

# Chromatography

## The Importance of Understanding Pump Flow

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Pumps are one of the most common tools enabling liquids to be moved, delivered, pushed through a separation column, supplied as a dose or transferred from one container to another. Several different pumping technologies are available to choose from, depending on your application or problem. As you might expect - a pump designed to pump crude oil through a pipeline has very different requirements compared to a pump designed to deliver a constant stream of medication into a patient's vein. As such, the term 'flow' can be interpreted very differently, depending on the context.

This article aims to shed some light onto flow of liquid supplied by pumps most used in a laboratory for analytical purposes such as chromatography, and for lab-level flow chemistry applications.

### The Context

Most modern analytical labs are today equipped with Liquid Chromatography (HPLC, UHPLC, GPC, SEC, Ion-Chromatography) systems. The flexibility of this technology makes it applicable for an almost endless number of analytical problems. Independently of the particular setup or application, all liquid chromatography systems share the necessity of pushing the carrier solvent (eluent) through the separation column for an undefined period of time with the same flow rate (defined as volume of Eluent / minute) with high accuracy, low fluctuations and, in most cases, overcoming the resistance given by all connected components including the column, which becomes what we generally define backpressure of a system. This pressure can easily exceed 1200 bar (c. 17,000 PSI), representing therefore a huge challenge for what we would like to be a 'constant' flow.

Today, reciprocating piston pumps (see *Figure 1*) have become the gold standard for delivering liquids in liquid chromatography systems. This type of pump is proven to be reliable and accurate, though is sensitive to problems associated with dissolved gases and air bubbles in particular. Reciprocating piston pumps are also widely used when dosage of chemicals in a high-pressure system is needed, for example to produce nanoparticles.

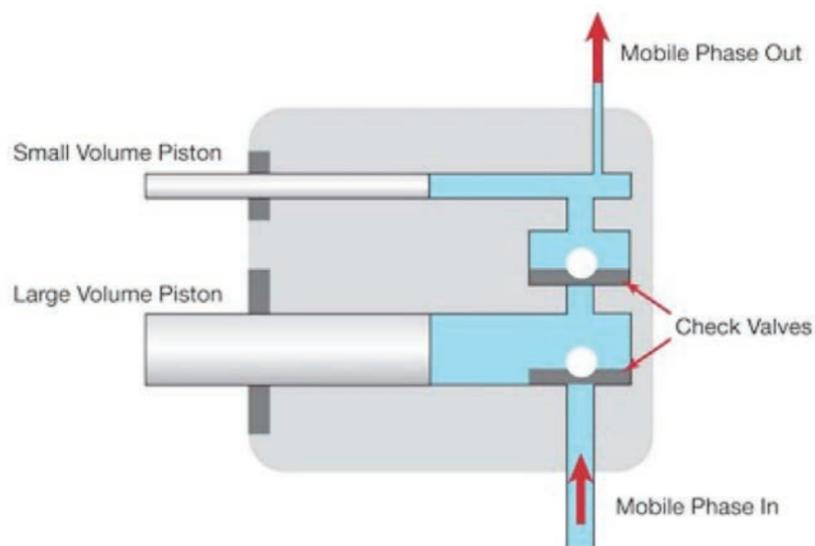
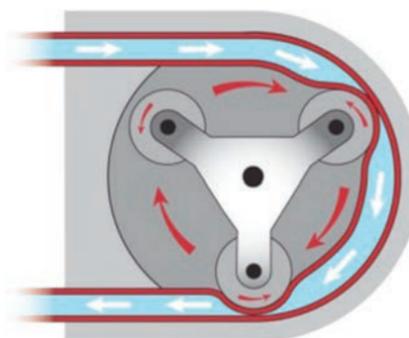


Figure 1: Schematic of a reciprocating piston pump.

A second type of pump, commonly used in laboratories for flow chemistry, are peristaltic pumps (see *Figure 2*). The principle of these pumps is extremely simple, a wheel with two or more runners rotates having a flexible tube fixed on the perimeter of the wheel. The runners therefore squeeze the tubing during rotation creating a moving closure which in turn forces the liquid to move in the direction of rotation.



Peristaltic pumps are widely acknowledged to be very easy to use and maintain and are relatively inexpensive. While they cannot achieve high pressure, they can achieve higher flow rates than reciprocating piston pumps.

### Flow, Flow Rate and Mysteries

Figure 2: Schematic of a peristaltic pump.

Whatever technology the pump in use is based upon, you would trust that the delivered flow was constant, and this is where troubles begin. When flow is constant, this simplifies a series of further considerations, like quantities delivered within any given time, fluctuation of pressure and an expectation of reproducible elution times. As such, a pump delivering a constant flow simplifies our lab life considerably. Now, the trouble of 'constant flow' is its intrinsic relation to a timeframe. If we were to measure the volume delivered by a pump over a period of 1 hour, and we repeat this experiment finding exactly the very same volume after each hour, then we could indeed define the flow to be constant. Moreover, within a given experiment in which 1 hour is a small timeframe compared to the total length of the experiment itself, this definition of constant flow may very well be accurate and correct. This investigation, however, will tell us nothing about the properties of the flow within any one minute of that hour. More accurately, we should therefore examine whether the flow rate is constant, therefore we should examine the amount of liquid delivered within a set time.

Let us consider a 15-minute long HPLC application run at 1 ml/min, here 1 minute is the defined time period. In this experiment the flow rate should be considered constant only if within 1 minute the fluctuation is zero or extremely small. To prove this, real-time measurement of the flow is necessary, sufficiently fast to supply a relevant number of measurement points within 1 minute so to allow statistical evaluation of the obtained data. The frequency of data collection will have an impact on the obtained flow rate, average flow rate and extreme values. It must be therefore selected carefully in regard to the time period and set flow rate of the application.

