

Process Analytics is the application of analytical science to the monitoring and control of industrial processes. The goal is always to both control and optimize the performance of processes or products. Key factors are productivity, quality, cost, and reduction of emissions to the environment. Depending on the type of problem, a distinction is made between off-line, at-line and in-line analyses.

## Process Analytics



## Instrumentation for process control

All industrial processes need to be controlled to reduce process variation that causes lack of quality. Measuring is an integral part of any control scheme. Fundamental questions related to these measurements are: what to measure, when (at what point in the process) and how (which instruments to use). Since these questions are connected, a systematic approach is required to tackle the problems simultaneously.

In collaboration with the University of Amsterdam, we use dynamic process models to help answer these questions. The basic idea is to determine to what extent different types of disturbance produce unwanted process variation and to find a measurement configuration that optimally monitors this variation. The properties of the measurement device can then be matched to the requirements of the process in terms of accuracy, speed and cost. We help our customers in selecting the optimal measurement equipment and, if required, in adapting the equipment to specific needs. We also develop and maintain the calibration procedures that ensure that these analysers produce reliable data at all times.

Typical examples of recent projects in this field are:

- development of a leak tester for polymer LEDs based on the measurement of luminescence decay;
- contribution to the development of a fast in-line leak tester based on mass spectrometry for a lamp factory in France (see fig. 1).



Fig. 1: Development of a fast in-line leak tester based on mass spectrometry as part of a lamp manufacturing line.

## Troubleshooting and failure analysis

Failure analysis in the process industry is a team effort. Once a project team is assembled and critical data are collected, the problem must be analysed. In the Root Cause Failure Analysis (RCFA) methodology, several steps are distinguished:

- stating the failure event;
- stating the failure modes;
- hypothesizing;
- verifying hypotheses;
- determining underlying causes (physical, human and latent).

The team asks a series of 'How Can?' questions to come up with their hypotheses. Procedures such as Failure Modes & Effects Analysis (FMEA), Fault Tree Analysis, or Failure

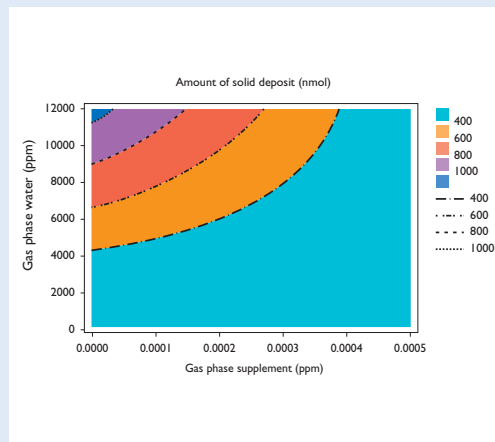
Modes, Effects & Criticality Analysis (FMECA), as well as prior knowledge and experience, can help to determine potential failure modes. Subsequently, data and samples from the field have to be used to prove or disprove the analysis team's hypotheses. Often small designed experiments are also performed to assess the effects of selected factors. Process Analytics specialists participate in failure analysis teams because of their expertise in analytical techniques and statistical data analysis. In addition, we have a broad experience with failure analysis problems from a wide variety of fields. This allows us to suggest solutions from different industries that might otherwise escape attention.

Fig. 2: Contour plot illustrating the amount of unwanted solid deposit as a function of gas composition. Water appears to have a detrimental effect; the gas phase supplement results in a beneficial effect.

Once all hypotheses have been proven or disproved, we can assess whether the underlying causes are physical, human or latent. These are the three main categories of problem or failure causes. Finally, findings and solutions must be communicated to the decision-makers. We provide detailed reports to help decision-makers understand the effectiveness of the failure analysis and the recommendations given.

Typical examples of recent projects in this field are:

- finding the root cause of lifetime problems for a US manufacturer of high-power LEDs for lighting applications;
- successfully investigating the origins of contaminant deposits on lenses in illumination systems (see fig. 2);
- participating in failure analysis teams to improve emission robustness in electron emitters for cathode-ray tubes.



