

C7 - Water iron concentration determination
Spectrophotometric method, with phenantrolin - 1,10

1. Principle

In raw water (surface water, groundwater...), iron concentration can be very high (until 10ppm) and the maximum for a raw water destined to drinking water production is about 2 mg/L. In drinking waters, the maximum recommended is 0.3 mg / L (the recommended allowance is 3mg Fe per day).

Inconvenience of iron presence in drinking waters :

- colour (red : Fe³⁺)
- taste
- water supply network corrosion

Dissolved Fe²⁺ and Fe³⁺ (which proportion depend on water redox and pH) can react with phenantrolin - 1,10 to form a red complex absorbing 510 nm wavelengths.

A range is realised with Fe²⁺ (Mohr's salt) ; hydroxylamine chlorhydrate allows to reduce Fe³⁺ in Fe²⁺ ; acetic acid and ammonia acetate is a buffer.

2. Procedure

In 100ml volumetric flasks (or 50 mL) , realise this range :

volumetric flask	0	1	2	3	4	5
Iron standard solution Mohr's salt 0.1g/l (mL)	0	1	2	3	4	5
demineralized water until 50mL or 25mL						
acetic acid and ammonium acetate (ml)	2	2	2	2	2	2
hydroxylamine chlorhydrate (ml) mix	1	1	1	1	1	1
phenantrolin (ml) mix	22	2	2	2	2	

Fill up until 100ml (to 50 mL) with demineralized water ; keep in the darkness during 15 minutes.

Determinate the absorbance at 510 nm.

Samples preparation : realise 2 tests for each type of water

Sample volume = 50ml (or 25 mL) **Pay attention to the tested water iron concentration**

add 2 ml acetic acid and ammonium acetate , mix

add 1 ml hydroxylamine chlorhydrate

add 2 ml phenantrolin

fill up until 100ml (or 50mL) ; keep in the darkness during 15 minutes.

Determinate the absorbance at 510 nm.

The range correlation must be superior to 99.9%

For the tested waters, $\Delta A / A_{\text{average}}$ must be < 0.05 (precision test)

3. Report

3.1. Draw a board presenting, for the range and the tested waters :

- stock solution volume and sample solution volume introduced in the volumetric flasks
- iron weight per volumetric flask (μg iron per volumetric flask)
- iron concentration (mg/L) in each volumetric flask
- absorbance values

3.2. Plot , on the same graph, the straight lines representing absorbance = f(iron weight, mg) and absorbance = f($[\text{fer}]_{\text{flask}}$, mg / L)

Determinate the correlation coefficient and interpret it.

3.3. Calculate the molar extinction coefficient ($\text{L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$) and write the Beer Lambert's law

3.4. Calculate the tested water iron concentration (mg / L)

3.5. Interpret these concentrations in comparison to :

- limits for raw water destined to drinking water production
- limits for drinking waters

4. Material and reagents requirement for 12 students

Spectrophotometers + measuring flasks

6 sets of 10 volumetric flasks : 50 or 100 mL

1L Mohr's salt $[(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2, 6 \text{H}_2\text{O}]$: 100 **mg** Fe^{2+} / L : annotated "standard solution"

2L Mohr's salt 10 **mg** Fe^{2+} / L : annotated "raw water"

2L Mohr's salt 0.2 **mg** Fe^{2+} / L : annotated "drinking water "

acetic acid and ammonia acetate : 400 ml

ammonia acetate : 40g

acetic acid 1.06g/mL : 50 mL complete to 100 mL with deionized water

hydroxylamin chlorhydrate ($\text{NH}_2\text{OH}, \text{HCl}$) 100 g / L : 300 mL

Phenantrolin chlorhydrate ($\text{C}_{12}\text{H}_9\text{ClN}_2\text{H}_2\text{O}$) 0.05 g / L : 400 mL

Water phosphorus concentration determination Spectrophotometric method

1. Principle

In waste waters, phosphorus concentration is about 10 to 25 mg P / L ; it can cause surface waters eutrophication.

Phosphorus origins in sewage are fecal matters phosphoproteins and detergents.

Phosphorus can be :

- mineral : phosphates PO_4^{3-} , HPO_4^{2-} , H_2PO_4^- depending on pH, polyphosphates
- organic in proteins

P concentration in treated waters must be inferior to 2 ppm.

Phosphates PO_4^{3-} can react with molybdate ammonia to form a blue complex absorbing 880 nm wavelengths.

A range is realised with phosphates.

2. Procedure

In 50ml volumetric flasks, realise this range :

Standard solution volume 2mg P / L (mL)	0	2	4	6	8	10
Add water until about 40 mL						
ascorbic acid 1 mL						
ammonium molybdate 1 mL						
Fill up until 50 mL mix determinate absorbance at 880nm						

Samples preparation : realise 2 tests for each type of water

Sample volume = 5 to 40 ml : **Pay attention to the tested water phosphorus concentration : this sample volume must present a P concentration included in the range**

Add reagents as indicated in the broad .

Determinate the absorbance at 880 nm.

The range correlation must be superior to 99,9%

For the tested waters, $\Delta A / A_{\text{average}}$ must be < 0.05 (precision test)

3. Report

3.1. Draw a board presenting, for the range and the tested waters :

- stock solution volume and sample solution volume introduced in the volumetric flasks

- phosphorus weight per volumetric flask ($\mu\text{g P}$ per volumetric flask)
- P concentration (mgP/L) in each volumetric flask
- absorbance values

3.2. Plot , on the same graph, the straight lines representing absorbance = $f(\text{P weight, mg})$ and absorbance = $f([\text{P}]_{\text{flask, mg / L}})$

Determine the correlation coefficient and interpret it.

3.3. Calculate the molar extinction coefficient ($\text{L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$) and write the Beer Lambert's law

3.4. Calculate the tested water P concentration (mgP / L)

3.5. Interpret these concentrations in comparison to limits for treated waste waters

4. Material order

Spectrophotometers + measuring flasks

6 sets of 10 volumetric flasks : 50 mL

1L standard solution 2 mg P / L : annotated "standard solution"

2L solution 10 mg P / L : annotated "raw waste water"

2L solution 2 mg P / L : annotated "treated waste water"

ascorbic acid : 300 ml

ammonia molybdate : 300mL