ProGasMix FC

User Manual

NorECs AS

List of contents

1 Introduction

Welcome to the ProGasMix FC (mixer, instrument, PGM or ProGasMix from here on) manual.

The manual provides you first with all essential information for

- Safety
- Installation

These parts are to be studied and used in the sequence they are provided here. We consider it essential that no parts are omitted. These two parts form fundamentals of the training for the user responsible of the mixer.

The section 'safety' includes risk assessments and recommended safety procedures. The section is also provided as two separate copies: to be read and to be signed during the PGM installation, one to remain always at the mixer and one to be taken back for the manufacturer. Further definitions relating to instrument safety can be found in chapter 7.

Thereafter we provide instructions for

- Different modes of operation
- Maintenance

which show how the PGM is used in normal use after installation. It thus forms the second part of the training, intended for all users, after they have been trained in safety and fundamental aspects by the user responsible.

Finally, we provide sections of more specialized operations and maintenance. We also provide a more detailed reference for safety and conformity.

- Special procedures and advanced information
- Technical specifications

NorECs AS

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2 Safety

In this section we list important aspects of safety of using the ProGasMix. Included are brief descriptions of normal operation, assessments of the possible risks and finally recommended safety procedures.

2.1 Normal use according to specifications

The ProGasMix can connect to three gases and mix them in wide ranges of ratios into 2 mixtures and control the water vapor levels for both mixtures individually.

Input gas A and input gas I forms the output mixture 1.

Input gas B and input gas I forms the output mixture 2.

2.2 Risk assessment

The mixer itself is passive and poses no threat of toxic, electrical or mechanical kind.

The gases connected to the mixer are selected by the user, and this forms the **main risk: misuse and human error.**

This manual defines the safe use, but the mixer itself is 'stupid', it offers no identification of or regulation according to gas species: the mixer physically treats any gas exactly the same way, even if dangerous or non-permitted gases were connected to it.

The gases used, the mixing action and the ventilation of excess gases are all decided, selected and operated purely by the user, so the possibility of a misuse is the main risk to prepare for. The mixer is designed to minimize these risks and possible consequences.

Secondary risks consist of possible malfunction situations.

2.2.1 Misuse hazards

1. Mixing flammable gas and oxidant as result of connecting such gases at wrong inputs
2. Mixing flammable gas and oxidant as result of not flushing the mixer
3. a) Ventilation outlets not connected while using toxic gases
   b) Humidifier out of water
4. Leakage

2.2.2 Safety regulations

1. Input Gas I (GI) must always be inert gas that is safe to be mixed with the other gases (GA and GB) connected to the mixer. All ingoing gases should be marked at all times.

2. a) When changing input Gas A or input Gas B one must consider that there is still gas of the previously used type inside the mixer, that may react with the new gas connected to the mixer. Such switching is to be avoided. If switching is required, the user must flush the target gas input with inert gas before connecting the new gas; this is described in detail later in the manual.

   b) The Mix4S selector can only be turned one step at a time. The flowmeter F7 and F8 both must have at least 30mm float travel, and the user must wait one minute before turning the Mix4S another step.

3. a) The mixer uses bubblers to keep constant pressure conditions. The bubbled gas is led to ventilation outlets. If these outlets are not connected to proper ventilation, they will let the gas into the room; a problem if toxic gases are used.

   b) If all the water in the humidifier is spent, the gas in the mixer has 'easier' way out through the refill line than through the rest of the mixer. To prevent this, the refill lines are connected to the ventilation lines.

   In case of using the mixer with toxic gas, the ventilation outlets must be connected to a working ventilation system, each ventilation outlet as its own separate line.

4. In case of a leakage, the main risk is posed by odorless toxic gases such as CO. If such gases are used, extra warning labels should be placed on the mixer. A corresponding gas detector with sound alarm must be used to guard against the possibility of a leakage or user error.

2.2.2.1 Additional information on safety regulation 2b

The valve Mix4S allows selecting the source gas for mixture 4. One may select either the mixture 2 or input Gas B. Now, it is possible that the mixture 2 might be undiluted input Gas A, possibly a fuel type gas. The input Gas B might be an oxidant. White the Mix4S valve effectively cuts off one feeds the other one to flowmeter F7 and prevents continuous mixing of Gas A and Gas B, there will be gas of the previous selection inside the system that will get into contact with the new

selection. For such reason on the four step selector the two positions between Mix2 and GB are both connected to GI, the inert gas. The user has to go over the GI selection, wait with it, and thus flush with inert gas, when operating according the safety regulation 2b.

2.3 Safety recommendations

The main risk is misuse; therefore the access to the mixer should be restricted to qualified persons. Each person with access to the mixer should understand the properties of all gases they are going to use. The users should be obliged to read the manual, especially this safety sheet and all accompanied documents, and to sign that they have done so, using the table at the end of this document.

2.4 Non-hazardous aspects and built-in safety features

There are no residual hazards with ProGasMix. It is built from metal, glass, silicon, O-rings and so on. The most exotic component ProGasMix contains is Halocarbon 6.3 oil, which is completely inert and non-toxic. Material safety sheet for the oil is included with the ProGasMix shipment. Otherwise ProGasMix contains only what the user feeds into the mixer such as distilled or de-ionized water, and the gases of user's choice.

The gas flow is controlled with manual flowmeters and manual valves and the mixer does nothing else than allows different gas species to mix; thus, the mixer does nothing else than what naturally occurs when mixing gases in general and therefore there are no other hazards that what the mixing itself poses; a responsibility of the user.

In case of system damage and a leakage the mixer is built in a well-ventilated construct. Volumes and pressures are small and the majority of the construct is non-flammable. The mixer is contained in a non-gastight metal frame with a polycarbonate front panel; in the unlikely case of explosion inside, the casing acts as muffler for pressure and possible glass shards. The polycarbonate front panel does not fracture into dangerous shards.

2.5 Additional safety considerations

Application of input pressures higher than 15 bars may lead to damage and to leakage in the tubing and pressure control parts. Apply input gases at pressure above 2 bar a and not exceeding 15 bar a. The mixer has built-in pressure reducers set to reduce input pressures to approximately 1.2 bars a, which is sufficient pressure to operate the mixer.

Corrosive gases: Cl_2, NH_3, SO_2 are examples of gases that will corrode and destroy the ProGasMix. Refer to PGM specifications sheet for material info.

When operated according to the rules, it is not possible to continuously mix gases that from flammable or explosive mixtures. However, should inflammation or explosion take place, the ProGasMix has been designed with a minimum of volume and of glass parts so that the size and consequence of such inflammation or explosion will be small, and contained in the casing.

Not dangerous but inconvenient event called 'back suction risk' is explained later in the manual.

2.6 Equipment classification

Pressures in the mixer are near atmospheric, except at the inputs before the pressure reduction valves. 'Regulations for pressure equipment' does not apply to ProGasMix as the volume between the inputs and the pressure regulator are extremely small, few cubic centimeters, well below the lower limit of the regulations.

The mixer uses an external adaptor to lower the voltages used to 24V DC. The documentation for the adapter is shipped with the mixer. On the mixer itself, the voltage used is 24V DC, which does not pose any electric hazard or require any certifications.

Further information about the mixer can be found in chapter Material and Safety reference

2.7 Statement of responsibility

The persons signed below agree and understand that

- The manufacturer NorECs Norwegian Electro Ceramics AS has provided sufficient information for safe and proper use of the mixer.
- The manufacturer NorECs has provided sufficient information on the possible risks and how to avoid them and prepare for them in case of misuse.
- The safety of operation of the mixer depends solely of the user's choices and actions.
- That NorECs cannot be held responsible for any consequences of any use of the mixer.

Name, date and organization	Name, date and organization
Signature	Signature
Name, date and organization	Name, date and organization
Signature	Signature
Name, date and organization	Name, date and organization
Signature	Signature

3 Installation

Installation means sequentially doing ***all*** steps of this section.

3.1 Before unpacking: Read this first!

Before you unpack the ProGasMix, there are a few instructions we want you to have read in beforehand:

3.1.1 Initial valve positions

During unpacking and initial installation of ProGasMix, do NOT turn any of its valves. This ensures that preinstalled drying stages remain closed and are not unnecessarily exposed to ambient humidity.

3.1.2 Initial pressure regulator positions

During unpacking and initial installation of ProGasMix, do NOT alter the valve positions of the pressure regulators. They are set to suitable pressure levels during manufacturing of the mixer.

3.1.3 Transport indicator

The ProGasMix package may be equipped with an external indicator that is ruptured if the package has been handled too roughly during transport. Please check for it and notify NorECs and the transporter without delay if there is such an indicator and it is broken.

3.2 Unpacking and inspection

3.2.1 Unpack

Remove any outer packaging until the ProGasMix stands free on its four legs. Please note that the center of mass is relatively high so use extra care removing the mixer from the pallet.

3.2.2 Documents

An envelope is located at the roof of ProGasMix, containing set of documents for the mixer.

3.2.3 Cabinet and front plate

Inspect the cabinet for signs of damage from transport. In particular, inspect the front polycarbonate plate for cracks.

3.2.4 Flowmeters

Likewise identify the flowmeters on the front plate and check that the glass tube inside each flowmeter is standing upright and looks OK.

3.2.5 Glass tubes

Your ProGasMix is delivered with a number of glass tubes 3 cm in diameter and about 50 cm long. They are mounted upright in the ProGasMix or packed separately. Check that they look undamaged from transport.

3.2.6 Remove the side and rear panels

Use appropriate screwdriver(s) to loosen necessary screws and remove the two side panels and the rear panel. Do not attempt to loosen the front plate.

3.2.7 Wetting stage box

An insulating box containing the wetting stage(s) is located at the bottom back part of the mixer. Remove the back side of the foam box, and retrieve the items stored there. Inspect any wetting stage construction for damage.

