

Mass Spectrometry

GasBench Plus

Operating Manual

BRE0029232

Revision B

November 2022

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Original Operating Instructions

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General Lab Equipment, Not for Clinical, Patient or Diagnostic Use.

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Technical Data of the GasBench Plus Device

The table lists the most important technical data of the GasBench Plus device, in relation to the installation. See the respective chapters of this manual for details and additional instrument properties.

Parameter	Specification	Value
Instrument Properties		
	Length × width	345 mm × 550 mm
	Length × width incl. TriPlus RSH ^a and temperature controller	780 mm × 810 mm
	Weight of GasBench Plus device	14 kg
	Weight of thermostatted tray, TriPlus RSH ^a and baseplate	43 kg
	Noise emission	below 70 dB(A)
Power Requirements^b		
	Nominal voltage	230 V, 50–60 Hz, single phase
	Voltage fluctuation	± 10% (207–253 V working voltage)
	Maximum current	1.6 A
	Power	0.5 W
	Fuse	3.15 A
	Degree of protection	IP 20
	Protection class	Class I
	Overvoltage category	II
Operating Environment		
	Laboratory temperature	18–24 °C
	Maximum Temperature fluctuation ^c	≤ ±1 °C/h
	Humidity	40–60%, non-condensing and non-corrosive atmosphere
	Maximum altitude	2000 m above sea level
	Pollution degree	2
Gas Requirements		
He	5.0 (99.999%)	2.5 bar (as carrier gas) 4 bar (as flush gas)
CO ₂	4.5 (99.995%)	2.5 bar (as reference gas)

Parameter	Specification	Value
H ₂	≥ 5.0 (99.9999%) ^d	2.5 bar (as reference gas)
He/CO ₂	He 5.0 (99.999%) with 0.3–1% CO ₂ 4.5 (99.995%)	2.5 bar (for ¹⁸ O water equilibration)
He/H ₂	He 5.0 (99.999%) with 1–2% H ₂ 5.0 (99.999%)	2.5 bar (for ² H water equilibration)
Compressed air ^b		4 bar (for carbonate option)

^a TriPlus RSH SMART for GasBench Plus - Adv and TriPlus RSH SMART for GasBench Plus - Std

^b Supplied by the IRMS

^c Temperature fluctuations exceeding the given range might affect instrument performance.

^d The reference H₂ gas isotopic signature (d2H) must not be lower than -300‰

Using this Manual

Welcome to the Thermo Scientific™ GasBench Plus device. The GasBench Plus device is a continuous flow interface for the Thermo Scientific isotope ratio mass spectrometers of the DELTA Series and the 253 Plus™ MS.

Contents

- [About this Manual](#) on page 1-1
- [Typographical Conventions](#) on page 1-2
- [Reference Documentation](#) on page 1-4
- [Contacting Us](#) on page 1-5
- [Training](#) on page 1-6

About this Manual

This *GasBench Plus Operating Manual* contains precautionary statements that can prevent personal injury, instrument damage, and loss of data if they are properly followed. It describes the modes of operation and principle hardware components of your GasBench Plus instrument and provides step-by-step instructions for cleaning and maintaining it.

This *GasBench Plus Operating Manual* is intended for all personnel that need a thorough understanding of the instrument (to perform maintenance or troubleshooting, for example).

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

NOTICE

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.



Consult Manual. Read this manual carefully before using the instrument and keep it for future reference.

Typographical Conventions

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

Signal Words

Make sure that you follow the precautionary statements presented in this manual. The special notices appear different from the main flow of text:

NOTICE

Points out possible material damage, data loss, impaired data quality and other important information in connection with the instrument.

Tip Highlights helpful information that can make a task easier.

Safety Symbols

Notices that concern the safety of the personnel who operate the GasBench Plus mass spectrometer appear different from the main flow of text:



Always be aware of what to do with and the effect of safety information.

CAUTION

Points out a hazardous situation that can lead to minor or medium injury if it is not avoided.

WARNING

Points out a hazardous situation that can lead to severe injury or death if it is not avoided.

DANGER

Points out a hazardous situation that will lead to severe injury or death if it is not avoided.

Viewpoint Orientation

The expressions left and right used in this manual always refer to the viewpoint of a person that is facing the front side of the instrument.

Data Input

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

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

Topic Headings

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

Chapter Name

Second Level Topics

Third Level Topics

Fourth Level Topics

Reference Documentation

This *GasBench Plus Operating Manual* represents the Original Operating Instructions.

Thermo Fisher Scientific provides additional documents for the GasBench Plus instrument that are not part of the Original Operating Instructions.

Reference documentation for the GasBench Plus instrument includes the following documents:

- *Gas Isotope Ratio MS Pre-Installation Requirements Guide*

This guide contains information on the required environmental conditions in the intended location for the instrument.

- *Qtegra ISDS Software for the Gas Isotope Ratio Mass Spectrometers Installation Guide*

- *GasBench II Consumables and Parts Catalog*

This document lists part numbers of key consumables and spare part items for the GasBench II device.

- *TriPlus RSH SMART Hardware Manual*

- *253 Plus Operating Manual*

- *DELTA Q Operating Manual*

- *ConFlo IV Operating Manual*

- *PreCon Operating Manual*

- *PreCon Consumables and Parts Catalog*

This document lists part numbers of key consumables and spare part items for the PreCon device.

You can access PDF files of the documents listed above and of this manual from the data system computer or other electronic storage devices.

A printed version of this *GasBench Plus Operating Manual* is shipped with the instrument.





Also refer to the user documentation provided by the manufacturers of third party components:


- Forepump(s)
- Turbomolecular pump(s)
- Temperature controller

- Data system computer and monitor
- Safety Data Sheets (SDSs)

Contacting Us

There are several ways to contact Thermo Fisher Scientific. You can use your smartphone to scan a QR code™, which opens your email application or browser.

Contact	Link / Remarks	QR code
Brochures and Ordering Information	www.thermofisher.com/IRMSPeripherals	
Patent Information	Your Thermo Scientific product may be manufactured under or covered by at least one or more U.S. patents and other patents pending. See www.thermofisher.com/patents for details.	
Service Contact	For technical support related to your instrument or software, visit the Services & Support tab at www.thermofisher.com or visit www.unitylabservices.com to find the customer care telephone line or email address for your geographical region.	
Technical Documentation SharePoint EU REACH Statement Health and Safety Form	<p>❖ To get user manuals for your product</p> <ol style="list-style-type: none"> 1. With the serial number (S/N) of your instrument, request access to our customer SharePoint as a customer at www.thermofisher.com/Technicaldocumentation. You find the serial number of your instrument on the name plate. See “Name Plate” on page 4-3 for information about its location. 2. For the first login, you have to create an account. Follow the instructions given on screen. Accept the invitation within six days and log in with your created Microsoft™ password. 3. Download new revisions of user manuals and other customer-oriented documents for your product. Translations into other languages may be available there as well. <p>See “Accessing the Technical Documentation SharePoint” on page B-1 for details.</p>	

Contact	Link / Remarks	QR code
Customer Feedback	<p>❖ To suggest changes to this manual</p> <p>You are encouraged to report errors or omissions in the manual. Send an email to the Technical Documentation at documentation.bremen@thermofisher.com.</p> <p>The PDF versions of our manuals allow adding comments with Adobe Acrobat Reader or other freely available PDF reader programs.</p>	

Training

Thermo Fisher Scientific offers worldwide training on instruments and software. Experience has shown that maximum results can be obtained from a scientific instrument if the instrument operators receive an adequate training.

We recommend that the key users participate in a basic operator training. For information on training courses and enrollment, contact your local Thermo Fisher Scientific office.

Scope of Delivery

This chapter lists the standard components of your GasBench Plus device and optional components.

Contents

- [Standard System Components](#) on page 2-1
- [Optional System Components](#) on page 2-1
- [Scope of Delivery of the Predecessor GasBench II System](#) on page 2-2

Standard System Components

The GasBench Plus device comprises the following components:

- GasBench
- Autosampler
 - TriPlus RSH SMART for GasBench Plus - Std
 - TriPlus RSH SMART for GasBench Plus - Adv
- Installation Kit including
 - Equipment for connecting the above components (tubings, cables)
 - PoraPLOT™ Q capillary column
 - Tools for installation and maintenance
 - Spare parts
- Printed manuals
 - *GasBench Plus Operating Manual*

Optional System Components

For reference, the following list contains components that are frequently shipped with the standard GasBench Plus device. They are not part of the standard system and may therefore not be present in your laboratory:

- Carbonate Reaction Kit (“carbonate option”)

Scope of Delivery

Scope of Delivery of the Predecessor GasBench II System

- Denitrification Kit
- 96 sample tray (ambient temperature)
- 96 sample tray (temperature controlled)
- Single cryotrap
- Dual cryotrap (PostCon)
- Autosampler extensions (TriPlus RSH for GasBench - Std and TriPlus RSH for GasBench - Adv)
- Hydrophobic Pt catalyst sticks for H₂/H₂O equilibration
- 5 Å PLOT capillary column

Scope of Delivery of the Predecessor GasBench II System

The predecessor GasBench II system comprises the following main components in deviation to the GasBench Plus system and is described in the - *GasBench II Operating Manual*.

- GasBench II device
- Autosampler CTC Pal 80 in A200S mode (“GC Pal”)

Optional system components in deviation to the GasBench Plus

- Denitrification Kit (including *Denitrification Kit for GasBench II - Installation Guide*)

NOTICE

Refer to the *GasBench II Operating Manual* for complete scope of delivery of the GasBench II system. The *GasBench Plus Operating Manual* describes the Qtegra installation and extra handling of the GasBench II system with Qtegra ISDS Software for GIRMS.

Hardware Components

This chapter describes important hardware components of the GasBench Plus device.

Contents

- [Layout of the GasBench Plus Device](#) on page 3-2
- [Autosamplers](#) on page 3-8
- [Sample Trays Used with the GasBench Plus Device](#) on page 3-9
- [Needles for the GasBench Plus Device](#) on page 3-15
- [Water Removal Devices](#) on page 3-17
- [Valco Eight Port Valve](#) on page 3-18
- [GC Oven](#) on page 3-22
- [Open Splits](#) on page 3-26

NOTICE

Refer to the *GasBench II Operating Manual* for GasBench II hardware components.

Layout of the GasBench Plus Device

This section describes the layout of the GasBench Plus device and important hardware components.

Figure 3-1 shows the GasBench Plus device with the TriPlus RSH SMART for GasBench Plus - Std autosampler.



Figure 3-1. GasBench Plus device with TriPlus RSH SMART for GasBench Plus - Std autosampler

Figure 3-2 shows the GasBench Plus device with the TriPlus RSH SMART for GasBench Plus - Adv autosampler.



Figure 3-2. GasBench Plus device with TriPlus RSH SMART for GasBench Plus - Adv autosampler

Working Principle

As Figure 3-3 schematically shows, the GasBench Plus device consists of:

- a user programmable autosampler (1)
- a gas sampling system (2)
- a maintenance-free water removal system (3)
- a loop injection system (4)
- an isothermal gas chromatograph (GC) (5)
- an active open split interface (6)
- a reference gas injection system with three reference ports (7) and
- one or two optional LN2 traps for cryofocusing

Hardware Components

Layout of the GasBench Plus Device

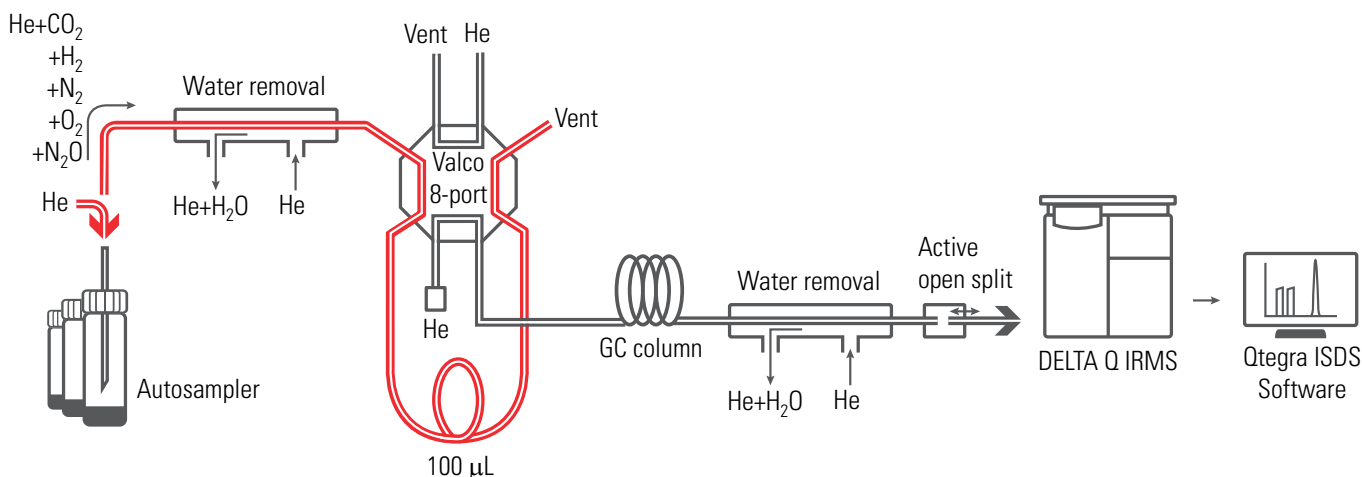


Figure 3-3. Schematics of GasBench Plus device

The autosampler can be equipped with a variety of sample trays. A precisely thermostatted sample tray is used for carbonates and water equilibration.

The gas sampling system includes a two port GasBenchTool 55, which can hold one or two needles for dual injection. The attached measurement needle adds a gentle flow of He into the sample vial, thus diluting and displacing the sample gas. Water is removed from the sample gas through diffusion traps. The loop injector aliquots the sample gas onto the GC column, which separates the molecular species. The reference gas injection system allows accurate referencing of each sample aliquot to isotopic standards. The system is designed for unattended measurements with high throughput, ensuring high productivity and high reliability.

The GasBench Plus device can be used for the isotopic characterization of CO₂, N₂O, O₂ or N₂ between 200 nmol and 20 µmol of total sample size. The CO₂ can be in the original gas sample (air or breath), be released through a preparation step (carbonates, DIC), or be added to the original liquid sample (water-CO₂ equilibration).

Front Side

At the front panel of the GasBench Plus device, the pressure regulators and the pressure gauges for the reference gases and helium are located. See [Figure 3-4](#).

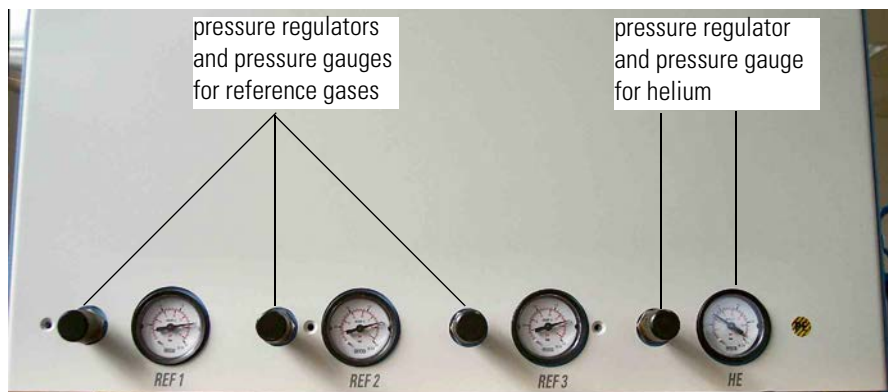
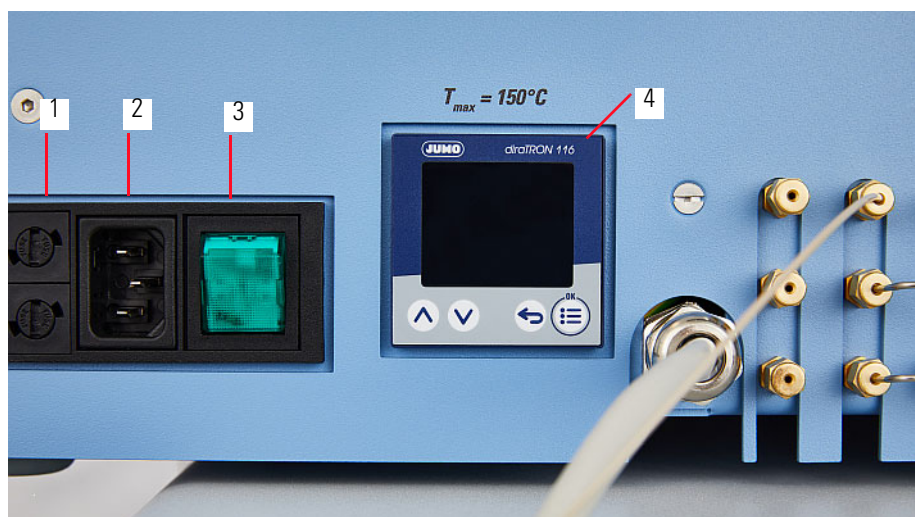


Figure 3-4. Pressure regulators and pressure gauges at front panel

Left Side

[Figure 3-5](#) shows the main fuses (3.15 A; **1**), the main power socket **2**, the main power switch **3** and the JUMO diraTRON 116 temperature controller **4**. They are all located at the lower part of the left side panel.

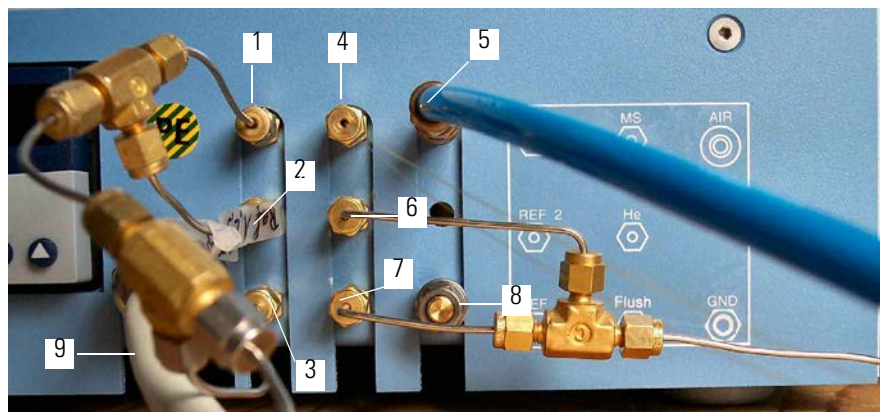


Labeled Components: 1=main fuses, 2=main power socket, 3=main power switch (on/off), 4=temperature controller

Figure 3-5. Power switch and JUMO diraTRON 116 temperature controller

Figure 3-6 shows the gas connections and the cable for connecting the GasBench Plus device to the IRMS. They are all located at the lower left side panel. See the connection scheme (Figure 5-4) as well.

Tip For flush gases (CO₂ in He, H₂ in He, for example), a dedicated plug must be installed.



Labeled Components: 1–3=connections for reference gases, 4=capillary feedthrough to IRMS, 5=connection for compressed air, 6=helium carrier gas connection, 7=flush connection, 8=GND (ground), 9=cable for connection to IRMS

Figure 3-6. Gas connections at left side panel

Figure 3-7 shows the fan for H₂ exhaustion located at the upper left side panel.



Figure 3-7. Fan at left side panel

Rear Side

Figure 3-8 shows the connections for the optional cryo traps at the rear panel. Each of both optional cryo traps (trap 1, trap 2) has a control output (Control) and an output for compressed air (Supply). See “Cryo Trap Options” on page 11-2 for more information about the cryo traps.

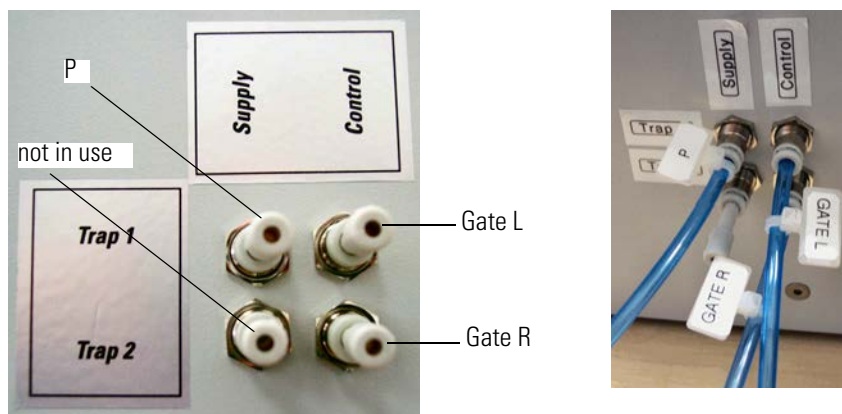
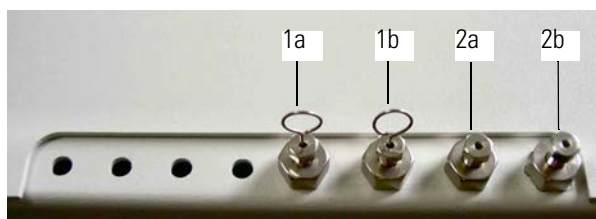


Figure 3-8. Ports for connecting optional cryo traps at rear panel

Top Side

Figure 3-9 shows the connections for the sample needles. They are located on top of the GasBench Plus device. If you bought a second needle holder (complete), you can flush with a second needle. This shortens the flushing time of a tray by 50%. See “Needles for the GasBench Plus Device” on page 3-15 for more information about connecting the needles to the GasBench Plus device.



Labeled Components: 1a=flush connection for single needle flush, 1b=flush connection for dual needle flush, 2a=input of carrier gas into sample needle, 2b=output of carrier gas with sample gas to the first Nafion water trap

Figure 3-9. Connections for sample needles (view from rear panel)

Figure 3-10 shows the GasBench Plus device after removing its cover.

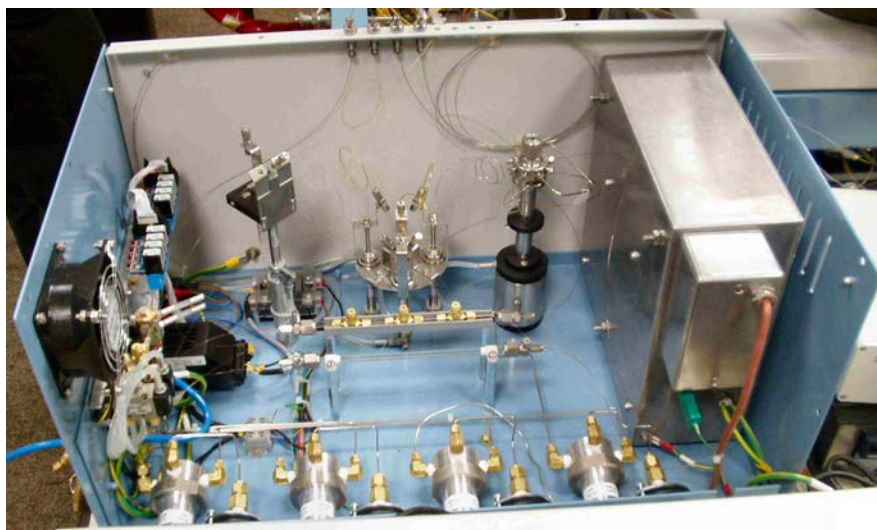


Figure 3-10. GasBench Plus device – cover removed

Autosamplers

Two TriPlus RSH SMART for GasBench Plus autosamplers are provided, the Standard and the Advanced type. Both autosamplers can be equipped with trays as described in “Sample Trays Used with the GasBench Plus Device” on page 3-9.

Figure 3-11 shows a drawing of the TriPlus RSH SMART for GasBench Plus - Std autosampler.

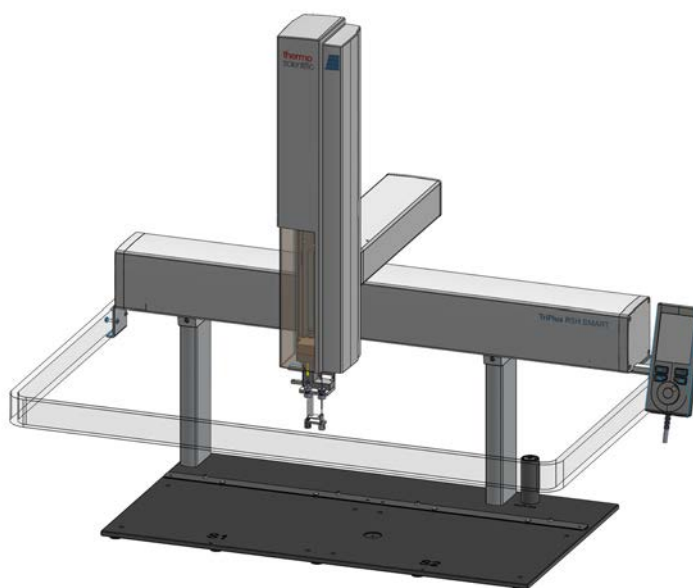


Figure 3-11. TriPlus RSH SMART for GasBench Plus - Std autosampler

Figure 3-12 shows a drawing of the TriPlus RSH SMART for GasBench Plus - Adv autosampler.

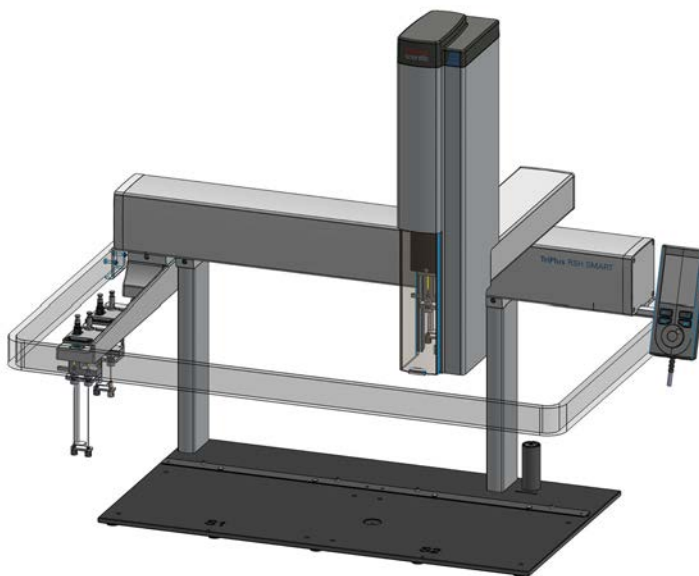


Figure 3-12. TriPlus RSH SMART for GasBench Plus - Adv autosampler

Sample Trays Used with the GasBench Plus Device

By default, the GasBench Plus device is delivered without any sample tray. Therefore, a sample tray must additionally be ordered unless a customized tray or standards trays that are delivered together with the TriPlus RSH SMART autosampler. Depending on the particular application, the sample trays described in the following topics are available for the GasBench Plus device.

Non-Thermostatted Sample Tray

For the GasBench Plus device, standard TriPlus RSH SMART for GasBench Plus autosamplers can be used (for DIC analysis, for example). By default, the TriPlus RSH SMART for GasBench Plus autosamplers are delivered without any trays.

Both autosampler units contain a standard GasBench Tool 55 and a needle protection guide to be installed on the autosampler.

Hardware Components

Sample Trays Used with the GasBench Plus Device

The non-thermostatted sample tray is suitable for DIC equilibrium work or breath gas analysis. See [Figure 3-13](#).

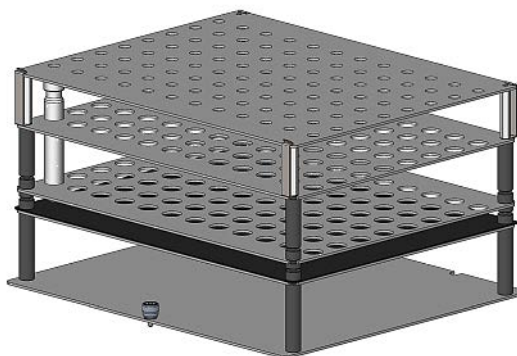


Figure 3-13. A 12 mL non-thermostatted sample tray

[Table 3-1](#) lists the sample trays for TriPlus RSH SMART for GasBench Plus - Std or TriPlus RSH SMART for GasBench Plus - Adv.

Table 3-1. Sample trays for TriPlus RSH SMART for GasBench Plus - Std or TriPlus RSH SMART for GasBench Plus - Adv

P/N	Description
BRE0029065	Mounting plate with SLOT 1 & SLOT 2 for all trays below
BRE0029300	thermostatted sample tray for 96 sample vials of 12 mL size each, 8 rows, 12 columns
BRE0029064	Non-thermostatted sample tray for 96 sample vials of 12 mL size each, 8 rows, 12 columns
BRE0029066	Non-thermostatted sample tray for 77 sample vials of 20 mL size each, 7 rows, 11 columns
BRE0029067	Non-thermostatted sample tray for 48 sample vials of 50 mL size each, 6 rows, 8 columns
BRE0029068	Non-thermostatted sample tray for 20 sample vials of 100 mL size each, 4 rows, 5 columns
BRE0029069	Non-thermostatted sample tray for 12 sample vials of 250 mL size each, 3 rows, 4 columns
BRE0029075	Non-thermostatted sample tray for 250 sample vials of 12 mL size each, 10 rows, 25 columns

Thermostatted Sample Tray

If temperature control is required for your application (carbonate analysis, equilibration measurements, high temperature stability measurements, for example) a thermostatted sample tray should be used. See [Figure 3-14](#).

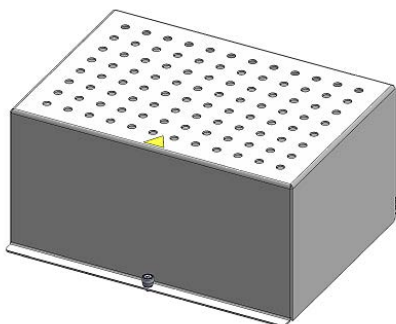


Figure 3-14. Thermostatted sample tray – top view

When using the thermostatted sample tray, take into account that:

- the GasBench Plus has a mounting plate with two slots (1 and 2). Each of the slots allows mounting different trays. The only exception is that a thermostatted tray cannot be attached to the right while a non-thermostatted tray is attached to the left. All combinations including doubling itself on the two slots, do work.
- the thermostatted sample tray is optimized for carbonate measurements. See [“Analyzing Carbonates”](#) on [page 9-38](#).
- depending on the carbonate reaction time, the recommended delay between acid dosing and measurement is 30–60 min.
- the acid reservoir is thermostatted.

Additional Options for IRMS, Autosampler and Peripheral Couplings

When coupling the GasBench Plus device with a PreCon or a GC, additional options for IRMS, autosamplers and various peripherals have to be installed:

- TriPlus RSH SMART for GasBench Plus - Std. Autosampler for PreCon including sample tray for 96 x 12 mL vials, thermostatted tray base plate, measurement needle (55 mm), autosampler mounting brackets, installation parts
- TriPlus RSH SMART for GasBench Plus - Adv. Autosampler for PreCon including sample tray for 96 x 12 mL vials, thermostatted tray base plate, measurement needle (120 mm), autosampler mounting brackets, installation parts

Layout of 96 Sample Trays with 12 mL Vials

This section describes the 96 sample trays (that is 8×12 and 12×8 trays) with 12 mL vials and 4.5 mL vials. The movement of the autosampler across the 8×12 tray is shown in [Figure 3-15](#) and [Figure 3-18](#).

They are all pre-installed in the TriPlus RSH autosampler firmware and can be optionally taken from the Qtegra software by the user if installed on the mounting plate. An automated tray detection is not implemented. Check teaching of the GasBench tools to the autosampler.

See “[Checking Calibration and Teaching Installed GasBench Trays](#)” on [page 5-37](#). Refer to the *TriPlus RSH SMART Installation Guide*. See “[Installing an RSH for GasBench](#)” on [page 5-17](#) as well.

The tray contains 96 holes with the properties listed in [Table 3-2](#). This tray needs to be positioned below the RSH autosampler. Make sure that you have sufficient free space below the x-arm of the RSH autosampler.

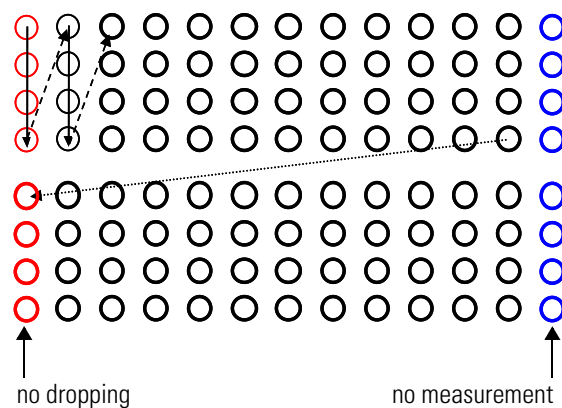
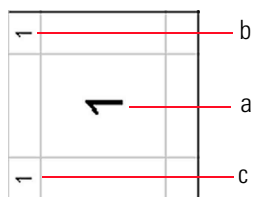


Figure 3-15. Autosampler movement across 8×12 tray (ex factory)

Table 3-2. Properties of tray holes

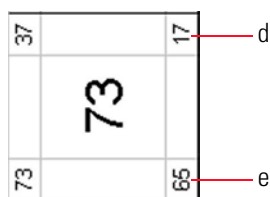
Parameter	Value
spacing of holes	26 mm × 26 mm
diameter of holes	15.7 mm
depth of holes	85 mm

To attach a TriPlus RSH tray, refer to the *TriPlus RSH Operating Manual*.



Labeled Components: a=autosampler position in sequence, b=row number in sequence (carbonates), c=row number in sequence (equilibration)

Figure 3-16. Autosampler position and row number in sequence



Labeled Components: d=dual needle flush, e=flush fill

Figure 3-17. Dual needle flush and flush fill

1	1	9	5	17	9	25	13	33	17	41	21	49	25	57	29	65	33	73	37	81	41	89	45
1		9		17		25		33		41		49		57		65		73		81		89	
2	2	10	6	18	10	26	14	34	18	42	22	50	26	58	30	66	34	74	38	82	42	90	46
2		10		18		26		34		42		50		58		66		74		82		90	
3	3	11	7	19	11	27	15	35	19	43	23	51	27	59	31	67	35	75	39	83	43	91	47
3		11		19		27		35		43		51		59		67		75		83		91	
4	4	12	8	20	12	28	16	36	20	44	24	52	28	60	32	68	36	76	40	84	44	92	48
4		12		20		28		36		44		52		60		68		76		84		92	
5	45	13	49	21	53	29	57	37	61	45	65	53	69	61	73	69	77	77	81	85	85	93	89
5		13		21		29		37		45		53		61		69		77		85		93	
6	46	14	50	22	54	30	58	38	62	46	66	54	70	62	74	70	78	78	82	86	86	94	90
6		14		22		30		38		46		54		62		70		78		86		94	
7	47	15	51	23	55	31	59	39	63	47	67	55	71	63	75	71	79	79	83	87	87	95	91
7		15		23		31		39		47		55		63		71		79		87		95	
8	48	16	52	24	56	32	60	40	64	48	68	56	72	64	76	72	80	80	84	88	88	96	92
8		16		24		32		40		48		56		64		72		80		88		96	
	8	16	52	24	24	24	24	32	28	40	28	48	32	56	32	64	36	72	36	80	40	88	

Figure 3-18. Sampling positions for 8x12 sample tray (non-thermostatted and thermostatted)

Needles for the GasBench Plus Device

This section describes the various needles that are in use with the GasBench Plus device.

Sample Needle

The sample needle (Figure 3-19), is located in the TriPlus RSH. The correct connection is important to guarantee high GC performance. All sample needles pass a quality control ex-factory. See “Connecting the Sample Needle” on page 3-15.

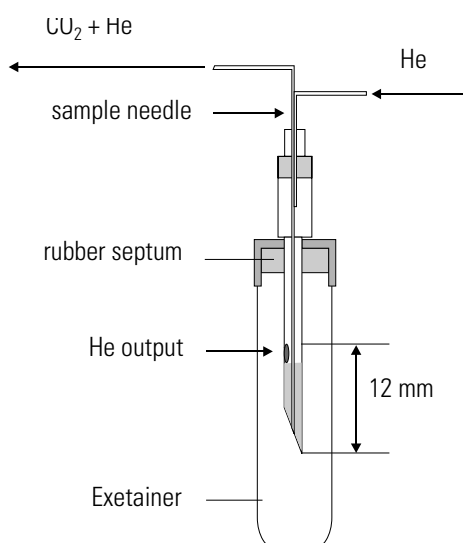


Figure 3-19. Sample needle

Tip The sample needle is sometimes synonymously called transfer needle or measurement needle.

Connecting the Sample Needle

Connect the sample needle as shown in Figure 3-20. The sample needle should direct the helium flow through the side hole and take up the sample through the needle tip. This ensures dead volume-free and therefore memory-free sampling. The $\text{CO}_2 + \text{He}$ carrying capillary and the corresponding bulkhead connector should be marked by a flag. See Figure 3-20.

Now, helium gently moves CO_2 from the headspace of the Exetainer™ into the fused silica capillary within the needle tip. From here, the sample is transferred through the water removal (1, see “Principle of Water Removal” on page 3-18) and the Valco loop for GC injection. The helium flow should be at approximately 0.4–0.5 mL/min (measured at the vent of the Valco valve; see Figure 3-25).

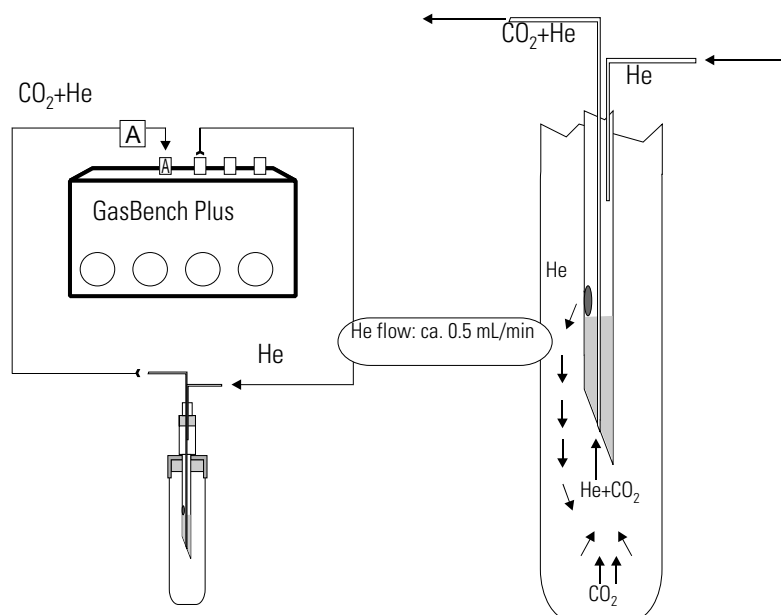


Figure 3-20. Connecting the sample needle

Flush Needle

The flush needle is a modified sample needle. It operates for a normal 12 mL sample vial at enhanced flush flow to exchange the headspace of the sample from ambient air or to exchange with an equilibration gas (for example CO₂ in He, H₂ in He).

Connecting the Flush Needle

The measurement needle will be changed. The fused silica capillary will be cut using a wafer or cutter approximately 10–20 cm from the top of the needle connection. This allows less restriction and a higher flow of flush gas through the sample vial and the needle.

Tip If a sample needle is defective, a measurement needle can be used as a sample needle. Connect the fused silica capillary of a flush needle with a 0.32-0.32 or fit-to-all press-fit connector. See “GC Oven” on page 3-22.

It is connected as a single version either to port 2a in Figure 3-9 on page 3-7 or in dual needle version to ports 2a and 2b in Figure 3-9. For the dual needle version, one extra sample needle needs to be changed to a flush needle.

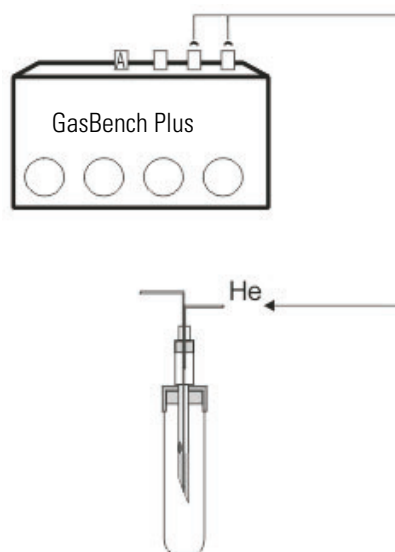


Figure 3-21. Connecting the flush needle

With the flush needle, different flush flows and flush gases are in use:

- Flushing with helium (≈ 100 mL/min during 4–6 minutes) in case of carbonates and DIC.
- Filling with a gas mixture of 0.3–0.4% CO_2 in helium and a flow of 50 mL/min makes the use of glove bags and glove boxes unnecessary.
- Filling of the sample vial headspace with CO_2 in He or H_2 in He.

Tip It is possible to connect two flush needles and operate them simultaneously by using our GasBench Tool 55.

For dual needle flush, an extra sample needle can be cut at the 0.32 mm ID deactivated fused silica capillary. This enables dual needle flush using two flush needles.

Water Removal Devices

The GasBench Plus device is equipped with two on-line water removal devices. See [Figure 3-3](#) on [page 3-4](#). One of them is positioned in front of the Valco eight port valve. The other one is used as a guard trap in front of the open split interface to the IRMS. The second water trap keeps the water background stable of helium coming from the GC column and the parts in front of it. See [Figure 3-22](#).

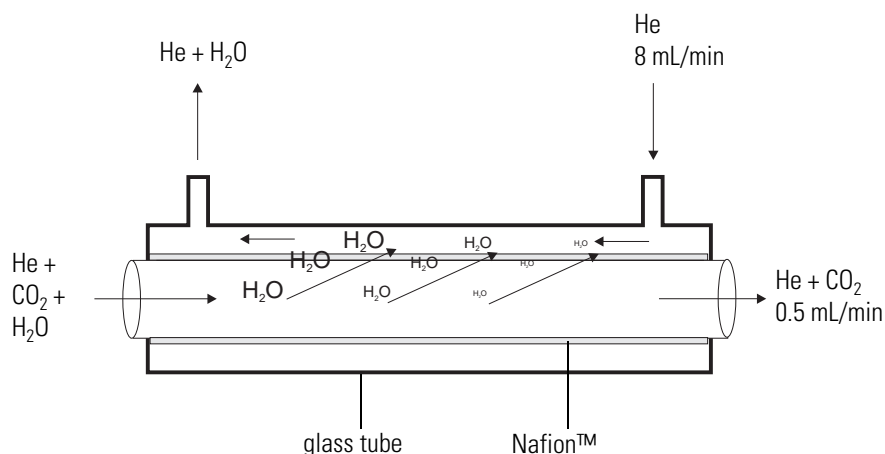


Figure 3-22. Schematic of water removal device

Principle of Water Removal

Water is removed from the transfer sample stream by a gastight but hygroscopic Nafion™ tubing. The sample flow (He + CO₂ + H₂O, 0.5 mL/min) passes through the Nafion tubing, which is mounted co-axially inside a glass tube. This glass tube, and therefore the outer surface of the Nafion tubing, is constantly kept dry by a He flow of approximately 8–20 mL/min.

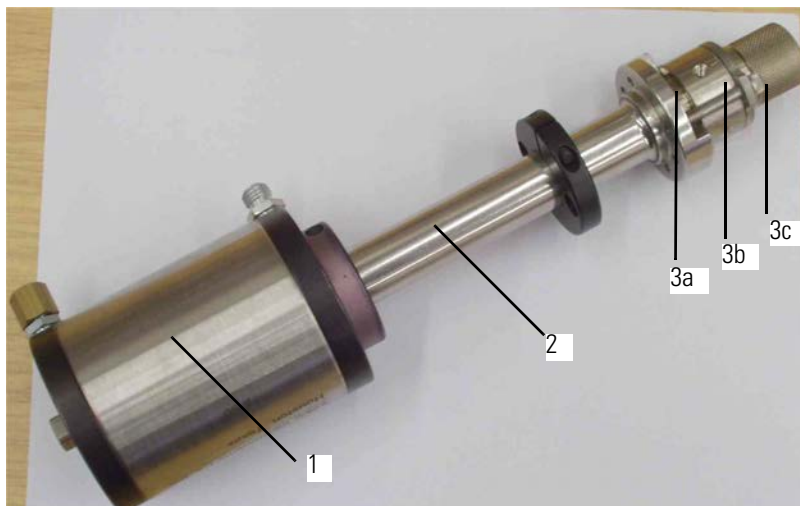
Owing to the water gradient through the Nafion wall, any water in the sample flow will move through the Nafion. A dry (He + CO₂) gas results, which flows towards the Valco loop. Alternatively, water-free nitrogen can be used for water removal.

Valco Eight Port Valve

The Valco eight port valve is an alternatively switching multiposition valve. It operates with dry, oil-free compressed air moving within an electropolished stainless steel body. It allows changing the gas flow by a pneumatic port switch under vacuum conditions.

Parts of the Valco Eight Port Valve

[Figure 3-23](#) shows the Valco eight port valve. The long shank allows to introduce the functional head into the oven as head and control are thermally separated by the distance. The chequered knob on top must be unscrewed, if you want to withdraw the rotor. After inserting the rotor, screw in the chequered knob again.



Labeled Components: 1=compressed air control, 2=long shank, 3=functional head (3a=mounting plate, 3b=n-port, 3c=chequered knob)

Figure 3-23. Valco eight port valve - side view

NOTICE

Take the direction of the letter on top of the rotors into account. After exchange or cleaning, the positioning of the letter must exactly be the same as it was before.

If the Valco valve leaks (argon background: switch to Instrument Control and measure Ar40 signal), as a first measure carefully clean the sensitive rotor.



Refer to Technical Note 201: Operation Notes and Cleaning Instructions of Valco Instruments Co. Inc. (VICI) at www.vici.com. It is also part of your equipment.



Refer to Technical Note 410: Multiposition Air Actuator O-Ring Replacement and to Technical Note 701: Operation Notes and Alignment Instructions, Air Actuated Multiposition Valves at www.vici.com.

Load Mode vs. Inject Mode

The Valco eight port valve is used in a six port setup. Two ports are in “standby” for each injection mode. See [Figure 3-24](#) and [Table 3-3](#).

Table 3-3. Load mode vs. Injection mode of Valco eight port valve

Load mode	Inject mode
Ports 1 and 8 are in “standby”.	The gas content of the sampling loop is directly transferred onto the GC column by the GC flow (2 mL/min, for example) via the ports 5 6 3 4.
The sample flow (He + CO ₂) purges the sampling loop (100 mL, for example) via the ports 2 3 6 7.	The sample flow is directly connected to Vent via the ports 2 1.
The GC column is directly connected to the He pressure via the ports 5 4.	

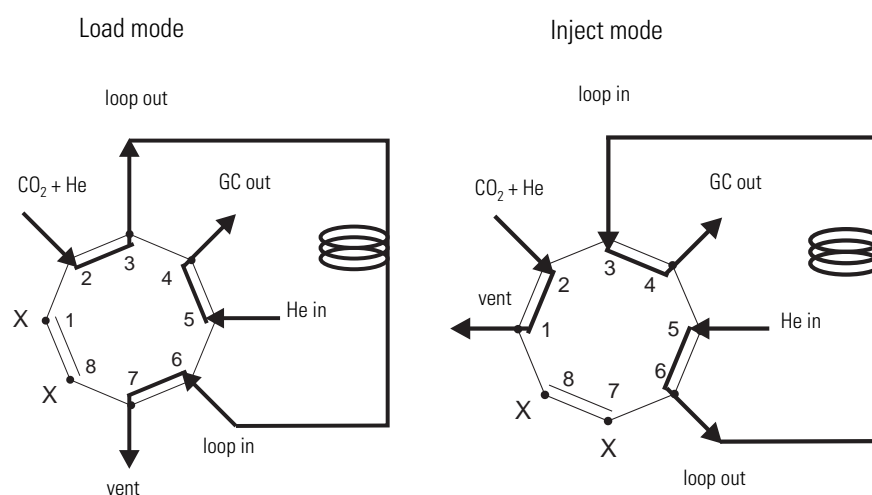


Figure 3-24. Valco eight port valve - Load mode vs. Inject mode

Changing the Loop Size



Refer to *Technical Note 201: Operation Notes and Cleaning Instructions* of Valco Instruments Co. Inc. (VICI) at www.vici.com. It is also part of your equipment.



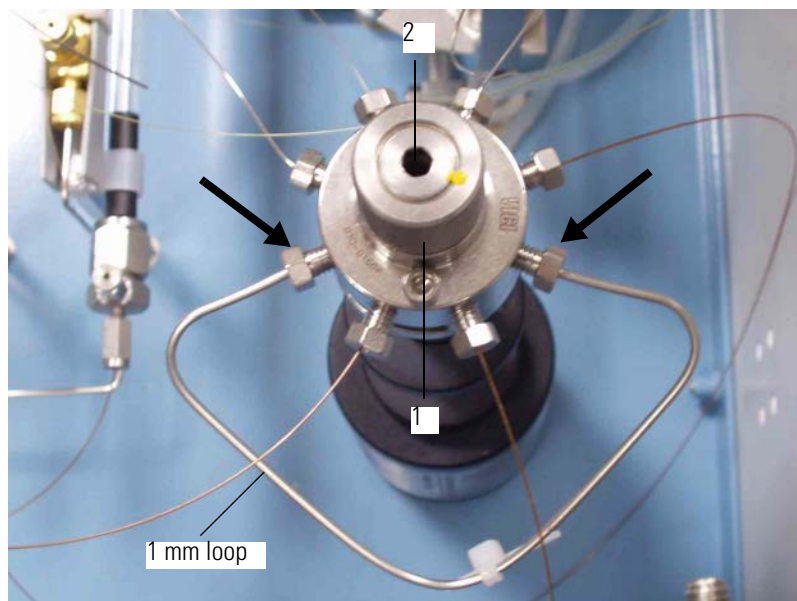
For proper loop installation, refer to *Technical Note 105: Installing a Loop* at www.vici.com.

NOTICE

Make sure that the Valco valve is in Load Mode. Changing the loop in Inject Mode interrupts the GC column flow. This will cause damage to the GC column.



Always use Valco stainless steel ferrules for mounting the loop. Refer to *Technical Note 503: Fitting Instructions* at www.vici.com.



Labeled Components: 1=chequered screw, 2=socket head screw

Figure 3-25. Valco valve with loop - top view

The arrows in [Figure 3-25](#) show the two screws **1** and **2** that fasten the loop:

- The chequered screw **1** is used to fix the internal rotor, which is flexibly fitted within in the stator by a conical seal.
- The socket head screw **2** is used after fixing the internal rotor by the chequered screw. It allows adjusting the pressure acting from above upon the cone. By increasing this pressure, the internal rotor is tightened against the side walls.

❖ **To change the loop size**

1. Switch the Valco valve to Load Mode.
2. Open the nuts on port 3 and port 6. See [Figure 3-25](#).
3. Replace the loop.

Use loop sizes less than 250 mL for the two column types.

4. Tighten the nuts.

5. Inject the sample needle into a helium-filled vial and purge the loop before switching to Inject Mode.
6. At the Valco vent (port 7) check for a purge flow of 0.3–0.5 mL.

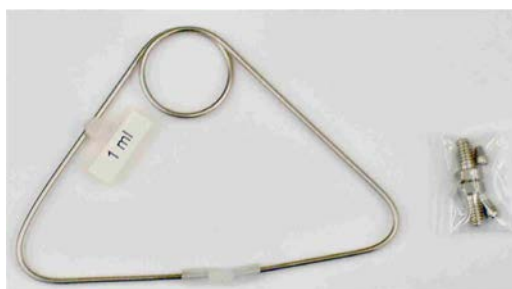


Figure 3-26. 1 mL loop

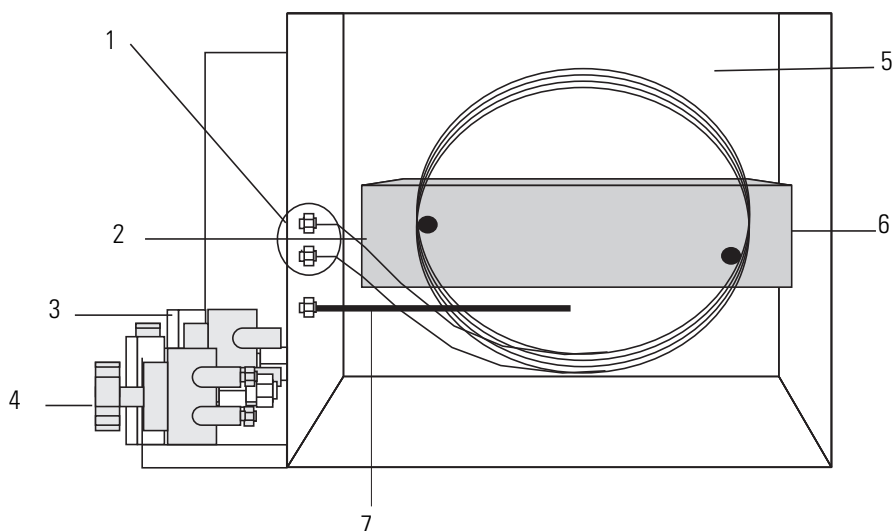


Figure 3-27. 2 mL loop

[Figure 3-26](#) and [Figure 3-27](#) are given as examples. Loops of 100 μL , 250 μL , and 1 mL are already part of your equipment provided by Thermo Fisher Scientific. If necessary, loops of even larger volumes are available. The 1 mL loop and the 100 μL loop are very similar. For nitrogen analysis, also smaller loops (1 μL) are available.

GC Oven

The GC oven is either equipped with a “HayeSep D” micro-packed stainless steel column or a PoraPLOT™ Q fused silica cap column (standard GC column). A JUMO diraTRON 116 temperature controller and a type K thermocouple guarantee stable isothermal conditions. The opened right side panel of the GasBench Plus device shows the GC oven with the column. See [Figure 3-28](#) and [Figure 13-5](#).



Labeled Components: 1=in/out, 2=heater assembly, 3=manometer, 4=gauge, 5=GC box (inside view), 6=GC column (PoraPLOT™ Q or HayeSep D), 7=thermocouple

Figure 3-28. GC oven – open

The GC column separates the different gas compounds released from the sample loop, N₂ and CO₂, for example. The compounds eluting from the GC column are transferred through the Nafion™ trap and via open split into IRMS.

NOTICE

Strictly avoid exceeding the maximum temperature of your column.

The change of the setpoints must be validated with a temperature calibrated multimeter and a thermocouple attached to it.

Type PoraPLOT Q GC Column

This column type is used in the current versions of the GasBench Plus device and is part of your equipment. See [Table 3-4](#).

Table 3-4. Properties of PoraPLOT Q GC column

Parameter	Value
type	fused silica column
length	25 m
inner diameter	0.32 mm
helium pressure	700–830 mbar
helium flow	approximately 2–3 mL/min
GC column temperature	room temperature, that is 24 °C

NOTICE

Strictly avoid exceeding the maximum temperature of the column.
Avoid fast pressure variations along the column ($E_p < 34.5$ mbar/s).

Type HayeSep D GC Column

This column type has been used in former versions of the GasBench device. See [Table 3-5](#).

Table 3-5. Properties of HayeSep D" GC column

Parameter	Value
type	1/16 in stainless steel micro-packed column
length	2 m
inner diameter	0.76 mm
packing material	polymer HayeSep D; 80/100 mesh
helium pressure	700–1000 mbar
helium flow	3–4 mL/min
GC column temperature	50–60 °C

NOTICE

Strictly avoid exceeding the maximum temperature of the column.

Temperature Controller of the GC Oven

The JUMO diraTRON 116 temperature controller allows controlling the temperature of the GC oven. It is located at the side panel of the GasBench Plus device. See [Figure 3-29](#).

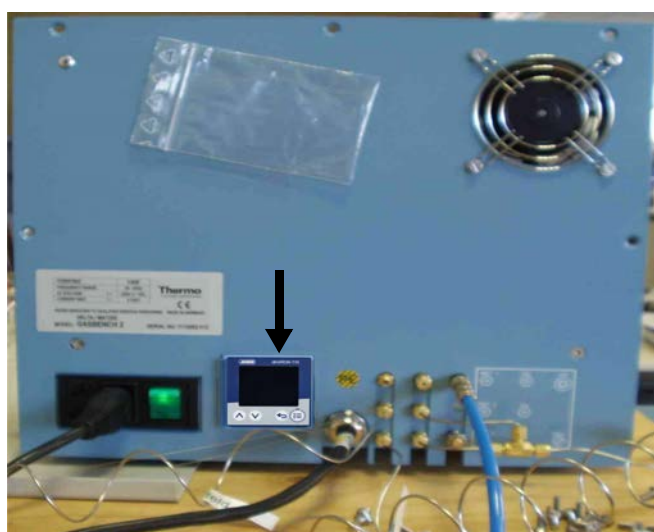


Figure 3-29. JUMO diraTRON 116 temperature controller at side panel

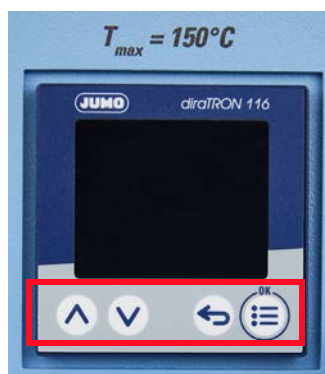






Figure 3-30. Buttons of JUMO diraTRON 116 temperature controller

Figure 3-30 and Table 3-6 show the four buttons of the JUMO diraTRON 116 temperature controller.

Table 3-6. Buttons of JUMO diraTRON 116 temperature controller

Button	Function
Up	 <ul style="list-style-type: none"> to increase a value to select the previous menu item or parameter
Down	 <ul style="list-style-type: none"> to decrease a value to select the next menu item or parameter
Back	 <ul style="list-style-type: none"> to go back to the previous menu level to leave Edit mode without applying changes
Menu/OK	 <ul style="list-style-type: none"> to activate the main menu to switch to a submenu to change the menu level to enter Edit mode to leave Edit mode with applying changes

The parameter values to be used for manual programming of the JUMO diraTRON 116 temperature controller have been pre-set by the factory. The change of the setpoints must be validated using a temperature-calibrated multimeter and a thermocouple attached to it.

NOTICE

Do not use self optimization of the JUMO diraTRON 116 temperature controller. The GC column may be destroyed.

The maximum temperature to be regulated by the JUMO diraTRON 116 temperature controller may not exceed 150 °C.



For details of manual programming, refer to the manual of the JUMO diraTRON 116 temperature controller.

Open Splits

In the GasBench Plus device, two open splits allow a continuous flow of the sample gas to the IRMS at atmospheric pressure:

- sample injection (active open split) and
- reference injection (reference open split)

Reference Injection

This section describes the function of the reference section of the GasBench Plus device. Three reference gases can be injected via a three-port open split interface. A He stream of 2 mL/min permanently flushes the interface tube. See [Figure 3-31](#) and [Figure 3-32](#). A permanent flow of 0.25 mL/min transports the content of the interface tube to the IRMS.

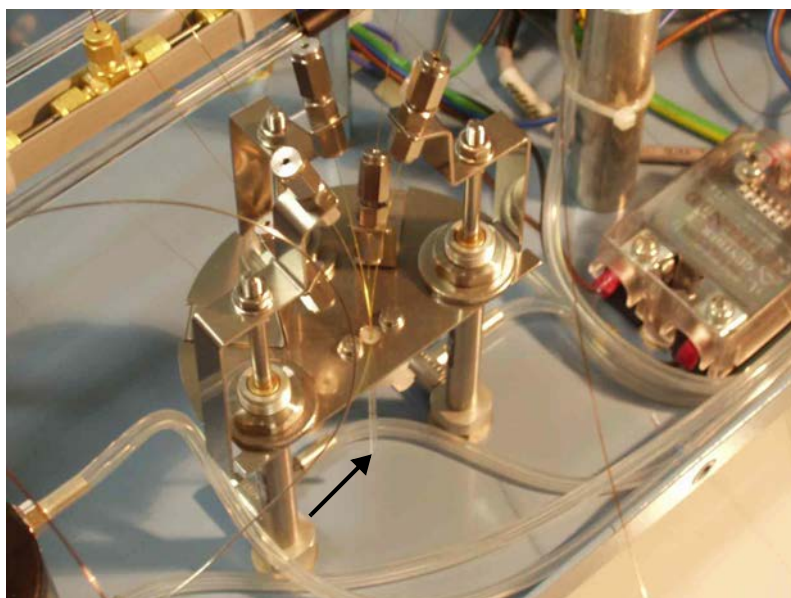


Figure 3-31. Reference inlet (open split)

Principle of Reference Gas Introduction

To inject a reference gas, the corresponding reference capillary moves to the bottom of the open split interface. See [Figure 3-32](#). The reference gas, CO₂ for example, is then mixed with the 4 mL/min He flow.

Now, 0.25 mL/min of this (He + CO₂) mixture is transferred to the IRMS resulting in a rectangular shaped reference gas pulse. The width of this pulse, 20 s for example, is defined by the time between injecting and removing the reference gas capillary.

NOTICE

No CO₂ or air must be visible in Instrument Control. Otherwise, the capillaries must be redesigned.

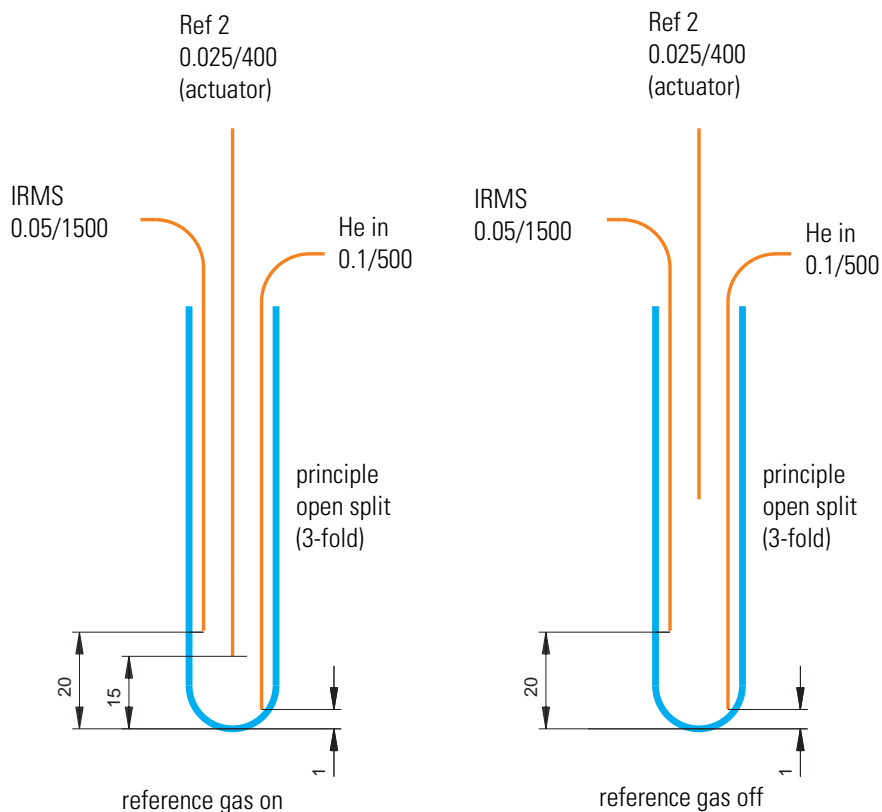


Figure 3-32. Principle of reference gas introduction

Sample Injection and Dilution

The sample injection (active open split) enables the injection of sample gas carried by helium flow into the IRMS. The sample gas is injected from below into a helium-purged mixing zone. Consequently, the gas pressure of the ion source is kept constant as defined by atmospheric pre-pressure, length and inner diameter of the capillary.

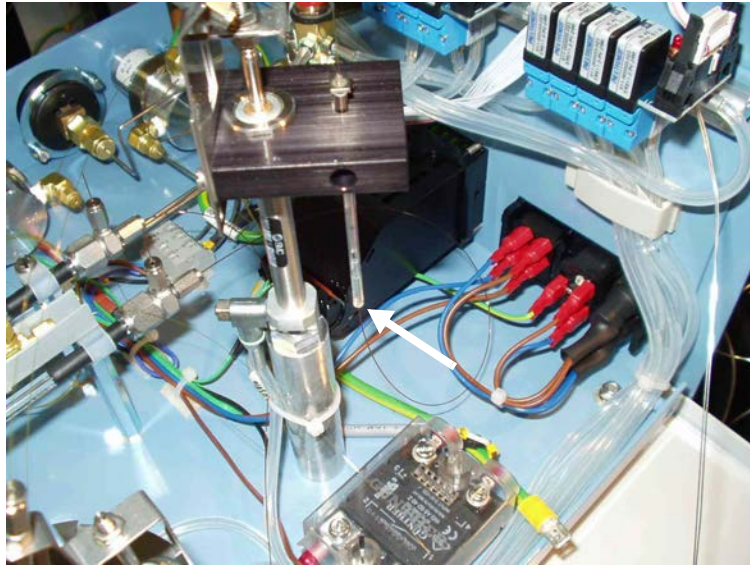


Figure 3-33. Sample inlet (open split)

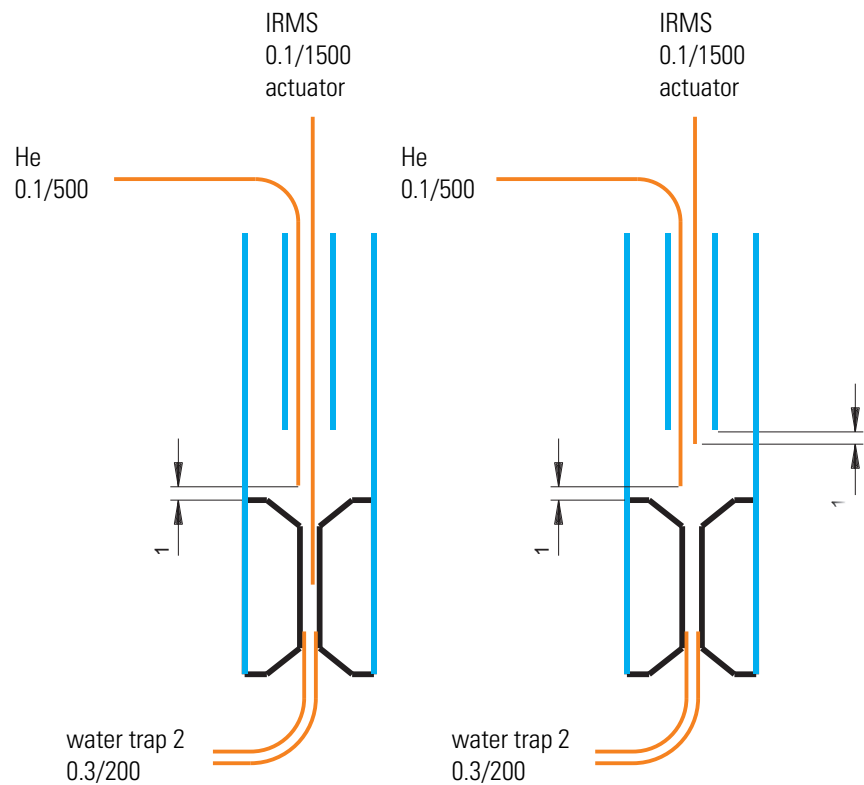


Figure 3-34. Sample injection and dilution

Principle of Active Open Split

The sample gas pressure is never ideally the same (for example, the amount of carbonates alternates). To be able to keep all CO₂ generated from different amounts of carbonates at a constant CO₂ pressure range, the active open split has been developed by Thermo Fisher Scientific.

Moving the IRMS capillary into the direct transfer connection (90–100%) or mixing zone (1:4) enables to dilute the sample gas concentration within the helium flow.

- left side of [Figure 3-34](#): no dilution
- right side of [Figure 3-34](#): dilution active

The transfer of the sample stream into the IRMS is achieved via the open split. The capillary that leaves the second water trap enters the open split interface as well as the retractable sampling capillary of the IRMS. A third capillary (protection capillary) delivers a constant stream of dry helium, which purges the exit volume of the open split at any time.

“IN” position

- The IRMS capillary is moved to the bottom of the open split.
- The IRMS capillary “sniffs” the sample stream eluted by the capillary that comes from the second water trap.

“OUT” position

- The gas from the protection capillary mixes with the sample flow.
- The IRMS capillary “sniffs” the diluted sample stream.

Tip In case of GC applications, the mass spectrometer capillary is completely decoupled from the GC.

In case of GasBench Plus applications, only partial decoupling occurs.

Hardware Components

Open Splits

Safety

This chapter contains information about machine safety. For your own safety, the safety of others and to prevent damage to the instrument, read this chapter carefully before installing, operating or coming into contact with the instrument and its accessories.

NOTICE

Refer to the *GasBench II Operating Manual* for GasBench II safety information.



To comply with safety and warranty requirements, the instrument and accessories described in this manual are designed to be used by properly trained personnel only.

Any installation, adjustment and repair of this equipment must be carried out only by a certified Thermo Fisher Scientific service representative who is aware of the hazards involved.





To protect our operating personnel, we ask you to adhere to special precautions when you send back parts to the factory for exchange or repair. See “Returning Parts” on page 13-9.

Contents

- [Safety Symbols and Signal Words in this Manual](#) on page 4-2
- [Safety Symbols on the Instrument](#) on page 4-2
- [Intended Use](#) on page 4-4
- [Handling Liquid Nitrogen](#) on page 4-7
- [Handling the Dewar Vessel](#) on page 4-12
- [Precautions for Handling of Gases](#) on page 4-14
- [Electric Safety Precautions](#) on page 4-14
- [Installing a Residual Current Device in the Laboratory Building](#) on page 4-17
- [Safety Instructions for Wearers of Medical Implants](#) on page 4-18
- [In Case of Emergency](#) on page 4-19
- [Residual Hazards](#) on page 4-20


Safety Symbols and Signal Words in this Manual

Notices concerning the safety of the personnel who operate the GasBench Plus device appear different from the main flow of text. Safety notices include the following:

	Always be aware of what to do with and the effect of safety information.
	Points out a hazardous situation that can lead to minor or medium injury if it is not avoided.
	Points out a hazardous situation that can lead to severe injury or death if it is not avoided.
	Points out a hazardous situation that will lead to severe injury or death if it is not avoided.

Observing this Manual

Always keep this manual near the instrument to have it available for quick reference.

	<p>Before you operate your instrument, read and understand all the safety information described in this manual.</p> <p>Be sure to read and comply with all precautions described in this manual.</p>
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System configurations and specifications in this manual supersede all previous information received by the purchaser.

Safety Symbols on the Instrument

Table 4-1 lists all safety labels on the instrument and their respective positions. See the indicated safety notices to prevent risk of harm to the operator and to protect the instrument against damage. If they are present, read and obey the instructions on the labels.

Table 4-1. Safety labels on the instrument




Label	Label description	Label position
	Hot Surface. Risk of Burn. See manual for instructions.	At the side of the housing of the GC oven. See Figure 13-2 .

Table 4-1. Safety labels on the instrument, continued

Label	Label description	Label position
	Hot Surface. Risk of Burn. See manual for instructions.	At the top of the housing of the GC oven. See Figure 13-2 .
	Read and fully understand operator's manual before you use this machine. Failure to follow the operating instructions could result in death or serious injury.	

If you use the Carbonate Option, see [“Safety Symbols on the Acid Pump”](#) on [page 9-3](#).

Refer to the safety regulations of the TriPlus RSH SMART autosamplers.

Name Plate

To correctly identify the instrument when you contact Thermo Fisher Scientific, always have the information of the name plate available. The name plate is attached to the left side panel. See [Figure 4-1](#) as an example.

The name plate contains the serial number, which is important in any type of communication with Thermo Fisher Scientific. Especially, the serial number is needed to access the SharePoint of the Bremen Technical Documentation group. See [“Contacting Us”](#) on [page 1-5](#).



Figure 4-1. Name plate of GasBench Plus – example

Figure 4-2 shows the name plate of the heated (that is, thermostatted) tray of the RSH.

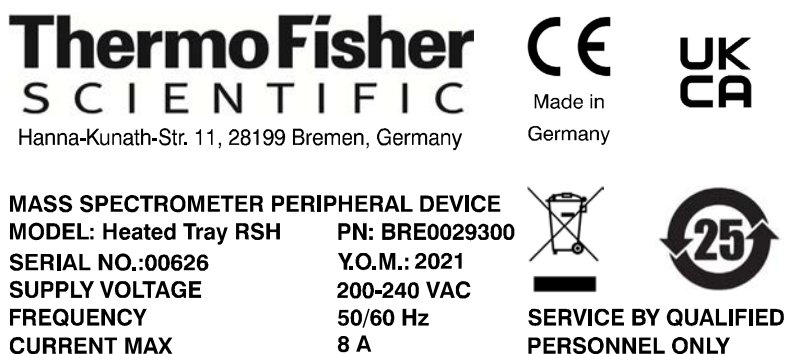


Figure 4-2. Name plate of heated tray of RSH – example

Intended Use

The GasBench Plus device is a universal on-line gas preparation and introduction system for Isotope Ratio MS.

The GasBench Plus device is attached to Thermo Scientific isotope ratio mass spectrometers and will be placed either on top of the IRMS or on a peripherals support table.



Obey the following guidelines when you operate your GasBench Plus instrument:

- The instrument is designed to be used in a laboratory. It is not designed for the use outdoors.
- The instrument is intended to be used by trained personnel within a laboratory environment for the purpose of chemical analysis. The instrument should not be used for any other purpose or within any other environment.
- The instrument is designed for use as general lab equipment. It is not designed for use in diagnostic or medical therapeutic procedures.
- If the instrument is used in a manner not specified by Thermo Fisher Scientific, the protection provided by the instrument could be impaired. Thermo Fisher Scientific assumes no responsibility and will not be liable for instrument damage and/or operator injury.

When the safety protection of the instrument has been compromised, disconnect the instrument from all power sources and secure the unit against unintended operation.

- Unintended use encompasses any use for the purpose other than those described, for example, operating the instrument at improper ambient conditions, use of improper accessories or spare parts.
- The instrument must not be used in an unspecified manner. Operate the instrument exclusively with the gases that are specified in this manual. Do not operate the instrument with combustible, corrosive, or poisonous gases.
- Operating the instrument requires the use of chemical substances having different hazard specifications. Before you use chemicals, read the hazard indications and information reported in the Material Safety Data Sheet (MSDS) supplied by the manufacturer referring to the relevant CAS (Chemical Abstract Service) number.

Notice on the Susceptibility to Electromagnetic Transmissions

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

Notice on Lifting and Handling of Thermo Scientific Instruments

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

Qualification of the Personnel

The primary audience for this manual consists of analytical chemists and laboratory technicians. To use this manual effectively, you should have a basic knowledge of chemistry, a basic knowledge of electronic sampling equipment, at least a beginning level of computer experience, and working knowledge of the analytical instrument.

The personnel must be trained to handle liquid nitrogen in pressurized and non-pressurized containers.



Personnel that install or operate the instrument must have the following qualifications:

- **Electrical Connections**

The electrical installation must be carried out by qualified and skilled personnel (electrician) according to the appropriate regulations (cable cross-sections, fuses, earth grounding connection, for example). Refer to the *Gas Isotope Ratio MS Pre-Installation Requirements Guide* for the specifications.

- **Installation**

Only certified Thermo Fisher Scientific service representatives or personnel who act on behalf of Thermo Fisher Scientific are allowed to install the instrument.

- **General Operation**

The instrument is designed to be operated by qualified laboratory personnel. Before they start, all users must be instructed about the hazards presented by the instrument and the chemicals applied. The users must be advised to read and obey the relevant Material Safety Data Sheets (MSDSs).

- **Decommissioning the system**

When you do not plan to use the system for an extended period, you have to decommission it. See also “[WEEE Compliance](#)” on [page A-2](#).

Only qualified employees of Thermo Fisher Scientific or qualified personnel who act on behalf of Thermo Fisher Scientific are allowed to decommission the instrument.

- **Disposal of the system**

It is your responsibility to dispose of contaminated parts complying with legal regulations.

Only qualified employees of Thermo Fisher Scientific or qualified personnel who act on behalf of Thermo Fisher Scientific are allowed to dispose of the instrument.

Key Operator

Experience has shown that the maximum benefit can be derived from a scientific instrument if there is one person, a key operator, who has major responsibility for that instrument. Thermo Fisher Scientific recommends that you designate a key operator to oversee the operation and maintenance of the system in your laboratory. The key operator should be available to the installing engineer throughout the installation. This person will also be the key figure in the communication between your laboratory and Thermo Fisher Scientific.

Permitted Materials

The GasBench Plus instrument is designed to be operated with the materials listed in [Table 4-2](#). Depending on the installed options, additional materials may be necessary. See the relevant chapters of this manual for details.

Table 4-2. Materials and their use

Material	Used
Gases	
Argon	for leak checking
Carbon dioxide	as reference gas and samples gas
Compressed air	to actuate valves
Helium	as reference gas and samples gas as carrier gas/flush gas
Hydrogen	as reference gas and samples gas
Chemicals	
Liquid nitrogen	for the liquid nitrogen-cooled traps
Orthophosphoric acid	for preparing phosphoric acid
Phosphorus pentoxide	for preparing phosphoric acid
Solvents	
Deionized water	for preparing phosphorus acid and as cleaning agent
Acetone	for cleaning vials and weighing instruments
Others	
Calibration compounds	
Samples	

Handling Liquid Nitrogen

When handling (filling and emptying, transporting, operating, etc.) liquid nitrogen, do the following:

- Wear protective gloves, goggles and protective clothing.
- Be well-informed about the risks.
- Be able to handle liquid nitrogen safely.

Safety

Handling Liquid Nitrogen

- Pay attention to the precautions given in this section to minimize the risk of accidents.
- Pay attention to the internal in-house safety guidelines as well as the guidelines of the Employers' Liability Insurance Association.

Site Requirements for Handling Liquid Nitrogen

- The laboratory must allow safe handling of liquid nitrogen (filling, inspecting, cleaning and maintenance of the Dewar vessel, for example) by the user.
- The laboratory must be equipped with a ventilation system of sufficient capacity that runs permanently when liquid nitrogen is handled (especially when decanting or refilling it).
- The laboratory must be prevented against entry of unauthorized persons.
- The floor must be level and not porous.
- The Materials and Safety Data Sheets (MSDSs) of liquid nitrogen must be easily accessible for the user.

Risks when Handling Liquid Nitrogen

Risks related to handling liquid nitrogen are:

- Cryogenic burns
- Fire and/or explosion
- Oxygen deficiency (suffocation)
- Oxygen accumulation

Cryogenic Burns

CAUTION

Cold Liquid. Risk of frostbite. Liquid nitrogen may flow out if the Dewar vessel is tilted or upset. Liquid nitrogen may be spilled or spout if the Dewar vessel is shaken.

Be careful when carrying the Dewar vessel, for example when inserting it into the cold trap assembly. Wear safety goggles, cryogenic gloves and protective clothing when handling cryogenically cooled samples or when decanting liquid nitrogen.

Liquid nitrogen is extremely cold (-196 °C). Liquid nitrogen (directly) or surfaces of containers that have been exposed to liquid nitrogen (indirectly) may cause burns when coming into contact with the skin.

Liquid nitrogen may make materials brittle that are not suitable for low temperatures. Depending on the humidity of the air, liquid nitrogen may cause strong fog formation. Cryogenic burns may be caused either by direct contact or by splashes of liquid nitrogen.

To prevent cryogenic burns

- Always wear individual protective equipment (suitable gloves, safety goggles, eye and face protection, safety shoes).
- Use only suitable equipment to refill the Dewar vessel (hose made of metal or PTFE).
- Keep the laboratory personnel well trained.
- Keep the Dewar vessel vertically upright.
- Do not expose your skin to liquid nitrogen.
- Do not touch surfaces or objects that have been exposed to liquid nitrogen (the inner walls of the Dewar vessel, uninsulated or cooled equipment, for example).

If splashes of liquid nitrogen have hit your *eyes*, rinse them abundantly with water for at least 15 minutes, consult a physician and pay attention to the in-house instructions in case of an emergency.

If splashes of liquid nitrogen have hit your *skin*, do not rub across the contaminated area and do not apply anything to it, loosen or remove your clothes (if possible), consult a physician and pay attention to the in-house instructions in case of an emergency.

Fire and/or Explosion



Fire and Explosion Hazard. Avoid accumulation of oxygen in the Dewar vessel as this increases the risk of fire and explosion.

Evaporating liquid nitrogen may produce excess pressure in recipients if they are closed. Reasons for the excess pressure may be the use of a tightly closing lid (without an evaporation hole) as well as ice formation at the neck or lid.

To prevent an explosion

- Use a suitable lid, with an evaporation hole.
- Avoid too high filling levels in the Dewar vessel as this prevents ice formation at its neck and lid.
- Use the Dewar vessel only in-house, in laboratory rooms.

Safety

Handling Liquid Nitrogen

- Monitor the humidity in the laboratory: avoid too high humidity levels.
- Regularly check the Dewar vessel for accumulated water condensation.
- Regularly check the Dewar vessel for damages of the glassy surface, as scratches, cracks, dents and flaws.

Oxygen Deficiency (Suffocation)



Suffocation Hazard. Nitrogen might cause suffocation if it accumulates in the laboratory due to a lack of oxygen in ambient air.

If the extraction is turned off, sufficient air must be supplied to prevent the nitrogen concentration from reaching a harmful level. Ensure that the laboratory is well ventilated.

The atmospheric gases (about 78% nitrogen, 21% oxygen, 1% argon, trace gases) are not toxic, but changes of their concentrations, especially of the oxygen concentration, markedly affect biochemical processes. In particular, inhaled air must contain sufficient oxygen (above 19%). As the gases are colorless and odorless, human senses cannot detect changes in their concentrations sufficiently quickly.

Oxygen replacement by nitrogen may decrease the oxygen concentration below the critical threshold value, especially in closed rooms without appropriate ventilation. This oxygen deficiency involves the risk of suffocation. Reasons for oxygen replacement may be:

- Normal evaporation of liquid nitrogen to nitrogen gas
- Decanting or refilling liquid nitrogen
- Leakage at containers used for liquid or gaseous nitrogen
- Overturning the Dewar vessel or other nitrogen containers
- Defective air supply or air suction

To prevent oxygen deficiency (suffocation)

- Always wear individual protective equipment (suitable gloves, safety goggles or eye and face protection, safety shoes).
- Keep the laboratory personnel well trained.
- Always carry an oxygen measurement device.
- Continuously monitor the oxygen concentration.
- Keep the Dewar vessel vertically upright.

- Close the Dewar vessel with a suitable isolating lid.
- Keep the Dewar vessel away from direct sunlight and heat sources.
- Do not transport the Dewar vessel in vehicles or over stairs. Use a lift instead. Do not ride on the same lift as the filled Dewar vessel. Operate the lift from outside. Take precautions that no one else enters the lift.
- Ensure permanent and sufficient ventilation of the laboratory rooms where the Dewar vessel is used.
- Prevent the Dewar vessel from strokes, shocks and rapid movements.

If an accident with liquid nitrogen has happened

- Mark and secure the surroundings to avoid secondary accidents.
- Pay attention to the in-house instructions in case of an emergency.
- Act quickly and deliberately. Take measures for self-protection.
- Carefully and quickly move injured people out of the area of risk.
- Ventilate the affected rooms sufficiently and permanently.

Oxygen Accumulation

When handling liquid nitrogen, oxygen may condense from ambient air and become liquid as well, because the boiling point of oxygen (-183 °C) is above that of nitrogen (-196 °C). Liquid oxygen will accumulate at the bottom of the Dewar vessel in this case.



Fire and Explosion Hazard. Avoid accumulation of oxygen in the Dewar vessel as this increases the risk of fire and explosion.

❖ To avoid oxygen accumulation

- Always wear individual protective equipment (suitable gloves, safety goggles or eye and face protection, safety shoes).
- Keep the laboratory personnel well trained.
- Do not smoke.
- Remove any source of fire (light, open fire, matches, lighters, sparking materials, for example).
- Keep highly flammable materials away from the Dewar vessel. Do not clean Dewar vessels using flammable liquids such as Ethanol.
- Clean the floor regularly.

Handling the Dewar Vessel

The inner container of the Dewar vessel consists of borosilicate glass. It is suitable to store coolants or liquids in the range between -196 °C and +150 °C, as for example liquid nitrogen.

Before handling the Dewar vessel, thoroughly inspect it for damages of the glassy surface, as scratches, cracks, dents and flaws. Do not use the Dewar vessel, if its glassy surface is damaged until it is repaired, because it may implode due to mechanic or thermal strain.

Filling the Dewar Vessel

CAUTION

Cold Liquid. Risk of frostbite. Liquid nitrogen may flow out if the Dewar vessel is tilted or upset. Liquid nitrogen may be spilled or spout if the Dewar vessel is shaken.

Be careful when carrying the Dewar vessel, for example when inserting it into the cold trap assembly. Wear safety goggles, cryogenic gloves and protective clothing when handling cryogenically cooled samples or when decanting liquid nitrogen.

❖ **To fill the Dewar vessel with liquid nitrogen**

1. Carefully insert a filling hose with a phase separator into the Dewar vessel.

When you alternatively use a filling funnel that is positioned on the neck of the Dewar vessel, carefully insert the filling hose into the funnel.

2. Ensure that the hose is lying steadily and safely in the neck of the Dewar vessel during the entire filling process.
3. To avoid that hose or phase separator touch and damage the glassy wall of the Dewar vessel, secure the hose.
4. Take care that the filling pressure in the hose does not exceed 1.5 bar (150 kPa).

Transporting the Filled Dewar Vessel

When transporting the filled Dewar vessel, do the following:

- Always position the lid loosely fitted on the Dewar vessel when it contains liquid nitrogen. The slot in the lid avoids creation of excess pressure due to evaporation.
- Transport and operate the filled Dewar vessel only in-house.

- Grasp the Dewar vessel at its handle. Avoid swinging it during transport because liquid nitrogen may be spilled or spout.
- Transport and operate the Dewar vessel only in a steady, upright position.
- Do not transport the filled Dewar vessel over stairs as they act as a tripping hazard. In case of stumbling, liquid nitrogen may be spilled or spout. Use a well ventilated lift instead.
- Avoid shocks or other mechanic influences on the Dewar vessel to minimize evaporation of liquid nitrogen and to increase the lifetime of the borosilicate glass.

Decanting Liquid Nitrogen

CAUTION

Cold Liquid. Risk of frostbite. Liquid nitrogen may flow out if the Dewar vessel is tilted or upset. Liquid nitrogen may be spilled or spout if the Dewar vessel is shaken.

Be careful when carrying the Dewar vessel, for example when inserting it into the cold trap assembly. Wear safety goggles, cryogenic gloves and protective clothing when handling cryogenically cooled samples or when decanting liquid nitrogen.

❖ **To decant liquid nitrogen**

1. Place the Dewar vessel securely on a workbench, not too close to its edges.
2. Remove the lid of the Dewar vessel.
3. With one hand, grasp the handle of the Dewar vessel.
4. Carefully raise the Dewar vessel straight upright, without swinging it.
5. With the other hand, grasp the handle edge located on the base of the protective shell.

This facilitates handling and decanting the liquid nitrogen safely.

6. Carefully tip the Dewar vessel and slowly decant the liquid nitrogen.

Precautions for Handling of Gases



Before using gases, carefully read the hazard indications and information in the Material Safety Data Sheet (MSDS) supplied by the manufacturer referring to the CAS (Chemical Abstract Service) number. It is your responsibility to ensure compliance with all local safety regulations for the use of gases.

Precaution for Helium

Helium is a nontoxic, odorless, colorless, nonflammable gas stored in cylinders at high pressure. It can cause rapid suffocation when concentrations are sufficient to reduce oxygen levels below 19.5%. It is lighter than air and may collect in high points or along ceilings.

Precaution for Oxygen

Oxygen is an odorless, colorless, nonflammable gas stored in cylinders at high pressure. It is an oxidizing gas and vigorously accelerates combustion. Keep away from oils or grease. Rescue personnel should be aware of the extreme fire hazards associated with oxygen-enriched (greater than 23%) atmospheres.

Precaution for Hydrogen

Hydrogen is a colorless, odorless, highly flammable gas. The use of hydrogen requires the operator's strict attention and compliance with special precautions due to the hazards involved. Hydrogen is a dangerous gas, particularly in an enclosed area when it reaches a concentration corresponding to its lower explosion level (4% in volume). When mixed with air it can create an explosive mixture.

Precaution for Carbon Monoxide

Carbon monoxide is a colorless gas. May be fatal if inhaled. Causes severe respiratory tract. Use only with adequate ventilation.

Electric Safety Precautions



High Voltage. High voltages capable of causing an electric shock and personal injury are used in the instrument.

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

- The instrument must be shut down and disconnected from line power before service is performed.

- 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 chassis of the instrument to ensure safe operation.

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

- Wrong usage of the ports might endanger personnel. Read and understand this Operating Manual to prevent risk of harm to the operator and to protect equipment against damage.
- Do not run the system without the housing on. Opening the instrument housing is only allowed for maintenance purposes as described in this manual (and after pulling out the mains plug) or by Thermo Fisher Scientific personnel. Do not remove protective covers from PCBs. Permanent damage or an electric shock can occur.

When you leave the system, make sure that all protective covers and doors are properly connected and closed, and that heated areas are separated and labeled to protect unqualified personnel.

Do not rig or override any safety switches or safety functions. Risk of electric shock, burn hazard or damage to your system can occur.

- Do not turn on the instrument if you suspect that any kind of electrical damage has incurred. Disconnect the power cords and contact a Thermo Fisher Scientific field service engineer for an evaluation.

Do not try to use the instrument until it has been evaluated. Electrical damage might have occurred if the system shows visible signs of damage or has been transported under severe stress.

- Do not place any objects upon the instrument—especially not containers with liquids—unless it is requested by the user documentation. Leaking liquids might get into contact with electronic components and cause a short circuit.
- If liquid is spilled on or adjacent to the instrument, immediately isolate the instrument and the accessories from the electrical supply by turning off the power remote to the instrumentation.

 **CAUTION**

Tripping Hazard. Lay electric feed lines so that there is no risk of tripping over these.

Safety Protection Provided by the Instrument

The GasBench Plus device provides protection according to several isolation and safety standards for electronic instruments. See the “[EU Declaration of Conformity](#)” on [page A-3](#) for information about the relevant standards.

