

## METHOD STATEMENT

### **Determinand:**

Manual determination of Sulphide

### **Matrix:**

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

### **Principle of Method:**

Hydrogen Sulphide and acid-soluble metal sulphides in acidic solution react with N,N-dimethyl-p-phenylenediamine to form Methylene Blue. The intensity of the Methylene Blue formed is proportional to the concentration of sulphide present and is measured in a spectrophotometer at 665 nm. The instrument gives a readout of sulphide concentration in  $\mu\text{g/l}$ .

### **Interferences:**

Strong reducing substances such as sulphite, thiosulphate and hydrosulphite can interfere by reducing the blue colour produced or preventing its development. High concentrations of sulphide may inhibit the full colour development.

Highly coloured or turbid samples will interfere and give a false high result. These samples should be analysed using the Waste Sulphide method, which accounts for sample colour/turbidity.

### **Performance of the Method:**

#### ***Range of Application:***

LOD - 800  $\mu\text{g/l S}^{2-}$  for a 10 ml sample volume.

The analytical range may be extended by sample dilution. Samples with a concentration higher than that of the top standard of 800  $\mu\text{g/l S}^{2-}$  should be diluted with deionised water, prepared again and analysed.

Reporting Limit is 7  $\mu\text{g/l S}^{2-}$

#### ***Limit of Detection***

6.67  $\mu\text{g/l S}^{2-}$

#### ***Recoveries of Compounds and Uncertainty of measurement:***

Sample type	Mean sample result ( $\mu\text{g/l}$ )	Mean sample spike result ( $\mu\text{g/l}$ )	Spike recovery (%)	Bias (%)	% uncertainty
Soft- Langsett	2.7	623.3	96.96	-	-
Medium - Wakefield	1.6	619.8	96.60	-	-
Hard - Bristol	1.4	623.7	97.22	-	-
Borehole - Nutwell	1.2	616.8	96.18		
Surface -Derwent	1.5	622.9	97.09	-	-
160 $\mu\text{g/l}$ Std	158.6	-	-	-0.90	$\pm 8.35$
640 $\mu\text{g/l}$ Std	603.6	-	-	-5.69	$\pm 8.84$

### **References:**

Based on Chemical Disinfecting Agents in Water and Effluents and Chlorine Demand 1980 (HMSO) ISBN 0117514934.

Hach Lange Sulfide Methylene Blue Method 8131

