الشركة القابضة
لمياه الشرب والصرف الصحى

برنامج المسار الوظيفي للعاملين بقطاع مياه الشرب والصرف الصحي

دليل المتدرب

التحاليل الفيزيائية والكيمائية

Physical and Chemical analysis

Part I

كيميائي مياه- الدرجة الثالثة

تم إعداد المادة بواسطة الشركة القابضة لمياه الشرب والصرف الصحي
قطاع تنمية الموارد البشرية - الادارة العامة لتخطيط المسار الوظيفي
الإصدار الثانى - 2019.

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مقدمة الإصدار الثانى

تهدف مجموعة البرامج التدريبية المعدة من إدارة المسار الوظيفى بالشركة القابضة لمياه الشرب والصرف الصحى إلى رفع كفاءة الكيميائين العاملين بالشركة القابضة لمياه الشرب والصرف الصحى والشركات التابعة لها وتنمية مهاراتهم ومعارفهم بالشكل الذى يضمن الوصول إلى كوب مياه نظيف وبيئة آمنة يرضى متطلبات وإحتياجات العملاء الكرام.

ويعتبر الإصدار الثانى من برامج المسار الوظيفى لوظيفة كيميائى مياه الشرب هو ثمرة جهود الكيميائيين العاملين بمعامل الشركات التابعة والمعمل المرجعى لمياه الشرب بالشركة القابضة بما تحمله من مزيج متجانس من الخبرات والكفاءات الذين لم يدخروا جهدا حتى يخرج هذا العمل بالطريقة اللائقة.

وجدير بالذكر أن هذا الإصدار يعتبر مكتبة مرجعية وافية وشاملة لجميع الجدارات المتضمنة المهارات والمعارف التي تجعل الكيميائى كفؤا لوظيفته.

ومما تجدر الإشارة إليه بأنه تم الاعتماد على منهجية للمسار التدريبى بحيث يكون المتدرب قد تلقى الدورات الحقلية بداية من التعرف على مراحل التنقية والمعالجة ثم الانتقال إلى الدورات المعملية داخل معمله طبقا للإطار الزمنى المحدد للمدد البينية لكل درجة وظيفية.

ولقد اعتمدنا على وضع معايير لكل مرحلة فى إعداد هذا الاصدار وكان من أهم هذه المعايير:

- المشاركة الفعالة للخبرات والكفاءات التدريبية بالشركات التابعة فى وضع المناهج بما يناسب عموم الكيميائيين على مستوى الجمهورية.
- عقد ورشة عمل متخصصة لكل مادة تدريبية يشارك بها جميع المدربين ذوى التخصص والخبرات سواء من المعمل المرجعى أو معامل الشركات فضلا عن أن يكون المدرب قد قام بتدريس هذه المادة مرات عديدة.
- استخدام وسيلة اتصال غير تزامنى بين جميع المدربين المعتمدين لكل مادة على حدة من خلال انشاء جروب على الفيس بوك لكل مادة على حده (مذكور فى دليل المدرب).
- وضع حقيبة تدريبية كاملة لكل برنامج معدة طبقا لأحدث النظم والمعايير العالمية تحتوى على (دليل المتدرب- شرائح العرض- ملحقات مقرؤة ومرئية- دليل المدرب- بنك الأسئلة).
- بناء المحتوى لكل برنامج تدريبى طبقاً لأحدث المراجع العالمية ومن أهمها كتاب الطرق القياسية لتحليل مياه الشرب والصرف الصحى (الإصدار رقم 23) وبما يتوافق مع متطلبات آخر إصدارات الايزو(17025)، مع مراعاة التحديثات الخاصة بالتشريعات والقوانين ذات الصلة.

وجدير بالذكر أن الإصدار الثانى من البرامج التدريبية اعتمد فى تصميمه على عرض مبسط للمعلومات قدر الامكان طبقاً للأسس العلمية مع حذف المواضيع المكررة والإسهاب الزائد طبقاً للجدارات المعتمدة على تحديد أهداف واضحة وصريحة لتدريب المتدربين، وتشتق تلك الجدارات من الفهم الواضح لدور المتدرب طبقا لبطاقة الوصف الوظيفى، وتتضمن معارف ومهارات وسلوك. مما يضمن إكساب المتدرب مهارات سلوكية بالإضافة إلى المواد التخصصية.

كما تم تصميم العديد من ورش العمل على أساس تسهيل و تسريع عمليتي التعلم و كسب المهارات بما يسمح بتعظيم الفائدة من العملية التدريبية.

كذلك تم استخدام أساليب التدريب الحديثة والاعتماد على التدريب التفاعلى والتركيز على الجوانب التطبيقية في استخدام الوسائل والأساليب المختلفة ، كما تم استخدام الطرق الحديثة للتعليم التفاعلي والغير تزامني كمصادر مساندة للتعلم من خلال انشاء جروب على الفيس بوك للمدربين المعتمدين **(HCWW Trainers)** .

وفى الختام نرجوا من الله أن يتقبل منا هذا العمل كما نأمل أن يكون هذا العمل علما نافعا للعاملين بقطاع المعامل بالشركة القابضة والشركات التابعة لما يشمله من معلومات فنية قيمة وأن يفيد العاملين الجدد بها ليصبحوا قادرين على تنفيذ مهامهم الوظيفية بالشكل الأمثل .

والله ولي التوفيق.

1. TEMPERATURE

1.1. Scope and Application:

- Measurements of temperature are required in studies of self-purification of rivers and reservoirs, and for the control of waste treatment plants.
- Water temperature is important in relation to fish life. In limnological studies; temperature at different depths is measured.
- Identification of the water source, such as deep wells, is often possible by temperature measurement alone.
- The temperature of drinking water has an influence on its taste. It is also important in connection with bathing and agricultural irrigation.
- Temperature reading is used in the calculation of various forms of alkalinity in studies of saturation and stability with respect to calcium carbonate.

1.2. Apparatus:

- A thermometer has a quick response with 0.1°C divisions, checked against a precision thermometer and sensor electrode.
- Normally, temperature measurements may be made with any good grade, mercury filled, centigrade thermometer, provided it is checked occasionally against a precision thermometer certified by the bureau of standards.
- Field instruments should be provided with a metal case to prevent breakage.

1.3. Procedure:

- Measurement should be made with the thermometer immersed directly in the water body, after a period of time sufficient to permit constant reading.

1.4. Units of temperature:

- $C^o = K-273$
- $K= C^o +273$
- $C^o = (F - 32)/ 1.8$
- $F = (C^o * 1.8) +32$

2. ELECTRICAL CONDUCTIVITY

2.1. Principle:

- Conductivity, k, is a measure of the ability of an aqueous solution to carry an electric current.
- Positive and negative ions in a solution (free ions) will move to the oppositely charged electrode when an electric charge is applied to the solution, thus conducting current.
- Ion movement is affected by the solvent properties (temperature, viscosity) and the physical properties of the ion (size, charge, concentration...).
- As temperature increases, ions move faster and conduct more current and as viscosity increases, the ions move slower and conduct less current.
- Solutions of most inorganic compounds are relatively good conductors, conversely, molecules of organic compounds that do not dissociate in aqueous solution conduct a current very poorly, if at all.
- For our purposes, conductivity is measured by placing a cell (probe) in an electrolytic solution. A cell consists of two electrodes of a specific size, spaced at a specific distance apart.
- The conductivity of a liquid is the ratio of current to voltage between the electrodes. The conductivity value changes if the electrodes are spaced closer or further from each other. To have useful analyses, the distance between the two electrodes must be considered in the conductivity calculation.

2.2. Definitions and Units of Expression

- Conductance, G, is defined as the reciprocal of resistance, R:

$$G = \frac{1}{R}$$

  - ➢ where the unit of R is ohm and G is ohm-1 (sometimes written mho).
  - ➢ ohm-1 = mho

- Conductance of a solution is measured between two spatially fixed and chemically inert electrodes. To avoid polarization at the electrode surfaces the conductance measurement is made with an alternating current signal.
- The conductance of a solution, G, is directly proportional to the electrode surface area, A, cm2, and inversely proportional to the distance between the electrodes, L, cm.
- The constant of proportionality, k, such that::

$$G = k\left(\frac{A}{L}\right)$$

•

- is called “conductivity” (preferred to “specific conductance”). It is a characteristic property of the solution between the electrodes. The units of k are 1/ohm-cm or mho per centimeter.
- Conductivity is customarily reported in micromhos per centimeter (µmho/cm).
- In the International System of Units (SI) the reciprocal of the ohm is the siemens (S)

ohm-1 = mho= siemens (S)

- Conductivity is reported as millisiemens per meter (mS/m);

1 mS/m = 1000 µmhos/100cm= 10 µmhos/cm

But 1 µS/cm = 1 µmho/cm

So, 1 mS/m = 10 µS/cm

- To report results in SI units of mS/m divide µmhos/cm by 10.
- To compare conductivities, values of k are reported relative to electrodes with A=1 cm2 and L=1 cm.
- Absolute conductance, Gs, of standard potassium chloride solutions between electrodes of precise geometry have been measured; the corresponding standard conductivities, ks, are shown in the table below showing equivalent conductivity, , and conductivity, k, of potassium chloride at 25.0 °C.

KCl Concentration M or equivalent/L	**Equivalent Conductivity, mho-cm^2/equivalent**	**Conductivity, ks umho/cm**
0	149.9	
0.0001	148.9	14.9

0.0005	147.7	73.9
0.001	146.9	146.9
0.005	143.6	717.5
0.01	141.2	1 412
0.02	138.2	2 765
0.05	133.3	6 667
0.1	128.9	12 890
0.2	124.0	24 800
0.5	117.3	58 670
1	111.9	111 900

2.3. Equipment:

- Conductivity meter with temperature compensation.
- Conductivity probe (Electrode).

2.4. Chemical and reagents:

- Standard conductivity solution (1000, 1410, 1413 or 500µS/cm) any of them is available.

2.5. Conductivity water:

- Any of several methods can be used to prepare reagent-grade water. The methods discussed in Section1080 are recommended. The conductivity should be small compared to the value being measured.

2.6. Standard potassium chloride solution (KCl), 0.0100M:

- Dissolve 745.6 mg anhydrous KCl in conductivity water and dilute to 1000 mL in a class A volumetric flask at 25°C and store in a CO_2^-free atmosphere.
- This is the standard reference solution, which at 25°C has a conductivity of 1412 mhos/cm. It is satisfactory for most samples when the cell has a constant between 1 and 2 cm1.

- For other cell constants, use stronger or weaker KCl solutions listed in Table 2510: I. Care must be taken when using KCl solutions less than 0.001M, which can be unstable because of the influence of carbon dioxide on pure water.
- For low conductivity standards, Standard Reference Material 3190, with a certified conductivity of 25.0 S/cm 0.3 S/cm, may be obtained from NIST.
- Store in a glass-stoppered borosilicate glass bottle.

2.7. Precaution:

- If the conductivity meter gets dirty, wipe the surface with a damp cloth.
- During normal use, rinse the probe with Deionized water between measurements. (This will minimize the interfering substances on the probe element).
- The cell must be stored dry.
- The solution must be clear from particles or suspended matter, don't allow the cell to come into contact with any sediment which may be present by leaving sample for sedimentation and measure conductivity for supernatant.
- You can store the electrode either wet or dry but if it is stored dry you will need to recondition the electrode before use.

2.8. Procedure:

2.8.1. Instrument calibration:

- Calibrating with a known standard: The conductivity meter must be calibrated before use by using standards with known electrolytic conductivity (1000, 1410, 1413 µS/cm at 25˚C, as available) the calibration instrumental according to manufacturing instructions.

2.8.2. Measurement:

- For measurement, rinse the probe with tap water then Deionized water then dry it.
- Immerse the probe in sample, press ENTER, and allow the readout to stabilize.
- Record conductivity reading when stabilize on sampling recording sheet.

2.8.3. Calculation:

- Internal calculation in Conductivity meter.

2.9. References:

- Ref: Standard Methods 23rd Edition,2017.

3. TOTAL SOLIDS (Dried at 103–105°C)

3.1. Principle:

- A well-mixed sample is evaporated in a weighed dish and dried to constant weight in an oven at 103 to 105°C.
- The increase in weight over that of the empty dish represents the total solids.
- The results may not represent the weight of actual dissolved and suspended solids in wastewater samples.