IEC Degree of Protection: IP20

The GasBench Plus device is resistant to solid foreign bodies from 12.5 mm in diameter (accidental finger contact), but it is not protected against the ingress of water.

IEC Protection Class: I

The GasBench Plus device has a protective ground connection and a basic insulation between accessible parts and the protective earth. A proper protective ground connection is compulsory.

IEC Overvoltage Category: II

The GasBench Plus device has a plug and socket connector or a fixed connection supplied from the electrical system of a building.

IEC Pollution Degree: 2

The GasBench Plus device must be used in an environment where only non-conductive pollution occurs. Occasionally, temporary conductivity caused by condensation is to be expected.

Impaired Safety Protection

When the safety protection has been impaired, the instrument and the accessories must be made inoperative and secured against any unintended operation.

The matter should then be referred to the local Thermo Fisher Scientific service organization. Safety protection is likely to be impaired, if the instrument fails to operate normally or shows visible damage. If the equipment is used in a manner that is not specified by the manufacturer, the safety protection provided by the equipment might be impaired.

Installing a Residual Current Device in the Laboratory Building

WARNING

Risk of Electric Shock in case of an Instrument driven with Cooling Water. Under extremely rare conditions, the cooling water supply inside the instrument might become leaky and water might come into contact with the mains voltage.

The leakage of water could endanger personnel when they come into contact with the water and could lead to a life-endangering electric shock in the worst case scenario.

If water is leaking from the instrument, do not touch the water or step into it. Do not touch the instrument except for the power off switch. Switch off the instrument immediately. Contact the local Thermo Fisher Scientific service or your local distributor.

Thermo Fisher Scientific recommends installing a residual current device (RCD) in the laboratory building.

A residual current device is also called Ground Fault Circuit Interrupter (GFCI). When it senses an imbalance between the outgoing and incoming current, this device instantly breaks the electric circuit to prevent serious harm from an outgoing electric shock. For further details, refer to the Technical Information of the residual current device.

NOTICE

For the installation of an RCD in the building, the local installation requirements must be followed.

The instrument must be protected by an RCD with a tripping current of maximum 30 mA. This can be accomplished by installing an RCD in the laboratory building that fuses the power socket of the instrument. If this is not possible, Thermo Fisher Scientific can alternatively provide an in-line RCD box. In this case, contact your local service office.

If an insulating type transformer, insulating type UPS or insulating type power conditioner is used, the RCD must be connected between the insulating device and the instrument. The in-line RCD box can be used here as well:

- in-line RCD box for North America
- in-line RCD box for other countries

Safety Instructions for Wearers of Medical Implants



HF/RF magnetic field emitting instruments may influence active and passive body implants, such as pacemakers, infusion pumps, hip joint replacements, etc.



If you are wearing a cardiac pacemaker, an implanted heart defibrillator or medical implants that might contain ferromagnetic materials (clips or prostheses, for example), read and follow these safety instructions

WARNING

Magnetic and Electromagnetic Radiation. The electromagnet might affect the function of cardiac pacemakers, implanted heart defibrillators and medical implants that might contain ferromagnetic materials (clips or prostheses, for example). If you wear these devices, keep a sufficient safety distance to the magnet.

Parts of a pump or a gauge emit electromagnetic radiation. This radiation can interfere with the operation of cardiac pacemakers and implanted heart defibrillators, possibly causing death or serious injury. If you wear these devices, keep a sufficient safety distance to the pump or gauge.

Current static magnetic field guidelines restrict exposures for wearers of cardiac pacemakers to 0.5 mT. To avoid exposure to stray fields, restrict access to personnel with pacemakers or other implanted electronic devices to a minimum distance from the edge of the magnet coil assemblies.



Medical implants (clips or prostheses, for example) that may contain ferromagnetic materials, would be subject to strong attractive forces near to the magnet. All people with such implants should be excluded from the laboratory rooms and appropriate warning signs be displayed.



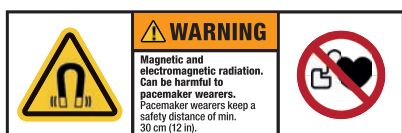
Strong magnetic fields might affect the operation of electronic components. They can cause permanent damage of magnetically sensitive devices.

Strong attractive forces can cause metal objects to move uncontrollably towards them. Do not bring compressed gas cylinders, tools, or other metal objects near to the magnet. Keep computers, credit cards and other magnetically sensitive devices sufficiently far away from the instrument.

To avoid hazards from flying metal objects, do not insert items, such as tools made of magnetically permeable material, in the gap between upper and lower magnet coil assemblies when the magnet circuit is switched on.



Read the Operating Manual of your instrument to get comprehensive information about the specific hazards that are presented by it. The manual also provides information about the necessary countermeasures (safety distances, for example).



If not stated otherwise in the Operating Manual, carriers of active implants must keep a safety distance of at least 30 cm to the magnet, magnet coil or pump.

In Case of Emergency

The emergency shutdown of the mass spectrometer should not be considered as a standard procedure. For the regular system shutdown, refer to the *DELTA Q Operating Manual*.



To allow shutting off the instrument in an emergency, make sure that you can get access to the main power switch at any time. The main power switch is located at the rear side of the instrument.

❖ To shut down the system in case of emergency

1. Turn the main switch of the IRMS to the **Off** position.

All power to the mass spectrometer, including the internal vacuum pumps, is shut off.

2. Pull the mains plug out of the power socket.
3. Switch off the computer by pressing the On/Off switch.



Electric Current. Electric shock hazard. Capacitors inside the instrument might still be charged, even if the instrument is turned off.

4. Close the compressed air supply.
5. Switch off all peripherals.
6. Close all gas supplies, except the helium supply.
7. Close the liquid nitrogen supply.
8. After about two hours, close the helium supply.
9. Secure the mains switch of the IRMS against being switched on by using a lock. Fill in and sign the card.
10. Inform your local service organization.

Residual Hazards

Users of the GasBench Plus device must pay attention to the following residual hazards.

 **WARNING**

Explosion Hazard. Hydrogen forms explosive mixtures with air. A leak in the hydrogen (H₂) supply might cause a fire or an explosion.

 **WARNING**

High Voltage. High voltages capable of creating an electric shock are used in the instrument. Do not remove protective covers from PCBs. Opening the instrument housing is only allowed for maintenance purposes by Thermo Fisher Scientific personnel.

To ensure that the instrument is free from all electric current, always disconnect the power cord before you try any type of maintenance.

 **WARNING**

Corrosive Chemicals. Phosphoric acid causes severe skin burns and eye damage. Wear protective clothing, protective gloves, and a face plate when you handle phosphoric acid. Goggles are not sufficient.

Wear protective clothing, protective gloves and a face mask when you handle phosphorous pentoxide. Goggles are not sufficient.

Also contain waste streams and use proper ventilation. Operate under a fume hood. Operate according to the Material Safety Data Sheet (MSDS).

As phosphoric acid is not a very strong acid, contamination of personnel by touching parts might not be noticed immediately, but delayed (contamination of fingers by droplets, passing to the eyes later on, for example).

⚠ CAUTION

All gas lines must be free of oil. Gas containers must be properly secured to avoid damages at the electric cables and gas lines. Strictly observe the applicable national and international safety regulations regarding the storage and transport of pressurized gas.

⚠ CAUTION

Sharp Edges. Risk of cuts. Follow appropriate care and safety procedures to avoid breaking any glassware or capillaries and causing injury to the operator. Handle any broken glassware or capillaries with appropriate care and wear protective gloves and goggles.

⚠ CAUTION

Hazardous Chemicals. If potentially hazardous chemicals or solvents are used in procedures throughout this Operating Manual:

- Careless handling of hazardous chemicals might cause severe personal injury. Avoid exposure to potentially harmful materials.
- Samples, solvents and containers might contain toxic, carcinogenic, mutagenic, corrosive, or irritant chemicals. Always wear protective clothing, gloves, and safety glasses when you handle solvents, corrosives, containers or samples.
- Store and handle all chemicals in accordance with standard safety procedures. Contain waste streams and use proper ventilation. Use approved containers and procedures for disposal of waste solution.
- Read and obey the Material Safety Data Sheet (MSDS) of your suppliers for a description of the specific hazards and proper handling of each compound you use:

Material Safety Data Sheets provide summarized information on the hazard and toxicity of specific chemical compounds. The MSDS also provides information on the proper handling of compounds, first aid for accidental exposure, and procedures for the remedy of spills or leaks.

The Material Safety Data Sheets describing the chemicals being used need to be freely available to laboratory personnel for them to examine at any time. Producers and suppliers of chemical compounds are required by law to provide their customers with the most current health and safety information in the form of an MSDS.

NOTICE

To ensure safety and proper cooling, always operate the instrument with its covers in place. This is also necessary to comply with product safety and electromagnetic interference regulations.

NOTICE

Always use original Thermo Fisher Scientific materials and products. The use of materials that do not meet the technical specifications of our products does not ensure good operation of the instrument and may even cause damage to it.



Service by the customer must be performed by trained qualified personnel only and is restricted to servicing mechanical parts. Service on electronic parts must be performed by Thermo Fisher Scientific field service engineers only.

Do not try to repair or replace any component of the system that is not described in this manual without the assistance of your Thermo Fisher Scientific field service engineer.

Personal Protective Equipment

Appropriate safety clothing must be worn at all times while operating or maintaining the instrument, particularly when handling hazardous materials.

This manual can only give general suggestions for personal protective equipment (PPE), which protects the wearer from hazardous substances. Refer to the Material Safety Data Sheets (MSDSs) of the chemicals handled in your laboratory for advice on specific hazards or additional equipment.

Eye Protection

The type of eye protection required depends on the hazard. For most situations, safety glasses with side shields are adequate. Where there is a risk of splashing chemicals, goggles are required.

Protective Clothing

When the possibility of chemical contamination exists, protective clothing that resists physical and chemical hazards should be worn over street clothes. Lab coats are appropriate for minor chemical splashes and solids contamination, whereas plastic or rubber aprons are best for protection from corrosive or irritating liquids.

Gloves

For handling many chemical compounds, Thermo Fisher Scientific recommends white nitrile clean room gloves from [Fisher Scientific](#) or [Unity Lab Services](#).

For handling organic solvents and acids however, nitrile gloves may not be sufficient. Strictly adhere to the respective MSDS.

For handling hot objects, gloves made of heat-resistant materials (leather, for example) should be available.

For handling liquid nitrogen, appropriate cryogenic gloves for the temperature range of your field of work are required.

Safety Boots and Shoes

Safety boots and shoes, which are penetration-resistant and feature protective toecaps, can protect operating personnel from falling objects, vehicles and other heavy loads, for example when moving the instrument.

Wear them whenever you perform operation or maintenance procedures on the instrument and whenever you lift it.

Safety

Residual Hazards

Installation

This chapter describes the conditions for an operating environment that will ensure continued high performance of your GasBench Plus device.

NOTICE

Refer to the *GasBench II Operating Manual* for the GasBench II installation.

To be sure that your laboratory is ready for the installation of the GasBench Plus device, you have to meet all requirements specified in the *Gas Isotope Ratio MS Pre-Installation Requirements Guide*. This guide also provides comprehensive information to assist in planning and preparing your lab site.

NOTICE

Do not remove items from the shipping containers—the service engineer unpacks, inspects, and installs the system.

After arrival, the instrument must be stored as packed by Thermo Fisher Scientific for at least 24 hours at an environmental condition of 15–35 °C and a relative humidity of 20–80% (< 30 °C ambient) or 20–60% (≥ 30 °C ambient) before connecting it to the power supply.



To comply with safety and warranty requirements, the instrument and accessories described in this manual are designed to be used by properly trained personnel only.

Any installation, adjustment and repair of this equipment must be carried out only by a certified Thermo Fisher Scientific service representative who is aware of the hazards involved.

Contents

- [Placing the Instrument](#) on [page 5-2](#)
- [Laboratory Conditions](#) on [page 5-4](#)
- [Installing the Autosampler](#) on [page 5-15](#)
- [Installing an RSH for GasBench](#) on [page 5-17](#)
- [Tools for the TriPlus RSH SMART for GasBench Plus Standard and Advanced Autosamplers](#) on [page 5-41](#)
- [Installing a CTC Pal 80 Autosampler for GasBench II](#) on [page 5-51](#)

NOTICE

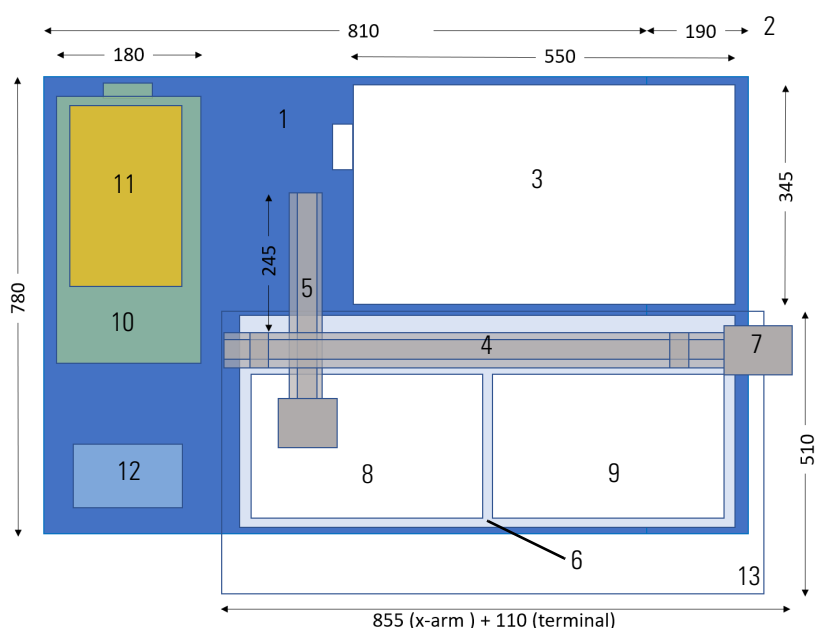
The installation of the CTC Pal 80 A200S autosampler is described in “[Installing a CTC Pal 80 Autosampler for GasBench II](#)” on [page 5-51](#).

Placing the Instrument

This section provides information that helps you positioning the instrument in the laboratory.

Instrument Dimensions

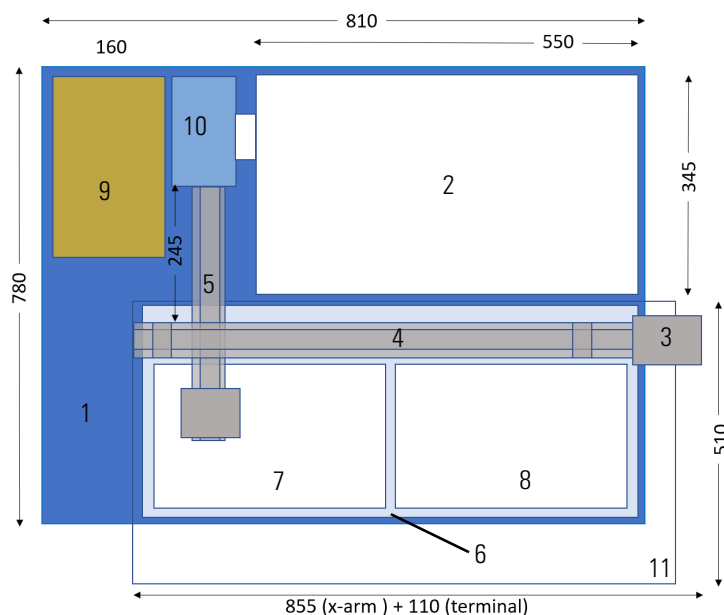
The GasBench Plus device is attached to a Thermo Scientific isotope ratio mass spectrometer (DELTA Q MS, 253 Plus™ MS). It is placed either on top of the IRMS or on a peripherals support table. The space required is 810 (+190) mm width × 780 mm depth. See [Figure 5-1](#) for GasBench Plus with ConFlo IV and [Figure 5-2](#) for GasBench Plus without ConFlo IV.



Labeled Components: 1=DELTA Q Series, 2=side panel, 3=GasBench Plus main module, 4=TriPlus RSH SMART (x-arm), 5=TriPlus RSH SMART (y-arm), 6=rack mounting plate, 7=TriPlus RSH terminal, 8=Slot1 & tray, 9=Slot2 & tray, 10=ConFlo IV, 11=temperature controller (thermostatted tray), 12=acid pump, 13=rack shield

Figure 5-1. Site requirements of GasBench Plus device with ConFlo IV^a

^a Dimensions given in mm



Labeled Components: 1=DELTA Q Series, 2=GasBench Plus main module, 3=TriPlus RSH terminal, 4=TriPlus RSH SMART (x-arm), 5=TriPlus RSH SMART (y-arm), 6=rack mounting plate, 7=Slot1 & tray, 8=Slot2 & tray, 9=temperature controller (thermostatted tray), 10=acid pump, 11=rack shield

Figure 5-2. Site requirements of GasBench Plus device w/o ConFlo IV^a

^a Dimensions given in mm

Both figures show symbolic areas for different instruments and peripherals. Their dimensions are listed in [Table 5-1](#).

Table 5-1. Space requirements for instruments and peripherals (mm)

Part	Width (horizontal)	Length (vertical)
DELTA Q	810	780
Side Panel	190	780
GasBench Plus (main module)	550	345
TriPlus RSH SMART (x-arm)	855 + 110 (terminal)	
TriPlus RSH SMART (y-arm), protruding from x-arm back side		245
Slot1 & Tray / Slot2 & Tray	345	345
Rack Mounting Plate	710	400
Rack Shield	915	510
ConFlo IV	180	470

Table 5-1. Space requirements for instruments and peripherals (mm),

Part	Width (horizontal)	Length (vertical)
Temperature Controller	160	265
Acid pump	150	100

Moving the Instrument

CAUTION

Heavy Load. Because of its weight, handling the instrument alone might cause muscle strain and back injury. Lifting and moving the instrument requires the effort of two persons to keep the individual load within acceptable limits (maximum 40 kg for men or 15 kg for women for a duration of 5 seconds).

The carriers must be trained in how to carry loads properly (for example, by rising from the knees with a straight back). Thermo Fisher Scientific recommends using a pallet jack to lift the instrument onto the workbench.

Laboratory Conditions

This section gives an overview of important requirements for the laboratory where the GasBench Plus instrument is placed. For details, refer to the *Gas Isotope Ratio MS Pre-Installation Requirements Guide*.

Site Services

This section lists the services that the GasBench Plus instrument requires to operate successfully. Depending on the installed options, additional site services may be necessary. See the relevant chapters of this manual.

Power Supply

Electric power to the GasBench Plus device is supplied by the IRMS line distributor (230 V, single phase, 3.15 A), which increases the total IRMS power consumption by 0.5 kW.

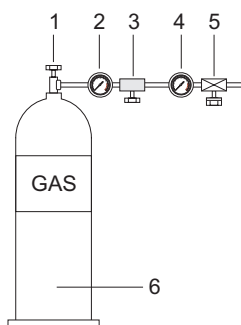
Gas Supply

For all applications, helium is needed as carrier gas. Its purity should be at least 99.999% He. We recommend using a second cylinder switchover to prevent pressure loss during overnight operation and contamination by atmospheric gases during bottle change. A standard 50 L gas tank has a lifetime of half a year in continuous operation.

For all applications with CO₂ as molecule of interest (that is water equilibration, DIC, or carbonates), CO₂ having a purity of 99.995% CO₂ is recommended as reference gas. A 40 L tank will last longer than one year in continuous operation.

In case of CO₂ water equilibration, additionally a mixture of CO₂ in He is needed for headspace flushing. The purities are recommended to be as stated above for He and CO₂ respectively. A CO₂ content of 0.3% leads to an ideal signal height of 9 V. In case of H/D measurements, H₂ is needed as reference gas. Its purity should be 99.999% H₂ (5.0, --200‰ vs. SMOW; not lower than -300‰ vs. SMOW). In case of headspace flushing, a mixture of 4% H₂ in He should result in a signal height of 9 V, which is optimal with regard to error margins.

Tip The pressure of new gas tanks is up to 200 bar (helium tank). The pressure must be adjusted to approximately 4 bar using the pressure regulator mounted at the gas tank.



Labeled Components: 1=main valve, 2=manometer 200 bar (He), for pre-pressure, 3=line pressure regulator, 4=manometer 4 bar (He), 5=on/off valve, 6=high pressure gas tank

Figure 5-3. Gas tank with regulators

NOTICE

All stainless steel gas lines should be oil-free and preferably flame-dried. The connectors can be made either of stainless steel or brass. The gas lines, or gas tanks respectively, should be at a distance of 1–1.5 m to the instrument.

All regulators should be oil- and fat-free and be specified for gases of high purity. The supply lines should terminate with 1/8 inch male Swagelok™-type connectors.

Gas Requirements of the GasBench Plus Device

The gases listed in [Table 5-2](#) are required for installing the GasBench Plus device by a Thermo Fisher Scientific service engineer.

Table 5-2. Gas requirements of GasBench Plus device

Purity	Pressure and usage
He 5.0 (that is 99.999%)	2.5 bar as carrier gas 4 bar as flush gas (to flush sample vials)
Tip Thermo Fisher Scientific recommends installing a high capacity purifier to ensure constant and affordable high quality of the helium carrier gas.	
He 4.6 with 0.3% CO ₂ 4.5 or He 4.6 with 0.5% CO ₂ 4.5	2.5 bar for acceptance tests
CO ₂	as reference gas (2.5 bar)
H ₂	

Tip Sometimes, it may be necessary to check the unit for leaks. Use an argon bottle for this.

Gas Requirements for Options

The following tables summarize the gas requirements for the various options.

Table 5-3. Gas requirements for water equilibration

Analysis	Purity	Pressure	Usage
¹⁸ O/ ¹⁶ O	He 4.6 with 0.3–1% CO ₂ 4.5		Auxiliary gas
	CO ₂ 4.5 (that is 99.995%)		Reference gas
² H/ ¹ H	He 4.6 with 2% H ₂ or He 4.6 with 4% H ₂	2.5 bar	Auxiliary gas
	H ₂ 5.0, --200‰ vs. SMOW; not lower than -300‰ vs. SMOW (that is 99.9%) ^a		Reference gas

^a generated by electrolysis. See “Water Equilibration (²H/¹H Equilibration)” on page 8-12.

Table 5-4. Gas requirements for DIC (Dissolved Inorganic Carbon)

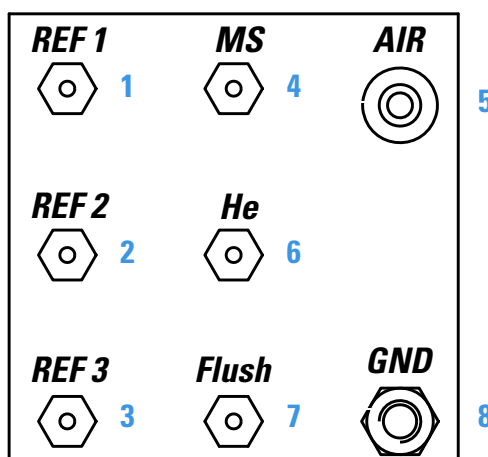
Purity	Pressure	Usage
CO ₂ 4.5 (that is 99.995%)	2.5 bar	Reference gas

Table 5-5. Gas requirements for carbonates

Purity	Pressure	Usage
CO ₂ 4.5 (that is 99.995%)	2.5 bar	Reference gas

Gas Connections of the GasBench Plus Device

To operate the GasBench Plus device and the IRMS, several gases are needed either from gas bottles or from the main gas supply of the laboratory (compressed air, for example). [Figure 5-4](#) shows the connection scheme at the left side panel of the GasBench Plus device.



Labeled Components: 1–3=connections for reference gases, 4=capillary feedthrough to IRMS, 5=connection for compressed air from MS, 6=helium carrier gas connection, 7=flush connection, 8=GND (ground)

Figure 5-4. Connection scheme at left side panel

Two capillaries leading the gas flow to the mass spectrometer input valve must be installed. See **4** in [Figure 5-4](#). The connections 1 to 3 are used for the reference gases used in the various applications. Flush gases must be connected to the respective connector. For a detailed explanation, see “[Measurement Procedures for Real Samples](#)” on [page 8-1](#).

Tip

- When you install the CO₂ reference gas bottles, keep in mind that standard high pressure bottles for CO₂ contain a liquid phase that is subject to fractionation when temperature changes. These bottles must be stored at constant temperature to obtain stable isotope values for your reference gas.
- When using hydrogen (H₂) as reference gas, it is necessary to shorten the internal flow restricting capillary (that is, the capillary leading from the reference pressure regulator to the open split, 3-fold) to approximately 50% of its original length. This ensures that enough hydrogen enters the reference port of the mass spectrometer.

It is intended to connect only one equilibration gas to the flush port. Ex factory, the helium inlet port is connected to a T-piece that feeds the flush port with helium. The service engineer will connect helium at the upper inlet port and the required flush gas at the lower inlet port.

Installing the Gas Bottles

After you have installed the gas bottles, you must perform a leak check outside the working area before you start the system.

CAUTION

Compressed Gas. Explosion Hazard. Sudden, uncontrolled release of cylinder contents might cause severe injuries to laboratory personnel. Strictly adhere to safety regulations for laboratories when handling compressed gas cylinders. All laboratory personnel must be qualified for working with compressed gases.

Use appropriate regulators. Secure all cylinders in an upright position while in storage and in use. Ventilate all cylinder storage areas. Transport the cylinders securely on a cart that is designed for cylinder use and secure all cylinders during the transport.

WARNING

Explosion Hazard. Hydrogen forms explosive mixtures with air. A leak in the hydrogen (H₂) supply might cause fire or an explosion.

❖ To install the gas bottles

1. Connect the reference gases.
2. Connect the measurement gases.

3. Connect the equilibration gases, that is the flush gases:

Either [CO₂ + He] or [H₂ + He] are used as equilibration gases (0.5% CO₂ in He because of 50 V dynamic range).

❖ **To perform a leak check**

1. After mounting the reducing valve to the gas bottle, both valves should be open (that is, the on/off valve and the reducing valve). See [Figure 5-3](#).
2. Open the main valve for two or three seconds to let the gas purge the whole valve system. See [Figure 3-4](#).
3. Close the on/off valve.
4. Close the main valve.
5. Mark the manometer positions of on/off valve and main valve.
Wait for 10–15 minutes.
6. If the manometer positions have changed, a leak might exist.
7. To detect the leak, brush all valves and connections carefully with soap sud. A possible leak is indicated by gas bubbles.

Compressed Air

To operate the open split levers, the Valco™ valve and the optional traps, compressed air of 4 bar is required (see **5** in [Figure 5-4](#)). It can be provided by the compressed air distributor of the IRMS. Use the quick release connection to connect the blue compressed air tubing to the compressed air connectors of the IRMS.

Because the IRMS has four connectors, four screws (wing unions for compressed air, quick release connections) are provided with either the GasBench Plus device or the IRMS.

Thermo Fisher Scientific recommends that you filter the compressed air to remove particulate matter and moisture. The main supply must be connected to the compressed air service unit of the GasBench Plus mass spectrometer with the provided PVC tubing 6×1 mm.



Keep the access to the compressed air supply free at all times so that you can switch off the supply in case the supply line is torn off.

Exhaust System and Solvent Waste

The proper performance of your system can be affected by the waste and exhaust arrangements for the instrument. Vacuum and solvent wastes must be vented separately. Wastes must be collected and disposed of properly.

Tip It is your responsibility as the user to provide proper waste and exhaust systems for the operation of your system.

Exhaust System

Consider the following safety guidelines for exhaust and ventilation.

NOTICE

This instrumentation is designed for operation in clean air conditions. The laboratory must be free of all contaminants that could have a degrading effect on the instrument components. Dust, acid and organic vapors must be excluded from the work area. The warranty does not cover operation in substandard conditions.

NOTICE

The exhaust system for the instrument must not be affected by external weather conditions or other recipients potentially connected to the same system.

The exhaust system must not let the fumes or the fumes condensation return toward the instrument. It must be tested for leakage before connecting it to the instrument.

Peripheral devices might need additional exhaust connections. Refer to the manuals of the peripheral devices.

Ambient Conditions

This section describes recommended properties for the location where you intend to operate the instrument.

Temperature

The instrument is designed to operate at the temperatures specified in [Table 5-6](#).

Table 5-6. Temperature requirements for GasBench Plus

Specification	Value
Laboratory temperature	18–24 °C
Maximum temperature fluctuation ^a	±1 °C/h

^a Temperature fluctuations exceeding the given range might affect instrument performance.

As laboratory temperature increases, system reliability decreases. All electronics components generate heat while operating. This heat must be dissipated to the surrounding air for the components to continue to operate reliably.

Tip Avoid direct sunlight, proximity to heating or cooling sources and air drafts. Do not place the instrument where sudden changes in temperature can occur:

- air ducts
- air conditioning vents
- heating vents
- doors
- windows

Air Conditioning

The air conditioning must be capable of maintaining a constant temperature as specified at “[Temperature](#)” on [page 5-10](#) in the immediate vicinity of the system without producing excessive draft.

Thermo Fisher Scientific recommends installing an air conditioner if the specified limits are exceeded due to unfavorable climatic conditions.

Any expenditures for air conditioning are more than offset by good sample throughput and reduced repair costs.

Humidity

The relative humidity of the operating environment should not exceed 40 to 60%, with non-condensing and non-corrosive atmosphere.

Thermo Fisher Scientific recommends your laboratory being equipped with a temperature and humidity monitor to ensure that it is always within the required temperature and humidity specifications.

NOTICE

Operating the instrument at very low humidity might cause accumulation and discharge of static electricity, which can shorten the life of electronic components.

Operating the instrument at high humidity might cause condensation, oxidation, and short circuits and also blocks the filters of the fans.

Lighting

Good lighting makes any work area more enjoyable. Since a lot of work is done on the computer terminal, it may be convenient to have dimmed lights to reduce eyestrain. A small, high-intensity lamp is recommended for cleaning instrument components, source inspection, and manipulation of small components. Contact your local safety officer for advice and regulations on adequate working place conditions.

Altitude

The GasBench Plus device is designed for indoor use at an altitude of up to 2000 m above sea level.

Disturbances

This section describes disturbances that might affect the performance of the instrument.

Vibrations

Select a laboratory location that is free from mechanical vibrations. Floors at ground level usually have less vibration. When selecting the location, be aware of adjacent rooms with equipment that could transmit vibrations through the floor to the mass spectrometer.

Propagation of vibrations and their influence on complex instrumentations are difficult to predict. Contact your local service organization if you have questions and concerns about your laboratory.

Airborne Noise Emission

The A-weighted emission sound pressure level created by the instrument does not exceed 70 dB(A). Therefore, wearing ear protection is not necessary.

Dust-Free Environment

The laboratory in which the system is installed must conform to IEC 61010-1 pollution degree 2.

The air in your laboratory must not contain excessive dust, smoke, or other particulate matter. For reference, the air should contain fewer than 35×10^6 particles per cubic meter (1×10^6 particles per cubic foot) in excess of 5 μm .

Particulate matter might contaminate the instrument, the samples and the sample introduction as well as the ion source and may limit the background level of the instrument.

Dust can clog the air filters, causing a reduction in air flow around electronic components. Dust also forms a layer on electronic components that acts as an insulating blanket and thus reduce the heat transfer from the components to the surrounding air.

NOTICE

Open the instrument only when you have to perform maintenance in the instrument. Otherwise, keep the instrument closed.

High dust levels increase the probability of a computer disc drive head crash, surface discharge and short circuits.

NOTICE

Deposits of conductive matter on electronic components (PCBs) might cause a short circuit that can destroy them. Do not operate your instrument in an environment where it might be exposed to such matter (metal dust, for example).

Electromagnetic Fields

Sources of disturbing fields¹ are for example:

- Other analytical instruments which use large magnets or high frequencies with high power (NMR systems, for example)
- Industrial machines or welding tools
- Trains, trams, or subways
- High power cables crossing the ceiling
- Large electric motors (elevators)
- Radio stations nearby
- Cell phones

Radio Frequencies

NOTICE

Your instrument is designed to work in a controlled electromagnetic environment as defined in IEC/EN61326-1. Do not use radio frequency transmitters, such as mobile phones, in close proximity to the instrument.

¹ The GasBench Plus complies with the EMC product specifications as defined in EN 50081-1. Refer to the Declaration of Conformity in the Operating Manual.

If strong radio transmitters are operating in or close to your laboratory, contact your local support for advice. Due to the complexity of such influences, no general suggestion can be given in this Operating Manual.

Telephone

Thermo Fisher Scientific recommends having a telephone installed in your laboratory near the instrument. So, if necessary, you can operate the instrument while you are on the telephone with a Thermo Fisher Scientific field service engineer. The voice telephone outlet should be within 2 m of your instrument.

Electrostatic Discharge

Static charges and electrostatic discharge (ESD) are common natural phenomena that occur in many ways. Although ESD may not always be perceptible to a human being, it can damage the electronic components of your instrument.

Thermo Scientific instruments are designed to withstand electrostatic discharges (ESD) up to 4 kV (air discharge) and 4 kV (contact discharge) with all panels in place. However, if the panels are removed and the PCBs are handled without proper precautions, the electronic components might be damaged or fail prematurely. Static electricity can develop in a variety of ways. A few examples of how electrostatic charge can develop are as follows:

- When walking across a carpet in a room that is at 20% relative humidity, as much as 35,000 V of electrostatic potential can be generated on the surface of your body. This same motion in a room at 80% relative humidity generates about 1,500 V of electrostatic potential.
- Sitting and working in a chair padded with polyurethane foam in a room at 20% relative humidity can cause as much as 18,000 V of electrostatic potential to develop on your skin, or 1,500 V at 80% relative humidity.
- Working in laboratory coats and clothing made of synthetic fibers can cause accumulation of static electricity on your skin.
- Polystyrene cups and packing materials typically have a considerable electrostatic charge on them.

Many electronic components can be damaged by a discharge of electrostatic potential of as little as 50 V. ESD damage can be catastrophic causing your system to cease functioning. More commonly however, ESD damage might cause latent problems that are detrimental to sensitive electrical components, causing premature failures.

Therefore, Thermo Fisher Scientific recommends the following precautions, especially when operating your system at the lower end of the relative humidity specification listed above:

- Use a static-dissipating floor covering (such as tile or conductive linoleum) in the room that houses your instrument.
- Use laboratory chairs covered with natural fiber or other static-dissipating material.
- When operating the instrument, wear laboratory coats and clothing made of natural fiber or other static-dissipating material.
- Do not place polystyrene cups or packing materials on the instrument.

Installing the Autosampler

For details on the installation, refer to the *TriPlus RSH SMART Installation Guide*. The x-axis is the long axis at the autosampler, whereas the y-axis is directed forward, and the z-axis downwards, respectively. To install the z-arm head and the cover ([step 1](#) of the autosampler installation) see the relevant chapters of the *TriPlus RSH SMART Hardware Manual*.

Short Installation Description for the TriPlus RSH

❖ To install the autosampler

1. Unpack the box containing the components of the autosampler.
2. Screw the feet of the autosampler onto the base plate.

Tip The base plate is packed into the box that contains the GasBench Plus device. The feet, however, are packed into the autosampler box.

Use the delivered x-arm position calibration tool (left pole) for the TriPlus RSH for GasBench - Std and the TriPlus RSH for GasBench - Adv.

NOTICE

Use the correct markers for the autosampler (indication marks on the marker).

Installation

Installing the Autosampler

3. Place the sample tray and the heating block onto the base plate. The base plate has prefabricated cut-outs, where the heating block and all non-heated tray types are simply inserted. Owing to its heaviness, the heating block must not be fixed by screws underside.
4. Unpack the temperature controller for the heating block. The lid of the heating block needs to be screwed sideways onto the heating block by two provided knurled head screws.



In case of the carbonate option, a cut-out must be rasped at the right rear edge of the lid. The cut-out is used as feedthrough for the acid line of the acid reservoir. Usually, this is performed by a Thermo Fisher Scientific field service engineer.

5. Take out the z-arm.
6. Mount the x-axis guidance upon the feet and fasten it there using a TORX™ screwdriver. Three TORX screwdrivers are provided with the autosampler: 360/T10×80, 360/T20×100 and 360/T25×100.
7. Loosen the retaining screws out of the y-arm.
8. Attach the z-arm at the y-arm.

Tip When the autosampler is switched off (during installation, for example), in most cases the plunger falls completely down and can then be moved freely. The plunger cannot be moved when the autosampler is switched on.

Connecting the Autosampler to the Measurement Computer

For details on the installation, refer to the *TriPlus RSH SMART Installation Guide*.

To connect the autosampler, connect the LAN cable to the measurement computer LAN slot.

Installing an RSH for GasBench

This section describes the firmware and the hardware installation of the TriPlus RSH, including needle, tray and tool installation. For a generic overview about teaching the TriPlus RSH SMART, e.g., tool installation, refer to the *TriPlus RSH SMART Hardware Manual*. The description contains information for the factory-set configuration installation for:

- TriPlus RSH SMART for GasBench Plus autosamplers
- GasBench Tool 55 and GasBench Tool 120
- GasBench Plus tray settings in the autosampler firmware

Hardware Installation

❖ To install the hardware

Use the installation description.

1. Prepare the GasBench laboratory area as described in [“Placing the Instrument”](#) on [page 5-2](#).
2. Install the legs on the GasBench mounting plate using appropriate screws.
3. Put the x-arm on the legs.
4. Use the distance calibration tool (block) for TriPlus RSH for GasBench - Std (RSI mark) and for TriPlus RSH for GasBench - Adv (RTC mark).
5. Move the x arm appropriately to these positions and tighten the screw properly.

The TriPlus RSH SMART for GasBench Plus has a position for the ATC version.

NOTICE

Remove the protection before installation, or better do not install it before calibrating the arm positions with the tool.

Installation

Installing an RSH for GasBench

Figure 5-5 shows the distance calibration tool for the TriPlus RSH for GasBench - Adv.



Figure 5-5. Distance calibration tool for TriPlus RSH SMART Std. and Adv.

Tip Each type of RSH autosampler is equipped with one distance calibration tool for both TriPlus RSH SMART for GasBench Plus Standard and Advanced. Set the mark for the model inside the tool.

6. Position the reference point pole to Slot2. If not in use, put it to its dedicated parking position.
7. If the IP address deviates from the factory set IP address, install it correctly.

The firmware has been pre-installed ex-factory. The configuration contains a pre-set configuration for all trays, tools and reference teaching if the calibration guide for the x-arm (TriPlus RSH Advanced with ATC position set points).

Only for a special release, an appropriate firmware must be set for the TriPlus RSH for GasBench as shown in the following steps (all pre-installed trays and tools shall be available):

- a. Teach the arm positions.
- b. Teach the ATC.
- c. Teach the tray holder positions. See [step 6](#).
- d. Teach the appropriate installed rack system or rack to the Slot1 or 2 position.
- e. Install the tools (TriPlus RSH for GasBench - Std by hand). Put the tools into the ATC and teach the tools to be able to pick up.

NOTICE

The Maintenance-Installation menu will guide in general by step through the aforementioned installation workflow.

General Notes for the TriPlus RSH SMART for GasBench Plus Terminal

The TriPlus RSH has three modes. In the manual, most of the modes are shown with extended user mode.

1. Standard user mode - No sign on the panel.
2. Extended user mode
3. Service Technician mode. A USB stick is shown on the upper panel.



NOTICE

A virtual terminal is integrated in Qtegra TriPlus RSH SMART. See “[TriPlus RSH SMART Autosamplers](#)” on [page 7-13](#).

Teaching

❖ To teach the TriPlus RSH

1. Move the arm to the position.
2. Click **Save**.
3. Click the **Arrow up** and **down** buttons to perform a fine adjustment for x-y-z positioning.
4. Type <Ok>.

Messages and status

On top of the panel, an envelop is shown. The status of the TriPlus RSH can show different status reports.

1. Green: RSH is completely and correctly configured and taught. The TriPlus RSH is ready to operate.
2. Yellow: A teaching or configuration is not yet finished properly. The TriPlus RSH will not be ready to operate with Qtegra.

TriPlus RSH SMART - Advanced and TriPlus RSH SMART - Standard Installation

Standard RSH Installation and Configuration Check

The delivered TriPlus RSH SMART for GasBench Plus autosamplers have a pre-installed firmware for GasBench Plus installed, and all parts are pre-configured appropriately.

The pre-installed firmware contains:

- All pre-installed trays

Installation

Installing an RSH for GasBench

- The delivered GasBench Tool 55 and GasBench Tool 120 pre-installed

Tip A firmware update is not needed on a standard TriPlus RSH SMART for GasBench Plus.



Figure 5-6. GasBench Tool 55



Figure 5-7. GasBench Tool 120

Installing a New TriPlus RSH Firmware Using a Memory Stick (FAT 16)

❖ Quick guide for generic firmware update

1. Use a memory stick with a folder named **AutoRun**.
 - a. Save a text file named **ResetSystem.txt** to this folder.
 - b. Save the appropriate new firmware version to this folder.

2. Back up the TriPlus RSH before running a new firmware. Refer to the TriPlus RSH Firmware chapter in the *TriPlus RSH SMART Hardware Manual*.
3. Switch off the power supply of the RSH.
4. Insert the memory stick in the USB slot of the RSH on its rear panel. Refer to the *TriPlus RSH SMART Hardware Manual*.
5. Switch on the RSH.

The RSH starts the installation automatically:

- a. The LED shows a flashing blue light.
 - b. The terminal panel shows the installation of the new configuration, and the new firmware version number, which is being installed.
 - c. If the installation stops, the file on the USB stick is corrupted. Get a new file on the USB stick.
 - d. The terminal panel shows when the memory stick can be safely removed.
6. The TriPlus RSH deletes the file **ResetSystem.txt** after successful installation.
 7. After removal, the TriPlus RSH reboots automatically without any user interaction.
 8. After reboot, the firmware version will be reported on the terminal panel.
 9. If a TriPlus RSH SMART - Adv is in use, the ATC has to be taught again.

- The successful installation shows a 'TriPlus RSH SMART' on the panel.

NOTICE

Use the memory stick again in Service Technician mode: store a text file named **ServiceTechnician.txt** on the main drive of the USB stick.

ResetSystem.txt will delete all preset firmware items and install the default firmware. No remnants of programming are being left.

The firmware removes the network connection, that is if DHCP is in use, the address needs to be reconfigured. Thermo Fisher Scientific does not recommend to use DHCP service. An extension of the delivered LAN cable and use of DHCP Thermo Fisher Scientific cannot be made liable for malfunction.

Read out the firmware on the terminal panel and update Qtegra with the new IP address. Refer to the *TriPlus RSH SMART Hardware Manual*.

❖ **Quick installation guideline for the GasBench specific firmware configurations and references**

Tip This guideline avoids that all tools and trays must be taught by the user again.

- Turn on the TriPlus RSH if not already done automatically or switch it off.
- Check the IP address. Refer to the *TriPlus RSH User Guide*.
 - The standard IP address used by TriPlus RSH and Qtegra is 192.168.99.230. Refer to the *Qtegra ISDS Software for the Gas Isotope Ratio Mass Spectrometers Installation Guide* to properly set IP addresses on the computer LAN slot.

At Control Panel > All Control Panel Items > Network Connections, name the slot **RSH**. Name one connection computer installed **RSH** (not the delivered USB LAN connector; the USB LAN is reserved for customer server connections), see [Figure 5-8](#).

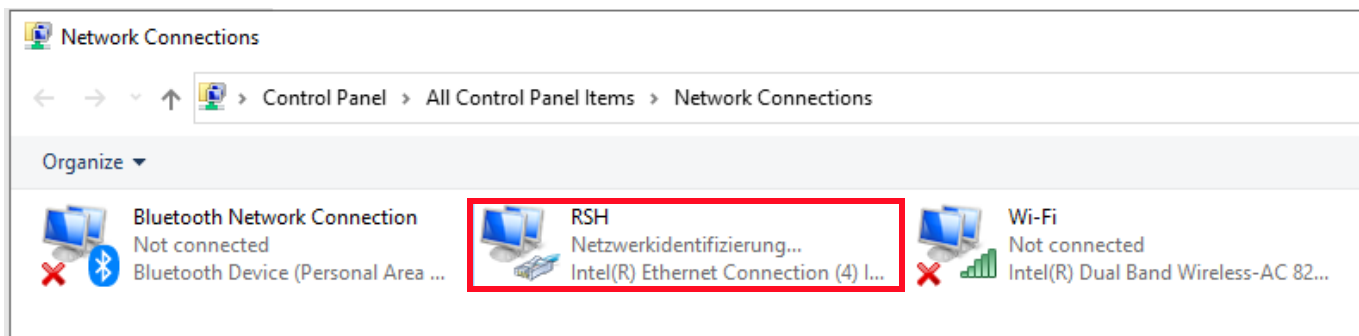


Figure 5-8. RSH among network connections

Table 5-7. IP and gateway addresses

Connection	IP address	Gateway
Autosampler	192.168.99.230	255.255.0.0
RSH LAN slot (IPv4)	192.168.99.100	255.255.0.0 ^a

^a is automatically set.

3. Ping in **cmd.exe**. Check the connection, see [Figure 5-9](#).

```

CA: Command Prompt
Microsoft Windows [Version 10.0.18363.1556]
(c) 2019 Microsoft Corporation. All rights reserved.

C:\>ping 192.168.99.230

Pinging 192.168.99.230 with 32 bytes of data:
Reply from 192.168.99.230: bytes=32 time=2ms TTL=128
Reply from 192.168.99.230: bytes=32 time=1ms TTL=128
Reply from 192.168.99.230: bytes=32 time<1ms TTL=128
Reply from 192.168.99.230: bytes=32 time<1ms TTL=128

Ping statistics for 192.168.99.230:
    Packets: Sent = 4, Received = 4, Lost = 0 (0% loss),
    Approximate round trip times in milli-seconds:
        Minimum = 0ms, Maximum = 2ms, Average = 0ms

C:\>
  
```

Figure 5-9. Ping connection to RSH

4. Open the Internet Explorer. LAN must be connected to the RSH.
5. Type the IP address **192.168.99.230**. Press **<Enter>**.

A panel is shown is the browser, see [Figure 5-10](#).

Installation

Installing an RSH for GasBench

6. In the lower part of the panel, click **Desktop setup**.

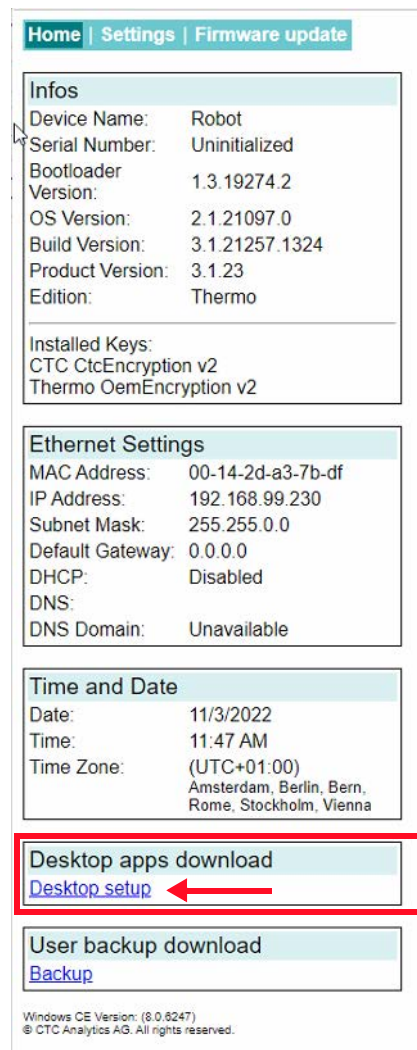


Figure 5-10. Clicking the link “Desktop setup”

Figure 5-11 appears.

7. Click **Run** to start the download.

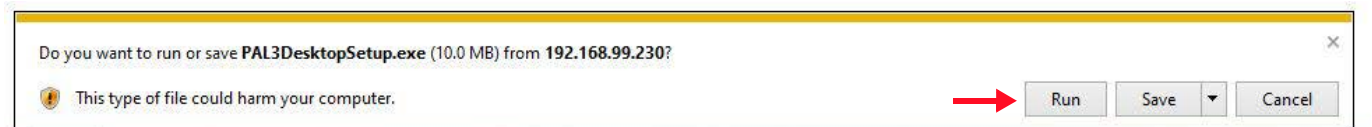


Figure 5-11. Starting the download

The Desktop installation wizard opens.

8. Use the proposed destination folder for the Desktop installation. See [Figure 5-12](#).

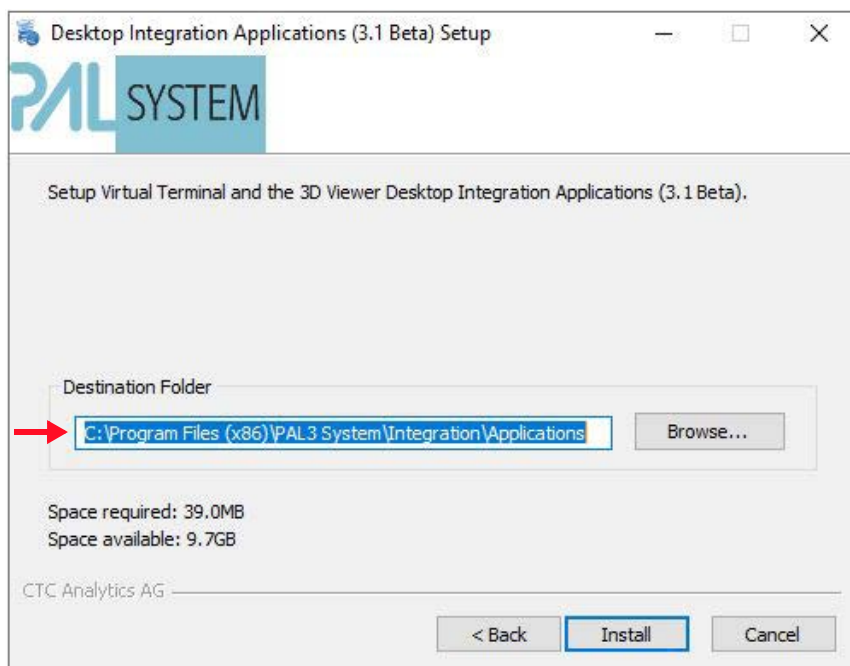


Figure 5-12. Using the destination folder for the Desktop installation

9. Confirm all following steps with **Next** until the Desktop installation is finished.

Select the languages you want to be installed. See [Figure 5-13](#),

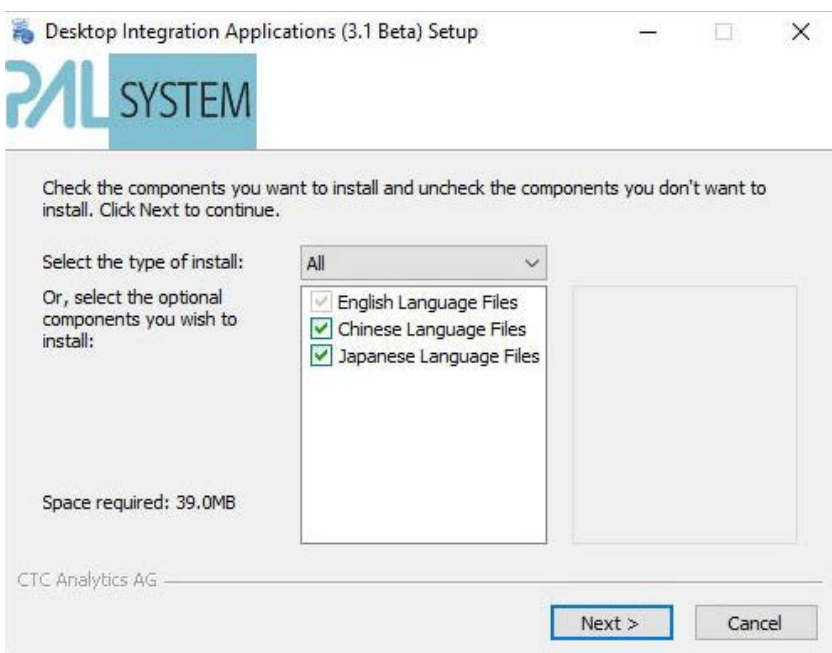


Figure 5-13. Selecting the languages to be installed

Installation

Installing an RSH for GasBench

9. When the installation is completed, click **Close**. See [Figure 5-14](#).

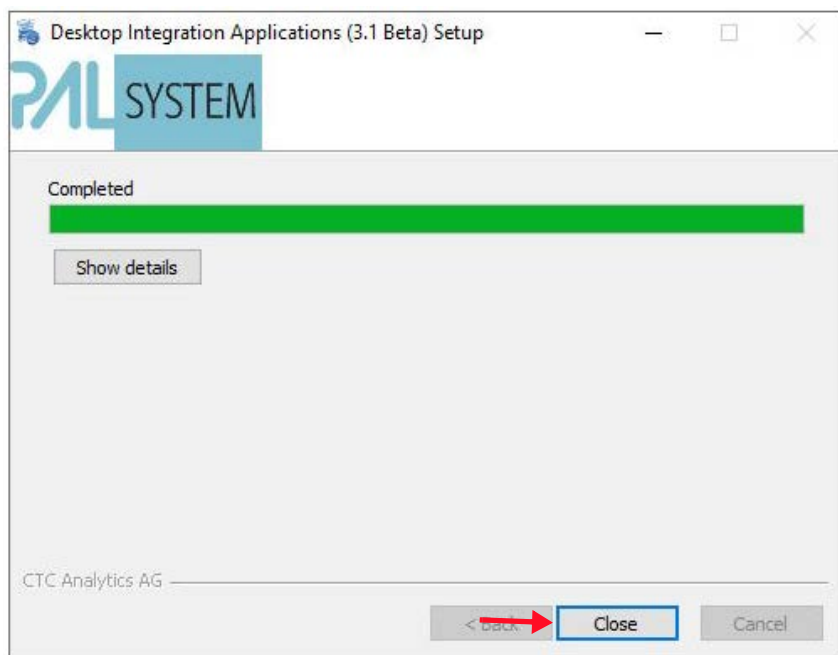


Figure 5-14. Installation is completed

10. Navigate to
C:\Program Files (x86)\PAL3 System\Integration\Applications\3.1

Double-click **TerminalDesktop.exe** to install the Terminal Desktop. See [Figure 5-15](#).

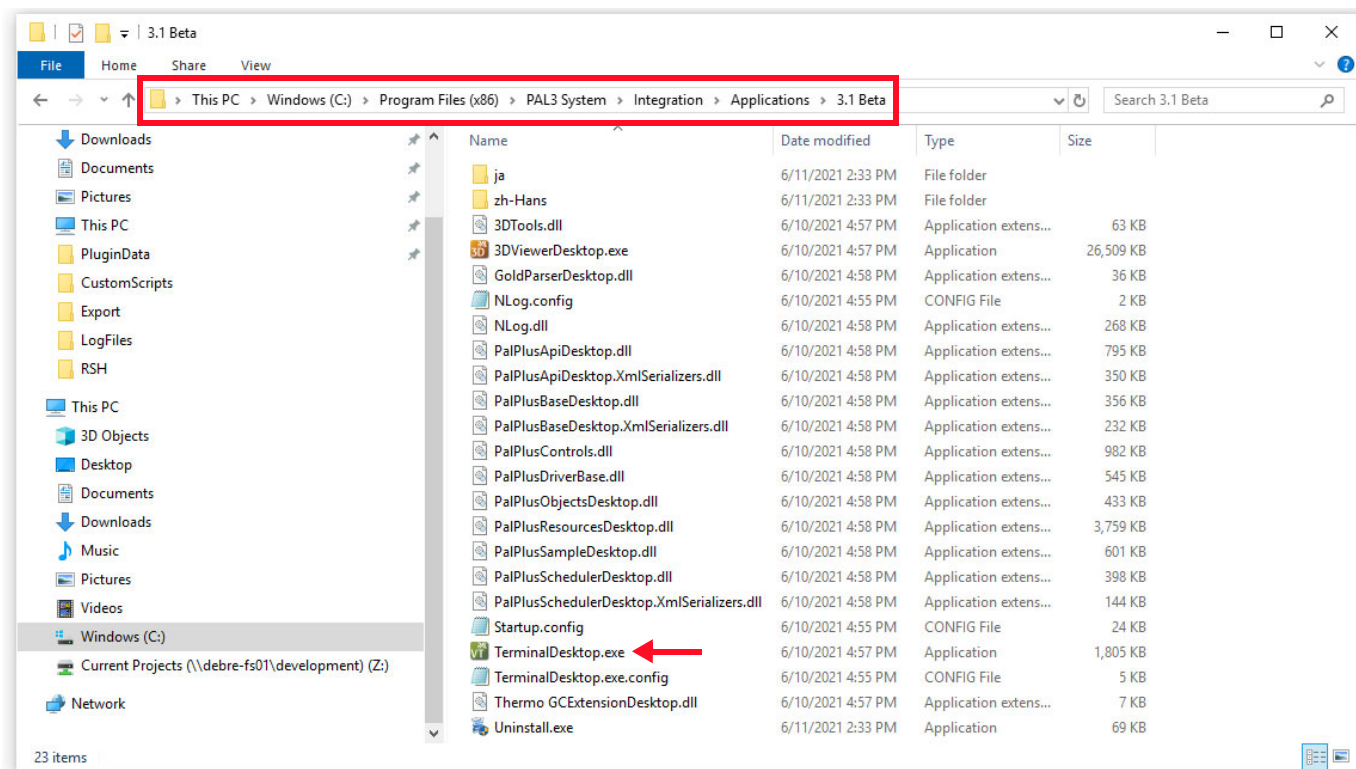


Figure 5-15. Installing the Terminal Desktop. Find the file with the appropriate folder name, which includes the TerminalDesktop.exe file.

11. Remove any predecessor Terminal Desktop from the taskbar.
12. At **Host**, type in the IP address, see [Figure 5-16](#).
13. At **Transport**, select **Http** from the dropdown menu.
14. Click **Connect**.

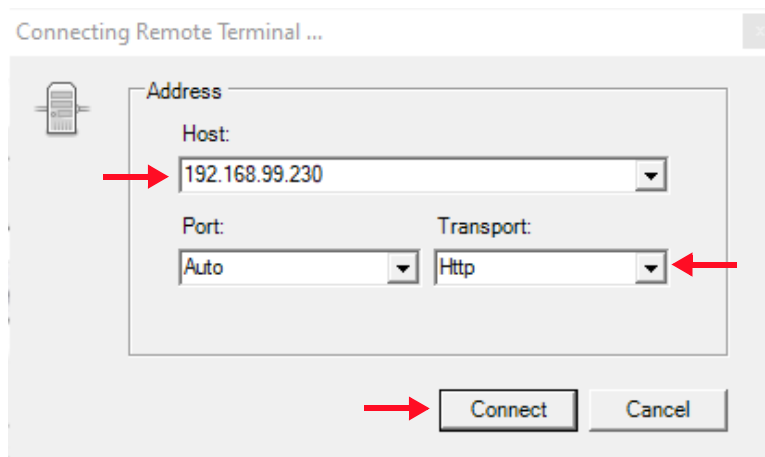


Figure 5-16. Connection window for the TriPlus Terminal Desktop

Installation

Installing an RSH for GasBench

The TriPlus RSH will be connected. This can be achieved as well by using the real panel.

15. On the lab connected computer, select **Extended User Level**. See [Figure 5-17](#).
16. Click **A** and **B** at the same time.

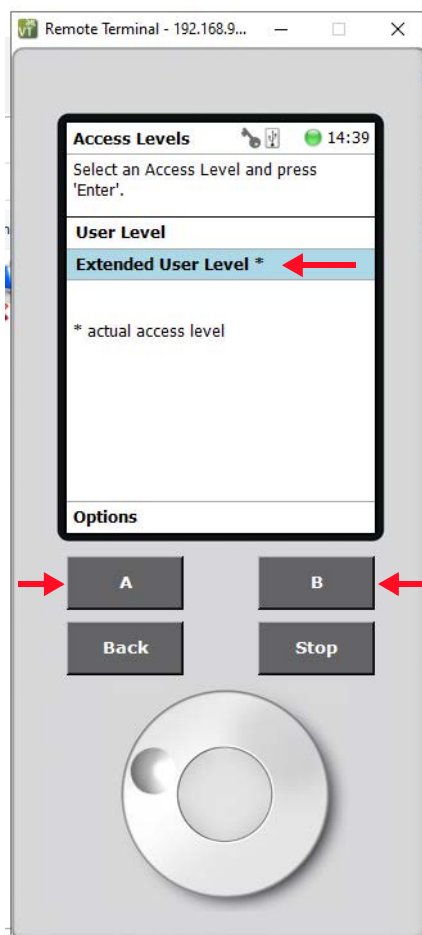


Figure 5-17. Selecting Extended User Level

17. Navigate to **Maintenance > Reset configuration**.

The Factory Settings page opens, see [Figure 5-18](#). The **GasBench Factory Setting** will be used.

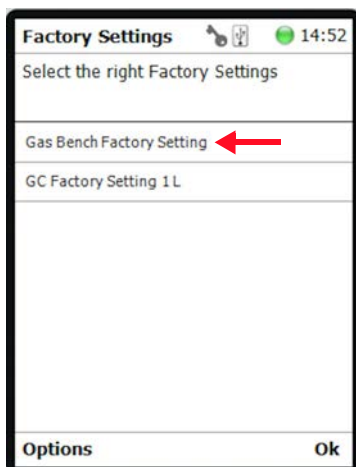


Figure 5-18. Using GasBench Factory Setting for TriPlus RSH for GasBench - Std

For a TriPlus RSH for GasBench - Adv, the Factory Settings page looks quite different, see [Figure 5-19](#).



Figure 5-19. Using GasBench Factory Setting for TriPlus RSH for GasBench - Adv

18. Click the GasBench configuration (**GasBench Factory Setting**). Confirm with **YES**.

The configuration is set. An automated reboot takes place.

19. After the reboot, the GasBench or GC configuration will be set.

For further calibrations, proceed with [Calibrating the TriPlus RSH for GasBench Plus Configuration](#).

Installation

Installing an RSH for GasBench

Three error messages are shown, because the x arm needs to be re-set, the racks and the reference point for the tools, tray holder and the tray need to be configured. Set the tools.

Installing, Updating, or Downgrading a TriPlus RSH Firmware Via an Internet Browser

With the release of the Rev II board and firmware 2.4.6 you can directly upgrade the TriPlus RSH firmware from the web server without using the USB key. The operation is possible with any firmware starting from 2.4.6 and with Chrome or Microsoft Edge as a browser.

❖ To upgrade the firmware from the web server

1. Store the firmware files on any folder on the PC.
2. Enter the IP address of the TriPlus RSH SMART for GasBench Plus in the browser (Chrome or Edge only).
3. Select the Firmware update tab.
 - a. Select the firmware you wish to install and select **upload**.
 - b. You will have the option to force a downgrade or a system reset as well
 - c. Just select the firmware and the update option, do not click any button yet.

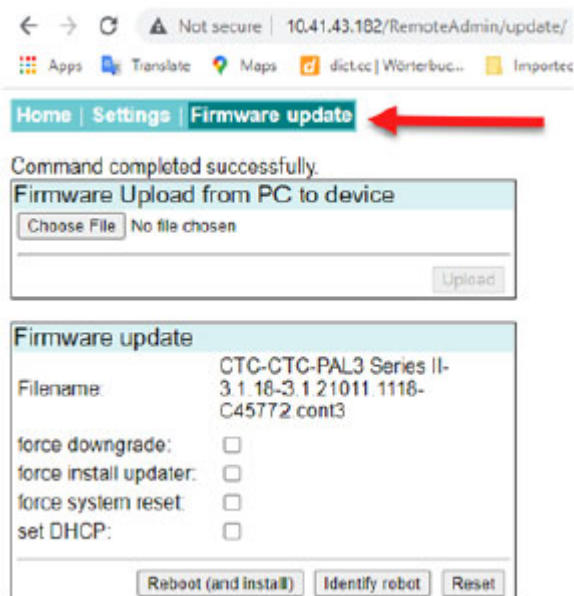


Figure 5-20. TriPlus RSH Firmware update tab

4. On the instrument terminal (virtual or handheld), change the **User Level** to *extended*.
5. From the Service menu, select **Allow LAN FW upgrade**.

Within the next 10 minutes, upgrade the system from the terminal by selecting the **reboot** and **install** option. This step prevents the instrument from being updated with an incorrect IP address.

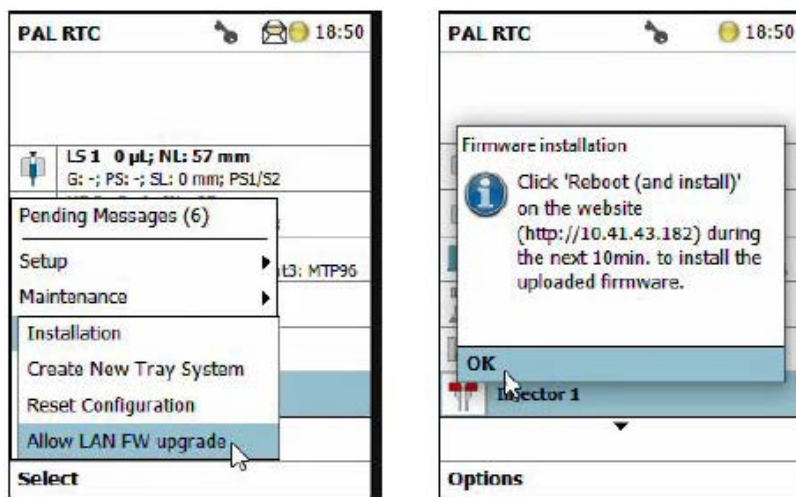


Figure 5-21. Terminal with Service menu and Allow LAN FW upgrade command

Calibrating the TriPlus RSH for GasBench Plus Configuration

This section describes the quick calibration after a firmware update of the GasBench RSH autosamplers. The instruction can be followed by using the terminal module as well.

❖ To calibrate the TriPlus RSH for GasBench Plus configuration

1. Go to the Extended mode by pressing the **A** button and the **B** button simultaneously. Confirm with a mouse click (or by pressing the Center button). Press the **Back** button to return to the Main menu.
2. Go to **Options**. Type `<service>`. Type `<installation>`.

A short sequence of installation steps will be shown. Languages and name changes are not presented.

Passive tools can be skipped if not installed.

3. Type **Calibrate motors**. See [Figure 5-22](#).

All motors will be calibrated automatically.

On the first wizard page, press **Calibrate**.

Installation

Installing an RSH for GasBench

The wizard guides you through the process. Follow its commands.

Release the tool:

- Std: by hand
- ATC: automatically by command



Figure 5-22. Calibrating the motors

4. Confirm with **OK**.

The arm moves automatically back to Home. The step is marked as Done on the panel.

5. If an ATC is installed, the exchange positions must be taught.

NOTICE

Do not move the z-arm too deep. The GasBench Tool 120 must fit under the arm.

For the TriPlus RSH for GasBench - Std, the exchange position can be taught here.

- a. Move the arm to the position and press **Save**.
 - b. Fine tune the x, y, and z positions using the wheel.
 - c. Confirm with **OK**.
 - d. Mount a tool as described in the *TriPlus RSH Hardware Manual*.
6. The exchange tool must be defined by the user beginning with [step 5](#) after a new tool is inserted. See also [step 9](#).
 - a. Use the GasBench needle guide type, see [Figure 5-23](#).

- b. Choose **GasBench**.

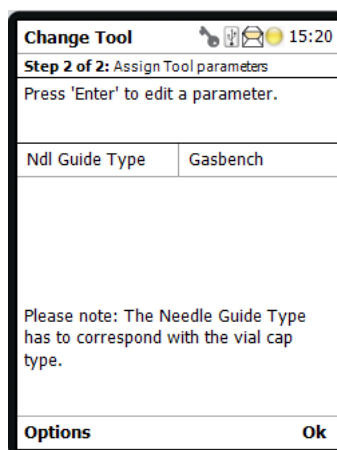


Figure 5-23. GasBench needle guide type

7. For each tool, a new reference must be set, see [Figure 5-24](#).

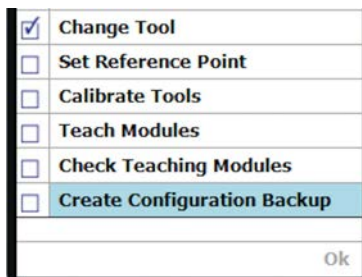


Figure 5-24. New calibration menus

8. Begin with **Set Reference Point**, see [Figure 5-25](#) and [Figure 5-26](#).
- a. Pick up the reference point from the GasBench Plus park position by hand. Install it in its position in slot 2.
 - b. Manually, move it to the reference point. Type **Save**.

NOTICE

Do not move the z-arm too deep. The GasBench Tool 120 must fit under the arm.

- c. Perform a fine tuning where the left needle guide from the GasBench Tool 55 touches the right circle.
- d. Move in z direction around 0.5–1 mm on top of the reference touch point.

NOTICE

Perform the fine tuning using the software or the terminal wheel. Do not exert any force with your hands. Use the center button to confirm the set x, y, and z values.

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Installing an RSH for GasBench

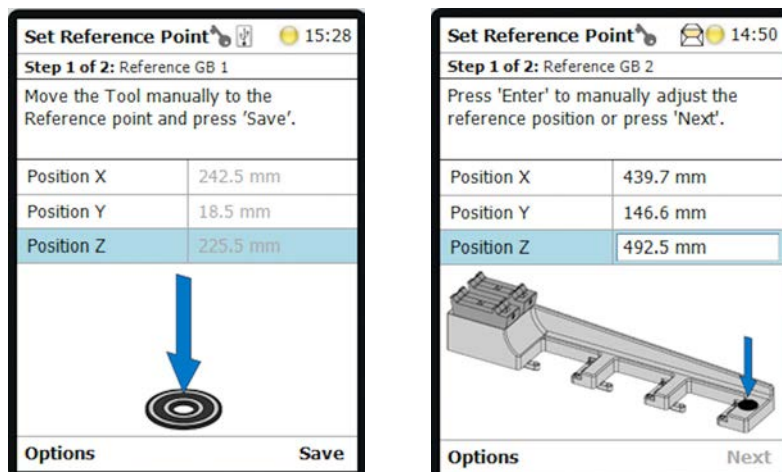


Figure 5-25. Reference point for referencing the TriPlus RSH for GasBench - Adv (left) and the TriPlus RSH for GasBench - Std (right)^a

^a This reference point will be the reference point for the ATC as well, but it is on the slot for ATC.



Figure 5-26. Reference point

9. Press **Calibrate tools**.

Use the same calibration set point as shown in [Figure 5-26](#). The GasBench Tool 55 and the GasBench Tool 120 must both be taught because the length of the needle guide is different. See [“TriPlus RSH SMART - Advanced and TriPlus RSH SMART - Standard Installation”](#) on [page 5-19](#).

a. TriPlus RSH for GasBench - Adv:

Install the tools (GasBench Tool 55 [standard tool] or GasBench Tool 120, or any hybrid of both) in the slot of the ATC. More tools appear.

b. TriPlus RSH for GasBench - Std:

The standard tool is not detected automatically. Therefore, manually set and install the tools (GasBench Tool 55 [standard tool] or GasBench Tool 120 or any hybrid of both) in the arm of the TriPlus RSH for GasBench - Std, see [Figure 5-27](#).

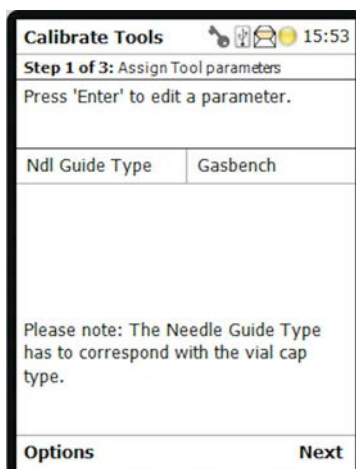


Figure 5-27. Calibrate Tools window for TriPlus RSH for GasBench - Std

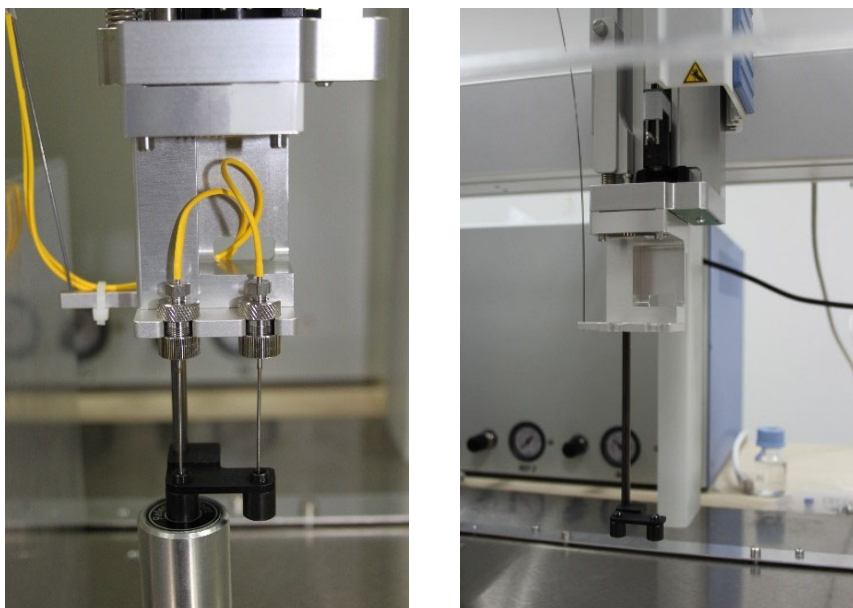


Figure 5-28. GasBench Tool 55 (left) and GasBench Tool 120 (right) to calibrate^a

^a Use the reference point for the calibration.

10. Move the installed tool to the tool referencing point, save and fine tune the x, y, z values. Click **Next**.

11. The next wizard page is shown (see [Figure 5-29](#)):

- a. Next tool: Choose **Next Tool** (GasBench Tool 120, for example).

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Installing an RSH for GasBench

- b. No other tool: Click **Finish**.
- TriPlus RSH for GasBench - Std: Move the arm to change positions.
- TriPlus RSH for GasBench - Adv: The arm will automatically pick up the GasBench Tool 120 if only this tool is in the slot. If two or more tools are available, you must define which tool you want to pick up for the next teaching. Only a tool with a different length should be logically taught.

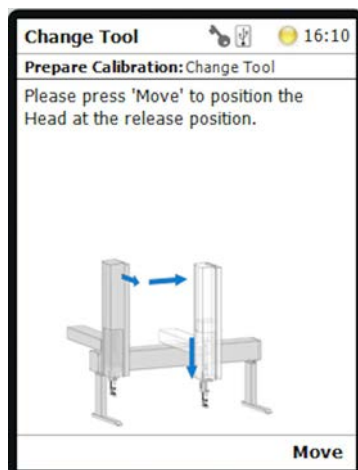


Figure 5-29. Changing the tool after Next has been clicked

12. Change the tool:

- manually (click **Move** to release point; change to GasBench Tool 120) or
- automatically (with ATC).

13. Repeat [step 9](#) after all available tools (also hybrids) have been taught.

NOTICE

All tools must be calibrated to be available as tools in Qtegra. Even hybrid tools must be taught, because they will get a specific name to be used for a certain application and injection.

14. Teach Modules: because a tray holder is a module, it must be taught. Move any tool to the reference point and define the reference point for the tray holder. Use GasBench Tool 55 to teach the tray holder.

15. Check Modules: The tray holder position will be checked:

- If the tray holder position is appropriate, confirm with **OK**.
- If the tray holder position is not appropriate, teach the tray holder again (see [step 14](#)).

16. Perform a backup.

The installation procedure teaching each tray position is finished. See [Figure 5-30](#). When you return to the main menu, the TriPlus RSH shows a green LED on the arm and on the virtual terminal.

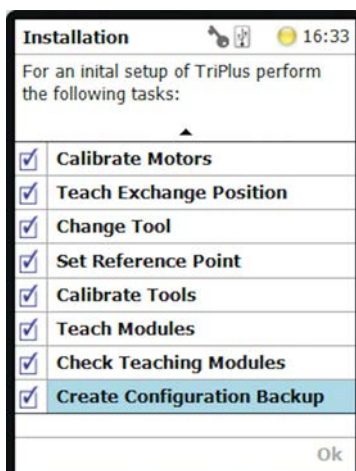


Figure 5-30. Report of all calibrated options shows a check mark when finished successfully

17. You must check the factory-installed tray with a tray (for example, with any of the non-thermostatted trays or with the 12 mL thermostatted tray).

NOTICE

A non-thermostatted tray in slot 1 and a thermostatted tray in slot 2 is the only combination, which is not supported by the autosampler firmware and consequently does not work with the setup in Qtegra ISDS Software.

18. Proceed with [Checking Calibration and Teaching Installed GasBench Trays](#).

Checking Calibration and Teaching Installed GasBench Trays

The available GasBench trays are described in [“Checking Calibration and Teaching Installed GasBench Trays”](#) on [page 5-37](#). Each tray and slot must be taught x-y-z with GasBench Tool 55 and GasBench Tool 120 individually if used with the thermostatted tray. The appropriate teaching adapters must be used (teaching adapter indicated with “heated tray” and teaching adapter indicated with “non-heated”). During the teaching, the autosampler will use the tool, which is mounted automatically for the teaching process, or it will pick up the first tool in the first ATC slot if no tool is mounted.

❖ **To check calibration and teach installed GasBench trays**

1. Install a tray in slot 1. If available, install a tray in slot 2. If not available, use the same tray as in slot 1 for teaching slot 2. See

Installation

Installing an RSH for GasBench

Figure 5-32. For example, the set points for the GasBench Tool 55 and GasBench Tool 120 for the 12 mL non-thermostatted tray are 43.1 / 50.3 / 392.3 for pos. 1, 43.1 / 232.2 / 392.3 for last pos. Y, and 716.4 / 232.2 / 392.3 for max. tray pos.

NOTICE

Three position calibrations for a tray: Each tray in each slot must be taught with each tool for the use in Qtegra. The TriPlus RSH will refuse to operate if a tool is taught for that rack. Use the position calibration for teaching.

NOTICE

Use the delivered teaching adapters for the x, y, z teaching positions. For the thermostatted tray remove the lid and put the teaching adapter into the heated block positions 1, 8, 96. Always the left needle must be used.

NOTICE

In Qtegra, the Needle penetration must be set to Zero.

NOTICE

After each teaching. Use Qtegra to send the autosampler arm into an intermediate position, e.g., for a 96 samples tray. Send it to position number 75. Check if the left needle guide will be exactly positioned to this position. If not, the autosampler has been teach wrongly.

After successful teaching. Put the maximum needle penetration to 28 mm for the GasBench Tool 55.

2. Click **Trayholder GB**.

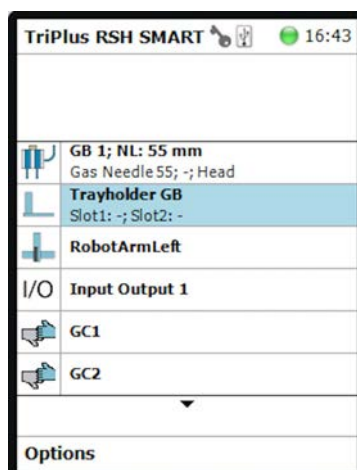


Figure 5-31. Clicking Trayholder GB

Figure 5-32 appears.

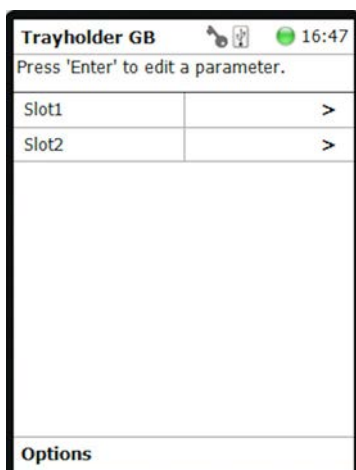


Figure 5-32. Selecting Slot1 or Slot2

3. Teach Slot1 as follows (use the same steps for Slot2, accordingly):
 - a. Select **Slot1**.
 - b. Click **Options**.
 - c. Click **New Tray**.

New Well plate: Not for GasBench Plus.
 - d. Set it up as **Tray 1**.

The wizard provides a page as shown in Figure 5-33 (left).

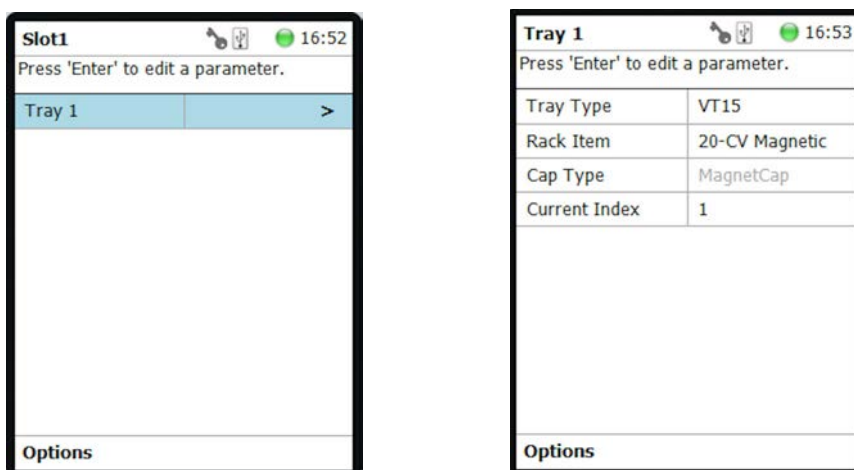


Figure 5-33. Setting up Tray 1 (left) and defining its properties (right)

4. Define the properties of Tray 1 as shown in Figure 5-33 (right).
 - Tray Type: “Rack 8×12 heated”, for example. See “[Layout of 96 Sample Trays with 12 mL Vials](#)” on page 3-12.

- Rack Item: Enter a vial type that can be used with the appropriate tray type.

NOTICE

With the Tray Type “8x12 heated”, only 4.5 mL vials and 12 mL vials can be used in Standard mode. To use other vials, the needle penetration must be edited before use.

- Cap Type: 20-CV-Magnetic (default)
- Current Index: 1 (default)

Figure 5-34 shows an example (“Rack 8x12 GB Heated”) of the standard heated tray programming of Slot1 (left) and Slot2 (right).

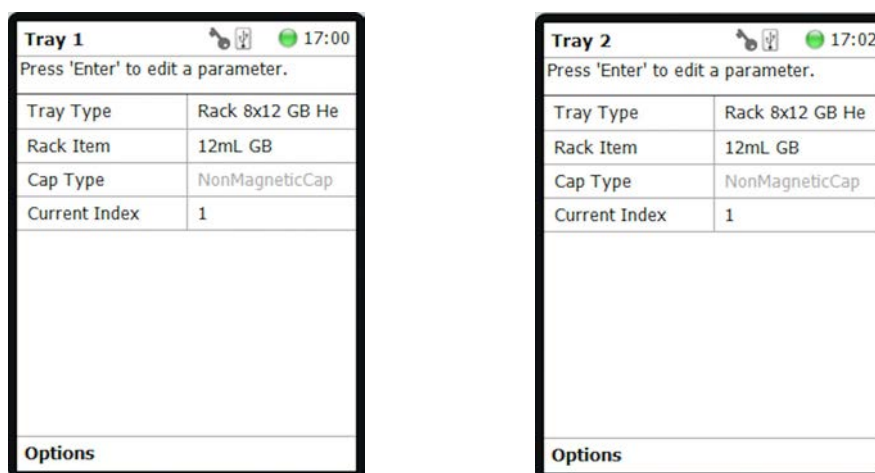


Figure 5-34. Standard heated tray programming of Slot1 (left) and Slot2 (right) – example

NOTICE

A tool must be taught for each slot with a defined tray.

5. Teach Slot1 as follows (use the same steps for Slot2, accordingly):
 - a. Select Tray 1.
 - b. Click **Options**.
 - c. Click **Check Teaching**.

The RSH software automatically guides you through the x, y and z check points.

Tip If only one tray is available and you want to check for future upgrades, install the tray of Slot1 into Slot2.

Tip A factory-set tray should exactly fit to the center of the vials positioned in the TriPlus RSH for GasBench - Std tray or in the TriPlus RSH for GasBench - Adv tray.

If the tray does not fit, each individual tray can be taught using the same menu given in [step 5](#), but the customized tray must fit at least into the dimensions of the thermostatted tray size (Slot1 size).

General aspects of teaching

1. Move the arm to the desired positions, i.e., first position, last y-direction position by using the **left** needle guide.

CAUTION

Material damage. When using the **right** needle guide, the needle can be destroyed.

2. Press **Save**.
3. Perform a fine adjustment by using the scroll button for x, y, and z positioning.

Tools for the TriPlus RSH SMART for GasBench Plus Standard and Advanced Autosamplers

The TriPlus RSH SMART for GasBench Plus autosamplers are delivered with a standard GasBench Tool 55 for install a standard measurement and purge needle (see needle setup with the GasBench Tool). For the Denitrification Kit or setups requiring a purge of a liquid, the Thermo Fisher Scientific GasBench Tool 120 can be used for any vial size. For each autosampler, more than one tool can be purchased and used.

Each of the autosampler type will be able to detect the tool and enumerate it accordingly as a hybrid tool with the same hardware operation. In Qtegra ISDS Software, the tools can be defined and be used for a specific applications. See “[TriPlus RSH SMART Autosamplers](#)” on page 7-13, “[Carbonate Isotope Analysis Workflow](#)” on page 9-41, and “[Setting up a ¹⁸O in Water Equilibration Sample List](#)” on page 8-24.

NOTICE

Each tool must be calibrated before use with the TriPlus RSH SMART terminal with the reference point. See [Figure 5-22](#) and [Figure 5-25](#).

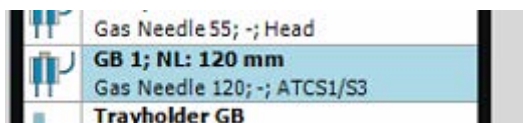
Installation

Tools for the TriPlus RSH SMART for GasBench Plus Standard and Advanced Autosamplers

Tool Calibration

❖ To calibrate a tool

1. Each new tool needs to be calibrated and configured, which is related to
 - The TriPlus RSH for GasBench - Std
 - The TriPlus RSH for GasBench - Adv
2. Each used tool will be configured automatically by the Autosamplers detection. If inserted in the ATC the Autosampler will detect the tool automatically and define if it is calibrated or not. If mounted into the TriPlus RSH SMART - Standard z-arm it will be detected but must be mounted before detection.
 - Any of the GasBench Tools, “GasBench Tool 55” or “GasBench Tool 120” need to be calibrated if not happened yet before. A message will appear if not calibrated properly.
3. Calibration
 - Use the terminal, Type Center button at the <tool level> or double-click on the tool (here GB 1):



4. Right-click under option (Extended user mode).
5. Press **Calibrate**. Following extra items can be done here:
 - a. Calibrate the tool
 - b. Rename the tool.
 - c. Check the tool calibration.

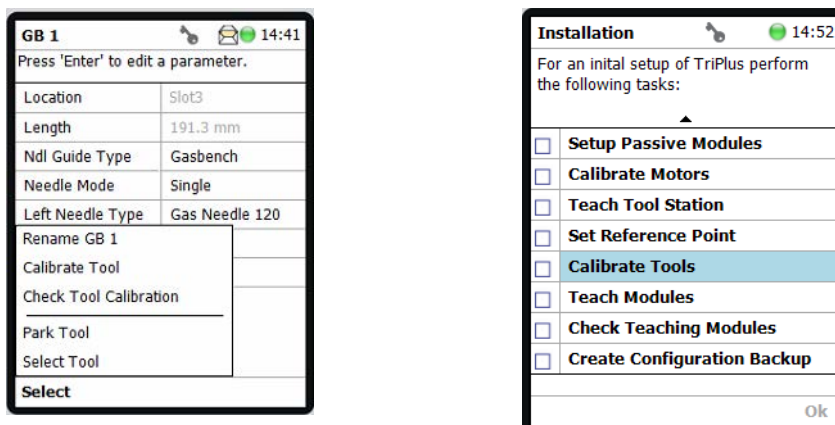


Figure 5-35. Tool calibration under the tool level (left) or under Service > Installation > Configuration > Calibrate (right)

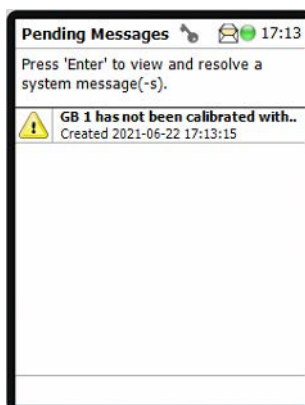


Figure 5-36. Warning message for a missing tool calibration

GB Tool Configuration in the Factory set TriPlus RSH Configuration (RSH terminal)

Tip All standard tools for the GasBench Plus are factory preset as follows.

1. GasBench Tool 55
 - a. Needle Guide type: GasBench
 - b. Needle Mode: Single
 - c. Left Needle Type: Gas Needle 55
 - d. Right Needle Type: None
 - e. Reserved Park Slot: N/A
2. GasBench Tool 120
 - a. Needle Guide type: GasBench

Installation

Tools for the TriPlus RSH SMART for GasBench Plus Standard and Advanced Autosamplers

- b. Needle Mode: Single
- c. Left Needle Type: Gas Needle 120
- d. Right Needle Type: None
- e. Reserved Park Slot: N/A

The following changes can be made in the RSH configuration or within Qtegra directly:

GasBench Tool 120 - The Robot Arm defines the calibration point and is only an internal factory information.

- a. Length: 191.3 mm (factory internal - cannot be changed)
- b. Needle Guide type: GasBench
- c. Needle Mode: Single or Double
- d. Left Needle Type: Gas Needle 120 / None
- e. Right Needle Type: Gas Needle 120 / None
- f. Reserved Park Slot: N/A

GasBench Tool 55

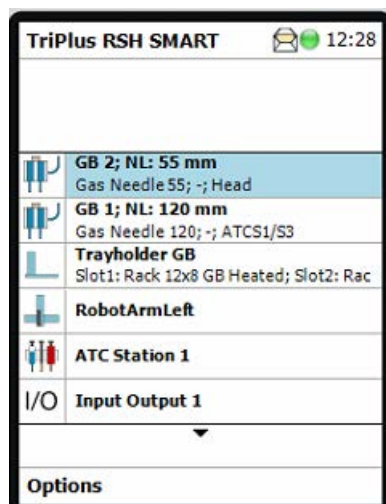
- a. Length: 126.3 mm (factory internal - cannot be changed)
- b. Needle Guide type: GasBench
- c. Needle Mode: Single or Double
- d. Left Needle Type: Gas Needle 55 / Acid Needle 48
- e. Right Needle Type: Gas Needle 55 / Acid Needle 48
- f. Reserved Park Slot: N/A

Tip The TriPlus RSH for GasBench - Adv autosampler will automatically detect a new tool even if it is a hybrid tool of the same design, e.g. GasBench Tool 55. The TriPlus RSH will detect it automatically as GasBench Tool 55 or 120 and will index it immediately and define it as not calibrated.

