Chemical Production

5 Ways In Situ Gas Analyzers Improve Safety

1 Fast Response Time
Catch Excursions Quickly

2 Prevent Downtime
By Eliminating Sampling Systems

3 Monitoring Vapor Recovery Units
Where Tight O_2 Levels Are Critical

4 Measure O_2 in Small Line Sizes
Adaptions for Unique Applications

5 Operating in Corrosive Environments
With Very Low Maintenance

METTLER TOLEDO

Reduce Explosion Risk in 2 Seconds
with In Situ Oxygen Analysis

Caprolactam production requires tight oxygen control to prevent explosive conditions developing in reactor vessels. For a Chinese manufacturer, the slow response and high maintenance of their O_2 analyzers was unacceptable. Changing to GPro® 500 in situ TDL sensors has reduced measurement time from 20 seconds to 2, and almost eliminated analyzer maintenance.

Rapidly growing demand for essential monomer

Caprolactam (CPL) is an important organic compound monomer. Approximately 70 % of caprolactam produced worldwide is used in the production of nylon 6 fibers for textiles, carpets, and heavy duty tires. With economic growth in Asia is driving CPL demand, the caprolactam market was USD 8,500 million in 2012 and is expected to reach USD 13,800 million by 2019.

90 % of caprolactam is produced through the ammoximation of cyclohexanone to cyclohexanone oxime with ammonia and hydrogen peroxide (H_2O_2). This is the preferred process due to mild reaction conditions, the production of fewer by-products, shorter process time, and less environmental risk.

Explosion risks must be mitigated

The ammoximation process has been consistently improved to increase product quality and yield, but the process still relies on large quantities of H_2O_2 which are fed constantly to the ammoximation reaction vessel. During the process H_2O_2 can undergo an undesired decomposition side-reaction, which not only consumes valuable precursor but also produces oxygen, leading to an explosion risk.

In order to ensure stable and safe production, typically, oxygen content must be kept below 1 %. If the concentration exceeds safe limits, an interlock program should be started to stop the reactor feed and introduce an inert gas to dilute the oxygen content.

Paramagnetic analyzers are commonly used in ammoximation reactors to monitor oxygen levels. After extraction, process gas must pass through sample conditioning to prevent incorrect measurement due to interference from background gases, or total failure of the analyzer if the measurement cell comes into contact with moisture in the process. Sample conditioning systems require regular maintenance to sustain operation.

Major CPL producer searches for fast oxygen measurement

One of China's largest CPL manufacturers was frustrated with the high level of maintenance required on their ammoximation reactors' sampling and conditioning systems, and they were regularly faced with either stopping production while maintenance was carried out, or running production without oxygen measurement. Further, even when the analyzer was operating correctly, slow response time was a constant cause of concern.

Production managers at the facility began looking for an alternative oxygen measurement technology that would require little maintenance and would respond rapidly to O_2 level changes.

Their search focused on tunable diode laser (TDL) analyzers as they have a very low maintenance requirement, do not suffer from interference from background gases, and are installed directly in reaction vessels, so response time is very fast and sample extraction and conditioning is not required. However, they usually require the installation of two flanges: one for the TDL's laser source and the other for the receiver that analyzes the laser light that has passed through the reactor headspace. This necessitates very precise alignment of the two units, and realignment after calibration.

METTLER TOLEDO approached the facility production managers with a TDL solution that would meet all their requirements and be easy to install.

Rapid oxygen measurement directly in the reactor

The GPro 500 oxygen analyzer requires only one flange for installation as both laser source and receiver are housed in a single unit. A probe attached to the GPro 500 that extends

