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# H. W. LABORATORY MANUAL

## 100 AREA SECTION

Compiled by

Analytical Section
Technical Services Division

July 1, 1950

HANFORD WORKS
RICHLAND, WASHINGTON

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# H. W. LABORATORY MANUAL

## 100 AREA SECTION

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# H. W. LABORATORY MANUAL

# 100 AREA JOB HAZARDS BREAKDOWN

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# PREFACE

The purpose of this manual is to present a Hazard Breakdown of all jobs normally encountered in the laboratory work of the three sections comprising the Analytical Section, Metallurgy and Control Division of the Technical Department. A Hazard Breakdown is a careful analysis of any job in which the source of possible dangers is clearly indicated for each particular step. The analysis is prepared by individuals who are thoroughly familiar with the specific job or procedure. It is felt that if the hazards herein outlined are recognized by the Laboratory personnel and the suggested safety cautions followed, the chance for injury will be minimized and the worker will become generally more safety conscious.

The manual, which is prefaced by the general safety rules applying to all the laboratories, is divided into three main sections, one for each of the three sections into which the Laboratories Division is divided. These sections are as follows:

1. Section I - 200 Area Control
2. Section II - 100 Area Control
3. Section III - 300 Area Control, Essential Materials, and Methods Improvement

Each main section is subdivided into three parts, as follows: (1) Particular Safety Rules; (2) Cleaning and Decontamination and (3) Procedures and Methods. Each section contains its own index in order that quick reference may be made to any particular item.

Each Hazard Breakdown is composed of three parts which are as follows:

1. STEPS: Alogical segment of the operation when something happens to advance the work.
2. KEY POINTS: Anything in a step which might "make or break" the job, make the work easier to do, i.e., "knack" or "trick", special timing or bit of special information.
3. SAFETY CAUTIONS: Anything that might injure the worker or the equipment or make doubtful the final result. Special consideration is given for recognized hazards in the work.

The greatest benefit from a Hazard Breakdown goes to the individual worker who prepares the analysis since each step must be carefully studied. Once made, however, a Hazard Breakdown is a valuable means of safety training for a new employee and a convenient form from which to check-up on good safety practice.

The Hazard Breakdown analyses given in this manual are not to be considered as final. To date they have proven adequate but are subject to revision depending on future practice and experience.

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## PARTICULAR SAFETY RULES FOR PROCEDURES AND METHODS

1. Observe general laboratory Safety Rules

2. Eye protection must be worn at all times while in the laboratory.

3. Visually inspect all glassware for chips, breaks, etc., before cleaning.

    a. Broken glassware which can be repaired should either be placed in the space provided for glassware which can be repaired or should be repaired immediately.

    b. Broken glassware which cannot be repaired should be discarded immediately in the proper waste container.

4. Operating machinery must be enclosed by a suitable guard.

5. Wear gloves when working with machinery.

6. Never attempt to make any adjustment or repairs while machine is running or the switch is in the "on" position. "Tag out" all switches before making adjustments. See "Lock and Tag Procedure", Safety Bulletin No. 6.

7. Do not wear loose clothing around moving machinery.

8. Inspect all equipment for defects. Never use faulty equipment.

9. Exercise extreme care while working around hot equipment. All combustible material, especially solvent vapors, should be excluded from the vicinity of the furnace or oven.

10. Be sure hands are dry when operating electrical equipment.

11. Use tongs to remove dishes, crucibles, etc., from ovens and furnaces. Have a firm grip on the tongs. If muffle furnace is at a high temperature, asbestos gloves should be worn in addition to the use of tongs.

12. Conduct all solvent extractions in a well ventilated, flame-free hood bay, which can be isolated in case of fire.

13. Know the location and method of operating all nearby fire extinguishers.

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14. For extinguishing solvent fires, use $CO_2$ extinguisher, if available; otherwise, use a "Pyrene" extinguisher.

15. Avoid contact of the skin with organic solvents.

16. Inflammations of the skin can be caused by continued exposure to grease and oils. Protect hands while working with these materials.

17. Gloves should be worn at all times when either handling or working around live steam lines.

18. Burns resulting from fused caustic or strong caustic solutions are among the most serious encountered in laboratory work. Flush any such burns with copious quantities of water and report to First Aid IMMEDIATELY.

19. Perchloric acid is an extremely dangerous reagent and should not be used except when absolutely necessary and then only with written permission of the supervisor in charge.

20. Filtering

    a. Non-suction
    Be sure the equipment is stable. Support glass funnels in racks.

    b. Suction
    Be sure the equipment is stable. Use large suction flasks for large Buchner funnels, etc. If the rubber tubing connecting the suction flask to the vacuum outlet is short or rigid, the apparatus can be easily upset and care must be used. Pliable tubing is preferable.

21. Put on leather gloves before attempting to remove glass stoppers which have become stuck. Do not twist the stoppers. Try to remove by gentle prying motions or by the use of hot water. In difficult cases use the stopper removing apparatus; and if that is not successful, discard the bottle.

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## CLEANING SAFETY RULES

1. Observe general laboratory Safety Rules.

2. Eye protection must be worn at all times while in the laboratory.

3. Visually inspect all glassware for chips, breaks, etc., before cleaning.

4. a. Broken glassware which can be repaired should either be placed in the space provided for glassware which can be repaired, or should be repaired immediately.

   b. Broken glassware which cannot be repaired should be discarded immediately in the proper waste container.

5. Rubber gloves which have been previously tested for leaks should be used to protect the hands.

6. Buret stopcocks should turn freely, grease before storing.

7. Use tongs when handling hot objects.

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# MASTER SAFETY RULES

1. All injuries, no matter how slight, shall be reported to First Aid and your Foreman immediately.

2. No machine shall be operated unless all guards are in place.

3. Do not start any machine or equipment which has a "DANGER" tag attached. Do not remove any "DANGER" tag from any machine or equipment unless you placed it there.

4. No "HORSEPLAY" or scuffling is permitted.

5. Compressed air is dangerous. Do not apply to body or use to clean off clothing.

6. All projecting nails shall be removed or turned down immediately.

7. Do not carry bottles or other glassware in clothes pockets.

8. Broken glass shall be deposited in special containers marked "For Broken Glass Only".

9. Do not wear finger rings, loose clothing, wrist watches, long neckties, or sweat rags, while working around or operating moving machinery.

10. "Roped-Off-Area" at all times are danger zones and shall be respected as such. Admittance to or passage through "Roped-Off-Areas" are prohibited except to those employees working within the "Roped-Off-Area".

11. Approved type goggles shall be worn when chipping, grinding, welding, handling corrosive liquids, and working with compressed air or when necessary to be close to such an operation being performed by another employee. Familiarize yourself with departmental rules as to "when and where" to wear goggles.

12. Milk and soft drink bottles shall not be used as sample bottles, oil or soap containers.

13. Exit lights shall be lighted at all times.

14. Fire extinguishers, fire alarm boxes, gass masks and stretcher locations shall be kept plainly marked and free from any interfering obstacles that would delay emergency use of this protective equipment.

[redacted]

15. Safety showers shall not be blocked. They shall be inspected daily to insure proper lighting and operation, and results recorded.

16. Do not attempt to lift heavy loads without adequate assistance.

17. Oily rags, waste and other combustible materials shall be kept in metal containers provided for that purpose.

18. Do not use any broken or defective tools or equipment. Use only the proper tool or equipment for any job you do.

19. Good housekeeping is necessary to safety and shall be maintained at all times.

20. Stay away from the scene of a fire unless you are a member of the fire-fighting personnel.

21. Only authorized persons are permitted to operate valves, electric switch and other process equipment.

22. In addition to abiding by the Master Safety Rules, all employees shall familiarize themselves with and abide by the Area and Departmental Safety Rules.

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# GENERAL LABORATORY SAFETY RULES

The general rules listed below cover the usual hazards to be encountered in the analytical laboratories at Hanford Engineer Works. Hazards inherent to particular laboratories will be covered by special safety rules in the various laboratory manuals. The general rules contained herein together with the special rules in the manuals must be complied with by all employees. Any suggested additions to, or proposed changes in the rules should be submitted to your immediate supervisor.

## I. Housekeeping

In general laboratory work is of a hazardous nature; however, a neat and orderly laboratory does much to reduce the hazard of many chemical operations and therefore makes good housekeeping an essential to any laboratory.

The flollowing are housekeeping "musts" to be observed in all laboratories of this division:

A. Floors, shelves and tables must be kept free from dirt, dust and all excess chemicals and equipment.

B. Spills or leaks must be cleaned up promptly.

1. Flammable Material

If the spilled material is flammable, every precaution must be taken to avoid igniting the material by extinguishing all flames or electric heaters immediately.

2. Toxic Material

If the spilled material is toxic, personnel must be evacuated immediately and not allowed to return to the area until all necessary protective measures have been taken and the area approved for occupancy by supervision.

C. Waste receptacles must be supplied for materials of all kinds, and as many different receptacles must be provided as may be necessary to avoid mixing dangerous materials.

D. Flammable packing materials such as excelsior must be disposed of immediately.

E. Clothing, such as coveralls, hats, coats, etc., must not be stored in the working area.

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F. Food, such as lunches, candy, chewing gum, and fruit, must neither be kept nor eaten in the working area nor placed in a refrigerator used to store chemicals.

G. Equipment and Chemicals

1. A definite storage place must be provided for each laboratory item and the item placed therein when not in use.

2. Equipment stored on shelves must be placed so that it cannot be jarred off.

3. All necessary stacks of material or equipment must be made structurally sound.

4. Items dropped on the floor must be removed at once.

II. Safeguards

Before undertaking any job, the operator must know the method of attack, hazards involved, and the location of all safety and fire protection equipment in the vicinity.

A. Eye Protection

Eye protection must be worn at all times, in the laboratories and shops, except as follows:

1. Standard chipping goggles or cover-all goggles must be worn during mechanical operations, such as chipping or grinding, where particles may fly.

2. Face shields may be substituted for cover-all goggles if protection from liquids only is required.

3. Colored glasses of the approved type must be worn when the following operations are being performed:

   a. Blowing glass.

   b. Observing an electric arc.

   c. Working quartz with an oxygen torch.

Those employees desiring hardened lens spectacles with correction should obtain their prescription as soon as possible and place their order with the Safety Department, through their immediate supervision.

[redacted]

Plain hardened lens spectacles and goggles may be obtained from the Storeroom.

B. Foot Protection

1. Foot protection is recommended whenever heavy objects are handled.
2. Open toe shoes must not be worn in the laboratory.

C. Hand Protection

1. Rubber or rubber impregnated gloves must be worn whenever large quantities of acid or other corrosive liquids are handled.
2. Cotton or leather gloves must be worn when handling alkalies or rough and/or heavy objects.
3. The hands must be thoroughly scrubbed before eating, smoking or leaving the plant.

D. Smoking

Smoking will not be permitted in the laboratories of buildings 222, 231, 321, 3706 or in rooms 68 and 70 of building 3706, but will be permitted in the offices and corridors of the above buildings.

Smoking will be permitted in all other laboratories except in those areas designated as "No Smoking".

E. Coveralls

Coveralls must be worn by all personnel working in laboratories and shops of buildings 222, 231, 321, 3706 and in rooms 68 and 70 of building 3706.

F. Pipetting

Use of mouth suction in pipetting is not permitted; instead a syringe, rubber bulb or other approved device must be used.

G. Glassware

1. For High Pressure or Vacuum Operations
    a. Equipment for pressure or vacuum work must be carefully designed, constructed and annealed to minimize hazards

[redacted]

[redacted]

    resulting from breakage and the danger of collapse. All operations with the above equipment must be carried out behind adequate safety shields.

    b. Flat-bottomed flasks must not be used under vacuum unless made of heavy glass.

    c. Vacuum desiccators must be protected by a suitable shield or heavy wooden box.

    d. Full pressure of the plant air system must not be applied to glass apparatus.

    e. Dewar flasks must be of "Pyrex" rather than soft glass construction whenever possible. In either case the flask must either be taped or inserted in a metal container to protect the user from explosion and flying glass.

2. Broken or chipped glassware must never be used or placed in storage. If the glassware is worth saving, it should be thoroughly cleaned and repaired.

3. Beakers must be carried by gripping around the side and not by the top edge. Both hands must be used in carrying large beakers.

4. Bottles or other containers, when filled, must always be labled. Five-gallon bottles or larger must be carried in wooden crates or pails.

5. A dust pan and broom or counter brush must be used when cleaning up equipment which has been dropped on the floor.

6. All-glass hand-type wash bottles of the type shown on page 71 of the Central Scientific Catalog as item 10718, are not considered safe, and therefore must not be used in the laboratories.

H. Electrical Equipment

1. Repairs to ordinary electrical equipment must be made by an electrician.

2. Repairs to specialized instruments are to be made only by authorized persons.

3. Electrical grounding is required on all equipment connected to any plant electrical services.

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4. All electric ovens must be equipped with "explosion-proof" safety latches.

5. Care must be excercised to prevent the formation of explosive air-vapor mixtures on the inside of electric refrigerators. The presence of motor and lamp switches inside the box may be responsible for sparks which can set off air-vapor mixtures.

I. Compressed Gases and Liquids

1. Whenever use of a compressed gas or liquid for an extended period is anticipated the gas must be piped into the laboratory from cylinders located in an outside storage.

2. Where the quantity of gas required is small, small cylinders should be used, if possible.

3. In any case the cylinder must be securely strapped or chained in place.

4. When opening valves on gas cylinders, the operator should stand as far away as possible from the valve and reducing mechanism and to one side of the discharge pipe.

5. Cylinders containing more than a few pounds of pressure must be equipped with a regulating needle valve.

6. It is imperative that even traces of oil be avoided on reducing valve assemblies for oxygen cylinders; oil will ignite spontaneously in the presence of compressed oxygen.

7. Cylinders must not be heated unless approval of supervision is obtained.

8. In any case where the cylinders are used inside the laboratory the following precautions must be observed.

   a. The cylinder valve must be closed when the gas is not being used.

   b. Gas cylinders must neither be set up directly beside a source of heat nor placed in a hood where reactions with flammable materials are being carried out.

   c. All "open flames" must be extinguished in any hood where gases of a flammable nature are being used.

[redacted]

9. Dry ice or liquid air must never be stored in a container which can be accidentally stoppered.

J. General

1. All-Service gas masks are available for emergencies. All employees should become familiar with the location of these masks.

2. Adequate ventilation must be maintained in all working areas.

3. Labor crews must be requisitioned for moving heavy equipment, as defined by supervision, in the laboratories.

4. Respirators are available and must be worn where ordinary dusts are prevalent.

5. Working alone is allowable only with the permission of supervision.

6. The cover of a centrifuge must not be removed until rotation has ceased.

7. Molten materials, oil baths, etc., used at high temperatures must be handled and protected in such a way that the material will not spill or splash on the user should the container break.

8. Gas burners must be turned off at the petcock rather than at the base of the burner.

9. Organic solvents must not be stored in the laboratory in quantities greater than one gallon.

10. Petcocks on compressed air, gas, vacuum and water lines must be closed before leaving unattended. Changes in pressure may cause rubber tubing to become loosened with resultant damage.

11. Castalloy clamps are manufactured from an alloy which has a low melting point and a low tensile strength; consequently, these clamps must not be used to support vessels being heated directly by gas or electric heaters.

III. Handling Acids and Alkalies

A. Gloves and goggles must be worn whenever appreciable quantities of strong acids or alkalies are being handled.

[redacted]

B. Acids must be diluted by adding acid to water, not the reverse.

C. Safety carriers must be used when one quart or more of strong acids, alkalies, or other corrosive chemicals are being transported from one location to another.

D. Storage. Strong acids or alkalies in bottles of five pint capacity or smaller must be stored in trays or individual containers. Bottles with a capacity of greater than five pints must be stored in individual containers.

For all practical purposes a strong acid or alkali is defined as one having a concentration of 6N or greater.

E. Disposal of strong acids and alkalies. These must be drained into a sink in small quantities while flushing with a continuous stream of water. Materials, such as the above, must never be placed in refuse containers.

F. Spilled Acids and Alkalies

1. Acids must be neutralized by treatment with solid sodium carbonate or bicarbonate and then cleaned up by the method which best suits the needs of the case.

2. Alkalies must be neutralized with boric acid and then cleaned up as above.

[redacted]

[redacted]

# HAZARDOUS CHEMICALS

In general, it should be stated that any chemical compound should be classed as potentially hazardous and handled accordingly, unless it is either known or has been tested and proven to be non-hazardous.

When handling chemicals it should be borne in mind that in some cases seemingly small amounts may produce serious effects. This is especially true if the chemical or solution of the chemical is taken internally and/or enters the blood stream. In view of this fact it should be apparent why all open cuts must be treated at the First Aid station immediately and all possible care used while working in the laboratory until the cut has healed completely.

In all cases hazardous chemicals must be disposed of under designated supervision by the analyst performing the analytical work.

The following section is to serve as a guide in handling, use and disposal of chemicals which are dangerous either because of their corrosive, inflammable or explosive nature, or because of the toxicity of their vapors. All hazardous materials are not included in the following section, but an attempt has been made to list the more dangerous ones of those most likely to be met with in the plant laboratories.

## I. ACIDS

See Section III of "General Laboratory Safety Rules" for approved method of handling and disposal.

In addition, the following precautions must be observed:

### A. Hydrofluroic Acid (Anhydrous or 48% Solution)

Hydrofluoric acid is a violently corrosive and volatile acid which causes slow-healing burns on contact with the skin and membranes of the respiratory passages. Consequently, whenever large quantities of the acid are handled, adequate protective measures, such as rubber or leather gloves, goggles, face shield and rubber suit or apron, must be used. In addition all work with hydrofluoric acid must be carried out in a well ventilated hood, which is within reasonable proximity of a safety shower.

Disposal of the acid should be carried out in a well ventilated hood as follows:

[redacted]

1. 48% Aqueous Solution - Drown the acid in a large volume of water and then proceed as in Section III of the General Laboratory Safety Rules.

2. Anhydrous Acid - Collect acid in HF-resistant plastic beakers, then set beaker in back of hood and allow acid to volatilize. Label the beaker and place a "Caution" sign in the immediate vicinity of the beaker.

   Whenever it is suspected or known that hydrofluoric acid has come in contact with the skin, immediately wash the affected parts with lime water, rinse with copious amounts of water and then report to the First Aid station for treatment. In case the affected part is in the immediate vicinity of the eyes, neutralization by use of lime water must not be attempted; instead, wash with large volumes of water and then report to the First Aid station for treatment.

