



INGOLD

Leading Process Analytics

Inductive conductivity-controlled acidity in zinc leaching process

One of the key aspects in the zinc leaching process is to maintain stability in the total acidity of the waste solution. The multiparameter transmitter M 700 and the conductivity sensor InPro 7250 showed superior performance and saved costs.

Customer background information

Our customer is one of the five largest Zinc producers in the world and the largest in Latin America. In 2004, significant investments were focused on production improvements.

Type of application

Zinc is usually found in nature in combination with Sulfur (ZnS). To be made marketable, Zinc sulfided minerals are concentrated by flotation. The most commonly used process around the world to concentrate zinc is based on hydrometallurgy, namely electrolytic zinc deposition. As the entire process is water-based, high importance is attached to pH of the associated concentrations, especially H_2SO_4 , for plant productivity. Refer to the basic process flow chart Fig. 1. Total acidity was

previously determined through lab analyses, and then controlled by greater or smaller quantities of waste solution being added to the process via manually actuated valves.

Customer's expectations

The project requirements of the customer aimed for total acidity stability to ensure higher output and quality. Conductivity control had previously been attempted by using the standard sulfuric acid curve, but it did not produce good results due to the presence of many contaminants that impaired the use of such standard curve.

METTLER TOLEDO's solution

After a detailed analysis of the customer requirements and the actual situation, the following measurement system was



suggested and thereafter also implemented:

- M 700 transmitter with PID controller, inductive module and Concentration Chart
- InPro 7250 HT conductivity sensor
- InDip housing

Benefits of this solution

The uniqueness of the “Concentration Chart” feature enabled METTLER TOLEDO to conduct a survey on the total acidity behavior in comparison with the conductivity at diverse temperatures and then input its respective results into a matrix, thereby allowing direct measurements in the unit already known by the customer. This procedure surely contributed significantly to the purchase decision.

The graph Fig. 2 visualizing the matrix mentioned above shows a comparison between the standard curve of H_2SO_4 70 °C/ 158 °F and 100 °C/212 °F and the curve resulting from different temperatures. The

selection of the best curve was aided by another feature of the M 700 – the Smart-Media™ data recording. On a daily basis, the customer provided the equipment reading data and the total acidity lab

the waste solution dosing but also the better performance of the leaching process directly related to the lower loss of zinc in the process. The customer was absolutely satisfied not only with the equipment reli-

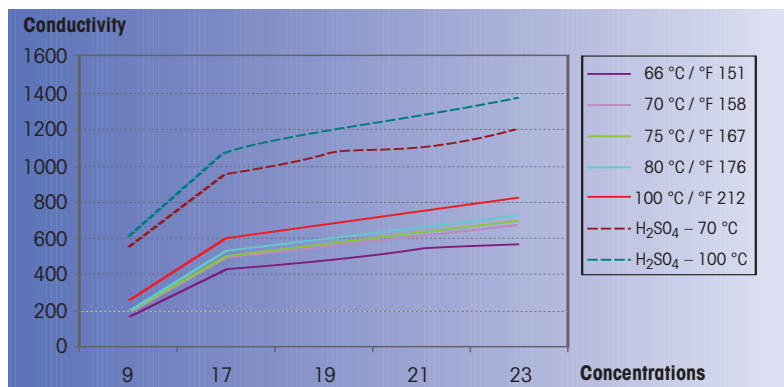


Fig 2: Total acidity behavior in comparison with the conductivity at diverse temperatures.

analyses for METTLER TOLEDO to adjust the graph point-by-point and correct any deviations until fully reliable measurements became possible.

Payback and customer satisfaction

The financial benefits are very meaningful thanks not only to the savings made on

ability but also the support we provided to develop a curve specific to their process.

The customer is now considering expanding INGOLD measurements to cover new application lines relative to peroxide (H_2O_2) concentrations.