3.2. Interferences:

- Highly mineralized water with a significant concentration of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and require prolonged drying, proper desiccation, and rapid weighing.
- 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.
- Disperse visible floating oil and grease with a blender before withdrawing a sample portion for analysis.

3.3. Apparatus:

- Evaporating dishes: Dishes of 100-ml capacity made of one of the following materials:
- Porcelain, 90-mm diam.
- Platinum—Generally satisfactory for all purposes.
- High-silica glass
- Muffle furnace for operation at 550°C.
- Steam bath.
- Desiccator
- Drying oven, for operation at 103 to 105°C.
- Analytical balance, capable of weighing to 0.1 mg.
- Magnetic stirrer with TFE stirring bar.
- Wide-bore pipets
- Graduated cylinder.
- Low-form beaker

3.4. Procedure:

3.4.1. Preparation of evaporating dish:

- If volatile solids are to be measured ignite clean evaporating dish at 550°C for 1h in a muffle furnace.
- If only total solids are to be measured, heat clean dish to 103 to 105°C for 1h.
- Store and cool dish in desiccator until needed. Weigh immediately before use.

3.4.2. Sample analysis:

- For homogeneous samples, pipet from the approximate midpoint of the container but not in the vortex.
- Choose a point both mid depth and midway between wall and vortex.
- Evaporate to dryness on a steam bath or in a drying oven.
- When evaporating in a drying oven, lower temperature to approximately 2°C below boiling to prevent splattering.
- Dry evaporated sample for at least 1h in an oven at 103 to 105°C, cool dish in desiccator to balance temperature, and weigh.
- Repeat cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained, or until weight change is less than 4% of previous weight or 0.5 mg, whichever is less.
- Analyze at least 10% of all samples in duplicate.
- Duplicate determinations should agree within 5% of their average weight.

3.4.3. Calculation:

$$\text{mg total solids/L} = \frac{(A - B) \times 1000}{\text{sample volume, mL}}$$

- ➢ Where:
- ➢ A = weight of dried residue + dish, mg.
- ➢ B = weight of dish, mg.

3.5. References:

- Ref: Standard Methods 23rd Edition,2017. 2540-B SOLIDS.

4. TOTAL DISSOLVED SOLIDS (TDS) (Dried at 180°C)

4.1. Principle:

- A well-mixed sample is filtered through a standard glass fiber filter, and the filtrate is evaporated to dryness in a weighed dish and dried to constant weight at 180°C.
- The increase in dish weight represents the total dissolved solids.
- The results may not agree with the theoretical value for solids calculated from chemical analysis of sample.

4.2. Interferences:

- Highly mineralized waters with a considerable calcium, magnesium, chloride, and/or sulfate content may be hygroscopic and require prolonged drying, proper desiccation, and rapid weighing.
- Samples high in bicarbonate require careful and possibly prolonged drying at 180°C to insure complete conversion of bicarbonate to carbonate.
- Because excessive residue in the dish may form a water-trapping crust, limit sample to no more than 200 mg residue.

4.3. Apparatus:

- Apparatus listed in Section 2540B.2 a-h (look at standard methods for the examination of water and wastewater) is required, and in addition:
- Glass-fiber filters disks without organic binder.
- Filtration apparatus: One of the following, suitable for the filter disk selected:
- Membrane filter funnel.
- Gooch crucible, 25-ml to 40-ml capacity, with Gooch crucible adapter.
- Suction flask, of sufficient capacity for sample size selected.
- Drying oven, for operation at 180 ± 2°C.

4.4. Procedure:

4.4.1. Preparation of glass-fiber filter disk:

- Insert disk with wrinkled side up into filtration apparatus.
- Apply vacuum and wash disk with three successive 20-ml volumes of reagent- grade water.
- Continue suction to remove all traces of water. Discard washings.

4.4.2. Preparation of evaporating dish:

- If only total dissolved solids are to be measured, heat clean dish to 180 ± 2°C for 1h in an oven.

- Store in desiccators until needed and Weigh immediately before use.

4.4.3. Selection of filter and sample sizes:

- Choose sample volume to yield between 2.5 and 200 mg dried residue.
- If more than 10 min are required to complete filtration, increase filter size or decrease sample volume.

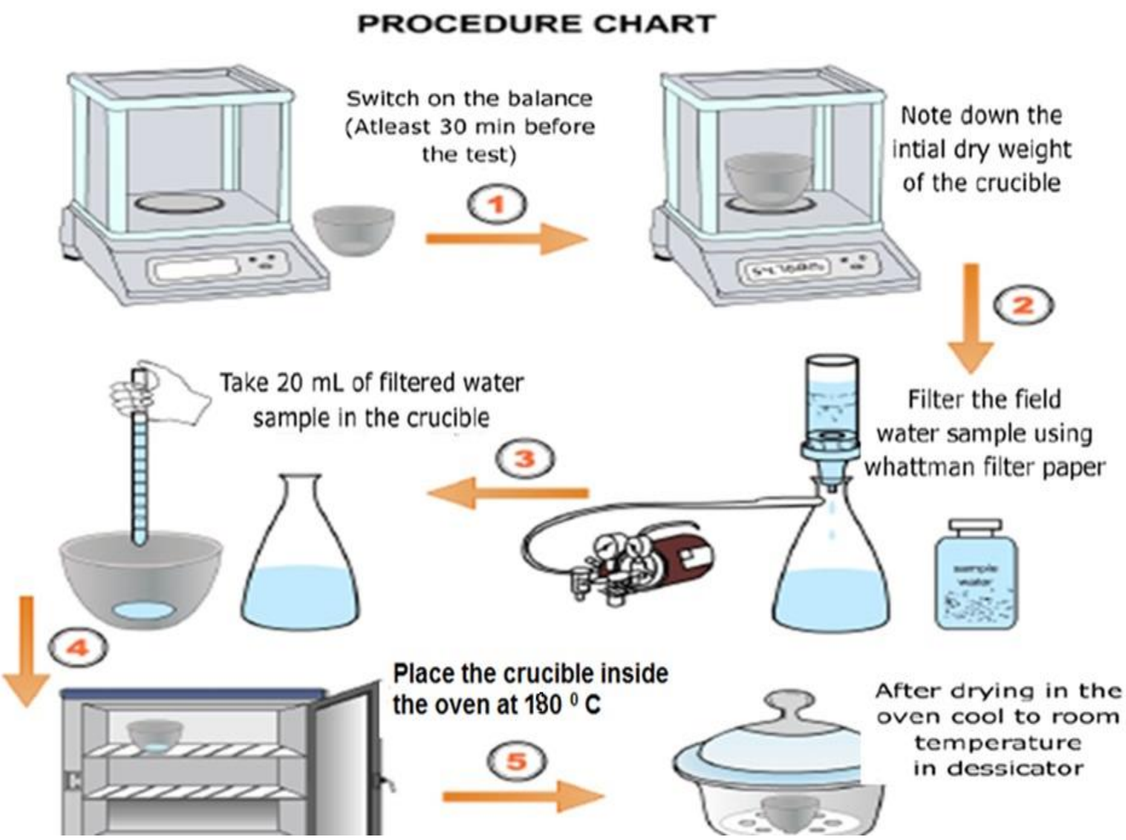
4.4.4. Sample analysis:

- Stir sample with a magnetic stirrer and pipet a measured volume onto a glass-fiber filter with applied vacuum.
- Wash with three successive 10-ml volumes of reagent-grade water, allowing complete drainage between washings, and continue suction for about 3 min after filtration is complete.
- Transfer total filtrate (with washings) to a weighed evaporating dish and evaporate to dryness on a steam bath or in a drying oven.
- Dry evaporated sample for at least1h in an oven at 180 ± 2°C, cool in a desiccator to balance temperature, and weigh.
- Repeat drying cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 4% of previous weight or 0.5 mg, whichever is less.
- Analyze at least 10% of all samples in duplicate.
- Duplicate determinations should agree within 5% of their average weight.

4.4.5. Calculation:

$$\text{mg total dissolved solids/L} = \frac{(A - B) \times 1000}{\text{sample volume, mL}}$$

- ➢ Where:
- ➢ A = weight of dried residue + dish, mg.
- ➢ B = weight of dish, mg.



4.5. References:

Ref: Standard Methods 22rd Edition,2017. 2540-C SOLIDS.

5. TOTAL SUSPENDED SOLIDS (Dried at 103 – 105°C)

5.1. Principle:

- A well-mixed sample is filtered through a weighed standard glass-fiber filter and the residue retained on the filter is dried to a constant weight 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, it may be necessary to increase the diameter of the filter or decrease the sample volume.
- To obtain an estimate of total suspended solids, calculate the difference between total dissolved solids and total solids.

5.2. Interferences:

- Exclude large floating particles or submerged agglomerates of nonhomogeneous materials from the sample if it is determined that their inclusion is not representative.
- Because excessive residue on the filter may form a water-entrapping crust, limit the sample size to that yielding no more than 200 mg residue.
- For samples high in dissolved solids thoroughly wash the filter to ensure removal of dissolved material.

5.3. Apparatus

- Apparatus listed in Section 2540B.2 and Section 2540C.2 (look at standard methods for the examination of water and wastewater) is required, except for evaporating dishes, steam bath and 180°C drying oven.

5.4. Procedure:

5.4.1. Preparation of glass-fiber filter disk:

- Apply vacuum and wash disk with three successive 20-mL portions of reagent-grade water.
- Continue suction to remove all traces of water and turn vacuum off, and discard washings.
- Remove filter from filtration apparatus and transfer to an inert aluminum weighing dish.
- If a Gooch crucible is used, remove crucible and filter combination.
- Dry in an oven at 103 to 105°C for 1h.
- Cool in desiccator to balance temperature and weigh.
- Repeat cycle of drying or igniting, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 4% of the previous weighing or 0.5 mg, whichever is less.

5.4.2. Selection of filter and sample sizes:

- Choose sample volume to yield between 2.5 and 200 mg dried residue.
- If volume filtered fails to meet minimum yield, increase sample volume up to 1 L.
- If complete filtration takes more than 10 min, increase filter diameter or decrease sample volume.

5.4.3. Sample analysis:

- Wet filter with a small volume of reagent-grade water to seat it.
- Stir sample with a magnetic stirrer at a speed to shear larger particles, if practical, to obtain a more uniform (preferably homogeneous) particle size.
- Note: Centrifugal force may separate particles by size and density, resulting in poor precision when point of sample withdrawal is varied.
- While stirring, pipet a measured volume onto the seated glass-fiber filter.
- For homogeneous samples, pipet from the approximate midpoint of container but not in vortex.
- Choose a point both mid depth and midway between wall and vortex.
- Wash filter with three successive 10-ml volumes of reagent-grade water, allowing complete drainage between washings, and continue suction for about 3 min after filtration is complete.
- Samples with high dissolved solids may require additional washings.
- Carefully remove filter from filtration apparatus and transfer to an aluminum weighing dish as a support.
- Dry for at least 1h at 103 to 105°C in an oven, cool in a desiccator to balance temperature, and weigh.
- Repeat the cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until the weight change is less than 4% of the previous weight or 0.5 mg, whichever is less.
- Analyze at least 10% of all samples in duplicate.
- Duplicate determinations should agree within 5% of their average weight.