B. Organic Materials

Organic materials must never be treated with either nitric acid or mixtures containing nitric acid and other strong acids, except under controlled conditions and direct supervision.

C. Fuming Sulfuric Acid

Fuming sulfuric acid should be disposed of by adding the fuming acid to an equal volume of C.P. sulfuric acid. The acid mixture can then be handled as outlined in the General Laboratory Safety Rules.

D. Halogen Acids

Concentrated solutions of these acids are corrosive but the principle danger is due to the volatilization of acid vapors which irritate the membranes of the respiratory system. Whenever significant quantities of these acids are used the work must be carried out in a well ventilated hood, especially if solutions containing the acids are to be heated.

E. Osmic Acid

The vapors of cosmic acid are toxic and have an irritating effect on the eyes in very low concentrations; consequently, solutions of the acid should be kept in tightly stoppered bottles when not in use. All work connected with preparation of solutions from the solid acid should be carried out in a well ventilated hood.

[redacted]

## II. ALKALIES (Potassium and Sodium Hydroxides, etc.)

See Section III of the General Laboratory Safety Rules for approved method of handling and disposal.

When handling alkalies in the finely divided form, protective devices such as respirator and cover-all goggles must be worn.

## III. GASES

In general, gases are hazardous either because of (1) their explosive and flammable nature, (2) their poisonous nature (3) or because they reduce the amount of oxygen present in the atmosphere to such an extent that it will not sustain life. One or more of the above categories may be common to any one gas. Due to the hazardous nature of most gases, all work necessitating release of hazardous gases to the atmosphere must be conducted in an efficient fume hood. The following are examples of the more toxic gases commonly found in laboratories as the pure cylinder gas or as a product of a chemical reaction:

### A. Arsine

Arsine is one of the more toxic gases, being generated by the action of acids on impure metals and arsenides.

### B. Hydrocyanic Acid

Hydrocyanic acid is one of the more toxic of the common gases. The gas is generated by the action of dilute acids on cyanides; therefore, cyanides or solutions containing cyanides must be disposed of in a hood sink, while flushing the sink and drain with a constant stream of water.

### C. Hydrogen Sulfide

Hydrogen sulfide ranks close to arsine and hydrocyanic acid in toxicity. The gas is prepared by the action of acids on metallic sulfides and is one of the more common laboratory gases.

### D. The Sulfur Oxides

The sulfur oxides rank with hydrogen sulfide in toxicity and are prepared either by the action of acids or heat on sulfites or may be encountered in work with sulfurous and fuming sulfuric acids.

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E. The Nitrogen Oxides

The nitrogen oxides, with the exception of nitrogen tetroxide, are extremely dangerous due to the fact that they lack a characteristic odor which would warn of their presence. Nitric acid and hot, strongly acid solutions of nitrates or nitrites are the principal sources of the nitrogen oxides.

F. The Halogens (Bromine, Chlorine, Fluorine, Iodine)

The halogens and their vapors are powerful irritants, readily blistering the skin and causing serious inflammation of the respiratory system, which usually results in "chemical" pneumonia.

## IV. METALS AND THEIR SALTS

A. Mercury

Mercury spills must be cleaned up at once, since mercury has an appreciable vapor pressure and prolonged exposure to the vapors will result in mercury poisoning.

B. Potassium and Sodium

Potassium and Sodium react violently with water and acids, liberating free hydrogen and heat. The quantity of heat liberated in this case is sufficient to ignite the hydrogen and result in a serious explosion. Potassium reacts rapidly with oxygen of the air forming peroxides and liberating quantities of heat sufficient to melt the metal and set fire to objects near by.

Potassium must be protected from oxygen and moisture by storing under dry benzene or kerosene. In the case of sodium, storage under benzene or in sealed containers is satisfactory. When handling these metals, long handled forceps or tongs must be used.

Small scraps of the metal must be disposed of by placing in butyl or some other "higher" alcohol, to which small quantities of methanol are added as necessary, under designated supervision.

C. The Salts of Antimony, Arsenic, Copper, Lead, Mercury and Silver

These salts are particularly hazardous due to their poisonous nature; therefore, all due precautions should be observed when handling them.

[redacted]

## V. OXIDIZING MATERIALS

### A. Perchloric Acid

Perchloric acid or its compounds must never be used unless under written instructions from the laboratory supervisor. In any case where permission is granted, the following regulations will apply:

1. Only persons thoroughly familiar with the properties of the chemical, the hazards involved and the precautionary measures necessary should be allowed to use these materials.

2. Perchlorates must not be used where other safer drying or oxidizing agents will give satisfactory results.

3. Anhydrous perchloric acid is a hazardous reagent; consequently, all reactions must be avoided where there is a possibility of forming the anhydrous acid as an end product.

4. While perchloric acid of 70% strength may be boiled without harm at approximately 200°C., it cannot be too strongly emphasized that contact of the boiling undiluted acid or hot vapor with organic matter, or even easily oxidized inorganic materials such as compounds of trivalent antimony, will lead to serious explosions. In any case where oxidizable substances are present, nitric acid must always be added to destroy the easily oxidizable material before the addition of perchloric acid. Contact of rubber with perchloric acid is particularly hazardous; consequently, rubber gloves, etc. should never be used where contact with this acid or its vapors is possible.

5. Perchloric acid or its compounds must always be used in a fume hood, with the window lowered to protect the face. Tongs must be used in handling beakers, etc., containing these materials. In any case where the above materials are used frequently, the hood walls should be thoroughly washed at least once per week, due to the possibility of forming explosive compounds by the reaction of perchloric acid with ammonia and nitric acid fumes.

### B. Peroxides

Peroxides are strong oxidizing agents which present a definite fire hazard in the presence of organic matter, such as paper, excelsior, etc.

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### C. Permanganates

Permanganates are strong oxidizing agents and as such react with easily oxidizable materials, such as organic matter, sulfur, sulfides, etc., to form explosive mixtures.

## IV. ORGANIC MATERIALS

Indiscriminate use of organic solvents in the laboratory usually results in explosion and fire hazards in addition to the usual hazard due to the toxicity of the material being handled. Confine all work with hazardous organic solvents either to a flame-free, well-ventilated room or to a flame-free bay of a fume hood. Organic solvents miscible with water may be discarded in a sink drain, accompanied by a steady stream of water. Non-miscible, flammable solvents in appreciable quantities must be collected in a closed metal container and disposed of.

Many organic solvents boil at low temperatures and care should be taken to store them at temperatures well below their boiling points.

Anhydrous ethers, on standing form unstable, explosive peroxides; however, formation of the peroxides can be retarded by (1) storing the ether in a dark place, (2) storing in tin lined containers, and (3) in the case of bottles, by the addition of a coil of fine copper wire to the container. In any case, the ether should be checked periodically for peroxide content by use of the method outlined in the laboratory manual (Essential Materials Code: EE-1a). Ethers containing more than 0.2 percent of peroxides as hydrogen peroxide must not be distilled or otherwise evaporated to dryness.

Carbon tetrachloride should not be used in extinguishing organic chemical fires if a more suitable extinguisher, such as carbon dioxide, etc., is available, due to the fact that phosgene is formed as a reaction product. When fires of this nature are extinguished with carbon tetrachloride all personnel should be evacuated and not allowed to return to the laboratory until supervision has definitely ascertained that the area is a safe working place.

[redacted]

# TABLE OF HAZARDOUS CHEMICALS

| SUBSTANCE | PERSONNEL HAZARD | FIRE HAZARD |
|---|---|---|
| 1. ACETIC ACID (Glacial) | 1. A corrosive chemical-which may cause painful burns to the skin. Inhalation of the fumes may cause irritation to mucous membranes. | 1. Dangerous in contact with cromic acid, sodium peroxide, or nitric acid; yields moderately flammable vapors above flash-point 104°F. Forms explosive mixtures with air. |
| 2. AMMONIA (ANHYDROUS) | 2. Irritant, especially in concentration of the order of ½% by volume. | 2. Not flammable in air except in comparatively high concentrations or when mixed with oil. |
| 3. AMMONIUM HYDROXIDE | 3. Irritant, especially in concentrated form. | 3. Decomposes explosively at 210°C. |
| 4. AMMONIUM NITRATE | 4. ---- | 4. ---- |
| 5. AMMONIUM SULFIDE | 5. Solution hydrolyzes and gives off hydrogen sulfide fumes which are toxic and care must be taken that the fumes are not inhaled during the preparation and subsequent storage of the solution. | 5. ---- |
| 6. ARSENIC | 6. All solutions and compounds of arsenic are poisonous and care must be taken to avoid ingestion, inhalation and contact with the skin. | 6. ---- |
| 7. AQUA REGIA | 7. Corrosive, suffocating, inhalation of fumes or contact with skin must be avoided. | 7. Dangerous, volatile, violently corrosive and will promote combustion in contact |

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| SUBSTANCE | PERSONNEL HAZARD | FIRE HAZARD |
|---|---|---|
| | | with most organic substances. |
| 8. BARIUM CHLORIDE | 8. Poisonous, avoid ingestion and inhalation. | 8. ---- |
| 9. BARIUM NITRATE | 9. Soluble in water, poisonous if taken internally. | 9. Oxidizing material. Hazard in a class with sodium peroxide. |
| 10. BARIUM PEROXIDE | 10. POISONOUS | 10. DANGEROUS fire hazard. |
| 11. BROMCRESOL GREEN | 11. Compares to Phenol-poisonous. | 11. ---- |
| 12. BROMINE (WATER) | 12. Corrosive at ordinary temperatures, gives off poisonous, suffocating vapors, burns skin. | 12. Causes oxidizing effect, resulting in heating and may cause fire when in contact with organic material. |
| 13. BARIUM HYDROXIDE | 13. CAUTION, Poisonous, absorbs carbon dioxide from the air; keep well stopered! | 13. ---- |
| 14. N-BUTYL DIETHANOL-AMINE | 14. Irritant to mucous membranes. | 14. DANGEROUS fire hazard, flash point 245°F. |
| 15. CARBON TETRACHLORIDE | 15. Poisonous, avoid ingestion. | 15. Non inflammable. |
| 16. COPPER NITRATE | 16. Poisonous, when taken internally. | 16. Oxidizing material, hazard classes with sodium nitrate. |
| 17. COLLODION | 17. ---- | 17. Dangerous fire hazard, flash point below 80°F., keep lights and fire away. |
| 18. CALCIUM CARBIDE | 18. Serious under fire conditions | 18. Gives off acetylene gas on contact with water or moisture. |

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| SUBSTANCE | PERSONNEL HAZARD | FIRE HAZARD |
|---|---|---|
| 19. CAMPHOR | 19. ---- | 19. Flammable; gives off flammable vapors when heated which may form explosive mixture with air. Flash point 180°F. |
| 20. CARBOLIC ACID (See Phenol) | 20. ---- | 20. ---- |
| 21. CARBON DISULFIDE | 21. Toxic. 3200 to 3850 parts of vapor per million (0.32 to 0.385% by volume) may cause dangerous illness in ½ to 1 hour. Direct contact with the skin should be avoided. Products of combustion contain sulphur dioxide, which in concentrations of 0.2% by volume in air may cause serious injury in ½ hour or less. Often poisonous carbon monoxide is present in the products of combustion. | 21. A highly volatile liquid with an offensive odor, giving off even at comparatively low temperatures vapors which form with air flammable and explosive mixtures. Flash point 30°C, (22°F) It is endothermic, and the vapor may be ignited by a heavy blow. The vapors are heavier than air and may trave a considerable distance to a source of ignition and flash back. More hazardous than gasoline. |
| 22. CHLORINE | 22. Corrosive. Irritating to the eyes and mucous membranes. Toxic 0.004% to 0.006% by volume in air causes dangerous illness in ½ to 1 hour. | 22. Is not combustible in air but reacts chemically with many common substances and may cause fire or explosio when in contact with them. It is considere dangerous to neutraliz chlorine in a room wit ammonia. |
| 23. CHROMIC ACID (Chromium Trioxide or Chromium Anhydride) | 23. Irritating to skin. Poisonous. | 23. Oxidizing material; will ignite on contact with acetic acid and |

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| SUBSTANCE | PERSONNEL HAZARD | FIRE HAZARD |
|---|---|---|
| | | alcohol. Chars organic material such as wood, sawdust, paper, or cotton, and may cause ignition. Combustible material in presence of chromic acid when ignited burns with great intensity. May cause explosion in fire. |
| 24. ETHYL ETHER | 24. Anaesthetic | 24. A highly volatile liquid, giving off even at comparatively low temperatures vapors which form with air or oxygen flammable and explosive mixtures. Explosive range 1.85% to 36.5% upward propagation. More hazardous than gasoline. |
| 25. HYDROCHLORIC ACID | 25. Aqueous solution is corrosive, irritating and poisonous. Fumes are corrosive and irritating to the mucous membranes. | 25. Not combustible, but if allowed to come in contact with common metals hydrogen is evolved, which may form explosive mixtures with air. |
| 26. HYDROCYANIC ACID (Prussic acid) | 26. POISONOUS. Avoid contact with skin; avoid inhalation. | 26. Forms flammable and explosives vapors with air. |
| 27. HYDROFLUORIC ACID (HF) | 27. Acid and its vapors highly toxic and irritating to the skin and eyes and to the respiratory tract. Fumes produced by contact with ammonia and many other metals poisonous. | 27. Colorless, volatile liquid. Not combustible but reacts with glass and most substances, platinum being an exception, aqueous solution also attacks glass and several metals. |

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| SUBSTANCE | PERSONNEL HAZARD | FIRE HAZARD |
|---|---|---|
| 28. HYDROGEN PEROXIDE | 28. Prolonged exposure to vapor irritating to eyes and lungs. Causes skin irritation. | 28. Oxidizing liquid, may cause ignition of combustible material. |
| 29. HYDROGEN SULFIDE | 29. Toxic. Irritant to eyes and respiratory tract after exposure in concentrations as low as 1 part in 10,000 by volume, higher concentrations dangerous. | 29. Flammable gas. Forms flammable and explosive mixture with air or oxygen. |
| 30. HEXONE | 30. Inhalation of vapors causes irritation of eyes and mucous membranes at concentrations below 0.1% by volume. | 30. Forms explosive mixture with air or oxygen. |
| 31. FLUORIDE | 31. Same as hydrofluoric acid. | 31. Same as hydrofluoric acid. |
| 32. HYDROXYLAMINE | 32. Poisonous, avoid ingestion. Has a corrosive action on the skin. | 32. ---- |
| 33. HYDROXYLAMINE HYDROCHLORIDE | 33. Same as above | 33. ---- |
| 34. IODINE | 34. Poisonous and corrosive, same as chlorine. | 34. Corrosive, same as chlorine. |
| 35. LEAD ACETATE | 35. Poisonous, avoid oral contact. | 35. ---- |
| 36. LEAD NITRATE | 36. Poisonous, avoid oral contact. | 36. Oxidizing material classes with sodium nitrate. |
| 37. METHANOL | 37. Poisonous, avoid ingestion. | 37. Volatile, inflammable liquid. |

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| SUBSTANCE | PERSONNEL HAZARD | FIRE HAZARD |
|---|---|---|
| 38. MERCURY | 38. Poisonous, clean up any spill immediately to avoid prolonged inhalation of vapors. | 38. ---- |
| 39. MERCURIC CHLORIDE | 39. Poisonous and corrosive. | 39. ---- |
| 40. NICKEL NITRATE | 40. Poisonous when taken internally. | 40. Oxidizing material, classes with sodium nitrate. |
| 41. NITRIC ACID | 41. Corrosive, causes severe burns by contact. DANGEROUS to inhale fumes. | 41. Caustic and corrosive, may cause ignition when in contact with combustible materials; corrodes iron or steel; may cause explosion when in contact with hydrogen sulfide or certain other chemicals. |
| 42. OSMIUM TETROXIDE | 42. Vapor irritating when breathed, highly poisonous. | 42. ---- |
| 43. OXALIC ACID | 43. Poisonous, has a local caustic action on the skin and mucous membranes, and causes discoloration and brittleness of the nails. It causes these symptons both as a dust and as a solution formed with the moisture in the atmosphere. | 43. ---- |
| 44. OSMIC ACID | 44. The vapors of osmic acid are toxic and have an irritating effect on the eyes even in very low concentrations; | 44. ---- |

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| SUBSTANCE | PERSONNEL HAZARD | FIRE HAZARD |
|---|---|---|
| | consequently, solutions of the acid should be kept in tightly stoppered bottles when not in use. All work connected with preparation of solutions from the solid acid should be carried out in a well ventilated hood. | |
| 45. PERCHLORIC ACID | 45. Perchloric acid or its compounds MUST NEVER be used unless under written instructions from supervision. In any case where permission IS granted Laboratory Safety Rules Handbook, Part III, p. 6 MUST be closely followed. | 45. ---- |
| 46. PERMANGANATES | 46. Permanganates are strong oxidizing agents and as such react with easily oxidizable materials, such as organic matter. | 46. ---- |
| 47. PEROXIDES | 47. ---- | 47. Peroxides are strong oxidizing agents which present a definite fire hazard in the presence of organic matter, such as paper, excelsior, etc. |
| 48. PHOSPHOROUS, RED | 48. Yields toxic fumes when burning. | 48. Flammable. Explosive when mixed with oxidizing materials. Not |

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| SUBSTANCE | PERSONNEL HAZARD | FIRE HAZARD |
|---|---|---|
| | | as dangerous to handle as white and more easily extinguished when afire. |
| 49. PHOSPHOROUS, WHITE (Or Yellow) | 49. Poisonous. Serious under fire conditions. Yields highly toxic fumes when burning. Contact of phosphorous with the skin causes severe burns. | 49. Highly flammable. Explosive in contact with oxidizing material. Ignites spontaneously on contact with air. |
| 50. PLUTONIUM | 50. Highly toxic. Emits alpha and beta particles. Cuts or scratches should be immediately treated to prevent contamination of the blood stream. | 50. ---- |
| 51. POTASSIUM (METALLIC) | 51. Strong caustic reaction. DANGEROUS. | 51. Oxidizes rapidly on exposure to atmosphere, igniting spontaneously if warm enough. Water is decomposed suddenly by contact with potassium, sufficient heat being generated to ignite spontaneously the evolved hydrogen, (in the presence of air). Its reaction with water is more violent than that of sodium. |
| 52. POTASSIUM CHLORATE | 52. Dangerous under fire conditions. Poisonous. | 52. Oxidizing material; explosive when in contact with combustible material. (Must not be ground with sugar, sulfur or other |