► www.mtpro.com/conductivity

► www.mtpro.com/transmitters

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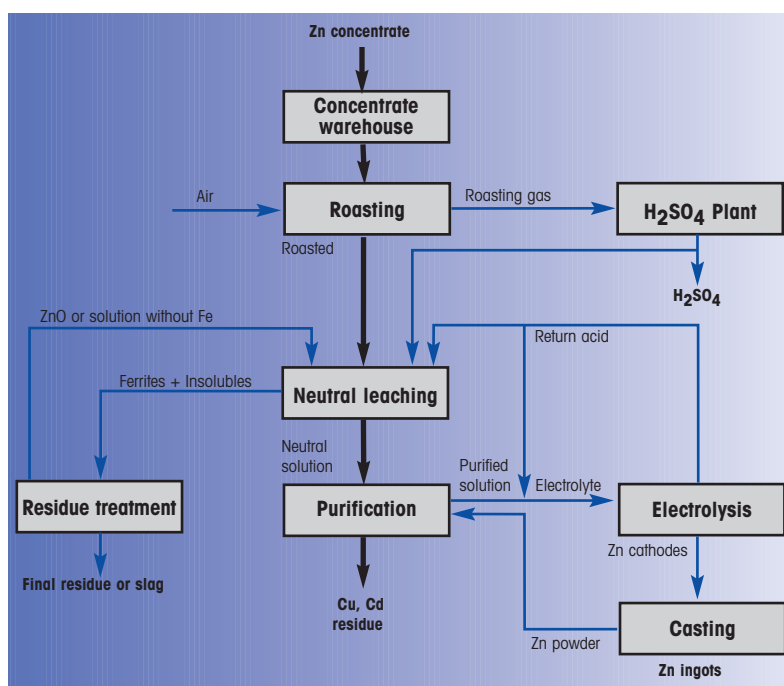


Fig. 1: Simplified flowchart of a zinc electrolytic plant.

With ISM[®] into the future of process analytics

METTLER TOLEDO's "Intelligent Sensor Management"[®] concept allows easier handling of process measuring systems, from initial installation, over maintenance, right through to change of sensor. This leads to lower maintenance costs and reduced risk of sensor failure.

Quick and easy installation thanks to "Plug and Measure"

- Sensors are immediately recognized during installation.
- Sensor-specific data is stored in the transmitter.
- Operational availability of a measuring point within seconds.

Pre-calibration of pH-electrodes and oxygen sensors in the lab

- Sensors can be pre-calibrated in the laboratory.
- Calibration at point of measurement is no longer necessary.
- The sensor can be replaced and operates within seconds, saving time as well as increasing operational availability.

