Future Chemistry

Smart, safe and clean chemistry

Gas Module

Operator's Manual

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Contents

1. Introduction

In this manual, the installation and operation of the Gas Module will be discussed step by step. Keep these installation and operating instructions at hand for future reference. The Gas Module is designed to mix a gas with a liquid to be dispensed in the flowstart *Evo*, but it can also be used as a reactor on its own.

- WARNING -

The use of this module requires careful attention of the user, so please read this manual thoroughly before installing and using the Gas Module. Damage to the Gas Module due to improper use is not covered under warranty.

1.1 Part list Gas Module

All parts of Gas Module are indicated and numbered in the image below.

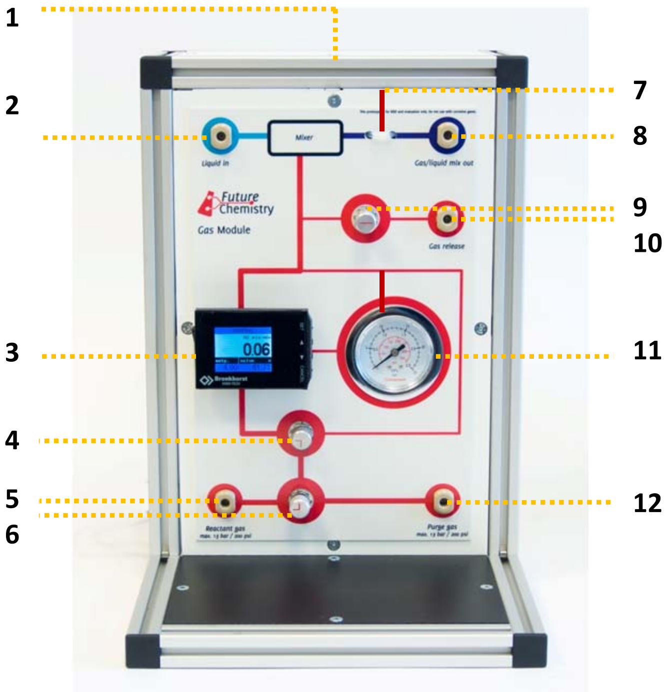

Figure 1.1: a schematic overview of the Gas Module.

1. Mixer
2. Liquid inlet
3. Gas flow controller
4. Valve B
5. Reagent gas inlet
6. Valve A
7. The "eye"
8. Liquid outlet
9. Valve C
10. Gas release
11. Manometer
12. Purge gas inlet

2 Installation Guide

In this section, the installation of the Gas Module will be discussed step by step. Read these installation instructions carefully before using the Gas Module. Follow the applicable safety instructions described below. Keep these installation instructions at hand for future reference.

2.1 Step 1: Parts Check and general preparations

Check the part list below to see if all the parts are included. If the supplied Gas Module is not corresponding to the part list, please contact us at support@futurechemistry.com. Place the instrument on a stable surface, e.g. fume hood. The Gas Module is manufactured from robust materials. However, avoid contact with liquids, chemicals, solvents, reagents etc. especially with the gas flow controller and the manometer. This to extend the lifetime of your instrument.

Table 1.1: Package contents.

Package name (SKU)	Package contents
Gas module 1 (B-480)	System
Mixer for Gas Module (B-481)	Special replaceable Mixer for mixing the gas with the liquid
Gas Inlet tubing (B-482)	Special Inlet tubing for the gas
Regulated Outlet (B-483)	Special Outlet for regulated dispensing of the gas/liquid to the pressure regulator
Gas Exhaust (B-484)	Special Gas Exhaust from the gas release
Power Supply (B-485)	Power Supply for Gas Module
Liquid Inlet tubing (B-486)	Tubing from liquid pump to inlet of Gas Module

2.2 Step 2: Fluidic Connection

Once the Gas Module has been placed on a stable surface, the fluidic and gas connections can be established. The Gas Module is supplied with one gas inlet module and a set of gas/liquid outlet modules.

