HOW TO HANDLE LOW LIQUID FLOWS

E-BOOK

Including insider tips and advice from our experts

www.bronkhorst.com



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SUMMARY

Why have we written this e-book?

What do micro reactors, catalyst research and odorant dosing have in common? Well, they all require the handling of low liquid flows. In the world of flow control & measurement, we distinguish between 'low flows' and 'high flows'. But what does this really mean? Bronkhorst High-Tech is a renowned supplier of flow meters and flow controllers in the 'low flow' range. So, time to explain what we mean when we refer to 'low liquid flow'.

This e-book is a summary over our blog series 'How to handle low liquid flow' supplemented with indepth information, technical advice and insider tips from our experts.

What's in it?

We have prepared this e-book with recommendations for low liquid flow setups, focusing on low liquid flows, flows < 100 g/h. Besides low flow definition and tips for flow meter selection, this e-book also gives advice on system lay-outs, connection material and liquid supply systems. Because flow setups and process conditions are rarely the same for different customers; there is no one-fix-for-all solution available. Providing the best advice requires insight into the customer application.

This E-book has been written in collaboration with several Bronkhorst colleagues from different departments; product management, training, field service engineering, calibration and R&D. They have all contributed useful tips and insights, in-depth information and advice to make this E-book a useful and valuable document which can be used as a reference work for you.

Enjoy reading

PART ONE

What are (ultra) low liquid flows?

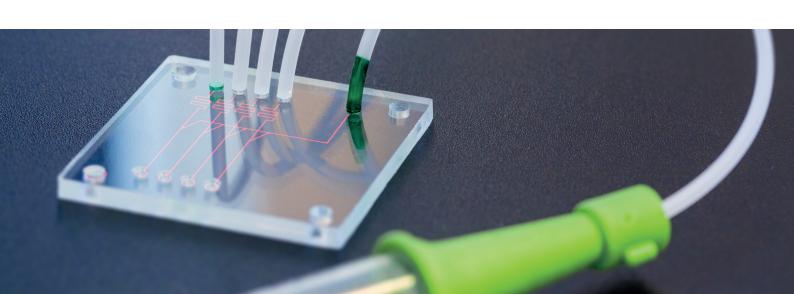
The definition of 'low' is arbitrary and depends on the area of business. In bulk industry, flows of much less than 500 kg/h are considered low flows, whereas in research this term is attributed to flows that are smaller than 100 g/h. This e-book focuses on handling - measuring as well as controlling - liquid flow rates up to 100 g/h and ultra low flows - which we define being in the range < 5 g/h.

To give you an idea, consider a water droplet. With a typical diameter of half a centimetre, 100 grams per hour is equivalent to about 2000 water droplets per hour - quite low. And 100 drops are an equivalent of 5 grams - to be dosed in an hour. Accurate instruments for measuring and controlling low liquid flows have proven their use in a wide array of applications. For example:

- The supply of 100 g/h of drilling oil as a lubricating agent is monitored during hole drilling in the manufacturing of aircraft fuselage parts.
- An ultra low liquid ethanol flow of 2 g/h is evaporated to generate a stable ethanol vapour flow as a carbon source in R&D for high-quality graphene production.

- In the investigation of catalysis at high pressure, low liquid flows of hydrocarbon compounds need to be dosed as a stable flow without pulsation.
- Labs-on-a-chip and other microfluidic devices in pharmaceutics and biotechnology significantly reduce the number of chemicals and experimental time compared to traditional means.
- The typical smell of natural gas and biogas originates from a 'warning agent' that has been added artificially to the gas, injected in a small but continuous amount as a liquid additive.

In all these cases, the measuring or dosing of the correct amount of liquid - not too much and not too little - is critical for the optimum performance of the process concerned.





Mass flow versus volume flow

In the previous paragraph, the flow is expressed in units of mass, such as grams/hour or milligrams/ second. However, many users think and work in units of volume. This is fine, at least when we are talking about the same reference conditions.

What is so typical about low flows?

How is a low liquid flow of less than 100 g/h different from 'normal' or 'high' flows? Well, (ultra) low flow applications involve some phenomena which are not observed in, or are not relevant to, larger flows. Due to the (very) small amount of liquid that is being moved, (ultra) low flows are so sensitive that even the tiniest disturbances in process or ambient conditions can have a massive effect on flow stability. The influence of external conditions on flow stability is therefore key here – as well as the means to control these external conditions.

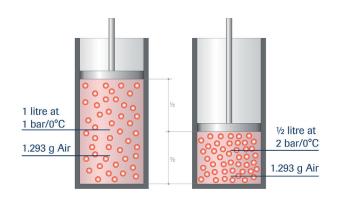
For example, even small leaks of gases or liquids into or out of the process have a considerable influence on the intended liquid flow. Furthermore, any obstruction caused by solid particles or contaminations in the small liquid flow lines will obviously reduce the flow in an undesired way. For low liquid flow dosing in particular, unstable pressures will lead to unstable flows. Variations in pre-pressure, pulsation due to excessive pump stroke volumes compared to the flow rate, and dissolution of gas (pressurised air) when pressurising the liquid to be dosed will all result

in an unstable flow. Knowledge of the application as well as the physical transport phenomena of the process are essential to deal with this complex matter of low flow handling. Optimising flow stability and performance of fluid systems requires in-depth knowledge of fluid characteristics and system components in a wide range of circumstances. Every component used in a fluid system can affect the behaviour of a fluid or interact with other components, especially when it comes to low flows.