Ideal tool for sensor documentation

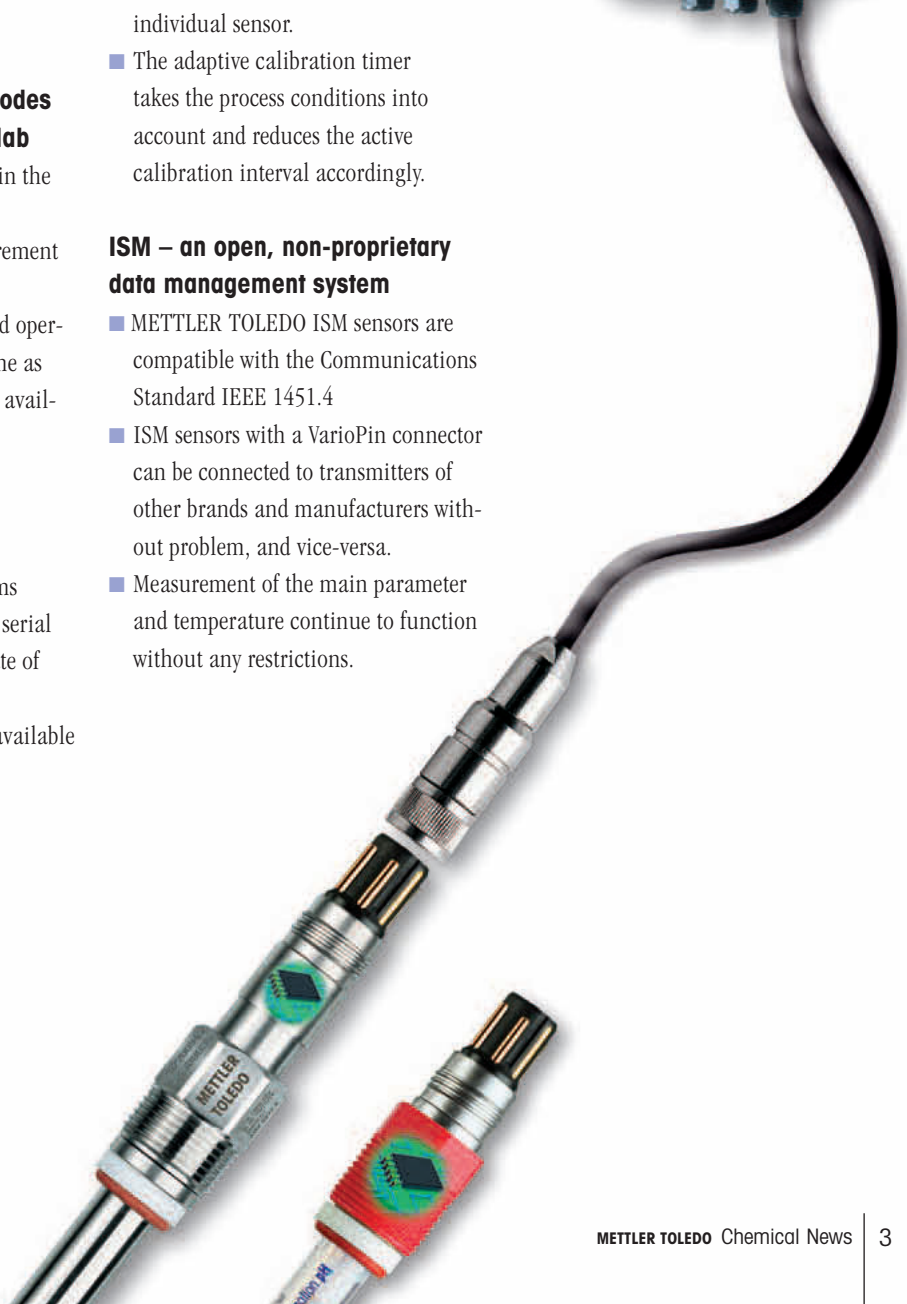
- ISM-enabled measuring systems recognize manufacturer, type, serial number, order number and date of last sensor calibration.
- Previous calibration datasets available for trend analysis.

Optimized maintenance with sensor wear indicator and adaptive calibration timer

- Measurement data are continuously monitored in order to obtain information on sensor wear.
- Operating hours are recorded for each individual sensor.
- The adaptive calibration timer takes the process conditions into account and reduces the active calibration interval accordingly.

ISM – an open, non-proprietary data management system

- METTLER TOLEDO ISM sensors are compatible with the Communications Standard IEEE 1451.4
- ISM sensors with a VarioPin connector can be connected to transmitters of other brands and manufacturers without problem, and vice-versa.
- Measurement of the main parameter and temperature continue to function without any restrictions.



▶ www.mtpro.com/pH

Three pH measuring loops guarantee long term accuracy and reliability

High pH-stability in the vessel guarantees keeping waste limits within the required specifications. A system of three pH measuring loops brought Degussa Fine Chemicals very satisfying results.

Degussa Fine Organics

Degussa Fine Organics are is of Degussa's Fine and Industrial Chemicals Division. Degussa produces highly profitable specialty chemicals and is market leader in this field. They maintain over 300 plants world wide, employing 45000 people generating over EU 11 billion sales in 2004.

