INSTRUCTIONS FOR THE QUANTITATIVE ANALYSIS OF WASTEWATER FOR WATER SOLUBLE POLYMERS (WSSP)

Kontakt

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CONTENT

SCOPE OF APPLICATION

The scope of this manual is the detection
of Water Soluble Synthetic Polymers (WSSP) in water.
WSSP can be detected and quantified by a chemical reaction
usings iodine, potassium iodide and boric acid.
The resulting reaction builds a colored complex of which
the intensity is linear to the concentration.

Because every WSSP reacts differently, a distinction
is made between two methods: a qualitative method
and a quantitative method.

If the WSSP or WSSP mixture in the sample is not known, the qualitative method can be used to determine how much WSSP is removed by measuring before and after the removal process.

If the WSSP to be analyzed in the sample is known, the quantitative method can be used to measure the exact concentration in a sample after a WSSP-specific calibration curve has been created.

EXCLUSION OF LIABILITY

This manual describes the standard procedure for the quantitative determination of WSSP.

The SOP is the property of Wasser 3.0 gGmbH.
It serves as the basis for our work.

Wasser 3.0 gGmbH accepts no liability for personal injury or damage to property resulting from the use of the SOP.

LABORATORY PREPARATION

- Work in a climatized environment. Temperature has a large influence on the experiments. The temperature should always remain constant for creating comparable results. We recommend 21 °C.
- All chemicals, solutions and samples should be at room temperature (21 °C) before starting the experiment.
- Only use clean equipment and always rinse laboratory glassware 3 times with distilled water before use.
- Make sure the expiration date of the used chemicals has not been exceeded.
- When working with chemicals, the general hazard warnings and protective measures apply, which can be found in the chemical's safety data sheets.

EXCLAMATION MARK (GHS07)

CAUTION!

Acute toxicity, skin corrosion/ irritation, serious eye irritation/damage, skin sensitisation, toxicity to specific target organs

CORROSIVE EFFECT (GHS05)

CAUTION!

Hazardous to environment

DANGER TO HEALTH (GHS08)

CAUTION!

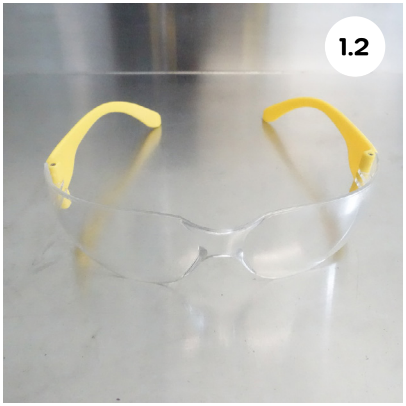
Respiratory sensitization; Germ cell mutagenicity; Carcinogenicity; Reproductive toxicity; Specific target organ toxicity (single or repeated exposure); Aspiration hazard

CAUTION!

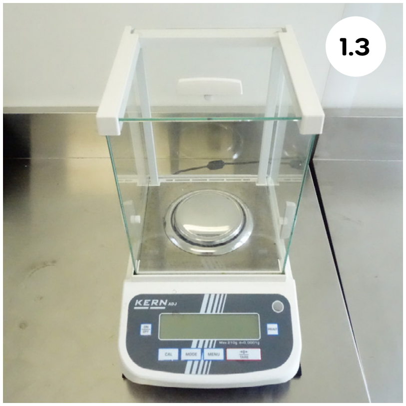
Danger of burning or scalding the skin.