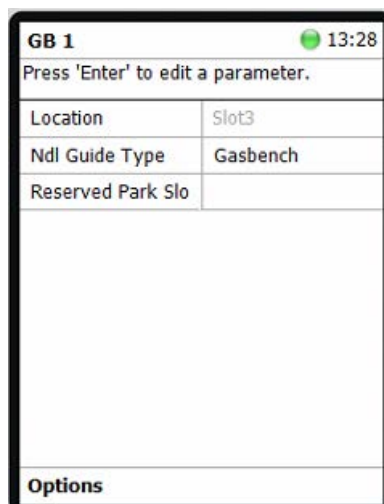
After calibration, the system can be set up with the appropriate tool and the trays being used for the application. The tool can be renamed and will be automatically saved on the TriPlus RSH autosampler with a new name.

TriPlus RSH SMART Standard and Advanced

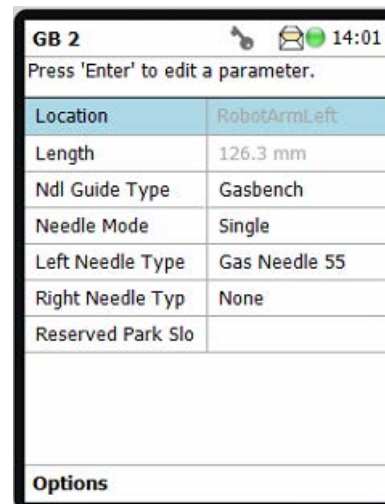
1. Opening the panel allows to see the configuration of the current TriPlus RSH installation.
2. Right-click the tool (see [Figure 5-37 a & b](#)) allows to see the Ndl Guide Type, here: “Gasbench” (see [Figure 5-37 c](#)).



a)



b)



c)

Figure 5-37. a) TriPlus RSH for GasBench - Adv with “GB 2: RT 55 mm” tool and “GB 1: RT 120 mm” tool; b) extended user mode: Settings of the GB 1 specific tool configuration; c) GB 2 specific tool configuration

GasBench Tool 55

The GasBench Tool 55 is to operate any needle with a 55 mm length including the acid needle (see [Figure 5-38](#)).

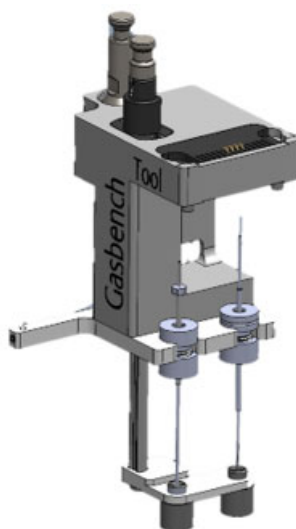


Figure 5-38. GasBench Tool 55

Installation

Tools for the TriPlus RSH SMART for GasBench Plus Standard and Advanced Autosamplers

GasBench Tool 120 - TriPlus RSH Standard and Advanced Autosampler

The GasBench Tool 120 is to operate any needle with a maximum length of 120 mm (see [Figure 5-39](#)).



Figure 5-39. GasBench Tool 120

Installation of the GasBench Tool 55 and the GasBench Tool 120 for the Standard and Advanced Autosampler

The TriPlus RSH GasBench Tools will be detected automatically. Only a needle must be installed properly.

Installation of a Needle in the GasBench Tool 55 or GasBench Tool 120

The measurement needle or a hybrid of the measurement needle (the purge needle by cutting the fused silica line of the measurement needle) and the acid needle of the GasBench can be installed in a the GasBench Tool 55 or 120. The TriPlus RSH SMART cannot detect the needle type automatically nor it can detect if a needle is installed in the left or the right of the needle holder and plunger.

Installation of a Needle to a Tool

Short description of a needle installation

1. Unpack any type of a needle, the spiral plastics, and a GasBench Plus Tool with the wire.
2. Put a needle through the back hole of the GasBench Plus Tool and move it to the front
3. Put the needle to the needle adapter and tighten it carefully to the tool, either on the left or the right.
4. Move the tubing straight and horizontally to the first attachment point of the guide wire. Very important to attach the tubing straight to the first wire attachment point and tighten it with a tie wrap at the distancing arm of the tool.
5. Attach all tubing properly firm to the wire alongside the wire. See [Figure 5-42](#).
6. Attach the wire thread (black) to the needle line protection jig.
7. Attach the tubings to the appropriate slot on the line protection slot. See [Figure 5-40 b](#).
8. Carefully check if no obstacle would be able to destroy the lines in operation, especially the arm when it moves into the ATC of the TriPlus RSH advanced.

CAUTION

Properly align the needle to the GasBench Plus tool that the needle lines cannot touch the x-arm tool window or the ATC (see [Figure 5-41](#)). If this is not properly aligned, any needle line connection will be destroyed.

CAUTION

Properly align the needle continuous flow tubings to the protection wire with a plastic spiral (see [Figure 5-41](#)). The tubings must be installed at two of the needle line protection jig ([Figure 5-40](#)) at the back of the TriPlus RSH unit. IF a tool is not in use the tool can be detached from the GasBench Plus but must be detached as well from the wire guide holder.

CAUTION

No wire and needle tubing is allowed to be placed in the operating space in front of or behind the TriPlus RSH. See [Figure 5-40](#). Needle line protection jig. The needle line protection wire including connector needs to be attached with the connector screw. The needle line protection wire including connector is available as spare part.

CAUTION

Do not bend the needle tubing too much and make a slight curve, that the fused silica does not break inside the yellow cable protection or inside the needle.

Installation

Tools for the TriPlus RSH SMART for GasBench Plus Standard and Advanced Autosamplers



Figure 5-40. Needle line protection jig. The needle line protection wire including connector (BRE0029734) needs to be attached with the connector screw. The needle line protection wire including connector (BRE0029734) is available as spare part.

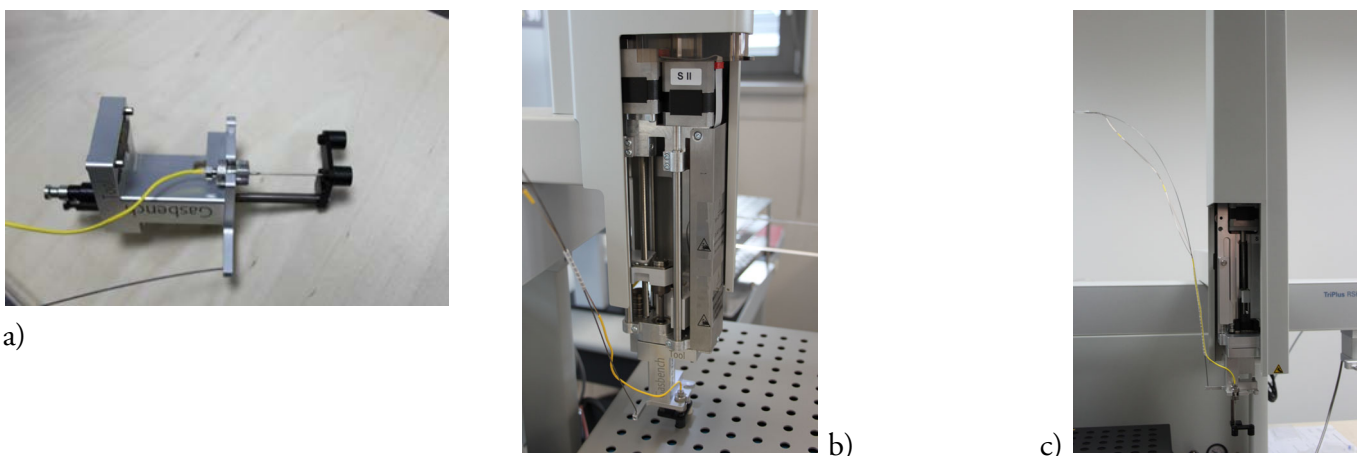


Figure 5-41. Appropriate installation of two needles into the GasBench Tool 55. a) front view b) side view c) with silicone tube attached. Leave the slider door open if possible.

[Figure 5-41 a\)](#) has a needle guide base (BRE0029732) installed on the left with a needle guide adapter for a gas needle (BRE0029730) and the same needle guide adapter type installed on the right with a needle guide adapter for the acid needle (BRE0029731). The needle guide base without adapter and both adapter can be purchased as spare parts.

All needle adapters can be changed on the needle guide base, i.e., a needle guide base can have only two needle guide adapters for a gas needle working making the GasBench Tool 55 a pure purge needle tool to be used on the advanced autosampler or a purge and a measurement needle tool for equilibration of hydrogen.

Since the equilibration of hydrogen needs only one hour of equilibration in normal pure water, a purge and a measurement needle can be used within one tool for the hydrogen isotope ratio determination of water.

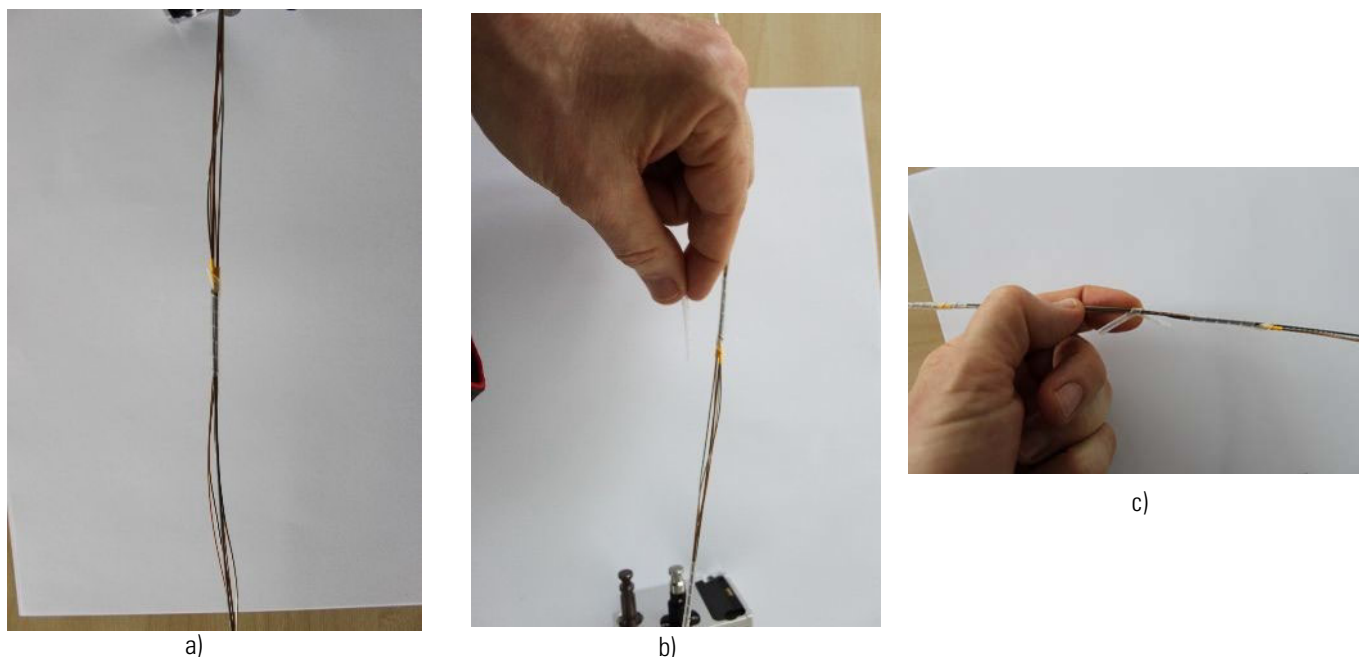


Figure 5-42. Needle line attachment with spiral tube (BRE0029733) available spare part. a) Correctly installed needle lines to the wire; b) spiral tube cut; c) How to curl a cut spiral tube around the needle wire and to tighten the needle lines to the wire.

GasBench Plus TriPlus RSH SMART for Gas Bench Trays and Tray Holder

The GasBench Plus has a mounting plate on which all trays can be mounted even in duplicates, except for the extended tray. Each of the trays is preprogrammed in the firmware and can be enabled as installed in Qtegra on the different slots. Any combination can be used except for a thermostatted tray on the right and non-thermostatted racks on the left.

Connect the capillaries to the corresponding ports on top of the GasBench Plus. Connect the capillary from the helium port to the long needle. Connect the capillary from the sample port to the short needle.



Figure 5-43. Capillary ports

Installation

Tools for the TriPlus RSH SMART for GasBench Plus Standard and Advanced Autosamplers

1. General Gas Supply

The fused silica capillary from the general gas supply of the GasBench Plus to the helium port should have a length of 1.9 m and 0.32 mm ID at 0.8 bar head pressure (flow rate of 20 mL according to the publication by Casciotti and Sigman. See [page 10-2](#)).

2. Vent line

Extend the vent line to 2 m to avoid a memory of the sample gas before. Use the provided stainless steel capillary.

3. 8-port valve

Connect the capillaries to the 8-port valve.

The installation of the sample needles is complete.

Installing a CTC Pal 80 Autosampler for GasBench II

The CTC Pal 80 autosampler is installed on a base plate, which is produced for the GasBench II system. The GasBench II system consists of the CTC Pal 80 autosampler and the GasBench II and specific tray systems (GasBench II trays). The *GasBench II Operating Manual* describes the hardware installation and how the communication of the CTC Pal 80 is implemented and set for operation with Qtegra ISDS for GIRMS.

NOTICE

Qtegra ISDS works on a Windows 10 operating system (64bit). Install the PAL Loader software version 2.1.1.

Software Installation for the CTC Pal 80 using Qtegra for GasBench II - Firmware, PalLoader and A200S Files

When running the CTC Pal 80 for GasBench II with Qtegra, ensure the following items are pre-installed on the CTC Pal Control board or on the Qtegra measurement computer. The files are delivered on the installation USB stick (...Firmware\CTC Pal 80 for GasBench II) - see [Figure 5-44](#).

1. PalLoader 2.1.1 (Windows 10 operating system)
Installation and Communication to the CTC Pal 80 and exchange of pre-defined CTC Pal 80 “x.sss” autosampler system installation files for GasBench
2. Check the Firmware and set to A200S mode (Firmware version: Pal-Firmware 2.3.3)
3. A200S files (xxx.sss files for GasBench autosampler installation).
See *GasBench II Operation Manual*.

Name	Date modified	Type
PAL Loader Version 2.1.1	6/2/2022 10:31 AM	File folder
sss files	6/2/2022 10:33 AM	File folder

Figure 5-44. Installation files for the CTC Pal 80 A200S mode for GasBench II

PalLoader 2.1.1 Installation and Communication to the CTC Pal 80

Make the PAL Loader active and available.

1. On the Qtegra computer, navigate to the PalLoader 2.1.1 zip file
\\Firmware\CTC Pal 80 for GasBench II\PAL Loader Version 2.1.1\

Installation

Installing a CTC Pal 80 Autosampler for GasBench II

2. Extract the zip archive to the Windows 10 program location (C:\Windows\).
3. Open the PAL Loader application and check the communication to the CTC Pal 80 (here: COM1 serial port in the Qtegra measurement computer). See [Figure 5-45](#).
4. Check the connection.

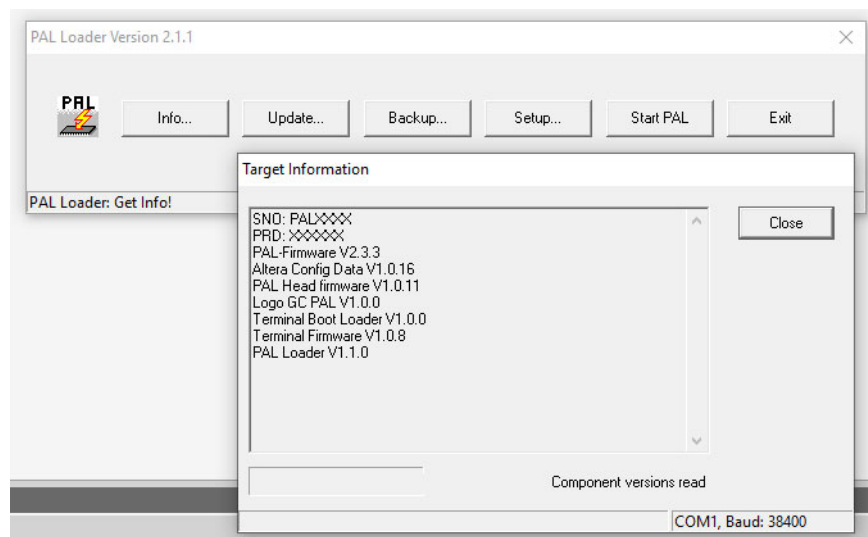


Figure 5-45. PAL Loader for Windows 10. Clicking Info opens the Target Information window showing the serial port COM1 to be used on Setup.

A200S Mode with the CTC Pal 80 - Firmware 2.3.3

The CTC Pal 80 must either be updated or downgraded to be able to operate in the A200S mode. Qtegra can only operate the CTC Pal 80 if the CTC Pal 80 is in A200S mode.

❖ To update the firmware

The steps are only needed if the Target Information window does not show “Firmware V2.3.3” as the installed firmware on the CTC Pal 80.

1. Use the “2-5-2_GC1_K.sss” file for a firmware update with the PAL Loader. Click **Update** and select the appropriate .sss file for the firmware update under: ...\\ThirdParty \CTC PAL 80 for GasBench II\sss files\...\\.
2. After installation of the .sss file, click **Start PAL** on the PAL Loader window to send a <Start PAL> command (see [Figure 5-45](#)).
3. On the CTC Pal 80, check if the firmware version is 2.3.3. If the system shows another version, it is not flashed on the CTC PAL control board.

4. Select the “BKGasbench.sss” file to update the PAL firmware update with the PAL Loader. Click **Update** and select the appropriate .sss file for the firmware update under: ...\`ThirdParty \CTC PAL 80 for GasBench II\sss files\...\`.
5. After installation of the .sss file, click **Start PAL** on the PAL Loader window to send a <Start PAL> command (see [Figure 5-45](#)).
6. Click **Exit**.

NOTICE

Do not disconnect the Pal from power during update.

NOTICE

Further .sss files are available as follows.

1. Select “BKGasBench New.sss” for a new setup to avoid injection into a flush or injector after stand-alone operation of the CTC Pal 80 with a PAL installed own sequence.
2. Select “GB 6x9_10ml_20ml_50ml_100ml_250ml trays Denitrification_Kit_2018-10-25 (1).sss” for a new setup of trays for Denitrification or PreCon using special GasBench II sample trays.
3. Click **Update**.
4. Click **Start PAL** on the PAL Loader window to send a <Start PAL> command (see [Figure 5-45](#)).
5. Click **Exit**.

Installation

Installing a CTC Pal 80 Autosampler for GasBench II

Basic Operations

This chapter describes basic operations to be performed with the GasBench Plus device.

NOTICE

CTC Pal 80 - A200S mode differences are explicitly described in this manual. For GasBench Series modes (GasBench Plus & GasBench II), the workflows are exactly the same. Hardware related items are described in the *GasBench II Operating Manual*.

Contents

- [Leak Check](#) on [page 6-2](#)
- [Checking the Column Flows](#) on [page 6-3](#)
- [Condition Test](#) on [page 6-5](#)
- [Blanking modes for GasBench Plus with and without ConFlo IV](#) on [page 6-6](#)
- [Starting a Qtegra LabBook](#) on [page 6-14](#)
- [Preparing a Test Sample](#) on [page 6-15](#)
- [Performance Test of the GasBench Plus Device](#) on [page 6-19](#)
- [Performing Sample Measurements with a Single Trap Sample Gas Injection Using Qtegra](#) on [page 6-20](#)

Leak Check

To check whether the IRMS is ready to operate, close the inlet valve and run a mass scan from 3000 magnet steps to 12000 magnet steps. Use the cup with the largest amplification factor (cup 3 with $3 \times 10^{10} \Omega$). For details, refer to the *DELTA Q Operating Manual*.

A mass scan shows the composition of the background gas in the source region and informs about the amount of gases present. Try to identify the following patterns and compare them with the maximum values below.

Water

For the isotope ratio measurement, the water background must be stable to ensure the principle of identical treatment of all samples and standard samples.

Water contains ions of m/z 16, m/z 17 and m/z 18. The peak intensity should be at most 1 V. The intensity ratio of the three peaks is 1:2:4.

Air

In principle, air response changes the stability of ion source ionization. Therefore, it is important to avoid any leakage of air into the ion source and the GasBench Plus device.

Air contains ions of m/z 28, m/z 32, and m/z 40. The maximum intensity for m/z 40 is 30 mV. The intensity ratio of the three peaks is 4:1:0.7.

Carbon Dioxide

Any CO_2 interference from a source other than the sample CO_2 changes the accuracy of the isotope ratio of samples. Therefore, a constant CO_2 response or better no CO_2 response shall be visible during measurement.

CO_2 contains ions of m/z 28 (CO) and m/z 44 (CO_2). The intensity of m/z 44 must be less than 50 mV. The CO portion can easily be confused with nitrogen from air.

If air appears in the spectrum, check the IRMS for leaks, for example by using argon from a tank. If the water level is too high, heat out the IRMS with the source heaters for at least 12 hours. When a high water level is present in the source, usually air is leaking into the mass spectrometer as well.

Once this check has been performed within the given limits, open the inlet valve and repeat the mass scan. If air appears in the spectrum again, check all gas connections at the GasBench Plus device for air leaks. Check all connections under excess pressure as they may leak, too. The best way to find leaks in the excess pressure section is to use a standard soap solution (SNOOP™, for example), which is applied to the connectors. Small bubbles appear when gas is leaking.

NOTICE

The connection between plot column and safety column located in the GasBench Plus oven is critical. Be careful when you tighten the connectors. Do not use excessive force. Tighten only if you are absolutely sure that the connection is leaking.

If the water level is too high after the leak check, heat out the GC column at 140 °C overnight. The GC column accumulates water by and by and releases it when heated. The water level only decreases after prolonged heating and continues to fall for some time even after heating is switched off (provided that there are no leaks).

Leak testing is especially laborious in the gas sampling section. A leak in this section has no continuous connection to the mass spectrometer. Instead, the Valco™ eight port valve needs to be switched to introduce a portion of the gas stream into the IRMS.

NOTICE

When you check this section comprising sample bottle, sampling needle connectors, water trap and the appropriate connectors at the Valco valve, do not to overtighten the connections.



When you replace ferrules in this section, make sure that you use only the listed Valco ferrules. Refer to Technical Note 503: Fitting Instructions at www.vici.com.

Checking the Column Flows

For an optimal operation, certain flows in the GasBench Plus device must be within a specific range. The bubble flow meter supplied with the GasBench Plus device can be used to check various flows throughout the system. Fill the small rubber ball with some soap solution and press it until bubbles appear in the inlet region. Connect the inlet tube to the capillary under test. The bubbles should then be transported along the tube by the gas flow under inspection. By measuring the time needed to fill a certain volume, the flow at the inlet tube can be calculated.

The flow through the sample needle should be checked regularly before each run using a flow meter. Measure at the exhaust capillary at the Valco valve, which is connected to port 7. Measure, while a closed bottle is attached to the sample needle and while the Valco valve is in Load

Basic Operations

Checking the Column Flows

mode. For normal operation, the flow should be in the range between 0.5 and 0.8 mL/min. Measuring at the exhaust of the loop allows checking the complete sample transfer path.

To check the flow through the flushing needle, a bottle must be connected and the flush valve be open. This flow is measured at the open exhaust capillary at the bottle connection of the flushing needle and should be in the range between 100 and 150 mL/min for normal operation.

Checking the flow in the GC column is more difficult. Because the GC column is the restriction for the gas flow, the flow can only be measured behind the column. The best point is the exit of the GC column. Carefully remove the capillary that leads to the second water trap and measure the flow, which should be between 1 and 1.5 mL/min for normal operation.

NOTICE

During the removal of the capillary, be careful when you tighten the ferrule. Excessive force may lead to the destruction of the ferrule or even the bulkhead connector at the GC housing.

Condition Test

A simple way to check the condition of the GasBench Plus device alone, that is without a set of individual sample vials, is to gently flush the sample line with a 0.3–0.5% (CO₂ in He) mixture. The check should be performed using a filled container of a larger volume, 500 mL, for example. The following parameters can be optimized by this check:

- temperature and flow of GC column (PoraPLOT™ Q)

GC temperature changes the separation between peaks belonging to the same sample injection (aliquot). GC column flow shifts all GC peaks in time: higher flows mean shorter retention times and vice versa.

- retention time and GC peak shapes (DtR N₂/CO₂)

Retention time depends on column type. Peak shapes tend to be tailed, if the column is heavily used and needs recovering. See “GC Oven” on page 3-22.

- time delay between METHOD/PROCESS (Dt loop injections)

Use this type of condition test when changing the timing to control the results of manipulations to the time events list.

- loop size (10–250 mL; sensitivity vs. peak shape)

Different loop sizes require different times for loading and injecting the loop. Calculate load times from loop volume and sample needle flow. Calculate inject times from loop volume and GC column flow. Allow extra times for safety.

- IRMS sensitivity (length of transfer line)

Frequently check the sensitivity of the whole apparatus by this test.

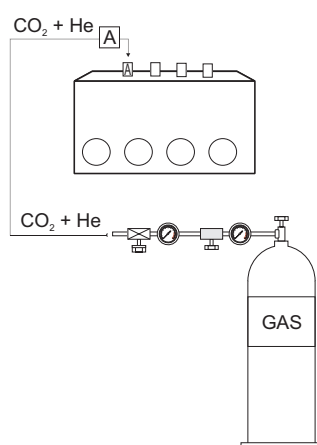


Figure 6-1. Basic test for sample section (autosampler and bottles excluded)

Blanking modes for GasBench Plus with and without ConFlo IV

Using the Autodilutor for Blanking with GasBench Plus without ConFlo IV

The atmospheric mixture used here contains lots of nitrogen and oxygen that severely distort operation of the source when reaching the inlet. To avoid this, the autodilutor arrangement has been modified to guarantee extreme dilution.

❖ **To obtain the modification**

1. Loosen the two screws.
2. Move the small metal plate that limits the movement of the pneumatic lever of the autodilutor fully upwards. See arrows in [Figure 6-2](#).

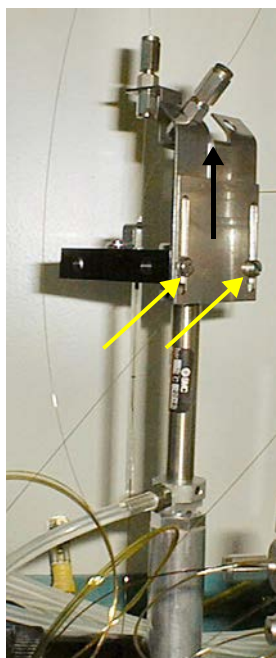


Figure 6-2. Adjusting open split for blanking

Additionally, the capillary feeding the split with helium needs to be retracted into the inner glass tube. See [Figure 6-2](#) and [Figure 6-3](#).

When unlimited in movement, the lever moves the capillary leading from the autodilutor to the IRMS into the inner tube of the autodilutor. In this position, the capillary samples almost entirely helium, and the dilution factor is larger than 100.

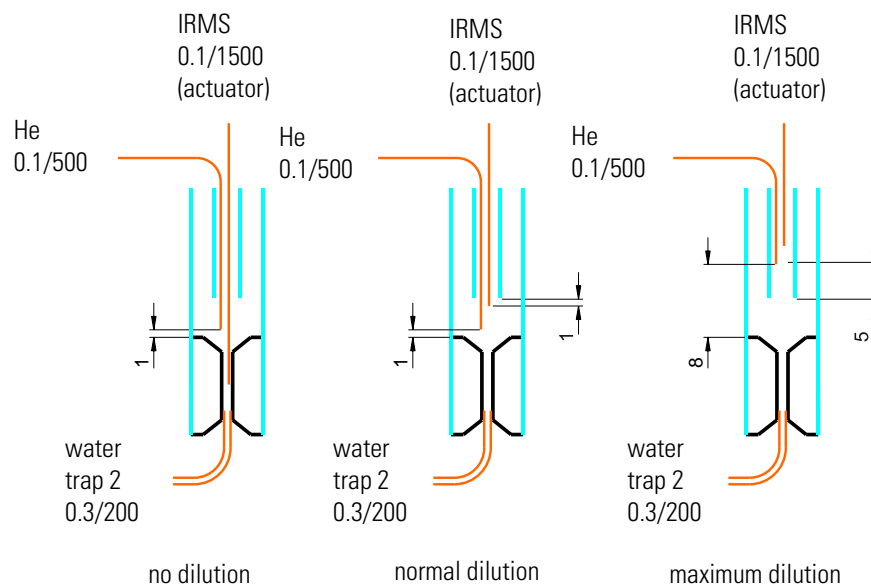


Figure 6-3. Principle of blanking

Using the Blanking Mode in GasBench Plus with ConFlo IV

If the blanking is activated under Qtegra LabBook time line item (see [“Using the Blanking Mode in Qtegra with ConFlo IV”](#) on page 6-8), it can be used. For blanking with ConFlo IV, Qtegra will only activate the blanking with the sample dilution and the MS in High Flow mode.

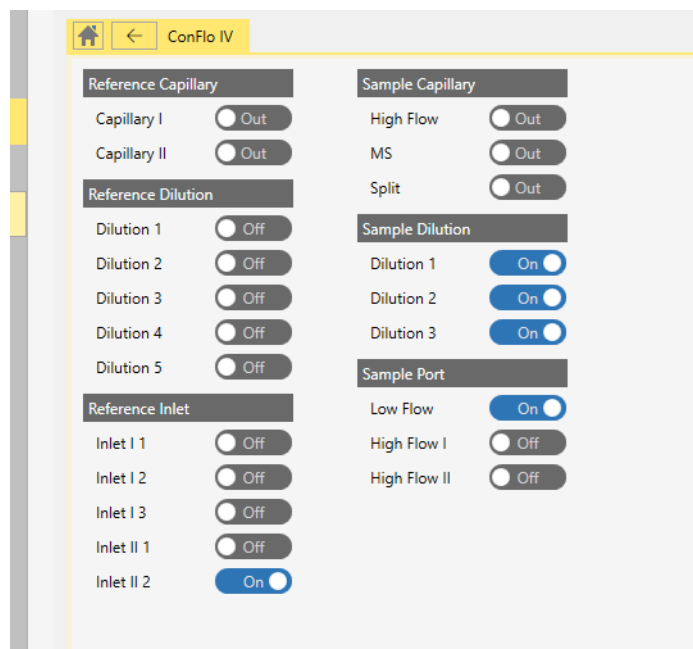


Figure 6-4. Valves setting in ConFlo IV if blanking mode is activated in the LabBook

NOTICE

The description is also valid for Qtegra with the GasBench II and ConFlo IV.

Using the Blanking Mode in Qtegra with ConFlo IV

Qtegra ISDS Software offers two modes for blanking:

- Blanking mode for multiple sample peaks - Blanking for each individual sample peak. Qtegra decides by the settings in the Qtegra Configurator if a blanking without a ConFlo IV (split changes- see [“Using the Autodilutor for Blanking with GasBench Plus without ConFlo IV”](#) on page 6-6) or with a ConFlo IV will be applied (see [“Using the Blanking Mode in GasBench Plus with ConFlo IV”](#) on page 6-8).

- Blanking mode for single peaks applied with a Single or Dual trap measurement. Here, the blanking must be independently set from the injection of the liquid nitrogen trap.

NOTICE

The two different blanking modes will be defined as described in the following chapters under the Continuous Flow Method Parameters (see [“Method Parameters - Continuous Flow”](#) on page 7-26).

Results in Blanking with GasBench Plus and Qtegra Setup for Blanking

Here a blanking is depicted, which shows the blanking of an interference peak (N_2) before the sample peak of CO_2 (not visible at the right side of the chromatogram (see [Figure 6-5](#)). The blanking is an almost 100% dilution of the sample gas during action by dilution with retracting the capillary into an inner glass tube or by the 100% sample gas dilution in the ConFlo IV and MS retraction into the HF side of the ConFlo IV.

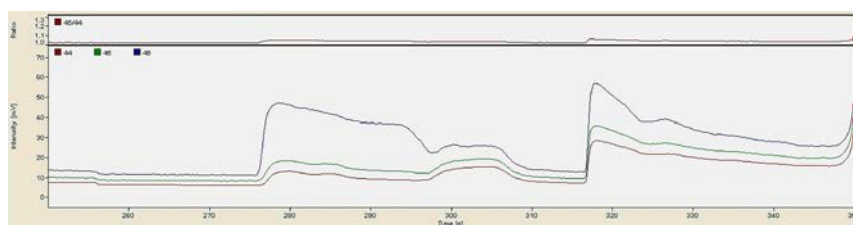


Figure 6-5. Blanking - chromatogram (principle shown)

Basic Operations

Blanking modes for GasBench Plus with and without ConFlo IV

Otegra Blanking Mode for Multiple Sample Peaks

Qtegra will use the correct blanking mode independent whether a ConFlo IV is installed or not, e.g. using ConFlo IV dilution (see [Figure 6-6](#)) or using blanking by GasBench split blanking without ConFlo IV (“GasBench standalone”, see [Figure 6-7](#); see chapter for hardware changes for blanking).

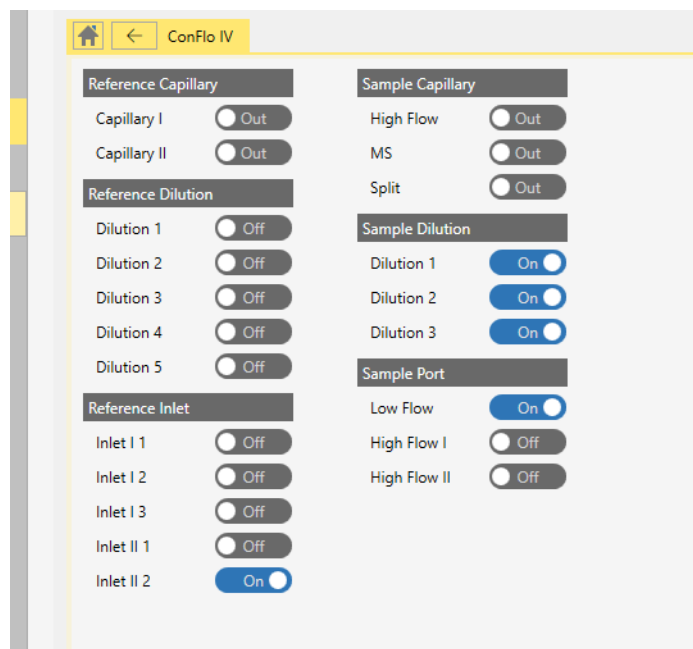


Figure 6-6. Blanking and dilution as with ConFlo IV

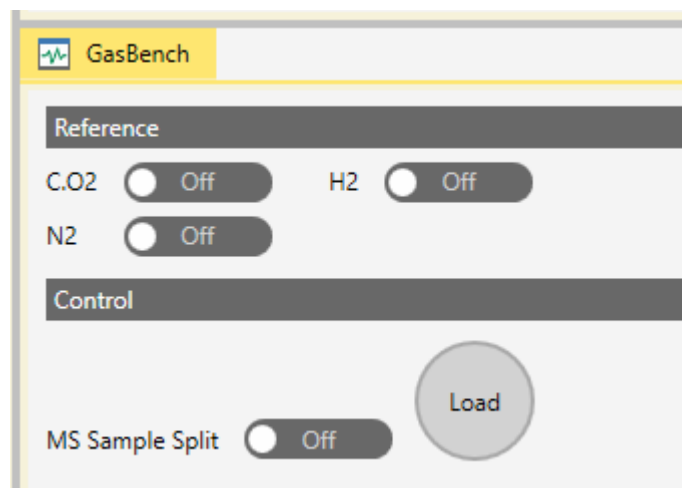


Figure 6-7. No blanking split in & blanking - Split out

Qtegra will be configured to the following mode for blanking with multiple peaks independent from a ConFlo IV is set up or not (see [Figure 6-8](#)).

The blanking can be set before, e.g. N₂O if CO₂ is analyzed or after the sample peak, e.g. CO₂ if N₂O is analyzed.

❖ To set the blanking timeline parameters

1. Set up reference gas peaks.
2. Set up a Sample Pulse Width 20 s (inject, Interval 30 s, load).
3. Set blanking to *On*.
 - a. Define the start of the blanking time.
 - Blanking Start: Time when the first peak appears, e.g., Sample Puls Start time plus the retention of the Interference Peak Start time. For example, as shown in [Figure 6-8](#), 60 s pulsing plus 70 s (blanking) result in an autodilution split or ConFlo IV dilutor action at 130 seconds.
 - b. Blanking Interval:
 - Interval time for active blanking time interval.
 - c. Blanking will pulse according to the sum of the pulse time and the interval, e.g. 50 s.
4. Set End time as after the last sample peak appears.

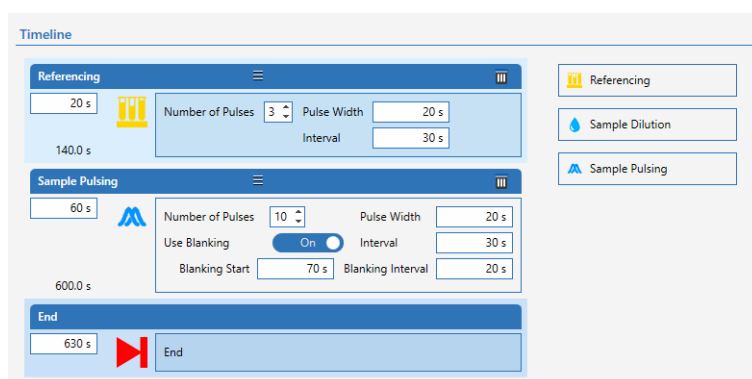


Figure 6-8. Qtegra timeline settings for blanking with multiple peaks, e.g., GasBench series configuration in standard mode without any traps

Blanking Mode for Single Peaks Applied with a Single and a Dual Trap Measurement

The blanking must be set independently from the injection of the liquid nitrogen trap. The load mode will not be activated anymore by setting the injection. The Dual Trap is used in the prescripts and the first trap of two will not have any active influence on the sample injection in the chromatogram.

For Single peak blanking Qtegra will be set up in the following process workflow (see [Figure 6-9](#)):

Basic Operations

Blanking modes for GasBench Plus with and without ConFlo IV

1. Prescripts will be active for sample processing (see [Qtegra for GasBench Applications](#)).
 - a. Initialization of the procedure
 - b. Autosampler activation
 - c. Trap Down. Last trap freezes the sample gas.
2. Set up the timeline.
3. Set up reference gas peaks.
4. Set the Trap with time and action in the timeline, e.g. 410 s. Set the State for injection to *Up*.
5. Set up a Sample pulsing only for one peak, e.g. 390. Consequently, pulsing is independent from injection.
 - a. pulse width: 450 s (inject)
 - b. Set the interval to 0 s. The Valco is set to *Load* for the total measurement time.
6. Set blanking to *On*.
 - a. Define the start of the blanking time, e.g. 70 s.
 - Blanking start: Time when the one interference peak will appear. Sample Pulsing time set plus Retention time of the Interference Peak Start, e.g., 390 s + 70 s = 460 s as Blanking Start, see [Figure 6-9](#).
 - b. Blanking Interval:
 - Interval time for active blanking time, e.g. 30 s

7. Set End time as after the sample peak or the blanking would have appeared (840 s), for N₂O sampling will appear before blank peak, for CO₂ the blanked peak will appear before the CO₂ analyte gas.

The screenshot displays the 'Timeline' configuration window. It is organized into four main sections:

- Referencing:** Duration is 20 s. Number of Pulses is 5. Pulse Width is 20 s. Interval is 30 s.
- Sample Pulsing:** Duration is 390 s. Number of Pulses is 1. Pulse Width is 450 s. Interval is 0 s. 'Use Blanking' is set to 'On'. Blanking Start is 70 s. Blanking Interval is 20 s.
- Trap:** Duration is 410 s. Trap is set to 'Trap 1'. State is 'Up'.
- End:** Duration is 840 s.

Figure 6-9. Blanking for GasBench series single trap mode. With setting a pulse to 1 and having blanking activated, the blank start can be set accordingly. Blanking action starts 70 s after 390 s pulsing, here: 460 s.

Starting a Qtegra LabBook

Before you start an automated sequence of valuable samples, check the GasBench Plus device for certain items:

- Frequently check the sample needle, flush needle and acid needle for remainders of the vial septa. Small parts can be removed using a syringe tip. Check the flow through the sample needle (0.5–0.8 mL/min) at the exhaust (vent) connection of the Valco eight port while a sample is connected.
- From time to time, at least once a month, heat out the GC column. Set the temperature regulator to 140 °C and keep this temperature constant for 12 hours.
- From time to time, check whether the water background of the IRMS is within acceptable limits, that is less than 3 V. See [“Water” on page 6-2](#).
- Check the fused silica capillary of the sample needle for remnants of phosphoric acid or water. Remove droplets before starting the analysis.



To start a LabBook, click the **Start** button in Qtegra Acquisition.

Setup for GasBench Plus

External Flush and Acid Addition sequence with the Qtegra GasBench Plus Dashboard. See description on [“Stand-Alone Flushing or Automated Flushing and Acid Addition \(“Sample Preparation”\)” on page 7-20](#).

Preparing a Test Sample

The basic principle of the GasBench Plus technique is the measurement of any gas (CO₂, for example) from the headspace in a vial. Therefore, it is unimportant for the GasBench Plus measurement how the gas was produced and released into the headspace. For a basic system check (that is, with no sample involved), a gas mixture is ideal.

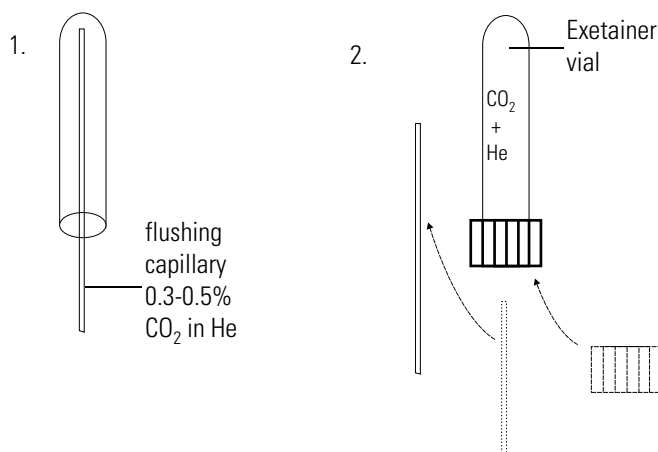


Figure 6-10. Flushing an empty Exetainer for preparation of a test sample

Prepare this gas into an Exetainer™ by flushing the vials with a mixture of 0.3-0.5% CO₂ in He. The flow should be about 100 mL/min. Hold the tube upside down onto the flushing capillary for approximately 20 seconds and close the tube immediately after flushing. See [Figure 6-10](#).

A more convenient way to fill the (He + CO₂) mixture into an Exetainer vial is to use the flushing needle (see [“Flush Needle”](#) on [page 3-16](#)) together with the TriPlus RSH SMART autosampler. To fill the Exetainer properly, each tube is rinsed for approximately 5 minutes with (He + CO₂) at a flow rate of 100 mL/min.

Tip To guarantee high performance, wash the Exetainer before using it. See [“Manual Cleaning of the Sample Vials”](#) on [page 9-15](#) and [“Machine Cleaning of the Sample Vials”](#) on [page 9-16](#).

NOTICE

The autosampler vials can be flushed automatically with an extra GasBench tool using a flush needle. Qtegra can operate this by enabling the flush mode in the LabBook or by using an external flushing within the GasBench Plus Dashboard sequence. See [“Stand-Alone Flushing or Automated Flushing and Acid Addition \(Sample Preparation\)”](#) on [page 7-20](#).

After Preparing a Test Sample

After preparation of the test sample, there are two options to proceed:

- Take a predefined LabBook of the CO₂ Acceptance Test for CO₂ and run it as a guideline for a measurement.
- Define a LabBook for Acceptance Test for a measurement.

Acceptance Test with a Purge Gas Mixture to Test Isotope Analysis Reproducibility, e.g., CO₂ Acceptance Test

An acceptance test is a flushing of the gas mixture into a number of empty vials and a measurement of those multi number of purged vials after addition of the purge gas, e.g. CO₂, H₂, N₂O or any other gas mixture used for IRMS analysis. Ensure that the reference gas peak appears 20 sec after purging.

The Acceptance test allows to judge on the ability of the system to measure reliably. It recommended for beginners to do this test more often. At a later stage, this test is not done so often anymore.

Using a Predefined LabBook for Acceptance Test

From the LabBook tab, select one predefined Qtegra LabBook. From the predefined LabBook containing a TuneBook setup for CO₂, a new LabBook can be created with a different TuneBook using a different Gas Configuration (see the *DELTA Q Operating Manual* for the description of using a LabBook).

❖ To prepare the instrument and LabBook for measurement

1. Make sure that the instrument Get Ready is started either with Peak Center or not.
2. Make sure that the vials are prepared and placed in the tray before flushing. Do not forget to install or open the appropriate flush gas connection for automated flushing of vials.
3. Set up the Continuous Flow parameters
 - a. For generic setup read the chapter “[Setup for GasBench Plus](#)” on [page 7-26](#)
 - b. Enable to pre flush sample. Do not equilibrate.
 - c. In the Timeline parameters (see [Figure 7-21](#)):
Set Referencing.
Set Sample Pulsing (10 times) with approx time of 60 to 80 seconds.

- Set End time.
- d. Add a reference gas standard.
 - e. Add the same as a Delta Standard.
4. Select an appropriate line in the sample list where the vials are positioned. Next to the first or second analyte, set the predefined gas standard as standard and the LabBook will do an automated evaluation against the standard delta values, e.g., ^{13}C of CO_2 or ^{18}O of CO_2 gas.
 5. Click **Run** to run the LabBook.

The external referencing delta values are calculated automatically during the run.

Thermo Fisher Scientific recommends performing a peak center before the acquisition. Define CO_2 as a reference three times (duration: 20 seconds) and take the middle one as standard. Qtegra ISDS Software has an appropriate default timing set.

Tip When the LabBook is running or finished, the averaged results for all vials are calculated fully automatically if the standards are set properly next to the sample list item. The standard deviation of these newly obtained results must be less than 0.1‰ for both $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$.

A measurement can be significantly shortened by using only four to five sample pulses in the timeline.

Figure 6-11 shows the CO_2 acceptance test Template.

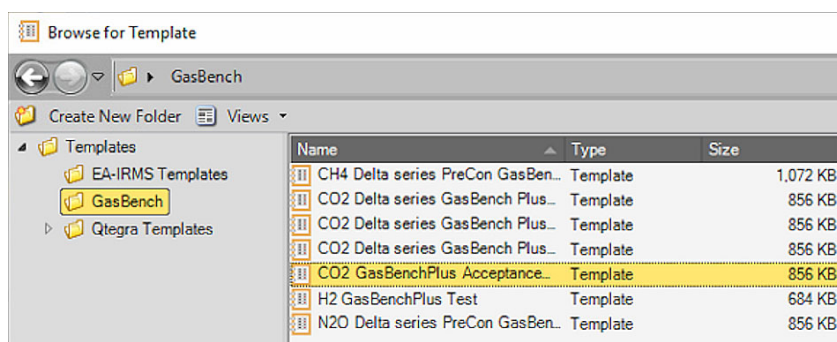


Figure 6-11. CO_2 acceptance test Template

Figure 6-12 shows Acceptance test standards.

The screenshot shows a software interface titled "Standards". It has two main sections: "Standard(s)" and "Selected Delta(s) for 'CO2'".

Standard(s) Table:

Name	Description
CO2	GB-IRMS: acceptance
CO2 lab tank 3	RefGas: GasBench

Selected Delta(s) for "CO2" Table:

Delta Identifier	Delta Value (%)	Primary Standard	Primary Standard Ratio
13C	0	VPDB 13C/12C	0.0111802
18O	0	VPDB 18O/16O	0.0020672

Figure 6-12. CO₂ acceptance test standards

Figure 6-12 shows a Sample List for a CO₂ acceptance test. A CO₂ acceptance test gas is used to determine the external standard deviation. Hydrogen can be used in analogy, but a H₃ factor must be determined beforehand.

The screenshot shows a "Sample List" table with 10 rows. The columns are: Label, Status, Comment, Action, Rack, Val, Evaluate, Sample Type, and Reference.

Label	Status	Comment	Action	Rack	Val	Evaluate	Sample Type	Reference
1 CO2 Acceptance	⊕	<Comment>	Measure		1	1	Delta Standard (BSIA)	CO2
2 CO2 Acceptance	⊕	<Comment>	Measure		1	1	Unknown	
3 CO2 Acceptance	⊕	<Comment>	Measure		1	1	Unknown	
4 CO2 Acceptance	⊕	<Comment>	Measure		1	1	Unknown	
5 CO2 Acceptance	⊕	<Comment>	Measure		1	1	Unknown	
6 CO2 Acceptance	⊕	<Comment>	Measure		1	1	Unknown	
7 CO2 Acceptance	⊕	<Comment>	Measure		1	1	Unknown	
8 CO2 Acceptance	⊕	<Comment>	Measure		1	1	Unknown	
9 CO2 Acceptance	⊕	<Comment>	Measure		1	1	Unknown	
10 CO2 Acceptance	⊕	<Comment>	Measure		1	1	Unknown	

Figure 6-13. CO₂ acceptance test standards

Performance Test of the GasBench Plus Device

From time to time, take a look at the checklist shown below. It shows the performance achievable by the TriPlus RSH SMART for GasBench Plus device plus IRMS. Check all mentioned items.

❖ To test the performance of the GasBench Plus device

1. Perform a basic test, that means test the IRMS alone as described in the *DELTA Q Operating Manual*.
2. Perform the Zero Enrichment test as described in the *DELTA Q Operating Manual*.
3. Carry out the Linearity test as described in the *DELTA Q Operating Manual*.
4. Make sure that also the sample side (that is Valco valve, GC column) operates properly by performing the Condition test. See “[Condition Test](#)” on [page 6-5](#).
5. Carry out the Acceptance Test with vial filling performed manually or automatically.
6. Only if all the previous items are performed to specifications, perform the measurement.

Performing Sample Measurements with a Single Trap Sample Gas Injection Using Qtegra

A general workflow principle for a single injection strategy must always be applied as described in this chapter to avoid background increase and cleaning of the trap before sample gas trapping in the concentration and injection trap. The procedure with Qtegra requires a prescript upload in the Peripheral part. In the Timeline section, configure a mode with the single injection of the trap plus blanking if an interference injection from air or similar gases to the sample gas peak is expected. The principle is described under *Fiebig et al. 2005*.

Preparation Workflow Principle

Peripheral Part (prescript)

1 s INJECT - Split out / HF capillary in to avoid increase background.
10 s Wait
Trap Down
20 s Wait
LOAD

Timeline

0 s Reference IN
20 s Reference OUT
Reference Pulsing as in the LabBook: Pulse and interval by number of required peaks), e.g. 20 s pulse, 30 s interval
240 s One new referencing pulse (optional ratio drift correction reference gas pulse), start 240, 20 s pulse, 30 s interval
391 s Sample Pulsing with only one pulse (see Notice below). Set pulse width until the measurement end, e.g. 790 s.
410 s Trap UP
490 s Reference IN
510 s Reference OUT
790 s End timeline.

NOTICE

Referencing must be optimized by the user.

NOTICE

The pulse width of the one pulse must be extended until the measurement end because Valco must not be in inject mode during the entire timeline.

For single trapping measurement sampling with and without blanking mode (see [“Analyzing CO₂ in Atmospheric Concentrations”](#) on page 8-7, [“Analyzing Carbonates”](#) on page 9-38).

Reference

Jens Fiebig, Bernd R. Schöne and Wolfgang Oschmann (2005)
High-precision oxygen and carbon isotope analysis of very small
(10-30 µg) amounts of carbonates using continuous flow isotope ratio
mass spectrometry. Rapid Commun. Mass Spectrom., 19: 2355-2358.

Basic Operations

Performing Sample Measurements with a Single Trap Sample Gas Injection Using Qtegra

Qtegra for GasBench Applications

This chapter includes all options (Single Trap/Dual Trap) and the TriPlus RSH autosamplers. It describes the standard configurations for the GasBench Plus and the configuration of the TriPlus RSH autosamplers for the GasBench Plus in Qtegra.

For using the Configurator and the Qtegra ISDS Software for GIRMS, see the *Qtegra ISDS Software for the Gas Isotope Ratio Mass Spectrometers Installation Guide*, the *DELTA Q Operating Manual*, and the *253 Plus Operating Manual*.

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- [Introduction](#) on page 7-2
- [Qtegra Configurator](#) on page 7-6
- [Qtegra Dashboard](#) on page 7-12
- [Qtegra Instrument Diagnostics](#) on page 7-24
- [Qtegra LabBooks](#) on page 7-26
- [Qtegra Templates](#) on page 7-52
- [Performing Preparation of Samples using Prescripts with Qtegra](#) on page 7-55

Introduction

The scope of this section is to explain main applications, schematic gas flow and the headspace gas sampling procedure with the GasBench Plus device.

The GasBench Plus device is a universal on-line interface, which allows automated isotope ratio determination of small gas samples (isotopic characterizations of CO₂ or N₂ between 200 nmol and 20 mmol of total sample size). The gas, that is CO₂, can either:

- be part of the original gas sample (breathed air, for example) or
- be released from liquid or solid phase into the headspace of the sample vial by different sample preparation methods (for DIC, carbonates) or
- be added to the original water sample (equilibration).

Using a gentle stream of helium, the CO₂ in the headspace of a sample container continuously passes through a Valco™ sampling port. Multiple analysis is achieved by switching the contents of the sample loop into a GC column every 90 seconds. Each switch corresponds to starting GC separation of the sample coming from the loop.

The GasBench Plus device is supported by TriPlus RSH SMART autosamplers for fully automated transfer of the gas samples, which are contained in a sample tube with a septum top. The GasBench Plus device covers a large variety of application areas. The same device can be used in:

- Hydrology (determination of ¹⁸O/¹⁶O and ²H/¹H from water samples)
- Global change research (¹³C/¹²C determination of dissolved inorganic carbon, DIC, from ocean water or fresh water) or
- Paleoclimatology (simultaneous ¹⁸O/¹⁶O and ¹³C/¹²C determination from carbonates of various sources).

Furthermore, it is possible to introduce traps to cryofocus methane and other trace gases in air mixtures or to determine ¹³C/¹²C concentrations in breath gas. The abilities in equilibration of oxygen and hydrogen isotopes can widely be used in food authentication.

The GasBench Plus device consists of the following components:

- a user-programmable autosampler
- a gas sampling system
- a maintenance-free water removal system
- a loop injection system

- an isothermal gas chromatograph (GC)
- an active open split interface
- a reference gas injection system with three reference ports
- an optional LN2 trap for cryofocusing
- an optional acid dosing system

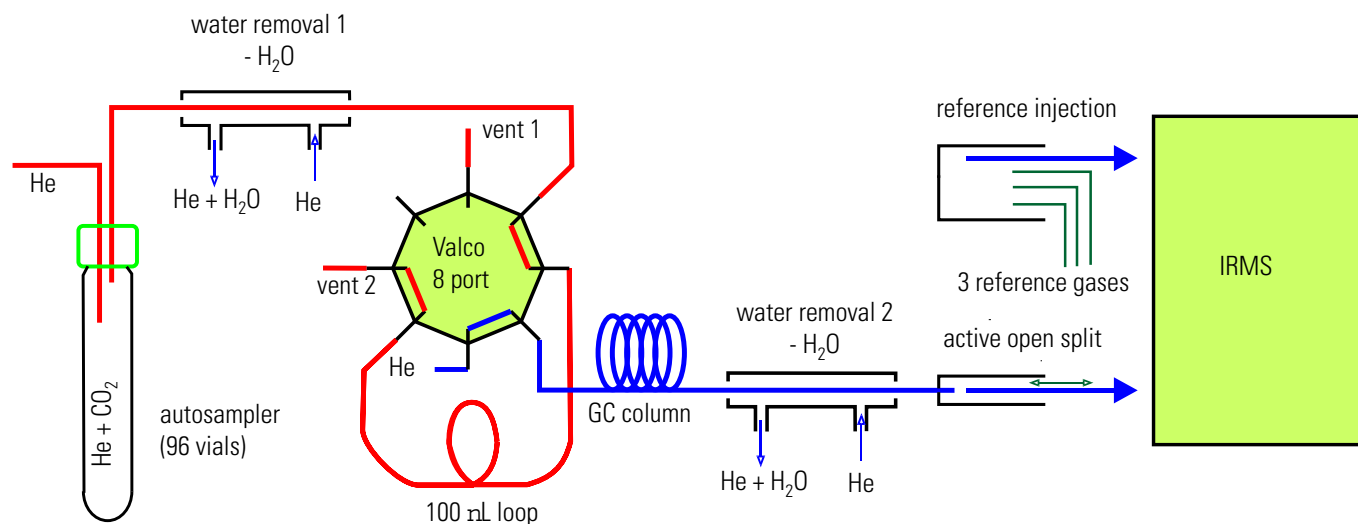


Figure 7-1. Schematic of components of the GasBench Plus device



For a description of the basic principles of Continuous Flow, refer to Habfast, K.: *Advanced Isotope Ratio Mass Spectrometry I: Magnetic Isotope Ratio Mass Spectrometers*. Chapter 3 in: Platzner, I.T., ed., *Modern Isotope Ratio Mass Spectrometry*, 1997, p. 11-82, John Wiley & Sons Ltd.

In all types of measurements, the isotopic composition of a sample gas is compared to the isotopic composition of a reference gas. The GasBench Plus device consists of a reference inlet system that allows to use three reference gases (Reference 1 or Reference 2 or Reference 3; only one of them per measurement). The GasBench can be attached to the Low Flow connection of the ConFlo IV and uses all its benefits. Only one standard low flow system can be attached, e.g., GC Isolink or GasBench Plus.

Usually, CO₂ and H₂ are chosen to cover all applications mentioned above. Reference gases are expected to be clean and stable with respect to their isotopic compositions. For a gas tank that contains a liquid phase like CO₂, this means absolute temperature stability.

The sample gas is fed into the GasBench Plus device by a specially designed headspace sampling needle. By a helium overpressure, the gas is transported through the capillaries into the GasBench Plus device

where a drying stage removes water from the sample gas mixture. Otherwise, it tends to clog the Valco valve or the mass spectrometer inlet valve.

The sample loop is filled with the analytic mixture. See “[Valco Eight Port Valve](#)” on [page 3-18](#). A portion of the sample gas mixture is cut from the continuous stream by switching the Valco valve to the inject position. The portion is injected into the GC column, where a separation in time between CO₂ and other gas components takes place.

To decouple the overpressure section of the GasBench Plus device from the mass spectrometer’s vacuum chamber, the gas mixture passes a second water trap and enters the open split arrangement. While a fixed amount of the gas mixture travels to the mass spectrometer, the excess gas leaves the split to the surrounding atmosphere.

The different gases contained in the original mixture arrive at the mass spectrometer source separated by polarity. Using a PoraPLOT™ Q, no time difference can be detected for O₂, N₂, H₂, and He. Their transfer time along the column is approximately 120 seconds depending on column pressure and temperature. CO₂ needs about 20 seconds longer, while more polar compounds like water or ethanol may transfer 300 to 500 seconds or get stuck on the column and “bleed off” only when the column is heated.

Headspace Sampling

In standard setup that is used for equilibration, DIC, and carbonate analysis, the sample gas is taken from the headspace of a sample bottle. In all of these cases, the gas to be measured is not identical to the substance whose isotopic value should be determined. This leads to numerous complications in sample preparation, sampling technique, and results interpretation.

First of all, the isotopic abundances in the liquid phases are different from those in the gas phase. This effect is most striking when measuring hydrogen isotopic ratios: here, the abundance of the heavier isotope in the gas phase is approximately four times lower than in the liquid phase due to thermodynamic mechanisms. The abundance of this isotopic dilution effect is described by a number usually denoted as α factor.



The α factor for HD is 4.00 and about 1.04 for CO₂ from dissolved CO₂.

Refer to Friedman, I. and O’Neill, J.R.: *Compilation of stable isotope fractionation factors of geochemical interest*. Chapter KK in: Fleischer, M., ed., *Data of geochemistry*, 6th ed., 1977, U.S. Geological Survey Professional Paper 440.

In equilibration techniques, the gas to be measured is added to the headspace. This requires the air in the headspace to be exchanged with helium or a mixture of helium and the gas to be analyzed. It is assumed that, after some time, an isotopic equilibrium is reached between the gas in the headspace and the molecules in the liquid. Only then the gas mixture can be analyzed. In carbonate analysis, the gas to be measured is released from the carbonate material by adding phosphoric acid.

A similar idea leads to DIC measurements. In both cases, the air in the headspace must be replaced prior to the reaction by helium which is inert and thus will not influence GC analysis. Measurement timing must take into consideration the times required for the reactions mentioned above as well as the times the autosampler needs to perform its injections. Nevertheless, one can use a single acquisition script for all analysis types.

If you take care of the reference gas settings in the LabBook (that is the reference port setting in the Dashboard or Diagnosis for GasBench and the reference port switching in the time events list), you can use the same LabBook as a Template for all GasBench Plus standard work. It is comprehensible that the LabBook must satisfy all analysis modes.

Tip Isotope ratio measurements with the GasBench Plus device can be performed especially quickly by using short methods. Recording only a few sample peaks considerably increases the number of samples measurable per day.

Qtegra Configurator

Standard Configurations for GasBench Plus and TriPlus RSH SMART Autosamplers

In the Qtegra Configurator, the GasBench Plus, PreCon and TriPlus RSH SMART are listed under **Peripherals** in the DELTA Q or 253 Plus mass spectrometers.

The following configurations are supported by Qtegra ISDS Software. See [Figure 7-2](#) and [Figure 7-3](#) as examples.

1. GasBench Plus with options, ConFlo IV, own referencing mass spectrometer and autosamplers
2. GasBench Plus with options, ConFlo IV and autosamplers
3. GasBench Plus with PreCon and the items listed in [step 1](#) and [step 2](#).

Refer to the *PreCon Operating Manual* for operation and measurement using Qtegra.

The GasBench settings and options are activated by a slider in the Experiment Configurator. See [Figure 7-4](#).

NOTICE

A standard IP address is installed via a Template in Qtegra ([Figure 7-3](#)) list under Qtegra Configurator including setup of a Default IP address by Qtegra TriPlus RSH.

If a DHCP controlled IP address is inserted, the user must configure the TriPlus RSH system and the LAN ports. The TriPlus RSH is set to standard LAN connection, and a disruption by DCHP cannot not be excluded, because of non-standard equipment setup at the installation site.

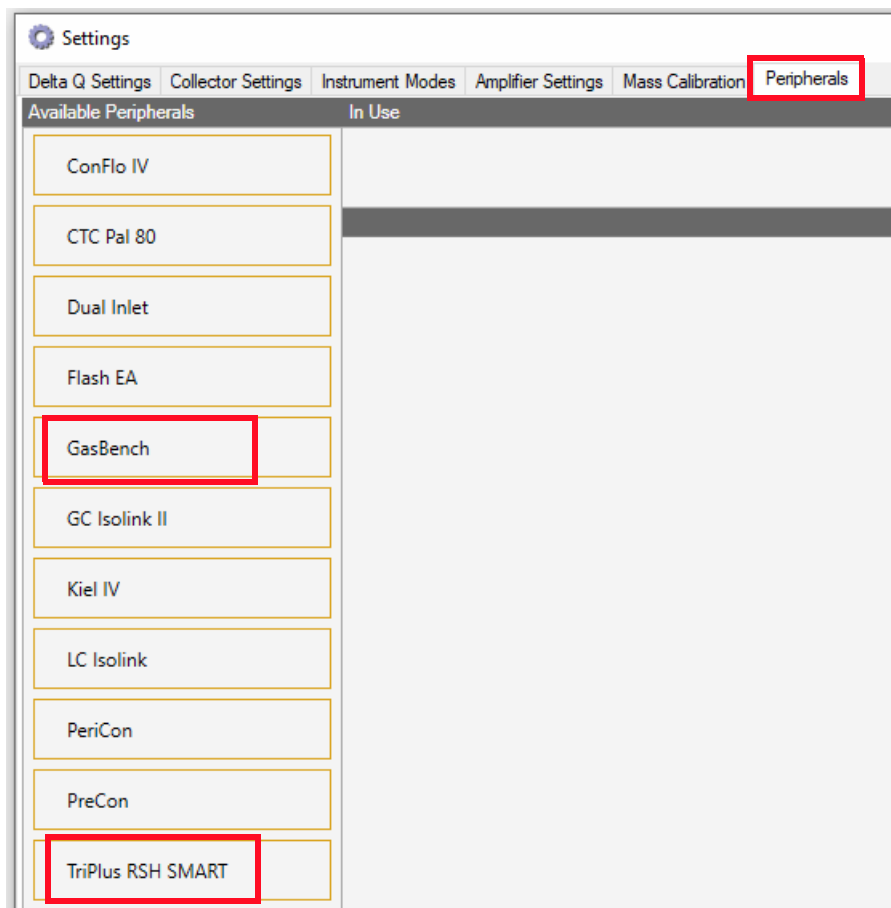


Figure 7-2. GasBench and TriPlus RSH SMART in the Peripherals list of the Qtegra Configurator

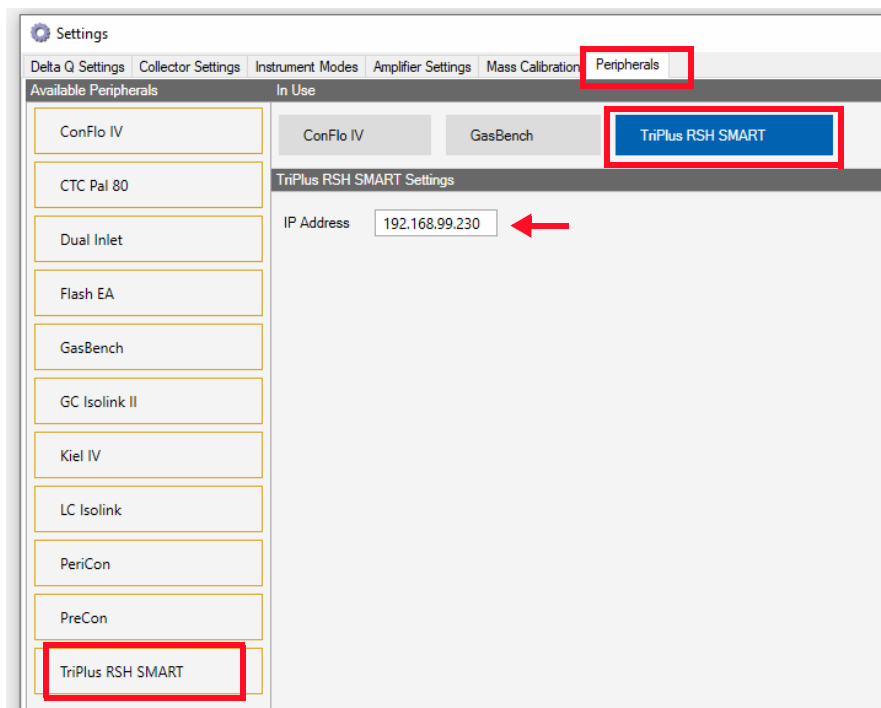


Figure 7-3. TriPlus RSH SMART in Peripherals list of Otegra Configurator – including setup of default IP address by Otegra for the two TriPlus RSH SMART for GasBench Plus models

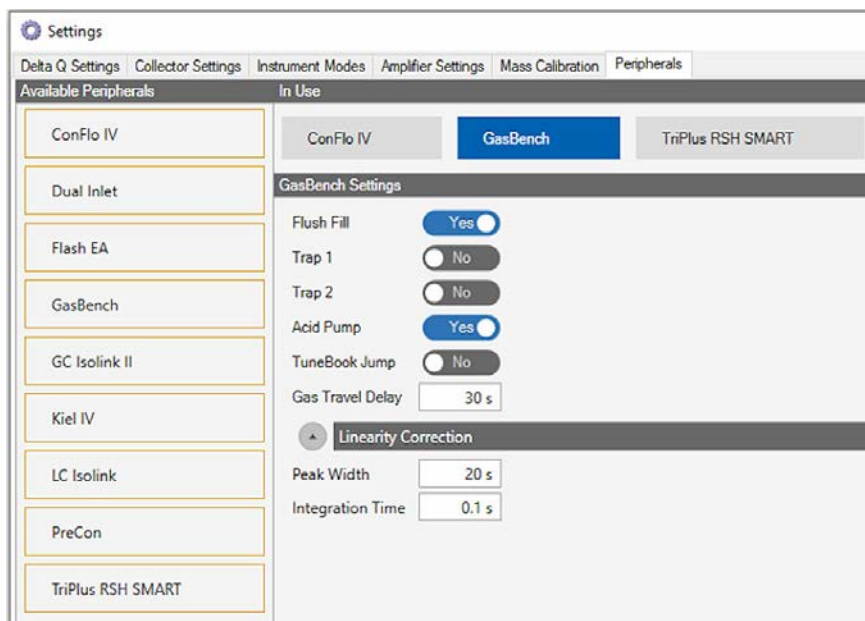


Figure 7-4. Settings for GasBench Plus with ConFlo IV referencing

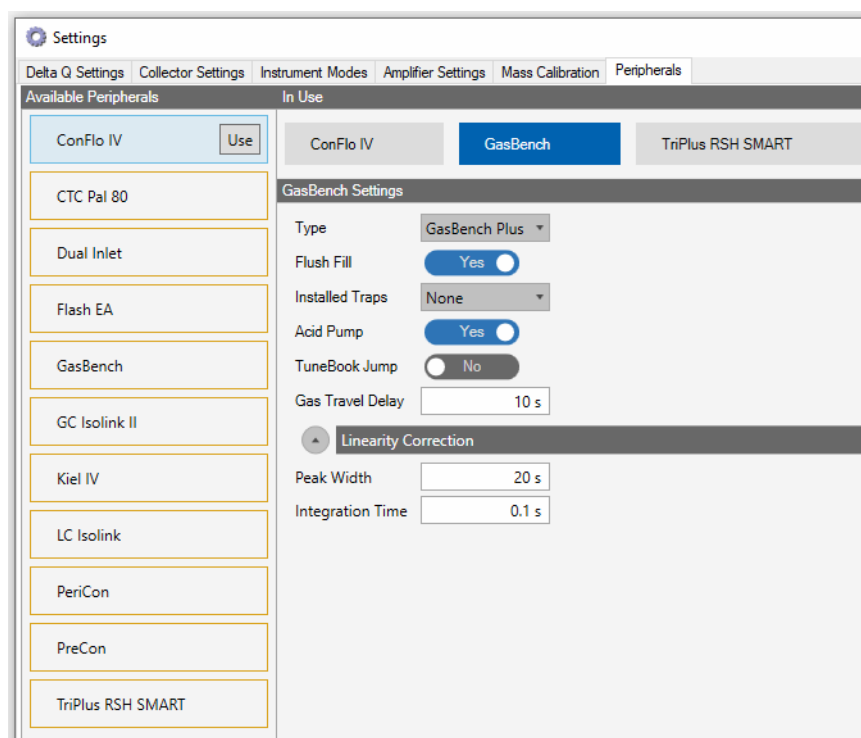


Figure 7-5. Settings for GasBench Plus with internal referencing

Tip The additional options or setting of the GasBench will be validated in the Qtegra ISDS Software. Options or items not in use will not be depicted or operational if not enabled or configured in the Configurator.

CTC Pal 80 - A200S Mode Autosampler Configuration with GasBench II

The CTC Pal 80 will be configured with a GasBench II in the Qtegra Experiment Configuration (see [Figure 7-6](#)). The generic CTC Pal 80 serial port is COM1. If another device is used, e.g., an EA IsoLink, the serial port of the CTC Pal 80 can be set to a different COM port of the measurement computer or a USB port connector. The COM port number has to be changed in the instrument Configurator (see [Figure 7-7](#)). Use GasBench II as the active Configuration with the CTC Pal 80.

Confirm with **OK** and save the Configuration. In the Experiment Configurator, the Configurations will be available under Qtegra Main. Note that Configuration and options like flush, acid pump, and trap will only be available if enabled under the GasBench Configuration.

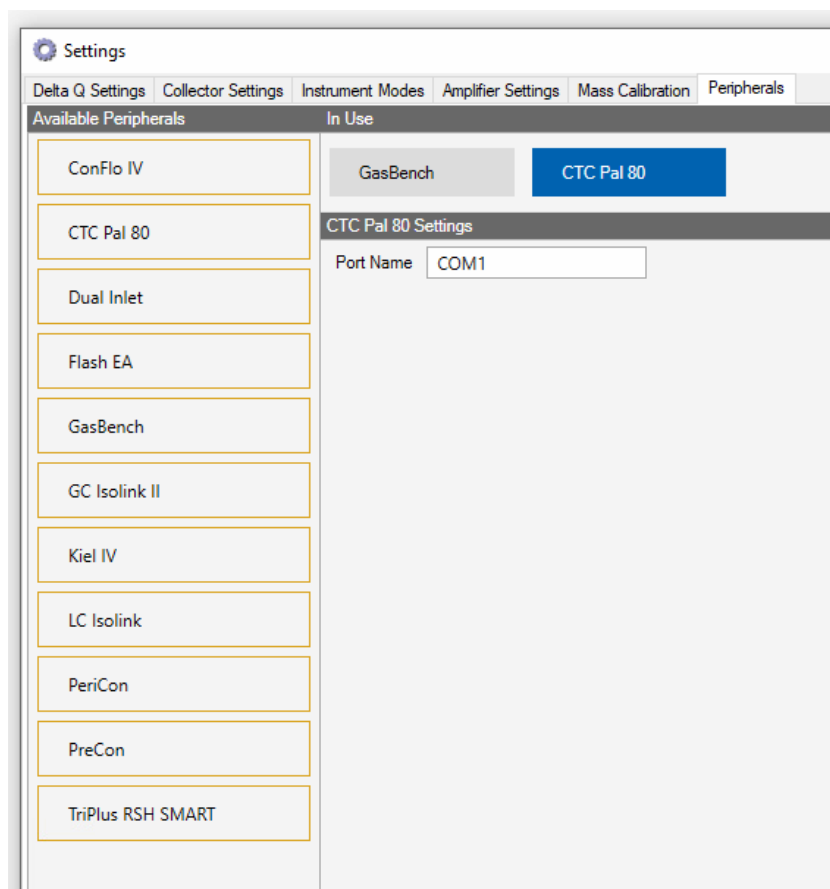


Figure 7-6. GasBench II installation with a CTC Pal 80 autosampler. Use GasBench II dropdown menu. Note: The additional options or settings of the GasBench will be validated in the Qtegra ISDS Software. Options or items not in use will not be depicted or operational if not enabled or configured in the Configurator.

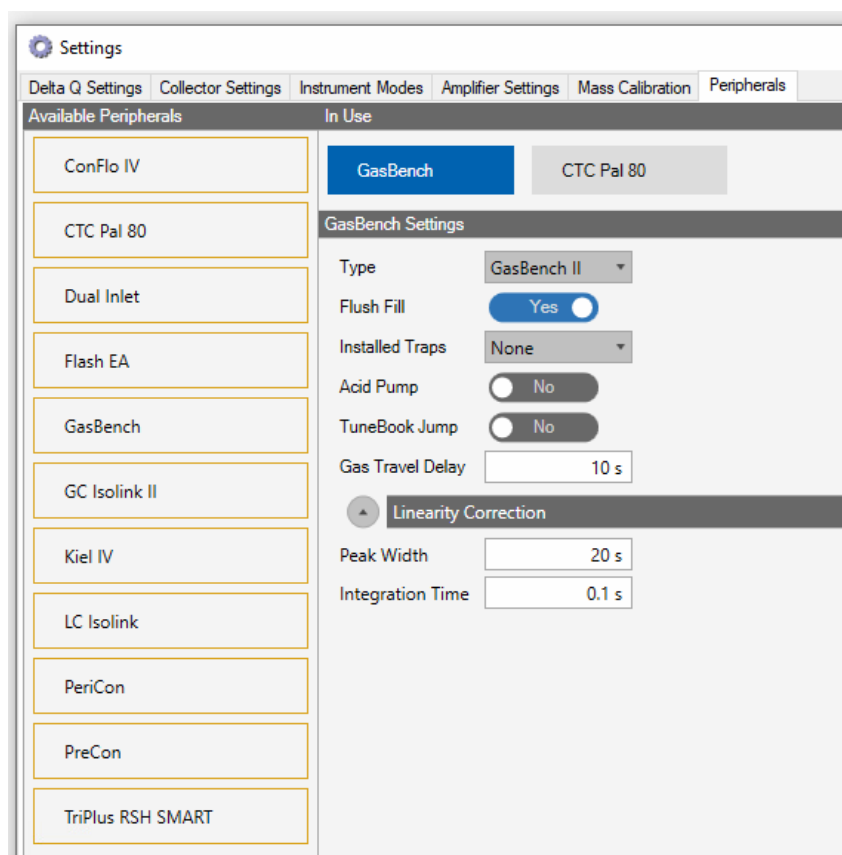


Figure 7-7. GasBench II installation with a CTC Pal 80 autosampler. Use COM1 for the standard installation of the CTC Pal 80 in Otegra.

Otegra Dashboard

GasBench Plus and TriPlus RSH SMART Autosamplers

The Otegra Dashboard shows all peripherals including the instrument control if the items are configured and enabled. See [Figure 7-8](#). Here, the peripherals are shown in peripheral tabs. The tabs can be organized on the screen. Refer to the *Otegra ISDS Software for the Gas Isotope Ratio Mass Spectrometers Installation Guide*.

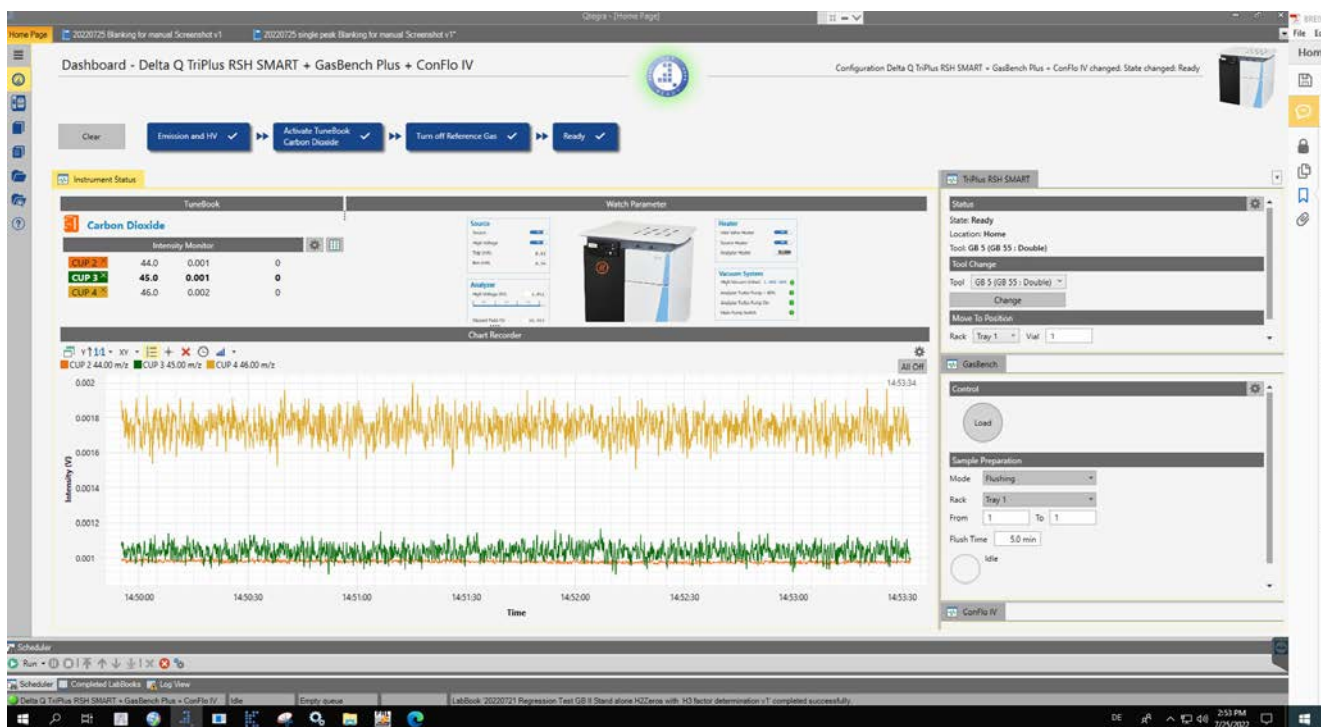


Figure 7-8. Otegra Dashboard with all peripherals tabs

TriPlus RSH SMART Autosamplers

The Dashboard for the TriPlus RSH shows the status of the autosampler and a testing slot for the autosampler, including the two slots and its installed racks. See [Figure 7-9](#).

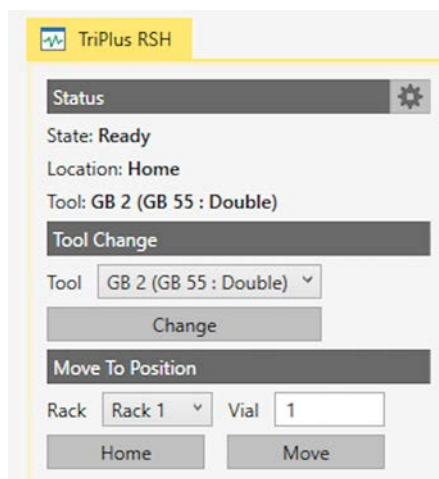


Figure 7-9. Dashboard for TriPlus RSH SMART for GasBench Plus

NOTICE

Each GasBench Plus tool must be taught on each one of the two slots (if both are in use) or rack definition to be able to operate properly. If the teaching (see [“TriPlus RSH SMART - Advanced and TriPlus RSH SMART - Standard Installation”](#) on [page 5-19](#)) is not correct or made on a wrong rack, any installed needle will break.

The TriPlus RSH panel shows the status of the RSH, the location of the tower and the installed GB Tool inside the z tower.

Under Tool Change, a tool change (TriPlus RSH Advanced model with automated tool changer) or a new tool mount can be activated (Standard model).

The needle can move to slot 1 (rack 1) or slot 2 (rack 2) by clicking the **Move** button. After finishing or stopping Qtegra, the arm can be moved to the home position by clicking the **Home** button.

The pull out menu (“grayish settings sign”) is used to define the tool type being used under Acquisition Tools for the two different rack types (see [Figure 7-10](#)) and the definition of the penetration depth for the

GasBench Tool 55 and GasBench Tool 120 types. The penetration and penetration speeds will be used in the LabBook sampling list as defined here. There is no differentiation between the slot types.

Tip Each rack 1 and rack 2 will have a flush and sampling tool. Only two different tool types exist, but they can be outfitted with different needles, for example:

- GB 2 (GB 55: Double) with two flush needles “Quick flushing for 18O equilibration & carbonates measurements”
- GB 3 (GB 55: Double) with an acid needle to the left and a measurement needle to the right - “d13C & d18O in carbonates measurements”
- GB 4 (GB 55: Double) with a flush needle to the right and a measurement needle to the left - “d2H in water measurements”
- GB 5 (GB 55: Single) with a single flush needle for a larger rack size for single needle sampling (≥ 20 mL vial sizes)
- GB 6 (GB 120: Single) with a 120 mm single measurement installed on the left.

Tip Any tool and tray position will be taught at the left position of either a GasBench Tool 55 or a GasBench Tool 120.

NOTICE

Each ATC can carry three Tools, but other not used tools can be put aside and detected if in use. The Tools in use must be defined in the Acquisition Tools menu.

If not defined, a LabBook will not operate and the orange LED of the RSH will flicker orange. The Tool type Single or Double must be defined in the TriPlus RSH firmware. See [“Installing a New TriPlus RSH Firmware Using a Memory Stick \(FAT 16\)”](#) on page 5-20.

NOTICE

The TriPlus RSH models allow using as many tools as put once a time into the ATC (ATC automatically assigns the tool with a number, but they must be calibrated, defined and taught properly to each of the rack and slots. See [“TriPlus RSH SMART - Advanced and TriPlus RSH SMART - Standard Installation”](#) on page 5-19.

NOTICE

Before using the commands Tool Change and Move to Position to check needle penetration and proper position with Qtegra, it is recommended putting the penetration depth to zero.

Do not forget to define the penetration depth for later sampling or flushing in a LabBook for each defined tool.

The maximum penetration depth for a GasBench Tool 55 and GasBench Tool 120 is 100 mm/s. The maximum penetration depth is defined in the firmware of the tool types (GB 55: 45 mm, GB 120: 95 mm). The default penetration depth for the GasBench Tool 55 is 28 mm.



Clicking the **Settings** button opens the RSH settings. See [Figure 7-10](#). All settings for the tools, the rack, the needle Penetration Depths for the individual flush and sampling tools are configured under the settings.

NOTICE

For an acid needle injection the penetration depth might be 1-2 mm deeper than 27 seconds to allow proper sampling by the measurement needle. Check correct penetration of the acid needle with zero penetration first.

The Penetration Speed and the Remote Terminal can be opened or edited.

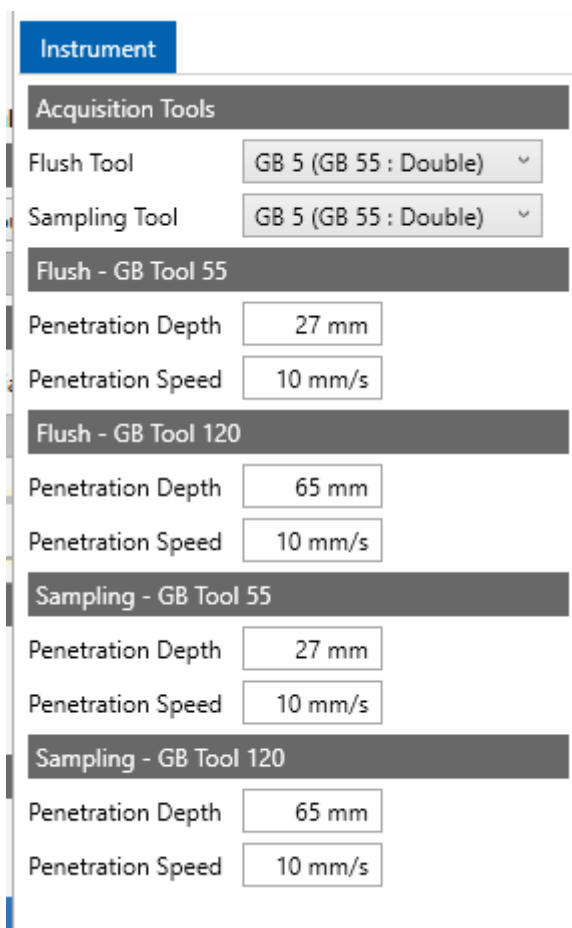


Figure 7-10. TriPlus RSH SMART Dashboard editing under Otegra

Click **Open** to open the remote terminal, see [Figure 7-11](#). Here, all settings can be made as with the standard terminal on the TriPlus RSH SMART autosamplers. See “[General Notes for the TriPlus RSH SMART for GasBench Plus Terminal](#)” on page 5-19.

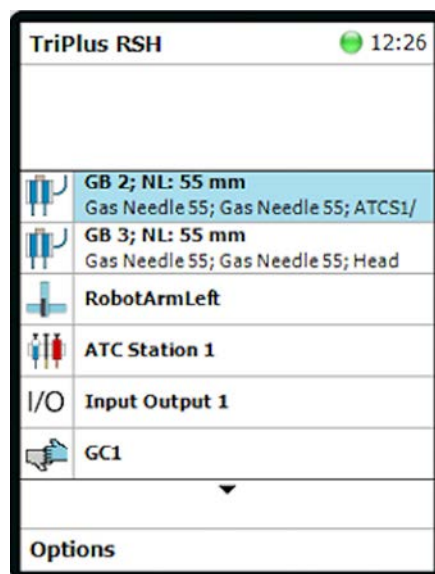


Figure 7-11. The remote terminal under Qtegra

GasBench II and CTC Pal 80 Autosampler A200S Mode

The Qtegra Dashboard lists all available peripherals including the instrument control, see [Figure 7-12](#). Here, the peripherals are shown in the peripherals tab. Tabs can be organized on the screen. Refer to the *Qtegra ISDS Software for the Gas Isotope Ratio Mass Spectrometers Installation Guide*.



Figure 7-12. Qtegra Dashboard with all peripherals tab. Settings for GasBench II with the CTC Pal 80 autosampler and (as shown here) ConFlo IV

Dashboard - CTC Pal 80 Autosampler

The Dashboard of the CTC Pal 80 autosampler shows the status of the peripheral (see [Figure 7-13](#)) and allows to set the autosampler rack (“tray 01” visible in the autosampler panel) and methods (A200S injection methods). Only 1 to 9 methods can be used from the autosampler terminal configuration. The A200S mode restricts the number of methods to a maximum of 9 available methods, which is sufficient for the GasBench II. Refer to the *CTC Pal 80 Autosampler Operating Manual* and the *GasBench II Operating Manual* for further information.

Open the settings to configure the rack and the method (see [Figure 7-14](#)). The autosampler terminal is not visualized in Qtegra ISDS Software.

