Supelco®

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Spectroquant®

CombiCheck 20

1. Method

With the help of two ready-to-use multiparameter solutions (standard solution and addition solution) of defined content, measurement values are compared with desired values to determine any analytical errors.

The error caused by the photometric measurement system (test kits, photometers, analytical procedures) and the mode of operation can be determined by means of the **standard solution**. This is used **without dilution** in place of the sample solution.

Sample-dependent effects on the measurement result (matrix effects) are determined by measuring the sample after spiking it with **addition solution** (standard addition). The addition solution is a concentrated mixture of the ions contained in the standard solution.

The standard solution and the addition solution are prepared by accurate weighing in, using an analytical scales controlled with standard weights. All scales are regularly calibrated and checked against PTB standards (**P**hysikalisch-**T**echnische **B**undesanstalt, Germany) and NIST standards (**N**ational **I**nstitute of **S**tandards and **T**echnology, USA).

2. Applications

The CombiCheck articles serve to check the quality of the photometric measurement system and of the mode of working, as well as to identify sample-dependent effects on the measurement result. The latter is an important indication of the necessity for sample pretreatment. The measurement results are traceable to NIST.

Recommended frequency of application:

Standard solution (reagent R-1)	Addition solution (reagent R-2)
• 1 x per series [1)] • approx. every 10th sample • upon a switch in operating personnel • upon the opening of a new test package • when measurement results do not appear plausible	• when the composition of the individual samples differs considerably from each other • when measurement results do not appear plausible

[1)] To determine the random error, it is recommended to repeat the measurement at least five times.

3. Desired values and number of quality checks

The "working tolerance" given in column 2 of the following tables does **not** express the variation in the contents of the CombiCheck solutions (this lies below 1%), but instead the permissible dispersion of the results caused by the photometric measurement system and the mode of operation when using the respective test kit (column 3). The working tolerance defined in this manner serves as an assessment criterion for the quality check (see section 5).

Standard solution (reagent R-1)

Parameter	Desired value/ working tolerance	Can be used for Cat. No.	R-1 (ml)	Number of quality checks
Ammonium	12.0 ± 1.0 mg/l NH_4-N	114544	0.50	192
Chloride	60 ± 10 mg/l Cl^-	114730	1.0	96
COD	750 ± 75 mg/l COD	114541	3.0	32
	750 ± 75 mg/l COD	118752	2.0	48
	750 ± 75 mg/l COD	C4/25	3.0	32
Nitrate	9.0 ± 0.9 mg/l NO_2-N	114563	1.0	96
	9.0 ± 0.9 mg/l NO_3-N	N2/25	1.0	96
	9.0 ± 0.9 mg/l NO_3-N	114542	1.5	64
	9.0 ± 0.9 mg/l NO_3-N	109713 [1)]	0.50	192
	9.0 ± 0.9 mg/l NO_3-N	114773 [1)]	1.5	64
	9.0 ± 0.9 mg/l NO_3-N	114942	1.0	96
Phosphate [2)]	8.0 ± 0.7 mg/l PO_4-P	114729	1.0	96
	8.0 ± 0.7 mg/l PO_4-P	100475	1.0	96
	8.0 ± 0.7 mg/l PO_4-P	P7/25	1.0	96
Sulfate	500 ± 75 mg/l SO_4^{2-}	114564	1.0	96

[1)] when a 10-mm rectangular cell is used
[2)] Only the determination of orthophosphate can be checked.

Addition solution (reagent R-2)

