# Water Treatment Technology – laboratory

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# 1.  COAGULATION PROCESS

## **INTRODUCTION**

The coagulation process in water treatment depends on the source of the water and the nature of the suspended, colloidal, and dissolved organic constituents.

Coagulation involves the addition of coagulants for conditioning the suspended, colloidal, and dissolved matter for subsequent processing by flocculation or to create conditions that will allow for the subsequent removal of particulate and dissolved matter.

Flocculation involves the aggregation of destabilised particles (particles from which the electrical surface charge has been reduced) and the precipitation products formed by the addition of coagulant into larger particles known as flocculant particles or, more commonly, floc that can be removed by gravity sedimentation and/or filtration.

Coagulation and flocculation can also be differentiated based on the time required for each of the processes. Coagulation (rapid mixing) typically occurs in less than 10 s whereas flocculation (slow mixing) occurs over a period of 20 to 45 min.

Coagulation process occurs due to the addition of the hydrolysing chemicals such as alum and iron salts and/or organic polymers.

The jar test procedure is used to simulate the expected or desired conditions in the coagulation-flocculation facilities. The test consists of a rapid mix phase (high mixing intensity) with a batch addition of the coagulant followed by a slow-mix period to simulate flocculation. Flocs are allowed to settle, and samples are taken from the supernatant. As part of the jar test routine some parameters can be measured: (1) turbidity or suspended solids removal; (2) NOM removal as measured by DOC or a surrogate measure of dissolved NOM, such as UV at 254 nm; (3) residual dissolved coagulant concentrations of Fe or Al coagulants; and (4) sludge volume that is produced.

Within the laboratory work, a series of jar tests will be performed to determine the optimum coagulant dose (alum) and pH for turbidity and colour removal for natural water.
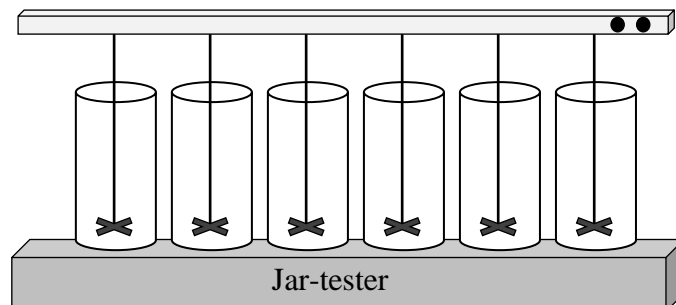


Setup for coagulation-flocculation process

# MATERIALS & EQUIPMENT

- Conical flasks
- Graduated cylinders
- Glass and plastic beakers
- Coagulant stock solution (alum, 1% concentration)
- 0.1 M HCl and 0.1 M NaOH solution
- pH-meter with electrode
- Turbidimeter
- Spectrophotometer
- Lab equipment for titration

# PROCEDURE

**Before you start:**
- Coagulant stock solution – 1% $Al_2(SO_4)_3 \cdot 18H_2O$ solution.
  Stock solution is prepared by dissolving 10.0 grams of alum in 1000 mL of distilled water. It means that each 1.0 mL of this stock solution equals 10 mg/L when added into 1000 mL of water sample.

- Determine the basic coagulant dose "$D$" in mg/L.
  Use an empirical formula which expresses the basic dose of coagulant needed in the coagulation step for colour (C) and turbidity (T) removal:

$$D = n_C \sqrt{C} \quad \text{and} \quad D = n_T \sqrt{T}$$

  where $D$ is the basic (theoretical) coagulant dose, mg/L; $C$ is the colour of raw water, mgPt/L; $T$ is the turbidity of raw water, NTU; $n$ is the constant, $n_C = 6 - 8$ for colour and $n_T = 7$ for turbidity.

  Three various alum doses will be tested according to the data sheet.
  Knowing the concentration of alum stock solution (it's 1%), calculate how many mL of alum should be added into each jar. Fill in the table 1 in data sheet with your calculations.