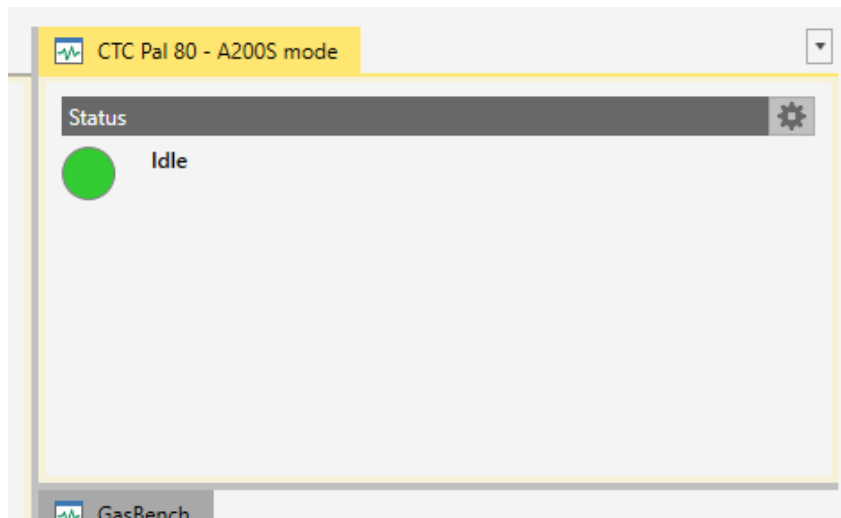


Figure 7-13. Qtegra Dashboard with CTC Pal 80 autosampler and the communication status. Open the settings from the gear icon in the upper right corner.

Click **Settings** to open the CTC Pal 80 settings. Under Installed Rack, the rack number is taken for later operation in the Sample Preparation (“[Performing Preparation of Samples using Prescripts with Qtegra](#)” on [page 7-55](#)) or running a Qtegra LabBook (see “[Qtegra LabBooks](#)” on [page 7-26](#)). The A200S mode allows only to use the rack named *Tray01*.

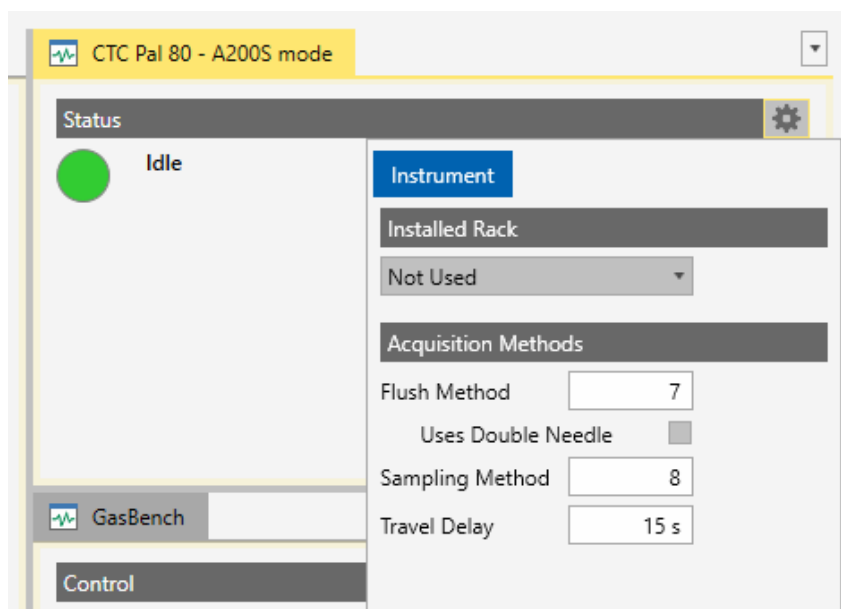


Figure 7-14. CTC Pal 80 settings. The A200S and the tray can be selected from the dropdown list.

Under **Acquisition Methods**, the A200S method injection method is taken for later operation in the Sample Preparation (“[Performing Preparation of Samples using Prescripts with Qtegra](#)” on [page 7-55](#)) or running a Qtegra LabBook (“[Qtegra LabBooks](#)” on [page 7-26](#)). The A200S mode allows only to use method numbers 1 to 9. The methods are programmed under the CTC Pal 80 terminal panel.

Enable a double needle installation on the GasBench II needle port (refer to the *GasBench II Operating Manual*) to allow faster flushing in the “Stand alone Flushing and Automated Flushing and Acid Addition” and in the LabBook/Method Parameters/Peripheral Parameters preparation. The next sample row or sample list preparation position will be omitted during flushing and the next sample row will be injected.

Under **Travel Delay**, the time of the movement of the autosampler to the sample or flushing position is defined. This time is dependent on the x, y, z motor velocity and must consequently be set by the user. It is used in any operation of the autosampler, i.e., prescript command for the CTC Pal 80 autosampler (single trap & denitrification, GasBench Series with PreCon).

ConFlo IV & PreCon

Refer to the *ConFlo IV Operating Manual* and to the *PreCon Operating Manual* for Qtegra operation and measurement.

GasBench Plus

The Dashboard shows the status of the configured GasBench Plus options, i.e Acid Pump, Single and Dual Trap (see [Figure 7-15](#)).

If a GasBench is used without the ConFlo IV universal interface the GasBench Reference options can be activated.

NOTICE

The drop out menu box (light gray settings sign) enables to define the reference gas names on the three different stand-alone reference ports and in a second tab inside the box, the definition of drops to stroke backwards and forward. Inside the LabBook, the Acid valve will only be activated or not.

NOTICE

Use the Diagnostics > GasBench tool to define the number of strokes to get a drop (see “[Acid Pump Calibration](#)” on [page 7-20](#)). Move the GasBench tool either to a 12 ml non-thermostatted rack in Slot2 or 1 or park or demount the tool and put the acid needle straight into an empty 12 ml vial to optimize the number of strokes per drop.

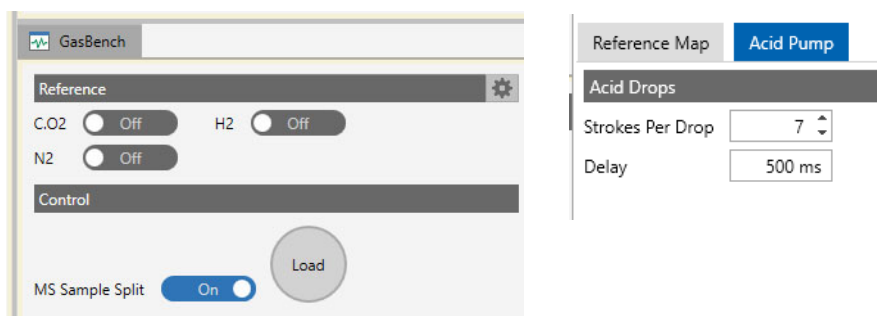


Figure 7-15. GasBench Plus Instrument Dashboard, Reference Map and Acid Pump

The GasBench Dashboard allows to see the status of the device tools and addresses during LabBook operation as well as the calibration of the acid pump.

Acid Pump Calibration

The GasBench Acid pump is calibrated in the GasBench Plus Dashboard. Number of strokes needs to be defined by using the **Manual** button below the pump to release one drop out of the tip of the acid needle or to retract one drop into the needle. Since the acid is very viscous and a slight pressure needed because of the surface tension of the stainless steel tubing, a delay of 500 milliseconds (0.5 sec) is needed.

Stand-Alone Flushing or Automated Flushing and Acid Addition (“Sample Preparation”)

Inside the GasBench Plus dashboard, Qtegra provides a stand-alone flushing and acid addition sample preparation. Three stand-alone sampling modes are implemented in Qtegra.

1. Flushing with a double or a single needle with a flush gas (Flushing)
2. Flushing at the right needle and acid addition at the left (Flushing & Acid Dosing)
3. Acid Addition (Acid Dosing)

NOTICE

The used tools are validated. If the acid pump is not enabled. It is not possible to use the acid dosing and flushing and acid dosing options.

General Description

The Sample preparation is used with a parallel process for flushing and acid dosing (see [Figure 7-16](#)), with acid dosing only (see [Figure 7-17](#)) or flushing only (see [Figure 7-18](#)). A flush time, number of drops, with Sequence Start and Stop is parameterized. **Rack** decision is made in the TriPlus RSH dashboard setup. Positioning **from** start number **to** end number can be taken but will be only go from start to end. Neither

individual positions can be taken, nor sequences scheduled. The **Flush Time** can be defined in minutes or seconds. Drop numbers **forward** and **reverse** must be defined. The GasBench Tools for flushing must be defined in the TriPlus RSH dashboard settings as well as the Acid stroke calibration and stroke delay.

The screenshot displays the 'Sample Preparation' configuration window. It includes the following settings:

- Mode:** Flushing & Acid Dosing (dropdown menu)
- Rack:** Tray 1 (dropdown menu)
- From:** 1 (input field)
- To:** 16 (input field)
- Flush Time:** 5.0 min (input field)
- Acid Drops:**
 - Forward:** 16 (spin box)
 - Reverse:** 3 (spin box)

At the bottom of the window, there are two blue buttons: 'Start' and 'Stop'. A large, faint circular icon is also visible in the lower-left area of the panel.

Figure 7-16. GasBench: External Flushing & Acid Dosing sequence

Figure 7-17 describes acid dosing with one needle positioned in either needle right or left. The right or left position depends on the operator's later measurement workflow.

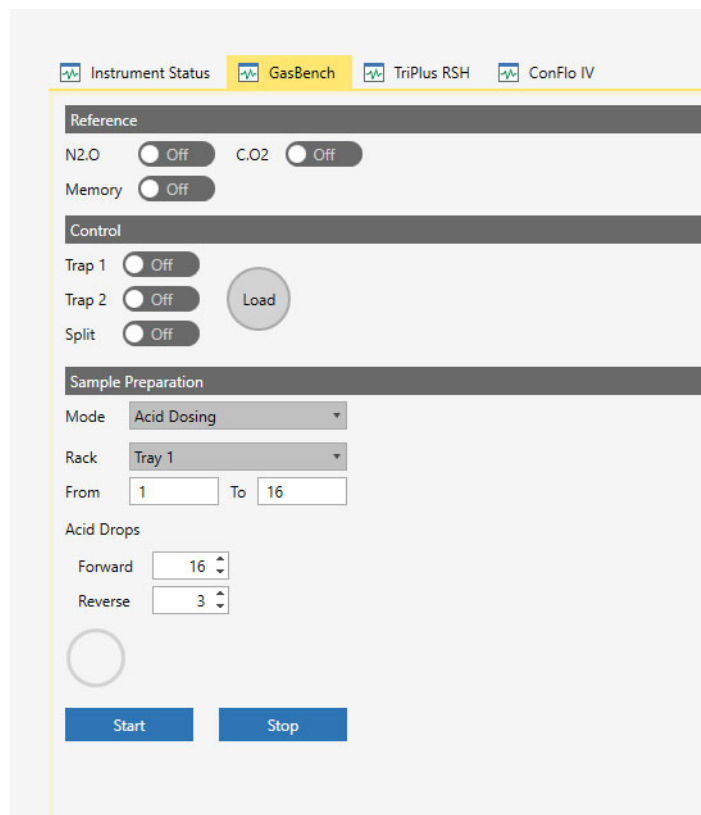


Figure 7-17. GasBench: External Acid Dosing sequence

Figure 7-18 describes the flushing workflow. Needle setup and rack setup must be defined in the dashboard for the TriPlus RSH. A rack will be used as configured in the TriPlus RSH (Slot1 / Slot2).

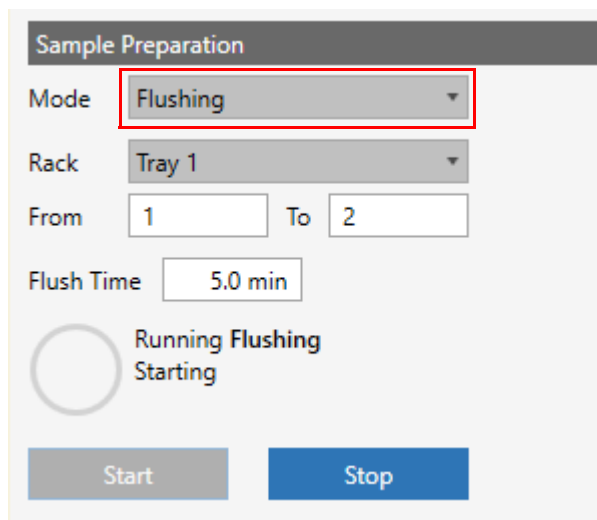


Figure 7-18. GasBench: External Flushing sequence

NOTICE

Qtegra does not validate the sample needle installation. Be aware that for acid dosing with an acid needle the right vials should always be positioned during operation.

NOTICE

On sample Preparation with GasBench II with the CTC Pal 80 autosampler Qtegra validates the autosamplers of the GasBench systems. For the GasBench II only one rack system is available for Sample Preparation operation. The time the autosampler needs to move to the position has been calibrated first by the user (“travel delay”).

Qtegra Instrument Diagnostics

GasBench Plus and Hardware Diagram

Refer to the *DELTA Q Operating Manual* to obtain a general understanding of the Diagnostics in Qtegra.

The GasBench Plus diagnostics show all hardware addresses available in the GasBench Plus and enable to switch them (see [Figure 7-19](#). Qtegra Diagnostics under Instrument for the GasBench Plus stand alone and with ConFlo IV.)

NOTICE

There are no safety measures in the Qtegra Diagnostics, which prevent the instrument from malfunction if wrongly operated.

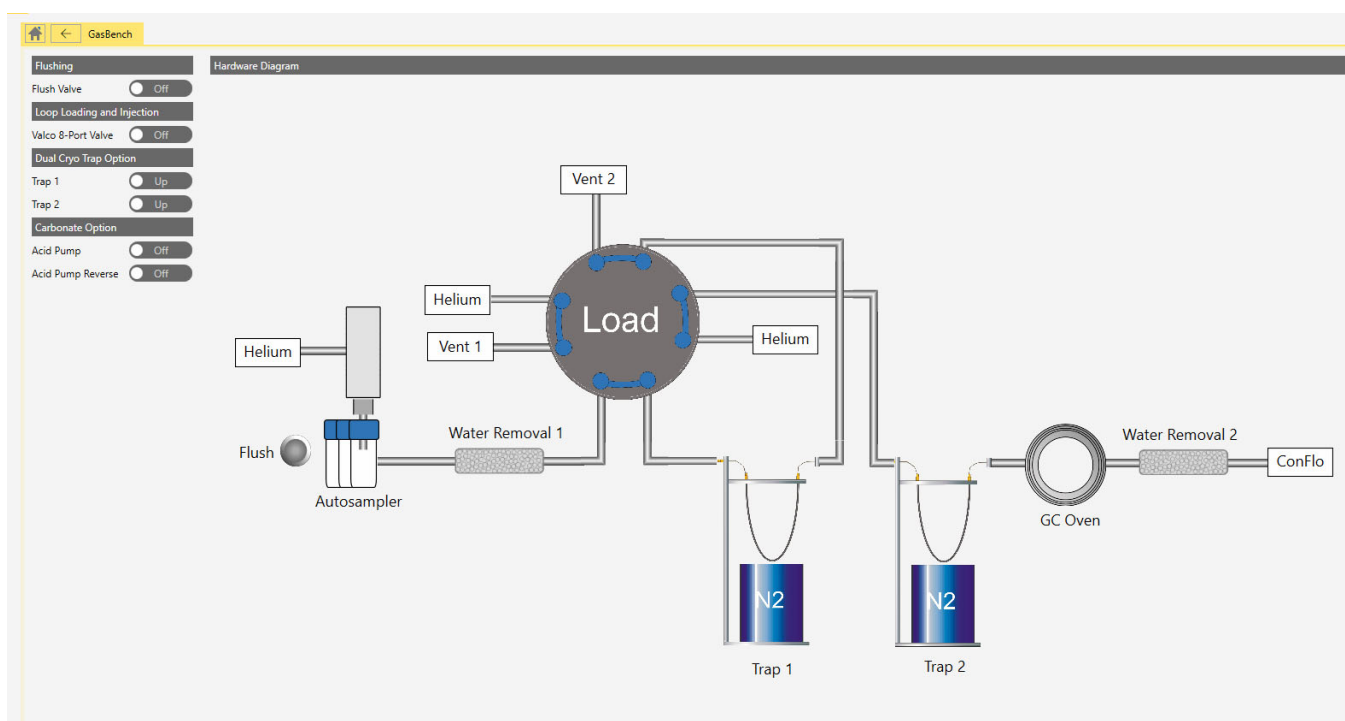


Figure 7-19. Qtegra Diagnostics under Instrument for the GasBench Plus stand alone and with ConFlo IV. Options, which are not enabled in the Configurator, will not be depicted.

GasBench Diagnosis objects for operation and testing

The **Port 1** shows all hardware objects, which are operated with the GasBench plug and measure adapter.

Acid Pump objects for operation and testing

The **Port 2** shows all hardware objects, which are operated with the Acid pump plug and measure adapter, i.e., acid pump number of steps (strokes).

1. Insert the number of strokes and number of drops in the test drop window. The time difference will be set as under the GasBench Plus acid pump settings under the Dashboard.
2. Pushing the test drop button will activate the Acid pump to pump. Test the number of strokes needed for a drop.
3. Insert the optimized number of strokes and time difference in the Acid pump settings.
4. Define the number of drops in the Acid Pump settings window for being used in the LabBook for sampling.

NOTICE

First move the autosampler acid needle to an empty needle or insert the needle into a vial without autosampler operation under the TriPlus RSH dashboard.

NOTICE

In the Qtegra LabBook, the acid pump will be only be activated for a sample list under method parameters - continuous flow. The number of strokes, drops and time difference between strokes is inserted under settings in the GasBench Plus instrument Dashboard after optimization of the acid pump. Acid flowing out after dropping will be prevented by backstrokes.

Instrument Diagnostics - CTC Pal 80 - A200S Mode

The Instrument Diagnostics enables to check the positioning and operation with the CTC Pal 80 (see [Figure 7-20](#)). Click **Start**. The travel delay can be checked as well.

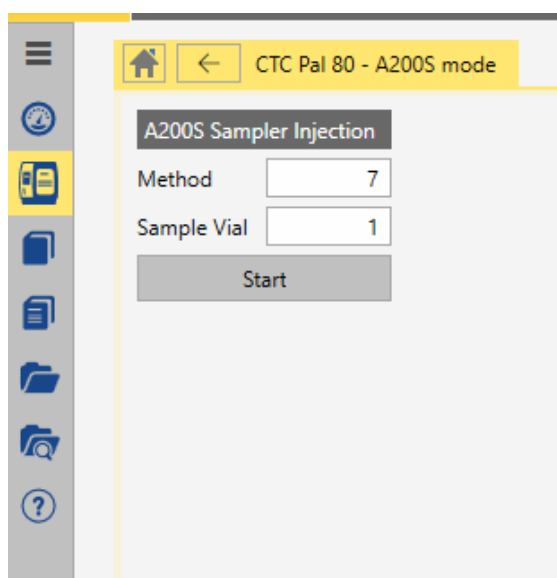


Figure 7-20. CTC Pal 80 Diagnostics. The A200S Sampler Injection method and the positioning can be tested under Diagnostics

Qtegra LabBooks

Setup for GasBench Plus

The general setup of a Qtegra LabBook is described in the *Qtegra ISDS Software Manual*, e.g., opening, creating and copy a new or existing LabBook for GasBench Plus as well as finding back up features of any Qtegra LabBook. For the GasBench Plus, LabBooks are found under the folder or above (*C:\ProgramData\Thermo\Qtegra_Application Data\Workspace\LabBooks\GasBench*).

By clicking on a newly, editable GasBench Plus LabBook in the upper menu bar, the full LabBook features will appear. Refer to the *DELTA Q Operating Manual*. An additional mouse-click on the Method Parameters opens the Method Parameters settings.

NOTICE

All subsequent chapters also refer to the GasBench II system connected with a CTC Pal 80 autosampler.

Method Parameters - Continuous Flow

The sample processing with the GasBench Plus is set in the continuous flow window of the LabBook. See [Figure 7-21](#). Refer to the *DELTA Q Operating Manual* for a general explanation of all features, saving time line objects and using drag & drop mode. Each timing can be set in minutes or seconds by typing a number and adding the unit symbol *s* or *m*.

NOTICE

At any time before a sample will be acquired or started.

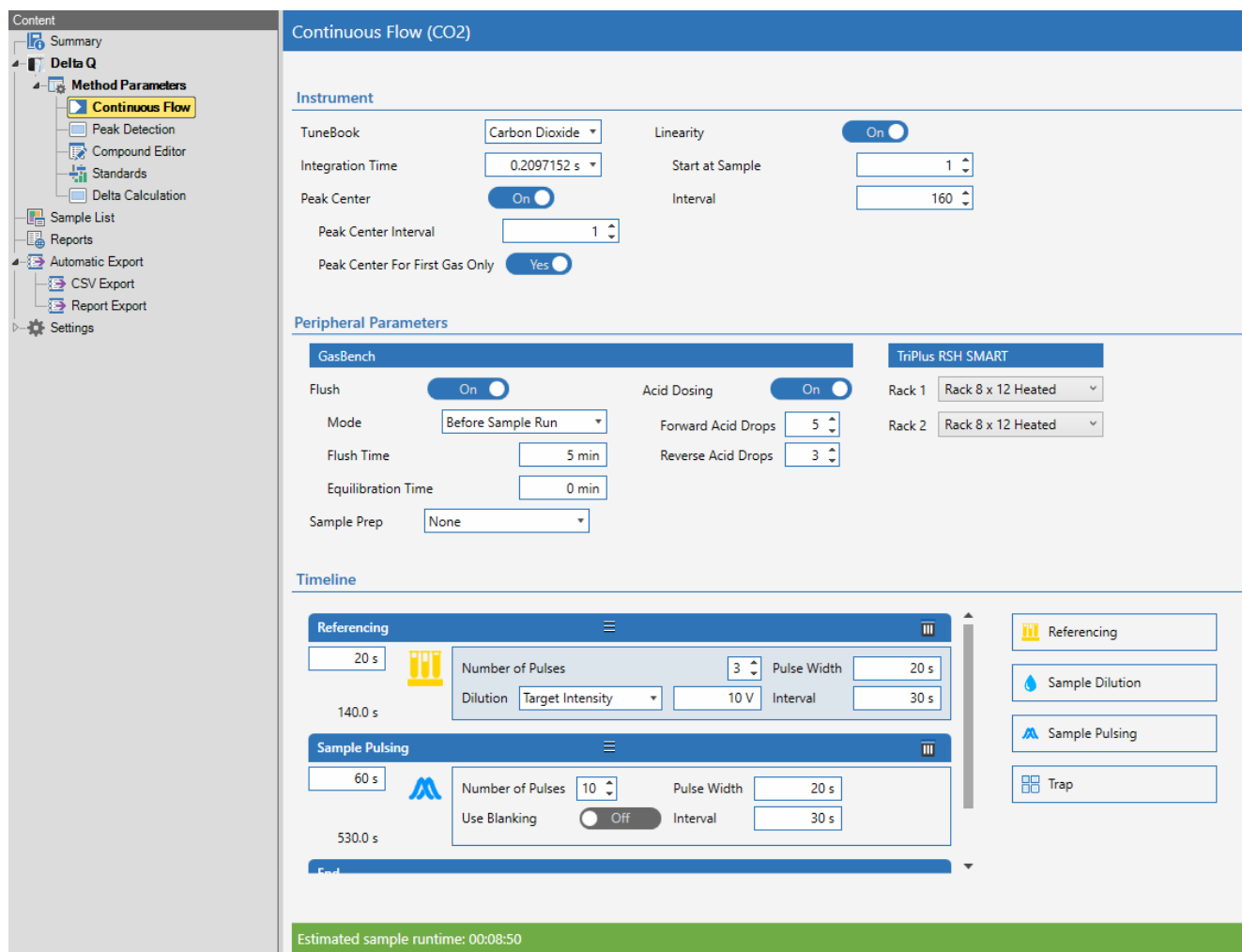


Figure 7-21. General continuous flow setup for the GasBench Plus. This generic LabBook shows all available options.

Instrument description

For instrument description, see the general *Qtegra ISDS Software Manual*. If a peak center is not used, disable the peak center. If a peak center shall appear in interval before a certain sample in the sample list, define the interval.

For measurements with one TuneBook only (no peak jump), the **Peak Center for First Gas Only** will be set to *Yes*.

NOTICE

Peak center properties and scan time of the peak center can be found under the following location in Qtegra: See under *\\Homepage\Instrument \ Collector Configuration Designer \ Peak Center settings*. Type *Advanced Settings* on the dropdown menu. Here, the Scan time will be shown, which is used in the LabBook.

NOTICE

Devices with ConFlo IV - After each Peak Center, the continuous flow will set a gas dependent travel time, which can be edited under the Experiment Configuration in the Qtegra Configurator.

NOTICE

The GasBench travel delay will be added to and after each Continuous Flow Gas Travel Delay action with a ConFlo IV (see “Otegra Configurator” on page 7-6).

NOTICE

If the peak center fails:

- a. use an appropriate backward peak center signal difference in percentage to the forward scan voltage
- b. choose the correct mass difference to the center mass
- c. step delay time for HD shall be ≥ 0.5 steps.

Peripheral Parameters

Parameter	Description
Flush	Flush turned off/on. See Figure 7-22. The flush time can be set.
Transfer Time	A transfer time is in general not needed. The transfer time is defined by the sample pulsing start time. NOTICE Only if peak center interval is used, the transfer time must be set accordingly.
Acid Dosing	Acid dosing can be activated for LabBook at a carbonate sample workflow. Acid dosing will happen independently from the other timelines, since the acid is injected independently in the right position of the GB Tool. The acid dosing time is not a part of the transfer time of the sample gas transfer to the Valco sample load.

TriPlus RSH

Here, the rack types for the RSH are defined for the running LabBook.

Parameter	Description
Rack 1	Definition of the inserted rack in Slot1.
Rack 2	Definition of the inserted rack in Slot2. NOTICE Take care that only an available slot can be used in this dropdown menu. Otherwise, a needle might break.

The Peripheral Parameters contain two different flushing modes.

CTC Pal 80

The CTC Pal 80 does not need a tray definition under **Peripheral Parameters** since the CTC Pal 80 can only drive to one set rack. The rack type is programmed on the CTC Pal 80 itself named *tray 01*. Consequently, the autosampler section on the right is kept empty.

1. Flushing before the timeline start for all sample positions in the sample list. Qtegra takes into account if a double needle or a single needle setup is used for flushing. See [Figure 7-22](#).
 - a. **Mode** to be set to *Before Sample Run*.
 - b. **Flush Time** to be set to, e.g., 5 minutes
 - c. **Equilibration Time** to be set minus the time the samples needed to run for flush, e.g. 20 h equilibration time needed equals 16 hours for equilibration time to be set minus the flushing of the whole tray with a double needle.

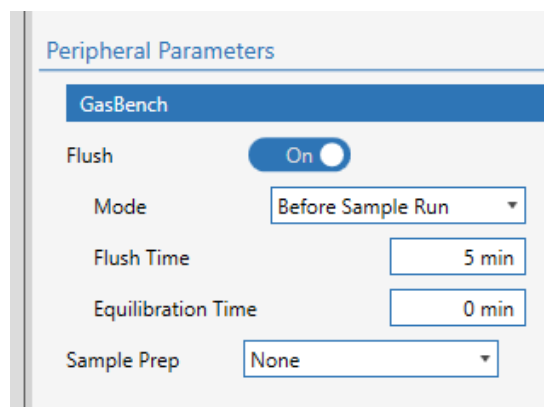


Figure 7-22. Flush mode before sample run

2. Flushing during the timeline. No staggering mode of the flushing is integrated in Qtegra. Staggering mode means the autosampler moved out of the time line process and does a different action even though the timeline process does still run.

Consequently, one needle is used for flushing during the timeline, e.g., measurement for hydrogen isotopes in water. See [Figure 7-23](#).

- a. **Mode** to be set to *During Sample Run*.
- b. **Flush Time** as the GasBench Tool sits in the sample position for sampling. Flush time opens as long as the flush needle is inserted in the vial.

c. No **Equilibration Time** set.

NOTICE

Significant consumption of flush gas can be reduced with the *during sample run* flush if the purge gas flow is reduced, e.g., 80 to 100 mL/min flow for 5 minutes can be reduced to approximately half of the head pressure from the flush gas bottle.

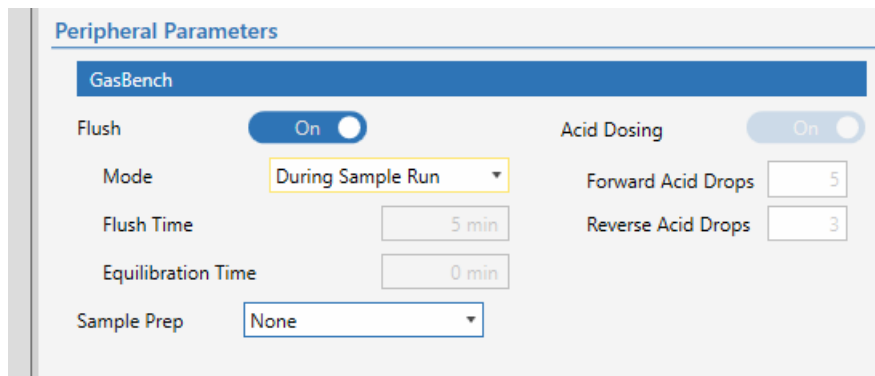


Figure 7-23. Flush mode During Sample Run. No equilibration time used. For equilibration configuration, the acid pump will not be enabled in the Instrument Experiment Configuration. Consequently, the Acid Dosing switch is not shown as here.

Timeline

For the GasBench Plus, the timing of the individual time duration of any Timeline object is switched off, e.g., the sample pulsing can start during a reference pulsing.

GasBench Plus with own referencing and GasBench Plus with ConFlo IV for referencing: The GasBench Plus can be operated in “stand-alone” referencing mode and in LowFlow (LF) mode with the ConFlo IV device.

Parameter	Description
Number of Pulses, Dilution, etc	Refer to the <i>DELTA Q Operating Manual</i> for the setup.
GasBench Plus with ConFlo IV	Refer to the <i>DELTA Q Operating Manual</i> for zero enrichment measurements and H ₃ ⁺ factor within a LabBook and within the H ₂ TuneBook (Linearity determination), e.g. CO ₂ , N ₂ , N ₂ O or H ₂ measurements. See Figure 7-24, a . A target value can be set.
GasBench Plus with own referencing	For gas analytes zero enrichment measurements with the GasBench Plus, the same LabBook mode will be used as with ConFlo IV and as described in the <i>DELTA Q Operating Manual</i> . The only difference is that the reference dilution cannot be set automatically and the signal height, respectively. See Figure 7-24, b . The current value as set by the pressure gauge can be taken. Adjust the signal to at least ≥ 4 V of a major signal.

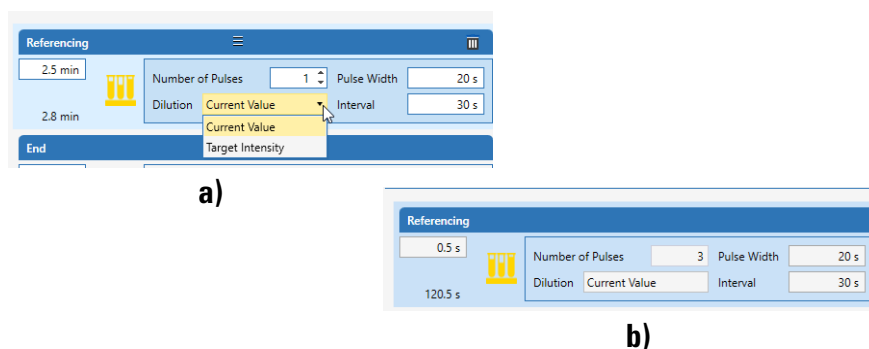


Figure 7-24. Method parameter for Continuous flow a) GasBench Plus with ConFlo IV b) GasBench Plus with own referencing mode does not allow a target intensity

Sample Pulsing

Drag and drop the sample pulsing after the referencing.

Parameter	Description
Number of Pulses, Dilution, etc	Number of required sample peak for the result reporting is determined here, e.g. 10 means ten sample pulse will appear.
Pulse Width	The timing of the injection from the Valco valve sample loop on the GC column (“Inject”).
Interval	Timing of the loading of the Valco valve sample loop before next injection (“Load”).
Dropdown	<p><i>Empty:</i> No blanking.</p> <p><i>Blanking:</i> With the dropdown window an editing window appears. Now, any interference gas peak can be removed and objected to enter the mass spectrometer without obscuring the baseline as in other mass spectrometers. See Figure 7-25 and Figure 7-26.</p>
Start time	Start of the dilution
End time:	End time of the dilution
Dilution	<p>In percent as for the Sample dilution and setting the length of an interval of the action. During the timeline, more than on dilution can be set within the timeline.</p> <p>A dilution factor is set in the Dashboard for ConFlo IV (refer to the <i>ConFlo IV Operating Manual</i> for the settings) or the GasBench Plus sample open split must be changed accordingly. Contact our local service organization to get the appropriate dilution kit.</p> <p>GasBench without ConFlo IV: The split will be used for the dilution action.</p> <p>GasBench with ConFlo IV: The split can be edited from the calibrated dilution table and its range in percent.</p>

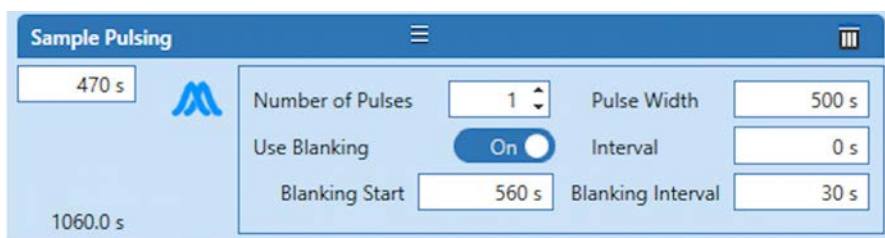


Figure 7-25. dropdown menu with editing parameters to set blanking for air measurements and for the single trap analyses to avoid air response before the analyte gas retention time, e.g. blanking of N₂

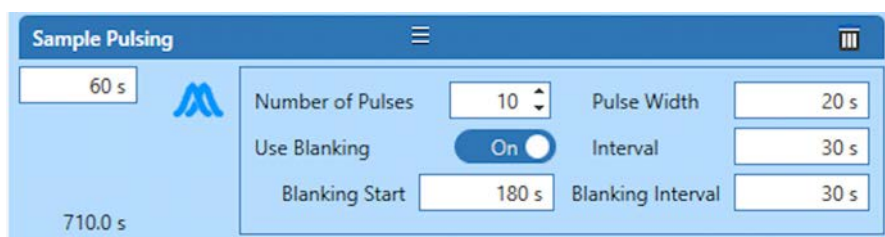


Figure 7-26. dropdown menu with editing parameters to set blanking for multiple peak blanking. The Blanking Start is added to the sample pulsing time, e.g., 180 s + 60 s = 240 s.

Method Parameters - Peak Detection

Refer to the *DELTA Q Operating Manual* for description of the peak detection modes and feature in the Qtegra LabBook. See [Figure 7-27](#).

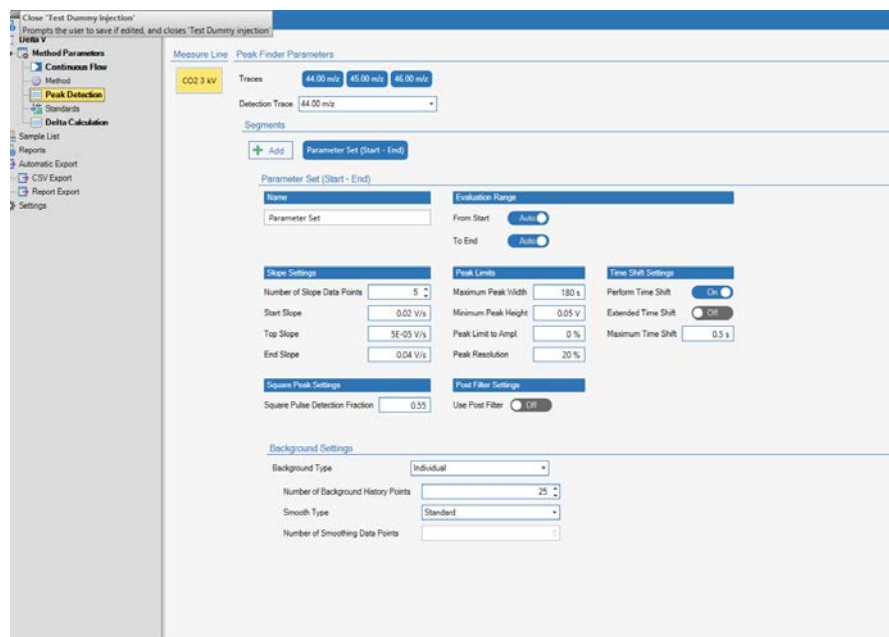


Figure 7-27. Qtegra peak detection settings for the GasBench Plus. For recommended peak detection mode see text and *DELTA Q Operating Manual*

For a short introduction the peak detection mode setting contains the following settings. The detection trace is in natural abundance measurements always the most abundant mass of an element or molecule.

Parameter	Description
Segments	With a start-end set values, by certain segments the detection mode can be changed at certain time ranges, e.g. if the background changes - see Evaluation Results (<i>DELTA Q Operating Manual</i>).
Evaluation range	Default: All automated. With a configured evaluation range, peaks or time ranges can be omitted from the chromatogram. The peak will not taken for calculation in the evaluation results. Peaks can also be in the chromatogram directly, see Evaluation Results (<i>DELTA Q Operating Manual</i>).

Parameter	Description
Slope settings	Default: Slope data points: 5, start slope: 0.001 V/s end slope: 0.001 V, Top slope: 5E-05 V/s. For all GasBench Plus measurements: 0.01 V/s (start slope), 0.04 V/s (end slope). Peak top: as default.
Peak limits	Defaults: Maximum peak width: 180 s, Minimum peak height: 0.05 V, Peak limit to amplitude: 0%, peak resolution: 20%.

For GasBench Equilibration with approx. 0.3 to 0.5% CO₂ helium and 1% to 4% H₂ in He in helium and carbonate CO₂ peaks or air measurements: 0.5 V. Smaller peak heights might be taken, but linearity correction could be necessary.

The generic detection peak limits will set for the full chromatogram. This is important for sample peaks which can be below detection limit and should be omitted, see Evaluation Results (*DELTA Q Operating Manual*).

Parameter	Description
Time shift settings	Default is on. Extended: off. Maximum time shift: 0.5 s. This is only important for optimization in peak detection of sample peaks in some gas isotope ratio measurement modes, e.g. hydrogen (see <i>DELTA Q Operating Manual</i>). Can be used for optimizing for your sample results but should not be changed for future sample analysis of the same sample and gas isotope ratio measurement acquisitions.
Square Peak Settings	Default value: 0.55. With a configured evaluation range, peaks or time ranges can be omitted from the chromatogram. The peak will not taken for calculation in the evaluation results. Peaks can also be in the chromatogram directly, see Evaluation Results (<i>DELTA Q Operating Manual</i>).
Post filter settings	Default: off. Never used for GasBench Plus measurements - see Evaluation Results (<i>DELTA Q Operating Manual</i>).
Background Settings	Background Types. Default: individual, number of background points 25, Smooth type: standard. Low Pass Filter: background points: 25, no start value, Tau value: 0.2 for all masses, 44, 45, 46, 2, etc. For GasBench Plus: Default [Individual] and for hydrogen [individual or Low Pass filter]. Low Pass Filter might not influence internal precision but external result accuracy. Optimize for dataset but do not change further onwards.

Parameter	Description
Acquisition	Set Stop measurement if no sample peaks were detected to <i>On</i> to stop the acquisition if no sample peak is detected. Reference gas injection and detection are not affected (see Figure 7-28).
Peak Averaging	Set Ignore first peak in averaging to <i>On</i> to ignore the first sample peak detection in the multiple peak detection mode. Set this parameter <i>Off</i> to include the first peak within GasBench applications. This feature does allow background conditioning of subsequent sample peaks (see Figure 7-28).

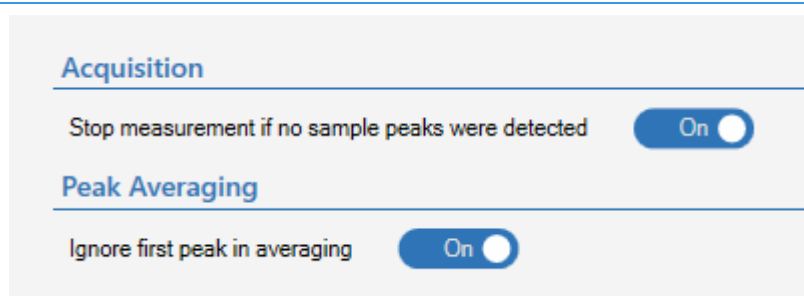


Figure 7-28. Stop measurement and peak averaging under Method Parameters > Peak Detection

Method Parameters - Compound Editor & Result Chromatogram Compound Reporting

Refer to the *DELTA Q Operating Manual* for generic description of the compound editor and how to define compounds and compound standards in the GasBench Plus chromatogram.

A pre-compound list can be edited for presentation of the result evaluation chromatogram peaks, for GasBench Plus, e.g., CO₂ and numbers of peaks or reference gas peaks (see Figure 8-15). The compounds can be created in the chromatogram by right-click, respectively.

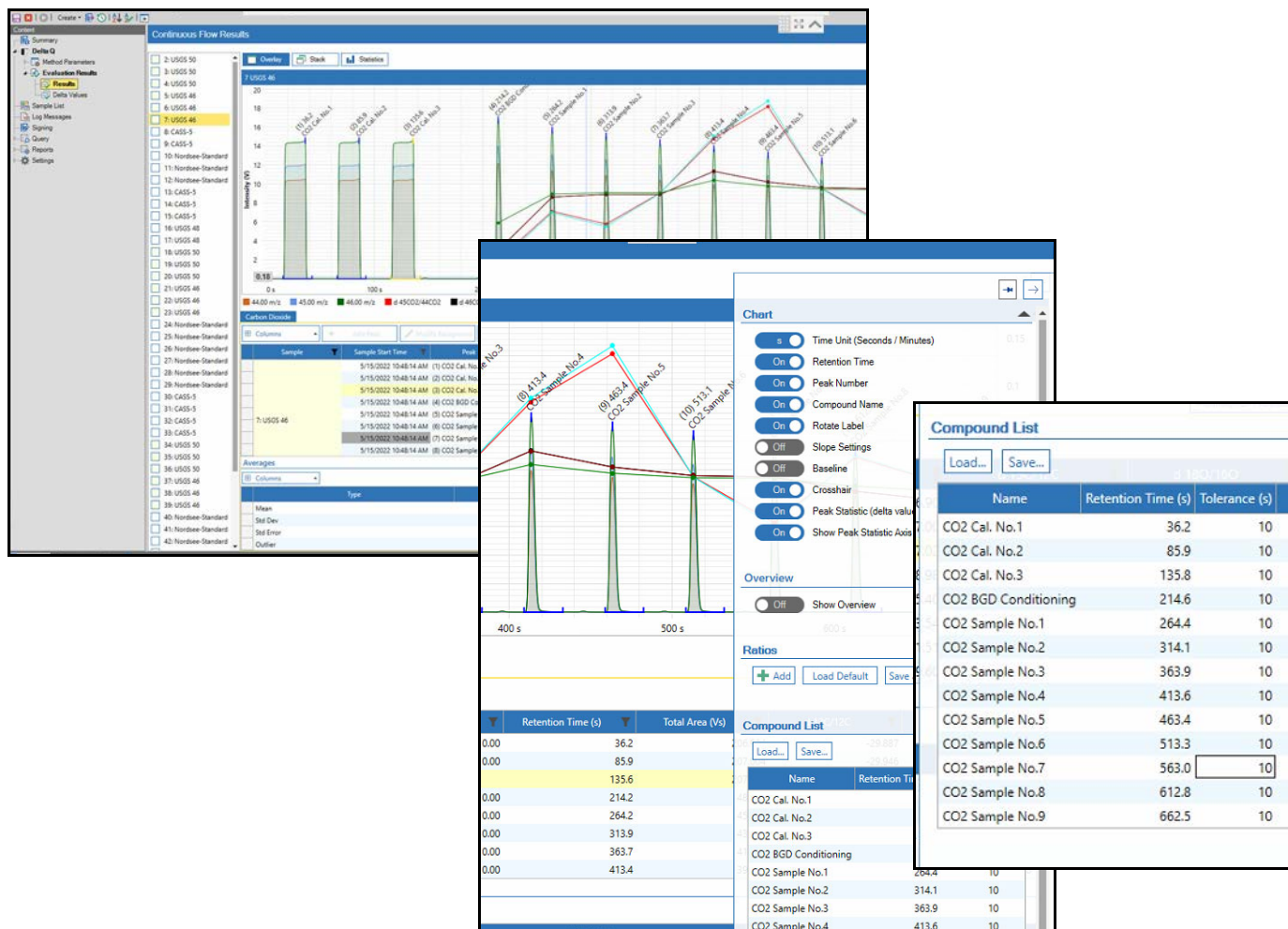


Figure 7-29. Result chromatogram by using right-click on compound diagram. Compound diagram can also be listed under evaluation results chromatogram and exported into the reports

Method Parameters - Standards and Delta Calculation

Refer to the *DELTA Q Operating Manual* for a generic description and adding isotopic standard ratios, exporting standards and importing standards into the LabBook Standard table (location of Standards.st file for backup). For a short example, see Figure 7-30, Figure 7-31, Figure 7-33).

- Isotope standards for GasBench Plus

Standard for GasBench are ordered under the category

1. “Bulk Isotope standards” - highlighted in a defined color, see Figure 7-31.

2. “Internal CO₂ chromatographic gas standard” - highlighted in a defined color (compound standard)
3. Reference Gas Standard - A liquid, solid or gas standard used as a standard to define the isotopic signal of the reference gas - highlighted in a defined color
4. Quality control standard for GasBench Plus (defined in dropdown list as replicate precision)

NOTICE

For the principle and features for using different standardization procedures refer to the *DELTA Q Operating Manual*.

NOTICE

The standardization principles for the main GasBench Plus applications are described under the applications, i.e., Carbonate isotope analysis versus VPDB / VSMOW scale using delta notation plus slope correction versus depleted standards (e.g., CO-9, LVSEC), oxygen isotope measurements of water (VSMOW / VSLAP normalization), hydrogen isotope measurements of water (VSMOW / VSLAP normalization).

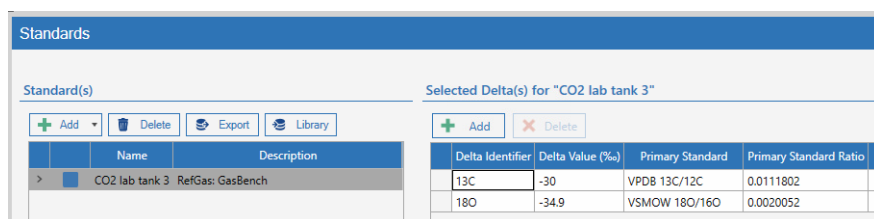


Figure 7-30. Standards. Reference Gas standard

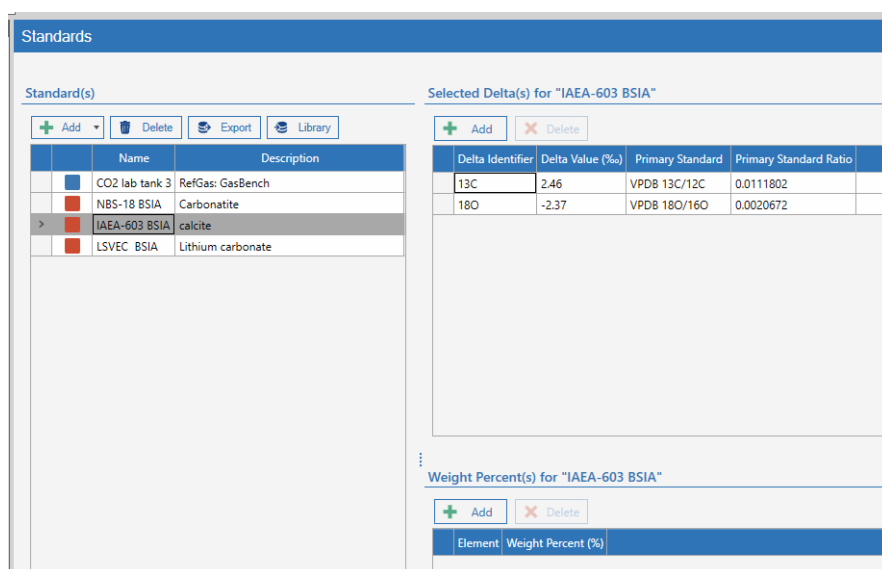


Figure 7-31. Standards. Bulk isotope standard (BSIA)

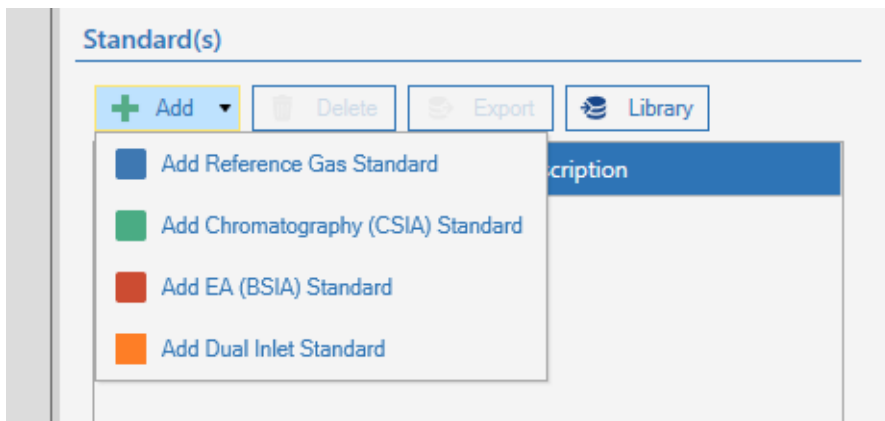


Figure 7-32. For GasBench, the BSIA standard must be added to be used for isotope standardisation



Figure 7-33. Standards. Chromatography (CSIA) Standard

Sample List - Reports - Automatic Export

Refer to the *DELTA Q Operating Manual* for the formatting and flagging on numbers and chromatographic features. The sample list will be covered with the individual main work flows.

#	Label	Status	Comment	Analysis No.	Action	Rack	Val	Evaluate	Sample Type	Reference
1	USGS 50		180 Equilibration	N/A	Measure	1	1	✓	Delta Standard (BSIA)	USGS 50
2	USGS 50		180 Equilibration	N/A	Measure	1	2	✓	Delta Standard (BSIA)	USGS 50
3	USGS 50		180 Equilibration	N/A	Measure	1	3	✓	Delta Standard (BSIA)	USGS 50
4	DI 1		180 Equilibration	N/A	Measure	1	4	✓	Unknown	
5	DI 1		180 Equilibration	N/A	Measure	1	5	✓	Unknown	
6	DI 1		180 Equilibration	N/A	Measure	1	6	✓	Unknown	
7	DI 2		180 Equilibration	N/A	Measure	1	7	✓	Unknown	
8	DI 2		180 Equilibration	N/A	Measure	1	8	✓	Unknown	
9	DI 2		180 Equilibration	N/A	Measure	1	9	✓	Unknown	
10	DI 3		180 Equilibration	N/A	Measure	1	10	✓	Unknown	
11	DI 3		180 Equilibration	N/A	Measure	1	11	✓	Unknown	
12	DI 3		180 Equilibration	N/A	Measure	1	12	✓	Unknown	
13	DI 4		180 Equilibration	N/A	Measure	1	13	✓	Unknown	
14	DI 4		180 Equilibration	N/A	Measure	1	14	✓	Unknown	
15	DI 4		180 Equilibration	N/A	Measure	1	15	✓	Unknown	
16	USGS 48		180 Equilibration	N/A	Measure	1	16	✓	QC Standard	USGS 48
17	USGS 48		180 Equilibration	N/A	Measure	1	17	✓	QC Standard	USGS 48
18	USGS 48		180 Equilibration	N/A	Measure	1	18	✓	QC Standard	USGS 48
19	DI 5		180 Equilibration	N/A	Measure	1	19	✓	Unknown	
20	DI 5		180 Equilibration	N/A	Measure	1	20	✓	Unknown	
21	DI 5		180 Equilibration	N/A	Measure	1	21	✓	Unknown	
22	USGS 46		180 Equilibration	N/A	Measure	1	22	✓	Delta Standard (BSIA)	USGS46
23	USGS 46		180 Equilibration	N/A	Measure	1	23	✓	Delta Standard (BSIA)	USGS46
24	USGS 46		180 Equilibration	N/A	Measure	1	24	✓	Delta Standard (BSIA)	USGS46
25	DI 6		180 Equilibration	N/A	Measure	1	25	✓	Unknown	
26	DI 6		180 Equilibration	N/A	Measure	1	26	✓	Unknown	
27	DI 6		<Comment>	N/A	Measure	1	27	✓	Unknown	
28	USGS 50		<Comment>	N/A	Measure	1	28	✓	Delta Standard (BSIA)	USGS 50
29	USGS 50		<Comment>	N/A	Measure	1	29	✓	Delta Standard (BSIA)	USGS 50
30	USGS 50		<Comment>	N/A	Measure	1	30	✓	Delta Standard (BSIA)	USGS 50
31	USGS 46		<Comment>	N/A	Measure	1	31	✓	Delta Standard (BSIA)	USGS46
32	USGS 46		<Comment>	N/A	Measure	1	32	✓	Delta Standard (BSIA)	USGS46
33	USGS 46		<Comment>	N/A	Measure	1	33	✓	Delta Standard (BSIA)	USGS46
34	USGS 48		<Comment>	N/A	Measure	1	34	✓	QC Standard	USGS 48
35	USGS 48		<Comment>	N/A	Measure	1	35	✓	QC Standard	USGS 48
36	USGS 48		<Comment>	N/A	Measure	1	36	✓	QC Standard	USGS 48

Figure 7-34. General LabBook for GasBench Plus, e.g., for equilibration measurements

Settings with Flags and Number Formatting for the LabBook Features

Refer to the *DELTA Q Operating Manual* for the formatting and flagging on numbers and chromatographic features. The flagging is quite useful to identify certain lines, e.g. outlier rejections, peak identification as reference peak, etc. formatting can be changed at any time in the LabBook.

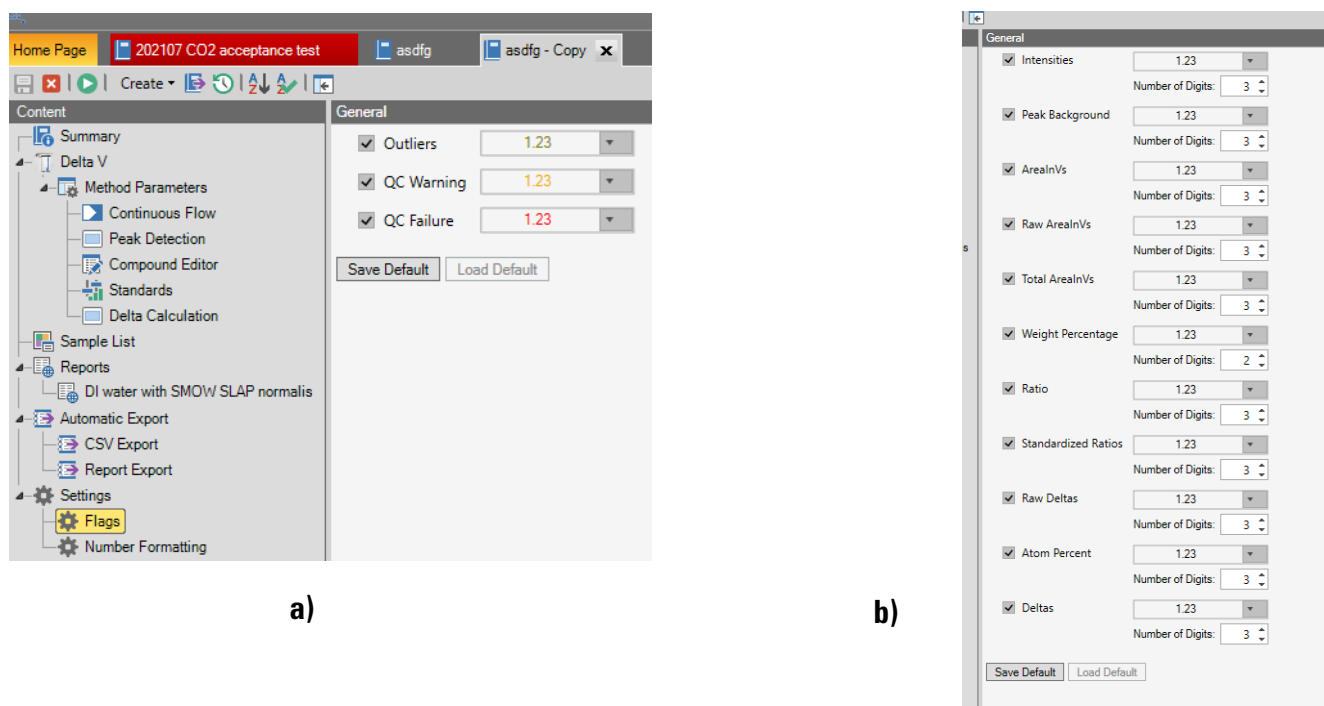


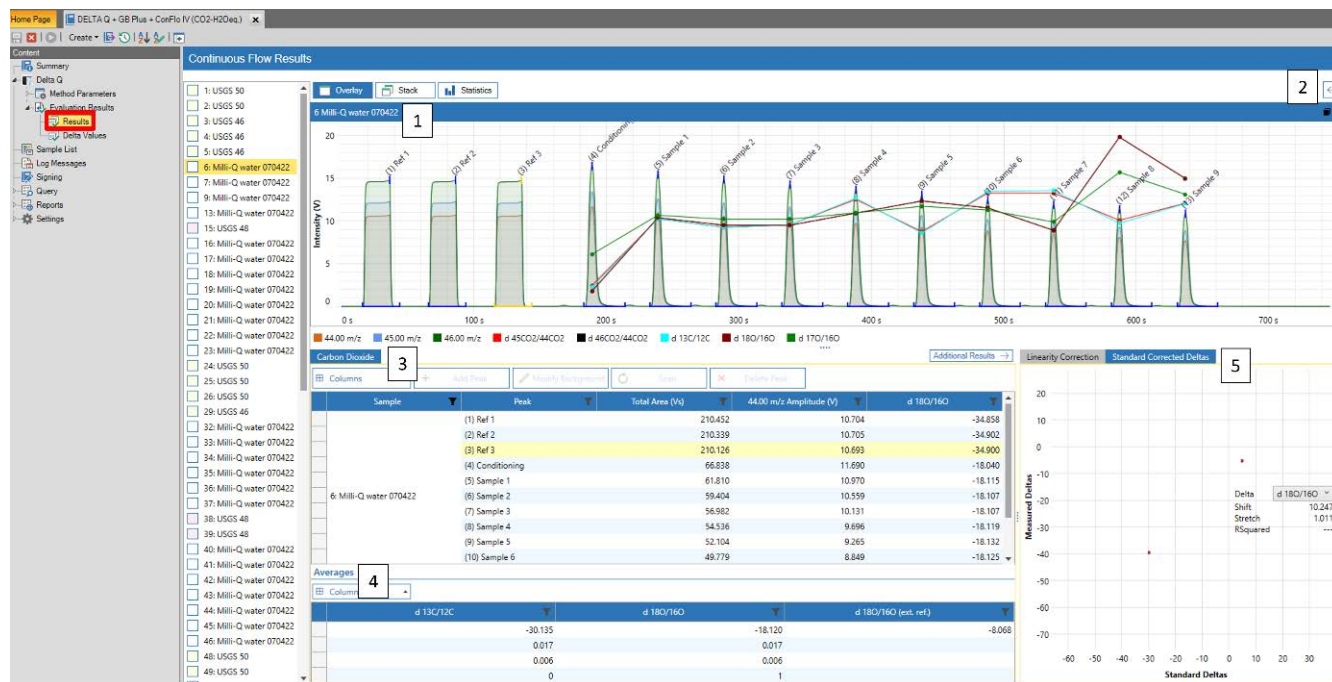
Figure 7-35. Example for a) setting flags and b) formatting numbers

Results View

The Results view of the LabBook shows the results and acquisition information reported during and after the acquisition. Each individual sample has its own entry (Results sheet), which can be selected in the vertical sample overview on the left side. The individual Results sheets comprise the components as indicated in [Figure 7-36](#).

NOTICE

Only samples that are marked as 'Evaluate' in the Sample List (see ?), will be displayed in the Results view.



Labeled Components: 1=Chromatogram, 2=Chart Settings, 3=Peak Data Table, 4=(Averaged) Sample Data Table, 5=Additional Results section (see Table 7-1 for more information)

Figure 7-36. Results view

Table 7-1. Results view items



No.	Item	Explanation
1	Chromatogram	An intensity vs. time plot which illustrates the recorded mass traces during the timeline run (see page 7-24). Reference and sample gas pulses are displayed.
2	Chart Settings ( slides out)	These settings can be used to adapt/optimize the layout of the chromatogram as well as accessing a few additional chart features (for more details see ‘Chart Settings’ on page 7-see-below).
3	Peak Data Table	The table can display the complete data set for each measured peak. The  Columns tab allows to define in detail which data should be illustrated in the columns. The other tabs above the table allow to manually edit the peak detection of individual peaks.

Table 7-1. Results view items, continued



No.	Item	Explanation
4	(Averaged) Sample Data Table	The averaged results of the delta calculations are displayed for the respective sample (see “Averaging” on page 7-45) as well as, e.g., the externally referenced or drift corrected data in case of unknown samples (see “External Referencing” on page 7-47 , “Drift Correction” on page 7-47). The  Columns tab allows to define in detail which data should be illustrated in the columns.
5	Additional Results section ( slides out)	This section displays information concerning linearity correction and the external referencing applied to the unknown samples (see “Linearity Correction” on page 7-44 and “External Referencing” on page 7-47).

Chart Settings

The Chart Settings comprise options to

- the chromatogram ('Chart'),
- an additional overview plot ('Overview'),
- an additional ratio plot ('Ratios'), and
- access to the Compound List ('Compound List') (see [Figure 7-37](#)).

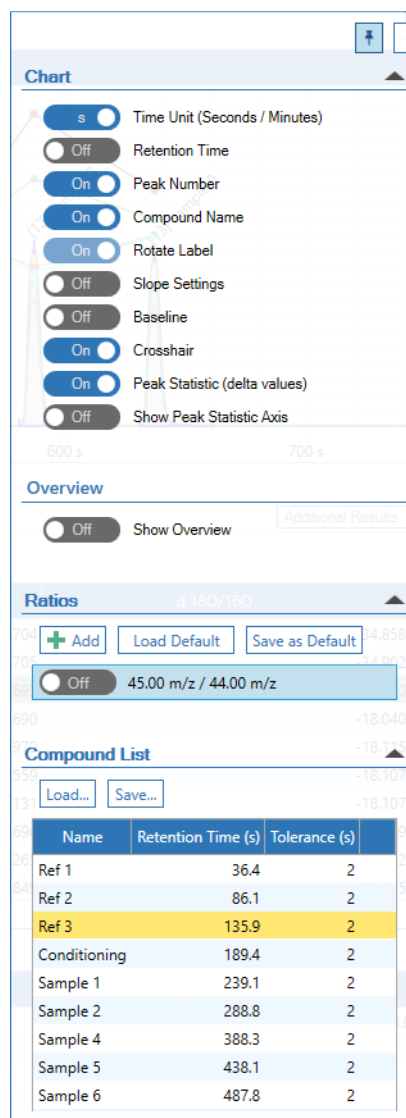


Figure 7-37. Chart Settings with sections Chart, Overview, Ratios, and Compound List

Linearity Correction

The linearity correction including H3 factor correction is explained in greater detail in the respective *Mass Spectrometer Operating Manual*. With respect to GasBench IRMS analyses it is important to know that

- for a GasBench-ConFlo IV system the linearity/H3 factor can be determined via an automatic process using Get Ready, the TuneBook, or as part of the LabBook method, and
- for a GasBench standalone device this action can be manually performed with the help of a wizard within the TuneBook (see [“Linearity Test with Qtegra with Standalone Reference Ports \(Pistons\)”](#) on page 7-53).

The Linearity Correction can be turned *On* in the LabBook in the Additional Results section 'Linearity Correction' (see **5** in Figure 7-36). The last determined correction factor is applied to the correction of the respective sample.

Averaging

In the course of GasBench IRMS analyses, the sample gas is (in most cases) pulsed several times using the injection loop and the Valco 8-port valve to determine the isotope composition of the sample gas with an increased precision. The Peak Data Table (see **3** in Figure 7-36) provides the delta values vs. reference gas of the individual sample peaks/pulses. The next step is to average the peak-wise delta values to calculate the isotope composition of the sample gas.

The Chart Setting 'Peak Statistics' (see "Method Parameters - Peak Detection" on page 7-34, see Figure 7-38) helps us to assess the quality of our peak-wise data and to determine if certain sample pulses should be excluded from the averaging/sample result calculation.

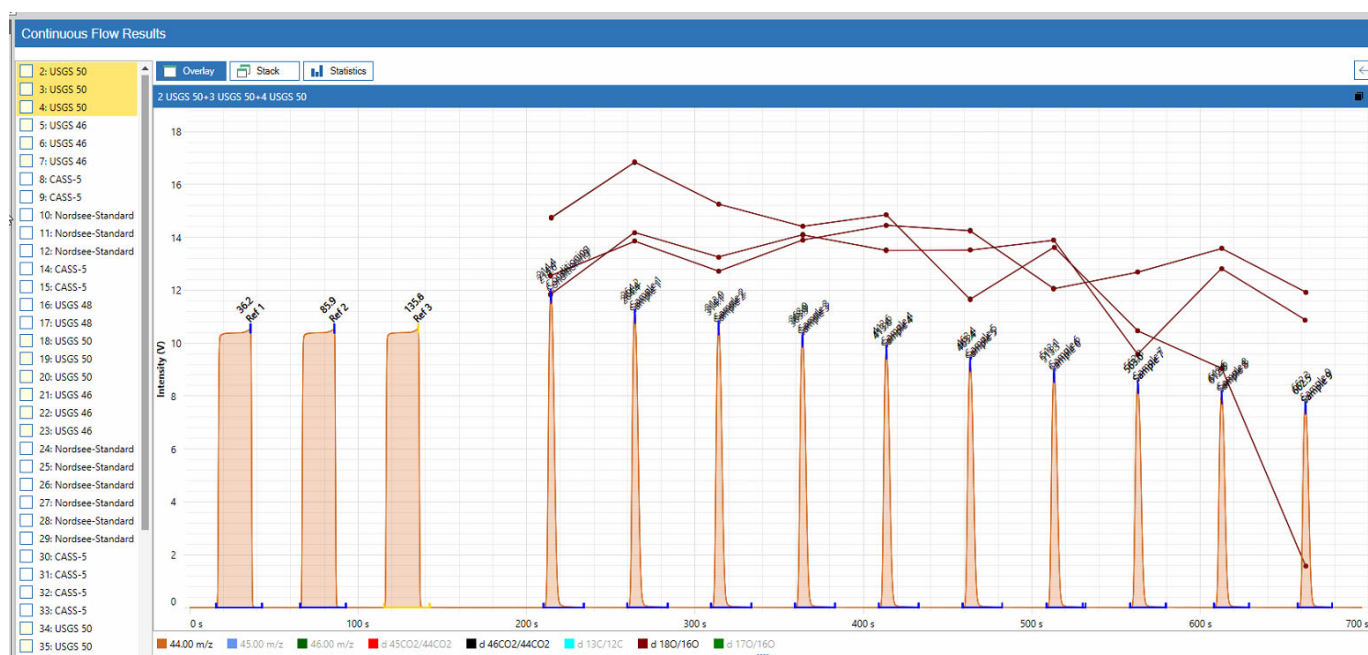


Figure 7-38. Peak Statistics view for data evaluation

Tip Sometimes it might help to exclude the first sample pulse because the system is not fully conditioned yet.

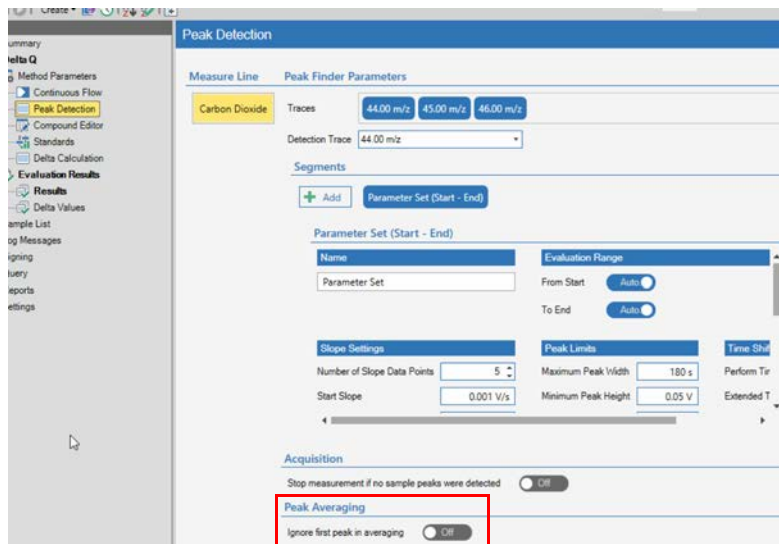


Figure 7-39. Enable the highlighted switch to exclude the first sample peak

Certain sample pulses can be excluded from the averaging in the Peak Data Table (see Figure 7-40). Insert the column 'Average' and switch *Off* the peak to exclude. The sample result calculation is immediately updated. This affects only the averaging of this specific sample. It is possible to exclude the first sample peak for all analyzed samples in Peak Detection > Peak Averaging.

Sample	Peak	Average	Total Area (Vs)	44.00 m/z Amplitude (V)	d 180/160
7: Milli-Q water 070422	(1) Ref 1		210.333	10.700	-34.843
	(2) Ref 2		210.160	10.686	-34.883
	(3) Ref 3		210.218	10.685	-34.900
	(4) Conditioning		67.861	11.858	-17.969
	(5) Sample 1	On	62.818	11.123	-17.956
	(6) Sample 2	On	60.229	10.701	-17.998
	(7) Sample 3	On	57.723	10.265	-18.047
	(8) Sample 4	On	55.238	9.815	-18.028
	(9) Sample 5	On	52.707	9.367	-18.052
	(10) Sample 6	On	50.274	8.917	-18.000

Type	d 180/160	d 180/160 (ext. ref.)
Mean	-18.041	-7.988
Std Dev	0.043	
Std Error	0.014	
Outlier	0	

Figure 7-40. Averaging peaks of specific samples

The results of averaging (Mean, Std Dev, Std Error, and Outlier) are displayed in the (Averaged) Sample Result Table (see 4 in Figure 7-36). All further corrections, like external referencing are (in this example) applied to the averaged delta values.

Drift Correction

Qtegra ISDS Software provides the option to correct the instrumental **drift** affecting the delta values of the samples (Sample Type: *Unknowns*) using the delta values of the standards (Sample Type: *Drift Correction*) measured under the same conditions as the samples. A linear drift correction function is used for the correction defined by the delta values and the Run ID of the standards, for more details refer to the *Data Processing Algorithms for GIRMS Software Manual*.

The Sample List in the LabBook is crucial to define the applied drift correction. Only if a drift correction standard was analyzed before and after the respective sample, the sample drift can be corrected. The drift correction is not extrapolated. More information can be found in the respective *Mass Spectrometer Operating Manual*.

NOTICE

The drift correction is applied to the averaged sample data and the results are stored in the (Averaged) Sample Data Table.

External Referencing

Qtegra ISDS Software provides the possibility of external referencing the delta values of the samples (Sample Type: *Unknowns*) using the delta values of the standards (Sample Type: *Delta Standard (BSIA)*) measured under the same conditions as the samples. Qtegra supports: one-point referencing, two-point referencing, and multiple point referencing (for more details refer to the *Data Processing Algorithms for GIRMS Software Manual*).

The Sample List in the LabBook is crucial to define the applied external referencing strategy. Only the standards, which are listed above the respective samples, are used as input for the external referencing of those samples. With every new analyzed standard or series of standards a new 'reference block' is started (see Figure 7-36). If all standards should be considered for the external referencing of all samples in the LabBook (one correction factor applied to all samples), it is necessary to drag all standards to the top of the Sample List after LabBook completion so that one large 'reference block' is created. The strategy of the external referencing can be adapted and changed at any time in the Sample List of the LabBook as soon as the LabBook has finished. If several measurements of the same standard are part of the 'reference block', the average value will be used as input for the external referencing. If several

different standards are part of the 'reference block', Qtegra will perform a two-point or multiple point referencing depending on the number of standards.

NOTICE

Externally referencing is applied to the averaged sample data and the results are stored in the (Averaged) Sample Data Table.

NOTICE

There is the option to restore the measurement order at any time by considering the fixed numbering in the 'Run ID' column of the Sample List.

NOTICE

QC Standards are treated as samples and are not considered as standards used for external referencing. However, it is possible, after LabBook completion, to switch QC Standards to Delta Standards and use them for the external referencing.

Sample List	Label	Status	Comment	Run ID	Action	Rack	Vial	Evaluate	Sample Type	Reference	Weight %	Amount
1	USGS 50	●	200 ul, 25 degree.	1	Measure		2	2	✓	Delta Standard (BSIA)	USGS 50	200
2	USGS 50	●	200 ul, 25 degree.	2	Measure		2	3	✓	Delta Standard (BSIA)	USGS 50	200
3	USGS 46	●	200 ul, 25 degree.	3	Measure		2	4	✓	Delta Standard (BSIA)	USGS46	200
4	USGS 46	●	200 ul, 25 degree.	4	Measure		2	5	✓	Delta Standard (BSIA)	USGS46	200
5	USGS 46	●	200 ul, 25 degree.	5	Measure		2	6	✓	Delta Standard (BSIA)	USGS46	200
6	Milli-Q water 0704	●	200 ul, 25 degree.	6	Measure		2	7	✓	Unknown		200
7	Milli-Q water 0704	●	200 ul, 25 degree.	7	Measure		2	8	✓	Unknown		200
8	Milli-Q water 0704	●	200 ul, 25 degree.	8	Measure		2	9	✓	Unknown		200
9	Milli-Q water 0704	●	200 ul, 25 degree.	9	Measure		2	10	✓	Unknown		200
10	Milli-Q water 0704	●	200 ul, 25 degree.	10	Measure		2	11	✓	Unknown		200
11	Milli-Q water 0704	●	200 ul, 25 degree.	11	Measure		2	12	✓	Unknown		200
12	Milli-Q water 0704	●	200 ul, 25 degree.	12	Measure		2	13	✓	Unknown		200
13	Milli-Q water 0704	●	200 ul, 25 degree.	13	Measure		2	14	✓	Unknown		200
14	USGS 48	●	200 ul, 25 degree.	14	Measure		2	15	✓	QC Standard	USGS 48	200
15	USGS 48	●	200 ul, 25 degree.	15	Measure		2	16	✓	QC Standard	USGS 48	200
16	Milli-Q water 0704	●	200 ul, 25 degree.	16	Measure		2	17	✓	Unknown		200
17	Milli-Q water 0704	●	200 ul, 25 degree.	17	Measure		2	18	✓	Unknown		200
18	Milli-Q water 0704	●	200 ul, 25 degree.	18	Measure		2	19	✓	Unknown		200
19	Milli-Q water 0704	●	200 ul, 25 degree.	19	Measure		2	20	✓	Unknown		200
20	Milli-Q water 0704	●	200 ul, 25 degree.	20	Measure		2	21	✓	Unknown		200
21	Milli-Q water 0704	●	200 ul, 25 degree.	21	Measure		2	22	✓	Unknown		200
22	Milli-Q water 0704	●	200 ul, 25 degree.	22	Measure		2	23	✓	Unknown		200
23	Milli-Q water 0704	●	200 ul, 25 degree.	23	Measure		2	24	✓	Unknown		200
24	USGS 50	●	500 ul, 25 degree.	24	Measure		2	25	✓	Delta Standard (BSIA)	USGS 50	500
25	USGS 50	●	500 ul, 25 degree.	25	Measure		2	26	✓	Delta Standard (BSIA)	USGS 50	500
26	USGS 50	●	500 ul, 25 degree.	26	Measure		2	27	✓	Delta Standard (BSIA)	USGS 50	500
27	USGS 46	●	500 ul, 25 degree.	27	Measure		2	28	✓	Delta Standard (BSIA)	USGS46	500
28	USGS 46	●	500 ul, 25 degree.	28	Measure		2	29	✓	Delta Standard (BSIA)	USGS46	500
29	USGS 46	●	500 ul, 25 degree.	29	Measure		2	30	✓	Delta Standard (BSIA)	USGS46	500
30	Milli-Q water 0704	●	500 ul, 25 degree.	30	Measure		2	31	✓	Unknown		500
31	Milli-Q water 0704	●	500 ul, 25 degree.	31	Measure		2	32	✓	Unknown		500
32	Milli-Q water 0704	●	500 ul, 25 degree.	32	Measure		2	33	✓	Unknown		500
33	Milli-Q water 0704	●	500 ul, 25 degree.	33	Measure		2	34	✓	Unknown		500

Figure 7-41. External referencing- example for the 'reference block' system.
 1=first reference block: two-point referencing will be applied to the samples (Milli-Q water/USGS 48 from sample lines 6-23) using the USGS 50 and USGS 46 (sample lines 1-2 and 3-5).
 2=second reference block: two-point referencing will be applied to the samples (Milli-Q water from sample lines 30-33) using the USGS 50 and USGS 46 (sample lines 24-26, 27-29).

The externally referenced results of the samples are shown in the (Averaged) Sample Data Table of the Results View (see **3** in [Figure 7-36](#), [Table 7-1](#)). Moreover, the parameters of the external referencing applied to the sample can be checked in the Additional Results section > Standard Corrected Deltas (see **5** in [Figure 7-36](#), [Table 7-1](#)). [Figure 7-42](#) opens and illustrates the measured (averaged if applicable) delta values of the standards from this 'reference block' versus the true values from the Standard Library including the external referencing parameters (shift, stretch) and the R2 value. The dropdown menu (highlighted in red in [Figure 7-42](#)) can be used to switch between different delta values.

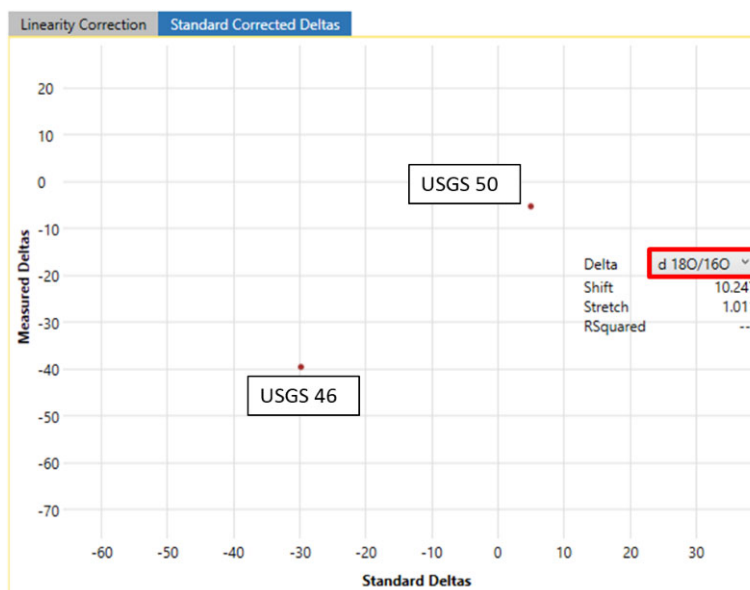


Figure 7-42. Plot illustrating the applied external referencing parameters concerning the $e^{18}O$. This example corresponds to the samples of the first reference block illustrated in [Figure 7-41](#) (two-point referencing: USGS 50, USGS 46).

Statistics View within One Sample

Qtegra ISDS Software provides a statistics tool to evaluate the data in the Peak Data Tables in greater detail.

1. Select data entries with the cursor.
2. Right-click and select **Open Statistic View** (see [Figure 7-43](#))




The screenshot shows a table with two columns: 'd 18O/16O (‰)' and 'D 47CO2/44CO2 (‰)'. The table contains 12 rows of data. A context menu is open over the 5th row, displaying the text 'Open Statistic View ...'. The data values are as follows:

d 18O/16O (‰)	D 47CO2/44CO2 (‰)
-5.158	4.608
-5.153	3.865
-5.164	3.736
-5.166	3.976
-5.188	3.999
-5.170	3.808
-5.181	3.687
-5.217	4.117
-5.211	4.109
-5.209	3.911
-5.226	3.524

Figure 7-43. Statistics view for one sample

A window is opened and provides a basic statistical evaluation of the selected data (e.g., mean value, standard deviation etc.)

Statistics View Across Several Samples

When  **Statistics** is chosen in the Results View and several samples are selected in the vertical sample overview, then the Statistics View across several samples is opened (see [Figure 7-44](#)). It allows to visualize the results gained during the GasBench IRMS run and to compare the results of selected samples. This is a powerful tool to interpret the results.

The Statistics View across several samples provides:

1. a table showing the individual results of the selected samples (Data Table),
2. a table containing the average delta values (internally and/or externally referenced), the standard deviation, maximum and minimum of the selected sample set (Results Table), and
3. plots illustrating each calculated delta value vs. Run ID (see [Figure 7-44](#)).

The columns settings of the tables can be accessed and modified in the upper left corner of the respective table. The plots can be enlarged and reduced by clicking on the window symbol in the upper right corner of the respective plot. It is also possible to visualize the externally referenced data in plots (see [“External Referencing”](#) on [page 7-47](#)).

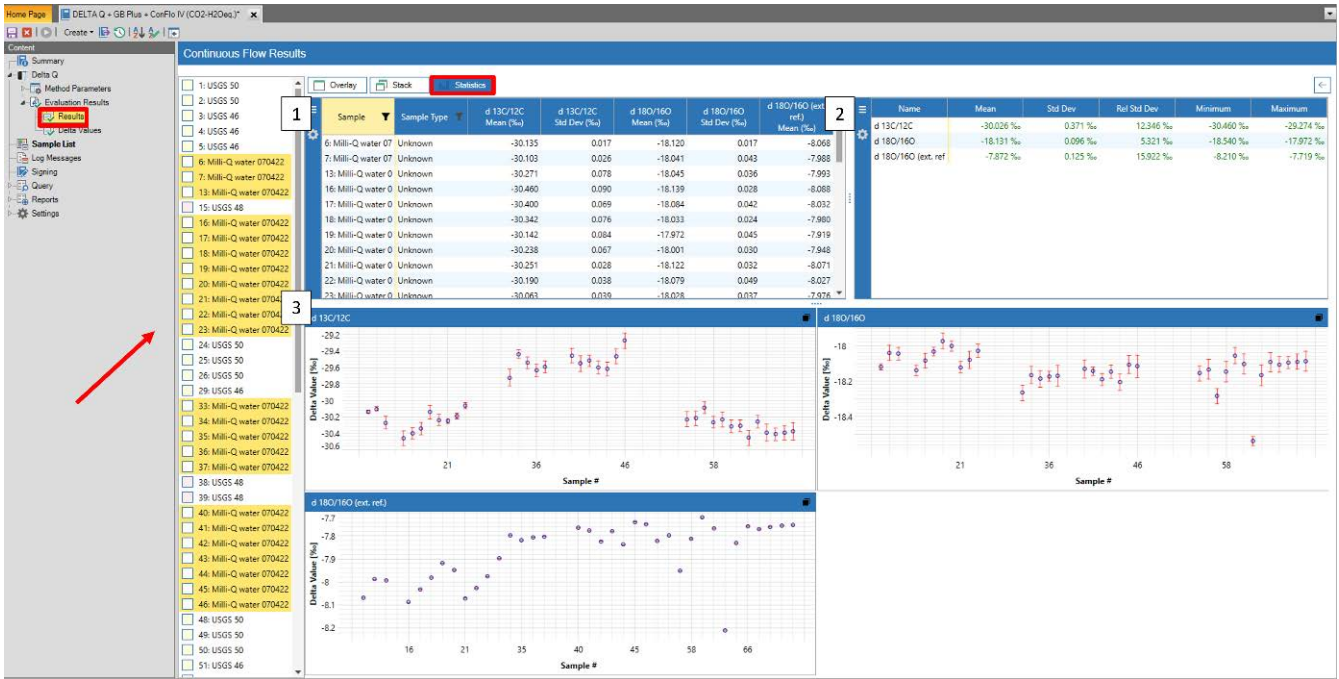


Figure 7-44. Statistics view across several samples

Qtegra Templates

File Manager, LabBook Query

The homepage objects do have no extra considering aspects for the GasBench Plus. Refer to the *DELTA Q Operating Manual Manual* for generic description of these objects.

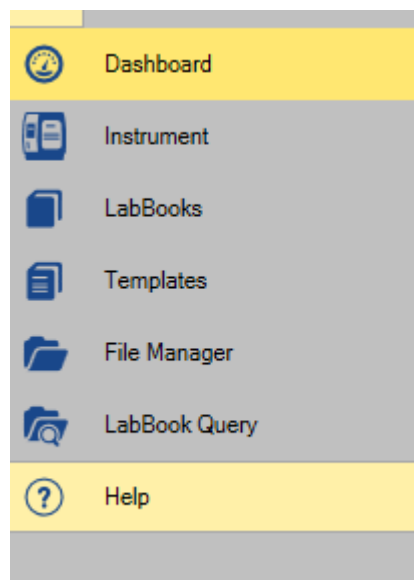


Figure 7-45. Qtegra homepage with Templates, File Manager and LabBook Query

Standard Off-test

The standard on-off test is to test the system stability for isotope ratio measurements and done on the same signal height. Refer to the *DELTA Q Operating Manual* for standard on off tests. For a GasBench with standalone referencing Qtegra will detect automatically that the reference pistons will be used for zero enrichment tests by the installed configuration without a ConFlo IV.

Linearity and H₃ Plus Factor Determination

The linearity test is to test the system linearity for isotope ratio measurements if a signal height changes. Refer to the *DELTA Q Operating Manual* for standard on off tests. For a GasBench with standalone referencing, Qtegra automatically detects that the reference pistons is used for linearity tests and H₃ Plus factor determination.

Linearity Test with Qtegra with Standalone Reference Ports (Pistons)

Qtegra with GasBench series allows to determine the linearity of the system under the instrument tab and the linearity determination button.

For the H₃ plus factor the same strategy will be follow except for that the H₃ plus factor will be determined and used automatically in the LabBook for measurement. A H₃ plus factor shall be determined at least daily. Qtegra will request a new determination if the last H₃⁺ factor determination is out of a reliable isotope measurement accuracy of samples.

❖ To perform the linearity test

1. Define the range of the linearity signal. Qtegra will request at least three steps to determine the linearity or the H₃ plus factor.
2. Use the appropriate focusing for H₃ plus factor determination. Refer to the *DELTA Q Operating Manual* to check the optimization for hydrogen isotope measurements or any different analyte gas.
3. The range of the pressure can be quickly determined in the Dashboard by going the lowest signal and the highest signal. Be aware that not the lowest signal can be taken for a proper linearity or H₃⁺ factor determination (see *DELTA Q Operating Manual*). The linearity can be determined for any other TuneBook, e.g., CO₂, N₂, N₂O TuneBook)
4. Click **Play**.

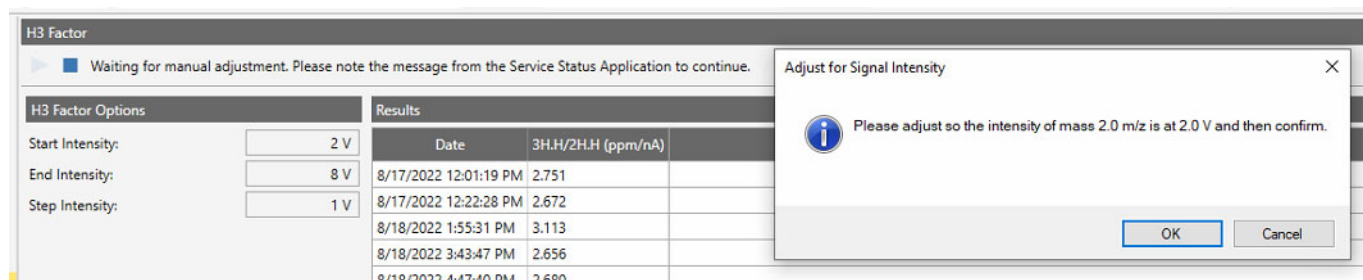


Figure 7-46. Starting the linearity determination wizard. **Play** will activate a wizard to adjust the required intensities for a linearity correction determination. Here, H₂ is used for the H₃ plus factor calculation.

5. Before each measurement Qtegra will request to adjust the appropriate pressure step. If confirmed it will start to determine the isotope ratio for that pressure after a delay time and request a next increase of pressure until the upper range is reached (see [Figure 7-47](#)).

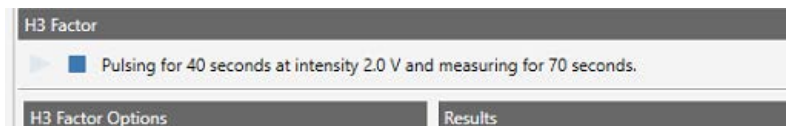


Figure 7-47. Starting the determination at 2 V intensity

- Qtegra calculates the linearity of the H₃ Plus factor accordingly (see *DELTA Q Operating Manual*).

