

**GENERAL STANDARD**

**FOR**

**WATER POLLUTION CONTROL**

**ORIGINAL EDITION**

**DEC. 1997**

**This standard specification is reviewed and updated by the relevant technical committee on Aug. 2005(1) and Feb. 2013(2). The approved modifications are included in the present issue of IPS.**

**FOREWORD**

The Iranian Petroleum Standards (IPS) reflect the views of the Iranian Ministry of Petroleum and are intended for use in the oil and gas production facilities, oil refineries, chemical and petrochemical plants, gas handling and processing installations and other such facilities.

IPS are based on internationally acceptable standards and include selections from the items stipulated in the referenced standards. They are also supplemented by additional requirements and/or modifications based on the experience acquired by the Iranian Petroleum Industry and the local market availability. The options which are not specified in the text of the standards are itemized in data sheet/s, so that, the user can select his appropriate preferences therein.

The IPS standards are therefore expected to be sufficiently flexible so that the users can adapt these standards to their requirements. However, they may not cover every requirement of each project. For such cases, an addendum to IPS Standard shall be prepared by the user which elaborates the particular requirements of the user. This addendum together with the relevant IPS shall form the job specification for the specific project or work.

The IPS is reviewed and up-dated approximately every five years. Each standards are subject to amendment or withdrawal, if required, thus the latest edition of IPS shall be applicable

The users of IPS are therefore requested to send their views and comments, including any addendum prepared for particular cases to the following address. These comments and recommendations will be reviewed by the relevant technical committee and in case of approval will be incorporated in the next revision of the standard.

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**GENERAL DEFINITIONS**

Throughout this Standard the following definitions shall apply.

**COMPANY :**

Refers to one of the related and/or affiliated companies of the Iranian Ministry of Petroleum such as National Iranian Oil Company, National Iranian Gas Company, National Petrochemical Company and National Iranian Oil Refinery And Distribution Company.

**PURCHASER :**

Means the "Company" where this standard is a part of direct purchaser order by the "Company", and the "Contractor" where this Standard is a part of contract document.

**VENDOR AND SUPPLIER:**

Refers to firm or person who will supply and/or fabricate the equipment or material.

**CONTRACTOR:**

Refers to the persons, firm or company whose tender has been accepted by the company.

**EXECUTOR :**

Executor is the party which carries out all or part of construction and/or commissioning for the project.

**INSPECTOR :**

The Inspector referred to in this Standard is a person/persons or a body appointed in writing by the company for the inspection of fabrication and installation work.

**SHALL:**

Is used where a provision is mandatory.

**SHOULD:**

Is used where a provision is advisory only.

**WILL:**

Is normally used in connection with the action by the "Company" rather than by a contractor, supplier or vendor.

**MAY:**

Is used where a provision is completely discretionary.

CONTENTS :	PAGE No.
0. INTRODUCTION .....	5
1. SCOPE .....	6
2. REFERENCES .....	6
3. DEFINITIONS AND TERMINOLOGY .....	7
4. UNITS .....	9
5. ON SITE PORTABLE INSTRUMENTS .....	9
5.1 Alternative Current Colorimeter .....	10
5.2 Calibration and Inspection .....	11
6. ON LINE FIXED MEASUREMENT OR CONTINUOUS MONITORING.....	11
6.1 Continuous Water Sampling and Clarification System.....	11
6.2 Calibration and Inspection .....	13
7. LABORATORY INSTRUMENTS .....	13
7.1 Collection and Preservation of Samples .....	13
8. PHYSICAL EXAMINATION .....	18
8.1 Color .....	18
8.2 Conductivity .....	18
8.3 Odor.....	19
8.4 Residue .....	19
8.5 Salinity.....	19
8.6 Specific Gravity .....	19
8.7 Taste .....	19
8.8 Temperature .....	20
8.9 Turbidity .....	20
9. DETERMINATION OF METALS (as defined in Chemical Periodic Table) .....	20
9.1 Atomic Absorption Spectroscopy.....	20
9.2 Polarography .....	21
9.3 UV-Visible Spectroscopy.....	22
9.4 ICP Spectrometry .....	22
10. DETERMINATION OF INORGANIC NON-METALLIC CONSTITUENTS.....	22
10.1 Acidity and Alkalinity.....	22
10.2 Arsenic .....	22
10.3 Boron.....	22
10.4 Carbon Dioxide.....	22
10.5 Chloride.....	22
10.6 Chlorine (Residual) .....	22
10.7 Cyanide, Fluoride, Iodide .....	23
10.8 Nitrogen (Ammonia, Nitrate, Organic) .....	23
10.9 Oxygen .....	23
10.10 Ozone .....	23
10.11 pH Value .....	23
TECHNICAL DATA .....	23
10.12 Phosphate.....	23
10.13 Silica.....	24
10.14 Sulfate .....	24
10.15 Sulfide .....	24

---

11. DETERMINATION OF ORGANIC CONSTITUENTS .....	24
11.1 Grease and Oil.....	24
11.2 Methane.....	28
11.3 Organic Acid and Volatile Acids.....	29
11.4 Organic Carbon (Total).....	29
11.5 Oxygen Demand (Biochemical) .....	29
11.6 Oxygen Demand (Chemical) .....	30
11.7 Pesticides .....	30
11.8 Phenols .....	30
11.9 Surfactants .....	31
12. EXAMINATION OF WATER AND WASTEWATER RADIOACTIVITY.....	31
12.1 Counting Room .....	31
12.2 Alpha Particle Counter Instrument.....	31
12.3 Beta Particle Radioactivity Instrument .....	32
12.4 Gamma-Ray Monitoring.....	32
13. MICROBIOLOGICAL EXAMINATION OF WATER .....	33
14. AUTOMATED LABORATORY EQUIPMENT FOR MONITORING WATER AND WASTEWATER .....	33
15. MARKING .....	34
16. SHIPMENT .....	34
17. SPARE PARTS .....	34
18. PRE-INSTALLATION AND INSTALLATION .....	34
19. WARRANTY.....	34
20. SERVICE AND INSPECTION.....	34
APPENDICES:	
APPENDIX A PHYSICAL CHARACTERISTIC OF POTABLE WATER .....	35

## 0. INTRODUCTION

The sources of pollution and T.L.V (Threshold Limit Value) of these pollutants in wastewater are described in [IPS-E-SF-880](#) and [IPS-E-PR-730](#). Here again, an effort has been made to present the most appropriate methods and equipment that apply as generally as possible for monitoring surface water, ground waters, cooling or circulating water, boiler water, boiler feed water, wastewater effluents after varying degrees of treatment, and untreated municipal or industrial wastewaters, but when monitoring of sample of highly unusual composition are encountered, the mentioned equipment of this manual may require modification, or may be wholly inappropriate.

## 1. SCOPE

This Standard specifies the minimum requirements for material, operation, tests, inspection and covers site equipment, process, laboratory measurements, calibration of instruments, sampling, sample preservation, preparing of reagents and quality control results.

### Note:

For further information refer to [IPS-E-SF-880](#) "Water Pollution Control" and [IPS-E-PR-730](#) "Process Design of Plant Waste Treatment and Recovery System".

### Note 1:

This standard specification is reviewed and updated by the relevant technical committee on Aug. 2005. The approved modifications by T.C. were sent to IPS users as amendment No. 1 by circular No. 306 on Aug. 2005. These modifications are included in the present issue of IPS.

### Note 2:

This standard specification is reviewed and updated by the relevant technical committee on Feb. 2013. The approved modifications by T.C. were sent to IPS users as amendment No. 2 by circular No. 382 on Feb. 2013. These modifications are included in the present issue of IPS.

## 2. REFERENCES

Throughout this Standard the following dated and undated standards/codes are referred to. These referenced documents shall, to the extent specified herein, form a part of this standard. For dated references, the edition cited applies. The applicability of changes in dated references that occur after the cited date shall be mutually agreed upon by the Company and the Vendor. For undated references, the latest edition of the referenced documents (including any supplements and amendments) applies.

