METHOD STATEMENT

Determinand:
Determination of Total Inorganic Phosphate including those phosphate compounds that hydrolyse to orthophosphate after pre-treatment with sulphuric acid.

Matrix:
Sample Types: Raw, Potable, Surface and Ground waters.

Principle of Method:
This method uses the Liebisch Digestion Block or similar and a laboratory Discrete Analyser for the final end point analysis.
The sample is hydrolysed with concentrated sulphuric acid to convert any condensed polyphosphates to orthophosphates. The extract is then reacted in an acid medium. Ammonium molybdate and antimony potassium tartrate react with orthophosphate to form an antimony-phosphor-molybdate complex. This is then reduced by ascorbic acid to form a blue complex, the intensity of which is proportional to the original concentration of orthophosphate ions present. The intensity of the colour formed is measured at a wavelength of 724 nm using automated discrete colorimetric analysis

Sampling and Sample Preparation:
Samples are normally collected in 500 ml PET bottles. Other size PET or HDPE bottles are also suitable.
No special preservation is required
If analysis cannot be immediately undertaken, samples should be stored at a temperature of 3±2°C until the day of digestion. Samples should be digested within 30 days sampling date. The sample can then be stored at a temperature of 3±2°C until the day of analysis.
The sample should be warmed up to room temperature prior to analysis and analysis should be carried out within 5 days of digestion.

Interferences
The samples are neutralised such that extreme pH should not affect the final analysis. Interferences associated with the end-point orthophosphate analysis include sulphide, arsenic and silicon.

Performance of Method:

Range of Application:
LOQ - 2.0 mg/l P
Samples with a concentration higher than that of the top standard of 2.0 mg/l P should be diluted with deionised water and reanalysed. This range may be extended by sample dilution post digestion with the digested blank.
Reporting Limit is 0.091 mg/l P

Limit of Quantification:
0.091 mg/l P

Recoveries of Compounds, Bias and Uncertainty of measurement:

Sample type	Mean sample result (mg/l P)	Mean sample spike result (mg/l P)	Conc. of spike (mg/l P)	Spike recovery (%)	% uncertainty
Soft	0.502	1.566	1.1	97.79	±6.01
Medium	0.861	1.509	0.7	93.27	± 9.91

Sample type	Mean sample result (mg/l P)	Mean sample spike result (mg/l P)	Conc. of spike (mg/l P)	Spike recovery (%)	% uncertainty
Hard	1.250	1.610	0.4	90.29	±11.86
Borehole	0.049	1.620	1.6	99.79	±2.56
Raw	0.028	1.600	0.8	99.79	±3.19

References:

Phosphorus- Methods for the Examination of Waters and Associated Materials 1980, ISBN 0 11 7515825
WPC64- Anions by Colorimetry in Potable, Surface and Ground Waters
WPC12 - Determination of Dissolved (Filtered) and Total Metals in Raw, Potable, Surface and Groundwaters by ICP-OES