NATIONAL DRINKING WATER MISSION

(Department of Rural Development, Government of India)

OPERATIONAL MAN UAL

for

WATER QUALITY TESTING LABORATORIES

MAY 1990

CONTENTS

I

SAFETY PRECAUTIONS IN LABORATORY

1.1 Precautions with Hazardous Chemicals:

- All containers must be clearly labeled and read before opening. If dispensing into another contained, put label alongwith warning.

- Minimal stocks not exceeding 500 ml of corrosive or flammable solvents only may be kept in work room. Keep in safe place in plastic covered metal cabinet.

- Glacial acetic acid must be regarded as a flammable solvent.

- Ether and low boiling point flammable liquids must not be kept in fridge.

- Large container of corrosive or flammable liquids should never be put on high shelves or where they can be knocked down or fall. Never put liquids that react violently together closely.

- Never carry bottles by neck alone. Open bottles with care.

- When diluting concentrated sulphuric acid or other strong acids, it should be added to water in heat resistant vessel.

1.2 Spillage of Hazardous Chemicals

- If amount is small, dilute with water or detergent. It amount is large protective apron, rubber gloves and boots are worn and treatment carried out according to wall chart showing how to manage chemical spillage.

- Hydrochloric acid and sulphuric acid can be neutralized with anhydrous sodium carbonate then shovelled into a plastic bucket which is subsequently diluted by water and run to waste. Ammonia solution, ethanol, methanol and formalin are best treated by diluting with water, collection and running to waste. Windows must be opened. Phenols must be diluted with atleast 20 times the volume of tap water before running to waste.

1.3 First Aid

The First Chart should be mounted on a nearby wall. The first aid box must be accessible with Laboratory Staff. An emergency eye wash bottle with a bottle of sterilised water should be readily available. A Universal *poison antidote* is useful. Activated aluminium oxide or a tin of evaporated milk should be readily available. A tin opener and some water proof dressing material should also be readily available.

1.4 Avoidance of Hazards of Equipment

- Trained staff should operate the equipments.

- Operating instructions should be followed.

- Check the autoclave filled with water to correct level before loading.

- Water or moisture on electrical fittings is dangerous.

- If fire breaks out, near electrical equipment should immediately be switched off and disconnected.

- Take care to avoid live wires.

- When not in use switch off and withdraw plug from socket.

- Avoid contact with wires as may think ‘dead’ but if wrongly connected may be ‘live’.

- Avoid use of multi-adaptors, if have to use it, must be fitted with fuses.

1.5 Fire extinguishers

- Water extinguishers are suitable for fires involving ordinary combustible materials e.g. wood, paper, textile, upholstery. Never use on electrical fires or liquids that will catch fire.

- Dry powder extinguishers or sand are suitable for liquids on fire, electrical fires and burning metals. CO_2 extinguishers suitable for all types of fire particularly for minimising damage to laboratory equipment.

II

DO's AND DON'T IN LABORATORY

- NEVER EAT, SMOKE OR DRINK IN LABORATORY

- NEVER MOUTH PIPETTE

- WASH HANDS BEFORE STARTIANG WORK, PREFERABLY WEAR A CLEAN LABORATORY COAT.

- REGULARLY CLEAN WORKING AREA WITH DISINFECTANT.

- USE ONLY MEDIA AND EQUIPMENT KNOWN TO BE STERILE.

- NEVER TOUCH PART OF PIPETTE TUBES ETC., WHICH WILL COME IN CONTACT WITH STERILE SAMPLE.

- OPEN TUBES OR PETRI DISHES FOR MINIMUM TIME.

- ALL CULTURES TUBES, PETRI DISHES SHOULD BE AUTOCLAVED BEFORE DISPOSAL OR RELEASE FOR WASHING UP.

- ALL USED PIPETTES MUST BE DISCARDED BY IMMERSING IN DISINFECTANT (e.g. > SAVLON) JARS FILLED TO ALLOW ALMOST COMPLETE IMMERSION. CHANGE DISINFECTANTLY DAILY.

- AVOID SPLASHING INFECTIOUS MATERIAL CREATING AEROSOLS OR DISPERSING INFECTIOUS MATERIAL INTO THE ENVRONMENT.

III

SAMPLING

3.1 It is well recognized that the result of any test procedure can be no better than the sample on which it is performed. The objective of sampling is to collect a portion of material small enough in volume which can be transported easily and handed in the laboratory while still accurately representing the material being collected.