### ASTM (AMERICAN SOCIETY FOR TESTING AND MATERIALS)

ASTM D 6855-2003	"Standard test Method for Determination of Turbidity below 5 NTU in Static Mode "
ASTM D 1067-2011	"Acidity and Alkalinity of Water"
ASTM D 513-2006	"Total and Dissolved Carbon Dioxide in Water"
ASTM D 1253-2008	"Residual Chlorine in Water"
ASTM D 888-2009	"Dissolved Oxygen in Water"
ASTM D 512-2010	"Standard Test Methods for Chloride Ion in Water"
ASTM E 203-2008	"Water Test Using Volumetric Karl Fisher Titration"
ASTM D 3864-2006	"Guide for Continual on-Line Monitoring Systems for Water Analysis"
ASTM D 0859-2010	"Test Methods for Silica in Water"
ASTM D 5317-2003	"Test Methods for Determination of Chlorinated Organic Acid Compounds in Water by Gas Chromatography with an Electrode Capture Detector"
ASTM D 6238-2003	"Test Method for Total Oxygen Demand in Water"
ASTM D 2580-2006	"Test Method for Phenols in Water by Gas-Liquid Chromatography"
ASTM D 3648-2011	"Practices for the Measurement of Radioactivity"

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ASTM D 1943-2005	"Test Method for Alpha Particles Radioactivity of Water"
ASTM D 7282-2006	"Practice for Setup, Calibration, and Quality Control of Instruments used for Radioactivity Measurements"
ASTM D 3863-2003	"Test Method for Retention Characteristics of 0.4 to 0.45 –g mm Membrane Filters used in Routine Filtration Procedures for the Evaluation of Microbiological Water Quality"

**BSI (BRITISH STANDARD INSTITUTE)**

BS 2690-9	"Methods of testing water used in industry"
BS 6068-6.2-1991	"Water quality- Guidance on Sampling Techniques"
BS 6068-2.24-1986	"Water quality- Method for the Determination of non-ionic Surfactants using Dragendorff Reagent"
BS 6920-2.3-2000	"Suitability of Non-Metallic Products for use in Contact with Water Intended for Human Consumption with Regard to their effect"

**DIN (DEUTSCHES INSTITUT FÜR NORMUNG EV.)**

DIN EN ISO 6878-2004	"Determination of Phosphorous-Ammonium Molybdate Spectrometric Method"
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**IPS (IRANIAN PETROLEUM STANDARDS)**

<a href="#">IPS-E-PR-730</a>	"Engineering Standard for Process Design of plant waste Treatment and Recovery System"
<a href="#">IPS-E-GN-100</a>	"Engineering standards for units"
<a href="#">IPS-G-SF-860</a>	"General Standard for Air Pollution Control"
<a href="#">IPS-G-SF-110</a>	"General Standard for Protection against Radioactive Sealed Sources"
<a href="#">IPS-E-SF-880</a>	"Engineering Standard for Water Pollution"
<a href="#">IPS-E-CE-340</a>	"Engineering Standard for Water Resources and Distribution Systems"

**3. DEFINITIONS AND TERMINOLOGY****3.1 Biochemical Oxygen Demand (BOD)**

Biochemical oxygen demand is oxygen demand by micro organisms during stabilization of organic matter under prescribed condition, usually over a 5 day period, BOD5 Specifically denotes the oxy demand over a 5 days period at 20°C.

**3.2 Chemical Oxygen Demand (COD)**

Chemical Oxygen Demand (COD) is the equivalent amount of oxygen consumed under specified conditions in the chemical oxidation of the organic and oxidizable inorganic matter contained in a wastewater corrected for the influence of chlorides. In American practice, unless otherwise specified, the chemical oxidizing agent is hot acid dichromate.

**3.3 Chemical Treatment**

A process involving the addition of chemicals to achieve a specific result.



**3.4 Contractor**

Contractor is the person, firm or company whose tender has been accepted by the Company and includes the contractor's personnel representative, successors and permitted assigns.

**3.5 Dissolved Oxygen (DO)**

Dissolved Oxygen (DO) is the oxygen dissolved in sewage, water, or other liquid, usually expressed in milligrams per liter or percent of saturation. It is the test used in BOD determination.

**3.6 Drinking Water, Potable Water**

Water of a quality based on WHO (World Health Organization).

**3.7 Effluent**

Effluent is (1) a liquid which flows out of a containing space, and/ or (2) sewage, water or other liquid partially or completely treated, or in its natural state, as the case may be flowing out of a reservoir, basin or treatment plant, or part thereof.

**3.8 Employer Company Owner**

Refers to any of the related affiliated companies of the Petroleum Industries of Iran such as National Iranian Oil Company (NIOC), National Iranian Gas Company (NIGC), National Petrochemical Company (NPC), etc., as a part of the Ministry of Petroleum.

**3.9 Ground Water**

Water found in the spaces between soil particles and cracks in rocks underground.(located in the saturation zone)

**3.10 Industrial Water**

Any water used for industrial purpose.

**3.11 Oxygen Consumed**

Oxygen consumed is the quantity of oxygen taken up from potassium permanganate in solution by a liquid containing organic matter commonly regarded as an index of the carbonaceous matter present. Time and temperature must be specified.

**3.12 Pollution**

Contamination of water with actively or potentially toxic or otherwise harmful materials.

**3.13 Rain Water**

Water arising from atmospheric precipitation, which has not yet collected soluble matter from the earth.

**3.14 Raw Water**

Water taken from natural sources, i.e. water wells or surface water.

**3.15 Sewage Effluent**

Treated sewage discharge from a sewage treatment works.

**3.16 Site**

Unit or plant where sampling is to be carried out.

**3.17 Sampling Point**

A specific location on a sampling line at which an individual sample is extracted.

**3.18 Standard Water**

Water with known amount of component.

**3.19 Surface Water**

Water which flows over, or rests on the surface of a land mass.

**3.20 Threshold Limit Values (TLVs)**

Refers to maximum concentration of substances which could be discharged in wastewater issued by the national standard of environmental protection agency for industrial waste.

**3.21 Total Organic Carbon (TOC)**

TOC is a measure of the amount of carbon in a sample originating from organic matter only. The test is run by burning the sample and measuring the CO<sub>2</sub> produced.

**3.22 Wastewater**

Water discharged after being used in, or produced by a process and is of no further immediate value to that process.

**3.23 Wet Chemical Method**

Method based on chemical reagent not instrumental.

**4. UNITS**

This standard is based on International System of Units (SI), as per [IPS-E-GN-100](#) except where otherwise specified.

**5. ON SITE PORTABLE INSTRUMENTS**

All parameters which have been mentioned in [IPS-E-SF-880](#) (Maximum Effluent Standard for Industrial Waste) could be monitored by this portable instrument excluding organic and radioactive substances.

This equipment has been developed to meet the need for a simplified, convenient, accurate means of testing water in the field. The colorimetric tests are made with colorimeter using precalibrated meter scales for direct read-out.

The volumetric tests are conducted by titration using a unique buret and titration stand with a

precision screw plunger.

It dispenses titration solution so that accurate, reliable results are obtained from test to test.

**5.1 Alternative Current Colorimeter**

**5.1.1** As mentioned above the basic of measurement is colorimetric which some reagent could be added to water sample to produce colored solution. Type of portable instrument could be determined by relevant authorities.

See also the following Technical Data:

**TECHNICAL DATA**

**ALKALINITY**

Titration for phenolphthalein and total alkalinity  
Enough of each indicator for 100 tests.  
Enough titrant for 100 average (125 ppm) tests.

**CARBON DIOXIDE**

Standard Titration procedure-  
Enough reagent for approximately 100 tests.

**CHLORIDE**

Mercuric Nitrate Titration-  
Enough reagent for approximately  
100 tests.

**CHLORINE**

Improved Orthotolidine Method  
Colorimetric Range: 0-1 ppm  
Enough reagent for 60 tests.

**CHROMATE**

Diphenylcarbohydrazide Method  
Colorimetric Range: 0-1.5 ppm  
Enough reagent for approximately 100  
tests.

**COLOR**

Colorimetric Range: 0-500 APHA  
platinum-cobalt units.  
No reagents required.

**IRON**

Simplified Phenanthroline Method  
Colorimetric Range: 0-3 ppm  
Enough reagent for approximately 100  
tests.

**MANGANESE**

Cold Periodate Oxidation Method  
Colorimetric Range: 1-10 ppm  
Enough reagent for approximately 100  
tests.

**NITRATE, NITROGEN**

Cadmium Reduction-Diazotization Method  
Colorimetric Range: 0-1.5 ppm N,  
0-15 ppm N. 0-150 ppm N Enough reagent  
for approximately 100 tests.

**NITRITE, NITROGEN**

Diazotization Method  
Colorimetric Range: 0-0.2 ppm N,  
Enough reagent for approximately 100  
tests.

**OXYGEN DISSOLVED**

Improved Winkler method, Alkaline-  
Iodide-Azide Modification  
Titration...1 drop=1 ppm,  
1 drop=.2 ppm.  
Enough reagent for approximately 100  
tests.

**pH, WIND RANGE**

Colorimetric Range: 4.0 -10.0  
Enough reagent for approximately  
100 test.

**COPPER**

Cuprethol Method  
 Colorimetric Rang: 0-3 ppm  
 Enough reagent for approximately 100 tests.

**FLUORIDE**

SPADNS Method  
 Colorimetric Range: 0-2 ppm  
 Enough reagent for approximately 10 tests.

**HARDNESS, CALCIUM**

EDTA Titration method  
 Enough reagent for approximately 100 tests.

**HARDNESS, TOTAL**

EDTA Titration method  
 100 tests.

**HYDROGEN SULFIDE**

Screen test color chart comparison  
 . . . 0.1-5 ppm  
 Enough test paper for 100 tests.

**PHOSPHATE, ORTHO & META**

Stannous Reduction Method  
 Colorimetric Range 0-2 ppm and 0-8 ppm.  
 Enough reagent for approximately 100 tests.

**SILICA**

Heteropoly Blue Method  
 Colorimetric Range: 0-3 ppm  
 Enough reagent for approximately 100 tests.

**SULFATE**

Turbidimetric Method  
 Range: 0-300 ppm  
 Enough reagent for approximately 100 tests.

**TURBIDITY**

Absorption method  
 Range: 0-500 JTU  
 No reagents required.

**5.1.2** For new equipment See catalog of laboratory equipment issued by environmental science and Technology or any Relevant Journal.

