Webinar 1: The Basics of Titration

Be Sure You Do It Right!

C. Haider

Thanks to its universal applicability, titration is a widely used method for quantitative analysis. In titration, the unknown concentration of an analyte in the sample solution (the titrand) is determined by standard solution of known concentration (the titrant). The chemical reaction between the analyte and titrant is tracked with an appropriate indication method. From the volume of titrant deployed at the endpoint (also referred to as the equivalence point), we can calculate the concentration of the analyte on the basis of the stoichiometry of the titration reaction.

For modern titration systems to deliver true and precise results with little effort, you have to ask yourself the following questions in advance:

What titration mode and which method should I choose for the indication of the endpoint?

Typically, the choice of titration method will be determined by the analyte, the sample matrix, and the precision required, but economic aspects are also relevant. The titration mode (endpoint titration, monotonic or dynamic equivalence point titration) and the endpoint indication method (e.g., visual, potentiometric, or thermometric indication) have most significant effects on precision. Most titrations are carried out in a dynamic titration mode, since this mode can generally be adapted easily to the reaction conditions.

In the selection of instruments and accessories, in particular the sensors, another decisive factor is the robustness of the titration procedure, e.g., the tolerance of the method to operation errors.

What should I be aware of regarding the preparation of the titration system, i.e., assembly, titer determination, sample preparation, etc.?

Everything you need to know about instruments, reagents, and accessories will generally be included in the titration procedure. Minor, often unintended deviations during the installation of the titration system can significantly affect the measurement results. Such deviations can include the positioning of the sensor or the titration vessel, or the rotation direction of the stirrer. We thus recommend that you pay careful attention to these points in the titration procedure, optimally with the help of illustrations or images. The same applies to the reagents and sample preparation – whether a forgotten titer determination or improper sample preparation: minor deviations can have a substantial impact.

What are the optimal titration parameters with respect to titration rate and the proper selection of the equivalence point?

You can ideally adapt a titration mode to the reaction kinetics in the titration vessel by selecting the appropriate method parameters. Rapid chemical reactions, such as in the titration of a strong acid with a strong base, can be conducted with a relatively high titration rate. Slower reaction rates (e.g., with weak acids/bases or slow mixing of the titrant and sample solution) or longer sensor response times require a lower titration rate, titration with smaller volume increments, or a prolonged waiting time between the individual volume additions. The parameters for determining the equivalence point must be adjusted accordingly.

How can I qualify my titration system or titration method?

After installation, the proper functioning of a titrator can be established using a wet-chemical process based on a primary standard. A classical method for this is the titration of tris(hydroxymethyl)aminomethane (TRIS) with HCl or potassium hydrogen phthalate with NaOH.

In principle, you can validate any application using a corresponding standardized sample, i.e., by spiking the sample with a defined amount of analyte. In addition, a titration method should always be tested using different sample sizes to check the stoichiometry or linearity of the result.

Webinar 2: Troubleshooting Titration

How to Get Back on Track Fast and Effectively!

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What can you do when a titration result is not within the expected concentration range or when the titrator provides no result? Quite often, you can quickly solve the problem yourself.

Checking the hardware and the titration method

Was the proper solution applied to the titration and was the appropriate sensor used to indicate the endpoint? Are the titer of the titrant and the calibration of the sensor still valid? Intelligent titration systems monitor the critical system components and prevent erroneous results. How about sample preparation? If the sample is manually pipetted, homogenized, or filtered, subsequent checking is difficult and reproducibility may possibly be insufficient. An automated sample preparation system significantly improves precision and reproducibility.

If you still have doubts about the results even though you did not get any error messages, the titrator was operating normally, and the titration curve reflected the expected course, then we recommend that you check the quality of the reagents, the titrant, and the solvent. If your samples are solid, then you must make sure that they are completely dissolved before beginning the titration.

Does the measuring signal exhibit unexpected potential fluctuations? Does reproducibility diminish over time?

If so, this is often due to the sensor. The response time or the height or slope of the potential jump can deteriorate at the equivalence point due to old or contaminated sensors. This usually cannot be seen in a single titration curve; only comparing the titration times or overlaying several titration curves can provide the necessary information here. These problems can be avoided through regular, careful maintenance and cleaning of sensors.

Unstable measured values or a large signal-to-noise ratio can be caused by electrostatic effects. On the one hand, these effects can occur due to defective electrode cables or damage to sensors. On the other hand, the sample solution or the laboratory environment may be to blame. This is particularly likely in sample solutions with low conductivity, such as when nonaqueous solvents are used. In these cases, you should always use a sensor with internal electrostatic shielding and try to prevent electrostatic effects in the lab environment.

Inadequate mixing of the sample solution is another common cause of erroneous results. The ratio between the size of the vessel and of the stir bar, for example, has a decisive impact on the efficiency of mixing and thus on the titration rate. The same applies to the type of stirring blade used with a propeller stirrer. Generally speaking, always employ the maximum possible stirring rate that does not form a vortex in the titration vessel.

If, in addition to maximum precision, you also want to work more efficiently and cost-effectively, you can achieve substantial savings in time and reagents by adjusting the titration parameters and stop conditions and by controlling the titration rate.

October, 29th, 2015, 2 p.m.

TROUBLESHOOTING

Learn all there is to know about "Titrations" during the webinar series presented by GIT and Metrohm! On October 29th the focus will be on troubleshooting and optimization. After the presentation, Dr. Haider will be available to answer your questions. Register now at: http://bit.ly/titration-webinar-en

Contact
Dr. Christian Haider
Metrohm AG
Herisau, Switzerland
hc@metrohm.com
www.metrohm.com

The Webinar will be held in German and English simultaneously