3.2 The sampling programme defines the portion of the whole to which rest results apply. The variations of the whole material with respect to time, area, depth, and occasionally, rate of flow must be taken into consideration to ensure representative nature of the sample. It is not possible to specify detailed procedure for collection of all types of samples. However, a general guidelines as suggested below may be followed:

3.3 **General Guidelines and Precautions**

- Collect a sample that conforms to the requirements of the sampling programme and handle it carefully so that it does not deteriorate or get contaminated during its transport to the laboratory. Before filling the container rinse it two or three times with the water being collected. Representative samples of some sources can be obtained only be making composites of samples collected over a period of time or at a number of different sampling points.

- While collecting a sample from the distribution system, flush lines adequately, taking into consideration the diameter and length of the pipe to be flushed and the velocity of flow.

- Collect samples from tube-wells only after sufficient pumping to ensure that the sample represents the ground water source.

- When samples are to be collected from a river or stream, analytical results may vary with depth, flow, distance from the shore and from one shore to the other. If equipments are available for collection of "integrated" sample from top to be bottom in the middle of the stream and composite the sample according to the flow, Grab or catch sample should be collected in the middle of the stream and at mid depth.

- Since lakes and reservoirs are generally undergo considerable variation due to normal natural causes, choose location, depth and frequency of sampling depending on local conditions and the objective of the study.

- Make detailed record of every sample collected and identify each contained by attaching a tag or label. Record information like date, time and exact location, weather condition, stream-flow etc.

3.4 Quality of Sample to be Collected

Normally a 2-litre sample would be sufficient for most physical and chemical tests. Never use the sample for chemical, bacteriological and microscopic examinations, because collection and test procedures for the latter tests are different.

3.5 Time Interval between Collection and Analysis.

The interval between collection and analysis of the sample should be shortest possible.

IV

RECORDING AND REPORTING OF DATA

4.1 The District Laboratory shall keep records of submitted samples and completed analysis in a manner that provides for the data retrievability, the sample preservation and the persons responsible for the sampling and analysis. All laboratory data sheets shall be dated and signed by the analysts and the Head of the Laboratory or his designee.

4.2 Electrical conductivity, pH, temperature, and turbidity should be recorded in units specified for the respective tests and the results of other chemical examinations shall be expressed in terms of substances or ions actually determined and reported as milligram per litre, which may be considered for all practical purposes as equivalent to parts per million (ppm).

4.3 A commonly used and functional method of recording laboratory data is a note book specifically printed for this purpose. The pages are serially numbered in pairs with a carbon between the pages to provide matching serial numbered copy of the data. These books are permanently bound but the duplicate page is performated for easy removal. The duplicate page may then be filed in a system wherefrom it may be readily retrieved. The pages of the note book are generally lined in a grid pattern with provision for such information as project identification, data, reference to the analytical procedure, the observations, sample calculations, as mg/1 and signature of the analyst.

4.4 **Analytical Quality Control**

The basic objective of a water analysis laboratory is to produce accurate data describing the physical-chemical characteristics of water samples under study. Quality assurance is the total programme for assuring the reliability of analytical data. Items discussed in this document can all be considered as contributing to the overall programme of quality assurance. Another essential component of a quality assurance is analytical quality control which refers to the routine application of procedures for controlling the measurement process.

4.4 Internal quality control or statistical quality control is the most important component of any laboratory quality control programme. Experience indicates that 10-20% of the resources of a laboratory should be devoted to such work. Suitable approaches to internal quality control should be followed.

4.5 External quality control is best applied after incorporating internal quality control practices in the laboratory, and consists in the periodic analysis of reference samples. These reference samples may not be different from the control samples which the laboratory has been preparing for its own use, with the exception that the amount of each substance present is unknown to the analysts.

V

PROCEDURE FOR ANALYSIS OF VARIOUS PARAMETERS

5.1 Colour

The platinum cobalt method of measuring colour is the standard method, the unit of colour being that produced by 1 mg platinum/ litre in the form of chloroplatinate ion. It is applicable to measure the colour of potable water and of water in which colour is due to naturally occurring materials.

Even a slight turbidity effects the colour. Remove turbidity by centrifugation for 1 hours. As colour varies with pH, specify the pH at which colour is measures.

Field Method

As the platinum-cobalt standard method is not convenient for field use, compare water colour with that of glass discs held at the end of metallic tubes containing glass comparator tubes filled with sample and colourless distilled water. Match sample colour with the colour of the tube of clear water plus the caliberated coloured glass when viewed by looking towards a white surface.

Collect representative samples in clean glassware and make the colour determination within a reasonable period.