5.4.4. Calculation:

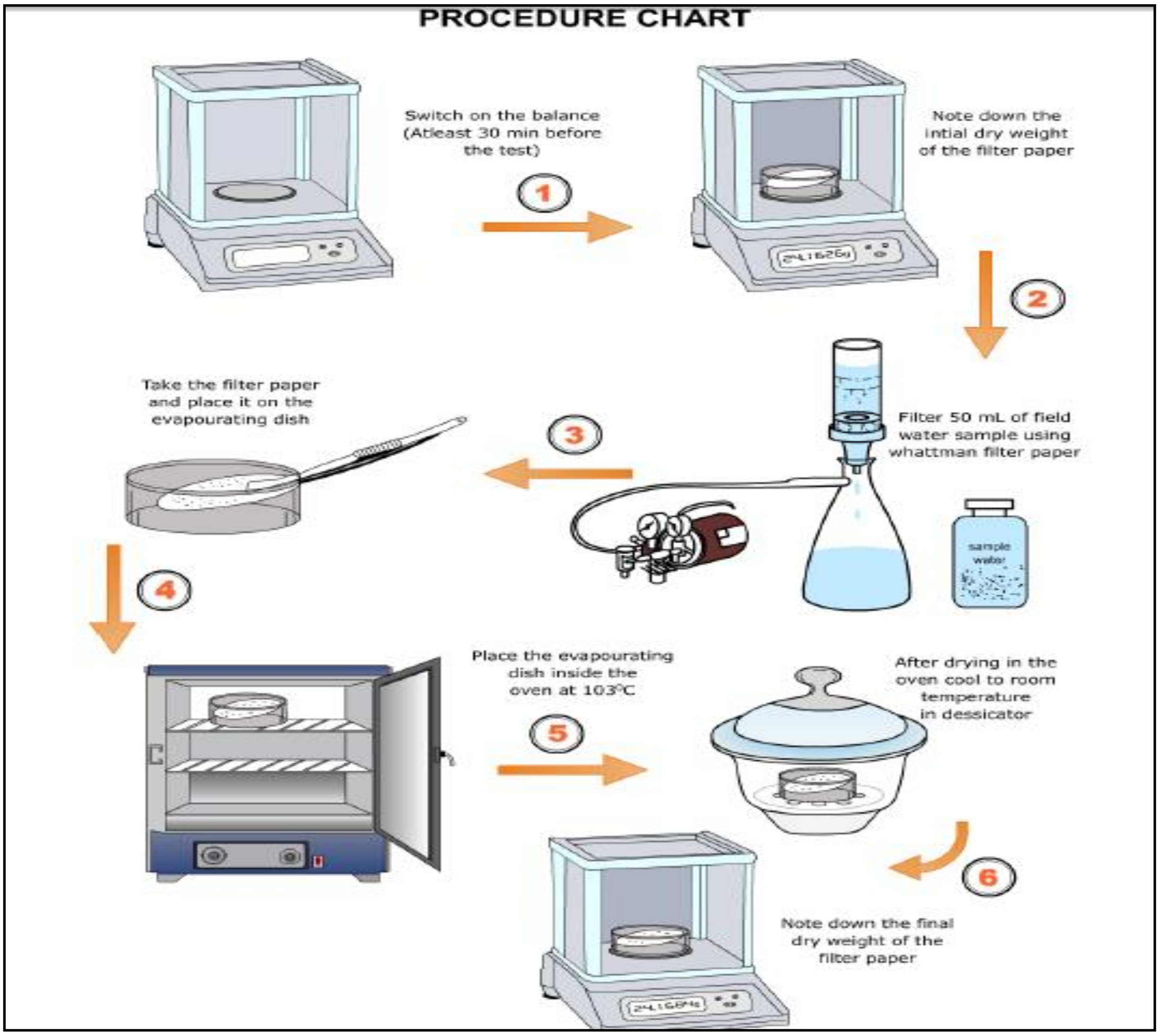
$$\text{mg total suspended solids/L} = \frac{(A - B) \times 1000}{\text{sample volume, mL}}$$

- Where:

- A = weight of filter + dried residue, mg.
- B = weight of filter, mg.

5.5. References:

- Standard Methods 23rd Edition,2017. 2540-D SOLIDS .


PROCEDURE CHART
Switch on the balance (Atleast 30 min before the test)
①
Note down the intial dry weight of the filter paper
②
Filter 50 mL of field water sample using whattman filter paper
sample water
③
Take the filter paper and place it on the evapourating dish
④
Place the evapourating dish inside the oven at 103°C
⑤
After drying in the oven cool to room temperature in dessicator
⑥
Note down the final dry weight of the filter paper

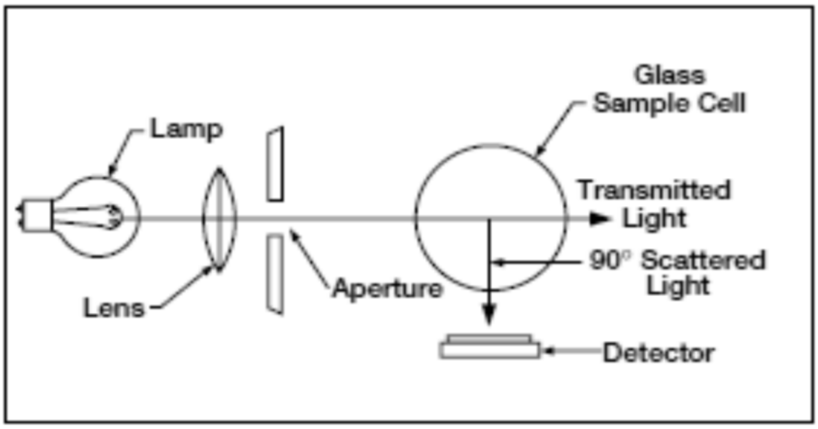
6. TURBIDITY

6.1. Sources and Significance:

- Turbidity in water is caused by suspended and colloidal matter such as clay, silt, finely divided organic and inorganic matter, and plankton and other microscopic organisms.
- Clarity of water is important in producing products destined for human consumption and in many manufacturing operations. Beverage producers, food processors, and potable water treatment plants drawing from a surface water source commonly rely on fluid-particle separation processes such as sedimentation and filtration to increase clarity and insure an acceptable product. The clarity of a natural body of water is an important determinant of its condition and productivity.

6.2. Principle:

- This method is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the intensity of scattered light, the higher the turbidity and vise versa



6.3. Interference:

- Debris and rapidly settling coarse sediment interfere with turbidity measurements. Dirty glassware and the presence of air bubbles give false results. “Tue color” i.e., Water color due to dissolved substances that absorb light causes measured turbidities to be low. This effect usually is not significant in treated water.

6.4. Apparatus:

6.4.1. Turbidimeter or Nephelometer

- Turbidity measurements are done conveniently through the use of photometers. A beam of light from a source produced by a standardized electric bulb is passed through a sample vial. The light that emerges from the sample is then directed to a photometer that measures the light absorbed.

6.5. Reagents:

- Deionized water its Turbidity less than limit of quantitation of the method.
- Formazin set of standards ready to use according to manufacturer's instructions.

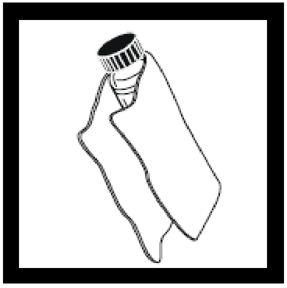
6.6. Procedure:

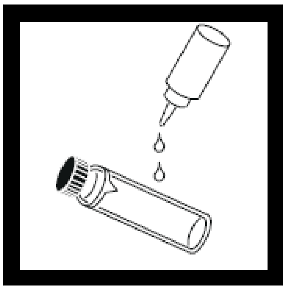
6.6.1. General measurement techniques:

- The measurement will be more accurate, precise, and repeatable if close attention is paid to proper measurement techniques.
- Measure turbidity immediately to prevent temperature changes and particle flocculation and sedimentation from changing sample characteristics.
- If flocculation is apparent, break up aggregates by agitation.
- Particles suspended in the original sample may dissolve or otherwise change characteristics when the temperature changes or when the sample is diluted.
- Remove air or other entrained gases in the sample before measurement. Preferably degas even if no bubbles are visible. Degas by applying a partial vacuum, adding a non-foaming-type of surfactant, using an ultrasonic bath, or applying heat.
- If degassing cannot be applied, bubble formation will be minimized if the samples are maintained at the temperature and pressure of the water before sampling.
- Do not remove air bubbles by letting sample stand for a period of time because during standing turbidity-causing particulates may settle and sample temperature may change.
- Condensation may occur on the outside surface of a sample cell when a cold sample is being measured in a warm, humid environment. This interferes with turbidity measurement.
- Remove all moisture from the outside of the sample cell before placing the cell in the instrument.
- If fogging recurs, let sample warm slightly by letting it stand at room temperature or by partially immersing it in a warm water bath for a short time.
- Make sure samples are again well mixed.

6.6.2. Measurement

- Fill the sample cell to the line, cap the sample cell.
- Hold the sample cell by the cap, and wipe to remove water spots and fingerprints.
- Gently mix the sample cells by slowly and gently inverted the sample cell to homogenize the sample, be sure to eliminate the air bubbles
- Apply a thin bead of silicone oil (if available) from the top to bottom of the cell just enough to coat the cell with a thin layer of oil. Using the oiling cloth provided spread the oil uniformly, then wipe off the excess. The cell should appear nearly dry.





- Insert the cell in the cell holder.
- The instrument displays the turbidity measure of the sample in NTUs (Nephelometric turbidity units)
- Note: The instrument’s response time is according to manufacturer's instructions.

6.6.3. Calculations:

- Sample results are read directly from the meter display.

6.6.4. Reporting:

- Turbidity value expressed by NTU unit
- Number of significant figures is three.

6.6.5. References:

- Standard Methods 23rd Edition,2017. 2130-B Nephelometric Method TURBIDITY.

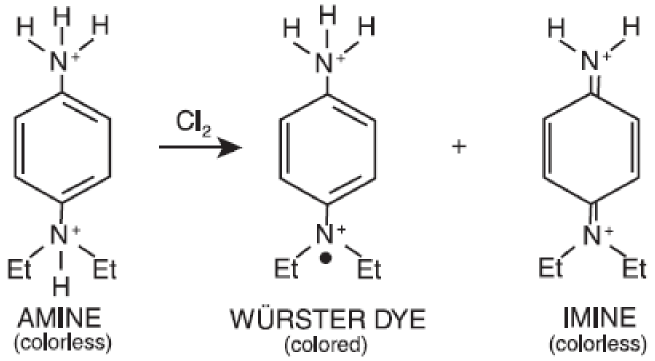
7. CHLORINE RESIDUAL BY DPD

7.1. Scope and Application

- Even in the l990s, the World Health Organization considered pathogenic microorganisms to be the most important danger to drinking water in both developed and developing countries. The primary purpose of chlorinating public water supplies is to prevent the spread of water borne diseases. Destruction of pathogenic organisms by chlorine is directly related to contact time and the concentration of the chlorine.
- Chlorine applied to water initially undergoes hydrolysis to form free available chlorine. The free chlorine reacts readily with ammonia and certain nitrogenous compounds to form combined chlorine. The presence of free and/or combined chlorine depends on pH, temperature, and initial chlorine to nitrogen ratio.
- When using Chlorine to disinfect water, the aim is to kill off the germs (pathogens) and then to leave a small amount of active (free) Chlorine in the water. This remaining Chlorine is also known as the “residual Chlorine” and is important as it is available to disinfect further contamination of the water.
- Total Chlorine is the sum of the free Chlorine available in the water to disinfect future germs and the Chlorine that has already been used and bound together with pathogenic molecules.
- It is vital to calculate the correct amount of Chlorine to be added:
  - too much is negative to health and wastes money,
  - too little can result in a high level of disease causing pathogens.
- The Chlorine demand can be determined by deducting the residual from the amount of Chlorine added.

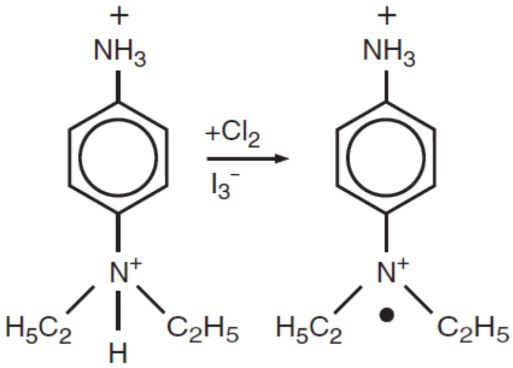
7.2. DPD Colorimetric Method

- The DPD (N, N-diethyl-p-phenylenediamine) method for residual chlorine was first introduced by Palin in 1957. Over the years it has become the most widely used method for determining free and total chlorine in water and wastewater.
- The chemical basis for the DPD chlorine reaction is depicted in the below Figure.



