APPLICATION REPORT
LABORATORY ANALYSIS & PROCESS ANALYSIS
ANALYTICAL QUALITY ASSURANCE

Recognised Results Based on Analytical Quality Assurance

Quality assurance and analysis are completely interlinked. There is much more to ensuring high precision of the measured results than simply the type of ➔*analytical process* (standard/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. The manufacturer actually carries out a substantial part of the quality assurance measures on behalf of users of LANGE cuvette tests.

This means that the relevant ➔*quality and batch certificates* are always available, e.g. on the Internet at www.hach-lange.com. Support is also provided for users when carrying out individual quality control measures. The ➔*ADDISTA* range of solutions provide support for the fundamental aspects of AQA.

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Why the need for quality control?

The quality of goods and services is crucially important in the current climate. 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 so that they can be valued and compared. 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.

Product quality
+ Application quality
+ Quality assurance measures
= Quality results

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, e.g. EN 45 001, ISO CD 13530.

The central points are:
- Defining the measures to be implemented
- Internal and external quality assurance measures
- Analytical equipment (checking and maintaining)
- Laboratory staff (skills and training)
- Documenting the measures that have been implemented

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, as well as for the manufacturers of devices and reagents and for the users. The requirements apply across the board in all industries.

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 manufacturer or between different (works) 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 operation.

