

## pH measurement improves process safety at the DOMO Works in Leuna, Belgium

**In the new facilities in Leuna pH measurements play an important role particularly in the control of the oximation process. By using METTLER TOLEDO electrodes with integrated temperature sensor, temperature can now be measured in the process or during calibration exactly at the same point, significantly increasing process safety.**

After the DOMO group took over the Leuna site, it modernized the existing equipment and built a new plant for the polymerization of caprolactam to polyimide. A novelty for the Leuna facility is the spinning process downstream of polymerization, for the production of textile and carpet yarns.

Today, DOMO still strives to widen its portfolio. For instance, the company recently took over the polypropylene facility of the Basell Group in Rozenburg in the Netherlands. Caprolactam has been produced in Leuna for more than half a century, following the epochmaking breakthrough by the chemist Paul Schlack, in being able to make a synthetic fiber from caprolactam. To meet the ever rising demand for caprolactam, the production

capacity was continuously expanded, and in 1967 a new, larger plant was put into operation that still constitutes the nucleus of the production facilities in Leuna.

In a classic process, cyclohexanonoxim is arrived at through the oximation of cyclohexanon with hydroxylammonium-sulfate. This is then transformed into caprolactam by the Beckmann rearrangement method using oleum. Continuous monitoring of the process parameters and maintaining of set values is ensured by the use of various measuring systems with data transfer to a modern process control system.

The pH values play an important role in the process plant generally. For instance, oximation is controlled through this parameter. For many years, pH electrodes Type HA465 with Friscoylt-B



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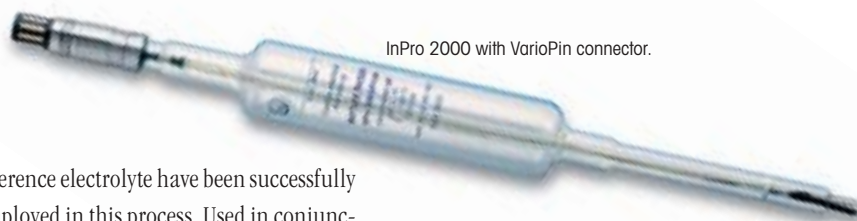
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InPro 2000 with VarioPin connector.

reference electrolyte have been successfully employed in this process. Used in conjunction with InTrac and InFit housings as well as the pH transmitter pH 2220X, (HART® communication as option) the complete installation has proved itself to be a fully reliable measurement system.

As the pH value relative to this process has to be kept within narrow limits, the process and calibration temperature necessary for temperature compensation of electrode slope is a decisive quantity for achieving improved measurement accuracy. Until recently, the temperature in the process was measured by a fixmounted Pt100 thermocouple which was however not located close enough to the pH electrode to make it suitable for calibration procedures. Moreover, the actual process temperature lies in the region of 82 °C (179.6 °F), whereas a calibration temperature of 25 °C (77 °F) was set on the transmitter, naturally only an approximation. For calibration, the electrode had to be removed from the housing and therefore assumed ambient temperature.

Following the introduction of the InPro 2000 electrodes with integrated temperature sensor, temperature measurements in the process as well as during calibration can now both be taken exactly at the same point, and the values passed to the transmitter for processing.

The use of the InPro 2000 with built-in Pt100 has contributed substantially to an improvement in the pH measurement accuracy and to overall process control.

InPro 2000 is equipped with the VarioPin connector (VP). The rugged construction of the VarioPin provides a watertight connection of a fixed cable with the convenience of a detachable cable, it allows quick connect and disconnect of the sensor from cable for easy maintenance and replacement. The IP68 certification ensures a dustproof watertight connection even in harsh environments.

Apart from the process sensitive pH measuring points, other loops (e.g. in the wastewater sector) will now also be converted to incorporate electrodes with an integrated temperature sensor. The requirements of those sectors where pH measurement is necessary can be fully covered by the electrodes InPro 4010 InPro 4250, InPro 3200 and InPro 2000.

# New InTrac retractable housings – when process safety is crucial!

The current family of retractable housings consists of several different models. Over the years, this particular range has become the most important type of housings in the METTLER TOLEDO portfolio, with successful applications in the chemical industry.

## The concept

The new, improved InTrac retractable housings offer enhanced reliability and operational safety due to a completely new sealing and bearing concept. These housings are dedicated to harsh and demanding applications in the chemical process industry where the use of pH/ORP probes with liquid or gel/polymer electrolyte is necessary to control and monitor the conditions of the process.

