

Attachment E.2.2.

Test Method Manuals

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Wastewater Sampling Method

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1:1 Personal Safety

Before sampling is undertaken familiarisation with the following documents is essential:

- Leitrim County Council Corporate Safety Statement.
 - Infrastructure Ancillary Safety Statement.
 - Site Specific Risk Assessments for Wastewater
 - Safe Work Practice Sheets Nos. 67, 72 and 81
1. Personal Protective Equipment must be worn at all times, visibility vests, protective footwear and gloves.
 2. When sampling near water, life belts must be used and if possible two persons should carry out the sampling.
 3. Easy access to and from the sampling location should be established before sampling commences.
 4. Information regarding the location(s) of sampling and estimated time of return to the laboratory should be made available before departure from the laboratory.
 5. Mobile phones should be carried at all times with contact numbers in case of emergency.

2:1 Introduction

A monitoring programme should be pre-determined before the start of each sampling year. The programme should set out each point at which samples are to be taken for particular parameters, how many samples are to be taken at each point for each parameter and when the samples are to be taken. The programme should ensure that the number of samples taken at each wastewater treatment plant (WWTP) is distributed as evenly as practicable throughout the year.

Samples collected in any monitoring programme must be representative of the quality of the bulk of the material being monitored. The act of collecting a sample must not alter the quality characteristics of the sample of the material collected for analysis from those of the bulk of the material from which the sample was drawn.

2:2 Purpose

The purpose of the wastewater sampling method is to define a sampling programme, to ensure confidence in analytical results and to ensure a suitable response to unsatisfactory results.

2:3 Scope

The wastewater sampling method relates to the sampling of all wastewater treatment plants in County Leitrim.

3:3 Sampling

3:3.1 Laboratory Check

Ensure that the materials necessary for sampling and analyses are present within the laboratory before undertaking sampling. A written work list identifying all samples to be taken and identifying compliance and operational samples should also be present within the laboratory.

3:3.2 Sampling Location

Ensure that the sampling location will give a representative sample.

3:3.3 Calibration of automatic samplers

1. Automatic samplers must be calibrated on-site at each sample location before use.
2. Set the time interval dial on the panel inside the sampler to 1hr.
3. Set the volume dial on the panel to 300mls.
4. Place the end of the sampling tube into a 500ml graduated cylinder and press start.
5. Measure the volume lifted.
6. If the volume is greater or less than 300mls, reset the volume dial and repeat step 4.
7. Continue steps 4 to 6 until the sampler lifts a 300ml volume.
8. The automatic sampler is now ready for use.

3:3.4 Sampling

1. Ensure that proper sample bottles are used and that adequate volumes of sample are taken.
2. Ensure that sample bottles are labelled with the sample location, date and time, along with any other relevant information deemed necessary. Where applicable ensure that appropriate preservatives are placed in bottles used to collect samples.

3:3.5 Dispatch to Laboratory

1. The time interval between sampling and arrival at the laboratory must be the minimum practicable consistent with local logistical problems.
2. As a minimum samples should be held at less than 10°C while being transported to the laboratory.
3. On arrival at the laboratory samples should be logged in the sample log book and given individual identification numbers. These references numbers should be written on the sample bottles and used throughout analysis and recording of the sample.
4. Samples should be analysed on the day of receipt at the laboratory wherever possible and refrigerated overnight if too late for processing on the same day as sampling.

4.1 Bibliography

Environmental Protection Agency (2003). **European Communities (Drinking Water) Regulations, 2000 (SI 439 of 2000) A Draft Handbook on Implementation for Sanitary Authorities.** Pages 1 to 147. Published by the EPA, PO Box 3000, Johnstown Castle Estate, Co. Wexford.

North Western Health Board (1999) **Microbiological Sampling of Water Supplies Serving Food Businesses – Standard Operating Procedures.** SOP 17.0, Revision No: 0, Issued: 25/01/99, Approved By: National Steering Committee. Pages 1 to 5.

Biochemical Oxygen Demand

Electrode Method

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1:1 Laboratory Safety

Safety is the responsibility of every individual working within a laboratory environment (Hach, 2002).

The following safety guidelines apply to individuals working within a laboratory environment. These guidelines are not intended to cover all aspects of safety:

General Safety

1. Do not block exits or access to emergency equipment within the laboratory.
2. Do not smoke, eat or drink within the laboratory.
3. Keep work areas neat and clean within the laboratory.
4. Never pipette by mouth within the laboratory.
5. Wipe up spills promptly within the laboratory. Appropriate training in spill management should be obtained.

(Hach, 2002)

Protective Equipment

1. Wear safety glasses to protect eyes from flying objects or chemical splashes.
2. Wear gloves to protect skin from toxic or corrosive materials, sharp objects, very hot or very cold materials or broken glass.
3. Wear laboratory coats to protect skin and clothing from splashes.
4. Fume hoods should be used when directed to do so by the procedure, or as recommended in the material safety data sheet (MSDS).

(Hach, 2002)

First Aid Equipment and Supplies

1. First aid kits should be provided within the laboratory. Appropriate training in first aid should be obtained.
2. Eyewash and shower stations should be provided within the laboratory.
3. Fume hoods and appropriate fire extinguishers should be provided within the laboratory. Appropriate training in fire extinguisher use should be obtained.

(Hach, 2002)

Appropriate protective equipment should be worn when undertaking the BOD (Biochemical Oxygen Demand) electrode method.

2:1 Introduction

The BOD electrode method is suitable for determining the BOD of water and wastewater.

2:2 Principle

The BOD determination is an empirical test in which standardised laboratory procedures are used to determine the relative oxygen requirements of wastewaters, effluents and polluted waters (APHA, AWWA and WPCF, 1985).

