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Recent Advances in Discrete Analysis Technology for Automated Simultaneous Multi-parameter Wet Chemistry Testing in Drinking Water, Wastewater, and Soil Samples

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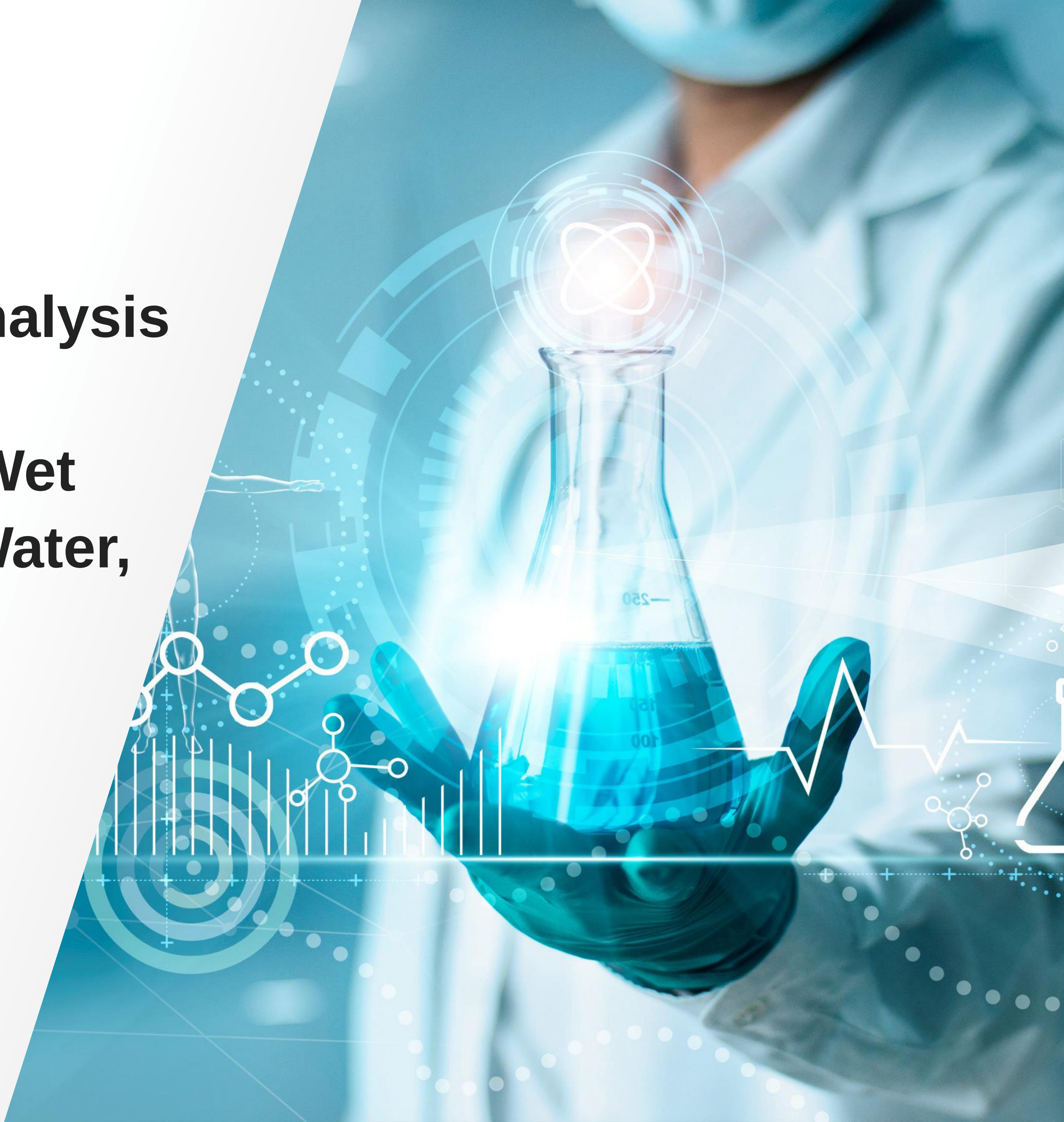


Agenda

1 *Limited lab resource vs. demand for improved throughput?*

2 *Limitations of traditional wet chemistry workflow*

3 *Improve lab productivity by workflow automation*

4 *Recent advances of Gallery Aqua Master systems*

Water analysis

Same analytics – Different purpose

- Drinking water
  - Safe drinking water
  - Regulatory compliance

- Process water
  - Protect process equipment from corrosion, scaling
  - Energy efficiency
  - Regulatory compliance

- Wastewater
  - Environmental Protection Agency – clean water act
  - Reuse – Repurpose
  - Regulatory compliance

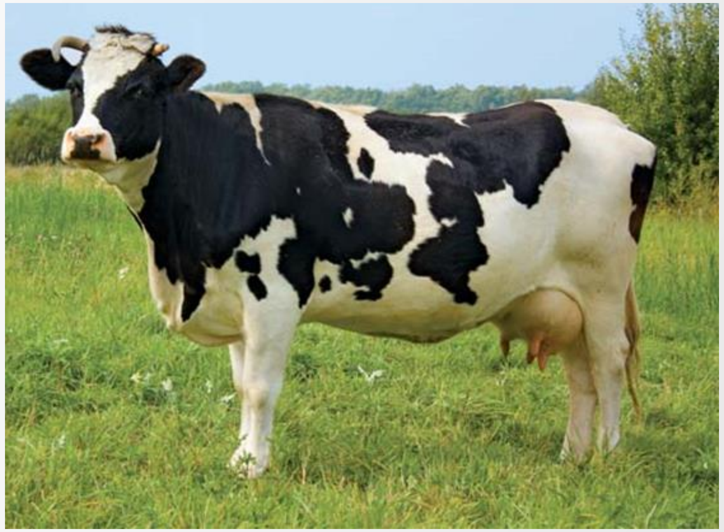
Water analysis is critical across all industries, water utilities and contract testing labs

Soil analysis

Same analytics – Different purpose

- Environmental soil samples
  - to check pollution e.g. after flooding.
  - Contaminants near ground water area

- Agricultural nutrient
  - Soil samples analysis to instruct the quantity of fertilizers needed for enhanced crop yield