Appratus

a) Nessler tubes, 50 ml, tall form

b) pH – meter

Preparation of standards

Dissolve 1.246 g potassium chloroplatinate, K_2PtCl_6 (equivalent to 500 mg metallic Pt) and 1.00 g. crystallized $CoCl_2{}_2O$ (equivalent to about 250 mg metallic Co) in distilled water with 100 ml conc. HCl and dilute to 1000 ml distilled water. This stock standard has a colour of 500 units.

Prepare standards having colours of 5, 10, 15, 20,…….70 by diluting the stock colour standard with distilled water to 50 ml in Nessler tubes.

Procedure

Observe sample colour by filling a matched Nessler tube, to the ml mark with sample and comparing it with standards. Looking vertically downwards through tubes towards a while surface placed at such an angle that light is reflected upward through the columns of liquid. If turbidity is present, report as apparent colour. If value exceeds 70, dilute sample according so that value is within 70.

Measure pH of the sample

Report nearest to 1 if value is 1-50, nearest to 5 for 51-100, to 10 for 101-250 & to 20 for 251-500.

5.2 Hydrogen ion Concentration (pH)

Purpose

It is one of the most important and useful tests in the control of water.

Many chemical and biochemical reactions like place at a certain pH value or within narrow pH range. Control of pH is particularly important in the chemical cogaulation of water, softening etc.

Theory

The pH of a solution denotes the intensity of the acidity or alkalinity of a solution and is defined by the relationship:

$PH + - \log_{10} C_H$

Where C_H = *the concentration of Hydrogen ions in gm. Ion per letre.*

The pH scale runs from 0.0 to 14 with 7.0 being neutral. Many surface water have pH between 6.0 to 8.5.

The pH can be measured by the indicator method and by the electerometric method using a pH meter. The equipments needed include colour standards covering the pH range desired, indicator solutions corresponding to the colour standards, a comparator designed to facilitate the matching of the colour and special test tubes to hold the test sample. The electrometric method requires a pH metere which is a sensitive instrument.

Procedure

In this method the pH metere is first calibrated against a solution of known pH and the test well is then filled with the sample. The pH is read directly either from the scale on the instrument of digital display as the case may be. This method is most accurate and almost free from interferences. It is imperative to follow strictly the manufacturer's direction.

5.3 Turbidity

Suspension of particles in water interfering with passage of light is called turbidity. Turbid waters are undesirable from aesthetic point of view in drinking water supplies and may also affect products in industries. Turbid water also possess a number of problems in water treatment plants. Turbidity is measured to evaluate the performance of water treatment plants.

Principle

The method is based on a comparison of the intensity of light scattered by a sample and a standard reference under the same conditions. Higher the intensity of scattered light higher the turbidity.

Interference

Colour is the main source of interference the measurement of turbidity.

Appratus

Turbidimeter or Klett Summerson.

Reagents

1. *Solution I:* Dissolve 1.0 g hydrazine sulfats and dilute to 100 ml.

2. *Solution II:* Dissolve 10.g hexamethylene-tetramine and dilute to 100 ml.

3. Mix 5 ml of I with 5 ml of II. Allow to stand for 24 hrs. and dilute to 1000 ml. This will have turbidity of 400 units.

4. *Standard turbidity suspension:* Dilute 100 ml of solution III as prepared above to 100 ml too have turbidity of 40 units.

Procedure

1. Prepare calibration curves in the rage 0-400 unit by carrying out appropriate dilutions of solutions III and IV above and taking readings on turbidimeter.

2. Take sample or a suitably diluted aliquot and determine its turbidity either by visual comparison with the diluted standards or by reading on turbidimeter.

3. Read turbidity from the standard curves and apply correction due to dilution, if necessary.

4. Report the readings in turbidity unit.

5.4 Conductivity

Conductivity is a numerical expression of the ability of an aqueous solution to carry an electric current. This ability depends upon the presence of ions, their total concentration, mobility, valence and relative concentrations and on the temperature of measurement.

Apparatus

1. A good conductivity meter with cell.
2. Thermometer

Reagents

1. Conductivity water with conductivity less than 1 mho/ cm.

2. Standard potassium chloride solution (0.01 N). Dissolve 745.6 mg anhydrons KC1 in conductivity water and dilute to 1000 ml at 25^{o}C. This has a conductivity of 1413 mhos/ cm at 25^{o}C.