The test measures the oxygen required for the biochemical degradation of organic material (carbonaceous demand) and the oxygen used to oxidise inorganic material such as sulphides and ferrous iron. It may also measure the oxygen used to oxidise reduced forms of nitrogen (nitrogenous demand) unless their oxidation is prevented by an inhibitor (APHA, AWWA and WPCF, 1985).

The method consists of placing a sample in a full, airtight bottle and incubating the bottle under specified conditions for a specific time. Dissolved oxygen (DO) is measured initially and after

incubation. The BOD is calculated from the difference between the initial and final DO (APHA, AWWA and WPCF, 1985). Normally a 5-day BOD test period (English legend has it that 5 days was the time period taken between sewage entering a river and reaching the ocean) is used where samples are incubated in the dark (to restrict algal growth) at 20°C (the temperature believed to be reasonably representative of field conditions).

Most wastewaters contain more oxygen demanding materials than the amount of DO available in air-saturated water. Therefore it is necessary to dilute certain samples before incubation to bring the oxygen demand and supply into appropriate balance (APHA, AWWA and WPCF, 1985).

Bacterial growth requires nutrients such as nitrogen, phosphorus and trace metals. These are added to dilution water, which is buffered to ensure that the pH of the incubated sample remains in a range suitable for bacterial growth (APHA, AWWA and WPCF, 1985).

3:1 Materials

1. BOD nutrient buffer pillows (Hach Catalogue No.'s 14861-66 and 14862-66 or equivalent)
2. Nitrification inhibitor (Hach Catalogue No. 2533-35 or equivalent)
3. D(+)Glucose (BDH Product No. 101174Y or equivalent)
4. L-Glutamic acid (BDH Product No. 371024T or equivalent)
5. Purified water or equivalent
6. BOD dilution water container
7. BOD dilution water aerator
8. BOD bottles
9. DO meter and electrode
10. Water bath (20 to 30°C)
11. Balance (4 decimal places)
12. Weighing boats
13. Porcelain crucibles
14. Dessicator
15. Spatula
16. Magnetic stirrer
17. Incubator (20°C +/- 1°C)
18. Oven (103°C)
19. Volumetric flask (1000mL)
20. Graduated cylinders (10 to 1000mL volume)
21. Parafilm
22. Pasteur pipettes
23. Gloves
24. Marker Pen
25. Scissors

4:1 Preservation Method

If more than a 2-hour delay before analysis of grab sample(s) takes place the sample(s) should be kept at or below 4°C from the time of collection and analysis should begin within 6 hours of collection. If this is not possible store sample(s) at or below 4°C and report length and temperature of storage with the results. In no case start analysis more than 24 hours after grab sample collection. Keep composite samples at or below 4°C during composting. Limit composting period to 24 hours. Use the same criteria as for storage of grab samples, starting the measurement of holding time from the end of the composting period. State storage time and conditions as part of the results (APHA, AWWA and WPCF, 1985).

5:1 Methods

5:1.1 Preparation of BOD Dilution Water

1. Place a desired volume of purified water in a BOD dilution water container.
2. Place a BOD aerator in the BOD dilution water
3. Aerate the BOD dilution water overnight.
4. Add a BOD nutrient buffer pillow to the BOD dilution water.
5. Continue aeration of the BOD dilution water until sample dilution is ready to take place.

5:1.2 Preparation of 2% Glucose-Glutamic Acid Solution (Prepare immediately before use)

1. Dry D(+)Glucose and L-Glutamic acid at 103°C for 1 hour.
2. Add 0.150g of D(+)Glucose and 0.150g of L-Glutamic acid to a 1000mL volumetric flask containing purified water, mix thoroughly and make up to the mark with purified water.
3. Prepare a 1:50 (2%) dilution of the Glucose-Glutamic Acid solution using BOD dilution water.

5:1.3 BOD Determination

1. Prepare dilutions, where appropriate, of the sample(s) to be tested. Dilutions that result in a residual DO of at least 1mg/L and a DO uptake of at least 2mg/L after 5 days incubation provide the most reliable results (APHA, AWWA and WPCF, 1985).
2. Transfer the diluted or undiluted sample(s) to a corresponding glass stoppered BOD bottle(s).
3. Bring the diluted or undiluted sample(s) to 20°C.
4. Measure the DO₀ of the sample(s) in mg/L using a DO meter and electrode. The DO should be approximately 9.2mg/L at 20°C.
5. Add an appropriate quantity of nitrification inhibitor to the sample(s).
6. Stopper the BOD bottle.
7. Treat a BOD blank, containing BOD dilution water instead of sample in the same manner as the sample.
8. Treat the 2% Glucose-Glutamic Acid Solution in the same manner as the sample.
9. Incubate the sample(s), blank and Glucose-Glutamic Acid Solution at 20±1°C for 5 days.
10. Measure the DO₅ of the sample(s), blank and Glucose-Glutamic Acid solution and determine their BOD₅ according to the formula in section 6:0.

The DO uptake of the blank after 5 days should not be more than 0.2mg/L and preferably not more than 0.1mg/L. If the oxygen depletion exceeds 0.2mg/L obtain satisfactory water by improving purification or from another source balance (APHA, AWWA and WPCF, 1985).

The BOD₅ of the Glucose-Glutamic Acid solution should be 200±37mg/L. If it is outside this range reject any BOD determinations made with the BOD dilution water and seek the cause of the problem balance (APHA, AWWA and WPCF, 1985).

If more than one sample dilution meets the criteria of a residual DO of at least 1mg/L and a DO depletion of at least 2mg/L and there is no evidence of toxicity at higher sample concentrations or the existence of an obvious anomaly, average results in the acceptable range (APHA, AWWA and WPCF, 1985).

