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The acid test
Analysis in process and in the lab

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Nowadays, there are very high demands for product quality in pharmaceutical, food and chemical production. It can be ensured by a stable process with careful analysis. The product quality is ensured by analysis that is as close to the process as possible. Adherence to legal, technical and customer requirements are typically demonstrated in the lab.
Process-specific steps are monitored online and inline at critical control points. The measurement precision must be determined such that the process runs steadily. The goal of these measurements is to monitor and optimize the production parameters. Time is of essence here because reactions must be fast and targeted in certain circumstances.

Process-specific parameters that are not specified directly in the process are determined at-line close in time to the system operation in the operations lab. The analyses and rapid tests or simple methods (pH value, screening) conducted here can also be performed by employees without expert knowledge in lab work. The second part of the at-line analysis impacts the finished product. Each product is characterized by main features, which if met contain the remaining parameters within the specification. Thus, for many products, it can be stated that if the pH value is OK, the content is thus within specification.

The off-line analysis is performed by suitably trained employees in the quality control lab. The finished product is tested for the points in the specification. Lab employees have considerably more analysis tools and methods available than is the case in the operations lab. The goal here is to subject the product to the "acid test" and confirm its conformity to the corresponding specifications. Time is also of essence here even if not as serious as with online analysis. The goods must be released before filling with the different containers (packaging) are filled. The completely packed goods must be inspected before they can be sold.

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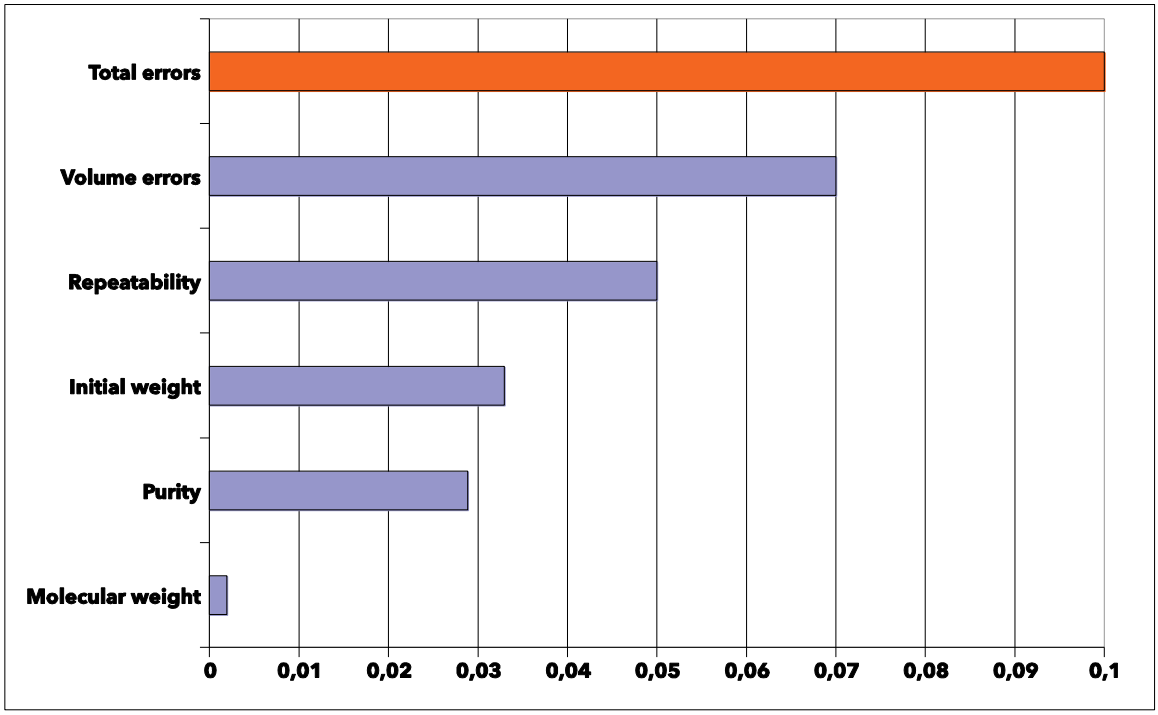
In practice

Jungbunzlauer is one of the world's leading manufacturers of natural and nature-identical biologically biodegradable materials. The roots of this internationally active company, domiciled in Switzerland, extends back to 1867. Jungbunzlauer is now a specialist for citric acid, xanthan, gluconate, specialties, special salts and sweeteners for food, drinks, pharmaceuticals and cosmetics as well as for various other industrial applications.

All of the products manufactured at Jungbunzlauer are subject to the regulations from current laws as well as the Food and Utensils Act (LMBG) for products supplied to the food industry and regulations of the European Pharmaceutical and USP for products supplied to the pharmaceutical industry.