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| SUBSTANCE | PERSONNEL HAZARD | FIRE HAZARD |
|---|---|---|
| | | combustible substance as it may cause explosion). |
| 53. POTASSIUM CYANIDE | 53. Highly poisonous when taken internally. Evolves hydrocyanic acid gas (poisonous) on contact with acids or moisture. | 53. Cyanides are not flammable but evolve hydrocyanic acid on contact with acids or moisture. |
| 54. POTASSIUM HYDROXIDE | 54. Extremely caustic; may cause severe burns. | 54. Generates heat on contact with water. Classes with calcium oxide (lime) in hazard. |
| 55. POTASSIUM NITRATE | 55. ---- | 55. In contact with organic materials causes violent combustion on ignition. Classes with sodium nitrate. |
| 56. POTASSIUM PERCHLORATE | 56. ---- | 56. Oxidizing material. Combustible in contact with organic materials. More stable than chlorates. Explosive in contact with concentrated sulfuric acid. |
| 57. POTASSIUM PERMANGANATE | 57. ---- | 57. Oxidizing material. Explosive when treated with sulfuric acid, and in contact with alcohol, ether, flammable gases, and combustible materials. |
| 58. POTASSIUM PEROXIDE | 58. Strong caustic reaction and dangerous under fire conditions. Avoid breathing | 58. Does not burn or explode but mixtures of potassium peroxide and combustible |

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| SUBSTANCE | PERSONNEL HAZARD | FIRE HAZARD |
|---|---|---|
| | dust in handling, and wear goggles to protect eyes. | substances are explosive and ignite easily even by friction or on contact with a small amount of water. Reacts violently with water and in large quantities this reaction may be explosive. |
| 59. POTASSIUM SULFIDE | 59. Yields irritating and corrosive gases when burning. Deliquescent in air, keep well stoppered. | 59. Moderately flammable; yields flammable hydrogen sulfide on contact with mineral acids and sulfur dioxide when burning. |
| 60. SILVER NITRATE | 60. Corrosive and poisonous. | 60. Oxidizing material. |
| 61. SODIUM | 61. Strong caustic reaction, Dangerous. | 61. Water is suddenly decomposed by contact with sodium with the evolution of hydrogen, which may ignite spontaneously (in the presence of air). Classes with potassium in respect to fire hazard but its reaction is less violent than that of potassium. |
| 62. SODIUM CHLORATE | 62. Dangerous under fire conditions. | 62. Oxidizing material; explosive when in contact with combustible material. |
| 63. SODIUM CHLORITE | 63. Poisonous when taken internally. Dangerous. | 63. Strong oxidizing material. Decomposes with evolution of heat at about 175°C. (347°F). Explosive in contact with combustible material. In |

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| SUBSTANCE | PERSONNEL HAZARD | FIRE HAZARD |
|---|---|---|
| | | contact with strong acid liberates chlorine dioxide, an extra hazardous gas. |
| 64. SODIUM CYANIDE | 64. Same as Potassium Cyanide. | 64. ---- |
| 65. SODIUM NITRATE | 65. ---- | 65. Oxidizing material-in contact with organic or other readily combustible substances will cause violent combustion on ignition. |
| 66. SULFUR | 66. When burning, forms sulfur dioxide, which in concentrations of 0.2% by volume in air may cause serious injury in ½ hour or less. | 66. Flammable. Dust or vapor forms explosive mixtures with air. Hazardous in contact with oxidizing material. |
| 67. SULFURIC ACID | 67. Corrosive; dangerous fumes under fire conditions. | 67. May cause ignition by contact with combustible materials. Corrodes metal. |
| 68. URANIUM | 68. Highly toxic. See plutonium. | 68. ---- |
| 69. ZINC CHLORATE | 69. Serious under fire conditions. | 69. When in contact with organic material explodes by slight friction, percussion or shock. |
| 70. ZINC, POWDER OR DUST | 70. Zinc is comparatively volatile at elevated temperatures: Under fire conditions precautionsshould be taken to avoid breathing fumes, which may | 70. Hydrogen is evolved when commercial zinc is in contact with acids, sodium hydroxide, or potassium hydroxide. Hydrogen is also evolved by |

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| SUBSTANCES | PERSONNEL HAZARD | FIRE HAZARD |
|---|---|---|
| | cause metal fume fever. | acid forming combinations containing zinc, such as zinc chloride and moisture. Dust may form explosive mixtures with air. Zinc dust in bulk in a damp state may heat and ignite spontaneously on exposure to air. |

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# CLEANING OF CHEMICAL GLASSWARE

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Rinse the glassware with water after use. | | 1. All glassware should be cleaned immediately after use. |
| 2. Wash the glassware with soap and water. | | 2. Apply the soap with the proper size brush. |
| 3. Rinse the glassware with water. | | 3a. When using a hot water rinse, use tongs to handle the glassware. |
| | | b. Flow of water from the faucet should not be too rapid. |
| 4. Clean the glassware with sulfuric acid-dichromate cleaning solution. | | 4. Sulfuric acid-dichromate cleaning solution is highly corrosive; use clean rubber gloves which have been previously tested for leaks and found in good condition when working with this solution. |
| 5. Rinse the glassware with tap water and then with distilled water. | 5. Residual cleaning solution can be as disastrous to an analysis as dirty glassware. | 5. Flow of water from the faucet should not be rapid enough to cause splashing. Local concentrations of water and acid should be avoided. |
| 6. Return the glassware to the proper storage place. | | |

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| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Transfer the sample to the proper container and place container in the centrifuge cup. | 1a. Be sure rubber cushions are in cups.<br>b. If only one sample is to be run, use tube weighed with water to counterbalance the tube containing the sample. | 1. Be sure opposing tubes are counterbalanced. Due to the speed of rotation of the centrifuge "head", considerable centrifugal force is developed. An unbalanced machine presents a serious hazard due to the possible failure of components under the strain. |
| 2. Centrifuge the sample for the required period of time. | 2a. Increase speed slowly.<br>b. When operation is complete, turn off current and slowly "brake" machine to a dead stop. | 2. Close and lock lid. Do not open until rotation has ceased completely. |

The above Job Break-Down Method covers WS-11c, colorimetric Sodium.

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## SETTING UP ACID DISPENSING BURET OR REPLACING STOCK BOTTLE OF ACID

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Drain buret and dispensing tip. | | 1. Remove the last drop of acid from the dispensing tip by touching the tip against the side of a beaker. |
| 2. Remove the buret from the stock bottle and rinse with cold water. | | 2. Flow of water should not be strong enough to cause splashing outside sink. |
| 3. Clean and dry buret by method described on page 2 in "Cleaning" section. | | 3a. Visually inspect the buret for breaks, cracks, etc.<br>b. Follow safety cautions outlined in "Cleaning of Chemical Glassware". |
| 4. Remove cap from acid bottle and attach polystyrene adapter. | | 4. Open bottle carefully to avoid spurting. |
| 5. Apply stopcock grease to ground glass joints of buret and place stem of buret in bottle. | | 5. Care should be excersized to prevent splashing on the body. |
| 6. Place bottle in assigned location and attach tubing from the vacuum line | | |
| 7. Fill buret | 7. Burets are provided with a side arm for an aspirator bulb if a vacuum filling system is not to be used. | 7. Wash hands and forearms thoroughly when work has been completed. |

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| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Assemble apparatus. | 1a. All parts should be supported and balanced properly. | 1a. All connections should be tight so that no vapors can escape. |
| | b. Water should be flowing up through the condenser. | b. In steam distillations all ground glass joints should be properly seated, well greased, and clamped in place. Be sure the proper valves are open and closed. |
| | c. If volatile and/or inflammable vapors will distill over, place the apparatus in hood and observe all necessary cautions. | c. Add four or five glass beads to distilling flask to prevent bumping. |
| | d. The distilling flask should not be more than two-thirds full. | |
| 2. Distill sample. | 2a. Watch for possible leakage from (1) top of distilling flask and (2) between condenser and side arm. | 2a. Keep the distilling flask supplied with solution. |
| | b. Adjust intensity of heat and quantity of solution in distilling flask so that solution can not boil over into condenser. | b. If the end of the condenser is below the surface of the distillate, either remove collecting flask or remove stopper of distilling flask before reducing heat. |
| 3. Dismantle, clean, and store apparatus. | | 3. If parts are hot, follow laboratory safety rules. |

The above Job Break-Down covers Aluminoid Nitrogen, methods WN-2a and 3a.

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| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Place the crucibles etc. in the oven. | 1. Equipment should always be in an oven so a free circulation of air is assured. | |
| 2. Ignitions (when called for in the analytical procedure). | | 2. Ignitions should be carried out in a well-ventilated hood. |
| a. Over an open flame. | a. If paper is used as the filtering medium, "smoke off" the paper at low temperature before igniting. | a. 1) When igniting materials over an open flame, place the crucibles on a firm base so there will be no danger of tipping.<br>2) When a burner "flashes back", turn the gas off at the petcock, rather than at the base of the burner. Allow the base of the burner to cool before adjusting and lighting again. |
| b. By use of a muffle. | b. Dry first in oven. | b. 1) Keep hands, arms and clothing away from open muffle door.<br>2) When observing items in furnace held at temperatures exceeding 1500°F, use a |

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| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| | | Cobalt glass shield to protect the eyes. |
| 3. Remove the crucibles etc. from the oven or muffle furnace and place in a desiccator to cool. | 3a. If several hot crucibles etc. are placed in the desiccator at the same time, the lid should be left ajar until the crucibles have cooled somewhat. This will prevent the formation of a partial vacuum in the desiccator, which sometimes causes the lid to "freeze".<br>b. The desiccant should be changed at least once per month in all desiccators; oftener if necessary. | 3. When moving a desiccator, grasp it by the flange so that the lid cannot slide off. |

The foregoing Job Break-Down covers the following analytical methods:

1. Magnesium, Gravimetric------------WM-2a
2. $R_2O_3$------------------------------WR-1a
3. Suspended Silica------------------WS-2a
4. Silica in Residues----------------WS-3a
5. Dissolved and Suspended Solids----WS-4a and 5a

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| STEPS | KEY POINTS | SAFEY CAUTIONS |
|---|---|---|
| 1. Set up and adjust stirrer. | 1. Adjust level of stirrer and position of container so that stirrer paddle will not strike sides or bottle of vessel or break surface of the liquid. | 1a. Be sure motor is securely loced in position and that stirring assembly is true and well balanced. |
| | | b. If an air stirrer is being used, the air line should be free of kinks and other obstructions. |
| 2. Operation of the stirrer. | 2a. Apply power slowly. | 2a. Hands should be dry when operating electrical equipment. |
| | b. Be sure that all rotation has ceased before removing stirrer paddle from liquid. | b. Wash stirring assembly immediately after use. |

The above Job Break-Down covers method WA-3a.

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| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Pour solution to be measured into a clean beaker. | 1. Never pipet directly from reagent or sample bottle. | 1a. If solutions are strongly acidic or alkaline, treat as instructed in "General Laboratory Safety Rules".<br>b. Volatile solutions such as concentrated nitric acid and hydrofluoric acid should be pipetted in the hood. |
| 2. Attach rubber bulb to end of pipet. | 2. Never pipet by mouth. | 2a. Hold pipet firmly but avoid pressing excessively with fingers.<br>b. Apply bulb to the pipet stem so as to establish the minimum contact necessary for a vacuum. If the bulb is on too far or fits too tightly, it cannot be quickly removed and there is danger of breakage or spilling. |
| 3. Draw solution into pipet. | 3. Control the bulb with one hand and grasp the pipet firmly with the other hand at a point low enough to prevent wobbling of pipet and possible upsetting of beaker. | 3a. Do not force pipet into bottom of beaker.<br>b. Draw up solution slowly so as to prevent any solution from going up into the bulb. If solution does get into bulb, it should be immediately cleaned and another clean, dry bulb should be used. |

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 4. Release bulb and adjust solution level within pipet with finger or thumb. Deliver measured amount into nearby container. | | 4a. Spilling drops of solution can be minimized by (1) touching pipet tip to side of beaker before delivering solution and by (2) having awaiting beaker nearby.<br>b. If any solution is spilled, clean it up immediately. |
| 5. Clean pipet and place in pipet rack. | 5. Pipets should never be left lying around whether clean or dirty. | 5. Pipets should be cleaned immediately after use. |

## ANALYTICAL METHODS COVERED

| | Constituent | Method |
|---|---|---|
| 1. | Sodium Silicate (Alk.) | WA-3a |
| 2. | Calcium | WC-1a |
| 3. | Copper, Colorimetric | WC-3a |
| 4. | Chlorides | WC-4a |
| 5. | Fluorine | WF-1a |
| 6. | Hydrogen Peroxide | WH-2a |
| 7. | Iron (0.3 to 1.5 ppm) | WI-1a |
| 8. | Magnesium | WM-1a and 2a |
| 9. | Manganese | WM-3a and 3b |
| 10. | Nickel | WN-1a |
| 11. | Albuminoid Nitrogen | WN-2a and 3a |
| 12. | Nitrate Nitrogen | WN-4a |
| 13. | Dissolved Oxygen | WO-1a |
| 14. | $R_2O_3$ | WR-1a |
| 15. | Sulphate (Turbidimetric) | WS-6a |
| 16. | Sodium Dichromate | WS-7a |
| 17. | Sodium (Colorimetric) | WS-11a |

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Prepare sample bottles. (Bottles should be cleaned by the method outlined in "Cleaning Chemical Glassware"). | 1. Bottles must be thoroughly clean and equipped with the proper size stopper. | 1. When cleaning bottles, use method outlined in "Cleaning Chemical Glassware". |
| 2. Collect the sample. | 2. In case it is necessary to adjust a sampling valve, wait 10 minutes after adjusting before sampling. | 2a. Keep walkways clear; also clean and dry.<br>b. Use a bottle carrier. Never carry bottles in hands or in pockets of clothing.<br>c. Use handrails when ascending or descending stairs.<br>d. Laboratory personnel are not cleared to operate valves other than sampling taps. Valves which have been tagged out with "Do Not Operate" signs should be operated only by the appropriate Operating Supervisor. |

Analyses covered by the Above Job Break-Down:

1. Alkalinity--------------WA-2a
2. Calcium-----------------WC-1a
3. Copper------------------WC-3a
4. Chloride----------------WC-4a
5. Free Chlorine-----------WC-5a
6. Iron--------------------WI-1a
7. Magnesium---------------WM-1a
8. Manganese---------------WM-3a
9. Diss. Oxygen------------WO-1a
10. pH----------------------WP-1a
11. Diss. Silica------------WS-1a and 1b
12. Diss. Solids------------WS-4a
13. Sulfate-----------------WS-6a
14. Sod. Dichromate---------WS-7a
15. Turbidity---------------WT-1a and 1b

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Clean equipment by method outlined in "Cleaning Chemical Glassware". | | 1. Follow precautions outlined in "Cleaning Chemical Glassware". |
| 2. Measure accurately the desired quantity of chemical or reagent. | | 2a. With corrosive chemicals, wear rubber gloves and coverall goggles.<br>b. Clean up spills immediately.<br>c. With poisonous chemicals, wash hands after handling.<br>d. Use inflammable chemicals only in a flame-free area. Know the location of the nearest fire extinguisher and how to use it.<br>e. For fuming chemicals all work should be performed in a well-ventilated hood.<br>f. Know the hazard of the chemical used. |
| 3. Transfer chemical or reagent to a measured portion of the water to be used for solution. | | 3a. Avoid splashing.<br>b. Always add acids to water; <u>Never</u> the reverse.<br>c. Add reagent slowly; if heat is evolve, allow solution to cool between additions. |

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| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 4. Dilute solution to volume and shake thoroughly. | | |
| | | 5. Wash hands and fore-arms thoroughly. |

The foregoing Job Break-Down covers the following analytical Methods:

1. Alkalinity of Sodium Silicate--------------WA-3a
2. Calcium------------------------------------WC-1a
3. Copper, Colorimetric-----------------------WC-3a
4. Chlorides, by Spectrophotometer------------WC-4a
5. Fluorine-----------------------------------WF-1a
6. Soap Hardness------------------------------WH-1a
7. Hydrogen Poroxide, Colorimetric------------WH-2a
8. Magnesium, Colorimetric--------------------WM-1a
9. Manganese----------------------------------WM-3a and 3b
10. Nickel, Colorimetric----------------------WN-1a
11. Albuminoid Nitrogen-----------------------WN-2a
12. Nitrate Nitrogen--------------------------WN-4a
13. Diss. Oxygen------------------------------WO-1a
14. Oxalic Acid-------------------------------WO-2a
15. $R_2O_3$----------------------------------WR-1a
16. Diss. Silica------------------------------WS-1a and 1b
17. Sulphates, Turbidimetric Method-----------WS-6a
18. Sodium Dichromate-------------------------WS-7a
19. Sodium Sulphate---------------------------WS-9a
20. Sodium Sulfite----------------------------WS-10a

[redacted]

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Turn line voltage switch on. | | 1. Transformer terminals must be shielded. |
| 2. Turn instrument switch to "galvanometer on" and adjust setting to zero on scale. | | |
| 3. Turn instrument switch to "on" position. | | |
| 4. Clean and dry cuvettes, fill with distilled water and place in carrier. | 4a. Clean cuvettes with dilute ammonium hydroxide and rinse with distilled water.<br>b. Never place hot solutions in tank cell cuvettes. | 4a. Cuvettes are made of soft glass and may be easily chipped or broken.<br>b. Visually inspect the cuvettes for chips, breaks, etc. Soft glass cuvettes cannot be repaired unless they are of the test tube type; and therefore should be discarded immediately in the proper waste container. |
| 5. Place carrier so left hand cuvette is in the light path and adjust scale setting to 100% transmission. | 5. Shade the scale from room light so that a more accurate reading may be taken. | |
| 6. Move carrier to left until right hand cuvette is in the light path and read percent transmission. If this is not 100%, reclean the cuvette indicating the least percent transmission and repeat steps 4 through 6. | | 6. Optical surface of cuvette should always be wiped or polished with "Kleenex" or "Wipes", never with cheesecloth. |

