Increasing Response Speed in Trace Moisture Measurements

Twelve process parameters affecting moisture measurement response speed, and ways to handle them, are examined.

Many process parameters have an impact on the speed of response for moisture measurement. Some you can't control; others, you can. The parameters listed below affect all moisture analyzers because they are oriented to the process and system rather than the sensor:

- Magnitude and direction of a step change—vapor pressure range
- System leakage
- Flow rate across the sensor
- Volume in the sample system—fast loop
- Materials of construction (both sample system and process piping)
- Piping surface area and finish—condition and contamination
- Carrier gas molecular weight relative to water
- Sample temperature
- Sample pressure
- Phase change with pressure reduction

Sensor-related issues include:

- Sensor technology
- Unit of measure—apparent speed of response

These parameters are listed in the order in which they are typically addressed when you are seeking to improve the speed of response of any moisture measurement.

STEP CHANGE MAGNITUDE AND DIRECTION

Vapor Pressure Range. The percentage of the step change to the overall range of a sensor often determines how fast the sensor is perceived to respond. This can be compared to overcoming momentum in a vehicle, where accelerating from a dead stop to the car's top speed could take many miles and several minutes. Going from 35 mph to 45 mph, however, might take only a few seconds. The direction of the change is also significant. For a given rate of change, stopping a car usually takes less time than accelerating it.

The vapor pressure range or starting point is important, too. As in the example of the car, the beginning velocity is a factor in determining how soon the final velocity will be achieved. Operating in the middle of the sensor range should produce faster response.

Consider for Your Application. If the process is normally very dry, with a dew point range of -70°C and below, and any upscale moisture reading is important, the sensor may wet up slowly. This is due to operating at the extreme low end of the sensor's range. When setting high alarms, use a tight setting, just over the allowable limit.

If the process is normally dry, in the -60°C to -25°C dew point range, and any upscale moisture reading is important, the wet-up of the sensor will be evident more quickly. When you set high alarms, you can allow a little more buffer over normal moisture levels without being concerned about a lag in response.

If the process is normally operated in the -25°C to 0°C dew point range, and any upscale moisture reading is important, the sensor will wet up very fast. Since this is often the high end of the range for trace sensors, set high alarms close to the allowable limit. This will allow the greatest amount of warning to help you avert sensor damage, as well as to correct the process more quickly.

For the occasional process operated in the -20°C to 20°C dew point range, where downscale moisture reading, indicating a loss of critical moisture, is important, sensor dry-down will be quickly evident. These processes are often moisture critical. When setting low alarms, keep them tight to overcome any process lag while adding moisture to the system.

SYSTEM LEAKAGE AND CORRECTIVES

A leaking process and/or sample system will not only slow response, but also offset the final reading so that the predicted moisture value is never reached. There is little to say about leaks other than that they happen even in the best of system designs.

To improve response speed, a tight system is a must. A leak is a two-way street. Leaks allow the process fluid out *and* the surrounding atmosphere into the process piping or vessel—even at high pressure (see Figure 1). We can't crawl inside the piping to test the ingress of the atmosphere, but we can often see evidence of a leak at a loose fitting or valve stem. It seems unnatural to imagine that air is getting into a pipe through a loose fitting when you hear all that hissing indicating a high flow velocity of stuff coming out—but it is.'

Selecting the components that will prevent all reasonable leakage is essential. In normal processes, connections with pipe threads and compression fittings are capable of maintaining a relatively tight system. For high-purity systems in the high-tech process applications, special fittings and techniques must be used to create a tight system.

There is a simple test for leakage ahead of a moisture sensor: simply change the flow through the sample system or the fast loop.

If an *increase* in flow produces a *decrease* in the moisture reading, there is probably a leak ahead of the measurement point. The moisture reading decrease is a result of the leak's being diluted by the increased dry sample flow. To verify the presence of a leak, *decrease* the flow. If the moisture indication *increases*, a leak is almost a certainty.

