MISCHIEVOUS MOISTURE

Jung-il Kim, AMETEK Process Instruments, USA, emphasises the importance of accurate measurement and analysis of moisture in process gas.

Moisture affects the physical, chemical, and electrical properties of virtually everything. Moisture (water vapour) is an impurity in the gas processing and chemical manufacturing industries, and its presence in process gas streams adversely affects the manufacturing process and the final product quality. In fact, moisture is the most commonly monitored impurity in any industry. If left undetected, moisture can lead to process downtime or lost end product. Moisture can cause freezing in gas process lines which, in addition to adversely affecting the product, can also damage process lines and equipment. Accurate measurement and analysis of moisture in process gas minimises corrosion to equipment, improves process yields, lowers plant operating costs, and slows degradation of expensive catalysts. Control of the moisture level is critical for product quality and process optimisation.

Background

The 'dew point' or 'frost point' of water is the temperature at which the water vapour changes physical state to a liquid (condensation) or a solid (freezing). This is a physical state measurement which is expressed in degrees (typically Celsius or Fahrenheit), rather than a quantitative measurement of how much water vapour is present in the gas.

The amount of moisture in the process gas may be expressed in a variety of units, most of which are different ways of expressing mole fraction, or the fraction of water that is contained in the process gas. Examples of typical quantity units used in moisture measurement are: parts per million by volume (ppm[v]) or weight (ppm[w]), nanograms per normal cubic metre (ng/nm^3), pounds per million standard cubic feet (lbs/mmscf), or indirectly as partial pressure exerted by the water vapour in a gas mixture, which varies by the amount of water present. Units such as ppm(v) are insensitive to changes in process temperature or process pressure. Measurements using these units do not need to compensate for changes in pressure or temperature, such as required when using partial pressure to measure moisture. For example, in a 1 litre closed volume of sample gas containing a known amount of moisture, reducing the volume by 50% does not change the concentration of water molecules

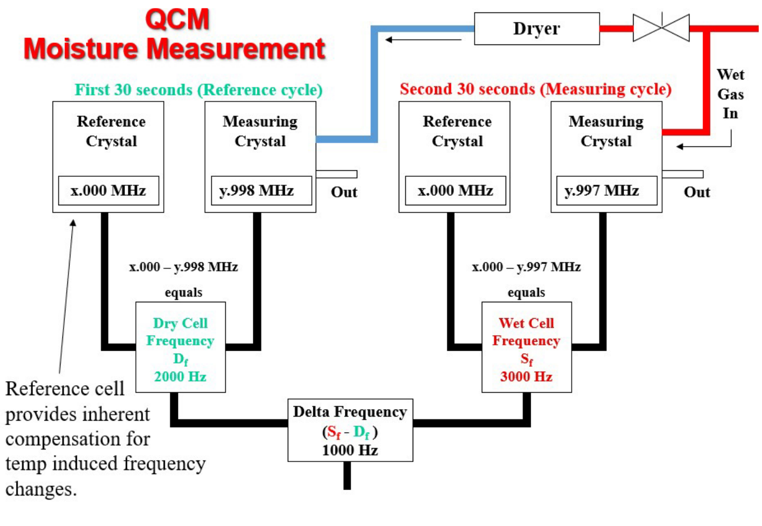

Figure 1. QCM moisture measurement.

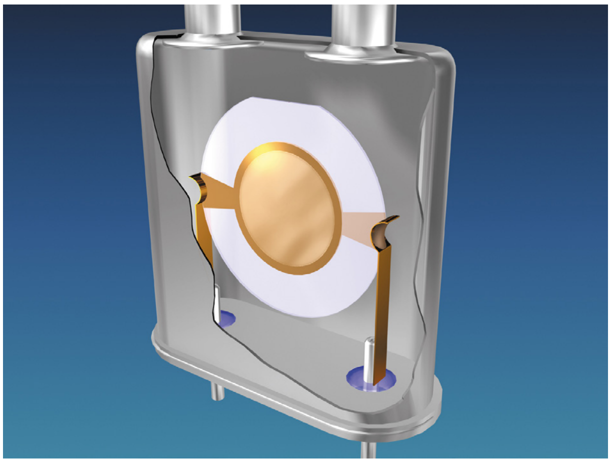

Figure 2. The quartz crystal oscillator is the heart of AMETEK Process Instruments' 3050-OLV moisture analyser.