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| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 7. When cuvettes balance the meter is ready to be used for analytical determinations. | | |
| 8. Calibration of the Spectrophotometer against PC-7 filter. (Note: This step is not necessary every time the spectrophotometer is used. The calibration of the instrument will be performed once per week by the chemist or supervisor, or oftener if necessary). | | 8. Calibration of the spectrophotometer involves adjusting set screw on mechanism inside the spectrophotometer case. Care should be exercised to prevent cuts on screw heads etc., during the adjusting operation. |

The above Job Break-Down covers the following analytical methods:

1. Sodium Silicate (Alk.)--------WA-3a
2. Copper, Colorimetric----------WC-3a
3. Chlorides---------------------WC-4a
4. Soap Hardness-----------------WH-1a
5. Iron--------------------------WI-1a and 1b
6. Magnesium, Colorimetric-------WM-1a
7. Manganese---------------------WM-3a and 3b
8. Nickel, Colorimetric----------WN-1a
9. Albuminoid Nitrogen-----------WN-2a and 3a
10. Phosphate---------------------WP-2a
11. Diss. Silica------------------WS-1a and 1b
12. Sulphates, Turbidimetric------WS-6a
13. Sodium Dichromate-------------WS-7a
14. Turbidity---------------------WT-1a

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| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Fill the buret with the solution required and then adjust the "zero" mark on the buret. | 1a. If suction line is used to fill buret, exercise care to prevent material from passing into line. Place a trap in the line as an added precaution.<br>b. To fill buret from a bottle, pour the reagent from the stock bottle into a clean dry beaker. Fill the buret from the beaker, using a small funnel. | 1a. Never pour liquids from above eye level. Lower top of buret below eye level before filling.<br>b. Use extreme care to prevent splashing or spilling.<br>c. Do not stand on rung of stool to raise eye to proper level. Use footstool or steps.<br>d. Buret should be firmly supported in two places. |
| 2. Titrate the sample, following the specified analytical procedure. | 2. Exercise care in manipulation to prevent loss of sample. | 2. If solution to be titrated is hot, handle according to laboratory safety rules. |
| 3. Dispose of sample and clean equipment. | | 3a. Rinse container with water before leaving it to be washed.<br>b. If stopcock ever becomes stuck, put on leather gloves and remove by gentle pulling and prying motions. Laboratory stopcock puller may also be used. |

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The foregoing Job Break-Down covers the folowing analytical methods:

1. Free Mineral Acidity--------------------------------WA-1a
2. Phenolphthalein and Methyl Orange Alkalinity------WA-2a
3. Calcium--------------------------------------------WC-1a
4. Free $CO_2$----------------------------------------WC-2a
5. Soap Hardness--------------------------------------WH-1a
6. Magnesium (Gravimetric)----------------------------WM-2a
7. Nitrate Nitrogen-----------------------------------WN-4a
8. Diss. Oxygen---------------------------------------WO-1a
9. Oxalic Acid----------------------------------------WO-2a
10. Total Solids---------------------------------------WS-4b
11. Sodium Chloride------------------------------------WS-8a
12. Sodium Sulfate-------------------------------------WS-9a
13. Sodium Sulfite-------------------------------------WS-10a

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Clamp mechanical stirrer onto ring stand. | 1. It should be clamped in two places; and, because of its weight it should be clamped at a low point on ring stand. | 1a. The mechanical stirrer is heavy and must be handled with care.<br>b. Check to see that the stirring rod is firmly attached and that the attachment point is shielded. |
| 2. Set up and fill buret. Follow the complete Job Break-Down for Titrations on page 10. | | |
| 3. Clamp on electrodes so that their tips are about ½" above the tip of the stirring rod. | 3. See "Use of L. and N. or Beckman pH Meter", page 13. | 3. Use extreme care in handling electrodes. Be sure they are well out of the path of the stirring rod. |
| 4. Clamp on beaker containing buffer solution. | 4. The electrodes, the stirring rod, and the buret tip should all be near the bottom of the beaker. No part should touch another or the beaker. | |
| 5. Turn on the stirrer and push the "on" button of the pH meter. | 5a. Start stirrer slowly.<br>b. See "Use of L. and N. or Beckman pH Meter", page 13.<br>c. See "Use of Mechanical Stirrer", Pg. 14. | |
| 6. Turn off the pH meter and the stirrer. Remove beaker containing buffer solution. | 6. Never leave the electrodes exposed to the air when the meter is reading. | 6a. Hands should be dry when using electrical equipment. |

[redacted]

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| | | b. Be careful not to knock the electrodes while unclamping and removing the beaker. |
| 7. Wash electrodes, buret tip, and stirring rod with distillled water. | | 7. Use wash bottle carefully to avoid spilling or knocking of electrodes. |
| 8. Clamp on clean beaker containing sample and run the analysis. | | 8a. See "Use of L. and N. Beckman pH Meter", page 13, and Laboratory Manual. |
| | | b. Keep face and hair away from stirring rod when reading buret. |
| 9. Turn off stirrer and pH meter. Remove beaker and replace with a clean beaker containing distilled water. | | 9. Check to see that all connections are off, and wipe up any spills. |

The above Job Break-Down covers method WA-3a.

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## RECLAIMING USED SOLVENT BY DISTILLATION

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Assemble and inspect the distillation set-up for leaks. | | 1a. All ground joints should be properly seated, well greased and clamped in place.<br>b. Condenser water should be on. |
| 2. If necessary, pre-dry the solvent using the proper desiccant. | 2. Many organic solvents form binaries with water which cannot be readily separated by distillation. In case of this type, a desiccant must be used to remove the water. | 2. Know the hazards and properties of the desiccant used. Observe all necessary precautions for the safe handling of the material. |
| 3. Add the solvent to the distilling flask and distill. | 3. Collect for re-use only that portion of the distillate which has a constant boiling temperature. | 3a. Avoid leaks and spills. Clean up spilled material immediately. If the spilled material is flammable, first disconnect all electrical equipment and extinguish all flames in the vicinity and then clean up.<br>b. Distilling flasks should not be filled more than two-thirds full.<br>c. Heat input to distillation set-up should be adjusted so the solvent condenses on the first 1-2 inches of the condenser.<br>d. Discard all waste solvent and distillation residue to proper waste disposal. (See |

[redacted]

[redacted]

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| | | Safety Rules in Employee's Handbook of Safety Rules". |
| 4. Transfer reclaimed solvent to proper container and place in storage. | | 4. For specific rules on the storage of solvents, see the "Employee's Handbook of Safety Rules". |

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| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Weigh sample into proper receptacles. | | |
| a. Solids | 1a. If sample is hygroscopic, it should be contained in a weighing bottle; otherwise the use of a balance pan is recommended. | 1a. 1) Do not allow corrosive solids to come in contact with the skin. Clean up spills immediately.<br>2) Lime dust is irritating to the eyes, nose, and throat. When working in a lime-laden atmosphere, use respirator and cover-all goggles. |
| b. Liquids and volatile solvents. | b. Use dropper type weighing bottle. | b. Exercise extreme care to prevent contact of the skin with corrosive materials. Do not breathe vapors. |
| | | 2. Wash hands and forearms thoroughly after handling chemicals. |

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| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Obtain sample. | | 1. See "Sampling", p. 11. |
| 2. Measure required amount of sample into graduated cylinder. | | 2. See "Preparation of Solutions", p. 12 & 13. |
| 3. Add methyl orange and neutralize with concentrated HCl. | 3. Add acid from buret. | 3. Add carefully to avoid spattering. |
| 4. Add oxalte solution, and heat to 80° to 90°C. | 4a. Pipet oxalate<br>b. Use of hot plate. | 4a. See "Use of Pipets", p. 9 & 10.<br>b. Be sure beaker is dry on outside before placing on hot plate to avoid spattering. Take care to keep hands and arms away from hot plate. |
| 5. Neutralize hot solution with Ammonium Hydroxide. | | 5. Follow master and laboratory safety rules. |
| 6. Cool solution. | | 6. Put solution in a place where it will not be accidently touched or upset. |
| 7. Filter solution through glass crucible using suction. | | 7. See "Suction Filters" under "Particular Laboratory Safety Rules". |
| 8. Add 5 ml. of concentrated sulphuric acid to the crucible (without suction). | 8. Use of concentrated acid. | 8. Keep hands and arms away from acid stream if using acid dispensing buret. Acid dispensing buret should be placed in protective stall with buret tip back of front edge of stall. |

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| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 9. Add 8 to 10 ml. of distilled water to the crucible containing concentrated acid and precipitate. Then filter. | 9a. Adding water to acid is especially hazardous.<br>b. Connecting vacuum to flask containing hot acid solution. | 9a. Add water very slowly, pouring it down the sides of the crucible. Wear rubber gloves, rubber apron and face mask.<br>b. Do not touch hot crucible, and take care not to upset the flask while connect-to vacuum outlet. |
| 10. Heat the solution to 80°C. and titrate with $KMnO_4$. | 10a. Handling of hot beakers and use of hot plate.<br>b. Titrating heated solution. | 10a. See "Safety Caution" #4.<br>b. See "Titrations I", page 16. |

The above Job Break-Down covers Method WC-1a (Calcium).

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## DETERMINATION OF COPPER (COLORIMETRIC)

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Obtain sample. | | 1. See "Sampling", p. 11 |
| 2. Add 1 ml. of concentrated HCl and 4 ml. of concentrated nitric acid to 100 ml. of sample in evaporating dish. | 2a. Pipet volatile acids under hood.<br>b. Add acid slowly, letting it run down the sides of the dish. | 2a. Use extreme care when handling the acids to minimize spattering and avoid spilling.<br>b. Follow "Use of Pipets", pg. 9 and 10. |
| 3. Place dish on hot plate and evaporate to dryness. | | 3. Be sure dish is dry before placing on hot plate. |
| 4. Remove dish on hot plate and allow to cool. Add 5 ml. of concentrated HCl and 1 ml. of water to residue. | 4. Add water to acid with extreme care. | 4. Wear gloves and add water slowly. |
| 5. Add ammonium hydroxide and rotate dish. | | 5. Take care to avoid spilling. |
| 6. Filter contents of dish into a 50 ml. graduated cylinder. | 6a. See "Use of Suction Filter" under "Particular Laboratory Safety Rules".<br>b. Do not use cylinder smaller than 50 ml. as it would make an unstable set-up. | 6. Use special suction filter apparatus designed for receiving filtrate in graduated cylinder. |
| 7. Wash dish with distilled water and add washings through filter. Do same with sodium diethyldithiocarbamate. | | 7. The filtering set-up is not too stable and care must be used in working with and near it. |
| 8. Separate cylinder and filter. Dilute filtrate to volume with distilled water. | | 8. Turn off vacuum before attempting to separate parts of the filtering set-up. |

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| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 9. Allow the solution to stand 30 minutes and then read on spectrophotometer. | | 9. See "Operation of Coleman Model 11 Spectrophotometer", p. 14 and 15. |

The above Job Break-Down covers method WC-3a (Copper) and method WN-1a (Nickel).

[redacted]

## pH DETERMINATIONS WITH L. AND N. OR BECKMAN METERS

| STEPS | KEY POINT | SAFETY CAUTIONS |
|---|---|---|
| 1. Install electrodes in meter. | 1a. Make certain calomel electrode contains both solid $KCl$ and $KCl$ solution.<br>b. Soak glass electrode in 1 N $HCl$ for at lease 8 hours before use. | |
| 2. Adjust machine with buffer solution. | 2a. pH meter should be in "on" position at least 2 minutes before reading is made.<br>b. To check for faulty electrodes use buffer solution at two pH ranges. (Usually 4.0 & 7.0).<br>c. Buffer solution must be within 3°C. of temperature of solution on which pH is to be determined. | |
| 3. Determine pH of unknown solution. | 3. Rinse electrodes thoroughly with solution to be tested. | 3. If constituents in water are not known, care should be taken to avoid contact with the skin. |
| 4. Shutt off meter. | 4. Allow electrodes to stand in distilled water when meter is not in use. | 4. Wash hands and forearms when work is completed. |

The above Job Break-Down covers the following Analytical Methods:

1. pH------------------WP-1a
2. Sodium Silicate------WA-3a

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## PHOTOMETRIC ANALYSIS (DIRECT)

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Clean equipment by method outlined in "Cleaning Chemical Glassware". | | 1. Follow precautions outlined in "Cleaning Chemical Glassware". |
| 2. Transfer the quantity of sample necessary to the containing vessel and add reagents in analytical procedure. | | 2. Use the proper size glass stopper for mixing cylinders to prevent spattering. Never use your thumb as a substitute for a stopper. |
| 3. Transfer sample to spectrophotometer cuvet and determine % light transmission. | 3. Make certain spectrophotometer galv. is balanced and that cuvettes balance. See "Operation of Coleman Model 11 Spectrophotometer". | 3. Cuvettes are made of soft glass and may be easily broken or chipped. |
| 4. Convert % light transmission to ppm of desired constituent using the proper curve. | | 4. Wash hands and forearms after completing analysis. |

Analyses covered by the above Job-Break-Down:

1. Alk. of Sod. Silicate--------WA-3a
2. Chlorides--------------------WC-4a
3. Hydrogen Peroxide------------WH-2a
4. Magnesium--------------------WM-1a
5. Manganese--------------------WM-3b
6. Albuminoid Nitrogen----------WN-2a and 3a
7. Phosphate--------------------WP-2a
8. Silica-----------------------WS-1a and 1b
9. Sulphates--------------------WS-6a
10. Sodium Dichromate------------WS-7a
11. Turbidity--------------------WT-1a

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Clean beakers by method outlined in "Cleaning Chemical Glassware". | | 1. Observe "Safety Cautions" outlined in "Cleaning Chemical Glassware". |
| 2. Add sample and chemicals to beaker. | 2. Add acid by allowing it to run down the side of the beaker. | 2a. Exercise extreme care when handling acids and peroxide; both are corrosive.<br>b. In adding acid as outlined under "Key Points" the hazard of spattering is minimized. |
| 3. Place beaker containing sample on hot plate and allow sample to evaporate until white fumes of $SO_3$ appear. | 3a. Tip beaker back and forth several times during the evaporation process to prevent iron ect. from plating in bottom of beaker.<br>b. Do not evaporate to dryness.<br>c. To minimize the chances of contamination, beakers must not be passed over the top of each other. | 3. Beaker bottom must be dry before beaker is placed on hot plate. |
| 4. Remove beaker from the hot plate and allow to cool. | 4. Finish the analysis as soon as possible after cooling to avoid contamination from dust particles in the air. | 4. Use beaker tongs when removing beakers from the hot plate. |
| 5. Rinse inside walls of beaker with a fine stream of distilled water. Heat to dissolve salts. | | 5. To prevent spattering:<br>a. The acid in the beaker must be cool. |

## PHOTOMETRIC ANALYSIS (WHERE SAMPLE IS CONCENTRATED)

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| | | b. Use only a fine stream of distilled water. |
| 6. Transfer sample to mixing cylinder and rinse beaker thoroughly. | 6. Mixing cylinder must be chemically clean. Use the method outlined in "Cleaning Chemical Glassware". | |
| 7. Add reagents to mixing cylinder containing sample and mix. | 7a. Rinse mixing cylinder stopper with distilled water before using. | |
| | b. Use care when inverting cylinder to mix sample; stopper may tend to pop out. | |
| 8. Determine the percent transmission of the sample using a spectrophotometer. | 8a. Use chemically clean cells cleaned by the method given in Step 4, "Operation of Coleman Model 11 Spectorphotometer. | |
| | b. See Job Break-Down sheet for spectrophotometers. | |
| | c. Optical surfaces of cuvettes should always be wiped or polished with Kleenex or Wips, never with cheesecloth. | |
| | | 9. Wash hands and forearms thoroughly when analysis is complete. |

The above Job Break-Down covers the following analytical methods:

1. Iron-----------WI-1a and 1b
2. Copper---------WC-3a
3. Manganese------WM-3a

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| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Clean the platinum ware. | 1a. Platinum ware can be conveniently cleaned by fusing potassium bisulphate in the vessel. The outside of the vessel can be cleaned with pumice.<br>b. Platinum ware should always be handled with platinum-tipped tongs. | 1. Care should be exercised to avoid splashing the molten potassium bisulphate. |
| 2. Weigh sample into a previously ignited platinum crucible. | 2. When hot objects are placed in a desiccator, the lid should be left slightly ajar for a few minutes to prevent the formation of a partial vacuum in the desiccator. Difficulty is sometimes encountered when a desiccator under a partial vacuum is to be opened. | |
| 3. Add sulfuric acid, evaporate sample to dryness and then ignite. | 3. An Infrarediator should be used in evaporating the sample to the fuming stage. Then sample should be placed on a hot plate, with a thin asbestos mat or mica sheet between the bottom of the platinum crucible and the top of the hot plate. When the sample is dry, ignite in a muffle furnace. | 3a. $SO_3$ fumes are very irritating to the nose and throat. Carry out all fuming operations in a well-ventilated hood.<br>b. Exercise care to prevent acid burns. |
| 4. Cool the crucible in a desiccator and then weigh. | 4. See Step 2, "Key Points". | |

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 5. Treat sample with sulfuric and hydrofluoric acids, evaporate to dryness and then ignite. | | 5a. Wear acid (coverall) goggles and rubber gloves when handling hydrofluoric acid.<br>b. Exercise extreme care to avoid spills. Clean up spills immediately.<br>c. Work involving the use of hydrofluoric acid should be confined to a well-ventilated hood.<br>d. Asbestos gloves provide additional protection for the hands when removing crucibles, etc. from a muffle furnace with tongs. |
| 6. Cool the crucible in a desiccator and reweigh. | 6. See Step 2, "Key Points". | |
| | | 7. Wash hands and forearms thoroughly when analysis is complete. |

The above Job-Break-Down covers method WS-3a.

[redacted]

## DETERMINATION OF ARTIFICIAL TURBIDITY

| STEPS | KEY POINTS | SAFETY CAUTIONS |
|---|---|---|
| 1. Set up Coleman spectrophotometer near valve pit header sample taps. | 1. Provide spare bulbs, spare cuvettes, kleenex, cheesecloth, a screw driver and a chart based on ppm turbidity vs. percent transmission. | |
| 2. Check spectrophotometer with a PC-7 filter and adjust if necessary. | | 2. When calibrating spectrophotometer observe precautions outlined under "Operation of Coleman Model 11 spectrophotometer". |
| 3. Take sample directly into cuvette and read % transmission on scale of photometer. | 3. Run samples as quickly as possible to avoid erroneous readings due to settling out of turbidity. | |

The above Job Break-Down covers method WT-1b.

