 <b>Institut de Camarles</b> EXPERIMENTAL SCIENCES DEPARTMENT	<b>EXPERIMENTAL SCIENCES</b>	
	<b>Group/class: 3th ESO</b>	
	<b>WATER ANALYSIS</b>	

## 1. Objective

Use simple techniques of water analysis to identify and measure some physical and chemical characteristic parameters .

## 2. Introduction

Water is a vital resource for living beings. If we want to control its quality and detect possibly contamination, physicochemical analysis must be performed periodically, both for drinking water that is suitable for consumption, and for guaranteeing the "health" of natural river courses.

**pH** : is a mesure of how acidic or basic a substance is. The escale of pH runs from 0 to 14,  $\text{pH} < 7$  (acid range),  $\text{pH} = 7$  (neutral state) i  $\text{pH} > 7$  (alkaline range).

**Alkalinity**: is the quantitative capacity of a water sample to neutralize an acid to a set pH. Alkalinity is not a chemical in water, but, rather, it is a property of water that is dependent on the presence of certain chemicals in the water, such as bicarbonates, carbonates, and hydroxides. Alkalinity is expressed in mg/l of calcium carbonate ( $\text{CaCO}_3$ ).

**1°d=10 mg/l CaO = 17.85 mg/l  $\text{CaCO}_3$ =1,25°e**

**1mmol/l  $\text{Ca}^{+2}$  =5,6°d = 7°e**

Water hardness				
Assessment of the water	Water hardness in °d	Water hardness in °e	in mmol/l (Ca + Mg)	in mg/l $\text{CaCO}_3$
soft	< 8.4	< 10.5	< 1.5	< 150
moderately hard	8.4 - 14	10.5 - 17.5	1.5 - 2.5	150 - 250
hard	> 14	> 17.5	> 2.5	> 250

**Water hardness-Total hardness**: water hardness is the amount of dissolved calcium and magnesium in the water. Total hardness is a measurement of the mineral content in a water sample. More specifically, total hardness is determined by the concentration of multivalent cations in water. Is expressed in mmols/l de  $\text{Ca}^{+2}$ .

TAULA DE CORRESPONDÈNCIA ENTRE UNITATS					
Unitats de duresa	mg $\text{CaCO}_3$ /L	mmol $\text{CaCO}_3$ /L	°f (° francès)	°d (° alemany)	°e (° anglès)
1 mg $\text{CaCO}_3$ /L	1	0,01	0,10	0,056	0,07
1 mmol $\text{CaCO}_3$ /L	100	1	10	5,6	7
1 °f	10	0,1	1	0,56	0,702
1 °d	17,9	0,179	1,79	1	1,253
1 °e	14,3	0,143	1,43	0,798	1

**Dissolved Oxygen (DO)** : the concentration of dissolved oxygen in water is extremely important in nature as well as in man's environment and it is essential to the growth and development of marine life. Without oxygen the water can become toxic due to anaerobic decaying of organic matter. DO in oceans can range from 0 a 20 ppm ( $\text{mgO}_2$ /l). Typical ranges generally fall around 5 and in colder waters one would expect to find higher values..

**Ammonia (NH<sub>3</sub>) and Ammonium (NH<sub>4</sub><sup>+</sup>)** : In aquatic systems we can find nitrogen (N) in form of ammonia or ammonium from excretion of animals. Ammonium (NH<sub>4</sub><sup>+</sup>) or, its uncharged form ammonia (NH<sub>3</sub>), is a form of nitrogen which aquatic plants can absorb and incorporate into proteins, aminoacids, and other molecules. High concentrations of ammonium can enhance the growth of algae and aquatic plants. Bacteria can also convert high ammonium to nitrate (NO<sub>3</sub><sup>-</sup>) in the process of nitrification, which lowers dissolved oxygen. Typically, the value reported is the sum of both forms and is reported as total ammonia or simply - ammonia. The relative proportion of the two forms present in water is highly affected by pH.

