Optimization and Monitoring of Oxygen in the Vapor Phase Environment

Abstract: Mettler Toledo/Ingold's oxygen measurement system is utilized to optimize and monitor vapor phase systems reducing maintenance costs and facilitating automated calibration. Three applications detail performance in nitrogen blanketing and reactor off-gas applications. The focus is both safety and economic based.

Background: Measurement of oxygen in the gas phase can be a critical optimization parameter for nitrogen blanketing, reactor off-gas and OSHA controlled areas. The discussed applications have critical economic and safety components.

Challenge: Accurate and low maintenance continuous oxygen measurements require: A probe that is stable, fast and resistant to fouling or destruction from aggressive solvents and other constituents. It often requires electronics and electrical connections unaffected by moisture, temperature extremes as well as power surges or dips common in industrial settings. A user friendly operator interface and a means of communication with external control and monitoring systems is also a must. Frequently the end user will require a means of automatically verifying the performance of the measurement systems for safety considerations.

Capital associated with sample conditioning must also be minimized to keep the system cost effective for the industrial end user.

Solution: Pioneering experience with polarographic oxygen measurement in the liquid phase has lead to the development of oxygen measurement technology that meets the challenge of the vapor phase environment. Special membrane technology (Teflon or Silicon) equipped with Kalrez o-rings will hold up to the harshest solvent laden streams. Stainless steel (316), Hastelloy (C22) and polysulfone electrode bodies fit most conditions experienced in industrial environments.

Accuracy of 1 ppb can be achieved with Ingold polarographic electrodes. Response times are typically 20 seconds for 95% response with 100% response being achieved within 90 seconds. This is sufficient for most dynamic processes.

Electronics are available for FM Class 1 Div 1 or 2 Group A-D environments.

Limitations of the technology are based on the vapor pressure of oxygen within water (The higher the pressure, the higher the operating temperature). Applications up to 100C have been documented.

Application 1: A Fortune 100 chemical manufacturer was looking for a solution to update oxygen monitoring on reactor off-gas. Their requirements were for the lowest

maintenance system that required the least complicated/costly installation. Additionally, they needed a system that could be automatically validated every 8 hours.

The plant had extensive success with Mettler oxygen equipment in the liquid phase. They were also very happy with the performance of Mettler retractable housings.

777 housing

Retractable housings give the end user the ability to insert or retract an electrode from a continuous operating process at any time. They come equipped with a flushing chamber available during retraction and can be equipped with pneumatic retraction as well as inductive position indicators.

The conditions of the vapor stream were aggressive and required a Hastelloy probe with Kalrez o-rings. Temperature was consistent at 75C and the pressure was 15 psig. Requirements included a transmitter with the ability to compensate for the increased pressure yet operate in a FM Class 1 Div 2 environment. The ultimate goal was to trim oxygen to approximately 8% for combustion.

The plant now utilizes the InPro 6000 series electrode with the 777 retractable housing and a 4100 oxygen transmitter. The system automates probe retraction (pneumatically) every 8 hours and runs a 5% oxygen standard through the flushing chamber to check performance. They are looking for a 95% response within 30 seconds and an approach to 100% final value in 2 minutes.

Mettler Toledo 4100 Field Mount Transmitter

Results: After 5 plus years of operation the plant has successfully applied the technology to trim oxygen in the reactor off-gas. Maintenance requirements have been standardized and involve a biannual membrane change and electrolyte refill of the electrodes. This is a task that takes approximately 10 minutes. It is considered a very successful application. No sample conditioning is required as the probe inserts directly into the vapor stream.

Application 2: A manufacturer of pigments requires vapor phase oxygen measurement to monitor the oxygen content in an aniline rich stream. The stream runs approximately 10% oxygen at 25 psig and 100C. Higher oxygen content is can potentially lead to a runaway reaction, while lower oxygen content can limit the optimal reaction conditions. The goal was to replace an oxygen monitoring system that required heavy and expensive maintenance as well as a timely calibration procedure. A complicated sample conditioning system, eliminating all vapors was also required for the incumbent system. Their system also required installation and maintenance of a nitrogen purged instrument box.

Although the plant had invested in the incument system only a year prior to the Mettler Toledo trial, the cost and convenience of the Ingold system was

attractive enough to generate considerable interest.