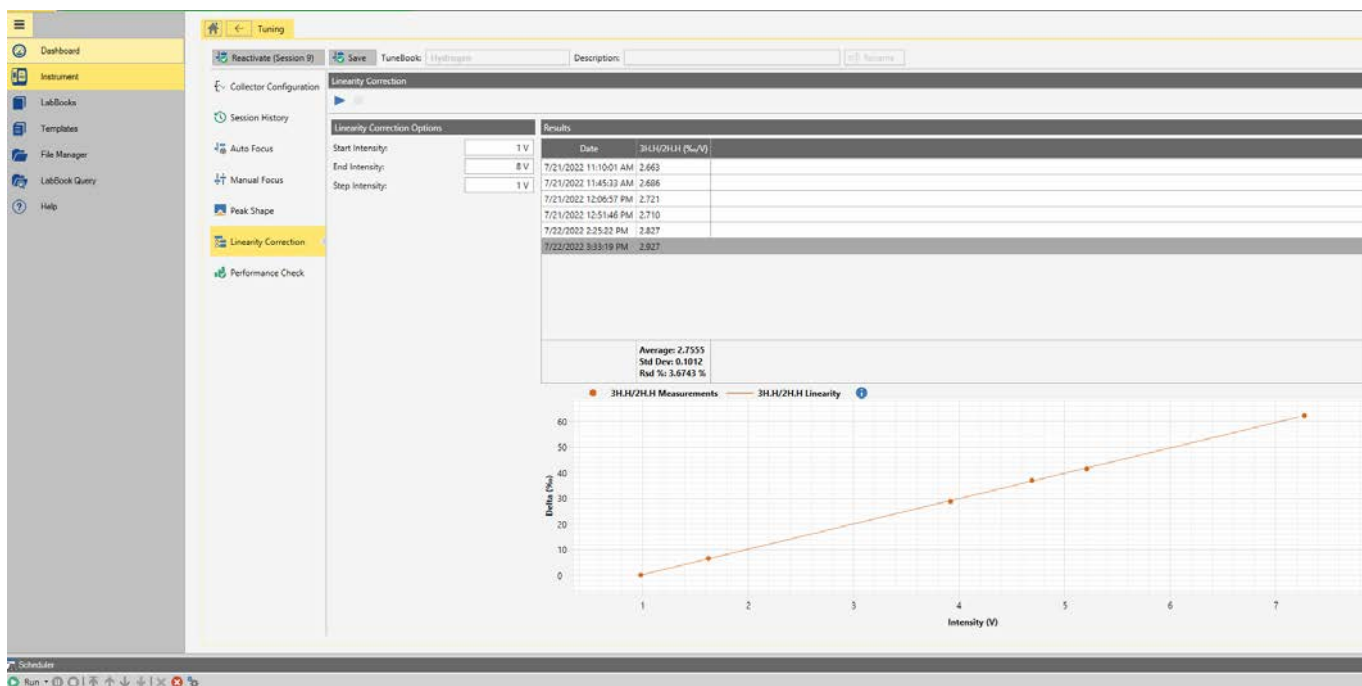


Figure 7-48. Linearity test under Instrument Tuning to determine the isotope ratio linearity of the system including ConFlo IV. The H₃ factor determination with GasBench Plus without ConFlo IV is workflow-guided and the results are saved in the same history files. If in H₂ tune mode, the linearity test is used for the H₃ factor determination, which is used for the LabBook samples.

See “Preparing a Test Sample” on page 6-15.

Performing Preparation of Samples using Prescripts with Qtegra

Some applications require a pre-preparation process before the timeline starts. The Qtegra LabBook measurement enables to start a prescript process including autosampler injection of the TriPlus RSH SMART autosampler during the process.

Configurations which require prescripting are:

1. GasBench with Trapping
2. GasBench with PreCon Applications
3. GC IsoLink with PreCon Applications

Tip This is a general description of prescripts. The individual scripts are pre-installed during the Qtegra ISDS Software installation. The specific script is described at the dedicated application workflow, for example, using GasBench traps for low concentration measurements or GC IsoLink II prescript for PreCon preparation and GC 1310 start.

NOTICE

GasBench II Sample Preparation scripts can be executed with GasBench Plus scripts since the CTC autosampler will be validated automatically. The configured autosampler and the GasBench model of the Experiment Configuration will be validated, and the autosampler is automatically used.

Description of Scripts and Implementation

From the folder *C:\ProgramData\Thermo\Qtegra_Application Data\PluginData\TFS253Plus\Scripts\SamplePreparation*, Scripts can be opened with a text editor and then be saved under the location. See [Figure 7-49](#). The scripts can be used for all GIRMS mass spectrometers, i.e., DELTA series & 253 Plus.

Opening the scripts shows basic commands and executed action, e.g., open, close, up, down, load, inject, “valve actions”, etc.

Folders named “Performance Tests PreCon GC IsoLink II” and “PreConTestScripts” contain test scripts to test the PreCon function and the GC separation of N₂O or CO₂ with the PreCon. Refer to the *PreCon Operating Manual*.

Performance Tests PreCon GC Isolink	7/19/2022 2:01 PM	File folder	
PreConTestScripts	7/19/2022 2:01 PM	File folder	
GasBench - A2005 - TraceGC Example.txt	7/13/2022 1:01 AM	Text Document	1 KB
GasBench Plus Denitrification.txt	7/13/2022 1:01 AM	Text Document	2 KB
GasBench Plus N2O static memory gas.txt	7/13/2022 1:01 AM	Text Document	1 KB
GasBench Plus Single Trap Small Carbonates.txt	7/13/2022 1:01 AM	Text Document	1 KB
GasBench Plus Single Trap Small Low CO2 in Air.txt	7/13/2022 1:01 AM	Text Document	1 KB
GasBench.aliases	7/13/2022 1:01 AM	ALIASES File	1 KB
PeriCon Example User Prompt.txt	7/13/2022 1:01 AM	Text Document	1 KB
PeriCon Example.txt	7/13/2022 1:01 AM	Text Document	1 KB
PreCon GasBench Plus Blank.txt	7/13/2022 1:01 AM	Text Document	1 KB
PreCon GasBench Plus UsePreCon_PreinstalledSampleLoop.txt	7/13/2022 1:01 AM	Text Document	1 KB
PreCon GasBench Plus UsePreCon_Sample Port.txt	7/13/2022 1:01 AM	Text Document	2 KB
PreCon GasBench Plus vRSH.txt	7/13/2022 1:01 AM	Text Document	2 KB
PreCon GC Isolink II PreCon_Use_PreinstalledSampleLoop.txt	7/13/2022 1:01 AM	Text Document	2 KB
PreCon GC Isolink II PreCon_Use_Sample Port.txt	7/13/2022 1:01 AM	Text Document	2 KB
PreCon GC Isolink II vRSH.txt	7/13/2022 1:01 AM	Text Document	2 KB
PreCon GC Isolink II_Blank.txt	7/13/2022 1:01 AM	Text Document	2 KB
TransferandFreeze.txt	7/13/2022 1:01 AM	Text Document	1 KB
TrapTransferInject.txt	7/13/2022 1:01 AM	Text Document	1 KB

Figure 7-49. Scripts in Sample Preparation folder

Consider the following rules to prevent from typical errors:

- Commands are case sensitive.
- Numbers and units, e.g. “second”: s, do not require a space.
- Spaces must be set between
 - A command must have a space in between the description.
 - //A command is noted, would not execute the script.
 - // A command is noted, would not give an error.

The following command structure exists (see tables):

Table 7-2. Generic commands and actions for Qtegra prescripts

Command	Action / Unit	Comment
Wait	s min h	No space needed.
“units”	%	For dilution
//	comments	Space must be precede after “//”
Info	“Text”, information displayed in the Qtegra information bar	Space
Prompt	Dialog box	Must be executed before the next script command.
Call <i>with</i> or <i>without</i> .txt extension	Script call	Library scripts (used in multiple scripts) can be executed with a call. Must be stored in the same sample preparation folder.

Table 7-3. Commands and actions for ConFlo IV

Command	Action / Unit	Comment
ConFloIV Sample Dilute	% value	Picks up calibrated dilution
ConFloIV Reference Dilute	% value	Picks up calibrated dilution
ConFloIV Reference I_1	On Off	Defined reference
ConFloIV Reference C.O2	On Off	Name as in ConFlo IV Dashboard

Table 7-4. Commands and actions for GasBench

Command	Action / Unit	Comment
GasBench Valco Inject	Load Inject	
GasBench Trap 1	Down Up	
GasBench Trap 2	Down Up	
GasBench Split	In Out	
GasBench FlushFill	On Off	
GasBench Reference 1	On Off	
GasBench Reference 2	On Off	
GasBench Reference 3	On Off	
GasBench Reference C.O2	On Off	Name as in GasBench Dashboard

Table 7-5. Commands and actions for PreCon

Command	Action / Unit	Comment
PreCon Vent 2	Close Open	
PreCon Purge	Close Open	
PreCon Valco	Load Inject	
PreCon Trap 2	Down Up	
PreCon Trap 3	Down Up	

Table 7-6. Commands and actions for GC Isolink II

Command	Action / Unit	Comment
GCIsoLinkII Vent	Close Open	
GCIsoLinkII He	Close Open	
GCIsoLinkII 4PV	Close Open	Switching valve between pyrolysis and combustion furnace.
GCIsoLinkII Trap	Down Up	
GCIsoLinkII FM	Close Open	Flow Meter
GCIsoLinkII BF	Close Open	Backflush Valve

Table 7-6. Commands and actions for GC Isolink II, continued

Command	Action / Unit	Comment
GCIsoLinkII O2	Close Open	O ₂ Valve
GCIsoLinkII CH4	Close Open	Methane Valve
GCIsoLinkII MS	In Out	In: MS LF & MS capillary in Out: LF out and MS out
GCIsoLinkII Vent	Close Open	Vent

Table 7-7. Commands and actions for TriPlus RSH SMART autosamplers

Command	Action / Unit	Comment
GasBench AS Next	Moves AS	Executes move to Qtegra sample list position, “GasBench” might change.
GasBench AS Home	Moves AS to home	Executes autosampler home position move. “GasBench” might change.

Table 7-8. Commands and actions for TriPlus RSH SMART autosamplers for GasBench

Command	Action / Unit	Comment
GasBench AS Next	Moves AS	Executes move to Qtegra sample list position
GasBench AS Home	Moves AS to home	Executes autosampler home position move

Commands and actions for TriPlus RSH SMART specifically for GC IsoLink II and the TriPlus RSH SMART for GasBench; used for the Trace GC 1310 operation with the GC IsoLink II using prescript (see [Table 7-8](#)).

Table 7-9. Commands and actions for TriPlus RSH SMART for GC IsoLink II and for TriPlus RSH SMART for GasBench or PreCon (same SMART autosampler firmware) Important for CTC Pal 80 with Trace GC 1310 with PreCon application^a

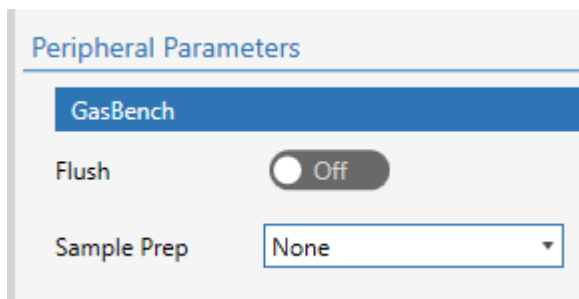
Command	Action / Unit	Comment
GCIsoLinkII AS WaitForSignal GC1	Waits for GC Ready signal from GC1	Waits for the GC ready which had been uploaded to GC by Qtegra ISDS Software before starting the prescript. Qtegra receives signal from GC1.
GCIsoLinkII AS WaitForSignal GC2	Waits for GC Ready signal from GC2	Waits for the GC ready which had been uploaded to GC by Qtegra Main before starting the prescript. Qtegra receives signal from GC2.
GCIsoLinkII AS Next	Moves AS	Executes move to Qtegra sample list position

Table 7-9. Commands and actions for TriPlus RSH SMART for GC IsoLink II and for TriPlus RSH SMART for GasBench or PreCon (same SMART autosampler firmware) Important for CTC Pal 80 with Trace GC 1310 with PreCon application^a, continued

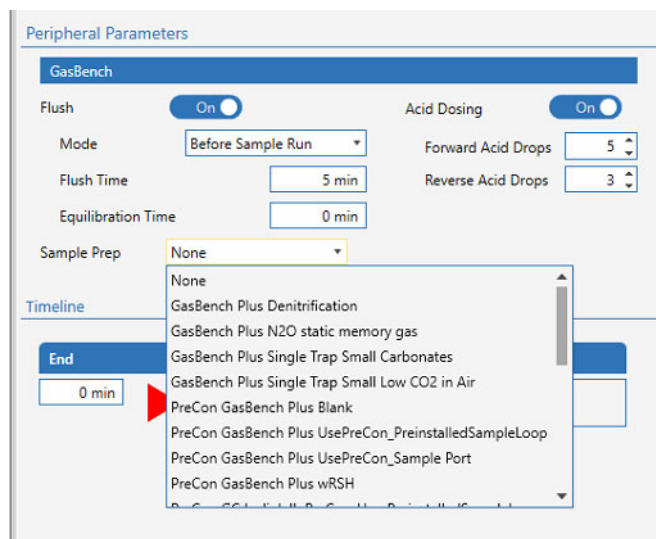
Command	Action / Unit	Comment
GCIsoLinkII AS Injected	GC Start signal send	Last command directly after an injection on the GC column
GCIsoLinkII AS Home	n/a	Executes autosampler home position move “GasBench” might change.
GCIsoLinkII WaitFor	GC Ready	Waits for Trace GC 1310 sample preparation and equilibration of the GC (seen on the GC control screen and programmed in the Trace GC 1310 GC method under ChromControl. Important for use of the CTC Pal 80 with Trace GC 1310 and PreCon applications. No communication cable is needed to operate the CTC Pal 80 with the Trace GC 1310.

^a used for the TraceGC 1310 operation with the GC IsoLink II using prescript

The scripts will be executed in Qtegra LabBook > Method Parameters under Peripheral Parameters > GasBench modes.



a



b

Labeled Components: a) None. No Script will be executed. b) A script will be executed. Qtegra will provide a set of prescript examples and all available objects, e.g., *GasBench Trap 1*. The same dropdown menu is available for the GC IsoLink II device with PreCon option. The dropdown menu shows the available scripts in C:\ProgramData\Thermo\Qtegra_Application Data\PluginData\TFS253Plus\Scripts\SamplePreparation.

Figure 7-50. Script execution drop down

NOTICE

Only the TriPlus RSH SMART for GasBench Plus, performance scripts for GC IsoLink II, and the CTC Pal 80 - A200S mode for PreCon applications with Trace 1310 and GC IsoLink II can be executed in Qtegra prescripts.

NOTICE

In the folder “Sample Preparation” (see above), two folders provide scripts - if copied in the main Sample Preparation folder location, they will be shown in the Sample Prep dropdown menu:

- a. Under “SamplePreparation”: all script commands (see above), which can be executed via LabBook Peripheral Parameters dropdown menu. An alias file can be used for transferring a command into a simple text information within a script. An example alias is being saved here with an A200S file containing an alias. The file extension is a .txt file with the extension .aliases.
- b. Extra folder under “Sample Preparation”: PreCon test scripts (with fast mode, to check the PreCon functionality). The freeze script and the inject script must be copied into the main folder “SamplePreparation” as well.
- c. Extra folder under “SamplePreparation”: “Performance Tests PreCon GC IsoLink II”

Measurement Procedures for Real Samples

This chapter describes the measurement procedures of the GasBench Plus device for various common sample types.

Contents

- [Safety Guidelines for Operation](#) on page 8-2
- [Referencing vs. VPDB](#) on page 8-2
- [Analyzing Dissolved Inorganic Carbon \(DIC\)](#) on page 8-3
- [Breath Gas Analysis](#) on page 8-6
- [Analyzing CO₂ in Atmospheric Concentrations](#) on page 8-7
- [Water Equilibration \(²H/¹H Equilibration\)](#) on page 8-12
- [¹⁸O Equilibration LabBook](#) on page 8-18
- [Operating the GasBench Plus Device with a ConFlo IV Interface](#) on page 8-27

Safety Guidelines for Operation

When operating the GasBench Plus system, pay attention to the following general safety guidelines.

WARNING

High Voltage. High voltages capable of causing an electric shock are used in the instrument. Do not remove protective covers from PCBs.

Before opening the GC housing, switch off the GasBench Plus at the right side panel and unplug the 230 V power supply cable.

CAUTION

Hot Surface. When the GC oven is operating (during a heating out period), the temperature of the heater located inside is above 100 °C. Touching the GC oven or the heater might cause severe burns. Stay away from the GC oven and the heater. Let them cool to ambient temperature before you work on them.

CAUTION

Hot Surface. When the thermostatted tray (“heated tray”) is operating, touching it might cause severe burns. Stay away from the thermostatted tray. Let it cool to ambient temperature before you work on them.

CAUTION

Hazardous Chemicals. Samples and solvents might contain toxic, carcinogenic, mutagenic, or corrosive/irritant chemicals. Avoid exposure to potentially harmful materials.

Always wear protective clothing, gloves, and safety glasses when you handle solvents or samples.

Contain waste streams and use proper ventilation. Refer to your supplier's Material Safety Data Sheet (MSDS) for proper handling of a particular compound.

Referencing vs. VPDB

All carbonate δ values must be referenced to the international standard VPDB (Vienna Pee Dee Belemnite), the successor of PDB as PDB is exhausted. However, VPDB with $\delta^{13}\text{C} = 0$ and $\delta^{18}\text{O} = 0$ as one would expect, does not exist. Instead, standards exist which are related to this virtual, that is unreal, definition.



Refer to “*Reference and intercomparison materials for stable isotopes of light elements*”. In: *IAEA-TECDOC-825, IAEA, ed., Vienna, 1995.*

Analyzing Dissolved Inorganic Carbon (DIC)

Dissolved Inorganic Carbon (DIC) is of large interest for global carbon cycle research, metabolic research and carbon flux studies of different water sources. The isotope ratio determination of DIC helps to identify and quantify processes within those different scientific areas.

Dissolved Inorganic Carbon (DIC) in Brief

The preparation of DIC samples for $^{13}\text{C}/^{12}\text{C}$ isotope ratios with the GasBench Plus device is explained briefly in this section. The preparation before the phosphoric acid reaction is performed in different ways. Each of these ways have different advantages and disadvantages:

1. Field collection of DIC samples and poisoning in the field.

Store the samples in destined sample vials. Add a sample to a preflushed sample vial containing phosphoric acid in the laboratory. See [Figure 8-1](#).

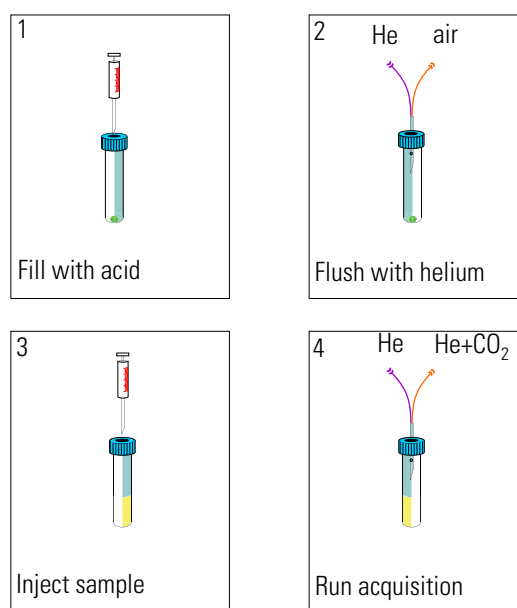


Figure 8-1. Sample preparation for DIC measurement

2. Field collection of DIC samples in helium-preflushed sample vials adding phosphoric acid to the samples in the field

The disadvantage of the first method is the poisoning with HgCl_2 . An advantage is the stopping of any bacterial degradation changing the original isotopic $^{13}\text{C}/^{12}\text{C}$ ratio of DIC.

The second approach has the advantage of clean analysis, but implies the possibility of biological degradation, if the analysis in the laboratory is not performed fast enough.

When real samples are collected, they must be poisoned using a saturated HgCl_2 solution to stop all biological activity.

WARNING

Poisonous Compound. Strictly avoid any exposure to the severely toxic HgCl_2 . Always wear protective gloves. Refer to your supplier's Material Safety Data Sheet (MSDS) for proper handling.

In the following section, common pitfalls with the preparation of DIC samples before the analysis with the GasBench Plus device are described.

- Almost no signal occurs on m/z 46 between the CO_2 peaks.
- Decreasing peak height indicates proper transport of sample/helium mixture.

NOTICE

When filling a number of tubes from the same water standard, do not fill them from a sealed vessel with septum. A negative pressure is created that could cause fractionation.

When filling real samples, use a new syringe for each sample. When running standards for acceptance tests, a single syringe is sufficient. Take care to allow any ocean water to remain on the inside of the septum.

Wipe the outside of the needle prior to puncturing the septum. When filling the flushed vial with ocean water, do not puncture the septum in the center, but close to the edge.

If samples are stored for a longer period (that is for several months), only use large sample amounts (above 100 mL). This avoids isotopic fractionation due to evaporation. Carefully close the bottles using Parafilm™. Avoid headspaces filled with air and store them in a cooler at 4 °C.

To maintain water as a working standard stable in isotopic composition over a longer time, it has been proven useful to store them in large canisters. Use at least a 50 L stainless steel barrel and vent it using only dry inert gas, N_2 , for example. It is not dissolved in the water and thus the CO_2 content will not change.

❖ **To perform a DIC measurement (see Figure 8-1)**

1. Fill some drops (about 30 μL) of 45% to 98% H_3PO_4 into an empty vial. See “Preparing Phosphoric Acid” on page 9-13 for instructions on how to prepare phosphoric acid.

For DIC measurements, smaller concentrations of H_3PO_4 can be used, simplifying the addition of H_3PO_4 into the vials.

2. Close the vial and place it in the tray.
3. Exchange the headspace (that is via the needle, helium streams in and replaces the gas in the vial, which in turn streams out of it).

- Inject the sample (about 700 μL) through the septum into the closed vial using a syringe. CO_2 will be released from these different origins and will then be mixed with the helium in the headspace.

NOTICE

A syringe must be used to prevent the sample from contacting and exchanging with ambient air.

- Allow 18 h to equilibrate.
- The sample is measured.

Referencing Strategies for DIC

For referencing the $^{13}\text{C}/^{12}\text{C}$ isotope ratios of working standards and samples to the international VPDB scale, different analytical procedures exist:

- Analysis of a solid carbonate standard (NBS 19 or working standard carbonate material) prior to analysis of DIC samples. For the working standard, baking powder (NaHCO_3 , pro analysis) can be used.
- Running a working standard as a DIC standard (dissolved in deionized water; remove CO_2 via ultrasonification). The working standard is prepared in different concentrations to make an area correction of the $^{13}\text{C}/^{12}\text{C}$ ratios of samples possible. See “[Analyzing Carbonates](#)” on [page 9-38](#). The concentration must reflect the DIC concentration range of the samples.

Tip The liquid NaHCO_3 (NaHCO_3 solved in water) is prepared before each analysis to avoid contamination of the standard NaHCO_3 with CO_2 coming from atmospheric air.

For different bicarbonates, different $^{13}\text{C}/^{12}\text{C}$ acid fractionation factors exist.

NOTICE

The DIC measurement with Qtegra is exactly the same methodology, except for

- using different standardization strategies
 - NBS 19 as a solid normalization standard
 - Define a in water dissolved bicarbonate as liquid standard. Define the bicarbonate as solid versus NBS 19
 - Run both the solid and the liquid bicarbonate against NBS 19 in one sequence and reevaluate it afterwards.
 - Use bicarbonate quality control standard.

Breath Gas Analysis

Isotopic analysis of breath CO_2 has important applications in physiology, ecology and medicine. $^{13}\text{C}/^{12}\text{C}$ of CO_2 in breath (breath gas analysis) helps identifying *Helicobacter pylori* and is used in metabolic research. *Helicobacter pylori* is one of the major elicitors to cause stomach ulcer and stomach cancer.

To detect the occurrence of *Helicobacter pylori*, ^{13}C -labeled urea is given to the patient. Demethylation of the bacteria is detected in the ^{13}C -enriched CO_2 of breath. The $\delta^{18}\text{O}$ of CO_2 reflects $\delta^{18}\text{O}$ values of body water. It matches the $\delta^{18}\text{O}$ of drinking water or from food sources (fruits with high water content).

Breath Gas Analysis in Brief

To perform breath gas analysis, the sample loop of the Valco valve must be replaced by a 10 μL volume. See [“Changing the Loop Size”](#) on [page 3-20](#).

❖ **To perform a breath gas analysis**

1. Fill empty sample vials with breath using a straw.
2. Close them with a fresh cap and septum. Place them in the sample tray.
3. You can modify the Qtegra method by using the blank mode in the LabBook continuous flow method as described in the generic Qtegra Workflow section.
4. A sequence of its own is not necessary. Instead, use the LabBook as described under equilibration sequence to perform a measurement.

Results of Breath Gas Analysis

For breath gas analysis, the GasBench Plus chromatogram looks different from a normal chromatogram for equilibration or carbonate measurements. Blanking is used, and therefore N_2O gas (production in the ion source coming from N_2 in air) or breath is removed.

High water contents in breath may result in less precision of $\delta^{18}\text{O}$ in CO_2 results, if water is not quantitatively removed. Adding a magnesium perchlorate tube helps to quantitatively remove breath water.

NOTICE

For breath gas analysis, the blanking mode must be applied as described under [“Blanking modes for GasBench Plus with and without ConFlo IV”](#) on [page 6-6](#).

Analyzing CO₂ in Atmospheric Concentrations

The analysis of $\delta^{13}\text{C}$ of CO₂ in air is to measure different flow rates of CO₂ coming from different carbon sources in the environment. It is largely used for keeling plots and for example for eddy correlation studies.

Editing a LabBook Continuous Flow Method

To measure CO₂ in atmospheric concentrations, create a LabBook method. Similar to breath gas analysis, blanking is used to avoid production of N₂O. See [“Breath Gas Analysis”](#) on page 8-6.

Tip The method differs only with respect to the Time End list used for carbonate LabBooks.

Sample Pulsing for a Single Blanking before each Sample Peak without Trapping

Similar to breath gas analysis, blanking is used to avoid production of N₂O. See [“Breath Gas Analysis”](#) on page 8-6.

Tip There are differences comparing the Sample Pulsing used for carbonate measurements.

When you expect an air peak in the chromatogram, it must be masked out. This is achieved by setting the split to dilution position on time or the ConFlo IV maximum dilution (HF capillary inserted, all sample dilution valves open during blanking interval), see [“Qtegra LabBooks”](#) on page 7-26 and [“Using the Blanking Mode in Qtegra with ConFlo IV”](#) on page 6-8.

Setting the split time off in the Split-In column will stop dilution. The split will be pushed in again. This ensures that most of the sample can be measured. Overall, this change between on and off positions takes place ten times.

For ConFlo IV blanking, see [“Operating the GasBench Plus Device with a ConFlo IV Interface”](#) on page 8-27.

1. Use the blanking described at the blanking modes.
2. Use a LabBook for blanking events, single peak with liquid nitrogen trapping for low concentration measurements or multiple blanking mode with larger sample amount measurement.

For blanking modes with single trap injection, see [“Blanking Mode for Single Peaks Applied with a Single and a Dual Trap Measurement”](#) on page 6-11.

Qtegra Setup for Single Blanking of Air

After the prescript injection and preparation with the Script *GasBench Plus Single Trap Small Low CO₂ in Air.txt* file.

Single scripts must be executed as described at [“Performing Sample Measurements with a Single Trap Sample Gas Injection Using Qtegra”](#) on page 6-20.

Run blanking modes with and without ConFlo IV with the GasBench Plus (using the Blanking Modes in Qtegra).

Qtegra Setup for Multiple Blanking of Air CO₂ Runs

For multiple peak analysis (sample pulsing greater than <1>) blanking of air (breath gas), the multiple blanking will be performed. No prescript is in use.

Analyzing Small Carbonate Samples

Small carbonate analysis of carbonates can follow two different workflow principles:

1. Analysis with sample pulsing by Valco injection of multiple CO₂ gas samples.
2. Analysis of very small carbonate concentrations by using single trap and CO₂ injection of CO₂ from the gas reaction.

Analysis With Sample Pulsing by Valco Injection of Multiple CO₂ Gas Samples

The analysis with sample pulsing by Valco injection follows the principle of the standard carbonate analysis. The only process difference is that small sample vials are used in the thermostatted sample tray, e.g., LABCOTM 4.5 ml vials down to custom-made 2 ml vial, following the procedure by Fiebig et al. 2005, Breitenbach and Bernasconi 2011.

Analysis of Very Small Carbonate Concentrations by Using the Single Trap Down to 3 µg Sample Amount

Small carbonate sample amounts in the µg scale are analyzed with single trapping strategy with the GasBench using single trapping. Since the head space is not influenced due to no interfering gases before the sample peak using pure helium, no chromatogram “clean up” by blanking is needed. Very small sample analysis is possible by the method applied by Vonhof et. al 2021.

The following main steps are required:

1. Single trap use.
2. Prescripting with the single trap and workflow principle for preparation of the single trap. See “[Blanking Mode for Single Peaks Applied with a Single and a Dual Trap Measurement](#)” on page 6-11.
3. Optional: blank determination and isotope linearity determination dependent on sample amount. In very small amount sample analysis required.

Follow the standard prescript and single trap template given by the standard Qtegra installation (file name: *GasBench Plus Single Trap Small Carbonates.txt*).

The LabBook method parameters timeline will be set as in [Figure 8-2](#). For analysis with a short time difference between flushing and a short distance of the measurement start after acid addition, the flushing can

be set during sampling. It uses the flush valve command in a prescript if the shortest time difference should be achieved between flushing and measurement.

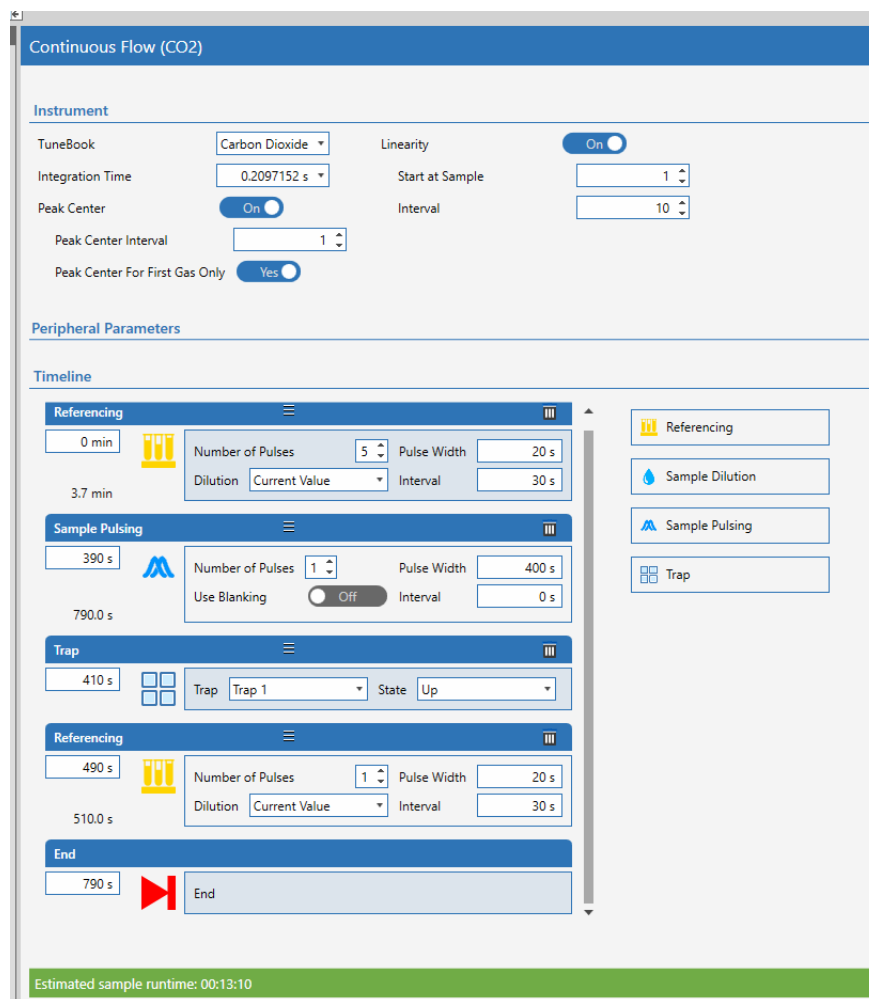


Figure 8-2. LabBook method parameters for the peripheral preparation (purging and acid addition) and timeline processing with the single trap without blanking. Under Peripheral Parameters use the “GasBench Plus Single Trap Small Carbonates” script for GasBench series measurements.

Reference



Sebastian F. M. Breitenbach and Stefano M. Bernasconi (2011) Carbon and oxygen isotope analysis of small carbonate samples (20 to 100 µg) with a GasBench II preparation device. *Rapid Commun. Mass Spectrom.*, 25, 1910-1914.



Jens Fiebig, Bernd R. Schöne and Wolfgang Oschmann (2005) High-precision oxygen and carbon isotope analysis of very small (10-30 µg) amounts of carbonates using continuous flow isotope ratio mass spectrometry. *Rapid Commun. Mass Spectrom.*, 19: 2355-2358.



Hubert B. Vonhof, Stefan de Graaf, Howard J. Spero, Ralf Schiebel, Suzan J.A. Verdegaal, Brett Metcalfe, Gerald H. Haug (2020). High-precision stable isotope analysis of <5 μg CaCO₃ samples by continuous-flow mass spectrometry. *Rapid Commun Mass Spectrom.*, 34:e8878.

Analyzing Dissolved Organic Carbon Samples (DOC)

Dissolved organic carbon can be analyzed in an analog strategy to the GasBench carbonate analysis except for a peroxydisulfate reaction (chemical oxidation) to produce CO₂ from organic-bound carbon together with the thermostatted tray to increase reaction activation (Lang et al. 2012).

Reference



Susan Q. Lang*, Stefano M. Bernasconi and Gretchen L. Früh-Green (2012). Stable isotope analysis of organic carbon in small (μg C) samples and dissolved organic matter using a GasBench preparation device. *Rapid Commun. Mass Spectrom.*, 26: 9-16.

Water Equilibration ($^2\text{H}/^1\text{H}$ Equilibration)

The hydrogen equilibration is also called HD (hydrogen-deuterium) equilibration.

❖ To perform a hydrogen equilibration

Keep the tray at room temperature.

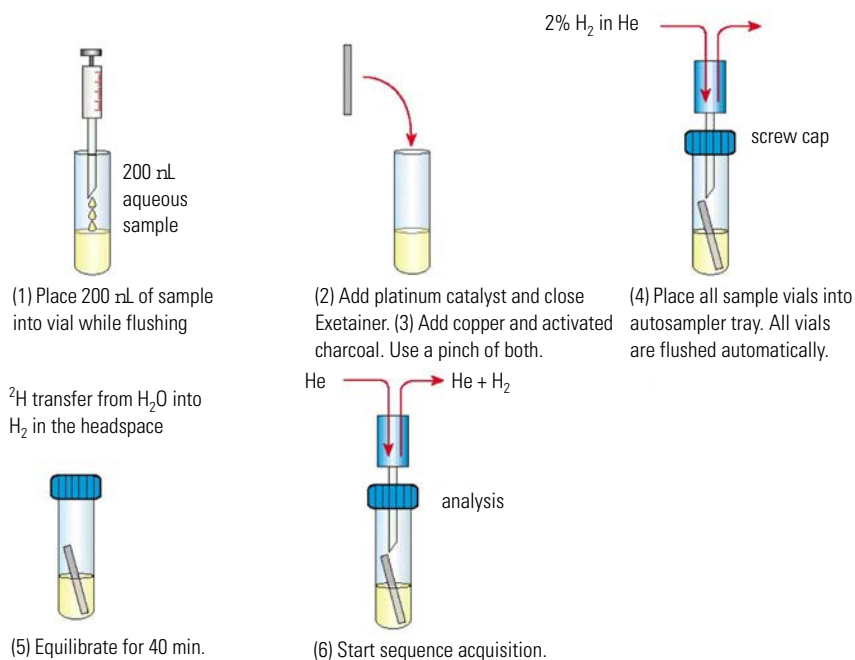


Figure 8-3. Sample preparation for $^2\text{H}/^1\text{H}$ equilibration

1. Insert the sample into vials and insert the catalytic platinum sticks.

This avoids formation of H_2S and to removes adsorbed water molecules on dissolved organic carbon (DOC). Activated charcoal adsorbs dissolved organic carbon (DOC).

See “[Catalyst for Hydrogen Equilibration](#)” on [page 11-17](#) for a description of the catalyst.

See “[Cleaning the Platinum Sticks](#)” on [page 11-17](#) for information about cleaning the platinum sticks.

2. Flush all samples with 1 to 4% H_2 in helium. Run a Qtegra LabBook with Flushing during measurement by enabling the internal flushing mode under peripheral parameters.

The Qtegra LabBook is very similar to the ^{18}O equilibration LabBook, but uses a H_2 TuneBook, the need to determine the H_3 factor at least every day, determine its stability after peripheral switch or TuneBook with different gas configurations. Use the appropriate standards for D/H measurement and reference gas.

The equilibration is finished within 40 min. It is not necessary to wait additional time.

NOTICE

Adjust the Sample List and position to the appropriate timing.

3. Exchange the rinsing needle with the sampling needle. There are various needle sets using the same needle type: one set of needles exists for flushing (that is rinsing needle) and another one for measuring (that is sampling needle). Different strategies are offered by the description under the GasBench Tools.

The rinsing needle is used to rinse the vials: the recurrent capillary must be broken off at 20 cm to let the rinsing agent pass into ambient air. In case of the sampling needle, the recurrent capillary leads into the GasBench Plus device.

4. Run a LabBook.

Tip Adding activated charcoal and copper pieces increases the accuracy of the results. Salt water needs an increased equilibration time.

Figure 8-4 shows standards for water equilibration with H_2/H_1 . H_3 factor does only run automatically with ConFlo IV. For GasBench without ConFlo IV, the H_3 factor must be determined daily under the instrument and an active H_2 TuneBook. Qtegra requests an automatic H_3 factor determination, if the determination is too old.

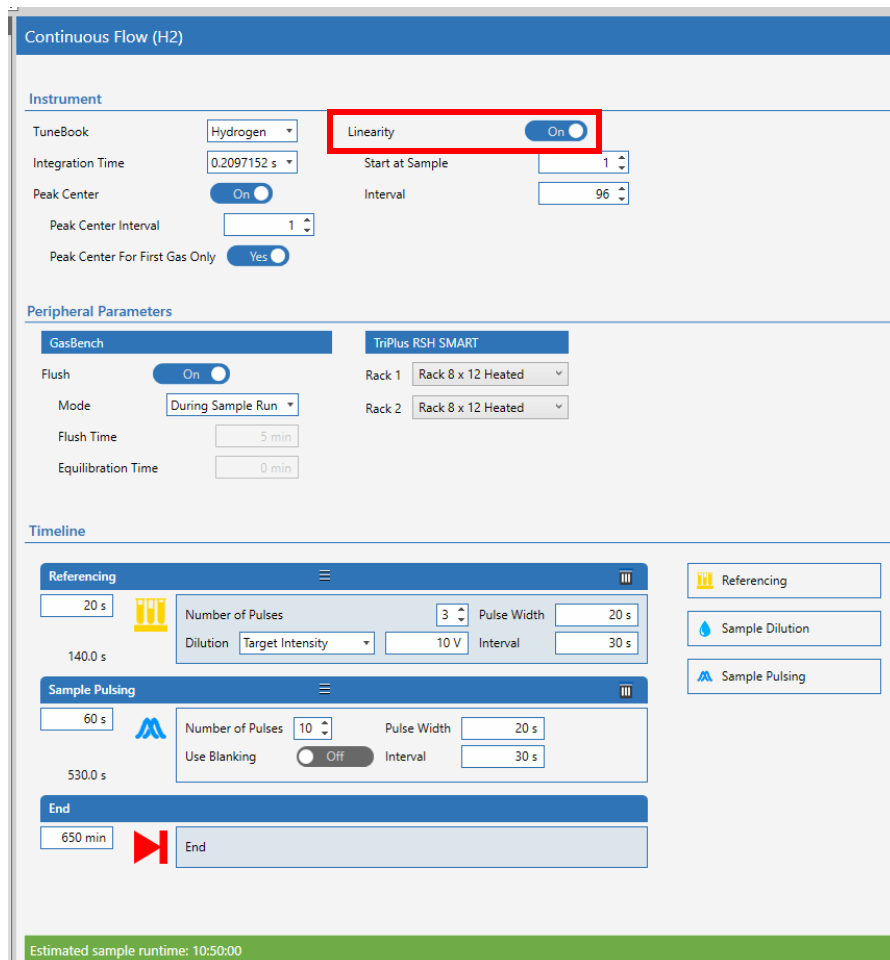


Figure 8-4. Standards for water equilibration with H_2/H_1 . H_3 factor does only run automatically with ConFlo IV

Figure 8-5 shows a Qtegra LabBook for water equilibration H2/1H equilibration: LabBook peripheral set with a GasBench tool having a flush needle inserted at the right position and a measurement needle at the left position.

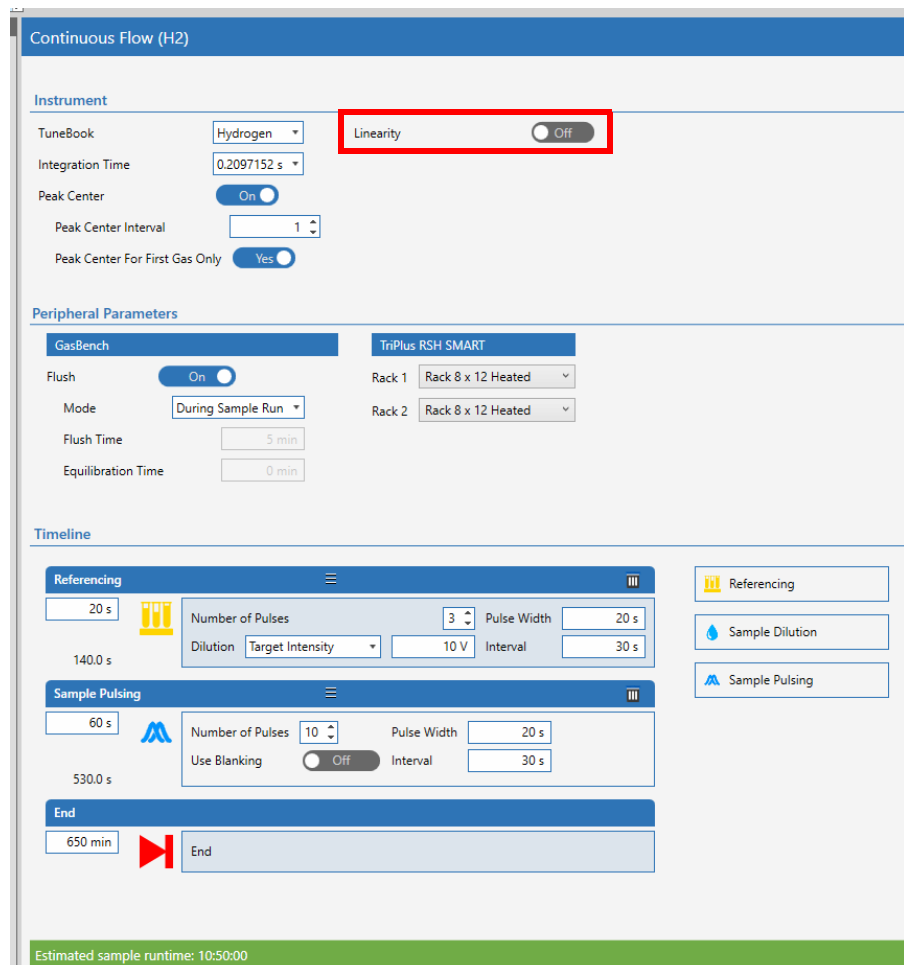


Figure 8-5. Qtegra LabBook for water equilibration H2/1H

Adjusting Reference Signal Height for GasBench Plus without ConFlo IV and H3 Factor Determination

To achieve optimal performance it must be possible to set the reference signal height to 8 V. Therefore, it is necessary to cut the flow restricting capillary by 30% from its original length.

Tip Ask your Thermo Fisher Scientific field service engineer upon installation of the GasBench Plus device to do this.



Refer to Nelson, S. T.: *A simple, practical methodology for routine VSMOW/SLAP normalization of water samples analyzed by continuous flow methods. Rapid Communications in Mass Spectrometry* 14:1044-1046 (2000). John Wiley & Sons Ltd.

Qtegra ISDS Software does not allow to automatically adjust the signal height without ConFlo IV.

Determining a H_3 Factor under Qtegra Instrument Control

Figure 8-6 shows a H_3 factor determination with GasBench without ConFlo IV using H_2 reference gas at the Qtegra Instrument Control Linearity Correction.

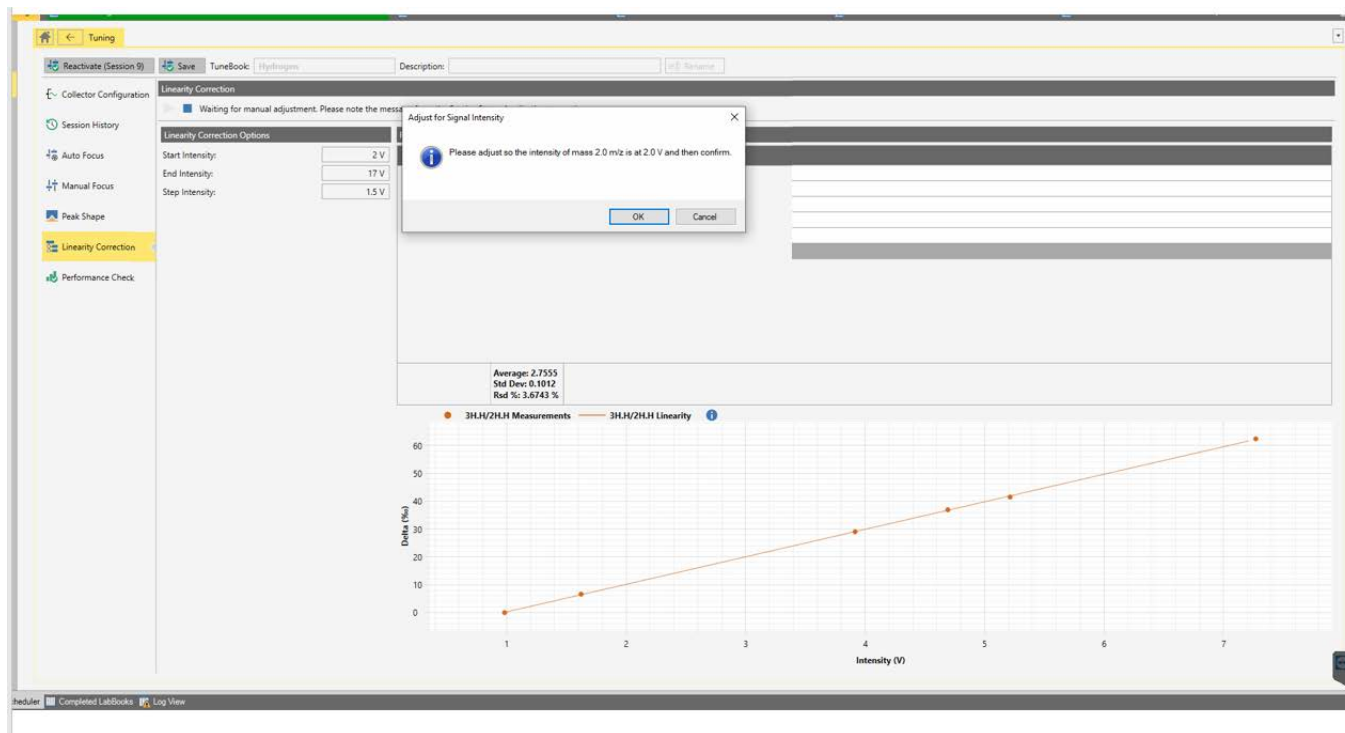


Figure 8-6. H_3 factor determination with GasBench without ConFlo IV using H_2 reference gas at the Qtegra Instrument Control Linearity Correction. Open a Hydrogen TuneBook and the Linearity Correction Tap. Click Run to activate the process for H_3 factor determination (see text for description of the manual step calibration).

❖ To determine a H_3 factor at Qtegra Instrument Control

1. Open an active H_2 TuneBook.

At “Linearity correction”, the H_3 factor will be determined (“Linearity Correction Options”). Use 1 V to 8 V in 1 V steps.

2. Before determining the steps width in bar or psi (for example 8 steps for the example above) vs. the voltage on the H_2 reference gas pressure gauge, use the Qtegra Dashboard.
3. Define the step width needed for the H_3 factor determination. For example 1 V to 8 V in 1 V steps would require 8 steps. For example, 1 V mass 2- H_2 with 0.5 bar and 2.5 bar with 8 V would require a bar step width of approximately 0.25 bar increase per voltage step. Check and mark each increment on the pressure gauge.
4. Go back to the Linearity correction. Pull the pressure gauge back to 0.5 bar and click **Run**.

5. Qtegra prompts asking to prepare the pressure gauge to set at the requested voltage start value.
6. Qtegra determines the signal for each step, move the pressure gauge in the predetermined increments (here: 0.25 bar).
7. Qtegra automatically checks whether the edited step width could be reached before the next measurement step starts.
8. After finishing the determination, Qtegra automatically determines the H₃ factor and saves it in the H₃ factor history. For H₃ factor determination, refer to the *DELTA Q Operating Manual*.

Sample Amount Considerations for Both Water Equilibration Types

In this section, the sample amount needed for both types of water equilibration is estimated via an approximate calculation. It helps to decide whether a mass balance calculation needs to be performed for a particular sample or not.

Depending on how much gas of a particular ϵ value has been filled into the headspace and how much water has been added to the sample (δ value unknown), a final δ value between these two original δ values results.

Tip Remember that 1 mol of water equals 18 mL and 1 mol of an ideal gas commensurates to 22.4 L.

One sample vial contains 12 mL, that is $12/22400$ mol $\approx 5.357 \times 10^{-4}$ mol of an ideal gas. We do not use pure CO₂, but 0.5% CO₂ in He and consider this mixture to be an ideal gas. Therefore, one sample vial contains $(12/22400) \times 0.005$ mol CO₂ $\approx 2.679 \times 10^{-6}$ mol CO₂.

Regarding the water: 1 mL of water equals 1/18 mol of water. Using 1 mL of sample in the sample vial yields 10000 times more oxygen atoms in the water phase compared to the gas phase.

As a good estimation, we can therefore assume the isotope value of the gas to be equal to the initial isotope value of the sample. This means, the isotope value will not shift, but the gas will indeed take the original value of the sample. Thus, using 1 mL (or 500 μ L or 200 μ L) of sample, no mass balance calculation is required.

¹⁸O Equilibration LabBook

Water Equilibration (¹⁸O/¹⁶O Equilibration)

❖ To perform an oxygen equilibration

1. Fill the sample into the clean open Exetainer vial (12 mL) by using an adjustable pipette with disposable pipette tips. It is not necessary to pierce the septum using the needle. The filling volume should be 0.5 mL.
2. Close the vial. Place it into the TriPlus RSH autosampler tray.
3. The flushing gas is a mixture of He and CO₂, that usually has already been properly mixed and filled into a He/CO₂ tank. Open the He/CO₂ tank connected to the flush gas input.
4. Increase the pressure to result in a flow of the He/CO₂ mixture of approximately 100–150 mL/min at the vent of the flush needle. When using a new gas mixture, wait for 10–15 minutes until all the lines are completely filled with this new mixture, that means, until it is ensured that the former gas mixture has been completely exchanged with the new one.

Tip The flush needle is synonymously called flushing needle, rinsing needle, or filling needle. Accordingly, one speaks of flush valve and flush connection.

5. Make sure that the flush needle is properly mounted to the autosampler tool.
6. Depending on your hardware, use the appropriate Qtegra workflow as described below with flush sequence or the dual needle flush sequence to fill the vials automatically. By default, the LabBook is set up to flush each vial with a helium stream of 100 mL for 5 minutes, see [“LabBook for the ¹⁸O Isotopic Application Workflow”](#) on page 8-20.
7. Close the He/CO₂ mixture tank when the flush sequence is finished.
8. Qtegra waits automatically until the LabBook with equilibration has been enabled. Wait for approximately 18 hours for proper equilibration.

9. Start the measurement sequence.

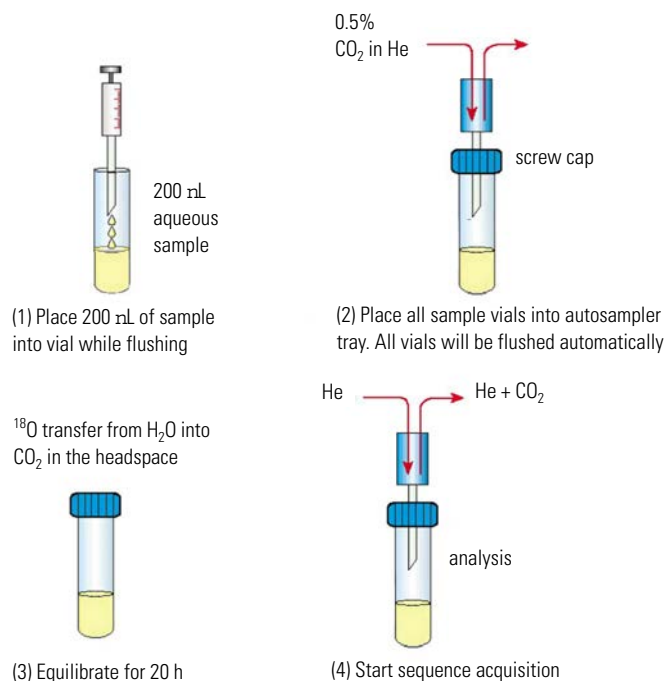


Figure 8-7. Sample preparation for ¹⁸O/¹⁶O equilibration

Tip Salt water needs to be equilibrated for a maximum of 2-5 days to improve accuracy of sample results. Flushing can be done externally on the GasBench Qtegra Dashboard.

Temperature Control of the Sample Tray

For high precision ¹⁸O/¹⁶O equilibration, the temperature of the sample tray needs to be stabilized. Two operation modes are available:

- Passive tray at room temperature, that is 21 °C or below

The thermal mass of the cast aluminum tray and its isolation allow keeping the temperature control of the tray deactivated. Only long-term drifts in tray temperature occur within a certain time interval. Placing the reference samples allows correcting for possible temperature drifts (one reference sample for six unknown samples, for example).

- Active temperature control at 21 °C or below

Make sure that room temperature is approximately 2-5 °C below the set tray temperature. Check the temperature stability over several hours. The controller readout may not alter by more than 0.1 °C.

LabBook for the ¹⁸O Isotopic Application Workflow

There are different ways to run a stable isotopic ratio of oxygen in water ($\delta^{18}\text{O} - \text{H}_2\text{O}$) workflow.

Flush with Single or Double Needle

CO₂ in Helium purge in an extra flush sequence under diagnosis, i.e., go from sample 1-96 with either a single or a double needle installed in the tool.

LabBook Continuous Flow Peripheral Parameter Sets for Certain Process Preparations

Certain preparation processes can be done automatically with Qtegra within one LabBook under the peripheral parameters (see [Figure 9-47](#)). Tool changes will be switched or requested automatically.

1. The whole preparation and measurement (see [Figure 9-48](#)) is done in one LabBook with
 - a. Flush of all samples
 - b. Acid addition during the sample measurement (drop numbers defined under acid pump in the GasBench dashboard instrument)
 - c. Measurement of samples by sample pulsing and the same time referencing.
 - d. Measurement with sample if the sample response is too high (see [Figure 9-47](#)), e.g. for soil or DIC samples with an unknown carbonate concentration.

Qtegra will take the information from the installed Tool set up under the RSH settings in the LabBook and does a flush processing of all sample before a sample list. Indicated by an empty chromatogram screen and a green arrow without movement over the next sample number in the LabBook.

Deviations for the process if sample have been processed according to the flush (see 1 & 2)

Disable the flush mode (see [Figure 8-8](#)).

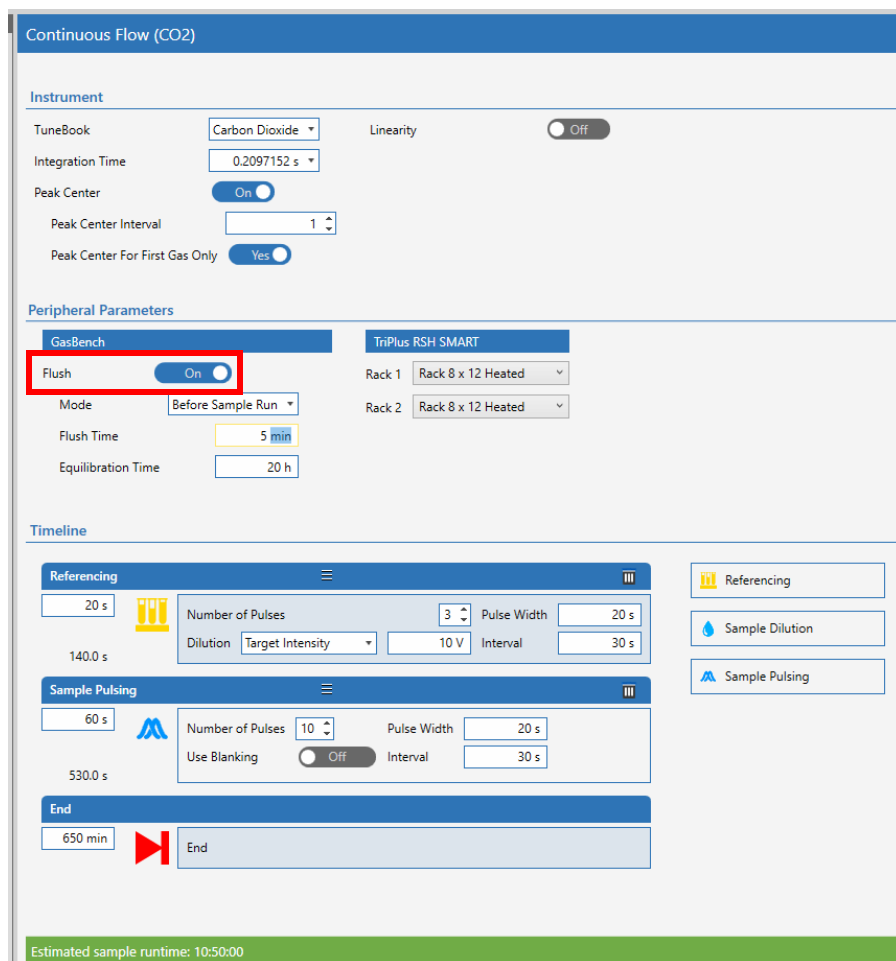


Figure 8-8. Peripheral preparation for ¹⁸O in water, with flushing
 Flush mode is disabled (see [Figure 8-9](#)).

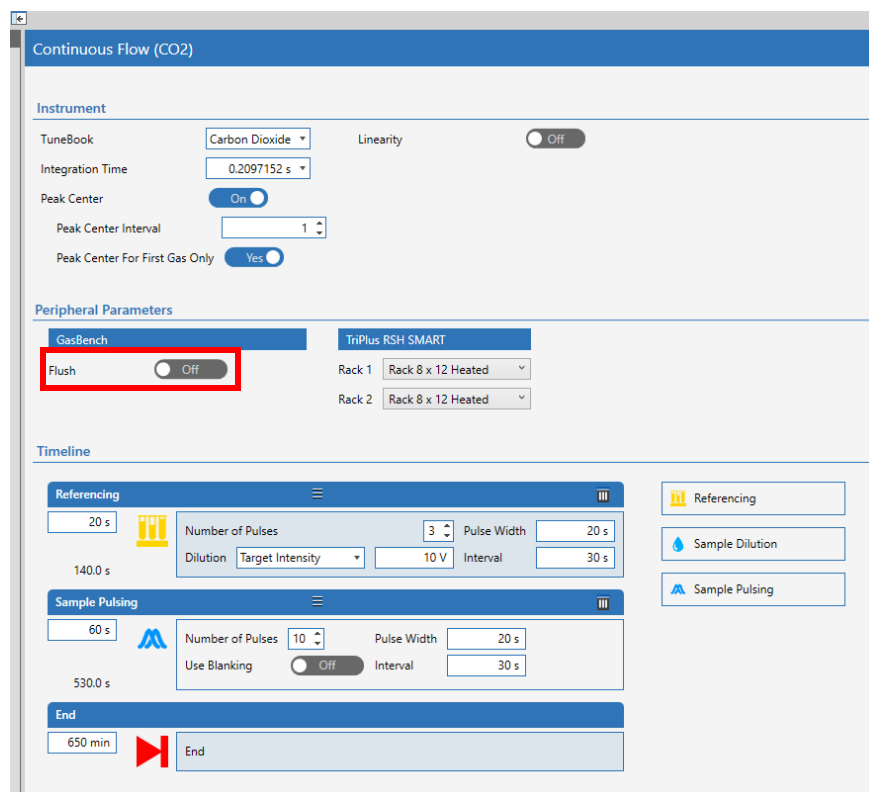


Figure 8-9. ¹⁸O in water workflow, without flushing

Setting up Water Isotope Standards and Delta Standard

Refer to the *DELTA Q Operating Manual* for the generic standardization procedure in Qtegra). Set up the standardization including a set of usable standards (see Figure 9-50 and including calibration gas standard (see Figure 9-51) The calibration standard will be used to calibrate the sample peaks to this standard for internal normalization.

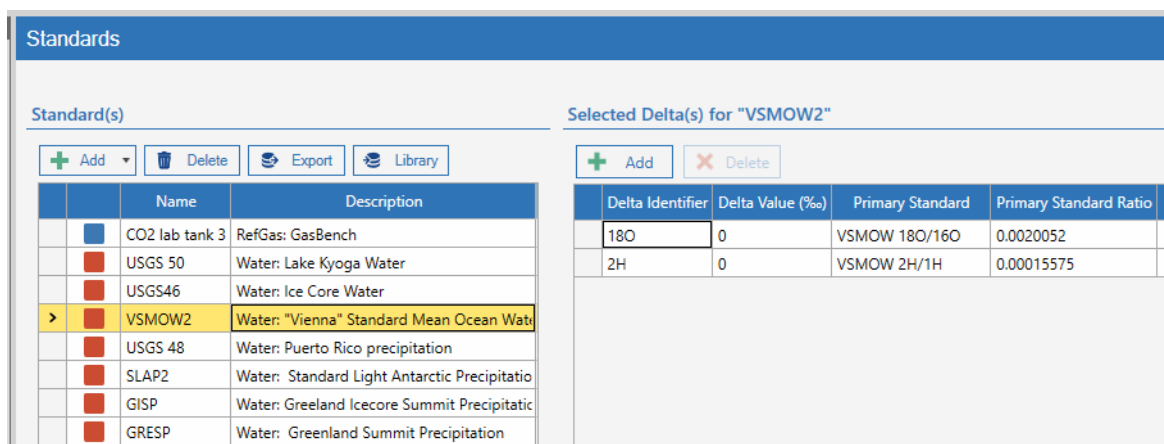


Figure 8-10. International ¹⁸O in water BSIA standards (red color) including a reference gas (calibration gas, blue color)

Figure 8-11. Delta calibration gas used for calibrating the sample peaks against VSMOW as defined in the calibration gas standard (see [Figure 9-51](#))

For the sample list, a standard for international standardization can be used, e.g. USGS 50 (close to VSMOW values). The standard is only used as reference to the defined value (e.g. USGS 50 vs. VSMOW, see [Figure 9-55](#)) if defined in the Reference column. If a different scale, e.g. USGS 48 scale (similar to GISP values) shall be used, the same standard can be recalculated against the VSMOW scale but with different values and assignments in the BSIA standard list (see *DELTA Q Operating Manual*).

Label	Status	Comment	Analysis No.	Action	Rack	Val	Evaluate	Sample Type	Reference
1	USGS 50	18O Equilibration	N/A	Measure	1	1	✓	Delta Standard (BSIA)	USGS 50
2	USGS 50	18O Equilibration	N/A	Measure	1	2	✓	Delta Standard (BSIA)	USGS 50
3	USGS 50	18O Equilibration	N/A	Measure	1	3	✓	Delta Standard (BSIA)	USGS 50
4	Di 1	18O Equilibration	N/A	Measure	1	4	✓	Unknown	
5	Di 1	18O Equilibration	N/A	Measure	1	5	✓	Unknown	
6	Di 1	18O Equilibration	N/A	Measure	1	6	✓	Unknown	
7	Di 2	18O Equilibration	N/A	Measure	1	7	✓	Unknown	
8	Di 2	18O Equilibration	N/A	Measure	1	8	✓	Unknown	
9	Di 2	18O Equilibration	N/A	Measure	1	9	✓	Unknown	
10	Di 3	18O Equilibration	N/A	Measure	1	10	✓	Unknown	
11	Di 3	18O Equilibration	N/A	Measure	1	11	✓	Unknown	
12	Di 3	18O Equilibration	N/A	Measure	1	12	✓	Unknown	
13	Di 4	18O Equilibration	N/A	Measure	1	13	✓	Unknown	
14	Di 4	18O Equilibration	N/A	Measure	1	14	✓	Unknown	
15	Di 4	18O Equilibration	N/A	Measure	1	15	✓	Unknown	
16	USGS 48	18O Equilibration	N/A	Measure	1	16	✓	QC Standard	USGS 48
17	USGS 48	18O Equilibration	N/A	Measure	1	17	✓	QC Standard	USGS 48
18	USGS 48	18O Equilibration	N/A	Measure	1	18	✓	QC Standard	USGS 48
19	Di 5	18O Equilibration	N/A	Measure	1	19	✓	Unknown	
20	Di 5	18O Equilibration	N/A	Measure	1	20	✓	Unknown	
21	Di 5	18O Equilibration	N/A	Measure	1	21	✓	Unknown	
22	USGS 46	18O Equilibration	N/A	Measure	1	22	✓	Delta Standard (BSIA)	USGS46
23	USGS 46	18O Equilibration	N/A	Measure	1	23	✓	Delta Standard (BSIA)	USGS46
24	USGS 46	18O Equilibration	N/A	Measure	1	24	✓	Delta Standard (BSIA)	USGS46
25	Di 6	18O Equilibration	N/A	Measure	1	25	✓	Unknown	
26	Di 6	18O Equilibration	N/A	Measure	1	26	✓	Unknown	
27	Di 6	<Comment>	N/A	Measure	1	27	✓	Unknown	
28	USGS 50	<Comment>	N/A	Measure	1	28	✓	Delta Standard (BSIA)	USGS 50
29	USGS 50	<Comment>	N/A	Measure	1	29	✓	Delta Standard (BSIA)	USGS 50
30	USGS 50	<Comment>	N/A	Measure	1	30	✓	Delta Standard (BSIA)	USGS 50
31	USGS 46	<Comment>	N/A	Measure	1	31	✓	Delta Standard (BSIA)	USGS46
32	USGS 46	<Comment>	N/A	Measure	1	32	✓	Delta Standard (BSIA)	USGS46
33	USGS 46	<Comment>	N/A	Measure	1	33	✓	Delta Standard (BSIA)	USGS46
34	USGS 48	<Comment>	N/A	Measure	1	34	✓	QC Standard	USGS 48
35	USGS 48	<Comment>	N/A	Measure	1	35	✓	QC Standard	USGS 48
36	USGS 48	<Comment>	N/A	Measure	1	36	✓	QC Standard	USGS 48

Figure 8-12. Sample list with international normalization, samples and slope correction standards each in triplicates

A Quality control standard can be run within the sample list. The QC limits will be applied as defined or edited, see “Method Parameters - Standards and Delta Calculation” on page 7-37.

Setting up a ¹⁸O in Water Equilibration Sample List

In the water equilibration sample list standards, a) normalization standard vs. VSMOW scale and samples are defined. A quality control standard can also be included and defined as quality control standard (see Figure 9-55), e.g. USGS 46.

NOTICE

Qtegra will automatically define the flush mode, i.e., either single needle or double needle flush. Consequently, by defining the positions Qtegra will automatically run the flush as the needle mode is defined in the TriPlus RSH settings under the Dashboard. Flushing will be defined by enabling flushing in the continuous flow method.

Label	Status	Comment	Action	Rack	Vial	Evaluate	Sample Type	Reference
DI 1	●	<Comment>	Measure	1	17	✓	Unknown	
DI 1	●	<Comment>	Measure	1	18	✓	Unknown	
DI 1	●	<Comment>	Measure	1	19	✓	Unknown	
USGS 50	●	<Comment>	Measure	1	20	✓	Delta Standard	USGS 50
USGS 50	●	<Comment>	Measure	1	21	✓	Delta Standard	USGS 50
USGS 50	●	<Comment>	Measure	1	22	✓	Delta Standard	USGS 50
USGS 46	●	<Comment>	Measure	1	38	✓	Delta Standard	USGS 46
USGS 46	●	<Comment>	Measure	1	39	✓	Delta Standard	USGS 46
USGS 46	●	<Comment>	Measure	1	40	✓	Delta Standard	USGS 46
DI 2	●	<Comment>	Measure	1	23	✓	Unknown	
DI 2	●	<Comment>	Measure	1	24	✓	Unknown	
DI 2	●	<Comment>	Measure	1	25	✓	Unknown	
DI 3	●	<Comment>	Measure	1	26	✓	Unknown	
DI 3	●	<Comment>	Measure	1	27	✓	Unknown	
DI 3	●	<Comment>	Measure	1	28	✓	Unknown	
DI 4	●	<Comment>	Measure	1	29	✓	Unknown	
DI 4	●	<Comment>	Measure	1	30	✓	Unknown	
DI 4	●	<Comment>	Measure	1	31	✓	Unknown	
USGS48	●	<Comment>	Measure	1	32	✓	Unknown	
USGS48	●	<Comment>	Measure	1	33	✓	Unknown	
USGS48	●	<Comment>	Measure	1	34	✓	Unknown	
DI 5	●	<Comment>	Measure	1	35	✓	Unknown	
DI 5	●	<Comment>	Measure	1	36	✓	Unknown	
DI 5	●	<Comment>	Measure	1	37	✓	Unknown	
DI 6	●	<Comment>	Measure	1	41	✓	Unknown	
DI 6	●	<Comment>	Measure	1	42	✓	Unknown	
DI 6	●	<Comment>	Measure	1	43	✓	Unknown	
USGS 50	●	<Comment>	Measure	1	44	✓	Delta Standard	USGS 50
USGS 50	●	<Comment>	Measure	1	45	✓	Delta Standard	USGS 50
USGS 50	●	<Comment>	Measure	1	46	✓	Delta Standard	USGS 50
USGS 46	●	<Comment>	Measure	1	47	✓	Delta Standard	USGS 46
USGS 46	●	<Comment>	Measure	1	48	✓	Delta Standard	USGS 46
USGS 46	●	<Comment>	Measure	1	49	✓	Delta Standard	USGS 46
USGS48	●	<Comment>	Measure	1	50	✓	Unknown	
USGS48	●	<Comment>	Measure	1	51	✓	Unknown	
USGS48	●	<Comment>	Measure	1	52	✓	Unknown	

Figure 8-13. Sample List for ¹⁸O in water runs

Normalization of $\delta^{18}\text{O}$ in Water Versus VSMOW Excluding or Including Scale Contraction (Slope Correction or Isotopic Linearity Correction)

Qtegra ISDS Software allows referencing of water samples for international standardization and scale contraction correction.

Evaluation after a LabBook run by doing normalization and scale compression. In general, the user does not want to analyze the standards in the first run, consequently standards are in different position.

The standardization does allow to use more than one standard. If more than one standard is used, the next normalization will start where the second set is positioned. Usually, a multiple number of standards is used as doublets, triplicates or more.

When multiple standards are moved to a direct position to the first scaling standard, the second standard is used for scale contraction (see [Figure 8-14](#)). Move the scale compression standard to the position right after the normalization standard. Qtegra will automatically do the scale compression and determine the isotope ratio in the external reference data column. To evaluate replicate runs, external referencing is done by using right-click on the data and do statistics on the data.

1	acid addition	<Comment>	Measure	1	1	<input checked="" type="checkbox"/>	Delta Standard	CO2 Calibration gas II
2	acid addition	<Comment>	Measure	1	2	<input checked="" type="checkbox"/>	Unknown	
3	acid addition	<Comment>	Measure	1	3	<input checked="" type="checkbox"/>	Unknown	
4	acid addition	<Comment>	Measure	1	4	<input checked="" type="checkbox"/>	Unknown	
5	SHK	<Comment>	Measure	1	9	<input checked="" type="checkbox"/>	Unknown	
6	SHK	<Comment>	Measure	1	10	<input checked="" type="checkbox"/>	Unknown	
7	SHK	<Comment>	Measure	1	11	<input checked="" type="checkbox"/>	Unknown	
8	NBS 19	<Comment>	Measure	1	12	<input checked="" type="checkbox"/>	Delta Standard	NBS 19 vs. VPDB
9	NBS 19	<Comment>	Measure	1	17	<input checked="" type="checkbox"/>	Delta Standard	NBS 19 vs. VPDB
10	NBS 19	<Comment>	Measure	1	18	<input checked="" type="checkbox"/>	Delta Standard	NBS 19 vs. VPDB
11	LVSEC	<Comment>	Measure	1	50	<input checked="" type="checkbox"/>	Delta Standard	IAEA LVSEC
12	LVSEC	<Comment>	Measure	1	51	<input checked="" type="checkbox"/>	Delta Standard	IAEA LVSEC
13	LVSEC	<Comment>	Measure	1	52	<input checked="" type="checkbox"/>	Delta Standard	IAEA LVSEC
14	ETH 1	<Comment>	Measure	1	19	<input checked="" type="checkbox"/>	Unknown	
15	ETH 1	<Comment>	Measure	1	20	<input checked="" type="checkbox"/>	Unknown	
16	ETH 1	<Comment>	Measure	1	25	<input checked="" type="checkbox"/>	Unknown	
17	CO8	<Comment>	Measure	1	26	<input checked="" type="checkbox"/>	Unknown	
18	CO8	<Comment>	Measure	1	27	<input checked="" type="checkbox"/>	Unknown	
19	CO8	<Comment>	Measure	1	28	<input checked="" type="checkbox"/>	Unknown	
20	NBS 18	<Comment>	Measure	1	33	<input checked="" type="checkbox"/>	Unknown	
21	NBS 18	<Comment>	Measure	1	34	<input checked="" type="checkbox"/>	Unknown	
22	NBS 18	<Comment>	Measure	1	35	<input checked="" type="checkbox"/>	Unknown	
23	ETH 3	<Comment>	Measure	1	36	<input checked="" type="checkbox"/>	Unknown	
24	ETH 3	<Comment>	Measure	1	41	<input checked="" type="checkbox"/>	Unknown	
25	ETH 3	<Comment>	Measure	1	42	<input checked="" type="checkbox"/>	Unknown	
26	ETH 4	<Comment>	Measure	1	43	<input checked="" type="checkbox"/>	Unknown	
27	ETH 4	<Comment>	Measure	1	44	<input checked="" type="checkbox"/>	Unknown	
28	ETH 4	<Comment>	Measure	1	49	<input checked="" type="checkbox"/>	Unknown	

Figure 8-14. Standardization for samples to the international scale and scale contraction correction

Refer to the *DELTA Q Operating Manual* for peak determination and obscuring sample results. See [Figure 8-15](#).



Figure 8-15. LabBook Cont Results Chromatogram only Sample without conditioning peak without reference

TriPlus RSH and Rack Setup

The TriPlus RSH rack setup can be set up for several different modes.

1. Thermostatted racks must be set in the continuous flow method and must be installed in nature.
 - a. Slot1 only
 - b. Slot2 only, but not with non-thermostatted rack taught at the left
 - c. Slot1 & Slot2
2. Double needle flush and measure
3. Single or double needle flush in a flush sequence and measurement in the LabBook

Each tool needle set-up must be defined in the autosampler firmware in the tool section (see [“TriPlus RSH SMART - Advanced and TriPlus RSH SMART - Standard Installation”](#) on page 5-19). Qtegra reads out the defined tool needle set up automatically from the RSH tool settings. The user must define for the rack, which tool is in use either for the LabBook or the flush or acid addition sequence (see [“Stand-Alone Flushing or Automated Flushing and Acid Addition \(“Sample Preparation”\)”](#) on page 7-20). LabBook is defined as being used for automated flushing but always sample measurement will be involved in the workflow of a LabBook.

CTC Pal 80 - A200S Mode Setup

For GasBench II applications, use the methods as set under the Dashboard for flushing and sampling. The LabBook does not require any rack. Qtegra will validate the autosamplers installed and will run the LabBook.

Operating the GasBench Plus Device with a ConFlo IV Interface

The ConFlo IV device is a universal continuous flow interface to a DELTA Series MS or a 253 Plus Series MS. It has two high-flow sample gas connection ports (for EA) and one low-flow sample gas connection port (for a GasBench Plus device or a GC).

It is equipped with three sample gas dilution systems in order to adjust the sample gas signal. Five reference gas ports are permanently connected for referencing. This allows automated H₃ factor determination and linearity tests of different gases. For details, refer to the *DELTA Q Operating Manual*.

Connecting the GasBench Plus Device to the ConFlo IV Device

To install the ConFlo IV interface itself and for its pre-installation requirements, refer to the *ConFlo IV Operating Manual*.

When assembling the hardware, establish the sample gas connection to the ConFlo IV interface. The ConFlo IV interface must already be in operation.

The GasBench Plus device must be connected to the low-flow port of the ConFlo IV interface. The “sample dilution 1” step of the automated sample gas dilution procedure replaces the formerly known autodilution of the GasBench Plus device.

❖ To connect the GasBench Plus device to the ConFlo IV interface

1. Close the needle valve of the GasBench Plus sample and reference capillaries.
2. Cut the sample capillary that connects the water trap 2 to the sample open split (see [Figure 9-46](#) on [page 9-39](#)). Use a press-fit to connect the 0.32 mm ID fused silica capillary to the water trap 2. See [Figure 8-16](#).

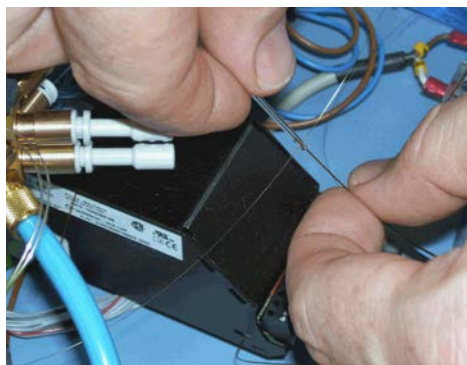


Figure 8-16. Cutting sample transfer capillary to IRMS

Measurement Procedures for Real Samples

Operating the GasBench Plus Device with a ConFlo IV Interface

3. Connect one end of a 0.32 mm ID fused silica capillary to a 0.32 mm ID-0.32 mm ID press-fit (BgB-Analytik, for example). See [Figure 9-46](#).
4. Connect the 0.32 mm ID fused silica capillary to the LF port of the ConFlo IV interface. See [Figure 8-17](#) which shows the low flow connector for the sample gas capillary of the GasBench Plus device at the rear panel of the ConFlo IV interface.

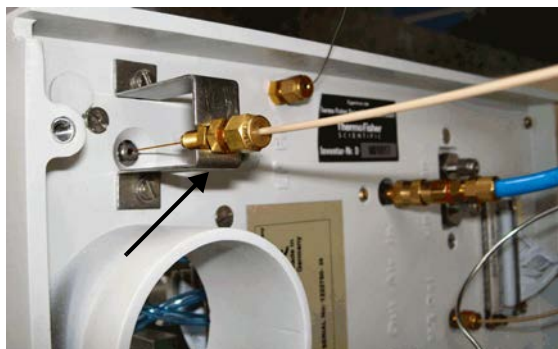


Figure 8-17. Low flow connector for GasBench Plus sample gas capillary

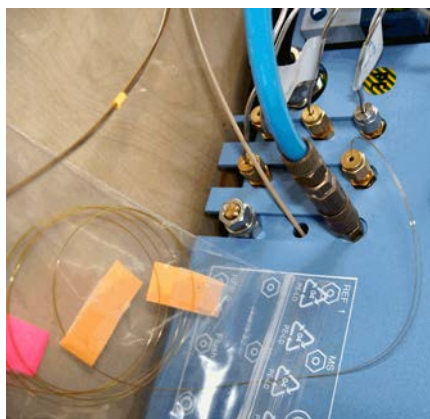


Figure 8-18. Feedthrough of sample gas capillary to LF port of ConFlo IV interface

Thermo Fisher Scientific recommends leading the 0.32 mm ID fused silica capillary through the hole below the compressed air connection of the GasBench Plus device. See [Figure 8-18](#) (feedthrough of the new sample gas capillary to low flow port of the ConFlo IV interface).

To connect the ConFlo IV interface to the reference gas capillary of the IRMS, refer to the *ConFlo IV Operating Manual*.

Carbonate Option

The carbonate option is used to measure $\delta^{13}\text{C}/^{12}\text{C}$ and $\delta^{18}\text{O}/^{16}\text{O}$ values simultaneously from carbonates. It allows for fully automated measurements of calcite, dolomite, foraminifera, or bulk sediments.

NOTICE

Make sure that only washed sample vials (with blue caps) are used with the carbonate option: a package consists of 100 sample vials made of borosilicate glass, 300 caps and 300 septa to hermetically close the vials. Sample vials, caps and septa are all washed. Alternatively, unwashed sample vials are available. Other caps are not dimensionally stable at the elevated temperatures.

Contents

- [Placing the Components of the Carbonate Option](#) on page 9-2
- [Preparing Phosphoric Acid](#) on page 9-13
- [Handling the Sample Vials](#) on page 9-15
- [Maintenance of the Carbonate Option](#) on page 9-17
- [Analyzing Carbonates](#) on page 9-38

Carbonate Option

Placing the Components of the Carbonate Option

Placing the Components of the Carbonate Option

❖ To place the components of the carbonate option

1. Place the acid reservoir in the rightmost row of the sample tray and the acid pump behind the tray. Connect them using the tubing supplied with the reservoir. See [Figure 9-9](#).
2. Do not cut the tubing length. The small diameter tubing is for venting the reservoir. Place it below the cover of the tray.
3. To ensure proper closing of the tray cover, make a small cut at the edge of the cover with a file.
4. Connect the acid needle tubing to the acid pump. See [Figure 9-8](#). Place the needle in the GasBench Tool 55 on the right side.
5. Mount the protection tray on the pump head. Carefully feed the acid needle tubing and the tubing to the reservoir through the slits on the protection tray.
6. Place the sample needle in the left slot of the dual needle holder.

Acid Pump

[Figure 9-1](#) shows the acid pump in top view (left) and in front view (right).

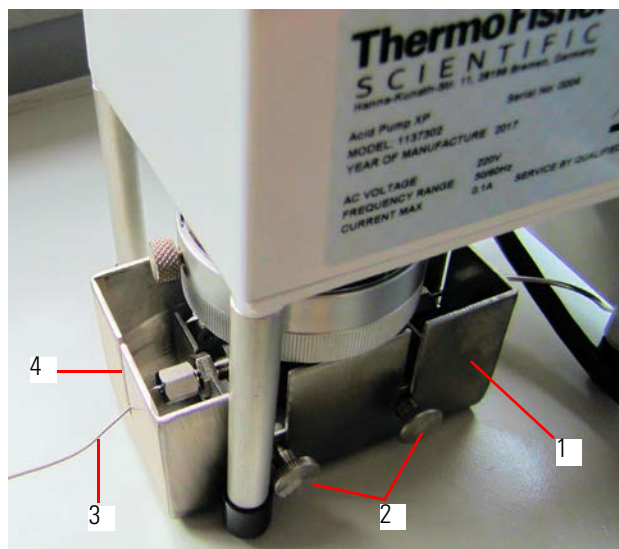


Figure 9-1. Acid pump in top view (left) and front view (right)

Protective Cover of the Acid Pump Head

The protective cover, **1** in [Figure 9-2](#), encloses the acid pump head and its fittings. In the unlikely event of acid drops leaking at the connectors, they are collected at the bottom of the protective cover. This minimizes the risk of contamination for personnel and equipment.

Four knurled screws **2**, two on either side, fix the protective cover in place. The protective cover is made of stainless steel. Because stainless steel is inert against phosphoric acid, the cover can be cleaned in a laboratory dishwasher.



Labeled Components: 1=protective cover, 2=knurled screws, 3=capillary, 4=lateral slot

Figure 9-2. Protective cover of acid pump

Safety Symbols on the Acid Pump

[Table 9-1](#) lists all safety labels on the acid pump and their respective positions. See the indicated safety notices to prevent risk of harm to the operator and to protect the acid pump against damage. If they are present, read and follow the instructions on the labels.

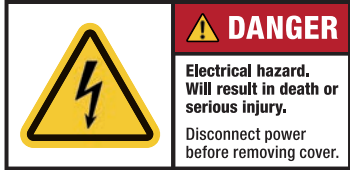
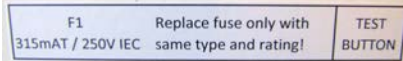
Table 9-1. Safety labels on the acid pump

Label	Label description	Label position
	It indicates the presence of concentrated phosphoric acid as a corrosive, hazardous liquid. See page 9-17 for details.	This label is attached to the front panel of the acid pump.
	The label indicates that only authorized personnel may perform service operations. See page 9-17 for details.	This label is attached to the top panel of the acid pump.

Carbonate Option

Placing the Components of the Carbonate Option

Table 9-1. Safety labels on the acid pump, continued

Label	Label description	Label position
 The label features a yellow triangular warning symbol with a black lightning bolt. To the right of the symbol is a red rectangular box with the word "DANGER" in white. Below this, the text reads: "Electrical hazard. Will result in death or serious injury. Disconnect power before removing cover."	The label indicates the presence of an electrical hazard that results in death or serious injury. See page 9-17 for details.	This label is attached to the top panel of the acid pump.
 The label is rectangular and contains the following text: "F1" on the left, "Replace fuse only with same type and rating!" in the center, and "TEST BUTTON" on the right.	The label indicates to only replace a fuse by one of the same type and rating.	This label is attached to the front panel of the acid pump.

Name Plate of the Acid Pump

To identify the acid pump correctly when you contact Thermo Fisher Scientific, always have the information from the name plate (also called rating plate) available. The name plate of the acid pump is attached to its rear side. See [Figure 9-3](#) and [Figure 9-4](#). It contains the serial number, which is important in any type of communication with Thermo Fisher Scientific.

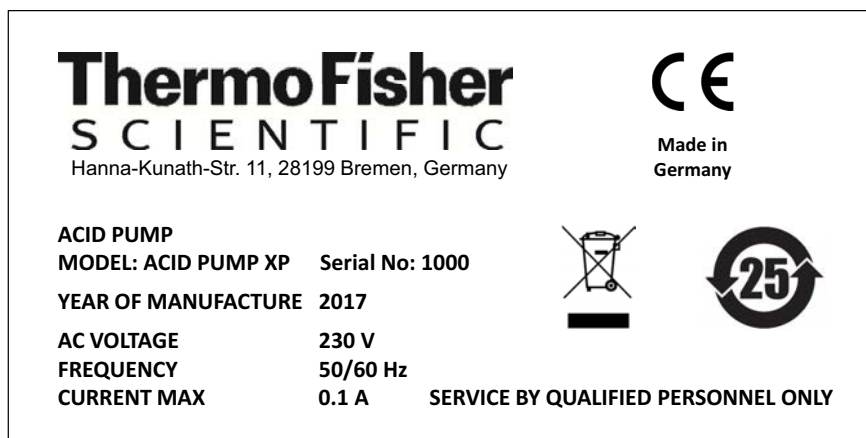


Figure 9-3. Name plate of acid pump (example)



Figure 9-4. Name plate of acid pump attached to rear panel

Intended Use of the Acid Pump

The acid pump is designed for use in connection with a GasBench Plus device and an Thermo Scientific IRMS.



Obey the following usage guidelines when you operate your acid pump:

- The acid pump is designed to be placed on a bench in the laboratory. It is not designed for the use outdoors.
- The acid pump is designed for laboratory research use only. It is not designed for use in diagnostic or medical therapeutic procedures.
- The acid pump must be used only for pure phosphoric acid (99%, that is of 1.85 g/mL density).

If the acid pump is used in a manner not specified by Thermo Fisher Scientific, the protection provided by the instrument could be impaired. Thermo Fisher Scientific assumes no responsibility and will not be liable for instrument damage and/or operator injury that might result from using the instrument improperly.

Qualification of the Personnel



Personnel that install or operate the acid pump must have the following qualifications:

- **Electrical Connections**
The electrical installation must be carried out by qualified and skilled personnel (electrician) according to the applicable regulations (for example, cable cross-sections, fuses, earth grounding connection). Refer to the *Gas Isotope Ratio MS Pre-Installation Requirements Guide* for the specifications.
- **General Operation**
The acid pump is designed to be operated by qualified laboratory personnel. Before they start, all users must be instructed about the hazards presented by the instrument and the chemicals applied. The users must be advised to read the relevant Material Safety Data Sheets (MSDSs).

Residual Hazards

Users of the acid pump must pay attention to the following residual hazards.

WARNING

Corrosive Chemicals. Phosphoric acid causes severe skin burns and eye damage. Wear protective clothing, protective gloves, and a face plate when you handle phosphoric acid. Goggles are not sufficient.

Wear protective clothing, protective gloves and a face mask when you handle phosphorous pentoxide. Goggles are not sufficient.

Also contain waste streams and use proper ventilation. Operate under a fume hood. Operate according to the Material Safety Data Sheet (MSDS).

WARNING

High Voltage. High voltages capable of causing an electric shock are used in the instrument. The housing of the acid pump does not contain any serviceable parts. Do not open the housing. Opening the housing is only allowed for maintenance purposes by Thermo Fisher Scientific personnel.

To make sure that the instrument is free from all electric current, always disconnect the power cord before you try any type of maintenance.



It is the user's responsibility to maintain the system properly by performing the system maintenance procedures on a regular basis.

Service by the customer must be performed by trained qualified personnel only and is restricted to servicing mechanical parts. Service on electronic parts must be performed by Thermo Fisher Scientific field service engineers only.

Do not try to repair or replace any component of the system that is not described in this manual without the assistance of your Thermo Fisher Scientific field service engineer.

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

Capillaries Connected to the Acid Pump

Figure 9-5 shows the stainless steel capillaries (**3** in Figure 9-2) connected to the acid pump. They enter and leave the protective cover via lateral slots (**4** in Figure 9-2):

- Capillary **1** ("pump line"; left direction) leads to the acid needle.

Carbonate Option

Placing the Components of the Carbonate Option

- Capillary **2** (“suction line”; right direction) leads to the acid reservoir that contains up to 12 mL of phosphoric acid.



