

METHOD STATEMENT

Determinand:

Total and dissolved sulphide

Matrix:

Leachates, effluents and waste waters

Principle of Method:

In an acidic solution, sulphide reacts with N-N-diethyl-p-phenylene-diamine in the presence of dichromate ions to produce the coloured complex DPD Blue. The intensity of the colour is proportional to the concentration of sulphide present and is measured using a spectrophotometer set at a wavelength of 670nm.

Samples must be preserved using sodium carbonate and zinc acetate at the point of sampling. For dissolved sulphide, the sample is filtered prior to preservation. (A result for dissolved sulphide cannot be obtained for an unfiltered sample after it has been preserved).

Sampling and Sample Preparation:

Sulphide is readily lost to the atmosphere, especially in acidic conditions. All samples must be preserved at the time they are taken by adding 1ml ± 0.1ml of the sodium carbonate solution to immediately neutralise any acid, followed by 1 ± 0.1ml of the zinc acetate solution to precipitate the sulphide within the sample as stable zinc sulphide.

Samples are stable for 28 days (Standard Methods:-ISBN 0-87553-161-X) from sampling. Samples are kept refrigerated before and after analysis.

Interferences:

Although sample turbidity is corrected for, excessively turbid sample may not give as reliable results as clear solutions. Similarly, highly coloured samples may be detrimental to colour measurement, despite a correction being applied.

Thiosulphate above 10mg/l and sulphites above 2mg/l will cause loss of colour. Iodides above 2mg/l and cyanides above 500mg/l will delay colour formation.

Performance of Method:

Range of Application: 0.029 to 2.5 mg/l for a 10ml sample volume

Limit of Detection: 0.0289 mg/l

Normal Reporting Level: <0.029 mg/l

Determinand	Low standard		High standard	
	Tot. RSD %	Bias %	Tot. RSD %	Bias %
Sulphide	5.09	4.81	5.10	2.31

Determinand		Final Effluent		Trade Effluent		Untreated Effluent	
		20%	80%	20%	80%	20%	80%
Sulphide	Rec. %	105.93	103.10	103.99	103.91	101.24	99.34
	%RSD	7.67	4.90	6.46	3.65	6.52	4.98



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Uncertainty of Measurement

The reported uncertainty is an expanded uncertainty calculated using a coverage factor of 2, which gives a level of confidence of approximately 95%.

Determinand	Uncertainty of Measurement (%)
Sulphide	16.45

References:

Sulphide in Waters and effluents 1983, Tentative Methods. HMSO Methods for the examination of waters and Associated Materials. ISBN 0117517186.

Standard Methods for the examination of Waters and Wastewater, 20th edition, 1998, American Public Health Association / American water Works Association / Water Environment Federation. ISBN 0-87553-235-7

Second Site Property Environmental Assessment Guidelines Version 3 - March 2003.

ISBN 0-87553-161-X, Standard Methods for the Examination of Water and Wastewater.