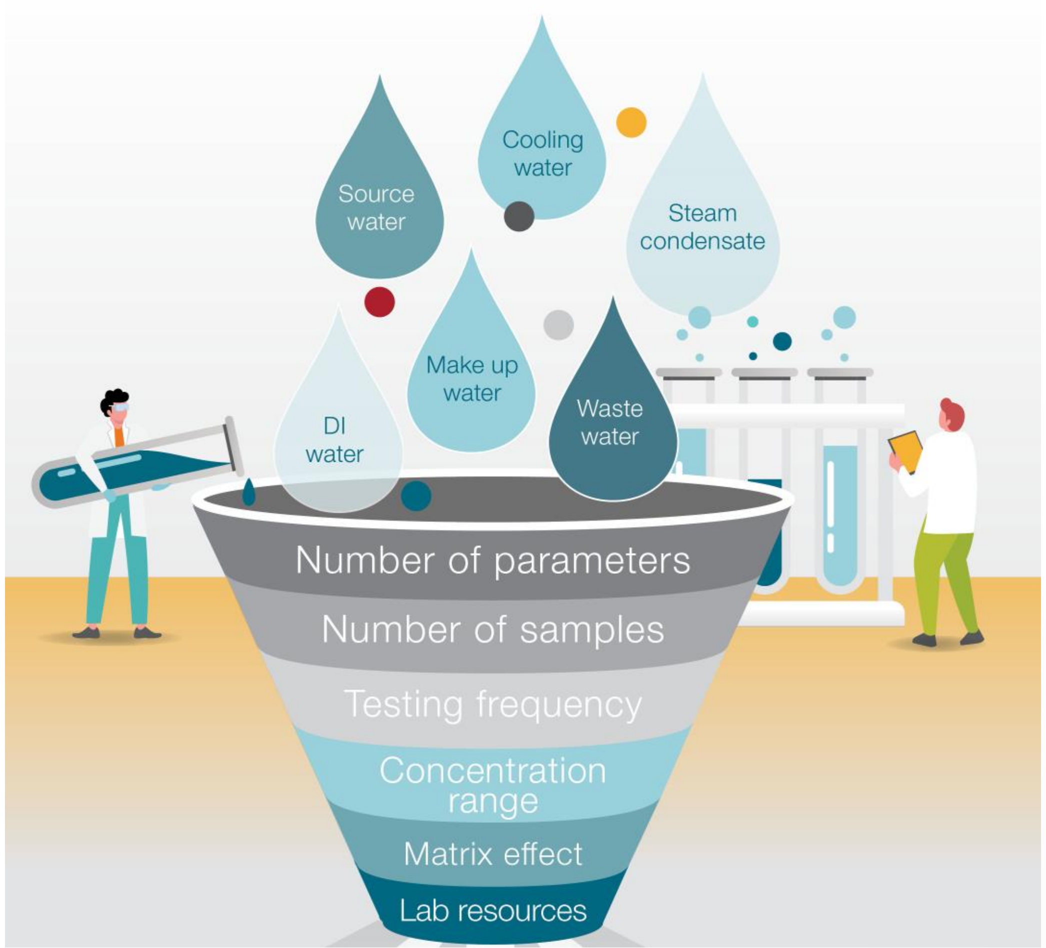


- Animal nutritional value
  - Farm animal manure for nutritional values and to give advice on animal feeding
  - Cows, hogs etc.

Soil analysis is critical for the determination of nutrients and contaminants

How does routine water and soil testing impact laboratory operations?

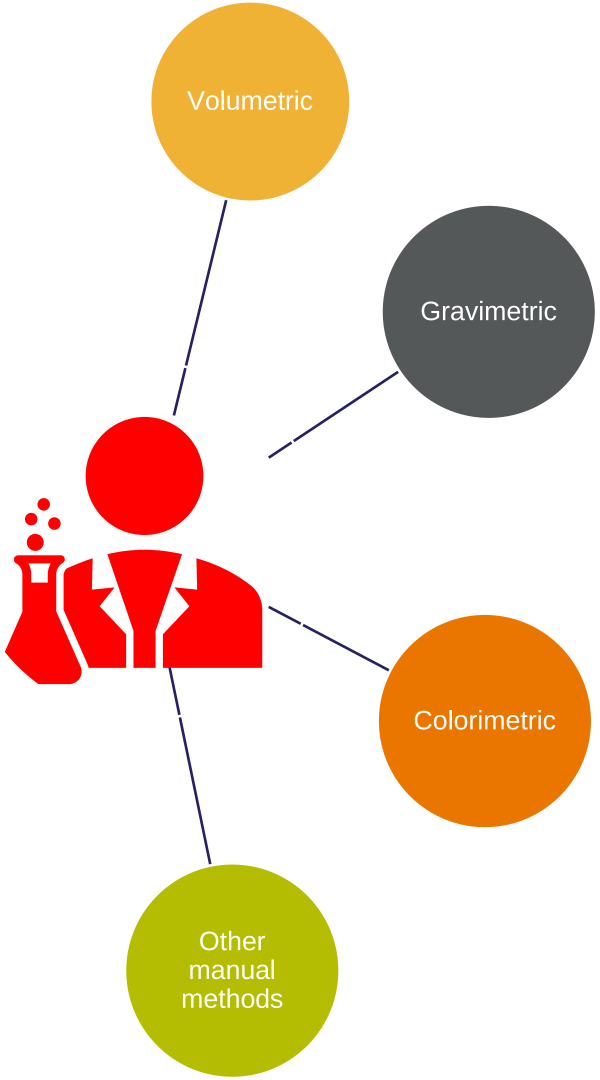
Many analytes, and diverse samples and concentrations



- Testing many samples for diverse parameters and concentrations can create a bottleneck with limited lab resources.
- Traditional analytical methods are slow, only process one parameter at a time—resulting in low throughput.

Analyte concentration varies from very low to high depending on the purpose and process

Wet chemistry analysis techniques and challenges



Spectrophotometers

Auto titrators

Flow injection analyzers (FIA)

Segmented flow analyzers (SFA)

- Manual techniques
- Visual detection
- Labor intensive
- Time consuming
- Large volume of reagent consumption per test
- Large waste generated

Water and soil testing can create a bottleneck for laboratories with limited resources

Consolidate water and soil analysis parameters

Basic water testing: pH, conductivity, alkalinity, total hardness

Comprehensive waste water testing as per regulatory methods: Total Kjeldal Nitrogen (TKN), total phosphate, total phenol, Total Oxidizable Nitrogen (TON), phosphate, nitrite, nitrate, boron, aluminium

Corrosive anions: Fluoride, chloride, sulfate, sulfide, nitrite, nitrate, phosphate, thiocyanate

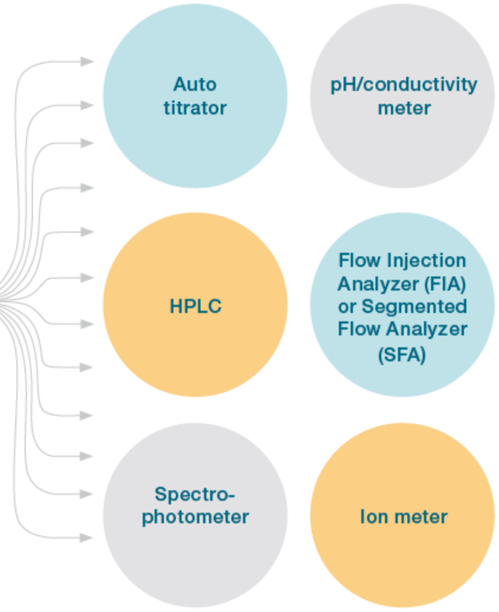
Scale formers: Silica, calcium, magnesium

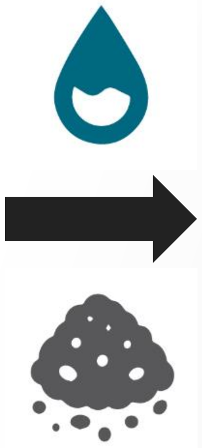
Corrosion inhibitors: Ammonia, zinc, molybdenum, nitrite

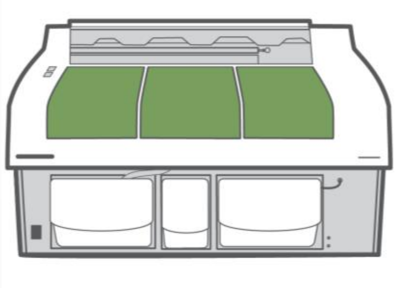
Corrosion indicators: Total iron, hexavalent chromium, zinc

Free and total cyanide

Regulatory fulfillment: Waste water analysis as per U.S. EPA and other standard methods







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Multiple parameters –
Multiple instruments – Multiple operators

Multiple parameters –
One instrument – One operator

What is wet chemical colorimetric analysis?