Labeled Components: 1=capillary to acid needle (“pump line”), 2=capillary to acid reservoir (“suction line”)

Figure 9-5. Acid pump in rear view with capillaries connected to it

Connections of the Acid Pump to the MS

Figure 9-6 shows the power plug **1** (“power cable”) of the acid pump and the ribbon cable plug (10-pole; “data cable”) **2** of the acid pump.



Labeled Components: 1=power plug of acid pump, 2=ribbon cable plug of acid pump

Figure 9-6. Plugs of acid pump

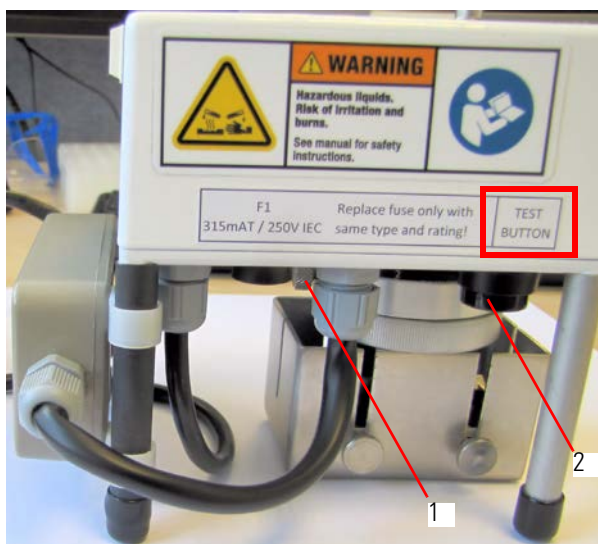
- The power plug **1** needs to be connected to a socket at the rear panel of the MS (not to a wall socket):
 - indirectly, using an extension cable (in case of a 253 Plus Series MS)
 - directly (in case of a DELTA Series MS)
- The ribbon cable plug **2** needs to be connected to the plug and measure adapter (pnm adapter) at the rear panel of the MS.

For details, see “Replacing the Acid Pump Head” on page 9-20.

Acid Pump Control

The adjusting screw, **1** in Figure 9-7, controls the volume pumped per stroke via a mark. See “Adjusting the Acid Pump” on page 9-10. This volume pumped per stroke is reported to Qtegra.

Pressing the button **2** (below the label “test button” on the housing) triggers a single stroke at the pump manually.



Labeled Components: 1=adjusting screw, 2=button

Figure 9-7. Adjusting screw and button

To run the acid pump, an Qtegra version higher than 3.0.0.4.12 is required:

- If you have the Qtegra version 3.0.0.4.12 or lower, you need the patch that comes on your USB stick.
- If you have an Qtegra version below 2.0, contact your local service representative.

Adjusting the Acid Pump

The acid pump needs to be adjusted before operation as follows:

❖ To adjust the acid pump

1. Set the acid pump to minimal pumping volume.

This allows exact dosing of the acid and pumping the viscous concentrated phosphoric acid.

2. Adjust the pumping volume until you obtain one drop of acid by every ten pump strokes:

Press the button at the acid pump to force a single stroke. Wait for 1–3 seconds between single strokes.

These settings are a precondition for retracting the acid from the needle tip. This also avoids spoiling the acid to the septum.

To change the pump rate within the range, turn the control ring:

- turning it clockwise decreases the pump rate.
- turning it counterclockwise increases the pump rate.

NOTICE

Do not use solvents to test the pump, because the O-rings might be destroyed.

Tip It is useful to set a larger pumping volume during the initial filling of pump and tubing.

Connecting the Acid Needle

❖ To connect the acid needle

1. Connect the acid needle to the bulk head connector.
2. Tighten the thread of the bulkhead connector with an intermediate suitable O-ring and a correctly directed metal seal.
3. Check for air bubbles at the tip of the acid needle. Leak tightness is indicated by absence of air bubbles.

Tip If no drops appear, check whether the flow direction has been correctly set in Qtegra. See [Figure 9-9](#).

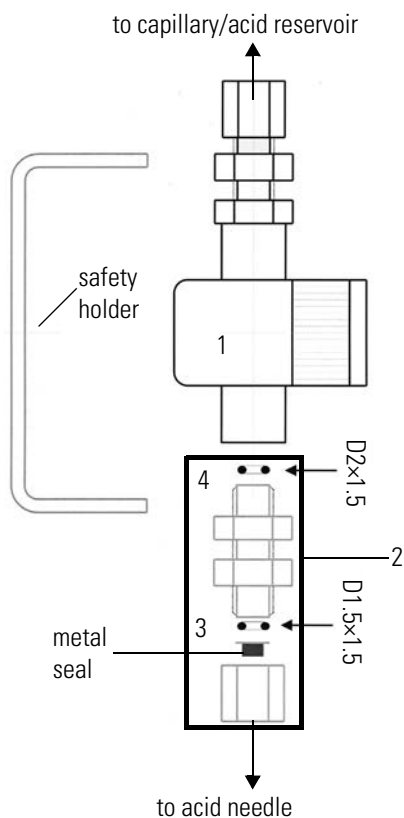


Figure 9-8. Details of pump head arrangement of seals

Pos.	Qty.	Designation
1	1	acid pump head
2	1	bulkhead connection (SERTO, 2 mm)
3	2	O-ring (1.5×1.5)
4	2	O-ring (2×1.5)

The O-rings **3** and **4** are not part of the bulkhead connection **2**, in contrast to the metal seal.



Figure 9-9. Placement of acid reservoir bottle in tray

Carbonate Option

Placing the Components of the Carbonate Option

Connect the acid reservoir to the acid pump with the appropriate O rings and connectors as given in [Figure 9-8](#).

[Figure 9-9](#) shows the standard placement of the reservoir bottle in the thermostatted tray. Place the acid pump beside the tray and feed the 1/16 in. stainless steel capillary underneath the tray cover to the acid pump.

NOTICE

Strictly observe the sequence of O-rings and metal seal parts given in [Figure 9-8](#). Otherwise, the assembly might not seal to air, and then the pump will not deliver acid.

When mounting the acid needle in the GasBench Tool 55 setup, use only the adapter (acid needle holder, [Figure 9-10](#)). This adapter has a slightly bigger bore diameter than the original adapter and can be recognized by a groove around the knurl. For the acid needle plunger adapter use only the GasBench Tool 55. This will prevent the acid needle from breaking at any time.

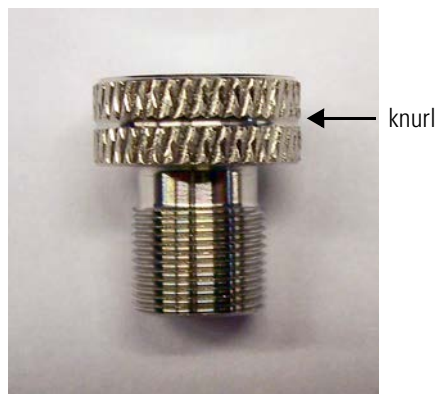


Figure 9-10. Acid needle holder

Preparing Phosphoric Acid

WARNING

Corrosive Chemicals. Phosphoric acid causes severe skin burns and eye damage. Wear protective clothing, protective gloves, and a face plate when you handle phosphoric acid. Goggles are not sufficient.

Wear protective clothing, protective gloves and a face mask when you handle phosphorous pentoxide. Goggles are not sufficient.

Also contain waste streams and use proper ventilation. Operate under a fume hood. Operate according to the Material Safety Data Sheet (MSDS).

As phosphoric acid is not a very strong acid, contamination of personnel by touching parts might not be noticed immediately, but delayed (contamination of fingers by droplets, passing to the eyes later on, for example).

NOTICE

For use with the GasBench Plus device, do not use phosphoric acid with densities above 1.85 g/mL. This would inevitably cause the acid pump to get clogged.

Phosphoric acid with a density of 1.85 g/mL is prepared on the basis of 99% phosphoric acid. No phosphorous pentoxide needs to be added.¹

H₃PO₄ is available in brown glass containers that contain 500 mL of 99% phosphoric acid. Because the phosphoric acid is crystallized at 25 °C, carefully heat it in its brown glass container until it becomes liquid.

NOTICE

Do not heat the phosphoric acid in a non-glass container.

Check the fused silica capillary of the measurement needle for droplets of remnant phosphoric acid every day and for droplets of water (DIC), which may contain phosphoric acid. Phosphoric acid destroys the Nafion water trap, the Valco valve rotor and the PoraPLOT™ GC column.

¹ For a discussion of preparation methods, refer to the following publications:

J. Burman, O. Gustafsson, M. Segl and B. Schmitz: A simplified method of preparing phosphoric acid for stable isotope analyses of carbonates. *Rapid Commun. Mass Spectrom.* 2005, **19**, 3086–3088

E.A. Wachter and J.M. Hayes: Exchange of oxygen isotopes in carbon dioxide–phosphoric acid systems. *Chem. Geol. (Isotope Geoscience Section)* 1985, **52**, 365–374

Carbonate Option

Preparing Phosphoric Acid

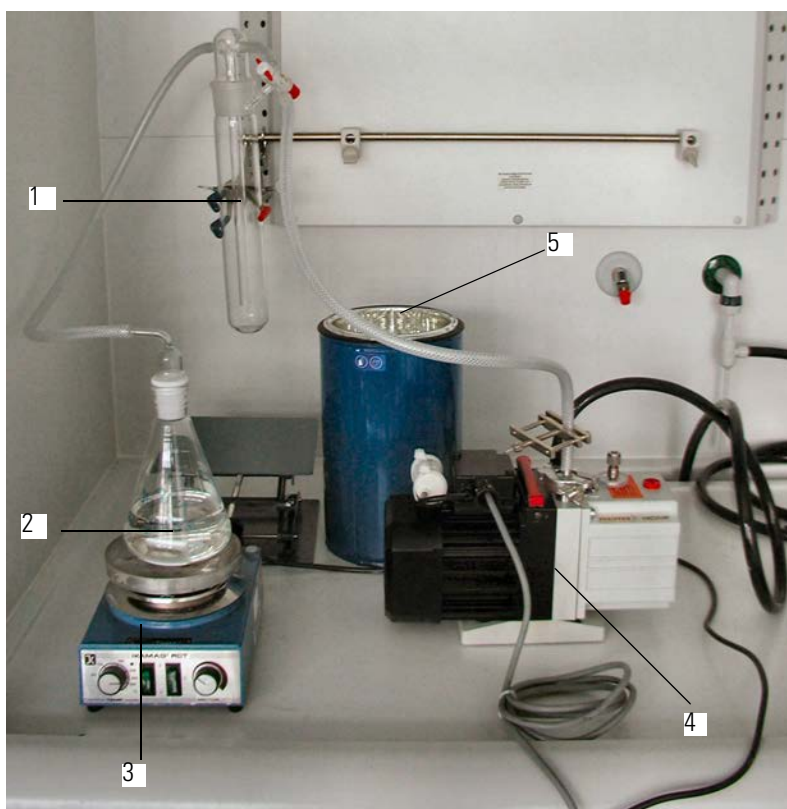
Checking the Density of th Phosphoric Acid

If necessary, allow the acid to cool down to room temperature and check its density as shown in [Figure 9-11](#). If it is less than 1.85 g/mL, remove the water. See “[Removing Water from the Phosphoric Acid](#)” on [page 9-14](#).



Figure 9-11. Checking density of phosphoric acid

Removing Water from the Phosphoric Acid



Labeled Components: 1=500 mL water trap, 2=phosphoric acid, 3=hot plate and magnetic stirrer, 4=vacuum pump, 5=dewar vessel for liquid nitrogen

Figure 9-12. Removing water from phosphoric acid

Figure 9-12 shows the apparatus that can be used to remove water (and absorbed gases) from phosphoric acid. Excess water is removed by freezing it in a liquid nitrogen trap under low vapor pressure. This apparatus can be used to regularly dewater prepared 95% H_3PO_4 .

Handling 99% Phosphoric Acid

Handling during a measurement:

- Keep the phosphoric acid in a closed container within a heating cabinet at 70 °C.

Handling when no measurement is performed:

- Keep the phosphoric acid in a closed container in a heating cabinet at above 50 °C.
- Keep the phosphoric acid in a tightly closed container at 25 °C. Use Parafilm™ to seal the screw cap. In case of recrystallization, carefully heat it anew and check its density.

Handling the Sample Vials

The sample vials used for carbonate measurements should be free of organic and inorganic contaminations before they are loaded with carbonate. You can clean the sample vials either manually or automatically in a dishwasher.

To ensure the complete removal of all residues, consider the following recommendations for cleaning the vials:

- Do not use plastic containers, only use glassware. Especially, do not use acetone in plastic bottles.
- Only use liquid detergents and no detergent in powder form.
- Use only deionized water for cleaning. Last rinsed water must be at least reversed phase water.

Manual Cleaning of the Sample Vials

❖ To clean the sample vials manually

1. Fill up the vials with warm diluted phosphoric acid (that is phosphoric acid plus warm distilled water) and leave them for eight hours. Alternatively, put the vials into distilled water immediately after analysis.
2. Repeatedly rinse the vials with distilled water using a washing bottle. Best rinse it twice in deionized water of 70 °C.

Tip You may rinse it a third time in millipore water or reversed phase deionized water (resistance $\leq 2 \text{ M}\Omega$).

3. Rinse the vials with acetone using a washing bottle, too. This helps to dry the vials faster. Acetone does not impact isotope results. This ensures removal of residual water that may contain acid-soluble minerals as well.

Tip Acetone contains residues. Therefore, dry the vials upside down.

4. Dry the vials in a drying chamber at 72 °C for one hour (if acetone was used to dry the vials) or up to 2.5 hours (if no organic solvent was used to dry the vials). Cover them with aluminum foil to protect them against contamination.

Machine Cleaning of the Sample Vials

Used sample vials can be cleaned automatically in a laboratory dishwasher made of stainless steel. The dishwasher must be connected to deionized water of high quality.

❖ To clean the sample vials automatically

1. Immediately after analysis, put the vials into distilled water.
2. Use automated liquid detergent dosing containing 0.5–1% of KOH or NaOH (suggested detergent: neodisher™ LM3, manufacturer: Dr. Weigert; see www.drweigert.de).
3. Use automated liquid neutralization dosing containing 0.1% of acetic acid or citric acid (suggested detergent: neodisher™ Z, manufacturer: Dr. Weigert; see www.drweigert.de).
4. Rinse twice. If only deionized water is available, a third manual cleaning step with at least reversed phase deionized water is mandatory.
5. Dewater the vials with acetone (acetone does not impact isotope results). This step is not mandatory.

If possible, dry the vials turned upside down.

Maintenance of the Carbonate Option

This chapter describes how the acid pump system must be maintained and put into idle mode if no carbonate material or acid addition is needed.

Cleaning the Acid Pump Housing

❖ **To clean the acid pump housing from outside**

1. Take a piece of cloth (preferably made of microfiber) and dampen it only slightly with distilled water.

NOTICE

Do not make the piece of cloth too wet. Do not immerse it into water.

2. Wipe the acid pump housing with the piece of cloth.

Cleaning the Acid Pump Head and Other Parts

CAUTION

Corrosive Chemical. Phosphoric acid might cause severe skin burns and eye damage. Always wear protective clothing, gloves, and safety glasses when you handle phosphoric acid. Also contain waste streams and use proper ventilation. Refer to your supplier's Material Safety Data Sheet (MSDS) for proper handling of phosphoric acid.

As phosphoric acid is not a very strong acid, contamination of personnel by touching parts might not be noticed immediately, but delayed (contamination of fingers by droplets, passing to the eyes later on, for example).

If you clean the head of the acid pump or other parts (acid needle, stainless steel capillaries, tubings, etc.), they might be contaminated by phosphoric acid.

Before you replace a seal of the head of the acid pump, remove potential remnants of phosphoric acid.

NOTICE

To clean the parts from remnants of phosphoric acid, carry out **only** the procedure described below. Do not use any alternative cleaning strategies on your own.

❖ **To clean the acid pump head and other parts**

1. Remove the connection of the tubing to the acid reservoir.
2. Place the acid needle into an empty vial of at least about 50 mL size.

3. Let the pump run by continuously pressing the button at the housing.

Wait until no more acid droplets drip off the tip.

NOTICE

Do not let the pump run dry for longer times than needed. The pump head may be overheated and damaged.

4. Connect the pump inlet to a vial filled with distilled water.
Continue pumping until about 5 mL of water have been flowed out of the needle. Dispose of the acid.
5. Remove the tubing from the water vial.
Continue pumping until no more water flows out of the needle. Dispose of the water.
6. Connect the pump inlet to a vial filled with isopropanol.
Continue pumping until about 5 mL of isopropanol have been flowed out of the needle.
7. Remove the tubing from the isopropanol vial.
Continue pumping until no more isopropanol flows out of the needle. Dispose of the isopropanol.
8. Demount the pump head. Leave it for drying.
9. Place the capillary tubing in an oven. Let it dry at 80 °C for 3 hours.

Avoiding Clogging of the Acid Pump

Acid clogging may occur after improper handling of the acid pump, phosphoric acid preparation and using phosphoric acid thereafter. Thermo Fisher Scientific recommends using only pure phosphoric acid as prepared according to [“Preparing Phosphoric Acid”](#) on [page 9-13](#).

Clogging of phosphoric acid in the acid delivery capillaries of the acid pump, the phosphoric acid capillaries of the GasBench Plus and acid needle can be avoided, if you take care:

- to use only pure phosphoric acid (99%, that is of 1.85 g/mL density).
- not to use phosphoric acid of higher density as it would crystallize inside the acid pump head and the capillaries. A crystal core may be formed. After cleaning, even a small crystal may lead to crystallization again due to its reduced lattice energy, and therefore to repeated clogging.

- to avoid any leakage between the acid container and the tip of the acid needle. A leakage may lead to clogging of acid inside the acid pump head.

Removing Crystallized Phosphoric Acid from Clogged Parts

WARNING

Corrosive Chemical. Phosphoric acid might cause severe skin burns and eye damage. Always wear protective clothing, gloves, and safety glasses when you handle phosphoric acid. Also contain waste streams and use proper ventilation. Refer to your supplier's Material Safety Data Sheet (MSDS) for proper handling of phosphoric acid.

As phosphoric acid is not a very strong acid, contamination of personnel by touching parts might not be noticed immediately, but delayed (contamination of fingers by droplets, passing to the eyes later on, for example).

Concentrated phosphoric acid has a high viscosity and tends to crystallize. As a consequence, a pressure may be built up in the acid pump. Thereby, small leaks may be formed at the screw connections and on the membrane of the acid pump head.

Due to its high viscosity, concentrated phosphoric acid does not splash out at leaks, but rather droplets pass out. These acid drops may contaminate other parts of the acid pump (connectors, membrane, etc.) and parts of the mass spectrometer.

Several parts (tubings, stainless steel capillary to the acid needle, for example) may get clogged by crystallized phosphoric acid. Removing the crystals becomes necessary as a maintenance operation.

WARNING

To remove crystallized phosphoric acid from clogged parts, **only** carry out the procedure described below. Do not use any alternative heating or liquefying procedures on your own. These might cause fire or an electric shock, or damage the electrical isolation.

❖ **To remove crystallized phosphoric acid from clogged parts**

1. Remove the capillary with the acid needle connected to it from the acid pump head.
2. Place the capillary together with the acid needle in an ultrasonic bath. Leave it at 80 °C for 3 hours.
3. Perform the decontamination procedure described at [“To clean the acid pump head and other parts”](#) on page 9-17.
4. If the clogging still persists,
 - a. Dispose of the capillary and of the acid needle. Both parts are firmly connected to each other.

- b. Order a new capillary together with a new acid needle.

Unclogging the Sample Needle

The sample needle might get clogged by and by due to phosphoric acid crystallizing within the fused silica capillary.

❖ To unclog the sample needle

1. Detach the fused silica capillary from the GasBench Plus device to avoid destroying the Nafion™ polymer of the water trap.
2. Fill an Exetainer™ with isopropanol.
3. Heat it to approximately 40 °C.
4. Use the pressure of the helium flow from the GasBench Plus device through the stainless steel capillary to pressurize the Exetainer.

If you have a setup of the GasBench Plus device with a flush needle for flushing Exetainers with gas (as used for water equilibration), you can connect the stainless steel capillary even to this gas line and use a higher pressure. Be careful and put a beaker under the end of the fused silica capillary because isopropanol will run very fast as soon as the line is unclogged.

5. A 0.1 mm ID capillary can be moved through the side hole or bottom end of the needle. Accidentally deposited septa butyl rubber from pinching might be removed that way.

Replacing the Acid Pump Head



Corrosive Chemical. Phosphoric acid might cause severe skin burns and eye damage. Always wear protective clothing, gloves, and safety glasses when you handle phosphoric acid. Also contain waste streams and use proper ventilation. Refer to your supplier's Material Safety Data Sheet (MSDS) for proper handling of phosphoric acid.

As phosphoric acid is not a very strong acid, contamination of personnel by touching parts may not be noticed immediately, but delayed (contamination of fingers by droplets, passing to the eyes later on, for example).

Replace the acid pump head if:

- the decontamination procedure (see “To remove crystallized phosphoric acid from clogged parts” on page 9-19) has been performed, but was not successful:

The concentrated phosphoric acid that crystallized inside the acid pump head could not be removed and prevents the acid pump from working properly.

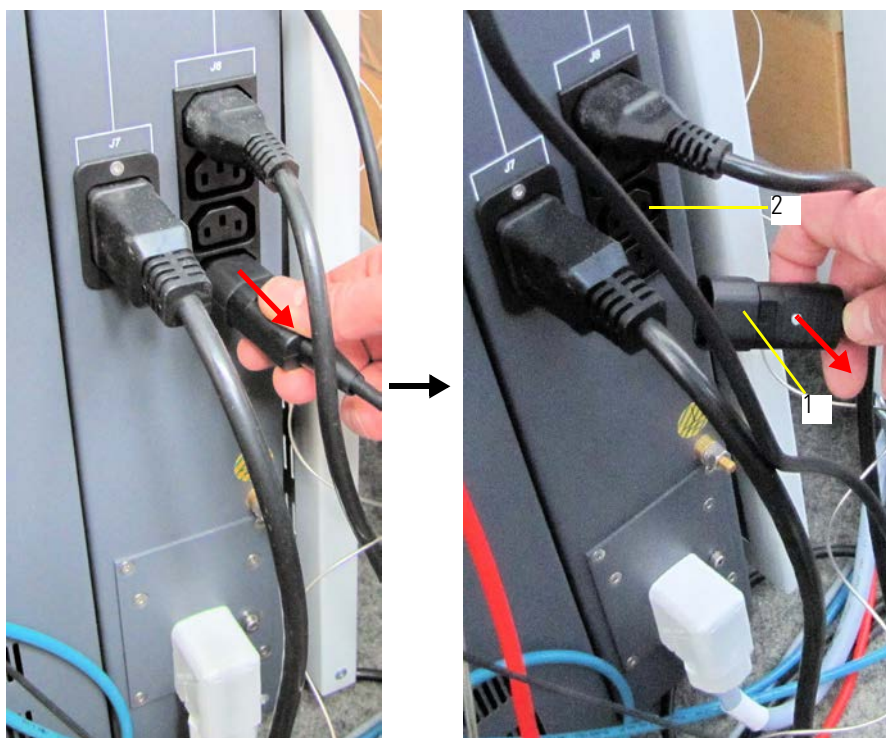
- drops of concentrated phosphoric acid leak out of the acid pump head:

At the minimum, the seals must be replaced.

Step 1: Disassembling the Old Acid Pump Head

❖ To disassemble the old acid pump head

1. Disconnect the acid pump from the mass spectrometer as follows:
 - In case of a **DELTA Q Series MS**:
 - a. As [Figure 9-13](#) shows, pull the power plug **1** (“power cable”) of the acid pump out of the socket **2** at the rear panel of the DELTA Q Series MS.



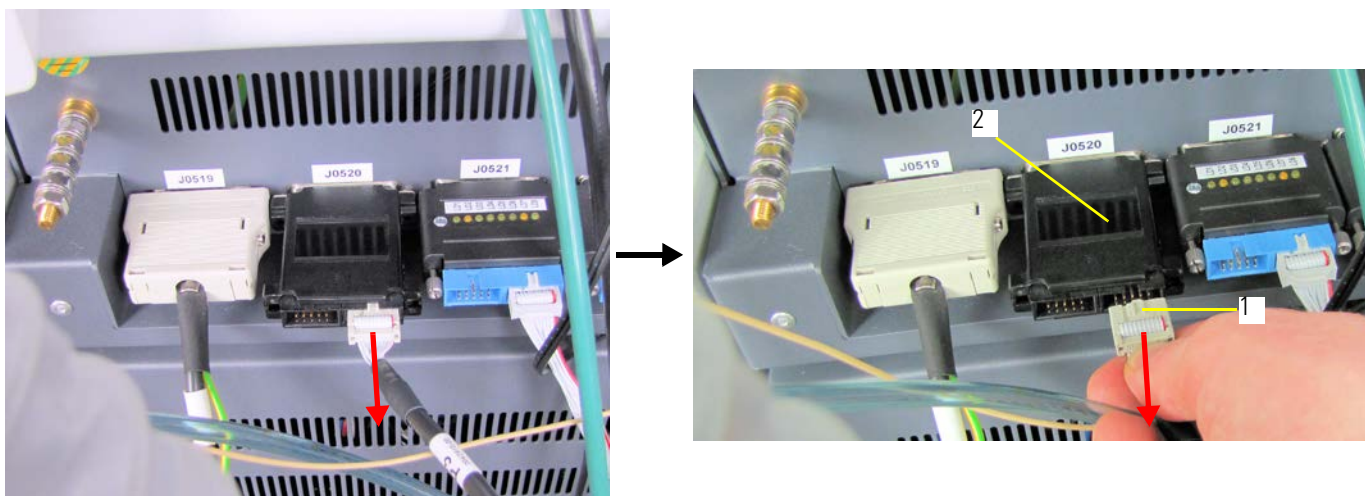
Labeled Components: 1=power plug of acid pump, 2=socket at rear panel of DELTA Q Series MS

Figure 9-13. Pulling power plug of acid pump out of socket at rear panel of DELTA Q Series MS

Carbonate Option

Maintenance of the Carbonate Option

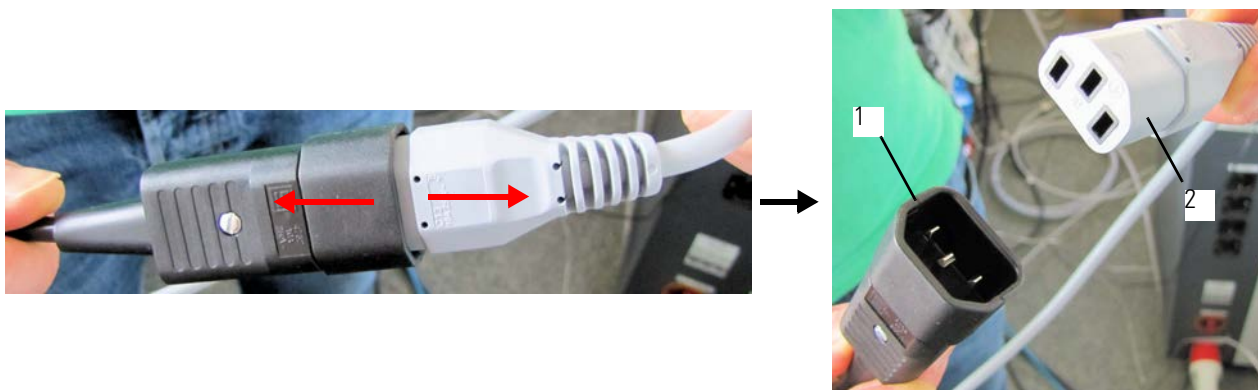
- b. As [Figure 9-14](#) shows, pull the ribbon cable plug **1** (“data cable”) of the acid pump out of the pnm adapter **2** at the rear panel of the DELTA Q Series MS.



Labeled Components: 1=ribbon cable plug of acid pump, 2=pnm adapter

Figure 9-14. Pulling ribbon cable plug of acid pump out of pnm adapter at rear panel of DELTA Q Series MS

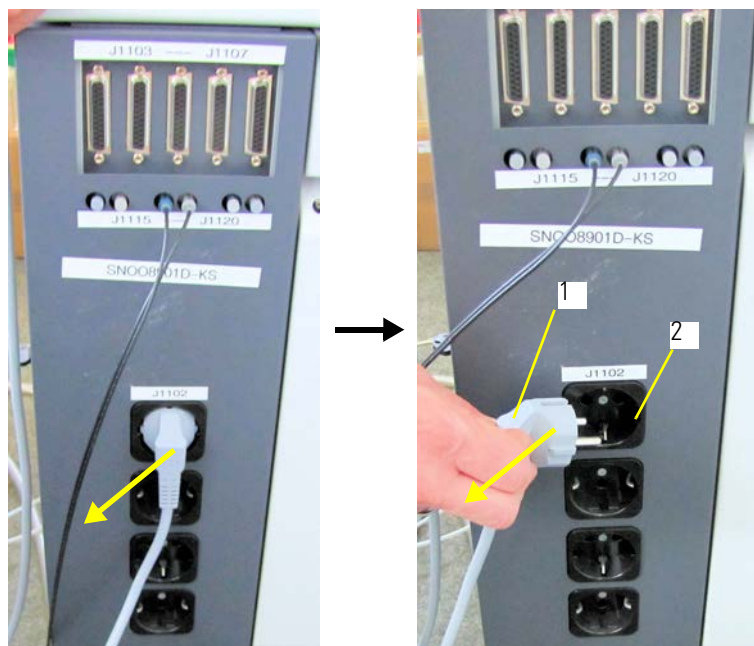
- In case of a **253 Plus** Series MS:
 - a. As [Figure 9-15](#) shows, pull the power plug **1** of the acid pump out of the female end **2** of the extension cable.



Labeled Components: 1=power plug of acid pump, 2=female end of extension cable

Figure 9-15. Pulling power plug of acid pump out of female end of extension cable

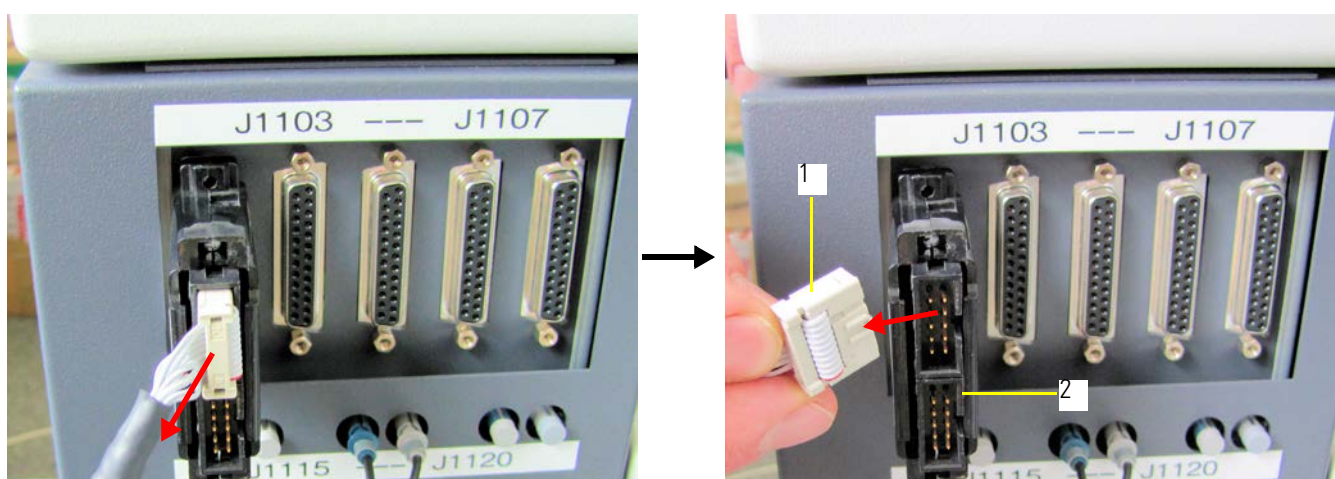
- b. As Figure 9-16 shows, pull the male end **1** of the extension cable out of the socket **2** at the rear panel of the 253 Plus MS or the MAT 253 MS.



Labeled Components: 1=male end of extension cable, 2=socket at rear panel of 253 Plus MS or MAT 253 MS

Figure 9-16. Pulling male end of extension cable out of socket at rear panel of 253 Plus MS or MAT 253 MS

- c. As Figure 9-17 shows, pull the ribbon cable plug **1** of the acid pump out of the pnm adapter **2** at the rear panel of the 253 Plus MS or the MAT 253 MS.



Labeled Components: 1=ribbon cable plug of acid pump, 2=pnm adapter

Figure 9-17. Pulling ribbon cable plug of acid pump out of pnm adapter at rear panel of 253 Plus MS or MAT 253 MS

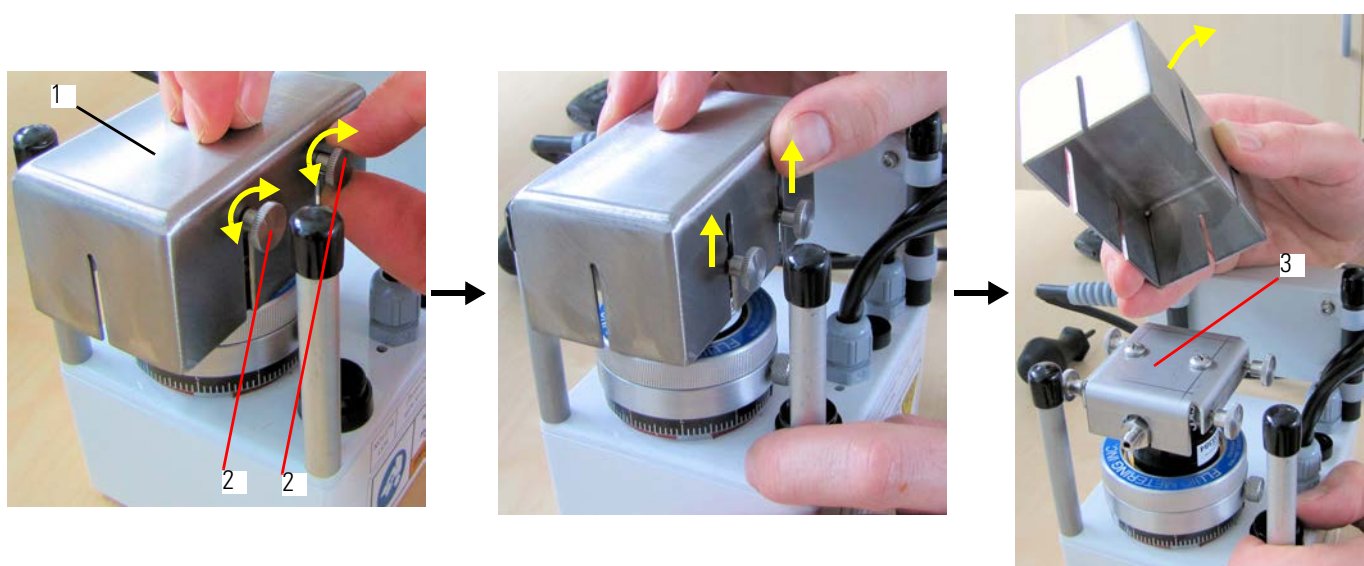
2. Unscrew the capillary that leads to the acid needle.

Carbonate Option

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3. Unscrew the capillary that leads to the acid container.
4. Place the acid pump upside down on a workbench to easily access the protective cover.
5. As [Figure 9-18](#) shows, remove the protective cover **1** as follows:
 - a. Manually loosen the four knurled screws **2** (two on either side).
 - b. Carefully pull the protective cover straight upwards.
 - c. Take the protective cover away.

The bracket **3** becomes accessible. Two crosshead screws fix not only the bracket, but also the protective cap of the acid pump below it.



Labeled Components: 1=protective cover, 2=knurled screw, 3=bracket with two crosshead screws

Figure 9-18. Removing protective cover to access bracket

6. Remove both crosshead screws using a crosstip screwdriver. See [Figure 9-19](#).

7. Manually loosen the four knurled screws a bit. Do not remove them entirely from the bracket. See [Figure 9-19](#).

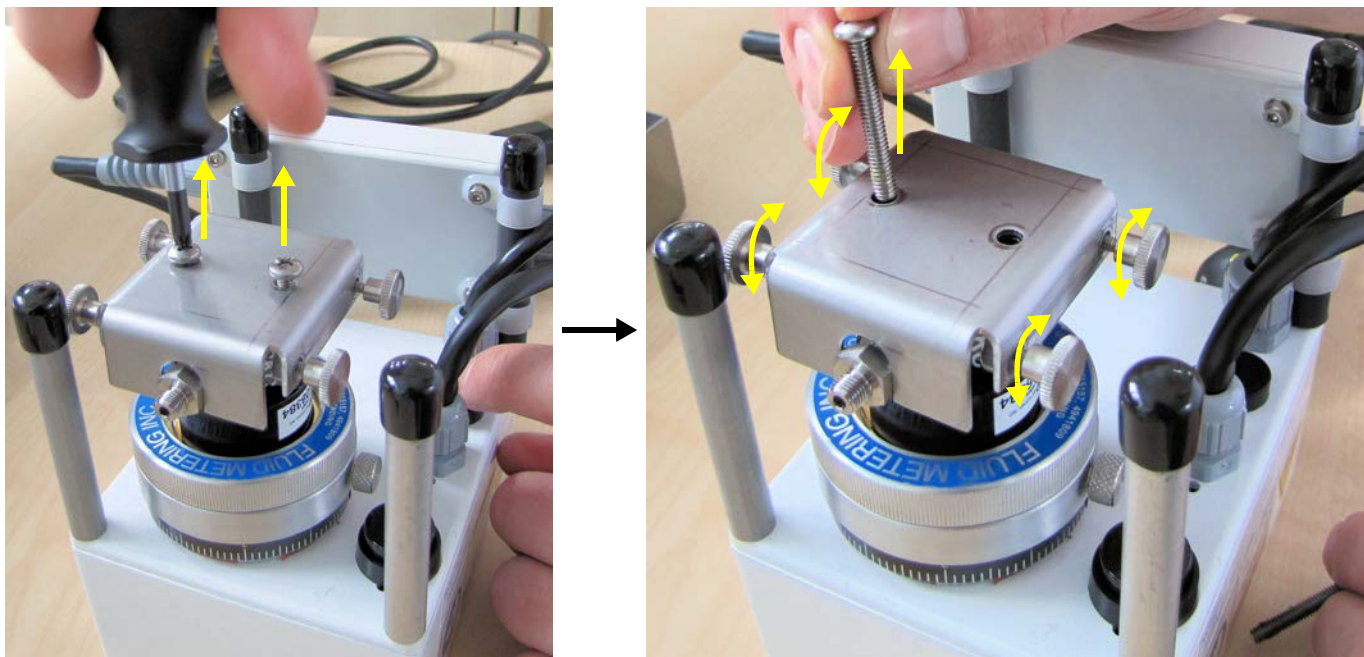


Figure 9-19. Removing both crosshead screws and loosening four knurled screws a bit

8. Carefully pull the bracket upwards. Take the bracket away together with the knurled screws still attached to it. See [Figure 9-20](#).

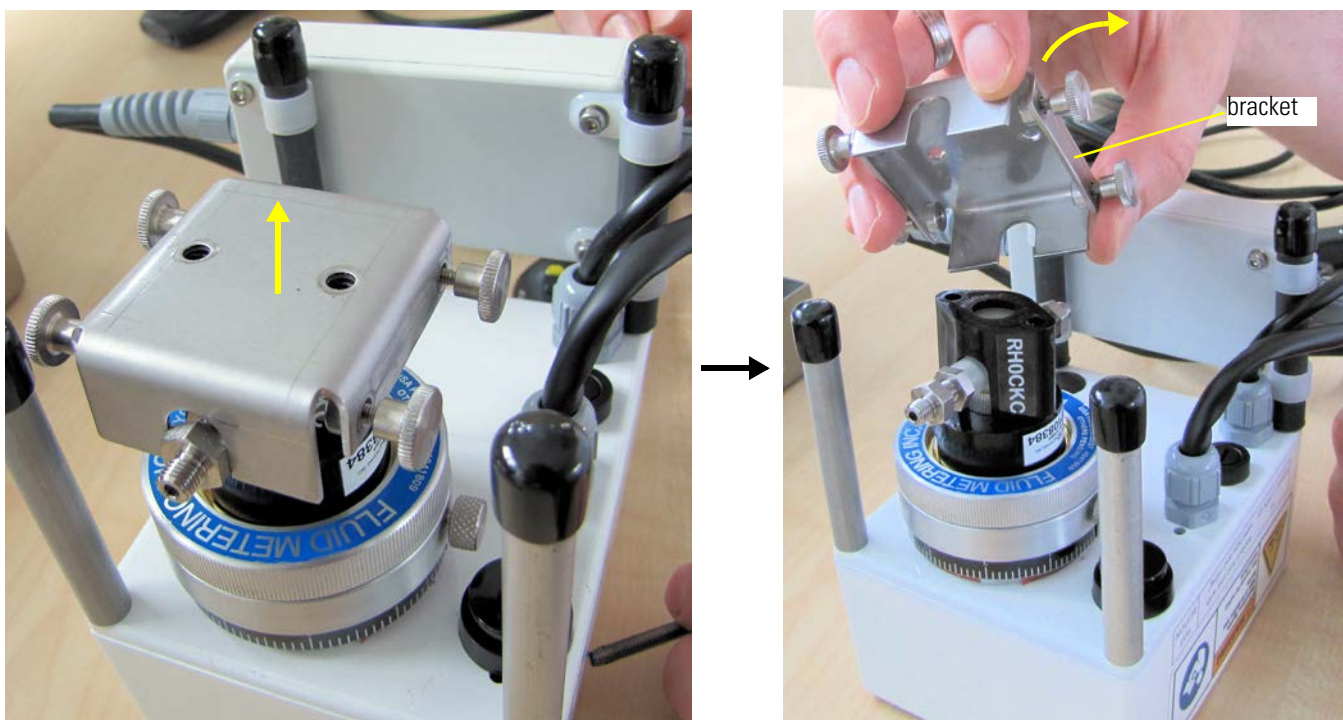


Figure 9-20. Taking bracket away together with knurled screws still attached to it

Carbonate Option

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9. As [Figure 9-21](#) shows, take away the protective cap **1** of the acid pump head **2**.



Labeled Components: 1=protective cap, 2=acid pump head

Figure 9-21. Taking away protective cap to access acid pump head

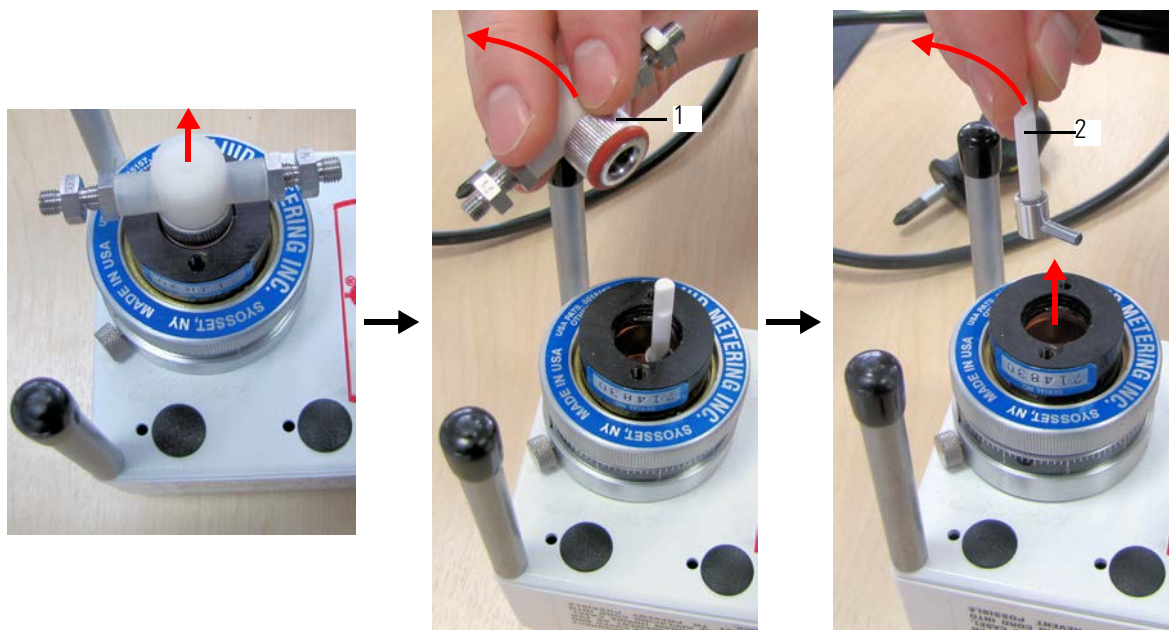
The acid pump head becomes accessible.

10. As [Figure 9-22](#) shows:

- a. Carefully pull the acid pump head **1** outwards as a whole.

The plunger **2** becomes accessible.

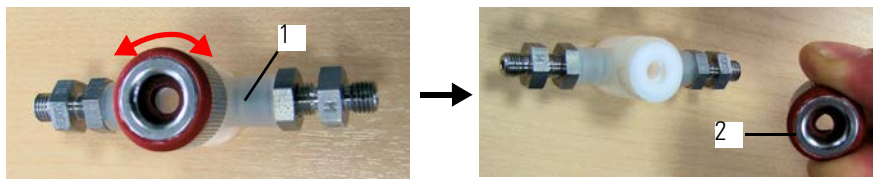
- b. Only if you must replace the plunger (that is, in case of massive mechanical wear), pull it outwards.



Labeled Components: 1=acid pump head, 2=plunger

Figure 9-22. Pulling acid pump head and plunger outwards

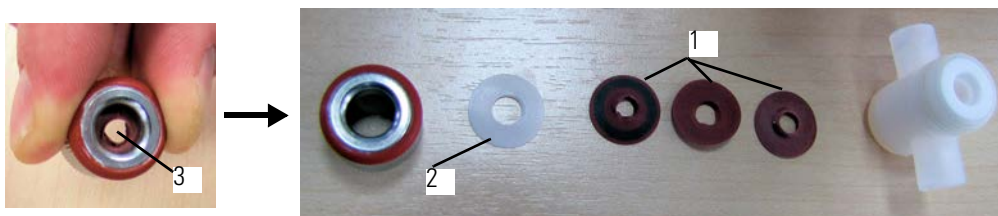
11. Disassemble the acid pump head (**1** in [Figure 9-23](#)) as follows:
 - a. Unscrew the cap (**2** in [Figure 9-23](#)) of the acid pump head.



Labeled Components: 1=acid pump head, 2=cap

Figure 9-23. Unscrewing cap of acid pump head

- b. As [Figure 9-24](#) shows, pull the three fluorocarbon seals **1** and the white Teflon™ washer **2** out of the cap **3** by using tweezers.



Labeled Components: 1=fluorocarbon seal, 2=white Teflon™ washer, 3=cap

Figure 9-24. Pulling three fluorocarbon seals and washer out of cap

- c. Unscrew the SERTO™ fitting on each of the two sides of the T-piece. Use a 10 mm open-ended spanner. See [Figure 9-25](#).

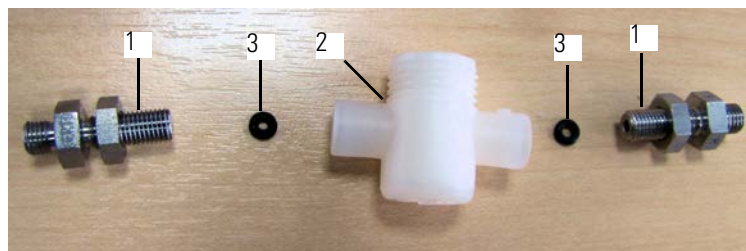


Figure 9-25. Unscrewing SERTO fitting on each side of T-piece

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- d. Remove the Viton™ O-ring on each side of the T-piece. See [Figure 9-26](#).



Labeled Components: 1=SERTO fitting, 2=T-piece, 3=Viton O-ring

Figure 9-26. SERTO fittings, Viton O-rings and T-piece of acid pump head

[Figure 9-27](#) shows the parts that result from dismantling the acid pump head.



Figure 9-27. Parts resulting from dismantling of acid pump head

Step 2: Reassembling the New Acid Pump Head

❖ To reassemble the new acid pump head

1. Be equipped with a new white Teflon™ washer and new fluorocarbon seals, if the ones in use should have been damaged.
2. If you do not use new parts, clean the ones in use (cap, plunger, T-piece, fluorocarbon seals, white Teflon washer). See [“To clean the acid pump head and other parts”](#) on [page 9-17](#).
3. Slide the cap over the plunger.
4. As the white Teflon washer must be placed lowermost, slide it over the plunger, deepest into the cap. See arrows in [Figure 9-28](#).

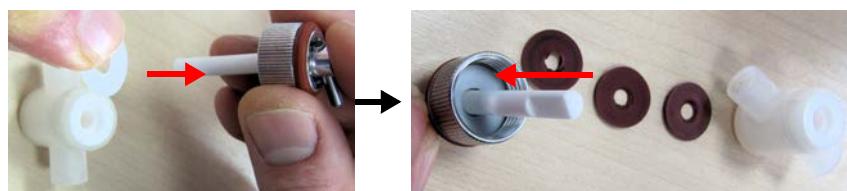


Figure 9-28. Sliding white Teflon washer over plunger, down into cap

5. Arrange the first fluorocarbon seal properly upon the plunger as shown in [Figure 9-29](#):
 - a. Carefully turn the lip of the seal over the plunger.
 - b. To widen the seal, gently push the seal a bit down the plunger (lip side **last**).
 - c. Carefully remove the seal from the plunger.
 - d. Reverse the seal by 180 °.
 - e. Carefully turn the lip of the seal over the plunger again (lip side **first**).
 - f. Gently push the seal as wide as possible down the plunger.
6. Arrange the second fluorocarbon seal properly upon the plunger (directly with lip side **first**). See [Figure 9-29](#).
7. Arrange the third fluorocarbon seal properly upon the plunger (directly with lip side **last**). See [Figure 9-29](#).

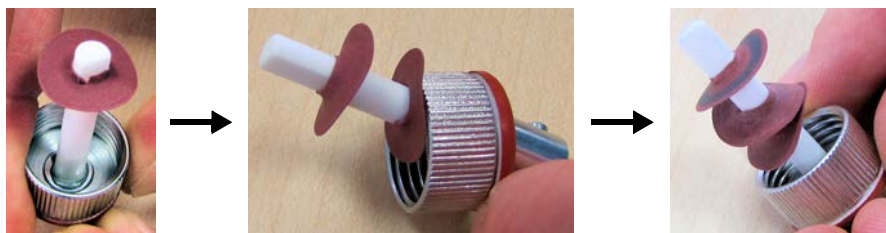


Figure 9-29. Arranging three fluorocarbon seals properly upon plunger

8. Carefully and slowly push the three fluorocarbon seals by a “rotating movement” towards the cap.
9. Screw the arrangement consisting of plunger, seals and cap onto the T-piece. See [Figure 9-30](#).

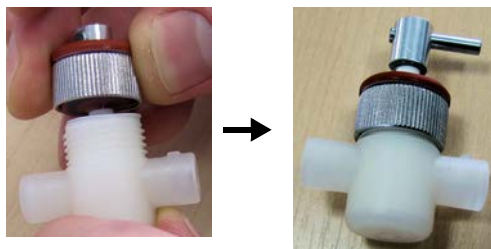


Figure 9-30. Screwing arrangement onto T-piece

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Maintenance of the Carbonate Option

10. Insert one Viton™ O-ring into each side of the T-piece. See [Figure 9-31](#).



Figure 9-31. Inserting one Viton O-ring into each side of T-piece

11. Very carefully screw one SERTO fitting with its **long** end into each side of the T-piece using a 10 mm open-ended spanner. See [Figure 9-32](#).

NOTICE

Exert only a very small force with the spanner to prevent damage to the sensitive thread of the T-piece, which is made of plastic.

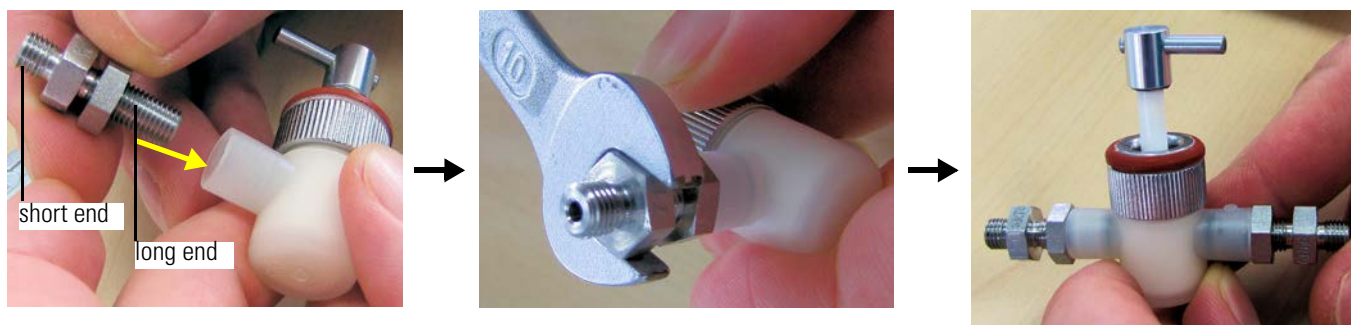


Figure 9-32. Screwing one SERTO fitting with its long end into each side of T-piece

The acid pump head is reassembled.

12. As shown by the arrows in [Figure 9-33](#):
 - a. Insert the metallic part of the plunger into the opening of the acid pump. See [Figure 9-33](#), left.

- b. Let the metallic bolt of the plunger carefully slide into the lateral opening inside the acid pump. See [Figure 9-33](#), right.

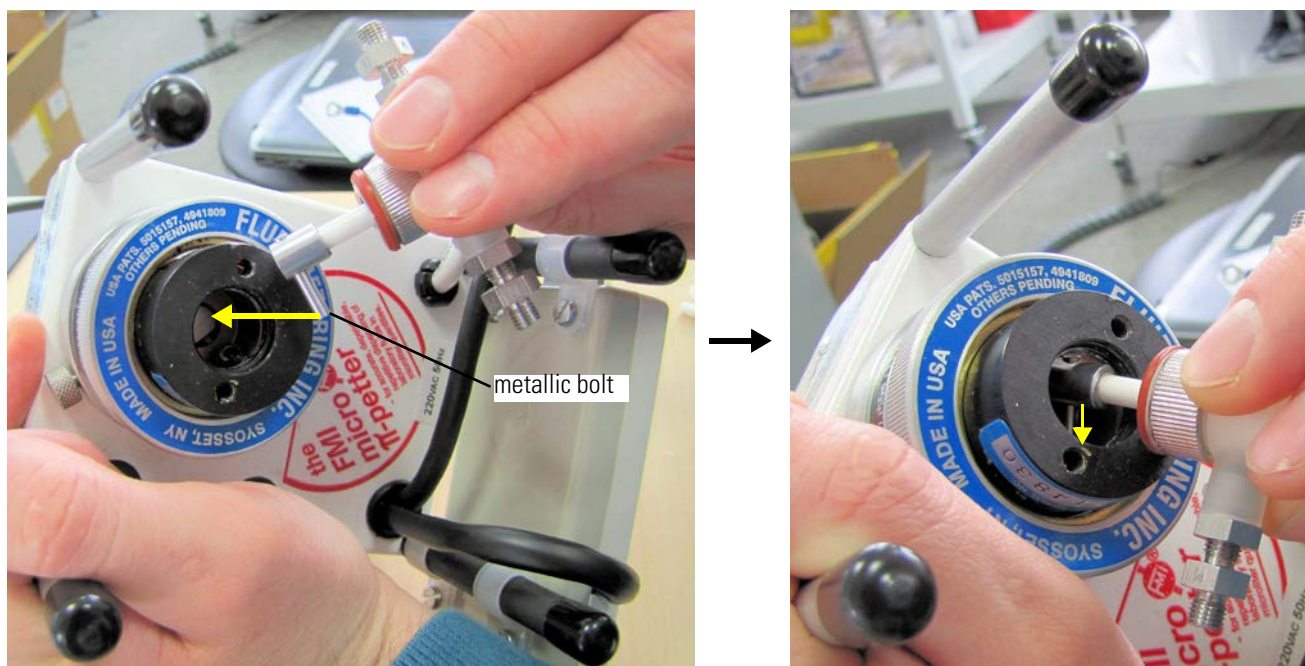


Figure 9-33. Sliding bolt of plunger into lateral opening inside acid pump

13. As [Figure 9-34](#) shows:

- a. Carefully push the acid pump head into the opening of the acid pump. See [Figure 9-34](#), left.
- b. Attach the protective cap upon the acid pump head. See [Figure 9-34](#), right.

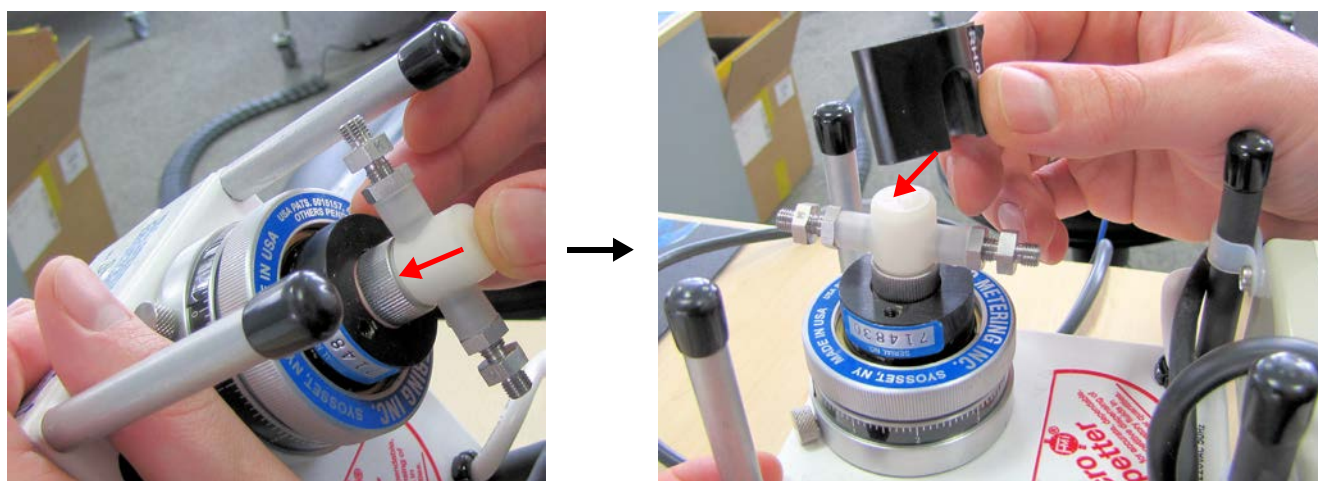


Figure 9-34. Attaching acid pump head to acid pump

14. Check as follows whether the plunger is moving properly within the acid pump:
 - a. Connect the power plug of the acid pump to the socket at the rear panel of the mass spectrometer.
 - b. Turn the acid pump so that you can look upon the bottom of the T-piece. See [Figure 9-35](#).

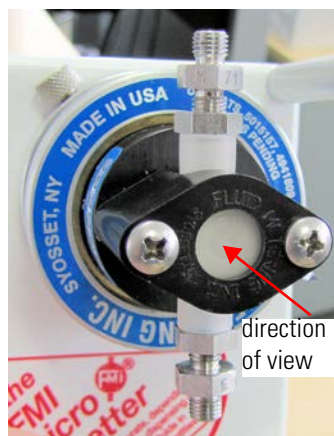


Figure 9-35. Turning acid pump to check movement of plunger

- c. Press the button at the housing of the acid pump.

The acid pump executes one stroke. The movement of the plunger can be seen from outside.

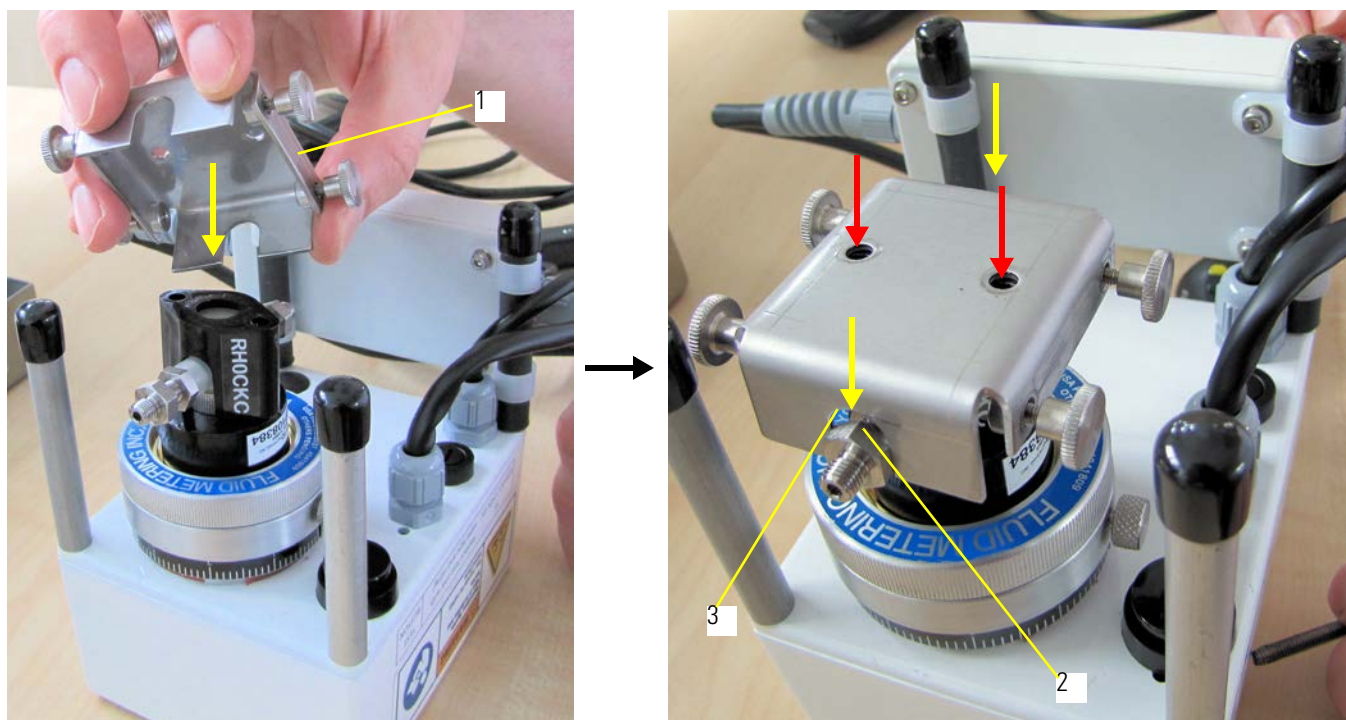
NOTICE

To avoid wear, the moving plunger must not touch the bottom of the T-piece (“housing”) or even bump against it. See [Figure 9-35](#).

- If the moving plunger does not touch the bottom of the T-piece, adjust the acid pump. See “[To adjust the acid pump](#)” on [page 9-10](#).
 - If the moving plunger touches the bottom of the T-piece or even bumps against it, contact your local service representative.
15. When the plunger is moving properly, carefully place the bracket centrally upon the protective cap (see [Figure 9-36](#)):
 - a. The two nuts must exactly fit into their cut-outs. Do not cant the bracket.

If the nuts do not exactly fit into their cut-outs, loosen or fasten the nuts a bit using an open-ended spanner.

- b. The holes of the protective cap underneath must coincide with the holes of the bracket.



Labeled Components: 1=bracket, 2=nut, 3=cut-out

Figure 9-36. Placing bracket exactly upon protective cap

16. Insert both crosshead screws into the coinciding holes. Fix them finger-tight using a cross-tip screwdriver. See [Figure 9-37](#).

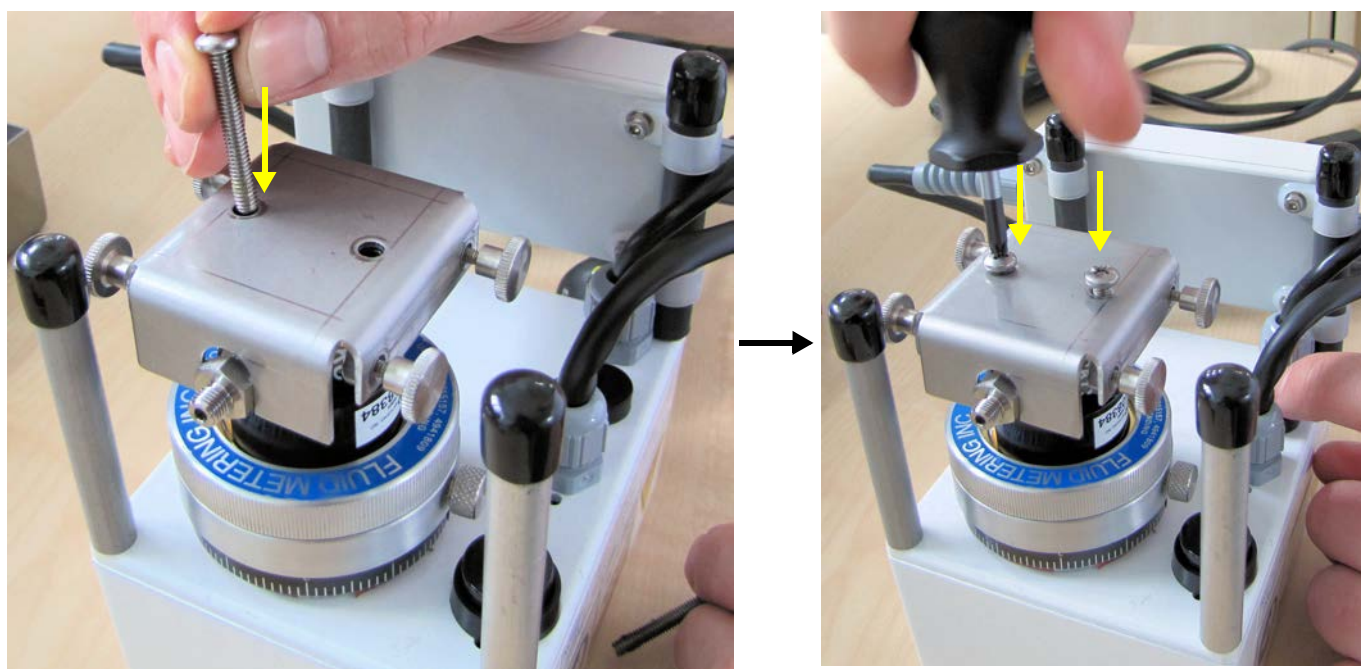
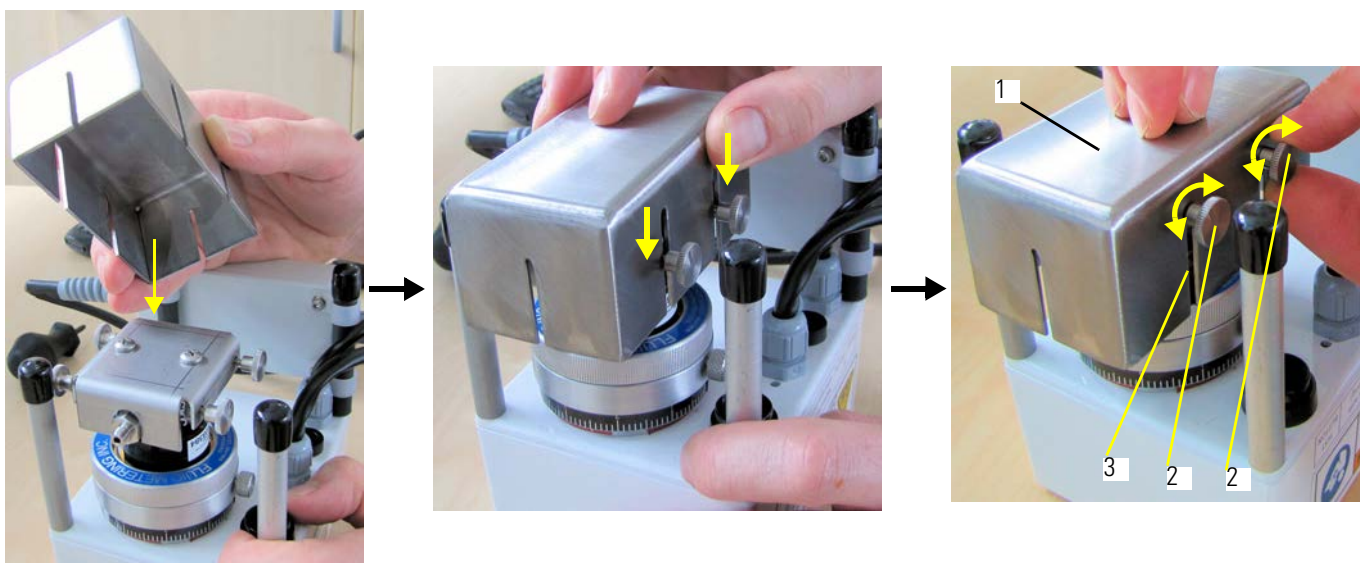


Figure 9-37. Fixing both crosshead screws

Carbonate Option

Maintenance of the Carbonate Option

17. As [Figure 9-38](#) shows, attach the protective cover **1** as follows:
 - a. Loosen the four knurled screws **2** wide enough (but do not remove them entirely).
 - b. Place the protective cover exactly straight upon the bracket. The four knurled screws must fit into the four lateral slots **3** of the protective cover (“snapping in”).
 - c. Carefully push the protective cover downwards upon the bracket as far as it will go.
 - d. Manually turn in the four knurled screws finger-tight.



Labeled Components: 1=protective cover, 2=knurled screw, 3=lateral slot

Figure 9-38. Attaching protective cover

The protective cover is fixed in place. The acid pump is reassembled. See [Figure 9-39](#).



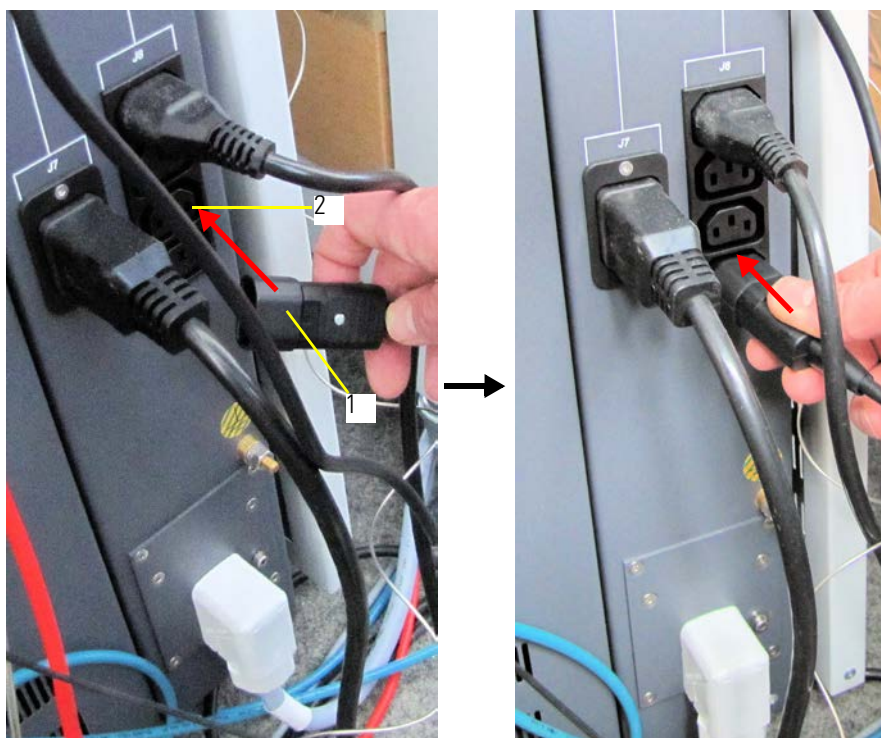
Figure 9-39. Acid pump reassembled

18. Connect the capillary that leads to the acid reservoir to the acid pump.
19. Connect the capillary that leads to the acid needle to the acid pump.
20. Connect the acid pump to the mass spectrometer as follows:

- In case of a **DELTA Q Series MS**:



As [Figure 9-40](#) shows, always insert the power plug **1** (“power cable”) of the acid pump only into the socket **2** at the rear panel of the DELTA Q Series MS (not to a wall socket).



Labeled Components: 1=power plug of acid pump, 2=socket at rear panel of DELTA Q Series MS

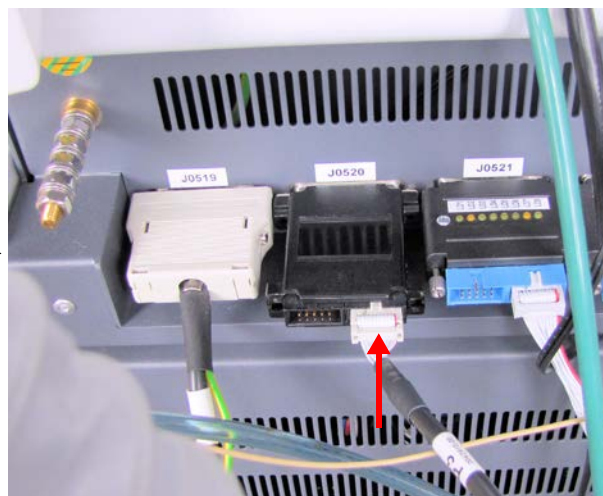
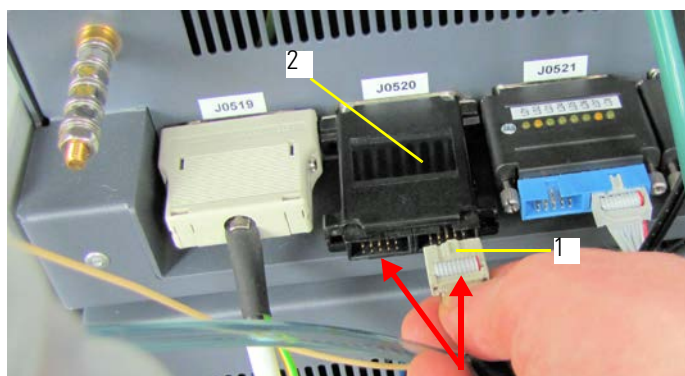
Figure 9-40. Inserting power plug of acid pump into socket at rear panel of DELTA Q Series MS

- a. As [Figure 9-41](#) shows, insert the ribbon cable plug (10 pole; “data cable”) **1** of the acid pump into an arbitrary connector of the pnm adapter **2** at the rear panel of the DELTA Q Series MS.

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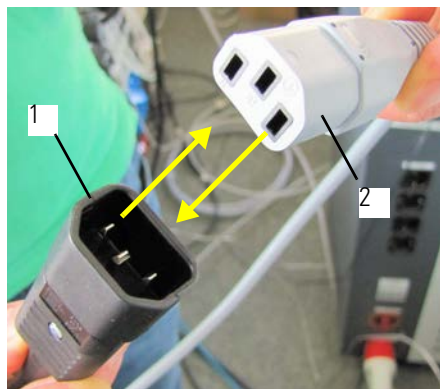
To ensure correct insertion, consider the “nose” of the ribbon cable plug.



Labeled Components: 1=ribbon cable plug of acid pump, 2=pnm adapter

Figure 9-41. Inserting ribbon cable plug of acid pump into pnm adapter at rear panel of DELTA Q Series MS

- In case of a **253 Plus** Series MS:
 - a. As [Figure 9-42](#) shows, connect the power plug **1** (“power cable”) of the acid pump to the female end **2** of the extension cable.

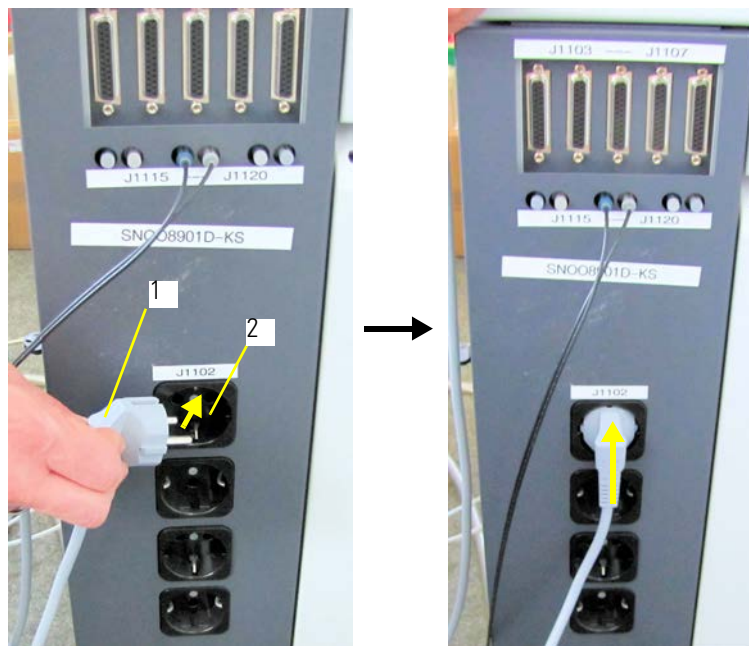


Labeled Components: 1=power plug of acid pump, 2=female end of extension cable

Figure 9-42. Connecting power plug of acid pump to female end of extension cable



As [Figure 9-43](#) shows, always insert the male end **1** of the extension cable only into the socket **2** at the rear panel of the 253 Plus MS or the MAT 253 MS (not to a wall socket).

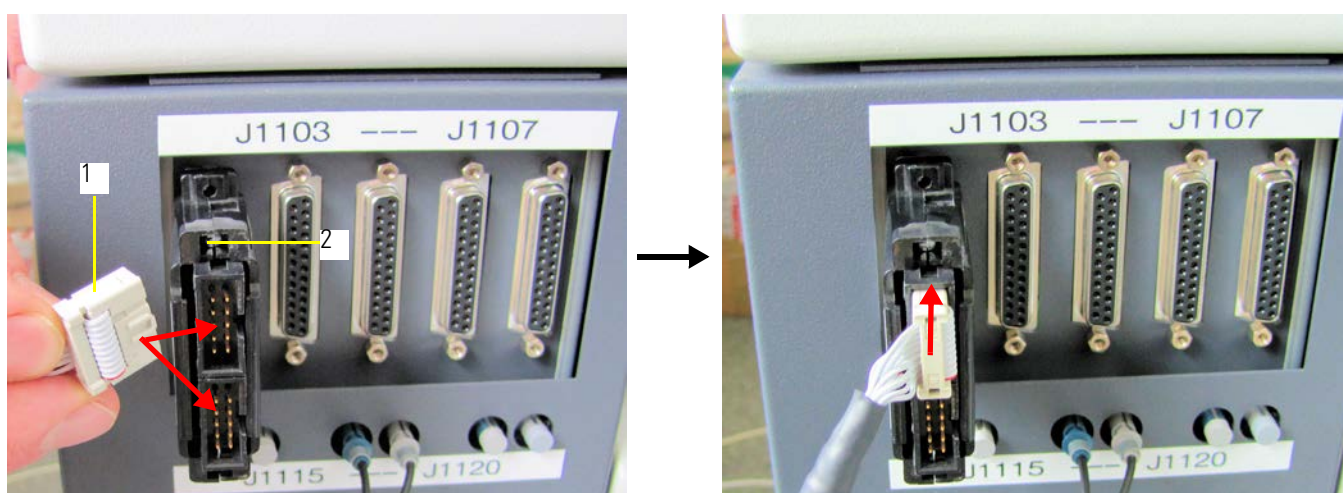


Labeled Components: 1=male end of extension cable, 2=socket at rear panel of 253 Plus MS or MAT 253 MS

Figure 9-43. Inserting male end of extension cable into socket at rear panel of 253 Plus MS or MAT 253 MS

- b. As [Figure 9-44](#) shows, insert the ribbon cable plug **1** of the acid pump (“data cable”) into an arbitrary connector of the pnm adapter **2** at the rear panel of the 253 Plus MS or the MAT 253 MS.

To ensure correct insertion, consider the “nose” of the ribbon cable plug.



Labeled Components: 1=ribbon cable plug of acid pump, 2=pnm adapter

Figure 9-44. Inserting ribbon cable plug of acid pump into pnm adapter at rear panel of 253 Plus MS or MAT 253 MS

Analyzing Carbonates

To measure carbonates, you need the carbonate option. This option contains special borosilicate vials suitable for carbonate analysis. The soda glass vials delivered with the basic GasBench Plus package are not suitable for carbonate analysis. Furthermore, the option contains blue Exetainer™ caps to be used with a thermostatted tray.

In this section, simultaneous measurement of $^{13}\text{C}/^{12}\text{C}$ and $^{18}\text{O}/^{16}\text{O}$ isotopic ratios in calcite, aragonite (that is, mainly CaCO_3) or dolomite (that is, MgCO_3) will be covered. The latter is subject to a lot of discussion, and results should be discussed carefully. The idea is to react the carbonate species with phosphoric acid to yield CO_2 that carries an image of the isotopic value of the carbonate ion CO_3^{2-} .

Dual Needle Setup

The dual needle setup allows acid dosing to a sample while measuring another one. See [Figure 9-45](#), [Table 9-2](#) and “Flush Needle” on [page 3-16](#). While the right needle transports acid to a bottle filled with helium, the left needle takes sample gas from the headspace.

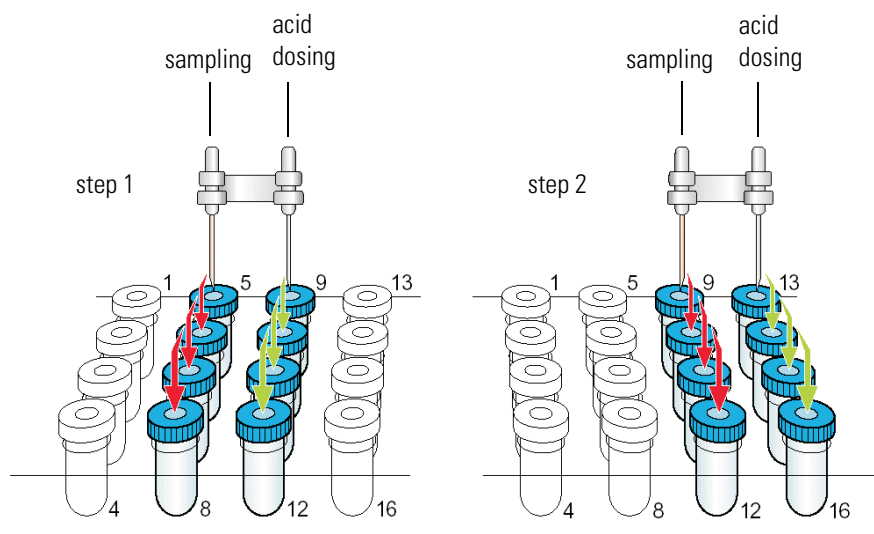


Figure 9-45. Defining the sequence - dual needle setup with Qtegra workflow mode

Carbonates in Brief

For optimized carbonate reaction and best performance results, carbonate sampling, the measurement procedure and contaminant-free, best performed chromatograms are explained in this section.

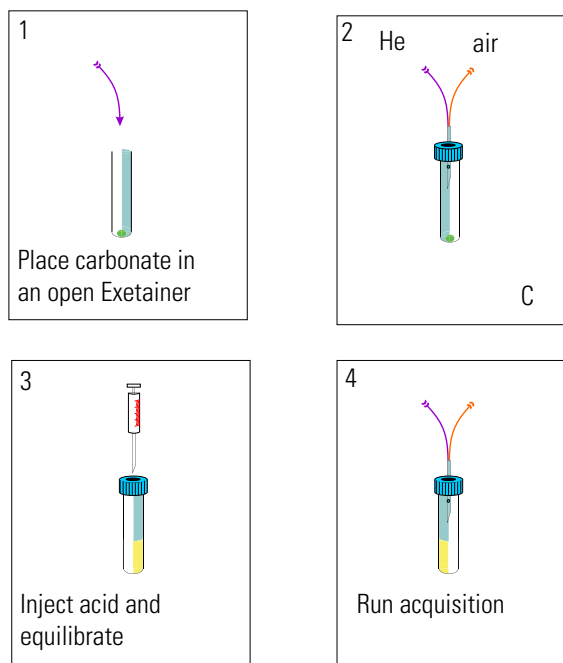


Figure 9-46. Sample preparation for carbonate measurements

❖ To perform a carbonate measurement

1. Heat the tray to 72 °C. This will speed up the reaction between the carbonates (that is mainly CaCO_3) and phosphoric acid (that is, H_3PO_4) and shortens the time required to reach isotopic equilibrium.
2. Place 50–600 μg of solid, carbonate-containing sample (dolomite, calcite, foraminifera, for example) into a clean sample vial.
3. Close the vial with a new cap and a new septum.
4. Place the vials within the tray.
5. Make sure that the rinsing/filling needle is properly mounted in the autosampler.
6. Depending on your flushing needle setup, either choose the flush or dual needle flush sequence. Select the appropriate line numbers and start the sequence. By default, the sequence is set up to flush each vial with a helium stream of 100 mL for 5 minutes.
7. Make sure that the sampling needle is properly mounted in the autosampler.

8. Make sure that the GasBench Plus device is absolutely free of contaminants (phosphoric acid and water in the fused silica capillaries).

Tip Thermo Fisher Scientific strongly recommends choosing a dual needle setup (that is sample needle plus acid needle) for fully automated measurement of carbonates. This ensures proper timing of the measurement. Mount the sampling needle on the left side of the dual needle holder of the autosampler.

9. Start the analysis sequence with a dual needle setup. See “Dual Needle Setup” on page 9-38. Use the Carbonates sequence. Select the appropriate lines.

The method used in connection with this sequence ensures that the following steps will take place:

- a. Dosage of H_3PO_4 using our automatic device. Reaction between the carbonate-containing sample and H_3PO_4 begins.¹ CO_2 will be released into the headspace.
- b. Waiting for about 1 hour for equilibration of the CO_2 .
- c. During measurement, helium enters the system, and a mixture of helium and CO_2 (as the sample gas) passes to the GasBench Plus device.

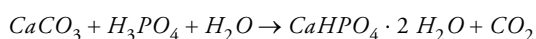
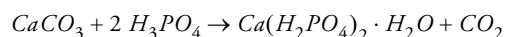
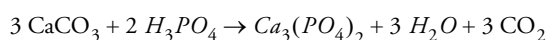
Tip The vials on the positions 1–9 are filled neither with carbonates nor with acid, but they will be flushed with helium. These vials are used as dummies for the sampling needle while the acid needle is dosing the phosphoric acid in vials 9–16.

The analysis pathway follows the positions 1–4, 9–12, 17–20 and so on. This defines a reaction time four times as large as the acquisition time for a single sample. See Figure 9-45, Table 9-2, and Figure 3-19.

If the system is operating properly, you should receive a result chromatogram for each sample.

¹ Formation of carbon dioxide from limestone

When dropping water-free phosphoric acid upon limestone (that is calcite or aragonite), phosphates of calcium, carbon dioxide and water will be formed. Possible reactions are:



Note that water is formed in each step.

Decreasing peak height indicates proper transport of the sample/helium mixture.

Tip We use the term “chromatogram” even though it may not be a chromatogram in a narrower sense. However, one obtains ten or less repetitions of the same sample, that is of the same small chromatogram.

Carbonate Isotope Analysis Workflow

TriPlus RSH and Rack Setup

The TriPlus RSH rack set up can be set up for several different modes.

1. thermostatted racks must be set in the continuous flow method and must be installed in nature.
 - a. Slot1 only
 - b. Slot2 only; but not with non-thermostatted rack taught at the left and a thermostatted rack at the Slot2 as this is a configuration that is not allowed by the RSH firmware.
 - c. Slot1 & Slot2
2. Double needle flush and measure (left) & acid addition (right)
3. Single or double needle flush in a flush sequence and measurement in the LabBook & acid adding on the right.
4. Single needle flushing on the right and acid addition on the left in a flush sequence.

Each tool needle set-up must be defined in the autosampler firmware in the tool section. See [“Standard RSH Installation and Configuration Check”](#) on page 5-19. Qtegra reads out the defined tool needle set up automatically from the RSH tool settings. The user must define for the rack, which tool is in use either for the LabBook or the flush or acid addition sequence. See [“Stand-Alone Flushing or Automated Flushing and Acid Addition \(“Sample Preparation”\)”](#) on page 7-20. LabBook is defined as being used for automated flushing but always sample measurement will be involved in the workflow of a LabBook.

LabBook for the Carbonate Isotopic Application Workflow

There are different ways to run a carbonate workflow.

Purge & Acid addition

1. Helium purge in an extra flush sequence under diagnosis, i.e. go from sample 1-96 with either a single or a double needle installed in the tool (see “[Stand-Alone Flushing or Automated Flushing and Acid Addition \(“Sample Preparation”\)](#)” on page 7-20).
2. Helium purge right and acid addition left in an extra flush sequence under diagnosis, i.e. go from sample 1-96.
3. Acid addition only with one needle in any position of the rack.

LabBook continuous flow peripheral parameter sets for certain process preparations

Certain preparation processes can be done automatically with Qtegra within one LabBook under the peripheral parameters (see [Figure 9-47](#)). Tool changes will be switched or requested automatically.

1. The whole preparation and measurement (see [Figure 9-48](#)) is done in one LabBook with
 - a. Flush of all samples. Flush can be done before or during the LabBook is running (see [Figure 9-47](#), [Figure 9-48](#)).
 - b. Acid addition during the sample measurement (drop numbers defined under acid pump in the GasBench dashboard instrument)
 - c. Measurement of samples by sample pulsing and the same time referencing.
 - d. Measurement with sample if the sample response is too high (see [Figure 9-47](#)), e.g. for soil or DIC samples with an unknown carbonate concentration.

Qtegra will take the information from the installed Tool set up under the TriPlus RSH configuration in the LabBook and does a flush processing of all sample before a sample list. Indicated by an empty chromatogram screen and a green arrow without movement over the next sample number in the LabBook. If flushing during sample run is selected, Qtegra will use the flush time and will not equilibrate after the run.

The acid will be added independently in a vial, which has been set 8 samples before the sample being measured.

Deviations for the process if sample have been processed according to the purge & acid addition (see [Figure 9-48](#)).