Utilizing a 316SS probe, Kalrez o-rings on the Teflon membrane and a manually actuated 777 housing, the plant underwent a trial. The 4220X HART transmitter met the intrinsically safe (IS) requirements for their classified environment and provided a HART (AMS) output to their DCS system.

After 3 months of operation, the Mettler system tracked the existing system precisely. The Mettler system required no maintenance or calibration, while the existing system required enough maintenance to pay for the capital cost of the Ingold loop. Additionally, the Mettler/Ingold system, required no sample conditioning.

Results: Based on the trial performance, the plant was able to justify redundant Mettler systems (adding an additional level of comfort) to their safety committee. The return on investment (ROI) from maintenance costs alone was enough to justify a redundant Mettler/Ingold oxygen system, meeting the plant's ONE YEAR ROI requirement.

Application 3: A major manufacturer of ink for the printing industry currently utilizes an oxygen monitoring system requiring extensive sampling conditioning to monitor a nitrogen blanketed space above a reactor vessel. Because they are manually opening the vessel to add solid product to solvent based media, maintaining a nitrogen blanket is a major safety consideration. The current system requires excessive maintenance and frequently suffers from inaccuracy. ***The inaccuracy was great enough to cause the system to be completely ignored***. Safety was ensured by running tremendous amounts of overpressure on the nitrogen blanketing system.

Both safety and economics were being compromised with the existing system. Additionally there were water requirements for running the vacuum eductor and sample condenser.

In search of a better system, the plant assigned an engineer to a 4 month evaluation of the Mettler system.

Two Mettler/Ingold oxygen loops were installed in stationary housings, in both the sample conditioning system and directly into the reactor. The goal was to establish the efficacy of installing the Ingold system directly into the tank where it would be exposed directly to condensable solvent vapors and potential fouling. The conditioned sample was chosen to compare the response time and accuracy to the existing system that was maintained and calibrated weekly.

The performance evaluation showed that the Mettler oxygen system in-tank tracked directly with the sample conditioned Mettler system (eliminating time delay). Additionally, the Mettler system required no maintenance during the entire trial period, significantly reducing time requirements by plant personnel.

The results of the trial showed that the Mettler/Ingold system was:

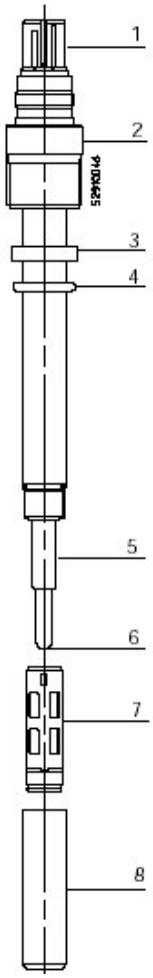
1. More accurate.
2. Easier to maintain.
3. Faster to respond.
4. Required no additional utilities.

Results: The Mettler Toledo/Ingold oxygen system was recommended as the corporate wide standard for all future plant oxygen monitoring systems.

4220X Intrinsically Safe Transmitter

Features and Benefits of the Mettler Toledo/Ingold System

Feature	Benefit
Polarographic measurement technique	More accurate than galvanic cells. Lower cost of ownership based on anode/cathode life. Low electrolyte consumption.
12 mm Teflon membrane cap design with integral O Ring	Highly resistant to fouling and durable. Quick change procedure. No individual O Ring parts required.
IP68 rated VP connection at probe	Housing leaks will not impede performance. Gold plated contacts eliminate corrosion. Connection at probe eases maintenance.
Pictorial Sensor Diagnostics	Calibration and operating conditions continuously monitored.
777 Retractable Housing	Enables manual or automated removal of sensor from a continuous process. Flushing chamber facilitates cleaning and/or calibration.
NEMA 4X FM Class 1 Div 2 A-D or Intrinsically Safe transmitter construction	Suitable for outdoor operation in all climates and all electrical classifications. Loop powered, HART and Profibus communication.



InPro 6000 Series Oxygen Sensor

1. IP 68 VP quick connect cable
2. Serial number coded threaded cap.
3. O Ring/housing seal.
4. PPS plastic body, 316SS or Hastelloy C22.
5. Silver Anode with integral RTD.
6. Platinum Cathode.
7. Teflon membrane body/integral O Ring (Kalrez o-rings optional).
8. Membrane cap.

IP68 Rated VP Cable