For a basic assay, in this case sulphate

- Add an aliquot of sample (100µl)
- Shine light (420 nm) through the sample and measure the intensity in absorbance units (Au)
- Add an aliquot of barium chloride reagent (40 µl)
- Wait for the reaction to occur (300 s)

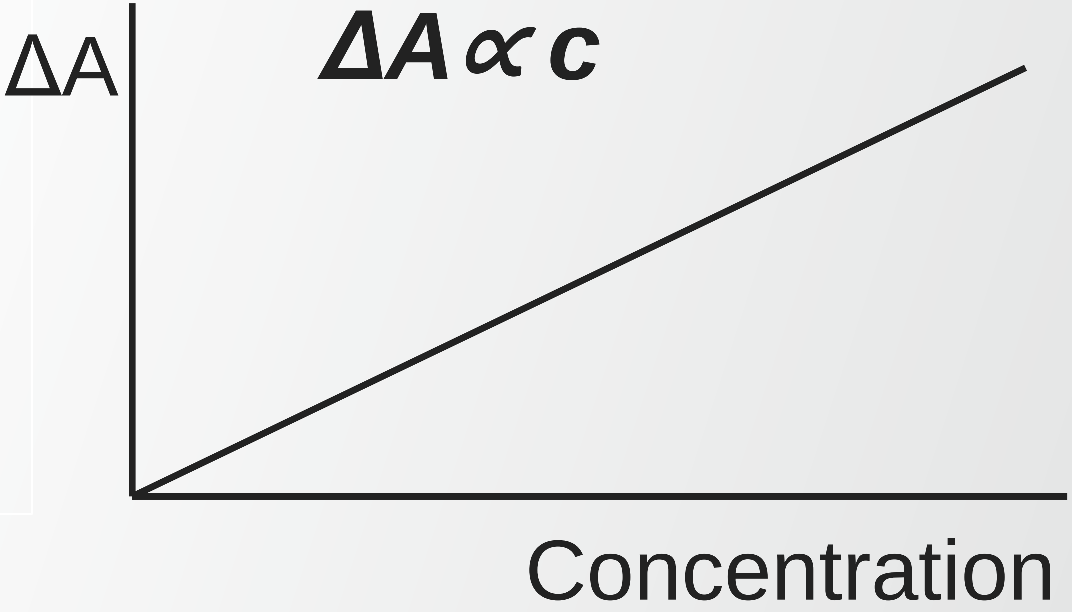
  $[SO_4^- + BaCl = BaSO4 + Cl^-]$

- Shine light (420 nm) through again and the difference in intensity is the change in absorbance ΔA

Beer–Lambert law

$$\Delta A = \varepsilon \times l \times c$$

$$\Delta A \propto c$$



Discrete analyzers in a nutshell

Photometric detection
- Colorimetric
- Enzymatic
- Electrochemistry

+

Liquid handling
- Sample aspiration
- Reagent addition
- Mixing and washing
- Calibration, QC, spiking, and dilution
- Incubation

+

Automation
- Random access
- Parallel analysis
- Auto-dilutions on overrange samples
- High throughput

Dispensing → Mixing → Incubation → Photometric measurement → Result interpretation

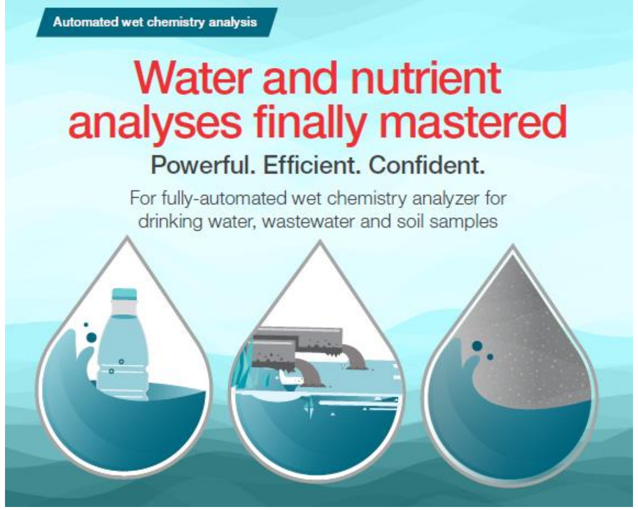
High throughput photometric analyzer

Simplified workflow solution

Highly automated workflows built for regulatory compliance

Recent advances of Gallery Aqua Master systems

Gallery™ Aqua Master systems for regulated water & soil testing



Software features for regulatory (EPA) compliance

- ✓ Choice of calibration order and relative standard error calculation (RSE)
- ✓ Automated spiking procedure for easy identification of sample-matrix-related interferences
- ✓ More flexibility for automated QC procedures
- ✓ More flexible results reporting
- ✓ Easy dilution rerun of multiple samples

New automation features reduce manual workarounds and risk of errors

Choice of calibration order, relative standard error calculation (RSE)