REQUIRED EQUIPMENT

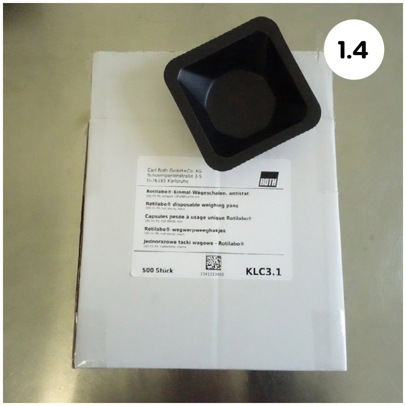
Nitril gloves



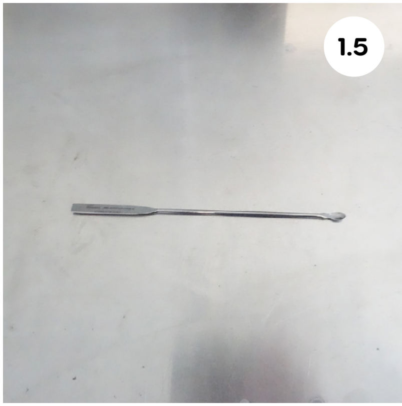
Protective glasses



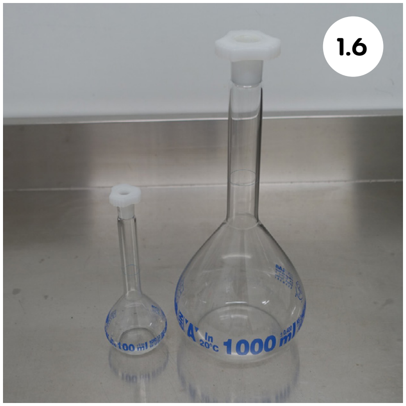
Analytical balance
Model: ADJ 200-4
Manufacturer: Kern



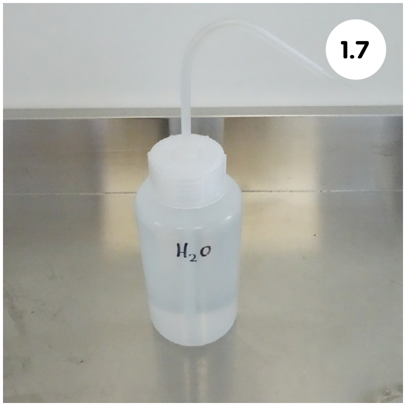
Weighing pans



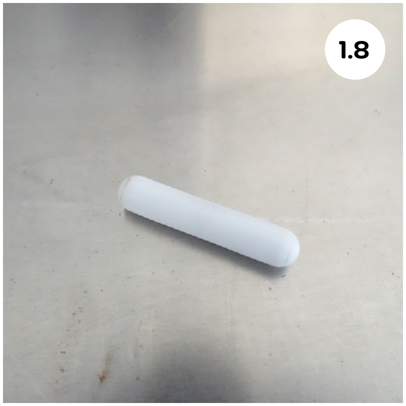
Spatula



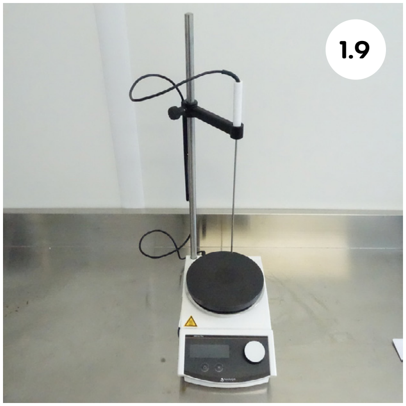
100 mL and 1 L volumetric flasks



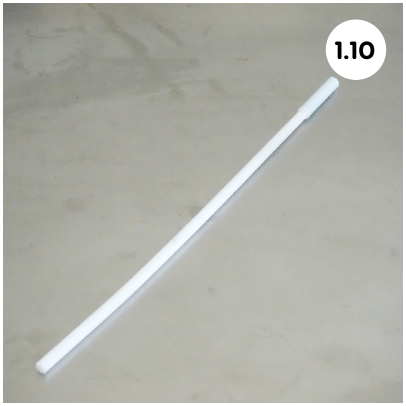
Wash bottle with distilled water



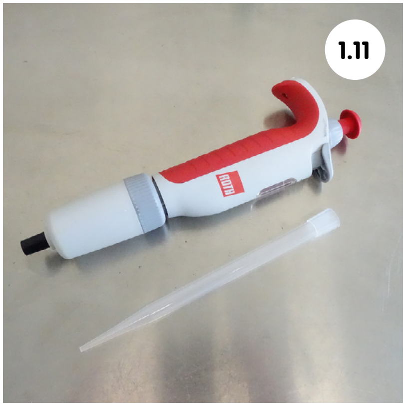
Magnetic stirring bar



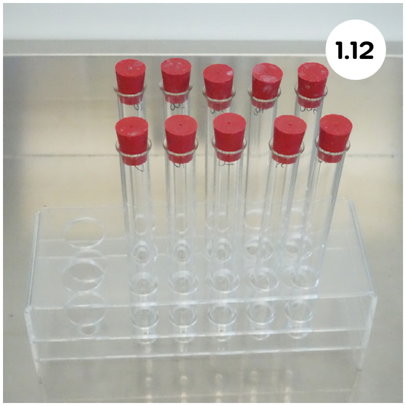
Magnetic stirrer with hotplate
Model: MR Hei-Tec + pt1000
Temperatur Fühler
Manufacturer: Heidolph