Parameter	Desired value/ confidence interval	Can be used for Cat. No.	Sample + R-2 (ml)	Number of quality checks
Ammonium	8.0 ± 0.8 mg/l NH_4-N	114544	0.50 + 0.10	280
Chloride	40 ± 7 mg/l Cl^-	114730	1.0 + 0.10	280
COD	200 ± 40 mg/l COD	114541	3.0 + 0.10	280
	300 ± 40 mg/l COD	118752	2.0 + 0.10	280
	200 ± 40 mg/l COD	C4/25	3.0 + 0.10	280
Nitrate	7.5 ± 0.8 mg/l NO_3-N	114563	1.0 + 0.10	280
	7.5 ± 0.8 mg/l NO_3-N	N2/25	1.0 + 0.10	280
	5.0 ± 0.6 mg/l NO_3-N	114542	1.5 + 0.10	280
	15.0 ± 1.5 mg/l NO_3-N	109713 [1)]	0.50 + 0.10	280
	5.0 ± 0.6 mg/l NO_3-N	114773 [1)]	1.5 + 0.10	280
	7.5 ± 0.8 mg/l NO_3-N	114942	1.0 + 0.10	280
Phosphate [2)]	5.0 ± 0.5 mg/l PO_4-P	114729	1.0 + 0.10	280
	5.0 ± 0.5 mg/l PO_4-P	100475	1.0 + 0.10	280
	5.0 ± 0.5 mg/l PO_4-P	P7/25	1.0 + 0.10	280
Sulfate	150 ± 30 mg/l SO_4^{2-}	114564	1.0 + 0.10	280

[1)] when a 10-mm rectangular cell is used
[2)] Only the determination of orthophosphate can be checked.

4. Reagents and auxiliaries

The solutions are stable up to the date stated on the pack when stored closed at +15 to +25 °C.

Package contents:

1 bottle of reagent R-1 (standard solution)
1 bottle of reagent R-2 (addition solution)
1 control chart

5. Quality check

Standard solution (reagent R-1)

Preparation

- Make photocopies of the enclosed control chart.
- Take the desired value and the working tolerance for the standard solution for the respective test kit from the table in section 3 and enter them in the enclosed control chart:
  desired value at ➲, upper tolerance limit at ☊, lower tolerance limit at ☋.

Procedure

Proceed according to the instructions given in the package insert of the respective test kit and in the manual of the photometer used. In this case, however, use undiluted reagent R-1 in place of the sample without adjusting the pH!

Evaluation

Enter the measurement value as a number in a copy of the control chart and mark the grid at the corresponding place.
If the measurement value is **within the working tolerance** (grey background), the working materials and handling are in order.
If the measurement value is **outside the working tolerance**, a systematic error is present. In such a case, among other things the following aspects must be checked:

Standard solution and reagents
- expiry date not yet exceeded?
- storage under the recommended conditions?

Pipettes
- adjusted correctly?
- no contamination?
- correct handling?
- new pipette tip used?

Cells
- proper size?
- clean?

Sampling
- correct sample volume?

Sample pretreatment
- thermoreactor set correctly?
- solution colorless and without turbidity?

Procedure
- operating instructions adhered to?
- correct quantity and sequence of reagents?
- thoroughly mixed/dissolved after addition of reagents?
- reaction time and temperature adhered to?

Photometric measurement
- correct wavelength (filter) selected?
- correct factors entered?
- light path free from foreign matter/dust?

Addition solution (reagent R-2)

Procedure

In the following analyses always proceed according to the instructions given in the package insert of the test kit to be checked and in the manual of the photometer used.

- Analysis of the sample **(measurement value A)**
- Analysis of a spiked sample:
  When preparing the measurement solution 0.10 ml of reagent R-2 is added by pipette **immediately after the sample is added (measurement value B)**.

Evaluation

Calculate the concentration difference C:

C = B - A

If C is **within the working tolerance** for the addition solution (see table in section 3), the sample does not contain any compounds that interfere with the measurement.
If C is **outside the working tolerance**, a sample-dependent interference is present, and the measurement value A obtained with the sample is incorrect. **To obtain a correct result, the sample must be appropriately pretreated.**

Notes:

- Spiking with the addition solution must not cause the measuring range for the respective test to be exceeded!
  If this possibility is to be expected, the original sample must be appropriately diluted.
- The identification of interferences that cancel each other out and of individual interference factors is not possible using this method alone.

6. Notes

- Reclose the reagent bottles immediately after use.
- For photometric measurement the cells must be clean.
  Wipe, if necessary, with a clean dry cloth.
- For quality and batch certificates for Spectroquant® test kits see the website.
- Additional notes see under **www.qa-test-kits.com**.

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