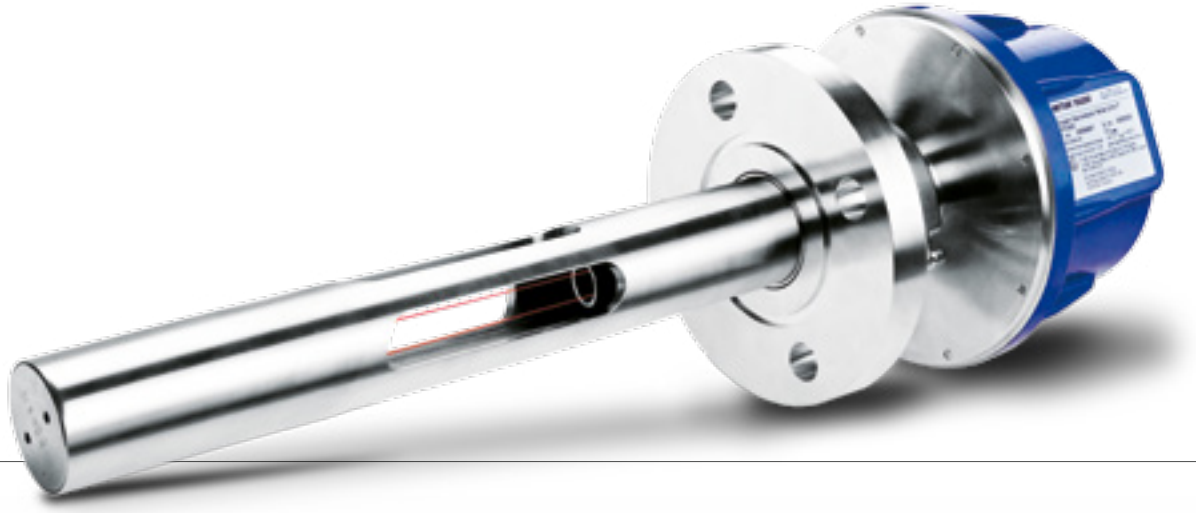
into the reaction vessel, contains a corner cube at its end. This 'prism' directs the laser beam back to the receiver, so loss of alignment is never a concern.

Tests showed that oxygen measurements from the GPro 500 were as accurate as from one of the installed paramagnetic analyzers. And unlike the 20 second response time of the paramagnetic units, the GPro 500 responded to O_2 changes in only 2 seconds, so process safety would always be assured.

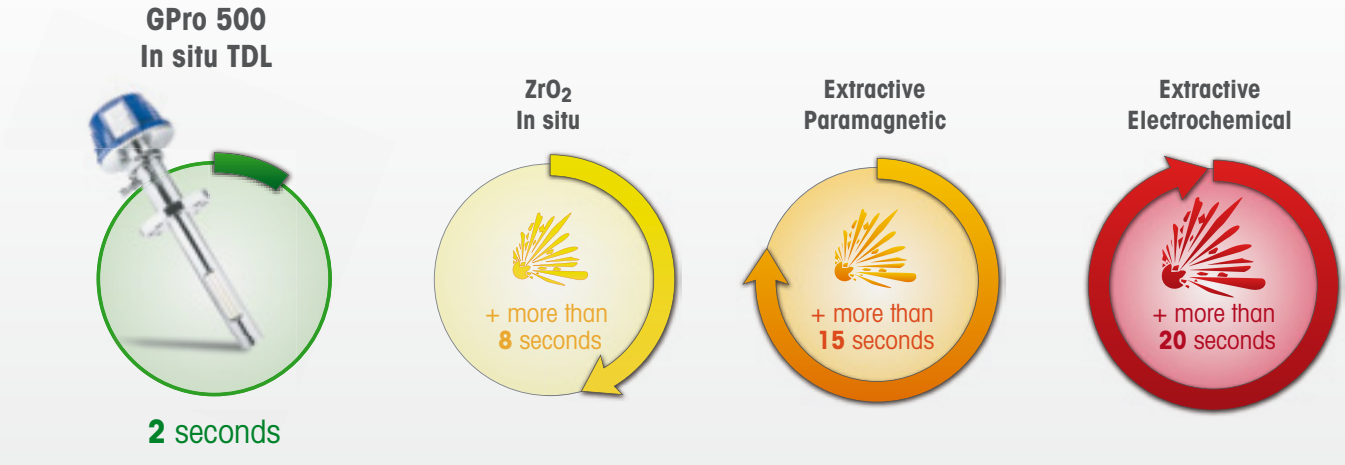
In addition, as the GPro 500 has no moving parts, there is very little that can go wrong with it.