[redacted]

WA-1a

## FREE MINERAL ACIDITY

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.

2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Titrations, pages 15 and 16.

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The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.

2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.

3. Sampling, page 10.

4. Titrations, pages 15 and 16.

[redacted]

WA-3a

## ALKALINITY OF SODIUM SILICATE

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Use of Pipets, pages 8 and 9.
4. Use of Mechanical Stirrer, page 7.
5. Weighing of Samples, page 21.
6. Preparation of Solutions, pages 11 and 12.
7. Titrations I and II, pages 15, 16, 17 and 18.

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WC-2a

## FREE CARBON DIOXIDE

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Titrations, pages 15 and 16.

[redacted]

[redacted]

WC-4a

## CHLORIDES

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Sampling, page 10.
4. Preparation of Solutions, pages 11 and 12.
5. Photometric Analysis (Direct), page 27.
6. Use of Pipets, pages 8 and 9.

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WC-5a

## FREE CHLORINE

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Sampling, page 10.

[redacted]

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WF-1a

## FLUORINE

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Use of Pipets, pages 8 and 9.
4. Preparation of Solutions, pages 11 and 12.

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## WH-1a

## SOAP HARDNESS

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules.
3. Preparation of Solutions, pages 11 and 12.
4. Titrations I, page 15 and 16.

[redacted]

WH-2a

## HYDROGEN PEROXIDE, COLORIMETRIC METHOD

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Preparation of Solutions, pages 11 and 12.
4. Use of Pipets, pages 8 and 9.
5. Photometric Analysis, page 27.

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WI-1a

# IRON

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Use of Pipets, pages 8 and 9.
4. Preparation of Solutions, pages 11 and 12.
5. Photometric Determinations (Where Sample is Concentrated), pages 28 and 29.

[redacted]

[redacted]

WI-1b

# IRON

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Sampling, page 10.
4. Photometric Determinations (Where Sample is Concentrated) - pages 28 and 29.

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WI-2a

# IODIDE

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Distillations, page 4.
4. Preparation of Solutions, pages 11 and 12.

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WM-1a

## MAGNESIUM, COLORIMETRIC METHOD

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Sampling, page 10.
4. Use of Pipets, pages 8 and 9.
5. Preparation of Solutions, pages 11 and 12.
6. Photometric Analysis, page 27.

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WM-2a

## MAGNESIUM, GRAVIMETRIC METHOD

The hazards and necessary precatuions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.

2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.

3. Weighing of Samples, page 21.

4. Titrations I, pages 15 and 16.

5. Ignitions and Oven Drying, pages 5 and 6.

[redacted]

[redacted]

WM-3a

## MANGANESE, COLORIMETRIC PERIODATE METHOD

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Sampling, page 10.
4. Use of Pipets, pages 8 and 9.
5. Preparation of Solutions, pages 11 and 12.
6. Photometric Determinations (Where Sample is Concentrated), pages 28 and 29.

[redacted]

WM-3b

## MANGANESE, COLORIMETRIC PERSULPHATE METHOD

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Sampling, page 10.
4. Use of Pipets, pages 8 and 9.
5. Preparation of Solutions, page 11 and 12.
6. Photometric Determinations (Where Sample is Concentrated), pages 28 and 29.

[redacted]

WN-1a

## NICKEL, COLORIMETRIC METHOD

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Colorimetric Copper, pages 24 and 25.

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WN-2a

## DETERMINATION OF AMMONIA-NITROGEN

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Use of Pipets, pages 8 and 9.
4. Preparation of Solutions, pages 11 and 12.
5. Distillations, page 4.
6. Photometric Analysis, page 27.

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[redacted]

WN-3a

## ALBUMINOID NITROGEN

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Use of Pipets, pages 8 and 9.
4. Distillations, page 4.
5. Photometric Analysis, page 27.

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WN-4a

## NITRATE NITROGEN

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Use of Pipets, pages 8 and 9.
4. Titrations, page 15 and 16.
5. Preparation of Solutions, pages 11 and 12.
6. Photometric Analysis (Where Sample is Concentrated), pages 28 and 29.

[redacted]

[redacted]

WO-1a

## DISSOLVED OXYGEN

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Use of Pipets, pages 8 and 9.
4. Preparation of Solutions, pages 11 and 12.
5. Titrations, pages 15 and 16.

January, 1949

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WO-2a

## OXALIC ACID

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Preparation of Solutions, pages 11 and 12.
4. Titrations, pages 15 and 16.

[redacted]

[redacted]

WP-2a

## PHOSPHATE IN BOILER WATER

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Photometric Analysis, page 27.

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## WR-1a

## $R_2O_3$

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Safety Rules. (Note carefully the section concerning halogen acids).

2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.

3. Use of Pipetes, pages 8 and 9.

4. Preparation of Solutions, pages 11 and 12.

5. Ignition and Oven Drying, pages 5 and 6.

[redacted]

[redacted]

WS-1a

## DISSOLVED $SiO_2$ IN TREATED WATER (COLORIMETRIC)

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Sampling, page 10.
4. Preparation of Solutions, pages 11 and 12.
5. Photometric Analysis, page 27.

January, 1949

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[redacted]

## WS-1b

## DISSOLVED $SiO_2$ IN UNTREATED WATER, COLORIMETRIC METHOD

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Sampling, page 10.
4. Preparation of Solutions, pages 11 and 12.
5. Photometric Analysis, page 27.

[redacted]

WS-2a

## DETERMINATION OF SUSPENDED SILICA

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Ignitions and Oven Drying, pages 5 and 6.
4. "Key Points" and "Cautions" under "Gravimetric Analysis - Silica", pages 30 and 31.

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[redacted]

WS-4a

## (1) TOTAL DISSOLVED SOLIDS
## (2) LOSS ON IGNITION OF DISSOLVED SOLIDS
## (3) NON-VOLATILE DISSOLVED SOLIDS

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Sampling, page 10.
4. Ignition and Oven Drying, pages 5 and 6.

[redacted]

## WS-4b

## TOTAL SOLIDS IN 200 AREA PROCESS WATER

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Titrations, pages 15 and 16.

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WS-5a

(1) SUSPENDED SOLIDS
(2) LOSS ON IGNITION OF SUSPENDED SOLIDS
(3) NON-VOLATILE SUSPENDED SOLIDS

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Ignition and Oven Drying, pages 5 and 6.

[redacted]

[redacted]

WS-6a

## DETERMINATION OF SULPHATES, TURBIDIMETRIC METHOD

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Use of Pipets, pages 8 and 9.
4. Preparation of Solutions, pages 11 and 12.
5. Photometric Analysis, page 27.

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[redacted]

WS-7a

## SODIUM DICHROMATE (COLORIMETRIC)

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Sampling, page 10.
4. Preparation of Solutions, pages 11 and 12.
5. Photometric Analysis, page 27.

[redacted]

[redacted]

WS-8a

## SODIUM CHLORIDE

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Titrations, page 15 and 16.

January, 1949

[redacted]

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[redacted]

WS-9a

## SODIUM SULPHATE

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Preparation of Solutions, pages 11 and 12.
4. Titrations, pages 15 and 16.

January, 1949

[redacted]

[redacted]

WS-10a

## SODIUM SULPHITE

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Preparation of Solutions, pages 11 and 12.
4. Titrations I, pages 15 and 16.

January, 1949

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UNCLASSIFIED

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WS-11a

## SODIUM, COLORIMETRIC METHOD

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Use of Mechanical Stirrer, page 7.
4. Use of Pipets, pages 8 and 9.
5. Use of a Centrifuge, page 2.
6. Photometric Analysis, page 27.

[redacted]

[redacted]

WT-1a

## TURBIDITY

The hazards and necessary precautions involved in this analysis are covered by the following:

1. General Laboratory Safety Rules.
2. Particular Safety Rules for Procedures and Methods, pages 1 and 2.
3. Sampling, page 10.
4. Photometric Analysis, page 27.

[redacted]

## H. W. LABORATORY MANUAL

## WATER METHODS SECTION

## TABLE OF CONTENTS

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WA-1a
Reissued: December, 1948
Status: Standard
By: W.J.M.

# FREE MINERAL ACIDITY
## (Range 0 - 100 ppm)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

A.P.H.A., "Standard Methods for the Examination of Water and Sewage", 8th Ed., Lancaster Press, Inc., 70, (1936).

### Principles and Limitations

The free mineral acidity of water is determined by titration with a standard alkali solution using methyl orange indicator. The procedure has been in use as standard practice for a number of years. Results can be duplicated to ± 1 ppm.

### Reactions

$$HA + NaOH \text{ --- } NaA + H_2O$$

### Chemicals and Solutions

1. Sodium Hydroxide, 0.02N (Solutions Code: SS-8a)
2. Methyl orange indicator (Solutions Code: SM-5a)

### Procedure

1. Measure a 100 ml. sample into a clean 100 ml. graduated cylinder and transfer to a clean porcelain dish.
2. Add a like amount of sample to a second porcelain dish to serve as a blank. (This amount need not be measured accurately).
3. Add 4 drops of methyl orange to the sample and to the blank. A reddish orange color will develop if ther is any free mineral acidity.
4. Titrate with the sodium hydroxide to the first visible color change when referred to the blank.

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## Calculations

$V_1 \times 10.0 = $ ppm. free mineral acidity as $CaCO_3$

where: $V_1 = $ ml. sodium hydroxide

## Remarks

It has been found that the temperature of the sample affects the pH at which methyl orange changes color. As the temperature is increased, the color change occurs at a lower pH. This effect is negligible for temperatures between 10° and 30°C. so the temperatures of the sample should be adjusted to within this range.

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H. W. Laboratory Manual
Water Methods

Code: WA-2a
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By: W.J.M.

## PHENOLPHTHALEIN AND METHYL ORANGE ALKALINITY

(Range 0 - 100 ppm)

### Safety Precautions

Observe general Laboratory Safety Rules.

### Literature References

1. A.P.H.A., "Standard Methods for the Examination of Water and Sewage", 8th Ed., Lancaster Press, Inc., 64 (1936).
2. Theroux, Eldridge, and Mallmann, "Analysis of Water and Sewage", 2nd Ed., McGraw-Hill Book Co., Inc., 8 (1936).

### Principles and Limitations

The alkalinity of water is represented by its content of carbonate, bicarbonate, hydroxide and occasionally borates, silicates and phosphates. The alkalinity is determined by titration with a standard acid solution using phenolphthalein and methyl orange as indicators.

This is a standard procedure which has been in use for a number of years. Results are reproducible to about 1 ppm.

### Chemicals and Solutions

| | | |
|---|---|---|
| 1. | Sulfuric acid, 0.040N | (Solutions Code: SS-23a) |
| 2. | Phenolphthalein indicator, 1% | (Solutions Code: SP-8a) |
| 3. | Methyl orange indicator, 0.1% | (Solutions Code: SM-5a) |

### Procedure

A. Phenolphthalein Alkalinity

1. Measure a 100 ml. sample (at room temperature) into a clean 100 ml. graduated cylinder and transfer to a flask or porcelain dish. Filter if turbid.
2. Measure a like amount of sample into a second flask or dish to serve as a blank.

[redacted]

[redacted]

3. Add 4 drops of phenolphthalein indicator to the sample only. If a pink color develops, phenolphthalein alkalinity is present.

4. Titrate with the sulfuric acid slowly with constant stirring until the coloration just disappears.

Calculations

$V_1 \times 20 = $ ppm. of phenolphthalein alkalinity as $CaCO_3$

where: $V_1 = $ ml. of the sulfuric acid

B. Methyl Orange Alkalinity

1. To the same sample used for determining the phenolphthalein alkalinity, add 4 drops of methyl orange indicator. A yellow color will develop if methyl orange alkalinity is present.

2. Add a like amount of indicator to the blank.

3. Titrate the sample with the sulfuric acid slowly with constant stirring until the first visible color change is noted when referred to the blank.

4. Use the total ml. of acid added to the sample (including that used for the phenolphthalein alkalinity) for the calculation.

Calculation

$V_1 \times 20 = $ ppm. methyl orange alkalinity, or "total alkalinity", as $CaCO_3$.

where: $V_1 = $ total volume of sulfuric acid used, ml.

Remarks

It has been found that the temperature of the sample affects the pH at which methyl orange changes color. As the temperature goes up the color change occurs at a lower pH. Samples for titration should be at a temperature between 10 and 30°C. In this region, the effect is very slight.

[redacted]

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WC-1a

Reissued: December, 1948
Status: Standard
By: W.J.M.

# CALCIUM
(Range 0-1000 ppm)

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

1. Furman, "Scott's Standard Methods of Chemical Analysis", 5th Ed., C. Van Nostrand, 211 (1939).
2. A.P.H.A., "Standard Methods for the Examination of Water and Sewage", 8th Ed., Lancaster Press, 79 (1936).

## Principle and Limitations

The calcium in the sample is precipitated as the oxalate from an acid solution. The calcium oxalate is filtered from solution, dissolved in hot acid, and the oxalate titrated with permanganate. From the amount of permanganate required the amount of calcium present can be calculated and expressed as a percentage of the original sample.

In general this method is one of the standard analytical procedures. A few modifications have been made to suit the special purpose. For control analyses it gives accurate results to about 0.1 mg. of calcium. This is approximately a 5% maximum error. In checking the procedure for the addition of known calcium to actual water samples excellent recovery was made.

## Reactions

$$Ca^{++} + (NH_4)_2C_2O_4 \text{ --- } CaC_2O_4 \text{ (ppt) } + 2NH_4^{++}$$

$$5CaC_2O_4 + 2KMnO_4 + 8H_2SO_4 \text{ --- } 5CaSO_4 + K_2SO_4 + 2MnSO_4 + 10CO_2 + 8H_2O$$

$$1 \text{ ml. } .1\underline{N} \text{ } KMnO_4 = \frac{5 \times 40.08}{2 \times 5 \times 1000} = .020 \text{ g/l. Ca} = 20 \text{ ppm Ca.}$$

## Chemicals and Solutions

1. Hydrochloric acid, C.P.
   HCl, 36%

[redacted]

2. Sulfuric acid, C.P., $H_2SO_4$, 96%

3. Methyl orange, 0.1% (Solutions Code: SM-5a)

4. Ammonium hydroxide, 6N (Solutions Code: SA-12a)

5. Ammonium hydroxide, 5:95 saturated (Solutions Code: SA-12a)

6. Ammonium oxalate, saturated (Solutions Code: SA-16b)

7. Potassium permanganate solution, 0.1N (Solutions Code: SP-16a)

Procedure

1. Measure 200 ml. of the sample to be analyzed into a 400 ml. beaker.

2. Add 2-3 drops of methyl orange and neutralize with concentrated hydrochloric acid adding 2 ml. in excess.

3. Add 10 ml. of the ammonium oxalate solution and heat to 80-90°C.

4. While the solution is hot, neutralize with 6N ammonium hydroxide, and add a small excess to insure the solution's being basic.

5. Let the solution cool and stand for one hour.

6. Filter off the calcium oxalate through a fine sintered glass crucible.

7. Wash the precipitate including the beaker and crucible with dilute ammonium hydroxide a number of times, wetting the sides of the crucible as all excess oxalate ions must be removed.

8. Rinse the filter flask and replace it under the crucible funnel, add 5 ml. of the conc. sulfuric acid to the crucible without suction pouring it down the sides, completely covering the surface.

9. Next add carefully 8 to 10 ml. of distilled water, slowly pouring it down the sides of the crucible. This hot acid solution dissolves the precipitate.

10. Apply suction and filter the solution.

11. Wash the crucible with several small portions of distilled water.

12. Heat the solution to approximately 80°C. and titrate with the potassium permanganate to a faint permanent pink (Remark 1, 2, and 3).

Calculations

$$\frac{V_1 \times N_1 \times .020 \times 10^6}{V_2} = \text{ppm Ca}$$

or for the above conditions:

$$V_1 \times N_1 \times 100 = \text{ppm Ca}$$

where: $V_1$ = ml. of potassium permanganate

$N_1$ = Normality of potassium permanganate

$V_2$ = ml. of sample

Remarks

1. After addition of the first drop of potassium permanganate it may require some time for the reaction to start.

2. It may be advantageous to use 0.01N potassium permanganate when very small amounts of calcium oxalate are obtained.

3. During the titration the temperature of the solution should not be allowed to fall below 60°C.

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WC-2a

Reissued: December, 1948
Status: Standard
By: W.J.M.

# FREE CARBON DIOXIDE
(Range 0 - 100 ppm)

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

1. A.P.H.A., "Standard Methods for the Examination of Water and Sewage", 8th Ed., Lancaster Press, Inc., 69 (1936).
2. Theroux, Eldridge, and Mallmann, "Analysis of Water and Sewage", 2nd Ed., McGraw-Hill Book Co., Inc., 9 (1936).

## Principles and Limitations

The free carbon dioxide is titrated with a standard alkali to an arbitrary standard pH datum as indicated by the color change of phenolphthalein indicator.

This is a standard method which has been in use for many years. It is difficult to reproduce results due to the carbon dioxide losses in sampling.

## Reactions

$$CO_2 + H_2O \text{ --- } H_2CO_3$$

$$H_2CO_3 + NaOH \text{ --- } NaHCO_3 + H_2O$$

## Chemicals and Solutions

| | |
|---|---|
| 1. Sodium hydroxide, $.02\underline{N}$ NaOH | (Solutions Code: SS-8a) |
| 2. Phenolphthalein indicator, 1% | (Solutions Code: SP-8a) |

## Procedure

Special care must be taken in collecting the sample. A 100 ml. sample must be collected at the sampling tap in a clean Nessler tube equipped with a rubber stopper.

1. Flush the tap and rinse the tube with water.

[redacted]

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2. Throttle the flow of water from the tap and collect the sample by allowing the water to run down the inside of the tube.

3. Stopper the tube immediately and make the titration within, five minutes after collecting the sample.

4. Make a test titration to determine the approximate total amount of alkali necessary by adding 10 drops of phenolphthalein to the sample and rapidly titrating to a faint pink end point, using the sodium hydroxide.

5. Remove the stopper for each addition of sodium hydroxide and mix the sodium hydroxide with the sample by gently inverting the stoppered tube.

