the magnet.
- ***Fast*** magnet setting of Jump Calibration
- ***HV*** high voltage setting of Jump Calibration

> **Note.** Before pressing the **Recalibrate** button, make sure that CO_2 reference gas is activated, i.e. that CO_2 enters the source.

- Press the ***Recalibrate*** button.

Comment: If no Jump Calibration is Available

If ***no*** Jump Calibration is available in the list, ***create a new one*** as follows:

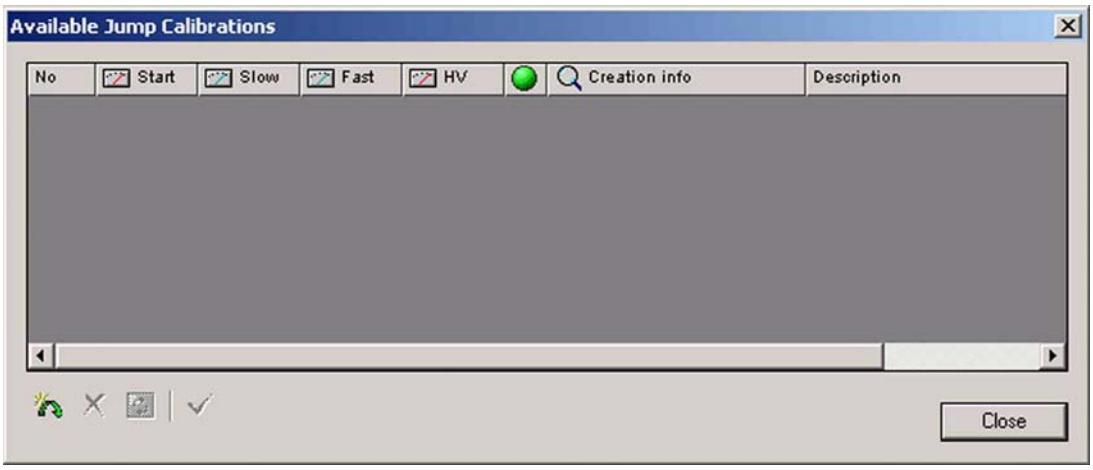


Figure 6-6. No Jump Calibration Available

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

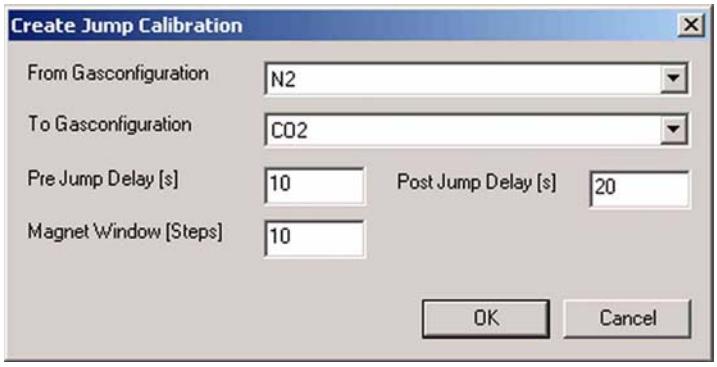
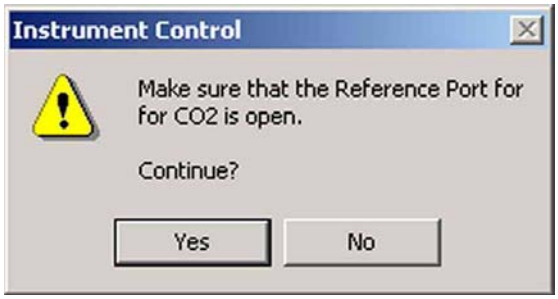
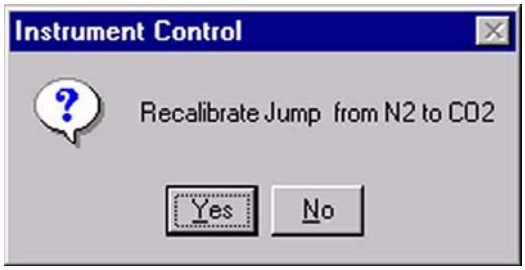


Figure 6-7. Define the Parameters of the new Jump Calibration

- At ***From Gas Configuration*** select ***N2***.
- At ***To Gas Configuration*** select ***CO2***.
- At ***Pre Jump Delay [s]*** accept the default value.
- At ***Post Jump Delay [s]*** accept the default value.
- At ***Magnet Window [Steps]*** accept the default value.
- Confirm by ***OK***.



- Confirm by ***Yes***, if you have already opened the Reference Port for CO_2 as shown in Figure 6-3.



- Confirm by ***Yes*** to start Jump Calibration.

Jump Calibration - Procedure

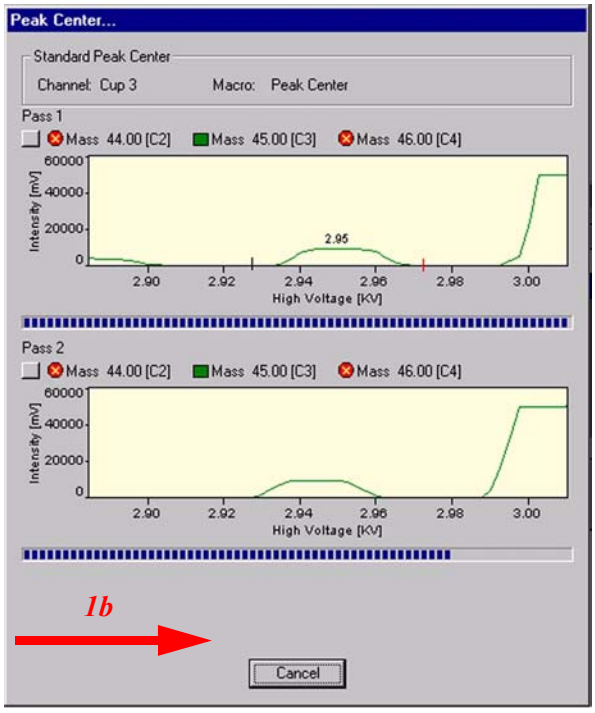
1a Jump to ***CO2*** (along hysteresis curve).

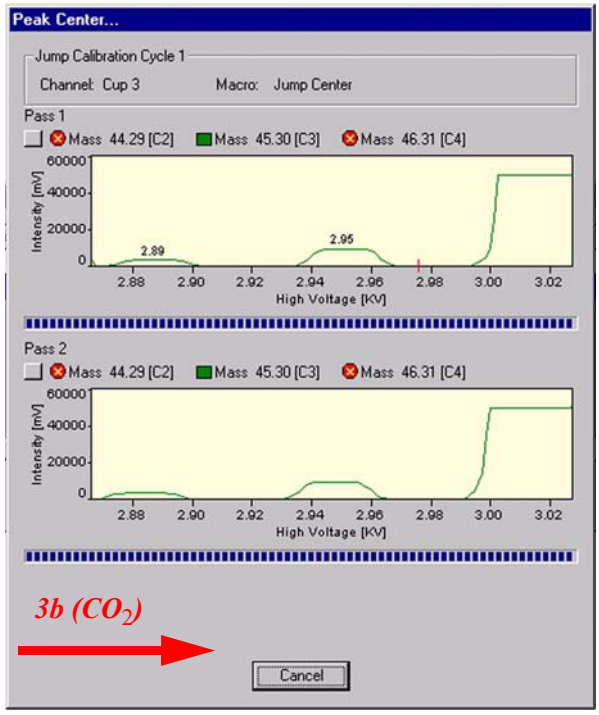
1b Perform a peak center for ***CO2*** to get the signal height.

2 Jump to ***N2*** (along hysteresis curve; ***N2*** is origin).

3a Jump to ***CO2*** (***not*** along hysteresis curve).

3b Perform a peak center for ***CO2*** to catch the peak.





Chapter 7
Sulfur Measurement

7.1 Introduction

In comparison to isotope ratio measurements of nitrogen (^{15}N) and carbon (^{13}C) in organic and inorganic matter, the analysis of sulfur (^{34}S) has always been difficult. Analyzing biological sulfur makes experimental difficulties caused by the low abundance of sulfur in organisms (e.g. 0.2 wt % [mg/mg] in plants), as well as by the fact that sulfur is present as a mixture of organic and inorganic compounds. Difficulties in using the Elemental Analyzer arise due to a large amount of carbon in the same samples, which quickly exceeds the capacity of combustion reactors. This causes incomplete combustion.

Thermo Electron (Bremen) has developed a technique for precise, accurate and fast sulfur measurement, which puts it on a par with carbon and nitrogen in terms of ease of use and sample size.

Due to the high natural abundance of the heavier isotope ^{34}S, less amplification is required and may become necessary for all Delta IRMS before Delta^{plus} XP (since 2002). It is recommended to use a smaller resistor ($1 * 10^{10}\ \Omega$) on the cup for mass 66 (usually $3 * 10^{10}\ \Omega$).

7.2 Procedure

Sulfur measurements are performed using a specially equipped Elemental Analyzer. Combustion and reduction are carried out in a single reactor filled with tungsten oxide (WO_3) and copper (Cu) as reducing agent. Alternatively, CuO can be used instead of WO_3.

The technique used for sulfur determination is based on the quantitative "Dynamic Flash Combustion" method. The samples - sometimes together with V_2O_5 - are wrapped in tin capsules and placed into the autosampler. Then they are continuously purged with helium (or oxygen using NA 2500 or EA 1110) to remove any traces of water and nitrogen. When a sample is dropped into the reactor, the helium stream is temporarily enriched with pure oxygen. The sample and its container melt as the tin promotes a violent reaction (flash combustion). Under these favorable conditions, even thermally resistant substances are completely oxidized.