- Process conditions:
  | | |
  |---|---|
  | Rapid mix | 2 min. with 200 rpm |
  | Flocculation | 20 min. with 15 rpm |
  | Settling | 20 min. |

**Procedure Jar 1-3:**
- Using the 1L graduated cylinder, pour 1L of raw water into the jar-test beakers no 1, 2 and 3.
- Using the prepared alum stock solution dose each beaker with a proper amount of the reagent according to table 1 (data sheet).

- After dosing, operate the stirrer at 200 rpm for 2 minutes.
- When rapid mixing is over turn on the stirrer and set up the revolutions on 15 rpm for 20 min.
- During flocculation observe the floc formation.
- Turn off the stirrer and allow the flocs to settle for 20 min.
- Pour the water from jars into glass beakers; be careful - don't allow deposit to enter the cell.
- In treated water determine the alkalinity, pH, turbidity and colour.

**Procedure Jar 4-6:**
- Using the 1L graduated cylinder, pour 1L of raw water into the jar-test beakers no 4, 5 and 6.
- Using the 0.1 M HCl/0.1 M NaOH solution adjust the water pH to the proper value according to table 1 in data sheet.
- Using the alum stock solution dose the beaker with a proper amount of reagent.
- After dosing, operate the stirrer at 200 rpm for 2 minutes and next set up the revolutions on 15 rpm for 20 min.
- Turn off the stirrer and allow the flocs to settle for 20 min.
- Pour the water from jars into glass beakers; be careful - don't allow deposit to enter the cell.
- In treated water determine the alkalinity, pH, turbidity and colour.

# DATA SHEET
## COAGULATION PROCESS

**Table 1**

|  | Tested coagulant dose | Calculated dose of coagulant | Amount of coagulant added | pH of the process |
|---|---|---|---|---|
|  | - | mg/L | mL | - |
| Jar 1 | 0.5 D |  |  | natural |
| Jar 2 | D |  |  | natural |
| Jar 3 | 1.5 D |  |  | natural |
| Jar 4 | D |  |  | 5 |
| Jar 5 | D |  |  | 6 |
| Jar 6 | D |  |  | 7 |

**Table 2**

|  | Turbidity | Colour | pH | Alkalinity |
|---|---|---|---|---|
|  | NTU | mgPt/L | - | mL of HCl used for titration |
| Raw water |  |  |  |  |
| Jar 1 |  |  |  |  |
| Jar 2 |  |  |  |  |
| Jar 3 |  |  |  |  |
| Jar 4 |  |  |  |  |
| Jar 5 |  |  |  |  |
| Jar 6 |  |  |  |  |

# LABORATORY REPORT
## COAGULATION PROCESS

Date: ………………                    Student: ……….……

**Table 1**

|        | Tested coagulant dose | Calculated dose of coagulant | pH of the process |
|--------|:---------------------:|:----------------------------:|:-----------------:|
|        | -                     | mg/L                         | -                 |
| Jar 1  | 0.5 D                 |                              |                   |
| Jar 2  | D                     |                              |                   |
| Jar 3  | 1.5 D                 |                              |                   |
| Jar 4  | D                     |                              |                   |
| Jar 5  | D                     |                              |                   |
| Jar 6  | D                     |                              |                   |

**Table 2**

|           | Turbidity | Turbidity removal | Colour | Colour removal | pH | Alkalinity |
|-----------|:---------:|:-----------------:|:------:|:--------------:|:--:|:----------:|
|           | NTU       | %                 | mgPt/L | %              | -  | mgCaCO$_3$/L |
| Raw water |           |                   |        |                |    |            |
| Jar 1     |           |                   |        |                |    |            |
| Jar 2     |           |                   |        |                |    |            |
| Jar 3     |           |                   |        |                |    |            |
| Jar 4     |           |                   |        |                |    |            |
| Jar 5     |           |                   |        |                |    |            |
| Jar 6     |           |                   |        |                |    |            |

- Prepare the graphs using the data from table 2 and compare the efficiency of the coagulation process performed with various alum dose and pH.
- Discuss and summarize obtained results.