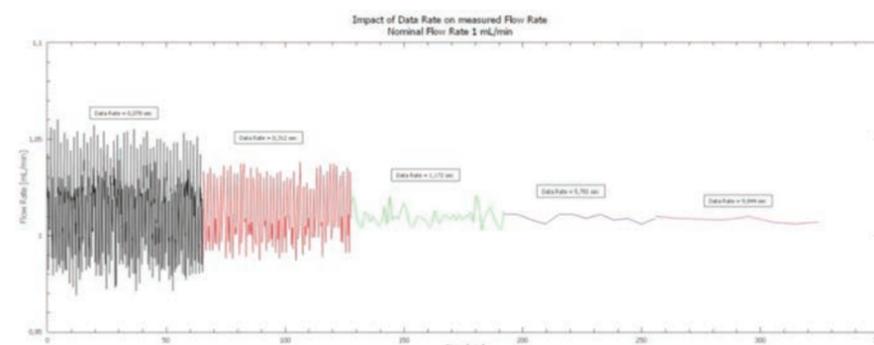


Figure 3: The impact on HPLC flow data acquired at different frequencies.

However, if we consider the flow rate from a peristaltic pump the situation is different. In most cases, the absolute flow rate is secondary. The relation of flow rates from different peristaltic pumps used simultaneously in the same system is by far more important. The fluctuation of the flow delivered by a peristaltic pump is by the nature of the technological mechanism very high. Therefore a representative average must be selected and put into comparison with averages delivered by the other pumps present in the system.

Similarly, to the above HPLC application example, the calculated flow average for a peristaltic pump will be affected by the frequency of data collection of the flow measurement. This makes 'real time monitoring' of flow values even more important as it might directly impact any pump flow rate adjustment required to optimise the process the peristaltic pump is servicing.

## Gas or Air bubbles - a common enemy

Just to make things slightly more realistic, we should also evaluate the impact of air or gas bubbles in combination with pumping of a liquid. The amount of gas that a liquid can absorb depends on factors including pressure and temperature gradients, the nature and type of liquid and the gas. Unfortunately, gas bubbles are produced in a liquid when there is more available gas than the liquid can absorb. We must however differentiate the effects created by dissolved gas and bubbles on the pump technologies described above, as the effects are very different.

Dissolved gas has an impact on reciprocating piston pumps as it influences (increases) the compressibility of the eluent resulting in higher fluctuation (pulsations) of the flow. This causes elution times reproducibility to worsen and increased baseline instability for the connected detectors.

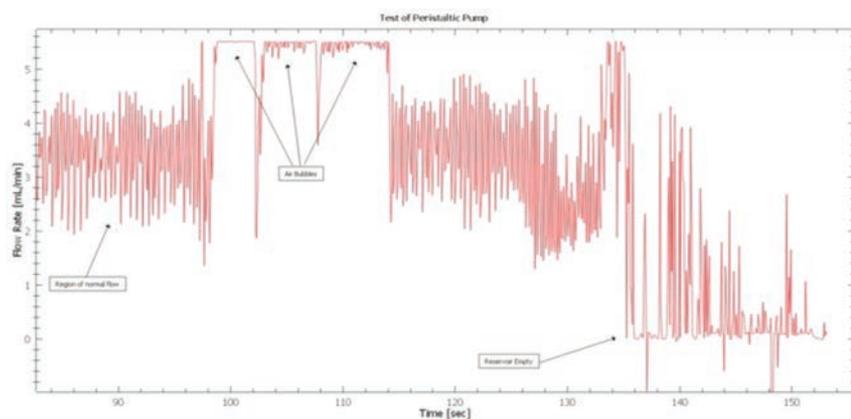


Figure 4: The impact of air bubbles and an empty solvent reservoir on peristaltic pump flow rate.

A proven remedy for the described problem is liquid stream degassers placed on the inlet line of the pump. A degasser will effectively reduce the amount of dissolved gas present in the eluent thus preventing most of the problems arising from dissolved gas.

Air or gas bubbles, however, are an even bigger problem for reciprocating piston pumps, as they may cause a system to suddenly stop operating. For instance, if a bubble is sufficiently large, it might cause a pump to stop delivering solvent altogether.

Peristaltic pumps are less sensitive to dissolved gas compared to reciprocating piston pumps, therefore extraction of dissolved gas is usually not necessary, though I would recommend it. While bubbles rarely lead to a stop in liquid delivery by a peristaltic pump, they are a well-recognised source of large fluctuations and apparent spikes in flow rate. A peristaltic pump can therefore be considered even more sensitive to bubbles, as it will produce erroneous results without any indication of a problem with the pumping system.

The only remedy to air or gas bubbles, no matter what pump is used, is constant real-time monitoring of the status of the tubing connecting the pump to the solvent reservoir, and a strategic handling of alarm conditions detected by such a monitor.

## Conclusions

Although the terms Flow and Flow Rates are commonly understood to define the volume streamed within a time, they do require more specific interpretation relating to the pump technology and application being undertaken. Moreover, flow accuracy and precision are not easily defined in real world applications, as the determination of these parameters is easily affected by parameters of the measurement itself.

Real time monitoring of flow using suitable non-invasive flowmeters, however, offers a powerful tool and a safe harbour for all those applications in which flow, and flow rate, are a critical parameter for achieving the set aim. Combined with such flowmeters, vacuum degassers, and powerful bubble detectors, are the way forward to create a reliable fluidic path, reducing operational downtime and increasing the reliability and productivity of any liquid chromatography or flow chemistry system.

## Author details

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