Solutions for optimal performance

In the Bronkhorst range of products, thermal-based μ -FLOW and LIQUI-FLOW mass flow meters and controllers, as well as Coriolis-based mini CORI-FLOW ML120 and mini CORI-FLOW M12 device, are particularly suitable for (ultra) low liquid flow applications. Where a mass flow meter consists of a sensor that only measures the flow rate of the medium, a mass flow controller combines such a sensor with a control valve to control the medium flow rate. Check out the 'mass flow controller theory'.

Flow controllers are typically used to generate a stable flow. However, optimal performance requires a good deal more than just an excellent flow controller. For example, make sure that there are no leaks in the setup and use small volume tubing. Moreover, in pressurised containers, avoid using gas that dissolves in liquid, or use means to remove this gas. In part 2 of this e-book we will discuss this more into detail by focusing on practical tips on how to select the right low flow device.



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PART TWO

Tips for flow meter selection

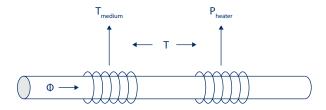
As explained in part 1 of this e-book, tiny disturbances in the process or ambient conditions can have a large impact on flow stability for (ultra) low liquid flows. In part 2 we share our tips and recommendations for dimensioning, choice of material and best practice procedures, based on years of experience, to help you optimise the stability and performance of your system.

The core of the low-flow fluid system is obviously a liquid flow meter or flow controller. Deciding which instrument type is best for the low-flow application largely depends on accuracy and stability requirements. However, ambient or media conditions which are difficult to control can also play an important role in choosing the best solution.

When to choose a thermal or Coriolis flow meter or controller

Within the Bronkhorst range of products, some thermal and Coriolis flow devices are particularly suitable for (ultra) low liquid flow applications. The different working principles for thermal and Coriolis devices make them specifically suitable for different applications, requirements and conditions.

In thermal-based mass flow devices, a constant temperature difference is created between two positions along a (capillary) tube. When liquid flows through this tube, the energy needed to maintain this temperature difference is proportional to the mass flow rate. Read more about the thermal-based mass flow measurement working principle.



In the Coriolis setup, the liquid flow through a tube with a small diameter causes this tube to twist, and the change in deflection is a direct measure of the mass flow through the tube. Moreover, the resulting change in vibration frequency of the (filled) tube is proportional to the medium density. Read more about the <u>Coriolis mass flow</u> measurement principle





Generally speaking, a Coriolis flow meter/controller

- performs very well in situations where absolute accuracy and flow stability are essential;
- has long-term stability and negligible thermal sensitivity;
- is a good choice when the medium density needs to be measured or monitored, in addition to the flow;
- can be used for mixtures of liquids with unknown properties (i.e. is media independent);
- is somewhat receptive to vibrations around the resonance frequency, which may necessitate the use of shock absorbing measures.

On the other hand, a thermal flow meter/controller ...

- is a more economical choice if the processed liquids and (ambient) temperatures are stable and distributed evenly;
- performs well when reproducibility is more essential than accuracy;
- requires specification of the liquid density,
 viscosity, thermal conductivity and heat capacity;
- generally causes a relatively small pressure drop, which can help to keep the flow stable if the liquid contains a considerable amount of dissolved gas.

Tips & tricks

Bearing in mind the above-mentioned information, please read and observe the following tips with respect to low liquid flows.

Tip 1 / Choose a flow meter or controller that is suited to the process and ambient conditions

The Bronkhorst website offers a product selector tool to help you choose the right low <u>liquid flow</u> meter or controller based on input parameters such as maximum (mass or volumetric) mass flow rate, operating pressure and operating temperature. Thermal-based <u>µ-FLOW</u> and <u>LIQUI-FLOW</u> devices can handle liquids up to 2 g/h resp. 0,25 to 5 g/h in the lowest range. Coriolis-based mini CORI-FLOW ML120 mass flow meters have 200 g/h as their maximum full scale flow rate but can be easily scaled down to a minimum full scale flow rate of 5 g/h, with the same relative accuracy. They also have a minimum flow rate of 50 mg/h.

Tip 2 / Provide a stable (inlet) pressure to the fluid system

A highly stable inlet pressure of a flow controller is a prerequisite for a stable low liquid flow rate. Two methods are popular to achieve this: use a pressure vessel where gas is used to pressurise the liquid or use a pump. Please refer to parts 3 and 4 of this e-book for more background details.

Tip 3 / In the case of a pressure vessel, minimise the containment or dissolution of gas in the liquid to be processed

Air or other gas bubbles dissolved in the liquid, or moving along the liquid flow, have a negative effect on the flow stability.





If gas is used to pressurise the liquid, prevent the gas from getting into direct contact with the liquid by using a membrane.

Alternatively, use a gas with low solubility such as helium or nitrogen to pressurise the liquid when the gas needs to be in direct contact with the liquid. Apply the lowest possible pressure to the liquid and keep pressure drops throughout the fluid path as small as possible. This obviously depends on the working pressure of the application. As a last resort, use a degasser to remove the gas from the liquid.

Read more about this in part 3 of this e-book 'Liquid supply using a pressure vessel'.

Tip 4 / Choose a piezo valve to accurately control low liquid flows

The quick response time, low internal volume and low heat generation of piezo valves are especially advantageous when using gas to pressurise the liquid. For an operating pressure higher than **5 bars**, solenoid valves are an alternative. Preferably install the meter part of the flow controller between the valve and the process.



In depth information Piezo valve or Solenoid Valve?

Piezo valve

When it comes to accurately controlling low flow rates, a piezo valve is the best choice. It has a quick response time and is very precise, two essential qualities for optimal controllability of the process.

- Compared to conventional Solenoid valves, the Piezo valve has almost no dead volume and no heat generation. Piezo valves are generally limited to an operating pressure of 5 bar and have a limited ingress protection (IP) rating.
- Another drawback of a piezo valve can be its higher cost.

