



# Recognised Results Based on Analytical Quality Assurance (AQA)

## Introduction

Analysis and quality assurance measures are inseparably interlinked. There is much more to ensuring high quality measurement results than simply the type of **analytical method** (standard/norm vs. operational analysis) that is used. The care taken over the individual work steps and the **quality assurance measures** that are implemented, play a much greater role. Hach® supports users of LCK Cuvette Tests by carrying out a substantial part of the quality assurance measures on their behalf. This means that the relevant **quality and batch certificates** are always available, e.g. on the Hach website. Support is also provided for users when carrying out individual quality control measures.

### Why the need for quality control?

Nowadays the quality of goods and services is crucially important. Purchasers and users have come to expect high quality standards from suppliers and manufacturers. This is why the quality of the services and products on offer is checked and documented several times over (e.g. in accordance with ISO 9001:2000).

The results of analyses can also be considered as goods and they have to be able to prove their quality. Responsibility for the resultant data lies with the users themselves or their supervisors. Both are therefore liable for any incorrect interpretations and decisions that are made as a consequence of incorrect analysis results. Integrating appropriate quality control measures at the relevant points of the analysis process ensures reliable analysis and minimises the risk of exposure to liability.

### How quality assurance is organised in the laboratory

Organising and carrying out Analytical Quality Assurance in laboratories involves dealing with a variety of international and local standards. The central points are:

- Defining the measures to be implemented based on applicable standards.
- Internal and external quality assurance measures
- Analytical equipment (monitoring and maintenance)
- Laboratory staff (skills and training)
- Documentation of implemented measures

The main aim is to define uniform quality standards for measured results from operational analysis. Fundamental requirements will be established for the operating methods themselves, for the manufacturers of equipment and reagents and for the users. The requirements apply across all industrial and municipal sectors.

### Building blocks of quality control

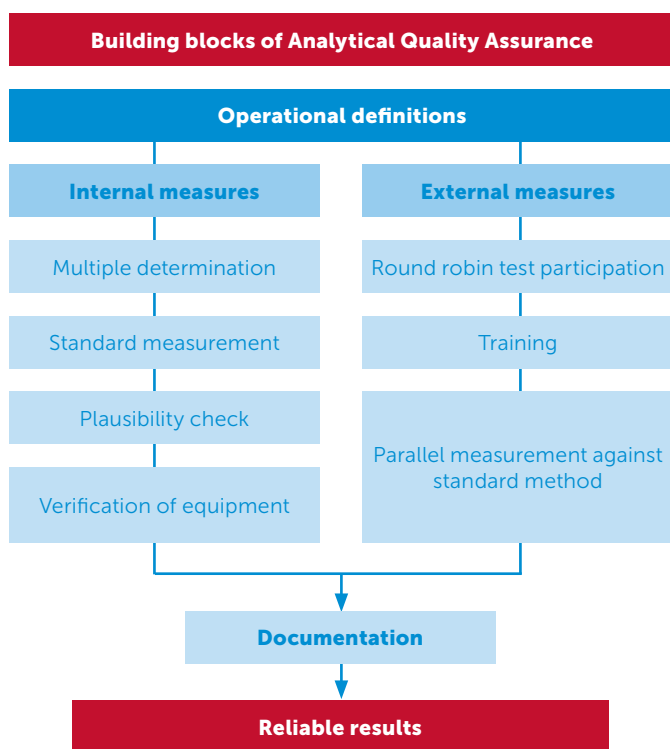
AQA can be subdivided into two areas:

**1. Internal quality assurance** – This is carried out by the user themselves.

**2. External quality assurance** – For example, this results from a collaboration between the user and the equipment manufacturer or between different laboratories.

The operational definitions (definition of measures, frequency and quality control objectives) ensure that individual measures are tailored to suit the needs of the relevant plant.

	<b>Product Quality</b>
+	<b>Workflow Quality</b>
+	<b>Quality Assurance Measures</b>
=	<b>Quality Results</b>



Internal and external quality assurance measures

## AQA Glossary

### Internal measures

#### Multiple determination

Multiple determinations for a sample or for the repetition of individual steps of an analysis (e.g. sampling) increase the reliability of the individual measurement result. Multiple determinations allow major outliers to be recognised immediately. Averaging the measured values substantially improves the precision of the results. Duplicate determinations should be part of everyday analysis, regardless of the analysis procedure that is used.

#### Standard measurement

Regular analyses of a standard solution form the basic framework of any quality assurance process. This is done by analysing solutions with a known content and documenting the readings on a standard control card. If the readings are within a predetermined confidence interval (permitted scatter around the setpoint value), this confirms that the equipment used, such as the photometer, cuvette test, pipettes etc, are working correctly and that the analysis was carried out properly.

With Addista Hach has created a practical system for operational analysis that unites the individual building blocks of Analytical Quality Assurance. The Addista range includes a standard/spiking solution that is suitable for LCK Cuvette Tests, as well as two round robin test solutions.