To 1. Disable the flush mode (Flush set to *Off*)

To 2. Disable acid addition (Acid Dosing set to *Off*)

To 3. Disable flush mode and acid addition

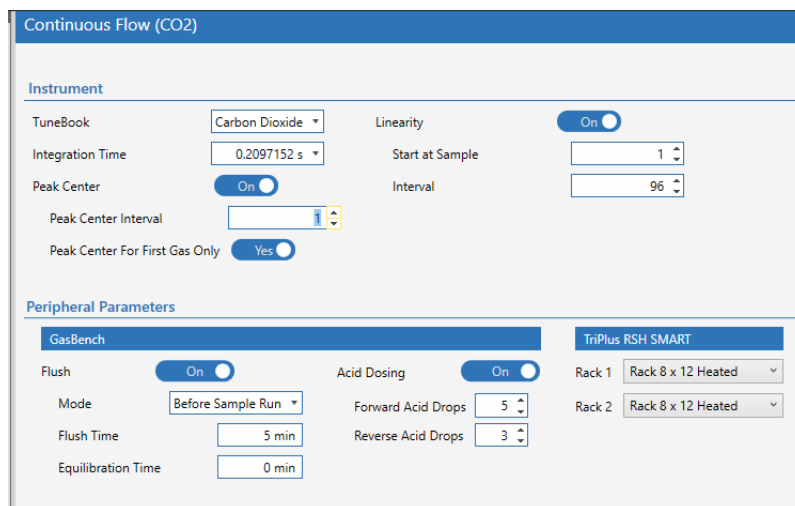


Figure 9-47. Peripheral preparation for carbonates, e.g., with purge (flush) and acid dosing as well as sample pulsing and sample dilution in one LabBook

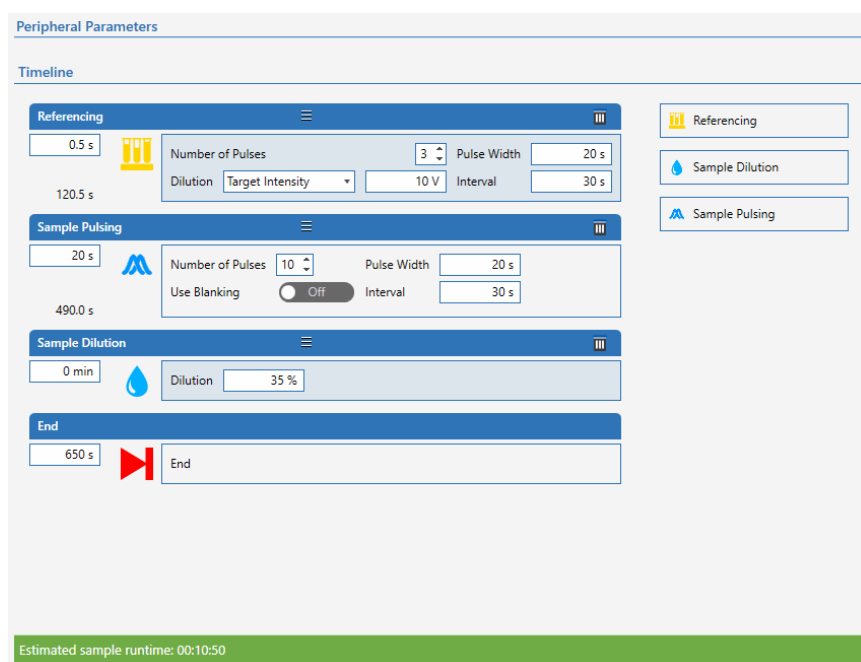


Figure 9-48. Carbonate workflow with acid dosing and flushing

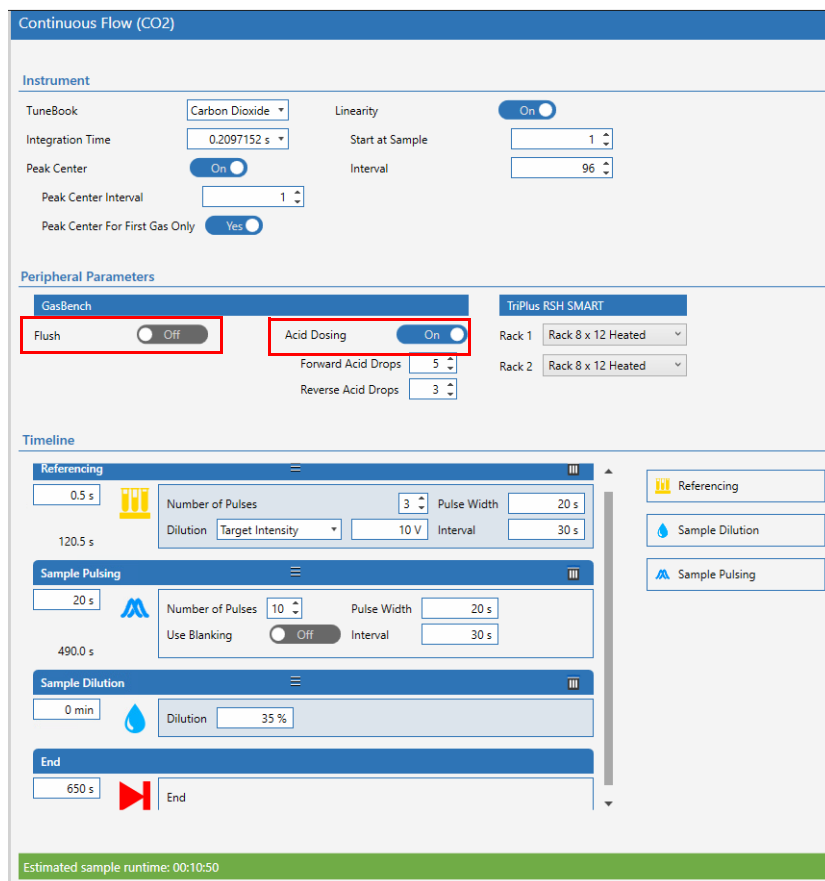


Figure 9-49. Carbonate workflow with acid dosing but without flushing

Setting up Carbonate isotope standards and Delta Standard



Refer to the *DELTA Q Operating Manual* for the generic standardization procedure in Qtegra.

Set up the standardization including a set of usable solid carbonate standards (see [Figure 9-50](#) and including calibration gas standard (see [Figure 9-51](#)) The calibration standard will be used to calibrate the sample peaks to this standard for internal normalization.

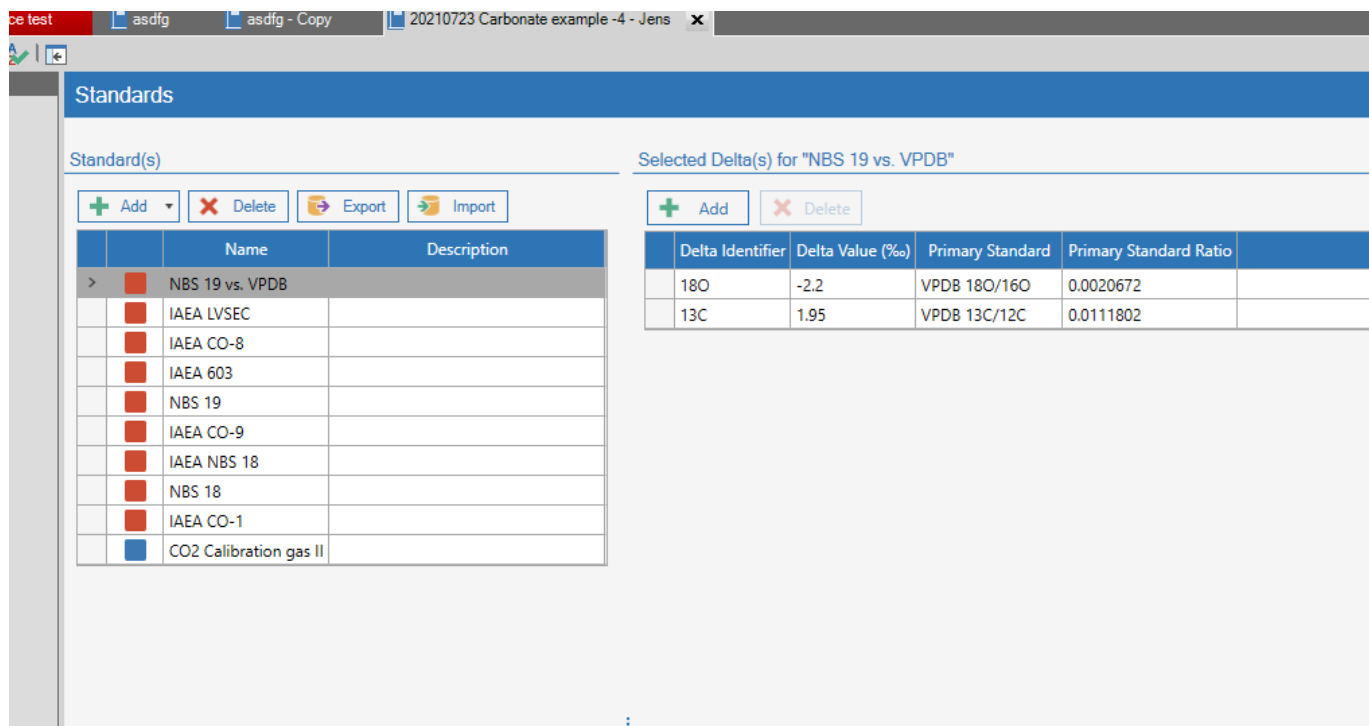


Figure 9-50. Carbonate BSIA standards (red color) including a reference gas (calibration gas, blue color)

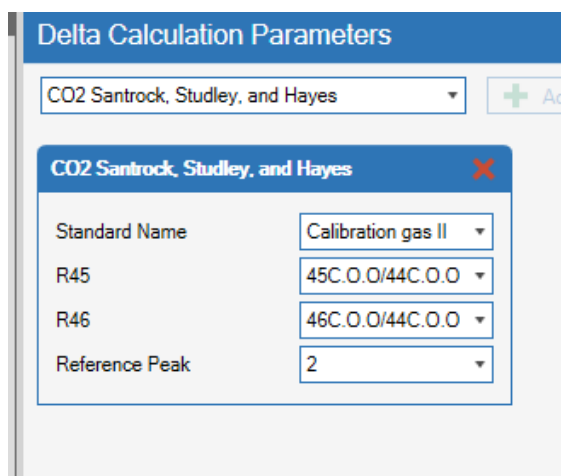


Figure 9-51. Delta calibration gas used for calibrating the sample peaks. Different calibrations can be taken for 170 corrections. See *DELTA Q Operating Manual*.

For the Sample List, a standard for international standardization can be used, e.g. NBS 19. The standard is only used as reference to the defined value (e.g. NBS 19 vs. VPDB, see [Figure 9-52](#)) if defined in the Reference column. If a different scale, e.g. SMOW scale, shall be used, the same standard can be recalculated against the SMOW scale but with different values and assignments in the BSIA standard list (see *DELTA Q*

Carbonate Option

Analyzing Carbonates

Operating Manual). For the scale contraction, a different standard, e.g. LVSEC, can be used only on the same scale (e.g. SMOW or PDB scale) as the primary normalization standard (e.g. NBS 19 in this example).

	Label	Status	Comment	Analysis No.	Action	Rack	Vial	Evaluate	Sample Type	Reference
1	acid addition	●	<Comment>	N/A	Prep	1	1	☑	Unknown	
2	acid addition	●	<Comment>	N/A	Prep	1	2	☑	Unknown	
3	acid addition	●	<Comment>	N/A	Prep	1	3	☑	Unknown	
4	acid addition	●	<Comment>	N/A	Prep	1	4	☑	Unknown	
5	IAEA-603	●	<Comment>	N/A	Prep + Measure	1	9	☑	Delta Standard (BSIA)	IAEA-603 BSIA
6	IAEA-603	●	<Comment>	N/A	Prep + Measure	1	10	☑	Delta Standard (BSIA)	IAEA-603 BSIA
7	IAEA-603	●	<Comment>	N/A	Prep + Measure	1	11	☑	Delta Standard (BSIA)	IAEA-603 BSIA
8	Carbonate Sample 1	●	<Comment>	N/A	Prep + Measure	1	12	☑	Unknown	
9	Carbonate Sample 1	●	<Comment>	N/A	Prep + Measure	1	25	☑	Unknown	
10	Carbonate Sample 1	●	<Comment>	N/A	Prep + Measure	1	26	☑	Unknown	
11	Carbonate Sample 2	●	<Comment>	N/A	Prep + Measure	1	27	☑	Unknown	
12	Carbonate Sample 2	●	<Comment>	N/A	Prep + Measure	1	28	☑	Unknown	
13	Carbonate Sample 2	●	<Comment>	N/A	Prep + Measure	1	31	☑	Unknown	
14	Carbonate Sample 3	●	<Comment>	N/A	Prep + Measure	1	32	☑	Unknown	
15	Carbonate Sample 3	●	<Comment>	N/A	Prep + Measure	1	33	☑	Unknown	
16	Carbonate Sample 3	●	<Comment>	N/A	Prep + Measure	1	34	☑	Unknown	
17	NBS-18	●	<Comment>	N/A	Prep + Measure	1	41	☑	QC Standard	NBS-18 BSIA
18	NBS-18	●	<Comment>	N/A	Prep + Measure	1	42	☑	QC Standard	NBS-18 BSIA
19	NBS-18	●	<Comment>	N/A	Prep + Measure	1	43	☑	QC Standard	NBS-18 BSIA
20	Carbonate Sample 4	●	<Comment>	N/A	Prep + Measure	1	44	☑	Unknown	
21	Carbonate Sample 4	●	<Comment>	N/A	Prep + Measure	1	49	☑	Unknown	
22	Carbonate Sample 4	●	<Comment>	N/A	Prep + Measure	1	50	☑	Unknown	
23	Carbonate Sample 5	●	<Comment>	N/A	Prep + Measure	1	51	☐	Unknown	
24	Carbonate Sample 5	●	<Comment>	N/A	Prep + Measure	1	52	☑	Unknown	
25	Carbonate Sample 5	●	<Comment>	N/A	Measure	1	57	☑	Unknown	
26	LVSEC	●	<Comment>	N/A	Measure	1	58	☑	Delta Standard (BSIA)	LVSEC BSIA
27	LVSEC	●	<Comment>	N/A	Measure	1	59	☑	Delta Standard (BSIA)	LVSEC BSIA
28	LVSEC	●	<Comment>	N/A	Measure	1	60	☑	Delta Standard (BSIA)	LVSEC BSIA

Figure 9-52. Sample list with international normalization, samples, QC standards, and slope correction standards each in triplicates

For acid addition during sampling, the first rows must only add the acid to the sample (*Prep*). At the action Prep the Valco valve stays on *Load*. Add the acid and measure at the same time (*Pre + Measure*) and only measure without any acid addition (*Measure*, see [Figure 9-53](#)).

	Label	Status	Comment	Analysis No.	Action	Rack	Vial	Evaluate	Sample Type	Reference
1	acid addition	●	<Comment>	N/A	Prep	1	1	☑	Unknown	
2	acid addition	●	<Comment>	N/A	Prep	1	1	☑	Unknown	
3	acid addition and measure	●	<Comment>	N/A	Prep + Measure	1	1	☑	Unknown	
4	acid addition and measure	●	<Comment>	N/A	Prep + Measure	1	1	☑	Unknown	
5	Measure only	●	<Comment>	N/A	Measure	1	1	☑	Unknown	
6	Measure only	●	<Comment>	N/A	Measure	1	1	☑	Unknown	

Figure 9-53. Measurement with acid addition

Sample preparation for a carbonate run

Sample must be pre-flushed.

- In the first two rows of the sample list, acid will be added,

- third and fourth row: acid and reacted sample will be measured,
- fifth and sixth row: only prepared sample will be measured.

Sampling needle will be in the left and the acid needle in the right needle connection port on the tool.

Setting up a Carbonate Sample List

In the carbonate sample list standards, a normalization standard vs. PDB or SMOW scale and samples are defined.



Refer to “Reference and intercomparison materials for stable isotopes of light elements.” In: IAEA-TECDOC-825, IAEA, ed., Vienna, 1995. Also refer to Nelson, S.T.: A simple, practical methodology for routine VSMOW/SLAP normalization of water samples analyzed by continuous flow methods. *Rapid Communications in Mass Spectrometry* **14**:1044-1046 (2000). John Wiley & Sons Ltd.

A quality control standard can also be included and defined as quality control standard (see [Figure 9-54](#)).

NOTICE

Qtegra will automatically define the flush mode, i.e. either single needle or double needle flush. Consequently, by defining the positions Qtegra will automatically run the flush as the needle mode is defined in the TriPlus RSH settings under the Dashboard. Flushing will be defined by enabling flushing in the continuous flow method.

Carbonate Option

Analyzing Carbonates

	Label	Status	Comment	Analysis No.	Action	Rack	Vial	Evaluate	Sample Type	Reference
1	acid addition	●	<Comment>	N/A	Prep		1	<input checked="" type="checkbox"/>	Unknown	
2	acid addition	●	<Comment>	N/A	Prep		1	<input checked="" type="checkbox"/>	Unknown	
3	acid addition	●	<Comment>	N/A	Prep		1	<input checked="" type="checkbox"/>	Unknown	
4	acid addition	●	<Comment>	N/A	Prep		1	<input checked="" type="checkbox"/>	Unknown	
5	IAEA-603	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Delta Standard (BSIA)	IAEA-603 BSIA
6	IAEA-603	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Delta Standard (BSIA)	IAEA-603 BSIA
7	IAEA-603	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Delta Standard (BSIA)	IAEA-603 BSIA
8	Carbonate Sample 1	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Unknown	
9	Carbonate Sample 1	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Unknown	
10	Carbonate Sample 1	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Unknown	
11	Carbonate Sample 2	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Unknown	
12	Carbonate Sample 2	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Unknown	
13	Carbonate Sample 2	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Unknown	
14	Carbonate Sample 3	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Unknown	
15	Carbonate Sample 3	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Unknown	
16	Carbonate Sample 3	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Unknown	
17	NBS-18	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	QC Standard	NBS-18 BSIA
18	NBS-18	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	QC Standard	NBS-18 BSIA
19	NBS-18	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	QC Standard	NBS-18 BSIA
20	Carbonate Sample 4	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Unknown	
21	Carbonate Sample 4	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Unknown	
22	Carbonate Sample 4	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Unknown	
23	Carbonate Sample 5	●	<Comment>	N/A	Prep + Measure		1	<input type="checkbox"/>	Unknown	
24	Carbonate Sample 5	●	<Comment>	N/A	Prep + Measure		1	<input checked="" type="checkbox"/>	Unknown	
25	Carbonate Sample 5	●	<Comment>	N/A	Measure		1	<input checked="" type="checkbox"/>	Unknown	
26	LVSEC	●	<Comment>	N/A	Measure		1	<input checked="" type="checkbox"/>	Delta Standard (BSIA)	LVSEC BSIA
27	LVSEC	●	<Comment>	N/A	Measure		1	<input checked="" type="checkbox"/>	Delta Standard (BSIA)	LVSEC BSIA
28	LVSEC	●	<Comment>	N/A	Measure		1	<input checked="" type="checkbox"/>	Delta Standard (BSIA)	LVSEC BSIA

Figure 9-54. Sample list for carbonate runs highlighting different action modes

Normalization of Carbonate versus VPDB excluding or including scale contraction (slope correction or isotopic linearity correction)

Qtegra ISDS Software allows referencing of carbonate samples for international standardization and scale contraction correction.

Evaluation after a LabBook run by doing normalization and scale compression. In general, the user does not want to analyze the standards in the first run, consequently standards are in a different position as duplicates, triplicates or higher.

When a standard is moved to in direct position after the first scaling standard, the second standard will be used for scale contraction (see Figure 9-55). Move the scale compression standard to the normalization standard. Qtegra will automatically do the scale compression and determine the isotope ratio in the external reference data column. To evaluate replicate runs the external referencing must be done by using right-click on the data and do statistics on the data.

NOTICE

It is not recommended to start a non-measured LabBook if the normalization standard had been run a few hours before. Create a new LabBook and weight in new standards.

	Label	Status	Comment	Analysis No.	Action	Rack	Vial	Evaluate	Sample Type	Reference
1	acid addition		<Comment>	N/A	Prep		1	<input checked="" type="checkbox"/>	Unknown	
2	acid addition		<Comment>	N/A	Prep		2	<input checked="" type="checkbox"/>	Unknown	
3	acid addition		<Comment>	N/A	Prep		3	<input checked="" type="checkbox"/>	Unknown	
4	acid addition		<Comment>	N/A	Prep		4	<input checked="" type="checkbox"/>	Unknown	
5	IAEA-603		<Comment>	N/A	Prep + Measure		9	<input checked="" type="checkbox"/>	Delta Standard (BSIA)	IAEA-603 BSIA
6	IAEA-603		<Comment>	N/A	Prep + Measure		10	<input checked="" type="checkbox"/>	Delta Standard (BSIA)	IAEA-603 BSIA
7	IAEA-603		<Comment>	N/A	Prep + Measure		11	<input checked="" type="checkbox"/>	Delta Standard (BSIA)	IAEA-603 BSIA
8	Carbonate Sample 1		<Comment>	N/A	Prep + Measure		12	<input checked="" type="checkbox"/>	Unknown	
9	Carbonate Sample 1		<Comment>	N/A	Prep + Measure		25	<input checked="" type="checkbox"/>	Unknown	
10	Carbonate Sample 1		<Comment>	N/A	Prep + Measure		26	<input checked="" type="checkbox"/>	Unknown	
11	Carbonate Sample 2		<Comment>	N/A	Prep + Measure		27	<input checked="" type="checkbox"/>	Unknown	
12	Carbonate Sample 2		<Comment>	N/A	Prep + Measure		28	<input checked="" type="checkbox"/>	Unknown	
13	Carbonate Sample 2		<Comment>	N/A	Prep + Measure		31	<input checked="" type="checkbox"/>	Unknown	
14	Carbonate Sample 3		<Comment>	N/A	Prep + Measure		32	<input checked="" type="checkbox"/>	Unknown	
15	Carbonate Sample 3		<Comment>	N/A	Prep + Measure		33	<input checked="" type="checkbox"/>	Unknown	
16	Carbonate Sample 3		<Comment>	N/A	Prep + Measure		34	<input checked="" type="checkbox"/>	Unknown	
17	NBS-18		<Comment>	N/A	Prep + Measure		41	<input checked="" type="checkbox"/>	QC Standard	NBS-18 BSIA
18	NBS-18		<Comment>	N/A	Prep + Measure		42	<input checked="" type="checkbox"/>	QC Standard	NBS-18 BSIA
19	NBS-18		<Comment>	N/A	Prep + Measure		43	<input checked="" type="checkbox"/>	QC Standard	NBS-18 BSIA
20	Carbonate Sample 4		<Comment>	N/A	Prep + Measure		44	<input checked="" type="checkbox"/>	Unknown	
21	Carbonate Sample 4		<Comment>	N/A	Prep + Measure		49	<input checked="" type="checkbox"/>	Unknown	
22	Carbonate Sample 4		<Comment>	N/A	Prep + Measure		50	<input checked="" type="checkbox"/>	Unknown	
23	Carbonate Sample 5		<Comment>	N/A	Prep + Measure		51	<input type="checkbox"/>	Unknown	
24	Carbonate Sample 5		<Comment>	N/A	Prep + Measure		52	<input checked="" type="checkbox"/>	Unknown	
25	Carbonate Sample 5		<Comment>	N/A	Measure		57	<input checked="" type="checkbox"/>	Unknown	
26	LVSEC		<Comment>	N/A	Measure		58	<input checked="" type="checkbox"/>	Delta Standard (BSIA)	LVSEC BSIA
27	LVSEC		<Comment>	N/A	Measure		59	<input checked="" type="checkbox"/>	Delta Standard (BSIA)	LVSEC BSIA
28	LVSEC		<Comment>	N/A	Measure		60	<input checked="" type="checkbox"/>	Delta Standard (BSIA)	LVSEC BSIA

Figure 9-55. Standardization for samples to the international scale and scale contraction correction

The results for carbonate sample will be shown individually. Refer to the *DELTA Q Operating Manual* for peak determination and obscuring sample results.

Linearity Correction from Exported Qtegra Result Files

The system GasBench Plus device - IRMS with its different gas flows and slightly varying temperatures is never perfectly linear. To achieve the best possible result with respect to both accuracy and stability, either tune your instrument to optimal conditions in every run or apply a mathematical correction for the effects.

The effects that influence fractionation of masses by the system include temperature first of all. Temperature variations change the viscosity of helium and thereby affect flow speeds. They also change the δ value of your reference gas, if you use a pressurized CO₂ tank with a liquid phase inside.



Refer to *Grootes, P.M., Mook, W.G. and Vogel, J.C.: Isotopic fractionation between gaseous and condensed carbon dioxide. Zeitschrift für Physik 221:257-273 (1969).*

Experiment-to-experiment variations of fractionation occur, if you tune the source or change the timing of the acquisition. More reasons for applying corrections to the signal-to- δ value-scale and to the measured-to-real δ value-scale can easily be found. This topic, “[Linearity Correction from Exported Qtegra Result Files](#)”, covers the relationship between measured δ value and signal height. The relationship between measured δ value and real δ value is described at “[Referencing vs. VPDB](#)” on [page 8-2](#).

[Table 9-2](#) shows an uncorrected result, that is raw data from a series of measurements of the same sample.

Table 9-2. Raw data example to illustrate linearity correction

Position	Bottle number	Weight	Average area	$\delta^{13}\text{C}$	$\delta^{18}\text{O}$
1	dummy sample	0	11.40	-39.332	-5.838
2	dummy sample	0	16.46	-39.662	-0.380
3	dummy sample	0	11.58	-39.401	-1.342
4	dummy sample	0	17.38	-39.532	-2.523
9	CaCO3 Merck	100	12.53	-30.292	-12.049
10	CaCO3 Merck	41	3.63	-29.885	-12.154
11	CaCO3 Merck	39	3.57	-30.083	-12.219
12	CaCO3 Merck	112	9.96	-30.333	-12.171
17	CaCO3 Merck	77	7.18	-30.198	-12.070
18	CaCO3 Merck	188	19.02	-30.350	-12.054
19	CaCO3 Merck	80	6.29	-30.193	-12.192
20	CaCO3 Merck	34	3.42	-30.196	-12.277
25	CaCO3 Merck	72	6.82	-30.199	-12.296
26	CaCO3 Merck	139	13.63	-30.340	-12.230
27	CaCO3 Merck	176	15.19	-30.381	-12.170
28	CaCO3 Merck	147	15.11	-30.390	-12.107
33	CaCO3 Merck	38	3.16	-30.155	-12.250
34	CaCO3 Merck	78	6.52	-30.316	-12.386
35	CaCO3 Merck	142	12.93	-30.374	-12.208
36	CaCO3 Merck	67	5.78	-30.330	-12.373
41	CaCO3 Merck	36	3.69	-30.356	-12.370
42	CaCO3 Merck	48	5.14	-30.130	-12.157
43	CaCO3 Merck	12	0.51	-29.484	-12.100
44	CaCO3 Merck	311	32.57	-30.449	-11.849
49	CaCO3 Merck	52	5.29	-30.501	-12.250

Table 9-2. Raw data example to illustrate linearity correction, continued

Position	Bottle number	Weight	Average area	$\delta^{13}\text{C}$	$\delta^{18}\text{O}$
50	CaCO3 Merck	303	32.99	-30.394	-11.827
51	CaCO3 Merck	48	4.06	-30.289	-12.235
52	CaCO3 Merck	26	2.14	-30.309	-12.211
57	CaCO3 Merck	143	13.47	-30.474	-12.196
58	CaCO3 Merck	108	10.79	-30.437	-12.205
59	CaCO3 Merck	48	2.55	-30.292	-12.346
60	CaCO3 Merck	201	9.84	-30.445	-12.097
65	CaCO3 Merck	21	1.58	-30.411	-12.387
66	CaCO3 Merck	57	5.64	-30.323	-12.340
67	CaCO3 Merck	250	25.03	-30.474	-12.035
68	CaCO3 Merck	235	24.41	-30.511	-12.031
73	CaCO3 Merck	86	9.23	-30.441	-12.149

If you plot the δ value versus the peak area or the peak amplitude, which is strictly proportional to it, a graph like [Figure 9-56](#) is obtained.

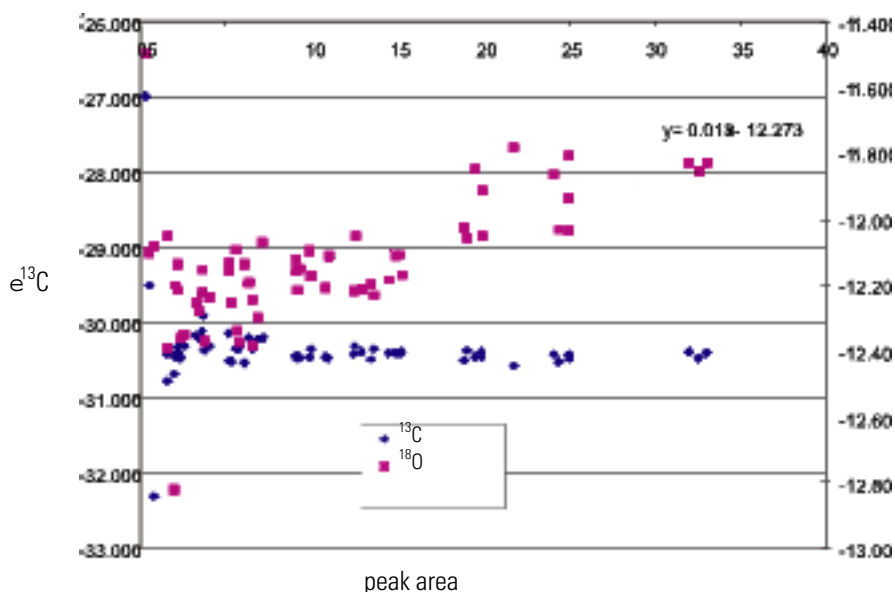


Figure 9-56. Measured δ value versus peak area for a set of measurements^a

^a same sample but different sample amounts

Experience teaches that the functional dependence between δ value and peak area (or peak amplitude) always is a linear one. Thus, within the statistical error limits all results are distributed along a line with a small

slope. The slope is small (0.013‰/Vs for $^{18}\text{O}/^{16}\text{O}$ and 0‰/Vs for $^{13}\text{C}/^{12}\text{C}$ in the example above), but depends on all of the factors mentioned above. The following correction procedure is recommended:

The correction can be approximated by a linear function $\delta_{\text{meas}}(A)$:

$$\delta_{\text{meas}} = m \times A + \delta_{\text{real}}$$

δ_{meas} denotes the measured δ value, δ_{real} the real one. A describes the peak area.

Determine the correction factor m , that is the slope, from reference samples (that is working standards) by plotting the measured δ value for $^{18}\text{O}/^{16}\text{O}$ and $^{13}\text{C}/^{12}\text{C}$ δ_{meas} versus peak area or peak amplitude.

The correction factor δ_{real} must be evaluated from absolute standards (IAEA). For details, see [“Referencing vs. VPDB”](#) on [page 8-2](#).

To achieve proper results, you must include working standards in your sequence of measurements. It is absolutely necessary to keep all possible sources of fractionation constant during the sequence. The reference samples (that is working standards) should be well distributed in the sample tray. To obtain a proper estimate for the slope, sample amount should vary. Furthermore, this procedure allows quality control during the entire data acquisition.

Denitrification Kit

Contents

- [Introduction to the Denitrification Kit](#) on page 10-1
- [Installing the Denitrification Kit for the GasBench Plus Device](#) on page 10-3
- [Additional Information for Installing the Denitrification Kit](#) on page 10-23

Introduction to the Denitrification Kit

This section describes the working principle of the pure Denitrification Kit and the Denitrification Kit for the GasBench Plus device including AS Kit. Some parts are not provided with the Denitrification Kit. They must be purchased additionally:

- GasBench Plus device
- Dual cold trap
- TriPlus RSH autosampler

The Denitrification Kit is an option for the Thermo Scientific GasBench Plus device. It allows, for example, measuring gaseous nitrogen or nitrogen oxides created by biological decomposition of nitrate samples or by chemical reduction using cadmium. Additionally, $N_2O > 1$ ppm can be analyzed.



Refer to *McIlvin M.R., Altabet M.A. Chemical conversion of nitrate and nitrite to nitrous oxide for nitrogen and oxygen isotopic analysis in freshwater and seawater. Anal. Chem. 2005, 77, 5589–5595.*



Refer to *Sigman, D.M., Casciotti, K.L., Andreani, M., Barford, C., Galanter, M., Böhlke, J.K. A bacterial method for the nitrogen isotopic analysis of nitrate in seawater and freshwater. Anal. Chem. 2001, 73, 4145–4153.*

Working Principle

Figure 10-1 shows the working principle of nitrogen analysis with the GasBench Plus. The red frame indicates the parts of the Denitrification Kit.

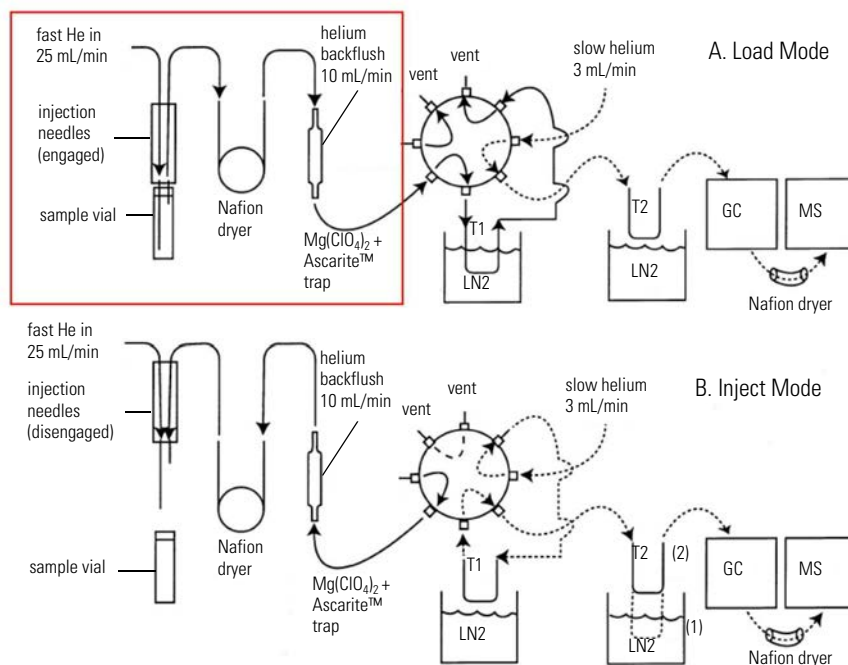


Figure 10-1. Working principle of Denitrification Kit

The description of the measurement method itself is beyond the scope of this section. For detailed information, refer to the following scientific paper and the papers cited therein:



Refer to *Casciotti, K. L., Sigman, D. M., Galanter Hastings, M., Böhlke, J. K., Hilkert, A. Anal. Chem. 2002, 74, 4905–4912.*

Cold Trap and Dual Cold Trap

The two cold traps shown in Figure 10-1 are not part of the Denitrification Kit. They are part of the Dual cold trap, which must be purchased separately. See “Cryo Trap Options” on page 11-2. If a Cold trap was purchased earlier, a second Cold trap is required.

T1 (Trap 1) is connected with a stainless steel capillary (length of 950 mm, 1/16 in. OD, 0.8 mm ID). T2 (Trap 2) is connected with a fused silica capillary (length of 560 mm, ID 0.32 mm).

Installing the Denitrification Kit for the GasBench Plus Device

This section describes how to install the Denitrification Kit for GasBench Plus or the Denitrification Kit for GasBench Plus including AS Kit. As a guideline use the schematic shown in [Figure 10-2](#).

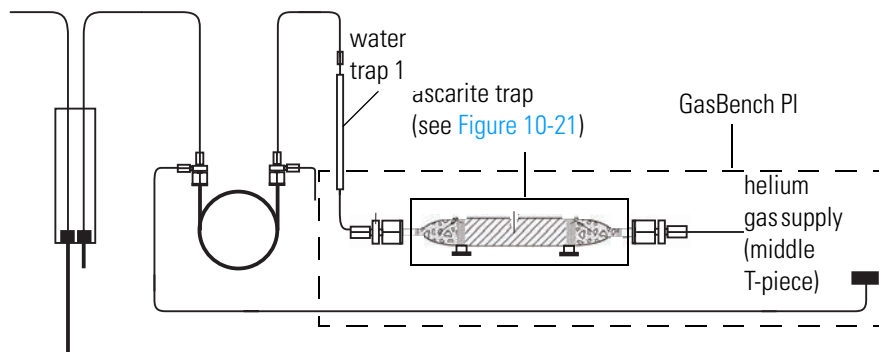


Figure 10-2. Assembling parts of Denitrification Kit

The section treats the following topics:

- “[Installing the Hardware Parts of the Denitrification Kit](#)” on [page 10-4](#)
- “[Assembling the Ascarite Trap](#)” on [page 10-8](#)
- “[Fixing the Cajon Connectors to the Ascarite Trap](#)” on [page 10-10](#)
- “[Installing the Perma Pure Water Trap](#)” on [page 10-12](#)
- “[Installing the Water Trap and the Ascarite Trap to the GasBench Plus Device](#)” on [page 10-14](#)
- “[Mounting the Dual Trap](#)” on [page 10-16](#)
- “[Connecting the Dual Trap to the GasBench Plus Device](#)” on [page 10-18](#)
- “[Installing the Needles](#)” on [page 10-20](#)
- “[Setting up Qtegra to Measure Isotope Ratios of N₂O Using the GasBench Plus Device and the Dual Trap](#)” on [page 10-19](#)

Tip After assembling the Denitrification Kit, check whether the assembly is gas-tight. Flush the assembly with nitrogen or argon and monitor the signal in the mass spectrometer. Use the following parameters:

- cup for m/z 28 (N₂) or m/z 40 (argon)
- amplification factor 3×10^{10}

Denitrification Kit

Installing the Denitrification Kit for the GasBench Plus Device

Installing the Hardware Parts of the Denitrification Kit

This section lists the steps of the installation procedure as an overview. See [Figure 10-3](#) and [Figure 10-4](#). The individual steps of the overview will then be described in turn in detail.

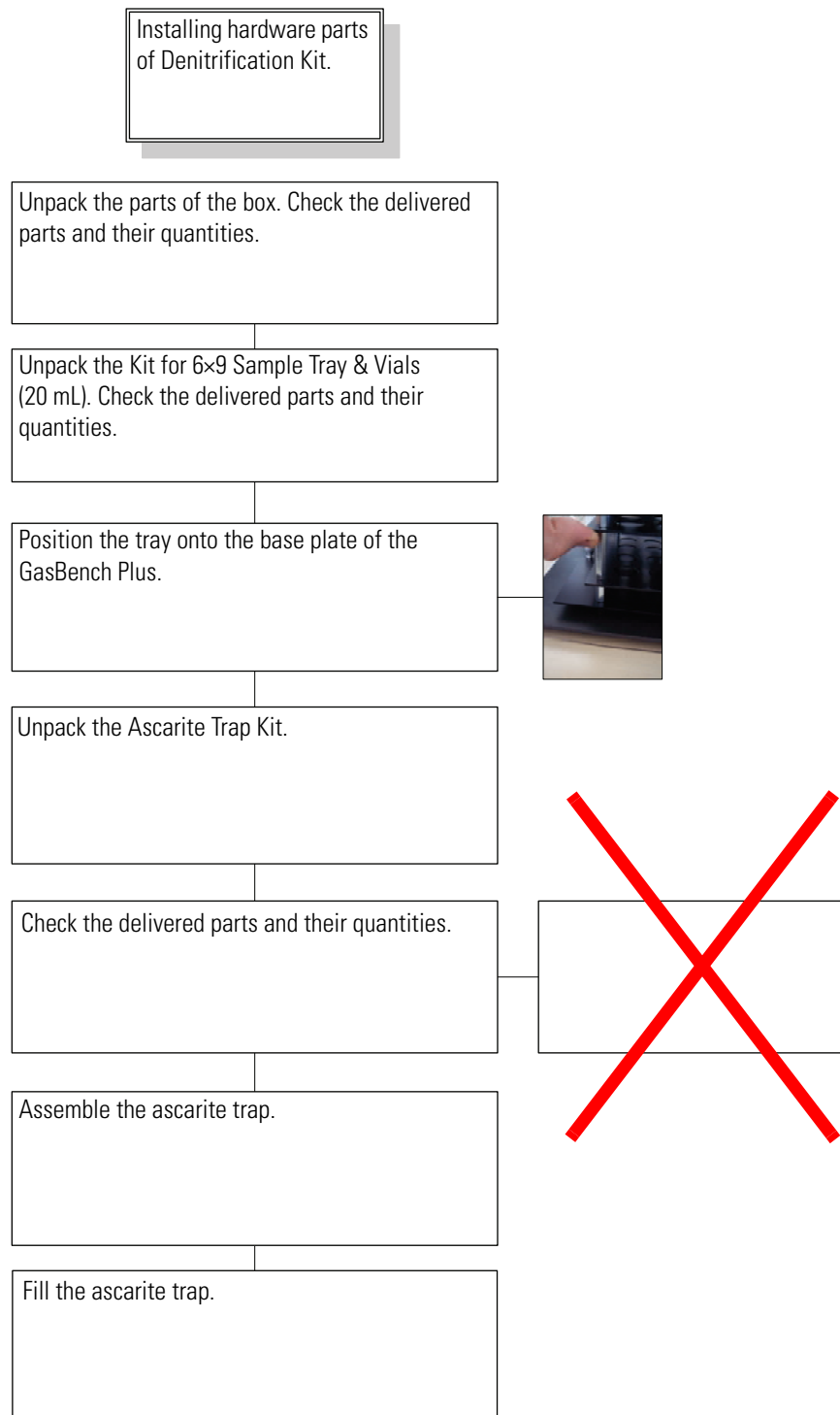


Figure 10-3. Installing hardware parts of Denitrification Kit - Part 1

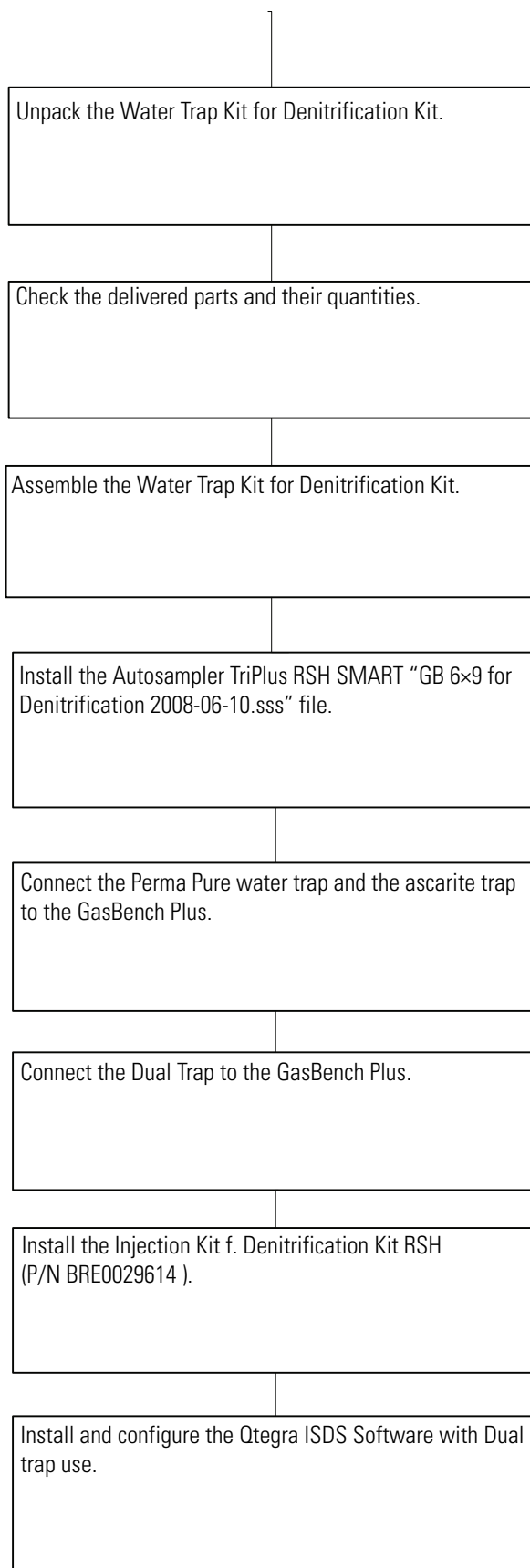


Figure 10-4. Installing hardware parts of Denitrification Kit - Part 2

Denitrification Kit

Installing the Denitrification Kit for the GasBench Plus Device

General Remarks

GVF (Vespel®) ferrules can generally be re-used, if capillaries with the same outer diameter as the former ones are exchanged. The stainless steel and fused silica capillary are usually tight after one or a few wrench turns.

If no tightness is reached after a few turns, open the bulkhead union again and re-attach the capillary into the old ferrule! If the old ferrule was damaged, exchange it with a new appropriate GVF ferrule.

Tightness is reached, if the capillary inside the ferrule cannot be moved by a short finger pull. A quarter of a turn will lead to leak tightness.

Full leak tightness can be checked using a leak hunter or a flow controller.

Tools

The following tools are needed to install the hardware parts of the Denitrification Kit.

- scissors
- small tweezers with tip
- 10" tweezers (if available)
- powder-free gloves
- SiO₂ safety goggles
- face mask (against spill of liquid nitrogen)
- cold-resistant gloves
- small spatula
- bigger spatula or funnel
- small funnel (tube dimensions: OD 6 mm, ID 4 mm)
- PTFE tube (OD 8 mm, ID 6 mm)
- wire (for pressing Silane fibers)
- vacuum grease (Apiezon™ N, for example)
- two 1/2" wrenches
- 1/4" wrench
- wafer (for cutting fused silica)
- expandable spanner

- 5/16" wrench
- 7/16" wrench
- TORX™ T10 wrench

NOTICE

Turn off the Emission (filament) prior to perform any installation at the GasBench Plus and close the needle valve. Check the high vacuum pressure before switching on the filament! If the pressure exceeds 1×10^{-7} mbar, check for leaks.

Denitrification Kit

Installing the Denitrification Kit for the GasBench Plus Device

Assembling the Ascarite Trap

To assemble the Ascarite™ trap, follow [Figure 10-5](#) and [Figure 10-6](#).

Tip Close the needle valve to the IRMS prior to any assembly.

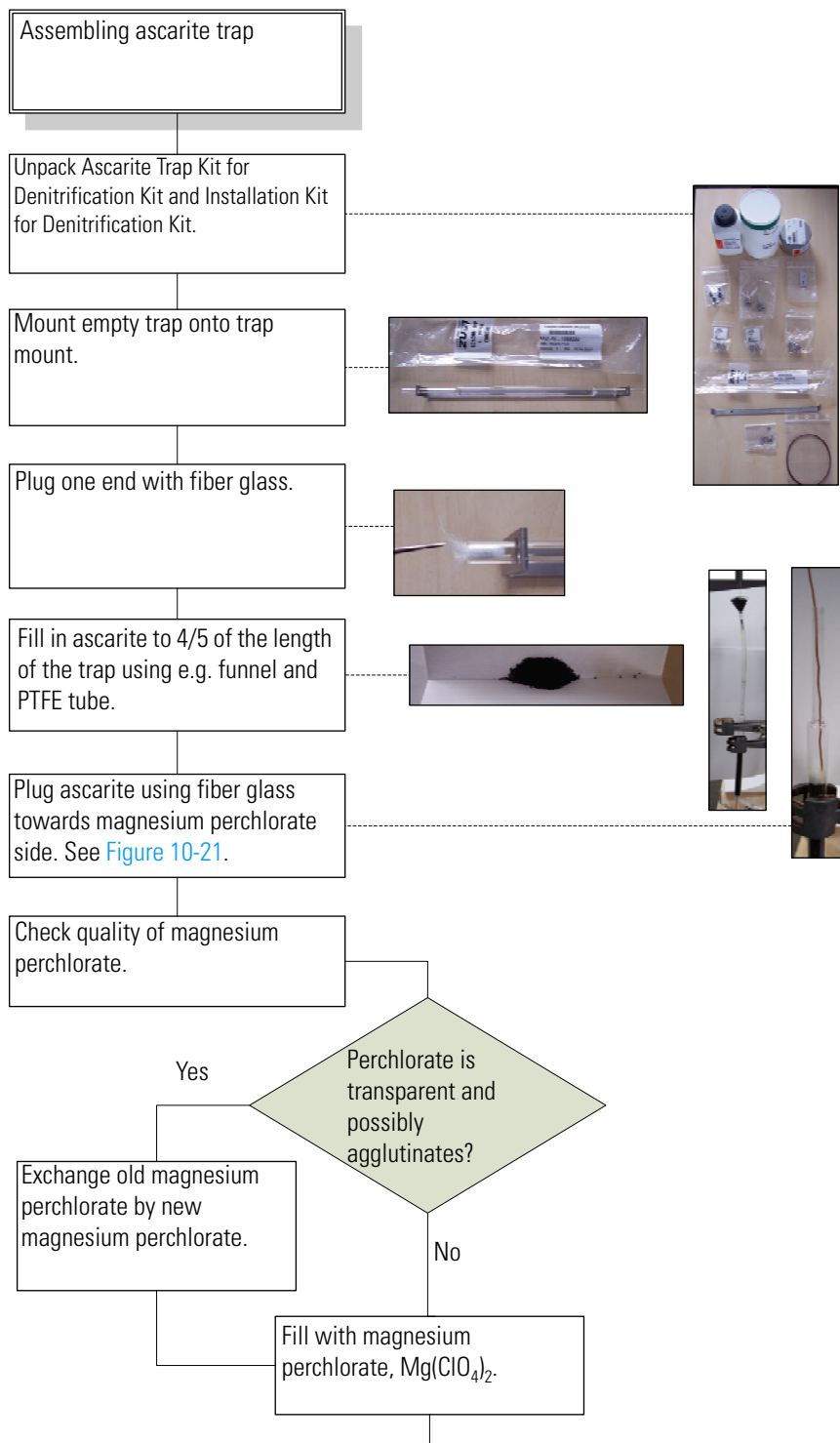


Figure 10-5. Assembling ascarite trap - Part 1

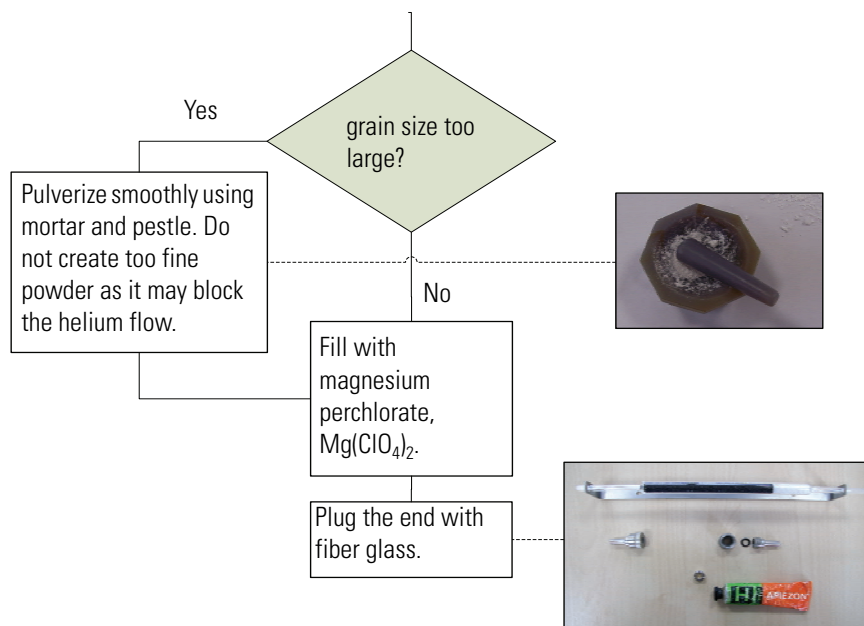


Figure 10-6. Assembling ascarite trap - Part 2

Denitrification Kit

Installing the Denitrification Kit for the GasBench Plus Device

Fixing the Cajon Connectors to the Ascarite Trap

To fix the Cajon connectors to the ascarite trap, follow the instructions given in [Figure 10-7](#) and [Figure 10-8](#).

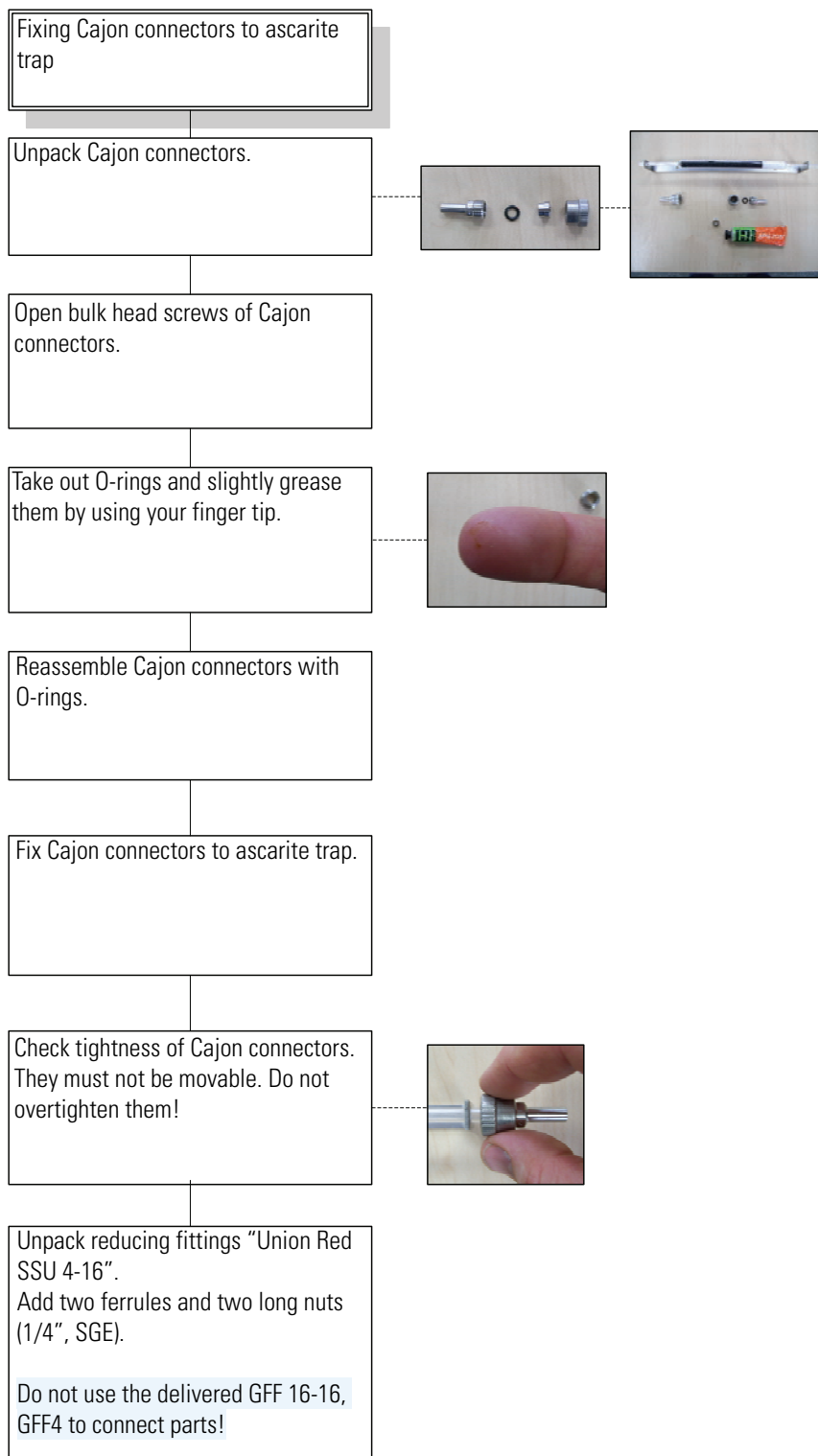


Figure 10-7. Fixing Cajon connectors to ascarite trap - Part 1

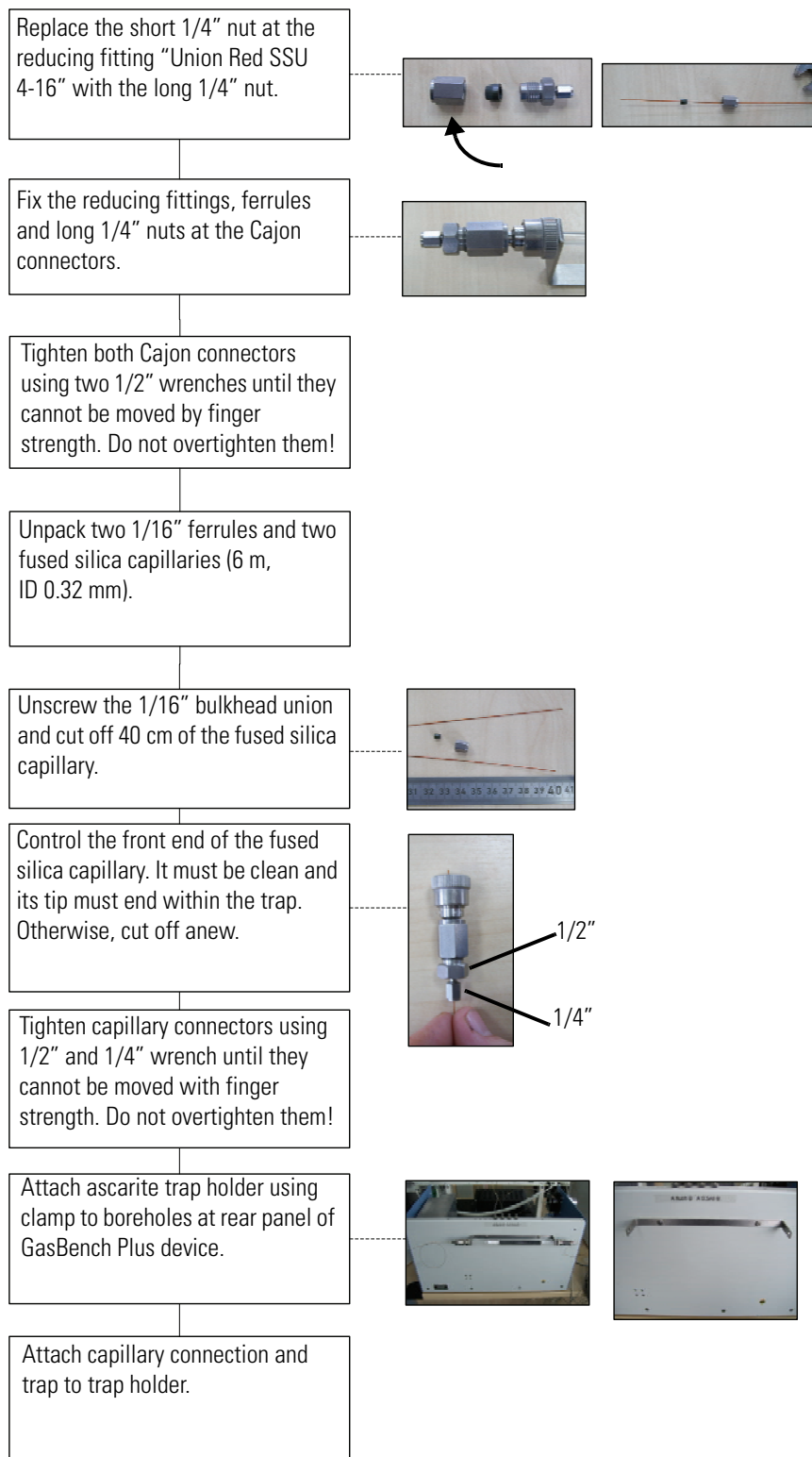


Figure 10-8. Fixing Cajon connectors to ascarite trap - Part 2

Denitrification Kit

Installing the Denitrification Kit for the GasBench Plus Device

Installing the Perma Pure Water Trap

To install the Perma Pure water trap, follow the instructions given in [Figure 10-9](#) and [Figure 10-10](#).

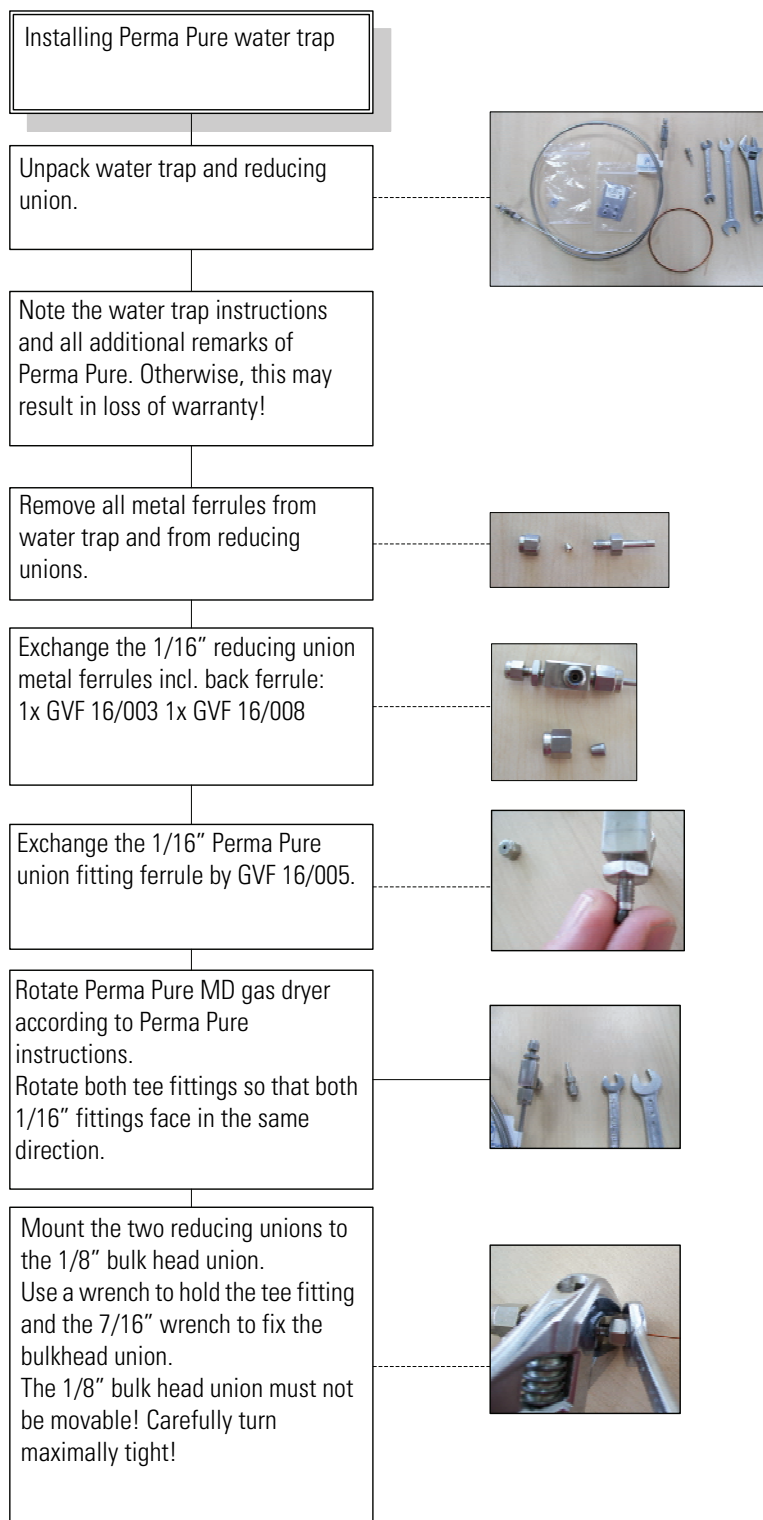


Figure 10-9. Installing Perma Pure water trap - Part 1

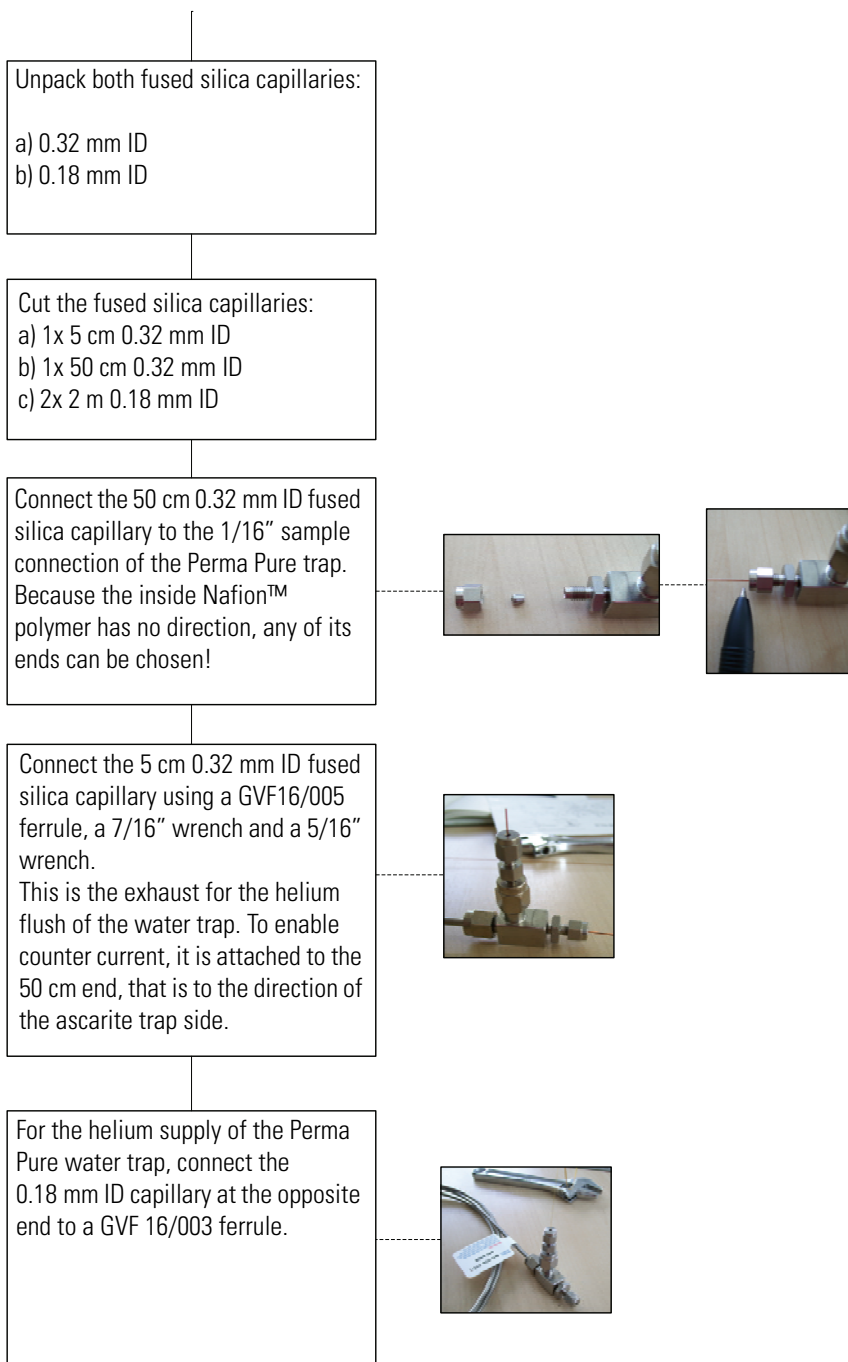


Figure 10-10. Installing Perma Pure water trap - Part 2

Denitrification Kit

Installing the Denitrification Kit for the GasBench Plus Device

Installing the Water Trap and the Ascarite Trap to the GasBench Plus Device

To install the water trap and the ascarite trap to the GasBench Plus device, follow the instructions given in [Figure 10-11](#), [Figure 10-12](#), and [Figure 10-13](#). The rearrangement allows a fast switch to normal flow GasBench Plus applications.

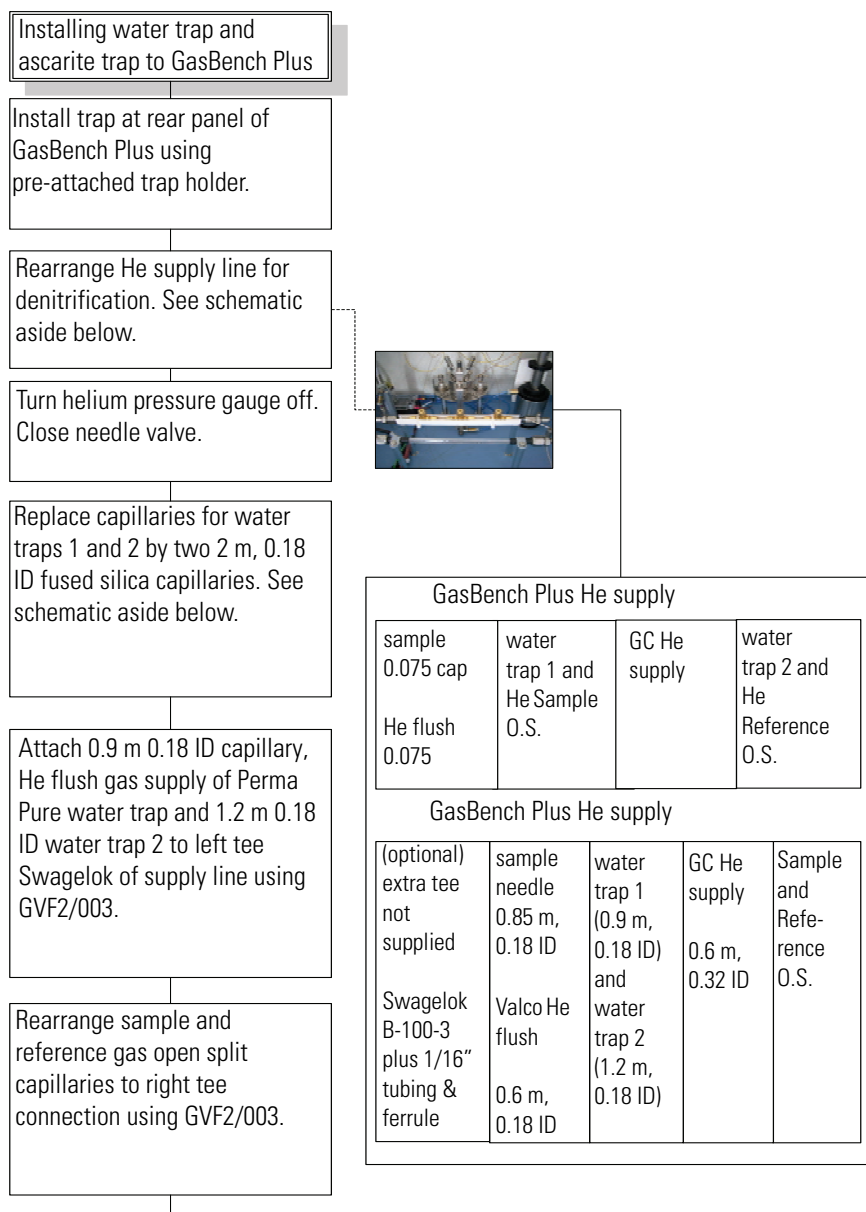


Figure 10-11. Installing water trap and ascarite trap to GasBench Plus - Part 1

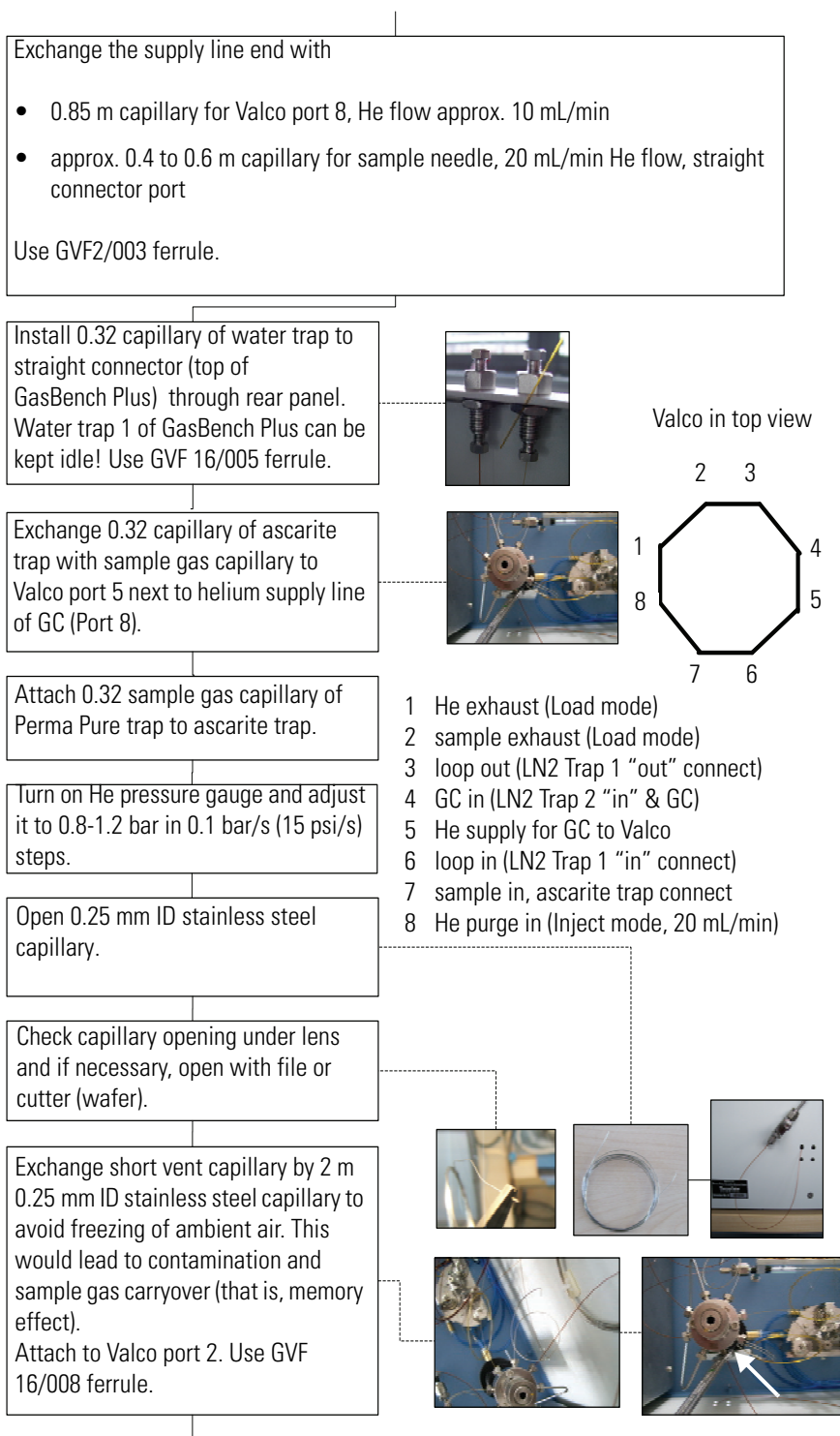


Figure 10-12. Installing water trap and ascarite trap to GasBench Plus - Part 2

Denitrification Kit

Installing the Denitrification Kit for the GasBench Plus Device

Attach short vent capillary to Valco port 1. If necessary, exchange with GVF 16/008 ferrule.

Check flows:

Water trap 2 6-8 mL/min at vent

Perma Pure 6-8 mL/min at vent

He vent (Valco) 8-12 mL/min at vent

Sample needle He 18-22 mL/min at straight connector with 3 cm 0.32 mm ID capillary supply

Needle flow acetone bubble check

Tip Water trap 1 (not in use) can be closed by using a clean GC septum.

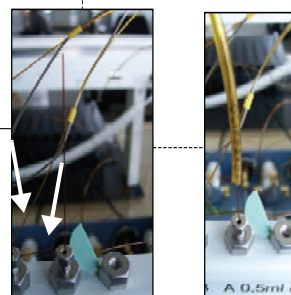


Figure 10-13. Installing water trap and ascarite trap to GasBench Plus - Part 3

Mounting the Dual Trap

To mount the Dual Trap, follow the instructions given in [Figure 10-14](#). See also “[Cryo Trap Options](#)” on [page 11-2](#), “[GasBench Plus Trapping System](#)” on [page 11-9](#), and “[Changing the Loop Size](#)” on [page 3-20](#).

CAUTION

Cold Liquid. Note the safety regulations for liquid nitrogen. Wear a safety face mask and liquid nitrogen-resistant safety gloves!

NOTICE

Avoid any overturn of the Trap Unit by fixing the unit to a stable anchorage point!

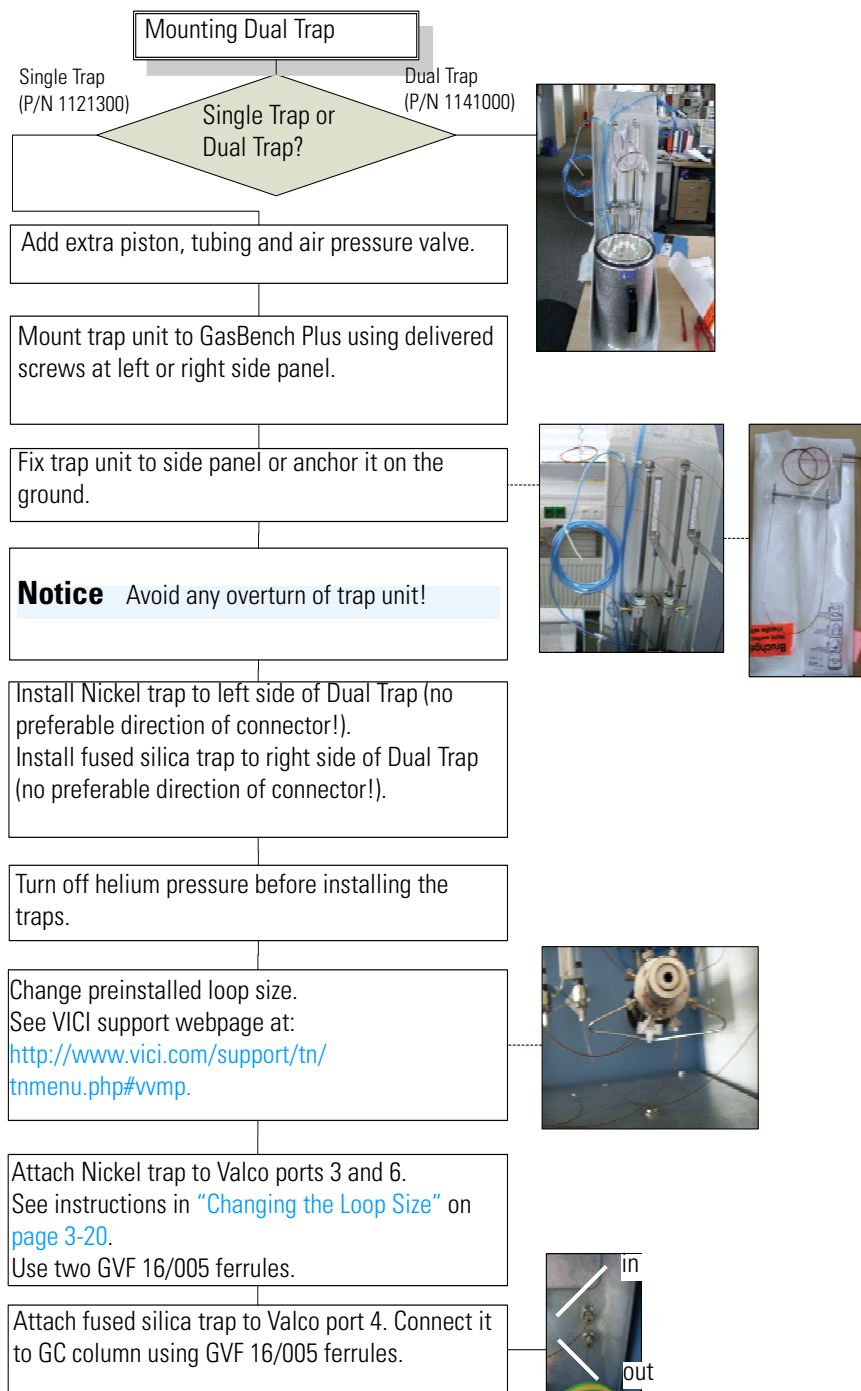


Figure 10-14. Mounting Dual Trap

Denitrification Kit

Installing the Denitrification Kit for the GasBench Plus Device

Connecting the Dual Trap to the GasBench Plus Device

To connect the Dual Trap to the GasBench Plus device, follow the instructions given in [Figure 10-15](#) and [Figure 10-16](#). See also “[Cryo Trap Options](#)” on [page 11-2](#), “[GasBench Plus Trapping System](#)” on [page 11-9](#), and “[Changing the Loop Size](#)” on [page 3-20](#).

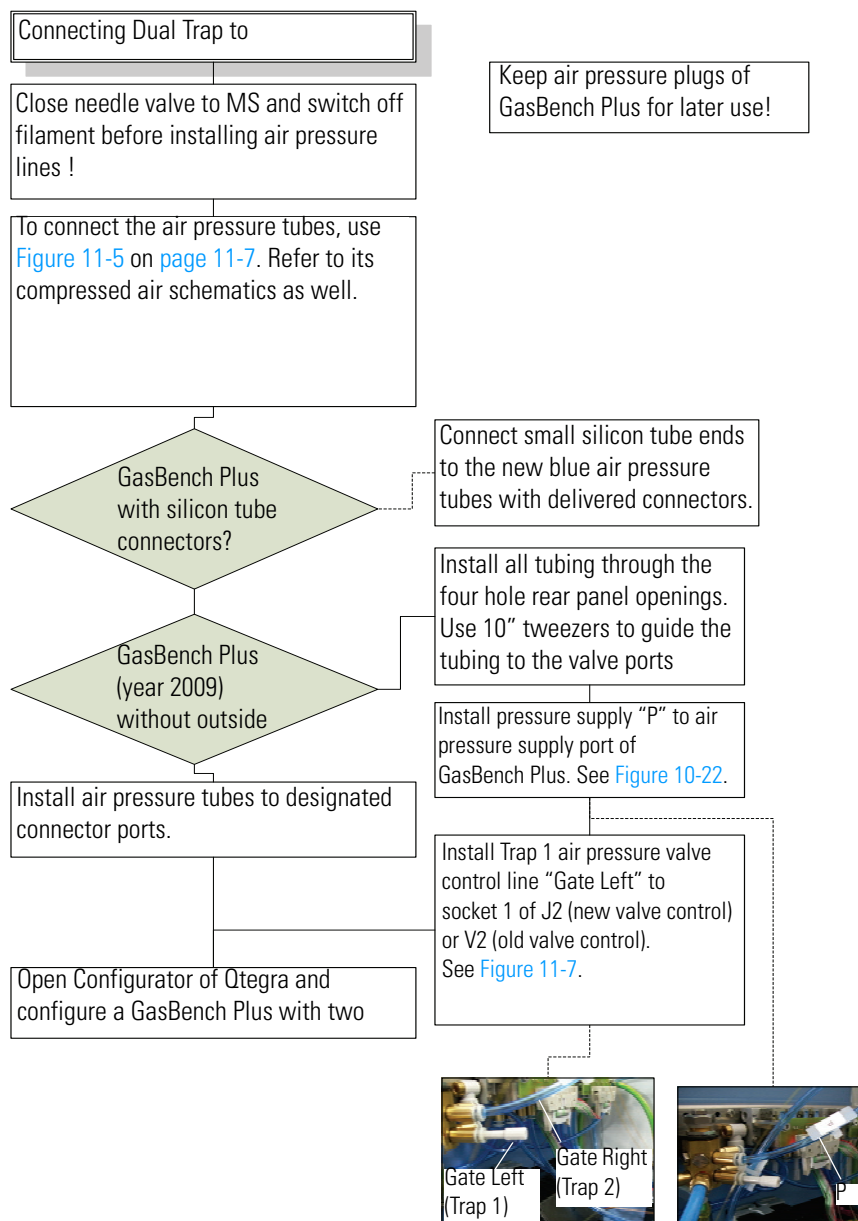


Figure 10-15. Connecting Dual Trap to GasBench Plus – Part 1

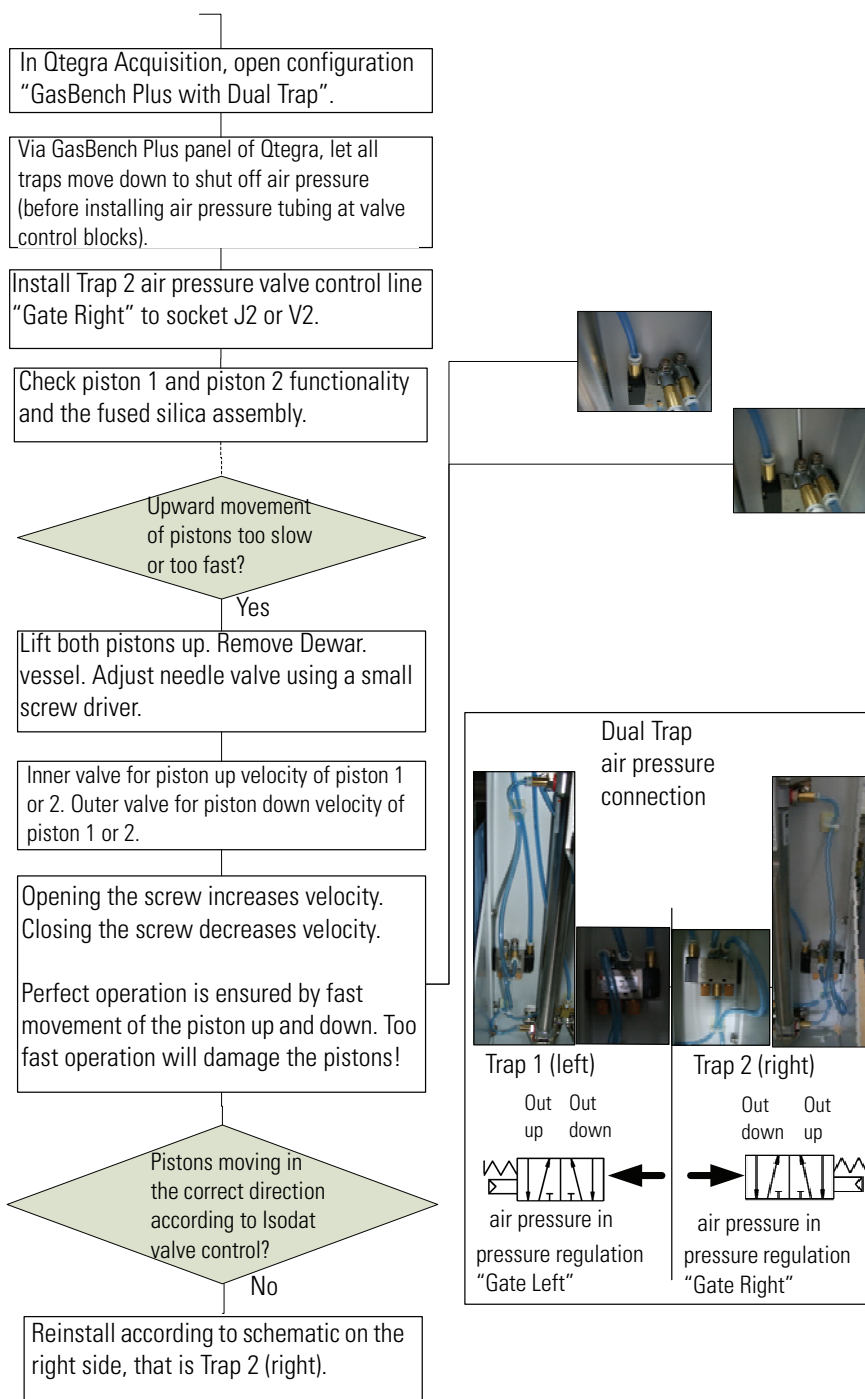


Figure 10-16. Connecting Dual Trap to GasBench Plus – Part 2

The installation of the traps is now finished.

Setting up Qtegra to Measure Isotope Ratios of N₂O Using the GasBench Plus Device and the Dual Trap

Set up Qtegra for measurement of isotope ratios of N₂O with the GasBench Plus and the Dual Trap.

Denitrification Kit

Installing the Denitrification Kit for the GasBench Plus Device

Installing the Needles

This section describes how the 120 mm needle is installed for the Denitrification Kit into the GasBench Tool 120.

❖ To install the needles

1. Unpack “Injection Kit f. Denitrification Kit RSH (P/N BRE0029614)”. Unpack “Sample Kit RSH:77 sample tray incl. parts” (P/N BRE0029609).



Figure 10-17. Injection and Sample Kits

2. Install needle as in any common GasBench Tool 55 and 120 (see [“TriPlus RSH SMART - Advanced and TriPlus RSH SMART - Standard Installation”](#) on page 5-19).
3. Create a Denitrification Kit LabBook as described below.
4. After successful installation, press Start to enable the TriPlus RSH with GasBench Tool 120. Do not use any needles.



Figure 10-18. GasBench Tool 120

5. Configure tray and tray positions. See [“Calibrating the TriPlus RSH for GasBench Plus Configuration”](#) on page 5-31.
6. Refer to generic teaching with tools. See [“Checking Calibration and Teaching Installed GasBench Trays”](#) on page 5-37.

7. Teach. Refer to “Teaching” on page 5-19.
8. Check positioning visually: no needle must touch rim and bottom of vial!
See “Checking Calibration and Teaching Installed GasBench Trays” on page 5-37.
9. Remove the needles and check the other positions.
10. If no proper injection is possible, redo teaching.
11. Carefully insert the tool 120 with pre-installed needles.
12. Install the He purge needle in tool 55 with the GVF 16/008 ferrule at the second from the left connector.