1. **Visual check**
   Check if the compression ring and ferrule are still in good condition as shown in Figure 2.1. Over time, the ferrule may wear out due to mechanic stress and heat.

Figure 2.1: Fitting in good condition

2. Installation of Inlet Modules and Outlet Module

A schematic overview of the connections is depicted in the scheme below.

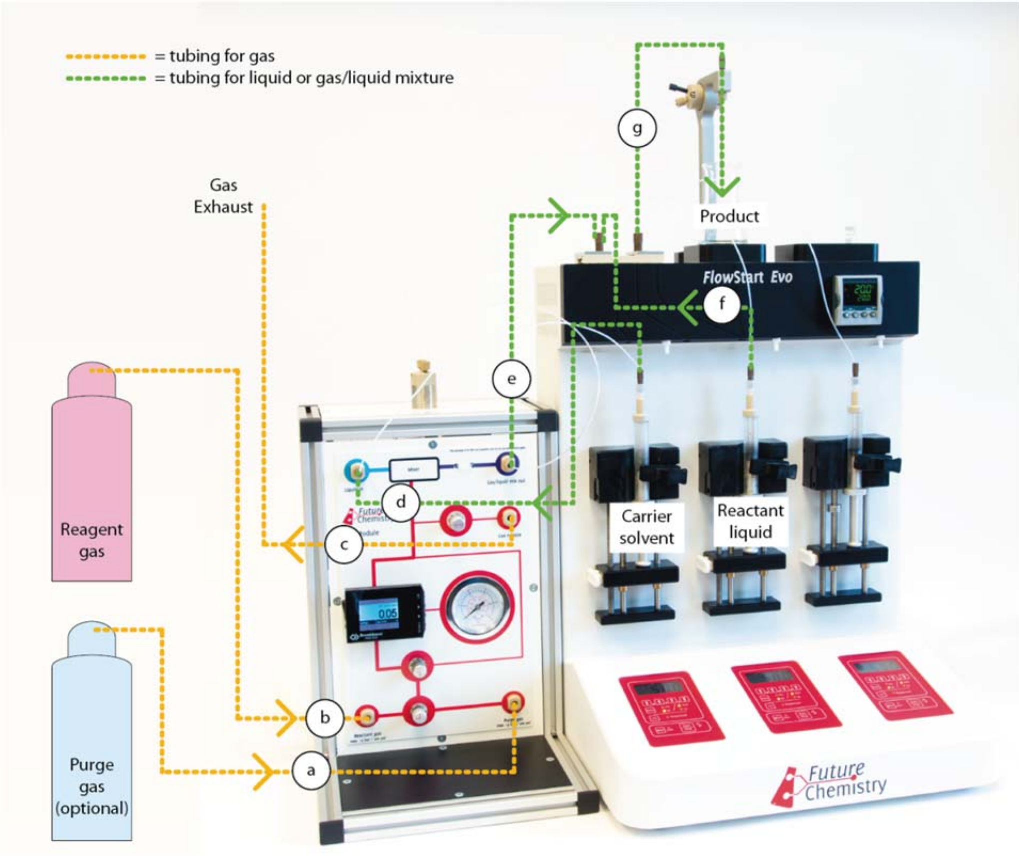

Figure 2.2: a schematic overview of the tubing connections of the Gas Module.

a. **Gas tubing from purge gas to purge inlet (optional, only when system needs to be purged, e.g. after using a reactive or corrosive gas, e.g. HCl) (not included).**
b. **Gas tubing from reagent gas to reagent gas inlet (B-482).**
c. **Gas tubing from gas release outlet to exhaust (mount tubing on high position in fume hood or use scrubber for reactive or toxic gases) (B-484).**
d. **Liquid tubing from <u>first</u> FlowStart Evo pump to liquid inlet of gas module (B-486).**
e. **Liquid tubing from gas module outlet to microreactor of FlowStart Evo, carrying liquid/gas mixture (Regulated Outlet B-483).**
f. **Liquid tubing from <u>second</u> FlowStart Evo pump to microreactor, carrying liquid with reactant, to be reacted with gas reagent.**
g. **Liquid tubing from the FlowStart Evo microreactor to collection flask, carrying liquid with product.**

2.3 Step 3: Power Connection

The FlowStart Gas Module can be connected to AC power outlet 110-120 V or 220-240 V, 50-60 Hz with the supplied power cable.

