



Challenge

Use of online measurement technology for more efficient measurability in processes with a spectroscopically non-detectable reactant.

Scope of Application

Continuous monitoring and precise reaction endpoint determination in purification processes

Area of Application

- # Monitoring of purification processes
- # Production of active ingredients
- # Chemical Industry
- # Pharmaceutical Industry

Method

- # Process Analytical Technology
- # Spectroscopy
- # Real-time measurements
- # Online Spectroscopy

More efficient reaction monitoring through online measurement techniques, even for processes with spectroscopically inactive components

Online process control for precise reaction endpoint determination in a purification process

> Situation

Online measurement methods enable reliable reaction tracking in real-time

The analysis of substances using spectroscopic measuring methods is commonly used in applications in the chemical and pharmaceutical industry. The key benefit is the quantitative and qualitative determination of a defined substance or parameter with high precision and reproducibility. This enables optimum process control and process reliability. Conventional methods, by contrast, such as gas chromatography (GC) are, primarily due to the time delay in the measurement results, problematic for certain types of processes, where a precise reaction endpoint determination is imperative. This is the case with purification processes, besides others.

To meet the strict purity requirements of the chemical, food and pharmaceutical industries, an additional cleaning step is often used during the manufacturing process. A common purification process is the washing out of impurities from a system by adding a washing solution.

Challenge

In the production of an organic compound, the washout of an impurity is determined by measuring the amount of impurity in the washing solution at the end of the purification process. The impurity to be washed out cannot be detected spectroscopically, but only by using gas chromatography.

This makes online process monitoring more difficult because a chromatographic separation method represents a technical limitation for process measurements that require continuous analysis or very fast analysis times.

Application

During a purification process, impurities must be washed out of the filter cake that forms during the washing process. The washing solvent downstream of the reactor must be monitored and the washing process interrupted if it falls below a defined value.