In the reactor, e.g. $BaSO_4$ is thermally decomposed within a tin capsule. The following reactions can then take place (see Bailey, S.A. and Smith, J.W., 1972):

$$BaSO_4 \rightarrow BaO + SO_2 + \frac{1}{2}O_2$$

$$BaSO_4 \rightarrow BaO + SO_3$$

$$SO_3 \rightarrow SO_2 + \frac{1}{2}O_2$$

Note. Bailey, S.A. and Smith, J.W. (1972): An improved method for the preparation of sulfur dioxide from barium sulfate for isotope ratio studies. Anal. Chem. **44**, 1542-1543.

Although the process does not require oxygen, better combustion has been experienced when O_2 is injected and vanadium pentoxide (V_2O_5) is added to the sample. If either the O_2 pressure is low or a bad catalyst is selected or the reactor has too much ash, combustion will proceed slowly. A slow stream of SO_2 through the system causes adsorption at the tubing wall.

Note. It is important that enough reduced copper is present in the combustion tube. If this is not assured, SO_3 will only be reduced partially and isotope fractionation will occur.

"Light" isotopes ($^{32}SO_3$) are reduced more easily than "heavy" isotopes ($^{34}SO_3$). Therefore, SO_2 gas is depleted in ^{34}S compared to the original sample, and the δ value becomes more negative.

7.3 Sulfur Measurement Kits

To perform a sulfur measurement you can get two ***sulfur measurement kits***, provided by Thermo Electron (Bremen):

- Sulfur Measurement Kit for ConFlo III
- Sulfur Measurement Kit for Flash Elemental Analyzer

Sulfur Measurement Kit for ConFlo III

It consists of the following parts:

Table 7-1. Parts of Sulfur Measurement Kit for ConFlo III (Part No. 115 7100)

Quantity	Description	Part No.
1	attachment for exhaust tube	112 1390
1	self-adhesive heating foil for ConFlo III	114 1180
1	power supply for self-adhesive heating foil for ConFlo III	204 8580

Sulfur Measurement Kit for Flash Elemental Analyzer

It consists of the following parts:

Table 7-2. Parts of Sulfur Measurement Kit for Flash Elemental Analyzer (Part No. 115 7110)

Quantity	Description	Part No.
1	sulfanilamide	106 9140
1	Teflon GC column	114 1170
5	Al olive (2 mm)	114 1210
5	nut	112 1370
3.5 m	Teflon tube	114 1220*
2	combustion reactor (packed, “ready for use”)	111 8121
3	union	114 1230
0.05 m	capillary tube (1/16” * 0.8 mm)	060 5470

*For additional part numbers (e.g. of nuts) see **Installing the Teflon Tubing** on page 7-6 and especially Figure 7-2.

Warning. When working with sulfur dioxide (SO_2), good ventilation is essential. Otherwise, the gas can be hazardous to your health.

Warning. To ensure operational safety, a CO, SO_2 and H_2 detector with an alarm must be installed!

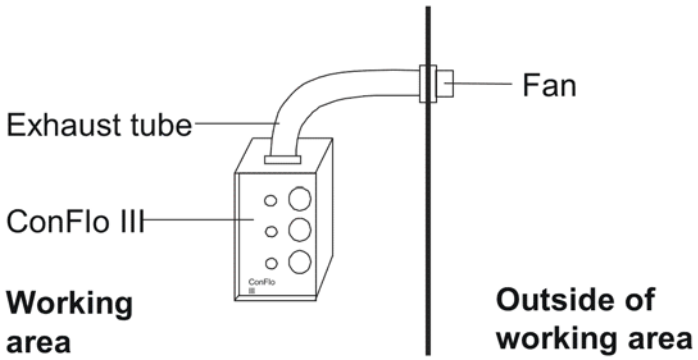


Figure 7-1. ConFlo III Interface with Exhaust Tube and Fan

The exhaust tube should be installed on top of the ConFlo III interface as shown in Figure 7-1 to remove the toxic sulfur dioxide (SO_2) from inside of ConFlo III out of your working area.

7.4 Preparing the System for a Sulfur Measurement

1. Install the exhaust tube on ConFlo III as shown in Figure 7-1.
2. Install the self-adhesive heating foil as shown in Figure 7-3.
3. Install the properly packed reactor within the Elemental Analyzer.
4. Replace all stainless steel tubing with Teflon tubing as shown in Figure 7-2.
5. Install the SO_2 GC column.

Installing the Teflon Tubing

To perform a sulfur measurement, a part of the stainless steel tubing needs to be replaced by Teflon tubing. The scheme below provides an overview:

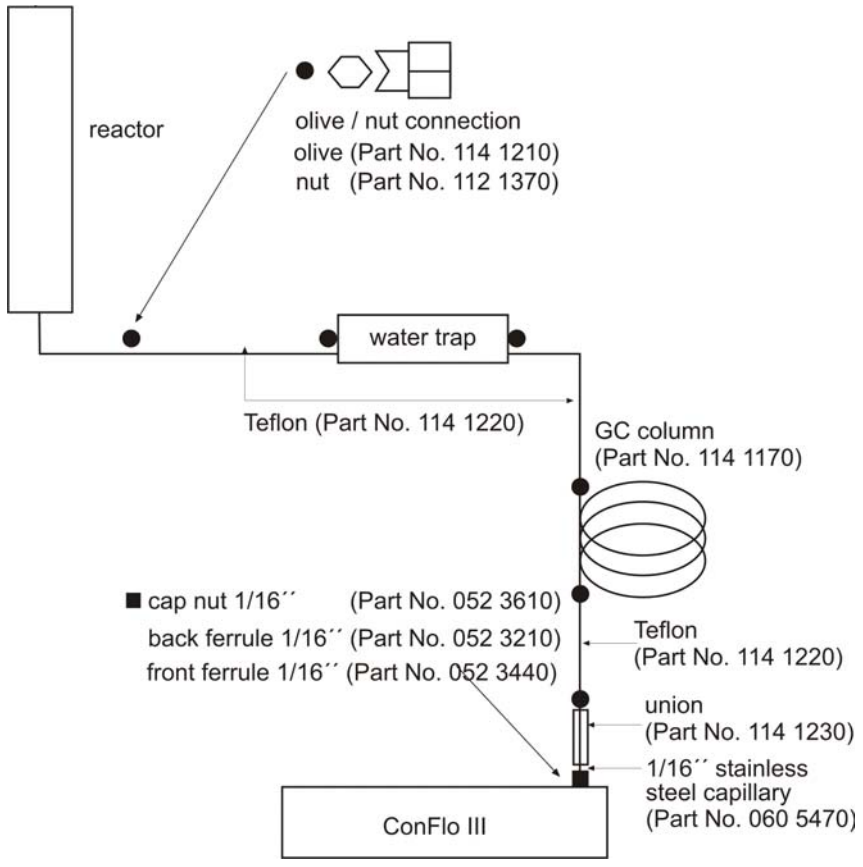


Figure 7-2. Installing the Teflon Tubing

Installing the Self-Adhesive Heating Foil

Note. SO_2 is liquid at higher pressures. Therefore, the manometer should be heated to avoid condensation of SO_2. The temperature must be between 60 °C and 70 °C. It may vary with ventilation.

1. Remove the backing paper from the self-adhesive heating foil.
2. Paste the heating foil in the middle of the manometer's rear side (of Ref 2) as shown in Figure 7-3.

Note. Take care that the wires of the heating foil point downwards when pasting it on the manometer's rear side.

3. Simply insert the wires into the plug socket where they fix themselves.

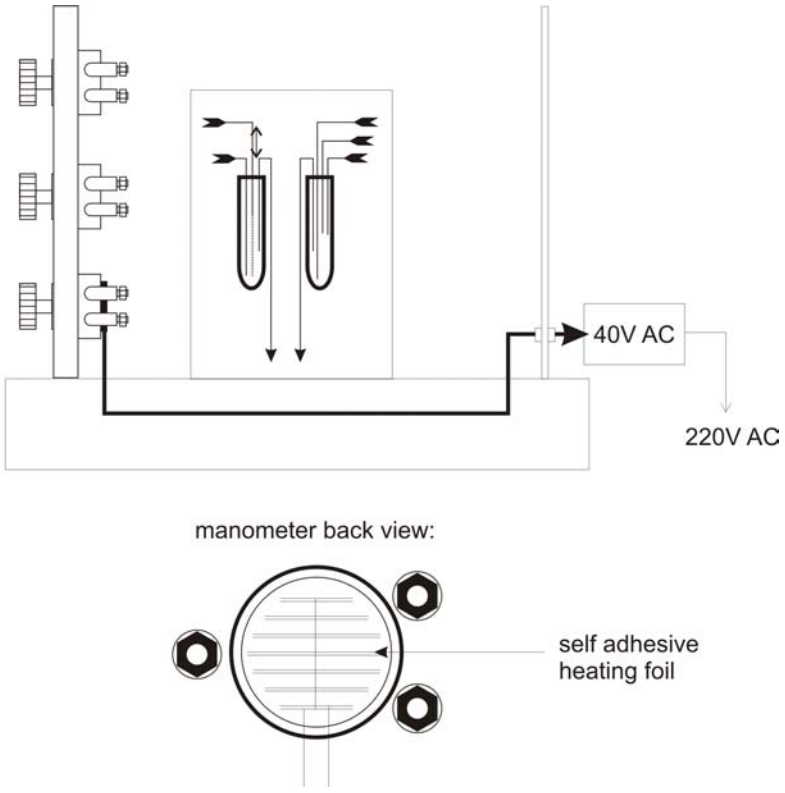
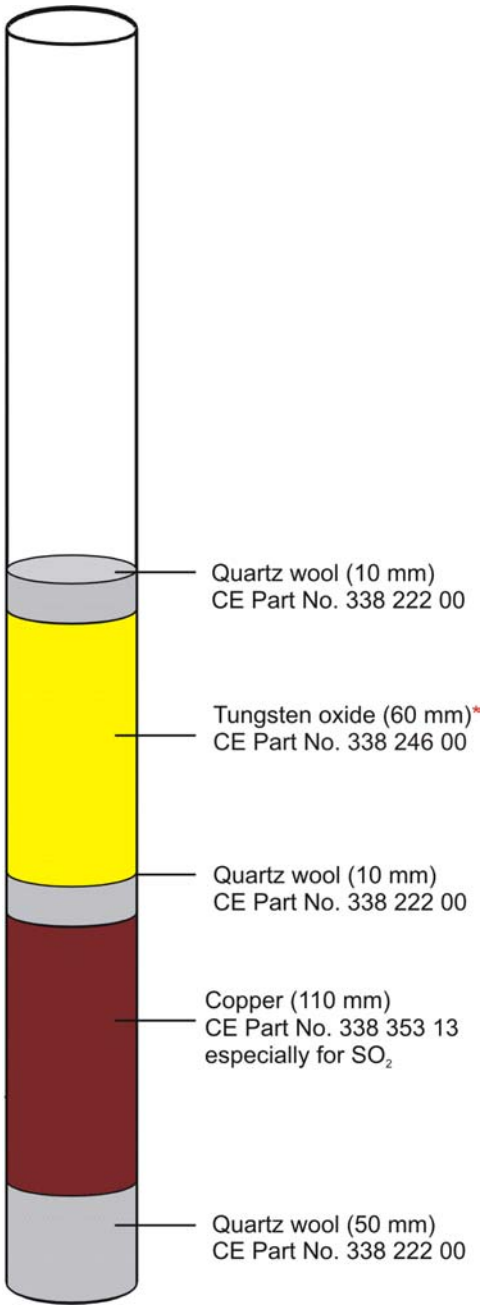