# 2.  RAPID SAND FILTRATION

## **INTRODUCTION**

Filtration is a process widely used for removing particulate matter from water. Nearly all surface treatment facilities and some groundwater employ some form of filtration. Most surface waters contain algae, sediment, clay and other organic and inorganic particulate matter, and filtration improves the clarity of the water by removing these particles. Groundwater is often low in particles but may require filtration when other treatment processes (such as oxidation or softening) generate particles that must be removed.

Granular media filters have used for more than a century and are still the most common type of filters in use today. In granular filtration, waterborne particles are removed as the influent water passes through a submerged granular material, or filter medium. The most common process - rapid filtration, operates over a cycle consisting of two stages: (1) a filtration stage, during which particles accumulate, and (2) a backwash stage, during which the accumulated material is flushed from the system. During the filtration stage, water flows downward through the filter bed, and particles collect within the bed. During the backwash stage, water flows in the opposite direction to the filtration stage to remove the particles that have collected in the filter bed. Efficient removal of collected solids is a key component of rapid filtration systems, so while the backwashing stage is very short (minutes) compared to the filtration stage (days), it is a very important part of the filtration cycle.

Thus, the laboratory work aims to run the rapid filtration process with the used of sand as a granular media followed by backwashing using water. During the filter operation, the water quality and the head losses will be analysed. The various intensity of backwashing will be examined.



Setup for rapid sand filtration process

# MATERIALS & EQUIPMENT

- Conical flasks
- Graduated cylinders
- Glass or plastic beakers
- Timer and ruler
- pH-meter with electrode
- Turbidimeter
- Spectrophotometer

# PROCEDURE

There are two steps in this exercise: (1) filtration process, (2) filter backwashing.

## Filtration process

Necessary information:
- Carry out the process with the constant filtration velocity ($v$) of 6 m/h.
- Basing on the value of filtration velocity and the filter area calculate the filtration capacity according to the equation:

$$Q_{fl} = v \cdot A$$

where: $Q_{fl}$ – the filtration flow rate, m$^3$/h; $v$ – the filtration velocity, m/h; $A$ – the filter cross sectional area, m$^2$

$$A = \frac{\pi \cdot d^2}{4}$$

where: $d$ – the filter diameter, m

- Control the head losses during the process.
- Note that the values of the head losses read from the piezometers table are in centimeters of H$_2$O.
- Calculate the head losses using the following equation:

$$\Delta p = \rho_{H2O} \cdot g \cdot h$$

where: $\Delta p$ – the head loss, N/m$^2$; $\rho_{H2O}$ – water density, kg/m$^3$; $g$ – the acceleration of gravity, m/s$^2$; $h$ – the head loss, mH$_2$O

## Procedure for filtration process:
- Determine the quality of raw water (pH, colour, turbidity).
- Familiarise with a filtration set-up and piezometers table.
- Set the required filtration velocity; to do it the calculated filtration flow rate ($Q_{fl}$) in m$^3$/h convert into mL/30 s. The required filtration velocity is reached when during 30 s you collect in the cylinder the calculated amount of filtrate (in mL).
- Open the valve with the source of raw water and a filter inlet line.
- Set he filtration velocity; for velocity, adjustment use a rotameter.
- When the velocity is fixed, open the piezometers valves and start the process.
- From an outlet line collect the filtrate samples after 15, 30, 45 and 60 minutes.
- Use the collected filtrate for measuring the pH, colour and turbidity.

- Simultaneously, after 15, 30, 45 and 60 min, observe the head losses at the piezometers table and write down results in the table 3a-3d (data sheet).
- To finish the exercise close in the following order: the piezometers valves, filter inlet line and at the end the valve at the raw water source.

## **Filter backwashing**

Necessary information:
- After the filtration process, backwash the clogged sand bed using tab water.
- Carry out the process with 3 different intensities.
- Change the backwash rates so that they cause 3 different bed expansions (the height of expansions is marked on the filter column with a black lines).
- Calculate:
   - flow rates of water used for backwashing,
   - backwash intensities,
   - bed expansions.