Símbol	Concentració Amoni (mg N-NH <sub>3</sub> /l)	Interpretació
●	< 0.1	Aigües netes. Sense risc de toxicitat per als organismes.
●	0,1 - 0,4	Aigües on el risc de toxicitat pot ser significatiu depenent del pH i del temps de permanència.
●	0,5 - 0,9	Aigües amb risc de toxicitat si el pH és alt.
●	1-4	Aigües que comporten un risc de toxicitat elevat per a moltes espècies, sobretot a pH > 8.
●	> 4	Aigües amb un grau de toxicitat agut per als organismes.

#### Nitrate and Nitrite :

Nitrate (NO<sub>3</sub><sup>-</sup>) and nitrite (NO<sub>2</sub><sup>-</sup>) are forms of nitrogen in the environment, both natural and human-made. Nitrate ions are present in trace amounts in surface water and in high levels in some groundwater. Many times the cause could be human-made due to fertilizers used in agrarian areas. Large amounts of nitrate in drinking water can be harmful for a person's health because nitrate can change into nitrite in the human body. Nitrite is the intermediate oxidized state of nitrogen, as a result of the oxidation of ammonia (NH<sub>3</sub>/NH<sub>4</sub><sup>+</sup>) or the reduction of nitrate (NO<sub>3</sub><sup>-</sup>). Nitrite are found in small quantities due to its unstable nature, but small concentrations are toxic for aquatic and human organisms.

#### Phosphate :

Phosphorus compounds are plant nutrients . Phosphate stimulates growth of plankton and marine plants. It can be found as a natural mineral in rocks, however, they are widely introduced into the environment from such sources as agricultural fertilizers, for example. Eutrophication effect occurs when excessive quantities of phosphates are present in water. This can lead to fish kills and the degradation of habitat with loss of species.

### 3. Material

- Several glass jar with different water samples.
- Beakers, small plastic vessels with cap.
- Glass bottle with stopper.
- Titration syringe.
- pH indicator paper and pH/EC/TDS/Temp tester.



- Reagents from *Marine Science Education Test Kit* de Hanna Instruments indicating the different parameters we are going to test.
- Safety goggles and protective gloves.
- Notebook, pencil and rubber.
- Record the extra material used (comparator cuvettes....)

## 4. Procedure

- Collect three different water samples (sea, Bassa de les Olles, canal, river, fountain, etc) from places close to your home. Write down in each jar the origin of water. How collecting water samples?. Rinse the bottle and cap three times with sample water and fill the bottle from the top. Avoid air bubbles formation. Plastic bottles must be used (1l)
  - Observe physical characteristics of each water sample: describe its color, smell, appearance...
  - Make a study about their chemical characteristics: pH, ammonia, alkalinity, total alkalinity, dissolved oxygen, ammonia, nitrite, nitrate, phosphate using indicator paper, pH tester and the suitable Marine Science test kit. On the table you'll find a sheet with instructions. To prepare the vessels for the samples, clean them before with tap water and rinse with distilled water first and then with the same sample to avoid cross-contamination.
- **pH** : measure the pH of the samples with indicator paper. Then, do the same with the pH-tester. Turn it on by pressing and holding the MODE button for 2-3 seconds and submerge the electrode in the solution to be tested while stirring it gently. The measure should be taken when the stability symbol of the top left of LCD disappears. To freeze the display, pres the SET/HOLD button for 2-3 seconds. After recording pH and temperature rinse the electrode with distilled water and turn it off.
  - **Ammonia** : prepare a plastic vessel with cap and rinse with water sample before filling it up to the 10 ml mark. Add 5 drops of *Ammonia Reagent 1 for Sea Water*, replace the cap mix by carefully swirling the vessel in tight circles. Add 8 drops of *Nessler reagent*, replace the cap and mix by carefully swirling the vessel. Remove the cap and transfer the solution into the color comparator cube marked as a «Ammonia». Wait 5 minutes to allow color to develop. Determine which color matches the solution in the cube and record the result in (mg/l NH<sub>3</sub>-N). It is better to match the color with a white sheet at about 10 cm behind the comparator.
  - **Alkalinity**: Remove the cap from the small plastic vessel. Rinse the plastic vessel with water sample, fill to the 5ml mark and replace the cap. Add 1 drop of *Reagent 1* and mix carefully swirling the vessel in tight circles. If the solution remains colorless, record the phenophthalein alkalinity as zero, and proceed with the procedure for the determination of Total Alkalinity (see below). If the solution is **pink or red**, take the titration syringe and plush plunger completely into the syringe. Insert tip into *Reagent 3* solution and pull plunger out until the lower edge of the plunger seal is on the 0 ml mark of the syringe. Place syringe tip into the cap port of the plastic vessel and slowly add the titration solution