Figure 7-3. Installing the Self-Adhesive Heating Foil

Reactor Filling



We recommend to use a Thermo Electron "ready for use" reactor (Part No. 111 8121). The reactor is 470 mm long. If the reactor must be packed by the user, the packing should be as shown left.

About 150 to 200 analyses can be performed using the reactor type described above. With the exception of tungsten oxide, no parts of the reactor can be used a second time.

Figure 7-4. Packing of the Reactor

***Option.** Instead of tungsten oxide also 60 mm of copper oxide (CE Part No. 338 217 10) can be used for the packing of the reactor.

7.5 Before Starting a Sulfur Measurement

We assume that the user already has working experience with the isotope ratio mass spectrometer. Before starting a sulfur measurement make sure that:

1. The Elemental Analyzer is set up, that is:
   - The SO_2 reactor is packed and installed.
   - The water trap is installed.
2. The special SO_2 stainless steel (or Teflon) GC column is installed.
3. The connection between reactor, water trap, GC column and ConFlo III is made up of Teflon tubing.
4. The gases needed (He, O_2) are available and have been connected to the corresponding positions.
5. Leak-check all connections outside of ConFlo III by brushing all fittings carefully with soapsuds.
6. The exhaust tube is connected to the ConFlo III and ventilation is working.
7. SO_2 is connected to Ref 2 of ConFlo III.
8. The self-adhesive heating foil is installed / switched on.
9. Compressed air is connected to ConFlo III and Elemental Analyzer.
10. ConFlo III configuration is installed and has already been in use for e.g. nitrogen or carbon analysis.
11. The IRMS has been calibrated for SO_2 measurements.
12. Switch the Elemental Analyzer on.
13. Adjust the gas pressure of Elemental Analyzer and ConFlo III Take the following settings as a guideline.

For EA 1108, EA 1110 and NA 2500

Table 7-3. Settings for EA 1108, EA 1110 and EA 2500

He	Elemental Analyzer	100 kPa
O_2	Elemental Analyzer	150 kPa
Purge with	O_2	40 ml/min
Purge with	He	110 ml/min (depending on the autosampler)
He	ConFlo III	1 bar
Ref. 2	ConFlo III	1 bar

For Flash Elemental Analyzer

Table 7-4. Settings for Flash Elemental Analyzer in Eager Software

Carrier	300 ml/min
Oxygen	175 ml/min
Reference	110 ml/min (depending on autosampler)
Cycle run time	60 s
Sampling delay	23 s
Oxygen Injection End	3 s

14. Switch the ion source ***OFF*** and inject the He-dilution capillary.
15. Slowly open the needle valve. Make sure that the needle valve is heated.
16. Make sure that the ion source heater and the change over valve heater are operating, if available.
17. Switch the ion source ***ON***.
18. After approximately 30 min. the expected background at the respective reactor and column temperature should be as follows:

Stainless Steel GC Column

Table 7-5. Expected Background Using a Stainless Steel GC Column

reactor temperature	column temperature	mass	28	29	Ar	H_2O	64	66
		cup	28	29	29	29	64	*66
		range	mV	mV	mV	V	mV	mV
30 °C	30 °C		50	60	180	1.5		
400 °C	100 °C		110	140	350	1.8		
1020 °C	100 °C		70	65	220	1.2	< 5	< 10

Note. Background values may vary depending on sensitivity and focus settings. They are given here as a general guideline.

Teflon GC Column

Table 7-6. Expected Background Using a Teflon GC Column

reactor temperature	column temperature	mass	28	29	Ar	H_2O	64	66
		cup	28	29	29	29	64	*66
		range	mV	mV	V	V	mV	mV
30 °C	30 °C		295	223	0.93	3.9		
400 °C	100 °C		422	337	1.30	5.6		
1020° C	100 °C		330	243	0.99	2.1	<5	<10

(* $R_i=10^{10}\ \Omega$)

Note. Background values may vary depending on sensitivity and focus settings. They are given here as a general guideline.

Note. Teflon tubes are not absolutely tight against atmosphere. Therefore, the backgrounds of argon and nitrogen are higher than those of carbon and nitrogen measurements. Nevertheless, take care of leaks.

Note. When using normal amplification (i.e. resistor of $3 * 10^{10}\ \Omega$ on cup for mass 66), the background values will be three times higher.

7.6 Create a Gas Configuration for a Sulfur Measurement

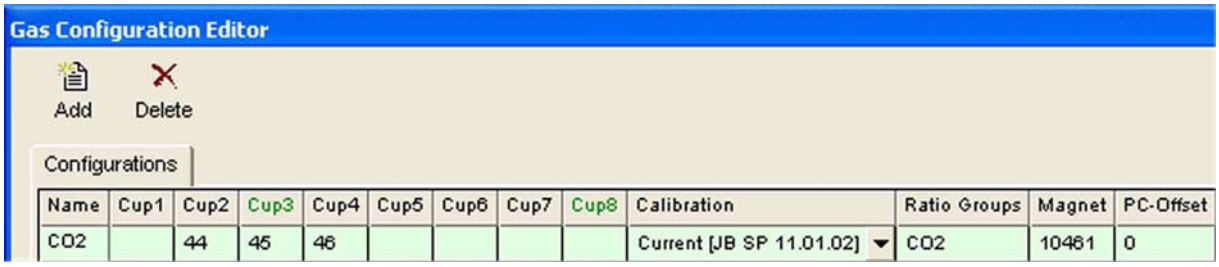
A Gas Configuration determines a combination of masses, which are collected in the cups, for evaluation of ratios and eventually δ values. The Gas Configuration is specific for the particular gas and is combined with a magnet field value taken from the mass calibration of your IRMS. The ratio groups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

Prior to defining this Gas Configuration ensure that the connected IRMS has the cups for the simultaneous detection of masses 64 and 66 and mass calibration for these cups has already been performed.

For a ^{34}S measurement, a Gas Configuration must be available for the masses 64 (i.e. $^{34}S^{16}O^{16}O$) and 66 (i.e. $^{32}S^{16}O^{16}O$).
Otherwise, it must be created as follows.

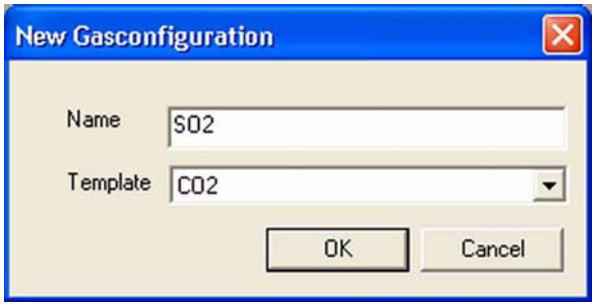
- Open the ***Acquisition*** module.

- Open the ***Gas Configuration Editor***.
  It is only available if no acquisition is running.



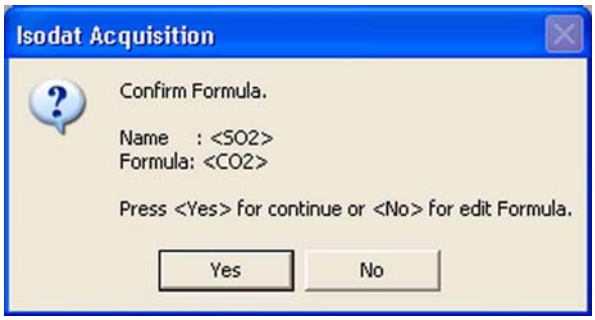
- Per default, the Gas Configuration ***CO2*** is being created as the first one.
- If the Gas Configuration ***SO2*** has already been created, it occurs in the list above.
- If the Gas Configuration ***SO2*** has not been created yet, it does not occur in the list above. Then, proceed as follows.