- The flow rates of water used for backwashing:

$$Q_{bw} = \frac{V_{bw}}{t_{bw}}$$

   where: $Q_{bw}$ – the flow rate of water used for backwashing, m$^3$/s; $V_{bw}$ – the volume of water required for filter backwashing, m$^3$; $t_{bw}$ – the duration of the filter backwashing, s

- The backwash intensity:

$$I_{bw} = \frac{Q_{bw}}{A}$$

   where: $I_{bw}$ – the backwash intensity, m$^3$/m$^2\cdot$s; $Q_{bw}$ – the flow rate of water used for backwashing, m$^3$/s; $A$ – the filter cross sectional area, m$^2$

- The bed expansion:

$$E = \frac{H_{ex} - H_{fl}}{H_{fl}} \cdot 100\%$$

   where: E – the bed expansion, %; $H_{ex}$ – the bed height during backwash, m; $H_{fl}$ – the bed height during the filter run, m

## **Procedure for filter backwashing:**
- Open the backwash line and valve with tab water.
- Set up the flow rate of tab water to raise the bed to the first level of expansion and collect from overflow 500 mL ($V_{bw}$) of backwash water into a cylinder.
- Determine time ($t_{bw}$) required for filling up the cylinder with backwash water at the 1st level.
- Increase the tab water intensity to raise the bed to the 2nd level of expansion, collect the water from overflow and control time.
- Follow the same procedure at the 3rd level of expansion.
- After finishing the backwashing, close the tab water and then the backwash line in the filter system.

## DATA SHEET
## RAPID SAND FILTRATION

**Table 1**

| Filter data | |
|---|---|
| The filter diameter ($d$), $m$ | |
| The bed height ($H_{fl}$), $m$ | |
| The bed height during 1st expansion ($H_{ex1}$), $m$ | |
| The bed height during 2nd expansion ($H_{ex2}$), $m$ | |
| The bed height during 3rd expansion ($H_{ex3}$), $m$ | |

**Table 2**

| Water quality | Turbidity | Colour | pH |
|---|---|---|---|
| | NTU | mgPt/L | - |
| Raw water | | | |
| After 15 min | | | |
| After 30 min | | | |
| After 45 min | | | |
| After 60 min | | | |

**Table 3a**

| Head losses after 15 min | Bed height at which the head losses are measured ($h_{pz}$) | Head losses ($h$) |
|---|---|---|
| | cm | cm $H_2O$ |
| Piezometer 1 | | |
| Piezometer 2 | | |
| Piezometer 3 | | |
| Piezometer 4 | | |
| Piezometer 5 | | |
| Piezometer 6 | | |
| Piezometer 7 | | |
| Piezometer 8 | | |

**Table 3b**

| Head losses after 30 min | Bed height at which the head losses are measured ($h_{pz}$) | Head losses ($h$) |
|---|---|---|
| | cm | cm H$_2$O |
| Piezometer 1 | | |
| Piezometer 2 | | |
| Piezometer 3 | | |
| Piezometer 4 | | |
| Piezometer 5 | | |
| Piezometer 6 | | |
| Piezometer 7 | | |
| Piezometer 8 | | |

**Table 3c**

| Head losses after 45 min | Bed height at which the head losses are measured ($h_{pz}$) | Head losses ($h$) |
|---|---|---|
| | cm | cm H$_2$O |
| Piezometer 1 | | |
| Piezometer 2 | | |
| Piezometer 3 | | |
| Piezometer 4 | | |
| Piezometer 5 | | |
| Piezometer 6 | | |
| Piezometer 7 | | |
| Piezometer 8 | | |

**Table 3d**

| Head losses after 60 min | Bed height at which the head losses are measured ($h_{pz}$) | Head losses ($h$) |
|---|---|---|
| | cm | cm H$_2$O |
| Piezometer 1 | | |
| Piezometer 2 | | |
| Piezometer 3 | | |
| Piezometer 4 | | |
| Piezometer 5 | | |
| Piezometer 6 | | |
| Piezometer 7 | | |
| Piezometer 8 | | |