### Plausibility check

Samples can contain substances that distort an analysis (e.g. high concentrations of COD when determining nitrate). These can be checked by means of diluting or spiking.

*Diluting:* For example, the sample is diluted 1:10, i.e. 1 mL sample + 9 mL distilled water, and then analysed according to the working procedure. The result produced must be comparable with the measured result of the original sample once the dilution factor is taken into account. Note: It is important that the measurement range limits are observed when selecting the level of dilution. If the measured result of the original sample is already in the lower measurement range of the cuvette test, the sample should be spiked.

*Spiking:* This involves mixing the sample with the spiking solution and then measuring this with the relevant cuvette test (E1). The sample is also measured without spiking solution in parallel to this (E2). The spiking rate is calculated as follows:  $\text{Spiking rate} = E1 - E2/2$

The calculated spiking rate should now be within the predetermined confidence interval. If it is outside this interval, the sample contains interfering ions. It must then be prepared prior to analysis using a suitable method to reduce the concentration of interfering substances (dilution, digestion etc, depending on the type of sample).

# AQA – Recommendations on frequency and quality targets

MEASURE	AIM/BENEFIT	MINIMUM FREQUENCY	QUALITY AIM <sup>3</sup>	
LABORATORY ANALYSIS	Multiple determination	Once per month <sup>1</sup> per operator and matrix	Difference ≤10%	
	Standard measurement	With each 10 <sup>th</sup> sample; at least once per month <sup>1</sup> per operator	Maintenance of confidence interval	
	Plausibility check	If implausible results are obtained or matrix changes occur, or at least once per quarter <sup>1</sup>	Difference ≤20%	
	Round robin tests / comparative measurements (with operational methods)	Once or twice per year per operator	Difference ≤20%	
	Parallel measurement (with reference method)	Once per year per cuvette test	Difference ≤20%	
	Pipettes (volume checks)	Twice per year	Difference ≤1%	
	pH meter	Once per month	Difference ≤0.2 pH	
	Thermostat	Once per year	Difference ≤3°C	
	Photometer	Once per year	In accordance with manufacturer's specifications	
	PROCESS	Standard measurement	Once per month	Maintenance of confidence interval
		Plausibility checks	If implausible results are obtained or matrix changes occur	Difference ≤20%
		Comparative measurements with laboratory	Once per week	Difference ≤20%

<sup>1</sup> Also for important studies (e.g. comparison with official monitoring methods)

<sup>2</sup> E.g. per test filter set or during instrument maintenance

<sup>3</sup> It might be worthwhile to define the permissible difference in mg/L rather than as a percentage, e.g. for readings in the very low concentration range.

### Verification of equipment

A frequent cause of error is incorrectly measured volumes, e.g. using an incorrectly adjusted pipette or not handling the pipette properly. Regular checks mean that these sources of error can be recognised and rectified quickly.

For photometers sets of calibration filters are available for checking stray light and photometric accuracy. These make it quick and easy for users to check their devices themselves. The resultant data is documented into an inspection sheet. Booking a Hach Service Program is another option to ensure the reliability of your instruments.

### External measures

#### Round robin test participation

The round robin test is an important element of external quality assurance. The principle behind it is that identical samples are analysed independently by several participants under comparable conditions. The work of the individual participants can be assessed using the individual results. The process also provides information about the precision and correctness of the analysis procedure. Participation in a round robin test is often a requirement for the recognition of the parity of operational analysis methods.

#### Parallel measurement

For almost all normal sample matrices the operational analysis procedures deliver results that are comparable with the standard procedures. Still the question of comparability of results with the reference method stays in focus. It is therefore recommended during regulatory monitoring that the sample be split and analysed in parallel with the cuvette test, including the necessary quality assurance measures.

### Training

Regular participation in training seminars keeps analytical knowledge up-to-date. Understanding analytical correlations, recognising possible sources of error and doing analyses with a group of peers increase the ability to make the best possible use of operational analysis and to evaluate results correctly.

### Documentation

Analytical Quality Assurance supports the verification of results and documents that the measuring system is operated correctly. This starts with pulling a sample and ends with an analysis report in the laboratory or operational logbook. The documentation must be accurate and clearly arranged. It must be obvious who produced what analysis data and when. All results of quality assurance measures should be entered onto the relevant control cards.

#### What do you need to pay attention to?

- All measured results should be within the confidence interval.
- Aim to improve working methods by narrowing the confidence interval.
- Observe trends.

Empirical values are also an important component in evaluating the results. Changes in the concentration of substances may depend on a variety of factors, such as total water quantity, period of time spent in the plant, pH value etc. Analysis values and empirical values must also match.

### Conclusion

Regular application of AQA ensures that:

- The results of analyses are traceable.
- The correct status of the analysis system is documented.
- Handling errors can be recognised immediately.
- Comparison of measured results is possible.
- Results of analyses are recognised.

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