Stirring bar retriever



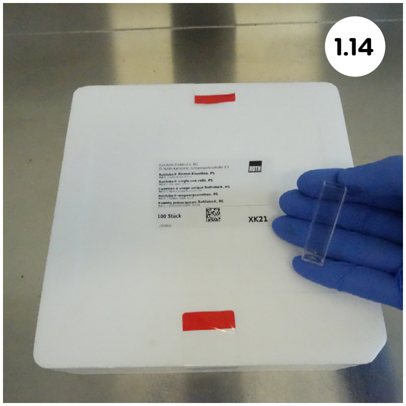
Micropipette + tips



Test tubes with stoppers and stand

REQUIRED EQUIPMENT

Timer

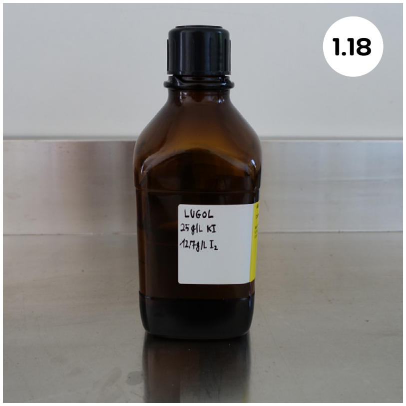


10 mm – PS Makro Cuvettes
Art. No.: XK21
Manufacturer: Carl Roth

Spectrophotometer
Model: Nanocolor - UV/VIS II
Manufacturer: Macherey Nagel

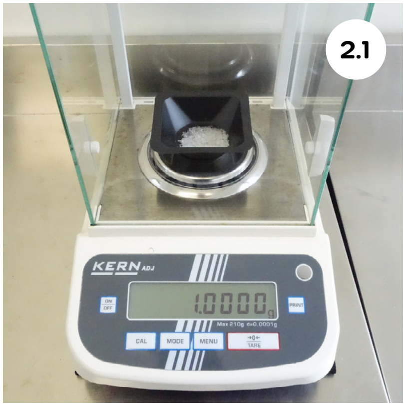
abcr Poly(vinyl alcohol), Mowiol®
28-99, Mw ±145.000
AB 578256

abcr Boric acid; 40 g/L

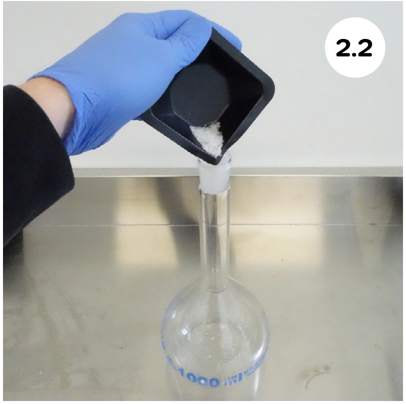


abcr Lugol´s solution
25 g/L KI + 12.7 g/L I_2

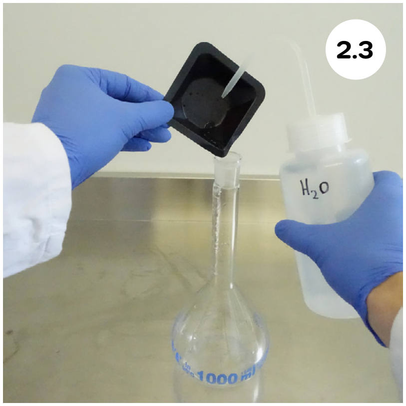
PREPARATION OF THE STOCK STANDARD SOLUTION



Measure 1 g of the WSSP

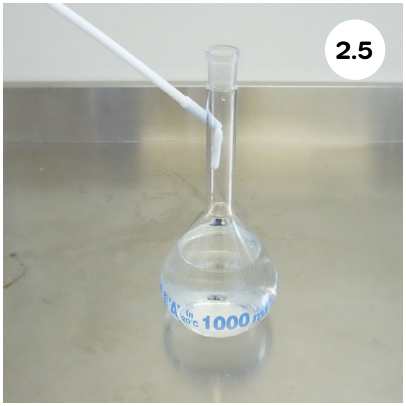


Transfer to a 1 L volumetric flask

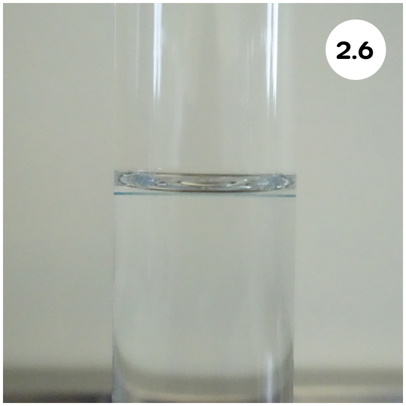


Rinse and fill the flask with about 800 mL of distilled water

Add a stirring bar and heat to 90°C while stirring at 300 rotations per minute



Let the solution cool down until room temperature is reached and remove the stirring bar. Don't forget to rinse the stirring bar into the volumetric flask.