Degussa's demand for determined pH limits of waste

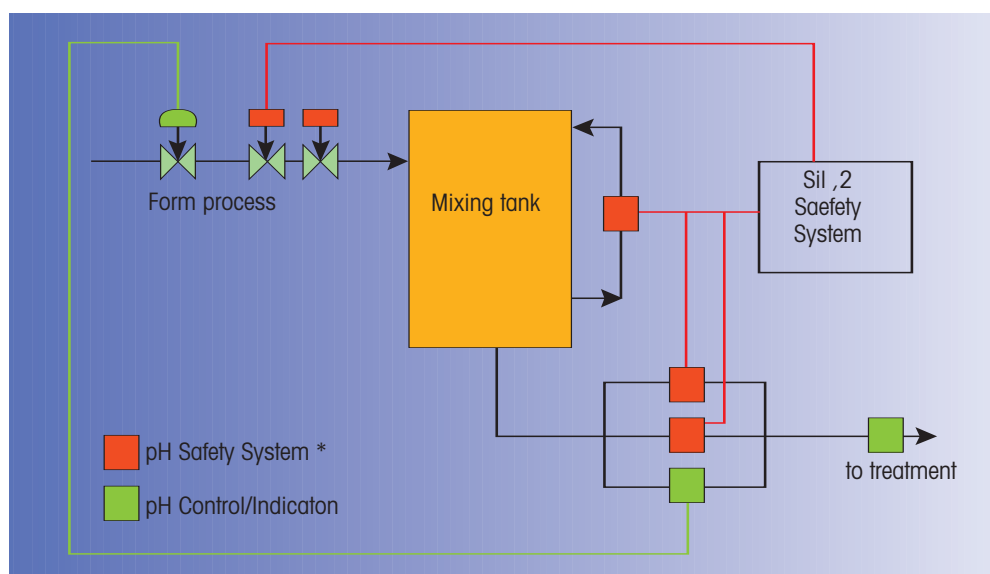
Degussa Fine Organics at Seal Sands in the North East of England have installed METTLER TOLEDO in-line pH measurement systems with automated cleaning to ensure that waste passed off site is within the pre determined limits set by a third party treatment company.

Changing conditions require detailed clarification

pH was defined as one of the most important analytical parameters to ensure that the waste would be within the required specification and would not cause any degradation of the treatment company's pipe transporting it to the treatment plant some distance away. The waste, which comes from many different processes, is collected in a storage vessel where it is blended together before leaving site. In order to fully understand and control this waste collection procedure it is important to know the pH value of the liquid in the tank (this may change greatly over time depending upon which process it comes from), the pH value of the mixed waste as well as that of the waste as it leaves site.

Correctly placed measuring loops for optimal surveillance

It was decided that due to the fact that the greatest variability in pH would be seen at the outlet from the tank. The 3 loops would be installed on a redundancy safety voting system to ensure accuracy of measurement. Two of the safety loops were installed on the outlet and one on the recirculation leg of the vessel to measure the homogenized liquid. A normal control loop was installed alongside the 2 safety loops on the tank outlet and the fifth in the pipe which leaves site to give the final measurement. If the control pH deviates from set parameters there are valves on the outlet which switch from offsite transfer to tank recirculation until the desired pH is achieved. If the pH value falls even lower than the control setting then the Safety System will close the inlet to the tank.



* InPro 2000, InTrac 776, pH 2100e, EasyClean 200

High accuracy and reliability determine product selection

InPro 2000 electrodes were chosen as they could offer the most accuracy and reliability in the changing composition of the waste and were installed in InTrac 776 housings.

As it was known that the liquid would foul the glass membranes of the electrodes, an EasyClean 200 was installed at each measuring point to automatically withdraw the electrodes, flush them with water and clean them with hydrochloric acid before washing the acid away with water and re-inserting the electrode.

The pH 2100e transmitters are programmed to stagger the cleaning cycles so that only one electrode is removed from the process at a time. During the cleaning process the output from the transmitter is held at the last measured value.

Fixation of a constant pH-value

Once long term accuracy and reliability had been ensured the re-circulation pH measurement could be used to control pH correction in the storage vessel.

Further problems to be solved

Since solving this difficult measurement Degussa Fine Organics have come to rely on METTLER TOLEDO for many of its in-line and R&D analytic measurements including pH, conductivity, O₂ concentration in inert environments and turbidity in crystallization.

Customer benefits

- Highest measuring accuracy and reliability with InPro 2000 pH electrode
- Automatically and individual cleaning at each measuring point of electrode with EasyClean 200
- Staggered cleaning cycles with pH 2100e transmitters

InPro 2000 liquid-electrolyte pH-electrode

- Used as problem solver in a wide range of applications
- Automatic temperature compensation during calibration and operation
- Excellent response time and accuracy
- No fouling of diaphragm in sulfide bearing media due to silver-ion trap



EasyClean 200

- Automatic sensor flushing with water
- Additional cleaning with cleaning solution
- Free choice of cleaning agent
- Especially designed for heavily contaminated processes



InTrac 776 retractable housing

- With built-in flushing chamber
- Electrodes can be cleaned and recalibrated without interruption of the ongoing process
- Designed for chemical applications manual or pneumatic versions



pH 2100 transmitter

- Reliable and safe transmitter in operation
- Self-explanatory user interface
- Several possibilities of diagnosis



▶ www.mtpro.com/pH

▶ www.mtpro.com/cleaning

Longer lifetime of pH-electrodes with the Cleaning System EC 350

To achieve high-yield production of pure cobalt hydroxide, the pH-value has to be measured accurately and continuously. Lifetime of the pH-electrodes improved remarkably when regular cleaning took place.