DPD-Chlorine Reaction Products

- The DPD amine is oxidized by chlorine to two oxidation products. At a near neutral pH, the primary oxidation product is a semi-quinoid cationic compound known as a Würster dye. This relatively stable free radical species accounts for the magenta color in the DPD colorimetric test. DPD can be further oxidized to a relatively unstable, colorless imine compound.
- When DPD reacts with small amounts of chlorine at a near neutral pH, the Würster dye is the principal oxidation product. At higher oxidant levels, the formation of the unstable colorless imine is favored resulting in apparent "fading" of the colored solution.



(Colorless) Würster Dye (Red)

- The triiodide, in turn, reacts with DPD, forming the Würster oxidation product. There is very little confirmed evidence that trichloramine species can be quantified when using iodide with DPD. In practice, only a trace of iodide is required at pH 6.2-6.5 to resolve monochloramine.
- Standard Methods for the Examination of Water and Wastewater stipulates the addition of approximately 0.1 mg of potassium iodide to a 10-mL sample to determine monochloramine. By adding excess potassium iodide (an additional 0.1 gram or more per 10-mL sample), dichloramine is included. It is not entirely clear at what level of iodide the dichloramine fraction begins to intrude into the monochloramine results.
  - DPD1 measures free (residual) Chlorine, the Chlorine available to kill bacteria.
  - DPD2 measures Monochloramine, a less effective and less widely used disinfectant than Chlorine.
  - DPD3 measures combined Chlorine, so add DPD3 to the same sample as DPD1 and you will be able to calculate the total Chlorine.
  - DPD4 is used to measure total Chlorine only.
- DPD tablets can accurately measure Chlorine levels from 0.001 ppm to 10 ppm. When a sample has a chlorine level that is higher than 10ppm, the color is bleached out of

the Chlorine tablet, (there is a quick show of pink color and then the sample returns to its original color. Dilutions can be done to determine high range Chlorine levels.

7.3. Summery

- This test describes the procedures for determining free chlorine, total chlorine, and combined chlorine residuals in various water samples. Low concentrations of chlorine (less than 4 ppm) react with DPD (N, N-Diethyl-p-phenylenediamine) giving a red color, which varies in its intensity with the chlorine concentration. For concentrations of chlorine greater than 4 ppm, samples must be diluted with chlorine-demand-free water.

7.4. Interferences

- Concentrations of free chlorine greater than 4 ppm lead to disappearing or fading of the red color as soon as it is formed. For concentrations of chlorine greater than 4 ppm, the sample must be diluted with chlorine-demand-free water.

7.5. Sample Handling

- Chlorine residual determinations must be carried out immediately (within 10 to 15 minutes) after sample collection due to the instability of chlorine.

7.6. Equipment and Appartus

- Colorimetric comparator, discs, and test vessels.

7.7. Reagents and Standards

- Two DPD tablets are required as follows:
  - DPD No. l Tablet
  - DPD No. 3 Tablet

7.7.1. Phosphate buffer solution:

- Dissolve 24 g anhydrous Na2HPO4 and 46 g anhydrous KH2PO4 in distilled water. Combine with 100 mL distilled water in which 800 mg disodiumethylenediamine tetraacetate dihydrate (EDTA) have been dissolved.
- Dilute to 1 L with distilled water and optionally add either 20 mg HgCl2 or 2 drops toluene to prevent mold growth.
- Interference from trace amounts of iodide in the reagents can be negated by optional addition of 20 mg HgCl2 to the solution.
- *CAUTION: $HgCl_2$ is toxic—take care to avoid ingestion.*

7.7.2. N,N-Diethyl-p-phenylenediamine (DPD) indicator solution:

- Dissolve 1 g DPD oxalate,or 1.5 g DPD sulfate pentahydrate, or 1.1 g anhydrous DPD sulfate in chlorine-free distilled water containing 8 mL 1 _ 3 H_2SO_4 and 200 mg disodium EDTA.
- Make up to 1 L, store in a brown glass-stoppered bottle in the dark, and discard when discolored.
- Periodically check solution blank for absorbance and discard when absorbance at 515 nm exceeds 0.002/cm. (The buffer and indicator sulfate are available commercially as a combined reagent in stable powder form.)
- CAUTION: The oxalate is toxic—take care to avoid ingestion

7.7.3. Potassium permanganate solutions—Prepare a stock solution

- containing 891 mg $KMnO_4$/1000 mL. Dilute 10.00 mL stock solution to 100 mL with distilled water in a volumetric flask. When 1 mL of this solution is diluted to 100 mL with distilled water, a chlorine equivalent of 1.00 mg/L will be produced in the DPD reaction.

7.8. Procedure

7.8.1. Free Chlorine

- Fill the test vessel in the left-hand compartment with approximately 10 mL of water sample.
- Rinse out the vessel in the right-hand compartment with sample and leave in a few drops.
- Add a DPD No. l tablet and crush with a stirring rod.
- Fill to 10 mL with the sample and mix, then place the vessel in the right-hand compartment.
- Rotate the disc until the color of the sample is matched by one of the glass standards.
- Read immediately the value expressed in ppm from the indicator window. This is value “A”.

7.8.2. Total Chlorine

- To the colored sample above, add a DPD No. 3 tablet, mix to dissolve and allow to stand for 2 minutes.
- Rotate the disc until the color of the sample is matched by one of the glass standards.
- Read immediately the value expressed in ppm from the indicator window. This is value “B”.

7.8.3. Precaution:

- Analysis for free chlorine: Reading shall be taken immediately upon addition of DPD to avoid change in colour.

7.8.4. Calculations

- Free Chlorine Residual = A in ppm
- Total Chlorine Residual = B in ppm
- Combined Chlorine Residual = (B - A) in ppm

7.8.5. Reporting

- The free chlorine residual shall be measured every two hours for the distributor, each Sedimentation Basins, and the treated water and recorded in the Daily Log Book. For treated water, the number of samples (normally 12), and the minimum, the maximum, and the average chlorine residual shall be recorded every day on the WTP Daily Report.
- Results are reported to the nearest one-tenth (for example, 1.6 ppm).

7.8.6. Quality Control

- Results can be controlled by analyzing a duplicate sample.

Chemical Analysis

8. Review on Quantitative Analytical Chemistry.

- We use the atomic mass unit (amu) as a unit for the mass number of each single atom in periodic table.
- The atomic mass number is the weight of a single atom in (amu).
- If we multiply mass number by Avogadro's number (6.022140857 × 10 23) we get the atomic molar mass which is the same number as atomic mass number but in (gm).
- Solute + Solvent = Solution

8.1. Molarity (M):

Number of molar mass(M.Wt) of solute per one Liter of solution.

- Eg. If we put 80 gm of NaOH (80/40=2 molar mass) in 1 L volumetric flask, containing little water and dissolve it then we complete water to mark, then we get 2 molar NaOH solution (2mole/Liter of solution).
- (Na=23 gm/mole O=16 gm/mole, H=1 gm/mole NaOH=40 gm/mole)

8.2. Molality (m):

Number of molar mass(M.Wt) of solute per 1Kg of solvent.

- Eg. If we put 200 gm of $CaCO_3$ (200/100=2 molar mass) in 1 L volumetric flask, containing 1 Kg(1L) water and dissolve it, then we get 2 molal NaOH solution (2mole/Kg of solvent).
- (Ca=40 gm/mole, C=12 gm/mole, O=16 gm/mole, $CaCO_3$=100 gm/mole)

8.3. Strength (S):

Number of grams of solute per Liter of solution. (gm/Liter of solution.)

8.4. Normality (N):

Number of equivalent weights of solute per one Liter of solution (g.equivalent/Liter of solution).

$$Eq.Wt = M.Wt/x$$

- In case of acids: x=No. of liable H eg. HCl: x=1, H_2SO_4: x=2, H_3PO_4: x=3
- In case of base: x=No. of OH eg. NaOH: x=1, Ca $(OH)_2$ x=2, Al $(OH)_3$: x=3
- In case of salts x= (No of anions*its valency) = (No of cations*its valency) eg. NaCl: x= (1*1=1), $CaCl_2$ x= (1*2=2), $Al_2(SO_4)_3$ x= (2*3=6)
- In case of chemical compound undergoes oxidation reduction x= no of e's involved.

- Normality (N)=x*Molarity (M)

$$\boldsymbol{S = N * Eq.Wt}$$

$$\boldsymbol{S = M * M.Wt}$$

- To prepare diluted concentration (C2) of volume (V2), we take Volume (V1) from original concentration (C1), as per the following relation:

$$\boldsymbol{(C1 * V1)\ before\ dilution = (C2 * V2)\ after\ dilution}$$

- **eg.** To prepare 600 ml of 0.1 M HCl from 3 M HCl

$$(3 * V_1)_{\text{before dilution}} = (0.1 * 600)_{\text{after dilution}}$$

$$V_1 = 0.1 * 600/3$$

$$V_1 = 20\ \text{ml}$$

- So we take 20 ml of stock solution and dilute it with pure water to 600 ml, then mix well.
- **In titration we use:**

$$(N1 * V1) Titrant = (N2 * V2)\ Titrate$$

or

$$\frac{(M1 * V1)}{\mathrm{n1}} Titrant = \frac{(M2 * V2)}{\mathrm{n2}}\ Titrate$$

  - where n is the number of moles in reaction equation

- To prepare a volume V (ml) of Normality N of solid compound we use the following relation:

$$Wt(gm) = N * Eq.Wt * V(ml)/1000, \text{OR}$$

$$Wt(gm) = M * M.Wt * V(ml)/1000$$

- So we dissolve the calculated wt(gm) in portion of solvent, then we dilute it to the volume V.

- To prepare a volume V2 (ml) of Normality N or Molarity M of liquid material we use the following relation:

$$N = P * d * 10/\,Eq.Wt \quad \text{or} \quad M = P * d * 10/\,M.Wt$$

In combination with

$$\boldsymbol{(C1 \; * \; V1)\ before\ dilution \; = \; (C2 \; * \; V2)\ after\ dilution}$$

so

$$((P*d*10/\ Eq.Wt) * V1)_{\text{before dilution}} = (N2 * V2)_{\text{after dilution}}$$

$$V_{1\ \text{before dilution}} = Eq.Wt * (N_2 * V_2)_{\text{after dilution}}/(P*d*10)$$

or

$$((P*d*10/\ M.Wt) * V_1)_{\text{before dilution}} = (M_2 * V_2)_{\text{after dilution}}$$

$$\boldsymbol{V1\ \ before\ dilution = M.Wt * (M2 * V2)\ after\ dilution/(P*d*10)}$$

- Where : P=%, D=denisty
- So we take the calculated volume of stock V1, then we dilute it to the volume V2.
- ppm = mg/L = µg/ml

- To prepare 2 liters of 100 ppm NaCl:
    - 100ppm = 100mg/L = 200mg/2L
- So we weigh 200mg of NaCl and dissolve it in portion of pure water then complete to the mark in 2L volumetric flask
- To prepare 100 ppm Cl from NaCl:
    - NaCl-----------------Cl
    - 23+35.5=58.5 35.5 gm
    - X 100mg/L (which is X is unknown)
    - ?????????= 100(mg/L)*58.5~~gm~~/35.5~~gm~~
    - = 164.789 gm of NaCl to be dissolved in portion of pure water then diluted to 1 Liter to get 100 ppm Cl

9. pH

9.1. Principle:

- Measurement of pH is one of the most important and frequently used tests in water chemistry. Practically every phase of water supply and wastewater treatment, e.g., acid-base neutralization, water softening, precipitation, coagulation, disinfection, and corrosion control, is pH-dependent.
- pH is used in alkalinity and carbon dioxide measurements and many other acid-base equilibrium. At a given temperature the intensity of the acidic or basic character of a solution is indicated by pH or hydrogen ion activity.
- Alkalinity and acidity are the acid- and base-neutralizing capacities of water and usually are expressed as milligrams $CaCO_3$ per liter.
- Buffer capacity is the amount of strong acid or base, usually expressed in moles per liter, and needed to change the pH value of a L-1 sample by one unit.
- pH as defined as $-\log [H^+]$; it is the ''intensity'' factor of acidity. Pure water is very slightly ionized and at equilibrium the ion product is:

$$Kw = [H+][OH-] \qquad (1)$$

$$= 1.01 \times 10^{-14}\ at\ 25°C$$

and

$$[H+] = [OH-] = 1.005 \times 10-7$$

- ➢ Where:
- ➢ $[H^+]$ = activity of hydrogen ions, moles/L,
- ➢ $[OH^-]$ = activity of hydroxyl ions, moles/L.
- ➢ Kw = ion product of water.