Procedure

Rinse conductivity cell with atleast three portions of 0.01 N KC1 solution. Adjust temperature of a fourth portion to 25.0 $\pm$ 0.1^{o}C. Measure conductivity which should be 1413 mhos/ cm. If not adjust the meter to read this value using the cell constant knob. Note the cell constant.

Rinse cell with one or more portions of sample. Adjust temperature of a final portion to 25.0 $\pm$ 0.1^{o}C. Read the conductivity value and note the temperature.

5.5 Hardness

Hardness in water causes scale formation in boilers. It is also objectionable from view point of water use for laundry and domestic purposes since it consumes a large quantity of soap. Generally, salts of Ca and Mg contribute hardness to natural waters. Hardness may be classified either as (1) carbonate and non-carbonate hardness or (2) calcium and magnesium hardness or (3) temporary and permanent hardness.

Principle

In alkaline condition EDTA reacts with Ca and Mg to form a soluble chelated complex. Ca and Mg ions develop wine red colour with eriochrome black 'T' under alkaline condition. When EDTA is added as a titrant the Ca and Mg divalent ions get complexed resulting in sharp change from wine read to blue which indicates end point of the fitration. The pH for this titration has to be maintained at 10.0 ± 0.1. At a higher pH, i.e., about 12.0 Mg ion precipitates and only Ca ion remains in solution. At this pH Murex indicator form a pink colour with Ca^{++} When Edta is added Ca^{++} gets complexed resulting in a change from pink to purple which indicates end point of the reaction.

Interference

Metal ions do interfere but lkexan be overcome by addition of inhibitors.

Reagents

1. *Buffer solution:* Dissolve 16.9 g NH_4 Cl in 143 NH_4OH. Add 1.25 g EDTA Mg salt to obtain sharp change in indicator and dilute to 250 ml.

This has to be titrated with standard calcium solution to avoid interference produced by addition of EDAT to the buffer.

2. *Inhibitor:* Dissolve 4.5 g hydroxyl amine hydrochloride in 100 ml 95% ethyl alcohol.

3. *Erichrome black T Indicator:* Mix 0.5 dye with 100g NaCl to prepare dry powder.

4. Murex indicator: Dry powder.

5. Sodium hydroxide 2N: dissolve 80 g NaOh and dilute to 1000 ml.

6. *Standard* EDTA *solution* 0.01 M: Dissolve 3.723 g EDTA sodium salt and dilute to 1000 ml. Standardize against standard Ca solution, 1 ml = 1 mg $CaCO_3$.

7. *Standard calcium solution:* Weigh accurately 1.0 g AR grade $CaCO_3$ and transfer to 250 ml conical flask. Place a funnel in the neck of a flask and add 1+1 HCL till $CaCO_3$ dissolves completely. Add 200 ml distilled water and boil for 20-3- min. to expel CO_2 Cool and methyl red indicator. Ad NH_4OH 3N dropwise till intermediate orange colour develops. Dilute to 1000 ml to obtain 1 ml – 1 mg $CaCO_3$.

Procedure

A. **Total Hardness**

1. Take 25 or 50 ml well mixed sample in porcelain dish or conical flask.

2. Add 1-2 ml buffer solution followed by 1 ml inhibitor.

3. Add a pinch of Erichrome black T and titrate with standard EDTA (0.01 M) till wine red colour changes to blue. Note down to vol. Of EDTA required. (A)

4. Run a reagent blank if buffer is not checked properly. Note the vol. Of EDTA or Ca required by blank (b).

5. Calculate vol. of EDTA required by sampe, C= (A-B) from vol. of EDTA required in steps 3 & 4.

6. Calculate as follows:

$$\text{Total hardness mg/l as } CaCO_3 = \frac{C \times D^3 \times 1000}{ml \text{ of sample}}$$

where

C = Vol. of EDTA required by sample.

D = mg $CaCO_3$ – per 1.0 ml EDTA 0.01 M used as titrant.

B. **Calcium Hardness**

1. Take 25 or 30 ml sample in a porcelain dish.

2. Add 1 ml NaOh to raise pH to 12.0 and a pinch of indicator.

3. Titrate with EDTA till pink colour changes to purple. Note the vol. of EDTA used (A').

4. Calculate as follows:

Calcium hardness mg/l as $CaCO_3 = \frac{A^1 \times D^3 \times 1000}{ml\ of\ Sample}$

where

C = Vol. of EDTA required by sample.

D = mg $CaCO_3$ – per 1.0 ml EDTA 0.01 M used as titrant.

Magnesium hardness = Total hardness – Ca hardness.

5.6 Chloride

Purpose

The chloride estimation has two different purposes.

