FLUORIDE IN WATER
Determining the fluoride concentration in a water sample

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Languages available
English, Hungarian, Slovenian

Summary
Everywhere we go we are exposed to fluoride: when we drink tap or spring water, use toothpaste, drink tea and in many other ways. In many instances, the fluoride content is very small, but in others, it is high. In this activity, fluoride is determined using an ion selective electrode (ISE). The question is: what is the lowest concentration of fluoride that can be detected reliably - the detection limit?

Activity type **B**
Use of scientific knowledge and understanding to solve problems
Working in teams to solve problems
Communication

Techniques
Potentiometry

Field
Foods and beverages

Time
Practical lessons: 180 minutes
Theory lessons: 45 minutes
Out of class time: 90 minutes

StandardBase procedures
Determination of fluoride ion content of toothpaste using potentiometry

StandardBase techniques
Potentiometry

Other resources
GlaxoSmith Kline; www.gsk.com
www.iupac.org/publications/pac/1994/pdf/6612x2527.pdf
www.fluoride-journal.com/98-31-2/31274-80.htm
www.nico2000.net/Book/Guide1.html

Student's document

FLUORIDE IN WATER: Determining the fluoride concentration in a water sample

Find two labels from bottled mineral water (there are two here). Look at the fluoride concentration given on the labels.

- How would you determine fluoride in samples of water?
- What type of fluoride determination do you know about?
- What is the lowest concentration of fluoride you can measure?

Figure 1. Label from *Margitszigeti kristályviz* bottled water

Figure 2. Label from *Apenta Optima* bottled water

Your brief
Your laboratory receives several samples of bottled mineral water. You have to determine the fluoride content in them. The analytical method available is potentiometry with fluoride ion selective electrode. Before you start the fluoride determination you have to decide what the detection limit is for your method, because you think that the fluoride content may be less than 0.1-0.2 mg/dm^3.

Your investigation
You will work as a member of a group. At least two groups will be needed to carry out this activity:
Group A will determine the detection limit of the available analytical method (fluoride ISE).
Group B will measure the fluoride content in at least two water samples.

Afterwards, groups should exchange results and write a final report. Groups should also write an article for the School Newspaper about the ProBase project and the results of the investigation.

A risk assessment must be carried out before starting practical work.

Estimated time
Teacher's introduction to the scenario: - 1 x 45 minutes
Gathering information from library and from Internet - 1 x 45 minutes
Laboratory work for determination of DL (detection limit) - 3 x 45 minutes (group A)
Laboratory work measuring some real water samples - 3 x 45 minutes (group B)
Data evaluation and report - 45 minutes
Writing an article for the School Newspaper – 45 minutes

Your findings
Group A
Compare your result (detection limit) with the stated parameters for fluoride ISE (see data sheets).
Group B
Compare your results with the analysis given by the manufacturers on the bottle label (from two or three samples).

Write your results and conclusions in:
1. a report (1-2 pages), attaching the calibration curve and graph for the determination of the detection limit, and
2. an article for the School Newspaper (100-150 words).

Student self assessment
Complete the checklist below. If there are more than three "NO" answers, please consult your teacher.

Using scientific knowledge and understanding to solve problems
1. Can you search for information after putting in a keyword on the Internet?
2. Have you found useful information other than the suggested articles and websites?

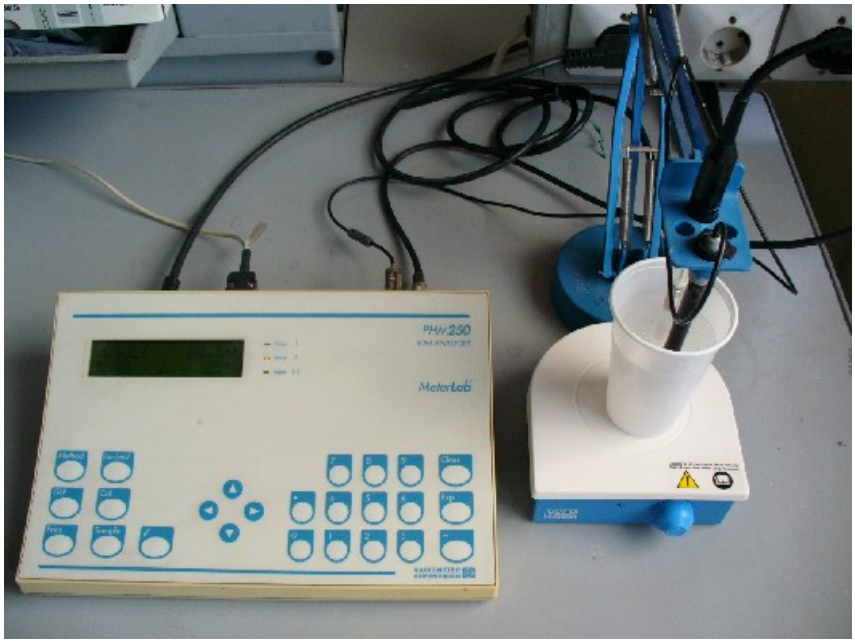
3. Can you summarise the advantages and disadvantages of using ISE electrodes?
4. Do you know the Nernst equation?
5. Do you use the pH/mV-meter in practice?
6. Did you understand the meaning of DL (detection limit)?

