

NANYANG TECHNOLOGICAL UNIVERSITY

School of Civil and Environmental Engineering

CV2701: Laboratory 2A

Laboratory Manual

For

Experiment Lab 2A-5 (ENV)

Water Quality Analysis

Location: Environment Laboratory II, N1-B3c-49

Session 2004/2005

WATER QUALITY ANALYSIS

TURBIDITY, COLOUR, pH, AND ALKALINITY

1. OBJECTIVES

- (i) To perform turbidity and colour tests on a given set of water samples and to examine their progressive change as the water flows from one treatment unit to another in a waterworks;
- (ii) To perform pH and alkalinity analyses on a given set of water samples and to examine the results with respect to the water treatment processes.

2. THEORY

2.1 Turbidity

Turbidity is caused by suspended materials which absorb and scatter light. These colloidal and finely dispersed turbidity-causing materials do not settle under quiescent conditions and are difficult to remove by sedimentation. Turbidity is a key parameter in water supply engineering, because turbidity will both cause water to be aesthetically unpleasant and cause problems in water treatment processes, such as filtration and disinfection. Turbidity is also often used as indicative evidence of the possibility of bacteria being present.

Turbidity measurements performed using proprietary nephelometric instruments are expressed as Nephelometric Turbidity Units (NTU). The nephelometric apparatus is designed to measure forward scattering of light at 90° to the path of an incandescent light beam. Suspended particles present in a water sample reflect a portion of the incident light off the particle surface. The light reflected at 90° is measured by a photoelectric detector and is compared against light reflected by a reference standard. No interference exists for the turbidity test. Locally, the Public Utilities Board (PUB) of Singapore requires all water treatment facilities to produce water containing less than 1 NTU.

2.2 Colour

Many surface waters are coloured, due primarily to decomposition of organics, metallic salts or coloured clays. This colour is considered as "apparent colour" as it is seen in the presence of suspended matter, whereas "true colour" is derived only from dissolved inorganic and organic matters. Samples can be centrifuged and/or filtered to remove turbidity in order to measure true colour.

Waters which obtain their colour from natural organic matter usually pose no health hazard. However, because of the yellowish brown appearance of such waters, the consumers may not find the water aesthetically acceptable. Consumers of highly coloured but already properly treated water may not believe the water is in fact properly

treated. Many processing industries require low coloured water. PUB requires drinking water to meet the "highest desirable" World Health Organisation (WHO) standards of less than 5 colour units.

One standard colour unit is defined as a 1 mg/L concentration of platinum in the form of potassium chloroplatinate K_2PtCl_6 . Measurement of colour is by comparison of the sample with standard colour solutions using a spectrophotometer. A straight line calibration curve is initially developed by plotting absorbance versus platinum-cobalt colour standard. In practice, the absorbance of a sample is determined and corresponding concentration is read off the calibrated curve.

When measuring true colour, pre-treatment has to be carried out to remove turbidity. Unfortunately, either centrifugation or filtration has some effect on true colour. So when reporting the true colour value, specify the details of the pre-treatment method and its operating conditions. Likewise, the colour value of water is extremely pH dependent, too, and invariably increases as the pH of the water is raised. When reporting a colour value, specify the pH at which colour is determined.

2.3 pH and Alkalinity

pH is a way of expressing the hydrogen-ion concentration of a solution. As acids and bases in solution dissociate to yield hydrogen ions $[H^+]$ and hydroxyl ions $[OH^-]$ respectively, pH is used to indicate the intensity of the acidic or alkaline condition of a solution.

Alkalinity is a measure of the acid-neutralizing capacity of dissolved substances in water and equals the amount of strong acid required to lower the solution from initial pH to about 4.5. Many materials may contribute to the alkalinity of water. For most practical purposes, it is due primarily to presence of salts of weak acids (mainly bicarbonate and carbonate) and hydroxide (at high pH).

pH and alkalinity are key water quality parameters in environmental engineering practice. In the water supply and treatment fields, these parameters have great influence on the chemical coagulation, disinfection and softening processes, and corrosion control for water distribution pipe networks. Effective chemical coagulation of water, for instance, occurs only within a specific pH range. Chemicals used for coagulation release, as a by-product of their reactions with water to form insoluble hydroxide precipitates, hydrogen ions (acid-causing). If unchecked, these hydrogen ions could lower the pH of the water sufficiently to render the coagulants ineffective. The presence of sufficient amount of alkalinity in the water can react and remove the hydrogen ions released by the coagulants, thus buffering the water in the pH range where the coagulant can be effective.