Consider for Your Application. If the process is *very* dry, in the -70°C and below dew point range, leakage will be crucial to the process as well as to the moisture reading. Use helium leak tests or other precautions to ensure a very tight system.

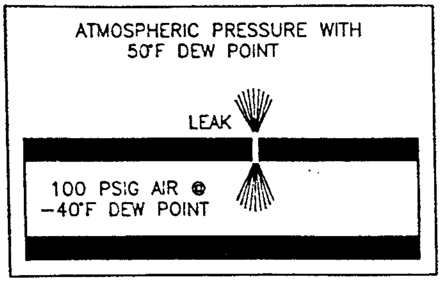

Figure 1. *Pipe leaks work both ways—air escapes and moisture enters. In this example, an 8:1 pressure ratio pushes the air out, and a 20:1 vapor pressure ratio pushes moisture in.*

If the process is normally dry, in the -70°C to -25°C dew point range, leakage will cause process problems and offset the moisture readings. A soap solution is usually sufficient to find the leak in pressurized systems.

In processes operated in the -25°C to 20°C dew point range, leaks are generally just a nuisance since ambient dew points are in the same range. They are also more difficult to find by changing the flow. This is because of the small differential between the moisture in the system and the atmosphere.

FLOW RATE ACROSS THE SENSOR

Increased flow will aid in the response speed of any moisture measurement, except when the C_v, or pressure restriction of the system immediately downstream, significantly increases the pressure at the sensor.

The real issue here is the number of exchanges the sensor will "see" over time. The more sample exchanges over time, the faster the response speed.

Caution: Always avoid flow rates that may damage the sensor. For example, drastically reduce the flow to very low rates when particles or aerosols are entrained in the sample directed across the sensor.

Consider for Your Application. Keep in mind that high flow rates may be wasteful or even dangerous for personnel working nearby. Often, an excessively high flow is simply unnecessary for an acceptable speed of response.

VOLUME IN THE SAMPLE SYSTEM/FAST LOOP

The total volume of the process and sample system and flow through the system are related to the speed of response. They both affect the exchanges as above, but volume also affects the sensor location within the system. When you establish the sensor's location relative to the process, you should minimize the volume contained in the tubing that transports the sample to the sensor.

Caution: Small tubing assemblies are often more prone to leaks if the tube is flexed at a fitting, and tubing $< 1/8$ in. O.D. should not be used when the stream contains dust or aerosols.

A fixed volume, e.g., a vessel or chamber, poses other moisture measurement problems. Here, the sensor location should be as close to the center of the chamber as possible and mounted where there is a natural flow across the sensor. Avoid locations near walls and other obstructions that could have a dead space or low flow around the sensor.

Consider for Your Application. If the sensor requires only a small volume of flow, and the moisture measurement is critical to the process, a fast-loop design will aid in the rapid transport of fresh samples to the sensor. A fast loop is often called a high bypass system. Some sample systems must draw off the sample through many feet of tubing. A high bypass system provides an increase in the sample flow from the process, usually through larger tubing, and a small portion of this flow is drawn across the sensor for the measurement—kind of a sample of a sample. This method is very popular for analyzing liquid samples.

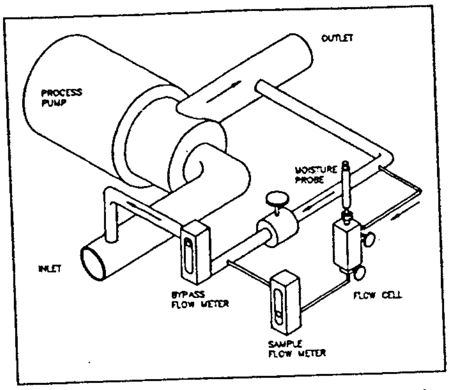

Figure 2. *All of the previous sample must be purged from a sample system before a new moisture value can be measured. One method of accomplishing this procedure is a high bypass sample system, an example of which is shown here.*

In order to measure a new or changing value of moisture, all the old sample must be purged. Every component in the sample path from the process line to the sensor must be counted in computing the volume for the exchange rate. This includes the high bypass piping. The components of the sensor downstream will have a negligible, if any, effect on the moisture measurement.