NOTE:

- Because of ionic interactions in all but very dilute solutions, it is necessary to use the ''activity'' of an ion and not its molar concentration. Use of the term pH assumes that the activity of the hydrogen ion, aH^+ is being considered.
- The approximate equivalence to molarity, $[H^+]$ can be presumed only in very dilute solutions (ionic strength <0.1). A logarithmic scale is convenient for expressing a wide range of ionic activities. Equation 1 in logarithmic form and corrected to reflect activity is:

$$(-\log_{10} a_{H^+}) + (-\log_{10} a_{OH^-}) = 14 \qquad (2)$$

or

$$pH + pOH = pKw$$

- Where:
- $pH = -\log10\ aH^+$
- $pOH = -\log10\ aOH^-$.

- Equation 2 states that as pH increases pOH decreases correspondingly and vice versa because pKw is constant for a given temperature.
- At 25°C, pH 7.0 is neutral, the activities of the hydrogen and hydroxyl ions are equal, and each corresponds to an approximate activity of 10^{-7} moles/L. The neutral point is temperature-dependent and is pH 7.5 at 0°C and pH 6.5 at 60°C.
- The pH value of a highly dilute solution is approximately the same as the negative common logarithm of the hydrogen ion concentration. Natural waters usually have pH values in the range of 4 to 9, and most are slightly basic because of the presence of bicarbonates and carbonates of the alkali and alkaline earth metals.

9.2. Apparatus:

9.2.1. pH meter:

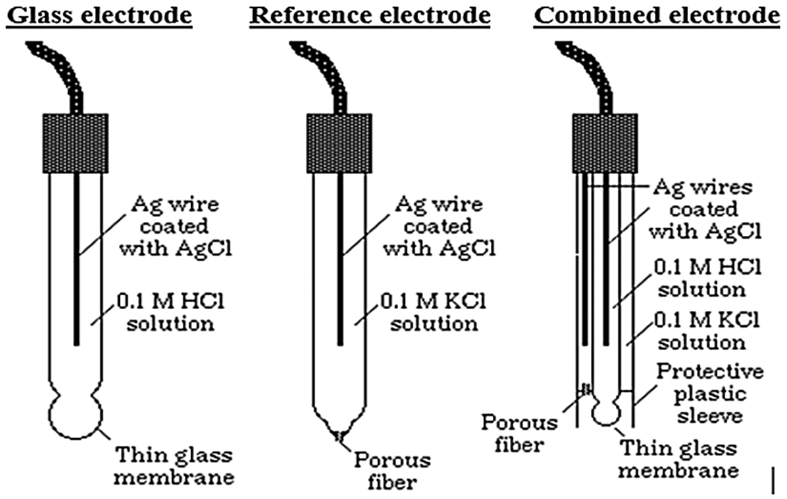
- consisting of potentiometer, a glass electrode, a reference electrode, and a temperature-compensating device. electrodes are immersed in the test solution.
- Many pH meters are capable of reading pH or mill volts and some have scale expansion that permits reading to 0.001 pH unit, but for routine work use a pH meter accurate and reproducible to 0.01 pH unit with a range of 0 to 14 and equipped with a temperature-compensation adjustment.

9.2.2. Reference electrode:

- consisting of a half cell that provides a constant electrode potential. Commonly used are calomel and silver/silver chloride electrodes. Either is available with several types of liquid junctions.

9.2.3. Glass electrode:

- The sensor electrode is a bulb of special glass containing a fixed concentration of HCl or a buffered chloride solution in contact with an internal reference electrode.



9.2.4. Reagents:

- Deionized water its conductivity less than 2 µmhos/cm.
- Standard buffer solutions of known pH prepare fresh as needed for accurate work at 25°C. or a set of buffer solution ready to use its values 4, 7 and 10.

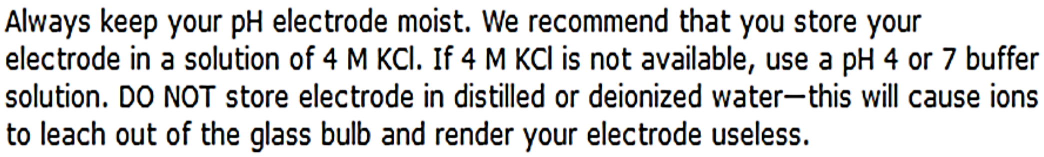
9.3. Procedure:

9.3.1. Instrument calibration:

- In each case follow manufacturer's instructions for pH meter calibration and preparation of electrodes for use.
- Before use, remove electrodes from storage solution, rinse, blot dry with a soft tissue, place in initial buffer solution, and set the isopotential point.
- Select a second buffer within 2 pH units of sample pH and bring sample and buffer to same temperature, 25°C, or the temperature of a fresh sample.
- Remove electrodes from first buffer, rinse thoroughly with distilled water, blot dry, and immerse in second buffer.
- Record temperature of measurement and adjust temperature dial on meter so that meter indicates pH value of buffer at test temperature (this is a slope adjustment).
- Use the pH value listed in the tables for the buffer used at the test temperature. Remove electrodes from second buffer, rinse thoroughly with distilled water and dry electrodes as indicated above.
- Immerse in a third buffer below pH 10, approximately 3 pH units different from the second; the reading should be within 0.1 units for the pH of the third buffer.
- If the meter response shows a difference greater than 0.1 pH unit from expected value, look for trouble with the electrodes or potentiometer.

9.3.2. Storage:

- Recommended solutions for short-term storage of electrodes vary with type of electrode and manufacturer.
- Keep electrodes wet by returning them to storage solution whenever pH meter is not in use.



Always keep your pH electrode moist. We recommend that you store your electrode in a solution of 4 M KCl. If 4 M KCl is not available, use a pH 4 or 7 buffer solution. DO NOT store electrode in distilled or deionized water—this will cause ions to leach out of the glass bulb and render your electrode useless.

Note:

Standardize the instrument before each measurement.

9.4. Sample analysis:

- Establish equilibrium between electrodes and sample by stirring sample to insure homogeneity; stir gently to minimize carbon dioxide entrainment.
- For buffered samples or those of high ionic strength, condition electrodes after cleaning by dipping them into sample for 1 min. dry, immerse in a fresh portion of the same sample, and read pH.
- With dilute, poorly buffered solutions, equilibrate electrodes by immersing in three or four successive portions of sample. Take a fresh sample to measure pH.

9.5. Calculations:

- Sample pH is read directly from the meter display.

9.6. References:

- Standard Methods 23rd Edition,2017. 4500-pH-B

10. Alkalinity

10.1. Principle:

- Alkalinity is a measure of water's ability to neutralize acids. In most types of water, alkalinity is the result of bicarbonates $(HCO_3)^{-1}$, carbonates $(CO_3)^{-2}$ and hydroxides (OH–) of the metals calcium, magnesium, and sodium.
- Alkalinity is expressed as total alkalinity, mg/l as calcium carbonate ($CaCO_3$).
- When $CaCO_3$ dissolves in water, the carbonate (CO_3^{2-}) can react with water to form bicarbonate (HCO3-), which produces hydroxide (OH-):
    - ➢ $CaCO_3 (s) \leftrightarrow Ca_2+ + CO_3^{2-}$
    - ➢ $CO_3^{2-} + H_2O \leftrightarrow HCO_3^- + OH^-$
- This alkalinity test provides results for use in controlling water treatment processes. Many chemicals used in treating water can change its alkalinity; the pronounced changes are caused by coagulants and softening chemicals.
- The results of this test are used to calculate chemical dosages needed in the coagulation and softening processes. Also, total alkalinity must be known for the Calcium Carbonate Saturation Test.

10.2. Interference:

- Soaps, oily matter, suspended solids, or precipitates may coat the glass electrode and cause a sluggish response. Allow additional time between titrant additions to let electrode come to equilibrium or clean the electrodes occasionally.
- Do not filter, dilute, concentrate, or alter sample.

10.3. Equipment:

- 50-ml volumetric pipette to measure the sample volume.
- 250-ml conical flasks.
- Dropping pipettes or medicine droppers of 0.05–1.00 ml for dispensing sodium thiosulfate ($Na_2S_2O_3.5H_2O$) and indicator solutions.
- Sensitive balance.
- 50-ml burette

10.4. Chemicals and reagents:

10.4.1. Sodium carbonate standard solution 0.05N

- Dry 3 to 5 g primary standard Na_2CO_3 at 250°C for 4 h and cool in a desiccator. Weigh 2.5 ± 0.2 g (to the nearest mg), transfer to 1L volumetric flask, fill flask to the mark with distilled water, and dissolve and mix reagent. (Do not keep longer than 1 week).
- 1 ml = 1.00 mg $CaCO_3$.
  - M.Wt of Na_2CO_3 =106 gm/mole
  - Eq.Wt of Na_2CO_3 =M.Wt/(2x1)=53gm
  - Wt(gm)=NxEq.WtxV(ml)/1000 = .05x53x1000/1000
  - =2.65 gm of Na_2CO_3 to be dissolved then diluted to 1000 ml with pure water to form 0.05 N Na_2CO_3.

10.4.2. Standard sulfuric acid 0.1N

- As indicated under *Preparation of Desk Reagents in the Standard Methods For the Examination of Water and Wastewater*, prepare sulfuric acid solution of approximate normality by cautiously adding 2.8 ml of concentrated sulfuric acids of ACS-grade (specific gravity = 1.834–1.836 at (20/4oC) & purity of 96-98 %), with mixing, to designated volume of proper type of distilled water. Dilute to 1000 mL and mix thoroughly.

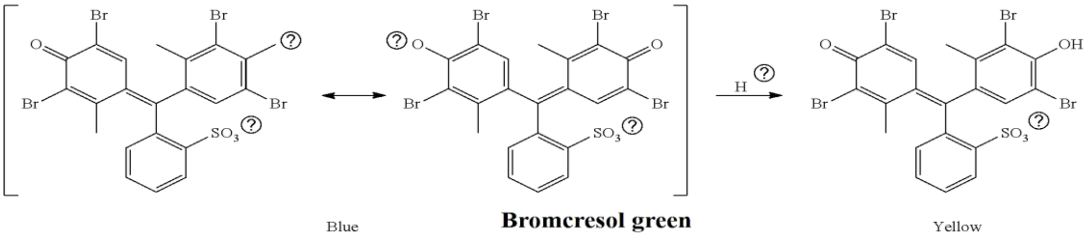
- Standardize against 50.00 ml 0.05N Na2CO3 solution using color indicator.
- Calculate the normality of acid:
  - Where:
    - $(N\,x\,V\,)H2SO4 \;=\; (N\,x\,V\,)Na2CO3$
    - $(\,?\;\;x\,50ml)H2SO4 \;=\; (\;0.05\;\;\;x\;Reading\;of\;burette\,)Na2CO3$
    - $(N)\;H2SO4 \;=\; \frac{0.05x\;Reading\;of\;burette}{50\;ml}$
  - Use measured normality in calculations or adjust to 0.1N:
  - 1 ml 0.1 N solution = 5.00mg $CaCO_3$.

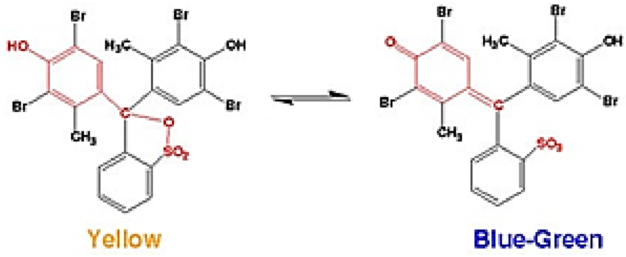
10.4.3. Standard sulfuric acid, 0.02N

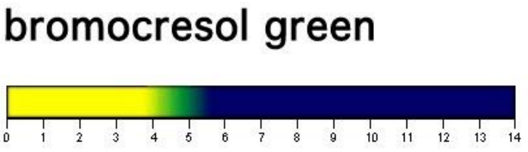
- Dilute 200.00 ml 0.100 N standard acid to 1000 ml with distilled or deionized water. (1ml = 1mg $CaCO_3$).