Solenoid valve

The Solenoid valve has some features which need to be taken into account depending on your application.

- The control behavior of a Solenoid valve can deteriorate due to trapped gas bubbles in the internal (dead) volumes.
- A Solenoid valve generates heat that can expand the fluid, causing the fluid to exit the valve at a higher volume flow rate than entering it.
- A temperature rise can cause degassing of the liquid, destabilizing the flow. The construction of a Piezo valve comprises an intrinsically smaller internal volume, reducing the risk of gas entrapment.



Tip 5 / Keep the internal volume between the flow device and the process as small as possible

This is to help minimise filling times and to limit external disturbances. For this purpose:

- Make tubing in the fluid system as short as possible and choose small diameter tubing.
- Use hard tubing such as stainless steel rather than flexible tubing.
- Avoid 'dead volumes' in bends and valves where air bubbles can be trapped. The <u>µ-FLOW</u> and <u>mini CORI-FLOW L120</u> devices each contain a continuous capillary with a limited dead volume.

Read more in our blog 'Why is the choice of piping important for thermal mass flow meters?'

Tip 6 / De-aerate the system before operation

This is especially important for skids. First connect all instruments and then de-aerate before starting to control or measure. To this end, the Bronkhorst skids contain a purge setting.



Insider tip

How to deal with the pressure drop of the valves?

An inevitable side effect of using any type of control valve is the relatively high pressure drop. Although the absolute pressure drop in a Piezo valve is limited (due to its limited pressure rating), the lower pressure after the valve can still cause dissolved gas to decompress and destabilize the flow. On the other hand, in fluid system, a control valve should always have the highest

pressure drop of all components, otherwise its controlling function may be impaired. Take this into account when selecting components such as valves, filters and pipes in particular. Instead of using a control valve, a pump can be an alternative way to control the flow rate as well. Read about pump setups in part 4 of this e-book.

PART THREE

Liquid supply using a pressure vessel

As indicated in the previous part, a highly stable inlet pressure of a flow controller is necessary to obtain a stable low liquid flow rate. While a gas as a compressible medium can have a cushioning effect in levelling the pressure of a system, a liquid is not very resilient. Although Bronkhorst flow controllers are capable of neutralising pressure fluctuations to some extent, rapid changes in the inlet pressure can destabilise the flow.

In general, two methods are suitable for providing a stable inlet pressure to the liquid system: using a pressure vessel where gas is applied to pressurise the liquid or a pump. Whichever method you choose is partly a practical decision based on what infrastructure is available at the customer's site.

Pressure vessel

A pressure vessel is a relatively safe option without needing electricity or moving parts, and it is a plus for some volatile liquids. However, gas bubbles dissolved in the liquid have a negative effect on the flow stability.

Pump

The advantage of a pump is that in principle there is no direct contact between gas and the liquid. This means that no gas can dissolve and a pump can be operated continuously. On the other hand, their moving parts make pumps sensitive to wear and they are often more expensive than pressure vessels.

Frequently it is a balance between the volume of liquid to be dosed, the pressure and the application. In a research lab, for example, helium gas for pressurising purposes may be available, whilst in a production environment another solution needs to be found, usually pump controlled. Read part 4 for more details about using a pump to provide a stable inlet pressure.

How to deal with dissolved gas

When using a pressure vessel, the main challenge is to minimise the dissolution of gas in the liquid to be processed. The less gas that comes into contact with the liquid, the better it is. Henry's law states that the amount of dissolved gas in a liquid is proportional to the pressure of the gas that is in direct contact with the liquid. This has some practical implications.

Gas used to pressurise the liquid and that has been dissolved in the liquid at high pressure will be released as gas bubbles at a lower pressure further downstream in the process. This is usually an unwanted phenomenon. Moreover, the solubility of a gas in a liquid decreases as the temperature gets higher. A temperature rise of the processed liquid will therefore also result in released gas bubbles.

In this respect, it is recommended that the pressure and temperature drop over the liquid path is kept as small as possible.



Tip 1 / Use a pressure vessel

If gas is used to pressurise the liquid, an important recommendation is therefore to prevent the gas from coming into direct contact with the liquid. For example, use a pressure vessel with a membrane that physically separates the liquid from the pressurising gas - in a similar way as an expansion tank is used in your central heating system at home.



Expert advice

What size of liquid vessel do you need?

The liquid vessel should be large enough to provide a stable flow for a sufficient amount of time between refills. Purging or flushing the fluid system can consume a relatively large amount of liquid; take this into account when selecting a suitable vessel size. The table below gives an indication of liquid consumption, based on different flow rates

Flow rate		Liquid consumption	
g/h	mg/min	g/workweek 40 hours	g/week 24 x 7 hours
0.1	1.6	4	16.8
1	16	40	168
10	160	400	1680
100	1600	4000	16800

Insider tip: Filling and draining of a vessel

A transparent tube alongside the liquid container can provide a visual indication of the liquid level inside. Note, however, that plastic tubing limits the maximum operating pressure of the total application. A vessel made out of one piece, usually has openings on both ends (inlet/outlet). The inlet also serves as an air exhaust when filling the vessel. For safety reasons, using a filling funnel on the inlet of the vessel is strongly recommended to prevent splashing and liquid spilling as a result of gas bubbles coming out of the inlet.

Adding a draining facility at the outlet of the liquid vessel offers the option for purging and draining. Note that, due to the small diameter of the inlet and outlet, it might be necessary to open the inlet of the vessel to allow draining.