A high bypass sample system (see Figure 2) is often a $3/8$ in. or $1/2$ in. tube connected to the process both above and below a pump. The sample is drawn from this tubing using $1/4$ in. or smaller tubing. The bypassed portion of the sample is returned to the suction side of the pump, and the sample exhaust from the sensor can often be routed there as well. For the added piping to be of value, the flow through the bypass should be a minimum of 10 × that of the sample loop routed to the sensor element.

The distance from the sample point to the sensor should be minimized; the return distance from the sensor to the process is not critical to the measurement.

When measuring moisture in a cham-

Table 1: Tubing Materials

Highly recommended for trace (<–40°C dew point) measurement	Acceptable (but not recommended) for mid-range (–40°C to 0°C dew point) measurement only	Not recommended for moisture measurement
Electropolished stainless steel	PTFE Teflon	Nylon
Stainless steel	Polyethylene	Polyurethane —Nalgene
Nickel alloy	Copper	PVC
PEEK	Galvanized or other steel	Tygon
Glass	Aluminum	Neoprene or rubber

For the fastest speed of response when measuring moisture, choose a material as high on the list as economically possible.

ber, e.g., a glove box, locate the sensor or sensor inlet to avoid processes that might affect or splash on the sensor fixture. If possible, suspend the sensor or inlet in the middle third of the volume. If there is a recirculation loop available outside the chamber, the sensor should be attached to it. An unmounted sensor on a cable or flexible sample inlet tubing can allow enough movement within a chamber to check specific areas.

MATERIALS OF CONSTRUCTION

Most decisions regarding component materials are made during the engineering of the process. They are often driven by comparative cost and compatibility. These decisions rarely involve the question, "How will this material affect our ability to track the moisture in the process?" With complex engineering processes, the effort is directed toward making the process work on schedule. In this environment, where moisture measurement often becomes an afterthought, the analyzer system must be able to make the best of an existing system. Obviously, moisture measurement should be considered during the initial system design, but when it isn't, at least the proper wetted materials for the sample system can be chosen.

The moisture molecule is polar and tends to stick to any surface it touches. Proper materials used for tanks, piping, valves, and tubing can contribute to response speed or moisture sensors. Certain wetted materials commonly found in processes and in some sample systems are so porous that they delay moisture equilibration for days. A different choice of materials could produce response times in minutes with the same mechanical system design and chemical compatibility.

Porosity and permeability are critical properties of construction materials. Materials such as rubber and some plastics are porous enough to allow moisture to permeate the tubing into the process or sample. Electropolished stainless steel is best in meeting the comparative cost and compatibility criteria while also optimizing the response speed of moisture analysis. In cases where stainless steel is not compatible with the stream, most materials of construction of moisture sensors would not survive either.

Consider for Your Application. Table 1 lists tubing materials for process or sample system plumbing in the order of their appropriate use for measuring moisture, not for their compatibility with a process.

SURFACE AREA AND FINISH

Surface area and condition both contribute to the response problem. When the process piping surface area is large, adsorption and desorption of moisture will take much longer to reach equilibrium. When these surfaces are dirty or rough, the effective surface area increases significantly. Components with a smooth mechanical finish are always preferred. Keeping these surfaces clean will always pay big dividends for moisture analysis.

Do not confuse the term electropolished with a mechanical polishing procedure. Electropolishing is an electrochemical procedure that satisfies the deficient ionic sites of surface metallic molecules. These ionic sites strongly attract the polar moisture molecule, causing a stronger adsorption effect at that site. For the best results, electropolishing is typically preceded by mechanical polishing.

