

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 Liebig 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

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 the Method:

Range of Application:

LOD - 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 0.046 mg/l P

Limit of Detection:

0.046 mg/l P

Recoveries of Compounds 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
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

