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Method of Analysis **PF30/83**		
Determination of the [redacted] using Ion Chromatography (IC)		
Product: [redacted]	Category: Finished Product	
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Pages: 6	*Tables:* 1	*Figures:* 1

1. PURPOSE AND APPLICATION FIELD

Quantitative determination of the content of [redacted] in samples of [redacted].

2. PRINCIPLE

Salification of a sample of [redacted] using NaOH and solubilisation in water. Determination of the fluoride ion (F^-) using ion chromatography (IC).

3. MATERIALS

3.1. Reagents

3.1.1. Standard solution of Fluorides 1000 µg/ml DIONEX type or equivalent.
3.1.2. 50% NaOH - J.T. Baker or reagent of similar purity.
3.1.3. 10% NaOH: *in a 50 ml volumetric flask place 10 ml of NaOH 3.1.2, accurately measured, and dilute to volume with milliQ water.*
3.1.4. MilliQ H_2O

3.2. Instruments and equipment

3.2.1. Dionex ICS2000 Ion Chromatograph or equivalent, equipped with conductometric cell and eluant generator + CR-TC
3.2.2. Dionex IONPAC AS14A 4mm guard column
3.2.3. Dionex IONPAC AS14A 4mm analytical column
3.2.4. Dionex ASRS-ULTRA 4mm Autosuppressor (External Water Mode)
3.2.5. pHmeter equipped with combined glass electrode
3.2.6. 50 ml and 100 ml volumetric flasks

4. SAMPLES

The samples to be analysed are [redacted].
CAUTION: Handle with care! (See point 7)

5. METHOD

5.1. Flow Diagram

Salification using NaOH
↓
Dilution
↓
IC analysis

5.2. Preparation of the standards and the sample

5.2.1. Preparation of the standards

Solutions of F^- at 0.3 – 0.5 – 1.0 – 3.0 mg / l

In four 100 ml volumetric flasks, using a precision pipet take 30, 50, 100, 300 µl respectively of the solution 3.1.1 and dilute to volume with milliQ H_2O.

5.2.2. Preparation of the sample

a) In a 100 ml beaker weigh 1 g of sample to a precision of 0.01 g
b) Add 50 ml of MilliQ H_2O and neutralise the solution very slowly with NaOH solution (3.1.3.) using the pHmeter until it is stable at pH 12.
c) Pour quantitatively into a 100 ml volumetric flask and dilute to volume with MilliQ H_2O.
d) Homogenise.

5.3. Operating conditions and IC analysis

- Loop: 50 µL
- Flow: 1.20 ml/min
- Current suppressor: 50 mA
- Eluant : 5.00 mM KOH generated using eluant generator
- CR-TC : On
- Retention time of F^-: 9 min (NB. Aging of the column leads to progressive reduction of retention times, which must be verified when calibration is carried out).
- Reading in area of peak
- Pump conditions: isocratic

5.3.1. Plotting the Calibration Curve

Set the operating conditions, allow the system to stabilise, verify that the conductivity is stable and inject the standards prepared in point 5.2.1 in sequence.
Plot the line of calibration, recording the peak areas for each of the concentrations of the standards.

5.3.2. Analysis of the samples

Inject the sample solution prepared in point 5.2.2. and, using the line of calibration, calculate the concentrations of fluorides in solution.
The injection is carried out immediately after the preparation.

5.4. Precision and accuracy (QA/QC)

The statistical elaboration of the data obtained from six consecutive repetitions carried out on the four standards used to establish the calibration curve is shown below (Tab.1):

where:

ST. DEV. = standard deviation $(\Sigma(x_{ij}\text{-MEAN})^2/(n\text{-}1))^{0.5}$

r = laboratory repeatability (2.8 * s)

(r) = % repeatability, (r / MEAN)*100

n = number of measurements

precision = (((max x_i – min x_i) / 2) / MEAN) * 100

In **Fig. 1** the verification of the linearity of the response of the instrument (integrated area) is shown in relation to the concentration of the standards (calibration line for fluorides, where: y = A (µS*min) ; x = C (mg/l))

Figure 1

6. CALCULATIONS

The concentration of [redacted] in the sample being examined, expressed in ppm (w/w), is calculated as follows:

[redacted]

where:
A = [redacted] in solution (µg/ml)
p = weight of the sample in g

7. SAFETY AND PRECAUTIONS

In handling the reagents used in the present method, refer to the relative safety data sheet which must be at the disposal of those using these substances:

- [redacted]
- Fluoride Standard (1000 mg/L)
- NaOH
- EGC KOH EluGen® cartridge (generation of KOH 5.00 mM using eluant generator)

The **samples of** [redacted]. Handle under properly functioning aspiration hood. Handle with care wearing protective gloves for acids and protective eyewear and using all other individual protective measures obligatory in the work area.

Sodium Hydroxide, which is dangerous due to its function as a base, is corrosive for mucous membranes, eyes and skin.

8. REFERENCES

Reference Library : Manuals, Technical Documents, Applications, Product Literature
DIONEX - P/N 053891-24