



HOW TO MANAGE VAPORIZATION IN AN ANALYTICAL SYSTEM

By Dean Slejko and Tony Waters

If the analyzer in your analytical system requires gas but your sample is liquid, the only option is to convert the liquid to gas. This process is called vaporization or flash vaporization. The objective is to convert a sample of all liquid to all vapor instantly – *without changing the composition*.

It is not easy to vaporize a sample, nor is it always possible, so make sure it's really necessary and possible before you try. You should always analyze a liquid in a liquid phase unless there are strong reasons for analyzing in a vapor phase.

If you proceed with vaporization, it's important to understand the difference between evaporation and vaporization. Evaporation occurs gradually with an increase in temperature. Vaporization occurs instantly with a drop in pressure. It's not possible to vaporize a sample by increasing temperature. Heat causes evaporation, and adding more heat simply makes evaporation happen faster.

In a mixed sample, evaporation will allow some compounds to evaporate before others, resulting in fractionation. Vaporization,

done properly, ensures that all of the compounds vaporize at the same time, preserving the sample's composition.

However, it is possible for things to go wrong when vaporizing. Instead of flashing the whole sample into a vapor, you could unintentionally cause a combination of vaporization and evaporation. The result would be fractionation. Once a sample of mixed compounds fractionates, it is no longer suitable for analysis. With fractionation, a common scenario is for lighter molecules to evaporate first and travel on toward the analyzer, while the heavier molecules remain behind in the liquid phase. Even if at some later point in the process a fractionated sample appears to be all gas, the mixture will not be of the same molecular proportions as it was before fractionation. It will no longer accurately represent the product taken from the process line.

Let's take a closer look at the process of vaporization and how we can manipulate the variables – temperature, pressure, and flow – to ensure proper vaporization and an accurate analytical result.

Understanding Vaporization

To vaporize a sample, one typically uses a vaporizing regulator, also called a vaporizer, which is a pressure-reducing regulator with the capacity to transfer heat to the sample at just the right location.

Vaporization consists of a three-stage process (Figure 1). First, the sample enters the vaporizer as a liquid. At this point, the liquid should not be bubbling or boiling.

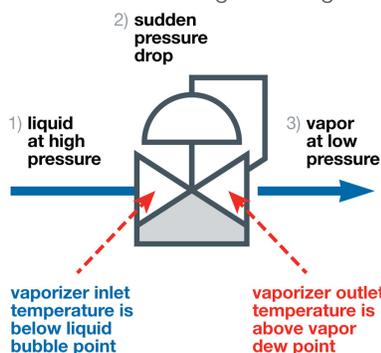


Figure 1. The three-stage vaporization process.

Second, the liquid passes through the regulating orifice in the vaporizer, resulting in a severe and sudden pressure drop, which vaporizes the liquid. At the same time, heat is applied, which enables the vaporized liquid to remain a vapor.

Third, the sample, now a gas, exits the vaporizer and travels to the analyzer to be read. Due to the immediate transition to the vapor phase, the composition of the gas is unchanged from that of the liquid, ensuring an accurate reading.

In this delicate process, there are many variables or inputs that determine success or failure. For the purpose of this discussion, let's say there are two main sets of inputs.

The first set of inputs concerns the composition of the sample. Depending on the composition of the sample, it will begin to bubble and finish vaporizing at different pressures and temperatures. We'll need to know what these pressures and temperatures are to successfully manage the process.



The second set of inputs concerns settings that you control in your sampling system: pressure, temperature, and flow. Pressure and temperature are controlled at the vaporizer, while flow is controlled downstream at a rotameter (variable area flowmeter) and needle valve. We set these inputs based on what we know

about the first set of inputs. Proper vaporization requires a delicate balance of all inputs.

Even when approaching vaporization in a systematic manner like this, the process does require some trial and error, so we'll also talk about how to diagnose and address problems.

Understanding Your Sample

The best way to understand the first set of inputs is with a phase diagram. A phase diagram plots pressure and temperature, showing at any pair of conditions whether a substance will be vapor, liquid, or solid. The lines indicate the interfaces between two phases.

Phase diagrams for most pure gases are available on the Internet, for example at <http://encyclopedia.airliquide.com>. But diagrams for gas mixtures are very difficult to create without commercial software.

Figure 2 represents a phase diagram for 20 percent hexane in pentane. When the sample is above the bubble point (blue line), it's all liquid. We want the sample to be all liquid when it enters the vaporizer. When the mixture is below the dew point (gold line), it's all vapor. The sample must be all vapor when it leaves the vaporizer.