1. Connecting the power cable to the instrument and to a wall socket

Figure 2.3: Side view of the instrument with power connector

3 Gas Flow controller (GFC) Settings

3.1 Step 1: Gas flow rate

Switch on the power of the LCD of the gas flow controller by inserting the power connection. To set the gas flow, this takes the following step:

Figure 3.1: Gas flow controller (GFC).

1. **Set point input**
   Values displayed on the LCD of the gas flow controller can be activated by using the set key (1). To adjust the flow rate, press the arrow key (2) until a red line appears above the square and then press set. Only then, the flow rate of the red marked number can be changed by using the arrow keys (2 and 3). After pressing the set key, the next number is marked red and can be changed. When all numbers are changed correctly, finally push set once more and the gas flow controller will adjust to the set point.

3.2 Step 2: Gas flow Stabilisation

To connect a consistent gas supply in the Gas Module it is important to establish a pressurized gas flow, this takes the following steps:

- WARNING –

CAUTION: To connect the Gas Module with gas cylinder via pressure regulator, the pressure regulator should have a Tube OD x 1/8 inch connection. FutureChemistry could help find the right configuration.

1. **Set point gas flow controller**
   Set the gas flow controller to 0.0 mL/min.

2. **Gas Connection**
   Connect the gas cylinder with pressure regulator to the reagent gas inlet with the Gas Inlet Tubing (B-482). Switch all three valves to position 1 (see Figure 3.2). Open the main valve onto the gas cylinder and raise the pressure gently to 80 PSI (5.5 bar) using the pressure regulator (See figure 3.3). Check the internal pressure onto the manometer of both the gas cylinder and the Gas Module (these should both assign 80 PSI). Switch valve B to position 2 (see Figure 3.4). Raise the pressure from the gas supply gently to 200 PSI (13.8 bar) with the pressure regulator. Set the gas flow to 1.0 mL/min.

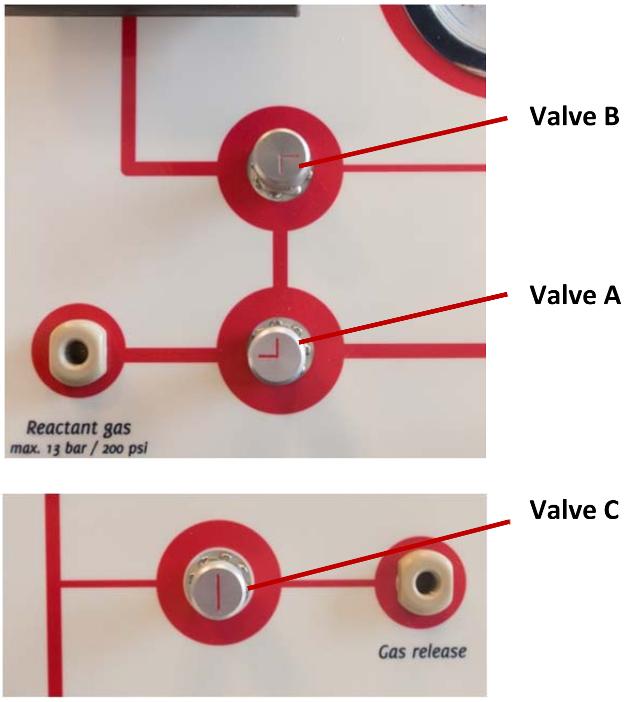

Figure 3.2: Valve positioning 1.

Figure 3.3: Pressure regulation from the gas cylinder.

