

F-analyse of Smyle test products.

Author : Dr. R.A.M. Exterkate

Amsterdam, mei 2021

Introduction

ACTA was asked to determine the content of total fluoride and free fluoride. The company Smyle has offered a number of test products for which they want to determine the fluoride release. The original report by ACTA was in Dutch. The report was translated and approval from ACTA was obtained for this version to be used by Smyle.

Material & methods

For the determination of the free-Fluoride content, approximately 1 g of product is accurately weighed. Then about 10 ml of milli-Q water is added at t=0. Then the container with product, water and a stirrer is shaken by hand for 1 min. The mixture is then placed on a magnetic stir plate and mixed for an additional 4 minutes. The total mixing time is then 5 minutes. After mixing, 1.4 ml of slurry is transferred to an eppendorf cup and centrifuged at 15000rpm for 8 minutes and 500 µl of the supernatant is pipetted into a new eppendorf cup. The F content in the supernatant is measured.

Contrary to the official procedure, we use gas-liquid chromatography (GLC) instead of a selective F electrode. In an earlier email message was already explained why the official procedure uses an electrode instead of GLC.

The official procedure is stated in the English text below:

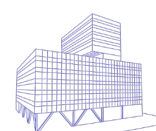
“Available fluoride will be determined using the procedure described for test Number 29 in the FDA Monograph using updated procedures.

TriPLICATE samples of approximately 2.5 g of each dentifrice (weighed to 0.001) will be mixed with 25 ml of de-ionized water (1:10) for 1 minute with a non-aerating mixer. A magnetic stir bar will be added to each slurry and mixed for an additional 4 minutes on a multipoint stir plate at 300 rpm (total mixing time of 5 minutes). Slurries will then be immediately centrifuged for 8 minutes at 11,000 g. 1.0 ml of each supernatant will be mixed with 1.0 ml of TISAB II. Analyses of these solutions will be performed using a fluoride ion specific electrode and an ion analyzer. A standard fluoride curve will be prepared and used for determination of the fluoride content of each of the dentifrice slurries.”

The total F content of the slurry is determined by mixing 100 µl of slurry in a glass vial with 1.3 ml 1 mol/L HCL and 400 µl toluene reagent. Toluene reagent consists of 9ml toluene, 0.9ml 1%

iso-pentane (internal standard) and 100 µl TMCS (trimethylchlorosilane). In an acid environment, TMCS is converted to TMFS (trimethylfluorosilane) and this can be measured in a gas chromatograph. By measuring a series of standards, a calibration curve can be drawn in which the area of the peak is plotted in relation to the F concentration. The internal standard is used to correct for small variations in injection volume. Dividing the area of the TMFS peak by the area of the iso-pentane peak produces a number that corrects the value of the F peak for the variation in injection volume.

The free F content is determined by analyzing 100 µl of the supernatant in a similar manner to the slurry. By measuring the F content of the supernatant you can determine which part of the fluoride is freely available.



Results

All products were weighed in duplicate. All solutions were measured in duplicate. The table shows the duplicate samples. So the 2 observations come from 2 independent samples. The column "free F" shows the F concentration in ppm that was present in the supernatant and thus released during the 5 minutes of mixing. The total F column shows the total concentration of F as determined by analyzing the slurry.

The Smyle company has delivered 2 products. These have been worked up and analyzed in a similar way.

Product	free F	totaal F		free F	total F
V7	1016	1416		1368	1409

The Smyle tabs V7 show a F-release of around 84%.

Remark: Besides a number of tests and internal controls the tested products also comprised a final Brushmint (V7). Only the results of the final Brushmint are shown in the table above. Other test products were for internal use only. The table was adapted accordingly from the original report.