In pure water, water molecules dissociate into equal amounts of hydrogen and hydroxyl ions (10^{-7} moles/L). From the law of mass action, it can be shown that, for pure water at about 25°C:

$$[H^+] \times [OH^-] = K_w = 10^{-14} \quad (1)$$

The pH value of a solution has been defined to be the negative log of the hydrogen ion concentration:

$$\text{pH} = -\log[\text{H}^+] \quad (2)$$

The pH scale runs from 0 to 14, with pH 7 representing neutrality. Acid conditions increase as pH values decrease, and alkaline (base) conditions increase as the pH values increase. Measurement of the hydrogen ion concentration is made by pH meters via a glass electrode and a calomel reference electrode.

The alkalinity of water is its quantitative capacity to neutralize acids. The three major forms of alkalinity ranked in order of their association with high pH values are

- (1) hydroxide alkalinity, $[\text{OH}^-]$,
- (2) carbonate alkalinity, $[\text{CO}_3^{2-}]$, and
- (3) bicarbonate alkalinity $[\text{HCO}_3^-]$.

Their ability to react with H^+ ends at pH 4.5 when both have turned into carbonic acid (H_2CO_3). In nature, bicarbonates are the major form of alkalinity because they result from the reactions of CO_2 on calcium and magnesium rocks. Some CaCO_3 (up to about 20 mg/L) may also go into solution as Ca^{2+} and CO_3^{2-} ions. For all practical purposes, alkalinity due to other sources in natural waters may be ignored.

Alkalinity of waters is measured by means of titration with a standard solution of a strong acid (usually H_2SO_4) to designated pHs, and is reported in terms of equivalent CaCO_3 . Alkalinity depends on the end-point pH or indicator used. Either titration curve technique or colour indicators can be used for the determination. The alkalinity measurement is based on the titration curve for a hydroxide-carbonate-bicarbonate mixture, as shown in Fig. 1.