Between the bubble point and dew point lines is what we call the no-go zone. This zone is the boiling range of the sample. Here, the mixture is in two phases, part liquid and part vapor. Once a sample falls into the no-go zone, it is fractionated and no longer suitable for analysis. The objective in vaporization is to set the temperature, flow, and pressure so that the sample skips instantly from the liquid side of the no-go zone to the vapor side of the no-go zone.

With pure and nearly pure samples, there is little to no boiling range or no-go zone. The bubble point and dew point lines are on top of each other or nearly so. Indeed, pure and nearly pure samples will convert to vapor of the same composition, whether through evaporation or vaporization. Some industrial samples approach this level of purity and convert easily.

On the other hand, some samples have such a wide boiling range or no-go zone that they cannot be successfully vaporized. There is no way to skip from the liquid side of the no-go zone to the vapor side of the no-go zone. We are unable to manipulate the variables – temperature, flow, and pressure – in such a way as to avoid fractionation.

Most samples fall between these two extremes. For example, in Figure 2, the band between bubble point and dew point is narrow enough that with the proper settings we can enable the sample to effectively skip from the liquid side of the no-go zone to the vapor side. At the same time, the band in Figure 2 is wide enough that we cannot afford to be careless. Indeed, we will need to be skillful in our manipulation of the variables or we will end up with a sample in the no-go zone.

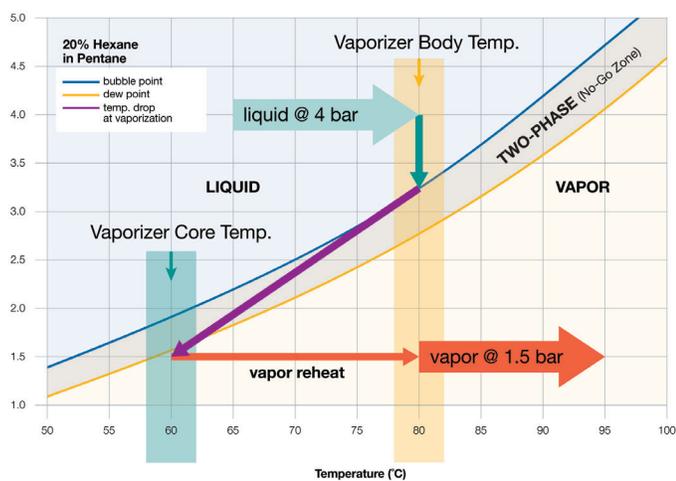


Figure 2. Phase diagram showing 20 percent hexane in pentane, with temperature settings



Setting Temperature, Pressure, and Flow

Let's continue to work with the sample in Figure 2 (20 percent hexane in pentane) and see how we can set our inputs to ensure successful vaporization.

In general, at the inlet, we want high pressure and low temperature. At the outlet, we want high temperature and low pressure. But there are limits as to how high and low these parameters can be, and not all of them are under our control completely. Vaporization is basically a balancing act between the variables.

Here is a four-step process for setting your inputs.

First, determine the inlet pressure at your vaporizer. This pressure, which is fixed, is your process pressure, provided your vaporizer is located close to your sample tap. In Figure 2, that pressure is 4 bar. Higher pressure is better because it allows you to keep the vaporizer temperature higher without boiling the incoming liquid.

Second, let's set your inlet temperature, or the temperature of your vaporizer. There are two objectives. First, the temperature must be low enough that when the sample enters the vaporizer it is entirely a liquid and isn't bubbling. In Figure 2, the bubble point at 4 bar is 88°C, but we want to build in a cushion, so let's choose 80°C, a round number far enough away from 88°C to be safe.

The second objective is that the temperature must be high enough to contribute to the complete flashing of the sample, ensuring that only vapor leaves the vaporizer. When you vaporize the sample, the temperature drops, in accordance with the laws of energy conservation. The sample temperature must be high enough at the outset so that after the pressure drop, the sample is not in the boiling range or no-go zone. In Figure 2, the vapor temperature after the pressure drop is 60°C, just on the vapor side of the dew point line

Third, let's set the outlet pressure at the vaporizer. Your objective is to drop the pressure below the gold dew point line. In Figure 2, the outlet pressure is set to 1.5 bar. If the outlet pressure were any higher in this example, the sample would not vaporize entirely. It would fractionate.

Fourth, set your flow. Flow is set downstream at a valve and rotameter, not at the vaporizer. In a sampling system, high vapor flow is desirable because it moves the sample to the analyzer faster. However, high flow can be problematic, too, because with high flow, more heat is required to vaporize the sample. In other words, high flow results in a greater drop in temperature at the time of vaporization. In Figure 2, the purple line illustrates the temperature drop. As flow increases, the purple line angles more sharply to the left.