Consider for Your Application. If a choice of finish is available for the materials dictated by the process or sample system, select the smoothest for faster response. If there is a possibility that particulate material will plate out and build up in the system upstream of the measuring point, try filtration and fast loop techniques to reduce the effects of the buildup. Withdrawing the sample from the middle third of the process piping can help avoid pulling in heavier contaminants that may be moving along the piping walls. Check the sensor periodically for evidence of contaminants carried to it from the stream when particulates may be entrained in the process.

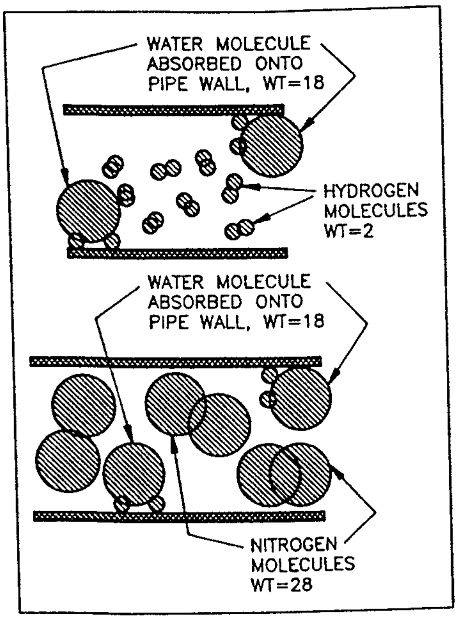

Figure 3. *In very dry systems using hydrogen and helium, the weight of the carrier gas relative to that of water must be given particular attention. The small molecular mass of these gases makes it difficult for them to move a heavier moisture molecule out of the system.*

CARRIER GAS MOLECULAR WEIGHT RELATIVE TO WATER

An often overlooked factor in response speed is the molecular weight of the carrier gas relative to that of water. With rare exceptions, this is a problem only in those very dry systems that use hydrogen and helium as purge gases. Because both these gases have a small molecular mass, it is a difficult matter for them to move a heavier moisture molecule out of the system (see Figure 3). It's like going bowling with a Ping-Pong ball—you just don't score well, if at all.

Consider for Your Application. These very dry systems have usually been through the normal leak prevention and detection procedures during their construction. Upon startup and/or after an upset in the process, the apparent slow speed of response sometimes comes into question. This is not the fault of the sensor, but rather of the slow dry-down of the process.

The best way to purge moisture out of the system is to use a heavier molecule gas for the purge cycle whenever possible. If the process valving can divert the purge gas into and then back out of the piping without contaminating the process, then purging can speed up the process. The usual purge gases are nitrogen and argon, but sometimes carbon dioxide is acceptable. In addition to selecting gas with a high molecular weight, the cost of the gas is usually a factor since it is typically vented overboard.

When designing process piping or sample systems, incorporating three- or four-way valves can simplify the purging function. For example, a customer had been using hydrogen in a silver-soldering furnace operation. He claimed that it took one to two weeks to purge the furnace using only hydrogen before it could be used for soldering the parts. If he purged first with argon, he could dry out the furnace in just one day and continue on with his process.

SAMPLE TEMPERATURE

The sample temperature affects the adsorption rate of moisture onto the surfaces of the system. Higher temperatures produce lower adsorption, while lower temperatures increase the moisture adsorbed into the inner walls of the system. Raising ,the temperature of the stream can sometimes help the speed of response, but the benefit might not justify the added cost. This technique should be used only when the quickest response is required.

Consider for Your Application. Maintaining a temperature above the normal ambient conditions can help the speed of response if it can be done economically. Because temperature impacts speed less than other factors, heaters and controls should be added only after the methods discussed above have been installed and optimized.

The following temperature-related procedure can provide an initial benefit when starting up a system that will operate in the very dry region:

1. With the sensor removed from the system and the sensor port plugged, use a good flow of nitrogen or argon as a purge gas through the system. To prevent a pressure-related hazard, the system pressure should not exceed a few psig.