drop-by-drop, swirling to mix after each drop. Continue adding titration solution until the solution in the plastic vessel turns **colorless**. Read off the milliliters of titration solution from the syringe scale and multiply by 300 to obtain mg/l  $\text{CaCO}_3$  (ppm) .

- **Total Alkalinity** : Remove the cap from the small plastic vessel. Rinse the plastic vessel with water sample, fill it up to the 5ml mark and replace the cap. Through the cap port, add 1 drop of *Reagent 2* and mix . If the solution is yellow, then it is acidic and an acidity test must be carried out. If the solution is green or blue, take the titration syringe and push plunger completely into the syringe. Insert tip into *Reagent 3* solution and pull plunger out until the lower edge of the plunger seal is on the 0 ml mark of the syringe. Place syringe tip into the cap port of the plastic vessel and slowly add the titration solution drop-by-drop, swirling to mix after each drop. Continue adding titration solution until the solution in the plastic vessel turns **yellow**. Read off the milliliters of titration solution from the syringe scale and multiply by 300 to obtain mg/l  $\text{CaCO}_3$  (ppm).

***Attention : if the result is lower than 100 mg/l, test should be repeated using 15 ml of sample, following the instructions step by step and multiplying by 100 to get the result in ppm  $\text{CaCO}_3$ .***

- **Dissolved Oxygen**: Fill in the glass bottle with water sample to overflow. Insert stopper carefully so as to avoid trapping of air bubbles in the bottle. Remove the stopper and add 5 drops each of *Reagent 1* and *Reagent 2*. Carefully stopper the bottle, shake vigorously and allow to stand for 1 minute. A flocculent precipitate will form. Remove the stopper, add 10 drops of *Reagent 3*, again stopper the bottle and shake vigorously until all particulate material is dissolved. If oxygen is present, the flocculent precipitate will disappear and the solution will turn a **yellow** color. Remove the cap from the plastic vessel. Rinse the plastic vessel with some of the solution in the bottle, fill to the 5ml mark and replace the cap. Add 1 drop of *Reagent 4* through the cap port and mix by carefully swirling the vessel in tight circles. The solution will turn a violet to blue color. Take the titration syringe and push plunger completely into the syringe. Insert tip into *Reagent 5* solution and pull plunger out until the lower edge of the plunger seal is on the 0 ml mark of the syringe. Place the syringe tip into the cap port of the plastic vessel and slowly add the titration solution drop-by-drop, swirling to mix after each drop. Continue adding titration solution until the solution in the plastic vessel changes from blue to **colorless**. Read off the milliliters of titration solution from the syringe scale and multiply by 10 to obtain mg/l  $\text{O}_2$  (ppm).

***Attention: if the result is lower than 5 mg/l, test should be repeated using 10 ml of sample, following the instructions step by step and multiplying by 5 to get the result in mg/l oxygen.***

- **Nitrates**: fill the glass cuvet up to the 10 ml mark with sample. Add 1 packet of *reagent HI 3874-0*, replace the cap and shake vigorously for 1 minute. Wait for 4 minutes to let the color develop. Remove the cap and fill the color comparator cube with 5ml of the treated sample. Determine which color matches the solution in the cube and record the result as a mg/l (ppm) of  $\text{NO}_3^-$ . It is better to match the color with a white sheet at about 10 cm behind the comparator. To convert the reading to mg/l of  $\text{NO}_3^-$ , multiply the reading by a factor of 4.43. Test range is 0 to 50 mg/l (ppm) of nitrates in 10 mg/l increments.