6:1 BOD Formulae

6:1.1 BOD formula when sample dilution is less than 1:10

$$\text{BOD}_5 = \left[\frac{(\text{DO}_0 - \text{DO}_5)_{\text{Sample}}}{X} - (\text{DO}_0 - \text{DO}_5)_{\text{Blank}} \right] \times X$$

X = Dilution factor

6:1.2 BOD formula when sample dilution is 1:10 or greater than 1:10

$$\text{BOD}_5 = \left[(\text{DO}_0 - \text{DO}_5)_{\text{Sample}} - (\text{DO}_0 - \text{DO}_5)_{\text{Blank}} \right] \times X$$

X = Dilution factor

6:1.3 BOD formula when sample is undiluted

$$\text{BOD}_5 = (\text{DO}_0 - \text{DO}_5)_{\text{Sample}}$$

7:1 Bibliography

APHA (American Public Health Association), AWWA (American Water Works Association) and WPCF (Water Pollution Control Federation) (1985). **Standard Methods for the Examination of Water and Wastewater. 16th Edition.** Prepared and Published Jointly By: APHA, AWWA and the WPCF. Joint Editorial Board: Greenberg, A.E. APHA Chairman; Trussell, R.R. AWWA and Clesceri, L.S. WPCF. Managing Editor: Franson, M.A.H. Publication Office: American Public Health Association, 1015 15th Street NW, Washington DC 20005, USA. ISBN: 0-87553-131-8, ISSN: 8755-3546.

APHA (American Public Health Association), AWWA (American Water Works Association) and WEF (Water Environment Federation) (1998). **Standard Methods for the Examination of Water and Wastewater. 20th Edition.** Prepared and Published Jointly By: APHA, AWWA and the WEF. Joint Editorial Board: Greenberg, A.E. APHA; Eaton, A.D. AWWA and Clesceri, L.S. WEF Chair. Managing Editor: Franson, M.A.H. Publication Office: American Public Health Association, 1015 15th Street NW, Washington DC 20005-2605, USA. ISBN: 0-87553-235-7, ISSN: 55-1979.

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Chemical Oxygen Demand

Reactor Digestion Method

(0-150mg/l and 0-1500mg/l)

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(Hach, 2002)

Protective Equipment

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6. Wear gloves to protect skin from toxic or corrosive materials, sharp objects, very hot or very cold materials or broken glass.
7. Wear laboratory coats to protect skin and clothing from splashes.
8. Fume hoods should be used when directed to do so by the procedure, or as recommended in the material safety data sheet (MSDS).

(Hach, 2002)

First Aid Equipment and Supplies

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5. Eyewash and shower stations should be provided within the laboratory.
6. Fume hoods and appropriate fire extinguishers should be provided within the laboratory. Appropriate training in fire extinguisher use should be obtained.

(Hach, 2002)

Appropriate protective equipment should be worn when undertaking the COD (Chemical Oxygen Demand) reactor digestion method (Jirka and Carter, 1975) (closed reflux-colourimetric method (APHA, AWWA and WEF, 1998)).

COD vials used in the COD reactor digestion method contain a range of ingredients including sulphuric acid and potassium dichromate. These vials may also contain mercury and silver solutions. An MSDS (Laboratory MSDS Number: C8. Appendix 1.1) should be consulted before attempting this method.

COD vials used in the COD reactor digestion method are heated to 150°C during the digestion stage of this method. During and, for a time, following this digestion stage of the COD reactor digestion method the COD reactors digestion block and the COD vials may be too hot to touch. The COD reactors digestion block and the COD vials must be allowed to cool to a 'hand-held' temperature before the COD vials are removed from the COD reactor digestion block.

2:1 Introduction

The COD reactor digestion method is suitable for determining the COD of water, wastewater and seawater (Jirka and Carter, 1975).

2:2 Principle

The COD is defined as the amount of a specified oxidant that reacts with a sample under controlled conditions. The quantity of oxidant consumed under controlled conditions is expressed in terms of its oxygen equivalence. Organic and inorganic components of a sample are subject to oxidation but in most cases the organic component predominates and is of the greater interest. COD is a defined test, in other words, the extent of sample oxidation can be affected by digestion time, reagent strength and sample COD concentration (APHA, AWWA and WEF, 1998).

COD is often used as a measurement of pollutants in wastewater and natural waters (APHA, AWWA and WEF, 1998). COD can be related empirically to biochemical oxygen demand (BOD), organic carbon or organic matter for samples from a specific source. The COD test is useful for monitoring and control after correlation has been established (APHA, AWWA and WPCF, 1985).

In the COD reactor digestion method a sample is digested for two hours, at 150°C, with potassium dichromate ($K_2Cr_2O_7$), which is a strong oxidising agent. Oxidisable organic compounds react with $K_2Cr_2O_7$, reducing the dichromate ion ($Cr_2O_7^{2-}$) to a green chromic ion (Cr^{3+}). When the reported 0 to 150mg/L COD method range is used, the amount of Cr^{6+} remaining is determined (Jirka and Carter, 1975).

The COD digestion solution contains mercuric sulphate (Hach Company, 2002)/mercury sulphate (Reagecon, 2003) and may contain silver sulphate (Hach Company, 2002). Mercury is used to complex interferences from chloride and silver acts a catalyst during the digestion step (Jirka and Carter, 1975).

2:3 Interferences

Chloride is the primary interference when determining the COD level of a sample. Chloride reacts with silver ions, present within the COD digestion solution, to precipitate silver chloride thus inhibiting the catalytic activity of silver. Bromide, iodide and any other reagent that inactivates these silver ions can lead to similar interferences. These interferences are negative in that they have the ability to restrict the oxidising action of the $Cr_2O_7^{2-}$. However under the rigorous digestion procedures for COD analysis, chloride, bromide or iodide can react with dichromate to produce the elemental form of the halogen and the chromic ion. Results are therefore in error on the high side. The difficulties caused by the presence of chloride can be overcome largely, though not completely, by complexing with mercuric sulphate ($HgSO_4$) before the refluxing procedure (APHA, AWWA and WEF, 1998).