**5.2 Calibration and Inspection**

**5.2.1** In general this type of instrument is factory calibrated, but can be calibrated by competent person in accordance with the manufacturer's recommended standard.

**5.2.2** All reagent and chemical must be freshed or in case of changing color and precipitation replaced by new one.

**6. ON LINE FIXED MEASUREMENT OR CONTINUOUS MONITORING**

The use of continuous monitoring equipment will permit the frequent measurement of several water quality parameters and the recording of this data at the measurement site or the transmitting of it to another location.

**6.1 Continuous Water Sampling and Clarification System**

**6.1.1** The monitoring instruments should contain a sampling module, the sensors, a signal conditioning module, and a data logging or transmission module. A continuous flow of sample must

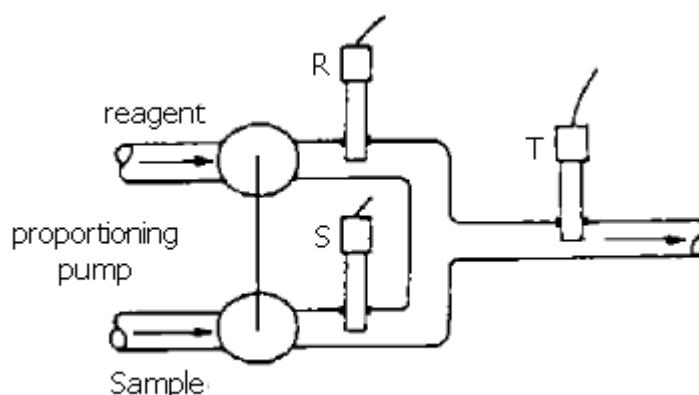
be provided to the sensors by a submersible pump.

Each sensor in the instrument should be equipped with its own signal conditioner which converts the input to a standard electrical output. For more detail on sensors and ion selective electrode. See manufacturer specification.

6.1.2 A typical electrode placement for continuous monitoring is shown in Fig. 1. Further information has been given hereunder (see also ASTM Volume 11.01(2011)-D3864-06).

**ELECTRODES, ION-SELECTIVE**

Ion Determined	Recommend Ref. Electrode
Ammonia	Not applicable
Ammonium	Calomel
Barium	Calomel
Bromide	Double junction
Cadmium	Calomel
Calcium	Calomel
Chloride	Double junction
Copper	Calomel
Cyanide	Double junction
Fluoride	Double junction
Fluoroborate	Not applicable
Iodide	Double junction
Lithium	Double junction
Nitrate	Double junction
Oxygen	Not applicable
Potassium	Double junction
Silver/Sulphide	Double junction
Sodium	Double junction
Sulphur	Not applicable



**ELECTRODE PLACEMENT FOR CONTINUOUS MONITORING**

**Fig. 1**

## **6.2 Calibration and Inspection**

**6.2.1** Calibration of the sensor signals should be carried out by adjustment of the controls on the signal conditioner.

**6.2.2** The sample intake should be shielded with a screen and must be inspected according to manufacturer's instructions to prevent debris from clogging the system or damaging the pump.

## **7. LABORATORY INSTRUMENTS**

All parameters which are summarized in the following sections could be monitored in laboratory using instrumental or wet chemical methods.

### **7.1 Collection and Preservation of Samples**

#### **7.1.1 Sampling of different sources**

The summary of different kind of sampling is mentioned here, for more details See BS 6068-6.2, Water Quality Sampling.

#### **7.1.2 Sampling of atmospheric precipitation**

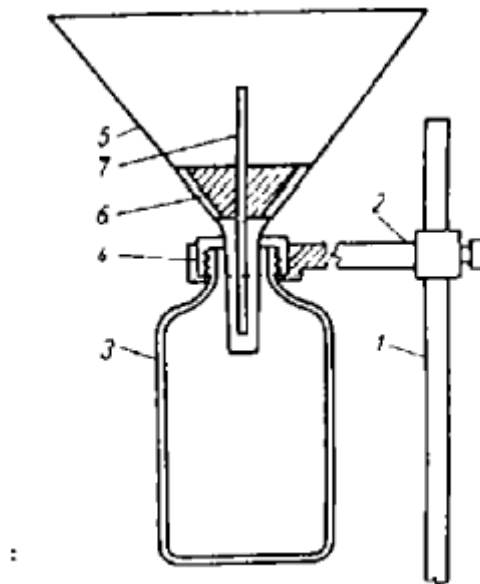
Atmospheric precipitation usually occurs in discrete events of limited duration.

##### **7.1.2.1 Sampling precipitation equipment**

Equipment for sampling precipitation, includes, a polyethylene polypropylene bucket and a stand.

##### **7.1.2.2 Construction**

The basic of this equipment is shown in Fig. 2. The equipment could be operated manually or automatically.



**Funnel system for precipitation water sampling:**

- 1) Stand
- 2) Adjustable support
- 3) Sample container
- 4) Threaded cap
- 5) Collecting funnel
- 6) Truncated grooved cone (fly trap)
- 7) Vent tube

## SAMPLING OF ATMOSPHERIC PRECIPITATION

**Fig. 2**

### 7.1.2.3 Inspection

The equipment must be designed to minimize pollution during its operation. It should be manufactured from chemically inert materials to avoid contamination.

### 7.1.3 Sampling surface water

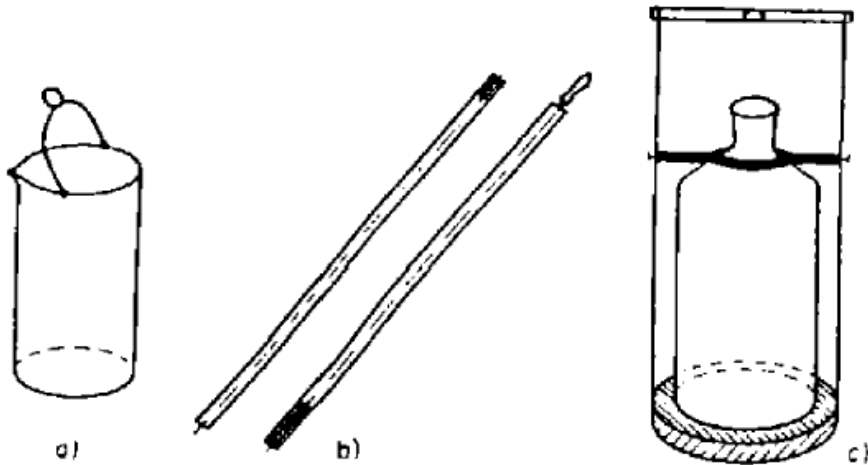
Surface water samples are usually taken directly into the sample container. It is not possible to take the sample by submerging the container by hand, laboratory forceps or a holder with a sliding sleeve should be used.

#### 7.1.3.1 Surface water container

For such sampling the container, which should have a volume of at least two liters, is usually made of polyethylene/polypropylene or glass.

#### 7.1.3.2 Construction

The typical container is shown in Fig. 3.



- a) can;  
b) segmented rod;  
c) submersible bottle with ballast and frame.

### SIMPLE AIDS FOR SURFACE WATER SAMPLING

Fig. 3

#### 7.1.4 Soil water sampling

Soil water is defined as being all of the water contained in the soil.

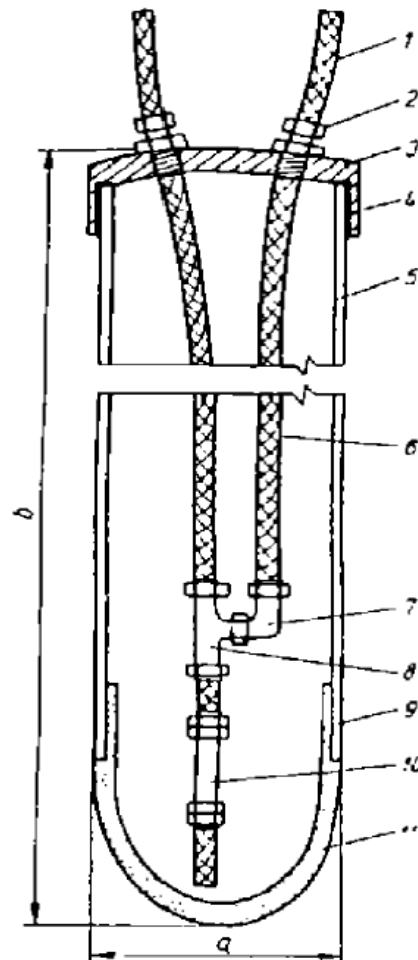
##### 7.1.4.1 Soil water sampling system

A common soil sampling system comprises a ceramic vessel with two pressure (vacuum) hoses inserted into it. The system operates by developing a vacuum through one hose, which causes the soil water to be drawn in through the porous walls of the vessel.

##### 7.1.4.2 Construction

A schematic of soil water sampler is shown in Fig. 4.





- 1) And 6, braided, flexible pressure hose
- 2) Hose connector
- 3) PVC sampler cover
- 4) Seal between cover and sample pot
- 5) Upper wall of sample container
- 6) Elbow connector
- 7) T-piece connector
- 8) Glued connection between upper wall of sample container (PVC) and porous ceramic pot
- 9) Non-return valve
- 10) Porous ceramic pot. Typical dimensions \_ a = 50 mm, b= 500 mm.