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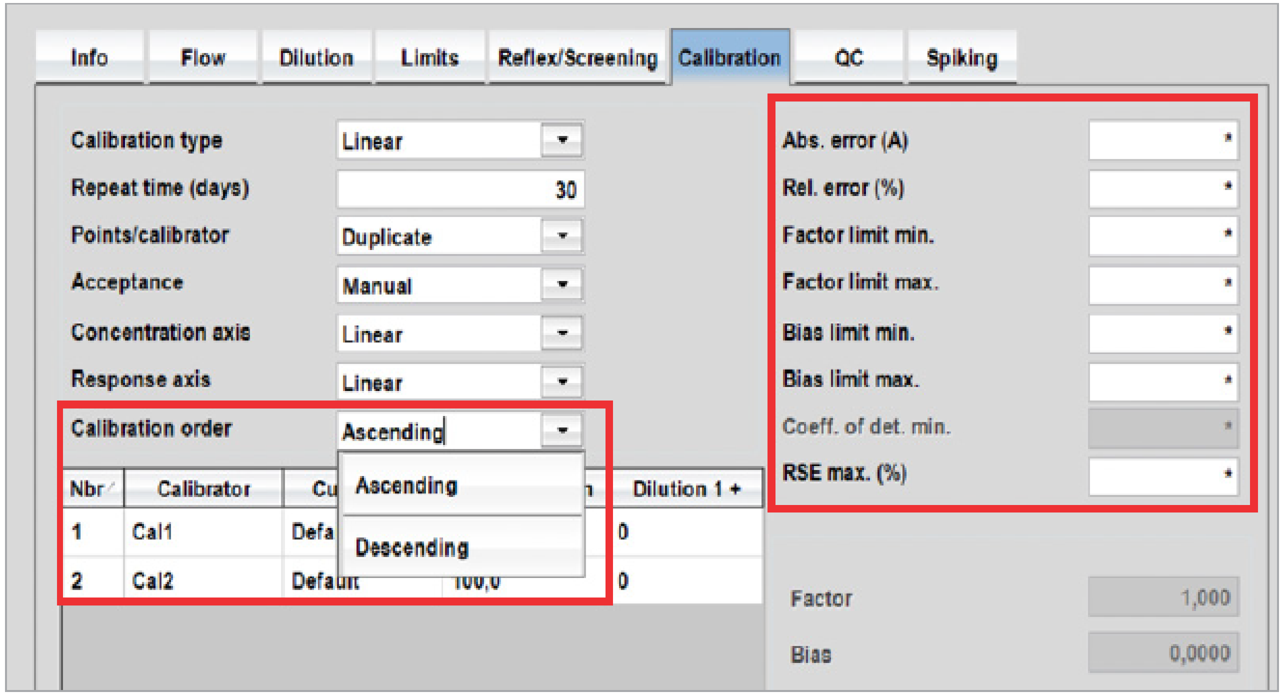
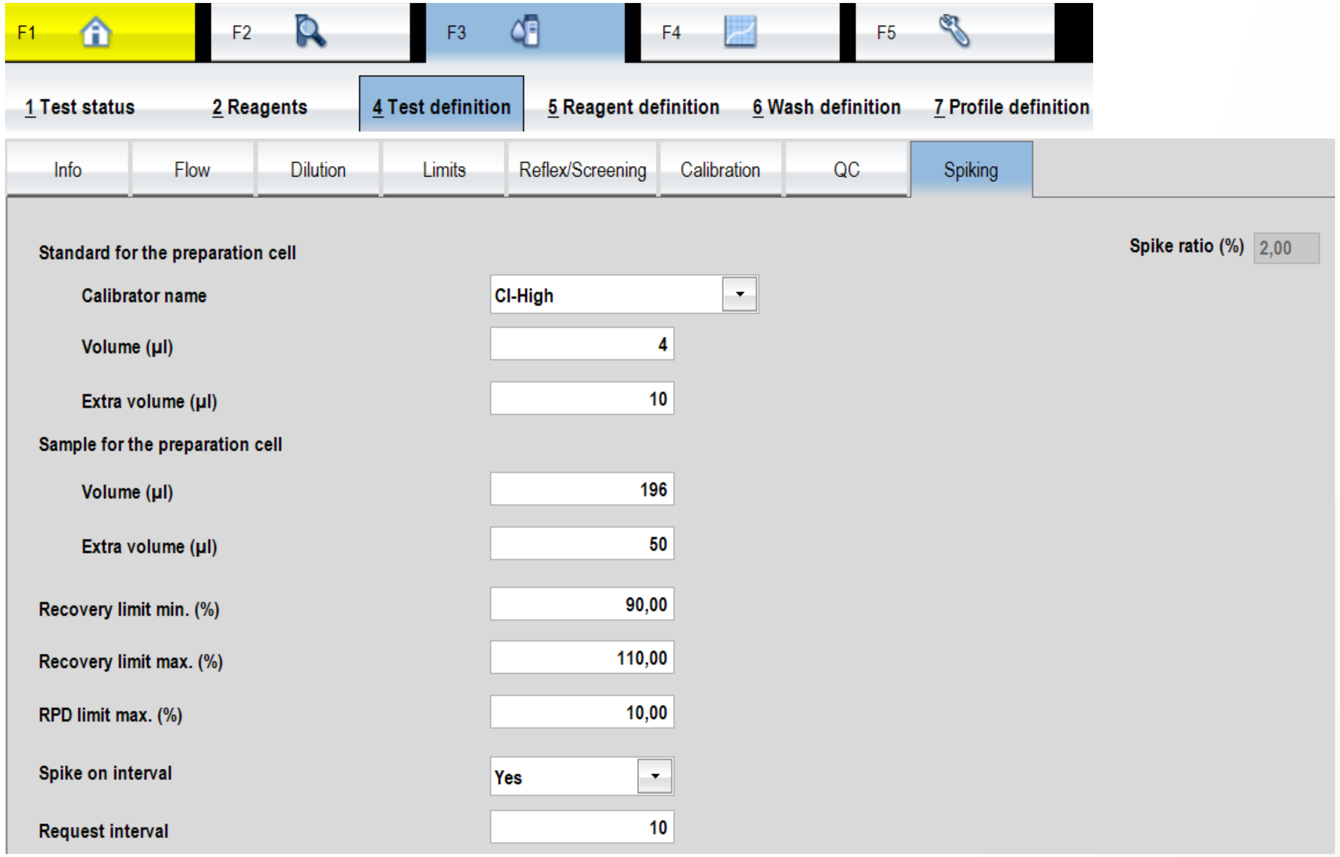
- Comprehensive calibration commands and flexibility
- Ascending or descending concentration calibration orders
- Automated RSE calculation with user-defined limits and automated error flagging if a limit is exceeded
- Instant feedback for calibration and goodness-of-fit — industry preferred



Choosing the calibration order to meet method requirements—either ascending or descending—is fast and easy using the Gallery Aqua Master discrete analyzer software.

Automated spiking procedure for easy identification of sample-matrix-related interferences

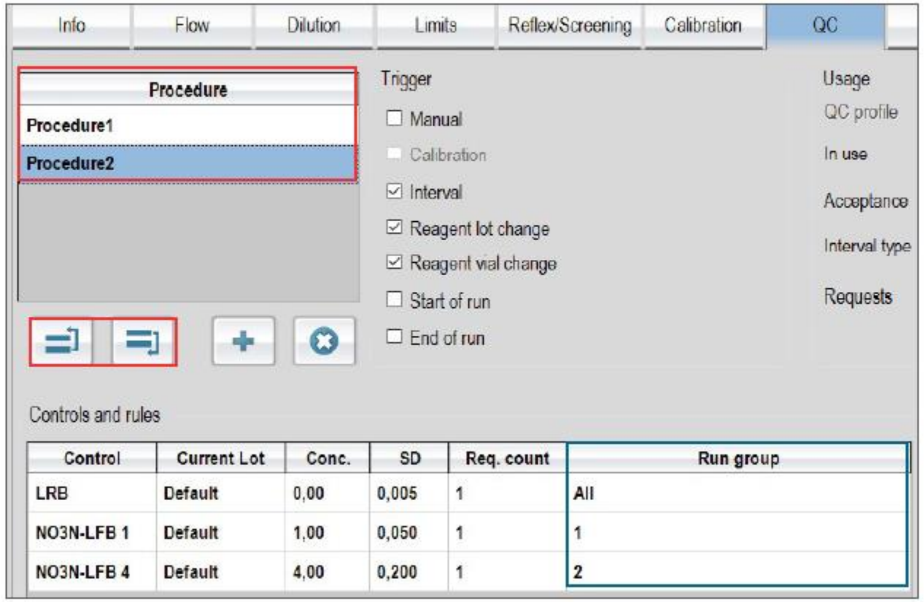


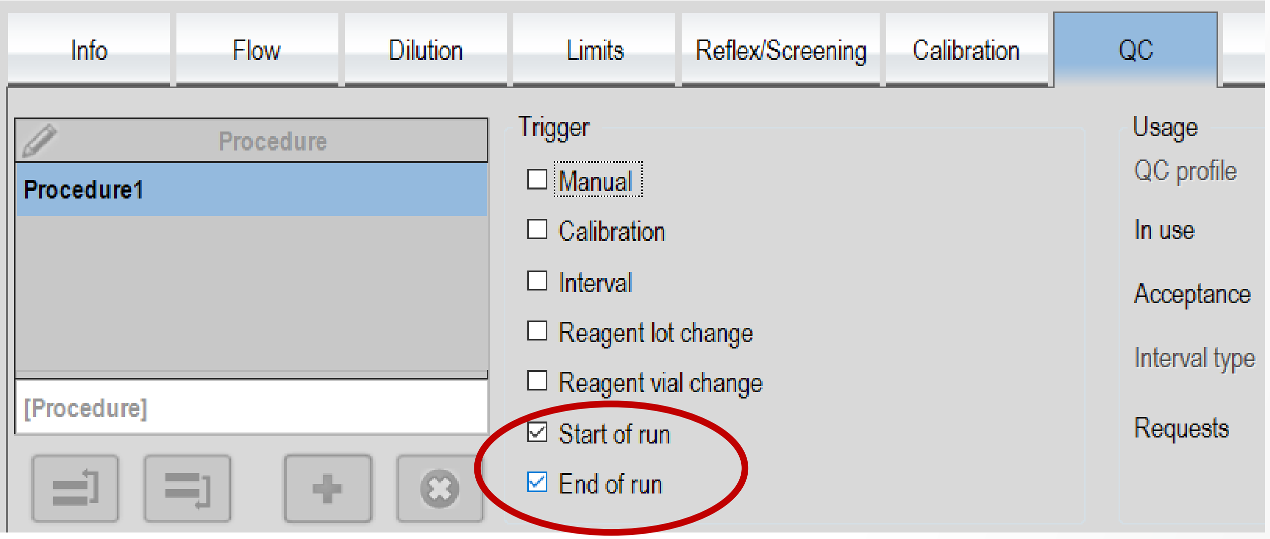
The Gallery Aqua Master discrete analyzer software provides for automated sample spiking that can streamline identification of sample-matrix-related interferences, including minimum and maximum limits for percent recovery and maximum limit for percent RPD