Mercuric sulphate (Hach Company, 2002)/mercury sulphate (Reagecon, 2003) is present within the COD digestion solution. This complexing solution will eliminate interference up to 2000mg/L Cl^- (Reported Hach method range: 0 to 150mg/L COD). Samples having higher chloride concentrations must be diluted. Samples must be diluted enough to reduce the chloride concentration to 1000mg/L Cl^- (Reported Hach method range: 0 to 150mg/L COD). If dilution of samples results in the COD level being too low to be accurately determined, 0.50g of $HgSO_4$ should be added to the COD digestion solution before the sample is added. The addition of $HgSO_4$ will raise the maximum allowable chloride level to 8000mg/L Cl^- (Reported Hach method range: 0 to 150mg/L COD) (Jirka and Carter, 1975).

Ammonia and its derivatives, in waste or generated from nitrogen-containing organic matter, are not oxidised. However elemental chlorine reacts with these compounds. Hence, corrections for chloride interferences are difficult (APHA, AWWA and WEF, 1998).

Nitrite (NO_2^-) interference, when determining the COD level of a sample, is considered insignificant and is usually ignored because concentrations in waters rarely exceed 1 or 2mg NO_2^-/L (APHA, AWWA and WEF, 1998).

Reduced inorganic species such as ferrous iron, sulphide, manganous, manganese, etc. are oxidised quantitatively under test conditions. For samples containing significant levels of these species, stoichiometric oxidation can be assumed from known initial concentration of the interfering species and corrections can be made to the COD value obtained (APHA, AWWA and WEF, 1998).

3:1 Materials

26. COD vials containing COD digestion solution (Reagecon Article/Product Number: 420720 (Laboratory MSDS Number: C8) or equivalent)
27. Analytical Quality Control (AQC) Standard 600mg/L (Reagecon Article/Product Number: COD600 (Laboratory MSDS Number: C3) or equivalent)
28. Analytical Quality Control (AQC) Standard 50mg/L (Reagecon Article/Product Number: COD50 or equivalent)
29. Purified water or equivalent
30. COD reactor (Hach Model Number: 45600-02. Serial Number: 960200003913 (Laboratory Identification Code: CODR-1) or equivalent)
31. COD reactor thermometer (Hach Part Number: PT5OR or equivalent)
32. COD reactor protective shield
33. Spectrophotometer (Hach Model Number: DR/4000V)
34. COD vial adapter and cover
35. Beakers
36. Volumetric flasks
37. Pipettors (Dr Lange Model Number: BBP 065. Volume Ranges: 1 to 5mLs. Serial Number's: N58118 and N58123 or equivalent) and tips (Dr Lange Pipette Code: BBP 068 or equivalent)
38. Rack.
39. Gloves.
40. Fume hood.

4:1 Preservation Method

1. If a delay before analysis of sample(s) is unavoidable the sample(s) should be preserved by acidifying them to a pH of less than or equal to (\leq) 2 using concentrated sulphuric acid (H_2SO_4) (APHA, AWWA and WEF, 1998) or equivalent and analysis should be undertaken within 28 days.
2. Acidified sample(s) should be returned to their original pH before analysis begins (Smith, 2003).

5:1 Methods

5:1.1 Pre-Digestion Treatment of Samples, Reagent Blank and Standards

The COD vials used in the analysis of the sample(s) should have the same lot number as the COD vials used in the analysis of the COD reagent blank, and COD standard.

1. Add 2mLs of homogenised or unhomogenised sample, diluted if necessary, to a labelled COD vial. Add 2mls of purified water and 2mls of pre-prepared Standard (600mg/l for range 0-1500 and 50mg/l for range 0-500) to labelled COD vials. This procedure should be undertaken within a fume hood.
2. Invert the labelled COD vials several times to mix contents.
3. Place the labelled COD vials in a rack until ready for digestion.

5:1.2 Digestion of Samples, Reagent Blank and Standards

1. Preheat the COD reactor to 150°C.
2. Place the COD vials in the metal block of the COD reactor.
3. Heat the COD vials in the COD reactor for 2 hours.
4. After two hours remove the vials from the reactor and place in a rack until cooled.

5:1.3 Spectrophotometric Determination of Samples and Standards

1. Select stored program for required COD range (Prog. No. 2710 for COD LR 150.0mg/l or Prog. No. 2720 for COD HR 1500mg/l).
2. Open the cell compartment holder on the spectrophotometer and place a COD vial adaptor into the holder. Ensure that the wavelength window is in line with the light source.
3. Wipe clean the COD reagent blank vial and place in the adaptor. Ensure that the text/logo on the vial is facing outwards.
4. Close the lid and press ZERO to blank the spectrophotometer.
5. Wipe clean the wall of the COD sample vial and place in the adaptor.
6. Close the lid and press READ.
7. Record the COD sample result.
8. Remove the vial from the adaptor.
9. Repeat steps 5 to 8 for all remaining samples and the appropriate standard.
10. Steps 3 and 4 must be carried out with the appropriate Reagent Blank for each COD range before samples and standard can be read.
11. Samples that are found to be 'over range' should be diluted and analysed at a higher range.