Tip 2 / Apply gases with low solubility

If it is necessary or inevitable for the pressurising gas to come into direct contact with the liquid, there are some solutions. For example: apply gases with a low solubility. Helium is usually the best choice for water-based liquids, followed by nitrogen. If possible, apply the lowest possible pressure to the liquid. This obviously depends on the working pressure of the application. In this respect, it may help to position the liquid vessel substantially higher than the flow controller - to let gravity do its work and thus require a lower gas pressure.

Tip 3 / Use a degasser

As a last resort, use a degasser to remove the gas from the liquid. This is a device with a porous hose to which a vacuum is applied on the outside, causing gas bubbles to be drawn out of the liquid inside the hose.

Standard degassers go well with water-like media. For low liquid flow rates below 50 g/h and depending on the type of application, we recommend using a degasser if stability and maximum accuracy are key and if gas bubble disturbances need to be deleted. A degasser is considered an expensive option, but if you have a high-end application, you look for the best available solution. Metaphorically speaking, you don't buy 25 euro tyres for a very expensive racing car, or do you?

Tip 4 / Pressure relief or buffer tank

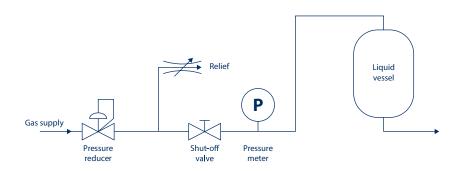
If the inlet pressure fluctuations in the pressure vessel setup are too fast for the flow controller to be able to compensate, there are some solutions.

A pressure relief or a buffer tank between the pressure reducer and the liquid vessel can smooth out these fluctuations to provide a stable inlet pressure.



Insider tipInstalling a pressure relief

In order to maintain a stable downstream liquid flow, the inlet pressure to the liquid vessel should be stable for a longer period of time. This can be achieved by installing a pressure reducer in the gas supply line of the liquid vessel. A pressure relief after the pressure reducer helps to control the gas pressure.





Tip 5 / How to deal with corrosion issues of welded parts

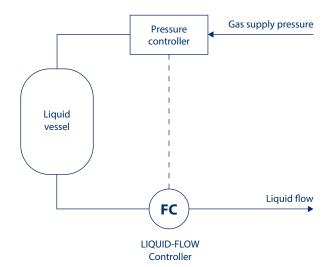
For use with water-based media, Bronkhorst recommends having a vessel made out of one piece of passivated steel to deal with corrosion issues of especially welded parts. With organic solvents like methanol, toluene or acetone, corrosion of welded parts in a vessel is not usually a problem. Because liquid flow control with a pressure vessel is usually a batch process, make sure that the vessel is large enough to provide a stable flow for a sufficient amount of time between refills. To prevent splashing and liquid spills when filling, use a filling funnel at the inlet.





Extra tip

Minimise gas pressure by indirect feedback The gas pressure for pressure vessels is commonly set at a fixed value. This value is determined or calculated for the application and is usually set higher to compensate for pressure loss due to liquid consumption. An indirect feedback setup gives you the possibility to minimise the gas pressure and control the flow accordingly. This can be done by adding a pressure controller. This example shows a liquid flow controller providing a control signal to a pressure control unit (pressure controller). The pressure needed to control 1 g/h water is about 2 mbar above outlet pressure (based on a μ -flow sensor). The pressure controller must be able to control low pressures from 1-100 mbar above atmospheric pressure.



Using an indirect feedback setup, the dissolved air in water is up to 1500x less compared with a pressurised tank with 4 bar(a). The pressure controller immediately adjusts the inlet pressure to achieve the required flow.

Bronkhorst <u>pressure instruments</u> can be used in various configurations to support this method of liquid supply.





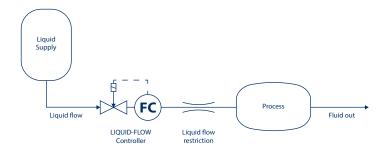
Extra tip

Reduce the impact of dissolved gas bubbles

Degassers are very effective at removing gas from liquids but are not always an option based on the liquid or application. Dissolved gas in the liquid is a potential risk, it can be released in the system unexpectedly and have an undesired impact. The impact of the gas release (bubbles), can be reduced by maintaining a constant pressure in the flow path.

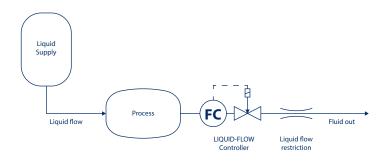
Example 1

Use a liquid flow restriction after the liquid flow controller to maintain a stable system pressure. It prevents the gas from escaping inside the flow controller and influencing the measurement and control signal. The potential impact of gas release (bubbles) in the process, or even further downstream, can be acceptable or even negligible. In some applications the process itself can act as a restriction.



Example 2

Maintain system pressure in the process and in the liquid flow controller. This prevents gas escaping in the process and inside the flow sensor. In this case the sensor is placed downstream of the process, which is a common method in certain industries. While this cannot be applied to all flow setups, understanding the principle might help with the design of future systems.







In-depth information (1/4)

Henry's Law, the theory of gas solution in liquids For a gas i that is soluble in a liquid, the mole fraction of dissolved gas in the liquid, $y_{i,liquid \, side}$ is related to the partial pressure of the gas $P_{i,gas \, side}$ by Henry's law:

$$y_{i,liquid \, side} \; = \frac{p_{i,gas \, side}}{H}$$

Where **H** is Henry's constant. The Henry constant increases with increasing temperature. For 300K some typical values are shown in the table below.