3.2.8 Bubbler liquid

ProGasMix is shipped with Halocarbon 6.3 which is inert and harmless. It will be used for filling the bubblers to pre-set levels, described later in the manual. Container of a liquid is packed inside ProGasMix separately and filled in the appropriate places in the ProGasMix during installation. Find out where the container is, extract it and set it aside.

3.2.9 In case of visible damage

In case of visible damage of any of the items listed above, or any other item, it is likely that this has resulted from rough handling during transport. Please notify NorECs and if possible and without delay the transporter.

3.3 Learn how to operate the valves before you begin

At this stage – before we have connected anything or filled any liquids, and as you go through the points below - feel free to try the action of the different types of valves, so that you are familiar with them and how they work. ***However, be sure to set each back to the original position***.

- The ProGasMix has **three types of valves**: The needle valves on all flowmeters, the 2-way selection valves and the 4-way selection valve.
- **Do not use excessive force on any valve**
- A **needle valve** on each flowmeter is a fine metering valve. It opens when turned counter clockwise. It is not a stop valve and must not be forced hard into full stop. Use a gentle force with two fingers – this will sufficiently stop the gas flow for our purpose. The valve knob turns fifteen full rotations.
- A 2-way valve can select between two open positions to connect to a third ‘common’ connect. It is closed in one middle position and can well be left in that position. It cannot be moved past the other two positions (sometimes called 3-way valve).
- A 4-way valve can select between four open positions to connect to a fifth ‘common’ connect. It ‘snaps’ to each four open positions (sometimes called 5-way valve).
- In addition the mixer hosts quick connects. Bulkhead part of the quick connect is mounted on the front panel and the stem part is, or will be, mounted on a 1/8 inch copper tube gas line to supply or to lead away the gas to or from the mixer. Both the bulkhead and the stem part are equipped with a valve that is open when the two parts are connected and closes when the two parts are separated from each other. To connect them just push the stem into the bulkhead. To disconnect push the rough ferrule on the outside of the bulkhead forward and pull the stem part away.

3.4 Assembly

The following procedures of assembly are done with the side and rear plates still removed. Assembly is normally performed by NorECs personnel on site. The acronyms for valves and other parts are all marked on the mixer, but it is good idea to have the flowchart of the mixer also in hand while doing these steps, to better understand the inner workings of the mixer. Copies of the flowchart can be found in the envelope on top of the mixer and also later in this manual.

3.4.1 Bubbler liquid filling

In case of an on-site installation of PGM, NorECs person will do the filling of the bubblers.

Each glass tube should be marked with bubbler ID and filling height mark. These should correspond approximately to heights of B1 = 45 cm, B2 = 35 cm, B3 = 10 cm, B4 = 25 cm, B5 = 10 cm.

Amongst the tools packed find a funnel and a piece of tubing and connect them to each other to make a convenient tool for bubbler filling. Pull the bubbler caps gently up, and slide the hose of the funnel to the tube. Use a small intermediate container to easily dose small amounts of liquid to the bubblers.

After filling, check visually that all levels are on or within few centimeters or so from the mark and then re plug the caps.

3.4.2 Bubbler maintenance

The bubbler liquid needs no maintenance. By normal use it lasts infinitely. A slight discoloration may occur (especially getting yellowish to light brown if you are using carbon monoxide, CO) but that is normal and has no consequence, and should not lead to replacement.

3.5 Wetting stages

Wetting stages bubble the used gas through temperature controlled water, setting a known water vapor pressure. W1 and W3 are working wetting stages, W2 and W4 are bypasses when wetting is not required. W2 and W4 also act as placeholders for custom wetting stages added by advanced users.

The transparent refill hose makes a downward loop from the wetting stage, acting as water lock, water level indicator and refill hose. The refill hose is also connected to the ventilation outlet (W1Re to VentA and W3Re to VentB) to improve the mixer safety in case of complete wetting stage dry-off and forgotten cap on wetting stage refill.

3.5.1 Water

For the wetting stages, use distilled or ion exchanged water. The water filled should not be above room temperature – rather below – when you fill it. Refilling will alter the temperature of the wetting stage and affect the partial pressure of water; a thing to keep in mind if refilling during operation.

3.5.2 Temperature

The temperature control unit (TC) for wetting stage cooling is preprogrammed to reach and hold a target temperature of 18°C.

This is a safe value definitely to be below any possible ambient laboratory temperature, eliminating risk of condensation of the wetted gas in the gas lines.

3.5.3 Fill the wetting stages

The mixer may be shipped with distilled water in the wetting stages, but it is good idea to familiarize with this process anyway. Before refilling make sure the wetting stage W1 has an open outlet; put Mix3Wout to W1, open F11 and F12 by turning it gently counter clockwise at least ten full turns or until you feel a resistance. Make sure the outputs have quick connect stem on them to open the valve on the quick connect bulkhead.

Remove the cap on wetting stage refill W1re and add distilled or de ionized water until the level indicator on W1 reaches mark Max1. Place back the cap on W1re.

Make sure the wetting stage W3 has an open outlet; put Mix5Wout to W3. Remove the cap on wetting stage refill W3Re, and add distilled or de ionized water until the level indicator reaches mark Max1. Place back the cap on W1Re. Select original positions for Mix3Wout and Mix5Wout and close the F11 and F12.

3.5.3.1 Maintenance of the wetting stages

Fill water as required, even during the operation but in case using toxic gases involved, be aware that the refill lines are in direct contact with the vent lines, and if the vent lines are not connected to actively working ventilation, the vent gases will come out of the mixer where the resistance is the least; the refill outlets without their caps on. This is the case when ventilation is arranged by a hose leading out, but with no ventilation machinery to actively move the gas out.

Each wetting stage has 4 markings on the refill tube, for example for WI the refilling tube has markings Min1, Min2, Max1 Max2. 1 is for refilling when the system has open outlet like in the previous chapter. Marks 2 are for refilling when the system is pressurized and in operation.

3.5.4 Extra and custom wetting stages

By default, W2 and W4 are bypasses at delivery of the ProGasMix.

Wetting stages W2 and W4 may however be equipped with e.g. D_2O-containing stages, or other custom stages.

One may also consider to use these for wetting stages that give much less H_2O contents than pure water, so that a larger range of mixing wet and dry can be reached by combining the two wetting stages. For instance, a solid-state mixture of a hydrate and an anhydrate may be considered.

One may also simply use W2 and W4 as extra wetting stages for H_2O just to increase the capacity.

Such installations at the user's site are the user/installer's responsibility. NorECs may provide assistance and advice.

One may note that the installation does not have to be safe against high pressures – the wetting stages are not exposed to high pressures.

3.5.5 Drying stages

By default, the ProGasMix is not equipped with drying stages and the input gases are assumed dry enough. The construction however allows for drying stages to be installed. Useful location for drying stages would be between Mix3DS and F6 and between Mix5DS and F10 for mixtures 3 and 5 respectively.

A suitable model will not cause pressure drop of more than few millibars. Be aware that the molecular sieves absorb CO_2 in addition to water, so that gases containing CO_2 as a component should have a way to bypass the installed drying stages. This can be achieved with similar selection mechanism as with the wetting stages. One recommended model is microporous absorbent, namely SGE analytical moisture trap model 103487. Under normal temperatures and the flows ProGasMix has, it removes H2O down to 10ppb and is expected to absorb about 12g of H2O.

The drying stages have a certain capacity to absorb various gases. That means that a flow of O2+Ar will initially get some O2 absorbed until the surfaces are saturated. After some flow it should stabilise at the correct ratio. As with water absorption will continue until the capacity of the stage is reached.

(The same effect also applies to any other desiccant, and also to the wetting stage: The liquid water has a capacity to dissolve gases which will make any change in the gas composition take a little time to stabilise.)

We recommend using the drying stage only when very dry gas is critical.

3.5.6 Connection of ventilation and overpressure outlets

The mixer uses bubblers to keep constant pressure levels and the gas through the bubbler is vented out. This section describes how and where to connect the outlets that dispose of surplus gas. VentA is connected to the bubblers and wetting stages using input gases GA and GI, and the VentB is connected to bubblers and wetting stages using gases GI and GB. Both the VentA and VentB lines must be led out to ventilation as separate lines.

Identify the outlets VentA and VentB.

It is in principle not necessary to connect these outlets anywhere if the mixer is not used for gases that pose any risk or smell, but if there is even a slight possibility that such gases may be used in the future or by accident, the ventilations must be connected as instructed.

If still used to lead the gases to a hood or ventilation system, lead both outlets as separate lines – do not merge them at any point. The ventilation must be an active one, a fan or a pump; it is not enough just to lead the vent lines out of the room. The materials and connections used after the bubblers are not considered high grade gas tight; it is the slight under pressure created by the ventilation machinery that makes sure the gases stay inside the ventilation lines and are led out of the system.

Important: Do not use quick-connects with built-in valves and do not use any other valves that may be deliberately or accidentally closed – these outlets must be kept open at all times. Also, do not use too narrow hose or tubing due to the pressure drop narrowness causes.

3.6 Connection of input gases

Connect input gases to the inlets at the front bottom of the mixer. Use only quick-connects *with* valves, so that they close upon disconnection and open when connected. Some quick connect counter pieces are supplied with the mixer, and the part number is included in the specifications of the mixer if more are needed.

Mark the input gases GA, GI and GB appropriately and clearly. GI stands for input Gas Inert, so never connect oxidant or fuel type gases on input GI. This is clearly marked on the mixer.

Input GA has capacity for much larger dilution than GB. In case of fuel cell type mixing, typically connect the fuel to GA and oxidant to GB. Never connect a new type of gas to GA or GB without first connecting an inert gas to the input first, and running the mixer for few minutes with significant gas flow to flush out the old gas.

3.7 Connection of outlets to measurement cells

Connect any mixer outlet O1 and/or O2 to any measurement cell chamber. The connection should use same type of quick-connect than the gas inputs. Use valve OS to select which way the Mix3 and Mix5 is connected to O1 or O2. This allows user for quick inversing the gases without stopping the gas flow.

