Process Analytical Technology/PAT

Process Analysis in the Chemical Industry

FT-NIR Spectroscopy as PAT

PAT has already been used in practice in the automobile industry for many years: A 100 % process validation and control system as the basis for process quality control and cost effectiveness. In the meantime, it is no different in the chemical industry. It is just that here the processes are not large open production lines, but are often sited within difficult to access processing tanks, sometimes under extremely high pressure and temperature conditions and often using aggressive media. Classical testing methods, operating procedures, or general tools for process control cannot always be used.

The control of processes has to take place at a molecular level; therefore use of modern Process Analytical Technology (PAT), e.g. using FT-NIR spectroscopy, is necessary. This allows for much greater production efficiency within many industries and production with a more constant quality.

FT-NIR spectroscopy is ideally suited for the direct monitoring of chemical reactions, as well as the quality assurance of intermediate and final products in chemical production plants. Modern analytical techniques can be used in all phases of production. Thus, it is possible to transmit information about single processes over long distances directly from a process tank or bypass using quartz fibre optics. The information can be evaluated at a distance from the process, away from extreme ambient conditions, such as high pressures or temperatures.

So that the information can be constantly evaluated in real time, immersion probes or measuring cells are used, which are designed to work reliably under extreme conditions. Different materials can be used for the immersion probes, depending on the chemical composition of the process. Many different materials are available, including various grades of stainless steel, robust Hastelloy alloys, or even extremely resistant tantalum.

The optical materials used in immersion probes and measuring cells can also be adapted to match the process. Quartz and sapphire excel by their ultra-high degree of precision. Sapphire, in particular, is frequently selected by customers because of its chemical resistance. The required immersion probe or measuring cell can be selected via a modular design principle and simply adapted to the individual process conditions prevailing on site.

Frequently used application areas for FT-NIR spectroscopy with immersion probes and measuring cells:

▪ Quality assurance: intermediates or final products are checked for purity, e.g. water content etc.
▪ Process monitoring: the material composition of intermediates and final products can be determined quantitatively
▪ Understanding of reactions: new processes can be monitored analytically in real time during the development phase
▪ Goods received inspections: delivered goods or products, for example, can be identified by their NIR spectra (fingerprint), in order to avoid faulty filling of storage tanks or material mix-ups

In addition to chemical production, these areas of application can also be found in the plastics and polymer industry, the food industry and many other industries.

A very frequent application in the pharmaceutical industry is the determination of the cloud point in the development of active substances for pharmaceuticals. In order to achieve the next phase of approval, active substances have to undergo various tests. An important test is the determination of the cloud point according to temperature. Normally, conventional turbidity measurements are used. As soon as a certain temperature is reached, the active substance begins to crystallise and the turbidity increases.

This is a constant process, in which the turbidity increases only gradually. It is very difficult to determine exactly at which temperature the cloud point is reached. By using an immersion probe coupled to a spectrophotometer it is possible to determine very exactly the temperature of the turbidity point in a simple and accurate way. At the very start of crystallisation, the absorption spectrum of the solution changes significantly. This abrupt and easily visible change in the optical spectrum allows very accurate monitoring of this process to be made.

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