Gases	Н
Air	7.4e10 ⁻⁴
Не	1.5e10 ⁵
N ₂	8.9e10 ⁴
H_2	7.2e10 ⁴
CO ₂	1.7e10 ³
O_2	4.5e10 4

Gas solubility can be illustrated by an example of a tank filled with water that is pressurised by air. At the air water interface, the air is saturated with water vapour. Both the air and vapour are ideal gases. The vapour pressure of water at 300K is 35mbar which is only a small part of the air at atmospheric pressure. It can be neglected for the overall picture.

The mole fraction of air dissolved in water below the water surface at atmospheric pressure is 1/7.4e10⁴ - 1.35e10⁻⁵. The molar mass of air is 28.97 g/mole. The molar mass of water is 18.02 g/mole. Therefore the mass fraction of air in water pressurized with 1 bara is 2.17e-5. With a known water density of 1kg/l the water in the tank at 1 bara contains 0.022g of air per liter of water.

And with known density of air at standard conditions of 1.2 g/l that is 0.018 l/l at 1 bara or 1.8 vol%/bar. This means that water exposed to air at atmospheric pressure contains the equivalent of 1.8 vol% of dissolved air.

According to Henry's law the amount of dissolved gas is proportional to the pressure. So at higher pressure more gas will dissolve in the water. When the pressure is relieved the dissolved gas cannot stay in the liquid and the gas will appear as bubbles. This is for instance the case when first opening a bottle of sparkling mineral water. For microfluidic systems with a high demand for stability this effect is unwanted.

According to the Henrys constants the solubility of helium is less than air and the solubility of carbon dioxide is more than air.

Bubbles in a microfluidic system

When controlling a flow using a setup as shown in figure A at 2 bard air pressure, the volume of air gassed out of the water at the end of the process is 2x1.8% = 3.6 vol%. The assumption is that the water is saturated with the pressurized air at 2 bard and all of the pressure drop is over the control valve. This will result in a theoretical volume expansion due to the air bubbles of 3.6% after the valve due to the decompression. This expansion of air will results in a mass flow increase after the valve in between the emerged air bubbles of 3.6%.

Because of mass flow in equals mass flow out, the average massflow including the airbubles has to the same. It is to be expected that reducing the pressure or using helium as a propellant gas reduces the amount of gas bubbles.





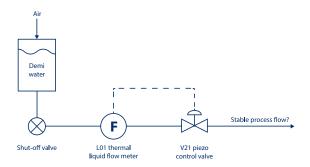
In-depth information (2/4)

Henry's Law, the theory of gas solution in liquids

Picture A

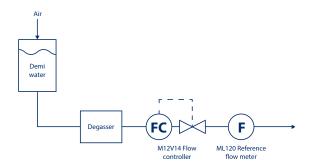
A basic setup, from left to right: A pressurized storage vessel with shutoff valve, L01 thermal liquid flow meter, V21 piezo actuated control valve. All components are interconnected with 1/16" tefzel tubing.

A degasser can be used to reduce the effects of dissolved gasses in liquids. A degasser consists of a gas permeable tube that is positioned in a vacuum chamber. As the liquid with dissolved gasses runs through the degasser the dissolved gasses are extracted from the liquid and this should result in a stable flow further downstream. Degassers are commercially available devices.



Picture B

Experimental setup for investigating the effect of air bubbles and degasser. From left to right: pressurized storage vessel with water, degasser, M12V14 flow controller and ML120 reference flowmeter.



Accuracy specifications of the used instruments as stated by the manufacturer

A measurement is done to monitor the process flow stability of the basic setup of Picture B with an ML120 flow sensor is placed after the M12V14 flow controller. Initially thew degasser is switched of so it leaves the dissolved air in the water. The M12 is set to control a flowrate of 1g/h. Subsequently the ML120 flowmeter measures the stability of the generated flow. After a few hours the degasser is switched on to reduce the amount of dissolved air.



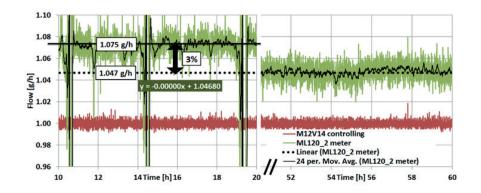


In-depth information (3/4)

Henry's Law, the theory of gas solution in liquids

Measure results

Chart 1 is cut into two sections. The first section on the left side shows the performance with the degasser switched off. The right section shows the system performance with the degasser switched on. Since it takes quite some time for the system to become stable, only the stable recordings for a few hours are shown for both cases.



(Chart: results of measurement with degasser off (L) and degasser on (R))
In the chart above the red line represents the measured flow of the M12 flow controller running continuously at 1 g/h for 60 hours. The green line represents the measured flow of the reference flowmeter ML120 positioned downstream after the control valve of the M12.

Clearly visible in the left section is a stable flow of the M12 as it controls while the ML120 sees a discontinuous flow caused by air bubbles. Also a final offset of 45 mg/h can be seen between the two instruments which can be caused by incorrect zeroing of the instruments or a calibration deviation. The deviation is within the combined specification limits of the flow meters as shown in the table below.

Property	M12V14	ML120
Accuracy [%RD]	0.2 %	0.2 %
Zero stability [mg/h]	100	10

Noticeable on the right side of the chart, where the degasser is active, a lower flow rate can be seen. The flow seems less when gas bubbles are not present. It results in a drop of the mass flow of approximately 3%.

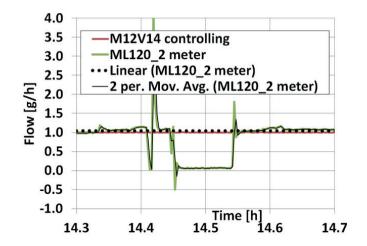
The dotted line in the left and right side of the chart is the calculated average value over the entire dataset of 60 hours. This shows that in both cases the mass flow is the same although it "seems" to be higher on the left side of the chart in between the air bubbles.