3.8 Learn the principles and terms of gases and mixtures

In order to operate onwards you need to know and understand some simple principles of what the mixer does and how we denote the gases and mixtures involved.

The ProGasMix starts with 3 input gases: GA, GI and GB. These are supplied at a pressure of normally a few bars (atm), but the mixer has internal pre-set pressure regulator reducing the pressures even further.

The gas GA is mixed with gas GI in several steps using pairs of flowmeters. F1 with gas GA and F2 with gas GI form a mixture 1 (Mix1).

The Mix1 is mixed with more gas GI with flowmeters F3 and F4 to form Mix2.

The Mix4 is made same way, but using gas GB or Mix2 for F7 and gas GI for F8.

Mix2 and Mix4 form the bases of the two final mixtures (Mix3 and Mix5), but first they will go through individual stages to control their water vapor levels: Mix2 is split in two portions, one to be wetted and one to remain dry, and these two portions are mixed by flowmeter pair F5+F6 into Mix3. Similarly, Mix4 is split in two portions, wetted or not, and these two portions are mixed by flowmeter pair F9+F10 into Mix5.

Each mixture Mix3 or Mix5 is a controlled composition including water vapor level. It is typically used for varying oxygen activity over a large range while keeping the water vapor level constant, or *vice versa*.

Each wetting stage has two pathways, W1 and W2 for Mix3 and W3 and W4 for Mix5. Normally, one is used for the wetting (W1 and W3) while one is used as a bypass (W2 and W4). However, other uses such as D_2O-wetting etc. are possible.

For each flowmeter pair and mixture Mix1...Mix5 a pressure column bubbler B1…B5 ensure that excess gas is let out, and in this way maintains constant pressure conditions throughout the mixer, an important feature when calculating the flow of gases through flowmeters. The bubbled gas goes to ventilation.

There are two outlets O1 and O2 from the mixer that are meant for connection to the user cell, e.g. to the two chambers of a ProboStat measurement cell. Each outlet has a flowmeter (F11 and F12) and a selection valve for inverting the output gases.

We repeat here the important list of gases and mixtures passing in the mixer, and ask that you check that you understand the difference between them:

- 3 Input gases GA, GI, GB G stands for Gas, I for Inert
- 5 Mixtures Mix1…Mix5 Various mixtures from the 3 input gases.
- 2 Outlets O1, O2 Selected from Mix3 and Mix5

3.9 Startup and checks during initial operation; first user operation training session

If this is the first time you are using the ProGasMix and you have had no other training, this will also function as the first step of your training, and we will thus explain things as we move on.

3.9.1 Mains power

Connect the mixer to mains power, using its external adaptor. By this, lights illuminate inside. Moreover, the Peltier element of the wetting stage box starts cooling, as indicated on the temperature display.

3.9.2 Initial settings of valves

Check that all (12) flowmeter needle valves are closed (gently turned fully clockwise).

3.9.3 Input gas

A gas should now be connected to at least two of the three input gas lines.

Check that there is no gas going to any of the glass tube columns in the mixer. If the columns are changing or eventually bubbling, check again to close all needle valves on the flowmeters (by turning clockwise to a gentle stop).

3.9.4 Flowmeters and overpressure bubblers

Flowmeter has fine valve and a flow indicator, a small ball in a hollow glass tube. The higher the ball, the more gas is flowing. The height of the ball is called float travel, and it is announced in millimeters and read from the scale printed on the tube.

Now turn flowmeter F1 up to a flow corresponding to, say 50 mm float-travel. This is fed from input gas GA.

By this the column on overpressure controller B1 starts to slowly move downwards. The gas goes down in a hollow glass tube against the pressure of the liquid in the external glass tube pushing the liquid away, making a visible indication of the flow.

Turn also flowmeter F2 up to a flow corresponding to, say, 50 mm float-travel. This is fed from Input gas GI.

F1 and F2 mix gases GA and GI into what becomes Mix1. F1 has a light glass float and supplies relatively small flows. F2 has a heavy steel float and contributes ca. 10 times as much gas as F1 at the same float travel height.

Soon, B1 will have reached the end of the column, and starts to bubble. This is the normal operational mode; it keeps the pressure of Mix1 constant, regardless of flows.

Test this; change one of the flows F1 or F2 and see how it does mainly *not* affect the other flowmeter (as long as B1 bubbles).

When B1 bubbles, you can use Mix1 to feed flowmeter F3. Turn it up to, say, 30 mm. You can use as much flow as you want, but not more than F1 and F2 supply, that is, only as much as B1 keeps bubbling. If B1 stops bubbling, you are losing the constant pressures situation, and the mixtures made cannot be precisely calculated.

NOTE: This is a good time to make sure you understand that the gas that bubbles in the overpressure bubblers – like now B1 – is not used for making the final mixtures: What bubbles is the excess gas that we don't need. While bubbling is essential for maintaining constant pressure conditions and for getting rid of

excess gas, it is of course also important to reduce unnecessary bubbling to a minimum to reduce gas consumption.

Now, you can mix in more gas into Mix2 by turning up flowmeter F4, to, say, 30 mm. This is fed from gas GI, similarly as flowmeter F2.

Now, we will run Mix2 through wetting stage and dry line, and mix the portions into Mix3 which is a final output mixture and which bubbles in B3.

Open the inlet (Mix3Win) and outlet (Mix3Wout) for wetting stage WI for gas coming from Mix2 and going into Mix3, by pointing both valves to WI. Turn up flowmeter F5 till a small flow (e.g. 30 mm). This flow is now being wetted in the wetting stage.

F6 will select the amount and type of gas that forms the dry part of Mix3, elected with Mix3DS between Mix2 and GI.

To make Mix 5, select a source for F7 using Mix4S and select GB. Turn up flowmeter F7 and F8 to a small flow (e.g. 30 mm).

Repeat the Mix3 steps but for F9, F10, Mix5Win, Mix5Wout to form Mix5.

There should by now run some gas in all of the 10 first flowmeters, and all pressure control columns should be bubbling.

3.9.5 Leakage test

When all pressure control columns have been bubbling for a few minutes, it is time to do the overall leakage test:

Starting from the last flowmeters (with biggest number) close all flowmeters. For each pair, the supply of gas stops, and no gas is consumed. Thus, the pressure control column should remain at the bottom of the bubbling tube. A gas-tight system keeps the columns at the bottom position for at least 10 minutes. Any tendency that the columns raise indicates leakage. Identify the leakage (e.g. by using Snoop), repair if possible, and try the test again.

Note: This test of the gas-tightness of the ProGasMix is sufficient for its normal and safe operation. It can be classified as low-vacuum gas-tight. It is not and is not intended to be high-vacuum gas-tight.

Note: Do *not* at this stage attempt to test gas-tightness using a vacuum pump – the vacuum will give violent back-suction in the bubbling stages with danger of contaminating tubing and flowmeters with bubbler liquid.

3.9.6 Leakage identification and elimination

Most changes in bubbler columns are due to temperature changes, this is best seen when all columns change same or similar amount or in clear proportions to their overall heights.

If the changes are not uniform, an overpressure column does not stay constant with others, there may be a leakage in the region connected to it.

Inspect all Swagelok nuts to visually confirm that they seem to be tightened to the same level. If one appears more distant from the bulk of the union or component, first check with your fingers whether it is still only finger-tight. If the nut can be loosened, check that it is otherwise OK, finger-tighten it, and then turn ¾ of a turn by wrenches.

If no obvious loose nut or other mistake is spotted, the leaky region may be tested by applying dilute soap-water (preferably Swagelok Snoop) to suspected joints and seals and looking for leak foam forming. (In this process, keep the overpressure up by letting the flowmeters flow in some gas.) Moreover, wait sufficient time to let the foam form; since the overpressures are small, the foam may form slowly and be difficult to spot. If a leakage is spotted, consider to remedy it:

If a nut looks slightly less than normally tightened, try tightening it gently.

If a nut leaks but looks normally or overly tightened, then open it, retract the nut, and check that ferrules are correctly in place and intact and that the tube and bulk part look intact. If OK, reassemble and tighten lightly using proper re-tightening procedure (not the one for new ferrules).

If no leak is spotted, contact NorECs for further advice.

3.9.7 Leave the mixer with overpressure

When not in use, you may well leave the whole or parts of the mixer with overpressure, especially if filled with an inert gas. You may even want to leave a tiny flow of inert gas going to be sure to keep it filled and ready. If there is no flow, be sure to close both ends of the wetting stages to prevent water vapour to back-diffuse into the system.

4 Normal operation

4.1 Use of software to calculate mixing result

In the following we will go through the use of the mixer. As we move on, the reader may feel overwhelmed by all the factors that affect the mixing and the complexity of obtaining the final composition of the output gas mixtures. Let us therefore rush in and say that these calculations are done by a computer program. Since the mixer is manually operated and with manually set flows, it is important to understand it, but the computer and software will do all the hard work after the flowmeter readings are entered. The software we provide with ProGasMix is described in a later section.

4.2 Output gas

4.2.1 Routing and use

Once you have established Mix3 and/or Mix5, either of the output lines O1, O2 may be directed to be fed from Mix3 or Mix5 via the output selection valve OS. Typically, a measurement cell like a NorECs ProboStat is connected with its two chambers being fed from O1 or O2 with the gas stream split after the mixer.

Transients or reversing of gas composition gradients can be accomplished by simply switching the position of OS.

4.2.2 Pressure, tubing, and flow

The final stages Mix3 and Mix5 of the ProGasMix have around 0.019 bar overpressure (corresponding to the 10 cm liquid overpressure control columns B3 and B5 and density of 1,9 of Halocarbon oil 6.3). This may limit the flow that can be supplied to the outputs if the tubing out of the ProGasMix is too thin and long. Consider adjusting bubbler liquid heights, or using more than 1/8'' tubing in this case, e.g. 3/16'' or 1/4'', via converting unions.