8:1 Bibliography

APHA (American Public Health Association), AWWA (American Water Works Association) and WPCF (Water Pollution Control Federation) (1985). **Standard Methods for the Examination of Water and Wastewater. 16th Edition.** Prepared and Published Jointly By: APHA, AWWA and the WPCF. Joint Editorial Board: Greenberg, A.E. APHA Chairman; Trussell, R.R. AWWA and Clesceri, L.S. WPCF. Managing Editor: Franson, M.A.H. Publication Office: American Public Health Association, 1015 15th Street NW, Washington DC 20005, USA. ISBN: 0-87553-131-8, ISSN: 8755-3546.

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Jirka, A.M. and Carter, M.J. (1975). **Analytical Chemistry.** 1975, 47(8), 1397. In: *Hach DR/2000 Spectrophotometer Handbook.*

Reagecon (2003). **Material Safety Data Sheet** (According to 91/155 EC, Version Number: 5). Trade Name: COD Standard 600mg/L. Article Number: COD600. Pages 1 to 4. Reviewed On: 16/05/2003, Printing Date: 11/07/2003. Laboratory MSDS Number: C3.

Reagecon (2003). **Material Safety Data Sheet** (According to 91/155 EC, Version Number: 28). Trade Name: COD-Cuvettes Low Range 0 to 150mg/L COD. Article Number: 420720. Pages 1 to 7. Reviewed On: 03/07/2003, Printing Date: 03/07/2003. Laboratory MSDS Number: C8.

VWR International (2001). **Material Safety Data Sheet** Product Name: Potassium hydrogen phthalate AnalaR. Catalogue Number: 10207. Pages 1 to 4. Laboratory MSDS Number: P7. VWR International Address: VWR International Ltd., Merck House, Poole, Dorset, BH15 1TD, England. Telephone Number: +44(0)1202669700. Telefax Number: +44(0)1202665599.

VWR International (2002). **Material Safety Data Sheet** Product Name: Sulphuric Acid sp.gr. 1.84, Analar®. Catalogue Number: 10276. Pages 1 to 5. Laboratory MSDS Number: S4. VWR International Address: VWR International Ltd., Merck House, Poole, Dorset, BH15 1TD, England. Telephone Number: +44(0)1202669700. Telefax Number: +44(0)1202665599.

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Total Suspended Solids

Gravimetric Method

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1:1 Laboratory Safety

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14. Never pipette by mouth within the laboratory.
15. Wipe up spills promptly within the laboratory. Appropriate training in spill management should be obtained.

(Hach, 2002)

Protective Equipment

9. Wear safety glasses to protect eyes from flying objects or chemical splashes.
10. Wear gloves to protect skin from toxic or corrosive materials, sharp objects, very hot or very cold materials or broken glass.
11. Wear laboratory coats to protect skin and clothing from splashes.
12. Fume hoods should be used when directed to do so by the procedure, or as recommended in the material safety data sheet (MSDS).

(Hach, 2002)

First Aid Equipment and Supplies

7. First aid kits should be provided within the laboratory. Appropriate training in first aid should be obtained.
8. Eyewash and shower stations should be provided within the laboratory.
9. Fume hoods and appropriate fire extinguishers should be provided within the laboratory. Appropriate training in fire extinguisher use should be obtained.

(Hach, 2002)

Appropriate protective equipment should be worn when undertaking the total suspended solids gravimetric method.

2:1 Introduction

Solids refer to matter suspended or dissolved in water or wastewater and may affect water or effluent quality in many ways e.g. waters high in suspended solids may be aesthetically unsatisfactory for such purposes as bathing. Solids analyses are important in the control of biological and physical wastewater treatment processes and for assessing compliance with regulatory agency wastewater effluent limitations (APHA, AWWA and WPCF, 1985).

The total suspended solids gravimetric method is suitable for determining the level of suspended solids in potable water, surface water, saline water, domestic wastewater and industrial wastewater (APHA, AWWA and WPCF, 1985).

2:2 Principle

The total suspended solids gravimetric method involves filtering a well-mixed sample through a weighed standard glass fibre filter and the solids retained on the filter is dried at 103 to 105°C. The increase in weight of the filter represents the total suspended solids. If the suspended material clogs the filter and prolongs filtration, the difference between the total solids and total dissolved solids may provide an estimate of the total suspended solids (APHA, AWWA and WPCF, 1985).

2:3 Interferences

1. Exclude large floating particles or submerged agglomerates of nonhomogeneous materials from the sample if it is determined that their inclusion is not desired in the final result (APHA, AWWA and WPCF, 1985).
2. Excessive residue on the filter may form a water-entrapping crust; therefore limit sample size to that yielding no more than 200mg of residue (APHA, AWWA and WPCF, 1985).
3. For samples high in dissolved solids thoroughly wash the filter to ensure removal of dissolved material (APHA, AWWA and WPCF, 1985).
4. Prolonged filtration times resulting from filter clogging may produce high results owing to excessive solids capture on the clogged filter (APHA, AWWA and WPCF, 1985).

3:1 Materials

41. Whatman GF/C 70mm glass microfibre filters (Catalogue No. 1822 070 or equivalent)
42. Funnel and Receiving Flask
43. Vacuum system
44. Merck microcrystalline cellulose (Product No. 1.02330.0500 or equivalent)
45. Purified water or equivalent
46. Balance (4 decimal places)
47. Calibration check weight (1.0000g)
48. Weighing boat
49. Dessicator
50. Forceps
51. Spatula
52. Magnetic stirrer
53. Oven (103 to 105°C)
54. Timer
55. Volumetric flask (1000mL)
56. Graduated cylinders (10 to 1000mL volume)
57. Pasteur pipettes
58. Gloves

4:1 Preservation Method

Use resistant-glass or plastic bottles, provided that the material in suspension does not adhere to the container walls. Begin analysis as soon as possible because of the impracticality of preserving the sample. Refrigerate samples at 4°C up to analysis to minimise microbiological decomposition of solids (APHA, AWWA and WPCF, 1985).