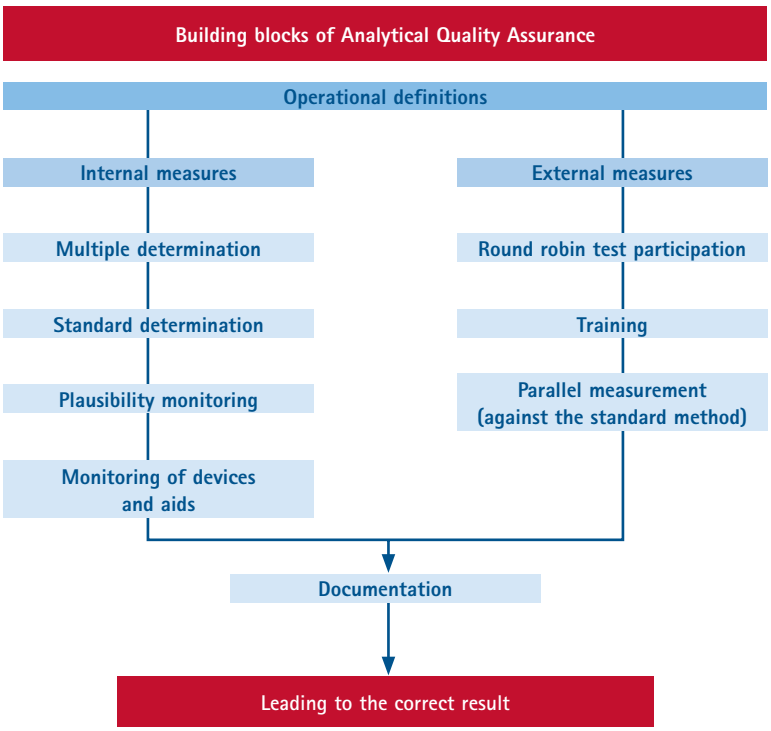


Fig. 1: Internal and external quality assurance measures

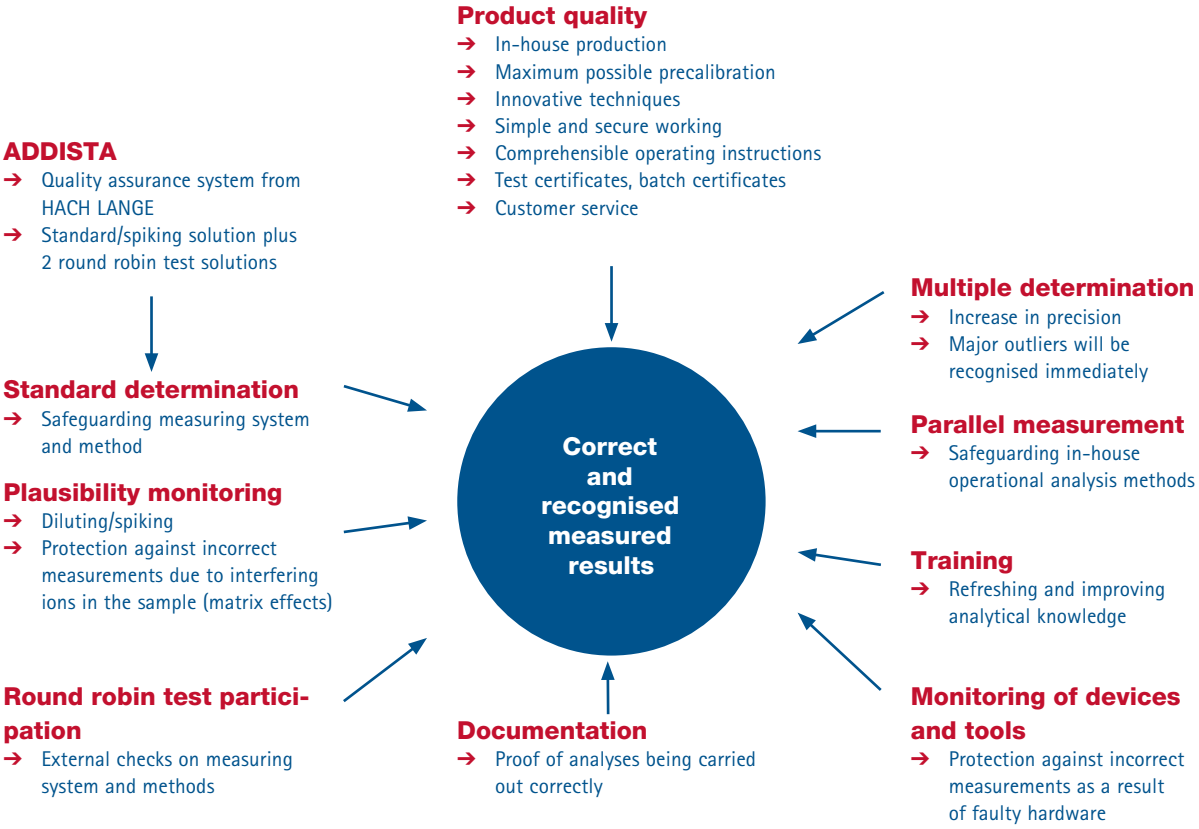


Fig. 2: The various quality assurance measures and their benefits

ADDISTA

HACH LANGE has created a practical system for operational analysis that unites the individual building blocks of Analytical Quality Assurance. The ADDISTA solutions can be used to carry out the fundamental measures of the Analytical Quality Assurance system. The ADDISTA range includes a standard/spiking solution that is suitable for LANGE cuvette tests, as well as two round robin test solutions. It contains both a standard and a round robin test solution for the field of nutrient process analysis.

Standard determination

Regular analyses of a standard solution form the basic framework of any Analytical 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.

Plausibility monitoring

Despite correct methods and equipment, 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 1:10, i.e. 1 ml sample + 9 ml distilled water, diluted and then analysed as normal in accordance with the work regulation. The result produced must be comparable with the measured result of the original sample once the dilution factor is taken into account. 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.

Round robin tests
In use for more than 20 years

ADDISTA for laboratory analysis with round robin test solutions A+B and standard/spiking solution

ADDISTA for the process test equipment AMTAX, NITRATAX and PHOSPHAX

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:

Spiking rate = E1 – E2/2

The spiking rate calculated should now be within a predetermined confidence interval (specified on the rear of the relevant ADDISTA package). If the value is outside this interval, the sample contains interfering ions and the sample must be developed using a suitable method (dilution, disintegration etc, depending on the type of sample). A simple method for removing interferences is often to dilute a sample, as this also reduces the concentration of interfering substances.

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. Officially recognised laboratories are required to take part in round robin tests that are carried out regularly. This is to ensure that the quality of the approved laboratories can be checked at any time. Participation in a round robin test is also often a requirement for the recognition of the parity of operational analysis methods. Successful participation in a HACH LANGE round robin test means that the participant receives a complete evaluation of the round robin test plus a certificate.

Multiple determination

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

Parallel measurement

Although the operational analysis procedures deliver results that are comparable with the standard procedures, for almost all normal sample matrices users will focus on the question of the comparability of the results with the reference procedure. It is therefore recommended during regulatory monitoring that the sample be split and analysed in parallel to the cuvette test, including the necessary quality assurance measures.