Do not use plastic or rubber hose tubing if inertness or dryness of the gas is essential; O_2 and especially H_2O in-diffuse quickly through most hose materials.

Normally, some tubing after the cell or furnace chamber is used to vent the off-gases. This also serves the purpose of preventing back-diffusion of unwanted components from air if inertness and/or dryness is important, but may again limit flow.

Bubbling the outlets of the cell or furnace chamber through a cm or two of liquid such as water gives a nice visual confirmation of flows, and a small overpressure that further prevents back-diffusion in the tubing and in-diffusion through a leaky cell. Use of oil prevents evaporation and back-diffusion of water vapor, but requires more care to avoid back-suction in case of e.g. rapid cooling of the furnace. In any case, these couple of cms of outlet overpressure again reduces the pressure gradient and flow out of the output stages of the ProGasMix.

4.3 Economic use of gases

4.3.1 Output flows

Consider how much gas flow you need: Equilibration in wet air or O_2 or when pH_2O does not matter for these gases, or in good buffer gases like $CO+CO_2$ mixtures or H_2+H_2O+Ar mixtures require little gas; Turn the output flow down and reduce the flow through the mixer correspondingly (see below).

If – on the other hand – the gas has the purpose to provide inert and/or dry conditions, a higher flow – sometimes even as high as possible - helps to minimize the effects of permeability, back-diffusion, and leakages. Remember, still, that flushing out a dead end of the mixer, tubing, or cell, may be equally effective and much more economic than a lengthy high flow.

Other cases where high flow is required are in supplying electrodes and surfaces in high-drain applications where constancy of gas composition is desired in certain types of fuel cell, reactor, and gas separation membrane tests are made. Also transients recorded after e.g. a change in gas composition may require flow as high as possible.

4.3.2 Backwards reduction of flows for mixtures

When the output flows have been set, it is good practice to regulate - in backwards order of the mixtures - the flows of gases to the minimum while still satisfying

- the mixing ratio,
- the use of acceptable ranges of the flowmeters,
- bubbling in all stages to ensure stable pressures and flows.

Thus, the experienced ProGasMix user often starts from Mix1 supplying ample amounts of gas so as to quickly and stably obtain bubbling and constant pressures and flows, and then, when the output flows are set, backwards down-regulating for minimum flows.

In this way, pure unmixed gases or mixtures not involving very large ratios can be made very economically. For somewhat higher ratios, using two or three stages (Mix1, Mix2 and/or Mix3) with small flows can in fact be more economic than one stage with one high flow. Nevertheless, one cannot escape from a large consumption of gas when very large ratios are attained; then necessarily a lot of the main (diluting) gas is running in two or three or four stages.

4.3.3 Quality of input gases

Here just a word on the need for purity of input gases. High temperature cells usually cannot maintain oxygen levels much below 10 ppm and not water levels below 30 ppm. Thus, the purity of the gases – especially the inert ones that are often used for dilution and in large quantities – need probably not be better than 99.999 %. In many cases, the main impurities are in any case another inert gas, with no or minimal consequences so that even lower purities may be considered.

4.4 Wetting stage control

The temperature of the wetting stage defines the vapor pressure H_2O and thus the maximum possible pH_2O of your mixtures. The higher temperature used here the higher pH_2O possible. If however the ambient temperature goes close or below the temperature inside the wetting stage the moisture begins to condense in the gas lines; this interferes with gas flow, renders the calculations wrong and causes other kind of trouble.

We recommend selecting a temperature of two degrees below the lowest foreseeable laboratory temperature and setting the wetting stage for that with 1°C hysteresis. Small difference from ambient also prolongs the life of the wetting stage fans. The cooling power and the insulation are sufficient to cool the wetting stage roughly 10°C below the ambient temperature at 25°C. At higher ambient temperatures the gain might be less.

In case no fluctuation in pH_2O is acceptable we recommend turning the cooling hysteresis to 0.5°C

When higher water vapor contents are required, external humidification stage can be used. NorECs builds such devices and also offer gas line heating solutions as well as ProboStat base unit heating systems.

4.4.1 Wetting stage temperature and vapor pressure of H_2O

Temp	8°C	9°C	10°C	11°C	12°C	13°C	14°C	15°C	16°C	17°C	18°C	19°C	20°C
kPa	1.073	1.148	1.228	1.313	1.403	1.498	1.599	1.706	1.819	1.938	2.064	2.198	2.339
Temp	21°C	22°C	23°C	24°C	25°C	26°C	27°C	28°C	29°C	30°C	31°C	32°C	33°C
kPa	2.487	2.645	2.810	2.985	3.169	3.363	3.567	3.782	4.008	4.245	4.495	4.758	5.034

4.5 Modes of operation

4.5.1 One gas (+ H_2O)

If you are simply going to supply one unmixed input gas as such – wet, dry or a mixture of wet and dry – you may select that gas as GB and supply it to Mix4 using F7 and having Mix4S selector on GB, setting pH_2O in Mix5 as needed (with Mix5DS selected as Mix4).

In this mode, the first parts of the mixer can be separately used to supply other gases GA, GI, pH_2O and their mixtures through Mix3 for other purposes.

4.5.2 Mixture of two gases (+ H_2O)

The most commonly applied mixes of two gases are O_2 in inert gas, air in inert gas, CO in CO_2, and H_2 in inert gas. All these typically have the purpose of controlling pO_2.

If you are mixing two gases, use GA as the one generally most dilute, and GI as the diluent, normally the inert gas or CO_2. GA can then be used undiluted or diluted roughly to 1:65 and to 1:4257 by the two first flowmeter pairs.

It is however more economical to use smaller mixing rations and more flowmeter pairs. To include one more flowmeter pair, route the Mix2 to F7 selecting Mix2 on Mix4S selector and dilute further (up to 1:132500) to make Mix4.

Even higher ratios can be obtained by using a pre-diluted input gas (e.g. air as diluted O_2 or 5 % H_2 in Ar) or by utilizing F9 and F10.

4.5.3 Mixture of three gases (+ H_2O)

This mode of operation is not possible with a standard ProGasMix FC. The FC in the name stands for Fuel Cell and for safety reasons it was made impossible to mix three gases in the mixer. This text was left here for the curiosity of the user. If such use is of interest, it is possible to use pre mixed gases or have the mixer modified.

> *Mixes of three different gases may comprise mixing CO and CO_2 and then adding Ar for control of the carbon activity, a_C. This may, in turn, be because you are interested in the effects of the activity of carbon (e.g. at constant pO_2) or because you want to reduce the degree of carbon precipitation via $2CO(g) = CO_2(g) + C(s)$ which is driven to the left by reduced total content of CO and CO_2.*
>
> *Another use of a third gas might be to mix e.g. N_2 and Ar and then mixing in O_2. Variations in the first stages may then change pN_2 to study effects of nitrogen activity at relatively constant pO_2. N_2 may be replaced by CO_2 to study effects of carbonatisation. N_2 may also be replaced by e.g. He; When He replaces Ar in a mixture, effects of gas phase diffusion on kinetics change and may be studied.*

4.5.4 Changing the water content (pH_2O) in a mixture

The water content of Mix3 can be varied down from wet (ca. 3 % H_2O at 25°C) by using F5, to dry by having Mix3DS at Mix2 and using F8 (similarly for Mix5).

In addition, the wet can be mixed with dry to cover three orders of magnitude of water content from 3 % down to ~0.05% (ca. 1.7 % H_2O at 15°C with maximum dilution)

This level of water does not influence much on the partial pressure of the other gases in the mixture; from dry to fully wet they decrease by 3 % relatively. For instance, pO_2 in dry pure O_2 is 1 atm, while pO_2 in wet pure O_2 is 0.97 atm. Such variations in pH_2O with nearly constant pO_2 is essential e.g. for studies of proton conductors under oxidizing conditions. Similarly, the pH_2O can be changed in $CO+CO_2$ buffer mixtures

without affecting pO_2 much. (But for H_2-based reducing atmospheres pH_2O directly affects pO_2 and another mode and approach is used, see next section.)

The maximum pH_2O depends of the wetting stage temperature.

4.5.5 Changing the water content (pH_2O) and that of another active component (e.g. pH_2) at the same ratio

Assume the mixture going through a wetting stage is H_2 in inert gas, e.g. Ar, from a premix and/or from mixing in the first two or three stages. Now, we switch Mix3DS to GI. Then the drying stage/bypass is not carrying the same mixture as the wetting stage, but instead dry GI = Ar. In the wetting stage we get wet H_2+Ar, i.e. H_2O+H_2+Ar. When this is mixed with the dry Ar, we are decreasing *both* the H_2O and the H_2 contents, but their ratio remains constant. At high temperature equilibrium this means that we are changing pH_2 and pH_2O, while pO_2 remains constant. This is essential for investigations of proton conductors under reducing conditions, in H_2+H_2O+inert gas atmospheres.

4.5.6 Large gradients; fuel cell and permeation tests

4.5.6.1 Fuel cells

One typical use of the ProGasMix is in supplying fuel and oxidant to the two sides of a fuel cell test. On the fuel side (Mix3) for example H_2 as GA, on oxidant side (Mix5) Air or O_2 as GB and the inert dilutant gas Ar or N_2 as GI.

While dry H_2-containing gases may seem the right for the fuel side, this is often too reducing and may embrittle platinum and reduce other components. Better and more stable operation is usually obtained by wetting the H_2 mixture fully. The ProGasMix can also wet the oxidant. This may be a good option for work with proton conducting fuel cells of various kinds.

Fuel cell can be started in soft manner with large dilution or pure inert gases.

It is good practice to flush all parts of the mixer – especially the wetting stage and bubblers – with inert gas before switching to and from gases that may form flammable or explosive mixtures.

Special care is to be taken not to alter the position of Mix4S when using the mixer in fuel cell mode. The valve selects between Mix2 (pure or diluted fuel) and GB (oxidant). While one is always cut off, the small volumes in the mixer are still filled with the previous type of selection. **When operating valve Mix4S, always comply with safety regulation 2b**.