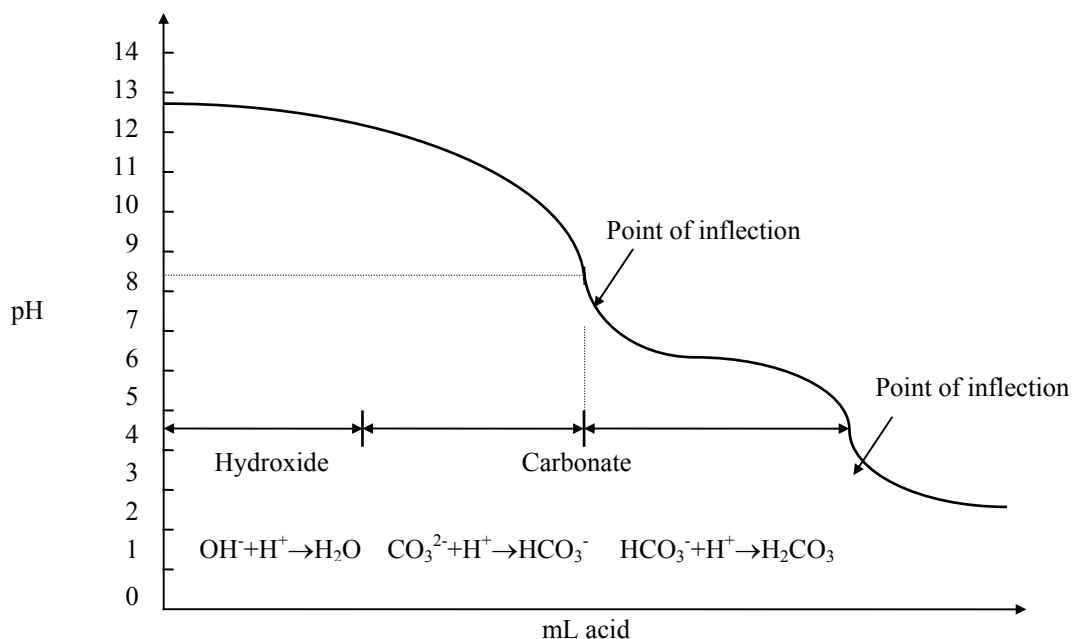


Fig. 1 Titration curve for a hydroxide-carbonate-bicarbonate mixture.

For samples whose initial pH is above 8.3, the titration is made in two steps. In the first step, the titration is conducted until the phenolphthalein indicator end-point is reached (i.e. pH of about 8.3) with a colour change from pink to colourless. During this first phase, the acid added to the sample reacts with $[\text{OH}^-]$ alkalinity, if present, and $[\text{CO}_3^{2-}]$ alkalinity as follows:



In the second phase, the titration is continued until the methyl orange indicator end-point is reached with a colour change from yellow to red (i.e. pH of about 4.5). During this phase, the addition of acid changes the HCO_3^- ions, initially present as well as those produced by Reaction (1-4), into carbonic acid;



In the above titration, the result of first step is known as "phenolphthalein alkalinity" and the overall titration is known as "total alkalinity" (the amount of acid required to react with all the hydroxide, carbonates and bicarbonates in the sample) respectively.

When the pH of a sample is less than 8.3, a single titration is made to the methyl orange end point. Also for routine work, it is common that only the total alkalinity is determined. It is possible to determine the various components of alkalinity (i.e. hydroxide, carbonate and bicarbonate fractions) from a combination of titration, pH measurements and chemical equilibrium equations.

An understanding of the buffering capacity of alkalinity can be derived from an evaluation of Figure 1. At the inflection points of pH 8.3 (phenolphthalein alkalinity) and pH 4.5 (total alkalinity), the carbonate system will react with a considerable pH change when only a small fraction of titrant is added. However, at the points where only half of the initial carbonate has been converted to bicarbonate and only half of the resultant bicarbonate has been converted to carbonic acid, considerably more titrant is required to effect a pH change. It is during these conditions that the buffering capacity is exhibited. Quantifying the alkalinity to the inflection points is a measure of this buffering capacity.

3. EQUIPMENT

3.1 Turbidity by HACH Turbidimeter

- (i) Select the operating range at "AUTO" mode.
- (ii) Fill a clean sample cell to the mark with the test sample and place it in the cell holder. The sample cell must be clean, dry and free of fingerprints. Wipe the outside of the cell with a lens tissue and align the dot on the sample cell with the raised mark on the spill ring around the cell holder opening. Be sure the cell is kept

down completely and held in place by the spring clip. Cover the sample with the light shield.

- (iii) The digital readout is in Nephelometric Turbidity Units (NTU).

3.2 Colour by Spectrophotometer

- (i) Place the cell containing the blank (distilled water) in the sample compartment with the transparent sides facing the light source. Close the sample compartment lid. (Note: Do not touch the transparent sides of the sample cell and keep it clean).
- (ii) Press "AUTOZERO" key to set the zero absorbance.
- (iii) Discard the distilled water and place the cell containing the sample in the measuring position. Close the sample compartment lid.
- (iv) Press "START" key to measure colour.
- (v) Record the "Conc" as colour units for the sample
- (vi) Report the results as follows:

<u>Colour Units</u>	<u>Record to nearest</u>
1 ~ 50	1
51 ~ 100	5
101 ~ 250	10
251 ~ 500	20

3.3 pH

- (i) Calibrate the pH meter according to instructions supplied by the Lab staff.
- (ii) Pour sample into a clean beaker.
- (iii) Rinse the probe thoroughly with distilled water to prevent any carry-over. Switch to pH mode.
- (iv) Immerse the probe in the sample.
- (v) Establish equilibrium between probe and sample by stirring to insure homogeneity. Gently drop a stirring bar into the sample and place the beaker on a magnetic stirrer. Start the magnetic stirrer and adjust the speed to give thorough but gentle mixing.
- (vi) Read and record the pH.
- (vii) Rinse the electrode thoroughly with distilled water.
- (viii) When not in use, the electrode should be replaced in the beaker containing water.