Fill the flask to the 1 L mark

PREPARATION OF THE SERIAL DILUTION

To prepare the serial dilution, the following experimental parameters need to be determined:

- The concentration range
- How many different concentrations will be prepared
- The step between concentrations
- Amount of solution required

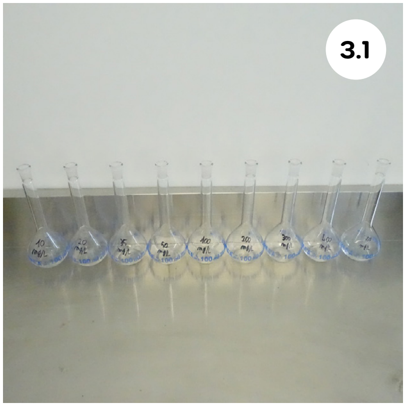
These parameters depend on the goal of the experiment and also on the type of WSSP being analyzed. This manual uses abcr Poly(vinyl alcohol), Mowiol® 28-99, Mw ±145.0, AB 578256; as an example. For other WSSPs, different concentrations may be more optimal.

The chosen parameters are 9 different concentrations ranging from 0 mg/L to 500 mg/L. To calculate how much of the stock solution is required and the corresponding amount of water needed, the following formula can be used:

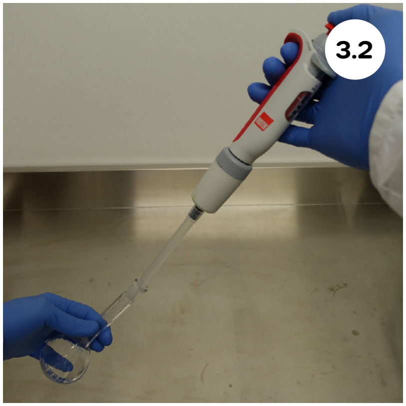
$$\frac{\textit{Wanted concentration}\ \frac{mg}{L} * \textit{Volume of the volumeric flask (mL)}}{\textit{Concentration of the stock solution}\ \frac{mg}{L}} = \textit{Volume of stock solution needed (mL)}$$

Below the results, starting from a 1000 mg/L stock solution using 100 mL volumetric flasks to make the serial dilution.

Wanted Concentration (mg/L)	0	10	20	35	50	100	200	300	400	500
Volume (in mL) of stock solution needed in 100 mL	0	1	2	3,5	5	10	20	30	40	50

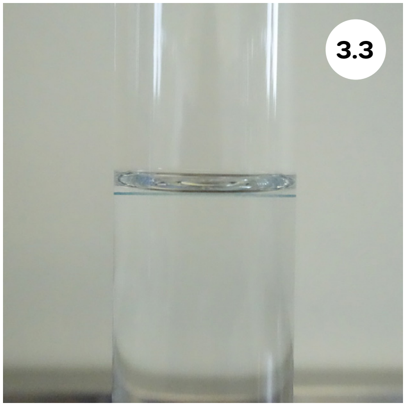


Rinse 9x 100 mL volumetric flasks with distilled water and name them according to the concentrations in the table on p. 14.



Transfer the correct amount of stock solution as described in the table to the flask for each concentration.

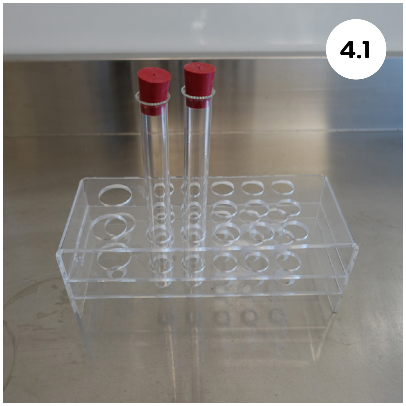
(Do this slowly to prevent formation of foam)



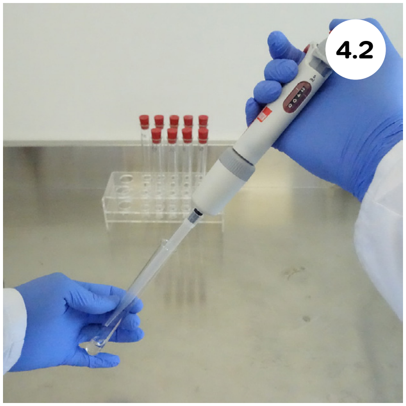
Fill the flasks to the 100 mL mark.

