

**PROGASMIX Sensor Testing
Set up USER Manual
Electrical Characterization Lab**



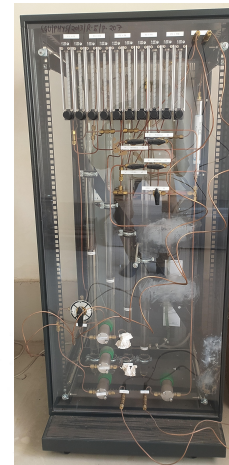
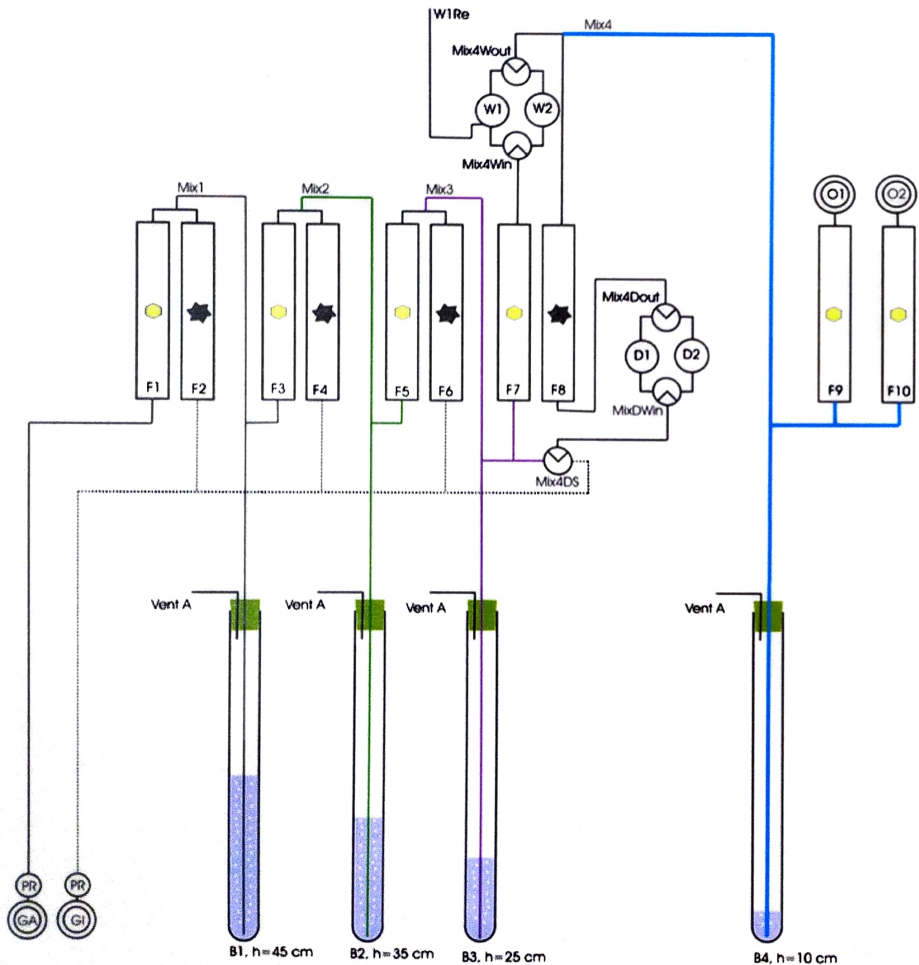
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(A Central University Established by the Central University Act 2009 No 25 of 2009)

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- 1. SPECIFICATION And OPERATIONAL MANUAL of FCMIX.**
- 2. SPECIFICATION And OPERATIONAL MANUAL of PROGASMIX.**





FCMix



User Manual

Version 1.000

April 2012

NorECs AS

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1 Introduction

Welcome to the FCMix manual.

It covers the FCMix and accompanying “Probble” humidifiers and pressure controls.

The manual intends to provide you 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. They form one part of the training for the user responsible.

Thereafter we provide instructions for

- Normal operation

which shows how the FCMix and Probble humidifiers and pressure controls are used in normal operation 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.

The section also contains some remarks on alternative uses.

Finally, we provide more detailed technical reference sections for safety and conformity.

NorECs AS

April 2012

2 Safety First

In this section we list important aspects of safety of using the FCMix. **Read this first** in order to be prepared and post it's *Do's and Don't's*. Later sections will provide a deeper understanding of using the FCMix correctly and safely as well as more detail and reference of safety aspects and technical specifications.

2.1 Normal safe use according to specifications

Purpose: The FCMix controls and displays flows of 3 input gases intended for use in a small fuel cell; oxidant (ox), inert gas (inert), and fuel (red). The flowmeters are marked and calibrated according to the actual gases selected upon ordering. Default is Air, Argon (Ar), and Hydrogen (H₂). The inert gas can be routed for mixing into the oxidant or into fuel gas, for safety, soft-start, and tests of effects of dilution of the fuel or oxygen.

The Probble humidifiers and pressure controls are connected after the FCMix, and are used to humidify oxidant and fuel separately and to provide overpressure safeguard. They furthermore receive the gases after passage through the fuel cell and there provide pressure control, flow visualisation, and outlet to ventilation.

Normal safe use implies careful control that oxidant and fuel gas lines are not intermixed anywhere along the way. We provide a sticker sheet with colour coding for markings on inlets and outlets, and devices, and gas lines for this purpose.

Input gases: As oxidant may be used the gas specified (default; air) or any of oxygen (O₂) or air (natural or artificial). As inert gas must be used the gas specified (default: argon, Ar) or any of nitrogen (N₂) or the noble gases (e.g. He, Ar). As fuel may be used the gas specified (default: hydrogen, H₂) or any of hydrogen H₂, methane CH₄, CO, propane C₃H₈, or mixtures of these or with inert gases. Ammonia (NH₃) may not be used with the present version of the FCMix because of corrosion, and not with the Probble as it will react with the water.

Gases must only be connected to the appropriate input. We suggest to adhere to the colour coding

- **blue** = oxidant
- **green** = inert
- **red** = fuel(flammable).

Mark new lines and devices accordingly.

NOTE: The presence and use of inert gas for flushing and soft-start according to this manual is required, and must not be omitted.

Pressures: Apply input gases to the FCMix at pressure preferably in the range 1 - 3.5 bar and not exceeding 6 bar.

2.2 Residual hazards

If the specifications (input gas types and pressures), correct connections of oxidant and fuel, and instructions for use of inert gas are adhered to, the FCMix and Probbles are inherently safe.

Any fuel cell may break down in ways that allow the fuel and oxidant to mix through leakages, cracks, etc. This will often lead to combustion into water and thus represent no direct danger. Unstabilities in the fuel cell may in the worst case still lead to mixtures of fuel and oxidant in the in- or outlets, and these may even reach the Probbles. For this reason the Probble is made with relatively small volumes and contain no glass parts in case of explosion.

2.3 Consequences of misuse

Application of input pressures higher than 6 bar may lead to damage to and leakage in the tubing and flow control and readout parts.

Use of other input gases than specified above may have the following consequences:

Corrosive gases: Cl_2 , NH_3 , SO_2 are examples of gases that will corrode and destroy the FCMix in its normal version, that utilises brass components and Cu tubing. Many of them will also react with the water in the Probbles.

Mixing up connections of gases anywhere on the inputs or outputs of the FCMix, Probbles, or the fuel cell, may lead to a mix of fuel and oxidant. Failure to flush with inert gas prior to use of fuel may have the same effect. Mixes of fuel and oxidant may combust in the fuel cell, or explode if ignited (by e.g. static discharge) in the FCMix, Probbles, fuel cell, or elsewhere in the gas lines. Because of small volumes and absence of glass parts in the FCMix and Probbles, the risks of serious damage or injuries are small. It is nevertheless strongly advised to wear safety goggles at all times when working with this and other apparatus handling flammable or explosive gases.

2.4 Do's and Don't's

Wear safety goggles at all times.

Connect appropriate gases only to the corresponding input.

Do not cross fuel and oxidant lines.

Adhere to the colour coding.

Do not omit the inert gas and its proper use.

Keep Probble water levels between the specified limits.

3 Installation

3.1 Unpacking and inspection

The package normally contains the 1 x FCMix, 2 x Probble humidifier and pressure control devices, 6 lengths of gas line tubing (PTFE) for connections between the FCMix and the Probbles and between the Probbles and the fuel cell and back, 2 lengths of PVC hose for output to ventilation, 3 + 2 Swagelok stems, the Manual and documentation, and minor accessories.

Note that the documentation – in addition to the Manual – contains also manufacturers' documentation for the flowmeters.

Check that the packaging and all parts appear without visible damage from the transport. In case of visible damage it is likely that this has resulted from rough handling during transport. Please notify NorECs and if possible also the transporter, without delay.

3.2 Introduction to the devices

3.2.1 FCMix

The FCMix is preassembled and need not be opened during installation or normal use. Its schematics are shown in Section 6.1.

Its flowmeters run on batteries that will last for approximately 2 years, and has thus no need for mains power. The batteries are preinstalled upon delivery, and the flowmeters are thus permanently on: Check that the LED panels of all flowmeters are on (e.g. showing zero flow).

Please identify and check that you understand the functions of the following items on the FCMix:

On the **rear** are three (3) gas inputs on one side (left-hand side when seen from the front, right-hand when seeing the box from the rear). These use Swagelok quick-connects with valves. They are marked with the oxidant gas (ox), inert gas (inert), and fuel gas (red).

On the **rear** are also two (2) outputs on the other side, oxidant (ox) and fuel (red). For simplicity, these are still marked the same as for the inlet, although the inert gas may be mixed into one or the other.



Figure 1. Rear and front of FCMix.

On the **front plate**, from left to right, you find for each of the gases a symbolic input, a stop-valve, a flowmeter with digital battery driven display and manual needle valves for flow regulation.

In addition, the inert line (default Ar) has a “3-way” selector with which it can be set to feed (mix into) the oxidant gas or the fuel gas, or neither (closed). It may be noted that the valve cannot be set so as to mix oxidant and fuel.

Finally, there are two symbolic outlets on the front plate.

