Manufacturing operations must keep output and quality their top priority. Therefore, initiatives aimed at replacing current instrumentation sometimes do not attract the management focus needed to support and fund such projects. One area of possible continuous improvement with a short payback time concerns process analytical systems for the measurement of oxygen in inerting or blanketing applications. Switching from maintenance-intensive on-line extractive systems to a more reliable and easy-to-maintain in-situ (in-line) solution will not only improve the bottom line, it will also help reduce unscheduled downtimes and assists production planning.

Know what is inside ...  
Manufacturing processes that require active measures to minimize the risk of explosion are inerted using nitrogen blanket-ing, i.e. displacing oxygen from the tank or vessel headspace with nitrogen. Basic inerting systems consist of inert gas being fed to the tank in order to create an overpressure and avoid any air ingress. Due to the cost of nitrogen, not all the oxygen is expelled from the vessel: only the amount of nitrogen necessary to keep the oxygen concentration below a safe level is introduced. The maximum concentration of oxygen that is acceptable is dependent on the mix of vapor gases and combustibles present in a tank. This level is called the limiting oxygen content (LOC). As long as the LOC is not exceeded, a source of ignition such as an electric discharge or overheated equipment will not cause an explosion.
Higher levels of protection against explosive conditions can be attained by using oxygen headspace measurement along with over-pressure control. By determining the value of residual oxygen in the nitrogen blanket, more cost-effective inert gas blowing can be attained, as with this method nitrogen is blown discontinuously, saving on nitrogen costs. A feedback-control loop can be installed that will immediately trigger the opening of the nitrogen valve if oxygen concentration in the vessel starts to rise.

Most commonly, extractive paramagnetic oxygen systems are used in inerting applications due to their high accuracy and wide measurement range from 0.1% to 100% O₂. Although their performance is largely sufficient for the requirements of inerting, paramagnetic systems cannot be directly connected to the vessel to be inerted, as the measurement cell must be protected from humidity and dust that might be present in the process gas mix. This protective function is conducted by a gas sampling and conditioning system that is installed between the process and the paramagnetic cell. Every component in the sampling and conditioning system such as pressure reducer, valves, filters or flow meters must be selected individually to work best with the gas mix to be measured.

...but not at any cost

The presence of a gas sampling and conditioning system greatly increases the complexity of oxygen measurement. Such systems have many potential sources of error (see box, right) and therefore frequently fail in operation and require regular maintenance.*

In a continuous process any measurement breakdown in the inerting system cannot be tolerated as it would automatically trigger process downtime to effect repairs. For this reason, sampling and conditioning systems are subjected to heavy pre-emptive maintenance in order to minimize the possibility of system failure.

Typical weak points of sampling and conditioning systems

- Condensation: Humidity is frequently present when a hot gas sample is cooled prior to measurement. Moisture in the process gas must be fully removed before the sample reaches the measurement cell. If it is not eliminated the cell will need replacement.
- Wet/Dry concentrations: In gas streams with high water content, the removal of condensation from the gas sample also means that the oxygen value measured by the paramagnetic cell will be overestimated, thus requiring a wet/dry reporting factor.
- Dust: Damage to the measurement cell will occur if dust is present in the gas sample.
- Line blocking: Piping in sampling and conditioning systems is often thin and follows complicated paths within the enclosure box. Moisture or dust can accumulate in corners or U-shaped lines and block the process gas.
- Filter blocking: Filters must be exchanged frequently if the dust load is high. Failure to do so will also block access lines.
- Sample flow: The paramagnetic cell can measure incorrectly if the gas flow is too high; therefore, it must be regulated. This is problematic if a sudden increase in oxygen level occurs in the tank as it can result in a delay in the response time, particularly if the sampling line is long.

In-line oxygen solutions are less complicated, and therefore far less error-prone than extractive systems.
Therefore, with extractive systems, the added safety of oxygen measurement in the tank headspace comes at a high price. System reliability is attained at the expense of intensive, resource-absorbing, pre-emptive maintenance. Is there an alternative to this trade-off between cost-of-ownership and process availability?

**Benefits of polarographic sensors**

Oxygen measurement systems from METTLER TOLEDO based on polarographic sensors offer an attractive alternative to extractive systems for inerting applications. Polarographic sensors are electrochemical devices with a constant polarization voltage applied between cathode and reference electrode. As opposed to galvanic cells (fuel cells), they do not need a lead anode to generate the polarization voltage. Since the polarization voltage is kept constant using an electrical circuit instead of a lead anode, this drastically reduces drift and extends the maintenance intervals. The current flowing between anode and cathode is proportional to the oxygen partial pressure in the media. With values as low as 70 nA for air, there is no measurement error due to the self-depleting effect of oxygen consumption at the cathode tip.