1. If the test is done regularly on a water supply and there is a sudden increase, it may indicate pollution due to ingress of sewerage or other chloride rich wastes.

2. Many ground waters have chloride content high enough to be of objectionable taste. By using the chloride test, the well with the lowest amount of chloride can be identified. If several well are being pumped, it can be planned in such a way that the lowest chloride content is obtained.

Principle

The Mohr method for the determination of chloride in water is based upon the fact that in solution containing chloride and chromate, silver reacts with all the chloride and precipitates before the reaction with chromate begins. The appearance of the brick-red colour of the silver chromate precipitate is the end-point of the titration.

Reagents

Potassium chromate indicator, 5% solution, standard silver nitrate solution 0.0282 N (1 ml = 1 mg of chloride).

Procedure

1. Take 100 ml of sample and adjust the pH between 7.0 and 8.0

2. Take 50 ml of well mixed sample adjusted to pH 7.0 – 8.0 and add 1 ml of potassium chromate indicator.

3. Titrate with standard silver nitrate solution till the colour changes from yellow to brick-red.

4. Titrate 50 ml of distilled water in the same way after adding 1 ml of potassium chromate indicator to establish reagent blank.

Calculation

$$\text{Chloride, mg/l as cl} = \frac{(A - B) \times 1000}{Ml\ of\ Sample}$$

Where

A = ml of $AgNO_3$ required for sample

B = ml of $AgNO_3$ required for blank.

5.7 **Iron**

Presence of iron causes nuisance in public water supplied. It occurs both in soluble and insoluble forms. When the iron is present in considerable amounts in water it imparts colour and also develops turbidity when exposed to air due to its conversion to ferric state. Hence, the water becomes unacceptable for drinking purposes from an aesthetic point of

view. Further, it interferes with laundering operation, imparts objectionable stains, causes difficulties in the distribution systems and imparts taste even at low concentrations (0.3 mg/l).

Principle

The ferric form of iron is reduced to ferrous form by boiling with HC1 and hydroxylamine hydrochloride. Later phenanthroline is added at pH between 3.2 and 3.3 to form soluble chelated complex of orange red colour with iron. The colour obeys Beer's Law and the intensity of colour is independent of pH between 3 and 9.

Interference

Stron oxidizing agents such as CN, NO_2 , polyphosphates, Cr, Zn in Conc. 10 times the Fe conc; Co and Cu if 5 mg/l, Ni if 2 mg/l colour and organic matter constitutes sources of interference in the development of colours.

Boiling with HCl and addition of hydroxylamine remove interferences due to CN, NO_2, PO_4 and other oxidizaing reagents. The metal ions get complexed with phenanthroline.

Apparatus

1. Colorimeter with an operating range of 400-700 mu.

2. Nessler's tubes.

Reagents

1. HCl cone.
2. Hydroloxylamine soloution: Dissolve 10g NH_2 OH HCl and dilute to 100 ml.
3. Ammonium acetate buffer: Dissolve 250 g $NH_4C_2H_3O_2$ in 50 ml distilled water. Add 700 ml conc. Glacial acetic acid. Final volume will be slightly more than 1000 ml.
4. Phenanthrolone solution: Dissolve 100 mg/l: 10 phenanthroline monobydrate in 100 ml distilled water warm slightly or add 2 drops cone. HCl if necessary. 1 ml of this colution can chelate 100 mg iron.
5. Stock iron solution: Add 20 ml conc. H_2SO_4 to 50 ml distilled water and dissolve 1.404 g Fe $(NH_4)_2 (SO_4)_2 6H_2O$. Add drop-wise 1 N KM nO_4 till faint pink colour persists. Dilute to 1000 ml. 1 ml = 200 mg Fe.
6. Standard iron solution: Dilute 50 ml stock Fe solution to 1000 ml. Prepare this solution freshly. 1 ml = 10 mg Fe.

Procedure

1. Take suitable aliquot (about 50 ml having 2 mg/l Fe) of well mixed sample in 125 ml conical flask.
2. Add 2 ml conc. HCl followed by 1 ml hydroxyl amine solution.
3. Add2-3 glass beads and boil for 20-25 min. to ensure dissolution of Fe.
4. Cool to room temp. and transfer to Nessler's tubes.
5. Add 10 ml amm. acetate buffer and 2 ml phenanthroline solution.
6. Dilute to 100 ml and mix well.
7. Prepare blank by substituting the sample by water.
8. For soluble iron determination, take known vol. of filtered sample, acidify by adding 2 ml. Conc. HCl per 100 ml of sample and treat from step 5 onwards for colour development.
9. Prepare calibration curve taking standard iron solution in the same way in the range 1000-4000 mg/l with 1 cm light path.
10. Measure the developed colour after 10 mn. at 510 mu.
11. Calculate the concon. of total or soluble Fe present in the sample from caliberation curve and express as mg/l.