The FCMix has particle filters mounted on its three gas inputs.

NOTE: FCMix can be used without the Probbles. Considerations for this is treated in a later chapter. However, here we will onwards assume you will use the Probbles.

3.2.2 Probble humidification and pressure control devices

The details of the Probble are shown in schematics in figure 2. Note that the two Probbles are physically identical, but may be pre-marked with oxidant (blue) and fuel (red) (default Air and H₂). Adhere strictly to this.

Each Probble is preassembled for default operation mode.

You may at this time fill both Probbles with water – preferentially distilled or deionised – to the maximum level.

Each Probble device has two plexiglass bubbler tubes and one small hose stub mounted on the lid.

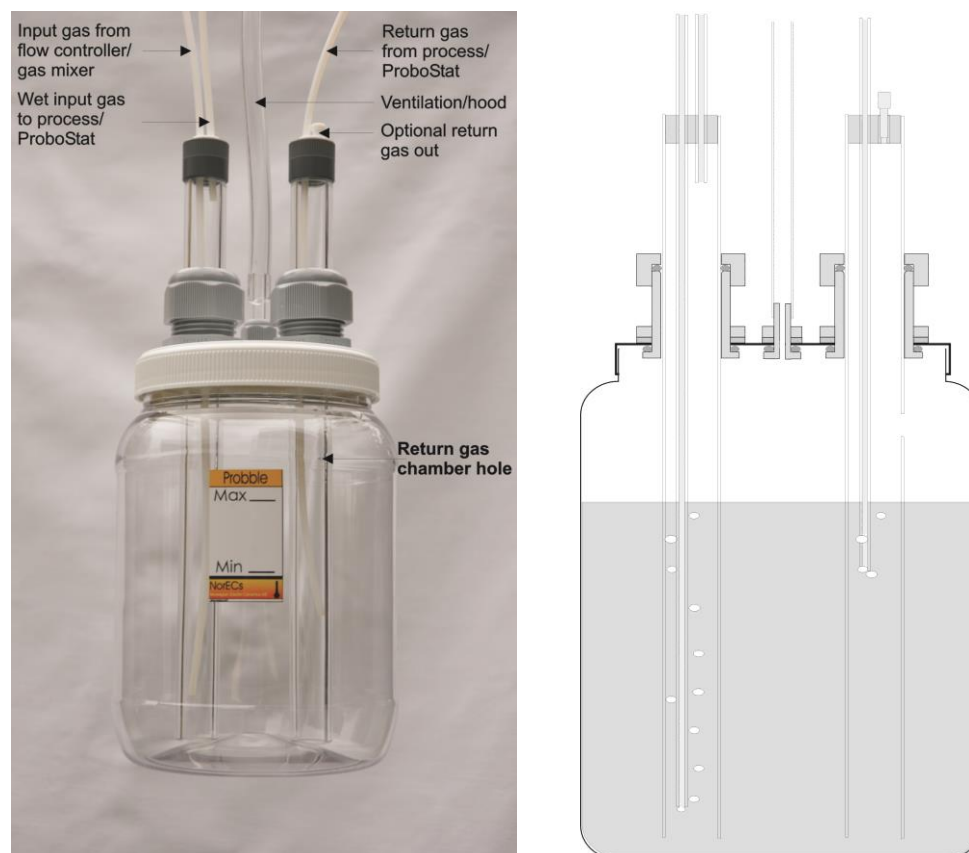


Figure 2. Left: “Probble” humidification and pressure control device delivered with FCMix. Right: Schematic of a Probble. Constructional details may vary.

The stub is the common outlet and should be led to ventilation using an open hose. This stub may also be used to refill water.

You must understand the difference between the two plexiglass bubbler tubes on each device, and their functions and connections. Look at one of the Probble devices and find its two bubbler tubes (PMMA plexiglass):

The *first* gas bubbler tube has two connections on the cap and *both* (input and output) are open.

The *second* gas bubbler tube has *only one* connection on the cap (input) that is open – the other is plugged. This bubbler has instead a small hole in the plexiglass tube wall above the water level, functioning as its output.

Here is how it works: The *first bubbler* receives gas from the FCMix oxidant (default Air) output into its thin, long PTFE hose (input) which then bubbles within the enclosing outer plexiglass tube, and out of the first bubbler's output (short PTFE hose). (Only if the path onwards is blocked will the gas press the water column down the outer tube and bubble outside it, acting as an overpressure relief.) The gas goes from the first bubbler to the gas input for the *appropriate electrode chamber – the cathode* - on the fuel cell. It returns from the same chamber and is entered into the thin PTFE hose of the *second* bubbler on the *same* Probble device. It exits through the hole in the plexiglass tube and continues out the stub to the ventilation hose.

The other device is exactly the same, and has the same functions, just supplied and working with the fuel gas from the FCMix to and from the anode chamber of the fuel cell.

We will next describe the connections and operating principles in more detail, but be certain that you understand what is meant by the two Probble devices (for oxidant and fuel), and their two bubblers (first and second), and where the gas goes in and out on each of the bubblers.

3.3 Gas line connections

3.3.1 General

We now describe the connections of the gas lines connecting the various parts. For pedagogical reasons we do this from left to right, from supply to exit, in Figure 3 below. However, keep all valves closed during mounting. We will first give some advice on location of units and how to connect tubing.

3.3.2 Locate the units

Place the Probbles preferably not more than 1 meter away from the FCMix, and as close as possible to the fuel cell. The latter is to reduce pressure loss in the tubing. Moreover, place the Probbles as low as possible compared to the fuel cell, in order to reduce the risk that water formed in the fuel cell clogs the gas line.

3.3.3 Tubing

The gas lines are made by PTFE tubing. The 6 lengths are intended used between the FCMix and Probbles and back and forth between the Probbles and the fuel cell.

When you have decided the locations of the FCMix, Probbles and fuel cell (ProboStat or other), cut the tubes – especially between the Probble and fuel cell - to keep the lengths not longer than necessary.

The PTFE tubing is connected to the Probble by simply inserting the tip of the tube into a hole on top of the plexiglass tubes and pushing down to the desired depth, as discussed later.

The PTFE tubing is connected to the FCMix and fuel cell (in case it is a ProboStat) using Swagelok tube fittings with stems, included with your FCMix and ProboStat..

3.3.4 How to make Swagelok tube fittings

When making Swagelok connections, be sure that you know the procedures. This can be learnt in Swagelok documentation, and is briefly repeated here:

When making a *new* connection, be sure that the ferrules are inside and correctly placed. You can trust this is the case if the tube connection is brand new from Swagelok and never has been opened. Insert and keep the tube all the way in, and fasten the nut with your fingers. From finger-tight, tighten $\frac{3}{4}$ turn (for 1/8'' tubing) by using two 7/16'' wrenches.

When re-connecting a *previously connected* Swagelok tube connection, the ferrules are already in place and have deformed the tube. In this case, only tighten 1/8 turn by the two wrenches, using only the force of two fingers.

3.3.5 Gas supply to FCMix (3 lines)

In the following, make reference to Figure 3 below. First connect each high pressure (max. 6 bar) supply of oxidant, inert gas, and fuel to the three respective inputs of the FCMix. For this purpose use the *three quick-connect stems with valves (B-QM2-D-200)* that are included with the FCMix shipment; they are to be connected to 1/8'' Cu or polymer tubing. The user should mark the stems with the appropriate colour coding:

- **blue** = oxidant
- **green** = inert
- **red** = fuel(flammable).

Check that all three inlet valves on the FCMix are closed. Then insert the three gas lines by pressing the ring on the bulkhead quick-connect inwards as the stem is inserted.

3.3.6 FCMix to first bubblers inputs (2 lines)

Next, connect the oxidant output of the FCMix to the input on the first bubbler on the oxidant Probble. Similarly, connect the fuel output of the FCMix to the input on the first bubbler on the fuel Probble. For these two lines use the *two stems without valves (B-QM2-S-200)* to connect to the FCMix outlets. These stems do not have colour coding.

The length of tubing between the FCMix and the Probbles is not important – there is plenty of driving pressure for the flow through these.

3.3.7 First bubblers outputs to fuel cell inputs (2 lines)

Next, connect the outlet of the first bubbler on the oxidant Probble to the oxidant (cathode side) inlet of the fuel cell. Similarly, connect the outlet of the first bubbler on the fuel Probble to the fuel (anode side) inlet of the fuel cell.

If the fuel cell is a NorECs ProboStat™, use two Swagelok quick-connect stems with valves that were supplied with the ProboStat™.

Keep tubing short.

NOTE: Figure 3 shows *an example* where the oxidant (cathode) side is the Inner chamber of a ProboStat™ and the fuel (anode) side is the Outer chamber. It is fully your responsibility to understand and implement the use of the correct chambers and their in- and outlets for your fuel cell in your laboratory.