Working in teams to solve problems
1. Did you share the work between the members of your group?
2. Did you help other team members with their problems?
3. Can you evaluate the confidence level of your result?
4. Did you compare your result with the expected result (i.e. the value given on the bottled water label)?

Student sheet: Procedures

Use the procedure *Determination of fluoride ion content of toothpaste using potentiometry* from www. standardbase.com

Note: You do not need to carry out point (4) – *Preparation (toothpaste preparation)*.



Student sheet:
Extract from Technical data for fluoride ISE electrode

Electrode slope at 25 °C: 54±5 mV/decade
Linear (Nernstian) measuring range: 0.1 to 1900 ppm
Detection limit: 0.02-0.1 ppm
Response time: < 10 seconds (Defined as time to complete 90% of the change in potential after immersion in the new solution.)
Time for stable reading after immersion: 2 to 3 minutes (partly dependent on reference)
Potential drift (in 1000 ppm): < 3 mV/day (8 hours)
(Measured at constant temperature and with ISE and reference electrode continually immersed)

Recommendations for nomenclature of ion selective electrodes (IUPAC Recommendations, 1994)
Ask your teacher for a printed version or see
www.iupac.org/publications/pac/1994/pdf/6612x2527.pdf

Teacher's document

Overview
Students are working in the analytical laboratory of a water bottling company. Working in groups, they are asked to measure of fluoride content in mineral (bottled) water and to determine the detection limit of the method used.

Developing students' skills
Students learn to:
- use scientific knowledge (potentiometry, Nernst equation, ion selective electrode, reference electrode, calibration curve)
- use electroanalytical methods
- determine detection limit
- use the Internet, gathering information
- work in teams to solve problems
- communicate by writing a newspaper report

Organising and managing the class
Arrange the class into at least two groups.

Group A will determine the detection limit of the fluoride ion selective electrode and draw a diagram for the detection limit of ISE
Group B will measure fluoride content in water samples (suggested fluoride range: above 0.2 mg/dm^3). They will prepare a 'normal' calibration curve (suitable software is *ORIGIN* or *Microsoft Excel*).

Session plan for teachers
- Teacher's introduction to the scenario (maximum 45 minutes)
  - outline the equipment and chemicals available
  - statement of the expected output (content, format)
- Students gather information and different water samples (45 minutes)

For Group A
- Measuring detection limit of fluoride ion selective electrode (3 x 45 minutes)
- Making final report, evaluating results (45 minutes)
- Writing article for School Newspaper (45 minutes)

For Group B
- Measuring fluoride content in different samples of bottled water (3 x 45 minutes)
- Making final report, evaluating results (45 minutes)
- Writing article for School Newspaper (45 minutes)

Technical notes

Limit of Detection and TISAB

For monovalent ions, the IUPAC definition is: that concentration at which the measured potential differs from that predicted by the linear regression by more than 18 mV.
The practical limit of detection can be calculated by plotting a calibration graph using several standards at the lower end of the concentration range, and below it, say, 100, 10, 1, 0.1, 0.05, 0.01 ppm - i.e. at least two to define the linear slope and two to show the position of the horizontal section below the limit of detection, where the electrode is unresponsive to concentration change. The limit of detection is then defined by the crossing point of the two straight lines drawn through these points. (*Reproduced by kind permission of Dr C Rundle*)

It is likely that students will not understand the term TISAB. In this method, a series of fluoride standards are prepared in a background matrix of TISAB (Total Ionic Strength Adjustment Buffer). The unknown is prepared using TISAB on the basis that the matrix will be similar to the standards.