5.8. **Fluoride**

Fluoride ions have dual significance in water supplied. Excess concentration of F causes dental fluorosis (disfigurement of the teeth).

At the same time, a concentration less than 0.8 mg/l results in `dental caries'. Hence, it is essential to maintain the F conc. Between 0.8 to 1.0 mg/l in drinking water.

Principle

Under acid condition fluorides (HF) react with zirconium SPADNS solution and the lake (colour of SPADNS reagent) gets bleached due to formation of ZRF_6. Since bleaching is a function of fluoride ions, it is directly proportional to the conc. of F. It obeys Beer's Law in a reverse manner.

Interference

Alkalinity 5000 mg/l, aluminium 0.1 mg/l, chloride 7000 mg/l. Fe 10 mg/l, PO_4 16 mg/l, SO4 200 mg/l and hexametaphosphate 1.0 mg/l interfere in the bleaching action. Residual Cl2 interferes in bleaching action. In presence of interfering radicals distillation of sample is recommended.

Apparatus

1. Colorimeter for use at 570 M/U.
2. Nessler's tubes cap. 100 ml.

Reagents

1. SPADNS solution: Dissolve 958 mg SPADNS and dilute to 500 ml.

2. *Zirconyl acid ragent*: Disslove 133 mg $ZrOCL_2$. $8H_2O$ in 25 ml water. Add 350 ml conc. HCl and dilute to 500 ml.
3. Mix equal volume of 1 and 2 to produce a single reagent. Protect from direct light.
4. *Reference solution*: Add 10 ml SPADNS solution to 100 ml distilled water. Dilute 7 ml conc. HCl to 10 ml and add to diluted SPANDS solution.
5. *Sodium arsenite solution*: Dissolve 15.0 g $NaASo_2$ and dilute to 1000 ml.
6. *Stock* F *solution:* Dissolve 221.0 mg anhydrous NaF and dilute to 1000 ml. 1 ml = 100 mg F.
7. *Standard* F: Dilute stock solution 10 times to obtain 1 ml = 10 mgF.

Procedure

1. Prepare standard curve in the range 0.0 to 1.40 mg/l by diluting appropriate volume of standard F solution to 50 ml in Nessler's tubes.
2. Add 10.0 ml mixed reagent prepared as in 3 above to all the samples, mix well and read optical density to bleached colour at 570 mu using reference solution for setting zero absorbance.
3. Plot cone. vs % transmission.
4. If sample contains residual chlorine remove it by adding $NaAsO_2$ solution 1 drop (o.05 ml = o.1 mg Cl) $NaAsO_2$ conc. should not exceed 1300 mg/l to avoid error due to $NaAsO_2$.
5. Take suitable aliquots of sample and dilute to 50 ml in Nessler's tubes.
6. Add mix reagent 10 ml; mix well and read % transmission.
7. Calculate the mg F present in the sample using standard curve.
8. Calculate F mg/l as follows:

$$= \frac{mg\ F\ in\ aliquot \times 1000}{ml\ of\ Sample}$$

5.9 **Residual Chlorine**

A. **Iodometric method**

This method is suitable for measuring total chlorine concentration greater than 1.0 mg/l.

Reagents

Glacil acetic acid, potassium iodide crystals, 0.01 N sodium thiosulphate solution, starch indicator.

Procedure

1. Add about 1 gm. of potassium iodide to each of the bottle followed by 10 ml of acetic acid; stopper the bottle and mix.

2. Titrate released iodine with standard thiosulphate solution using starch as indicator. Note the volume required for each bottle.

Calculation

$$\text{Residual Chlorine mg/l} = \frac{\textit{ml of Thiosulphate x N x 35.5 x 1000}}{\textit{ml of Sample}}$$

where

N = Normality of the thiosulphate solution.

B. **Orthotolidine Method Principle**

Orthotolidine is an aromatic organic compound which reacts with both free and combined in acid solution to form a yellow coloured compound, the intensity of the colour being directly proportional to the concentration of chlorine in the sample. The orthotolidine reacts instantaneously with free chlorine but its reaction with combined chlorine is rather slow. The method is more sensitive to lower concentrations of chlorine and is affected by temperature and contact time. The maximum and minimum concentration of residual chlorine that can be measured by this method is 10 mg/l and 0.01 mg/l respectively.