## The safety

Complete separation of the sealing and bearing functions improves operation and reliability even in highly contaminating applications. TRI-LOCK™ features several safety elements, consisting of sensor detector, safety immersion tube and positive position locks. All safety features aim towards avoiding unintentional release of process fluids. The sensor detector prevents the housing from being inserted without an

electrode or sensor being installed. The patented immersion tube seals the housing from the process at all times during operation, and the positive position locks always keep the electrode/sensor in an optimal measuring position.

## The versatility

METTLER TOLEDO also offers a broad range of sensor versions for other measuring parameters such as dissolved oxygen, conductivity, CO<sub>2</sub> or turbidity able to work with this housing.

## The cleaning

In combination with an EasyClean cleaning and calibration system, a fully automated measuring loop is available, bringing about an enormous reduction in operation costs.

The new InTrac housings are the first to be fully compliant with ATEX guideline 94/9/EC and suitable for installation in

hazardous areas Zone 0 and 1 as well as with PED 97/23/EC Category III. A wide selection of process fittings enables unrivalled flexibility for any installation purpose.

## The compliancy

The new retractable housings combined with the EasyClean automatic cleaning and calibration system form unique measuring systems. These systems accommodate specific maintenance objectives aimed at optimizing process safety as well as maintenance costs – even in the most demanding applications. ■

### Benefits of new InTrac housings

- Bundle of safety elements with TriLock™
- Fully automated measuring loop
- Fully compliant with ATEX
- Built for hazardous zones 0 and 1

## Automatic control with EasyClean

### Examples of a complete measurement loop

Retractable housing	Parameter	Electrodes/Sensors	Transmitter	Cleaning & Calibration System	
InTrac776 e	pH/Redox	Liquid electrolyte electrodes	pH 2100 e	EC150	
			pH 2500	EC200	
			pH 2220X	EC300	
			M 700*	EC300EX EC350	
InTrac777 e	pH/Redox	Gel and solid polymer electrolyte electrodes			
			DO	InPro 6800	O <sub>2</sub> 4100 e
			Conductivity	InPro 7001-VP	Cond 7100 e
InTrac 779 e	Turbidity	InPro 8100 InPro 8200			
					Trb 8300



InTrac 776 e  
(pneumatic type,  
made of PP).



InTrac 777 e  
(pneumatic version,  
made of PVDF).

# pH measurements in electroplating process

**The yardstick for quality assurance is the sum of current demands of market and customers, combined with technological advances in electroplating technology and environmental protection measures. The scope of analytical requirements demanded from businesses in the plating industry has increased substantially during recent years.**

## Quality aspects in galvanization

Galvanizers are suppliers to industrial sectors such as the automobile, aerospace, computer and semiconductor industries as well as to producers of electrical and electronic components. In the plating industry, product quality and environmental compatibility are given pride of place. Over and above regular checking of effluents, analytical procedures relating to the plating baths have taken on increasing importance. Decisive in being able to achieve high-quality end products are the individual characteristics of the relative process baths. A stable condition in these baths is all-important. Depending upon the actual type of production or on the requirements of ISO 9002, it may be necessary to check the various bath parameters several times a day or even better to carry out overall monitoring on a continuous basis. These various parameters are for instance, temperature, metal ion concentration,