- WARNING –

CAUTION: The gas cylinder and pressure regulator is not part of the Gas Module. For clarity, the part is included in this manual.

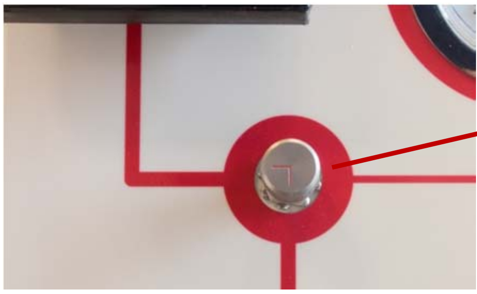

Figure 3.4: Valve positioning 2.

- WARNING –

CAUTION: A maximum pressure of 200 PSI (13.8 bar) can be supplied to the system!

3. **Set point gas flow controller**
   Set the gas flow controller to the flow rate desired. The gas flow rate ranges from 0.15 to 20 milliliters per minute. Depending on the choice of gas and liquid, there is maximum gas solubility. In addition, an increase of the gas flow rate causes a pressure raise within the system. The maximum gas flow rate therefore corresponds to the maximum pressure within the system, which can be read from the manometer. Make sure that the system pressure is always lower than the inlet pressure of 200 PSI (13.8 bar).

- WARNING –

CAUTION: With increased gas flow rate, the pressure increases into the Mixer. Stabilization of that pressure takes several minutes.

- WARNING –

CAUTION: For an accurate use of the gas flow controller, the inlet pressure from the gas supply always should exceed the pressure in the Mixer.

3.3 Step 3: Gas/Liquid flow Stabilisation

With the gas flow stabilized, it is important to avoid air (bubbles) accumulating in the system. To create a consistent gas/liquid flow of the Gas Module, it takes the following steps:

1. **Liquid flow stabilization**
   Set the flow rate of the pump to 350 µL/min and start pumping. Check if the system is free of gas (bubbles) using “the eye” (see Figure 3.5). When the system is gas (bubble) free, set the flow rate to the flow rate desired. The minimum liquid flow rate is 10 µL/min.

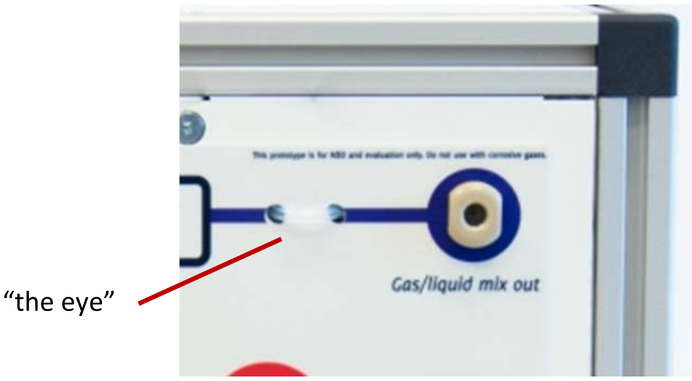

Figure 3.5: the "eye".

3.4 Refill the syringe

To refill the syringe of the syringe pump, stop the liquid flow and refill the syringe (see manual FlowStart Evo). Then start pumping and stabilize the gas/liquid flow again as described in paragraph 3.3.

3.5 Shut down

To shut down the Gas Module, it takes the following steps:

1. **Stop all pumps of FlowStart Evo.**
2. **Close the gas supply onto the gas cylinder.**
3. **Set the gas flow of the gas flow controller to 0.00 mL/min.**
4. **Switch valve B to position 1 (Figure 3.2).**
5. **Open the gas release switching valve C to position 2 (see figure 3.6). Wait 1 minute to release the gas pressure from the Gas Module.**
6. **The FlowStart Evo system is still under pressure because it contains gas saturated liquid. First, without removing the solvent syringe from pump 1, flush the microreactor at a flow rate of 350 µL/min with the solvent from pump 1, for 1 minute. Fill the syringe of pump 1 with isopropanol and repeat flushing for several minutes with a flow rate of 350 µL/min.**
7. **Close the gas release switching valve C to position 1 (see figure 3.2).**
8. **Turn off the power by plugging off the power connector.**