Certificate

Jenny Miller

successfully completed the round-robin test for the following parameters:

Nitrate LCK339
COD LCI400

Sabine Kater
Manager round-robin tests

LANGE

Certificate demonstrating successful participation in the round robin tests

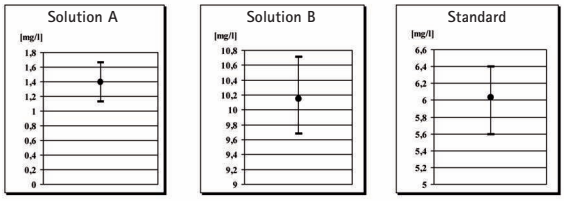
HACH LANGE
UNITED FOR WATER QUALITY

Round robin test evaluation

Mrs Miller
Main sewage treatment plant

LCK 339
Nitrate-N

Measurement range	0.23-13.5 mg/l
Date of entry	05/04/2007
Test tool	DR 5000



	Your measured result [mg/l]		Correct concentration [mg/l]	Confidence interval [mg/l]	Total average value [mg/l]	Number of participants
Solution A	1.38	1.42	1.4	±0.27	1.365	1355
Solution B	10.2	10.1	10.2	±0.52	10.04	1396
Standard	6.05	6.03	6	±0.4	6.007	5005

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Fig. 3: Round robin test evaluation using the example of the parameter for nitrate nitrogen. Each of the individual results calculated by the user is given, plus the correct concentration, confidence interval, total average value and the total number of participants in the round robin test.

Training

Regular participation in training seminars (e.g. annually) keeps analytical knowledge up-to-date and refreshes or improves it from a specialist point of view. Understanding analytical interrelations, recognising possible sources of error and performing practical work in groups increase the ability to make the best possible use of operational analysis and to evaluate measured results correctly.

Monitoring devices and aids

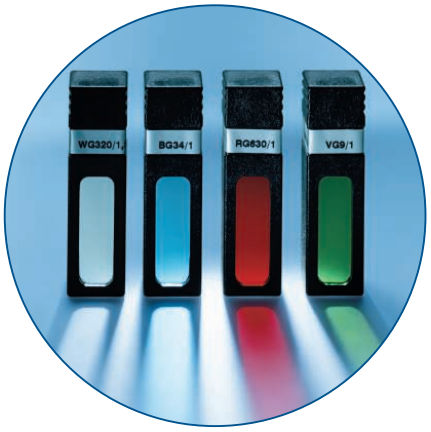
Sets of calibration filters for checking the stray light and the photometric accuracy are available for HACH LANGE photometers. These make it quick and easy for users to check their devices themselves. The resultant data is entered into a test log.

Taking out a HACH LANGE service package is another option to ensure the reliability of your analysis. Further details can be found at: www.hach-lange.com.

A frequent cause of error is incorrectly measured volumes, e.g. using an incorrectly adjusted pipette or not handling the pipette properly. Regular checks against the pipette test kit LCA722 means that these sources of error can be recognised and rectified quickly.

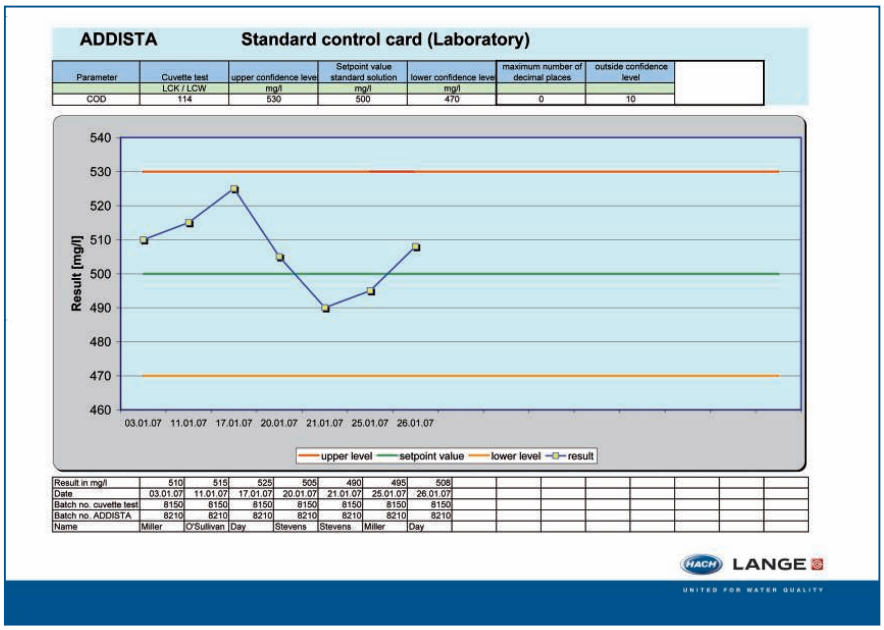
"Our free round robin testing in Europe has stood the test of time over more than 20 years. The impressive degree of acceptance of this simple and popular method of external quality control is demonstrated by the increasing number of participants we see each year. The high success rate of more than 85 % positive round robin tests is proof of the good analytical work done by HACH LANGE users."