Further testing convinced the plant managers that the METTLER TOLEDO solution would meet all their needs and five GPro 500s were installed in the facility's ammoximation reactors. In the 12 months since, all the GPro 500s have operated exactly as required – ensuring rapid detection of any oxygen upsets.

Discover more about the GPro 500:
www.mt.com/O2-Analyzer



Get 90 % Accuracy in 2 Seconds for Your Critical Safety Applications



"No Downtime Whatsoever"
High Reliability from TDL O_2 Analyzer

Oxygen sensor accuracy and speed of response are paramount when there is a risk of explosion in production vessels. For a leading producer of performance additives, METTLER TOLEDO's GPro® 500 is providing high performance with almost zero maintenance.

Safety is overriding

Phosphorus pentasulfide is an inorganic compound used in the production of lubricants, pesticides and flotation agents. It is extremely flammable; therefore, an inert atmosphere must be maintained during its production.

A closed extractive measurement system and paramagnetic analyzer can be used to monitor O_2 levels inside equipment along production lines. Keeping moisture in the gas sample at a very low level avoids negative effects on analyzer response time and performance.

Italian company needs fast, accurate O_2 measurement

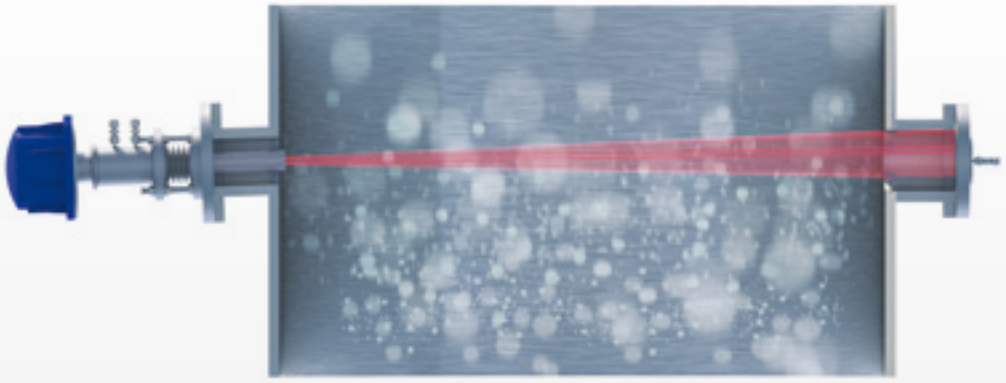
Italy's Italmatch is a leading supplier of performance additives for the lubricant, plastics, water and oil industries. They required an improved oxygen analyzer and sampling system to reduce measurement time and increase accuracy for phosphorus pentasulfide production in its plant in Spoleto.

Advanced technology provides measurement confidence

A tunable diode laser (TDL) gas analyzer uses laser light, tuned to a specific frequency, to rapidly detect the quantity of a target element in a gas stream. METTLER TOLEDO's growing GPro 500 series of analyzers offers TDLs with a wide range of process adaptions. This gives the series exceptional flexibility in respect to application suitability and installation possibilities.

Other than annual verification and periodic cleaning of the analyzer's optics, GPro 500 sensors require no maintenance; therefore, they can be relied on to provide continuous, reliable measurements throughout production processes. The GPro 500 can

Cross-Pipe Adaption for the GPro 500: Just Aim!



- Simply point the GPro 500 towards the retroreflector – that's all!
- No alignment of the retroreflector necessary
- Possibility to insert ball valves for GPro 500 removal without process interruption
- For pipes up to approx. 2 m diameter

be installed in situ when process conditions allow, or connected to an extraction system if the gas stream has a very high particle load and moisture content, as is the case at Italmatch.

Six months without maintenance

A demonstration of the GPro 500 at Italmatch's Spoleto facility convinced the instrumentation team that the analyzer's ease of use, speed of response, and low maintenance would meet all their needs. In addition, they appreciated the two alarm thresholds on the connected M400 transmitter that would signal when a lower and higher O_2 level in the production equipment had been breached; something that was not possible with their paramagnetic analyzer.