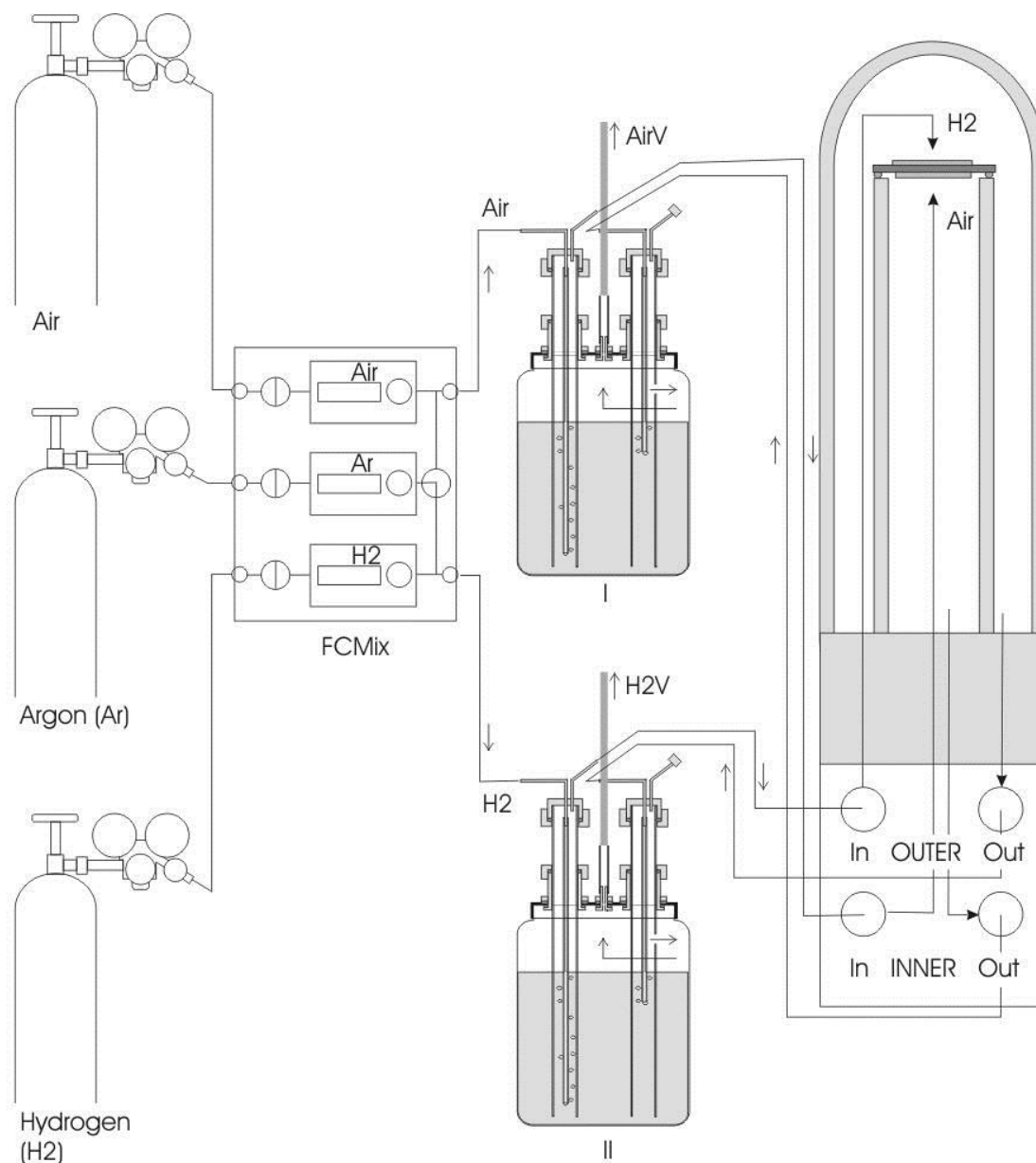


Figure 3. Connections of the inlets of the FCMix to oxidant, inert, and fuel gas supplies (default Air, Ar, and H₂) and of the outlets to two Probble humidifier and pressure control devices, and of these feeding the electrode chambers of a fuel cell test, and of the exit gases returned to and bubbled in the same device for pressure adjustment and flow visualisation. Both overpressure

safeguard relief gas and return gas exit to ventilation through two lines (AirV and H2V) that should be kept separate. **NOTE:** Care must be taken to ensure that the exit of one cell chamber returns to the same humidification device that feeds that chamber. The fuel cell configuration exemplified is that of a NorECs ProboStat™ (only gas connections shown, electrical connections omitted).

3.3.8 Fuel cell outputs return to second bubblers inputs (2 lines)

Next, connect the outlet of the oxidant (cathode) side of the fuel cell to the input of the second bubbler on the oxidant Probble. Similarly, connect the outlet of the fuel (anode) side of the fuel cell to the input of the second bubbler on the fuel Probble.

If the fuel cell is a NorECs ProboStat™ use two Swagelok quick-connect stems without valve that were supplied with the ProboStat™.

Keep tubing lengths short.

NOTE: *It is very important that you here do not cross lines; the oxidant gas must end up at the oxidant Probble and the fuel gas at the fuel Probble. Use colour coding and adhere to it.*

3.3.9 Common outlets to ventilation (2 hoses)

Finally, connect the common outlet stub of the oxidant Probble to safe ventilation or hood. Connect similarly the common outlet stub of the fuel Probble to safe ventilation or hood. For these, use separate hoses.

NOTE: *Make sure that the ventilation hoses are open and that there is no possibility that the gas flow in them can be obstructed in any way.*

3.3.10 Control and marking

Make a final visual check that all lines go as described above, and especially that oxidant and fuel lines never cross.

Consider to add marking (color or writing or other) that is clear and meaningful to you and your staff, and that will prevent anyone from making ill-connections.

3.4 Startup and checks during initial operation

3.4.1 Wear eye-protection

Wear eye-protection safety goggles.

3.4.2 Water level

If not done before, fill both Probbles with water – preferably distilled or deionised – to the maximum level. The maximum level must be below the height of the small hole in the plexiglass tube of the second bubbler.

3.4.3 Gas lines only (no need for an operating fuel cell)

With all valves on the FCMix closed, open for gas supply at controlled pressure not exceeding 6 bars to the inlets of the FCMix.

Check that all three flowmeter valves are closed (turned fully clockwise (don't use force!)).

Open the three inlet stop valves. The flow indicators should be at or come to rest at zero flow.

Set the inert gas routing valve to the oxidant side.

Turn up the flow on the middle, inert gas, flowmeter, using its needle valve. A positive flow should read in the LED window of the flowmeter. Observe that the inert gas is now led into the output of the oxidant line, and it should start bubbling inside the plexiglass tube of the first bubbler on the oxidant Probble.

If it bubbles, OK. If it does not, turn the flow down, close the inlet valves, and look for solutions:

- If there was no steady flow in the flowmeter window (and no bubbling) it is possible that
 - o there was no gas present on the input
 - o the inlet valve was not open
 - o the inert gas routing valve was not pointing to one of the output lines
 - o the wrong type of quick-connect stem was used
 - o or – in the least likely case - a Swagelok connection was made too tight so as to close the tube opening.
- If there was positive steady flow in the flowmeter, but no bubbling, it is possible that
 - o the oxidant line from the FCMix was connected to the wrong place on the oxidant Probble
 - o the oxidant line from the FCMix was connected to the wrong Probble
 - o the wrong type of quick-connect stem was used
 - o the inert gas routing valve was not pointing to the oxidant line.

Close valves, check and correct, carefully reopen valves and repeat the procedure until it works (flow and bubbling) as expected.

Now check whether the gas continues to bubble only on the inside of the plexiglass tube, or whether the column of water inside is pressed down and the gas eventually starts bubbling on the outside. Both are safe:

- If the gas continues to bubble on the inside and the water level stabilises this means that the gas is continuing out of the first bubbler and finds its way into the fuel cell (if connected).
- If the water level is pressed down and it eventually starts bubbling on the outside, this means that the onwards gas flow is more or less obstructed. The gas will now instead flow out of the Probble through the common outlet and to ventilation via the hose (which must be open and unobstructed).

If the gas flows outside because the pathway onwards is obstructed, this is OK.

If the gas flows outside while the pathway onwards is in fact open, you are trying to push through the system more gas than can be realised with your Probbles: The resistance in the tubing etc. requires a higher pressure and the column of the first

bubbler is not enough and it over-bubbles. Reduce the flow and/or the length of the tubing.

If the gas is indeed flowing well, you may try to stop it somewhere down the line (e.g. by retracting the stem from the fuel cell input – if the stem has a valve, like in the ProboStat™, the gas will stop). Then observe and check the bubbling out of the plexiglass tube (overpressure relief).

While inert gas is running to the oxidant line, add oxidant gas by turning up the flow on the oxidant gas flowmeter. Observe and check that the bubbling increases. Afterwards, turn the oxidant gas back down to zero.

Turn the inert gas routing valve to the fuel side. Observe and check flow and that bubbling starts in the fuel Probble. If not, see the suggestions above.

Rise the flow of inert gas to a considerable level. Let it run for a minute to flush out air. Add a small (less than 5 %) flow of fuel. Observe the flow in the flowmeter. Then turn the fuel flow back down to zero.

Turn the inert gas flow down to a moderate level.

You have now verified function and flow for all three gases in the FCMix and the first bubblers of the two Probbles. You have seen how the inert gas can be used for flow in the oxidant or fuel lines (but not both at the same time) for flushing and function testing, and how it can be used to dilute fuel to a safe level for startup operations.

3.4.4 Gas lines onwards through the fuel cell

The following assumes you have a fuel cell system of some kind – operating or not – connected. It could also be some other kind of application, like a membrane test setup.

Let inert gas flow through the one of the Probbles and onwards to the fuel cell.

If the cell is properly sealed towards the surroundings, the gas will be flowing back to the Probble and will bubble in the second bubbler. If the two chambers of the cell are not sealed, the gas may leak between chambers, and it will flow back to the Probble with lowest overpressure (bubbling height) of the second bubbler.

You may repeat this for the other Probble by directing the inert gas there at the FCMix.

You may vary the heights of the second bubbler by loosening the nut around it close to the lid and sliding the whole assembly up and down and re-fastening. Ensure that the hole in the plexiglass tube remains between the lid and the water level.

If everything operates as expected, the initial testing of gas lines and functions is hereby finished. You have learned to use the bubbling in the second bubblers as a coarse means to verify gas tightness to the surroundings and between chambers. You have also seen that you may vary the relative pressures by adjusting the heights of the second bubblers, and that this may be done to enforce leakage flow between chambers.

3.4.5 A word on water cooling and water condensation

Since the gases are humidified, no parts of the gas lines or ventilation or the fuel cell should be cooled below room temperature, as it may cause condensation of water. If such condensation occurs it makes itself known sometimes by fluctuations in the flow (as droplets move back and forth – up and down) or – in a low-pressure system like ours – by simply stopping the flow, so that the overpressure relief starts operating.

Therefore: Avoid having cooling water lines even near the gas lines.

And do not cool the fuel cell more than necessary. Let it rather be considerably above room temperature than near room temperature. If the fuel cell is a ProboStat™ there is probably no need at all to cool the base unit - if tolerates a lot of heat. (See also ProboStat™ manual.)

Later on we will return to this issue and further measures against water condensation.