Sabine Kater, Product Manager,
HACH LANGE Dusseldorf

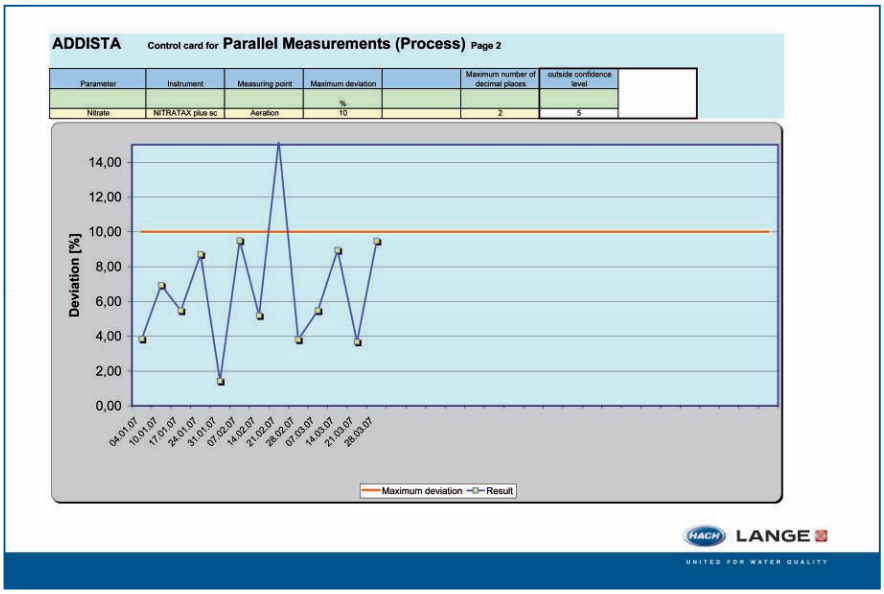


Set of calibration filters for checking photometric correctness

Documenting AQA measures correctly



Laboratory analysis: Standard control card



Control card process: Parallel measurements in the lab

Documentation

Analytical quality assurance supports the verification process and documents that the measuring system is operated correctly and analysis is carried out free of errors. This begins with sampling and ends with an analysis report in the laboratory journal or operational protocol. The documentation must be accurate and clear. It must be obvious who produced what analysis data and when.

Control cards

All results of quality assurance measures should be entered onto the relevant control cards. For example, the HACH LANGE quality control blocks or the Excel version on CD-ROM). A confidence interval is given for each cuvette test as an evaluation aid.

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

The Excel version of the quality control block also simplifies the whole procedure. The users enter their data into the computer and the programme calculates automatically whether or not the desired goal has been achieved (e.g. whether or not the results are within the confidence interval). The results can also be displayed in graphic form, depending on the measure in question.