A GPro 500 with an extractive cell adaption was duly installed to a new extraction system. Since its commissioning, the Spoleto plant has been very impressed with the GPro 500's measurement accuracy, rapid measurement and problem-free operation. The site manager said, "I'm delighted with the analyzer. It was surprisingly easy to install and since turning it on six months ago we've had no downtime from it whatsoever. I wish we'd known about it years ago." At Italmatch, confidence in the accuracy of production vessel O_2 levels has been well and truly restored.

Read more about the GPro 500:
www.mt.com/Cross-Pipe

In Situ Analyzer
Ensures Safe Vapor Recovery Unit O_2 Limits

Tunable diode lasers (TDLs) are rapidly replacing paramagnetic analyzers in many petrochemical unit operations. By providing fast, accurate measurements directly from within the process, TDLs maintain process safety.

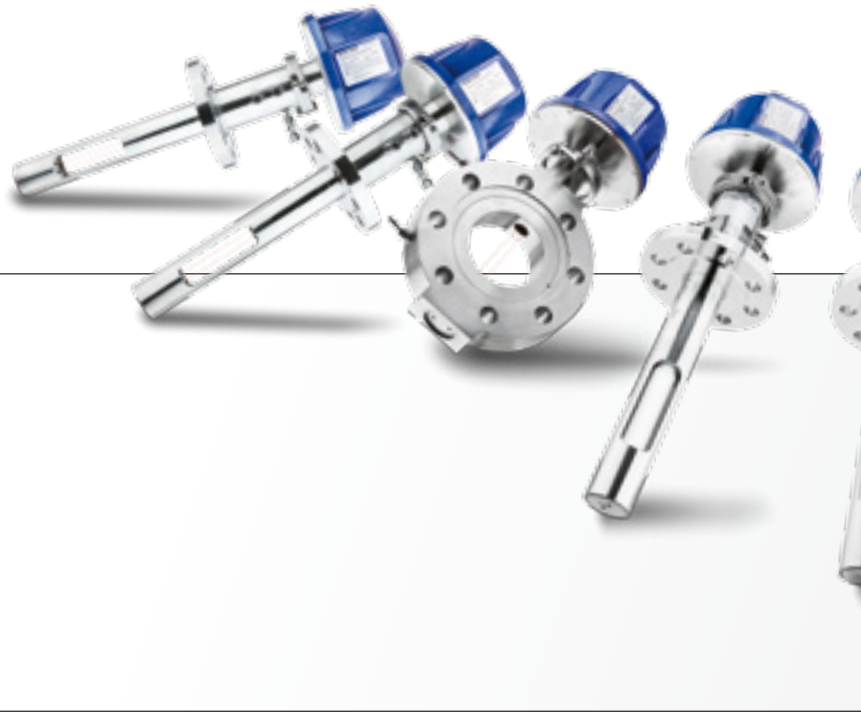
Strict oxygen levels are essential in vapor recovery units

Volatile organic compounds (VOCs) are present in many oil refinery and petrochemical plant unit operations. In some units flares are used to ignite the VOCs before they are released to the atmosphere. In other applications, such as oil/petrochem storage, handling, transport and waste systems, vapor recovery units (VRUs) collect the gas for processing as it commonly contains useful compounds.

If oxygen levels in VRUs are not kept below safe limits, there is an explosion risk; therefore, strict O_2 monitoring is employed.

Safe VRU operation requires swift oxygen measurement

One of China's largest petrochemical companies operates multiple facilities across the country. Like most companies, for monitoring oxygen levels in VRUs they traditionally installed paramagnetic analyzers, as that was the best available technology at the time. But a number of problems with the instruments, including slow response, poor measurement reliability due to cross-interference from background gases, and stability issues caused by fluctuations in the sample pretreatment system meant there were con-



stant concerns about oxygen levels in the VRUs.

In situ analyzer provides safety assurance

Consequently, the company was always looking for alternatives to paramagnetic instruments and were happy when METTLER TOLEDO China introduced to them our GPro® 500 tunable diode laser analyzer. TDLs operate in situ so sample treatment is not required. They are also largely immune to background gas cross-interference, dust in the process and require almost no maintenance. Critically, they have a rapid response time of two seconds, so any sudden increase in VRU oxygen levels is quickly identified.