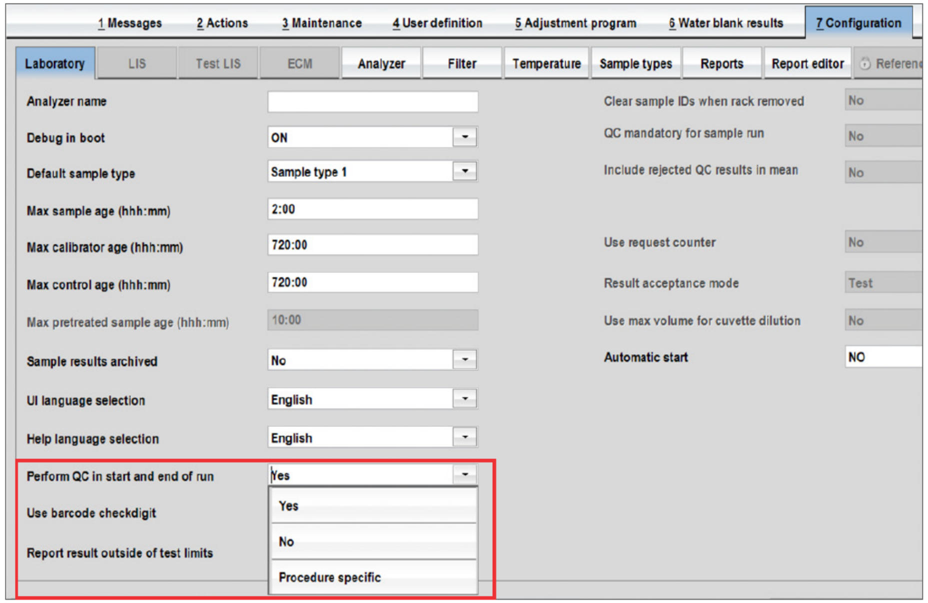
- Spiking series can be automatically and repeatedly performed at user-specified intervals
- Saves time and reduces error compared to manual sample spiking
- Automated calculation of the concentration of native and spiked samples, recovery for both spiked samples, and the Relative Percent Difference (RPD) between spiked samples
- User-defined minimum and maximum limits for percent recovery and maximum limit for RPD

More flexibility for automated QC procedures

- User-configurable QC elements and procedures can be set to run 1) at defined intervals, and 2) at the start or end of a run
- If multiple QC procedures must be run, their sequence is user definable