**SOIL WATER SUCTION SAMPLER**

**Fig. 4**

**7.1.5 Groundwater sampling**

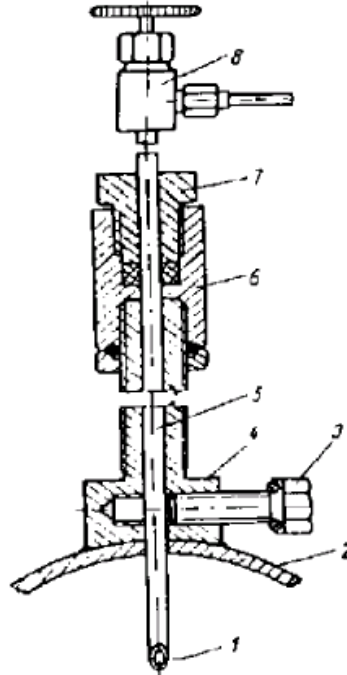
The sampling of groundwater is usually carried out with devices and equipment very similar to that used for surface waters.

**7.1.5.1 Ground water sampling system**

Similar containers are used for groundwater, mainly glass or polythene bottles.

### 7.1.5.2 Construction

A typical diagram of transferable sampler for use on wastewater pipelines is shown in Fig. 5.



- 1) Inlet (aperture cut obliquely across tube)
- 2) Pipeline wall
- 3) Sample point closing valve
- 4) Sampler inlet guide, welded to pipeline wall
- 5) Sampling tube
- 6) Connecting bush with o-ring seal
- 7) Locking nut
- 8) Needle valve

TRANSFERABLE SAMPLER FOR USE ON PIPELINES

Fig. 5

### 7.1.6 Different types of sampling

#### 7.1.6.1 Grab or catch samples

A sample collected at a particular time and place.

#### 7.1.6.2 Composite samples

In most cases the term composite sample refers to a mixture of grab samples collected at the same sampling point at different times.

#### 7.1.6.3 Integrated samples

For certain, the information needed is provided best by analysis of mixtures of grab samples collected from different points simultaneously, or as nearly so as possible.

**8. PHYSICAL EXAMINATION**

**8.1 Color**

Color in water may result from the presence of metallic ion humus and peat materials, plankton, weed and industrial waste.

The unit of color produced by 1 mg/L platinum in the form of the chloroplatinate ion.

For color monitoring see ASTM D 6855-2012 and BS 2690: Part 9, 1970. Maximum concentration tabulated in [IPS-E-SF-880](#).

**8.2 Conductivity**

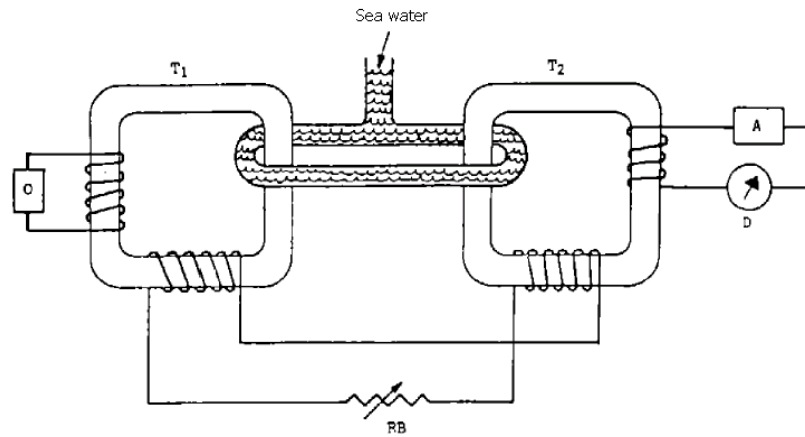
Conductivity is a numerical expression which is indicated ability of a water sample to carry an electric current and is defined as  $\mu$  moh/cm<sup>2</sup>. This number depends on the total concentration of the ionized substances in the water. See also BS 2690: Part 9 1970.

**8.2.1 Conductivity instruments**

Measurement of electrical conductance is usually based on the use of conductivity bridges (whetstone bridge). The bridge circuit always should be arranged so that at the balance point the detector indicates either zero or minimum potential. Basic of instrument is shown in Fig. 6.

The commercial form is an electrode connected to electrical circuit. Best result can be obtained with the following technical data:

	<u>TECHNICAL</u>	<u>DATA</u>
Ranges	0-1000	mS
	0-199.9	mS
	0-19.99	mS
	0-1999	$\mu$ S
	0-199.9	$\mu$ S
	0-19.99	$\mu$ S
	0-100.0	$^{\circ}$ C
	(automatic selection)	
Resolution	1	mS
	0.1	mS
	0.01	mS
	1	$\mu$ S
	0.1	$\mu$ S
	0.01	$\mu$ S
	0.1	$^{\circ}$ C
Accuracy	$\pm$ 0.5%	
Temperature compensation range	0 to 100 $^{\circ}$ C	
Slope auto temp comp	Variable from 0.5 to 4 %/ $^{\circ}$ C	
Cell constant	0.05 to 19.99 digitally settable	
Outputs	Analogue 1 mV/digit, RS 232	
Display	Conductivity 20 mm LED	
	Temperature 10 mm LED	
Overall dimensions (H x W x D)	120 x 300 x 180 mm	
Mass	2kg	
Power requirements	115/230 V ac $\pm$ 20% 50/60 Hz	



- T<sub>1</sub>    **First electrical circuit**
- T<sub>2</sub>    **Second electrical circuit**
- RB    **Variable resistance**
- A    **Ampere**
- D    **Conductivity meter**

**"INDUCTIVE-TYPE" CONDUCTIVITY CELL**

**Fig. 6**

**8.2.2 Calibration**

Calibration should be carried out with standard water.

**8.3 Odor**

Odor and taste are known as the "chemical senses" since they depend on actual contact of the stimulating substance with the appropriate human receptor cell.

For odor monitoring See M.C. Rand, A.E. Greenberg, standard methods for the examination of water and wastewater, American Public Health Association, 1987 and BS 2690 Part 9 1970. For maximum odor see [IPS-E-SF-880](#).

**8.4 Residue**

The term residue refers to solid matter suspended or dissolved in water or wastewater.

**8.5 Salinity**

It is defined as the total solid in water after all carbonates have been converted to oxides, all bromide, and iodide have been replaced by chloride and all organic matter oxidized.

**8.6 Specific Gravity**

Specific gravity is determined by comparing the weight of a volume of the sample of mud, sludge, or industrial waste with that of an equal volume of distilled water.

**8.7 Taste**

Taste, like odor, is one of the chemical senses.

### 8.8 Temperature

Normally, temperature measurement may be made by mercury-filled Celsius thermometer with scale marked for every 0.1°C.

### 8.9 Turbidity

Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in straight lines through sample.

Turbidity in water is caused by the presence of suspended matter, such as clay, silt, finely divided organic and inorganic matter, plankton, and other microscopic organisms. See also BS 2690: Part 9, 1970.

#### Note:

For Clauses 9.4 to 9.9 the above reference or ASTM Volume 11.01-D 6855-2003 should be seen. For maximum characteristic see [IPS-E-SF-880](#).

## 9. DETERMINATION OF METALS (as defined in Chemical Periodic Table)

The presence of metals in potable water, domestic wastewater and industrial effluents is a matter of serious concern. Metals could be monitored by atomic absorption spectroscopy, polarography, ICP and colorimetric methods.

### 9.1 Atomic Absorption Spectroscopy

In atomic absorption the sample is atomized into a flame, producing atomic vapor of the elements in question. The atoms absorb the radiation from the light source. The amount of light absorbed is proportional to the amount of the element.

#### 9.1.1 Atomic absorption instrument

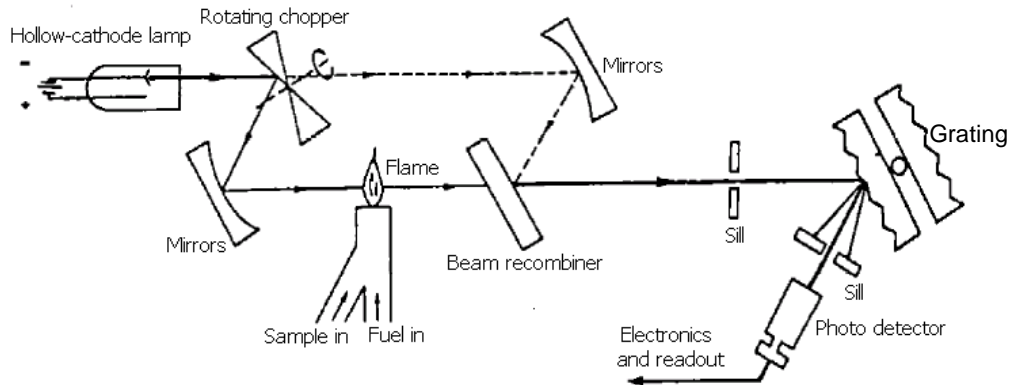
Fig. 7 shows schematic diagram of an instrument. A mechanical modulated chopper alternately passes and reflects the light beam. One beam by-passes the sample and its intensity is measured as  $I_0$  the sample's beam is measured as  $I_1$ . The absorbed light is as  $I_0 - I_1$ . See also the following technical data:

#### TECHNICAL DATA

Monochromator	:	Focal length 267 mm.
Wavelength	:	Range 190-870 nm. 1800.
Lines/mm		
Dispersion	:	1.6 nm/min
Photometer	:	Double-beam
Deuterium background corrector:		Internally mounted correction for molecular absorption
Wavelength drive	:	5 nm/min
Burner	:	Stainless steel
Power requirements	:	105-125 or 200-240 Volts, 50/60 Hz, 150 Watts

**9.1.2 Calibration**

The instrument should be calibrated according to manufacturer's manual book.