Tests of the GPro 500 on a VRU at one of the company's plants confirmed its ability to accurately monitor oxygen, despite changes in process conditions. With high satisfaction in the GPro 500's performance, the company has embarked on a program to install them in place of their VRU paramagnetic analyzers.

GPro 500 TDL Oxygen Analyzer

Dependable, industry-proven tunable diode laser technology

Direct measurement in the gas stream is fast and reliable

Response time of two seconds maintains process safety

Simple installation thanks to probe design and single flange connection

No sampling or conditioning equipment. Annual verification and periodic optics cleaning are the only maintenance

Low nitrogen purging consumption means low operating costs

Sensor diagnostics continually monitor optical path quality to determine when cleaning will be required

www.mt.com/GPro500

Minimizing Explosion Risk
Where Other Solutions Cannot

To run carbon disulfide recovery with the highest safety, a Chinese chemical company wanted to install a tunable diode laser (TDL) oxygen analyzer. But the line size of the pipe at the installation point was too small. A METTLER TOLEDO TDL analyzer provided the answer.

Recovery of valuable raw material

Polyacrylamide (PAM) is a polymer widely used as a flocculant in water treatment and processes such as paper manufacture. One PAM production method uses carbon disulfide (CS_2) as a raw material. Carbon disulfide is highly toxic and waste gases from PAM production that contain unused CS_2 must be processed before being released to the environment.

Regenerative thermal oxidizers (RTOs) are commonly used for such processing. But as CS_2 is a valuable raw material, some plants recover as much of it as possible before sending the waste gas to the RTO.

The world's leading manufacturer of PAM operates production facilities in more than 20 countries, including China. In one of its Chinese plants, measurements from a paramagnetic analyzer were being used to control O_2 levels in the facility's CS_2 recovery unit, as CS_2 is highly flammable.

Paramagnetic analyzers risk process safety

Production engineers were concerned that the sampling system on the paramagnetic analyzer was significantly delaying measurements and represented a safety risk.

In addition, the sample conditioning system attached to the paramagnetic analyzer would not always eliminate all hydrocarbons in the sample. As the presence of hydrocarbons interferes with paramagnetic analyzers, there

Wafer Cell
for small line size pipes

Non-Purged Probe
for low speed and static streams

Filter Probe
for dusty or wet gas streams

Extractive Cell
works with existing sampling system

GPro 500 Gas Analyzers – for all conditions

was always uncertainty regarding the accuracy of measurements.

In situ technology delivers an immediate measurement

Production engineers at the plant were eager to replace the paramagnetic unit with a tunable diode laser (TDL) oxygen analyzer, as this technology can be installed directly into process pipes to provide an almost instant measurement and is immune to hydrocarbon interference.

However, due to the small line size of the CS_2 recovery unit gas stream feeding lines, the engineers were disappointed to discover that the TDL analyzer they were considering could not be fitted. And proximity of other equipment would not allow installation in a bypass. METTLER TOLEDO had the solution.

Scalable TDL analyzer for a wide range of applications

GPro® 500 is a range of TDL gas analyzers that can be equipped with a wide variety of process adaptions to meet virtually any installation requirement. Crucially for the PAM production facility, these adaptions include a wafer cell that allows installation of the GPro 500 into pipes down to 2 inch. Also, the compact size of the GPro 500 meant the solution could easily be fitted in the available space at the facility.

In addition, production engineers at the plant were happy to learn that the only maintenance needed on the GPro 500 is occasional cleaning of the analyzer's optics, and annual verification.

High confidence in safety of volatile compound recovery

A GPro 500 oxygen analyzer with wafer cell adaption has been installed at the plant and is providing rapid, problem-free assurance of CS_2 recovery unit safety. Plans are in place to replace other paramagnetic analyzers at the facility with the GPro 500.

See the full range of adaptions at:
www.mt.com/Probe-Adaptions

Trouble-Free Oxygen Measurement
with a Unique Gas Analyzer

When corrosive gases damaged their paramagnetic oxygen analyzer, a major producer of manufactured fibers required a better solution. The GPro 500 is not only providing them with almost instant measurements, its very low maintenance is a significant time and cost saving.