**Table 4**

| Bed expansion | Volume of backwash water ($V_{bw}$) | Time ($t_{bw}$) (1st measurement) | Time ($t_{bw}$) (2nd measurement) | Time ($t_{bw}$) (3rd measurement) | Average |
|---|---|---|---|---|---|
| | m³ | s | s | s | s |
| 1st level | $5 \cdot 10^{-4}$ | | | | |
| 2nd level | $5 \cdot 10^{-4}$ | | | | |
| 3rd level | $5 \cdot 10^{-4}$ | | | | |

# LABORATORY REPORT
## RAPID SAND FILTRATION

Date: ………………                              Student: …….….……

Process conditions:

The filtration capacity          $Q_{fl}$ =……….m³/h

The filtration velocity          $v$ = ………..m/h

**Table 1**

| Filter data | |
|---|---|
| The filter diameter ($d$), $m$ | |
| The bed height ($H_{fl}$), $m$ | |
| The bed height during 1st expansion ($H_{ex1}$), $m$ | |
| The bed height during 2nd expansion ($H_{ex2}$), $m$ | |
| The bed height during 3rd expansion ($H_{ex3}$), $m$ | |

**Table 2**

| Water quality | Turbidity | Turbidity removal | Colour | Colour removal | pH |
|---|---|---|---|---|---|
| | NTU | % | mgPt/L | % | - |
| Raw water | | - | | - | |
| After 15 min | | | | | |
| After 30 min | | | | | |
| After 45 min | | | | | |
| After 60 min | | | | | |

**Table 3a**

| Head losses after 15 min | Bed height at which the head losses are measured ($h_{pz}$) | Head losses ($h$) | Head losses ($\Delta p$) |
|---|---|---|---|
| | cm | cm H$_2$O | N/m$^2$ |
| Piezometer 1 | | | |
| Piezometer 2 | | | |
| Piezometer 3 | | | |
| Piezometer 4 | | | |
| Piezometer 5 | | | |
| Piezometer 6 | | | |
| Piezometer 7 | | | |
| Piezometer 8 | | | |

**Table 3b**

| Head losses after 30 min | Bed height at which the head losses are measured ($h_{pz}$) | Head losses ($h$) | Head losses ($\Delta p$) |
|---|---|---|---|
| | cm | cm H$_2$O | N/m$^2$ |
| Piezometer 1 | | | |
| Piezometer 2 | | | |
| Piezometer 3 | | | |
| Piezometer 4 | | | |
| Piezometer 5 | | | |
| Piezometer 6 | | | |
| Piezometer 7 | | | |
| Piezometer 8 | | | |

**Table 3c**

| Head losses after 45 min | Bed height at which the head losses are measured ($h_{pz}$) | Head losses ($h$) | Head losses ($\Delta p$) |
|---|---|---|---|
| | cm | cm H$_2$O | N/m$^2$ |
| Piezometer 1 | | | |
| Piezometer 2 | | | |
| Piezometer 3 | | | |
| Piezometer 4 | | | |
| Piezometer 5 | | | |
| Piezometer 6 | | | |
| Piezometer 7 | | | |
| Piezometer 8 | | | |

**Table 3d**

| Head losses after 60 min | Bed height at which the head losses are measured ($h_{pz}$) | Head losses ($h$) | Head losses ($\Delta p$) |
|---|---|---|---|
| | cm | cm $H_2O$ | N/m$^2$ |
| Piezometer 1 | | | |
| Piezometer 2 | | | |
| Piezometer 3 | | | |
| Piezometer 4 | | | |
| Piezometer 5 | | | |
| Piezometer 6 | | | |
| Piezometer 7 | | | |
| Piezometer 8 | | | |

**Table 4**

| Bed expansion | Volume of backwash water ($V_{bw}$) | Time ($t_{bw}$) (1st measurement) | Time ($t_{bw}$) (2nd measurement) | Time ($t_{bw}$) (3rd measurement) | Average |
|---|---|---|---|---|---|
| | m$^3$ | s | s | s | s |
| 1st level | $5 \cdot 10^{-4}$ | | | | |
| 2nd level | $5 \cdot 10^{-4}$ | | | | |
| 3rd level | $5 \cdot 10^{-4}$ | | | | |

