# METHOD STATEMENT

## Determinand:
Determination of acid extractable fluoride

## Matrix:
Sample Type: soil and sludges

## Principle of Method:
A sample of soil (dried and ground) or sewage sludge (liquid) is extracted using 105℃ dilute sulphuric acid. A citrate buffer is used to adjust the final pH to 5.4 ± 0.2, release any complexed fluoride ions and to maintain the provision of constant high ionic strength. A fluoride selective electrode is used to measure the activity of fluoride in the prepared extract by means of standard addition technique, since the samples analysed contain substances likely to interfere.

## Interferences:
Fluoride ions readily form complexes with polyvalent cations such as $Al^{3+}$, $Ca^{2+}$, $Fe^{3+}$, $Mg^{2+}$ and $PO_4^{3-}$ making them undetectable by ion selective electrode. Citrate buffer contains complexing agents, which release fluoride ions from metal-fluoride complexes that form. Under acidic conditions the formation of HF reduces the concentration of fluoride ions. The citrate buffer is designed to avoid these interferences by maintaining the pH of the sample between 5.2 and 5.6

## Performance of Method:
***Range of Application:***

Reporting limit: 10mg/kg as F

***Limit of Detection:***

2.7mg/kg as F

***Bias and Recoveries of Compounds:***

| Sample type | Mean sample result (mg/kg) | Bias (%) | RSD (%) | Spike recovery (%) |
|---|---|---|---|---|
| Soil | 60.45 | - | 4.98 | 88.55 |
| Liquid sludge | 106.30 | - | 5.43 | 96.05 |

| Sample type | RSD (%) | Prediction interval, mg/kg | Mean result, mg/kg |
|---|---|---|---|
| Sandy loam CRM | 7.71 | 45.5 - 193 | 148.20 |
| Clay CRM | 7.51 | 77.9 - 412 | 291.80 |

## References:
Fluoride in waters, effluents, sludges, plants and soils 1982; Methods for the Examination of Waters and Associated Materials; HMSO 1983 (ISBN 011 751662 7).