(Do this slowly to prevent formation of foam)

To determine the optimal wavelength (λ_{max}), 3 of the serial dilution solutions are selected to cover the entire concentration range. Thus the measured absorbance falls once between 0 and 1, once between 1 and 2 and once between 2 and 3. The average of these 3 maxima determines the λ_{max} max for this WSSP.

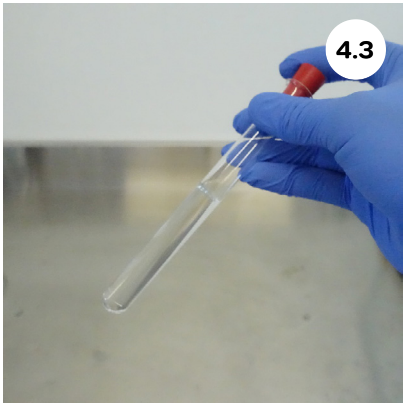


Prepare 2 clean and dry test tubes with plugs and name one of them according to the concentration that was selected. The other one is labeled as "Blank"



Using the pipette, extract 2.4 mL of one of the selected solution and put it into the labeled test tube. The test tube labeled "blank" gets 2.4 mL distilled water instead.

(Ensure the chemicals and solutions are at room temperature)



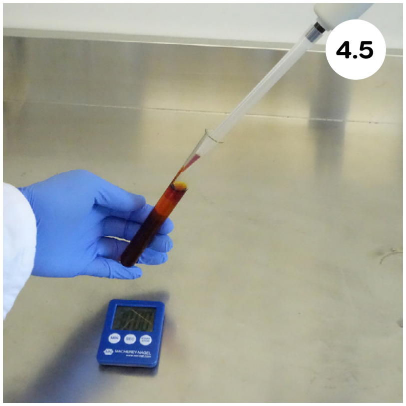
Add 9 mL of the 40 g/L abcr boric acid and shake the test tube.

(Ensure the chemicals and solutions are at room temperature.)

Once both test tubes are prepared with the solution and boric acid, the tests become time sensitive.

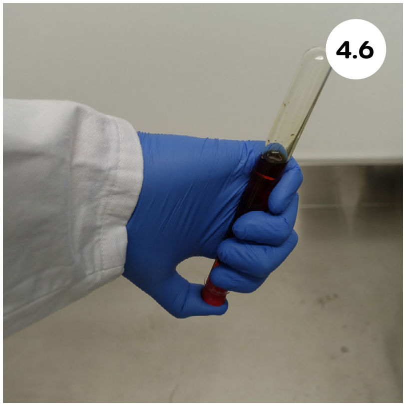
It is therefore important that everything is well prepared beforehand and that the described times are followed precisely.

Prepare a timer for 30 minutes.

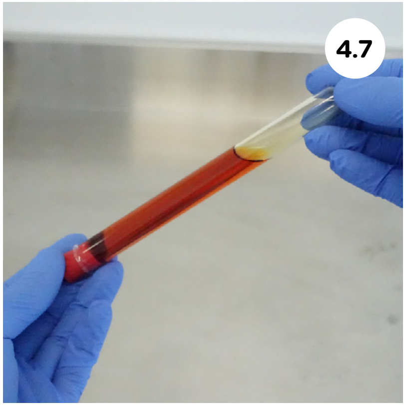


Begin with the blank: add 2.4 mL of abcr Lugol´s solution and immediately start the 30-minute timer.

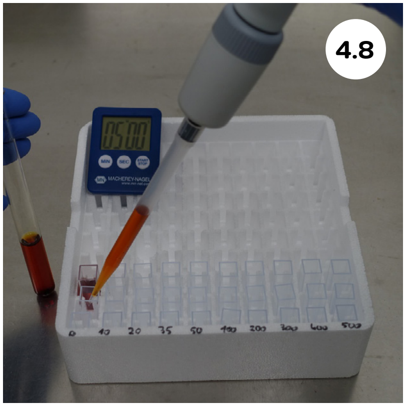
MEASURING THE OPTIMAL WAVELENGTH



Put a stopper on the test tube and mix it by inverting it 3 times.



After exactly one minute, add 2.4 mL of abcr Lugol´s solution to the test tube containing the WSSP solution) and just like the blank, put on a stop-per and mix it by inverting it 3 times.



5 minutes before the timer runs out, transfer 3 mL of each solution into the cuvettes.

Program the Spectrophotometer so that it measures an absorbance spectrum from 340 nm to 900 nm with a step size of 0.1 nm.