**ATOMIC ABSORPTION SPECTROMETER**

**Fig. 7**

For more detail see D.A. Skoog, Principles of instrumental analysis, Holt Rinehart and Winston, Inc., 1981. The toxic pollutants such as Pb, As, Hg with low concentration (PPb) part per billion should be monitored by special device (vapor Generation and Hydride Generation) which could be installed on atomic absorption instrument. Detail has been written in Atomic Absorption manual book.

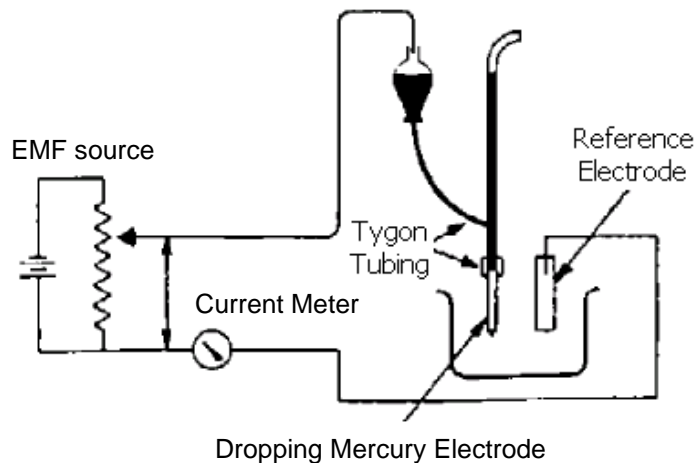
**9.2 Polarography**

Electrochemical methods are used for analysis of Raw water and wastewaters.

**9.2.1 Polarography instrument**

A schematic diagram of a classical polarograph is shown in Fig. 8.

Polarographic measurement is based on determining the time-averaged current of the Dropping Mercury Electrode (DME) under diffusion condition.



**SCHEMATIC DIAGRAM OF CLASSICAL POLAROGRAPH**

**Fig. 8**

For more details see the following Technical Data:

**TECHNICAL DATA**

Concentration range:	10 <sup>-2</sup> to 10 <sup>-6</sup> M (Mole)
Initial voltage:	Infinitely variable -5 and +5
Voltage range:	-0.75V, -1.5V, -3V
Voltage speed:	Adjustable from 1 to 500 mv/S
Current range:	Adjustable from 20 nA to 10 mA

**9.2.2 Calibration**

It should be calibrated according to instrument's manual book.

**9.3 UV-Visible Spectroscopy**

For UV-VIS spectroscopy See [IPS-G-SF-860](#).

**9.4 ICP Spectrometry**

For inductively coupled argon plasma spectrometry See [IPS-G-SF-860](#).

**10. DETERMINATION OF INORGANIC NON-METALLIC CONSTITUENTS****10.1 Acidity and Alkalinity**

For above chemicals See ASTM D 1067-2011

**10.2 Arsenic**

Arsenic could be monitored by wet chemical method or hydride generation technic. Detail is described in M.R. WPCF, "Standard Methods for The Examination of Water and Wastewater, American Public Health Association, 1987."

**10.3 Boron**

Monitored by UV-VIS spectroscopy see 9.3.

**10.4 Carbon Dioxide**

Measured by titrimetric method see ASTM D 513-2006.

**10.5 Chloride**

Chloride, in the form of Cl Ion, is one of the major inorganic anions in water and wastewater. Chloride could be monitored by titrimetric, potentiometric and Ion selective electrode. More detail could be obtained in ASTM D 512-2010 or W, Horwitz, Official Methods of Analysis, Association of Official Analytical Chemists 1975.

**10.6 Chlorine (Residual)**

The chlorination of water supplies and polluted water serves to destroy or deactivate disease, producing microorganisms. It could improve water quality by reaction with ammonia, Iron, Manganese, Sulfide and some organic substances. It should be monitored by titrimetric, colorimetric

techniques. More detail should be obtained in ASTM D 1253-2008.

### 10.7 Cyanide, Fluoride, Iodide

These three pollutants should be measured by titrimetric and/or colorimetric methods (see [IPS-G-SF-860](#), UV-VIS spectroscopy) and Ion selective electrodes.

Detail about Ion selective electrode could be provided in D.A Skoog, D.M. Wes, principles of instrumental analysis, Holt Rinehart Winston, 1981.

### 10.8 Nitrogen (Ammonia, Nitrate, Organic)

Nitrogen group should be monitored by titrimetric and/or colorimetric methods (see [IPS-G-SF-860](#)).

### 10.9 Oxygen

Oxygen should be monitored by specific electrode for ISE. See titrimetric method as detailed in ASTM D 888-2009.

### 10.10 Ozone

Ozone should be measured by titrimetric method as given in ASTM D 7677.

### 10.11 pH Value

The PH value of a solution is defined as the inverse logarithm of the hydrogen-ion concentration. Detail on effect of PH could be observed in API, manual on disposal of refinery wastes volume on liquid wastes, Chapter 2. PH should be monitored by electronic PH meter.

#### 10.11.1 pH meter instrument

The pH meter consists of electronic part, glass electrode, reference electrode. See also the following Technical Data.

#### TECHNICAL DATA

Digital pH meter range	:	0-00 to 14.00 PH
mV range	:	-1999 to +1999 mV
°C range	:	-50.0 to +199.9 °C
Accuracy	:	Better than 0-01 PH
Output, analogue	:	1 mv/Digit

#### 10.11.2 Calibration

It should be calibrated with standard buffer solution with known PH.

### 10.12 Phosphate

Phosphate content should be monitored by colorimetric method. For detail see DIN EN ISO 6878-2004 Section 2.28 1986.



**10.13 Silica**

Silica concentration could be measured by calorimetric, gravimetric method. For detail See ASTM 11.01(2011)-D 0859-2010.

**10.14 Sulfate**

Sulfate could be measured by gravimetric method. For detail See M.R. WPCF, standard methods for the examination of water and wastewater, American Public Health Association, 1985.

**10.15 Sulfide**

For sulfide monitoring see Clause 10.14 above, but in general it should be measured by colorimetric and titrimetric methods.

**11. DETERMINATION OF ORGANIC CONSTITUENTS**

**11.1 Grease and Oil**

**11.1.1 Solvent extraction infrared absorption**

In this type of monitoring the oil which is extracted from sample, measured by the infrared absorption method.

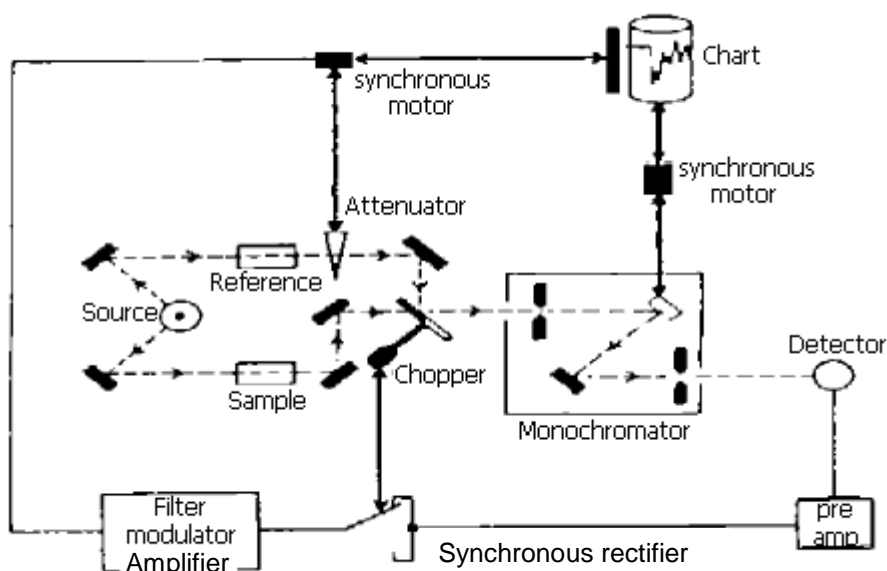
**11.1.1.1 Infrared spectrophotometers instrument**

Radiation from the source is split into two beams, half passing into the sample cell and the other half into the reference cell. The reference beam then passes through the attenuator and onto the chopper. After dispersion by the prism or grating, the alternating beams fall on the detector and are converted to an electrical signal.