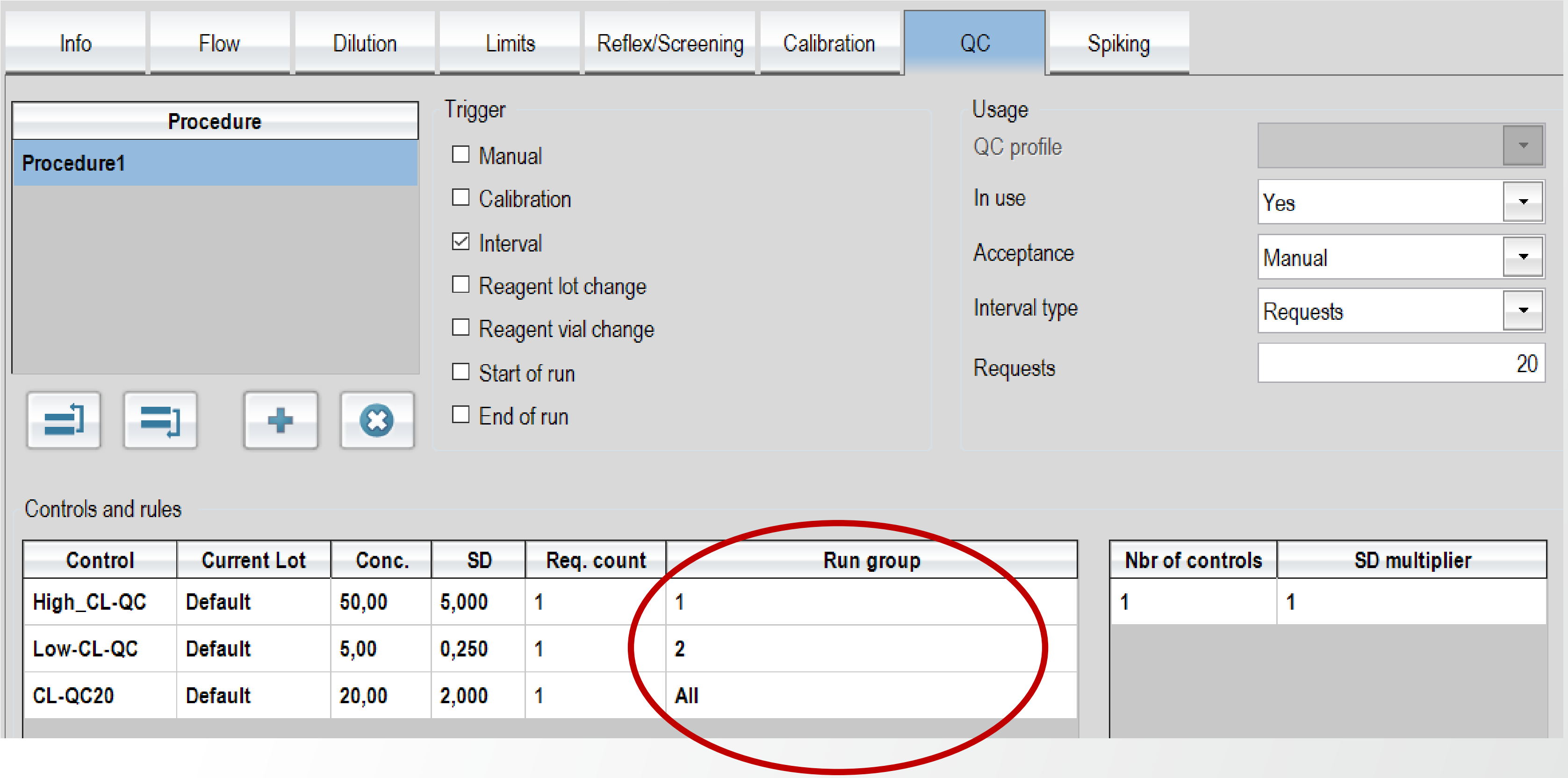






Run group for controls

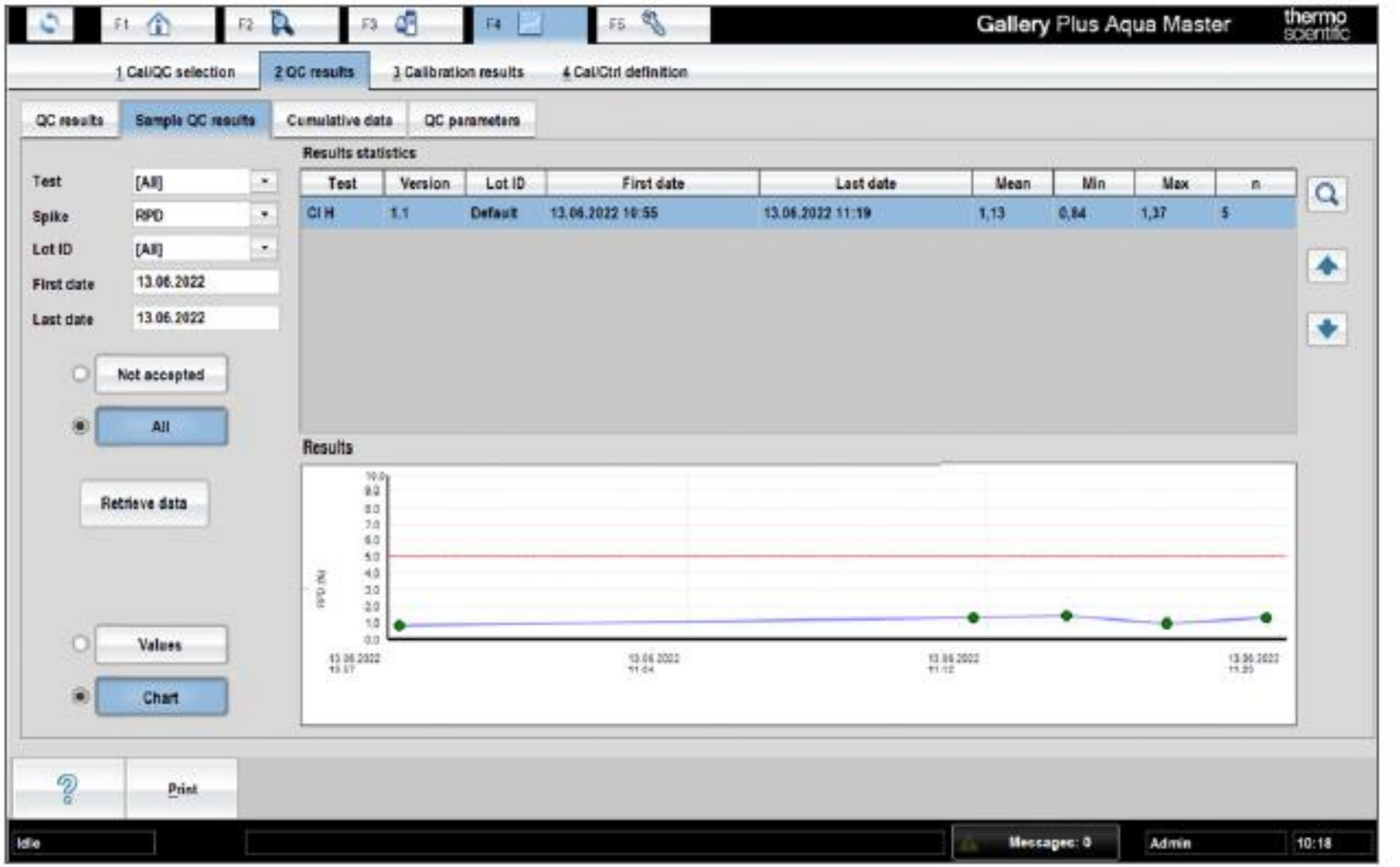
- Controls of a QC procedure have a new setting: Run group
- Controls can be defined to be part of a specific, or of all, run groups.
- Each time a QC procedure is run, the software will run the controls appropriate for the current run group
- The Run group setting provides flexibility for automatically alternating the controls that are run each time the procedure is run



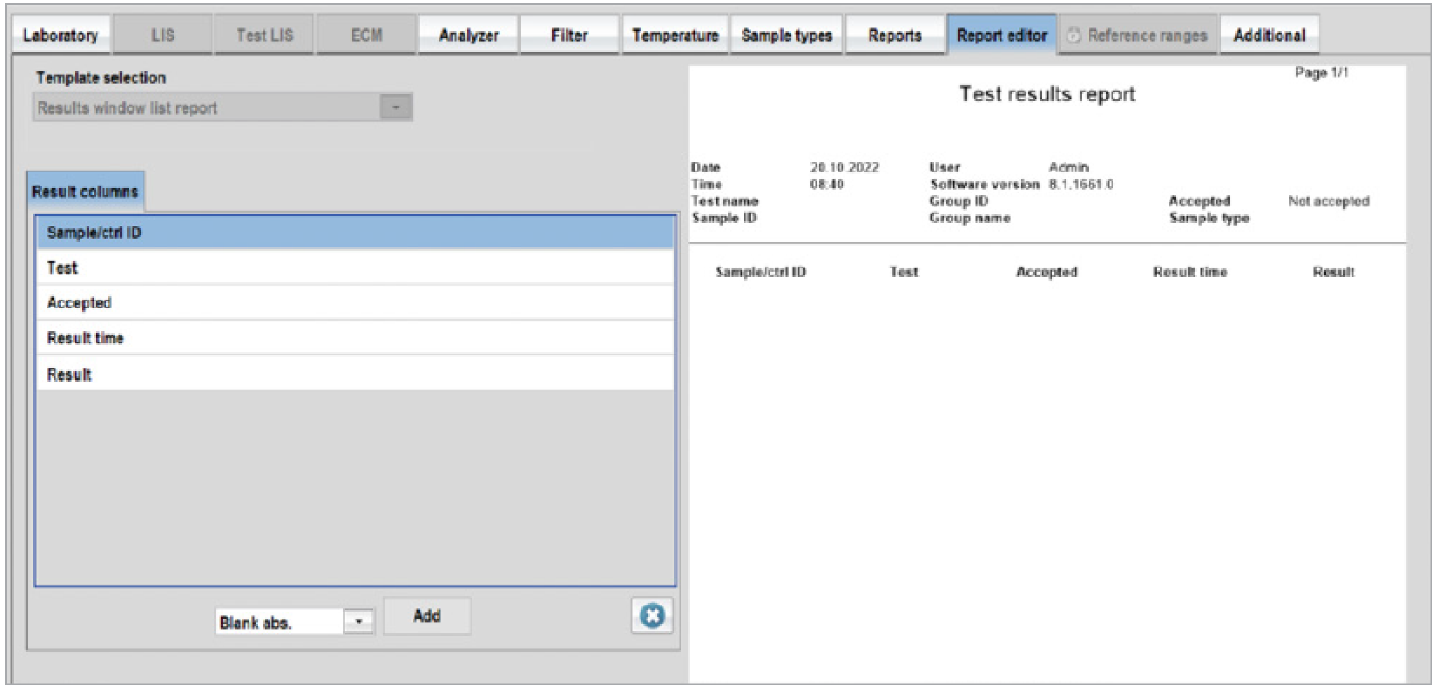
*Example above: The first time the procedure 1 is run, the control High-CL-QC belonging to **Run group 1** is run. The next time the procedure1 is run, the control Low-CL-QC belonging to **Run group 2** is run. As there are no more run groups defined, on the third round it starts again from run group 1. The control CL-QC20 is run every time the procedure1 is run, as it belongs to **Run group ”All”.***

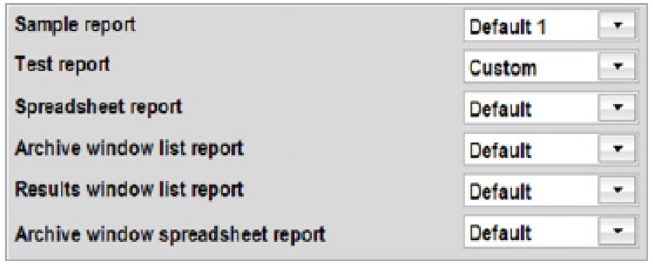
More flexibility for automated QC procedures

- Automated transfer of QC data into the Gallery Aqua Master discrete analyzer's QC charts to visualize trends
- Saves time compared to manually transferring data to LIMS or Shewhart control charts