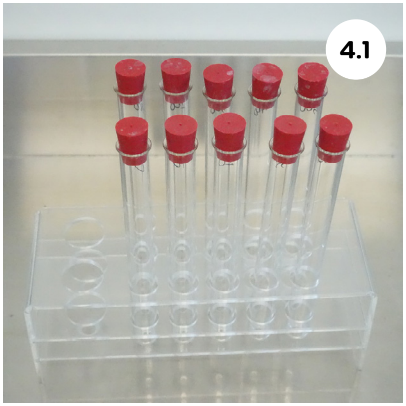
When the timer hits 0, first measure the blank and immediately scan the spectrum of the other sample.

Copy the results into excel and determine the wavelength where the absorbance is the highest.

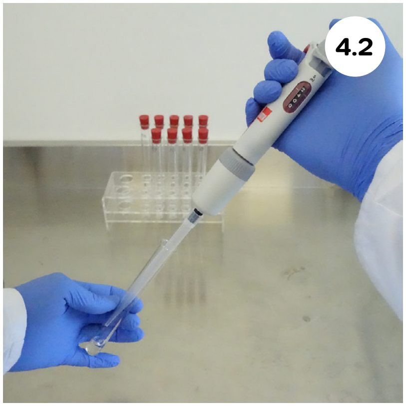
Repeat this for the other 2 selected solutions.

Calculate the average of the 3.

This is (= λ_{max}) for this specific solution.

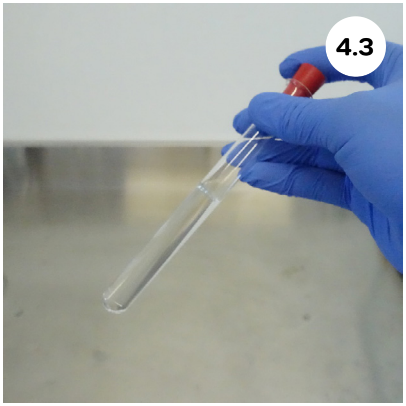


Prepare 10 clean and dry test tubes with plugs and name them according to the concentrations in the table above.
The leftover test tube gets the name “blank”.



For each concentration, pipette 2.4 mL solution into the respective test tube. The test tube labled “blank” gets 2.4 mL distilled water.

(Make sure the chemicals and solutions are at room temperature)



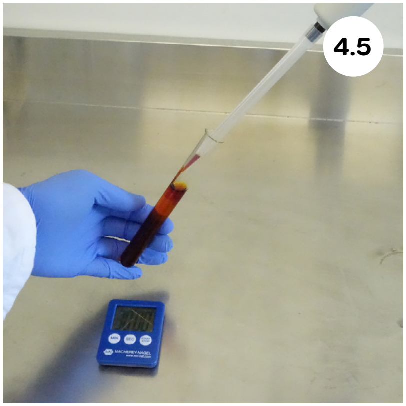
Add 9 mL of 40 g/L abcr boric acid and shake the test tube

(Make sure the chemicals and solutions are at room temperature)

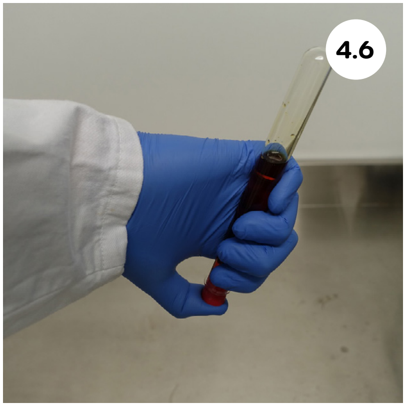
Once all test tubes are prepared with solution and boric acid, the test becomes time sensitive.

Therefore it is important that everything is well prepared beforehand and that the described times are followed precisely.

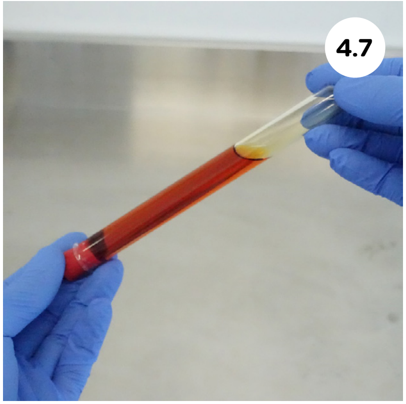
Prepare a timer for 30 minutes and order the test tubes from lowest to highest concentration.



Starting with the blank: add 2.4 mL abcr Lugol´s solution and immediately start the 30-minute timer.