10.4.4. Bromocresol green indicator solution, pH 4.5 indicator

- Dissolve 100 mg Bromcresol Green, sodium salt, in 100 ml distilled water.







10.4.5. Mixed bromcresol green-methyl red indicator solution, pH 4.5 indicator

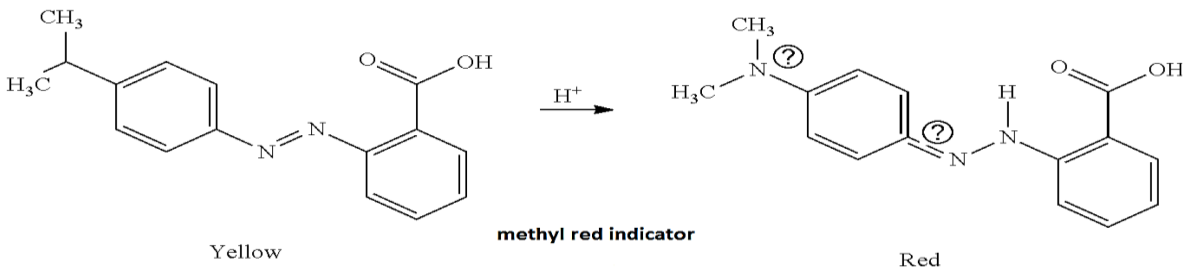
- Use either the aqueous or the alcoholic solution
- <u>**aqueous solution:**</u>
  - ➢ Dissolve 100 mg bromcresol green sodium salt and 20 mg methyl red sodium salt in100 ml distilled water.
- <u>**alcoholic solution:**</u>
  - ➢ Dissolve 100 mg bromcresol green and 20 mg methyl red in 100 ml 95% ethyl alcohol or isopropyl alcohol.

10.4.6. Bromcresol green indicator solution, pH 4.5 indicator:

- Dissolve 100 mg bromcresol green, sodium salt, in 100 mL distilled water

10.4.7. Phenolphthalein solution, alcoholic, pH 8.3 indicator

- Dissolve 5 mg Phenolphthalein in1L distilled water.
- Note: Methyl Orange indicator is not recommended in Alkalinity megerments



methyl red indicator

10.4.8. Sodium thiosulfate, 0.1N

- Dissolve 25g $Na_2S_2O_3.5H_2O$ in 1L distilled water.

10.5. Procedure:

10.5.1. Standardization of 0.02 N Sulfuric Acid

- Fill the burette with 0.02 N Sulfuric Acid.
- Place 10ml standard Sodium Carbonate solution in a 250 ml flask. Add 40ml distilled water to the flask.
- Also prepare a color comparison blank by placing 50 ml distilled water in 250ml flask.
- Add Bromcresol green indicator to each flask and mix.
- For the color comparison use a blank.
- Add 0.02N sulfuric Acid from the burette drop by drop, with continuous stirring, until the greenish color appears.
- Titrate the standard sodium carbonate solution by adding of 0.02N sulfuric acid slowly, with continuous stirring, until greenish color appears as the color comparison blank. Record the new reading.
- Calculate the net volume of titrant used by subtracting the initial reading (step 1) from the final reading (step 7).
- Calculate the blank correction by subtracting the burette reading in (step 1) from (step 5).
- Calculate the total volume of titrant used for the standard sodium carbonate solution alone by subtracting the result found in (step 8) from the result found in (step 9).

- Calculate mg Sodium carbonate equivalent to 1ml 0.02 N sulfuric acid as follows:
  - mg of $NaCO_3$ equivalent to 1 ml 0.02 N sulfuric acid = 10/total ml of 0.02 N sulfuric acid used to titrate 10ml standard Sodium Carbonate solution.

10.5.2. Analysis:

- Select sample size that use a sufficiently large volume of titrant (20ml or more from a 50ml burette) to obtain relatively good volumetric precision while keeping sample volume sufficiently small to permit sharp end points. Adjust sample to room temperature, if necessary, and with a pipette discharge sample into an Erlenmeyer flask.
- If free residual chlorine is present add 0.05ml (1 drop) 0.1M $Na_2S_2O_3$ solutions.
- Add 0.2ml (5 drops) of indicator solution and titrate over a white surface to a characteristic change of the end point.
- Check color at end point by adding the same concentration of indicator used with sample to a buffer solution at the designated pH.
- Titrate with sulfuric acid (0.02N):
- For total alkalinity (alkalinity to pH 4.5): use the mixed bromcresol green-methyl red indicator, the color turns from the bluish green color to colorless,
- While for phenolphthalein alkalinity (alkalinity to pH 8.3) the pink color of phenolphthalein turns to colorless.

10.6. Calculation:

$$(N * V)H2SO4 \ = \ (N * V) \ Sample$$

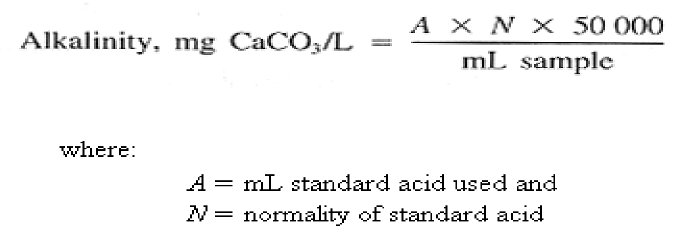
$$(N * V)H2SO4 \ / \ (V) \ Sample \ = \ (N) \ Sample$$

or

$$(N) \ Sample \ = \ (N * V)H2SO4 \ /(V) \ Sample$$

- But we know that Strength(S) in g/L = N*Eq.Wt
- So $\boldsymbol{Strength(S) \ Sample \ in \ g \ CaCO3/L \ = \ (N) \ Sample \ * \ Eq.Wt \ CaCO3}$
  - Sample Alkalinity in mg $CaCO_3$/L=1000*Eq.Wt $CaCO_3$*(N*V)H_2SO_4/(V) Sample
  - Sample Alkalinity in mg $CaCO3/L = 1000 * 50 * (N * V)H2SO4/(V)$ Sample
  - Sample Alkalinity mg $CaCO_3$/L=50000*(N*V) H_2SO_4/(V) $_{\text{Sample}}$
  - Sample Alkalinity as mg $CaCO_3$/L=50000*(N*V) H_2SO_4/(V) $_{\text{Sample}}$
- **Example:**
  - $(N \ x \ V \ )H2SO4 \ \ = \ \ (N \ x \ V \ )CaCO3$

- (0.02 x Reading of burette) H_2SO_4 = (? x 50ml) CaCO3
    - $(N)CaCO3 = \frac{0.02x\ Reading\ of\ burette}{50\ ml}$
- $CaCO3\ (g/L)\ \frac{= 0.02\ x\ 100/2\ x\ Reading\ of\ burette}{50\ ml}$
- $CaCO3\ (mg\ /L) = \frac{0.02\ x\ 50\ x\ 1000\ x\ Reading\ of\ burette}{50\ ml}$
- $CaCO3\ (mg\ /L) = 0.02x\ 1000\ x\ Reading\ of\ burette$
- $Ca\ CO3\ (mg\ /L) = 20\ x\ Reading\ of\ burette$



$$\text{Alkalinity, mg CaCO}_3\text{/L} = \frac{A \times N \times 50\,000}{\text{mL sample}}$$

where:

A = mL standard acid used and
N = normality of standard acid

10.7. Calculation of alkalinity relationships:

- The results obtained from the phenolphthalein and total alkalinity determinations offer a means for stoichiometric classification of the three principal forms of alkalinity present in many waters. The classification ascribes the entire alkalinity to bicarbonate, carbonate, and hydroxide, and assumes the absence of other (weak) inorganic or organic acids, such as silica, phosphoric, and boric acids.
- If further presupposes the incompatibility of hydroxide and bicarbonate alkalinities. Because the calculations are made on a stoichiometric basis, ion concentrations in the strictest sense are not represented in the results, which may differ significantly from actual concentrations especially at pH > 10.

According to this table:

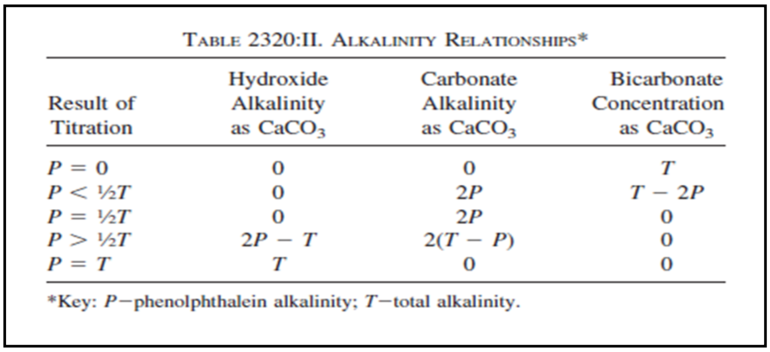


TABLE 2320:II. ALKALINITY RELATIONSHIPS*

Result of Titration	Hydroxide Alkalinity as $CaCO_3$	Carbonate Alkalinity as $CaCO_3$	Bicarbonate Concentration as $CaCO_3$
$P = 0$	0	0	T
$P < ½T$	0	$2P$	$T - 2P$
$P = ½T$	0	$2P$	0
$P > ½T$	$2P - T$	$2(T - P)$	0
$P = T$	T	0	0

*Key: P—phenolphthalein alkalinity; T—total alkalinity.

10.8. Reporting

- The results reported as mg/l $CaCO_3$.
- Number of significant figures is two.

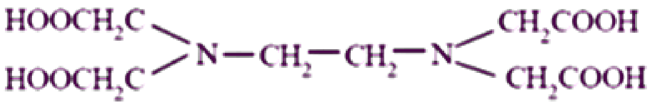
10.9. References

- Standard Methods 23rd Edition,2017. 2320-B ALKALINITY.

11. Total Hardness

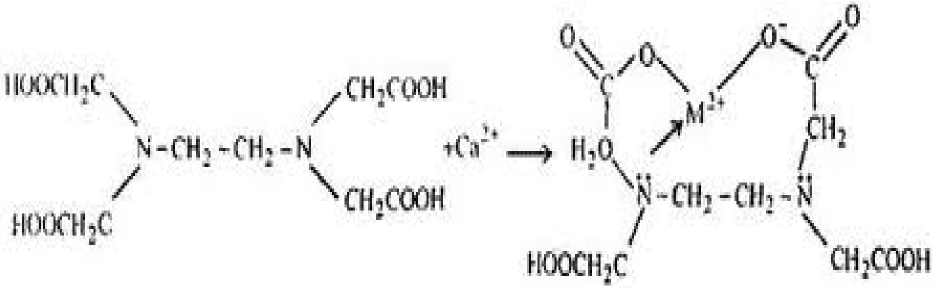
11.1. Principle:

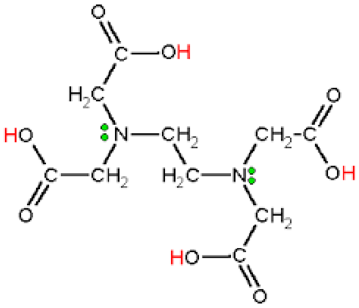
- Originally, water hardness was understood to be a measure of the capacity of water to precipitate soap. Calcium and magnesium in water cause hardness.
- Total hardness is defined as the sum of calcium and magnesium concentrations, both expressed as calcium carbonate in mg/l.
- The ethylene di amine tetra acetic acid (EDTA) titrimetric method can be used to determine total hardness or calcium hardness. This method is designed for the routine determination of hardness in drinking water.
- The ions involved in water hardness, i.e. Ca^{2+}(aq) and Mg^{2+}(aq), can be determined by titration with a chelating agent, ethylene di amine tetra acetic acid (EDTA), usually in the form of disodium salt (H^2Y^{2-}). The titration reaction is:

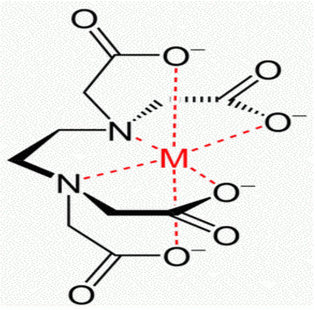


EDTA

$$Ca^{2+}(aq) + H_2Y^{2-}(aq) \longrightarrow CaY^{2-}(aq) + 2H^+(aq)$$







- Ethylene Diamine Tetra Acetic acid and its sodium salts (EDTA) form a chelated soluble complex when added to a solution of certain metal cations.