**Table 5**

| Bed expansion | Flow rate ($Q_{bw}$) | Backwash intensity ($I_{bw}$) | Bed expansion ($E$) |
|---|---|---|---|
| | m$^3$/s | m$^3$/m$^2 \cdot$s | % |
| 1st level | | | |
| 2nd level | | | |
| 3rd level | | | |

- Prepare the graphs using the data from table 2 (filtration time in min vs. process efficiency in %), from tables 3a-3d ($\Delta p$ in N/m$^2$ vs. $h_{pz}$ in m) and from table 5 ($I_{bw}$ in m$^3$/m$^2 \cdot$s vs. $E$ in %).
- Discuss and summarize obtained results.

# 3. IRON AND MANGANESE REMOVAL

## INTRODUCTION

Iron and manganese are typical constituents for groundwaters. Iron is relatively soluble in a reducing environment or natural waters, such as some low-oxygen containing groundwaters and low-oxygen surface water, such as eutrophic lakes. In these waters, iron may be found in the reduced or ferrous form (Fe(II)) such as ferrous sulphate ($FeSO_4$) and ferrous bicarbonate ($Fe(HCO_3)_2$), hydroxide forms, or complexed with NOM. However, iron is very insoluble in an oxidising environment or natural waters containing sufficient quantities of dissolved oxygen. Depending upon the water quality, iron can exist in three physical forms: as large oxidised particles, small oxidized colloidal particles, and the soluble reduced form. Manganese is similar to iron in that it is usually present in the +2 oxidation state ($Mn^{+2}$) in anoxic groundwaters and the hypolimnion region of reservoirs and eutrophic lakes. When the groundwater is pumped to the surface and when the hypolimnetic waters are mixed, the manganese is exposed to oxygen and begins to undergo a series of oxidation reactions to $Mn^{+4}$. This oxidation is accompanied by a decrease in pH and dissolved oxygen concentration and the formation of $MnO_2$ precipitate. Manganese and iron may cause aesthetic problems such as laundry and fixture staining. May also impart an unpleasant taste to water and can promote the growth of microorganisms in reservoirs and distribution systems.

The fundamental concepts involved with removal of soluble iron and manganese ions are similar and the two often occur together in water supplies. As already pointed out, these soluble species are typically unstable when exposed to oxidants such as dissolved oxygen and when processed in a water treatment plant will usually form precipitate during treatment, in the distribution piping system, or at the point of use. However, depending upon which treatment processes are used, it may be important to control these precipitation reactions. The most common treatment approach is to precipitate all the soluble forms of iron and manganese so that these constituents can be removed by other processes such as sedimentation and filtration. In addition to controlling the soluble species, colloidal and/or particulate iron and manganese are also important and must be considered in the overall removal.

The laboratory work aims at the removal of soluble $Fe^{+2}$ and $Mn^{+2}$ ions from water using the filtration system with sand bed coated with manganese dioxide. The efficiency of the process will be analysed and the precipitation of iron and manganese as $Fe(OH)_3$ and $MnO_2$ will be observed.
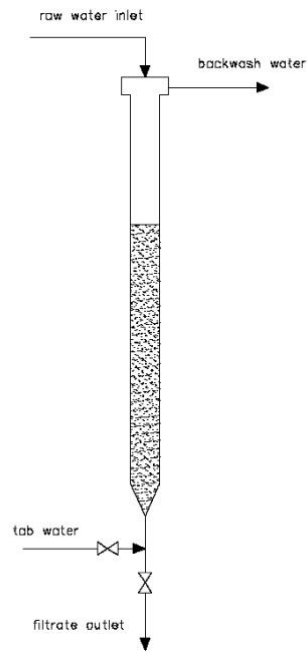
Setup for Fe and Mn removal

## MATERIALS & EQUIPMENT

- Conical flasks
- Graduated cylinders
- Nessler cylinders
- Glass or plastic beakers
- Timer and ruler
- Spectrophotometer