If it is desired to identify both free and combined residual chlorine and also to find out if any additional colour has been produced by interfering substances present in the sample, the Orthotolidine arsanite method should be employed.

Apparatus

1. Chlorine comparator.

Reagents

Orthotolidine Reagent and Sodium arsenite Reagent.

Procedure

1. Take 0.5 ml orthotolidine reagent in the 10 ml cell supplied with the equipment and add sample upto the mark and mix thoroughly.
2. Compare the colour developed immediately (within 10 seconds). The colour developed is that for free residual chlorine.
3. Compare the coklour again exactly after 5 minutes. The colour developed includes both for free combined chlorine.
4. Compare both the colours with that of the standards supplied with the equipment and express in results in mg/l chlorine.
5. The combined residual chlorine – Total residual –free residual.

5.10 **Bacteriological**

The following is the test method done as a routine for bacteriological analysis of water.

Presumptive coliform count (a quantitative test for all coliform bacilli/100ml)

Description of Presumptive Coliform Count method

The coliform count takes notices of only those organisms which are gram-negative, nonsporing aerobic or facultative anaerobic, red shaped micro organisms capable of fermenting lactose with the production of acid and gas within 48 hours at 37^{o}C.

Procedure

An estimation of the number of coliform bacilli in water supply is usually made by adding varying quantities of the water (from 50 ml to 0.1 ml) to MacConkey's broth or in a chemically defined medium contained in the bottle and test tubes with Durham's tubes to show the formation of gas. Depending on the quality of the water, the samples may be inoculated as follows:

	With Good & Medium Quality (filtered or chlorinated tubewell)			Poor Quality		All Qualities	
Media(MacConkey's broth)	50mlx1	10mlx5	5mlx5	10mlx5	5mlx5	5mlx5	10mlx5
Strength of Media	D.S.	D.S.	D.S.	D.S.	S.S.	S.S.	D.S.
Water	50ml	10mlx5	1mlx5	10mlx5	1mlx5	0.1mlx5	10mlx5
Incubation	37^{o}C (48 hours)			37^{o}C (48 hours)		37^{o}C (48 hours)	

(D.S. = Double strength, S.S. = Single strength)

Very bad water may required use of 1 in 100 or in 1000 or higher dilutions. For a relatively poor quality of water 5 tubes method may be used. In this method five 1 ml quantities each to 5 ml single strength MacConkey's broth are inoculated for 48 hours at 37^{o}C and results are recorded from probable tables.

Results

Those that show acid and sufficient gas to fill the concavity at the tope of the Durhan tube are considered to be "presumptive positive" as a result of growth of coliform bacilli.

Reporting Results

The results are reported in terms of the 'presumptive' number of coliform bacilli per 100 ml (i.e. MPN – Most Probable Number/100 ml) by computation from tables complied by McCrady.

MONTHLY REPORTING OF FUNCTIONS OF DISTRICT LAB

State :_____ Month :_____

1. Name of District :

   Mini Mission District :

   Other District:

2. Name of the Incharge of Laboratory:

3. No. of Samples received:

4. No. of Samples analysed:

   Physical & Chemical :

   Biological :

5. No. of Samples analysed:
   (Physical & Chemicals)

   For all parameters :

   For specific prameters :

6. Results of the samples for specific problem:

   No. of samples indicating Excess Iron :

   No. of samples indicating Excess Fluoride :

   No. of samples indicating Brackishness:

   No. of samples for any other problem:

   No. of samples for monitoring of existing
   DF/Fe/Deslination Plants:

7. Reporting of the results sent to:

   CE on date:

   District Medical Officer on date:

   Executive Engineer date:

   Regional Centre on date:

Reporting Channel

Incharge of laboratory will send one copy of above report to:

i. Executive Engineer, PHED
ii. District Health Officer
iii. Chief Engineer, PHED

and send 2 copies to their Regional Centre. The Regional Centre will give their own comments and forward it to DRD, New Delhi for compiling the data on National basis.