### Rapid lab-scale experimentation

Development of new products and processes requires a great deal of experimentation. To improve the cost-effectiveness of product and process development, it is imperative that problems are recognized and solved at an early stage, when experiments are relatively inexpensive. Thermal-analysis techniques in combination with experimental design procedures (DoE, see fig. 5) are excellently suited for rapid screening experiments. Thermal analysis refers to a group of techniques in which a property of the sample is monitored against time or temperature, while the temperature of the sample, in a specified gas atmosphere, is programmed.

Properties monitored can be the mass of the sample (thermogravimetric analysis, see fig. 4), the heat consumed or released (differential scanning calorimetry) or the concentrations of gases evolved (evolved-gas analysis, see fig. 3). Using different instruments, various sample types (powders, thin layers, etc.) can be measured. Mass changes of less than 1 nanogram can be detected using a quartz crystal microbalance. As samples and gases can be changed quickly, an initial screening of the effects of varying the process conditions and product compositions can be performed within days.

Typical examples of recent projects in this field are:

- optimizing the thermal processing of plasma display screens;
- screening processing conditions for the development of lamp coatings;
- analysis of corrosion problems in LCD manufacture by measuring permeation of water through photoresists.

Fig. 3: Monitoring gas phase contaminants in burning fluorescent tubes using mass spectrometry.

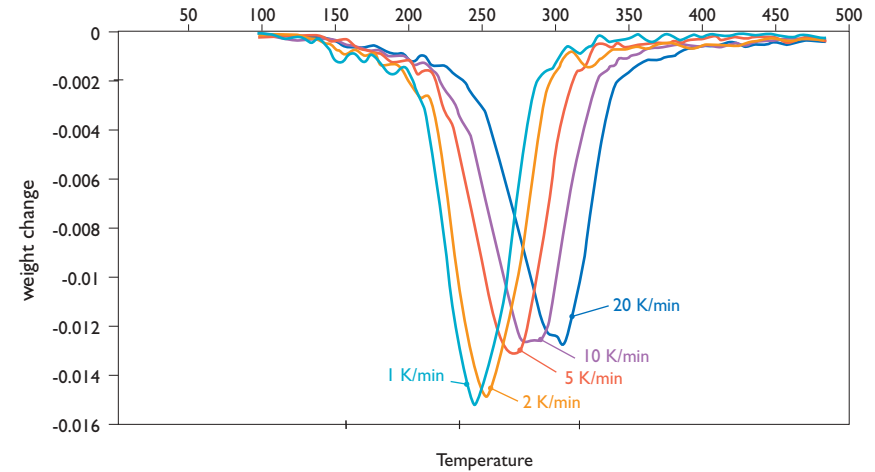


Fig. 4: Weight change vs. temperature during rib frit de-binding. In plasma screen production, the ribs that separate the pixels are formed by screen-printing of a glass frit (paste). In a subsequent step, organic binders in the paste need to be removed. Optimizing the processing time for the rib frit de-binding step is important in lowering the cost of plasma screen production.

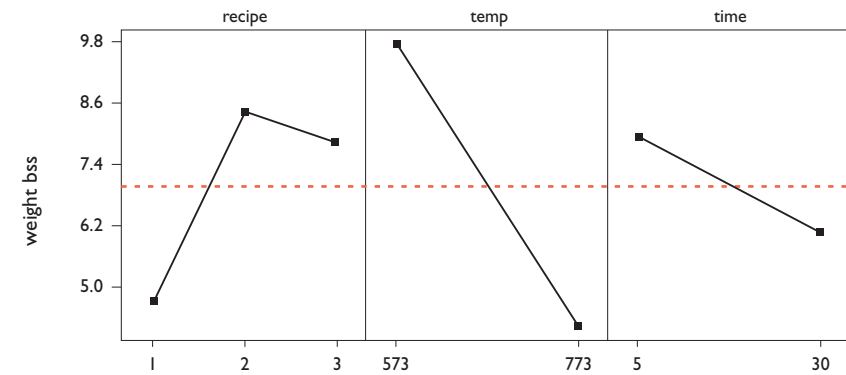


Fig. 5: Main-effects plot, showing the effects of recipe, temperature and processing time on the weight loss of a lamp coating material. The use of designed experiments (DoE) enables the experimenter to study the influence of factors and find optimum conditions using a minimum effort.

