Fluoride, previously thought to be an essential element for tooth development, is actually a toxin that can cause dental fluorosis, osteosarcoma, and neurological impairment. The risk of such harmful effects increases with children consuming formulas, as they are more sensitive to smaller concentrations than adults and are thus more likely to suffer the adverse effects. The recommended daily amounts for children under the age of 3 range from 0.01–0.07 mg per day.

In order to address these concerns, new and more stringent standard method performance requirements (SMPRs) are being developed for infant formulas by the SPINALA initiative. The official AOAC method for fluoride determination is not able to accommodate these SMPRs as it can only reach a limit of quantitation (LoQ) of 2.0 ppm and the results can vary widely from analyst to analyst due to the assay’s subjective nature. In response, a new and more robust method for determination of fluoride in infant formulas has been developed to address these concerns. Free fluoride is extracted from infant formula matrices under acidic conditions and the interfering analytes are removed by an alternative chromatograph. The fluoride is then converted to trimethylfluorosilane (TMFS) extracted into toluene, and analyzed by GC-FID. The fluoride content is determined at a LoQ of 0.20 ppm using a seven-point standard reference curve. Validation was performed with a Certified-in-house fluoride control as well as an in house fluoride control; it was found that the common infant formula matrix contained 0.33 ppm fluoride with a standard deviation of 0.03 ppm and an RSD of 10%. The novel method not only delivers more precise results for infant formula but could eventually replace the antiquated AOAC method.

### Methods and Materials

#### Experimental Design

Ionic fluoride was extracted from the sample matrices under acidic conditions. Several cleanup steps were employed in order to remove solid particles from the aqueous fraction. The deratization procedure was optimized by varying injection volumes, temperatures, and gas flow to achieve as small analyte discrimination, improved responses. Initially, splitless injection was used in order to achieve trace levels of quantitation. The instrument parameters listed in Table 1 allow for the removal of nearly all matrix interference. In addition, it necessitates the use of hazardous chemicals such as perchloric acid.

#### Results and Discussion

Overall precision is based on the comparative analysis of two in-house validated controls; rat Diet Meal Control and rat Diet Meal Control plus a commercial matrix (MBF by FID). At least 10 comparison results are displayed in Table 2: 1 replicates of the RCP control and of the MW control. The nominal values obtained are compared with their accepted ranges of 3.66 ± 0.13 µg/g for both controls and 3.52 ± 0.05 µg/g respectively. Analysis of the results showed a mean improvement in %RSD for these two matrixes with respect to the other methods.

#### Table 2. Control Comparisons on GC-FID Method (n=5)

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Mean (µg/g)</th>
<th>%RSD</th>
<th>Mean (µg/g)</th>
<th>%RSD</th>
<th>Mean (µg/g)</th>
<th>%RSD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rat Diet Meal Control</td>
<td>4.68</td>
<td>3.41</td>
<td>4.71</td>
<td>3.40</td>
<td>4.71</td>
<td>3.40</td>
</tr>
<tr>
<td>Rat Diet Meal Control + MBF</td>
<td>4.71</td>
<td>3.40</td>
<td>4.71</td>
<td>3.40</td>
<td>4.71</td>
<td>3.40</td>
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<tr>
<td>Standard Reference</td>
<td></td>
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</tbody>
</table>

#### References

2. **Official Methods of Analysis of AOAC INTERNATIONAL (2012) 19th Ed. AOAC INTERNATIONAL, Gaithersburg, MD, USA, Official Method 944.08 (Modified).**
Analysis of Fluoride Content in Infant Formula by GC-FID

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