Preparation, equipment, materials and procedure

Refer to *Determination of fluoride ion content of toothpaste using potentiometry* from www. standardbase.com but with the following amendment to the suggested standards -
- standards for calibration curve: 10^{-1} -10^{-2} -10^{-3} -10^{-4} F^- mol/dm^3
- low concentration standards for determination of detection limit:
10^{-1} -10^{-2} -10^{-3} -10^{-4} -10^{-5} -10^{-6} -10^{-7} F^- mol/dm^3

Sample results

On tests for the fluoride content of several examples of bottled (spring) water, the results corresponded well with the fluoride concentrations given on the labels. It is suggested that teachers accept the average fluoride concentration as labelled. If students' measurements differ by less than 10%, this will be an excellent determination; more than 50% deviation indicates weak accuracy.
The detection limit of an average fluoride ISE is about 0.1- 0.3 mg/dm^3.

Two sample graphs are given below, one for the normal calibration curve, **Diagram 1**, and one for the determination of detection limit, **Diagram 2**.

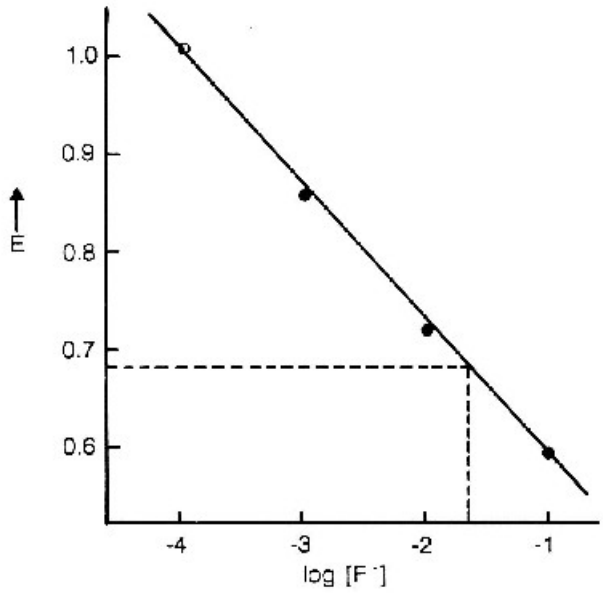


Diagram 1
E denotes electromotive force in V (or mV)

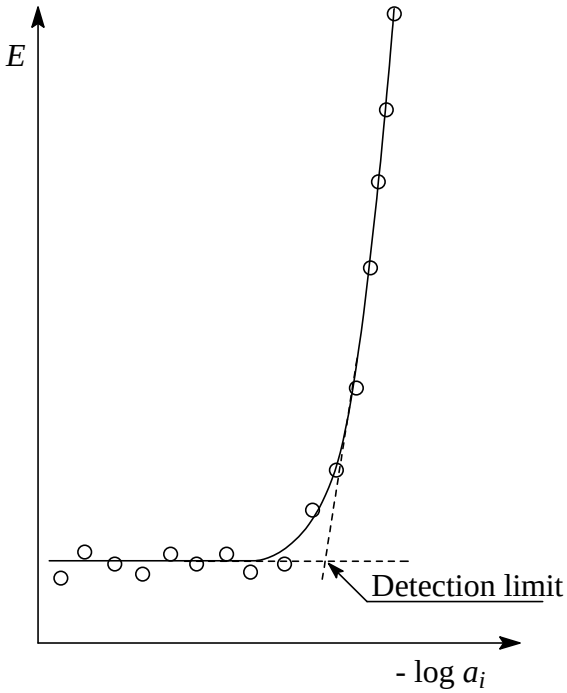


Diagram 2
E denotes electromotive force, a_i the ion activity but in practice, c_i (concentration) is normally used

TESTING PRIOR KNOWLEDGE

Choose the correct answer.

1. What is the F^- content limit in tap water, according to European Union Water Supply Regulations?
a. 1.8 mg/dm^3
b. 1.5 mg/dm^3
c. 0.8 mg/dm^3
d. 0.1 mg/dm^3

2. Which ions interfere significantly with the use of a fluoride ISE electrode?
a. chloride ions
b. bicarbonate ions
c. CO_2 gas
d. hydroxide ions

3. Calculate the pF of a solution with $c(F^-) = 3.15 \times 10^{-3}$ mol/dm^3.
a. 3.15
b. 11.72
c. 2.50
d. 3.67

4. The fluoride content in toothpaste...
a. increases the risk of osteoporosis.
b. helps to prevent tooth decay.
c. has no positive impact.
d. causes bone cancer.

Note: answers are given in red. These need to be removed before the test is used.

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