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



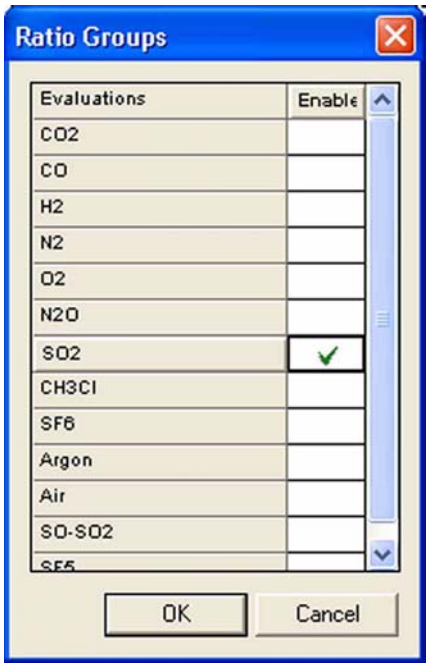
- Type ***SO2*** for the ***Name***.
- Select a Gas Configuration as ***Template*** (e.g. ***CO2***).

  In the pulldown menu, only the already existing Gas Configurations are displayed. When creating the first Gas Configuration, ***CO2*** is displayed.
- Confirm by ***OK***.



- Type ***No***.

  If you would type ***Yes***, this would automatically mark the template (i.e. ***CO2***) instead of ***SO2*** in the ***Ratio Groups*** window below.



- Mark ***SO2***.
- If Ratio Groups other than ***SO2*** are marked, unmark them all.
- Confirm by ***OK***.

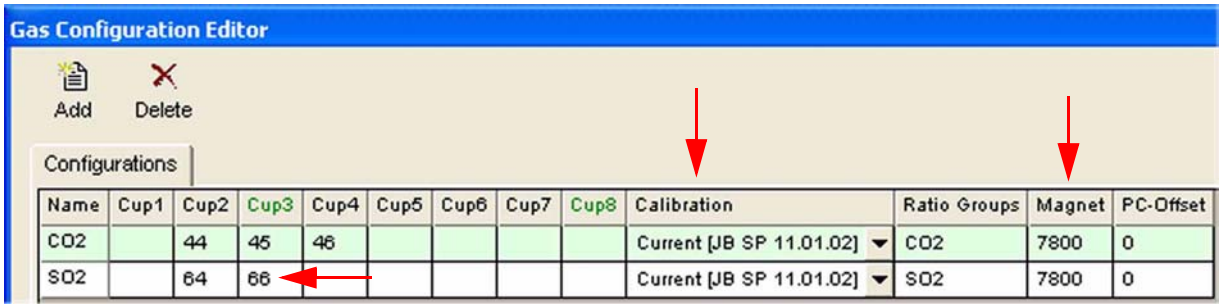
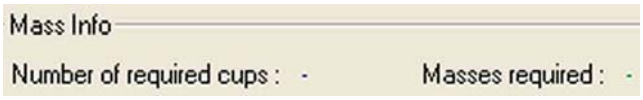


Figure 7-5. Creating a New Gas Configuration

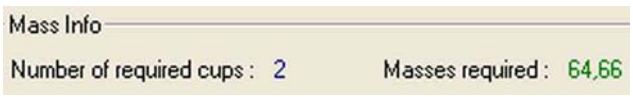
- The new Gas Configuration ***SO2*** appears in the list as a row of its own.
- In the ***Calibration*** column select your current calibration file.

> **Note.** The above figure shows a common cup configuration as used in most Delta mass spectrometers, i.e. universal triple collector.
>
> If you have a special cup configuration, the respective masses will be collected in other cups!

- Fill in the correct masses (64 and 66 replace e.g. 44, 45 and 46) to the appropriate cups specific for your IRMS.



When highlighting the specific gas configuration by a click on its row, the number of cups required for measurement is displayed together with the assigned masses.



- Select a ***Calibration***, which is valid for the selected cups.

Press the ***Save & Close*** button 

.

7.7 Zero Enrichment of Sulfur (Standard On/Off Test)

We assume that the user already has working experience with the ConFlo III Interface and IRMS. It is recommended to perform a simple check in order to test the analytical condition of ConFlo III and IRMS before measuring any samples.The most important checks to test the analytical condition are ***zero enrichment*** and ***linearity test***.

1. Only as a guideline use the following method from the File Browser's ***Examples*** folder (***SO2_zero.met***):

Instrument

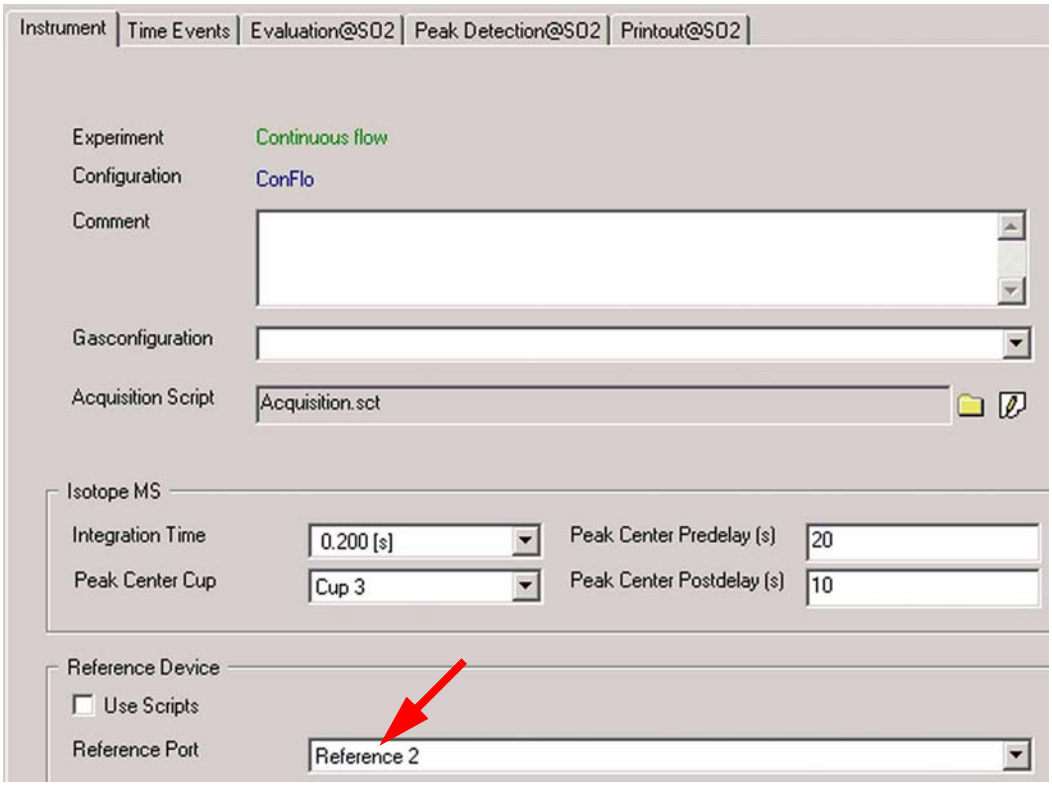
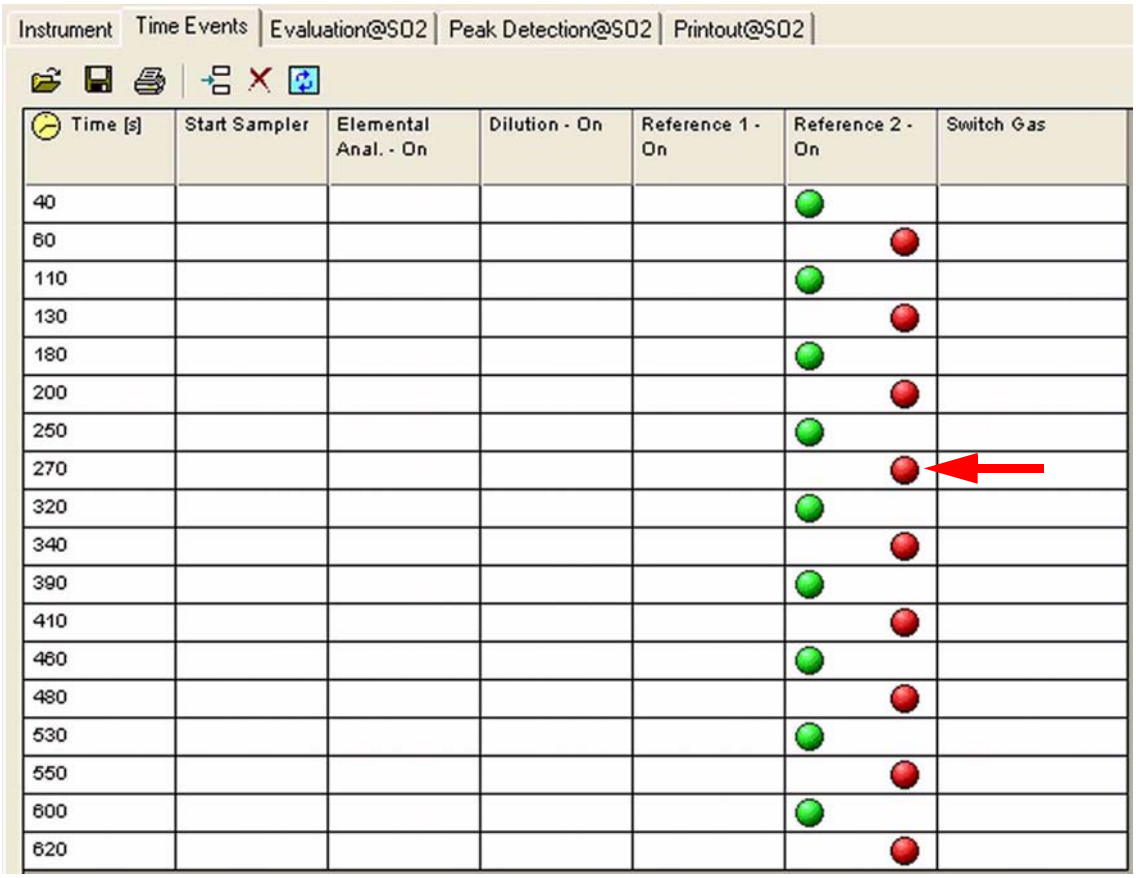


Figure 7-6. Zero Enrichment of Sulfur - Instrument Tab

- Select the reference port your reference gas is connected to at the ConFlo interface (e.g. Reference 2).
- Adapt the reference gas port to the respective column of the Time Events list: e.g. if you choose Reference 2, the on-off entries must occur in the Reference 2-On column of the Time Events list (see Figure 7-7).