# PROCEDURE

Necessary information:
- Carry out the process with the filtration velocity $v = 7$ m/h.
- Basing on the value of filtration velocity and the filter area, calculate the filtration capacity according to the equation:

$$Q_{fl} = v \cdot A$$

where: $Q_{fl}$ – the filtration flow rate, m³/h; $v$ – the filtration velocity, m/h; $A$ – the filter cross sectional area, m²

$$A = \frac{\pi \cdot d^2}{4}$$

where: $d$ – the filter diameter, m

- Calculate the bed volume:

$$BV = \pi r^2 H_{fl}$$

where: $BV$ – the bed volume, cm³; $r$ – the filter radius, cm; $H_{fl}$ – the filter bed height, cm

## Procedure of the process:
- Determine the quality of raw water according to the table 2 (data sheet).
- Familiarise yourself with the experimental set-up.
- Determine the required filtration velocity in mL/30 s. The required filtration velocity is reached when during 30 s you collect in the cylinder the calculated amount of filtrate (in mL).
- Open the valve with the source of raw water and the filter inlet line.
- Set up required filtration velocity (7 m/h); for controlling the velocity use a rotameter.
- From an outlet line allow flowing the filtrate in amount of 0.5 BV (don't collect this sample).
- After that, start the proper filtration run for 60 minutes.
- From the outlet line collect the filtrate samples 4 times, after 15 min, 30 min, 45 min and 60 min.
- Use the collected filtrate for measuring the Fe total and Mn.
- To finish run, close the filter inlet line and valve at the raw water source.
- Backwash the clogged filter bed for 5 minutes using the tab water.

# DATA SHEET
## IRON AND MANGANESE REMOVAL

**Table 1**

| Filter data | |
|---|---|
| The filter diameter ($d$), $m$ | |
| The bed height ($H_{fl}$), $m$ | |
| The bed volume, $m^3$ | |

**Table 2**

| Water quality | Fe total mgFe/L | Mn mgMn/L |
|---|---|---|
| Raw water | * | ** |
| After 15 min | | |
| After 30 min | | |
| After 45 min | | |
| After 60 min | | |

Raw water:
* prepare sample with dilution 1:2
** prepare sample with dilution 1:2

**IRON AND MANGANESE REMOVAL**


Date: ………………                 Student: ……….……


Process conditions:

The filtration capacity       $Q_{fl}$ =……….m³/h

The filtration velocity        $v$ = ………..m/h


**Table 1**

| Filter data | |
|---|---|
| The filter diameter ($d$), $m$ | |
| The bed height ($H_{fl}$), $m$ | |
| The bed volume, $m^3$ | |


**Table 2**

| Water quality | Fe total | Fe removal | Mn | Mn removal |
|---|---|---|---|---|
| | mgFe/L | % | mgMn/L | % |
| Raw water | | - | | - |
| After 15 min | | | | |
| After 30 min | | | | |
| After 45 min | | | | |
| After 60 min | | | | |


- Prepare the graphs using the data from table 2 (filtration time in min vs. process efficiency in %).
- Discuss and summarize obtained results.

# 4. ANALYTICAL METHODS

## pH

Measure the pH at a room temperature with a properly calibrated pH-meter.

Equipment
- pH-meter with electrode
- Small glass beaker
- Magnetic stirrer with a stirring bar

Method
- Remove electrode from the storage solution and rinse it with distilled water.
- Place the beaker with the sample on the magnetic stirrer and then immerse the pH electrode in the solution.
- Turn on the stirrer (set a gentle mixing) and measure the pH with constant stirring.
- Wait until the stable value of pH will be displayed.

## Alkalinity

Equipment and reagents
- Conical flasks
- Graduated cylinder
- Bromocresol green solution
- Burette with 0.1 M HCl

Method
- Using the graduated cylinder measure 100 mL of water sample and pour into the flask.
- Add 5 drops of bromocresol green solution (water sample will turn to green).
- Titrate with 0.1 M HCl till indicator turn from green to grey.
- Record the amount of HCl used for titration as "a".