3.4.6 Leakage tests

A simple and rough test of leakages to the surroundings may be done by letting gas flow in one or both lines so that there is bubbling in both first and second tubes. Then stop the flow by closing one or more valves in the FCMix. The bubbling stops. If the system is gastight the gas column in the thin inner tubes will remain constant. If there is leakage to the outside, these columns will decrease.

If the pressures and columns are different for the two chambers, the rate by which they level out is informative for the leakage between the chambers.

The tests above are best done with a cold fuel cell, to avoid thermal fluctuations.

Tests of gas tightness and sealing efficiency of an operating fuel cell is a more special issue, the we come back to below under.

3.4.7 How to leave the system after use

If the system will not be used for some time, it is suggested to flush the fuel line with inert gas, then simply close all gas inlets and otherwise leave the system as is.

An alternative may be to leave a minute flow of inert gas flow through the fuel line.

4 Normal operation

We describe the normal operation of the system in terms of a rather specific example; an SOFC test – discussing many detailed subjects along the way. However, the system can be used also for other types of fuel cells, with most of the considerations the same as for SOFC. Thus, other types of fuel cells, as well as other uses, are described only briefly after the full treatment of the SOFC case.

4.1 Solid Oxide Fuel Cell (SOFC) test

In the following we will go through a typical use of the mixer to manage a test of a small SOFC. We assume you have mounted the cell with electrodes and sealing such as a gold gasket. The cell has a typically a Ni-YSZ cermet anode, but before mounting this is NiO-YSZ; the NiO is reduced only by use of a reduced gas after sealing.

4.1.1 Heating up in a safe atmosphere before and during sealing

In order to start the process in as safe and gentle way, do the initial heating with a flow of oxidant to the cathode side and a flow of inert gas to the anode side.

4.1.2 The sealing process; utilise a pressure difference

We suggest that you before the sealing starts make a difference in pressure of a couple of cm of water column between the two chambers, so that the higher pressure is on one side of the fuel cell and presses it slightly towards the seal (and not away from the seal). If the fuel cell is a ProboStat™ this normally means applying a higher pressure to the outer (top) chamber, pressing the sample down towards the support (helping the spring load).

The pressure difference is set by retracting the second bubbler on the low-pressure side a couple of centimetres.

This will make gas from the high pressure side exit through the leak and through the low pressure bubbler.

We suggest at this stage to stop the supply of gas to the low pressure chamber, so that gas runs only into the high pressure chamber and out from any chamber depending on the leakage.

The sealing may now proceed. If it comprises a metal it may mean heating to close below the melting point. If it is a brazing or glass ceramic it requires heating to a specified temperature.

As the sealing proceeds, this flow on the low pressure side will get smaller and smaller and the flow out of the high pressure side higher. If the low pressure flow stops entirely, the sealing is qualitatively successful. In some cases it is then desirable to cool the cell considerably to prevent the seal to flow too much.

If there is still a minor flow on the low pressure side of gas out through a leakage, the user must make own judgements on whether to continue to let the seal heal, whether it is an acceptably small leak, or whether to abandon this run.

4.1.3 Equalling the flows and pressures

After the sealing is satisfactory, turn up the flow of the gas to the low pressure side (which we stopped during sealing). We suggest to operate with equal flows on both sides as a starting point.

The pressures should next be equalled by equalling the second bubbler heights (water column heights). This will minimise cross-flows and mixing of oxidant and fuel.

4.1.4 Monitoring the electrical performance of the cell

Before continuing, it is suggested to have done or running measurements of AC conductivity or of DC open circuit voltage (OCV), or both (via a manual switch or reconnection or an automatic switch/multiplexer). These data are valuable for evaluating what goes on during the forthcoming introduction of fuel and the anode reduction.

4.1.5 Reducing the anode

Now, the fuel side electrode (anode, normally NiO-based) may be reduced (to Ni) if required. This is normally done by going to the appropriate temperature selected for reduction, and then mixing in an increasing amount of fuel gas to the inert gas already running to the anode of the fuel cell.

Be aware that the reduction may increase the water vapour level in the exit gas considerably, and that water may condense in the gas line. For this purpose, you may want to introduce the hydrogen slowly, perhaps supplying it drier or taking measures to collect the water.

Normally one will observe a dramatic increase in OCV and conductance during the initial reduction. This will level out as the process completes.

The cell now is operative.

A good fuel cell stays stable in this operative state. A mediocre cell may degrade in OCV or conductance over time. A poor cell will immediately start to degrade – often sooner than the maximum OCV has been reached. This is why collecting data in the initial stage is important.

It may be mentioned that the reduction process itself may be a decisive factor for the microstructure of the anode, and thus for the stability and performance of the cell. Factors that may affect the reduction process are temperature, concentration of hydrogen, rate of increase of the concentration of hydrogen, and total flow rate of fuel side gas.

4.1.6 Living with leakage; optimising the OCV

Many fuel cells will have leakage in sealing or in the electrolyte itself. These leaks may be of various kinds and behaviour depending on the size of the openings, type of gas species, and temperature. There may in addition be diffusional leakage through sealing or other components, especially of hydrogen, which is permeable to most materials. There may finally also be electrochemical leakage through the electrolyte if it exhibits some mixed ionic electronic conduction.

Normally one would like to minimise the effect of such leakages. This can be done by increasing the flows of one or both gases. Furthermore, one may adjust the pressures

of the chambers by adjusting the second bubbler columns. Be aware that flows affect the pressures in the cell slightly, so that flow and pressure adjustments are interrelated.

Sometimes, it is beneficial to operate with a deliberate small flow of one gas from one chamber into the other. This may be because one electrode is less exposed to the leakage stream than the other. Moreover, if hydrogen is used in the pure state and flows more easily than other gases in small openings, it may reduce the overall mixing of gases if the openings are filled with the slower flowing oxidant gas, i.e. if there is a small deliberate overpressure on the oxidant side.

4.1.7 Hot-spots?

A fuel cell can operate well with some leakage. However, there will be evolved heat from the reaction between the fuel and oxidant – such leaky spots are thus named hot-spots. They may possibly destroy parts of the sealing or electrolyte. A leaky cell is therefore probably best kept running on diluted fuel.

4.1.8 Too much water from leakages?

Leakage and consequent mixing in the cell will (should) react immediately and form water. The amount of water depends on the leakage and on the flow and content of fuel and oxidant. The problem of the water is – as mentioned earlier - that it condenses and may obstruct or eventually block gas lines. We will return to remedies against this problem later on, but for now just mention that cells with large leakages for this and other reasons are best left running on diluted fuel.

4.1.9 Fuel cell performance

Fuel cells and their performance are complex and there are many parameters to characterise.

Firstly, the OCV should be measured and checked against the theoretical value. The OCV should vary with the hydrogen to water ratio of the fuel gas and with the oxidant content. Thus, mixing inert gas into either the fuel or oxidant gas will affect the OCV.

One furthermore wants to measure the I-U-curve of the cell, namely its voltage U vs the load current I . (U is often replaced by V , so that it would be V vs I (I-V-curve)).

The curve is often shaped like a straight line (the ohmic loss due to electrolyte and other resistances) with bends at low and/or high currents. We here make a few comments about the factors that tend to be dependent on the gas composition and flow:

The curvature at low currents (near open circuit) may be due to polarisation of the charge transfer process at the electrode; the resistance and slope gets smaller as the overpotential increases (Tafel behaviour). It may also reflect a change in the composition of the gas. With the two Probbles, both gases are humidified, so that gas compositions stay relatively constant at both electrodes, both for oxide ion and proton conducting SOFCs.

The curvature at high currents comprises approach to a limiting current. This signals that there is not enough supply of reactants to the electrodes, or not sufficiently fast removal of products; we enter into mass transport limitations. These may be lessened by increasing the flow of gas and usually by increasing the content of reactant (oxidant or fuel). Testing whether the content and flow of oxidant or of fuel make the main difference can thus tell you which electrode is the limiting one.

4.1.10 Operate with low or high fuel utilisation?

The above brings up an important question, namely whether to test the fuel cell under conditions of low fuel and oxidant utilisation, or under conditions of high fuel (and possibly oxidant) utilisation:

If the flow of fuel and oxidant is high compared to the current load, we have low fuel (and oxidant) utilisation. The limiting mass transfers are then usually reduced and the cell performs electrically at its best. This is beneficial for characterising the electrolyte and electrode kinetics (overpotentials) under relatively constant gas concentrations.

High flows to current ratio (low fuel utilisation) moreover has the advantage that the increase in content of water is minimal, reducing the risk of condensation in the outlets.

However, running at low fuel utilisation is of course not very realistic for a final fuel cell application; the overall efficiency from fuel to electrical energy becomes very poor, and it may be interesting to decrease the supply of fuel and perhaps also oxidant.

High fuel utilisation is obtained by supplying low flows of fuel, compared to the current load. This will bring about mass transport limitations and the performance of the cell will decrease, realistically.

One must furthermore be aware of the danger of producing enough water to get condensation.

A way to reduce the problem with water may be to reduce the content of fuel instead of the flow; by having a larger overall flow, the water content in the exhaust becomes smaller. There are, however, also other remedies, see below.

4.2 Further issues on water vapour

4.2.1 Reduced humidification

There may be reasons to reduce the level of water vapour in the humidified gases. This may be desire to tests applications with dryer gases, or it may be necessary to avoid condensation with cells that produce a lot of water internally.

In order to reduce the water level, one may simply open the first bubbler of the Probble and shorten the hose to above the water level, or simply remove it. The gas then obtains only a partial humidification from the surface of the water, depending on the flow rate.

4.2.2 Controlled temperature chiller bath for the humidifiers

The Probbles may be immersed in a chilled bath. This will give a smaller and more constant level of water vapour, and reduced chance for condensation later in the gas line.

4.2.3 Other remedies for problems of water condensation

First of all, avoid having cooling water lines near the gas lines. Also, do not cool the fuel cell or other devices too much – perhaps better not at all - they are best run well above room temperature.