4.5.6.2 Gas permeation

By supplying different gases over a membrane sample, analysis (e.g. with MS or GC) of the gas composition after the membrane passage on the feed or – in particular - permeate side may be used to obtain the permeation of species in the gases. For measurements of oxygen permeation one may for instance supply O_2+N_2 mixtures as feed gas on one side. On the permeate side, using Ar, one may analyse the content of N_2 and O_2. If the membrane is permeable only to oxygen (e.g. through ambipolar diffusion of oxide ions and electrons), the content of N_2 may be taken to reflect leakage through pores, cracks or poor seals, and after correcting for this using the fractions of N_2 and O_2 in the feed gas and assuming macroscopic flow in the leaks, one may obtain the real permeation of oxygen through the membrane material.

If the membrane is at high temperature, a dilute H_2+Ar mixture may be used instead, giving a much higher chemical driving force for oxygen, which flux is now measured via the H_2O content in the permeate gas.

In a similar manner, a mix of H_2 and an inert gas can be supplied as feed gas. The permeate is then typically another inert gas and the levels of H_2 and the feed inert gas are measured. Wetting the feed gas is probably a good choice. Wetting the permeate side may give the side effect that any transport of oxygen from the permeate side to the feed side may split water and leave hydrogen, which is then incorrectly assigned to permeation of hydrogen.

4.5.7 Small gradients for transport number measurements

4.5.7.1 General principles

By establishing a difference in chemical potential over a sample, a driving force for mobile species affected is set up. If the species *s* is charged a voltage – or electromotive force (EMF) - arises that is proportional to the transport number t_s of the species, defined as $t_s = \sigma_s/\sigma_{total}$.

This emf is measured at open circuit by using high impedance voltmeters, multimeters or electrometers, and is thus often also referred to as an open circuit voltage (OCV). The method we are to describe therefore are called measurements of transport numbers by the OCV of concentration cells, or simply the EMF method.

In its simplest form, the voltage over the cell is derived from so-called Wagner transport theory: The partial current of each charge carrier involved is expressed in terms of the driving force (gradient in electrochemical potential) it experiences, its conductivity, and charge. Then all partial currents are summed to a total current, the expression equaled to zero in the OCV case, and solved with respect to the electrical potential gradient. This is integrated from side I to side II to obtain the voltage over the sample:

$$U_{II-I} = -\sum_n \int_I^{II} \frac{t_n}{z_n e} d\mu_n$$

where U_{II-I} is the voltage between electrodes II and I, t_n is the transport number of ion n with charge z_n and where μ_n is the chemical potential of the corresponding neutral atomic species. The expression thus sums up over all ionic species n for which there is a significant gradient and transport number.

In the following, the equation is solved under the assumption that there is a gradient in only one species, and that the gradient is small, so that the transport number can be considered constant, whereafter the integration leads to

$$U_{II-I} = -\bar{t}_n \frac{kT}{ze} \int_I^{II} d\ln a_n$$

where E_N is the Nernst voltage. Finally,

$$\bar{t}_n = \frac{U_{II-I}}{E_N}$$

The software that comes with the ProGasMix will calculate the Nernst voltages involved after you have entered the readings of the flowmeters, and will then give transport numbers based on the voltages measured.

With the ProGasMix one may measure the transport number as a function of the gradient, i.e., the Nernst voltage, and extrapolate back to zero gradient to obtain the transport number closest to the assumption of a small gradient. However, making too small gradients will inevitably lead to immeasurably small voltages and consequently scatter in the transport number curve.

Below we give some examples of types of gradients and how they are obtained in the ProGasMix. However, first we discuss how you can measure small voltages by minimising or eliminating background offset voltages.

4.5.7.2 Background (offset) voltage minimization and elimination

Use identical materials for electrodes and make sure there are no thermal gradient over the sample. All junctions and transitions from one type of lead to another type must be identical in material and of temperature, as well as all cables must be identical to each other. Use shielded cables and keep away from power carrying leads.

Use identical atmosphere on both sides to find out presence of an offset. Alternatively with true atmospheres measure sample voltage, reverse the gases, measure the sample voltage and average voltages to find out the offset.

4.5.7.3 Oxygen activity gradients

Enter here.

4.5.7.4 Hydrogen activity gradients

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4.5.7.5 Other activity gradients

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4.5.8 Transients

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4.5.9 H/D isotope effects

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4.6 Characteristics of the flowmeters

The flowmeters used in the ProGasMix are so-called variable area flowmeters. This reflects the variable cross-section of the flowmeter tube; the float (in our case a small ball) floats higher up (larger area for passage of the gas) the higher the flow. The floats usually spin or rotate when they operate normally – hence the flowmeters are often also referred to as rotameters.

The flowmeters used in the ProGasMix are of type Vögtlin V-100. The tubes are borosilicate tubes with hexagonal inner to prevent the ball, called float, from getting stuck easily.

In general, the tube and the float both can be of different sizes. The ProGasMix utilizes three combinations, or three different types of flowmeters described in table below.

Symbol	Float looks	Flowmeters	Role	Flow range, air 30°C	Type
⬡	Black	F1, F3, F5, F9	S)mall flows	0.16 – 1.6 ln/h	M080E01G
✸	Metallic	F2, F4, F6, F10	L)arge flows	0.69 – 10.7 ln/h	K080H03G
●	Metallic	F7, F8, F11, F12	M)edium flows	0.16 – 5.0 ln/h	M080H01G

The tubes are equipped with a scale. The reading on the scale is in mm and is called float travel (FT). The scale is 0-65 mm. Flowmeters cannot detect very small flows, so there is a region of unknown flow between the 0 mm mark and the lowest possible location for the float. When the float is at the bottom of the tube and the needle valve is fully closed, the flow is considered to be zero, and in the program such condition is designated with FT=0. On the flowmeter scale 0 mm is however already a significant flow. Due this contradiction the lowest usable flow is 1 mm FT.

On the high end, the ball can pass beyond the top reading of FT = 65 mm, but is eventually stopped at a metallic stopper. The flow can thus be much higher than the maximum reading, but it won't be readable. Such high flows are usable for example for flushing the mixer.

The flow required to keep a ball floating at a certain height is dependent of the viscosity and density of the gas or gas mixture. Thus, as extremes, a certain FT with H_2 or He as the gas represents a much higher flow than of CO_2 or Ar. The flow ranges in specifications and general calculations are given in air, and these ranges and ratios are naturally depending on what gases are being used.

Flowmeter readings in FT (mm) must be converted into flows and the conversion is done with the help of calibration curves. The relationship between flow and float travel is a non-linear complex function that includes tube size, float weight, gas density and gas viscosity. It is additionally (greatly) affected by temperature of the input gas and pressure in the tube. The tube- and float types are known, the pressures inside the mixture are known, all the user needs to do is to feed the float travels and the type of gas used to the software and it will calculate the actual flows of gases and mixtures using the calibration curves obtained with range of typical input gases. The GasMix program will correct for the temperature difference, the gas species and so forth based on the provided information.

4.6.1 Additional details on absolute flows

The calibration curve flow is given in units of mln/min or ln/h. The ml (milliliters) or l (liters) is the volume of the gas in n (normalized) conditions; a volume that the specific gas occupies at certain temperature and pressure. These normalized conditions with ProGasMix are 0°C and 1013.25 mbars atmospheric pressure.

For example, the ingoing gas may be 27°C and the pressure in the flowmeter is 1.03 bars a, but the actual flow is reported in normalized conditions. Let us say the float travel and calibration curve would indicate 5 ln/h. If the measured flow of gas was to be captured for a specified time, say one hour. Only if the captured gas was cooled to 0°C and given a pressure of 1013.25 bars a, it would occupy the calibration-curve-reported volume, 5 liters.

Now, none of this is of much consequence to typical user, as he or she is not interested in the total flows, but in the ratios between the gases. While interference such as changes in the laboratory temperature

affects the accuracy of the total flow measurement, it affects all the flowmeters roughly the same, keeping the ratios between flows much less prone to error.

> If, however the total flow volume is of interest, it is not enough to know if the flow is denoted in 'standard' or 'normal' liters, but actually to know which temperature and pressure these 'standard' or 'normal' conditions refer to.
>
> Worldwide, the 'standard' or 'normalized' condition for pressure is variously defined as an absolute pressure of 101,325 Pascal, 1.0 bar, 14.73 psia, or 14.696 psia and the 'standard' or 'normalized' temperature is variously defined as 68°F, 60°F, 0°C, 15°C, 20°C, or 25°C.
>
> There is, in fact, no universally accepted set of standard conditions and these differences can cause tens of percents of errors.

But, in short, the flows and calibration curves handled by the software and these issues are of no concern to normal user.

4.6.2 Flowmeter mixing ratios general overview

It is suggested that the reading of the floating ball is taken at the middle of the ball. The accuracy of the flowmeters is not such that this actually matters, but it is wise to stay with one way of doing it for the best possible reproducibility and accuracy of relative changes.

However, there are considerable deviations from tube to tube and float to float. There are also deviations from the curve shape within each tube. Thus, in principle, each reading should use its own calibration curve for that tube and that float and that gas composition and those conditions (T, P). This is hardly possible – especially for the versatile ProGasMix – and one must instead find and use a practical compromise. This includes in the simplest case the use of one standard master curve for each float type, and just applying different sizing factors for the various gas species. The next level includes the use of several master curves for different types of gas. Alternatively or additionally, one may calibrate individual flowmeter-ball-pairs. Experience has shown that the latter give hardly any improvement in investigations of properties over many orders of magnitude in partial pressure of a gas, e.g. in the slope of log electrical conductivity vs log oxygen partial pressure.