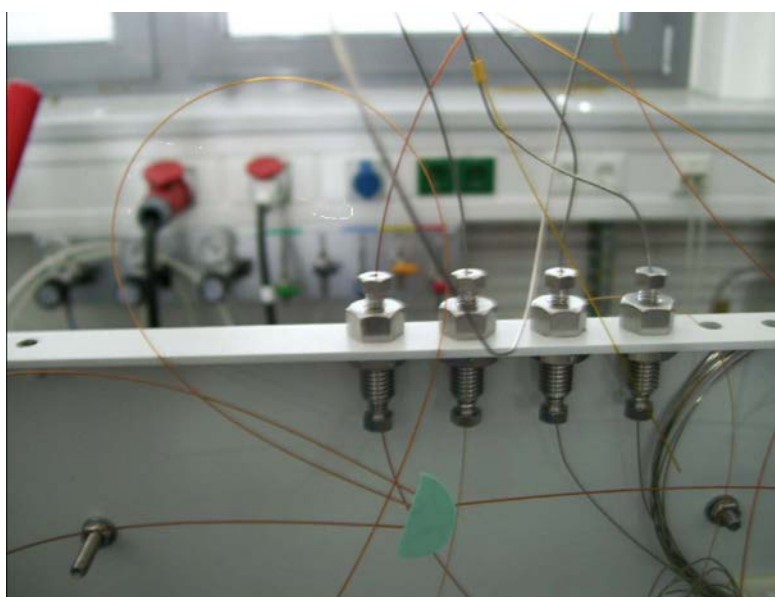


Figure 10-19. Helium Purge Needle installed

13. Stabilize the 0.32 mm ID capillary. Avoid bending it during operation of autosampler.
14. Install measurement needle at the leftmost straight connector with the necked GVF 005 ferrule.
15. Bubbles can be seen after injection into a sample vial.

Denitrification Kit

Installing the Denitrification Kit for the GasBench Plus Device

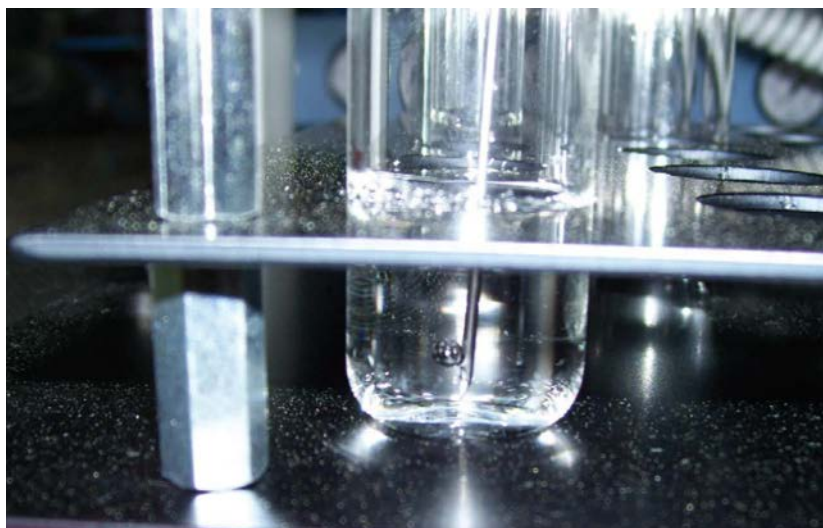


Figure 10-20. Bubbles after injection

NOTICE

GasBench II with CTC Pal 80. For autosampler installation of the CTC Pal 80 refer to the *GasBench II Operating Manual*. Qtegra does not evaluate the CTC Pal 80. Only the rack and the method must be adjusted on the Dashboard for the CTC Pal 80 - A200 S mode.

The hardware of the Denitrification Kit is now completely installed.

Additional Information for Installing the Denitrification Kit

This appendix gives information about the chemical trap and the connection of a Single Trap or Dual Trap.

Chemical Trap

The chemical trap is filled with $Mg(ClO_4)_2$ and Ascarite (NaOH on support material). See Figure 10-21. This trap captures close to 99.99% of the CO_2 from the sample. Water is also withheld in the trap and removed from the helium gas stream, whereas nitrogen and nitrogen oxides quantitatively escape the Ascarite trap.

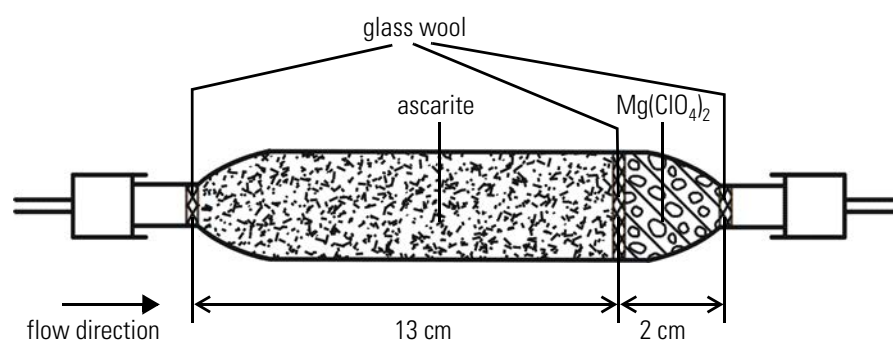


Figure 10-21. Schematic of chemical trap

Connecting the Single Trap or the Dual Trap

Figure 10-22 shows the ports at the GasBench Plus for connecting a Single Trap or a Dual Trap.

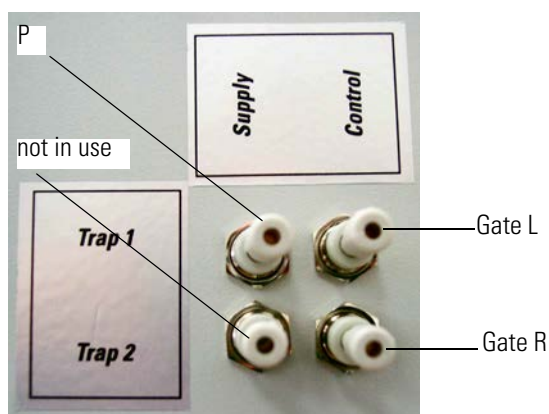


Figure 10-22. Ports for connecting single trap or Tool 55

Setting up Qtegra to Measure Isotope Ratios of N₂O Using the GasBench Plus Device

For standard GasBench measurements of N₂O, a dual trap system is needed to pre-concentrate the N₂O in the first trap at a higher flow rate with the GasBench Tool 120 or with the GasBench Tool 55. The GasBench Tool 120 is applied for complete purging of N₂O from the liquid. Some users use the GasBench Tool 55 and pre-concentrate only from the headspace.

The second liquid nitrogen trap is used to transfer the pre-concentrated N₂O on the low flow side of the Valco port. The GC injection on the second trap will cryofocus the N₂O at low flow to get the sample peaks consequently sharper.

Set up the Qtegra LabBook with a prescript (see [Figure 10-23](#), template delivered with Qtegra ISDS Software or by service, see “[Performing Preparation of Samples using Prescripts with Qtegra](#)” on [page 7-55](#)) and the LabBook timeline will be set as described in “[Performing Sample Measurements with a Single Trap Sample Gas Injection Using Qtegra](#)” on [page 6-20](#).

Continuous Flow (CO₂)

Instrument

TuneBook: Nitrous Dioxide Linearity: Off

Integration Time: 0.2097152 s

Peak Center: On

Peak Center Interval: 1

Peak Center For First Gas Only: Yes

Peripheral Parameters

GasBench: **GasBench** TriPlus RSH SMART

Flush: Off

Sample Prep: GasBench Plus Denitrification

Rack 1: Rack 7 x 11

Rack 2: Rack 7 x 11

Figure 10-23. Qtegra prescript for denitrification

Other Options

This chapter describes technical details about the standard options to be used together with the GasBench Plus device.

Contents

- [Cryo Trap Options](#) on page 11-2
- [PreCon Device](#) on page 11-13
- [Catalyst for Hydrogen Equilibration](#) on page 11-17

Cryo Trap Options

The basic problem to deal with is that a very small sample has to be analyzed from a relatively big gas volume. The Thermo Scientific cryo-option now renders this possible using the so-called GasBench Plus cryo-option. Two different types of the cryo-option can be delivered:

- Cold trap, (that is single trap version), comprising only one trap
- Dual cold trap, (that is comprising two traps)

In the Cold trap (that is single trap version), either a stainless steel capillary or a fused silica capillary is used depending on gas flow and sample amount. Both types of capillaries are used within the Dual cold trap, namely the fused silica capillary follows the stainless steel capillary. The general idea of the cryo-option is to obtain higher peak shapes by analyzing small samples in bigger gas volumes.

The cryo traps option contains an automated lever used to move a sample loop in and out of a Dewar filled with a cooling agent (to be supplied by the customer). By filling the Dewar with liquid nitrogen, substances like carbon dioxide, water, methane, or nitrous oxides can be frozen out (trapped). Via proper timing, it is possible to collect these substances in the trap and yield high amplitudes from low concentrations.

Operation Principle

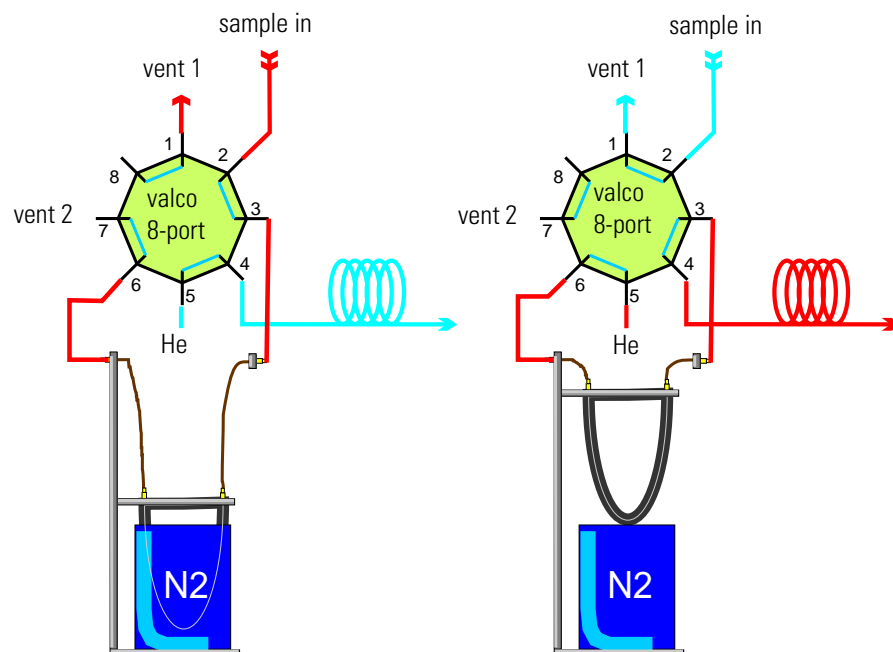


Figure 11-1. Fused silica trap connection for Cold trap

A sample loop is formed from a portion of a 3 m long piece of fused silica tubing. The rest of the full length is used to connect the trap setup to the Valco™ valve. The complete scheme replaces the standard sample loop that comes with GasBench Plus. See [Figure 11-1](#).

According to the time event list of the method, the trap is moved into liquid nitrogen (LN2) at regular intervals to achieve accumulation of CO₂ in the cold spot of the sample loop. When released from the Dewar, the trap heats up without significant time delay, and the CO₂ starts to travel towards the GC of the GasBench Plus device. Due to cryofocusing (Dual cold trap, see [Figure 11-9](#) on [page 11-11](#)) the peak shape is extraordinary sharp. The grade of CO₂ enrichment can be determined by varying the time during that the loop stays in liquid nitrogen (accumulation time).

Procedure

This topic describes the function of the Dual cold trap.

1. In a first step, the sample gas is carried through the sample needle into the nickel-filled stainless steel capillary by a gas flow of approximately 5–15 mL/min. There, the sample is frozen (Load Mode). In this case, the big surface of the stainless steel capillary plays an essential role as the entire sample can be frozen along a short distance. The stainless steel capillary is introduced into the sample gas flow instead of the loop of the Valco port that has been within the sample gas flow so far. For instructions about exchanging the loop, see [“Changing the Loop Size”](#) on [page 3-20](#).
2. After switching the Valco valve from Load Mode to Inject Mode, the entire sample is carried over into the fused silica capillary and frozen a second time.
3. Inject the sample gas into the IRMS by a continuous flow of less than 3 mL/min. Due to the lower diffusion in the fused silica capillary compared to the stainless steel capillary, a better peak shape is achieved.

Cryo Trap Option with Cold Trap (Single Trap)

Figure 11-2 shows a schematic of the Cold trap (that is single trap version).

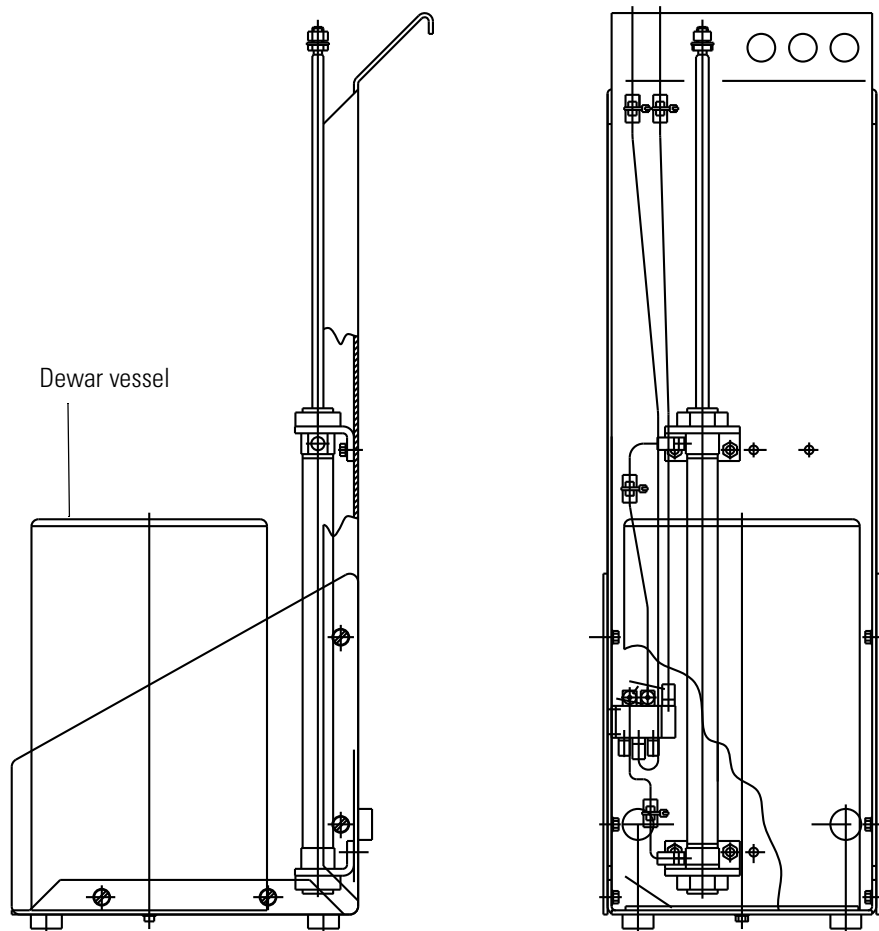


Figure 11-2. Cold trap (that is single trap version)

Cryo Trap Option with Dual Cold Trap

Figure 11-3 shows a schematic of the Dual cold trap.

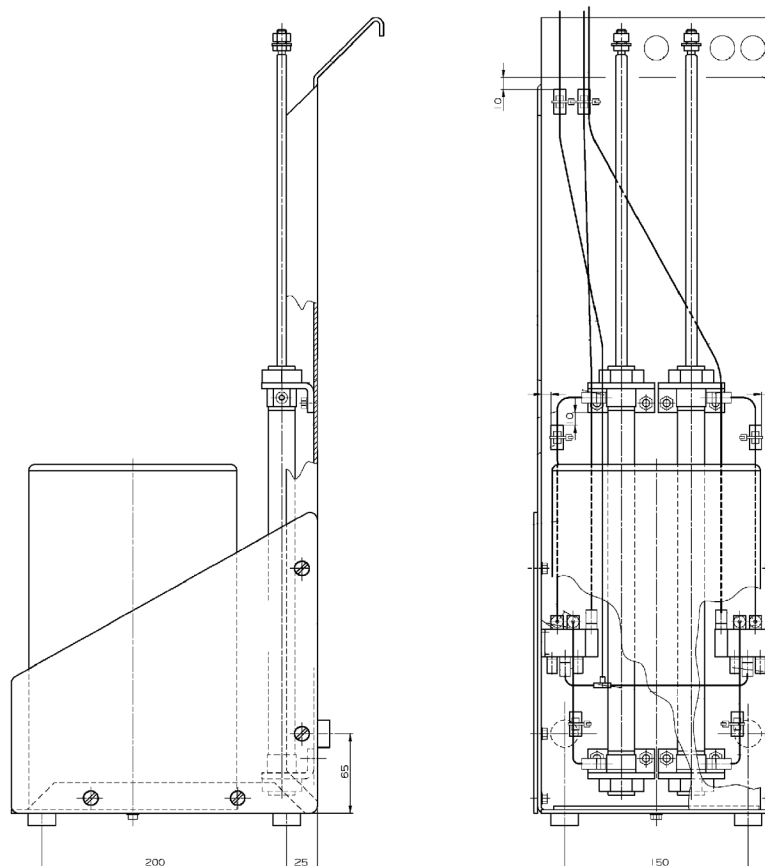


Figure 11-3. Dual cold trap

Important Parts of Both Cold Trap and Dual Cold Trap

Table 11-1 summarizes some important parts of both Cold trap and Dual cold trap.

Table 11-1. Important parts of Cold trap and Dual Cold Trap

Designation	Qty.
Cryo trap for GasBench Plus (mounted)	1
Nickel trap for GasBench Plus (mounted)	1
Capillary, 1/16 in × 0.8 mm	0.6 m

The cryo trap (Figure 11-4 left) can be used to trap water, carbon dioxide, and nitrous oxide but is not suitable for trapping nitrogen. The nickel trap (Figure 11-4 right) is equipped with a nickel wire that can be used to adsorb/trap nitrogen in this trap when submerged in liquid nitrogen.

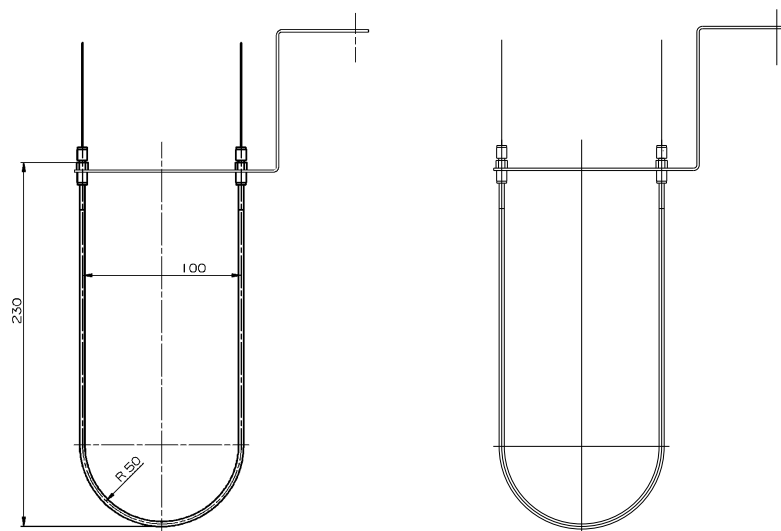


Figure 11-4. Cryo trap (left) and nickel trap (right)

Compressed Air Supply

Figure 11-5 shows the compressed air schematic of the Cold trap and the Dual cold trap. For a dual trap arrangement, trap 2 is added to the rack.

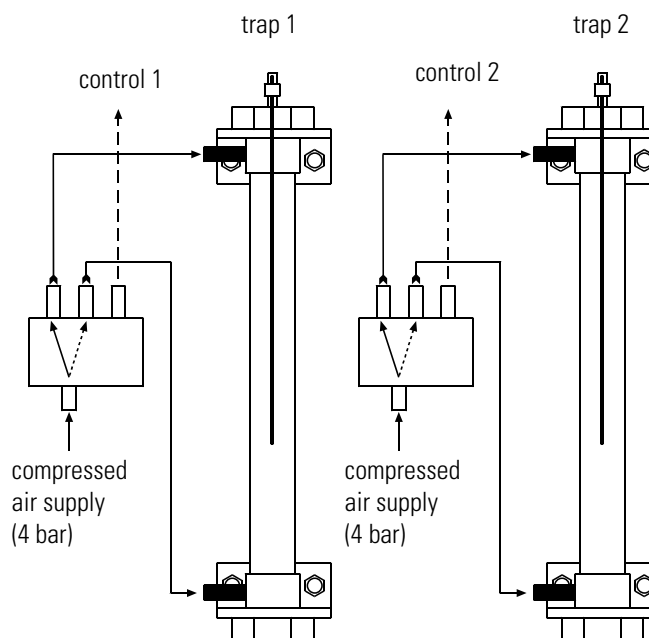


Figure 11-5. Compressed air schematic of Cold trap (trap 1) and Dual cold trap (trap 1 and trap 2)

The compressed air supply should always be set to approximately 4 bar.

Connecting the Cold Trap and the Dual Cold Trap

This topic informs about how to connect the Cold trap and the Dual cold trap.



Figure 11-6. Compressed air connections for Cold trap and Dual cold trap

In [Figure 11-6](#) and [Figure 11-7](#), the compressed air connections for the Cold trap and the Dual cold trap are shown (interior view).



Labeled Components: 1=P for Cold trap and Dual cold trap, 2=Gate Right to Dual cold trap (trap 2), 3=Gate Left to feedthrough for cold trap (trap 1)

Figure 11-7. Compressed air connections for Cold trap and Dual cold trap

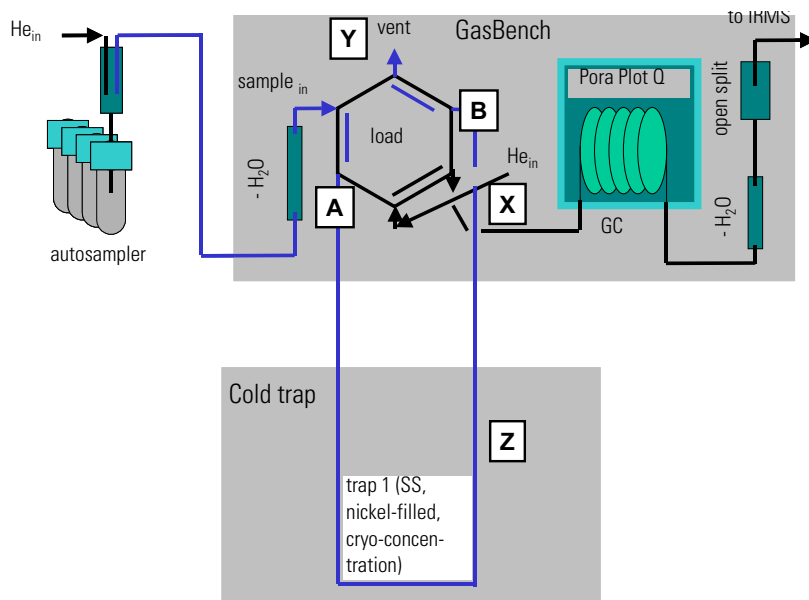
GasBench Plus Trapping System

The trapping system of the GasBench Plus is used to pre-concentrate or for peak sharpening. Additionally, it is used to trap and clean sample gas (N₂O from CO₂, for example) and to use it as an injection concentration to be able to switch from a medium flow to a normal gaschromatographic flow (that is, 2–3 mL/min).

The Cold trap serves as a cryogenic pre-concentration unit for flows in the range of the GC column flow, that is 0.5–5 mL/min. Depending on the GC performance needed, a fused silica trap (0.32 mm fused silica tubing from Valco port A to port B) for very sharp GC peaks or a

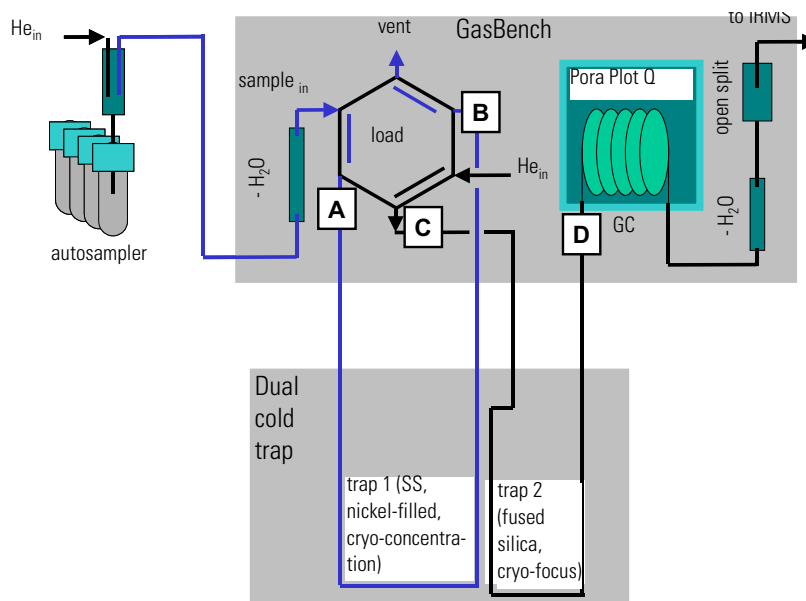
nickel-filled stainless steel trap (ID 1 mm) resulting in broad GC peaks can be used. In case of the stainless steel trap, the sample flow can also be increased up to 15 mL/min.

Tip A longer fused silica capillary needs to be installed in the vent exit (Y) of the Valco valve to avoid freezing of ambient air into the trap. See [Figure 11-8](#) (Cold trap) and [Figure 11-9](#) (Dual cold trap).



Labeled Components: A=injector loop in, B=injector loop out, X=change He in and sample out at Valco, Y=vent capillary needs to be prolonged, Z=depending on the sample flow, the capillary of trap 1 may be made of SS, fused silica or be nickel-filled.

Figure 11-8. Cold trap



Labeled Components: A=injector loop in, B=injector loop out, C=injector to trap 2, D=trap 2 to GC column

Figure 11-9. Dual cold trap

Table 11-2. Volumes of traps

No.	Inner diameter ID (mm)	Volume V ($\mu\text{L}/\text{m}$)
1	0.32	80
2	1.0	780

Now, general remarks are added to be taken care of during installation of Cold trap and Dual cold trap.

- Before you release the ferrules in the Valco valve, slowly reduce the He pressure in the GasBench Plus device to zero.

NOTICE

Close the needle valve leading into the ion source before reducing the He pressure.

- The Dual Trap system serves as a cryogenic pre-concentration unit for flows in the range of the GC column flow (0.5–15 mL/min) including a cryogenic focusing trap in front of the GC column.
- The cryofocusing trap is a fused silica trap (0.32 mm fused silica tubing from Valco port C to port D) for very sharp GC peaks. It also serves as a mediator between high sampling flows and low GC flows (the sample is dissolved in other gases. Here, the fraction that can be frozen out is collected from a bigger gas amount. To collect this fraction completely, high throughputs through the trap are used during a long period of time).
- The cryogenic pre-concentration trap is a nickel-filled stainless steel trap (ID 1 mm) connected from Valco port C to port D.

Other Options

Cryo Trap Options

- An application for one trap is given in “[Analyzing CO₂ in Atmospheric Concentrations](#)” on [page 8-7](#).
- Before you release the ferrule in the bulkhead union in front of the GC column, slowly reduce the He pressure in GasBench Plus to zero.

Trapping of N₂ at -196 °C

Liquid nitrogen can be adsorbed on silica gel or nickel surfaces at about -196 °C. Thus, it is possible to collect and cryofocus nitrogen for analysis by using a trap operating with liquid nitrogen. The trap used with the GasBench Plus device is equipped with a nickel wire to perform N₂ trapping.

Tip When you apply this kind of trap, keep in mind that other air compounds like CO₂ or water will also be collected therein.

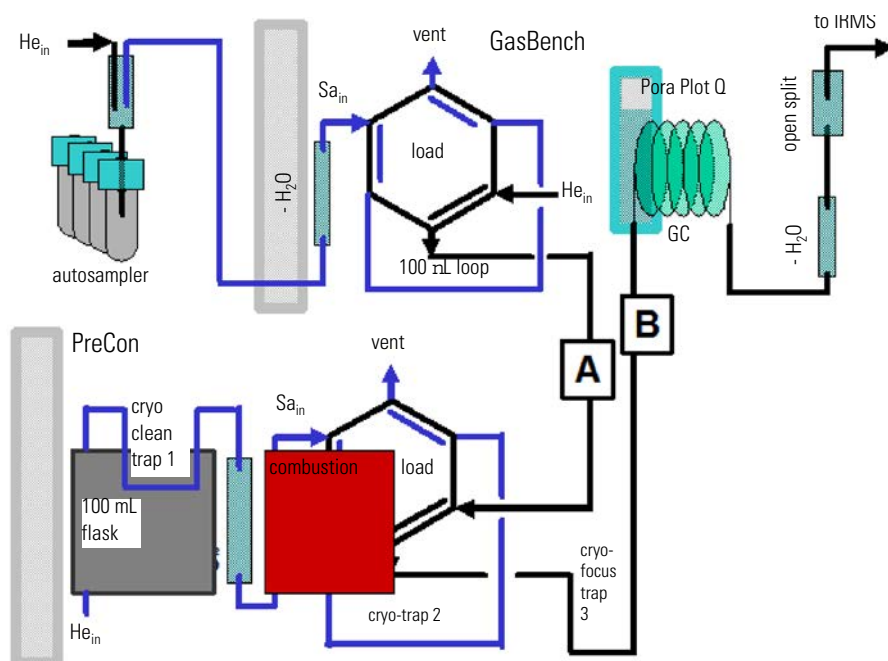
PreCon Device

The PreCon device is an additional peripheral to either the GasBench Plus device or a Trace GC IRMS. It is used to concentrate subambient gas concentrations (CO_2 in soils, N_2 in water, noble gases), to clean noble gases from air etc., to concentrate ambient N_2O , CO_2 and CH_4 and to combust CH_4 to CO_2 for $^{13}\text{C}/^{12}\text{C}$ ratio determination of CH_4 .

The PreCon device is designed for the preconcentration of trace gases in air or other samples to perform high precision isotope analysis. It allows to analyze trace gases with concentrations in the low ppm to ppb range (methane 1.7 ppm, N_2O 300 ppb, for example) using air sample sizes of 100 mL or less.

The PreCon device consists of two parts: a high flow part with helium flows of 20–25 mL/min (PreCon side) and a low flow part of 1–2 mL/min helium flow (GC side).

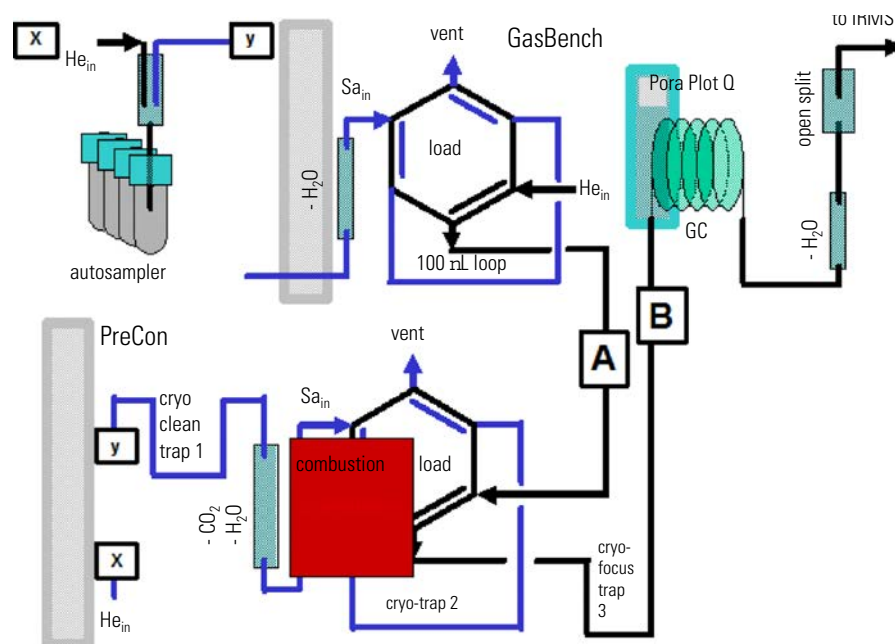
Trap T2 belongs to both sides depending on the Valco valve being switched to Vent or Load mode. The PreCon device is linked into the GasBench Plus device after the sample loop, but in front of the GC capillary column. See [Figure 11-10](#). If the PreCon Valco valve is in Load mode, the GasBench Plus device can be used normally.



Labeled Components: A=injector to PreCon device, B=trap 3 to GC column

Figure 11-10. Connection of PreCon device to GasBench Plus device for parallel operation

Alternatively, the autosampler of the GasBench Plus device can be used with the PreCon device, if the sample bottle is removed and the sampling needle is connected to the sample bottle ports. In this setup, it is not possible to use the GasBench Plus device normally.



Labeled Components: A=injector to PreCon device, B=trap 3 to GC column

Figure 11-11. Alternative setup where PreCon device uses autosampler of GasBench Plus device^a

^a x must be connected to x, and y to y.

Connecting the PreCon Device to the GasBench Plus Device

Tip Before attaching the PreCon device to the GasBench Plus device, make sure that the inlet valve of the mass spectrometer is closed and the helium supply at the GasBench Plus device and the PreCon device is shut off.

The mechanical position of the PreCon device is usually on the left hand side of the GasBench Plus device. Connect all peripherals to compressed air, electronics, helium and power supplies. The helium quality should be according to the pre-installation requirements.

❖ To connect the PreCon device to the GasBench Plus device

1. Connect the capillary designated as “Injection” (see [Figure 11-12](#)) to the vent port of the Valco valve of the GasBench Plus device (see [Figure 11-13](#)).

2. Connect the capillary designated as “Column” to the Column inlet of the GasBench Plus device. See [Figure 11-14](#).



Figure 11-12. PreCon inlet and outlet connections

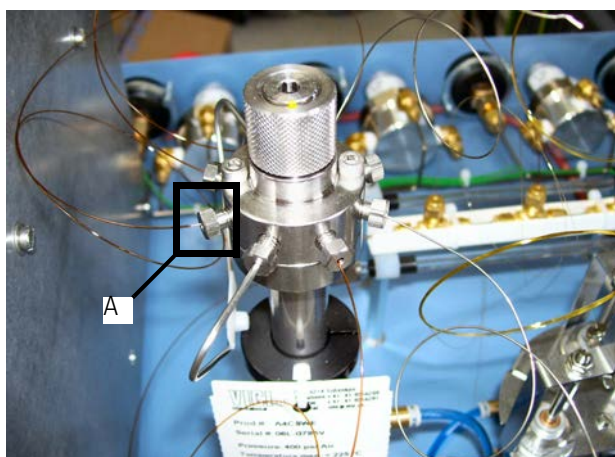


Figure 11-13. PreCon connection (A) at Valco valve of GasBench Plus device

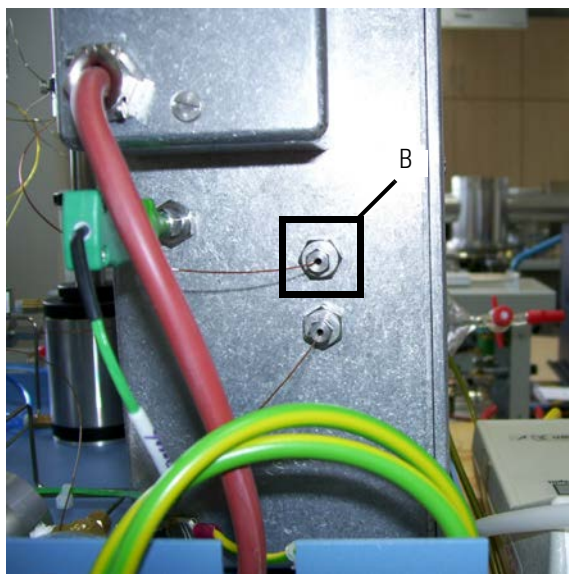


Figure 11-14. GasBench Plus-GC input connection (B)

Checking the Helium Supply Lines for Leaks

This check can only be performed, if a pressure meter is available in the main helium supply line to the PreCon/GC/C or PreCon/GC/GP.

❖ **To check the helium supply lines for leaks**

1. Adjust the main supply to 5 bar.
2. Close all helium pressure reduction units at the front panels of the PreCon device and the GasBench Plus device.
3. Close the main supply. The pressure may not drop within 30 minutes or longer.

Catalyst for Hydrogen Equilibration

The catalyst required for hydrogen equilibration is a platinum stick that is partly covered with platinum powder. See [Figure 11-15](#). It is delivered in a box containing 50 pieces.

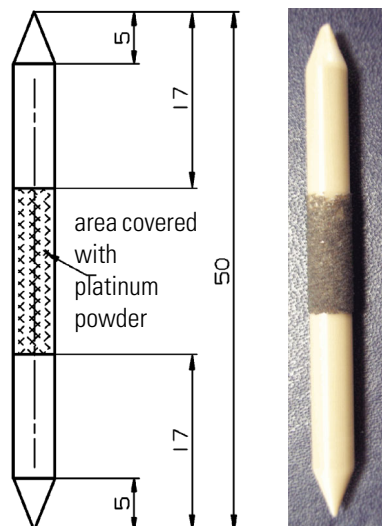


Figure 11-15. Catalyst for hydrogen equilibration^a

^a The distances are given in mm.

Cleaning the Platinum Sticks

Platinum sticks are used in HD equilibration technique (see “[Water Equilibration \(²H/¹H Equilibration\)](#)” on [page 8-12](#)). After each contact to the sample matrix, that is pure water or aqueous solutions, the platinum sticks must be cleaned with deionized water and dried appropriately to avoid any contamination with the previous sample or with remnants of inorganic or organic matter.

The platinum sticks provided by Thermo Fisher Scientific consist of Hokko beads, an epoxy resin and PEEK™ material. Each of these materials has its individual maximum cleaning temperature and chemical resistance. The color of the platinum does not reveal the degree of the catalytic capacity. The operating temperature of the platinum sticks ranges between 5 °C and 80 °C.

The maximum decomposition temperature of the Hokko beads is specified as 280 °C vs. a temperature range of the adherent epoxy glue between -40 and 100 °C.

The Hokko beads are resistant against humidity, oil, diluted acids, alkaline bases and many other organic solvents. Humidity, diluted acids and bases as well as mineral oil hardly impair the cohesive strength of the glue, even after longer dwelling times. Nevertheless, according to the

Other Options

Catalyst for Hydrogen Equilibration

manufacturer no general specification can be given, because multiple factors like chemical and physical influences, dwelling time and temperature affect the adhesive properties of the glue structure.

The epoxy glue is resistant to aging and against atmospheric influences. Cooling, even very low temperatures, do not affect it.

❖ To clean platinum sticks

1. Put on protective gloves to protect the platinum sticks from dirt.
2. Put the used platinum sticks into a clean beaker filled with pure distilled water at room temperature.
3. Rinse the platinum sticks as follows:
 - a. Leave the platinum sticks in the water.
 - b. Slowly and carefully pour the supernatant water (above the platinum sticks) out along the spout of the beaker.
 - c. Prevent the platinum sticks from falling out of the beaker by using, for example, a Petri dish.
 - d. Add new distilled water so that the platinum sticks are entirely covered.
4. Repeat [step 3](#) until no dirty sample matrix is visible anymore.

Aqueous samples like sea water, salt water from geological evaporates or drill mud, and/or particulate organic matter (urine, blood plasma, for example) require the addition of a worchester of pure copper chips to deactivate sulfates and pure activated charcoal without additive to scavenge organic material.

The copper chips and the charcoal are added to the sample before analysis and stay in the sample vial during analysis. After analysis, carefully remove all remnants of copper chips and activated charcoal from the platinum sticks during cleaning.

5. If medical (urine, blood plasma, for example) or dirty aqueous sample matrices have been used, put the platinum sticks into a clean beaker filled with distilled water and heat it up to maximum 80 °C.

- Put the platinum sticks into a wider beaker. Put the beaker into a drying oven and heat it to maximum 80 °C.

Tip Take care that the sticks do not get in contact with each other. This shortens the drying period in the oven. A rack helps avoiding mutual contact of the sticks.

If the sticks will not be used for a longer time, store them in aluminum foil within a plastic bag or a polypropylene box.



Check the chemical and physical resistance of one exemplary platinum stick if you are using any other aqueous matrix than mentioned in this manual. Do not use a full set of all sticks.

Thermo Fisher Scientific assumes no responsibility and will not be liable for the use of an unusual sample matrix that might decompose the sticks.

Other Options

Catalyst for Hydrogen Equilibration

Troubleshooting

Contents

- [Safety Guidelines for Troubleshooting](#) on page 12-2
- [Fault Table](#) on page 12-2
- [Unclogging the Sample Needle](#) on page 12-2
- [Common Pitfalls](#) on page 12-3

Safety Guidelines for Troubleshooting

When performing troubleshooting on the GasBench Plus device, pay attention to the following general safety guidelines.

WARNING

High Voltage. High voltages capable of creating an electric shock are used in the instrument. Do not remove protective covers from PCBs. Opening the instrument housing is only allowed for maintenance purposes by Thermo Fisher Scientific personnel.

To ensure that the instrument is free from all electric current, always disconnect the power cord before you try any type of maintenance.



Service by the customer must be performed by trained qualified personnel only and is restricted to servicing mechanical parts. Service on electronic parts must be performed by Thermo Fisher Scientific field service engineers only.

Do not try to repair or replace any component of the system that is not described in this manual without the assistance of your Thermo Fisher Scientific field service engineer.

Fault Table

If malfunctions on the system occur, you find possible causes and instructions for repair in [Table 12-1](#).

Table 12-1. Troubleshooting

Problem	Possible Causes	Remedy
Qtegra shows bad vacuum	System was vented because of a mains failure	page 6-2
	A leak exists	page 6-2
Check Measurement Needle	Sample needle is clogged	page 12-2
Avoid Clogging Acid Pump	Acid crystallization	page 9-13

Unclogging the Sample Needle

The sample needle might be clogged by and by due to phosphoric acid crystallizing within the fused silica capillary.

❖ To unclog the sample needle

1. Detach the fused silica capillary from the GasBench Plus device to prevent destroying the Nafion™ polymer of the water trap.
2. Fill an Exetainer™ with isopropanol.
3. Heat it up to approximately 40 °C.

4. Use the pressure of the helium flow from the GasBench Plus device through the stainless steel capillary to pressurize the Extainer.

If you have a setup of the GasBench Plus device with a flush needle for flushing Extainers with gas (as used for water equilibration), you can connect the stainless steel capillary even to this gas line and use a higher pressure. Be careful and put a beaker under the end of the fused silica capillary because isopropanol will run very fast as soon the line is unclogged.

5. A 0.1 mm ID capillary can be moved through the side hole or the bottom end of the needle. Accidentally deposited septa butyl rubber from pinching might be removed that way.

Common Pitfalls

In this section, some failures possible with the GasBench Plus device are described. You are responsible for cleanliness of all lines and leak-tightness. The GasBench Plus device allows long time highest performance. It is explained how a chromatogram of GC PoraPLOT™ gas separation looks like using the GasBench Plus device. The addition of a chromatogram - visual as one - allowing peaks from the first injection to appear under the injection chromatogram of the third one, is described.

Retention Times

When you must create or modify the methods used for any of the possible applications, the timing of the injection events becomes important. To understand the required timing, you must understand how the substances injected to the chromatographic column (a PoraPLOT™ Q is used as default) behave. As a general statement, substances are delayed relative to the carrier gas (usually helium) while travelling along the column. This delay is called retention time, and on a PoraPLOT Q column, the retention is higher if the substance is more polar.

Figure 12-1 shows a sample chromatogram for a PoraPLOT Q chromatographic column. Retention times are given for several substances. For example, ethanol has a retention time of 3.73 min, equal to 224 s. Keep in mind that retention times are strictly temperature and flow dependent. The retention time for ethanol in the example below is given at a column temperature of 150 °C. Other column temperatures cause other retention times.

In general, lower temperatures yield higher retention times. As an extreme, if the temperature is below a certain limit, the substance may not leave the column. This is the case for instance for water, if the column temperature is 21 °C.

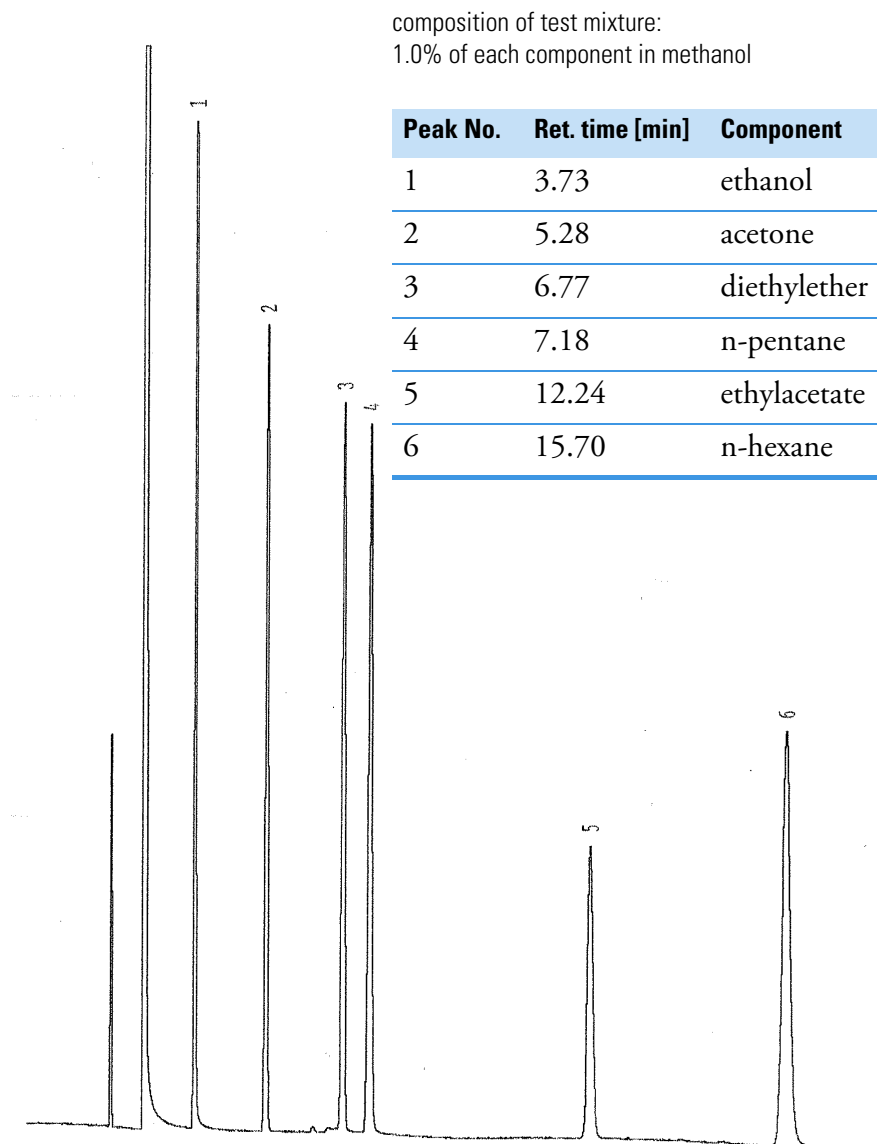


Figure 12-1. Sample chromatogram for PoraPLOT™ Q column

The substances that are of interest in GasBench Plus applications, namely CO₂, H₂O, Ar, O₂ and N₂, have much shorter retention times than the methanol given above. Under the normal conditions that we set in the GasBench Plus device, that is flow 1.5 mL/min and temperature 70 °C, O₂ and N₂ arrive in the mass spectrometer about 120 seconds after the injection. CO₂ arrives after 150 seconds.

If water is present in the substance mix in the loop, it arrives after about 300 s. The situation is complicated furthermore by the repeated loop injection that is normally performed to enhance the accuracy of the measurement. This repeated loop injection causes the peaks to “stack” in the chromatogram thus sometimes hiding important impurities. This is given in [Figure 12-2](#) which shows combined chromatograms.

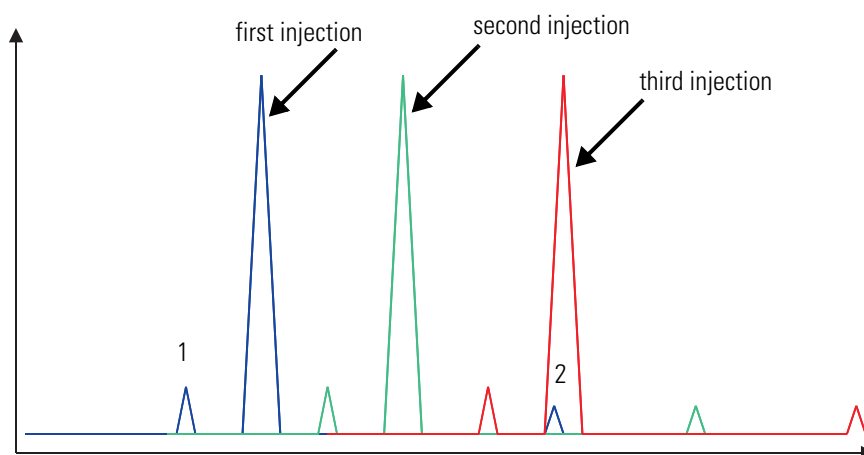


Figure 12-2. Upon retention times - combining chromatograms

The following features are then typical for the chromatogram:

1. Air peak precedes CO₂. See **1** in [Figure 12-2](#).
2. Water peak may follow CO₂ but must not interfere with CO₂. See **2** in [Figure 12-2](#).
3. Additional peaks, for example due to solvents, must not interfere with CO₂.

Wasting Acid

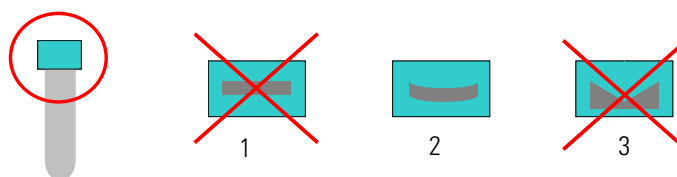
Due to an improper adjustment of the acid pump, acid drops might be deposited on the septa. If so, acid can enter the sample needle and travel towards the Valco valve.

NOTICE

Under all circumstances, prevent the acid from entering the sample needle. Damage to the water trap and the Valco valve would result. See [“Adjusting the Acid Pump”](#) on [page 9-10](#).

Handling the Septa

Make sure that the sample vials are screwed down correctly to be really closed. See [Figure 12-3](#).



Labeled Components: 1: is not leak tight, 2: fits correctly, 3: develops fissures and tends to be cut by the needle

Figure 12-3. Correct handling of septa

Water Condensation below the Septa

During the equilibration, when the tray temperature is only slightly above the room temperature, water vapor condenses below the septa. This effect is unavoidable and usually poses no problem. When the septa have been punctured by the needle, these water droplets accumulate to one large drop. If now this particular vial is measured again, there is a significant chance to pick up this drop. This leads to water traveling towards water trap and Valco valve, possibly clogging the system.

Tip Therefore, do not measure equilibrated samples twice.

Maintenance

This chapter describes maintenance procedures that must be performed by the user to ensure optimum performance of the GasBench Plus device.

Contents

- [Safety Guidelines for Maintenance](#) on page 13-2
- [General Advice for Maintenance](#) on page 13-3
- [Inspection- and Servicing Plan](#) on page 13-4
- [Maintaining the GC Column](#) on page 13-5

Safety Guidelines for Maintenance

When performing maintenance on the GasBench Plus system, pay attention to the following general safety guidelines.

WARNING

High Voltage. High voltages capable of causing an electric shock are used in the instrument. Do not remove protective covers from PCBs.

Before opening the GC housing, switch off the GasBench Plus at the right side panel and unplug the 230 V power supply cable.

To ensure that the instrument is free from all electric current, always disconnect the power cord before you try any type of maintenance.

CAUTION

Hot Surface. When the GC oven is operating (during a heating out period), the temperature of the heater located inside is above 100 °C. Touching the GC oven or the heater might cause severe burns. Stay away from the GC oven and the heater. Let them cool to ambient temperature before you work on them.

CAUTION

Hot Surface. When the thermostatted tray (“heated tray”) is operating, touching it might cause severe burns. Stay away from the thermostatted tray. Let it cool to ambient temperature before you work on them.

CAUTION

Hazardous Chemicals. Samples and solvents might contain toxic, carcinogenic, mutagenic, or corrosive/irritant chemicals. Avoid exposure to potentially harmful materials.

Always wear protective clothing, gloves, and safety glasses when you handle solvents or samples.

Contain waste streams and use proper ventilation. Refer to your supplier's Material Safety Data Sheet (MSDS) for proper handling of a particular compound.



It is the user's responsibility to maintain the system properly by performing the system maintenance procedures on a regular basis.

Service by the customer must be performed by trained qualified personnel only and is restricted to servicing mechanical parts. Service on electronic parts must be performed by Thermo Fisher Scientific field service engineers only.

Do not try to repair or replace any component of the system that is not described in this manual without the assistance of your Thermo Fisher Scientific field service engineer.

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

General Advice for Maintenance

When performing maintenance on the GasBench Plus mass spectrometer, observe the following advice:

- Accurate results can be obtained only if the system is in good condition and properly calibrated.
- Preventive maintenance must commence with installation, and must continue during the warranty period to maintain the warranty. Thermo Fisher Scientific offers maintenance and service contracts. Contact your local Thermo Fisher Scientific office for more information. Routine and infrequent maintenance procedures are listed in [Table 13-1](#).
- The user maintenance procedures described in this manual do not require removing the instrument housing. Thermo Fisher Scientific assumes no responsibility and will not be liable for instrument damage and/or operator injury that might result from using the instrument without the housing attached. Therefore, call a Thermo Fisher Scientific field service engineer if removal of the instrument housing is required.
- To successfully carry out the procedures listed in this chapter, observe the following rules:
 - Proceed methodically
 - Always wear clean, talc-free, and lint-free gloves when handling the components of the API source, ion optics, mass analyzer, and ion detection system. See “[Personal Protective Equipment](#)” on [page 4-22](#) for a specification for the required gloves.
 - Always place the components on a clean, lint-free surface.
 - Do not overtighten a screw or use excessive force.
 - Dirty tools can contaminate your system. Keep the tools clean and use them exclusively for maintenance and service work at the mass spectrometer.
 - Do not insert a test probe (for example, an oscilloscope probe) into the sockets of female cable connectors on PCBs.

Inspection- and Servicing Plan

Routine and infrequent maintenance procedures to be performed by the user are listed in [Table 13-1](#).

Table 13-1. User maintenance procedures

MS Component	Procedure	Frequency	Procedure Location
Instrument	Leak check gas lines	Annually	page 13-4

Cleaning the Surface of the Instrument

NOTICE

Prevent any liquids from entering the inside of the instrument. Leaking liquids might get into contact with electronic components and cause a short circuit.

Clean the outside of the instrument with a dry cloth. For removing stains or fingerprints on the surface of the instrument (panels, for example), slightly dampen the cloth (preferably made of microfiber) with distilled water.

Checking the Gas Lines for Leaks

Regularly leak check each gas line from the gas supply in the laboratory to the instrument.

❖ To perform a leak check for a gas line

1. After closing all valves in the instrument, monitor the manometer of the gas regulator for some minutes.
2. If the pressure falls significantly (for example, the nitrogen pressure falls by more than 10 psi / 690 mbar within two minutes), you should search for leaks in the gas line.
3. Search for leaks in the gas line (for example, by using a conventional thermal conductivity-based leak detector, such as is widely used to check leaks in gas chromatography equipment).
4. If you detect a leak (which is usually at a connection), verify the tightness of the connection. In case of doubt, replace it.
5. When you cannot find a leak in the gas line, we recommend calling a Thermo Fisher Scientific field service engineer to check for gas leaks within the instrument.

Maintaining the GC Column

Currently, the GC column is a static part of the GasBench Plus device because it rarely needs to be exchanged. Only maintenance is necessary from time to time. The GC oven is located at the right side of the GasBench Plus (front view).

Step 1 - Accessing the GC Column

❖ To access the GC column

1. When inserting the GC column for the first time or when exchanging it, first remove the cover of the GC oven (that is right side panel of the GasBench Plus). To do so, loosen all seven screws with a hex wrench. See [Figure 13-1](#).



Figure 13-1. GasBench Plus device - opening right side panel

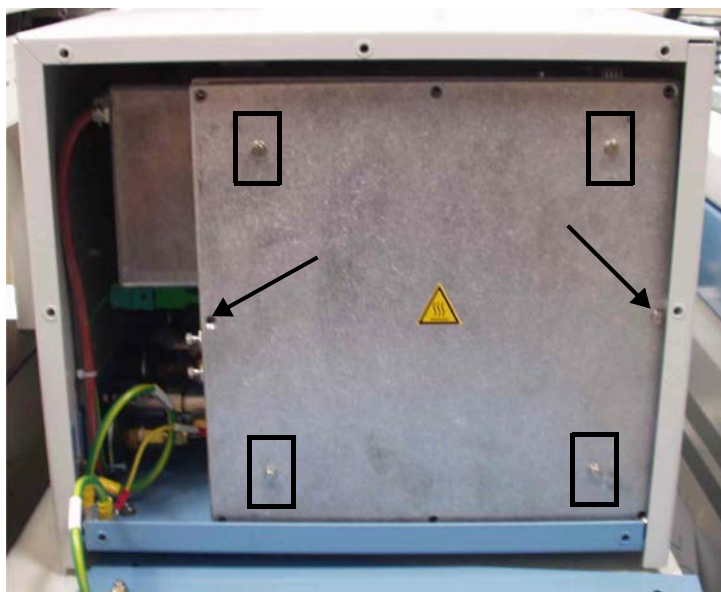


Figure 13-2. GasBench Plus device - right side panel removed

2. Remove the two screws marked by arrows in [Figure 13-2](#).

NOTICE

Leave the remaining four screws untouched that are marked by rectangles in [Figure 13-2](#), because they hold the insulation of the GC oven.

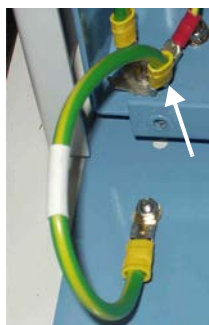


Figure 13-3. Grounding cable of right side panel

Each side panel has a grounding cable of its own to guarantee electrical security. Each grounding cable must be connected as shown in [Figure 13-3](#) as an example for the right side panel. Furthermore, the top side and the oven housing are grounded as well.

Step 2 - Exchanging the GC Column

The GC column is now visible and consists of two parts:

- the functional part (light yellow; **3** in [Figure 13-4](#)). It is the packed part of the column, that is the plot part.

- the post-column (nearly transparent; **1** in Figure 13-4)

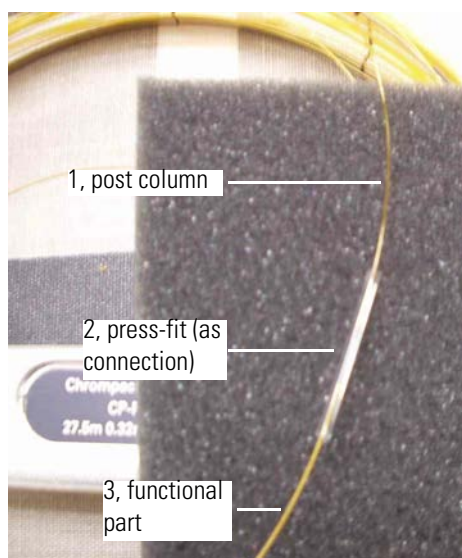
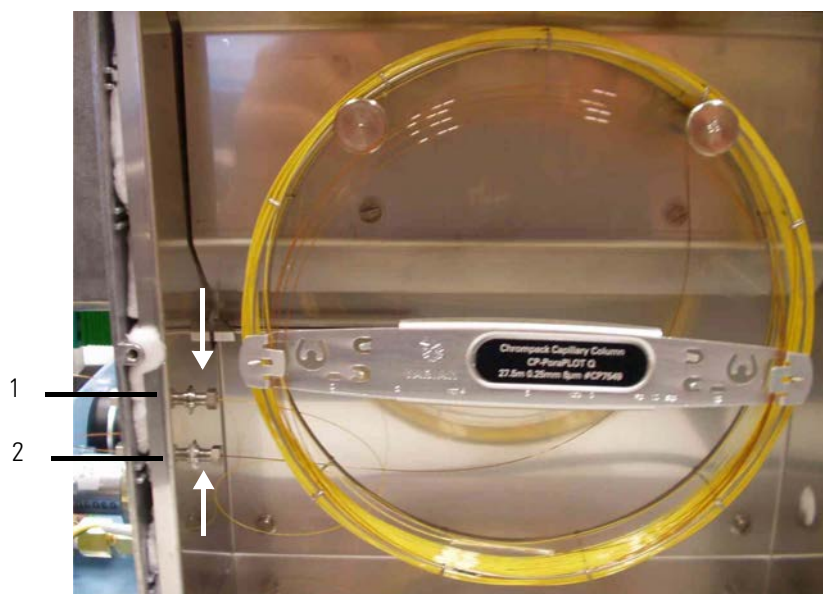


Figure 13-4. Junction between both parts of GC column

The connection between both parts is established by a press-fit (**2** in Figure 13-4).



Labeled Components: 1=GC in, 2=GC out

Figure 13-5. GC column installed^a

^a By default, the column is installed as 1=GC in and 2=GC out.

Maintenance

Maintaining the GC Column

Figure 13-6 shows the ends of the column, which are blocked by silicon plugs.

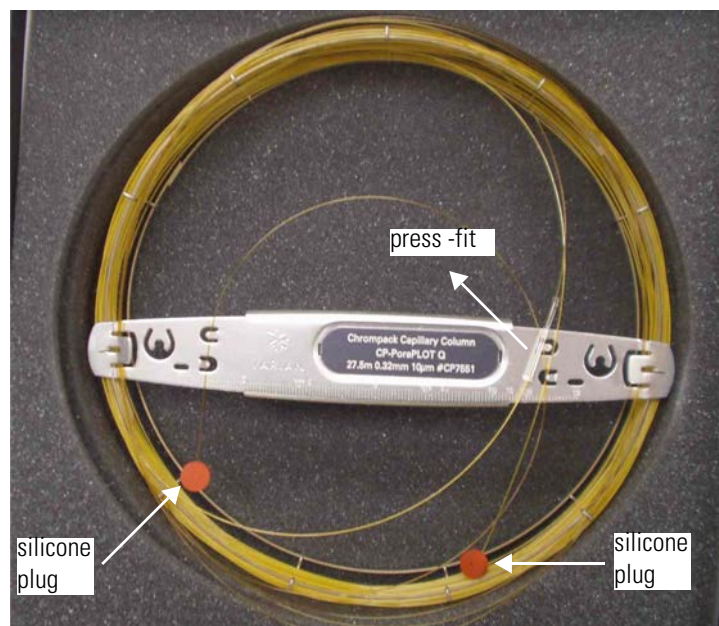


Figure 13-6. GC column with blocked ends

❖ To exchange the GC column

1. Cut the silicon plugs off using a capillary column cutter (wafer).
2. Insert each end into its bulkhead connection at the left side of the oven (**1** and **2** in Figure 13-5) as follows:
 - a. The bulkhead connection, which is connected to the Valco valve, is intended for the inlet of the column. It must be connected to the functional part of the column (light yellow).
 - b. As the outlet, the post-column must be connected to the bulkhead connection that is directed towards the water trap 2. The water trap, in turn, leads to the diluter, that is to the open active split.

The post-column (nearly transparent) acts as a particle trap, that is it prevents particles from reaching rear valves.

3. Screw the Swagelok™ connection or Valco valve connection on as follows:
 - a. Insert the respective ferrule.
 - b. Newly cut the capillary off.
 - c. Introduce the capillary.
 - d. Carefully tighten the ferrule until the capillary can no longer be pulled back. Do not tighten the ferrules/connections too strong.

NOTICE

If you want to tighten the ferrule further, you must perform a leak check first. Only tighten it further, if gas is still coming in after the leak test has been performed.

Act extremely carefully while opening and closing connections. Do not tighten any Swagelok™ connection around the column too strong because it will be destroyed.

Only connections made up by metal ferrules can be tightened strongly. Normally, the only connections to be touched by users are those of the column, the loop and for installing a flush needle or a sample needle, respectively.



For detailed information about how to install the column in the GC oven, about conditioning, storage and description refer to the *Capillary Column Test Report* by Varian/Chrompack. It is part of your Varian/Chrompack capillary column.

Returning Parts

CAUTION

Hazardous Chemicals. Hazardous material might contaminate certain parts of your system during analysis. To protect our operating personnel, we ask you to adhere to special precautions when you send back parts to the factory for exchange or repair.

If hazardous materials have contaminated instrument parts, Thermo Fisher Scientific can only accept these parts for repair if they have been properly decontaminated.

Materials that might be toxic due to their structure and the applied concentration or which are reported in publications to be toxic are regarded as hazardous. Materials that will cause synergetic hazardous effects in combination with other present materials are also considered hazardous.

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

Your signature on the Health and Safety Form confirms that the returned parts have been decontaminated and are free of hazardous materials.

You can download this form from the SharePoint. See “[Contacting Us](#)” on [page 1-5](#). Instead of copying or printing this page, request a copy from the Thermo Fisher Scientific field service engineer.

Maintenance

Maintaining the GC Column

Legal Documents

Contents

- [EU REACH Statement](#) on page A-1
- [WEEE Compliance](#) on page A-2
- [EU Declaration of Conformity](#) on page A-3
- [UK Declaration of Conformity](#) on page A-4

EU REACH Statement

The European Commission promulgated legislation that covers the registration, evaluation, authorization and restriction of chemicals within the European Union community under (EC) No 1907/2006. This regulation is commonly known as REACH. Thermo Fisher Scientific is committed to meeting all compliance obligations under REACH. As per Article 33 of the Regulation, this product may include items which contain more than 0.1% by weight of some SVHC Candidate Substance. Some electronic parts and copper alloys can contain lead.

WEEE Compliance

This product is required to comply with the European Union's Waste Electrical & Electronic Equipment (WEEE) Directive 2012/19/EU. It is marked with the following symbol:



Thermo Fisher Scientific is registered with B2B Compliance (B2Bcompliance.org.uk) in the UK and with the European Recycling Platform (ERP-recycling.org) in all other countries of the European Union and in Norway.

If this product is located in Europe and you want to participate in the Thermo Fisher Scientific Business-to-Business (B2B) Recycling Program, send an email request to weee.recycle@thermofisher.com with the following information:

- WEEE product class
- Name of the manufacturer or distributor (where you purchased the product)
- Number of product pieces, and the estimated total weight and volume
- Pick-up address and contact person (include contact information)
- Appropriate pick-up time
- Declaration of decontamination, stating that all hazardous fluids or material have been removed from the product



This recycling program is not for biological hazard products or for products that have been medically contaminated. You must treat these types of products as biohazard waste and dispose of them in accordance with your local regulations.

EU Declaration of Conformity

-Original-

EU-Konformitätserklärung EU Declaration of Conformity



ThermoFisher
SCIENTIFIC

Thermo Fisher Scientific (Bremen) GmbH
Hanna-Kunath-Str. 11
28199 Bremen, Germany

Wir erklären hiermit, dass die folgenden Produkte
We hereby declare that the following products

Bezeichnung: <i>Designation:</i>	Massenspektrometer <i>Mass Spectrometer</i>
Modell: <i>Model:</i>	Thermo Scientific Delta Serie <i>Thermo Scientific Delta Series</i> <i>(Delta V Plus, Delta V Advantage, Delta Q)</i>

alle einschlägigen Anforderungen der folgenden Richtlinien erfüllen:
fulfill all the relevant requirements of the following directives:

Niederspannungsrichtlinie <i>Low Voltage Directive</i>	2014/35/EU <i>2014/35/EU</i>
Richtlinie über elektromagnetische Verträglichkeit <i>Electromagnetic Compatibility Directive</i>	2014/30/EU <i>2014/30/EU</i>
RoHS-Richtlinie <i>RoHS Directive</i>	2011/65/EU and (EU) 2015/863 <i>2011/65/EU and (EU) 2015/863</i>

Die folgenden einschlägigen harmonisierten Normen wurden zugrunde gelegt:
The following relevant harmonized standards were used:

EN 61010-1:2010/A1:2019 <i>EN 61010-1:2010/A1:2019</i>	EN 61326-1:2013 <i>EN 61326-1:2013</i>
--	--

Für die Zusammenstellung der technischen Unterlagen ist bevollmächtigt:
Person authorized to compile the technical file:

Rainer Bröring (Geschäftsführer)
Thermo Fisher Scientific (Bremen) GmbH


Unterschrift
Signature

Bremen, September-13, 2021

Datum
Date

UK Declaration of Conformity

-Original-

UK Declaration of Conformity



ThermoFisher
SCIENTIFIC

Thermo Fisher Scientific (Bremen) GmbH
Hanna-Kunath-Str. 11
28199 Bremen, Germany

Declares, under sole responsibility, that products

Designation: Mass Spectrometer

Model: Thermo Scientific Delta Series
(Delta V Plus, Delta V Advantage, Delta Q)

as originally delivered complies with the essential requirements of the following applicable UK Regulations:

Electrical Equipment (Safety) Regulations 2016

Electromagnetic Compatibility Regulations 2016

The Restriction of the Use of Certain Hazardous Substances in Electrical and Electronic Equipment (ROHS) Regulations 2012

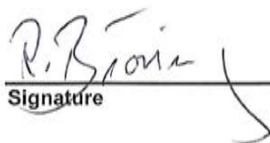
and complies with the following harmonized standards and other technical specifications:

BS EN 61010-1:2010

BS EN 61326-1:2013

Signed for and on behalf of: Thermo Fisher Scientific (Bremen) GmbH:

Rainer Bröring (Geschäftsführer)
Thermo Fisher Scientific (Bremen) GmbH


Signature

Bremen, 2021-09-20

Date

Accessing the Technical Documentation SharePoint

Use the Technical Documentation SharePoint to download current revisions of user manuals and other customer-facing documents for your product. Translations into other languages may be available as well. The SharePoint also provides access to videos and software.

Contents

- [Creating a User Account](#) on page B-1
- [Accessing the SharePoint for the First Time](#) on page B-3
- [Accessing the SharePoint as registered Customer](#) on page B-5
- [Using the SharePoint](#) on page B-6

Creating a User Account

❖ To create a user account

1. Click www.thermofisher.com/Technicaldocumentation or enter that address manually.

Your Internet browser displays a Start page.

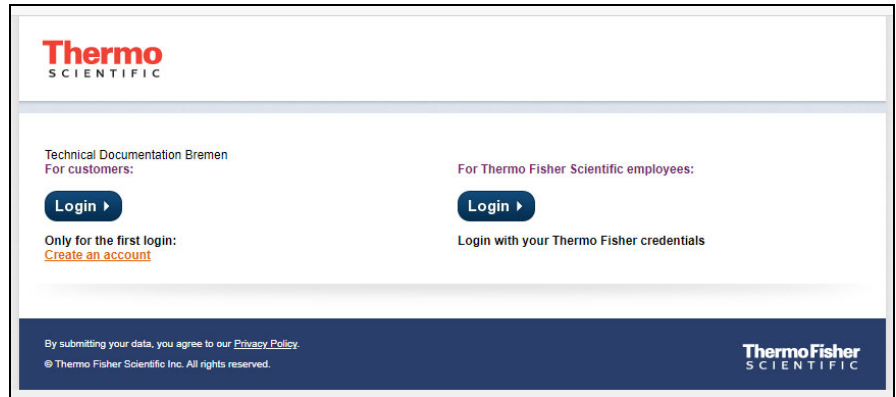


Figure B-1. Start page

2. Click [Create an account](#).

Your Internet browser displays a page with a short guide.

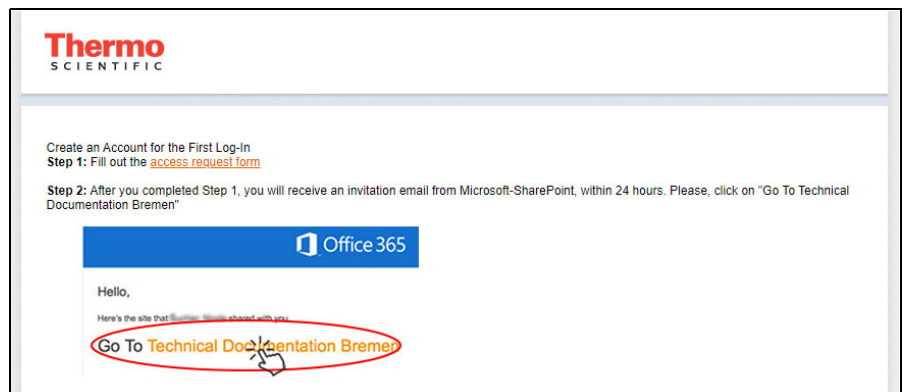


Figure B-2. Guide

3. Click the link in Step 1 of the guide to display the [access request form](#).

Your Internet browser displays the access request form.

Thermo Scientific

Technical Documentation Bremen
Please fill in the form to request access the Technical Documentation Bremen system.
Your request will be sent to our technical documentation group. You will soon receive an email with log in information.

First Name: *

Last Name: *

Company: *

Country: * -- Please select --

Email Address: *

Instrument Group: * -- Select Instrument Group --

LSMS: Exactive-, LTQ FT- and LTQ Orbitrap Series

Gas IRMS Instruments: Delta Series, Breath MAT Plus, MAT 252 and MAT 25

Gas IRMS Peripherals: ConFlo, Dual Inlet, Element Analyzers, Gas Bench, GC-TC, GC Isolink, H-Device, Kiel IV Carbonate Device, LC IsoLink, µ-Volumn

Service Number: (begins with SN)*

Submit Form ▶

By submitting your data, you agree to our [Privacy Policy](#).
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ThermoFisher Scientific

Figure B-3. Access request form

4. Enter the requested data in the appropriate fields. Select the requested entries in the list boxes for Country and Instrument Group.

All entries are mandatory. Provide the email address that you use in your company or institution. Do not provide any private email addresses.

Tip This form requests for a Service Number. Use the **Serial** Number instead. The Serial Number of your instrument is given on the name plate attached to your instrument. Refer to the Operating Manual for the location.

5. When you are finished, click **Submit Form** to send your data to Thermo Fisher Scientific.

Thermo Fisher Scientific will check your data to make sure that you are a legitimate customer. When the check is successful, you will receive an invitation to the SharePoint. Now, you have six days to log into the SharePoint with your Microsoft™ password.

Accessing the SharePoint for the First Time

The SharePoint requires that you have a Microsoft account for the email address that you use in your company or institution. If you have not already done so, create a Microsoft account by following the instructions on the Microsoft website.

Tip If your computer runs with Windows 10, you have most likely set up a Microsoft account during the installation.

❖ To access the SharePoint for the first time

1. On the invitation email from the Microsoft SharePoint, click **Go To Technical Documentation Bremen**.

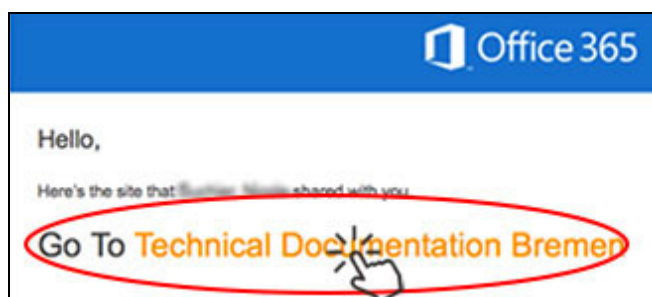


Figure B-4. Invitation email

2. On the Welcome page, click [Create a Microsoft SharePoint Online account](#).

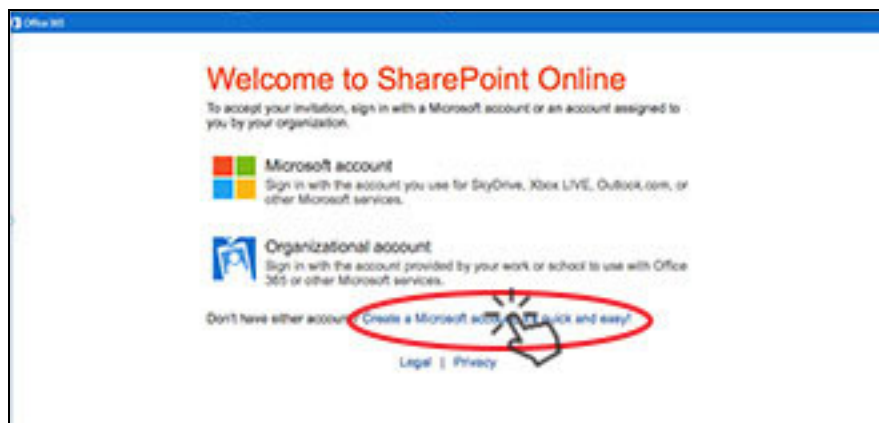


Figure B-5. Welcome page

3. Click **Microsoft account**.



Figure B-6. Choosing Microsoft account

4. Log in with your Microsoft credentials. Enter the email address and the password.

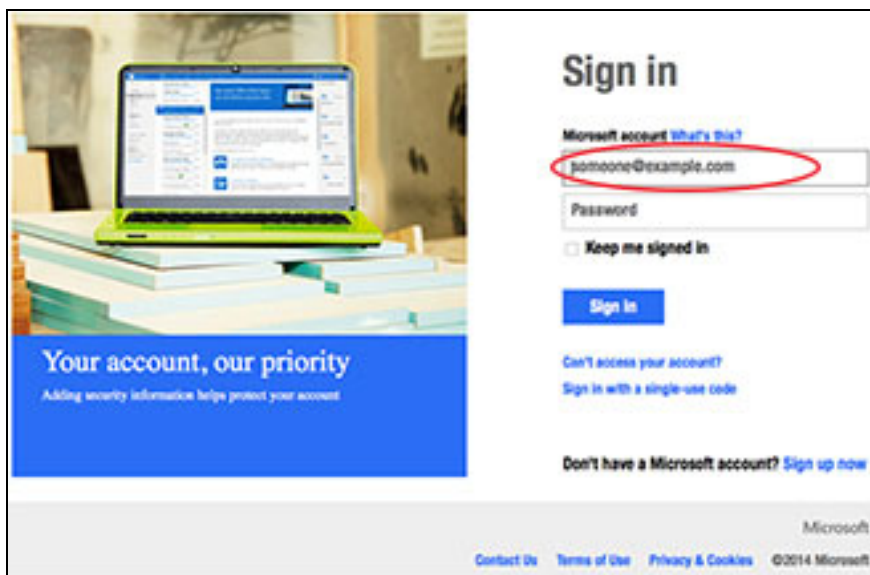


Figure B-7. Signing in

5. Click **Sign In**.

Accessing the SharePoint as Registered Customer

❖ To access the SharePoint as a registered customer

1. Click www.thermofisher.com/Technicaldocumentation or enter that address manually.
2. On the Start page, click the **Login** button on the **left** side.

Accessing the Technical Documentation SharePoint

Accessing the SharePoint as Registered Customer

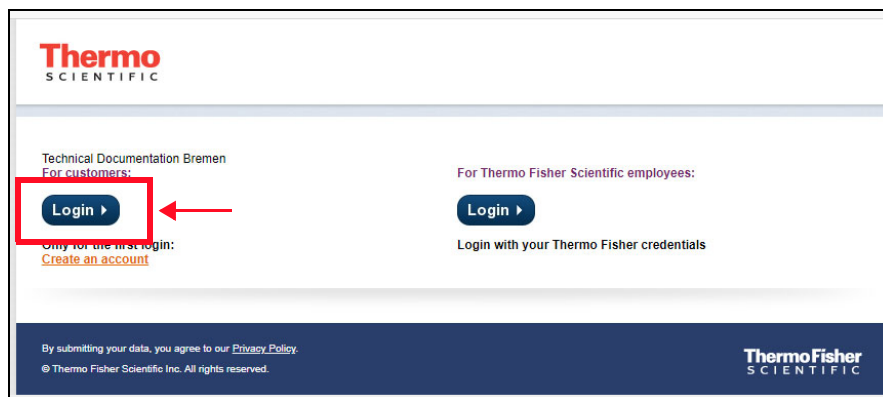


Figure B-8. Customer login

3. On the Login page, click [Technical Documentation Bremen Site](#).

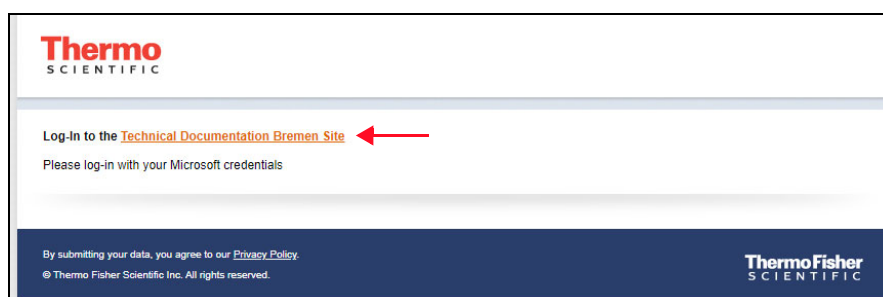


Figure B-9. Login page

4. Your Internet browser displays the Start page of the Technical Documentation SharePoint.

You have access to documents that are relevant for the instrument that you have registered at “[Creating a User Account](#)” on [page B-2](#) and to documents that are relevant for all users. The figure shows the Start page for a user who has registered a multicollector ICP-MS (a Neptune MS, for example).

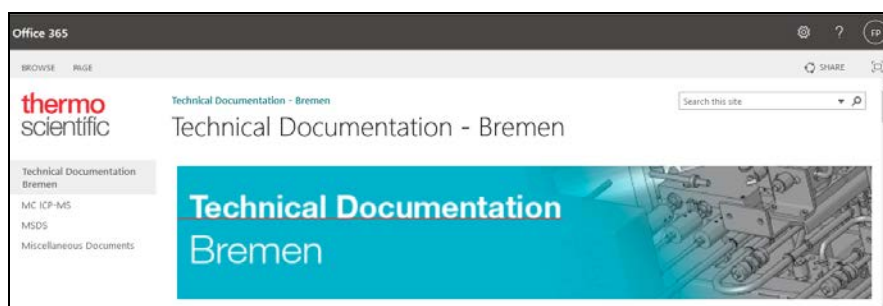


Figure B-10. Start page of the Technical Documentation SharePoint

Using the SharePoint

On the SharePoint, you can use either the links on the Navigation pane on the left or the links on the Content pane on the right.

If an instrument group contains several instruments, click the tiles to go to the page of your instrument.



Figure B-11. Instrument Group page

On an instrument page, the available documents are displayed in tables. Click the title of a column to sort the entries or to apply a filter. If more than five documents are available for your instrument, a scroll button appears at the bottom of the table. To download a document, right-click the entry under Name and choose **Save Link as**.

English Documents				
<input type="checkbox"/> Document type	Title	Information	Type	Name
Others				1250910_Jet_Interface_User_Guide_Rev_A
Others	High-Resolution Peak center			1378220_HighResolutionPeakCenter_Manual_Rev_A
Others	UK Declaration of Conformity			2020_DoC_UK_Neptune Series
Operating Manual	GCI 300 Transfer Line Operating Manual			BRE0006945_GCI 300_Operating Manual_RevD
Operating Manual	Neptune Series Operating Manual	1250710, Rev. G		NeptuneSeries_OM

6 - 10

Figure B-12. Available documents

Receiving Update Messages

You can configure the SharePoint to notify you when a page was updated.

❖ To activate the alerts for a SharePoint page

1. Browse to the page that you want to have alerts for.
2. Click the Page tab in the top left corner to display the toolbar.

Accessing the Technical Documentation SharePoint

Receiving Update Messages

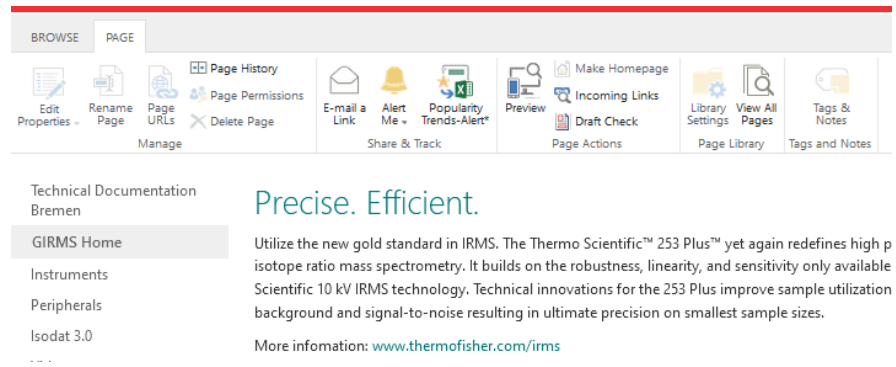


Figure B-13. Page tab

3. Click the **Alert Me** button to display the menu.



Figure B-14. Alert Me button

4. Choose **Set an alert on this page**.
5. On the alert settings dialog box, make the required changes. We recommend that you select a daily summary of the changes.

Site Pages: 253 Plus.aspx - New Alert

Alert Title
Enter the title for this alert. This is included in the subject of the notification sent for this alert.

Delivery Method
Specify how you want the alerts delivered.

Send Alerts for These Changes
Specify whether to filter alerts based on specific criteria. You may also restrict your alerts to only include items that show in a particular view.

When to Send Alerts
Specify how frequently you want to be alerted. (mobile alert is only available for immediately send)

Send me alerts by:

- E-mail john.doe@mycompany.com
- Text Message (SMS)
- Send URL in text message (SMS)

Send me an alert when:

- Anything changes
- Someone else changes a wiki page
- Someone else changes a wiki page created by me
- Someone else changes a wiki page last modified by me
- Someone changes an item that appears in the following view:
Created By Me

Time:
Wednesday 4:00 AM

Figure B-15. Alert settings dialog box

6. Click **OK** to save your changes and close the dialog box.
7. Depending on your settings, an email message or SMS message will confirm that you have activated the alert.

From now on, you will be notified when the page is updated.

Accessing the Technical Documentation SharePoint

Receiving Update Messages

Glossary

This section lists and defines terms used in this manual. It also includes acronyms, metric prefixes, symbols, and abbreviations.

A B C D E F G H I J K L M N O P Q R S T U V W X Y Z

- A**
- A** ampere
 - ac** alternating current
 - ADC** analog-to-digital converter
 - AP** acquisition processor
 - ASCII** American Standard Code for Information Interchange
- B**
- b** bit
 - B** byte (8 b)
 - baud rate** data transmission speed in events per second
- C**
- °C** degrees Celsius
 - cfm** cubic feet per minute
 - CI** chemical ionization
 - CID** collision-induced dissociation
 - cm** centimeter
 - cm³** cubic centimeter
 - CPU** central processing unit (of a computer)
 - CRC** cyclic redundancy check
 - CRM** consecutive reaction monitoring
- D**
- <Ctrl>** control key on the terminal keyboard
 - d** depth
 - Da** dalton
 - DAC** digital-to-analog converter
 - dc** direct current
 - driver** A device-specific control program that enables a computer to work with a particular device.
 - DS** data system
 - DSP** digital signal processor
- E**
- EI** electron ionization
 - <Enter>** Enter key on the terminal keyboard
 - ESD** electrostatic discharge
 - eV** electron volt
- F**
- f** femto (10^{-15})
 - °F** degrees Fahrenheit
 - forepump** The pump that evacuates the foreline. A rotary-vane pump is a type of forepump.
 - ft** foot
 - FTP** file transfer protocol

FWHM Full Width at Half Maximum

G

g gram

G Gauss; giga (10^9)

GC gas chromatograph; gas chromatography

GC/MS gas chromatograph / mass spectrometer

GUI graphical user interface

H

h hour

h height

HPLC high-performance liquid chromatograph

HV high voltage

Hz hertz (cycles per second)

I

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

ion optics Focuses and transmits ions from the ion source to the mass analyzer.

ion source A device that converts samples to gas-phase ions.

K

k kilo (10^3 , 1000)

K kilo (2^{10} , 1024)

kg kilogram

L

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

log file A text file, with a .log file extension, that is used to store lists of information.

n micro (10^{-6})

M

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 MSⁿ power: where n = 1

MS/MS MSⁿ power: where n = 2

MSⁿ MSⁿ power: where n = 1 through 10

m/z Mass-to-charge ratio. An abbreviation used to denote the quantity formed by dividing the mass of an ion (in u) by the number of charges carried by the ion. For example, for the ion C₇H₇²⁺, m/z=45.5.

N**n** nano (10^{-9})**NIST** National Institute of Standards and Technology (USA)**noise** Any random disturbance that obscures the clarity of a signal.**O****OD** outside diameter

X ohm

outlier A calibration data point that does not appear to correlate to other calibration data points within experimental error.**P****p** pico (10^{-12})**Pa** pascal**PCB** printed circuit board**PE** protective earth**PID** proportional / integral / differential**P/N** part number**P/P** peak-to-peak voltage**ppm** parts per million**psi** pounds per square inch**R****RAM** random access memory**relative standard deviation** A measure of the dispersion of a group of measurements relative to the mean of the group. Relative standard deviation is expressed as a percentage of the average value. The percent relative standard deviation is calculated as:

$$\%RSD = 100 (S / \bar{X})$$

where S is the [standard deviation](#) and \bar{X} is the sample mean.

RF radio frequency**RMS** root mean square**ROM** read-only memory**rotary-vane pump** A mechanical vacuum pump that establishes the vacuum necessary for the proper operation of the turbomolecular pump. (Also called a roughing pump or forepump.)**RS-232** An accepted industry standard for serial communication connections. This Recommended Standard (RS) defines the specific lines and signal characteristics used by serial communications controllers to standardize the transmission of serial data between devices.**S****s** second**serial port** An input/output location (channel) for serial data transmission.**standard deviation** In statistics, the standard deviation is a measure of the dispersion of a group of measurements. For example, masses, times, or intensities. Standard deviation is calculated as follows:

$$s = \sqrt{\text{Var}(x_1 \dots x_n)}$$

where Var ($X_1 \dots X_N$) is the variance.

See also [relative standard deviation](#).

T**TCP/IP** transmission control protocol / Internet protocol**TIC** total ion current**Torr** A unit of pressure, equal to 1 mm of mercury and 133.32 Pa.**turbomolecular pump** A vacuum pump that provides a high vacuum for the mass spectrometer and detector system.**U****u** atomic mass unit**UHV** ultra high vacuum

Glossary: V–W

V

V volt

V ac volts alternating current

V dc volts direct current

W

w width

W watt



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