If the fuel cell produces too much water by load or leakage, or during reduction of anode NiO, one may apply natural or active heating of the base units of the cell. Moreover, the return gas lines may be heated using e.g. heating tape.

If using a ProboStat, you may furthermore use a tap in the exit hole of the chamber with the wet gas – this will prevent water from exiting until the level stands above the height of the tap. Extra outlet tap for the outer chamber (equal to the inlet tap) can be obtained from NorECs.

Condensing water can be handled also by having a continuous downhill path of the tube from the fuel cell to the Probble.

It may be an idea to insert an unheated container in the line to collect condensed water before going back to the Probble or to another ventilation exit.

4.3 Other types of fuel cells

4.3.1 Polymer electrolyte membrane fuel cells (PEMFC)

PEMFCs have electrolytes such as Nafion© that need water and conduct H_3O^+ ions. The cells may thus run preferentially using both humidifiers of the FCMix.

4.3.2 High temperature PEMFC

High temperature polymers like phosphonated PBI work above 100 °C and are in principle purely proton conducting, but also here humidification of the gases – at least the fuel gas - is probably beneficial for long term operation.

4.3.3 Solid Acid Fuel Cells (SAFC)

These use acidic salts, such as CsHSO_4 or CsH_2PO_4 , as electrolytes and work around 200 °C. They are pure proton conductors and do in principle not require humid gases. Again, however, it is likely that the long term stability of the electrolytes is favoured by humidification.

4.3.4 Proton conducting solid oxide fuel cells (PC-SOFC)

Such cells use oxides with predominant proton conduction as electrolytes.

PC-SOFCs produce water on the cathode (oxidant) side instead of the anode. This must be taken into account when considering the possibilities of condensation.

Another issue with these cells is that water vapour is necessary the proton conduction as such. Thus, wetting both the oxidant and fuel may be beneficial for the operation of such cells.

4.4 Other applications

4.4.1 Gas separation membrane testing

The FCMix system may be used to supply two gases to the two sides of a gas separation membrane.

For instance, one may measure oxygen permeation between oxidant (O_2 or air) on one side and inert gas or inert gas with H_2 on the other side.

One may similarly measure hydrogen permeation between H₂ on one side and inert gas or inert gas with O₂ on the other.

One may choose to feed premixed gases in order to have another component for differentiating between leaks and diffusion.

4.5 What the FCMix & Probbles can not do

The Probbles is not designed to supply humidification above dew point of room temperature. It moreover cannot supply gas at pressures much more than atmospheric. If any of these are desirable, one may use the FCMix standalone.

The Probble can not support high flows, because its short water columns cannot uphold a high flow through the thin tubing, and it will start to flow over. In its standard setup it can take 50 ml/min, which can be increased to 100 ml/min if water level is kept high and tubing heights are adjusted. Light-flowing gases like H₂ can operate at somewhat higher flows.

4.6 Use of FCMix standalone without Probbles

The FCMix can be used without the Probbles. One must then take special care to ensure another way of overpressure relief.

One may consider to use a FCMix to simply mix two gases for any application.

4.7 Use of Probble standalone

The Probble humidifier and pressure control devices can be used separately. However, remember that they contain no flow control; there must be a needle valve to control the flow, e.g. as part of a flowmeter, in front of the Probble.

4.8 Calculations

4.8.1 Gas flows and mixing

The flowmeters are calibrated for the gas specified and display the flow directly.

Other gases may be used, but only within the same category (oxidant, inert, fuel/flammable). The flow must then be recalculated from the flow displayed by a conversion. Contact NorECs AS or the flowmeter manufacturer to obtain the conversion.

After mixing in inert gas in any of the oxidant or fuel gas, the total flow can be calculated as the sum. The content of the components after mixing is calculated as simple ratios given by the individual flows of the two gases divided by the total flow.

After humidification, around 3 % of H₂O(g) is added to the total flow so that the content of H₂O becomes 3 % and the other components are reduced to 97 % of their value in the dry gas.

The flows referred to here are in ml_n/min or l_n/h where the subscript “n” refers to standard conditions. In a high temperature cell, the flow will be the same in these

normalised units or in e.g. mol/s or mol/min or mol/h. However, the gas will be diluted and the velocity of the gas will be higher.

4.8.2 Humidification

The water vapour content in the gas after bubbling through the Probble will be close to the saturation pressure of water vapour over pure water at the temperature at hand (normally room temperature). This pressure can be found in Handbook tables or calculated by Thermochemical software.

The effectiveness of the bubbler will vary with flow, depth, temperature, bubble size etc. You may expect humidification of, 95-100 % of theoretical. Thus, the accuracy of the obtained p_{H_2O} is not very high.

As a first approximation of all the above factors, we suggest that 0.03 bar (0.03 atm, or 3 %) H_2O is used as a reasonable statement of p_{H_2O} in the wetted gas.

The precision of this may be increased by measuring the room temperature and looking up in the tables. Moreover, calibration or check with a humidity meter can be worthwhile, but such instruments are often not very accurate.

4.9 Maintenance and service

The only needs for maintenance are the refilling of water in the Probbles as necessary and replacement of flowmeter batteries every 2 years. (Note: If a battery goes low, it does not affect the flow, only the reading; it is entirely the manual valve that determines the flow.) For service, contact NorECs AS.

5 Troubleshooting

5.1 No display in FCMix flowmeter panel

Background: The panels LCD panels should show some letters and a reading of e.g. 0.0 even if the mixer is not in use or no gas is flowing.

Symptom: No display.

Cause: The battery is empty and needs to be replaced

Action: Order from NorECs or the flowmeter manufacturer directly.

Note: The FCMix can still be used in a qualitative manner for supplying one gas or flushing, using the bubbling in Probbles as indicator of flow.

5.2 No flow in FCMix flowmeter

Symptom: There is no positive reading of flow after opening the needle valve several turns counterclockwise.

Cause: The gas pathway is closed at some stage before or after the flowmeter or there is no supply of gas.

Action: Turn the flowmeter fully down (clockwise) before looking for the error and solving it. Check if the input valve (to the left of the flowmeter) is open (horizontal). If the gas is the inert gas (in the middle) check that the selector valve on its output (to the right of the flowmeter) is open to the output gas you want (fuel (up) or oxidant (down)). Finally, check that there gas line is connected to the appropriate FCMix input using a stem with valve (supplied with the FCMix), and that there is gas pressure on the line. When the error is found, close all valves, apply pressure on the input line, and carefully open the valves in the right order and finally open the flowmeter valve counterclockwise (several turns if necessary) to obtain a flow reading.

5.3 No bubbling in the Probble input despite flow in the flowmeter panel

Caution: Since you are working with a fuel cell and potentially with a fuel and an oxidant, this error may indicate a potentially dangerous situation, namely that you have misconnected one of these gases, with the potential danger of mixing fuel and oxidant

Cause 1: There is a disconnected or misconnected line between the FCMix and the Probble.

Action: Identify the missing or wrong connection and fix it.

Cause 2: The plastic tube is not inserted deep enough into the water.

Action: Inserte the tube till just a few millimetres from the bottom of the plexiglass tube.

Note: There is not really a problem if the tube doesn't go into the water. The gas still flows – it just takes with it less water vapour.

5.4 The Probble input bubbles over into its main chamber

Cause 1: The gas line further on is stopped. This may be normal if it is deliberate. The overbubbling is then your overpressure relief. If it is a mistake, identify the reason. A typical reason may be that the input to a ProboStat is made to the wrong entry or with the wrong quick-connect stem: ProboStat inputs have a valve and needs to be connected using a stem also with a valve.

Action: If the gas stoppage is undeliberate, identify the cause and fix it.

Cause 2: The gas line into or – more typically - out of your ProboStat or other process chamber is fully or partially clogged by condensed water. This can be a result of a cold-spot along the gas line (no point should be colder than the Probble humidifier). It can also be a result of water forming during drying/dehydration of a material, during a chemical reaction (e.g. reduction of NiO with H₂ or mixing of CO₂ and H₂), during operation of a fuel cell, or as a result of a leak in a fuel cell.

Action: Remove the water by opening and letting it out, blow it out if necessary. Remove coldspots if this is the problem. Reduce input water level by retracting the input tube into the Probble to above the water level. If a high water output is unavoidable, install a water trap directly at the outlet of the ProboStat or ensure that the gas line out of the ProboStat to the Probble return bubbler has a continuous downhill slope. The considerations and measures are also described in the parts on operation in the manual.

Cause 3: You are trying to have a larger flow than the tubing and pressures of the Probble can uphold.

Action: Reduce the flow. Fill the water level in the Probble higher. Retract the return input tube to just below the water surface.

6 Technical specifications and reference

6.1 FCMix

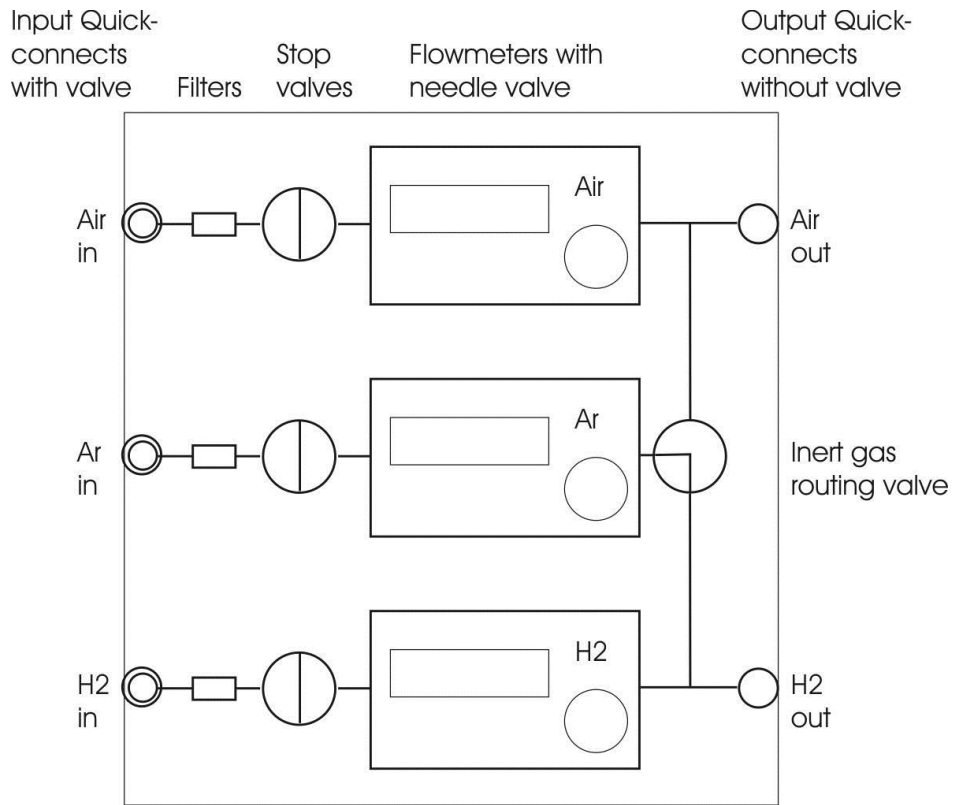


Figure 4. FCMix schematic.