Time Events



Time [s]	Start Sampler	Elemental Anal. - On	Dilution - On	Reference 1 - On	Reference 2 - On	Switch Gas
40						
60						
110						
130						
180						
200						
250						
270						
320						
340						
390						
410						
460						
480						
530						
550						
600						
620						

Figure 7-7. Zero Enrichment of Sulfur - Time Events Tab

Figure 7-8. Zero Enrichment of Sulfur - Acquisition Start and Acquisition End Time

- Recognize the nine on-off pulses shown above in the expected chromatogram (Figure 7-12).
- Note that the off-time of the e.g. forth reference gas pulse is 270 s (Figure 7-9).

Evaluation

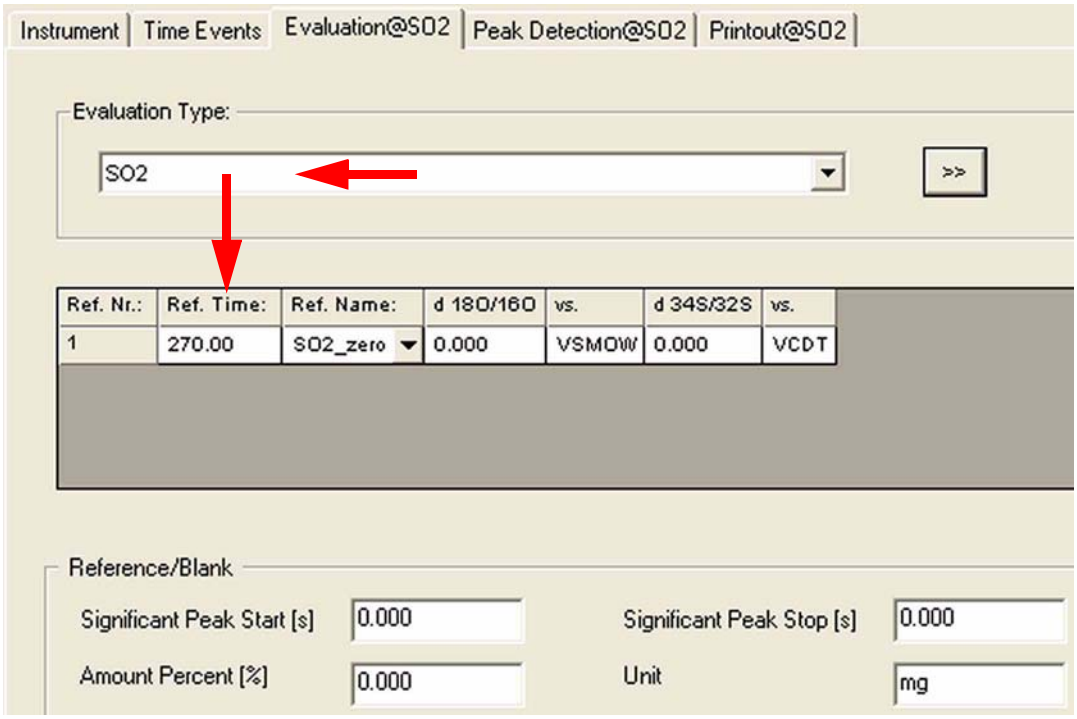


Figure 7-9. Zero Enrichment of Sulfur - Evaluation Tab

> **Note.** At **Ref. Time**, the **off**-value of an arbitrary reference gas peak according to the Time Events list must be typed (e.g. 270 s as the **off**-value of the forth reference gas peak, see Figure 7-7).

Peak Detection

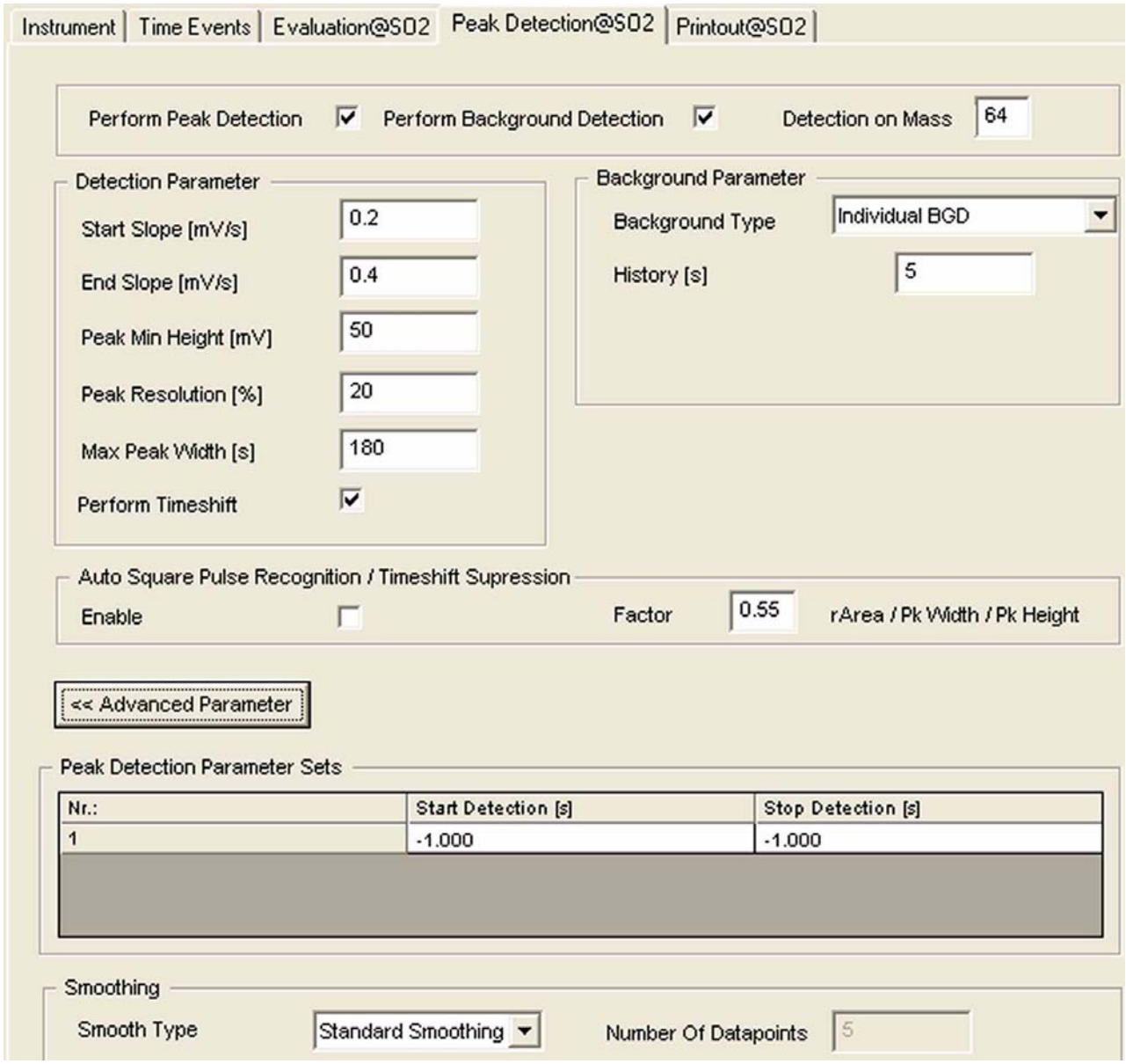


Figure 7-10. Zero Enrichment of Sulfur - Peak Detection Tab

> **Note.** A value of -1 denotes unlimited.

