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Determination of Fluoride Levels in Toothpaste using Ion Chromatography

Purpose: To Determine the amount of fluoride in one gram of Children's Toothpaste, and the amount of fluoride in one gram of Adult Toothpaste.

Procedure:

1. Calibrate Metrohm Ion Chromatograph using prepared standard solutions, or use those supplied by manufacturer, according to instructions provided in the chromatograph's user manual.

2. Prepare a 1 parts-per-million diluted solution using Adult Toothpaste*:
   a. Carefully measure 0.100 grams of Adult Toothpaste using a calibrated digital balance.
   b. Clean and prepare a 1000mL Volumetric Flask, filled approximately one-third with deionized water.
   c. Carefully add 0.100 grams of Adult Toothpaste to volumetric flask prepared above. Be sure to rinse any remaining toothpaste from weigh paper or spatula into the volumetric flask using deionized water.
   d. Carefully fill volumetric flask with deionized water to the "fill line" on the flask, making sure the meniscus (water's surface) just touches the line.
   e. Place glass stopper into the top of the volumetric flask, seal with parafilm, and shake the volumetric flask vigorously for 60 seconds to dissolve toothpaste within the flask completely.
   f. Allow the contents of the flask to settle for a minute, and shake again if necessary, until toothpaste is completely dissolved.

   *Perform these same steps to Prepare a diluted solution of the Children's Toothpaste.

3. Once chromatograph has completed the calibration run, prepare the following sample tubes (use only the sample tubes provided by chromatograph manufacturer, unless otherwise instructed):

Tube #	Contents
1	"Blank" sample of deionized water
2	Approximately 1mL of the diluted Adult Toothpaste solution prepared in step #2
3	"Blank" sample of deionized water
4	Approximately 1mL of the diluted Children's Toothpaste solution, using procedures in step #2

   Load each of the sample tubes into the chromatograph auto sampler, using the corresponding slots (1-4) on the carousel. Then move on to step #4.

4. Using the chromatograph's computer interface, program the test run as follows:

a. Select "Detrermination Series" from the main interface. This allows you to run a series of sample tubes.

b. With the first line of the series highlighted, go to the bottom of the dialogue window and click "Edit Line". This opens an edit dialogue box, where values for that particular sample can be entered.

c. In the edit dialogue, enter "1" for the position of the first sample tube in the autosampler. Select "Anions Without Rinse" as the method of analysis, Select "blank" as the content type, 20 micro liters (20uL) for the sample amount, 0 for the dilution value. Rather than complete the entry, at the bottom of the dialogue, click the "right arrow" button to add another entry.

d. For this second tube, enter "2" for the position of the second sample tube in the autosampler. As above, select "Anions Without Rinse" as the method of analysis, Select "sample" as the content type, 20 micro liters (20uL) for the sample amount, 1,000,000 for the dilution value (the toothpaste has been diluted 1 million times). Rather than complete the entry, at the bottom of the dialogue, click the "right arrow" button to add another entry. Repeat the entry process for the other two tubes in the autosampler.

e. With the autosampler loaded with prepared sample tubes, and the Series Determination set-up, on the chromatograph's computer interface, close the sample entry dialogue, and click the red "Run" button on the top left of the screen. This initiates the ion chromatograph to begin analysis.

5. Upon completion of the analysis, the ion chrmomatograph will display results of the analysis via an onscreen graph, which displays various peaks according to ion presence and concentration. Attached to this graph is also a set of numerical values organizes in to a table (a separate file accessible via the computer interface). The graph and table can be saved in pdf format and/or printed directly from the interface. On-screen display will also note important retention times used to identify ion species, and peak height, which denotes concentration. Total area under the curve for each ion identified by the chromatograph measures the amount present in the sample. The chromatograph has already factored in the dilution of the sample if you entered a dilution amount during the series determination set-up. Using these values, the levels of fluoride in toothpaste samples can be determined.