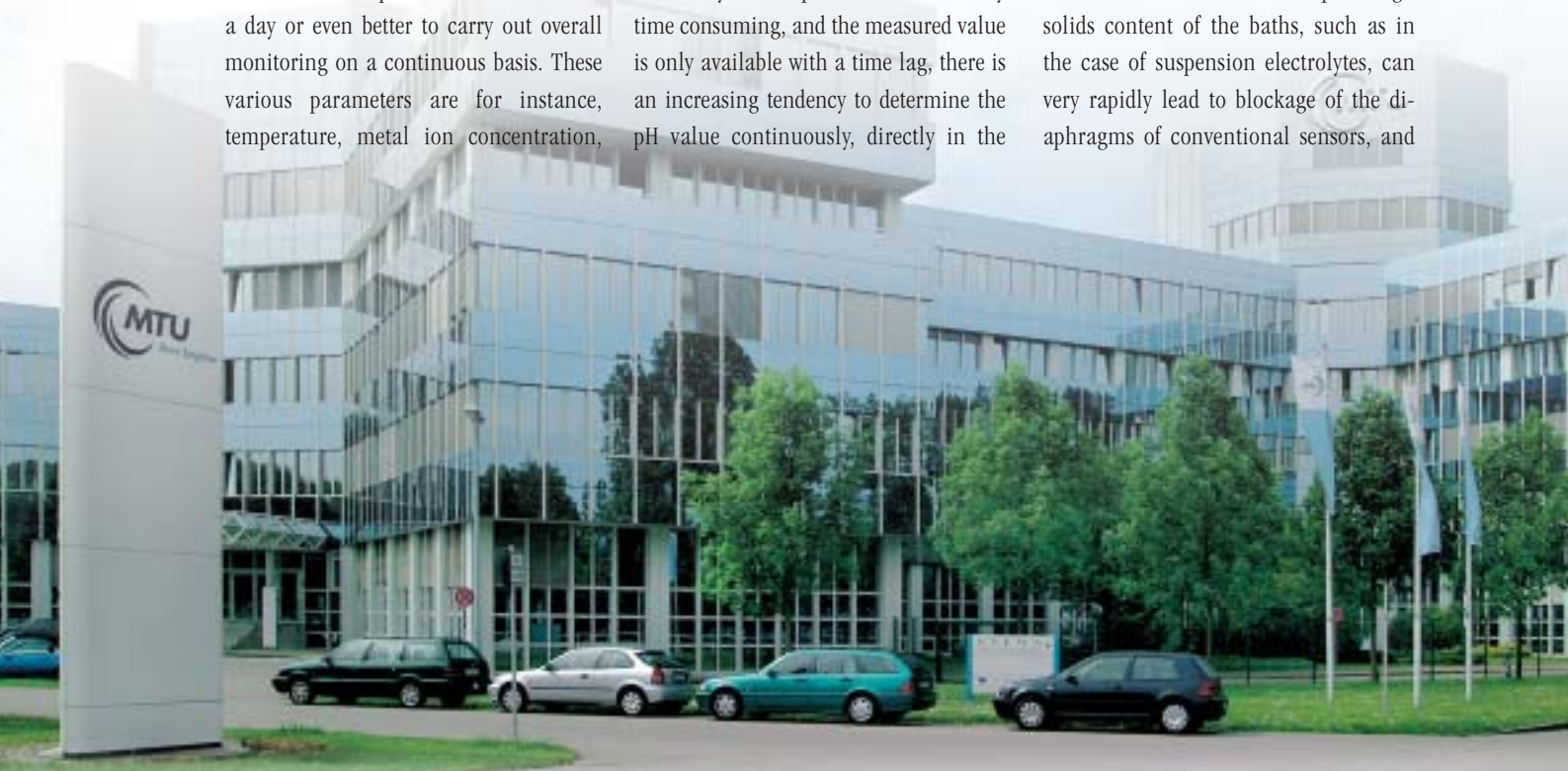
acid/base content or pH value, etc. In some process baths such as those containing chemical nickel, nickel sulfamate, bright nickel, and above all, suspension electrolytes (electrolyte containing solids), the pH value is an important quantity in any monitoring procedure. As the pH value in these baths is subject to continuous change, it is necessary to determine the pH parameter regularly (in suspension electrolyte, daily) and in the event of tolerance deviations, to be able to initiate immediate corrective measures such as the addition of acid or lye.

In the past, pH values were mostly measured offline, that is, a sample was taken from the bath and analyzed in the laboratory. As this procedure is extremely time consuming, and the measured value is only available with a time lag, there is an increasing tendency to determine the pH value continuously, directly in the

bath(s). Continuous measurement of the pH value clearly allows swifter reaction to any change noted, consequently providing improved process safety. At MTU in Munich, a company belonging to Daimler-Chrysler Aerospace, and Germany's leading aircraft engine manufacturer, a comparison test was made between continuous measurements in the above mentioned baths and standard laboratory (grab sample) measurements.

## Continuous measurements

Particularly problematic in continuous measurement procedures is the presence of strong electrical fields which could disturb the electronics of the signal amplifier. In addition it has to be taken into account that the often quite high solids content of the baths, such as in the case of suspension electrolytes, can very rapidly lead to blockage of the diaphragms of conventional sensors, and



consequently to erroneous measurement readings. A measurement system available from METTLER TOLEDO has proved to be especially suitable for direct measurements in plating baths. The measuring system for the tests at MTU consisted of the following components:

- transmitter pH 2100 (measuring range 0 – 14 and output 0/4 – 20 mA) for data acquisition,
- pH electrode HA-405-DXK-S8/120 (pH range 2 – 14 and temperature range 0 – 100 °C) fitted in
- InDip 550 housing made of PVC for immersion into the process bath. The electrode has a polymer (XEROLYT®) reference system containing KCl, and two open aperture junctions.