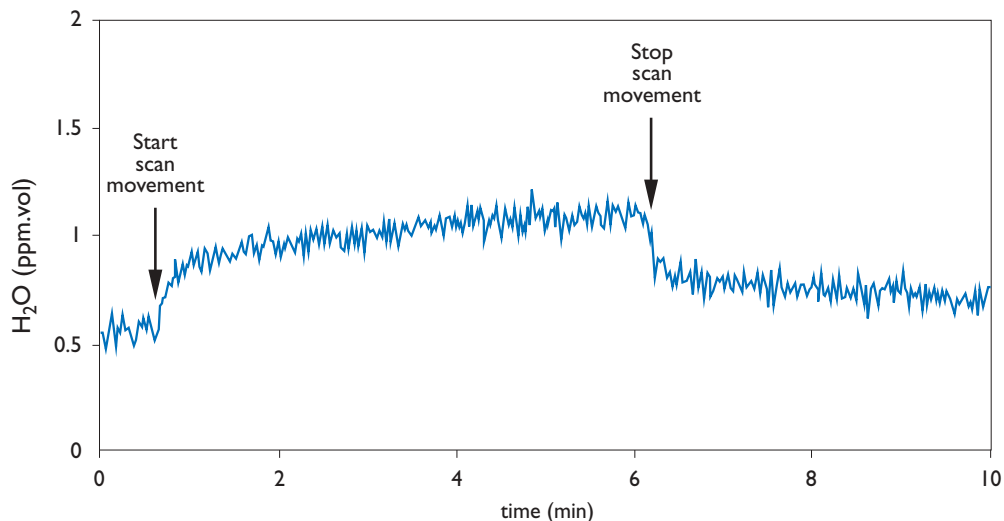


Fig. 6: At-site measurement of fast changes in gas-phase water content at (sub)ppm level.

#### At-site sampling and measurement

Quality and contamination control problems typically require sampling and measurement to be performed at the location where the problem occurs: in the factory. In case of liquids and solids, samples are generally collected in the factory and subsequently analysed in the central laboratory. For gas analysis, we have a number of (trans)portable instruments (see fig. 7) that allow direct measurement in the factory down to the high part-per-billion (ppb) levels (see fig. 6). Analytical techniques include mass spectrometry, infrared spectroscopy, near-infrared diode laser spectroscopy, electrochemical and capacitive sensing. For lower concentrations, the detection limits of these techniques are no longer sufficient.

We have developed proprietary sampling techniques that enable determination of airborne molecular contamination down to the low part-per-trillion (ppt) levels. Sample kits, containing specially prepared and packaged filters and air sampling pumps, are sent to the factory. Factory staff can perform sampling using the enclosed manual. The samples are then returned in a special container to avoid contamination during transport. Depending on the contaminant to be determined, gas chromatography - mass spectrometry (GC-MS), ion chromatography (IC) or laser ablation - inductively coupled plasma - mass spectrometry (LA-ICP-MS) are used for the analysis of the filters.

Typical examples of recent projects in this field are:

- determination of the efficiency of amine scrubbers for a ceramics production plant in Belgium;
- validation of the gas composition in glove boxes used in lamp production lines;
- determination of airborne total phosphorus in semiconductor production facilities in the USA and Taiwan;
- measuring fast changes in water content at (sub)ppm level for a manufacturer of lithographic instruments.

Fig. 7: Mass spectrometer that allows fast determination of elemental composition.



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### Application Note 15

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#### What we offer

- general consultancy (confidential, free of vendor bias)
- instrumentation for process control
- troubleshooting and failure analysis
- rapid lab-scale experimentation ('early problem solving')
- at-site sampling and measurement
- contamination control