Flexible results reporting





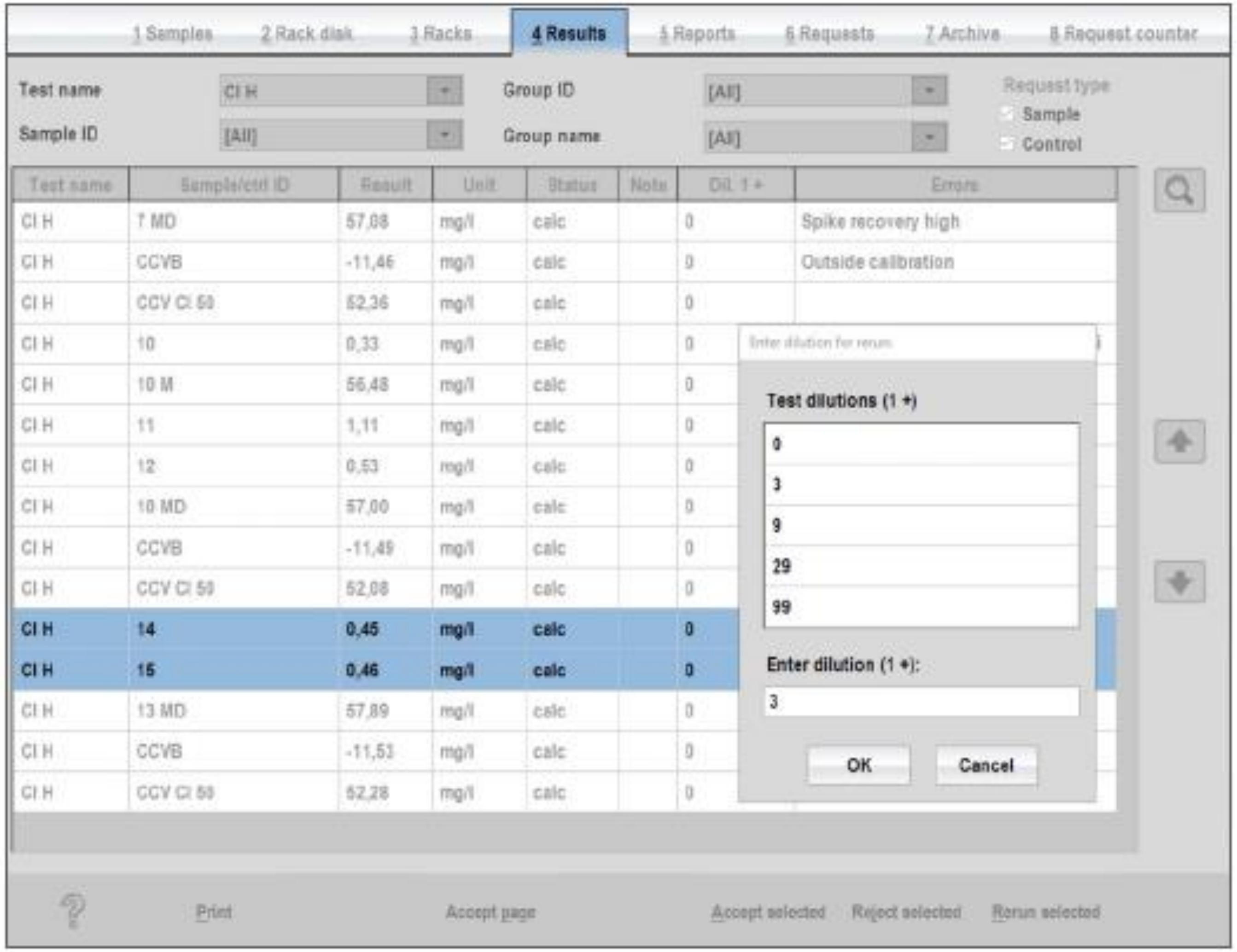
The report editor includes versatile capabilities to report test results, including report templates that can be easily customized to meet laboratory needs.

- Versatile results reporting, including predefined and easily customizable report templates available by test
- Environmental laboratories can report by tests, samples and or batches
- The report editor also allows customization of the header texts and logos used

Easy rerun of samples with new dilution factors

- Multiple samples can be rerun without having to individually define a new dilution factor for each

- Important usability improvement makes life a lot easier and enables time saved each day



To rerun samples using a new dilution factor simply select the samples to rerun and then enter the new dilution factor.

Built for regulated analyses

Environmental labs are highly regulated environments, as such analysis techniques & methods used must be compliant with recognized regulatory methods or standards.

✓ New automation features further reduce manual workaround and risk of errors

Gallery discrete analyzers

U.S. EPA wastewater reference methods

Analyte	Regulatory method
Alkalinity	EPA 310.2 (Rev. 1974)
Ammonia	EPA 350.1 (Rev. 2.0 1993)
	SM 4500-NH3-F
	SM 4500-NH3-G
COD*	EPA 410.4 (Rev. 2.0 1993)
Chloride	SM 4500-Cl-E
Chlorine (Total residual)*	SM 4500-Cl-G
Conductivity	EPA 120.1 (Rev. 1982)
Copper*	SM 3500-Cu-C
Cyanide (Amenable)*	SM 4500-CN-G
Cyanide (Total)*	EPA 335.4 (Rev. 1.0 1993)
	SM 4500-CN-E
Fluoride	SM 3500-F-D
Total hardness	EPA 130.1 (Issued 1971)
Chromium	SM 3500 Cr-B
Iron	SM 3500 Fe-B
Nitrate + Nitrite (TON) (Enzymatic reduction)	N07-0003
	ASTM D7781-14
	USGS I-2547-11
	USGS I-2548-11
Nitrate + Nitrite (TON) (Hydrazine reduction)	SM 4500-NO3-H
Nitrate + Nitrite (TON) (Vanadium reduction)	NEMI (Nitrate via manual Vanadium (III) reduction)
Nitrite	N07-0003 (Bypass enzymatic reduction)
	SM 4500 NO2-B
Orthophosphate	EPA 365.1 (Rev. 2.0 1993)
	SM 4500-P-E
pH	EPA 150.2 (Dec. 1982)
Silica	SM 4500 SiO2-C
	SM 4500 SiO2-D
Sulfate	SM 4500 SO4-E
	ASTM D516-16
Sulfide*	SM 4500-S2-D
Total Kjeldahl nitrogen (TKN)*	EPA 351.2 (Rev. 2.0 1993)
Total phenol*	EPA 420.1 (Rev. 1978)
Total phosphorous (TP)*	EPA 365.1 (Rev. 2.0 1993)
	EPA 365.4 (Issued 1974)
	SM 4500-P-E

* Third party reagent

U.S. EPA drinking water reference methods

Analyte	Regulatory method
Conductivity	SM 2510-B
Cyanide (Total)*	EPA 335.4
	SM 4500-E
Cyanide (Amenable)*	SM 4500-G
Fluoride	3500-F B, D
Nitrate + Nitrite (TON) (Enzymatic reduction)	NECi Nitrate-Reductase
Nitrite	NECi Nitrate-Reductase (Bypass enzyme)
	SM 4500-NO2-B
pH	EPA 150.2
Orthophosphate	EPA 365.1
	SM 4500 P-E
	SM 4500 P-F
Silica	SM 4500 SiO2-C
	SM 4500 SiO2-D