- **Nitrites:** fill the glass cuvet to the 10 ml mark with sample. Add 1 packet of *reagent HI 3873-0*, replace the cap and shake *gently for about* 15 seconds. Wait for 6 minutes to let the color develop. Remove the cap and fill the color comparator cube with 5ml of the treated sample. Determine which color matches the solution in the cube and record the result as a mg/l (ppm) of  $\text{NO}_2^-$ . It is better to match the color with a white sheet at about 10 cm behind the comparator. To convert the reading to mg/l de  $\text{NO}_2^-$ , multiply the reading by a factor of 3,28. Test range is 0 to 1mg/l (ppm) of nitrites in 0,2 mg/l increments.
  - **Phosphates :** Remove the cap from the plastic vessel. Rinse the plastic vessel with water sample, fill to the 10 ml mark . Add 1 packet of *reagent HI 3833-0*, replace the cap and mix solution until solids dissolve. Remove the cap and transfer the solution into the color comparator cube. Let set for 1 minute. Determine which color matches the solution in the vessel, and recorded the results as mg/l (ppm) de  $\text{PO}_4^{3-}$ . It is better to match the color with a white sheet at about 10 cm behind the comparator. Test range is 0 to 5 mg/l (ppm) of phosphates, in 1 g/l increments.
- d. Draw a table to collect all the observed and measured data.
- e. Check the table below about orientative values for grading the quality of water. Try to determine which water has more quality.

Auxiliary tables				
1 Orientational values for grading the quality of water bodies				
Quality class	I	II	III	IV
Organic load	un polluted to very slightly polluted	moderately polluted	strongly polluted	extremely polluted
BOD <sub>5</sub> value in mg/l	1 - 2	2 - 8	8 - 20	> 20
Oxygen minimum in mg/l	> 8	> 6	> 2	< 2
Oxygen saturation in %	86 - 100 100 - 110	50 - 85 110 - 150	20 - 40 150 - 200	< 10 > 230
pH acidic alkaline	6.5 - 7.0 7.0 - 7.5	6.0 - 6.5 8.0 - 8.5	5.0 - 5.5 9.0 - 9.5	< 5.0 10.0
Ammonium in mg/l	< 0.1	0.1 - 1	> 2	> 5
Nitrate in mg/l	< 1.0	1 - 5	> 5	
Nitrite in mg/l	< 0.1	0.2 - 0.5	4.0 - 6.0	8.0
Orthophosphate in mg/l	< 0.03	< 0.5	> 0.5	
Total hardness in mmol/l	approx. 3.6	approx. 5.3	approx. 7.1	
Acid-binding capacity (ANC) in mmol/l	0.5 - 1.0	0.25 - 0.5	0.03 - 0.1	0.05
Iron in mg/l	0 - 0.1	0.1 - 0.2	approx. 0.5	1.0
Chloride in mg/l	< 80	80 - 500	1500 - 3500	> 3500

## 5. Questions

- a. Is any of the three samples of water neutral?
- b. According to the pH parameters, is it possible to have a basic contaminant or acid in the samples? Which is the range of pH to be considered acidic rain?
- c. Which water sample has a higher content of nitrites? And in nitrates? What do you think it may be due to?
- d. Is there a relationship between the results and the origin of the samples?

## 6. Conclusions

## 7. Annexes

**Field Test Procedures - Phosphate** Marine

**Phosphates Test**

- 1 Remove the cap from the plastic vessel. Rinse the plastic vessel with water sample, fill it to 10 mL mark.
- 2 Add 1 packet of reagent HI 3833-0.
- 3 Replace the cap and mix solution until solids dissolve.
- 4 Remove the cap and transfer the solution into the color comparator cube. Let set for 1 minute.
- 5 Determine which color matches the solution in the vessel, and record the results as mg/L (or ppm)  $\text{PO}_4^{3-}$ .