Calculations

$$\text{Alkalinity, mg CaCO}_3/L = \frac{a \times 0.1 \times 1000}{V} \times 50$$

where:
a – amount of HCl solution used for titration, mL; 0.1 – conversion factor related with 0.1 M HCl; 50 – gram equivalent weight of $CaCO_3$; 1000 – conversion factor: mL to L; V – volume of water sample, mL

## Turbidity (Nephelometric Method)

Equipment
- Turbidimeter
- Measuring cell

Method
- Mix water sample very well and pour it into the measuring cell.
- Dry the cell and place it in the turbidimeter, close the cover and wait for stabilisation.
- The result of turbidity in NTU will be displayed.

## Colour (Single-Wavelength Method)

Equipment
- Spectrophotometer
- Glass cuvette with a light path length of 5 cm or 1 cm

Method
- Prepare the spectrophotometer and set the proper calibration curve.
- Use a blank cuvette with distilled water to remove the background.
- Pour the water sample into the cuvette and place it in the machine.
- Press the "start" button and record the result in mg/L.

Colour is determined spectrophotometrically with platinum-cobalt solutions as standards.

## UV254

Equipment
- Syringe and filter membrane
- Quartz cells with a light path length of 1cm or 5 cm
- Spectrophotometer

Method
- Prepare the spectrophotometer.
- Filter about 15 ml of your water sample using the syringe and filter membrane.
- Prepare the spectrophotometer and set wavelength of 254 nm.
- Use a blank cuvette with distilled water to remove the background.
- Pour the water sample into the cuvette and place it in the machine.
- Press the "start" button and record the result as absorbance (Abs).

Calculations

$$UV254, m^{-1} = \frac{Abs \times 100}{d}$$

where: UV254 – the calculated UV absorbance, $m^{-1}$; Abs – the absorbance units; d – the quartz cell path length, cm.

## Iron total

Equipment and reagents
- Nessler cylinder
- Glass cells with a light path length of 1 cm or 5 cm
- Spectrophotometer
- Hydroxylammonium chloride solution, $NH_2OH \cdot HCl$
- Octane buffer solution
- 1,10-phenanthroline solution, $C_{12}H_8N_2$

Method
- Pour 50 mL of water sample into the Nessler cylinder (or a proper volume resulting from the dilution and fill cylinder with distilled water up to 100 mL).
- Add 1 mL of $NH_2OH \cdot HCl$ solution.
- Add 2 mL of octane buffer solution.
- Add 2 mL of 1,10-phenanthroline solution and mix the sample.
- Put the sample in the dark place and wait 15 minutes.
- Prepare the spectrophotometer and set the proper calibration curve.
- Use a blank cuvette with distilled water to remove the background.
- Pour the water sample into the cuvette and place it in the machine.
- Press the "start" button and record the result in mg/L.

## Manganese

Equipment and reagents
- Conical flask
- Nessler cylinder
- Heater
- Glass cells with a light path length of 1 cm or 5 cm
- Spectrophotometer
- Ammonium persulfate ($(NH_4)_2S_2O_8$), dry solids
- Special reagent solution (mix of $HgSO_4$, $HNO_3$, $AgNO_3$)

Method
- Pour 100 mL of the water sample into the flask (or a proper volume resulting from the dilution but then prepare the dilution in cylinder and fill it up with distilled water up to 100 mL).
- Add 1 teaspoonful of $(NH_4)_2S_2O_8$ reagent.
- Add 5 mL of special reagent and mix.
- Place the flask into the heater and boil for 2 minutes.
- Quickly cool down the flask with cold tap water.
- Pour the sample into the Nessler cylinder, some of the sample will evaporate, thus fill up the cylinder to the 100 mL with distilled water and mix.
- Prepare the spectrophotometer and set the proper calibration curve.
- Use a blank cuvette with distilled water to remove the background.
- Pour the water sample into the cuvette and place it in the machine.
- Press the "start" button and record the result in mg/L.

# NOTES