The first manufactured fiber

Rayon, first produced in the 1880s, is the world's oldest manufactured fiber. It is a natural-based material that is usually made from cellulose derived from wood pulp. Improvements in the rayon production process have led to variations on the fiber such as modal which, due to its greater absorbency, is widely used in towels and bedsheets.

One of the world's largest producers of modal is the Lenzing Group, an international organization headquartered in Austria. Lenzing (Nanjing) Fiber, located in the Liuhe district of Nanjing, China was developed with investments from the Lenzing Group and the Nanjing Chemical Fiber Co., Ltd. The Lenzing project is currently China's largest Austrian investment. Lenzing (Nanjing) Fiber mainly produces rayon and Lenzing Modal™ for non-woven fabric and clothing manufacture.

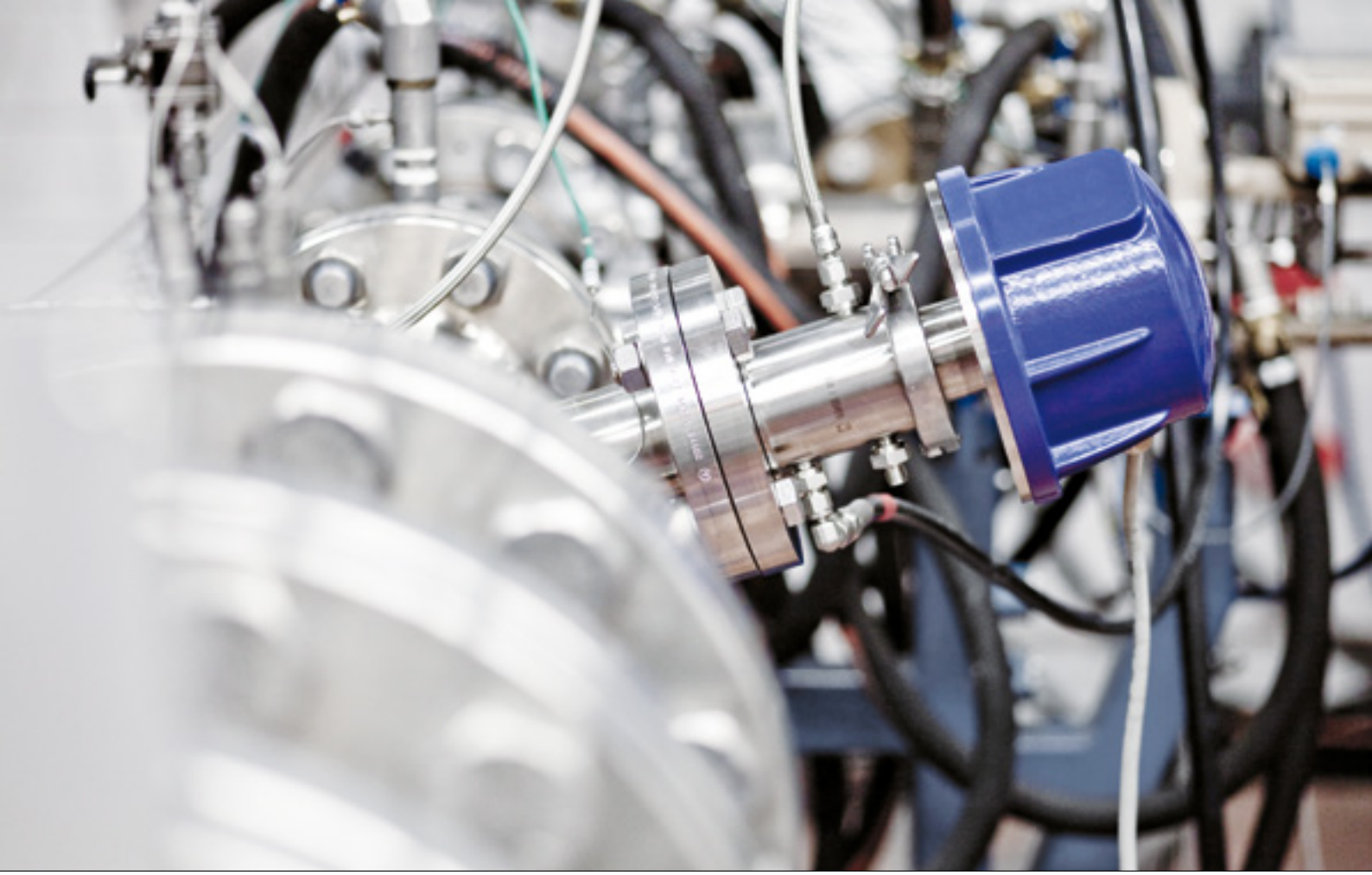
Oxygen in waste gas must be controlled

During production at Lenzing (Nanjing) Fiber, a significant amount of gas is produced. As it contains toxic combustibles such as hydrogen sulfide and carbon disulfide, the gas must be processed before it is vented to the atmosphere. During transportation by pipeline to the factory's waste gas processing unit, oxygen levels in the gas must be kept low to minimize the risk of explosion.

Corrosive compounds damage paramagnetic analyzer

A paramagnetic oxygen analyzer was installed in the gas pre-treatment system. Conditioning equipment was also required to remove liquid, dust, and gases that could interfere with the paramagnetic analyzer's ability to measure reliably. Despite this treatment, engineers noticed the analyzer's performance deteriorating over time.

GPro 500 oxygen sensor with sampling cell adaption



Analysis showed that the corrosive gases were not being removed entirely by the conditioning equipment and that the analyzer's expensive motherboard was being damaged. Engineers therefore looked for a more robust measurement system.

Reliable tunable diode laser oxygen sensor

They were immediately drawn to tunable diode laser (TDL) technology as it is a non-contacting measurement technique, so there is no possibility of process gases entering the sensor body. The available TDL analyzers in the marketplace were investigated and the Lenzing (Nanjing) Fiber engineers became particularly impressed with the METTLER TOLEDO solution.