The company

Lanzhou Jinchuan Metal Material Technology Co. Ltd. is a hi-tech chemical company producing cobalt oxide (Co_3O_4), a substance widely used in rechargeable batteries.

This subsidiary company of Jinchuan Group Ltd., a large integrated, non-ferrous metallurgical and chemical engineering enterprise in Jinchang City, Gansu Province, P.R.China, employs nearly 300 workers. The output of nickel- and platinum-group metals together account for more than 90 % of the total production in China, and 1,200,000 tons of chemical products are manufactured by the Jinchuan Group Ltd. annually.

The application

The chemical process reaction presented itself as follows:

- $\text{Co}(\text{NO}_3)_2 + \text{NaOH} - \text{Co}(\text{OH})_2 + \text{NaNO}_3$
- Temperature: 60-70 °C; 140-158 °F
- Pressure: 1bar; 14.5 psi
- Batch time: 8 hours

As a key parameter, the pH must be adjusted by ammonia to a value of 8 to 9. Precision of pH measurement for the control system should be ± 0.05 . If the pH value is controlled incorrectly, then the quality of the end product becomes unacceptable due to characteristics such as poor degree of purity and different size of grain, accompanied by low yield.

Customer's requirement

It was very difficult to measure the pH during the reaction because the fine powder of $\text{Co}(\text{OH})_2$ easily covered the membrane and diaphragm of the pH electrode. Whenever such situation occurred, the pH value was inaccurate. The customer required that the pH sensor be rinsed and calibrated frequently, for instance twice per batch.

- Buffer: pH 7.00 and 9.21
- Detergent: 0.1N HCl

Sensor performance convinced the customer

Our pH sensor InPro 4800 had been used during the pilot-scale trials without an EasyClean system. Comparing this pH sensor with those from other companies, the customer was particularly satisfied with the performance of the InPro 4800.

Because of the necessity for frequent rinsing and calibration of the sensor, METTLER TOLEDO recommended use of the EasyClean 350 system. The transmitter pH 2100e which matched the EasyClean 350 was part of the offered solution. The material of the wetted parts of the housing would have to be made of PVDF due to the aggressive detergent solution of 0.1N HCl. The housing InTrac 777e was therefore selected.

Eight vessels of each 50m³ volume were installed in a new production plant, so that the customer accordingly needed 8 pH measurement loops.

Strong reasons for METTLER TOLEDO

After rigorous evaluation of competitive offers it was determined that the solution offered by METTLER TOLEDO was the one fitting the customer's needs best regarding handling and technical requirements.

Over and above this, the customer also recognized that INGOLD service offering was indeed of superior standing.

METTLER TOLEDO solution

The following products were installed to meet the customer's requirements:

- pH 2100e transmitter
- InPro 4800 pH electrode
- InTrac 777 SLI retractable housing
- VP6-ST VarioPin cable
- EasyClean 350

The customer's benefits

The customer is extremely satisfied both with service from INGOLD and with the installed products. In particular, great importance was given to the following points:

Continuous operation

The incorporation of the EasyClean system made it possible for the customer to carry through the reaction on a continuous basis.

Longer lifetime of the pH sensors

The lifetime of the pH sensors (InPro 4800) during pilot-scale tests was 2 months, in the new plant however, it even reached 4 months on average.



InPro4800.

Higher yield

There was a remarkable improvement in yield after our loops had been installed.

Measurement accuracy

Accurate pH measurement values were achieved by our loops, enabling a high degree of purity of the end product. It was possible to control the pH so as to reach a value of 8.70 ± 0.05 within 15 minutes after start of the reaction. Subsequently the pH value of 8.70 ± 0.02 was maintained right through until the end of the reaction.