In-depth information (4/4)

Henry's Law, the theory of gas solution in liquids A zoom in on an air bubble passing the reference flowmeter reveals the following as depicted in chart 2. The airbubble takes about 0.1 hour to pass the system while the flow seems stable in between two air bubbles. The time in between two air bubbles is approximately 3.5 hours according to the left side of chart 1. The percentage of time where an air bubble is present equals 0.1/3.5 = 3% During this time the mass flow is nearly zero as the air bubbles passes at the same speed as the liquid while the density is 0.1% of the density of water.



Conclusion

Dissolved gasses in liquid will be released when pressure is reduced. This occurs in microfluidic systems when liquid reservoirs are gas pressurized and the flow is controlled by valves that cause the pressure tot drop. Gas bubbles cause instability and deviations in the mass flow.

The conclusion that can be drawn from the experiment is that in the case of air bubbles being present in a system initially pressurized at 2 bara, the mass flow rate is 3% higher for most of the time while in 3% of the time the mass flow rate is nearly no units. This results in the same average mass flow as if the gas is still dissolved. This effect seems very little but is very measurable with sensitive flow meters.

Although the average mass flow is the same at stable conditions it seems to be higher in between originated gas bubbles. Since flow in our example is controlled by an accurate flowmeter, it is to be expected that its delivered flow is accurate. However, in the presence of dissolved gas and pressure drop, the mass flow in between gas bubbles can be a few percent higher after the flow controller. The measured amount of deviation of 3% roughly matches the calculated 3.6%. A degasser reduces the disturbance and deviations otherwise caused by air bubbles.

PART FOUR

Liquid supply using a pump

To ensure a flow controller has a stable (ultra-) low liquid flow rate, a highly stable inlet pressure is a prerequisite. As you read in Part 3 of this e-book, the presence of dissolved gas in the liquid is the main issue to overcome when using a pressure vessel to generate a stable low liquid flow. If prevention of gas dissolution in the liquid is essential, applying a pump to generate the inlet pressure will be a good choice.

Why do we use a pump?

A pump is a device that can provide a **continuous**, **reliable** and **stable pressure or flow**. It will not introduce dissolved gas into the liquid flow because the mechanical action of the pump itself pressurises the liquid. If the pump draws liquid from a separate, non-pressurised vessel, this vessel can be refilled at any time without interrupting the process. It is recommended that such a vessel is positioned at the same level as the pump to prevent vacuum suction with the risk of gas bubbles. This is at the expense of an extra shut-off valve to prevent the vessel from spontaneously emptying into the pump. Make sure that this valve is mounted so that no extra dead volume is introduced in which air can accumulate.

Which pump type is recommended?

A displacement-type pump - either a gear pump or a piston pump - can be used to provide the necessary inlet pressure for the flow controller. In a gear pump, a fixed liquid volume in between interlocking teeth of rotating gears is

repeatedly displaced to generate a flow, whereas in a piston pump a liquid flow is generated at subsequent strokes of a piston filling and emptying a fixed volume. Overall, small pumps are favourable, as a pump with a small internal volume reduces the fill and refresh time of the system. Whichever pump type has been chosen, always make sure that the wetted pump materials are compatible with the processed liquid. Because the relevant displaced fixed liquid volume in gear pumps is generally smaller than in piston pumps, gear pumps are the preferred choice if a customer wants to have a reasonably stable flow control for low liquid flows. However, **gear pumps** are limited to a maximum operational pressure of 10 to 15 bars. Piston pumps can handle higher pressures, from several tens to more than 100 bars - which are frequently occurring process conditions in lowflow applications.

The dual-piston pumps we use at Bronkhorst are shifted 180° in phase, resulting in a very stable pressure/flow delivery which is important in the low-flow range. However, whether or not this is sufficiently accurate depends on the customer's application process. The required amount of liquid is accurately dosed, but due to the pumping principle it will be dosed as small pulses. When the process volume is sufficiently large, there will be enough mixing capacity to smoothen the pulses. However, if you want an evenly distributed dosing over a short period of seconds, such a piston pump setup is less ideal and a gear pump may still be the preferred choice.



Using mass flow meters with a pump to achieve control loops

Bronkhorst mass flow meters, particularly Coriolis-based ones, are ideal for working with pumps to achieve a control loop to establish a stable low flow rate. Another method of creating a stable low flow rate is to use a pressure control setup. Here, the pump speed is controlled by means of a (EL-PRESS) pressure controller to provide a constant and stable inlet pressure. At the same time, flow adjustment is performed with a control valve which is operated by a flow meter like the Bronkhorst mini CORI-FLOW ML120

For many low-flow applications, the control loop setup does not have to be as sophisticated as the combined flow & pressure control setup above. The most straightforward way to use a pump for

generating a liquid flow is to let it draw the liquid from a vessel and to steer the pump speed with the control signal of the flow meter. In practice, such a 'direct control' setup has been applied for the stable dosing of low liquid flows of hydrocarbon compounds without pulsation for high pressure catalysis R&D.

For smooth control behaviour and a stable flow throughout the flow range, a **bypass line** can be incorporated into the direct control setup with an adjustable restriction - typically a needle valve. Using the bypass method, the speed of the pump will be increased, resulting in a more stable flow/pressure control, allowing the pump to run at a more efficient and stable setpoint for a smoother liquid flow output.



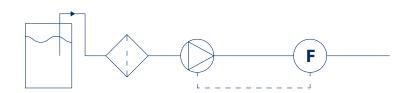
Expert advice

Possible pump setups

Direct control setup

The most straightforward way to use a pump for generating liquid flow is to use a direct control setup. In this setup the pump is directly controlled by the flow meter (with integrated PID controller). The supported flow range of the pump should be comparable to the measuring range of the flow meter.