Schematic diagram is shown in Fig. 9. See also the following Technical Data:

**TECHNICAL DATA**

Accuracy	:	$\pm 6 \text{ Cm}^{-1}$ from 4000 to 2000 $\text{Cm}^{-1}$ $\pm 3 \text{ Cm}^{-1}$ from 2000 to 600 $\text{Cm}^{-1}$
Repeatability	:	1 $\text{Cm}^{-1}$ from 4000 to 2000 $\text{Cm}^{-1}$ $\pm 0.5 \text{ Cm}^{-1}$ from 2000 to 600 $\text{Cm}^{-1}$
Resolution	:	6 $\text{Cm}^{-1}$ at 3000 $\text{Cm}^{-1}$
Spectral slit width:		2.8 $\text{Cm}^{-1}$ at 1000 $\text{Cm}^{-1}$
Stray light %T	:	Less than 0.3 %
Noise	:	0.8 %T



Heavy dark line= mechanical linkage;

Light line = electrical linkage;

Dotted line = radiation path

**SCHEMATIC DIAGRAM OF A DOUBLE-BEAM SPECTROPHOTOMETER**

Fig. 9

**11.1.1.2 Calibration**

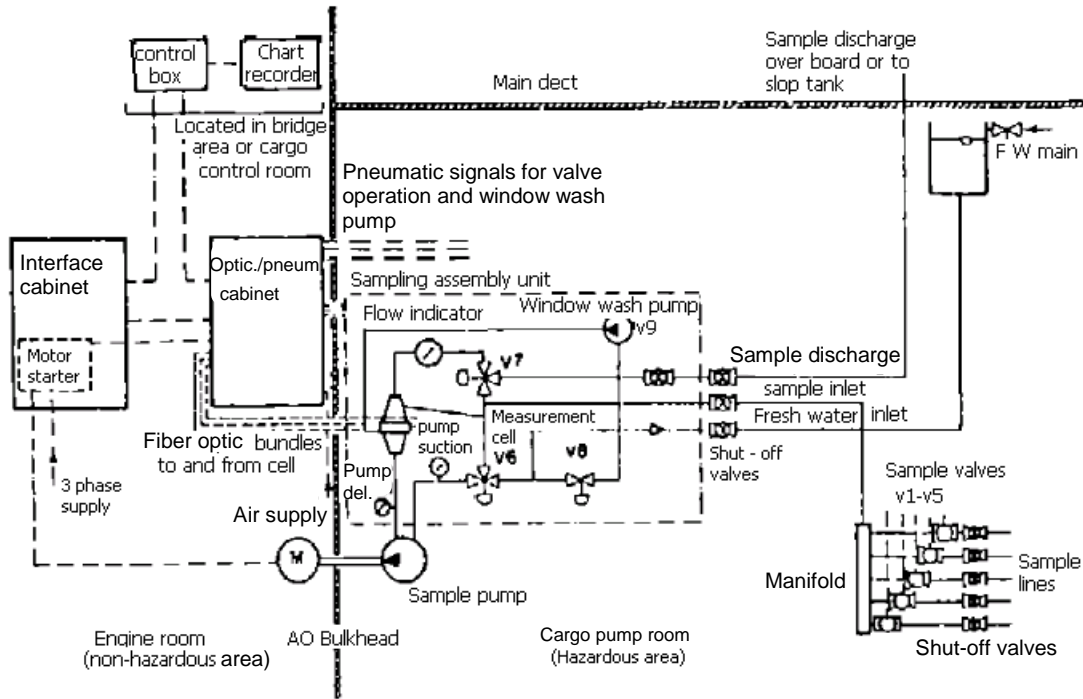
Calibration of instrument should be according to manufacturer’s manual book.

**11.1.2 Optical fiber**

This method is the use of bared optical fiber immersed in the discharge stream.

**11.1.2.1 Optical fiber monitoring instrument**

Water has a refractive index of 1.33-1.38 and oil or hydrocarbon have near 1.5 so radiation would be lost from the fiber when oil droplets became attached. For radiation helium-neon laser could be used. Schematic diagram of monitoring by this system is shown in Fig. 10.



A fibre optic link joins the measurement cell with the electronics box.  
**SCHEMATIC DIAGRAM OF A TANKER'S BALLAST MONITORING SYSTEM**

Fig. 10

**11.1.2.2 Calibration**

Calibration should be according to manufacturer's manual book.

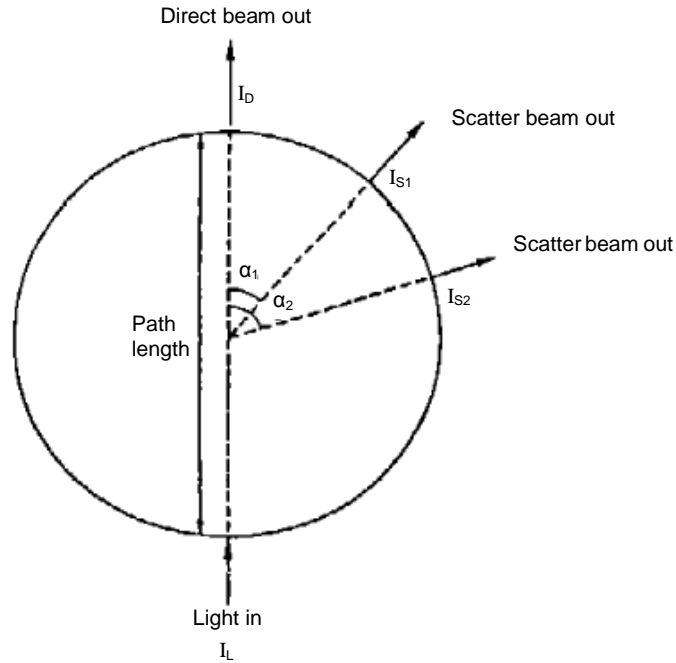
**11.1.3 Scatter and turbidity**

Oil concentration could be monitored by light scatter from oil droplets in the sample.

**11.1.3.1 Optical scatter instrument**

The light source should preferably have a wave length at which the water absorption is low. White light sources or near infrared emitters at about 0.9  $\mu\text{m}$  can be used.

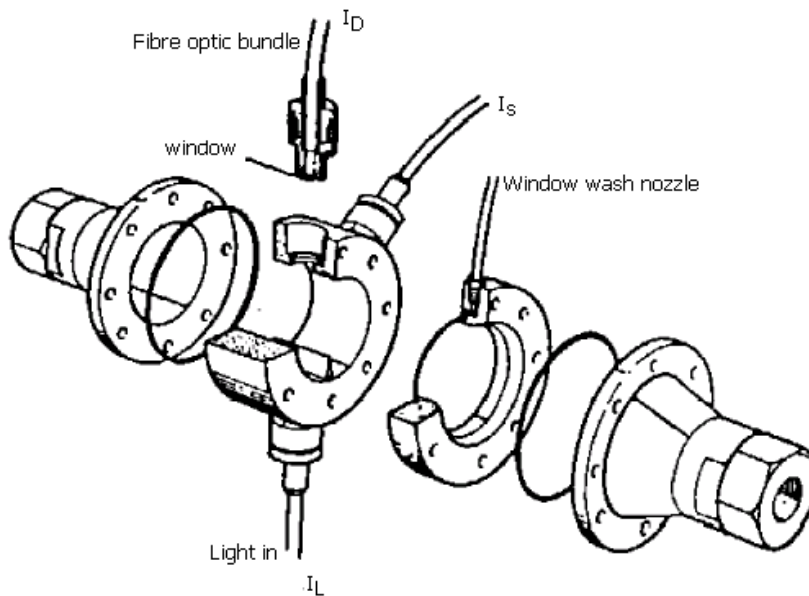
Basic of this method is shown in Fig. 11.



**SCATTER DIAGRAM, SCHEMATIC REPRESENTATION OF A TYPICAL SCATTER CELL WITH THE FLOW PERPENDICULAR TO THE PAGE**

**Fig. 11**

Also schematic diagram of an optical scatter cell is provided in Fig. 12. More detail is provided in H.D. Parker and G.D. PITT, Pollution Control Instrumentation for Oil and Effluents, Graham and Trotman, 1987.



The device is designed for incorporation of optical fibres. The windows are cleaned by periodically passing a jet of clean water through adjacent slots.

**SCHEMATIC DIAGRAM OF AN OPTICAL SCATTER CELL WITH DETECTION AT ONE FORWARD ANGLE**

**Fig. 12**

**11.1.3.2 Calibration**

Calibration should be according to manufacturer's manual book.

**11.2 Methane**

**11.2.1 Volumetric method**

For volumetric method. See ASTM E 203-2008.