VII

LIST OF DOCUMENTS

The following documents are to be maintained in each district laboratory :

a) Report on collection and codification of samples

b) Proforma for test report

c) Report on number of samples tested

d) Statement of recurring expenditure for operation of laboratory

e) Stock register of consumables

f) Stock register of equipment/furniture

g) Attendance register of the staff

NATIONAL DRINKING WATER MISSION

EXPORT ON COLLECTION AND CODIFICATION OF SAMPLES

STATE: **DISTRICT LAB**
AT:

Sl. No.	Name of Source	Date of Collection	Date of Receipt in Lab.	Quantity of Sample(Ltrs)	Mode of Transport	Parameters Required	Code No. Allocated	Remarl
1.	2.	3.	4.	5.	6.	7.	8.	9.

NATIONAL DRINKING MISSION

PROFORMA FOR WATER ANALYSIS REPORTS (TEST REPORTS)

STATE:_____ DISTRICT LAB AT:_____

1. DATE OF EXAMINATION :

2. SAMPLE CODE :

3. PURPOSE OF TEST REPORT (PARAMETERS REQUIRED) :

Sl.No.	PARAMETERS	VALUE
1.	2.	3.
i)	PH	
ii)	TEMPERATURE ($_{O}C$) COLOUR	
iii)	TURBIDITY	
iv)	CONDUCTIVITY	
v)	TDS	
vi)	TOTAL HARDNESS	
vii)	ALKALINITY, as $CaCO_3$	
viii)	IRON	
ix)	FLOURIDE	
x)	RESIDUAL CHLORINE	
xi)	PRESENCE OF COLIFORM	
xii)	ANY OTHER	

REMARKS:

SIGNATURE

INCHARGE LABORATORY

NATIONAL DRINKING MISSION

DAILY REPORT ON NUMBER OF SAMPLES TESTED
(DATE:_____)

STATE:_____ DISTRICT LABORATORY

AT:_____

SL.NO.	SAMPLE CODE	DATE OF COLLECTION OF SAMPLE	TYPE OF ANALYSIS CARRIED OUT	DATE OF ISSUE OF TEST REPORT	TYPE OF PROBLEM IDENTIFIED	REMARKS
1.	2.	3.	4.	5.	6.	7.

NATIONAL DRINKING WATER MISSION

MONTHLY STATEMENT OF RECURRING EXPENDITURE FOR OPERATION OF LABORATORY AND TESTING COST

FOR THE MONTH OF:_____

STATE:_____ DISTRICT LABORATORY AT:_____

Sl.No.	ITEM	AMOUNT (Rs.)	REMARKS
1.	2.	3.	4.
1.	SALARY, WAGES ETC.		
2.	TRAVEL, TOUR ETC.		
3.	PURCAHSE OF CONSUMABLES i) CHEMICALS ii) GLASSWARE iii) OTHERS		
4.	MAINTENANCE EXPENSES i) EQUIPMENT ii) BUILDING iii) TRANSPORT ETC.		
5.	COST OF UTILITIES (POWER, WATER, FUEL ETC.)		
6.	MISCELLANEOUS		
7.	SUB-TOTAL OF ITEMS 1-6		
8.	NUMBER OF SAMPLE TESTED		
9.	COST OF ANALYSIS OF ONE SAMPLE ITEM 7/8		

NATIONAL DRINKING WATER MISSION

STOCK REGISTER FOR EQUIPMENT FURNITURE

STATE:_____ DISTRICT LABORATORY AT:_____

(Separate sheet for each equipment/furniture)

1. Name of the Item :

2. Brief specification :

3. Invoice No. and date of receipt :

4. Status :

5. If damaged, state action taken for repairs :

6. If irrepairable, mention date of condemnation :

7. Indenting for its replacement with date :

8. Invoice No. and date of receipt :

9. Brief description of new piece :

NATIONAL DRINKING WATER MISSION

ATTENDANCE SHEET OF THE STAFF OF LABORATORY

FOR THE MONTH OF:_____
DISTRICT LABORATORY AT:_____

STATE:_____

Sl. No.	NAME	DESIGNATION	1	2	3	4	5	6	7	8	9	10	11	12	13
			14	15	16	17	18	19	20	21	22	23	24	25	26
			27	28	29	30	31								
1.															
2.															
3.															
4.															
5.															
6.															
7.															
8.															
9.															
10.															

NATIONAL DRINKING WATER MISSION

ATTENDANCE SHEET OF THE STAFF OF LABORATORY

FOR THE MONTH OF:_____
DISTRICT LABORATORY AT:_____

STATE:_____

Sl. No.	NAME	DESIGNATION	1	2	3	4	5	6	7	8	9	10	11	12	13
			14	15	16	17	18	19	20	21	22	23	24	25	26
			27	28	29	30	31								
1.															
2.															
3.															
4.															
5.															
6.															
7.															
8.															
9.															
10.															