- A filter is placed in front of the pump to protect it against particles.
- Bronkhorst flow meters offer firmware to program a response alarm preventing dry running of the pump.
- This setup can have a limited turndown ratio (20:1) as most pumps are more unstable in the lower flow ranges.

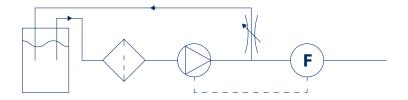




Bypass line setup

Using a bypass line behind the pump module has several benefits in low liquid flow applications. In some cases it might even be necessary. At very low flows, the pump might have to operate at the lower part of its speed range, which can result in unstable control behavior. Adding a bypass circuit allows the pump to run at a more efficient speed, while the amount of dosed liquid can still be very small. In many setups a bypass can help to run the pump at the most efficient speed. This setup also allows for a large turndown ratio, meaning the setup can be used at the higher flow range of the Liquid Flow Controller with the bypass closed, and at the lower range of the LFC with the bypass opened.

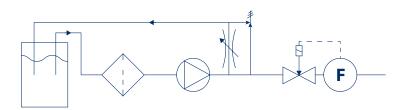
Blog How to deal with vibration using Coriolis mass flow meters



Fixed pump speed setup

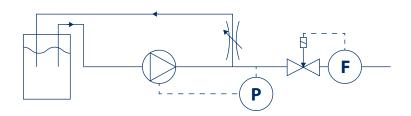
Another method is to run the pump at a fixed (optimum) speed, creating a continuous and stable flow of liquid at a constant pressure. The flow runs back into the storage tank via the bypass. A Liquid Flow Controller with a valve uses the constant supply flow to control the liquid into the process. Additional components are added to extend the lifetime of the system, a pressure relief valve to protect against any unexpected pressure spikes, and an extra particle filter because of multiple reasons, mentioned on the right.

- The pump is usually able to withstand larger particles than the control valve (especially if this is a piezo valve).
- Due to the larger surface area and pore size, the first filter has a substantially higher flow capacity than what the flow meter requires, which is necessary to keep up with the higher region of the pump's over dimensioned flow range.
- The second filter protects the control valve not only against particles that pass the first filter, but also against particles generated by the pump.



Pressure control setup

A final optimisation can be achieved by having the pump speed controlled by means of a pressure controller, to provide a stable inlet pressure to the further process.



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PART FIVE

How to deal with external conditions?

Bronkhorst flow meters mentioned in the previous parts of this e-book, that are capable of measuring (ultra) low flows, are very sensitive. This implies that even the tiniest disruptions in a customer's process or ambient conditions can be detected. So any disturbances that may have been present in the process now emerge, because the measurement accuracy is much better than before this sensitive flow meter was applied.

A customer might then respond with "there is something wrong with the meter". However, don't shoot the messenger! Instead, use the information in this e-book to optimise your own process. Check external aspects such as the tubing to and from the flow meter, the influence of surrounding vibrating equipment or the presence of solid particles in the liquid flow.

To put the previous consideration in a practical context: when choosing a Coriolis-based mass flow meter or flow controller, a relatively high pre-pressure will be necessary to overcome the relatively high pressure drop over the device. This is mainly the case when Coriolis instruments are operating in their nominal flow range. However, because Coriolis instruments have a large turndown ratio down to 1%, the pressure drop in the lower region is usually negligible and comparable with thermal.

Although the measurement conducted with such a Coriolis flow meter is much more accurate than with a thermal-based flow meter, a high prepressure from a pressure vessel will allow more gas to be dissolved in the liquid. This is released as gas bubbles downstream in the process at a lower pressure, resulting in instability. This e-book is therefore intended to create awareness of all the things you can do to improve the setup of your process, each with their own pros and cons and possible effects.

What tubing should I choose?

Choose the smallest possible tubing. By minimising the length and diameter of the liquid supply tubing between the flow meter and the process, the fill and refresh times will be as short as possible. The pressure drop over Coriolis-based mass flow meters is much larger than over thermal-based flow meters, because the capillary in the latter is about 20 times shorter and its diameter is larger. Find an optimum between the pressure drop and the smallest possible internal volume of the tubing. For low flows **up to 100 g/h**, tubing with an outer diameter of 1/16 inch (~ 1.6 mm) is recommended. For **higher** flow rates, we recommend **1/8 inch** (~ 3.2 mm) tubing to limit the pressure drop. Try to use as few connectors, bends or T-parts as possible, because they can cause air accumulation which results in flow instability. If necessary: use smallvolume connectors.



Expert adviceRecommended tube sizes

Piping with a smaller inside diameter (ID) involves a higher pressure drop, introducing a greater risk of gas dissolution. An optimum has to be found between the pressure drop and the smallest possible internal volume of the piping. In general: Low flows <100 g/h/OD: 1/16'' and Higher flows >100 g/h/OD: 1/8'' (OD = outside diameter)

Flow rate	Recommended tube size OD x ID	Pressure drop across 10 cm / 100 mm	Liquid speed (estimated)
0.1	1/16" x 0.01"	< 0.3 mbar	0.5 mm/s
	1/16" x 0.02"	negligible	0.1 mm/s
1	1/16" x 0.01"	< 3.0 mbar	5.5 mm/s
	1/16" x 0.02"	< 0.3 mbar	1.4 mm/s
2	1/16" x 0.01"	< 6.0 mbar	11 mm/s
	1/16" x 0.02"	< 0.5 mbar	2.7 mm/s
	1/8" x 0.0069"	negligible	0.2 mm/s
10	1/16" x 0.02"	< 0.2 mbar	13.7 mm/s
	1/8" x 0.069"	negligible	1.2 mm/s
100	1/16" x 0.02"	< 20 mbar	137 mm/s
	1/8" x 0.069"	< 0.2 mbar	11.5 mm/s