- If small amount of dye such as Eriochrome black T (EBT) is added to an aqueous solution containing calcium and magnesium ions at ph of 10.0 ±0.1, the solution becomes wine red.
- If EDTA is added as titrant, the calcium and magnesium will be complexes and the solution turns from wine red to blue, marking the end point of the titrant.
- Magnesium ion must be present to yield a satisfactory end point. To insure this small amount of complex metrically neutral magnesium salt of EDTA is added to the buffer. This automatically introduces sufficient magnesium and eliminates the need for blank correction.
- When hardness numerically is greater than the sum of carbonate and bicarbonate alkalinity, that amount of hardness equivalent to the total alkalinity is called "Carbonate hardness(temporary) ", the amount of hardness in excess of this is called ' Non carbonate hardness(permanent).
- When the hardness numerically is equal to or less than the sum of carbonate and bicarbonate alkalinity, all hardness is carbonate hardness and non-carbonate hardness is absent.

11.2. Interference

- Suspended or colloidal organic matter may interfere with the end point. Eliminate this interference by evaporating the sample to dryness on a steam bath and heating in a muffle furnace at 550°C until the organic matter is completely oxidized. Dissolve the residue in 20 ml in HCl, neutralize to pH 7 with NaOH and make up to 50 ml with distilled water. Cool to room temperature and continue according to the general procedure.
- The specific pH may produce an environment conductive to $CaCO_3$ precipitation. Although the titrant slowly re-dissolves such precipitates, a drifting end point often yields low results. Completion of the titration within 5 minutes minimizes the tendency for $CaCO_3$ to precipitate. The following three methods also reduce precipitation loss:
- Dilution of sample to reduce $CaCO_3$ concentration
- If the approximate hardness is known or is determined by a preliminary titration. Add 90% or more of titrant to sample before adjusting pH with buffer.
- Acidify sample and stir for 2 minutes to expel CO_2 before pH adjustment. Determine alkalinity to indicate amount of acid to be added.

11.3. Equipment

- Analytical Balance
- Cylinder 100ml.
- Measuring flasks (1000ml, 2000ml).
- Conical flasks 250ml
- Pipettes: 10ml graduated: 50ml volumetric pipette.
- Burette

11.4. Chemicals and reagents

11.4.1. Buffer Solution

- Dissolve 16.9 g ammonium chloride (NH_4Cl) in 143ml conc ammonium hydroxide (NH_4OH). Add 1.25 g magnesium salt of EDTA (available commercially) and dilute to 250ml with distilled water.
- If the magnesium salt of EDTA is unavailable, dissolve 1.179 g disodium salt of Ethylene Diamine Tetra Acetic acid dihydrate (analytical reagent grade) and 780 mg magnesium sulfate ($MgSO_4.7H_2O$) or 644mg magnesium chloride ($MgCl_2.6H_2O$) in 50 ml distilled water. Add this solution to 16.9 g NH_4Cl and 143 ml conc NH_4OH with mixing and dilute to 250ml with distilled water. Store Solution (1) or (2) in a plastic or borosilicate glass container for no longer than 1 month. Stopper tightly to prevent loss of ammonia (NH_3) or pickup of carbon dioxide (CO_2). Dispense buffer solution by means of a bulb-operated pipette. Discard buffer when 1 or 2 ml added to the sample fails to produce a pH of 10.0 ± 0.1 at the titration end point.

11.4.2. Indicator

- Eriochrome black T(EBT): dissolve 0.5 g dye in 100 g 2,2',2''-nitrilotriethanol(also called triethanolamine) or 2-methoxymethanol (also called ethylene glycol monomethyl ether).

or

- Eriochrome black T: Mix 0.2 g dye and 500 g NaCl and grind well.

11.4.3. Standard Calcium Solution

- Weigh 1.0 gm anhydrous $CaCO_3$ powder (primary standard or special reagent low in heavy metals, alkalis, and magnesium) into 500 ml Erlenmeyer flask, Place a funnel in the flask neck and add a little at a time 1: 1 HCl until all $CaCO_3$ has dissolved. Add 200 ml distilled or milliQ water and boil for a few minutes to expel CO_2.

- Cool, add a few drops of methyl red indicator then adjust to the intermediate orange color by adding 3N NH_4OH or 1:1 HCl as required.
- Transfer quantitatively and dilute to 1000 ml with distilled water; (1ml= 1mg $CaCO_3$).

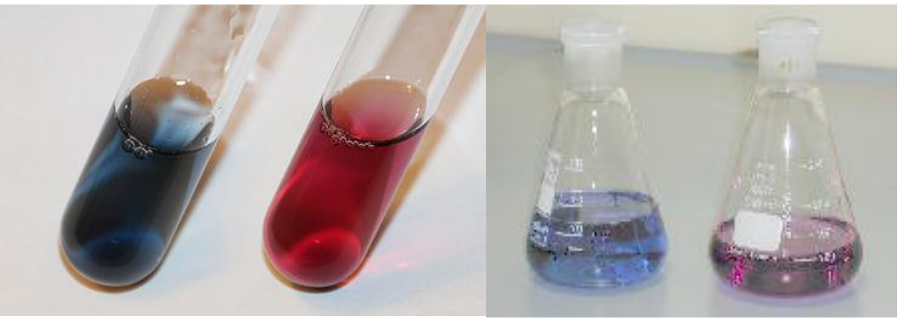
11.4.4. Standard EDTA titrant 0.01M

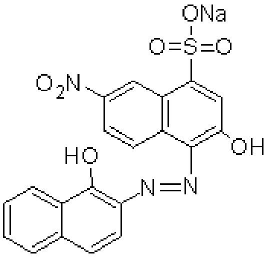
- Dissolve 3.723 gm of EDTA (Ethylene Diamine Tetra Acetic acid disodium salt) in distilled water and complete to 1000 ml. Store in polyethylene or borosilicate glass bottle.
- Standardize against standard calcium solution as follow:
- Dilute 10.0 ml of $CaCO_3$ to about 50 ml with distilled water in a conical flask.
- Add 1 to 2 ml ammonium buffer solution (Usually 1 ml will be sufficient to give a pH of 10.0 to 10.1),
- Add an appropriate amount of dry-powder indicator.
- Complete titration within 5 min, measured from time of buffer addition.
- Add standard EDTA titrant slowly, with continuous stirring, until the last reddish tinge disappears.
- Add the last few drops at 3- to 5-s intervals. At the end point the solution normally is blue. Calculate the Correction factor.

11.4.5. Procedure:

- Pretreatment of polluted water and wastewater samples(If necessary):
- Use nitric acid-sulfuric acid or nitric acid-perchloric acid digestion (Section 3030 SMWW).
- Titration of sample: Select a sample volume that requires less than 15 ml EDTA titrant and complete titration within 5 min, measured from time of buffer addition.
- Dilute 25.0 ml sample to about 50 ml with distilled water in a porcelain casserole or other suitable vessel. Add 1 to 2 ml buffer solution and an appropriate amount of indicator. Add standard EDTA titrant slowly, with continuous stirring, until the last reddish tinge disappears. Add the last few drops at 3- to 5-s intervals. At the end point the solution normally is blue.

- If sufficient sample is available and interference is absent, improve accuracy by increasing sample size.





ل Eriochrome Black T (sodium salt)

11.4.6. Calculation

$$\frac{(M1xV1)}{n1} Titrant = \frac{(M2xV2)}{n2} Titrate$$

where n is the number of moles in reaction equation

M=Molarity V =volume

and since n1 =n2 so they will be neglected…

$$(M_1xV_1)_{EDTA} = (M_2xV_2)_{SAMPLE}$$

$$(M_2)_{SAMPLE} = \frac{(M_1xV_1)_{EDTA}}{V_{2\ sample}}$$

$$(\text{Hardness as mg } CaCO3\ /L)\ SAMPLE = \frac{(M1xV1)\ EDTA\ xM.Wtx1000}{V2\ sample}$$

But we know that $Strength(S)\ (gm/L)\ or\ (mg/ml) = M * M.Wt$

$$(Hardness\ \ as\ mg\ CaCO3\ /L)\ SAMPLE = \frac{(V1x\ S)\ EDTA\ x1000}{V2\ sample}$$

Or as from the standard method:

Hardness (EDTA) as mg $CaCO_3$ /L = $\frac{A*B*1000}{mLsample}$

Where:

A= ml titration for sample

B= mg $CaCO_3$ equvilent to 1 ml EDTA titrant

Or

$$(\textbf{Hardness as mg CaCO3 /L) SAMPLE} = \frac{\textbf{(M1xV1) EDTA xM.Wtx1000}}{\textbf{V2 sample}}$$

$$(\textbf{Hardness as mg CaCO3 /L) SAMPLE} = \frac{\textbf{(0.01 xCF xV1) EDTA x100x1000}}{\textbf{50 sample}}$$

$\textbf{Total Hardness} = \textbf{ml of EDTA x 20xCF}$ (if 50ml of sample is used)

11.4.7. Reporting

- Total hardness expressed as mg/l $CaCO_3$ for two decimals.

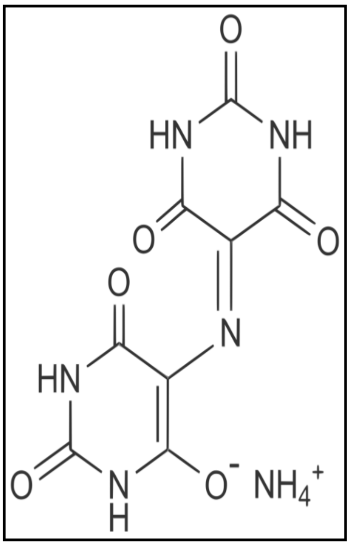
11.4.8. References

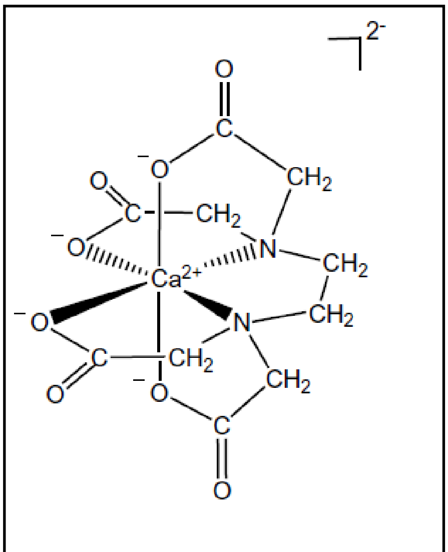
- Standard Methods 23rd Edition, 2017. 2340 C. EDTA Titrimetric Method

12. Calcium Hardness by EDTA titrimetric method

12.1. Principle:

- When EDTA (Ethylene Diamine Tetra Acetic acid or its salts) is added to water containing both calcium and magnesium, it combines first with the calcium. Calcium can be determined directly, with EDTA, when the pH is made sufficiently high that the magnesium is largely precipitated as the hydroxide and an indicator is used that combines with calcium only.
- Several indicators give a color change when all of the calcium has been complexes by the EDTA at a pH of 12 to 13 . Murexide (with Ca) pink color at pH 12:13 and after addition of EDTA, Ca-EDTA are formed and Murexide being alone at the range of this PH appearing a purple color.





Complex formed by EDTA and calcium ions

12.2. Interference:

- Orthophosphate precipitates calcium at the pH of the test. Strontium and barium give a positive interference.
- Alkalinity in excess of 300 mg/l may cause an indistinct end point in hard water so the sample must be diluted.

12.3. Apparatus

- Erlenmeyer flask, 250-ml
- Burette, 50-ml.

12.4. Reagent

- NaOH (1N) .Dissolve 40 gm in distilled. Water and dilute to L. by distilled Water

12.4.1. Indicator

- (presence in the stable form with NaCl) Mix 200 mg Murexide with 100 g NaCl and grinding it well.