6.2 *Probble*

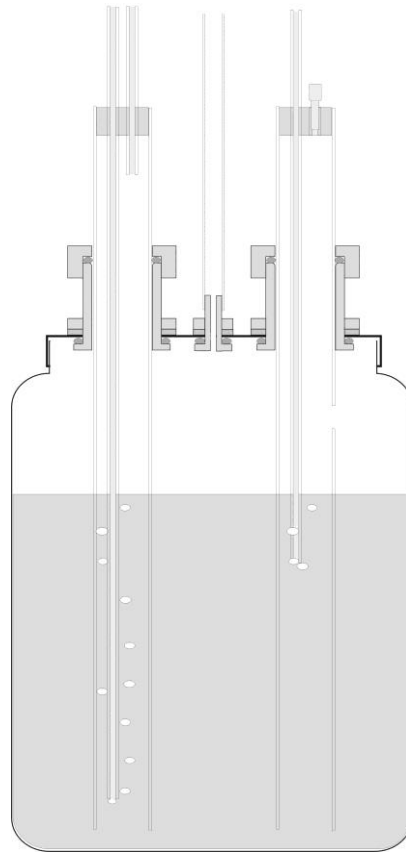


Figure 5. Schematic of Probble during normal operation.

7 Safety reference

7.1 Reference to the EC Pressure Equipment Directory (PED)

7.1.1 Conformity, Compliance, and General issues

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

The FCMix may receive and handle gases at pressures up to 6 bar.

The FCMix comprises pressurised accessories and tubing of a total volume less than 0.1 L.

The FCMix comprises pressurised tubing of DN = 6.

On this bases, referring to Annex II Tables 1-7, the FCMix 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 FCMix according to the PED, notably Annex I.

7.1.2 Design

7.1.2.1 General

The main design and component selection of the FCMix is based on normal principles for regulating and mixing flows of gases. The design of the Probble humidifier and overpressure control devices is based on many years of experience and testing of humidifying and pressure controlling bubblers. The units have been produced in prototypes and tested in real fuel cell tests.

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 the same or similar components in similar equipment.

7.1.2.3 Provisions to ensure safe handling and operation

The FCMix has no parts in danger of mistakes of handling and operation. The Probble humidifier and overpressure control devices are made without use of glass, and these and the FCMix are marked throughout with colour codes to help avoid mixing fuel and oxidant lines. The FCMix involves no voltages above 12 V. It has no hot external surfaces.

7.1.2.4 Means of examination

Each FCMix is manually assembled. It is tested for major leakages before shipment.

7.1.2.5 Means of draining and venting

FCMix and the Probble humidifier and overpressure control devices have venting outlets to be led to ventilation or hood. FCMix is equipped with an inlet for inert gas that enables flushing of the fuel cell and gas lines.

7.1.2.6 Corrosion or other chemical attack

The most corrosive gases considered that FCMix can tolerate used intermittently over many years is wet CO₂ and wet O₂.

7.1.2.7 Wear

No issues known.

7.1.2.8 Assemblies

Not Applicable (N.A.)

7.1.2.9 Provisions for filling and discharge

N.A.

7.1.2.10 Protection against exceeding the allowable limits of pressure equipment

If the user applies pressures higher than specified, some parts in the FCMix may eventually rupture, but only at much higher pressures (> 25 bar, i.e. 4 times the specified maximum pressure). Volumes are very small, and no further dangers from this are foreseen directly. However, the fuel gas may leak out. No special protection against this is implemented. However, the FCMix contains no electrical switches etc. that can ignite the fuel gas. Moreover, there are no glass parts.

7.1.2.11 Safety accessories

The unit is equipped with a set of safety goggles.

7.1.2.12 External fire

The FCMix 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

The FCMix is manufactured according to specifications for making Swagelok connections.

7.1.4 Materials

The FCMix in the standard version is made using copper tubing and brass components for all parts carrying pressure above atmospheric.

The Probble humidifier and overpressure control devices are made using polymers (plexiglass, PVC, rubber) and brass.

7.1.5 Fired or otherwise heated pressure equipment

N.A.

7.1.6 Piping

All piping is done with 1/8" Cu tubing and Swagelok tube fittings.

7.1.7 Specific quantitative requirements for certain pressure equipment

N.A.

7.2 Reference to other directories

The FCMix carries no voltages except the low voltage DC batteries internal in each of the flowmeters. There are thus no hazards related to electricity.

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

Gas Mix



User Manual

Version 0 December 2011

Update 0.602 September 2017

Truls Norby

NorECs AS and University of Oslo



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GasMix software

1.1 Introduction

1.1.1 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. The program has flexibility of configuration and use that may make it useful also for other gas mixers and applications.

1.1.2 History of GasMix – past and present

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

At present, the new GasMix software that you here are reading about and perhaps are trying out, is not new, but yet in its early development. This means that

- Some functions of the program aren't implemented yet.
- The chance of bugs is considerable.
- User friendliness and ease of use as well as screen information are not optimised.
- This manual is in the midst of being written; it may have parts where the intended sections are missing or uncompleted. There may be minor errors in references to menus etc. in the program.
- My apologies! Your comfort may be that it can only get better and that it is free! Look for updates!

1.1.3 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.

1.2 Installation and startup

1.2.1 GasMix package

The GasMix software package consists of 6 files:

GasMix.exe The program itself

GasMix Manual.pdf This manual (may also be integrated in another manual, e.g. a ProGasMix Manual.pdf)

Four types of parameter files, containing data for GasMix definition and calibration:

- ? .inp Input gases definitions
- ? .mix Mixer setup
- ? .cal Flowmeter calibration
- ? .cel Output cell setup

These may be only four, e.g. ?=Default. Or there may be more sets (?=Default, ProGasMix, UiO, etc.).

1.2.2 Copy to directory c:\Program Files\Gasmix

The package may be installed anywhere on your Windows computer. However, we suggest that you install it in a directory

c:\Program Files\GasMix\

If it does not exist, create it, and copy all GasMix package files there.

Note: On computers running with another language than English, “Program files” may appear with the local name, e.g. “Programfiler” in Norwegian. Use that directory.

1.2.3 Make a shortcut

Point at the Gasmix.exe icon. Right-click and Create Shortcut. Move the shortcut to your desktop or elsewhere. Rename it e.g. “Shortcut to GasMix”.

1.2.4 Start and run the program: GasMix.exe

Start the program GasMix.exe from the shortcut icon or in any other way. Go to Section 1.3 for a quick-start introduction and tutorial or to Section 1.4 for a more thorough treatment of GasMix operations.

1.2.5 Updates

If you later download or otherwise receive updates of the program, manual or parameter files, simply overwrite the existing ones. Consider, however, to make a copy of all existing files in a separate directory in case the original or edited parameter data will be needed or the new program version does not work on your computer for some reason.

1.3 GasMix quick-start and tutorial

This section intends to give the user a fast introduction to the GasMix software and practice in using it. For more thorough explanations refer to Section 1.4.

1.3.1 Start and run the program: **gasmix.exe**

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

1.3.2 Overview of the gas mixer and GasMix display

At the bottom of the display is 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. O₂, Air, N₂, Ar, 5% H₂ 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 coloured connecting lines – one colour for each Input gas.

Above the flowmeters, gas lines show how the flowmeters are connected to form Mixtures. Each Mixture has a colour, 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 H₂O (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. The number selector and “OK” button is for selecting the number (of Input mixture, Flowmeter, Calibration curve, or Output cell) to edit, and the “OK” button is for confirming and entering edited data.

Results of mixing and equilibrium calculations will appear on the top of the window once you start calculations.

At the bottom right is a table showing the four setup files presently loaded.

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

The “Calc Start” button executes mixing and starts equilibrium calculations.

The program has a top menu bar where file operations and editing are accessed. Moreover, it has a direct Flowmeter readings entry access, Calc start/stop, and settings access.

1.3.3 Run a simple session with typical actions

Load a set of parameter files to define everything. In the menus, go to File menu and select Open All. A file dialog allows you to find the location of the setup files (*.MIX, *.CAL, *.CEL, and *.INP). Click on one of them, and all (up to four) that are found with the same first part of the filename are loaded.

If the mixer that appears has something in at least one of the input mixture boxes at the bottom, and something in at least one flowmeter box at middle height, and lines making some connections between input mixtures and flowmeters and further to an

output cell at the top, the mixer appears to be configured with a real or tutorial example and should be ready to do something. In that case, leave the Edit menu unused for the moment.

Select FlowmeterReadings in the top menu. A table appears where you edit the readings of flowmeters. If the readings look sensible, leave them, or change one just for the test. Or you may enter for instance 3 as a reading for all flowmeters. Press the OK button. The edited readings should now be visible at the bottom of the flowmeter boxes.

Select Calc start/stop in the top menu, or the Calc start button. Now the program makes all calculations of mixtures and displays them in a table for output cells and partial pressures of gas species.

In the Settings menu change the results display between room temperature gas mixture contents and equilibrium values. Note that the selection of gas species in the two may be different, and that colours of the table change.

In the Settings menu you may also change the display from linear to logarithmic.

