



CERTIFICATE FOR

QC WW6

SUSPENDED SOLIDS (SS)

BATCH: VKI-31-3-0116

INSTRUCTIONS FOR USE OF THE REFERENCE MATERIAL

Description

This reference material consists of a bottle with concentrate for preparation of reference samples for quality control after dilution with water. The certificate includes documentation for the analytical parameter Suspended Solids (SS).

Quantity and Preservation

QC WW6 is a bottle containing 3.00 g concentrate from which 83 mL reference sample is prepared. The concentrate has been preserved by autoclaving.

Use

The reference material is intended for quality control, i.e. measurement and control of the accuracy and precision of analyses. It is typically intended for analyses of SS in wastewater samples. It may also be used in the quality control of other types of water samples and for the implementation and optimisation of analytical instruments and analytical methods.

It is important that the batch numbers of the reference material and on the certificate are identical.

Preparation for Use

Stabilise the bottle at room temperature (approx. 20°C). Open the bottle and add 80.0 mL water without a significant content of suspended solids (SS) to the bottle. Close the bottle and shake it well. Analyse the sample on the day when it is prepared. The certified concentration is given in the table on page 3 of this certificate.

<u>Note</u>: The reference material is to be prepared in the bottle. It is important to ensure sufficient purity of the water used for preparation.

If QC WW6 is used for other purposes than quality control of wastewater analyses, the dilution ratio between the concentrate and water can be altered.

Analysis

For quality control the reference material is analysed at the same time and in the same manner as other samples.

Storage and Durability

Store the bottle protected from sunlight, e.g. in the original box, and at room temperature or in a refrigerator. The certificate is valid until 1st of April 2024 provided the material is stored under the recommended conditions.

After preparation, the reference material has an expected storage time of up to 24 hours.

PRODUCTION OF THE REFERENCE MATERIAL AND DOCUMENTATION

Production

The production of this reference material is in accordance with the quality management procedures of Eurofins Miljø A/S, with the aim to obtain the intended quality of the material.

Documentation of Content

All documentation for the reference material has been performed after dilution as described above.

Internal control

The analytical quality of Eurofins Miljø has been documented and found satisfactory by regular participation in international proficiency tests.

Homogeneity:

The homogeneity has been investigated by measurements of SS in randomly selected bottles of QC WW6. Tests for homogeneity have been performed by comparing the standard deviation between the reference material units with the within batch standard deviation obtained from duplicate measurements of the reference material in the same bottle (F-test, 95%). In addition, homogeneity testing was included in the external control. No indication of heterogenity was found.

Stability:

The stability of the reference material is being followed at 5°C, 20°C and 37°C.

External control

The external laboratory documentation was performed by selected laboratories in an external documentation in April-May 2016. The participating laboratories are skilled and have documented good analytical quality by participation in interlaboratory comparisons and by analysis of a control sample in the certification. The laboratories were requested to analyse five bottles of QC WW6: three bottles in the same analytical series, one as a duplicate determination and the others as a single determination, and in addition two bottles in two different analytical series as single determinations. In addition, the laboratories were requested to analyse a control sample sent together with the reference material. The statistics are in accordance with the international standard: ISO Guide 35 /1/. On the basis of the analytical results submitted by the laboratories the following statistical parameters have been calculated:

 y_{char} : average, calculated in accordance with ISO Guide 35 (section A.2.4.)

s(y): standard deviation between the laboratories, calculated in accordance with ISO Guide 35 (section A.2.5.):

$$\sqrt{\frac{\sum (y_i - y_{char})^2}{p-1}}$$

The 95% confidence interval of the true mean value of analytical results is:

$$y_{char} \pm t_{0,025}(\nu) \cdot \frac{s(y)}{\sqrt{p}}$$

where

p: number of laboratories included in calculations

v: p-1, degrees of freedom

 $t_{0,025}(v)$: t value of 0,025 level at v degrees of freedom.

The criteria for selection of laboratories were as follows:

- the laboratory results in the above-mentioned proficiency tests diverged less than 2 standard deviations from the nominal value,
- the laboratory analyses more than 20 series of samples each year or holds an accreditation for SS,.
- the laboratory result for the control sample in the certification study deviated less than 15% from the nominal value, and
- the laboratory results in the certification study are not Cochran outliers, Grubbs outliers or deemed to be an outlier on the basis of a scientific evaluation.

The data included in the external control and names of the participating laboratories are listed in an annex to this certificate. On the basis of the selected results, the following has been calculated:

Certified Value

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DETERMINAND	UNIT	AVERAGE	BETWEEN LABORATORY STANDARD DEVIATION	95% CONFIDENCE LIMITS OF THE AVERAGE VALUE		NUMBER OF DATA SETS IN CALCULATIONS/ METHOD	EXCLUDED DATA SETS
				$y_{char} \pm t_{0.025}(v) \cdot \frac{s(y)}{\sqrt{p}}$			
		${\cal Y}_{char}$	s(<i>y</i>)	Lower	Upper	(p)	
SS	mg/L	241	3.5	240	243	5 / L 25 / M	4

Methods

L DS 207, NS 4733.