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**Field Test Procedures - Alkalinity** Marine

**Alkalinity Test**

- 1 15 mL 0-100 mg/L 5 mL 0-300 mg/L
- 2 **Phenolphthalein Alkalinity** **Total Alkalinity**
- 3 5 mL sample 15 mL sample
- 4 5 mL sample 15 mL sample

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**Field Test Procedures - Nitrite** Marine

**Nitrogen Test  $\text{NO}_2^-$**

- 1 Fill the glass cuvet to the 10 mL mark with sample.
- 2 Add 1 packet of reagent HI 3873-0.
- 3 Replace the cap and shake gently for about 15 seconds. A deposit may remain, but it will not affect measurement. Time and the manner of shaking can affect the results.
- 4 Wait for 6 minutes to let the color develop. Remove the cap and fill the color comparator cube with 5 mL of the treated sample (to the mark).
- 5 Determine which color matches the solution in the cube and record the result as mg/L (ppm) of nitrite-nitrogen.

It is better to match the color with a white sheet at about 10 cm behind the comparator.

To convert the reading to mg/L of nitrite ( $\text{NO}_2^-$ ), multiply the reading by a factor of 3.28.

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**Field Test Procedures - Nitrate** Marine

**Nitrate Test  $\text{NO}_3^-$**

- 1 Fill the glass cuvet to the 10 mL mark with sample.
- 2 Add 1 packet of reagent HI 3874-0.
- 3 Replace the cap and shake vigorously for exactly 1 minute.
- 4 Wait for 4 minutes to let the color develop. Remove the cap and fill the color comparator cube with 5 mL of the treated sample.
- 5 Determine which color matches the solution in the cube and record the result as mg/L (ppm) of nitrate-nitrogen.

It is better to match the color with a white sheet at about 10 cm behind the comparator.

To convert the reading to mg/L of nitrates ( $\text{NO}_3^-$ ), multiply the reading by a factor of 4.43.

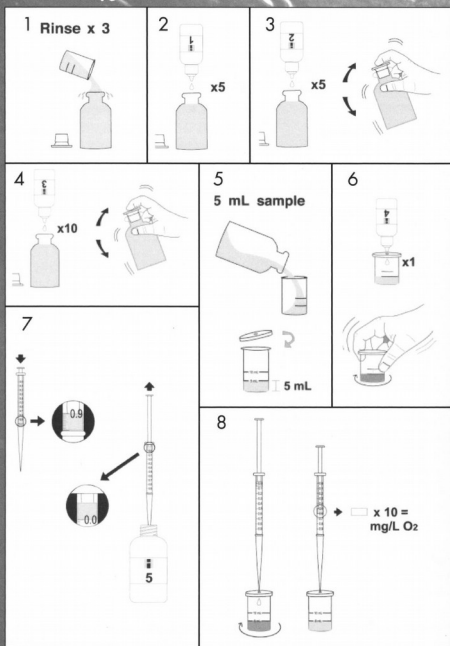
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## Field Test Procedures - Dissolved Oxygen

Marine

### Dissolved Oxygen Test



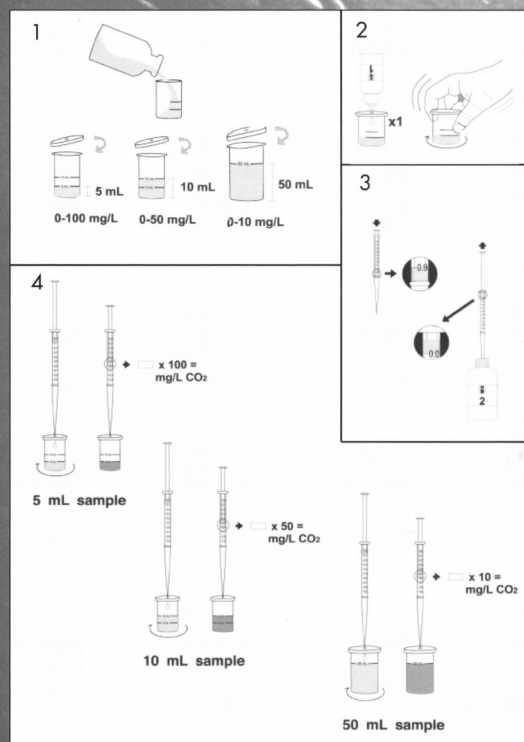
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## Field Test Procedures - Carbon Dioxide

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### Carbon Dioxide Test



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