5:1 Methods

5:1.1 Pretreatment of Glass Microfibre Filters

6. Filter 3 x 20mL aliquots of purified water through each filter.
7. Dry the filters in an oven between 103 and 105°C for at least 1 hour.
8. Allow the pretreated filters to cool in a dessicator before use.

5:1.2 Preparation of 100mg/L Total Suspended Solids Standard

4. Add 0.100g of microcrystalline cellulose to a 1000mL volumetric flask containing purified water, mix thoroughly, make up to the mark with purified water and mix thoroughly again.

5:1.3 Total Suspended Solids Determination

11. Filter a desired volume of sample through a pretreated, preweighed glass microfibre filter paper. Ensure that all solids within this sample volume are collected on the filter paper.
12. Dry the filter paper and solids in an oven set between 103 and 105°C for at least 1 hour.
13. Cool the filter paper and solids in a desiccator.
14. Reweigh the filter paper and calculate the level of total suspended solids within the sample according to the formula in section 6:0.
15. Determine the level of total suspended solids in the 100mg/L total suspended solids standard in the same manner as for the sample.

6:1 Total Suspended Solids Formula

$$\text{Total Suspended Solids} = \frac{A - B}{C} \times 1000$$

A = Weight of filter and solids

B = Weight of filter

C = Volume of sample filtered

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7:1 Bibliography

APHA (American Public Health Association), AWWA (American Water Works Association) and WPCF (Water Pollution Control Federation) (1985). **Standard Methods for the Examination of Water and Wastewater. 16th Edition.** Prepared and Published Jointly By: APHA, AWWA and the WPCF. Joint Editorial Board: Greenberg, A.E. APHA Chairman; Trussell, R.R. AWWA and Clesceri, L.S. WPCF. Managing Editor: Franson, M.A.H. Publication Office: American Public Health Association, 1015 15th Street NW, Washington DC 20005, USA. ISBN: 0-87553-131-8, ISSN: 8755-3546.

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pH Method

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1:1 Laboratory Safety

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20. Wipe up spills promptly within the laboratory. Appropriate training in spill management should be obtained.

(Hach, 2002)

Protective Equipment

13. Wear safety glasses to protect eyes from flying objects or chemical splashes.
14. Wear gloves to protect skin from toxic or corrosive materials, sharp objects, very hot or very cold materials or broken glass.
15. Wear laboratory coats to protect skin and clothing from splashes.
16. Fume hoods should be used when directed to do so by the procedure, or as recommended in the material safety data sheet (MSDS).

(Hach, 2002)

First Aid Equipment and Supplies

10. First aid kits should be provided within the laboratory. Appropriate training in first aid should be obtained.
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12. Fume hoods and appropriate fire extinguishers should be provided within the laboratory. Appropriate training in fire extinguisher use should be obtained.

(Hach, 2002)

Appropriate protective equipment should be worn when undertaking the total suspended solids gravimetric method.

2:1 Introduction

The method is suitable for the determination of pH in water and wastewater.

2:2 Principle

pH is the measure of the acidity or alkalinity of a solution and is defined as the negative logarithm (base 10) of the H^+ ion concentration. The measurement of the pH of a sample can be done by measuring the cell potential of the sample. A pH meter, comprising of a glass electrode (a silver wire coated with silver chloride immersed in dilute hydrochloric acid) is used for analysis. The electrode solution is separated from the sample by a thin glass membrane. The potential which develops across that glass membrane can be shown to be proportional to the hydrogen ion concentrations on the two surfaces. In the measurement instrument, a cell is made with the other electrode (commonly a mercury-mercury chloride electrode). The cell potential is then linearly proportional to the pH and the meter can then be calibrated to read directly in pH.

3:1 Materials

1. pH technical buffer solutions with the following values:
 - pH 4
 - pH 7
 - pH 5
 - pH 8
2. WTW pH meter
3. Electrode Filling Solution 3 M KCl
4. Glass beakers
5. Magnetic stirrer
6. Stirring bars

4:1 Preservation Method

Use resistant-glass or plastic bottles. Analysis should be done as soon as possible after the sample is taken and no more than 24hrs after.

5:1 Methods

5:1.1 pH meter calibration

9. The pH meter should be calibrated before use, using pH 4 and 7 to calibrate and pH 5 and 8 as a check.
10. Pour 50 mls of each pH technical buffer solution into labelled glass beakers and add a stirring bar to each. Calibration buffers and samples are placed on a magnetic stirrer for reading pH.
11. Turn meter on and press the CAL button, CT1 will appear on the screen.
12. Remove the electrode from the storage solution and rinse with de-ionised water. Uncover the fill hole by sliding the cover up.
13. Place the probe in pH buffer 4 (ensuring the sensor near the base is completely covered) and press RUN/ENTER, leave to stabilise and record reading. CT2 will appear on the screen.
14. Rinse the probe and place in pH buffer 7 (ensuring the sensor near the base is completely covered), press RUN/ENTER. Leave to stabilise and record reading.
15. Record slope and asymmetry values (Slope should be between -58.3 to -60.5mV. Asymmetry value is between -20 to 20 mV).
16. Press M to display the pH screen. Place the probe in the pH buffer 5 (ensuring the sensor near the base is completely covered) and leave to stabilise, record reading.
17. Place the probe in pH buffer 8 (ensuring the sensor near the base is completely covered) and leave to stabilise, record reading.
18. Calibration is complete and samples can now be read.

5:1.2 Reading samples

1. Place 50mls of sample in a labelled glass beaker and add a stirring bar.
2. Place the beaker on a magnetic stirrer and place probe in the beaker (ensuring the sensor near the base is completely covered).
3. Allow to stabilise and record reading.
4. Repeat steps 1 to 3 for all remaining samples, rinsing the probe with de-ionised water between samples.
5. Rinse probe with de-ionised water, replace the storage solution and cover the fill hole by sliding the cover down.