Experimental work in a first series of trials, the pH value of a nickel sulfamate bath was measured on a continuous basis over a period of several days and the readings compared with parallel laboratory sample measurements. It was shown that the continuous measurement values remained extremely stable and that there were practically no deviations from the measurement values established in the laboratory. In the second test series, measurements were taken in a bright nickel bath. In this instance, it became clearly apparent that changes in pH value in the bath occurred in relation to the throughput rate of components undergoing plating.



pH 2100e transmitter.



InPro 4250.

The reliability of the continuously measured values were confirmed by parallel laboratory measurements. In a third series, the pH values in the dispersion coating line were measured. Due to the particularly high content of solid particles ( $\text{Cr}_2\text{O}_3$ ), a certain amount of difficulty had been expected. But here also, it was possible to achieve stable and accurate continuous pH readings, with results verified in the laboratory.

### Assessment of the measuring point

The measurements taken continuously over a period of eighty days show that the system recommended by METTLER TOLEDO is indeed highly suitable for use in the continuous measurement of pH values in electroplating baths. The electrode delivered stable value in all of the baths included in the test program, and showed no signs of deterioration. It had to be recalibrated once a week. The system is remarkable for its robust features and ease of operation. The cost of installing such a pH measuring loop from METTLER TOLEDO could, in the instance described here, be recouped within the space of one month, alone through being able to forego sampling and laboratory analysis procedures. Connection to an automatic dosing system for pH correction is also possible, and this would provide still better process safety. Following the introduction of the InPro 4250 pH electrode, with an integrated temperature sensor and improved performance in the complete pH range 0 – 14, this electrode will provide even higher measurement safety and lower maintenance costs. This electrode has an upgraded polymer reference system (XEROLYT® PLUS) containing KCl and two open aperture junctions. A cleaning system, Easy Clean is to be recommended as an optimized application solution for efficient and safe measurement procedures in industrial processes. It provides fully automated unattended cleaning and calibrations of pH measuring electrodes in continuous processes, significantly reducing the maintenance costs. EasyClean is available in a range of models, each designed to satisfy a specific application.

### Beneficial test results

- stable pH value in all tests
- no sign of deterioration
- calibration only once a week
- ease of operation

# System Integration made easy

Analytical measuring systems from METTLER TOLEDO are particularly easy to link to modern process control systems and allow substantial cost savings for installation, configuration and maintenance. The communication protocols HART®, PROFIBUS® and FOUNDATION Fieldbus are fully supported by a variety of transmitters from METTLER TOLEDO.

METTLER TOLEDO offers measuring systems with standardized interfaces for HART®, PROFIBUS® and FOUNDATION Fieldbus. This enables full exploitation of the advantages of digital communication for ease of instrument parameterization and maintenance. Process and instrument data are recorded via the interfaces, using software in accordance with Guidelines 21 CFR Part 11.

## HART® integration

METTLER TOLEDO transmitters use registered device description (DD) technology for the hand-held instrument HART® Communicator 275. Various instruments are also suited to the Management Software included in process control systems, e.g. AMS™ Suite from Emerson.

METTLER TOLEDO transmitters available for HART® are:

- pH 2100e and pH 2220
- O<sub>2</sub> 4100e and O<sub>2</sub> 4220
- Cond 7100e and Cond 7220
- Cond Ind 7100e and Cond Ind 7220

## PROFIBUS® PA integration

PROFIBUS® PA (Process Automation) is a standardized, open digital communication system for all fields of application in process automation. PROFIBUS® PA (EN 50 170 P4) is a further development of PROFIBUS DP and includes two special features for process automation, namely bus powering and communication of field units using 2-wire technology. The transmitter units listed are also suitable for use in hazardous areas.

METTLER TOLEDO instruments are fully integrated into PDM (Process Device Manager) from SIEMENS.

METTLER TOLEDO offers a choice of PROFIBUS® transmitters including:

- pH 2100 PA
- O<sub>2</sub> 4100 PA
- Cond 7100 PA
- Cond Ind 7100 PA
- M 700 for pH, O<sub>2</sub> and Cond

## FOUNDATION Fieldbus integration

FOUNDATION Fieldbus (FF) is also a vendor-neutral, standardized and open digital communication protocol for all application fields. From the homepage of fieldbus.org, all relative integration data sets for the new FF transmitters from METTLER TOLEDO, pH 2100e and O<sub>2</sub> 4100e, can be downloaded.

METTLER TOLEDO transmitters suited to FOUNDATION Fieldbus are:

- pH 2100e
- O<sub>2</sub> 4100e
- Cond 7100e
- Cond Ind 7100e

Emerson has also tied in METTLER TOLEDO transmitters to its AMS™ Suite software.