We denote our tubes with small flow, S, and large flow, L and tube with medium flow, M. Flowmeter pairs F1&F2, F3&F4, F5&F6 and F9&F10 are combinations of S and L and allow ratios from 1:1 to 1:65 for dilution. Flowmeter pairs F7&F8 and F11&F12 are pairs of M, giving accurate medium flows and ratios from 1:30 to 30:1.

With following float travels one can get the general idea of the possible mixing ratios available.

S float travel mm	L float travel mm	L/S ratio
60	10	1
25	25	10
15	30	20
20	55	30
10	65	65

Also, for S, the ratio between flows at 10 mm and at 65 mm is always 1:10 and between those flows not too far off from linear dependency between those two points.

With same float travels for S and L, the ratio of flows is 10 at 25mm and goes down both on higher and smaller flows.

S float travel mm	L float travel mm	L/S ratio
15	15	8.74
25	25	9.83
35	35	8.99
45	45	7.88
55	55	7.13
65	65	6.56

The remaining float meter pairs F7&F8 and F11&F12 are both M type, and have the following characteristics.

M float travel mm	M float travel mm	L/S ratio
1	65	27.7
35	35	1
65	1	0.036

4.6.3 Possible flowmeter issues

Unstable float travel has normally two reasons:

Small fluctuations at a regular rate are normal and are due to the tiny variations in pressure arising from the forming and then released bubbles. If the float is fluctuating, the float travel reading is then taken at an average of the high and low positions. If these fluctuations get unusually large, it indicates that the available pressure difference working as the driving force is getting small and thus more subject to the variations from bubbles. This may indicate that bubbler liquid columns are out of normal state, or that there is too much water in a wetting stage.

Slow, sliding changes in the float travel are normally due to the valve of the flowmeter relaxing a little in its rubber O-ring seals and against its needle orifice. This is normal. It is for this reason not always easy to set the flowmeters to certain flows in one try. Instead, one typically set them to flows approximately at the desired flow, and then leaves the valve to relax for some minutes and fine tunes the flows.

Other behaviors of unstable float travel may have other, less normal flow. Irregular jumps indicate that there is liquid somewhere in the gas line (hopefully just condensed water). Check also that the wetting stage is not empty and thus leaking out.

If you discover that a float is stuck, and then check whether you may have opened the flow too much and that gas is flooding the bubblers – then turn it down before you proceed.

NOTE: It only takes a dust particle to get the ball stuck, especially near the bottom. It usually loosens by knocking gently at the front polycarbonate or the top of the flowmeter. Even if you *can* take the tube out for cleaning, it is actually difficult to do that procedure without entering more dust particles than you remove, so we recommend that you refrain from taking them out unless you have a remaining recurring problem. If there is a chance that it is stuck because of (condensed) water, it is best to just let dry gas run until the water is evaporated. See also special section/documentation on flowmeter maintenance and service.

The flowmeters are equipped with 15-turn needle valves. While these can close off a flow, they must never be forced and do not provide “vacuum tight” closure. If absolutely zero flow of something is needed, it must be achieved by other means.

4.7 Suggestions for flowmeter settings for various uses

4.7.1 Outputs

First of all, let us suggest that output flowmeters F11&F12 are set at FT = 15 mm as normal. This corresponds to flows of the order of 10 ml_n/min. Reduce flow throughout when possible to save gas, or increase if necessary for special needs.

4.7.2 Small variations in partial pressure

The flow is to a first approximation proportional to the float travel. The user will thus intuitively be able to set mixing with relatively linearly varying ratios. For instance, a pair of S and L tubes set with FTs = S10:L10 mm will give a 1:8.7 flow ratio. Changing to FTs = S35:L10 mm will give a 1:2.5 ratio and changing FTs = S65:L10 mm will give ratio of 1:0.87

4.7.3 Small differences between Mix3 and Mix5

Flowmeters F7 and F8 making Mix4 can be used to make small accurate differences between Mix3 and Mix5 for transport number studies (Mix4Se -> Mix2 in such case). Typically, F7 may be set to FT=35 mm and F8 varied through 1 mm to 65 mm to measure voltages over samples and obtain a series of transport numbers. One may for instance plot the transport number vs ratio or log ratio of partial pressure and extrapolate back to zero gradient (ratio = 1, log ratio = 0).

One may also make differences in pH_2O instead by setting the wet+dry mixing ratios different between Mix3 and Mix5 along similar principles.

Or – as explained above – set Mix3DS to GI and mix in dry GI in order to change for instance pH_2O and pH_2 without a difference in pO_2.

4.7.4 Logarithmic variations in partial pressure

The real advantage of the ProGasMix comes in the variations of e.g. pO_2 over large ranges. This can be done at fixed pH_2O, and we will show here some typical series.

Let us start at pure O_2 as Input gas A and mix it with Ar as Input gas I. After mixing in stages F1&2 and F3&4 it is then wetted by running at e.g. FT = 65 through F7 and W1. But what are the best settings for the flowmeters F1 to F4? Here is a suggestion, with rough ratio estimates:

F1	F2	F3	F4	Mix1	Mix2	Log Mix2
65	0	max	0	1:0	1:0	0
50	5	max	0	1:1	1:1	-0.3
20	10	max	0	1:5	1:5	-0.7
25	25	max	0	1:10	1:10	-1.0

We see here that the three latter series utilizes the rule of thumb “much + little, little + little and some + some” gives three points per order of magnitude, which may be suitable for many purposes.

Now instead of pressing more out of this stage and thereby wasting a lot of gas, it is more economic and accurate to leave it and do the same series on stage 2:

F1	F2	F3	F4	Mix1	Mix2	Log Mix2
25	25	50	5	1:10	1:20	-1.3
25	25	20	10	1:10	1:50	-1.7
25	25	25	25	1:10	1:100	-2.0

Same steps can be done for Mix4 with Mix4S as Mix2 (as in example below) or with the stages F5+F6 and F9+F10.

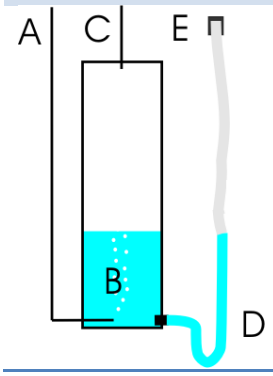
F1	F2	F3	F4	F7	F8	Mix1	Mix2	Mix4	Log Mix4
25	25	25	25	25	25	1:10	1:100	1:200	-2.3
25	25	25	25	20	55	1:10	1:100	1:500	-2.7
25	25	25	25	10	58	1:10	1:100	1:1000	-3.0

We have now covered three orders of magnitude with three stages, but each individual step can be pushed further for greater range, for example:

F1	F2	F3	F4	F7	F8	Mix1	Mix2	Mix4	Log Mix4
25	20	25	20	5	52	1:13	1:159	1:2000	-3.3
27	45	27	45	1	50	1:17	1:295	1:5072	-3.7
20	40	20	40	1	55	1:21	1:464	1:10000	-4
20	55	20	55	1	65	1:31	1:930	1:25730	-4.5
10	65	10	65	1	65	1:65	1:4225	1:116891	-5

4.8 Characteristics of wetting stages

Typically two identical wetting stages are located in the insulated and Peltier cooled container. One is W1 and the other is W3.



Label	Name	Notes
A	Incoming gas	From Mix3Win or Mix5Win
B	Distilled water	Gas bubbling through the water
C	Outgoing gas	To Mix3Wout or Mix5Wout
D	Transparent hose	Transparent hose acts as water level indicator. Loop downwards acts as water lock.
E	Refil & Cap	W1Re or W3Re. Cap for safety in case water lock dries out

4.9 Back suction risk

When a large volume such as sample holder (just 'volume' from now on) is connected to the mixer there is a risk of under pressure or 'back suction' in certain conditions.

If and when such volume is at high temperature and then cooled back to room temperature it will have low pressure inside, and gas will try to flow into it. Normally this is not a problem since the volume outlets are open, and will allow the pressure to equalize. However, if the volume outlets are closed (plugged, fitted with relief valve etc.) and the gas mixer is connected to the volume inlets and the gas supply is interrupted (the user has decided to save gas or gas ran out), and the mixer is not disconnected from the cell inlets, the under pressure developing inside the volume will 'suck' on the mixer itself. Depending on the valve positions this may or may not be a problem. If any of the wetting stages are connected to the outlet of the mixer, the replacement air will be sucked though the refill tube of the wetting stage to no consequence. If, however, none of the wetting stages are connected to the mixer outlet, the under pressure will try to suck up the bubbler liquid. The liquid is heavy and needs to be pulled up at least 20cm, but after that it will be in the gas tubes, and possibly enter into the flowmeters. This will render the mixer useless until a throughout cleaning of the system is performed. Such cleaning is a major undertaking that will take at least two days.

4.10 Calculational principles of mixtures

Enter here

4.11 High-temperature equilibria

Enter here

4.12 Normal operations maintenance

Enter here

4.13 Service procedures

Enter here

4.14 Typical extensions and modifications

Enter here

5 Special procedures

5.1 Flushing of gas lines to preserve purity of gases

5.1.1 Flushing of connections during gas bottle replacement

To be entered.

5.1.2 Flushing of input gas line for connection of a new gas to a line

When replacing an input gas with another gas, one must consider that there is leftover of the previous gas in the system. Some of the previous gas has higher pressure also, the short copper tube from the quick connect to the pressure regulator has the previous gas with the pressure that the feed line had. Past the pressure regulator the previously used gas has very low pressure, and it may exist in the mixer as diluted or pure. If the new gas is not safe to be used with the old gas, the mixer needs to be flushed with inert gas. This is done in following steps.

1. Stop all other use of the mixer.
2. Flush the volume between the quick connect and the pressure regulator by connecting an inert gas to the gas line to be flushed. This procedure will also flush bubblers B1 or B4.
    - If the gas line is input gas A, open F1 for a large flow for few minutes, close F1 and disconnect the flush gas.
    - If the gas line is input gas B, set Mix4S to input gas B, open F7 for a large flow for few minutes, close F7 and disconnect the flush gas.
3. To flush the rest of the mixer, have an inert gas connecter to input gas I. Open F4, F5, F8 and F9 to large flows. Set the Mix3Win and Mix3Wout to W1, and Mix5Win in and Mix5Wout to W3. Leave the mixer like this for few minutes and then close the flowmeters.