2. Apply heat to the process and/or sample system tubing and other components that can withstand the temperature. Begin at the upstream side of the system and work slowly toward the exhaust. The heat will drive adsorbed molecules off the walls of the piping and into the purge gas to be carried out.

3. After the sensor flow cell has been heated and purged, continue the flow of gas and allow the system to cool. Then remove the sensor port plug and replace the sensor.

4. The system can now be checked for

leakage either using the moisture sensor to detect elevated moisture levels or by some other method such as conducting a helium or Freon leak test.

5. Once all leakage has been eliminated, valve off the purge gas. If the process has not been started up again, leave the sensor section, including a fast loop, isolated until the process has been running for a sufficient time to purge all contamination out of the system. Then switch over the sample system or fast loop to the operating process.

SAMPLE PRESSURE

Often pressure can neither be changed nor selected. Measuring moisture at process pressure with an in-line sensor is such a case. Because water vapor pressure changes with total pressure, sample systems can allow the pressure at the sensor to be selected to enhance its speed of response. For example, if a dry process stream is at an elevated pressure, the dew point is elevated toward the middle of the range even though the concentration of moisture may be low. Why reduce the pressure over the trace moisture sensor within the sample system? Even if the sample will be vented overboard, operating the sensor at the highest pressure provides faster response. The pressure drop can then be taken after the sensor location.

Consider for Your Application. Maintaining the elevated pressure of the process can keep the vapor pressure of the sample significantly higher, which can benefit some sensors. For example, if the process pressure of a nitrogen stream is 100 psig with 5 ppmv of moisture, the dew point will be -50°C. If the sample pressure were dropped to atmospheric, the dew point would drop to near -66°C. This moves the moisture level into a region of the sensor that is generally slower to respond. The closer to the middle of the sensor range, the faster the sensor responds to changes. In this case, if the sample is to be vented to atmospheric, simply use the exhaust valve to regulate flow.

PHASE CHANGE BY REDUCING PRESSURE

This unique situation occurs when the stream is a light hydrocarbon liquid that vaporizes at a reduced pressure and ambient temperatures. Typically the liquid process pressures are from 100 psig to 1000 psig. Sample systems often contain a vaporizing (heated) regulator that drops the pressure while keeping the sample warm. This phase change will slow the response speed of the measurement because the liquid volume produces so much more volume in the gas phase. If the vapor phase sample is throttled to reduce the consumption for measurement, then the measurement for the sample may lag far behind, displaying readings for samples that have long since passed the sample point.

Consider for Your Application. Because our aluminum oxide sensor can be used directly in some liquid process streams, the need for a vaporizing sample system may sometimes be eliminated. Direct insertion would provide the best speed of response, as long as all the remaining sensor parameters were satisfied. Yet historical sampling methods often prevail. If vapor phase samples have been used, then this method should probably be continued—if only because comparisons between liquid and gaseous units of measure are practically impossible.

Endress + Hauser offers a fixture that allows the probe to be inserted directly into the process stream through a ball valve. This fixture permits the probe to be withdrawn while under pressure for servicing (see Figure 4). As long as we know the solubility of water in the liquid, we can read out directly in ppmw in the liquid phase.

Note: ppmw moisture in the liquid phase is not the same as ppmv in the gaseous phase.

SENSOR TECHNOLOGY

All sensors are not created equal. Some slower sensing techniques may be useful for their other characteristics. All moisture sensors will benefit from the issues we have covered here.

Searching out specifications for the speed of response of a particular sensing technology is difficult at best. Most of the data available come from the sensor manufacturer. A comparison test of techniques for measuring moisture must usually be completed with the process conditions in mind. Such a test is most effective if completed without the bias of factory claims.

Consider for Your Application. When time permits, the testing should be conducted against reliable standards, comparing not only response but also accuracy and repeatability. The testing program should begin with nominal process simulations and progress to more demanding process-upset simulations to determine sensor stability. These tests should include a repeated regimen of returning to a normal reading periodically to track sensor drift and survivability.