Printout

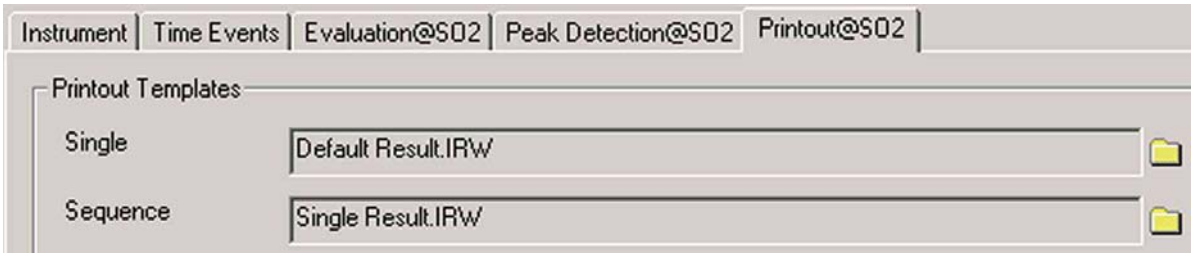


Figure 7-11. Zero Enrichment of Sulfur - Printout Tab

2. At the corresponding pressure regulator of ConFlo III, set the ion intensity of mass 64 (i.e. $^{32}S^{16}O^{16}O$) to approximately 3 V.
3. Create a new Sequence.
4. Press the ***Start*** button and confirm by ***OK***.
5. Expected data after three or four measurements:

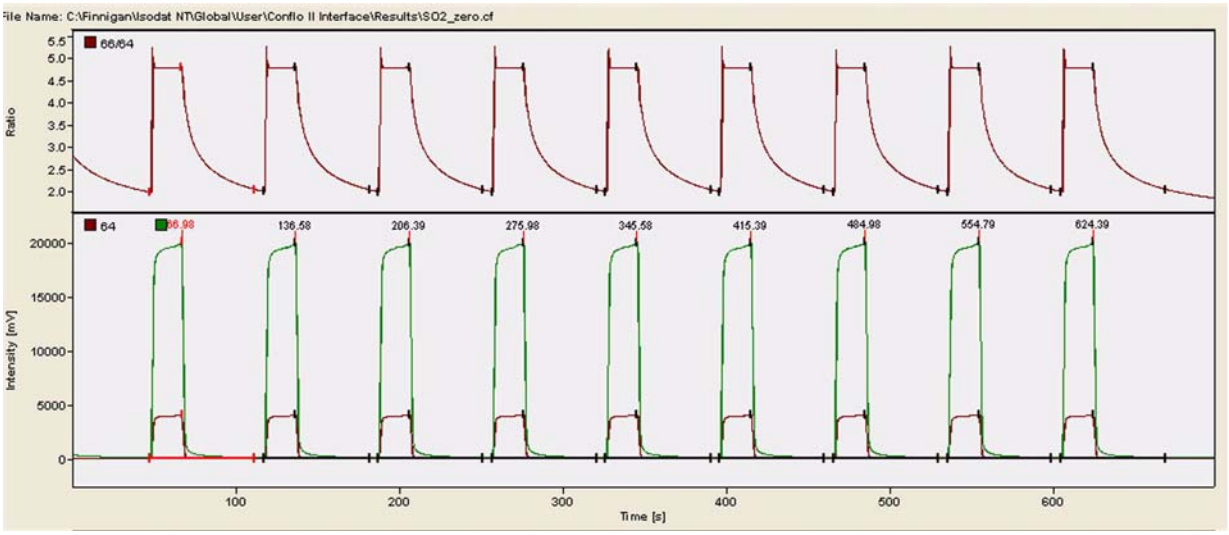


Figure 7-12. Zero Enrichment of Sulfur - Chromatogram

Recognize the nine peaks shown above as the nine on-off pulses in the Time Events list (Figure 7-7).

SO2 | Error | Extended | Sequence Line

Peak Nr.	Start [s]	Rt [s]	Width [s]	Ampl. 64 [mV]	Ampl. 66 [mV]	BGD 64 [mV]	BGD 66 [mV]	Area All [Vs]	Amt% [%]	d 66SO2/64SO2 [per mil] vs. SO2_zero	d 34S/32S [per mil] vs. VCDT
1	47.8	67.0	63.7	4085	19953	33.5	163.9	79.345	-	0.021	0.022
2*	117.4	136.6	64.4	4061	19838	34.1	166.8	79.141	-	0.000	0.000
3	187.2	206.4	64.0	4070	19878	34.0	166.3	79.308	-	-0.040	-0.044
4	256.8	276.0	64.4	4072	19887	33.9	165.8	79.329	-	-0.042	-0.046
5	326.4	345.6	64.2	4075	19904	33.9	165.9	79.465	-	-0.048	-0.052
6	396.2	415.4	63.7	4068	19869	33.8	165.8	79.164	-	-0.076	-0.083
7	465.8	485.0	63.7	4094	19995	33.8	165.8	79.399	-	-0.078	-0.085
8	535.6	554.8	63.7	4091	19981	33.8	165.9	79.386	-	-0.105	-0.114
9	605.2	624.4	64.0	4083	19944	33.9	166.2	79.401	-	-0.117	-0.128

Figure 7-13. Zero Enrichment of Sulfur - Result Grid

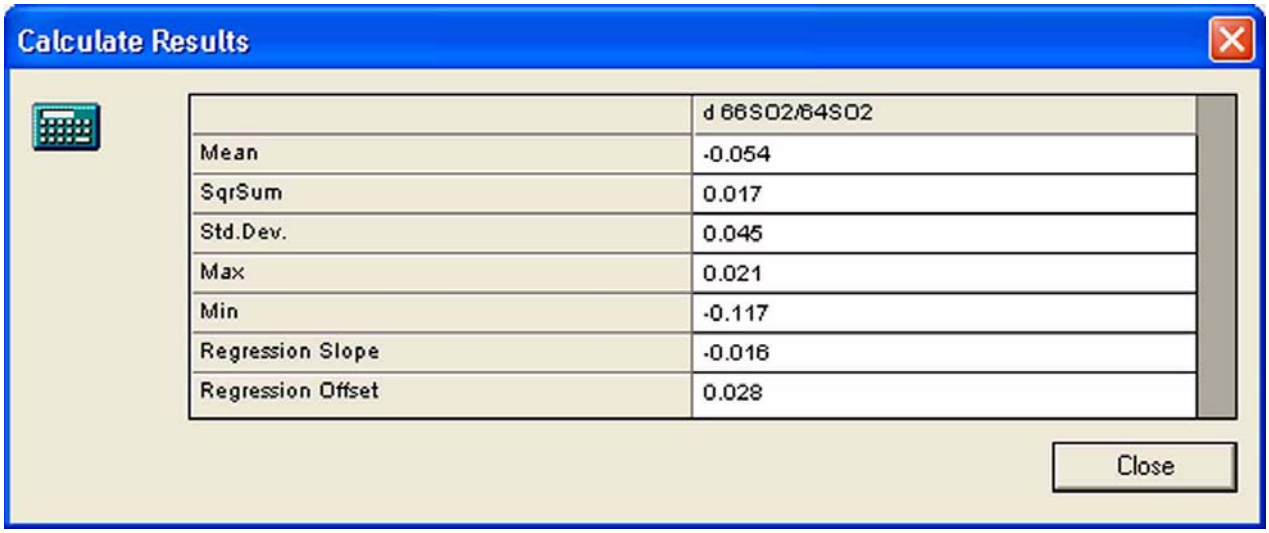
Note. The standard deviation of $\delta\ ^{66}SO_2/^{64}SO_2$ should be **0.05 ‰** or less.

To obtain the standard deviation of all nine peaks perform as follows:

d 66SO2/64SO2
0.021
0.000
-0.040
-0.042
-0.048
-0.076
-0.078
-0.105
-0.117

- Click on the column header of the ***d 66SO2/64SO2 [per mil] vs. SO2_zero*** column.
  It will be highlighted.

- Right-click on the column header.
- Choose ***Calculate***.



7.8 Linearity Test of Sulfur

- Use the same method as defined for ***zero enrichment***, e.g. ***SO2_zero.met***).
  See **Zero Enrichment of Sulfur (Standard On/Off Test)** on page 7-15.
- Start the acquisition as a single run.
- At each detection of a peak de- or increase the reference gas pressure at ConFlo III.

  Expected data after three or four measurements:

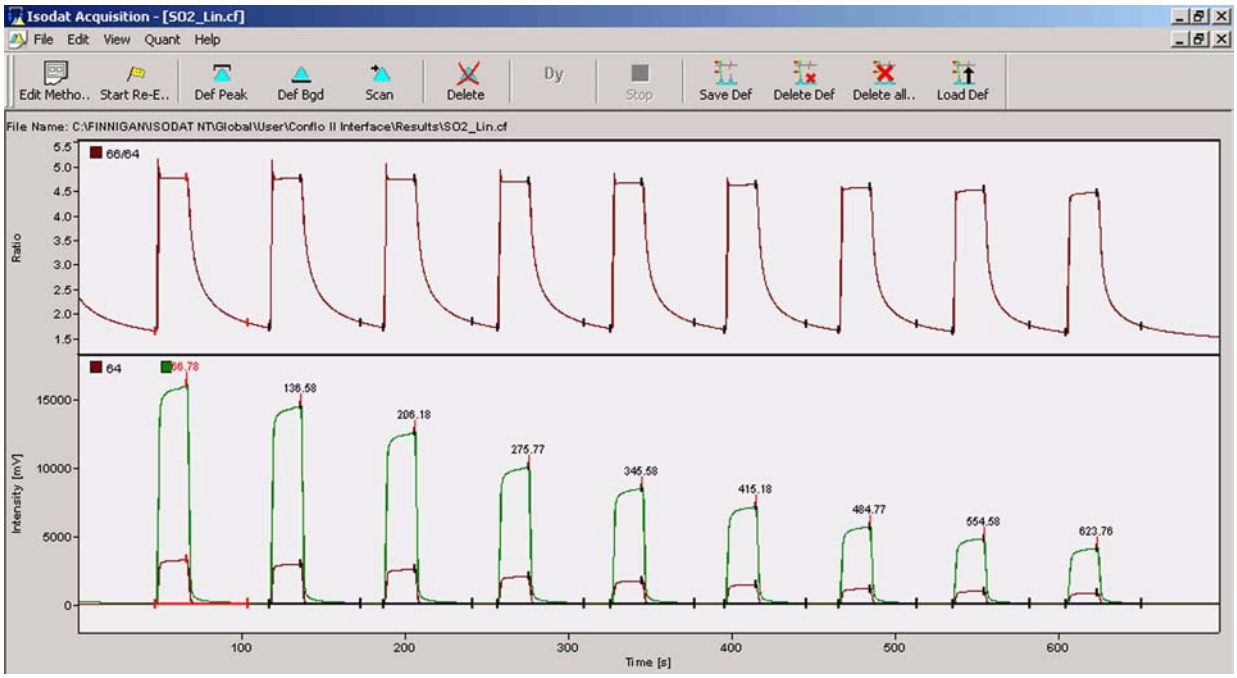
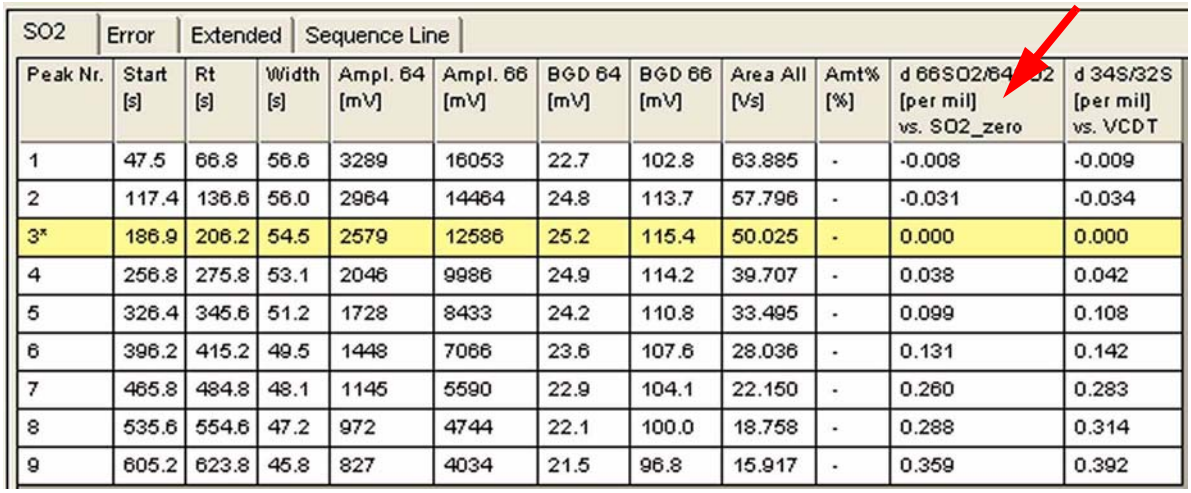