(Recommended tube sizes and expected pressure drops (calculated with water))

The choice for hard tubing like stainless steel or flexible tubing is mainly based on the operational pressure. In a production environment at high pressures, flexible hoses are rarely used. For flow rates **up to 2 g/h**, the use of hard tubing is strongly recommended because it prevents internal volume changes. For flow meters with Hastelloy capillaries, tubing made of Hastelloy is recommended. PEEK, PolyEther Ether Ketone, is preferably applied for aggressive liquids that attack stainless steel.

12.10

12.08

12.06

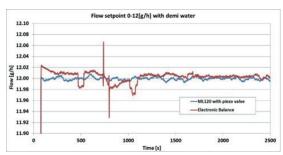
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Example Difference between result in setup using a 1/4 inch tube vs 1/16 inch tube



11.90 0 500 1000

Flow setpoint 0-12[g/h] with de

Figure A: 1/4 inch tube

Figure B: 1/16 inch tube

Use materials with a low expansion coefficient. When these materials are used the setup is less sensitive for ambient changes, such as temperature and pressure fluctuations. A larger tubing diameter makes the flow more susceptible to temperature gradients on the way, due to expansion of the tube and more volume inside the tube. Figure A and B give an indication of the flow stability in a tube section that is heated to 40 °C. The flow rate into the tube section is controlled by a Coriolis instrument (mini CORI-FLOW ML120) - blue line -, the outgoing flow rate is measured with an electronic scale - red line - Figure B with the 1/16 inch tubing clearly shows the increased stability.

Check our blog <u>Why is the choice of piping important for thermal mass flow meters</u> to learn more about the choice of piping.



Prevent water hammer by avoiding sudden changes in tubing diameters

A phenomenon that needs special attention is water hammer. Also familiar from your toilet or dishwasher at home, this is a hydraulic shock that occurs when a liquid in motion is suddenly forced to stop, start or change direction. This will result in pressure surges that are much higher than (static) pressure values for which a system has usually been dimensioned.

Prevent water hammer by avoiding sudden changes from large to small tubing diameters, by applying a small pulsation damper (where an isolated gas column has a cushioning effect), by gradually increasing an applied pressure or by avoiding the pump running against a closed valve.

How to deal with vibrations?

Vibrations from a pump or other surrounding equipment can negatively impact the performance of Coriolis-based mass flow meters. The working principle of Coriolis instruments is based on vibrations, so make sure that pumps and other surroundings machines are vibrating at a different frequency than the frequency of the Coriolis instrument.

To prevent these external vibrations from reaching the Coriolis flow meter, you can use (slightly flexible) PEEK tubing, or the flow meter/controller can be mechanically uncoupled by applying a curl in the hard tubing ('pigtail tubing'). For the Coriolis-based instruments, Bronkhorst has 2kg and 4 kg massblocks available with vibration dampeners, an extra buffer to absorb vibrations.

Blog <u>How to deal with vibrations using Coriolis</u> mass flow meters



What about Calibration?

We recommend calibrating thermal-based flow meters such as <u>u-FLOW</u> and <u>LIQUI-FLOW</u> devices once a year. For Coriolis devices like the <u>mini CORI-FLOW ML120</u>, no calibration is required because their measurement principle is less vulnerable to ageing. However, in sectors like automotive, pharmacy and food, calibration is required by legislation or by standardisation. In these industries, it is vitally important that a measuring device indicates the correct value. For calibration purposes, it may be useful to apply transparent flexible tubing such as Teflon, so that any gas bubbles in the liquid flow can be detected visually.

Use particle filters to avoid clogging

To prevent the small diameter tubing and capillaries of the flow meters from clogging, or to prevent damage to piezo control valves, it is recommended that one or more particle filters are incorporated. This becomes more important when channels and control valves are used for the lowest flow rates. The filter pore size should be at least ten times smaller than the smallest capillary, orifice or control gap in the system, and upstream of a piezo control valve the recommended pore size should be **5 microns**. A large filter surface area can compensate for a high pressure drop caused by a small pore size.





Insider tip Installing particle filter

In the low liquid flow range, the presence of even the smallest particles can increase the risk of clogging, dramatically resulting in damaging components. To provide stability in the low flow range, it is important that the liquid is absolutely free of impurities. However, even when you use liquid with the highest purity, it is strongly recommended to use particle filters. When selecting a suitable filter, observe the following recommendations:

Pore size

- At least 10x finer than the smallest capillary, orifice or control gap in the system
- Recommended pore size is 5 µm (for Piezo valves)
- If the processed liquid contains many particles, multiple filter stages with decreasing pore sizes might be an option

Surface area

• A high-pressure drop should be avoided, it can cause dissolved gas to expand

- The surface area of the filter should ensure a low pressure drop, even when starting up or purging the system
- A large surface area can compensate for a higher pressure drop caused by a small pore size
- A large surface area yields less clogging and therefore less frequent maintenance

Tips

- Mount the filter 'up-side down' (outlet up (if possible)), so gas bubbles can escape during purging
- Clean or replace clogged filters; make sure to reassemble it in the same direction as it was
- Make sure to install the filter in front of every component that is sensitive to clogging or damaging
- Filters will not filter dissolved elements like minerals (e.g. dissolved lime or sodium chloride) and chemical stabilizers

Thank you for reading

f you need any advice



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