6:1 Bibliography

APHA (American Public Health Association), AWWA (American Water Works Association) and WPCF (Water Pollution Control Federation) (1985). **Standard Methods for the Examination of Water and Wastewater. 16th Edition.** Prepared and Published Jointly By: APHA, AWWA and the WPCF. Joint Editorial Board: Greenberg, A.E. APHA Chairman; Trussell, R.R. AWWA and Clesceri, L.S. WPCF. Managing Editor: Franson, M.A.H. Publication Office: American Public Health Association, 1015 15th Street NW, Washington DC 20005, USA. ISBN: 0-87553-131-8, ISSN: 8755-3546.

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Mixed Liquor Suspended Solids

Gravimetric Method

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First Aid Equipment and Supplies

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Appropriate protective equipment should be worn when undertaking the total suspended solids gravimetric method.

2:1 Introduction

Mixed Liquor Suspended Solids (MLSS) refers to the suspended solids content of the mixed liquor contained within the aeration tank.

2:2 Principle

MLSS determination is done using the same method as is used for determining total suspended solids. The method involves filtering a well-mixed sample through a weighed standard glass fibre filter and the solids retained on the filter is dried at 103 to 105°C. The increase in weight of the filter represents the total suspended solids. If the suspended material clogs the filter and prolongs filtration, the difference between the total solids and total dissolved solids may provide an estimate of the total suspended solids (APHA, AWWA and WPCF, 1985).

3:1 Materials

59. Whatman GF/C 70mm glass microfibre filters (Catalogue No. 1822 070 or equivalent)
60. Funnel and Receiving Flask
61. Vacuum system
62. Merck microcrystalline cellulose (Product No. 1.02330.0500 or equivalent)
63. Purified water or equivalent
64. Balance (4 decimal places)
65. Calibration check weight (1.0000g)
66. Weighing boat
67. Dessicator
68. Forceps
69. Spatula
70. Magnetic stirrer
71. Oven (103 to 105°C)
72. Timer
73. Volumetric flask (1000mL)
74. Graduated cylinders (10 to 1000mL volume)
75. Pasteur pipettes
76. Gloves

4:1 Preservation Method

Use resistant-glass or plastic bottles, provided that the material in suspension does not adhere to the container walls. Begin analysis as soon as possible because of the impracticality of preserving the sample. Refrigerate samples at 4°C up to analysis to minimise microbiological decomposition of solids (APHA, AWWA and WPCF, 1985).

5:1 Methods

5:1.1 Pretreatment of Glass Microfibre Filters

19. Filter 3 x 20mL aliquots of purified water through each filter.
20. Dry the filters in an oven between 103 and 105°C for at least 1 hour.
21. Allow the pretreated filters to cool in a dessicator before use.

5:1.2 Preparation of 100mg/L Standard

5. Add 0.100g of microcrystalline cellulose to a 1000mL volumetric flask containing purified water, mix thoroughly, make up to the mark with purified water and mix thoroughly again.

5:1.3 Mixed Liquor Suspended Solids Determination

16. Filter a desired volume of sample through a pretreated, preweighed glass microfibre filter paper. Ensure that all solids within this sample volume are collected on the filter paper.
17. Dry the filter paper and solids in an oven set between 103 and 105°C for at least 1 hour.
18. Cool the filter paper and solids in a dessicator.
19. Reweigh the filter paper and calculate the level of mixed liquor suspended solids within the sample according to the formula in section 6:0.
20. Determine the level of mixed liquor suspended solids in the 100mg/L standard in the same manner as for the sample.

6:1 Total Suspended Solids Formula

Mixed Liquor Suspended Solids = $A - B \times 1000 \times 1000$

C

A = Weight of filter and solids

B = Weight of filter

C = Volume of sample filtered

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Settled Sludge Volume (Cone Test)

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Appropriate protective equipment should be worn when undertaking the total suspended solids gravimetric method.

2:1 Introduction

Settled Sludge Volume (SSV) determination or cone test is a standard test carried out on wastewater and is used to assess the settleability of the mixed liquor in the clarifier. It is used, along with the Mixed Liquor Suspended Solids to calculate the Sludge Volume Index (SVI).

2:2 Principle

The test is carried out using an Imhoff cone which is graded from 0mls to 1000mls. The Imhoff cone is filled to the 1000mls mark with a sample of mixed liquor from the aeration basin. This is left to settle for 30-minutes and the volume of clear supernatant left at the top is recorded. This volume is the SSV.

3:1 Materials

1. Imhoff cone
2. Timer

4:1 Preservation Method

Use resistant-glass or plastic bottles.

5:1 Method

22. Fill an Imhoff cone to the 1000ml mark with a well mixed sample of mixed liquor.
23. Allow to settle for 30-minutes.
24. Record the volume of supernatant in mls/l. This is the settled sludge volume

6:1 Calculation of Sludge Volume Index (SVI)

Sludge Volume Index can be calculated using the following Formula:

$$SVI = \frac{SSV \text{ (mls/l)} \times 1000}{MLSS}$$

7:1 Bibliography

APHA (American Public Health Association), AWWA (American Water Works Association) and WPCF (Water Pollution Control Federation) (1985). **Standard Methods for the Examination of Water and Wastewater. 16th Edition.** Prepared and Published Jointly By: APHA, AWWA and the WPCF. Joint Editorial Board: Greenberg, A.E. APHA Chairman; Trussell, R.R. AWWA and Clesceri, L.S. WPCF. Managing Editor: Franson, M.A.H. Publication Office: American Public Health Association, 1015 15th Street NW, Washington DC 20005, USA. ISBN: 0-87553-131-8, ISSN: 8755-3546.