Consistent powder grain size

Formation of tiny, equal-sized grains was ensured due to accurate measurement and control of the pH value.

Appreciable savings in manpower

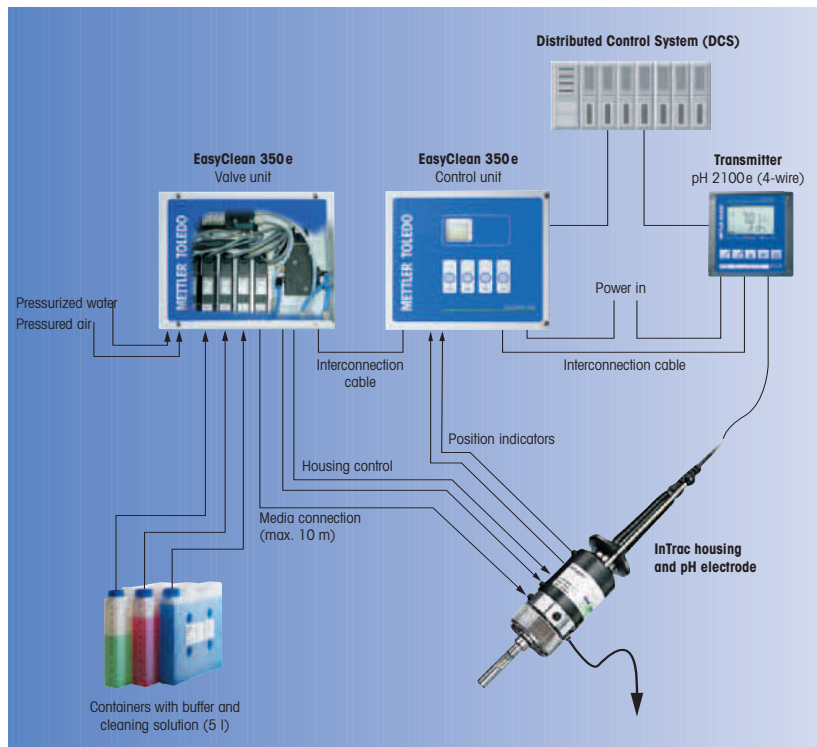
EasyClean 350 allows to effectively reduce the maintenance costs of measuring points while increasing the operational safety at the same time.

EasyClean 350 e is a robust and proven fully automated system which works reliably even in high maintenance measuring points.

InPro 4800 – top-class electrode for especially challenging chemical processes:

- Extended-life electrode reduces costs involved for maintenance and replacement.
- Reliable measurement under the harshest process conditions, even at high temperatures.
- The long, pressure-compensated diffusion path permits use in particularly high-pressure applications.
- Enhanced measurement accuracy due to the built-in temperature sensor and Vario Pin® (VP) connector.

www.mtpro.com/pH



Outstanding digital communication capabilities

METTLER TOLEDO "Advanced Line" transmitters offer HART®, PROFIBUS® PA and FOUNDATION Fieldbus® (FF) communication for measurements of pH, Redox (ORP), DO and conductivity.

The "advanced line" transmitters feature built-in diagnostics to provide continuous self-check of both sensor and transmitter, and allow users to perform proactive maintenance. A large LCD display with plain text and pictographs guarantees user-friendly handling and total control of all transmitter functions.

Designed for high reliability and safety, the "x100e PROFIBUS PA and FF" transmitters are available as intrinsically safe versions for operation in hazardous areas and are in accordance with the FISCO (Fieldbus Intrinsically Safe Concept) model. Fieldbus installations are employed worldwide in a wide range of applications in the chemical, petrochemical and pulp and paper industries as well as in pharmaceutical and food & beverage processes.

Fieldbus transmitters offer the benefits of digital communication such as a) convenient set-up and configuration of the transmitter from a central control room, or b) field diagnostics via the fieldbus.

With a broad-based portfolio of transmitters with HART, PROFIBUS PA and Foundation Fieldbus communication, hazardous or non-hazardous areas versions, METTLER TOLEDO enables full integration of your measuring system into a PLC.



pH transmitter 2100e PA.

► www.mtpro.com/transmitters

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