in that volume. No water molecules have been added or removed from the volume, which means that the moisture concentration in units such as ppm(v) remains the same and does not need to be compensated for process conditions. Partial pressure of water behaves differently. The same 50% reduction in volume doubles the total pressure of the water molecules, because the same number of water molecules are forced to occupy half the volume.

Technology

Various technologies are available to measure moisture. The manual chilled mirror instrument was the first widely used process moisture monitor. This device measures the temperature at which dew or frost forms: the water dew point or frost point temperature. The drawback of this method was that some process gases liquefy before the water vapour condenses. In the 1950s, driven by a need to measure water concentration in Freons, which remain liquid at relatively high temperatures, the DuPont Co. developed a new method for measuring the moisture concentration in ppm(v) that utilised an electrolytic sensor with phosphorus pentoxide (P_2O_5). Also, in the 1950s, the Department of Scientific and Industrial Research Torry Research Station developed an oxide moisture sensor of aluminium oxide (Al_2O_3) to measure moisture in fish drying ovens and later to measure humidity in weather balloons. A decade later the piezoelectric sorption method of moisture measurement (using a polymer coated quartz crystal) was developed by ESSO Research and Development, then purchased by the DuPont Co. in 1969. This piezoelectric method, known as quartz crystal microbalance (QCM) technology, was first used to monitor moisture in a catalytic reformer's hydrogen recycle gas. Laser-based moisture analysers were developed in the mid-1980s by Bell Labs and were first used to monitor semiconductor production gases.

QCM technology

QCM technology measures the difference in oscillation frequency of two independent quartz crystals. One quartz crystal is the reference crystal and the other is the measuring crystal. The reference crystal is isolated, and the measuring crystal is coated with a thin film of hygroscopic material that is in contact with the wet sample gas. The mass of the measuring crystal increases as the hygroscopic material adsorbs water molecules from the wet stream, thereby changing the oscillation frequency (wet cell frequency). At regular, periodic intervals, the sample gas is routed through a reference dryer (dry gas), and the mass of the measuring crystal decreases, changing the oscillation frequency (dry cell frequency). The wet cell frequency is then compared against the dry cell frequency, and the difference in oscillation frequencies between sample gas (wet cell frequency) and dry gas (dry cell frequency) is the delta frequency. The delta frequency translates into the moisture concentration of the sample gas. The moisture on the measuring crystal is not in equilibrium with the moisture in the sample gas due to the periodic exposure to both the wet and dry gas, and this provides a fast response for the moisture measurement.

DuPont Instruments (purchased by AMETEK in 1989) further differentiated the QCM moisture analyser by developing online verification (OLV). The verification is accomplished by including an NIST traceable internal moisture standard (moisture generator) within the QCM analyser to produce a known value of moisture against which the analyser performance is checked. These performance checks could be done on a regular basis or as needed, to assure confidence in the measurements provided by the instrument.

Low level moisture applications

Low level moisture measurement is especially crucial in the refining, gas processing, and petrochemical industries. Moisture measurement in hydrogen recycle gas in the catalytic reformer and hydrocracker processes is a primary application. In both processes, a catalyst is used to optimise the chemical reactions, and uncontrolled moisture can poison the catalyst. Measuring the moisture concentration in the hydrogen recycle gas is crucial to optimising product yield and catalyst life.

Figure 3. Typical moisture analyser, AMETEK Process Instruments' 3050-DO.