Methods compliant with U.S. EPA, NELAC, ASTM and other international standards

Overcome nitrate + nitrite (TON) measurement challenges

Using safer enzymatic methods in Gallery discrete analyzers

Limitations of traditional cadmium reduction coil methods

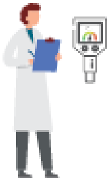
Carcinogenic health risk

Costly waste disposal

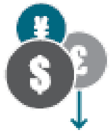
Time-consuming, manual methods

Advantages of enzymatic Nitrate + Nitrite method

Green chemistry

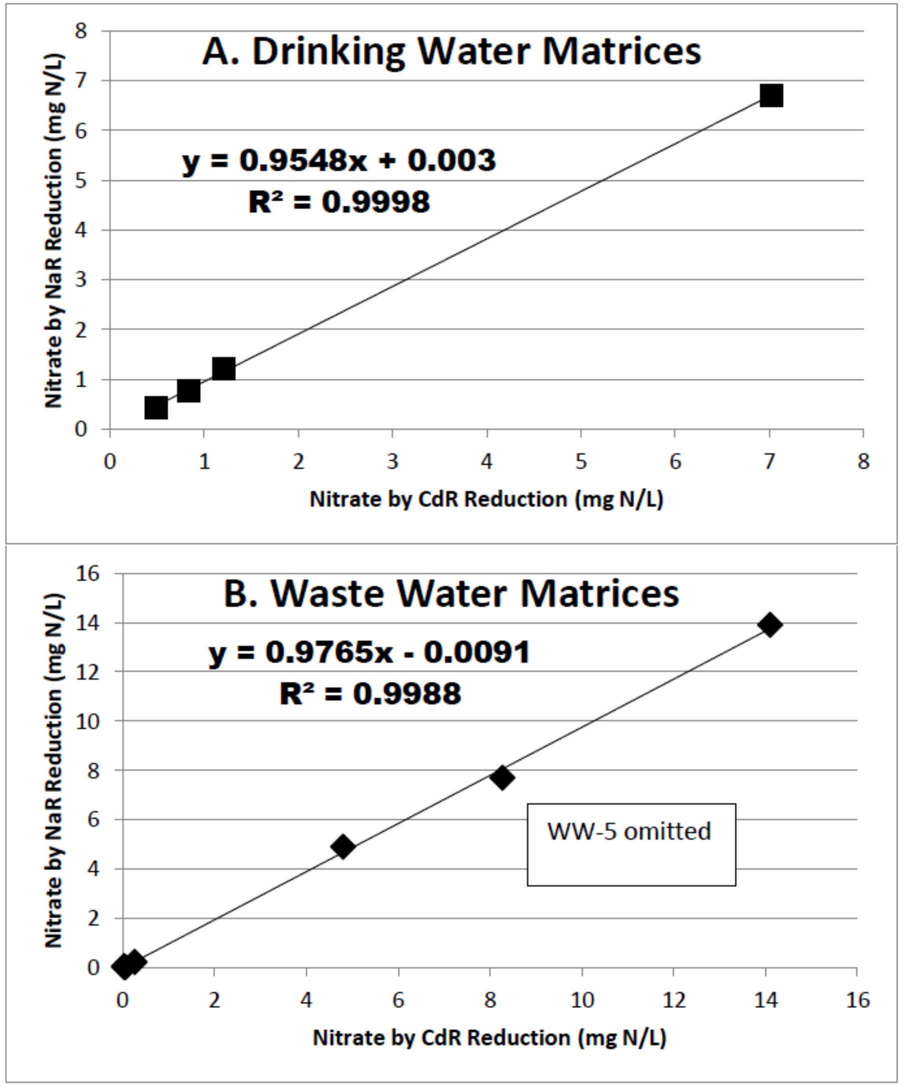


No sample preservation or pH adjustment



Cost effective

In compliance with 40 CFR Part 136, 40 CFR Part 141.23, 40 CFR Part 141, NECi–N07-0003, USGS I-2547-11, USGS I-2548-11 and, NECi Nitrate-Reductase regulatory standards.



Data courtesy to the NECi method report N07-0003

Figure 1A, 1B. Results comparison of cd reduction method and nitrate reductase method

Detecting nitrate + nitrite (TON) in drinking water

Official system method for EPA Safe Drinking Water Act (SDWA)

Automated method to detect the sum of nitrate + nitrite (TON) in drinking water using the Thermo Scientific Gallery discrete analyzer

Application benefits

- Meets requirements of regulated nitrate + nitrite total oxidized nitrogen methods
- Minimizes use of hazardous reagents and their associated costs and safety risks when carrying out nitrate + nitrite methods
- Automation and Thermo Scientific™ Gallery™ system reagents save time and reduce errors compared to manual approaches

Table 9. Performance of the Thermo Scientific drinking water method: drinking water nitrate + nitrite for Thermo Scientific Gallery discrete analyzer compared to reference method acceptance criteria

	QC acceptance criteria for reference method *Method for Nitrate Reductase Nitrate-Nitrogen Analysis of Drinking Water, Ver. 1.0 Rev. 2.0 Feb 2016*	Performance of nitrate + nitrite method for the Gallery Discrete Analyzer
MDL	To be determined by each laboratory according to 40 CFR Part 136 Appendix B. *(MDL results in reference method validation were between 0.0066 and 0.0097 mg N/L)*	MDL: 0.0038 mg N/L when done according to 40 CFR Part 136. Appendix B. MDL 0.009 mg N/L was applied*
MRL	NA	0.027 mg N/L
Reduction efficiency	90–110%	101–103%
Method blank	<MDL	*max. 0.0088 mg/L < MDL (0.009 mg N/L)
% Recovery initial calibration range (ICR)	±10% of true value, the equation between instrument responses and the nominal values of the standards must have regression coefficient (R^2) ≥ 0.999	9–110% for both primary and extended ranges (0.027–25 mg N/L). The R^2 was 1.000 for both ranges.
% Recovery QCS	±10%	94–97%
Recovery initial precision and recovery (IPR)	From CRM certificate *(ERA #698, lot: S234-698 acceptance limits 8.49–10.5 mg N/L)* or ±10% of known value, whichever is more restrictive.	Recovery IPR: 8.70–8.95 mg N/L (91–93% recovery)
% Recovery ongoing precision and recovery (OPR)	±10%	91–102%
% Recovery spike sample	±10%	91–100%
% RSD IPR	NA	1.2%
% RSD OPR	≤10%	1.4–1.9%
Relative percent difference (% RPD) spike duplicates	≤10%	0.1–0.4%

*The method criteria state that the LRB sample should never be higher than the MDL. The highest individual LRB result of the test was 0.0088 mg N/L.

Conclusion:

The results demonstrated that the Gallery discrete analyzer-based automated method meets the QC acceptance criteria in the EPA-approved reference method for testing drinking water.

Detecting orthophosphate in drinking water

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Official system method for EPA Safe Drinking Water Act (SDWA)

Automated method to detect orthophosphate in drinking water using the Thermo Scientific Gallery discrete analyzer

Application benefits

- Meets the requirements of regulated methods for orthophosphate testing in drinking water.
- Automation and ready-to-use reagents save time and reduce errors compared to manual approaches.

Table 10. Performance of the Thermo Scientific drinking water method: Drinking water orthophosphate for the Thermo Scientific Gallery discrete analyzer compared to the reference method acceptance criteria

	QC acceptance criteria for reference method: standard method 4500-P E.	Performance of drinking water ortho-PO4P method for the Gallery discrete analyzer
MDL	Minimum 7 replicates of MDL sample, conc. 1–5 × MDL estimate, analyzed in 3 days, ideally by different users. Recoveries should be 50–150% and % RSD ≤20%.	0.00036 mg P/L when the procedure in 40 CFR part 136, Appendix B was used. The procedure is similar to reference method.
MRL	Set at or above the lowest calibrator and verified with QC sample at 1 to 2 times the MRL conc. Result is acceptable if precision and accuracy meet laboratory method requirements.	0.0125 mg P/L. The MRL confirmation procedure was done per EPA requirements.[7]
Method blank	<1/2 MRL	Max. 0.0035 mg/L
% Recovery continuing calibration verification (CCV)	±10%	100–105%
% Recovery QCS	±15%	97–98%
% Recovery spike sample	Laboratory specific	92–98%
% RSD CCV	Laboratory specific	1.3–1.9%

Conclusion:

The results demonstrated that the Gallery discrete analyzer-based automated method meets the QC acceptance criteria in the EPA-approved reference method for testing drinking water.

Learn more about Gallery Aqua Master systems

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Thank you