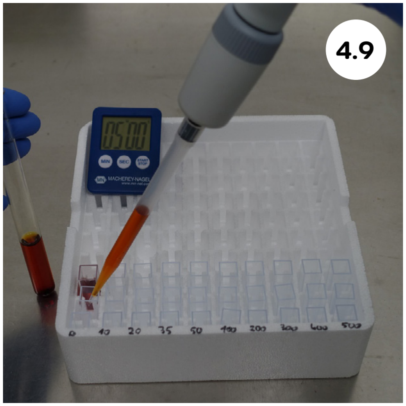


Put a stopper on the test tube and mix it by inverting it 3 times



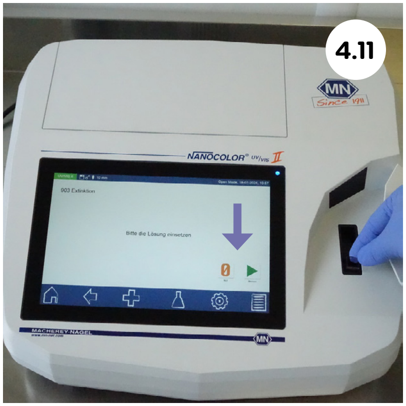
After exactly 1 minute, add 2.4 mL abcr Lugol´s solution to the next test tube and just like the blank, put on a stopper and mix it by inverting it 3 times.

Repeat this for all the solutions with an interval between each one of exactly 60 seconds.

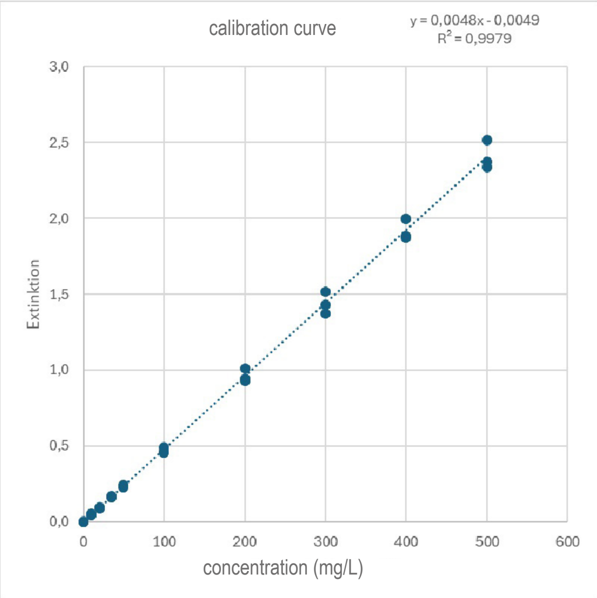


5 minutes before the timer runs out, for every solution transfer 3 x 3 mL into the cuvettes.

When the timer hits 0, restart it and measure the blank value (this can be either of the 3 blank cuvettes) and immediately measure the absorbance of the 3 blank cuvettes back-to-back at λ_{max}



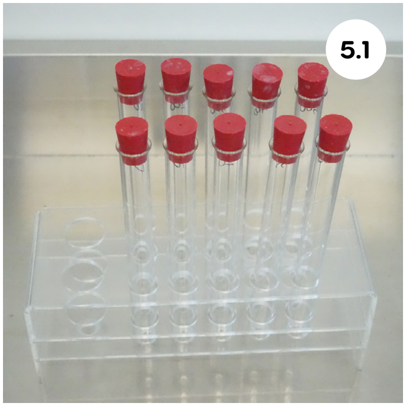
After exactly 60 seconds, measure the next 3 cuvettes of the next concentration back-to-back. Repeat this for the remaining concentrations. Make sure to keep the same order during the experiment.



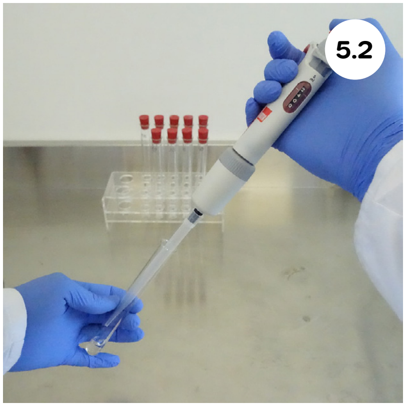
4.12

The absorbance can now be plotted against the concentration in excel. The resulting best fit line through these points can now be used to determine the concentration of a sample.