6. Proceed with a second sample by very rapidly adding the approximate total amount of alkali necessary all at one time.

7. Mix the sample well and titrate to the end point.

Calculations

$$V_1 \times 10 = \text{ppm } CO_2$$

where: $V_1$ = Volume of sodium hydroxide, ml.

Remarks

By following this procedure carbon dioxide losses are minimized.

[redacted]

| | |
|---|---|
| H. W. Laboratory Manual | Code: WC-3a |
| Water Methods | Reissued: December, 1948 |
| | Status: Standard |
| | By: W.J.M. |

# COPPER (COLORIMETRIC METHOD)

(Range 0.01-0.1 ppm)

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

1. Hopkins and Williams, "Organic Reagents for Metals", 3rd Ed., 120, (1938).
2. Memo: Story, to Safranski, "Determination of Copper in Water", May, 1945.

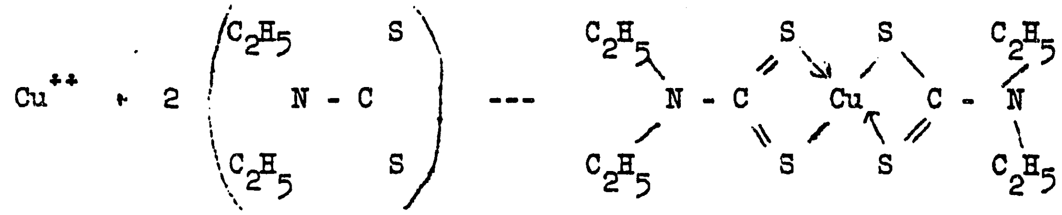
## Principles and Limitations

Sodium diethyldithiocarbamate reacts with small amounts of copper producing a golden brown color in acid, ammoniacal, or neutral solutions. The color obtained is proportional to the concentration of copper and is measured on a spectrophotometer at 450 mu.

Iron shows no interference when present in amount of 2 ppm or less. If the iron concentration exceeds this amount it may be removed by precipitation with ammonium hydroxide in the presence of ammonium salts.

This method gives good results down to about 0.005 mg. or 0.01 ppm. Since this is approximately the amount of copper in "W" water the method is not very satisfactory. Its accuracy in the range of 0.01 to 0.1 ppm is about 0.005 mg. or 0.01 ppm.

## Reactions



[redacted]

Special Apparatus

1. Coleman, model 11 spectrophotometer (Apparatus Code: AS-16b)
2. Absorption cells, 5 cm. Aminco (Apparatus Code: AC-21d)
3. Filter, sintered glass (coarse) (Apparatus Code: AF-9b)

Chemicals and Solutions

1. Hydrochloric acid, C.P.
   $HCl$, 36%
2. Hydrogen peroxide, 3% (Solutions Code: SH-26d)
3. Ammonium hydroxide, C.P.
   $NH_4OH$, 59%
4. Sodium diethyldithiocarbamate, 0.1% (Solutions Code: SS-6a)
5. Standard copper solution, 1 ml. = 100 ppm cu (Solutions Code: SC-16b)

Procedure

The Coleman Model 11 spectrophotometer is used in this determination. See Apparatus Code: AS-16a for details on the operation and maintenance of this instrument.

A. Preparation of the Calibration Curve

A calibration curve relating the per cent transmission to the concentration of copper must be prepared as a reference for unknown samples. This curve must be prepared under the same conditions as the samples to be run and is good only for the specific instrument on which it is determined. If such a curve is not available (Remark 1), prepare it as follows before analyzing the sample.

1. Deliver into separate clean, dry evaporating dishes 0, 1, 2, 3, 4, and 5 ml. portions of the standard copper solution. The dish containing no copper will serve as a blank on the reagents.
2. Proceed with the color development as described in steps 2 to 10 under part B.
3. Calculate the ppm of solution compared for each sample.

[redacted]

[redacted]

a. $$\text{ppm cu} = \frac{\text{ml. standard} \times 100}{\text{final volume of dilution}}$$

b. $$\%\ \text{transmission corrected} = \frac{\%\ \text{transmission of sample} \times 100}{\%\ \text{transmission of blank}}$$

4. On semi-log co-ordinate paper, plot ppm copper against percent transmittance corrected (the latter on the log scale).

B. Analysis of the Sample

In the analysis of a single sample, run duplicate determinations. Run at least one blank on the reagents with each sample (Remark 2).

1. To a clean porcelain evaporating dish, add 100 ml. of sample.
2. Add 1 ml. of the hydrochloric acid, and about 4 drops of of the hydrogen peroxide to the contents of the dish.
3. Transfer the dish and contents to a hot plate and evaporate the contents of the dish just to dryness.
4. Remove the dish from the hot plate and wet the residue completely by adding 0.5 ml. of hydrochloric acid; then add about 1 ml. of distilled water.
5. Add 2 ml. of ammonium hydroxide and rotate the dish to mix thoroughly.
6. Filter the contents of the dish through a coarse sintered glass suction filter directly into a 10 ml. graduated cylinder (Remark 3).
7. Wash the dish three times with 1 ml. portions of distilled water, adding the washing through the filter.
8. Measure 2 ml. of the sodium diethyldithiocarbamate and transfer to the filter, filtering into the cylinder.
9. Remove the graduated cylinder from the filter flask, dilute to 10 ml. with distilled water, mix, and allow 30 minutes for color development.
10. Fill a 5 cm. Aminco absorption cell with the solution, insert the PC-4 filter into the spectrophotometer, and measure the percent transmittance of the solution against against the blank at 450 mu.

[redacted]

11. Determine the ppm of copper present from the calibration curve.

Calculations

$$\text{ppm cu} = \frac{\text{ppm cu from curve}}{10}$$

Remarks

1. Prepare a new calibration curve whenever a new sodium diethyldithiocarbamate or copper standard solutions are introduced. A new calibration curve should be prepared at frequent intervals, the frequency to be determined by the results obtained with an old curve.

2. The blank should contain all the reagents, but the sample is omitted.

3. The final volume of solution is kept down to 10 ml. This may be conveniently done by the use of a 10 ml. graduated cylinder with the base cut down so that it will fit through the neck of a 500 ml. filter flask.

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Code: WC-4a

H. W. Laboratory Manual
Water Methods

Reissued: December, 1948
Status: Standard
By: W.J.M.

## CHLORIDES, BY SPECTROPHOTOMETER
(Range 0.05 - 2.5 ppm)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules for handling alcohol.

### Principle and Limitations

The addition of silver nitrate to a water sample containing chlorides (or other halogens or halogenoids) to which alcohol, nitric acid, and sodium sulfate has been added produces a turbid solution that transmits light with an intensity which is dependent on the amount of chloride present.

The procedure is accurate within 15% in the range of 0.05 to 2.5 ppm. Reproducibility is good and a straight line calibration curve is obtained.

### Reactions

$Ag^{+} + Cl^{-}$ --- AgCl

### Special Apparatus

1. Spectrophotometer, Coleman model 11 (Apparatus Code: AS-16a)
2. Absorption cells, 5 cm. (Apparatus Code: AC-21b)

### Chemicals and Solutions

1. Ethanol, C.P.
   $C_2H_5OH$, 95%
2. Nitric acid, 10% (Solutions Code: SN-9a)
3. Sodium sulfate, 0.2M (Solutions Code: SS-7a)
4. Silver nitrate, 0.1N (Solutions Code: SS-9a)
5. Sodium chloride, 1 ml. = gamma (Solutions Code: SS-4e)

[redacted]

## Procedure

The Coleman Universal Spectrophotometer is used in this determination. For details regarding its use and operation see Apparatus Code: AS-16a.

### A. Standardization of Instrument

A standard transmittance curve is prepared from a chloride solution of known strength and is used as a reference for unknown samples. This curve must be prepared under the same conditions as the sample to be run and is good only for the instrument on which it is determined.

1. Deliver into 100 ml. graduated cylinders portions of 1, 5, 10, 20, 30, and 40 ml. of the standard chloride solution. To another similar cylinder add 40 ml. of distilled water to be used as a blank.
2. Add 10 ml. of the ethanol, 10 ml. of the nitric acid and 3 ml. of the sodium sulfate solution to each cylinder.
3. Dilute to about 98 ml. with distilled water.
4. Mix the solutions well, being sure that they remain clear, since any color or turbidity at this point will interfere with the test.
5. Add 1 ml. of the silver nitrate to the series of standards and to the blank, then dilute both the samples and the blank to the mark with distilled water.
6. Mix by slowly inverting the cylinder once.
7. Allow the solutions to stand from 18 to 22 minutes <u>in a dark place</u>.
8. Transfer standards and blank successively to one of a pair of matched 50 mm. absorption cells and determine the per cent transmittance against distilled water at 450 mu using the PC-4 filter.
9. On semi-log co-ordinate paper plot ppm chloride against the corrected percent transmittance (the latter on the log scale).

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## Calculations

1. ppm chloride $= \frac{\text{ml. of standard} \times 5}{75}$

[redacted]

2. $$\%\ \text{transmittance corrected} = \frac{\%\ \text{transmittance of sample} \times 100}{\%\ \text{transmittance of blank}}$$

### B. Determination of Chloride in Unknown Sample

1. To each of two clean 100 ml. graduated cylinders add 75 ml. of filtered water samples, one to be used as a blank and the second for the development of turbidity.

2. Add 10 ml. of the ethanol, 10 ml. of the nitric acid, and 3 ml. of the sodium sulfate solution.

3. Mix the solutions well, being sure that they remain clear and colorless.

4. Add 1 ml. of the 0.1N silver nitrate to the turbidity sample only and dilute both samples to the mark with distilled water.

5. Mix by slowly inverting the cylinder once. Do not shake vigorously.

6. Allow the solution to stand from 18 to 22 minutes in a dark place.

7. Transfer the solutions to matched 50 mm. absorption cells and determine the percent transmittance against distilled water at 450 mu using the PC-4 filter.

## Calculations

1. $$\%\ \text{transmittance corrected} = \frac{\%\ \text{transmittance of sample} \times 100}{\%\ \text{transmittance of blank}}$$

2. From the calibration curves read the ppm of chloride for the % transmittance corrected. If other than a 75 ml. sample is used, multiply by a correction factor to convert to ppm as of a 75 ml. sample.

## Remarks

A straight line transmittance curve must be obtained. Any points that are off more than 10% should be checked.

[redacted]

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WC-5a
Reissued: December, 1948
Status: Standard
By: W.J.M.

## DETERMINATION OF FREE CHLORINE

(Range 0.0 to 1.0 ppm)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

Theroux, Eldridge and Mallmann, "Analysis of Water and Sewage", McGraw-Hill Co., 67 (1943).

### Principle and Limitations

Chlorine and certain other oxidizing agents produce a yellow compound with orthotolidine. The intensity of the color is proportional to the amount of chlorine present and this concentration can be estimated by comparison with color standards.

Hydrogen peroxide interferes with the reaction and must be absent. Dichromate also interferes, but in the range of 1-4 ppm the color developed by this reaction is insignificant at the end of a two minute period. Consequently, if the reading is made after such an interval, results reproducible to within about 0.05 ppm of chlorine can be obtained.

### Special Apparatus

Chlorimeter, Taylor (Apparatus Code: AC-7a)

### Chemicals and Solutions

Orthotolidine Solution, 0.1% (Solutions Code: ST-15a)

### Procedure

1. Fill the three cells provided with the chlorimeter to the mark with the water sample.

2. To the center cell add 0.5 cc. of the orthotolidine as shown by the mark on the dropper.

3. Mix the solution well and place in the chlorimeter.

[redacted]

4. Cover the chlorimeter and let stand exactly two minutes. (Remark 1).

5. Compare the test solution with the color standards and report in ppm of chlorine the value of the standard which most nearly matches the sample.

Remarks

The two minute standing time is critical in order to minimize the effect of any dichromate which might be present.

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WF-1a
Reissued: December, 1948
Status: Standard
By: W.J.M.

# FLUORINE
(Range 0.0-1.6 ppm)

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

Lamar, Ind. Engr. Chem., Anal. Ed. 13, 901 (1941)

## Principle and Limitations

A stable zirconium-alizarin solution is added to water containing fluorine. The red zirconium lake is bleached by the fluoride ion resulting in a yellow to greenish yellow solution. The fluoride content of water containing less than 500 ppm of sulfate and less than 1000 ppm of chloride may be determined within a limit of 0.1 ppm when a 100 ml. sample is used. Control of the pH of the sample eliminates error due to the alkalinity of the sample.

## Reactions

$$C_{14}H_5O_2(OH)_2SO_3Na \cdot H_2O + ZrONO_3 \text{ --- Red Violet Lake}$$

$$\text{Red Violet Lake} + F_2 \text{ --- } C_{14}H_5O_2(OH)_2SO_3Na \cdot H_2O$$

## Special Apparatus

Coleman model 11 Spectrophotometer (Apparatus Code: AC-16a)

## Chemicals and Solutions

1. Zirconium-alizarin indicator (Solutions Code: SZ-7a)
2. Nitric acid, 0.2N (Solutions Code: SN-9a)
3. Fluoride standard, F = 1 g/l (Solutions Code: SF-12a)

## Procedure

### A. Preparation of the Curve

1. Deliver into a 1 liter volumetric flask 10.0 ml. of the standard fluoride solution and dilute to the mark with

[redacted]

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redistilled water. Mix thoroughly.

2. Deliver 2, 4, 6, 8, and 10 ml. portions of the diluted standard fluoride solution into 100 ml. volumetric flasks, and dilute to the mark with redistilled water. These new standards represent 0.2 to 1.0 ppm of fluoride. Prepare a blank by adding 100 ml. of the redistilled water to a 100 ml. volumetric flask and run the blank along with the standards.

3. Add exactly 10 ml. of the zirconium-alizarin indicator to the standards and the blank, let stand for at least 1 hour.

4. Transfer standards and blank successively to one of a pair of matched 19 mm. cuvettes and determine the percent transmittance on the spectrophotometer against distilled water at 540 mu using the PC-4 filter.

5. On semi-log co-ordinate paper plot ppm chloride against the corrected percent transmittance (the latter on the log scale).

Calculations (Remark 1)

1. $\text{ppm fluoride} = \frac{\text{ml. of diluted standard}}{10}$

2. $\%\ \text{transmittance corrected} = \frac{\%\ \text{transmittance of blank} \times 100}{\%\ \text{transmittance of sample}}$

B. Determination of Fluoride in Unknown Sample

A 100 ml. distilled water blank is to be carried through the procedure along with the samples (Remark 2).

1. Measure out 100 ml. of each sample in a 100 ml. graduated cylinder.

2. To the samples and the blank add exactly 10.0 ml. of the zirconium-alizarin indicator, mix well, and allow to stand for at least 1 hour.

3. Transfer standards and blank successively to a pair of 19 mm. matched cuvettes and determine the percent transmittance against distilled water at 540 mu using the PC-4 filter.

4. Determine the percent transmittance corrected and read the ppm of fluoride from the curve.

Calculations

$$\% \text{ transmittance corrected} = \frac{\% \text{ transmittance of blank} \times 100}{\% \text{ transmittance of sample}}$$

Remarks

1. It is necessary to divide the percent transmittance of the blank by the percent transmittance of the sample because the fluoride ion causes the indicator color to fade. This affect is the opposite of that noticed in most spectrophotometric determinations.

2. In cases where the alkalinity or acidity of the samples exceeds about 100 ppm it is advisable to run one extra sample to which has been added a known amount of the fluoride and then determine the percent recovery of this added quantity. The percent recovery is used to correct the reading obtained from the standard curve.

Calculations

$$\text{ppm corrected} = \frac{\text{ppm read from standard curve}}{\% \text{ recovery of added fluoride}}$$

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[redacted]

Code: WH-1a

H. W. Laboratory Manual
Water Methods

Reissued: December, 1948
Status: Standard
By: W.J.M.

# SOAP HARDNESS

(Range 0-100 ppm)

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

1. A.P.H.A., "Standard Methods for the Examination of Water and Sewage", 8th Ed., Lancaster Press, Inc., 59 (1936).
2. Theroux, Eldridge and Mallmann, "Analysis of Water and Sewage",,2nd Ed., McGraw-Hill Book Co., Inc., 10 (1936).

## Principles and Limitations

The soap-consuming power of the water is proportional to the calcium and magnesium content of the water. Calcium, magnesium, aluminum and iron all contribute to the "hardness" of water.

This is a standard procedure that has been in use for a number of years. Results can be duplicated to within ± 1 ppm on the same sample. The soap hardness of a particular type of water should be compared with the hardness as determined by an actual analysis for calcium, magnesium, aluminum and iron before any confidence is placed in the soap hardness test.

## Chemicals and Solutions

1. Phenolphthalein indicator, 1.0% (Solutions Code: SP-8a)
2. Standard sodium hydroxide solution, 0.02N (Solutions Code: SS-8a)
3. Standard sulfuric acid solution, 0.04 N (Solutions Code: SS-23a)
4. Standard soap solution (1 ml. = 1 mg. $CaCO_3$ (Solutions Code: SS-1a)

## Procedure

1. Measure a 50 ml. sample into an 8 oz. glass-stoppered bottle and add several drops of phenolphthalein indicator.

[redacted]

2. Adjust to a faint pink color with standard sodium hydroxide. This color should be maintained throughout the test by additional adjustments if necessary.

3. Add 0.5 ml. of standard soap solution to the sample, stopper the bottle, and shake vigorously.

4. Continue adding soap solution in 0.5 ml. portions until a strong lather is secured which will stand for five minutes when the bottle is laid on its side after shaking as above. As the endpoint is approached, add the soap in 0.1 ml. increments (Remark 1).

5. If the quantity of soap solution used is greater than 7 ml., repeat the procedure using a smaller sample diluted to 50 ml.

Calculations (Remark 2)

$$\text{ppm total soap hardness as } CaCO_3 = \frac{(V_1 - V_2) \times 1000}{V_3}$$

where: $V_1$ = Volume of soap solution, ml.

$V_2$ = Volume of lather factor, ml.

$V_3$ = Volume of sample, ml.

Remarks

1. Oftentimes a "false" endpoint is reached on waters which contain magnesium. The false endpoint is assumed to be the dividing line between the soap's reaction with calcium and magnesium. Always add additional soap to the sample after an endpoint is secured to make sure that the endpoint was a true one. The lather will "break down" if additional soap is added to a sample which is at a false endpoint.