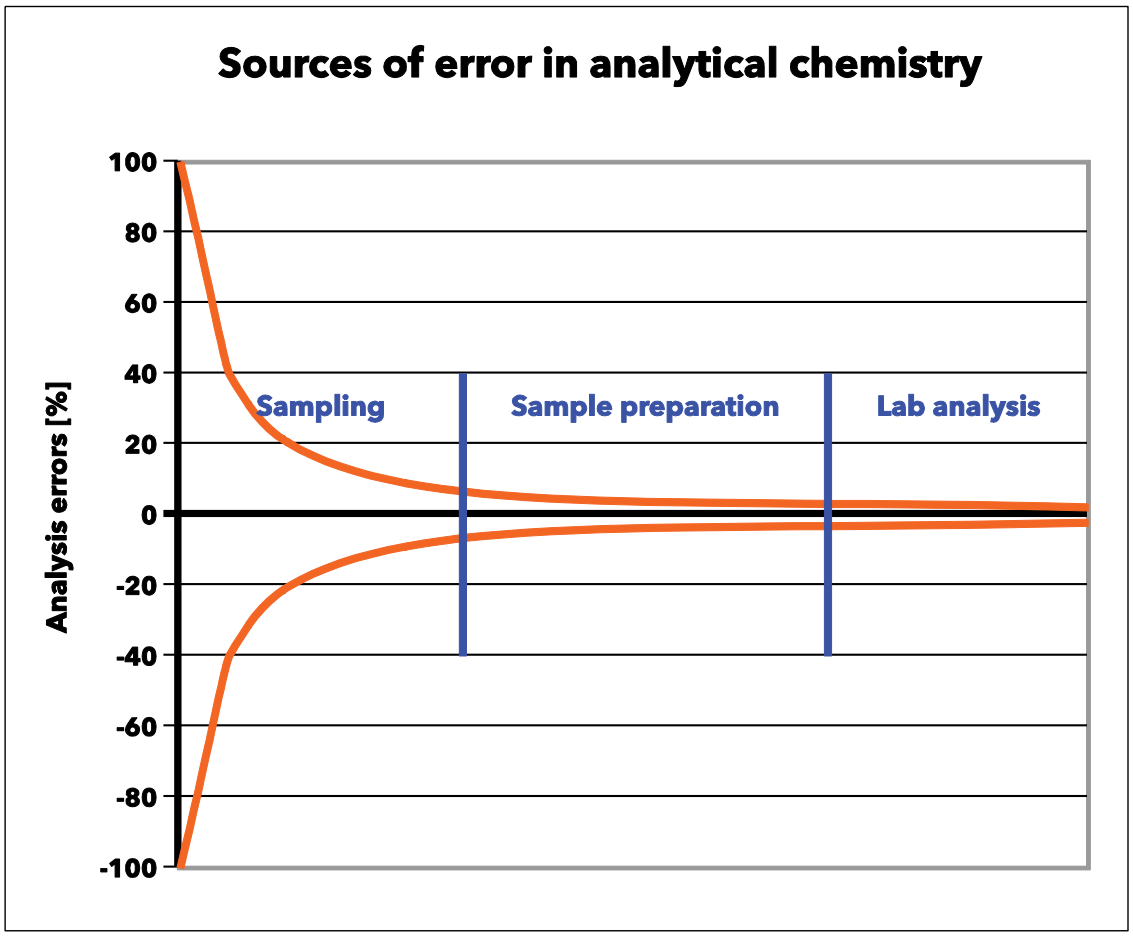
Tracking is a necessity and must be accessible from the outside. The cost effectiveness of an analysis system is of particular interest to the operator. They are primarily influenced by the personal combination time and the specimen capacity. Andrea Rüssel-Faas, Jungbunzlauer Ladenburg GmbH, Ladenburg Dr. Jürgen Peters, Schott Instruments GmbH, Mainz

In addition to accuracy and precise results, speed and personnel use also play a role in the lab routine. In order to work efficiently, the greatest possible precision must be achieved in the shortest time using the least personnel expense. Parameters such as planning of lab sequences, use of automated systems and good documentation play a major role here.



Typical uncertainties for setting a titer using potassium hydrogen phthalate.

During implementation, employees are supported by automated systems, which are controlled by a PC program and assumes the measurement and evaluation of specimens and which provides a high capacity with automated sampling. Computer programs take on a large portion of the documentation. It backs up the data to an internal company network to ensure the necessary data security.



One example: Titration
The citric acid produced by Jungbunzlauer in Austria is processed further in Ladenburg into different salts (e.g., tripotassium citrate, monosodium citrate, tricalcium citrate), citric acid esters and citric acid solutions. The content for different products is determined with the esters and titrimetically. There are several specifications, which are differentiated by their content area, for the citric acid solutions. Content adherence is determined by an acid base titration with 1 M soda lye.

The basic requirement of a method for determining content is precision, of course. The greatest possible errors from measurement uncertainty consideration can be determined using titration. If, for example, soda lye is adjusted with potassium hydrogen phthalate, burette volume errors are the greatest portion of errors. The burette precision is thus of considerable significance. Schott Instruments thus places considerable value on robust and highly precise motor piston burettes in which the accuracy is monitored regularly.

High level of assurance

The analyses generally undergo repeat determination in order to attain the highest possible assurance for the analysis. If there is 50% citric acid content, a deviation of maximum 0.2% absolute (0.4% relative) is accepted. Generally, deviations are only a tenth of that, although measurement of liquids demands a great deal of skill and precise handling. The accuracy of the results is ensured by weekly determining the soda lye factor against a primary titer substance (potassium hydrogen phthalate). The titer must be set so that the titer is dependent on the chemicals used as well as on the condition of the titrator and the electrodes. It would be fatal to rely on the calculation of the value using the titer information from the chemical packaging. The titer is determined using a 10x determination of the dry primary titer substance, with a standard deviation of not more than 0.0005 (relative: 0.05%). Values from 0.03-0.04% are normal.

The use of control software and sample providers allow for a high sampling rate with minimal personnel expense. If the samples are weighed and the program sequence is programmed, the titration runs automatically with intercalated cleaning steps, to prevent substances from dragging. The lab personnel can handle other duties during this time.

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