

METHOD STATEMENT



Determinand:

Chemical Oxygen Demand (COD), (Total, Settled & Filtered)

Matrix:

Leachates, effluents, sewage and waste waters

Principle of Method:

Samples are oxidised by heating in vials with sulphuric acid and potassium dichromate. Mercuric sulphate is added to suppress chloride interference. The dichromate is reduced to chromate during the digestion and the chromate produced is measured colorimetrically.

Sampling and Sample Preparation:

There is no sample preservative used. Samples should be analysed as soon after receipt as possible. Samples requiring settled COD should be allowed to settle for a minimum of 1 hour and not more than 1 hour 30 minutes.

Samples requiring filtered COD should be filtered through a Whatman 0.45µm filter.

Samples requiring pH7 COD should be adjusted to pH7±0.5, using 10% H₂SO₄ and 1M NaOH and allowed to settle for a minimum of one hour.

Samples are stable for 6 days (In-House Data) from sampling.

Interferences:

If not suppressed, chloride will cause positive interference. Inorganic reducing agents such as nitrites, sulphites and ferrous salts will contribute to the COD. Oxidising agents, e.g. Cr VI salts, can give false negative and low results. Bromide interference will not be controlled by mercuric sulphate.

Performance of Method:

Palintest P8000

Range of Application: 11-2000mg/l

Limit of Detection: 10.98mg/l

Normal Reporting Level: 11mg/l

Determinand	MCERTS Accreditation	Resorcinol		Low standard		High standard	
		RSD %	Bias %	RSD %	Bias %	RSD %	Bias %
COD	✓	3.38	+4.22	1.67	-0.53	0.74	+2.61

Determinand		Treated Sewage		Trade Effluent		Untreated Sewage		Ground Water	Land Leachate	Prepared Leachate	Surface Water	
		CLOI	80%	20%	80%	20%	80%	80%	80%	80%	CLOI	80%
COD	%Rec.	93.19	101.52	97.59	102.02	93.64	98.06	101.73	102.85	102.53	96.15	100.28
	%RSD	4.7	1.64	1.79	0.74	5.78	1.14	1.17	1.67	1.08	4.41	0.94

Critical Level of Interest (CLOI): 125mg/l (Urban Waste Water)

METHOD STATEMENT



Hach DR3800

Range of Application: 11-2000mg/l

Limit of Detection: 9.92mg/l

Normal Reporting Level: 11mg/l

Determinand	MCERTS Accreditation	Resorcinol		Low standard		High standard	
		RSD %	Bias %	RSD %	Bias %	RSD %	Bias %
COD		2.3	+4.87	0.68	+1.41	0.58	-0.09

Determinand		Treated Sewage		Trade Effluent		Untreated Sewage		Ground Water	Land Leachate	Prepared Leachate	Surface Water
		CLOI	80%	20%	80%	20%	80%	80%	80%	80%	80%
COD	%Rec.	96.21	94.55	97.30	99.17	96.81	96.37	99.43	96.77	106.83	97.55
	%RSD	2.79	1.68	3.15	0.52	4.05	1.83	1.65	0.48	1.17	0.68

Critical level of interest: (CLOI) 125mg/l (Urban Waste Water)

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 %
COD	10.04

References:

Chemical Oxygen Demand (Dichromate Value) of Polluted and Waste Waters 1977. HMSO Methods for the Examination of Waters and Associated Materials. ISBN: 011 7512494.

Chemical Oxygen Demand (Dichromate Value) of Polluted and Waste Waters 1986 (Second Edition). HMSO Methods for the Examination of Waters and Associated Materials. Method B. ISBN: 011 7519154.

HACH Water Analysis Handbook 1997. Method 8000, Oxygen Demand, Chemical. ISBN: 23196-01.

COD analysis of saline waters - an investigation into chloride interference suppression. In-house investigation report. P Clark MDPC 046. 2002.