



Bottle top burets and dispenser

Testing and calibration

Overview

An in depth look at the topic of measuring and test equipment monitoring of laboratory instruments. This article discusses the meaning of terms such as checking, calibrating and adjusting, clarifies who should carry out these activities and when it makes sense.

Introduction

The question of the correct volume is always present in chemical, biological or clinical laboratories. Measuring and testing equipment monitoring according to GLP/GMP and DIN EN ISO 9001 requires users to regularly check their bottle-top burets and dispensers. Many processes require precisely dosed fluids to achieve repeatable results and quality assurance.

The application areas are diverse:

+ Clinical diagnoses:

Was the correct amount of detection reagent added to the patient sample?

- + Analytical-chemical processes: Can I trust titration results?
- + Industrial filling processes:

Industrial filling processes are also relevant, as a regularly calibrated dispenser gives the supplier confidence that the liquid dispensed is accurate to the customers requirements.



Testing

Checking: Compare and evaluate the measured relevant physical quantity with a defined setpoint. A simple check of a bottle-top buret can be carried out, with a measuring flask. This volumetric flask corresponds to the nominal volume of the bottle top buret: You dose the medium from the bottle-top buret into a measuring flask until the meniscus reaches the ring mark in the measuring flask. Then stop dosing and read the volume delivered from the bottle top buret. This should then correspond to the nominal volume of the measuring piston, taking into account the device tolerances. Special test measuring flasks are available for dispensers.

The procedure described above is not a calibration within the relevant standards. This type of check is only used to quickly and easily obtain an overview of the device condition and to identify large deviations, e.g.:

- Functional check after disassembling a device (aspirating air through an improperly mounted valve)
- + At the beginning of the working week: Did a contamination clog a channel in the appliance over the weekend?

The method can also be used to evaluate the condition of the device after falling or other extreme external influences. This makes sense for users to check themselves at the site of use.



Measuring flasks with 3 marks. The 3 mark test plunger is used to check the function of a dispenser. The middle mark corresponds to the nominal volume, the upper and lower marks to the error limits

Calibration

Calibration: Determine the displayed measured values of a device with regard to a physical quantity and compare them with a reference value defined by measuring standards (e.g. a very precise weight piece) under specified conditions.

For volume determination with reference to the relevant standards, this means: The amount of fluid discharged from the device is measured with a scale. The weighing value is converted into the delivered volume using a correction factor Z (see current DIN EN ISO 8655-6) To calibrate the volumedelivering device, it is necessary to know the density of the fluid used at the measuring temperature, as well as the temperature of the fluid, the air temperature, the air pressure and the relative humidity that prevails during the measurement. If the manufacturer specifies temperature-dependent effects on the measured volume of the device, these must also be taken into account.

In order to control the specified environmental parameters, the room in which calibration is performed must be climatemonitored (according to ISO 8655:2022: Room temperature between 17 °C and 23 °C, fluctuations of a maximum of 0.5 °C per hour, relative humidity stable between 45 % and 80 %). The necessary investments and operating costs can be considerable if the users want to calibrate themselves.



Schematic curves of measuring points if the temperature changes of the test medium are not included in the volume calculations.

- Temperature increase
- O Temperature decrease
- Temperature fluctuates sharply or uneven operation of the device



In addition, various directives and standards require that the measurement uncertainty of the calibration procedure be considered. The measurement uncertainty is the uncertainty of the measurement procedure as used in the respective calibration laboratory. On the one hand, the systematic measurement uncertainty is determined. This takes into account, among other things, the measurement uncertainty contributions of the scale, the room climate and the water used for the measurement. On the other hand, the random measurement uncertainty is determined. This results from the repeatability of the measurement and is therefore significantly dependent on the skills of the operating personnel.

During calibration, measuring points are determined as well as statistically determined mean values of the measurement series. These measurement points and averages can be located in areas far enough away from the error limits, i. e. "safe". But also so close to the error limits that these measuring points and mean values must be described as "unsafe". If the measured value or the calculated mean value of a measurement series, together with its measurement uncertainty, lies within the error limits, the calibration has been passed (evaluation criterion A). If the measured value with its measurement uncertainty is outside the error limits, the calibration was not passed (evaluation criterion C). If the measured value with its measurement uncertainty is neither completely within nor completely outside the error limits, no clear evaluation is possible. It is not clear whether the evaluation has been passed or not (evaluation criterion B).



If the calibration fails on, and or is completed with evaluation criterion B, the instrument and also the test procedure must be carefully considered. The following questions include: Is the device leakproof? Is the appliance clean? Are there no defects? Was the test procedure performed correctly?

The manufacturer of the devices can answer these questions most reliably. So it makes sense to send bottle-top burets and dispensers to the manufacturer for calibration. In addition, the cost savings for room air conditioning and testing equipment (scales and thermostats for the water) and for the qualification of the test personnel are possible.

If qualified personnel and appropriate infrastructure are available, than users can calibrate themselves. Special calibration software (EASYCAL[™] 5) supports users in his processes.



Adjustment

If the calibration is completed with evaluation criteria B or C and all critical questions are answered in the affirmative, the determined volume of the device is correct. Even if the display deviates from the target value. To ensure the result, the calibration should be repeated.

It is possible to note the deviation determined and calculate it as the correction value for the next volume dispensing with the device, but in practice this is not done. Liquid handling devices can be adjusted in this case. Depending on the design, the piston stroke can be changed so the volume displayed corresponds to the volume actually delivered or the volume value displayed on the device is adjusted to the actual measured value. It is then necessary to calibrate again to confirm the calibration or, if necessary, to adjust it again. If the calibration fails several times, there is probably a device defect and requires repair by the device manufacturer or its service partner.



Adjusting a Dispensette S Digital - the volume displayed is adjusted to the actual measured volume.

Conclusion

To address the question asked at the beginning, "Who should carry out the test and who should carry out the calibration?", we can generally recommend: Testing by the user, calibration by the manufacturer of the devices, in particular if additional maintenance or repair of the device is to be expected.

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