**11.2.2 Gas chromatograph**

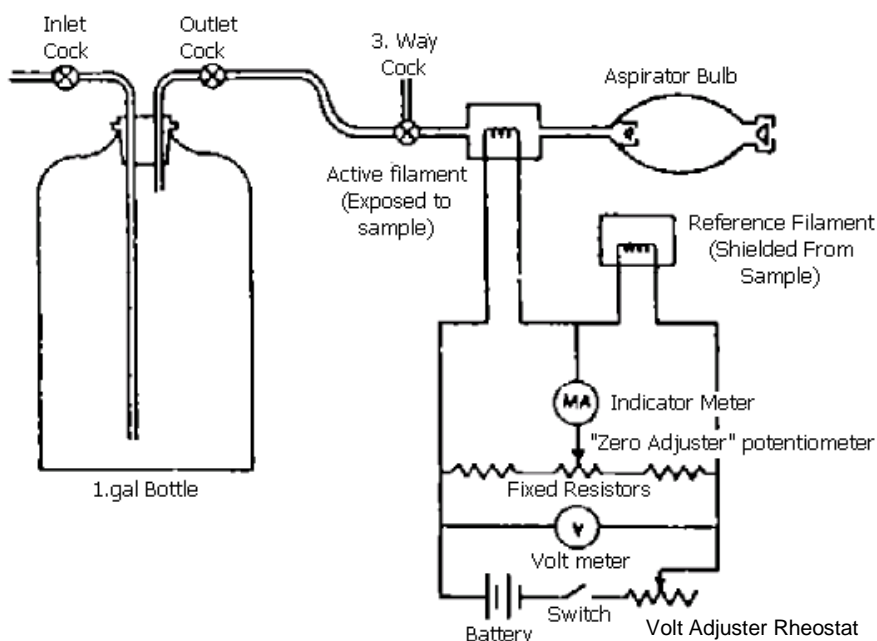
For equipment and basic of gas chromatograph. See [IPS-G-SF-860](#).

**11.2.3 Combustible gas indicator**

An equilibrium is established between methane in solution and the partial pressure of methane in the gas phase above the solution. The partial pressure of methane could be determined with a combustible gas indicator.

**11.2.3.1 Combustible-gas indicator instrument**

The heat generated by the oxidation of the gas increases the electrical resistance of the filament. The resulting imbalance of the electrical circuit causes deflection of a milliammeter. A diagrammatic view of the apparatus is shown in Fig. 13.



**COMBUSTIBLE-GAS INDICATOR CIRCUIT AND FLOW DIAGRAM**

**Fig. 13**

**11.2.3.2 Calibration**

The milliammeter should be calibrated in terms of the percentage of methane.

**11.3 Organic Acid and Volatile Acids**

Organic acid and volatile acids should be monitored by chromatographic and steam distillation. See ASTM Volume 11.02-D 5317-2003 and [IPS-G-SF-860](#).

**11.4 Organic Carbon (Total)**

Organic Carbon TOC could be monitored by carbon analyzer in the range of 1 to 150 mg/L in water and wastewater.

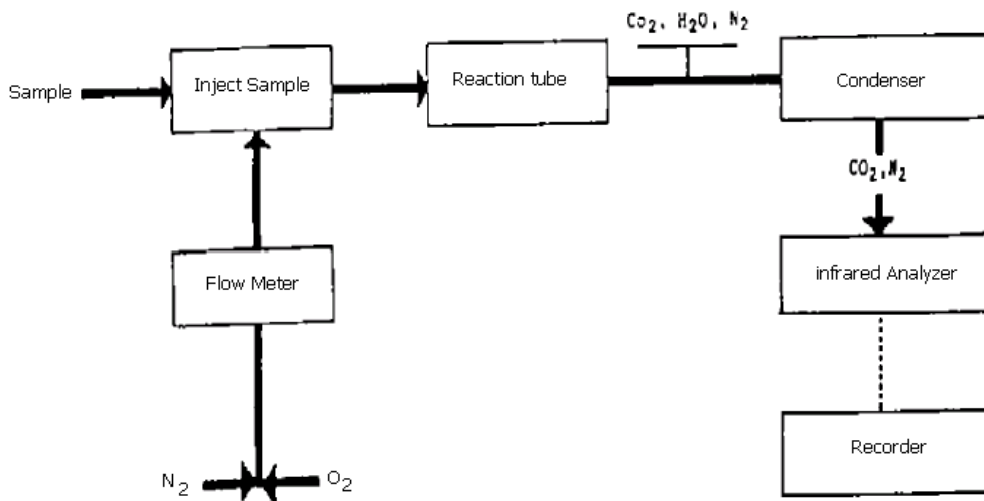
**11.4.1 Total carbon analyzer instrument**

Microportion of sample is injected into a heated packed tube in a stream of oxygen or purified air.

The water is vaporized and the organic matter is oxidized to carbon dioxide, which is measured by means of a nondispersive type of infrared analyser. Flow diagram is shown in Fig. 14. See also the following Technical Data:

**TECHNICAL DATA**

Sampler	:	Quartz boat
Combustion temperature	:	800 °C
Carrier gas	:	N <sub>2</sub>
Combustion gas	:	O <sub>2</sub>
Measuring range	:	1 g <sup>c</sup> /l
Sensitivity	:	0.1 mg <sup>c</sup> /l
Voltage	:	220 V, 50 Hz
Power input	:	2.35 kVA



**TOTAL CARBON ANALYZER FLOW DIAGRAM**

**Fig. 14**

**11.4.2 Calibration**

The instrument should be calibrated according to manufacturer's manual book.

**11.5 Oxygen Demand (Biochemical)**

The Biochemical Oxygen Demand (BOD) is a test in which standardized laboratory procedures should be used to determine the relative oxygen requirements of wastewaters, effluents, and polluted waters.

For this procedures see ASTM Volume 11.02-D 6238-2003.

**11.6 Oxygen Demand (Chemical)**

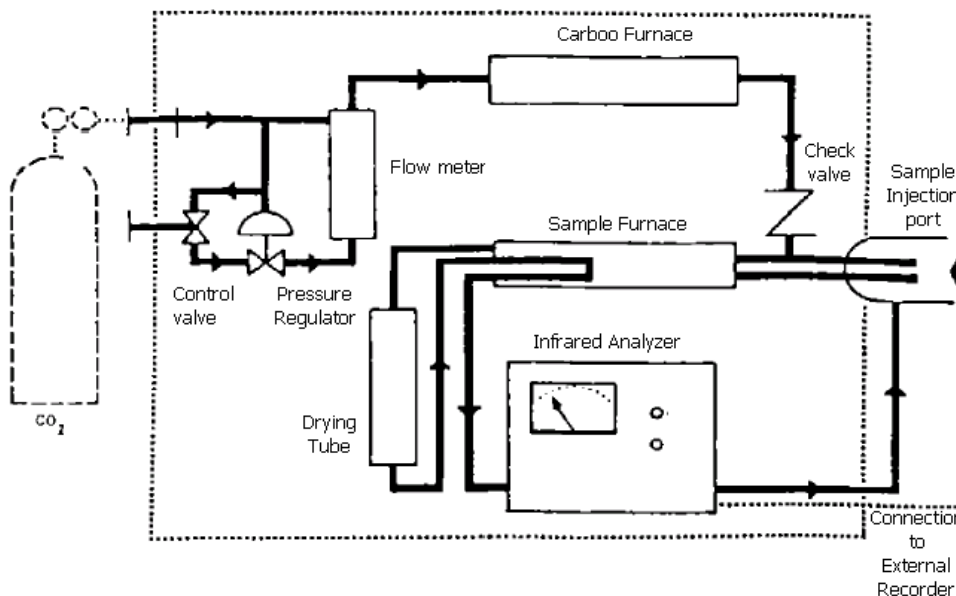
COD is an important measured parameter for stream and industrial waste control.

**11.6.1 COD analyzer instrument**

By using dry CO<sub>2</sub> to carry the organic matter through a platinum catalytic combustion furnace which oxidizes it to CO and H<sub>2</sub>O Following water removal and passage through a second catalytic treatment, the CO concentration is measured using an infrared analyzer. A schematic of instrument is shown in Fig. 15. See also the following Technical Data:

**TECHNICAL DATA**

Sampler	:	Quartz boat
Combustion temperature	:	800 °C
Carrier gas	:	CO <sub>2</sub>
Measuring range	:	1 g <sup>O<sub>2</sub></sup> /l
Sensitivity	:	0.1 mg <sup>O<sub>2</sub></sup> /l
Voltage	:	220 V, 50 Hz
Power input	:	2.35 KVA



**COD ANALYZER FLOW DIAGRAM**

**Fig. 15**

**11.6.2 Calibration**

Calibration should be according to instrument's manual book.

**11.7 Pesticides**

Gas chromatographic procedure should be used for quantitative determination of pesticides which is fully described in [IPS-G-SF-860](#).

**11.8 Phenols**

Phenols, defined as hydroxy derivatives of benzene and its condensed nucleate, may occur in domestic and industrial wastewaters. It should be monitored by photometric and gas chromatographic methods which are described in [IPS-G-SF-860](#). For column packing, carrier gas

refer to ASTM Volume 11.02-2011-D 2580-2006.

### 11.9 Surfactants

One of the pollutant which must be controlled in wastewater is surfactants (Synthetic Detergents). This should be monitored by infrared equipment or spectrophotometer. For infrared description see 11.1.1.1 and for spectrophotometer, [IPS-G-SF-860](#). For surfactants monitoring. See also BS 6068-2.24-1986.

## 12. EXAMINATION OF WATER AND WASTEWATER RADIOACTIVITY

The radioactivity in water and wastewater originates from natural and artificial or man-made sources. Artificial sources of radioactivity include fission, fusion, or particle acceleration, giving rise largely to alpha, beta and gamma radioactivity. For more detail See [IPS-G-SF-110](#) "Protection Against Radioactive Sealed Sources" and ASTM 11.02(2011)-D 3648-2011.

### 12.1 Counting Room

The room should be free of dust and fumes that may affect the electrical stability of instrument. The background could be stabilized and lowered considerably by making the walls, floor, and ceiling out of several centimeters of concrete. Generally, temperature could be constant within 3°C and should not exceed 30°C.

