Determination of Fluoride Content in Toothpaste

Modern toothpastes are designed to do more than just clean teeth. Hence they contain many additives, either for health or cosmetic reasons, including components to prevent tooth decay, colourants, flavours, and preservatives. Fluoride is an important component of toothpaste as it protects the tooth by making the enamel harder. However, toothpastes are classified as drugs, not cosmetics, therefore the level of fluoride must be carefully controlled and measured accurately.

Method
Conventional methods currently in use for determining the fluoride content (titration, ion selective electrodes or ion chromatography) are time consuming, require a trained analyst, and may involve the use of strong acids.

In contrast, low resolution Nuclear Magnetic Resonance (NMR) provides a method for fluoride determination that is rapid, easy to perform and simple to calibrate. NMR detects the soluble fluoride content of toothpaste which is the most important parameter in determining its effectiveness to prevent tooth decay.

The two main fluoride based additives are sodium fluoride (NaF) and sodium monofluorophosphate (MFP). Most toothpastes contain between 0.10 and 0.15 wt% (1000 to 1500 ppm) fluoride, a level easily detectable by NMR. The toothpastes can be either a gel or paste with widely different water and abrasive contents. However, NMR does not exhibit any change in sensitivity with different formulas.

Calibration and Results
Samples of commercial toothpastes and laboratory preparations were used to develop and validate the NMR method.

Tests on real toothpaste samples indicate that the NMR signal intensity is correlated with the fluoride content in the sample as shown in the figure below.

Calibration simply requires a minimum of six sodium fluoride standards (in distilled water) that span the concentrations of interest. The instrument is calibrated at installation and only requires ongoing calibration checks as a test of the performance.

Instrument repeatability was then tested by measuring one sample ten times without removing it from the instrument. Sample repeatability was tested by measuring five different portions of the same sample. The results of both are shown over the page.
Results of instrument and sample repeatability

<table>
<thead>
<tr>
<th>Ref. (ppm)</th>
<th>Repeat Measurements</th>
<th>MEAN</th>
<th>SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>1447</td>
<td>1465 1466 1444 1431 1445 1453 1441 1452 1438 1455</td>
<td>1449</td>
<td>11.3</td>
</tr>
<tr>
<td>Value</td>
<td>Portion Measurements</td>
<td>MEAN</td>
<td>SD</td>
</tr>
<tr>
<td>1410</td>
<td>1393 1422 1438 1413 1407 1415</td>
<td>1415</td>
<td>16.8</td>
</tr>
</tbody>
</table>

Recommended Instrument

The MQC-F with a 0.55 Tesla magnet, fitted with a 26 mm diameter (14 ml sample) Fluorine probe is a suitable instrument for this application. The Fluoride in Toothpaste package comprises:

- **MQC-F** with a built-in computer operating the latest version of Microsoft® Windows® (no separate PC is required)
- **MultiQuant** software including RI Calibration, RI Analysis, and the EasyCal ‘Fluoride in Toothpaste’ application
- 26 mm diameter glass tubes
- Installation manual
- Method sheet

In addition to this package you may also require:

- A dry heater and aluminium block with holes for sample conditioning at 40°C
- A precision balance

The instrument offers multiple advantages over other instruments on the market:

- High signal sensitivity
- Low maintenance
- The sample tubes are recyclable, lowering consumable costs
- Minimal sample preparation