3.4 Alkalinity (Total Alkalinity)

- (i) For each sample, place 100 mL of sample in an Erlenmeyer flask.
- (ii) Add 3 drops of methyl orange indicator solution to the flask.
- (iii) Titrate sample with 0.02 N H₂SO₄ (sulphuric acid), constantly swirling the flask content above a white surface until just after the colour of the flask content change from yellow to red.
- (iv) Record the volume of titrant used.
- (v) Calculate Total Alkalinity as follows:

$$\text{Total alkalinity as mg/L CaCO}_3 = \frac{A \times N \times 1,000 \times 50}{\text{mL of sample}} \quad (6)$$

where: A = mL 0.02 N H₂SO₄ used for methyl-orange end point.
N = Normality of H₂SO₄, 0.02 N.

4. EXPERIMENT

Four water samples (3 from Choa Chu Kang (CCK) Waterworks, each taken at a different stage of the water treatment process and the other one from NTU) will be determined for turbidity, colour, pH, and alkalinity. The samples are:

From CCK: Raw water
Settled water
Filtered water
From NTU: Tap water

5 REPORT

Enter the results in the worksheet similar to that shown in Table 1 and submit them together with the report including the discussion given below.

6 DISCUSSION

- (i) Discuss the progressive change in the colour and turbidity of the samples as the water flows from one process unit to another in the waterworks (see Fig. 2).
- (ii) From your results, are there any observable relationships between turbidity and apparent colour and between turbidity and true colour?
- (iii) What are the pH values of 0.002 N HCl and 0.002 N sodium hydroxide, respectively?
- (iv) Referring to Fig. 1 and your results, what is the predominant alkalinity species in your samples?
- (v) What form of alkalinity would you expect to predominate in most Singaporean and Malaysian natural waters?

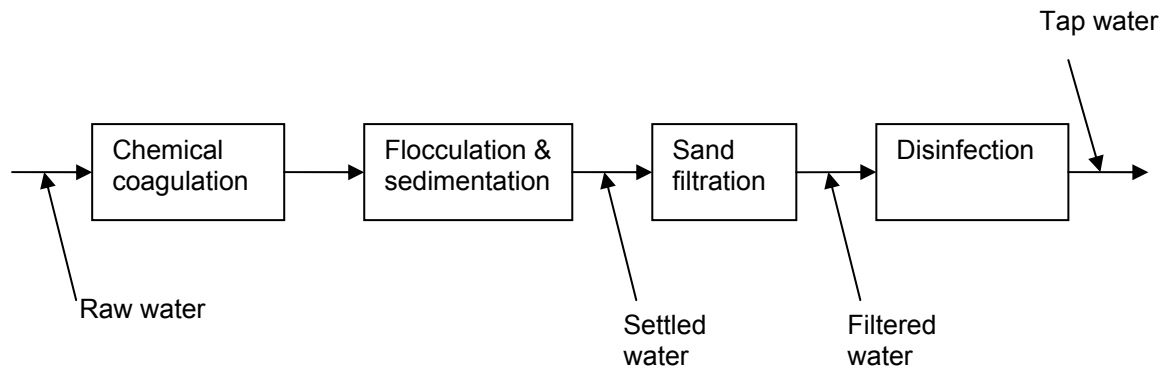


Fig. 2 Conventional water treatment processes.

Name: _____ Batch/Group: _____

Date: _____

Table 1 Test results of turbidity, colour, pH, and alkalinity of water samples from CCK Waterworks and NTU

Sample	Turbidity (NTU)	Apparent Colour (Colour Unit)	True Colour (Colour Unit)	pH	Alkalinity (mgCaCO ₃ /L)
Raw Water (CCK)					
Settled Water (CCK)					
Filtered Water (CCK)					
Tap Water (NTU)					