Samples containing appreciable activity should be stored at a distance so as not to affect instrument background counting rate.

### 12.2 Alpha Particle Counter Instrument

Alpha particle counter, consisting of either a proportional detector or a scintillation detector, and a scaler conforming to the following requirements. For method, see ASTM 11.02 (2011)-D 1943-2005 and Technical Data.

#### 12.2.1 Proportional detector

This may be one of several types commercially available. The material used in the construction of the detector should be free from detectable radioactivity.

The manufacturer should supply voltage plateau and background counting rate data. Voltage plateau data should show the threshold voltage, slope, and length of plateau for a particular input sensitivity.

#### 12.2.2 Scintillation detector

It should consist of an "activated" zinc sulfide phosphor having a minimum effective diameter of 36.5 mm. The phosphor should be mounted so that it could be attached and optically coupled to a multiplier photo tube.

#### 12.2.3 Scaler

Mechanical register, power supply, and amplifier are contained in a single chassis, generally termed the scaler.

#### 12.2.4 Sample mounting disks or dishes

Having a flat bottom of a diameter slightly less than the inside diameter of the detector. Flat disks are preferred, but dishes could be used that have 3.2 mm high side walls. Platinum and stainless



steel have been used for this purpose.

#### **12.2.5 Calibration and standardization for general monitoring**

Place a known amount of alpha standard into a volume of water sufficient to dissolve salts equivalent to those of the test samples and prepare for counting.

#### **12.3 Beta Particle Radioactivity Instrument**

Beta particle radioactivity of water and wastewater in general monitored by beta particle counter which consist of the following components:

##### **12.3.1 Detector**

The end-window Geiger-Muller tube and the internal or external proportional gas-flow chambers are the two most commercially available types of detector.

##### **12.3.2 Detector shield**

The detector assembly shall be surrounded by an external radiation shield of massive metal equivalent to approximately 51 mm of lead and lined with 3.2 mm thick aluminum.

##### **12.3.3 Scaler**

Described in 12.2.3.

##### **12.3.4 Sample mounting dishes**

Described in 12.2.4.

##### **12.3.5 Calibration for general monitoring**

See 12.2.5. For more technical data see ASTM Volume 11.02- D 7282-2006.

#### **12.4 Gamma-Ray Monitoring**

This section covers the monitoring equipment for gamma-ray emitting radio nuclides in water or wastewater by means of gamma-ray spectrometry.

##### **12.4.1 Gamma-Ray instrument**

Gamma-ray spectra are measured with modular equipment consisting of a detector, and analyzer, memory and a permanent data storage device. Lithium-drifted detectors, P-Type or N-Type, are used. A multichannel pulse-height analyzer should be used to determine the amplitude of each pulse originating in the detector.

##### **12.4.2 Shield**

See 12.3.2.

##### **12.4.3 Calibration and standardization**

See 12.2.5. For more technical data see ASTM Volume 11.02 - D 7282-2006.

**13. MICROBIOLOGICAL EXAMINATION OF WATER**

In this section there should be suitable procedures in making microbiological examinations of samples of water to determine sanitary quality and suitability for general use. For more detail about these procedures following standards could be seen; ASTM Volume 11.02 - D 3863-2003 and standard methods for the examination of water and wastewater, American Public Health Association, 1985. See also Appendix A and [IPS-E-CE-340](#) Water Resources and Distribution Systems.

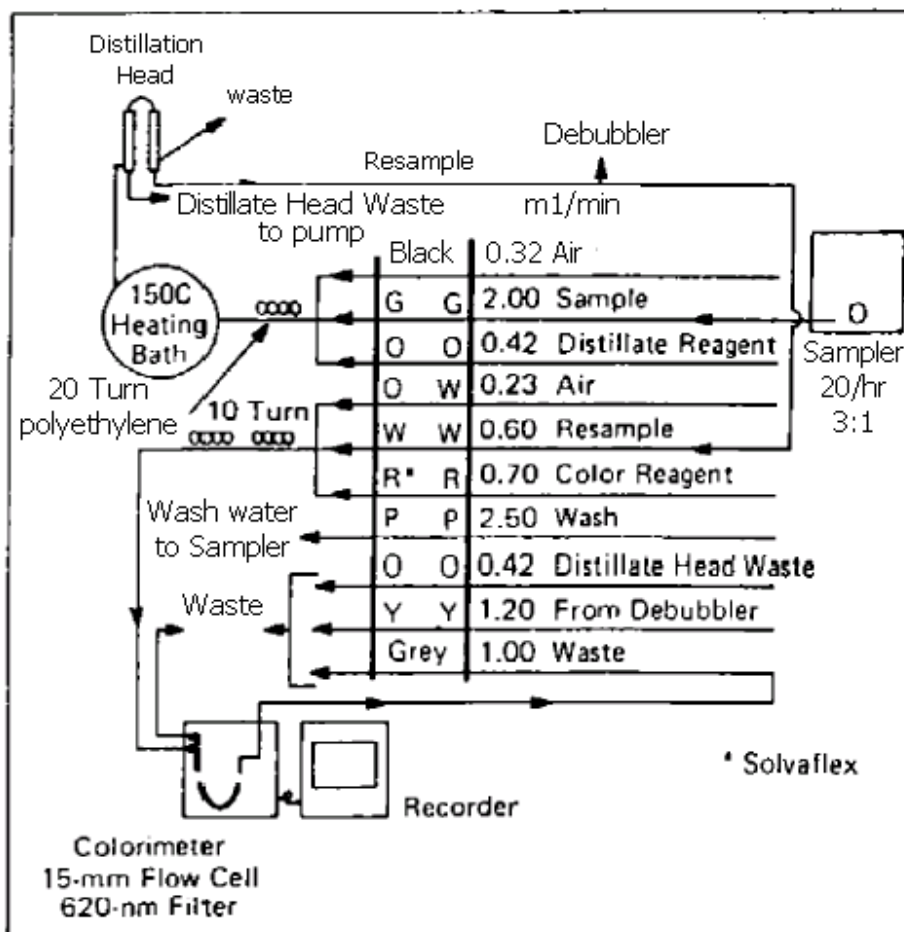
13.1 Physical and chemical characteristics of potable water. Appendix A defines the maximum and desired values of physical and chemical characteristics in separate tables.

**14. AUTOMATED LABORATORY EQUIPMENT FOR MONITORING WATER AND WASTEWATER**

Automated instrument are available and in use to monitor individual samples at rate of 10 to 60 samples/hr. The same instruments could be modified to make analysis for multiple constituents simultaneously from one sample. The read-out system includes sensing elements with indicators, alarms, and recorders.

Appropriate methodology could be supplied by the manufacturer for many of the common constituents of water and wastewater.

Monitoring of fluoride as a typical example could be totally automated. The basic of measurement is shown in Fig. 16. Uptaking of sample is automated and all valves which are indicated by different colors (B,O,Y,etc.) operated by computer in measuring time.



**FLUORIDE MANIFOLD**

Fig. 16

**15. MARKING**

Each equipment shall be marked using stamp and cast figure or metal nameplate with letters not less than 8 mm in height.

- 1) Name of manufacturer or identifying symbol.
- 2) Distinctive catalogue designation.
- 3) Date of manufacture.

**16. SHIPMENT**

Shipment of all equipment should be under warranty and unpacking must be within this period (time of insurance for shipment).

It must be supplied and rapped in moisture proof material and also it shall bear in a clearly visible manner, with appropriate instruction, for storage.

**17. SPARE PARTS**

Packaging list should be with instrument which include, instrument, spare parts and two manual books. Spare parts must be supplied for at least two years.

**18. PRE-INSTALLATION AND INSTALLATION**

Pre-installation and installation must be done by manufacturer or representative of supplier and purchaser. Representatives should witness tests carried out for installation and commissioning of equipment.

**19. WARRANTY**

The instruments should be under warranty for at least one year after test run.

**20. SERVICE AND INSPECTION**

Instruments should be inspected by qualified persons assigned by the manufacturer or user according to letter of agreement.

**APPENDICES**

**APPENDIX A**

**PHYSICAL CHARACTERISTIC OF POTABLE WATER**

CHARACTERISTIC	DESIRED THRESHOLD LIMIT VALUE (T.L.V)	MAXIMUM VALUE
Color	5 units	5 units
Odor	2 "	3 "
Turbidity	5 "	25 "
pH	7-8.5	6.5-9.2
Chemical characteristic of potable water mg/L		
CHARACTERISTIC	DESIRED (T.L.V)	MAXIMUM VALUE
As	0	0.05
Cd	0	0.01
Cn	0	0.05
Pb	0	0.1
Hg	0	0.001
Se	0	0.01
Cr	0	0.05
Ba	0	1
Ag	0	0.05
B	0	1
Hardness	150	500
Ca	75	200
Mg	50	150
Mn	0.05	0.5
Fe	0.3	1
Zn	5	15
Cr	0.5	1.5
So <sub>4</sub>	200	400
Cl	200	600
N	0.002	0.05
Detergent	0.1	0.2
P	0.1	0.2
Total Dissolved Solid	500	1500

"The guidelines for drinking water quality recommendation" Third edition 2004, (WHO), shall also be considered for characteristic of potable water.