Moisture is also monitored in the light olefin feed to the HF/H_2SO_4 alkylation process where sulfuric acid or hydrofluoric acid is used as the catalyst. The acid catalyst is corrosive in the presence of moisture. Heat generated in the exothermic process varies with the moisture content in the feedstock. Since high temperatures cause polymerisation, low temperatures are needed for good quality alkylate yield, optimising the consumption of acid, and reducing cooling costs. To protect the acid catalyst and optimise the operating temperature, it is critical to accurately measure low moisture concentrations in the feedstock.

Moisture measurement is critical in the manufacturing, storage, and distribution of olefins. Analysis of the moisture content on the feed gas pipelines from naphtha cracking and ethane cracking to polyethylene and polypropylene plants is critical to production. In gas phase polymerisation reactors, the moisture content of the feedstock, typically a highly purified (olefinic) monomer gas such as ethylene or propylene, is measured to protect the catalyst from poisoning. Between reactions, the reactor may be purged with nitrogen, and the moisture content of the purge gas is monitored to verify dry reactor conditions.

Moisture measurement is performed in the dehydration of natural gas during processing and natural gas liquids recovery. Along the natural gas pipeline, analysis of the moisture content is conducted at customer transfer locations to confirm the gas quality meets specifications. Moisture is also measured during transmission and storage to prevent hydrate formation, limit corrosion, and protect infrastructure.

Installation matters

Regardless of the technology chosen to measure the moisture content, the success of the measurement depends on proper installation of the instrument system. A reliable and accurate moisture reading depends on proper extraction, transport, conditioning, and delivery of the sample to the analyser.

Several factors should be considered to achieve a proper sample conditioning system:

- The speed of response is dependent on the distance from the sample tap to the analyser. The distance should be as short as possible, typically within 15 ft.
- Especially for low level moisture, it is recommended that the sample line be 1/8 in. electropolished tubing which is heat traced to 60˚C. Minimising the overall volume of tubing is desired. 1/4 in. tubing has 7.8 times more volume than 1/8 in. tubing.
- To reduce sample volume, it is recommended to reduce the sample gas pressure to a level required by the analyser at or close to the sample tap.
- To ensure that the analyser receives a fresh sample quickly, the overall sample flow through the sample conditioning system should be maintained at a high level. A fast loop or bypass can be installed at the sample inlet of the analyser. While the overall sample flow rate can be increased using a fast loop, the tradeoff is potentially wasted sample gas. The fast loop or bypass also provides the ability to troubleshoot higher than expected moisture readings. If the flow rate through the fast loop/bypass is increased, the moisture reading on the analyser should remain stable. However, if the moisture reading decreases, it indicates that additional moisture is being added to the sample gas. Conversely, if the flow rate decreases through the fast loop/bypass, the moisture reading should remain stable. However, if the moisture reading increases, it indicates the same problem of additional moisture intrusion in the system. The source of this added moisture could come from the wetted surfaces of the sample conditioning system due to insufficient purging and/or leak and/or dead volume(s) in the system.
- Purging the sample system sufficiently, before connecting the analyser, can reduce or eliminate any contaminants remaining in the sample conditioning system reaching the analyser. Prior to maintenance/shutdown of the process, it is important to isolate the inlet and outlet of the analyser to protect it from contamination. After the maintenance/shutdown of the process and before the analyser is connected to the sample conditioning system, the sample system should be sufficiently purged.
- Verify that continuous sample gas is being supplied to the analyser. Without a continuous sample gas supply, the analyser will get contaminated via the vent/exhaust ports on the analyser.
- Do not install any flow control or flow meter on the sample conditioning system upstream of the analyser. Flow control or flow meters (rotameters) are a source of dead volume and can introduce ambient moisture into the sample stream.

Conclusion

Accurate measurement and analysis of moisture in process gas is critical. Regardless of the technology chosen for crucial application, the success of measurement also depends on the proper installation of a moisture analyser. Therefore, obtaining a reliable and accurate moisture reading depends on choosing the right technology, proper extraction, transport, condition and delivery of sample to the right moisture analyser.