5.2 Procedures to optimize purity (e.g. inertness or dryness) of a gas in the mixer

Previously used gases remain in the mixer, specifically dissolved in the wetting stages for considerable amount of time. While flushing for some minutes is enough to prevent potential hazards, it does not purify the mixer completely of the previously used gases. The purity of made mixtures improves over time as and as function of flow.

5.3 Leak testing

To be entered.

5.4 Flowmeter calibration

In some cases, for example if flowmeter is replaced with another type or an existing calibration curves does not match the requirements (for example rare gas type to be used) the user may be interested to enter their own calibration curves. Exact details for such operation are beyond the scope of this manual, but the general idea is described below.

Flow table or calibration curve is a table of real flows measured at given float travels, typically ln/h per float travel. To create such table the user may acquire for example Vögtlin Smart series mass flow meter (mfm) calibrated with real gas(es) in question. The mfm is connected to a gas line before the flowmeter, and the measured flow is read from the mfms led display or from a computer connected to the mfm through USB cable. The flow is controlled with the needle valve of the flowmeter, and once the float has settled at a specific float travel, the actual gas flow is recorded to the calibration table. Once the table is ready with readings for each 5mm float travel, it is transferred to the GasMix software.

Calibration curves used in the software must share equal conditions of testing with other calibration data used, namely pressure in the flowmeter, gas temperature, and the unit of measured flow.

Once the data is obtained, adding the information to the GasMix software is straightforward, and is covered in the manual of the software.

5.5 Bubbler refill

In some cases a bubbler tube (B1…B5) may need refilling. Measure from the bottom of the tube and mark the height of your desired refill. The refilling is easy, but removing excess liquid is more complicated, so be careful with the amounts. Also be aware of what liquid your gas mixer has and use the same liquid. You need a long nosed funnel (max 8 mm nose outer diameter), and a light weight bottle or a jug. Do not try to pour from a heavy 2.5 L glass bottle or similar, instead have a small amount of liquid in a small jug or a plastic bottle so that it will be easy to pour small amounts to the funnel without spillage. Locate the tubes coming out of the metal cap on the chosen bubbler.

5.6 Temperature controller programming

The temperature control unit (TC) for wetting stage cooling operates with one of the two following modes: Target temperature mode (PR1 - Program 1). PR1 tries to cool down the interior of the wetting stage box to a fixed temperature set in the controller. We recommend using the Target temperature mode at 12°C, which is preprogrammed into the controller.

Preprogrammed mode definitions:

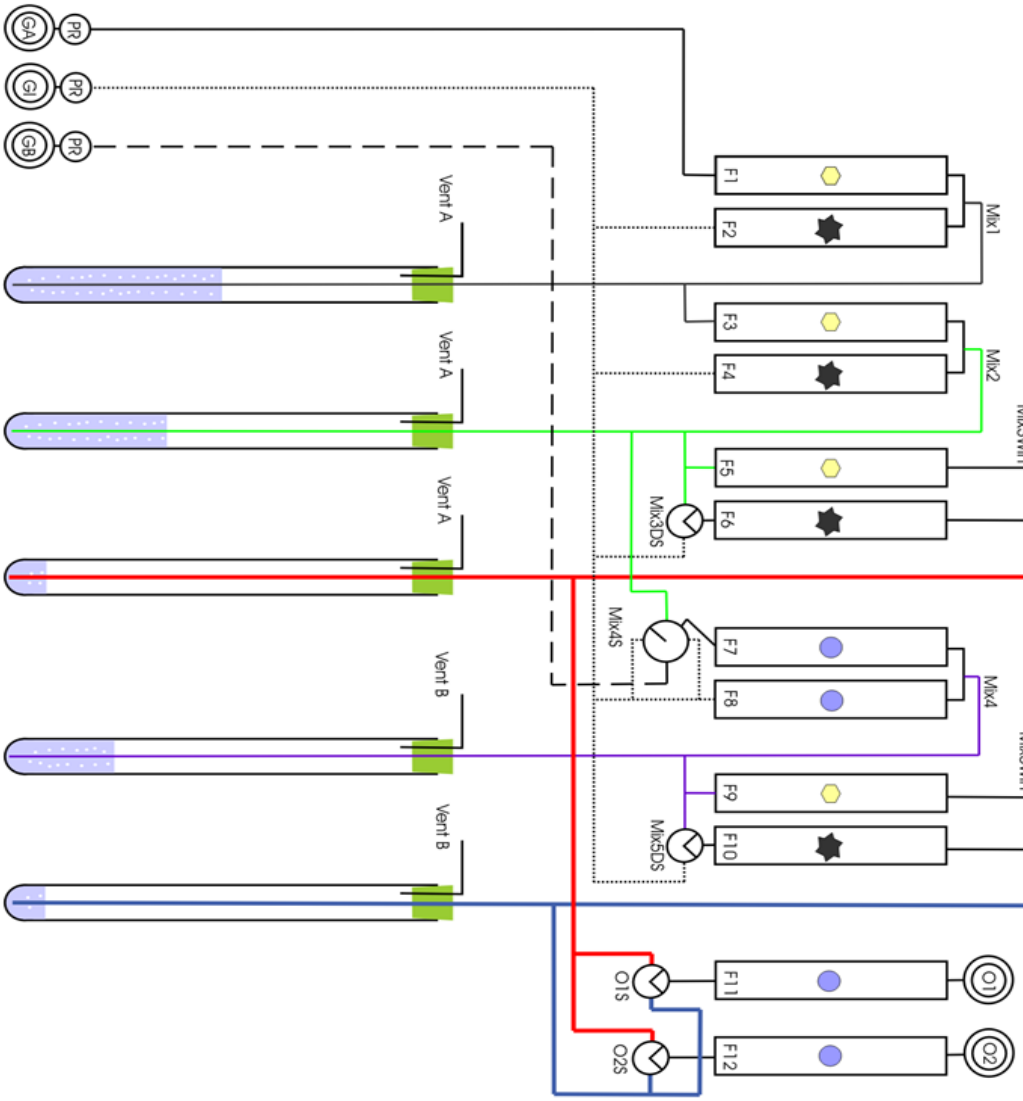
Mode: PR/1, Heating: H/0.0, Heating hysteresis: HY/0.5, Cooling: C/18.0, Cooling hysteresis: HY/0.5, Safety Shutdown: CH2/60, Fan follow-up time: LUFt/1.

This program tries to cool and maintain 18°C with 0.5°C hysteresis to save mechanical part lifetime. Power is cut off if Peltier heats above 60°C, which may be the case if the fans are damaged or the back door to the cooling box is open. Fans will run for 1 minute after the Peltier element power is cut off to dissipate the remaining heat on the hot side of the heat sink.

For more details about programming the temperature controller refer to ‘Temperature controller for Peltier module‘-document (by the manufacturer of the temperature controller unit) provided with ProGasMix.

6 Technical specifications and reference

6.1 ProGasMix flow sheet



Label	Name	Notes
GA	Input Gas A	Gas to be diluted. Quick connect with valve. **Safety regulation 2a**
GI	Input Gas I	Dilutant, inert gas. Quick connect with valve. **Safety regulation 1**
GB	Input Gas B	Gas to be diluted. Quick connect with valve. **Safety regulation 2a**
PR	Pressure regulator	Preset to roughly 1.2 bar a. Max input pressure 15 bar a
F1 ○	Small flow flowmeter	0.5 - 26 mln/min air
F2 ★	Large flow flowmeter	18 - 208 mln/min air
F7 ●	Medium flow flowmeter	3 - 83 mln/min air
Vent A	Ventilation A	A-vents are connected to each other. **Safety regulation 3**
Vent B	Ventilation B	B-vents are connected to each other. **Safety regulation 3**
B1...B5	Bubbler 1 to 5	Bubblers for constant pressure
Mix1...Mix5	Mixture 1 to 5	Gas mixtures
Mix3DS	Mixture 3 Dry Source	2-way selector between Mix2 and GI
Mix3Win	Mixture 3 Wet in	2-way selector between W1 and W2
Wix3Wout	Mixture 3 Wet out	2-way selector between W1 and W2
W1 and W3	Wetting stage 1 and 3	Peltier cooled wetting stages
W1re and W3re	Wetting stage 1 and 3 refill	Refill entry with cap
W2 and W4	Wetting stage 2 and 4	Bypass / placeholder for alternate wetting stage
Mix4S	Mixture 4 Source	4-way selector between Mix2 and GB. **Safety regulation 2b**
Mix5DS	Mixture 5 Dry Source	2-way selector between Mix4 and GI
Mix5Win	Mixture 5 Wet In	2-way selector between W3 and W4
Wix5Wout	Mixture 5 Wet out	2-way selector between W3 and W4
OS	Output selector	Connect Mix3 and Mix5 to F11 and F12 with possibility to inverse the order
O1 and O2	Output 1 and Output 2	Quick connect with valve

6.2 ProGasMix marking and definitions

6.2.1 The ProGasMix front panel markings and functions

Marking	Function	Notes
GA GI GB	Input Gas A Input Gas Inert Input Gas B	Swagelok 1/8 inch quick connects with shutoff valve Safety regulations 1 and 2a
F1…F12	Flowmeter 1…12	Flowmeters with needle valve F2, F4, F6, F8 and F10 have higher flow range than the rest.
Mix3DS	Mixture 3 Dry Source	Left: Mix2, Right: GI, Up: Closed
Mix5DS	Mixture 5 Dry Source	Left: Mix4, Right: GI, Up: Closed
Mix4S	Mixture 4 Source	Left: Mix2, Top: GI, Right: GB, Up: GI Safety regulation 2b
Mix3Win	Mixture 3 Wet in	Left: W1, Right: W2, Up: Closed
Mix3Wout	Mixture 3 Wet out	Left: W1, Right: W2, Up: Closed
Mix5Win	Mixture 5 Wet in	Left: W3, Right: W4, Up: Closed
Mix5Wout	Mixture 5 Wet out	Left: W3, Right: W4, Up: Closed

6.2.2 The ProGasMix exterior markings and functions

Marking	Function	Notes
Vent A	Ventilation A for GA and GI	**Safety regulation 3**
Vent B	Ventilation B for GB and GI	**Safety regulation 3**
W1Re and W3Re	Wetting stage 1 and 3 Refill access	Use distilled or de-ionized water only. Do not overfill.
24VDC	24VDC supply for Peltier element and lights	Round entry in the back panel.