You may now go on doing more things, or minimise the program, or exit from File, Exit.

1.4 GasMix operations in more detail

1.4.1 Edit the input mixtures

With input mixtures is meant what you may often call gases – the gases that are supplied to the mixer from pressured bottles or lines. They are actually generally mixtures, firstly because some of them are, such as air or hydrogen that is diluted with an inert gas to make it explosion safe. Secondly, even simple gases contain impurities, and these may be entered into the input gas and makes it a mixture.

Start editing input mixtures by Edit, InputMixtures.

Next to the table that appears, the name of the input mixture s entered in the separate window. This name has no meaning other than to identify the input mixture. If it is oxygen 99.99 %, then you may for instance call it O2 or Oxygen or O2-4N.

In the table itself, enter the names of the gas species and their content pairwise in the two columns. The name must be exactly the same as the one used in the calibration curves, and normally also exactly the same as the program uses for equilibrium calculations. Typically valid names are O2, N2, Ar, H2 and H2O. Names should here not reflect a mixture. (For instance Air is not meaningful here.)

The gases contained in the input mixture may be entered in any order.

The units of content may be any as long as it is consistent for each input mixture. The program only uses the partial pressure calculated as the content divided by the total content.

Note that water (H2O) may be entered as an impurity in an input mixture, but water as such is normally not entered as an input mixture – it comes in via humidification stages.

1.4.2 Edit the flowmeters and gas mixer configuration

Start editing flowmeters and their configuration by Edit, Flowmeters.

Go to the appropriate flowmeter by using the up/down controls. Edit the table. Remember to press OK before proceeding to the next flowmeter. The mixer layout scheme changes only upon pressing the OK button.

All information regarding the make, type, and float of a flowmeter is edited here. Remember that exact spelling is required for the calibration curves to recognise the flowmeter. (The actual flow reading is not edited here, but in a special table accessed directly from the FlowmeterReadings menu point.)

Moreover, it is specified what gas mixture the flowmeter is supplied with. This is done by writing I # (where # is a number) for Input Mixtures, and M # for mixed Mixtures.

It is also specified which mixture the flowmeter feeds to, numbered simply from 1 to 16. Note that the box specifying the mixture and its total flow is placed after the first flowmeter only (for practical reasons) while it actually signifies all the flowmeters going into that mixture.

A flowmeter's input or output can be set to 0 (zero) to signify that it's input or output are not connected or the flowmeter is not in use or not installed. In this case, the flowmeter appears as a greyed empty rectangle.

Finally, you can here specify Fixed gas pressure units – normally simply humidification or drying units. For this, the gas species to be fixated, the method, and the temperature (when relevant) are specified. In addition, the use of the unit can be switched between Yes and No, and you specify whether it is placed before or after a flowmeter.

For humidification, let the gas be H₂O and the method be Pure or KBr, and set the temperature to that of the humidification step – equal to the room temperature if you have no temperature control. For drying, let the method be Dry.

Note that when you press OK to confirm the changes made to a flowmeter, the mixer scheme changes on the screen.

1.4.3 Edit the output cells

Start editing flowmeters and their configuration by Edit, OutputCells.

Select which mixture to feed the cell. If set to 0 (zero) the cell is considered not in use and is displayed as an empty rectangle.

Give the impurity/background levels of gas species you consider appropriate to add to the final gas mixture fed to the cell. The source of these may be typical leakages, permeable gas line hoses, permeable high temperature cell wall materials, desorption, or impurities in the input mixtures not specified other places. The gases species you can add impurity or background levels to are O₂, H₂O, H₂, and N₂. For single walled high temperature cells we suggest O₂ = 1E-5 (10 ppm) and H₂O = 3E-5 (30 ppm) as realistic settings (with H₂ and N₂ zero).

Select the cell's temperature and pressure if used for equilibrium calculations..

Press OK to confirm the changes.

1.4.4 Edit the calibration curves

GasMix can store up to 1000 flowmeter calibration curves.

Each curve can hold up to 20 data pairs (reading, flow).

All curves must have the same unit for readings. The default – and the unit used in the calibration curves supplied as standard with GasMix – is litres/hour (l/h). Litres here of refers to standard litres (1 atm, 25 °C).

Start an editing session from Edit, Calibration.

The data specifying the flowmeter and conditions for each calibration curve are entered in the left hand side table. Note that the texts entered must be accurately the same as those used for flowmeters are gas species. A misprint means that the program will not recognise the fit of the calibration curve to the flowmeter or gas. In this table, you can enter data for several curves, but you must press the OK button in order to store the changes in memory. If you go to another operation without pressing OK, the changes are lost.

The numerical data for calibration curves are entered in the right hand table. Go to the calibration curve you want to edit by the number selector and press OK.

The data **MUST** be entered in increasing order of Reading. You do not need to use all 20 rows, but the last rows not used must hold only 0,0.

In running the program, readings below the lowest or above the highest valid calibration point are rejected.

The first entry may have a reading of 0, but 0 is anyway always interpreted as zero flow. Any value larger than zero is then interpreted in the normal manner.

If the lowest reading is larger than 1 (e.g. 2) then 1 is given a special meaning, namely to represent the maximum flow. The use of this will be dealt with later on.

You may enter data manually one by one, or you can copy them via the clipboard for instance from Excel. In order to do this, first go to Excel and mark and Copy a section, e.g. the Readings column. Then go to GasMix and press the first cell, then mark all cell in the range you need while holding the Shift key down. Then Right-click inside this range and select Extract from Clipboard.

As you make the calibration curve, check that it looks as expected from the plot that is displayed based on the numbers you enter.

For each calibration curve filled with numbers you must press the OK button to store the data in memory, otherwise the entered numbers are lost. (This is a safety against unintended data entry in established curves.)

When you have edited one curve, press the OK button in order to store the changes in memory before you proceed to another curve or another operation.

When you have entered a curve and correctly identified it in the table to the left, it will be taken into use by the program automatically when it searches for the best curve for any gas species and flowmeter type.

Remember to save calibration curves to disk before leaving the program.

1.4.5 Enter or edit the flowmeter readings

The flowmeter readings are entered from FlowmeterReadings. Press OK to confirm.

1.4.6 Start the calculations

Select Calc start (the button or in the menu) to do all calculations throughout the mixer. The result is shown in a table where all gas species appear for all Output Cells. The composition of a mixture is not shown unless the mixture is associated with (routed to) an output cell.

When the calculations start, the program first calculates all mixtures at room temperature and feeds them to the appropriate cells. Then it starts to calculate equilibrium compositions for the mixture fed to each cell at the temperature specified for that cell.

If the mixtures display table is set to room temperature mixture, it shows and keeps showing the mixture as fed to the cell, and has blue header column and row to indicate that these are non-equilibrium mixtures.

If the mixtures display table is set to equilibrium (high temperature) mixture, it shows the mixtures in the present state of the equilibrium calculations, and keeps updating them as equilibrium calculations proceed. The table has yellowish colour header cells to indicate that these are or are approaching equilibrium (high temperature) gas composition.

As mixing and equilibrium calculations proceed, a small indicator box in the upper right corner of each output cell rectangle changes colour. Black means that data are not yet calculated, light blue means that a room temperature mixture is calculated and OK. Yellow means that equilibrium calculations have started but are not finished with the required accuracy yet. Green means that calculations are finished and that the displayed values should represent high temperature equilibrium.

1.4.7 Settings: Change the display of the numerical output

In the Settings menu you can change between linear and logarithmic display of the gas partial pressures of the mixtures; lin/log toggle. The background colour of the table changes to show you at a glance the format of the data displayed.

Moreover, you can select whether the display shows the room temperature mixture as fed to the output cell or the equilibrium composition. The equilibrium display will show all species well defined by their thermodynamics in the program and where the required chemical elements are all present in the mixture fed to the cell. Note that colours of the table change to show you at a glance the type of data displayed.

1.4.8 Information and warning messages

There is a field for information messages (in blue) and warning messages (in red) at the bottom of the screen.

1.4.9 GasMix window

You may drag and resize the GasMix window, and maximise/minimise it. In some cases, resizing is a way to refresh the GasMix display if parts are not showing.

If you cannot see all items on screen, it is possible that your screen has a too small resolution. This is mostly a problem with video projectors or small laptops or old computers.

1.5 GasMix technical reference

1.5.1 GasMix capabilities and definitions

The GasMix program comprises a number of components and connections between them, to be described below.

Those that are user editable are accessed through the Edit menu.

1.5.1.1 Gas species

A number of chemical species (atoms or molecules) are predefined in the program. They are identified by their chemical formulae: H₂, N₂, O₂, Ar, H₂O, CO, CO₂, etc. Thermodynamic parameters of formation are connected with these species. These definitions and parameters can not be edited by the user.

The list of defined species is currently:

Species	Disabled	Brooks R215AAA Glass and Ta calibration curves
H ₂		Yes
He		Yes
C		
N ₂		Yes
O ₂		Yes
Ar		Yes
O ₃	disabled	
H ₂ O		Yes
D ₂ O		Yes
CO		Yes
CO ₂		Yes
NH ₃		

1.5.1.2 Input Gases

Up to 16 input gases can be named and defined by the user, corresponding to the gases connected to the inputs of the mixer. Each input gas is actually a mixture of gas species, and the definition is simply a fractional content of the gas species.

The definitions of the input gases can be edited via Edit, Input Gases.

The definitions of the input gases can be stored in files of type *.inp via the File menu and submenus.

1.5.1.3 Flowmeters and mixer configuration

Up to 16 flowmeters can be defined by the user. They are supposedly rotameters and thus identified by their make, model, tube size, and float material, but the make, model, and size can also be used to identify any other type of flowmeter. The definitions of the flowmeter will enable the program to find a suitable calibration curve when the flow is to be calculated.

Each flowmeter can be configured so as to feed any of 16 Mixtures. Further, each flowmeter can be configured so as to be fed from any of the Input Gases or Mixtures.

Each flowmeter can be configured with a gas fixing stage before it or after it. A gas fixing stage fixes the partial pressure of one gas component. Usually, this is a stage for H₂O which wets or dries the gas to a certain level. Various ways of operation and calculation can be set, and the temperature of operation also enters.