## Complete product offering

With HART®, PROFIBUS® and FOUNDATION Fieldbus, METTLER TOLEDO now has at its command, state-of-the-art digital fieldbus protocols able to be linked to the respective process control system without any difficulty. ■



pH 2100e transmitter.

HART® is a registered trademark of HCF  
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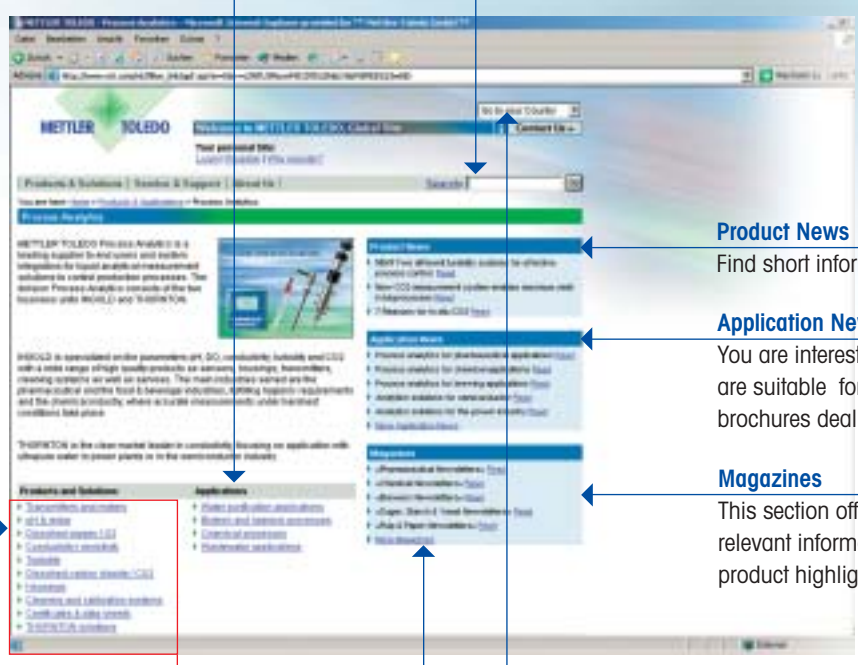
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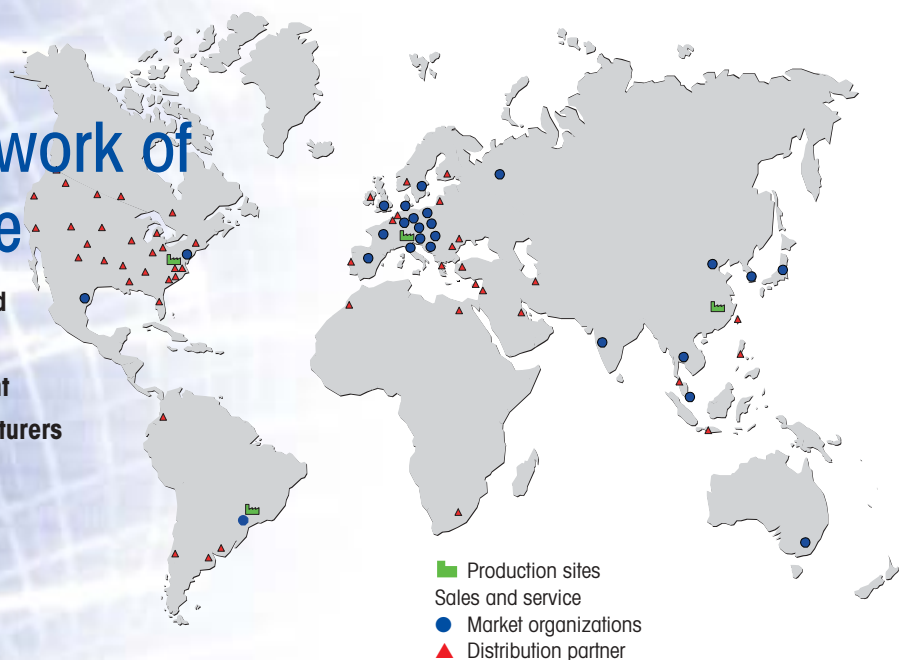
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## Distribution network

Based on its several global production sites, more than 20 market organizations, and numerous sales representatives,

METTLER TOLEDO maintains a wide distribution network all over the world. Satisfaction of our customers is based on three pillars:

## Consulting

With the knowledge of our experts, we support you in finding the best solution, including planning, product selection, and installation of our measurement solution.

## Top products

A complete range of products and systems to meet measurement requirements

## After-sales service

With our customized, lifelong service management, we are able to assist in maintaining measurement loops throughout their whole life-cycle.

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- Faster logistics
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