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Leitrim County Council – Sanitary and Water Services

MAINTENANCE AND CALIBRATION OF DO PROBES

Membrane Replacement

The following procedure describes membrane replacement

1. Remove the oxygen probe from the water and clean with a cloth or paper towel.
2. Unscrew the bottom end cap and discard the electrolyte solution.
3. Using a coin, unscrew the threaded retaining ring in the membrane cap, remove and discard the membrane and small `o` ring located below the membrane
4. Clean the inside of the membrane cap thoroughly and dry with a clean cloth.
5. Insert a new `o` ring, and then the membrane on top of the `o` ring. Screw down the threaded retaining ring until you feel tension, then give a further 1/4 turn. If the membrane wrinkles, you will need to replace the `o` ring and membrane and try again.
6. Clean the inside of the probe top. (You may clean the silver cathode with 1500 grade paper, take care as damage to the silver cathode can affect probe readings. You can clean the angular zinc anode with rough abrasive paper in order to remove any oxidation layer. Clean the probe in freshwater to remove any of the fines.)
7. Fill the membrane cap with electrolyte, and holding the probe vertically, screw the membrane cap on to the top of the probe, making sure that the large `o` ring is in place. Do not screw the cap on too quickly as this will stretch the membrane. Screw up the cap until it seals on the large `o` ring a then give it a further 1/4 turn.
8. Immerse the probe into the water, after 24 hours perform a calibration as per the instructions below.

Calibration

1. Remove probe from water, dry and clean the probe and membrane with a soft clean cloth.
2. Using a pin, clear the breather hole on top of the probe.
3. Suspended the probe in air above the surface of the water, try to keep the probe out of the wind and direct sunlight. The probe should be left for a period of at least 30 minutes, or until you are sure that the temperature of the probe is the same as the air temperature. (The best time to perform this task is early in the morning or late evening.)
4. The probes will now be reading the equivalent of 100% saturation. Take an accurate reading of the air temperature around the probes. Use the table below to establish the concentration in mg/l for the probe.

Probe Maintenance

The probe should be removed from the water once a week and the membrane cleaned with a soft clean cloth. The breather hole on top should be cleaned using a pin. If the readings become very erratic or stay very constant the membrane should be replaced.

Table for calibration of DO probe

If the DO probe is calibrated correctly it should read the same as the values in the table below, (i.e. The values not in bold) at the appropriate temperature.

T deg C	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
0	14.60	14.65	14.52	14.48	14.44	14.40	14.36	14.33	14.29	14.25
1	14.21	14.17	14.13	14.09	14.05	14.02	13.98	13.94	13.90	13.87
2	13.83	13.79	13.75	13.72	13.68	13.64	13.61	13.57	13.54	13.50
3	13.46	13.43	13.39	13.36	13.32	13.29	13.25	13.22	13.18	13.15
4	13.11	13.08	13.04	13.01	12.98	12.94	12.91	12.88	12.84	12.81
5	12.78	12.74	12.71	12.68	12.64	12.61	12.58	12.55	12.52	12.48
6	12.45	12.45	12.39	12.36	12.33	12.29	12.26	12.23	12.20	12.17
7	12.14	12.11	12.08	12.05	12.02	11.99	11.96	11.93	11.90	11.87
8	11.84	11.81	11.78	11.76	11.73	11.70	11.67	11.64	11.61	11.58
9	11.56	11.53	11.50	11.47	11.44	11.42	11.39	11.36	11.34	11.31
10	11.28	11.25	11.23	11.20	11.17	11.15	11.12	11.10	11.07	11.04
11	11.02	10.99	10.97	10.94	10.91	10.89	10.86	10.84	10.81	10.79
12	10.76	10.74	10.72	10.69	10.67	10.64	10.62	10.59	10.57	10.55
13	10.54	10.50	10.47	10.45	10.43	10.40	10.38	10.36	10.34	10.31
14	10.29	10.27	10.24	10.22	10.20	10.18	10.15	10.13	10.11	10.09
15	10.07	10.04	10.02	10.00	9.98	9.96	9.94	9.92	9.89	9.87
16	9.85	9.83	9.81	9.79	9.77	9.75	9.73	9.71	9.69	9.67
17	9.65	9.63	9.61	9.59	9.57	9.55	9.53	9.51	9.49	9.47
18	9.45	9.43	9.41	9.39	9.37	9.36	9.34	9.32	9.30	9.28
19	9.26	9.24	9.23	9.21	9.19	9.17	9.15	9.13	9.12	9.10
20	9.08	9.06	9.05	9.03	9.01	8.99	8.89	8.96	8.94	8.92
21	8.91	8.89	8.87	8.86	8.84	8.82	8.81	8.79	8.77	8.76
22	8.74	8.72	8.71	8.69	8.67	8.66	8.64	8.63	8.61	8.59
23	8.58	8.56	8.55	8.53	8.51	8.50	8.48	8.47	8.45	8.44
24	8.42	8.41	8.39	8.38	8.36	8.35	8.33	8.32	8.30	8.29
25	8.27	8.26	8.24	8.23	8.21	8.20	8.18	8.17	8.16	8.14
26	8.13	8.11	8.10	8.08	8.07	8.06	8.04	8.03	8.01	8.00
27	7.99	7.97	7.96	7.94	7.93	7.92	7.90	7.89	7.88	7.86
28	7.85	7.84	7.82	7.81	7.80	7.78	7.77	7.76	7.74	7.73
29	7.72	7.70	7.69	7.68	7.66	7.65	7.64	7.63	7.61	7.60
30	7.59	7.57	7.56	7.55	7.54	7.52	7.51	7.50	7.49	7.47