M EN 872

Use of the Certified Values

For laboratories with an analytical quality that is comparable with that of the laboratories who have contributed to this certification, the following applies:

1) For <u>single determinations</u>, analytical results will with a probability of 95% be in the interval:

$$y_{char} \pm t_{0.025}(v) \cdot s(y)$$

 Analytical results, calculated as the <u>average of two determinations</u> will with a probability of 95% be in the interval:

$$y_{char} \pm t_{0,025}(\nu) \cdot \frac{s(y)}{\sqrt{2}}$$

REFERENCES

- /1/ ISO guide 35:2017. Reference materials Guidance for characterization and assessment of homogeneity and stability.
- /2/ ISO guide 31:2015 Reference materials Contents of certificates, labels and accompanying documentation.

Date of issue: February 2023

RESPONSIBLE SCIENTIST

Rikke Mikkelsen, MSc Eurofins Miljø A/S DK-8464 Galten

Certificate revision history: February 2023 (expiry date extended; update to newest version of ISO Guide 31 and 35); April 2020 (expiry date extended); June 2016 (original certificate date)

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ANNEX TO CERTIFICATE QC WW6

Laboratory Measurements

SS									
y _i mg/L	s _{ri} mg/L	n _{ri}	s _{Li} mg/L	n _{Li}	IVICITIOU				
239.5	5.99	4	2.69	3	BM				
241.7	3.75	4	1.30	3	AM				
239.8	0.24	4	0.18	3	CM				
241	4.16	4	2.08	3	AL				
240.0	2.39	4	1.65	3	CM				
242	4.35	4	3.53	3	CM				
242	0.96	4	9.06	3	AM				
232.7	1.96	4	5.22	3	AM				
240.0	2.39	4	6.34	3	AM				
241.95	3.29	4	1.87	3	DM				
236.9	3.75	4	6.16	3	BM				
243	2.06	4	0.14	3	AM				
241.7	3.23	4	4.39	3	AM				
236.9	3.75	4	4.88	3	DM				
246.6	3.36	4	3.26	3	ZL				
238	1.41	4	0.00	3	СМ				
234.6	1.66	4	4.11	3	AM				
247.9	8.19	4	0.68	3	ZM				
246	1.41	4	4.04	3	BM				
246	8.37	4	2.08	3	AL				
242	1.15	4	1.53	3	BL				
245	5.94	4	1.53	3	СМ				
245	3.79	4	3.91	3	AL				
237	2.63	4	7.80	3	AM				
240.3	1.66	4	4.22	3	СМ				
244	4.35	4	3.53	3	AM				
244.5	1.22	4	1.31	3	AM				
241.93	3.01	4	2.47	3	СМ				
239.2	4.73	4	4.52	3	AM				
241.0	2.99	4	2.66	3	BM				

External Control Values

 y_i : average for laboratory i

s_{ri}: standard deviation for laboratory i within an

analytical series

 $n_{ri}\,$: number of results for determination of s_{ri}

 s_{Li} : standard deviation for laboratory i between

analytical series

 n_{Li} : number of results for determination of s_{Li}

Methods: See explanation on page 3.

ANNEX TO CERTIFICATE QC WW6

Certifying Laboratories

Denmark BIOFOS A/S, København K Eurofins Miljø A/S, Vejen Renseanlæg Bjergmarken, Roskilde Vandrens, Skælskør

Finland

Nab Labs Oy Ltd/Ambiotica, Jyväskylä

Norway
Alcontrol Skien, Skien
Eurofins Environment Testing Norway AS, Moss
Fishguard Avd. Måløy, Måløy
Hardanger Miljøsenter AS, Odda
Ivar IKS, Randaberg
Labora AS, Bodø
Mjøslab IKS, Gjøvik
Vestfoldlab A/S, Sem

Sweden

Iggesund Paperboard AB, Cell o. Miljölab, Iggesund INOVYN Sverige AB, Stenungsund Jönköpings Kommun VA-laboratoriet, Jönköping Karlskrona Kommuns Laboratorium, Lyckeby Motala Kommun, Tekn. Förvaltn. Vatten o. Avfall, Motala Norrköping Vatten o. Avfall AB, Laboratoriet Slottshagens Rv., Norrköping Nyköping Kommun / Vattenlaboratoriet, Nyköping Preemraff Lysekil, Lysekil Reningsverket Aggerud, Karlskoga St1 Refinery AB, Göteborg Stora Enso Paper AB, Hylte Mill, Hyltebruk Tekniska Verken i Linköping, Linköping Tekniske Förvaltningen, Verksamhetsstöd VA, Reningsverket Skebäck, Laboratoriet, Örebro VA SYD Ellingelaboratoriet, Malmö Vattenfall AB Heat Generation Uppsala, Kemilaboratoriet, Uppsala Vimmerby Energi o. Miljö, Vimmerby VIVAB, Varberg