2. The lather factor is the amount of soap necessary to produce a permanent lather in a 50 ml. sample of distilled water and it is determined at the time the soap solution is standardized.

[redacted]

Code: WH-2a

H. W. Laboratory Manual
Water Methods

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By: W.J.M.

## HYDROGEN PEROXIDE, COLORIMETRIC METHOD
(Range 1 x $10^{-5}$ to 2 x $10^{-4}$N)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

1. J. Chem. Soc. 792 (1880)
2. Furman, "Scott's Standard Methods of Chemical Analysis", II, 5th Ed., D. Van Nostrand, 2180 (1944).
3. U. of C., "Systematic Water Analysis", 9-13-43.

### Principles and Limitations

This method depends upon the release of iodine from a buffered solution of potassium iodide by the action of hydrogen peroxide using ammonium molybdate as a catalyst. The amount of iodine released is proportional to the peroxide concentration and is determined by measuring its light absorption with a spectrophotometer.

The method gives good results and is easy and rapid for control analyses. Reproducibility is excellent on duplicate samples. Based on a large number of calibration curves the error is less than 10% in the range of 1 x $10^{-5}$ to 2 x $10^{-4}$ normal.

### Reactiions

$2KI + H_2O_2 \text{ --- } 2KOH + I_2$

### Special Aparatus

1. Spectrophotometer, Coleman model 11 (Apparatus Code: AS-16a)
2. Matched cuvettes, 19 mm. round (Apparatus Code: AC-21a)

### Chemicals and Solutions

1. Potassium iodide-buffered, 6.5% (Solutions Code: SP-15c)
2. Ammonium molybdate, 5% (Solutions Code: SA-10a)

[redacted]

3. Standard peroxide solution, 0.0005 N (Solutions Code: SH26a)

## Procedure

A Coleman spectrophotometer model 11 is used in this determination. For details as to its use and operation see Apparatus Code: AS-16a.

### A. Standardization of Instrument

A standard transmittance curve is prepared from a peroxide solution of known strength and is used as a reference for unknown samples. This curve must be prepared under the same conditions as the samples to be run and is good only for the instrument on which it is determined.

1. Deliver into 50 ml. volumetric flasks 1, 5, 10, 15, and 20 ml. portions of standard peroxide solution.
2. At this point start a blank using 15 ml. of distilled water in place of the standard peroxide (Remark 1).
3. Make standard and blank neutral or slightly acid.
4. Add 9 ml. of the potassium iodide reagent and 2 ml. of ammonium molybdate solution.
5. Dilute to the mark with distilled water, mix well and let stand for 5 or 10 minutes.
6. Transfer the standard and blank successively to one of a pair of matched 19 mm. cuvettes and determine the per cent transmittance against distilled water at 450 mu, using the PC-4 filter.
7. On semi-log paper plot normality against corrected per cent transmittance with the latter on the log scale.

## Calculations

1. $$\text{N of sample} = \frac{\text{ml. standard solution x normality of std.}}{\text{diluted volume}}$$

2. $$\text{\% transmittance corrected} = \frac{\text{\% transmittance of sample x 100}}{\text{\% transmittance of blank}}$$

[redacted]

B. Determination of Peroxide in Unknown Sample

1. Pipet into separate 50 ml. volumetric flasks 25 ml. of water to be analyzed and 25 ml. of distilled water for a blank (Remark 1).
2. Make solutions neutral or slightly acid.
3. Add 9 ml. of the potassium iodide reagent and 2 ml. of ammonium molybdate to each flask.
4. Dilute to mark, mix well, and let stand 5 to 10 minutes.
5. Transfer to matched 19 mm. cuvettes and determine the per cent transmittance against distilled water at 450 mu using the PC-4 filter.
6. From the calibration curve read directly the N for the per cent transmittance corrected.

Calculations

1. % transmittance corrected =

$$\frac{\%\ \text{transmittance of sample}}{\%\ \text{transmittance of blank}} \times 100$$

2. $\underline{N}$ of sample =

$$\frac{\text{observed normality from curve} \times \text{volume of dilution}}{\text{volume of sample}}$$

Remarks

The potassium iodide reagent may become slightly colored; therefore, a blank must be run on each sample.

[redacted]

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WI-1a

Reissued: December, 1948
Status: Standard
By: W.J.M.

# IRON
(Range 0.3 to 1.5 ppm)

## Safety Precautions

Observe general laboratory Safety Rules.

## Procedure

1. Pipet 50 ml. portions of sample into each of two 150 ml. beakers.
2. Evaporate to a volume of 10 to 15 ml.
3. Rinse the evaporated samples into 25 ml. volumetric flasks with distilled water.
4. Continue as directed in Part B, Step 2, of Standard Procedure No. ESI-2a, except calculate as follows:

### Calculations

Let A = ppm of solution compared (from curve).

B = volume of original sample in ml.

$$\text{ppm Fe} = \frac{A \times 25}{B \times \text{sp.gr. of sample}}$$

or for the above conditions

$$\text{ppm Fe} = \frac{A \times 25}{50 \times 1} = \frac{A}{2}$$

[redacted]

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[redacted]

H. W. Laboratory Manual
Water Methods

Code: WI-1b
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By: W.J.M.

# IRON COLORIMETRIC METHOD
## (Range 0 - 1 ppm)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

1. Swank & Mellon, "Determination of Iron with Mercapto. acetic acid, Ind, Eng. Chem., An. Ed., 10, 7 (1938).
2. "Orgainic Reagants for Metal," 3rd. Ed., Hopkins & Williams Ltd. 132(1938).

### Principle and Limitations

Thioglycolic (mercapto-acetic) acid is used as the reagent for iron. In ammoniacal solutions, a reddish-purple color is produced. The intensity of the color is proportional to the amount of iron present.

The method will accurately detect less the 0.1 ppm of iron. It is relatively free of interferences. Cobalt, nickel, manganese and uranium interfere but are usually absent in water in amounts that intergere, cyanide must be absent. The method is rapid and very reproducible. It is advantageous for control work because it is simple and does not require precision measurement of reagents added.

### Special Apparatus

1. Spectrophotometer, Coleman Model 11 (Apparatus Code: AS-16a)
2. Absorption cells, 5 cm. square Coleman (Apparatus Code: AC-21b)

### Chemicals and Solutions

1. Sulfuric acid, C.P.
   $H_2SO_4$, 96%
2. Hydrogen peroxide, 3% (Solution Code: SH-26a)

[redacted]

3. Thoiglycolic acid 1% - neutralized (Solutions Code: ST-8a)

4. Ammonium hydroxide, 6N (Solutions Code: SA -12a)

5. Iron Standard - 10 mg. of $Fe^{+3}$/ml. (Solutions Code: SI-18c)

## Preparation of the Calibration Curve

A calibration curve relating the per cent transmission to the concentration of iron must be prepared for each instrument used. Use as a standard: 10 mg. $Fe^{+3}$/ml.

1. Pipet into 50 ml. glass stoppered cylinders 0.5, 1.0, 1.5, 2.0 2.5, 3.0 ml. of the iron standard. These correspond to 0.02, 0.04, 0.06, 0.08, 0.10, 0.12 ppm of iron when using a 250 ml. sample.

2. Starting with the addition of thioglycolic acid, proceed with the regular procedure as written below.

3. Run a reagnet blank.

4. Plot the per cent transmission of each solution as ordinate against the amount of iron (expressed as ppm of iron)on semi-log paper. Draw a curve through the various points.

## Determination of Iron in Unknown Sample

1. Measure 250 ml. of the water sample into a clean 600 or 800 ml beaker.

2. Prepare a reagent blank with each series of iron samples starting with Step 3.

3. Add 1 ml. of the sulfuric acid and 4 ml. of hydrogen peroxide on a hot plate to dense white fumes. At this point solution must be clear. If solution is not clear, add 4 ml. of hydrogen peroxide evaporate to white fumes again.

4. When the solution is colorless, cool add 10 to 15 ml. of distilled water. Heat to boiling.

5. Pour the solution into a 50 ml. graduated flask. Wash the beaker with distilled water, keeping the sample volume under 30 ml.

6. Add 10 ml. of neutral 1% thioglycolic acid and mix. Place a small piece of litmus paper in the cylinder and add the ammonuim hydroxide until solution is slightly alkaline. Mix well and dilute to 50 ml. with distilled water.

[redacted]

7. Determine the per cent transmittancy against distilled water at 520 millimicrons, using tank cells (light path 5.0 cm.).'

Calculation

1. $\frac{\%\ \text{transmission of sample}}{\%\ \text{transmission of blank}} \times 100 = \%$ transmission corrected
2. From the standard curve, read the ppm of iron for the % transmission corrected.
3. When using a 100 ml. sample, multiply answer by 2.5.

[redacted]

H. W. Laboratory Manual
Water Methods

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By: W.J.M.

# IODIDE

(Range 0.05 - 2.0 ppb)

## Safety Precautions

1. Observe general laboratory Safety Rules.

2. Observe rules for handling strong acids.

## Literature References

1. American Public Health Association, "Standard Methods for the Examination of Water and Sewage", 8th Ed. Lancaster Press Inc., Lancaster, Pa., 39(1936).

2. Snell and Snell "Colorimetric Methods of Analysis", P. D. Van Nostrand Co., Inc., New York, (1936).

## Principle and Limitations

Iodide, present in minute amounts in water, may be converted by colorless oxidizing agents such as nitrosyl-sulfuric acid, to free iodine and this extracted with organic solvents such as chloroform or carbon tetrachloride. In such solvents, the iodine gives a pink color which is suitable for colorimetric estimation of the iodine. Chlorides of sodium, magnesium, and calcium cause errors in the extraction. Sulfates do not affect the results.

## Reactions

$$2I^- + 2SO_2(OH)ONO \text{ --- } 2H^+ + 2SO_4^{=} + 2NO + I_2$$

## Chemicals and Solutions

1. Sodium hydroxide, 0.1N (Solutions Code: SS-8a)

2. Phosphoric acid C.P.
$H_3PO_4$, 85%

3. Arsenous acid, 0.1N (Solutions Code: SA-18a)

4. Carbon tetrachloride, purified (Solutions Code: SC-13a)

5. Nitrosyl-sulfuric acid solution, E.H. Sargent Co., Chicago, (Remark 1).

[redacted]

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6. Potassium iodide, standard (10 mg. I/ml.). (Solutions Code: SI-16a)

7. Phenol red, alcoholic (Solutions Code: SP-5a)

8. Sodium carbonate, 10% (Solutions Code: SS-3a)

Procedure

1. Evaporate approximately 100 liters of the sample water to about 1 liter as follows: Siphon the sample water (from the 5 gallon bottles) into a sufficiently large (12 liter) flat-botton flask connected to a water-cooled condenser adjusted so the condensate is discarded. Fill the flask nearly full with the sample water and make the solution alkaline with sodium carbonate, using several drops of the phenyl red as indicator (Remark 2). Continue the above proceedings until the 100 liters has been reduced to 1 liter.

2. Filter on No. 42 Whatman paper, the remaining volume of sample water, contained in the distilling flask, to remove solids. Collect the filtrate in a 1500 ml. Pyrex beaker.

3. Evaporate the filtrate to approximately 50 ml. and refilter on No. 42 Whatman paper. Wash the residue on the filter paper with several ml. of hot distilled water. Collect the filtrate and washings in a suitable-sized platinum dish.

4. Evaporate the contents of the platinum dish to dryness over a steam bath or in a 110°C oven.

5. Pulverize the residue, contained in the platinum dish, in a Mullite Mortar and Pestle, and transfer the residue to a Coors. No. 6 combustion boat.

6. Insert the combustion boat into the center of a Pyrex combustion tube which has a drawn out portion one-quarter inch in diameter on one end bent down and passing through one hole of a 2-hole rubber stopper into 30 ml. of the sodium hydroxide solution in a Pyrex test tube.

7. Through the other hole in the rubber stopper, place a piece of right-angled Pyrex tubing which does not reach down to the liquid in the test tube.

8. Connect the right-angled tube to a source of vacuum.

9. Open the vacuum line and pull air through the tube at a rate of 3 to 4 bubbles per second.

10. Heat the combustion tube and boat carefully with a Meker burner until combustion is complete, avoiding fusion of the ash (Remark 2).

11. Mechanically transfer the ash from the boat into a 100 ml. beaker. Pour the sodium hydroxide solution from the test tube into the beaker. Rinse out the tube with distilled water and pour the rinsings into the beaker.

12. Boil the solution in the beaker, stirring it until the residue is dissolved or thoroughly bleached out.

13. Evaporate the solution to approximately 20 ml.

14. Filter the residue through Whatman No. 42 paper.

15. Carefully adjust the pH of this filtrate with phosphoric acid to a pH of 3 to 4, using suitable pH paper.

16. Meanwhile prepare the iodine standards as follows:

    a. Deliver 1.0 ml. of the standard potassium iodide into a 1 liter volumetric flask, dilute to the mark with distilled water , and mix thoroughly.

    b. Deliver 0.5, 1.0, 5.0, 10.0 and 15.0 ml. portions of the diluted standard potassium iodide solution into five 30 ml. separatory funnels, and dilute each to 20 ml. with distilled water. The standards represent 5, 10, 50, 100, 150, and 200 ppb of iodine in the sample.

17. To the sample and standards, prepared in step 16, add a drop of arsenous acid.

18. Add 1 ml. of the carbon tetrachloride and one drop of arsenous acid.

19. Shake each funnel for 2 minutes and allow the solutions to settle.

20. Withdraw each carbon tetrachloride layer into separate small test tubes (Remark 3).

21. Add 1 ml. of the carbon tetrachloride to the aqueous phase remaining in each funnel and extract again.

22. Repeat step 21, combining the extracts in the test tube and visually compare the color of the sample extracts with the standards.

[redacted]

Calculations:

$$\text{ppb } I_2 = \frac{A}{B}$$

Where A = ppb in standard whose color matches the color of the sample.

B = Volume of sample water evaporated, liters.

Remarks

1. Because of the safety hazards involved in the laboratory preparation of nitrosyl-sulfuric acid, only the commercial reagent is to be used.

2. The powder will turn black, then white indicating that combustion is complete. If the sodium hydroxide solution becomes very discolored, it must be evaporated and burned in the combustion boat in exactly the same manner that the sample was burned, but the sample ash does not have to be treated again.

3. A Pyrex test tube, 10 x 75 mm., is suitable.

[redacted]

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WM-1a
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By: W.J.M.

# MAGNESIUM, COLORIMETRIC METHOD

(Range 0 to 10 ppm)

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

1. Ludwig and Johnson, "Spectophotometric Determination of Magnesium by Titan Yellow," Ind. Eng. Chem., An. Ed., 14 895 (1942).
2. Hopkins and Williams, Ltd. "Organic Reagents for Metal" 3rd ed. 136 (1938).

## Principle and Limitations

Dilute magnesium solutions, when precipitated by sodium hydroxide in the presence of titan yellow, give stable suspensions in the presence of excess calcium and a protective colloid, which appear clear and colored to the eye. The transmittance of these solutions under the proper conditions is proportional to the magnesium concentration.

This method has been found very satisfactory. The calibration curve does not obey the Lambert-Beer law perfectly but it is reproducible. The procedure is easy to follow and is rapid for control work. The accuracy of the method is about 0.03 mg. in the range of 0.10 to 0.50 mg. of magnesium. Excellent results are obtained on recovery of added magnesium to actual water samples.

## Special Apparatus

1. Spectrophotometer, Coleman Model 11 (Apparatus Code: AS-16a)
2. Matched cuvettes, 19 mm. (Apparatus Code: AC-21a)

## Chemicals and Solutions

1. Sulfuric acid, 1N (Solutions Code: SS-23a)
2. Starch solution 1% (not stabilized) (Solutions Code: SS-21b)

[redacted]

[redacted]

3. Calcium sulfate, saturated solution (Solutions Code: SC-3a)
4. Titan yellow indicator 0.05% (Solutions Code: ST-9a)
5. Sodium hydroxide, 4N (Solutions Code: SS-8a)
6. Magnesium standard 1 ml. = 100 ppm (Solutions Code: SM-4a)

Procedure

A Coleman Soectrophotometer Model 11 is used in this determination For details of its use and operation see Apparatus Code: AS-16a.

A. Standardization of Instrument

A standard transmittance curve is prepared from a magnesium solution of known strength and is used as a reference for unknown samples. This curve must be prepared under the same conditions as the samples to be run and is good only on the instrument on which it is determined.

1. Transfer into 100 ml. volumetric flasks samples of 2, 4, 6, 8 and 10 ml. portions of standard magnesium solution.
2. Dilute to less than 50 ml. with water.
3. Using distilled water prepare a blank of similar volume.
4. Add to each flask the following reagents in the order given and mix after each addition.
   a. 1 ml. of the sulfuric acid (this must make the sample acid)
   b. 10 ml. of the starch solution, freshly prepared.
   c. 20 ml. of the calcium sulfate solution.
   d. 10 ml. of the titan yellow
   e. 5 ml. of the sodium hydroxide (this must bring the pH to approximately 11 or higher).
5. Dilute to the mark with distilled water, place in a mechanical shaker and shake for five minutes.
6. Transfer standards and blank successively into one of a pair of 19 mm matched cuvettes and determine the percent transmittance against distilled water at 530 mu using the PC-4 filter.
7. On semi-log paper plot ppm against % transmittance corrected with the latter on the log scale.

[redacted]

Calculations

$$\text{ppm Mg} = \frac{\text{ml. standard} \times 100}{\text{volume of dilution}}$$

$$\text{\% transmittance corrected} = \frac{\text{\% transmittance of sample} \times 100}{\text{\% transmittance of blank}}$$

B. Determination of Magnesium in Unknown Sample

1. Filter the sample through a #1 filter paper into a clean flask. Rinse the flask with filtered sample before cottecting the sample which is to be used.

2. Pipet 50 ml. of filtered sample into a 100 ml. volumetric flask.

3. At this point start a blank using 50 ml. of distilled water . Carry this blank throughout the following procedure.

4. Add the following reagents to both sample and blank in the order named, shaking after each addition:

   a. 1 ml. of the sulfuric acid (this must make the solution acid)
   b. 10 ml. of the starch solution freshly prepared
   c. 20 ml. of the calcium sulfate solution.
   d. 10 ml. of the Titan yellow.
   e. 5 ml. of the sodium hydroxide (pH must be 11 of higher).