Figure 7-14. Linearity Test of Sulfur - Chromatogram



SO2 | Error | Extended | Sequence Line

Peak Nr.	Start [s]	Rt [s]	Width [s]	Ampl. 64 [mV]	Ampl. 66 [mV]	BGD 64 [mV]	BGD 66 [mV]	Area All [Vs]	Amt% [%]	d 66SO2/64SO2 [per mil] vs. SO2_zero	d 34S/32S [per mil] vs. VCDT
1	47.5	66.8	56.6	3289	16053	22.7	102.8	63.885	-	-0.008	-0.009
2	117.4	136.6	56.0	2964	14464	24.8	113.7	57.796	-	-0.031	-0.034
3*	186.9	206.2	54.5	2579	12586	25.2	115.4	50.025	-	0.000	0.000
4	256.8	275.8	53.1	2046	9986	24.9	114.2	39.707	-	0.038	0.042
5	326.4	345.6	51.2	1728	8433	24.2	110.8	33.495	-	0.099	0.108
6	396.2	415.2	49.5	1448	7066	23.6	107.6	28.036	-	0.131	0.142
7	465.8	484.8	48.1	1145	5590	22.9	104.1	22.150	-	0.260	0.283
8	535.6	554.6	47.2	972	4744	22.1	100.0	18.758	-	0.288	0.314
9	605.2	623.8	45.8	827	4034	21.5	96.8	15.917	-	0.359	0.392

Figure 7-15. Linearity Test of Sulfur - Result Grid

> **Note.** The linear regression of the $\delta\ ^{66}SO_2/^{64}SO_2$ values vs. the working standard should be less than ***0.06 ‰/V***.

Chapter 8
Technical Information

8.1 Mechanics

Note. This chapter is intended for use by trained Thermo Electron (Bremen) personnel only. Thermo Electron (Bremen) discourages use by and denies liability for the consequences of use by other than Thermo Electron (Bremen) personnel.

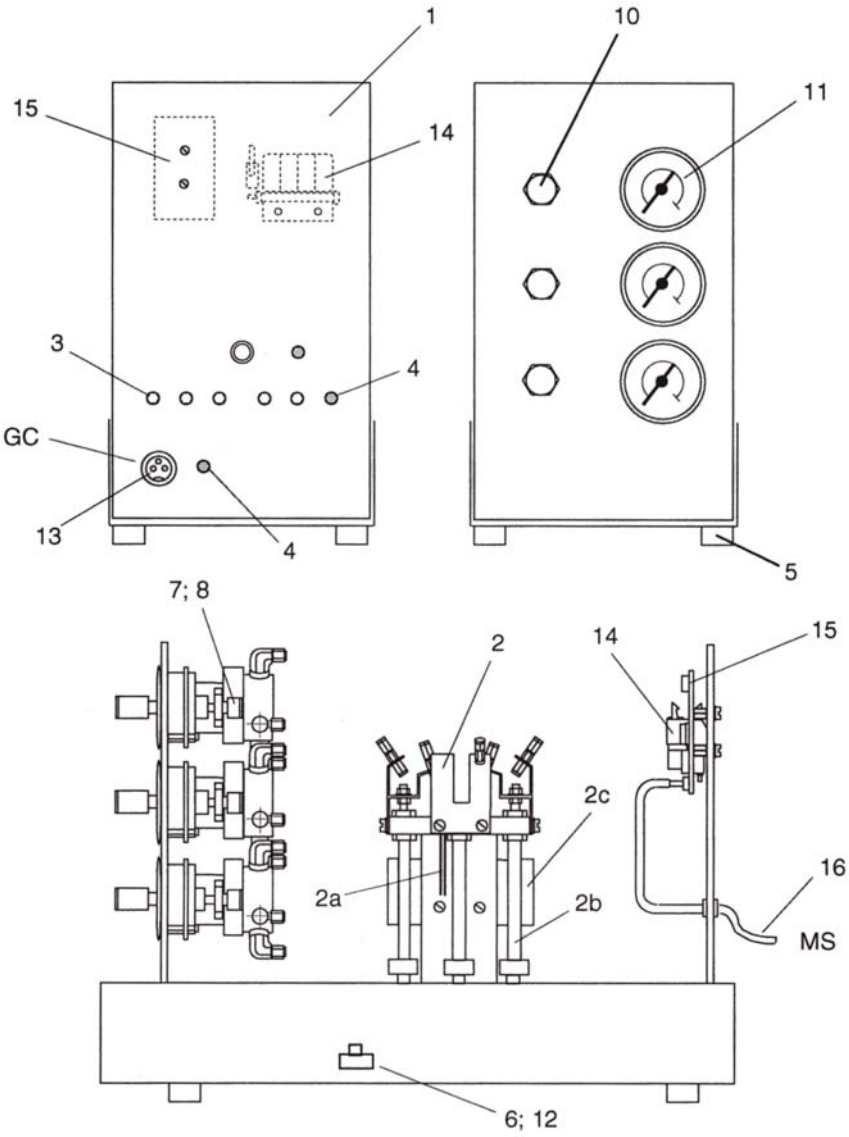


Figure 8-1. ConFlo III Interface (Part No. 113 7200)

Table 8-1. Parts of ConFlo III Interface

Pos. No.	Quantity	Designation	Part No.
1	1	housing	113 7190
2	1	combi inlet	113 2260
2a	2	glass tube with funnel	113 2270
2b	3	lifting cylinder, 8-25	067 4580
2c	1	valve, 5-2 port	113 2350
3	5	coupling	056 7350
4	2	coupling	067 4651
5	4	rubber plate, GM	050 0520
6	1	T-piece, M 5	052 2330
7	3	coupling, 1/8"	074 3360
8	3	ferrule, 1/8"	067 4790
9	1	installation kit ConFlo III	113 7250
10	3	pressure regulator	067 4880
11	3	manometer, 0 -2.5 bar	052 4391
12	2	gasket, 8 * 5	050 5260
13	1	flange type receptacle, BT 3263	032 3760
14	1	manifold card, 4 station, 10 PO	108 3241
15	1	EA start pcb	204 2350
16	1	connection cable	104 0351
17	2 m	silicon hose, 1.0 * 1.75 NF	101 5830

Pos. 9 and 17 are not shown in Figure 8-1.