If testing is out of the question, the best information on

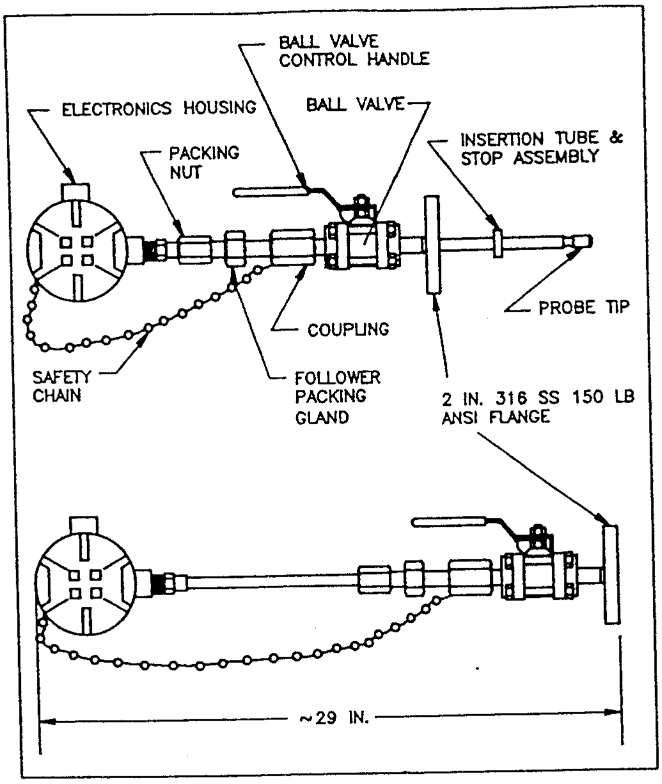

Figure 4. *In some sample systems, a vaporizing regulator is used to drop the pressure while keeping the sample warm. This phase change, however, creates a response speed lag because the liquid volume produces so much more volume in the gas phase. The problem can be eliminated in some cases by using a direct insertion probe in the process stream. A ball valve on the probe shown here allows the probe to be withdrawn while under pressure.*

accurate sensor performance can come from a customer who has used the sensor in a similar process. These processes might not have all the same characteristics as the desired application, but should be close enough to give added confidence that the application will perform well.

UNIT OF MEASURE

Apparent Speed of Response. The unit of measure may be a consideration in the function of a particular sensor. Some sensors operate as a function of dew point, ppmv, or RH. The differences in these units of measure can often indicate, at first blush, that one sensing technique is responding faster than another when actually the opposite may be true. For example, a sensor indicating in dew point might show a change of only -30°C to -50°C in 10 min., a change of 20°C. Another sensor indicating the concentration in ppm_V in the same stream would indicate a change from 376 ppmv to 38.8 ppmv at atmospheric pressure in nearly 20 min. But since this change is nearly an order of magnitude, it may appear faster.

The only way to ensure an accurate comparison is to normalize the readings to a common unit of measure and supply a sample to all sensors simultaneously while tracking the results.

Consider for Your Application. When selecting a new technique for a moisture measurement, keep in mind that different sensors respond to different moisture effects. Some respond to changes in concentration, some to RH, and some to changes in water vapor pressure.

If a new method is chosen to measure in an existing process, then operators using the instruments may need to be retrained to think in different units of measure for moisture and the comparative ranges of normal process operation. This training is often factory supported with documentation or actually conducted on site.

SUMMARY

Sensor selection is usually governed by a variety of considerations, among them speed of response, temperature, pressure, moisture range, flow rates, and compatibility.

Asking the right questions will help determine system design. Working with factory specialists will yield the best results, but process experience in the field is also important in determining the final system design parameters.

Flow and pressure changes, for example, may rule out the possibility of fast loops, or mean that some type of pumping will be required. This perspective can come only from the process engineer.

The procedures outlined in this article are based on fundamental physical principles. Individual results will depend on process conditions, materials, and other factors.