Another variable influencing the temperature drop is the heat transfer capability of the vaporizer. Some vaporizers are constructed in such a way that heat transfers more efficiently to the sample. When the liquid sample converts to a vapor and its temperature drops, it draws heat from the stainless steel surrounding it. The critical question is how efficiently can the vaporizer replace that heat and keep it flowing to the sample. The more heat the sample can draw, the less its temperature drops during vaporization.

In some instances, it is possible for the vaporizer to be hot to the touch on the outside but cold at the core inside. That's because the vaporized sample is drawing lots of heat and the vaporizer cannot transfer enough heat to keep up. The best solution is to reduce the flow.

In sum, the angle of the purple line in Figure 2 is a product of the flow rate and the heat transfer capability of the vaporizer. With a good vaporizer and low flow, the line will become more vertical. Unfortunately, there is no easy way to calculate the location of the purple line, and it is not generated by any known software program. As a result, vaporization involves some approximation. As a rule of thumb, keep the flow rate as low as possible without causing an unacceptable delay in the sample's travel time to the analyzer. It's better to start with a low flow rate and experiment with increasing it than to start with a higher flow rate.



Troubleshooting

Phase diagrams will enable you to approximate temperature, pressure, and flow settings, but some troubleshooting will still be required. One sure indication of a problem is poor repeatability in analyzer results.

There are two possibilities when the sample is fractionating instead of vaporizing, with Problem #1 being the more common:

Problem #1: Only part of the sample is being vaporized. Liquid is passing through the vaporizer and sitting in the tubing on the downstream side. Eventually, it evaporates. When it does, it draws heat from the surrounding tubing, making the tubing cold to the touch or causing frost or ice to form.

Indicators: Vaporizer outlet and downstream tubing is cold to the touch or has frost or ice on it.

(Note that in many cases, liquid on the downstream side of the vaporizer may pass beyond the area of the vaporizer and into other components, such as flowmeters and filters, where it can cause considerable damage.)

Solution: In the approach above, your best option would be to reduce the flow rate. Another option would be to lower the vaporizer outlet pressure, if that is possible. A third option would be to increase the heat to the vaporizer, but in this case you risk causing Problem #2 (see below).

Problem #2: The sample is boiling at the inlet to the vaporizer. It is fractionating before it can be vaporized. Lighter molecules evaporate and produce a large cloud of vapor molecules. Some of these lighter molecules move on to the analyzer, giving an inaccurate analysis. The remainder create a “vapor wall,” which pushes the liquid back into the process. A portion of that vapor wall then cools and condenses. Finally, the liquid sample moves again toward the vaporizer, where the lighter molecules evaporate, starting the cycle all over again. Eventually, the heavier molecules reach the vaporizer and move on toward the analyzer giving an entirely different analysis. This cyclical fractionation process causes the measurement values to oscillate.

Indicators: The inlet tube to the vaporizer twitches, sometimes violently, and the measurement values oscillate with the light component cycles out of phase with the heavy ones.

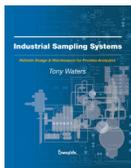
Solution: Lower the vaporizer temperature.

Conclusion

Vaporizing a liquid sample is challenging. In many sampling systems around the world, vaporizers are fractionating samples and sending unrepresentative samples to the analyzer every minute of every day. You can dramatically increase your chances of success by researching a phase diagram of your system’s particular mixture of compounds. You can further increase

your chances of success by understanding what is occurring in the process – specifically, by knowing what the variables are (temperature, pressure, and flow) and their role in influencing the process outcome. With this framework in place, you can reach the right settings, making adjustments in accordance with the signs and symptoms you observe.

Resource



For more information, see the Swagelok book *Industrial Sampling Systems* (2013), the definitive sampling systems reference guide by expert Tony Waters.



Author Biographies

Dean Slejko is product manager for analytical instrumentation for Swagelok Company, focusing on advancing the company's solutions and services in sample handling system applications. Prior to joining Swagelok, Dean held market and product management positions involving solutions for process control, power management, and utility metering applications. Dean earned a bachelor of science in environmental engineering and a master's degree in business administration from Case Western Reserve University and is a licensed professional engineer in the state of Ohio.

Tony Waters has 45 years' experience with process analyzers and their sampling systems. He has worked in engineering and marketing roles for an analyzer manufacturer, an end-user, and a systems integrator. He founded three companies to provide specialized analyzer services to the process industries and is an expert in the application of process analyzers in refineries and chemical plants. Waters is particularly well known for process analyzer training courses that he has presented in many of the countries of Asia, Europe, and the Middle East, as well as North and South America. His presentations are always popular and have equal appeal to engineers and maintenance technicians.