MEASURING SAMPLES

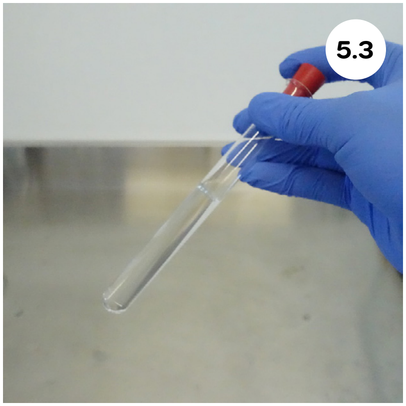


Prepare as many clean and dry test clean tubes with plugs as there are samples. Don’t forget to add one for the “blank”.



For each sample, pipette 2.4 mL solution into the respective test tube.The test tube labeled “blank” gets 2.4 mL distilled water.

(Make sure the chemicals and solutions are at room temperature)



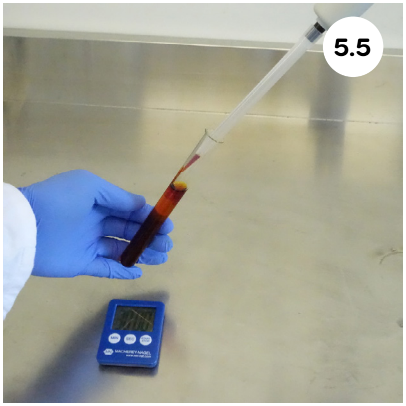
Add 9 mL of 40 g/L abcr boric acid and shake the test tube.

(Make sure the chemicals and solutions are at room temperature)

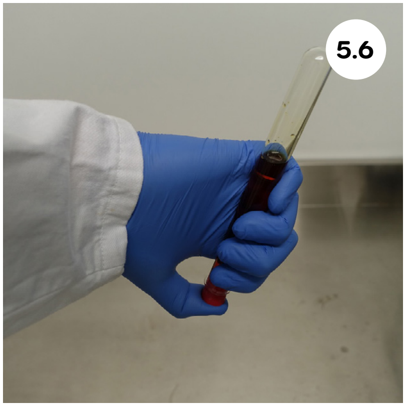
Once all test tubes are prepared with solution and boric acid the test becomes time sensitive.

Therefore it is important that everything is well prepared beforehand, and that the described times are followed exactly.

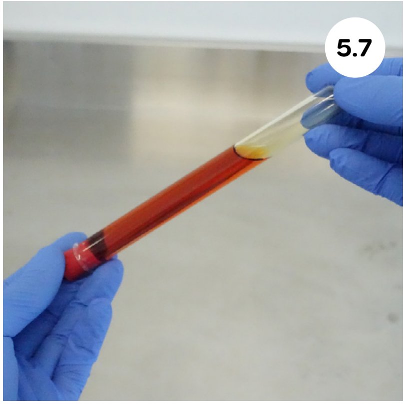
Prepare a timer for 30 minutes and order the test tubes. It is important that the “blank” comes first; the order of the other test tubes does not matter, but should stay the same for the rest of the experiment.



Starting with the blank: add 2.4 mL of abcr Lugol´s solution and immediately start the 30-minute timer.

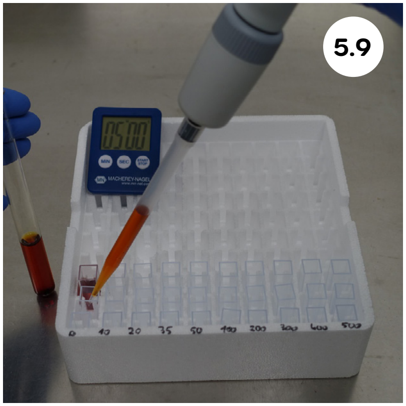


Put a stopper on the test tube and mix it by inverting it 3 times.



After exactly one minute, add 2.4 mL of abcr Lugol´s solution to the next test tube and just like the blank, put on a stopper and mix it by inverting it 3 times.

Repeat this for all the solutions with an interval of exactly 60 seconds.



5 minutes before the timer runs out, for each solution transfer 3 x 3 mL into the cuvettes.

When the timer hits 0, restart it and measure the blank value (this can be either of the 3 blank cuvettes) and immediately measure the absorbance of the 3 blank cuvettes back-to-back at λ_{max}.

After exactly 60 seconds, measure the next 3 cuvettes of the next samp-le back-to-back. Repeat this for the remaining samples. Make sure to keep the same order during the experiment.

CALCULATION OF THE CONCENTRATION OF THE WSSP

The measured absorbances can be converted into concentrations using the calibration curve described on p. 22.

Where the best fitting curve is represented by y=ax+b, so

$$x = \frac{y - b}{a}$$

And:
x is the concentration in mg/L,
y the measured absorbance,
a is the slope of the function,
b is the intersect with the y-axis

Any measurement with an absorbance greater than 3 are not representative and should be diluted.

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