NOTES:

- Titrate immediately after adding indicator because it is unstable under alkaline condition.
- Preparing color comparison blank contain 2.0 ml NaOH + 0.2 g indicator & only drop or two from EDTA to produce unchanged color

12.4.2. Standard EDTA (0.01M):

- As shown in TH

12.4.3. Standard Calcium Solution:

- As shown in TH

12.4.4. Standardization of (0.01 M) EDTA with Calcium Solution:

- As shown in TH

12.5. Procedure:

12.5.1. Sample preparation:

- Because of the high pH used in this procedure; titrate immediately after adding alkali & indicator and use the sample volume that give about 5 to 10 mg Ca content.
- Analyze hard waters with alkalinity higher than 300 mg $CaCO_3$/L by taking a smaller portion and diluting to 50ml.

12.5.2. Titration:

- Add 2.0 ml NaOH solution or volume sufficient to produce pH of 12 to 13. Stirring by adding 0.1 to 0.2 g indicator, then add EDTA titrant slowly, with continuous stirring to proper end point.
- When using Murexide add 1 or 2 drop of EDTA to make certain that no change in color occurs.

12.5.3. Calculation

$$\text{Hardness As mg } CaCO_3/L = \frac{\text{ml Of EDTA} \times \text{M.of EDTA} \times \text{M.wt of } CaCO_3}{\text{ml Of Sample}} \times 1000$$

$$\text{mg CaCO}_3\ /\ \text{l} = \frac{\text{ml EDTA} \times 0.01 \times 100 \times 1000}{\text{ml of sample}}$$

$$\textbf{mg Ca / l} = \textbf{mg CaCO}_3\textbf{/l} \times \textbf{0.4008}$$

12.6. Reference:

- Standard Methods 23rd Edition, 3500B Calcium HARDNESS EDTA Titrimetric Method.

13. Determination of Magnesium Hardness By Calculation

13.1. Equation

$$Mg\ hardness\ (mg/l) = Total\ hardness - Ca\ hardness$$

$$mg\ Mg/l = mg\ CaO_3/L\ x\ 0.24$$

13.2. Reference:

- Standard Methods 23rd Edition.2017. 3500-Mg B. Calculation Method.

14. Chloride

14.1. General discussion:

- Chloride, in the form of chloride (Cl–) ion, is one of the major inorganic anions in water. The salty taste produced by chloride concentrations is variable and dependent on the chemical composition of water. Some waters containing 250 mg Cl–/L may have a detectable salty taste if the cation is sodium.
- For example, when a solution of sodium chloride reacts with silver nitrate, the balanced equation can be written in complete form as

$$NaCl(aq) + AgNO3(aq) \rightarrow AgCl(s) + NaNO3(aq)$$

We can break each dissolved ionic compound into cations and anions to give

$$\boldsymbol{Na + (aq) + Cl - (aq) + Ag + (aq) + NO3 - (aq) \rightarrow AgCl(s) + Na + (aq) + NO3 - (aq)}$$

Note that the insoluble precipitate, AgCl, is not broken down into separate ions, since it is not disolved. The sodium and nitrate ions are spectators to the precipitation reaction between silver and chloride ions. If we remove the spectator ions, the net ionic equation can be written

$$\boldsymbol{Cl - (aq) + Ag + (aq) \rightarrow AgCl(s)}$$

- The reaction taking place during titration of chloride

$$Ag^{+} + Cl^{-} \rightarrow AgCl(s)$$

$$2Ag^{+} + CrO_4^{2-} \leftrightarrow \mathbf{Ag_2CrO_4}(s)$$

14.1.1. Principle:

- In neutral & slightly alkaline solution pot.Chromate can indicate the end point of the silver nitrate titration to chloride; silver chloride is precipitate quantitatively forming white precipitated before red silver chromate is formed

14.1.2. Interference:

- Substances in amounts normally found in potable waters will not interfere.
- Bromide, iodide, and cyanide register as equivalent chloride concentrations. Sulfide, thiosulfate, and sulfite ions interfere but can be removed by treatment with hydrogen peroxide. Orthophosphate in excess of 25 mg/L interferes by precipitating as silver phosphate. Iron in excess of 10 mg/L interferes by masking the end point.

14.2. Apparatus:

- Conical flask 250ml
- Burette 50ml

14.3. Reagent:

14.3.1. Standard Silver nitrate titrant (0.0141N):

- Silver nitrate M.Wt. = 169.87 g/mol
- Silver nitrate Eq.Wt. = M.Wt./1= 169.87 g
- Wt(gm)=N*Eq.Wt*V(ml)/1000
- Wt(gm)= 0.0141*169.87 *1000/1000
- Wt(gm)= 2.395

- Dissolve 2.395 g AgNO3 in distilled water and dilute to 1000 ml. Standardize against NaCl 0.0141 N solution.

14.3.2. Standard sodium chloride:

- To prepare 500 ppm Cl:

NaCl-----------------Cl

23+35.5=58.5 35.5 gm

????????? 500mg/L

?????????= 500(mg/L)*58.5~~gm~~/35.5~~gm~~

?????????= 500(mg/L)*58.5~~gm~~/35.5~~gm~~ =823.9437 mg

- Dissolve 824.0 mg NaCl (dried at 140°C) in distilled water and dilute to 1000 ml; 1.00 ml = 500 µg Cl–.

14.3.3. Potassium chromate indicator solution:

- Dissolve 50gm K2CrO4 in a little distilled water and add AgNO3 solution until a definite red precipitate is formed. Let stand 12h, filter and dilute to 1 liter with distilled water.

14.4. Procedure:

14.4.1. Silver nitrate standardization:

- Standardize silver nitrate standard using sodium chloride NaCl (0.0141N) using K2CrO4 indicator and then calculate the correction factor (CF).

14.4.2. Analysis:

- Fill the burette with standard silver nitrate.
- Place 50ml of sample in a suitable Erlenmeyer flask
- Add 1.0 ml K_2CrO_4 indicator solution to the sample.
- Titrate against $AgNO_3$ (0.0141N) until the appearance of a pinkish yellow color.
- Read the burette and record the volume of silver nitrate used.
- Standardize $AgNO_3$ titrant and establish reagent blank value by the titration method outlined above. A blank of 0.2 to 0.3 mL is usual.

14.4.3. Calculation:

$$(mg\ Cl-/L)\ SAMPLE\ =\frac{(N1xV1)\ AgNO3\ xEq.Wtx1000}{\text{V2 sample}}$$

$$(mg\ Cl-/L)\ SAMPLE\ =\frac{(0.0141xCF\ xV1)\ AgNO3\ x35.45x1000}{\text{50 sample}}$$

$$(mg\ Cl-/L)\ SAMPLE\ =\ ml\ of\ AgNO3\ x\ 10xCF\ \ (if\ 50ml\ of\ sample\ is\ used)$$

or

$$\text{mg Cl}-/\text{l}\quad=\ (\text{A}-\text{B})\ \text{x CF x 10}$$

- Where:
  - A = ml titration for sample.
  - B = ml titration for blank, and,
  - CF = correction factor of silver nitrate standardization.

14.4.4. Reporting:

- Results are reported as mg/l Cl^-

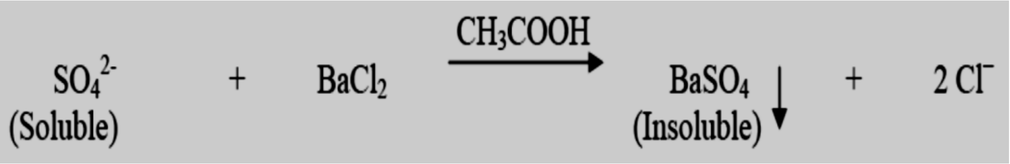
14.5. Reference

- Standard Methods 23rd Edition.2017. 4500-Cl^- B. Argentometric Method

15. Sulfate (4500- SO_4^{2-} E)

15.1. Principle:

- Sulfate ion (SO_4^{2-}) is precipitated in an acetic acid medium with barium chloride ($BaCl_2$) so as to form barium sulfate ($BaSO_4$) crystals of uniform size. Light Scattered is measured by a turbidity meter and the SO_4^{2-} concentration is determined by comparison of the reading with a standard curve.



$$\underset{\text{(Soluble)}}{SO_4^{2-}} + BaCl_2 \xrightarrow{CH_3COOH} \underset{\text{(Insoluble)}}{BaSO_4}\downarrow + 2\,Cl^-$$

15.2. Interference:

- Color or suspended matter in large amounts will interfere. Some suspended matter may be removed by filtration. Silica in excess of 500 mg/L will interfere, and in waters containing large quantities of organic material it may not be possible to precipitate BaSO4 satisfactorily.
- In potable waters there are no ions other than SO42– that will form insoluble compounds with barium under strongly acid conditions.

15.3. Equipment:

- Glassware
- Beakers 250ml
- Graduated pipettes 10ml.
- Magnetic Stirrer: provides a 400-rpm stirring speed.
- Tubidimeter.
- Stopwatch or electric timer.

15.4. Chemicals and reagents:

15.4.1. Buffer solution A:

- Dissolve 30g magnesium chloride, $MgCl_2.6H_2O$, 5g sodium acetate, CH3COONa.3H_2O, 1.0g potassium nitrate, KNO_3, and 20ml acetic acid, CH3COOH (99%), in 500 ml distilled water and make up to 1000 ml.

15.4.2. Buffer solution B:

- ***(required when the sample SO42- concentration is less than 10 mg/L)***:

- Dissolve 30g magnesium chloride, $MgCl_2.6H_2O$, 5g sodium acetate, $CH_3COONa.3H_2O$, 1.0g potassium nitrate, KNO_3, 0.111 gm sodium sulfate and 20ml acetic acid, CH3COOH (99%), in 500 ml distilled water and make up to 1000 ml.

15.5. Barium chloride:

- $BaCl_2$ crystals. In standardization, uniform turbidity is produced and the appropriate buffer.

15.5.1. Standard sulfate solution

- **Preparation of standard sulfate solution from sodium sulfate**
  - 1 ml = 100 µg SO_4^{2-}.
  - Dissolve 0.1479 g anhydrous Na_2SO_4 in distilled water and dilute to 1000 ml.
- **Preparation from stock standard**
  - Dilute directly from stock standard 1000mg/l sulfate.
- **Standard sulfuric acid 0.1N**
  - As indicated under *Preparation of Desk Reagents in the Standard Methods For the Examination of Water and Wastewater*, prepare sulfuric acid solution of approximate normality by cautiously adding 2.8 ml of concentrated sulfuric acids of ACS-grade (specific gravity = 1.834–1.836 at (20/4°C) & purity of 96-98 %), with mixing, to designated volume of proper type of distilled water. Dilute to 1000 mL and mix thoroughly.

15.6. Procedure:

15.6.1. Formation of barium sulfate turbidity

- Measure 100 ml sample or a suitable portion made up to 100 ml, into a 250-ml Erlenmeyer flask. Add 20 ml buffer solution and mix in stirring apparatus. While stirring, add 0.2gm of $BaCl_2$ crystals and begin timing immediately. Stir for 60±2 s at constant speed.

15.6.2. Measurement of barium sulfate turbidity:

- After stirring period has ended, pour solution into absorption cell of Turbidimeter or spectrophotometer and measure turbidity at 5±0.5 min.

15.6.3. Preparation of calibration curve

- Estimate SO_4 concentration in sample by comparing turbidity reading with a calibration curve prepared by carrying SO_4^{2-} standards through the entire procedure. Space standards at 5mg/l increments in the 0 to 40mg/l SO_4^{2-} range. Above 40mg/L accuracy decreases and $BaSO_4$ suspensions lose stability. Check reliability of calibration curve by running a standard with every three or four samples.

15.6.4. Correction for sample color and turbidity

- Correct for sample color and turbidity by subtracting turbidity of sample (when using Buffer-A) to which $BaCl_2$ is not added.

15.7. Calculations:

- The result is taken directly from the calibration curve.

15.8. Reporting:

- The results of this method expressed as mg/l SO4^{2-}
- Number of significant figures is two

15.9. References

- Standard Methods 23rd Edition.2017. 4500-SO_4 SULFATE E. Turbidimetric Method.

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