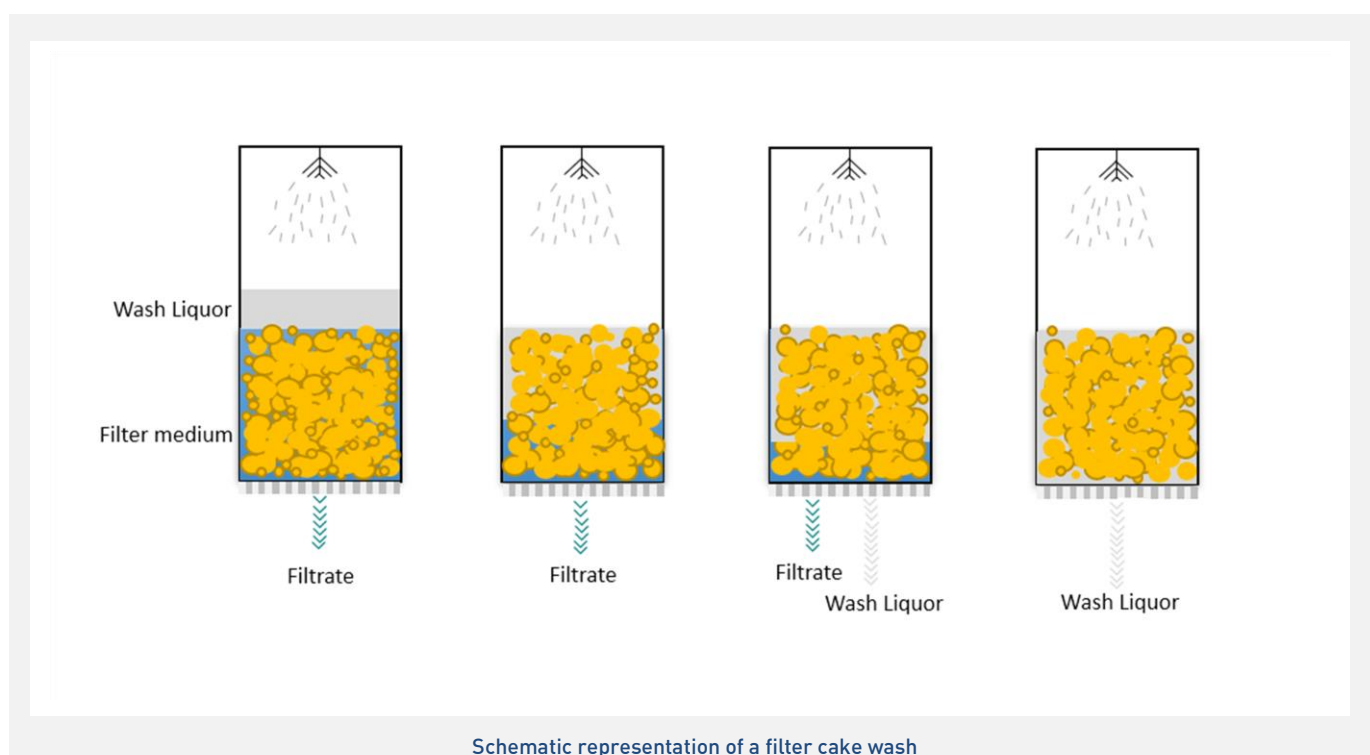
Solution

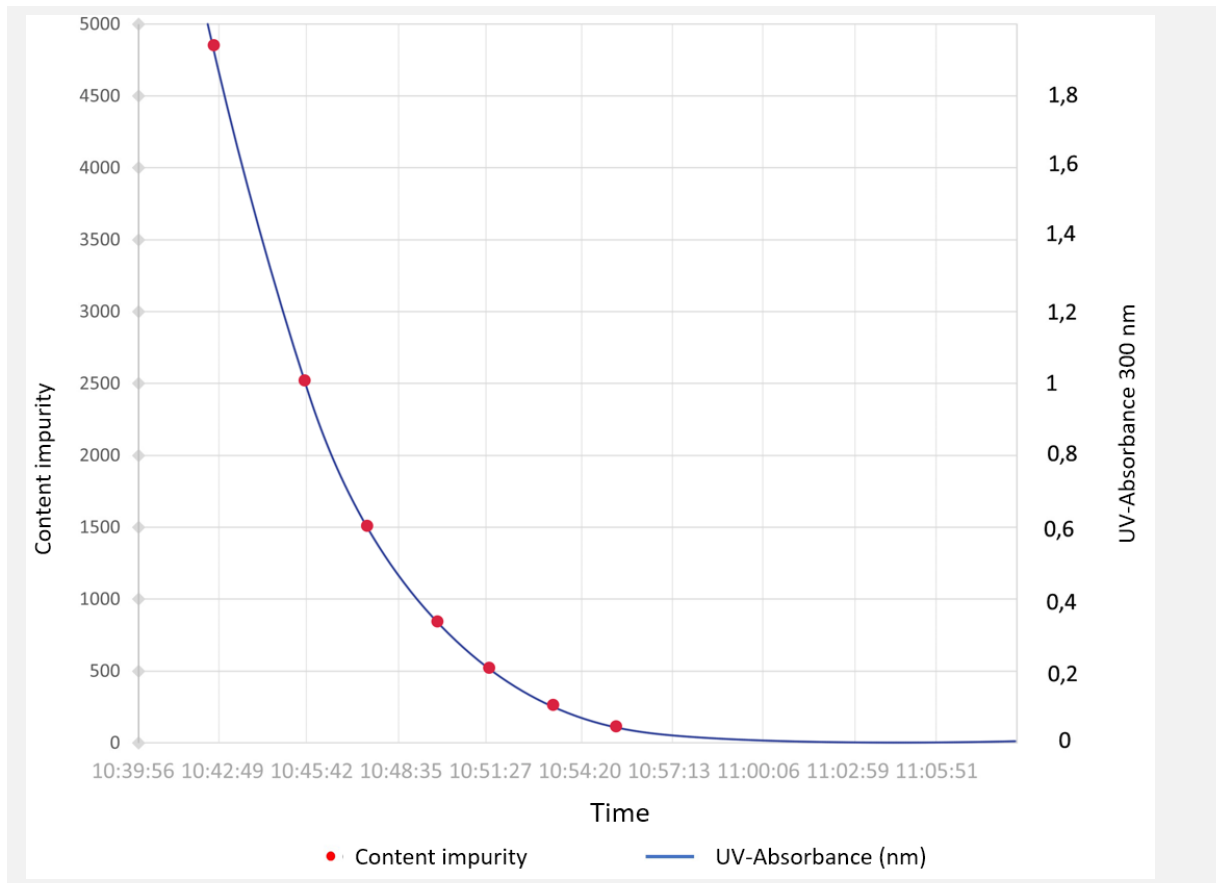
Use of a spectroscopically detectable component as a marker

Another impurity, which is present in much smaller quantities but can be detected spectroscopically in the ultraviolet (UV) spectrum, is also washed out of the filter cake.

By tracking the decrease of this detectable reactant, it is possible to determine the conversion of the reaction online, although the actual parameter is spectroscopically inactive.

Depending on the process environment, a transmission probe or measurement cell is installed via a bypass or directly into the reactor. The measurement signals recorded by the optical probe or measurement cell are forwarded to a spectrometer via optical fibers for evaluation.





Due to its compactness and flexibility, the Hellma [Excalibur Lab FFV](#) transmission measurement cell is particularly suitable for applications of this type.

Time-consuming sampling and analyses in the laboratory are no longer necessary and lead to considerable cost savings.



Compliant and safe production

Online measuring methods enable very precise and close-meshed process monitoring and guarantee the quality assurance of the manufacturing processes and environment necessary for compliance with regulatory requirements (GMP).

➤ Benefits

Optimum process control through continuous real-time measurements

The status of the washed-out contamination can be made available online and in real-time. Precise determination of the reaction end point is therefore possible.

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