All these definitions set up the overall connection and mixing scheme of the mixer. The connections are shown graphically on the program screen.

The definitions of the flowmeters, fixing stages, and connections of the mixer can be edited via Edit, Flowmeters.

The definitions of the flowmeters, fixing stages, and connections of the mixer can be stored in files of type *.mix via the File menu and submenus.

1.5.1.4 Calibration curves

Up to 1000 flowmeter calibration curves can be held by the program. They can be entered in any order. They are marked with the flowmeter name, tube size, float material, gas, individual flowmeter, temperature, and pressure that the calibration is valid for. Further, each curve contains up to 20 data points. The program selects the best curve available for any flow calculation.

The calibration curves can be entered or edited via Edit, Calibration.

The calibration curves can be stored in files of type *.cal via the File menu and submenus.

1.5.1.5 Output Cells

Up to 8 output cells may be defined, normally the measurement cells at high temperatures to which gas mixtures are routed and for which equilibria are calculated. The cell can be assigned a gas mixture, total pressure, and temperature. In addition, leakages of H₂, H₂O, and O₂ can be set that will be added to the Gas Mixture arriving.

The definitions of the output cells can be edited via Edit, Output Cells.

The definitions of the output cells can be stored in files of type *.cel via the File menu and submenus.

1.5.2 Parameter files

1.5.2.1 File definitions

The GasMix parameters are stored in four types of files according to the part of the mixer and parameters affected. The types have indicative extensions:

- *.inp Input gas definitions
- *.mix Mixer configuration
- *.cal Flowmeter calibration curves
- *.cel Output cells

1.5.2.2 File operations

The File menu allows the user to open or save files individually. They may then have individual names. Alternatively, they may all be opened or saved in one operation using one and the same name for all.

If one chooses the Open, Save, or Save As options, then a second level allows the selection of operation on one of the Input Gas, Flowmeters (Mixer), Calibration, or Output Cell parameter file. In this way, files with different names may be used for the different parameter sections.

If one chooses Open All, Save All, or Save All As, the operation will affect all four types and they will all address files by one and the same name (except the extension). In this way, entire sets of parameters may be opened or saved more easily.

1.5.3 Mixer calculations

1.5.3.1 Flow of a mixture of gas species through a flowmeter

When a mixture is passed through a flowmeter, the program calculates the flow of each gas species as if it was alone. Then its contribution to the total flow is obtained by multiplication by the partial pressure of the gas. By summation, the total flow is obtained. (There are other and more correct ways of doing this, and these may be implemented in future versions.)

Finally, each gas species gets a partial flow by multiplying the total flow by its partial pressure, and this flow is added to the total flow of the mixture.

It may be noted that a minor component of a gas in this way will be carried onwards by the mixture and would be carried onwards also in the calculations of its partial flow even if this species was not specifically calibrated for and therefore did not get a flow assigned to in the calculation of its contribution to the total flow. However, the way GasMix works, any gas will have a partial flow contribution calculated, using another gas' calibration curve if necessary.

1.5.3.2 Flow of a single gas species through a flowmeter

When the calibration curve is selected the flow of each gas species is calculated from the flowmeter reading and that curve by using a Lagrange interpolation routine.

Readings above the maximum calibration point are not accepted.

Readings below the minimum calibration point are not accepted, with the following exceptions:

The reading 0 (zero) always returns zero flow, even if the extrapolation of the curve gives another value or even if the calibration curve contains the reading 0 as the first entry and the flow associated with it is not zero.

If the minimum reading of the calibration curve is bigger than 1 (one), then the reading 1 is taken to mean the same as the maximum reading. The experienced user can use this for fast entry of readings in parts of the mixer where the actual flow does not matter (no mixing) and it is desirable to just tell the program that gas is running in plenty through the flowmeter.

1.5.3.3 Selection of a calibration curve

The conversion of a flowmeter into a flow requires a calibration curve. The program can hold a large number of calibration curves, and attempts to find the one best suited. At present the algorithm used is the following: The program looks for matching flowmeter make, flowmeter type, float type, gas species, and actual flowmeter. The one with the closest match is chosen. Normally, with standard curves, it will be easy to achieve the first four. To get the fifth it is assumed that you will have made individual curves for individual flowmeters of your mixer.

In future versions of the program it is planned to make the selection more advanced and safer, and to display and verify the selection. Moreover, more advanced manners of obtaining flows of mixtures instead of individual gas species may require different selection criteria.

1.5.3.4 Making mixtures

The partial flows from all flowmeters connected to a Mixture are calculated and added. When all are done, the program divides the individual partial flow of each species with the total flow to obtain the fractional content. If the total pressure is one, the fractional content will correspond to the partial pressure.

1.5.3.5 Fixation of a gas species

When a gas mixture passes a fixation stage, e.g. a wetting or drying stage, the new mixture is calculated according to the following principles:

Before the fixation stage, each gas i has a partial pressure $p_{i,b}$ and the sum of all these is $P_{tot,b}$. After the fixation stage, gas j has had its partial pressure changed from $p_{j,b}$ to $p_{j,a}$. This change may be positive (as in a wetting stage) or negative (as in a drying stage). All the other gases $i \neq j$ will have to share a corresponding decrease or increase in partial pressure, so as to arrive at a sum of the partial pressures $p_{i,a}$ of all gases i corresponding to the total pressure after the stage, $P_{tot,a}$.

If the total pressure is the same both before and after the fixation stage, the partial pressures after the fixation are

$$p_{j,a} \quad \text{and} \quad p_{i \neq j,a} = p_{i,b} \frac{P_{tot,a} - p_{j,a}}{P_{tot,b} - p_{j,b}}$$

If the total pressure is unity, as in the present version of GasMix, then this simplifies further into

$$p_{j,a} \quad \text{and} \quad p_{i \neq j,a} = p_{i,b} \frac{1 - p_{j,a}}{1 - p_{j,b}}$$

If the fixation stage is inserted before a flowmeter, the new composition is first set, and the flow then calculated using the composition with the calibration of the flowmeter to obtain the total and partial flows.

If the fixation stage is inserted after a flowmeter, the new post-fixation composition of the fixated gas and its new partial flow is calculated, and the new total flow and individual composition of each of the other species are calculated based on the increase or decrease in content of the fixated gas. The formulae used for the composition is the same as used above for pre-flowmeter fixation.

1.5.3.6 Calculations of fixed pressures for various gases and methods

The fixed pressure of water over pure water H₂O (method “Pure”) is calculated with the Goff-Gratch equation, with temperature as input.

The fixed pressure of water over a saturated solution of KBr(aq) (method “KBr”) is taken as 84 % of that calculated by the Goff-Gratch equation. This percentage is specified in the CRC Handbook for 20 °C.

The pressure of water over a drying or getter stage (method “Dry” or “Getter”) is set to zero.

The pressure over pure heavy water D₂O is calculated from the 5th order polynomial formula

$$p_{D_2O} \text{ (kPa)} = 0.413333333 + 0.046428671 * t + 0.000936772 * t^2 + 2.85781e-05 * t^3 + 2.5e-07 * t^4 + 2.82051e-09 * t^5$$

where t is the temperature in degrees Celsius. The curve is based on a fit using TableCurve 2D to values taken from the CRC Handbook¹.

The pressure over D₂O saturated with KBr is tentatively calculated as 84 % of that over pure D₂O.

The pressure of oxygen over a getter stage (method “Getter”) is set to zero.

The Method names are case insensitive.

1.5.4 Equilibrium calculations

1.5.4.1 Thermodynamic parameters

To appear here....

1.5.4.2 Numerical routine for equilibrium calculation

To appear here...

¹ *Handbook of Chemistry and Physics*, 73rd Edition, Lide, D.R. Ed.; CRC Press: Boca Raton 1992; Chapter 6, pg. 10

1.5.5 Definitions of arrays and indexing used in the program (mainly for us programmers)

1.5.5.1 Chemical Elements

The elements are numbered and named according to their atomic number and chemical formula. For instance, hydrogen is 1, H, helium is 2, He, etc.

Deuterium is given the number 122 and formula D, and tritium is given the number 123 and the formula T. No other isotopes are handled as such.

1.5.5.2 Compounds and gases

GasMix operates with predefined **compounds**, for which thermodynamic data are stored and their elemental compositions are defined. They are used in calculations of equilibria. They are indexed by an arbitrary number and identified by their chemical formulae. Any well-defined single phase can be what is here called a compound. Examples comprise monoatomic elemental gases (He, Ar), molecular elemental gases (H₂, O₂, O₃, N₂), gaseous compounds (CO, CO₂, H₂O), and condensed phases such as solid graphite (C).

GasMix furthermore operates with **gases**. These are gaseous species – in one sense the same as gaseous compounds. However, they need not in principle be defined thermodynamically – it is more important in the first instance that the flowmeters are well calibrated for them in order to obtain correct mixing ratios. If equilibria are required however, only gases corresponding to a defined compound (as above) can be properly split into the proper amount of defined elements. The remaining will enter as inert.

1.5.5.3 Non-equilibrated (room temperature) gas mixtures

GasMix stores gas mixtures in a number of arrays, depending on where in the mixing and calculation process they are:

The InputMixtures array hold all input mixtures – they can be opened from and saved to *.INP files. They can be edited by the user. They are copied into the Mixtures array when a calculation is initiated.

The Mixtures array is an array that holds input mixtures and mixed mixtures alike. It is rearranged in the course of mixing so that all mixtures have the same order of gases. The Mixtures feed the flowmeters and the output cells. The Mixtures array holds only room temperature mixtures, it does not contain equilibrated mixtures.

CompBuffer is a temporary buffer array for the gas species and is used during the calculations of partial flows and setting of fixed gas for each individual flowmeter.

The CellMixturesRT array is similar to the other mixtures, but holds the room temperature mixtures as fed to the output cells. It is copied from the Mixtures array after completion of all calculations in the mixer, and in addition gets the leakages of the gases specified for the individual cell.

1.5.5.4 Arrays used in equilibrium calculations

To appear here...