AQA recommendations for frequency and quality objectives

MEASURE	OBJECTIVE	USES	FREQUENCY	QUALITY OBJECTIVE
Multiple determinations	Increasing precision	Recognising outliers	Once per month; additionally for important checks; personal and matrix related	Difference ≤10 %*
Standard determination	Internal system checking	Safeguarding working methods	With every 10th sample, but at least once per month; personal related	Complying with the confidence interval
Spiking / dilution	Plausibility control	Protection against matrix related incorrect measurements	For implausible measured results; for matrix changes; but at least every 3 months	Deviation ≤20 %*
Round robin test participation	External confirmation of the good quality of the analyses	Recognition of scatter levels; proof of success	1-2 times per year; personal related	Deviation ≤20 %*
Parallel measurement	Safeguarding operating method	Proof of suitability of the cuvette test for sample in question	Once per year for each cuvette test and for implausible measurement results (regular splitting of samples for regulatory monitoring)	Deviation ≤20 %*
Documentation	Transparent analysis and proof	Traceability of laboratory activities	Always	

Table 1: Objective, use and frequency of the various AQA measures
* The percentage limits apply for the 20 % – 80 % interval of the measurement range. For very low concentrations, it can be more useful to specify the quality objective in mg/l.

Empirical values are also an important component in evaluating the results. Changes in the concentration of the parameters to be checked 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. For example: water transparency = 50 cm and COD = 38 mg/l O_2 => implausible.

Conclusion

Regular application of Analytical Quality Assurance ensures that:

- ➔ The results of analyses are traceable
- ➔ The analysis system is correct and can be demonstrated at any time
- ➔ Handling errors can be recognised immediately
- ➔ Comparison of measured results is possible
- ➔ Results of analyses are recognised

Multiple and individual standard solutions are available for many parameters including turbidity

Seamless documentation of the quality of the analyses

Tools for AQA

ADDISTA	CUVETTE TEST
LCA 700	LCK 238 LATON LCK 304 Ammonium LCK 311 Chloride LCK 328 Potassium LCK 348 Phosphate ortho LCK 414 COD
LCA 701	LCK 306 Lead LCK 321 Iron LCK 329 Copper LCK 337 Nickel LCK 353 Sulphate LCK 360 Zinc
LCA 702	LCK 301 Aluminium LCK 308 Cadmium LCK 313 Chromium (VI), total LCK 353 Sulphate
LCA 703	LCK 049 Ortho-phosphate LCK 114 COD LCK 303 Ammonium LCK 311 Chloride LCK 339 Nitrate LCK 350 Phosphate ortho LCK 353 Sulphate LCK 386 TOC
LCA 704	LCK 153 Sulphate LCK 305 Ammonium LCK 311 Chloride LCK 314 COD LCK 340 Nitrate LCK 349 Phosphate ortho LCK 385 TOC

ADDISTA	CUVETTE TEST
LCA 705	LCK 014 COD LCK 302 Ammonium LCK 311 Chloride LCK 387 TOC
LCA 706	LCK 521 Iron LCK 529 Copper LCK 537 Nickel LCW 032 Manganese
LCA 707	LCK 341 Nitrite LCK 348 Phosphate ortho/total LCK 614 COD
LCA 708	LCK 338 LATON LCK 350 Phosphate ortho/total LCK 514 COD
LCA 709	LCK 138 LATON LCK 342 Nitrite LCK 349 Phosphate ortho/total LCK 614 COD
LCA 310*	LCK 310 Free chlorine
LCA 333*	LCK 333 Non-ionic surfactants
LCA 390*	LCK 390 AOX
LCA 555*	LCK 555 BOD
LCA 753**	**ADDISTA process for** Ammonium (AMTAX) Nitrate (NITRATAX) Phosphate (PHOSPHAX)
LCA 754**	**ADDISTA process for** TOC (TOCTAX)

* Mono-standard without round robin test solutions

** Only standard and round robin test solution

HACH LANGE Services

Contact us to place an order, request information or receive technical support.

On site technical support.

Seminars and workshops: Practical and hands on training.

Quality assurance, complete with standard solutions, instrument checks and test solutions.

Service packages and extended warranty up to 5 years.

www.hach-lange.com
up to date and secure, with downloads, information and e-shop.

Test media

LZP 310	**Calibration filter set** for checking the stray light and the photometric correctness for CADAS and XION
LZV 537	**Calibration filter set** for checking the stray light and the photometric correctness for DR 2800/5000
LCA 722	**Pipette test kit** for checking the HACH LANGE piston pipettes
LZP 181	**Test kit** for checking photometric accuracy
LZV 086	**Holmium perchloride solution** for checking the wavelength accuracy (blended in the QA cuvette)

We reserve the right to make changes without notice.

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