Most TDLs are composed of two parts: a unit that outputs a laser beam of a frequency equal to the absorption frequency of oxygen molecules; and a unit that receives the laser light, analyzes it, and calculates the oxygen level in the process. The two units require very precise alignment, which is not easy to achieve.

The METTLER TOLEDO GPro 500 sensor, on the other hand, features a probe that protrudes into the gas stream. A corner cube at the end of the probe directs the laser beam back to the receiver/analyzer in the sensor head. Alignment is never required and because the laser beam travels twice through the process gas, measurement accuracy is very high. Also, the sensor has no moving parts. This means the analyzer requires no maintenance other than occasional cleaning of its optical windows and annual verification.

Unique process adaptions

The GPro 500 is available with a series of unique process adaptions that replace the standard probe. These adaptions greatly increase the range of applications suitable for the GPro 500. Lenzing (Nanjing) Fiber installed a GPro 500 with a sampling cell adaption. This has allowed them to use the GPro 500 along with their existing sample conditioning equipment. In addition, installation was conducted without any interruption to the gas treatment process.

Lenzing (Nanjing) Fiber are more than satisfied with their decision: "The analysis capability of the GPro 500 meets all our requirements. Furthermore, it's superior to the paramagnetic analyzer in terms of response speed and maintenance."

www.mt.com/TDL

Tunable Diode Laser Spectroscopy Booklet

Theory and Background

In this free booklet we discuss the theory of absorption spectroscopy and how TDL absorption spectroscopy analyzers are being employed in an increasing number of process and safety-critical applications.

The booklet covers a wide range of topics on TDL spectroscopy including:

- Laser spectroscopy theory
- Advantages of TDL spectroscopy in process gas analytics
- TDL application examples
- Signal processing techniques for TDL spectroscopy

Download your free copy of the METTLER TOLEDO Tunable Diode Laser Spectroscopy Booklet:

www.mt.com/tdl-booklet

METTLER TOLEDO Group
Process Analytics
Local contact: www.mt.com/pro-MOs

www.mt.com/pro

Visit for more information