8.2 Capillaries and Ferrules

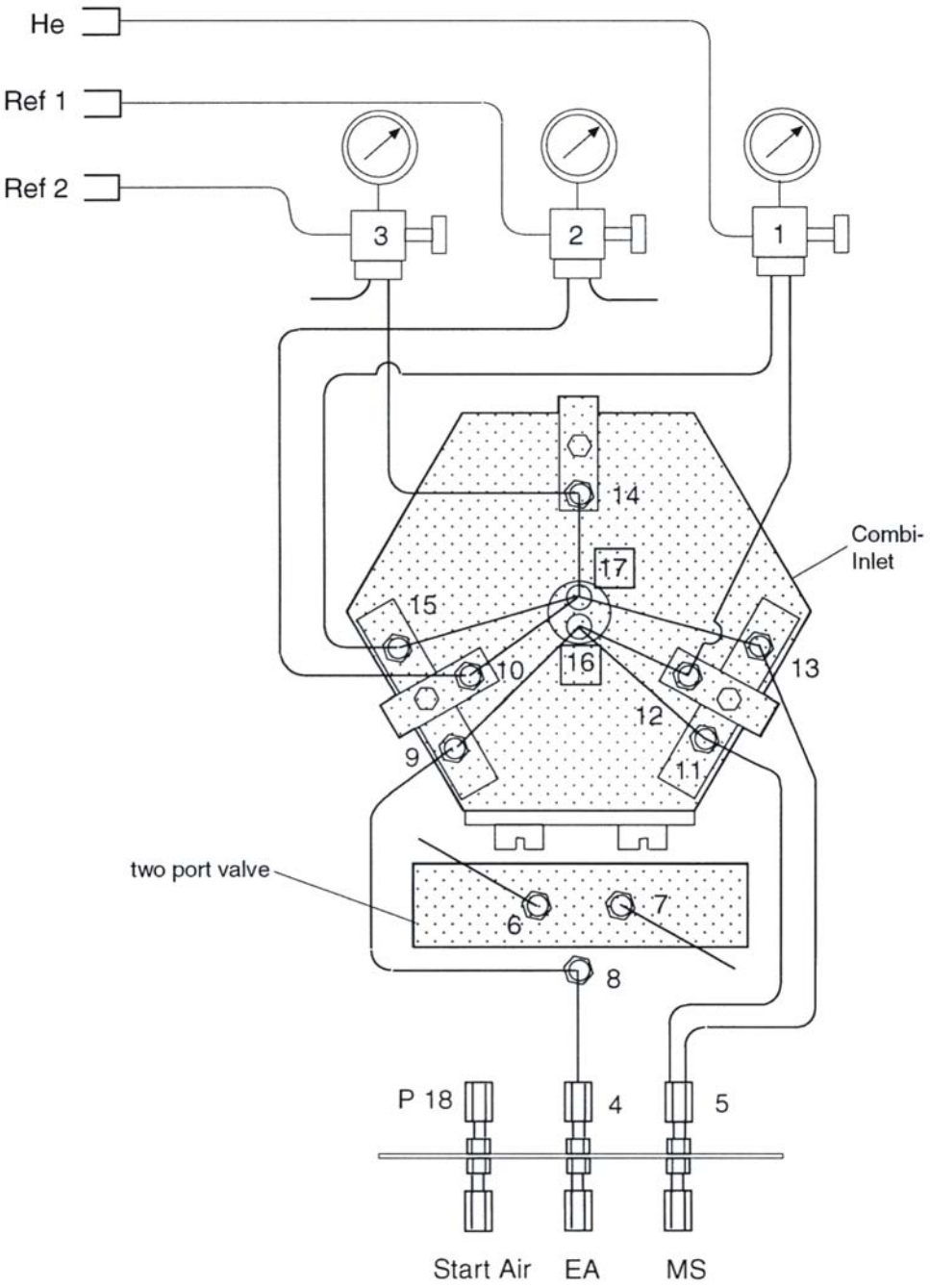


Figure 8-2. Combi Inlet of ConFlo III (Part No. 113 2260)

Table 8-2. Capillaries and Ferrules

Connection	Inner Diameter of *Capillary*	Length	Part No.	capillary-related *Ferrule*	Part No.
1 - 12* - 16	0.18 mm	2000 mm	054 2910	GVF2 / 003	106 0170
1 - 15* - 17	0.1 mm	250 mm	104 8990	GVF2 / 003	106 0170
2 - 10* - 17	0.05 mm	310 mm	054 3380	GVF2 / 005	100 4780
3 - 14* - 17	0.05 mm	340 mm	054 3380	GVF2 / 005	100 4780
8 - 9* - 16	0.32 mm	1000 mm	100 4640	GVF / 005	056 6390
4 - 8	1/16"-SS	fitting	060 5470	Swagelock	
5 - 11* - 16	0.1 mm	2000 mm	104 8990	GVF / 003	100 4850
5 - 13* - 17	0.05 mm	1500 mm	054 3380	GVF / 003	100 4850
6	0.32 mm	115 mm	100 4640	GVF / 005	056 6390
7	1/16"-SS	650 mm	060 5470	Swagelock	
2	0.1 mm	200 mm	104 8990	GVF2 / 003	106 0170
3	0.1 mm	200 mm	104 8990	GVF2 / 003	106 0170

* denotes the respective point of support for the capillaries

8.3 Function Schematic

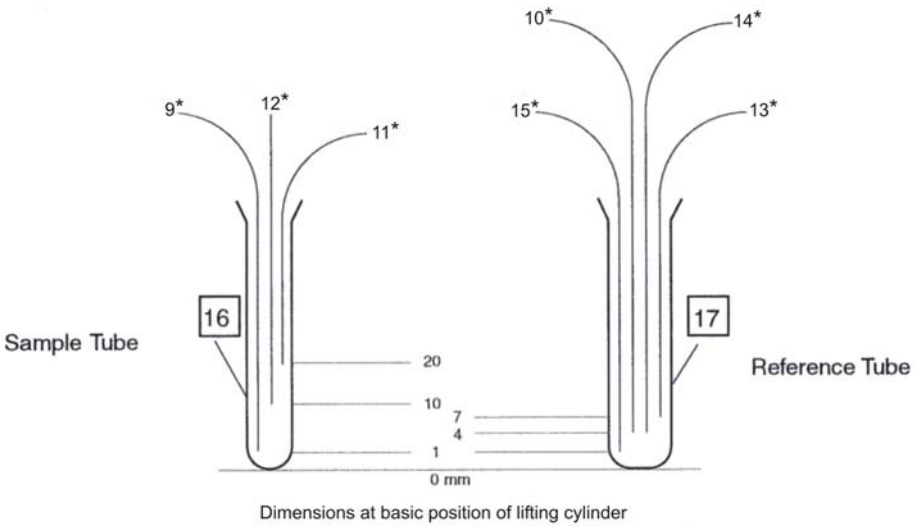


Figure 8-3. Sample Tube and Reference Tube

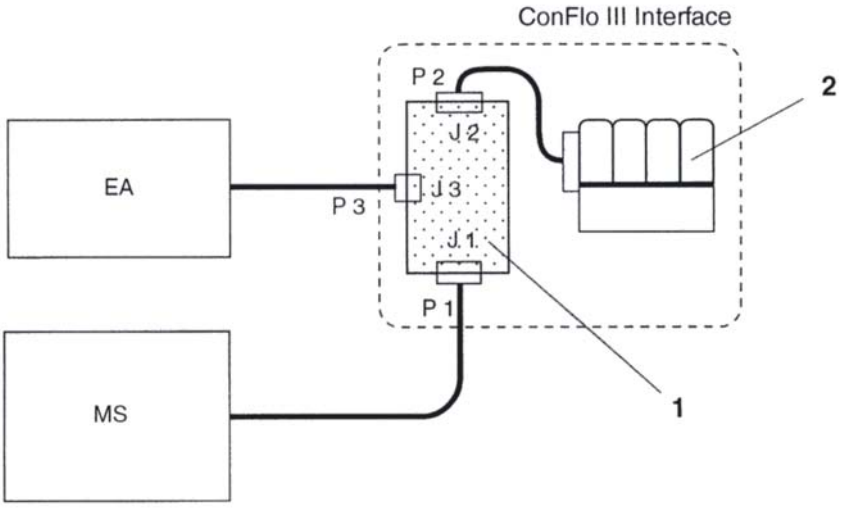


Figure 8-4. Circuit Diagram

Table 8-3. EA Start pcb and Manifold Card in the Circuit Diagram

Pos. No.	Quantity	Designation	Part No.
1	1	EA start pcb	204 2350
2	1	manifold card, 4 station	108 3241

8.4 Connections for Compressed Air

P 1 - P 18
P 2 - P 9
P 3 - P 10
P 4 - P 5
P 6 - P 11
P 7 - P 8

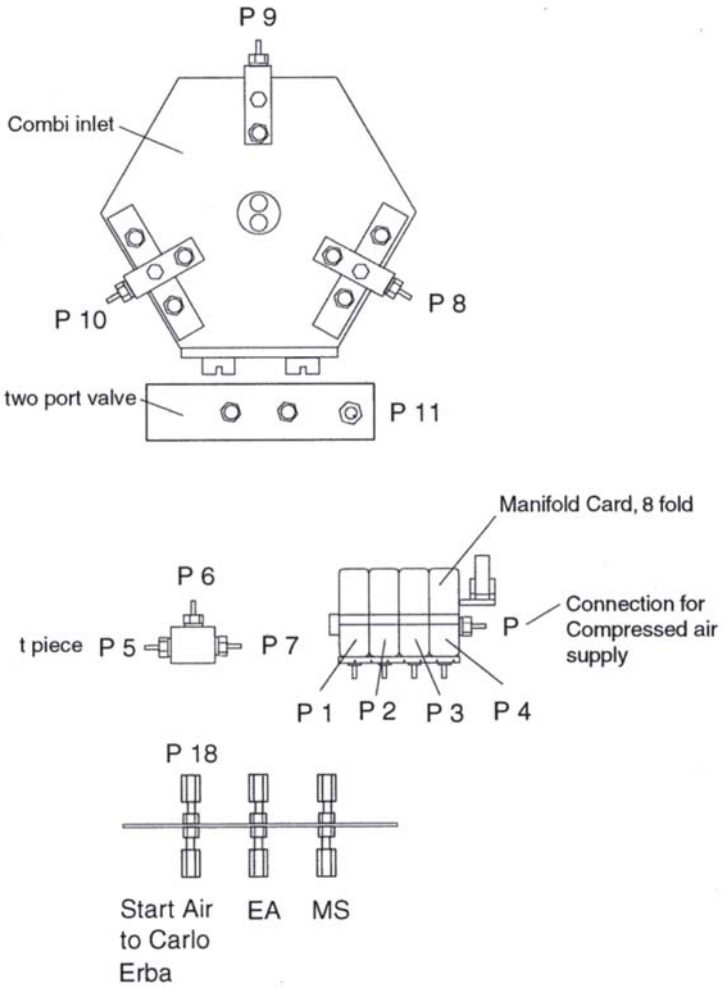


Figure 8-5. Connections for Compressed Air

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