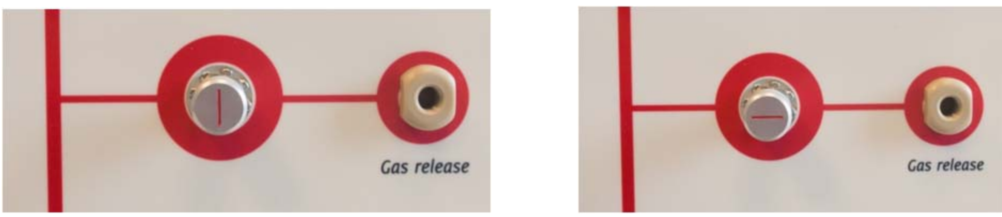

Figure 3.6: Valve C positioning 1 (L) and 2 (R).

4 Short handling overview for synthesis

In this chapter, the flow chart describes the protocol for synthesis based on a setup where the Gas Module is connected to the FlowStart Evo system.

4.1 Stabilization of the gas flow/liquid system

The protocol for producing a stable gas/liquid flow is summarized in this paragraph, it takes the following steps:

- Fill the three syringes with the solvent liquid (see manual FlowStart Evo).
- Connect the syringes and place them into the pump holder (see manual FlowStart Evo).
- Apply a controlled gas flow to the Mixer (see chapter 3.2, Gas Module).
- Pump the liquid through the mixer until it is gas (bubble) free as observed by the “eye” (see chapter 3.3, Gas Module).

4.2 Stabilization of the reaction

The protocol for synthesis is summarized in this paragraph, it takes the following steps:

- Stop the pumps (see manual FlowStart Evo).
- Exchange the solvents for the reagent solution (syringe 2) and if required, the quenching agent (syringe 3) (see manual FlowStart Evo).
- Start the pumps (see manual FlowStart Evo).
- Stabilize the system with a total liquid volume (pump 1) for 2 times the Mixer volume of the Gas Module (Liquid volume of the Mixer is 150 µL).

- WARNING –

CAUTION: Always apply the same order of events for a stable gas/liquid flow.

4.3 Flow chart

This paragraph contains a flow chart for a systematic stabilization of the gas/liquid flows for accurate results. Always apply the stabilization in the same order of events as depicted on the next page.

Gas supply stabilization

1. System setup

- Valve A directed to react gas
- Valve B directed to manometer
- Valve C closed
- Set flow rate GFC = 0.0 mL/min

⇩

2. System pressure

- Open gas reduction valve regulator to apply 80 PSI to the system

⇩

3. Controlled gas flow

- Switch valve B to the GFC
- Raise pressure to 200 PSI
- Set flow rate GFC = 1.0 mL/min

Liquid supply stabilization

4. System setup

- Set flow rate pump 1 = 350 µL/min
- Set flow rate pump 2 = 50 µL/min
- Set flow rate pump 3 = 50 µL/min

⇩

5. System stabilization

- Wait until the system is gas (bubble) free (typically 2 min)
- Set flow rate of pump 1 to 50 µL/min

⇩

6. Reagent

- Stop all pumps
- Exchange the organic solvent in syringe 2 for reagent solution
- Exchange the organic solvent in syringe 3 for the quench solution

⇩

7. Reaction stabilization

- Start all pumps
- Stabilize the system for 2 times the Mixer volume (2x150 µL)

⇩

8. Collection

- Start collecting the reaction solution

5 Calibration factors

The Gas Module's Gas Flow Controller is calibrated for use with hydrogen. For use with other gases, please use the following conversion factors:

Gas	Conversion factor
Hydrogen (H_2)	1.000
Ammonia (NH_3)	0.958
Hydrogen chloride (HCl)	1.271
Chlorine (Cl_2)	1.042
Isobutene (C_4H_8)	0.375
Fluorine (F_2)	1.250
Phosgene ($COCl_2$)	0.646
Propene (C_3H_6)	0.542

For example, when the meter indicates a value of 4.75 and you are using isobutene as a gas, the real flow value will be 4.75 * 0.375 = 1.78 mL/min.