6.2.3 The ProGasMix interior markings and functions

Marking	Function	Notes
PR	Pressure Regulator	Pre-set to suitable pressure ~1.2bar a
B1…B5	Bubblers 1 to 5	Glass tube bubbler system with bubbler liquid for respective mixes.
W1 and W3	Wetting stages 1 and 3	Temperature controlled steel bubblers
PELTIER	Peltier element	Peltier element for wetting stage cooling
TC	Temperature Control/indicator	Temperature controller and indicator

6.3 Other reference lists

6.3.1 Shipment contents

Item	Location	Function
ProGasMix	On a pallet	
8 x Quick connect stem Part number: B-QM2-D-200	Inside wetting stage	To connect the mixer inputs and outputs to gas 1/8 inch copper tube gas lines.

6.3.2 Included documents

Item	Description	Amount
Power supply CE declaration	Specifications and conformity for the external power supply used with ProGasMix.	1
Halocarbon 6.3 material safety sheet	Material safety data sheet for: Halocarbon 4.2, 6.3, 27, 56, 95, 200, 400, 700, 1000N oils	1
Halocarbon 6.3 properties page	Density and viscosity data	1
EC Declaration of Conformity	PGM conformity with applicable directives	1
Risk assessment	Extract from this manual printed separately	2
Flowchart	Flowchart of the mixer	4
Peltier manual	Peltier controller operation manual	1
GasMix manual	GasMix software manual	1

7 Material and safety reference

7.1 Reference to the EC Pressure Equipment Directory (PED)

7.1.1 Conformity, Compliance, and General issues

The ProGasMix may be used with fluids (gases) in Group 1 (Dangerous) and Group 2 (Other).

The ProGasMix may receive and handle gases at pressures up to 15 bar a.

The ProGasMix comprises pressurized accessories and tubing of a total volume less than 1 L.

The ProGasMix comprises pressurized tubing of DN = 6.

On this bases, referring to Annex II Tables 1-7, the ProGasMix falls below Category I of the PED. It is thus not CE-classified, but manufactured according to Sound Engineering Practices (SEP).

Nevertheless, it is instructive to treat the pressure-related safety aspects of the ProGasMix according to the PED, notably Annex I.

7.1.2 Design

7.1.2.1 General

The main design and component selection of the ProGasMix was made in the 1980s and is published in scientific literature[1]. It has since been built in a number of units, with minor changes and improvements. The main change is the replacement of wetting stages first using saturated solutions of $(NH_4)_2SO_4$ for dew point suppression into stages using saturated solutions of KBr (for lower vapor pressure of the salt), and finally into the present use of Peltier stage cooling instead of salt solutions. Furthermore, the drying stage has been changed from being P2O5-based to being based on a commercial mol-sieve stage and eventually skipped. Both the wetting and drying stages now are made of steel instead of glass, eliminating risk of breakage and damage from broken glass.

The design and the many units built and continuously used over more than 20 years makes the ProGasMix well-proven in terms of functionality, lifetime, and safety.

7.1.2.2 Design for adequate strength

The materials used for gas tubing and components are all qualified for the gases and pressures used, and this is well-proven over many years of use of preceding units over more than 20 years.

7.1.2.3 Provisions to ensure safe handling and operation

The ProGasMix has initial pressure reducers and continuous near-atmospheric pressure relief throughout. It relies on the user connecting safe gases at safe pressures. Should there still be occasions of overpressure supply, clogging of overpressure systems ventilation, or explosion, the volumes involved are small, the number of glass items kept to a minimum, and all such parts protected behind polycarbonate and metal casing. The ProGasMix involves no voltages above 24 V. It has no hot external surfaces.

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[1] T. Norby, "**EMF Method Determination of Conductivity Contributions from Protons and Other Foreign Ions in Oxides**", *Solid State Ionics,* **28-30** (1988) 1586-91.

7.1.2.4 Means of examination

Each ProGasMix is manually assembled. It is tested for leakages before shipment. Final function testing and renewed leakage testing is performed during installation, after filling of the overpressure columns with bubbler liquid on-site.

7.1.2.5 Means of draining and venting

Draining is normally not necessary for safety reasons, but may be done for improved purity and correctness of gas mixtures made, by flushing inert gas according to procedures in the manual.

Overflow gases (from the input vents or overpressure control columns) are led to vent outlets on the unit. These need normally not be led onwards to venting system, unless toxic or irritating gases are used, or venting is otherwise desirable.

7.1.2.6 Corrosion or other chemical attack

The continuous use of ProGasMix units for more than 20 years indicates that corrosion is not a problem. The most corrosive gases considered that it can tolerate used intermittently over many years is wet CO_2 and wet O_2; no problems have been observed with these gases.

7.1.2.7 Wear

The only wear expected is on flowmeter valves. While these can operate for many years without maintenance, they may also break down due to e.g. over tightening (user applying excessive force). Preventive maintenance and repair may be done according to standard procedures issued by the flowmeter manufacturer, supplied with the ProGasMix.

7.1.2.8 Assemblies

N.A.

7.1.2.9 Provisions for filling and discharge

Discharging the mixer is to be done according material information and local legislation.

7.1.2.10 Protection against exceeding the allowable limits of pressure equipment

To be entered.

7.1.2.11 Safety accessories

To be entered.

7.1.2.12 External fire

The ProGasMix is considered of too small pressure and volume of fluids (gases) of Group 1 (Dangerous) that any special precautions are necessary in case of fire.

7.1.3 Manufacturing

To be entered.

7.1.4 Materials

Material	Locations	Notes
Copper	Copper is used in gas lines	
Brass	All valves include brass. All copper tube joint adapters have brass ferrules.	
Stainless steel	Wetting stages	
Polycarbonate plate	Front panel	
Undefined painted metal	Casing and frame	
Glass	Bubblers	
Halocarbon 6.3 oil	Bubblers	Boiling Point: Decomposes > 200°C Vapor Pressure: <= 0.1 mm Hg (@21°C) Specific Gravity (H2O=1): ca 1.9 (@38°C) Solubility in Water: Negligible
Natural rubber	Bubbler caps	
Viton, Buna-N O-rings	Quick connects and needle valves	
Silicone hose	Bubblers, ventilation hoses	
Undefined plastic	Ventilation hose joints, ventilation hose caps	
Polyurethane tube	Wetting stage refill lines	Also silicon and PVC

7.1.5 Fired or otherwise heated pressure equipment

To be entered.

7.1.6 Piping

To be entered.

7.1.7 Specific quantitative requirements for certain pressure equipment

To be entered.

7.2 Reference to other directories

The ProGasMix carries no voltages higher than 24 V DC or AC, and thus has no hazards related to electricity.

The ProGasMix does not conform to the EC ATEX directory for equipment to be used in environments with flammable or explosive fluids.

8 GasMix software

This is a short introduction to GasMix program. Please see the full and individual GasMix manual.pdf for a more complete and updated version.

8.1 Introduction

8.1.1 What is GasMix?

The GasMix program is software for calculating gas mixing ratios and also equilibrium compositions resulting from gas mixers such as the ProGasMix. However, the program has flexibility of configuration and use that makes it useful also for other gas mixing applications.

8.1.2 History of GasMix

The GasMix for Windows is a development from the GasMix program written by Truls Norby for HPBASIC computers in the 1980s and used at University of Oslo till this date for calculating gas mixtures on gas mixers analogous to the ProGasMix.

8.1.3 Note: GasMix emulates but does not control your mixer

To avoid any misunderstanding: GasMix emulates the physical gas mixer and makes calculations based on the readings taken from the mixer. It is however not physically connected to the mixer in any way, it does not communicate with the mixer and it does not control the mixer in any way.

8.2 GasMix quick-start and tutorial

This section intends to give the user a fast introduction to the GasMix software and practice in using it.

8.2.1 Start and run the program: GasMix.exe

Start the program GasMix.exe from the shortcut icon or in any other way.

8.2.2 Fundamental elements of the gas mixer and GasMix display

At the bottom of the display are a row of small boxes, each representing an Input gas. Typical Input gases are the pure gases or mixtures that come from bottles or lines of gas in your laboratory, e.g. O_2, Air, N_2, Ar, 5% H_2 in Ar.

Next – at middle height of the display - comes a row of vertical rectangles, representing the Flowmeters. Some of the Flowmeters may be fed from the Input gases, and this is then illustrated by colored connecting lines – one color for each Input gas.

Above the flowmeters gas lines show how the flowmeters are connected to form Mixtures. Each Mixture has a color, and there is one small boxes attached to the Mixture line above the first flowmeter connected to that Mixture.

Additional small boxes directly below or above a flowmeter represent stages for fixing the partial pressure of some component, normally H_2O (wetting or drying stages).

On the top you find rectangles representing your measurement or reaction chambers, here called Output cells, for which temperature and total pressure can be set so as to allow calculation of equilibrium partial pressures.

The right hand side of the window is reserved for tables in use during editing and data entry. Moreover, results of mixing and equilibrium calculations will appear on the top of the window.

During editing of calibration curves also parts of the mixer schematic temporarily becomes covered with tables and graphs.