Contrary to paramagnetic systems, polarographic sensors are membrane-covered and do not need additional protection from moisture or dust. The membrane in METTLER TOLEDO’s sensors consists of a steel mesh embedded in a silicone layer for enhanced pressure resistance. Accumulation of droplets at the membrane surface do not affect the measurement, as long as equilibrium is reached between oxygen content in the liquid and gaseous phases. Also, formation of a diffusion-blocking dust layer on the sensor tip is drastically reduced by the use of a dirt-repellent PTFE outside layer. The maintenance requirement for polarographic sensors is significantly less than that for paramagnetic cells. Due to the former’s highly linear measurement range between 0 % and 100 %, there is no loss of measurement accuracy when air is used as a calibration gas, even if measurement is performed at the 2 – 5 % O₂ concentration range, as typically met in inerting applications. Because the zero-point value of the sensor characteristic does not drift significantly, air is the only calibration gas required to calibrate a polarographic sensor.

The air current drift that occurs in polarographic sensors is caused by the progressive contamination of the electrolyte inside the membrane module. This is why regular membrane and electrolyte exchange is recommended in order to reduce drift. The frequency of this necessary maintenance step depends largely on the process conditions and composition. But since a one-point air calibration can be performed on METTLER TOLEDO’s sensors in less than five minutes at the measurement point itself, optimal performance can be maintained at a high level throughout the service life of the membrane without significant impact on process availability.

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**The Universe of Intelligent Sensor Management**

**Intelligent**
- Digital sensor technology offers easy handling and enhanced sensor performance.
- Sensors can be pre-calibrated and stored for later use.
- Electronic documentation of calibration and maintenance for regulatory compliance.

**Tailorable**
- Industry specific solutions tailored to respective needs, from Biopharma to Chemical to Food and Beverage.

**Adaptive**
- Flexible system integration via transmitters, converters or Ethernet allows integration of diagnostic information.
- Digital technology permits temporary, remote and wireless solutions.

**Predictive**
- Avoid unscheduled maintenance with real-time diagnostics.
- Unique software provides instant evaluation of sensor condition.
ISM: Process Analytics 2.0

Industrial processes must meet growing requirements from the sustainability, environmental, and safety points of view. It is no longer sufficient that they run well, they also have to improve productivity for increased competitiveness, reduce emissions to comply with environmental standards, and demonstrate safe working conditions for people and assets.

Intelligent Sensor Management (ISM) is a METTLER TOLEDO concept that supports users in all these tasks. ISM sensors and analyzers are “tuned” to deliver more stable and accurate measurement using digital technology (see illustration on previous page). They also perform online diagnostics to identify potential sources of problems in sensors before they occur. For example, polarographic sensors with ISM are able to detect electrolyte depletion inside the membrane body, prompting the user to schedule maintenance.

ISM simplifies the handling of analytical equipment, enhances its reliability and reduces sensor lifecycle costs. By making analytical equipment run more efficiently, operations can better meet industry expectations.

Conclusion

METTLER TOLEDO polarographic oxygen measurement systems offer the advantages of direct, in-situ measurement without the burden of demanding and delicate sampling systems. Moreover, cost-of-ownership calculations show a fast payback time due to the particularly easy and user-friendly calibration and maintenance concept.


For more information, visit:

www.mt.com/o2-gas

Case study: CP Kelco Germany GmbH

CP Kelco is the leader in the production of polysaccharides by microbial fermentation. It is also the largest producer of pectin, a widely used high-quality gelling and texturizing agent. At Grossenbrode (Germany), pectin is obtained from processing citrus peels by extraction in hot, acidified water followed by isolation of the pectin from the ensuing solution. After clarification, propectin is precipitated with isopropyl alcohol (IPA) to form a slurry under inert conditions.

Due to the potentially explosive atmosphere created by the IPA, CP Kelco Germany GmbH used a centralized paramagnetic oxygen measurement to monitor the oxygen levels in over 50 reactors. The system was connected to the reactors using multiplexed gas lines, measuring one reactor at a time. While this setup ensured data consistency and quality, it also showed the dependability of the whole precipitation step on this single measurement point. Occasionally, splashes of slurry in a reactor would block the sampling line to the paramagnetic cell and the reactor had to be set on hold until the problem was rectified.

Unsatisfied with the process downtimes and high maintenance demand of the oxygen measurement system, the company turned to METTLER TOLEDO to supply a solution that was to be installed in-situ into each reactor, without the drawback of slurry blocking. Additionally, these sensors had to demonstrate lower costs of ownership compared to their current centralized solution.

CP Kelco Germany GmbH selected the award-winning InPro 6850i G polarographic sensor inserted into static InFit housings welded into the vessels’ domes. A first system was installed on a test basis in July 10 and its performance in real conditions analyzed over several months. The system not only fulfilled CP Kelco’s requirements, it also delivered more predictability into the sensor maintenance process. Because of its simplicity and short duration, the required maintenance did not disrupt manufacturing operations. After passing all the tests, CP Kelco Germany GmbH rolled out the installation of METTLER TOLEDO polarographic systems on all the precipitation reactors.