If you would like to have the GFC recalibrated, please contact FutureChemistry. The Gas Module will need to be returned to FutureChemistry for recalibration.

6 Chemical resistance

When using the Gas Module for chemical reactions, chemical compounds come into contact with several wetted parts, both gases and liquids. Even though FutureChemistry has selected the best high-performance materials for these parts, some chemicals may deteriorate the integrity over time. Even though the actual performance of materials highly depends on concentration, temperature and exposure time, we have built up a chart of chemical resistance to give an indication of how the wetted parts will perform in combination with several example chemicals. First, for each wetted part a list of materials used is given. Next, several classes of chemical compounds are listed with performance. The performance is segmented in the following way:

Good performance, can be used without problems are expected	+
Material could deteriorate over time. Please clean as soon as possible after use, and expect to replace parts after extended use	0
Not recommended: use for own risk	-
No data known	N/A

Overview of materials per part

		PEEK	ETFE	FEP, PFA or PTFE	Kalrez	Stainless steel 316
Gas module: gas contact	**Tubing**		■	■		
	Connector ports	■				
	Valves			■		
	Gas flow controller					■
	Manometer					■
Gas module: gas contact	**Tubing**		■	■		
	Connector ports	■				
Gas and liquid contact	**Mixer (replaceable)**		■	■		
	Back pressure regulator	■			■	

Overview of Chemical resistance per material

		PEEK	ETFE	FEP, PFA or PTFE	Kalrez	Strainless steel 316
Aliphatic compounds	**Heptane**	+	+	+	+	+
	Paraffin oil	+	N/A	+	+	+
Aromatic compounds	**Benzene**	+	+	+	+	+
	Nitrobenzenes	0	+	+	+	+
	Phenol (aq)	0	+	+	+	+
	Styrene	+	+	N/A	N/A	+
Ketones, alcohols etc.	**Acetone**	+	+	+	+	+
	Isopropanol	+	N/A	N/A	+	+
	Benzaldehyde	+	+	+	+	+
	DMF	+	0	+	+	+
	DMSO	0	0	+	+	+
	Formaldehyde (aq)	+	+	+	+	+
	Methylethylketone	0	N/A	N/A	+	+

		PEEK	ETFE	FEP, PFA or PTFE	Kalrez	Stainless steel
Amines	Aniline	+	+	+	+	+
	Ammonia (anhydrous liquid)	+	+	+	+	+
	Hydrazine hydrate (aq)	+	+	+	+	+
	Pyridine	+	0	+	N/A	+
Halogenated compounds	Chloroform	+	0	+	+	+
	Dichloromethane	+	+	+	+	+
	Iodoform	N/A	N/A	N/A	0	+
Acids	Aqua regia (3:1 HCl HNO3)	-	+	+	+	-
	Acetic acid (aq)	+	+	+	+	+
	Boric acid (aq)	+	+	+	+	+
	Chromic acid (aq)	+	+	+	+	+
	Hydrochloric acid (aq)	+	+	+	+	-
	Hydrofluoric acid (aq)	-	+	+	+	-
	Nitric acid (40% aq)	-	0	0	+	+
	Sulphuric acid (conc)	-	+	0	+	-

		PEEK	ETFE	FEP, PFA or PTFE	Kalrez	Stainless steel
Bases	**Sodium hydroxide (aq)**	+	0	+	+	+
Halogens	**Bromine (pure)**	-	0	+	+	-
	Fluorine gas	-	0	0	0	+
Oxidants	**Ozone**	+	+	+	+	+
	Hydrogen peroxide	N/A	+	+	+	+

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