5. Dilute to the mark with distilled water, place in a mechanical shaker and shake for five minutes.

6. Transfer into 19 mm matched cuvettes and determine the percent transmittance against distilled water at 530 mu using the PC-4 filter.

7. From the standard curve read the micrograms of magnesium per ml. for the % transmittance corrected.

Calculations

1. $$\text{\% transmittance corrected} = \frac{\text{\% transmittance of sample} \times 100}{\text{\% transmittance of blank}}$$

2. Where A = ppm Mg of solutions compared (from curve)

   B = volume of original sample

C = volume of sample prepared for comparison

$$\text{ppm of Mg} = \frac{A \times C \times 10^{-6} \times 10^{6}}{B} = \frac{A \times C}{B}$$

or for the conditions above

$$\text{ppm of Mg} = \frac{A \times 100}{50} = 2A$$

Remarks

1. The samples can not be measured against the blanks as the color fades on exposure to light.

2. If concentration of the sample is necessary take an appropriat sample, add 1ml. of the sulfuric acid and evaporate to about 50 ml. Cool and proceed as above except that a suitable correction factor must be applied to the calculation.

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WM-2a
Reissued: December, 1948
Status: SStandard
By: W.J.M.

## DETERMINATION OF MAGNESIUM, GRAVIMETRIC METHOD
(Range: 0 - 1000 ppm)

### Safety Precautions

Observe the general laboratory Safety Rules.

### Literature References

1. A.P.H.A., "Standard Method for the Examination of Water and Sewage", 8th Ed., Lancaster Press, Inc., 79 (1936).
2. Willard and Diehl, "Advanced Quantitative Analysis", D. Van Nostrand Co., 293 (1943).
3. Hillebrand and Lundell, "Applied Inorganic Analysis", John Wiley & Sons, 512 (1929).

### Principle and Limitations

An acid solution of the magnesium is treated with diammonium phosphate $(NH_4)_2HPO_4$. The solution is then made ammoniacal and the magnesium filtered off as magnesium ammonium phosphate. This salt is then ignited to the pyrophosphate and weighed as such. The amount of magnesium is calculated and expressed as a percentage of the original samples.

Many other elements interfere. Most of these can be removed by treatment with hydrogen sulfide gas in an acid solution, followed by ammonium hydroxide, ammonium sulfide, and ammonium oxalate leaving in the usual cases magnesium associated with ammonia salts and the alkalies which need not be removed. Barium however, unless present as the sulfate, must be removed by treatment with sulfuric acid.

### Reactions

$$Mg^{++} + (NH_4)_2HPO_4 \text{ --- } MgNH_4PO_4 + H^+ + NH_4^+$$

$$2MgNH_4PO_4 + \text{Heat} \text{ --- } Mg_2P_2O_7 + 2NH_3 + H_2O$$

$$1 \text{ gm. } Mg_2P_2O_3 - \frac{48.64}{222.68} = 0.2184 \text{ gm. Mg}$$

[redacted]

[redacted]

## Chemicals and Solutions

1. Hydrochloric acid, C.P.
   HCl, 36%
2. Ammonium phosphate, C.P.
   $(NH_4)_2HPO_4$, C.P.
3. Ammonium hydroxide, 6N (Solutions Code: SA-12a)
4. Ammonium hydroxide, 5:95 (Solutions Code: SA-12a)
5. Methyl red, alcoholic, 0.1% (Solutions Code: SM-6a)

## Procedure

1. After removing all interfering substances weigh or measure a portion of sample equivalent to from 0.002 to 0.2 g. of magnesium (Remark 1).
2. Transfer the sample to a new or non-etched 400 ml. beaker.
3. Dilute to about 250 ml. with distilled water and add 5 ml. of the hydrochloric acid warming if necessary to effect solution,
4. Cool the solution and to the cold solution add 1 to 2 g. of ammonium O-phosphate.
5. Add a few drops of methyl red indicator and cool the solution in an ice bath.
6. Add ammonium hydroxide dropwise with constant stirring until the solution is alkaline. Do not scratch the beaker while stirring.(Remark 2).
7. Add 15 ml. of 6N ammonium hydroxide in excess and stir for 5 to 10 minutes.
8. Leaving the stirring rod in place, cover the beaker and let stand for at least 4 hours.
9. Filter the solution through a #42 Whatman paper (Remark 3).
10. Policeman the beaker and stirring rod and transfer all of the precipitate to the paper.
11. Wash the beaker and precipitate with cold dilute (5:95) ammonium hydroxide until free of chlorides.

[redacted]

12. Transfer the precipitate into a tared crucible and dry in an oven or under an infra-red heater.

13. Ignite to constant weight a 1000°C. and weigh as magnesium pyrophosphate (Remarks 4 and 5).

Calculations

$$\% \text{ Mg} = \frac{\text{wt. of precipitate} \times 0.2184 \times 100}{\text{wt. of sample}}$$

$$\text{ppm Mg} = \% \text{ Mg} \times 10{,}000$$

Remarks

1. If the filtrate from the calcium determination is available, and contains enough magnesium, it may be used as the sample in this determination as it will be free of all interfering elements.

2. It is directed not to scratch the beaker because a clinging crystalline precipitate will form in such a mark.

3. This solution can not be filtered through a Gooch crucible since some asbestos is attacked by alkaline solutions containing soluble phosphates.

4. The ignition of the paper must be done slowly and at as low a temperature as possible until the carbon is destroyed. At high temperatures the carbon is apt to become "fireproofed" and can not be burned out.

5. If the precipitate is of the proper composition, 1000°C. is high enough for the ignition. In the case of larger precipitates (0.5 g) it is desirable to heat from 5 to 15 minutes at 1150 to 1200°C. after reaching constant weight at 1000°C. This converts any remaining magnesium phosphate to the pyrophosphate. However, the ignition to constant weight can not be carried out at this temperature since magnesium pyrophosphate loses phosphorous pentoxide under these conditions.

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WM-3a
Reissued: December, 1948
Status: Standard
By: W.J.M.

## MANGANESE, COLORIMETRIC PERIODATE METHOD

(Range: 0.02 - 200 ppm)

### Safety Precautions

1. Observe the general laboratory Safety Rules.
2. Observe rules for handling strong acids.

### Literature References

A.P.H.A., "Standard Methods for the Examination of Water and Sewage", 8th Ed., Lancaster Press, Inc., 83-84 (1936).

### Principle and Limitations

The method is based on the oxidation of the manganese to permanganate by periodate and the determination of the intensity of the permanganate color which is proportional to the concentration of manganese present.

This procedure will accurately detect a minimum of about 0.01 mg. of Mn in 100 ml. of sample. Since the manganese content of "W" water is very low, this method has been used only to show that Mn was below 0.02 ppm. If time permits the evaporation of large samples, manganese can be determined with a fair degree of accuracy.

### Reactions

$$2Mn^{++} + 5IO_4^- + 3H_2O \text{ --- } 2MnO_4^- + 5IO_3^- + 6H^+$$

### Special Apparatus

1. Spectrophotometer, Coleman Model 11 (Apparatus Code: AS-16a)
2. Absorption Cells, 50 mm, Coleman (Apparatus Code: AC-21b)

### Chemicals and Reagents

1. Sulfuric acid, C.P.
   $H_2SO_4$, 96%
2. Nitric acid, C.P.
   $HNO_3$, 70%

[redacted]

3. Sodium para periodate, C.P.
   $Na_2H_3IO_6$, C.P.

4. Sulfuric acid - sodium periodate solution (Solutions Code: SS-16a)

5. Standard Mn solution, 50 ppm (Solutions Code: SM-3a)

Procedure

A Coleman Spectrophotometer modell 11 is used in this determination. For details as to its use and operation see Apparatus Code: AS-16a.

A. Standardization of Instrument

A standard transmittance cureve is prepared from a manganese solution of known strength and is used as a reference for unknown samples. This curve must be prepared under the same conditions as the samples to be run and is good only for the instrument on which it is determined.

1. Deliver into 100 ml. beakers 1, 2, 3 and 4 ml. portions of standard manganese solution and add 1 ml. of the sulfuric acid to each.

2. At this point start a blank using 2 ml. of distilled water in place of the standard and adding 1 ml. of the sulfuric acid.

3. Evaporate to dryness.

4. Add 2 to 5 ml. of distilled water, 1 ml. of the sulfuric acid and warm to dissolve the residue.

5. Add 1 ml. of the nitric acid and evaporate to white fumes of sulfur trioxide until the solution is colorless. If the solution is not colorless repeat this step until the color disappears.

6. Add 25 ml. of the sulfuric acid - sodium periodate reagent and boil.

7. Cool, and filter the solutions through a #40 Whatman paper into clean beakers.

8. Add about 0.3 g. of sodium para periodate and 75 ml. of the sulfuric acid - sodium periodate reagent.

[redacted]

9. Heat to boiling and keep hot for 25 to 30 minutes.

10. Cool and transfer into a 100 ml. volumetric flask.

11. Dilute to mark with sulfuric acid - sodium periodate reagent.

12. Transfer standards and blank successively to one of a pair of matched 50 mm absorption cells and determine the percent transmittance against distilled water at 520 mu using the PC-4 filter.

13. On semi-log paper plot ppm manganese against corrected percent transmittance with the latter on the log scale.

Calculations

$$\text{ppm} = \frac{\text{ml. standard} \times 50}{\text{volume of dilution}}$$

$$\%\ \text{transmittance corrected} = \frac{\%\ \text{transmittance of sample}}{\%\ \text{transmittance of blank}} \times 100$$

B. Determination of Manganese in Unknown Sample

1. Transfer 500 ml. of sample into a beaker and add 1 ml. of the sulfuric acid.

2. Transfer a similar sample of distilled water to a beaker and add 1 ml. of the sulfuric acid. This is to be used as a blank.

3. Evaporate both samples to dryness and cool.

4. Add 2 to 5 ml. of distilled water, 1 ml. of the sulfuric acid and warm to dissolve the residue.

5. Add 1 ml. of the nitric acid and evaporate until the solution is colorless and white fumes of sulfur trioxide appear. If the solution is not colorless repeat this step until the color disappears.

6. Add 25 ml. of sulfuric acid - sodium periodate reagent and boil the solution.

7. Cool and filter the solutions through a #40 Whatman paper into clean beakers.

[redacted]

8. Add about 0.3 g. of sodium periodate crystals and 75 ml. of sulfuric acid - sodium periodate reagent.

9. Heat to boiling and keep hot for 25 to 30 minutes.

10. Cool and transfer into a 100 ml. volumetric flask.

11. Dilute to the mark with sulfuric acid - sodium periodate reagent.

12. Transfer to matched 50 mm absorption cells and determine the percent transmittance against distilled water at 520 mu using the PC-4 filter.

13. From the standard curve read the micrograms of Mn per ml. for the corrected percent transmittance.

Calculations

1. $\%\text{ transmittance corrected} = \frac{\%\text{ transmittance of sample}}{\%\text{ transmittance of blank}} \times 100$

2. $\text{ppm Mn} = \frac{A \times C \times 10^{-6} \times 10^{6}}{B} = \frac{A \times C}{B}$

or for the conditions above $\text{ppm Mn} = \frac{A}{5}$

where: A = ppm of solutions compared (from curve)

B = volume of original solution

C = volume of sample as prepared for comparison

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[redacted]

H. W. Laboratory Manual
Water Methods

Code: WM-3b
Reissued: December, 1948
Status: Standard
By: W.J.M.

## DETERMINATION OF MANGANESE, COLORIMETRIC PERSULFATE METHOD

(Range: 0.02 - 200 ppm)

### Safety Precautions

1. Observe the general laboratory Safety Rules.
2. Observe rules for handling strong acids.

### Literature References

A.P.H.A., "Standard Methods for the Examination of Water and Sewage", 8th Ed., 83-84 (1936).

### Principle and Limitations

This method is based on the oxidation of manganese to permanganate by persulfate, and the determination of the intensity of the permanganate color, which is proportional to the concentration of manganese present.

This procedure will accurately detect a minimum of about 0.01 mg. of Mn in a 100 ml. sample. The manganese concentration in "W" water is very low, and this method has been used only to show that this concentration was below 0.02 ppm. If time permits the evaporation of large samples, manganese can be determined with a fair degree of accuracy.

### Reactions

$$2Mn(NO_3)_2 + 5(NH_4)_2S_2O_8 + 8H_2O \text{ --- } 5(NH_4)_2SO_4 + 4HNO_3 + 5H_2SO_4 + 2HMnO_4$$

### Special Apparatus

1. Spectrophotometer, Coleman Model 11 (Apparatus Code: AS-16a)
2. Absorption cells, 50 mm, Coleman (Apparatus Code: AC-21b)

### Chemicals and Solutions

1. Sulfuric acid, C.P.
   $H_2SO_4$, 96%

[redacted]

2. Hydrogen peroxide, 3% (Solutions Code: SH-26d)

3. Ammonium persulfate, C.P.
   $(NH_4)_2S_2O_8$, C.P.

4. Silver nitrate, 0.10N (Solutions Code: SS-9a)

5. Standard manganese solution, 50 ppm (Solutions Code: SM-3a)

Procedure

A Coleman spectrophotometer model 11 is used in this determination. For details regarding its use and operation see Apparatus Code: AS-16a.

A. Standardization of Instrument

A standard transmittance curve is prepared from a manganese solution of known strength and is used as a reference for unknown samples. This curve must be prepared under the same conditions as the samples to be run and is good only on the instrument on which it is determined.

1. Deliver into 100 ml. beakers 1, 2, 3 and 4 ml. portions of standard manganese solution and add 1 ml. of the sulfuric acid.

2. At this point start a blank using 2 ml. of distilled water in place of the standard and adding 1 ml. of the sulfuric acid.

3. Evaporate to dryness and cool.

4. Add 2 to 5 ml. of distilled water and 1 ml. of the sulfuric acid to dissolve the residue.

5. Add 1 ml. of the hydrogen peroxide and evaporate until the solution is colorless and white fumes of sulfur trioxide appear. If the solution is not colorless repeat this step until the color disappears.

6. Add 1 ml. of the silver nitrate and dilute to 25 ml. with distilled water.

7. Heat to boiling, and filter into a clean beaker.

8. Wash with distilled water keeping the total volume less than 90 ml.

9. Add 0.3 g. of ammonium persulfate and heat to about 90°C.

10. Allow the color to develop.

11. Cool to room temperature and transfer into a 100 ml. volumetric flask.

12. Dilute to mark with distilled water and mix well.

13. Transfer standards and blank successively to one of a matched pair of 50 mm absorption cells and determine the percent transmittance against distilled water at 520 mu using the PC-4 filter.

14. On semi-log paper plot ppm against corrected percent transmittance with the latter on the log scale.

Calculations

1. $$\text{ppm} = \frac{\text{ml. standard} \times 50}{\text{volume of dilution}}$$

2. $$\%\text{ transmittance corrected} = \frac{\%\text{ transmittance of sample}}{\%\text{ transmittance of blank}} \times 100$$

B. Determination of Manganese in Unknown Sample

1. Measure 500 ml. of sample into a clean beaker.

2. Measure an equal quantity of distilled water to be used as a blank. Carry this blank throughout the procedure.

3. Add 1 ml. of the sulfuric acid and evaporate to dryness.

4. Cool, add 2 to 5 ml. of water and 1 ml. of the sulfuric acid to dissolve the residue.

5. Add 1 ml. of hydrogen peroxide and evaporate until the solution is colorless and white fumes of sulfur trioxide appear. If the solution is not colorless, repeat this step until the color disappears.

6. Add 1 ml. of the silver nitrate and dilute to 25 ml. with distilled water.

7. Heat to boiling, and filter into a clean beaker.

8. Wash with distilled water keeping the total volume less than 90 ml.

[redacted]

9. Add 0.3 g. of ammonium persulfate and heat to about 90°C. Do not boil.

10. Allow the color to develop.

11. Cool to room temperature and transfer into a 100 ml. volumetric flask.

12. Dilute to the mark with distilled water and mix well.

13. Transfer into 50 mm absorption cells and determine the percent transmittance against distilled water at 520 mu using the PC-4 filter.

14. Using the corrected percent transmittance read the ppm present in the sample from the curve.

Calculations

1. $\%\text{ transmittance corrected} = \frac{\%\text{ transmittance of sample}}{\%\text{ transmittance of blank}} \times 100$

2. $\text{ppm Mn} = \frac{A \times C \times 10^{-6} \times 10^{6}}{B} = \frac{A \times C}{B}$ or for the conditions

above $\text{ppm Mn} = \frac{A}{5}$

where: A = ppm of solutions compared (from curve)

B = Volume of original sample, ml.

C = Volume of sample prepared for comparison, ml.

[redacted]

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WM-4a

Reissued: December, 1948
Status: Standard
By: W.J.M.

## MOBILITY MEASUREMENT OF COLLOIDAL PARTICLES IN WATER

(Range 1.35 to 1.65 units)

### Safety Precautions

Observe general laboratory Safety Rules.

### Principles and Limitations

All colloidal particles in solution exhibit a positive or a negative charge. The kind and magnitude of the charge can be determined by placing the terminals of a dry cell battery in the solution and observing to which pole the particles move and the rate of movement.

In the mobility range of 1.35 to 1.65 the method is accurate to ± 0.15 units. Other ranges have not been investigated.

### Special Apparatus

Ultra microscope, Leitz slit and attachments. (Apparatus Code: Au-1a)
Stop watch.

### Chemicals and Reagents

Cored carbons, 6 mm x 150 mm.

### Procedure

The Leitz slit ultra microscope is used in this determination. See Appartus Code: AU-1a for details on the operation and maintnance of this instrument. Examination of water sample is to be made as soon as possible after sampling. Samples over four hours old begin to lose their charge.

### A. Standard Water

Collect 18 liters of water from the clear wells and hold at room temperature in a stoppered carboy. After 3 days there is no further "loss-of-charge", and the water may then be used from time to time to check the settings of the microscope.

[redacted]

B. Determination of Mobility in Sample

1. Collect sample in a cleaned bottle which has been rinse several times with the water sample being collected.

2. Rinse and flush the Mattson cell on the microscope with approximately 100 ml. of the sample.

3. Close the lower stopcock on the Mattson cell and then carefully fill the cell with the water sample to be tested. Avoid trapping air bubbles in the cell.

4. Close the upper stopcock when cell is filled.

5. Turn on the carbon arc lamp and adjust, if necessary, so that a concentrated light beam fills the precision slit.

6. Check the cell for air bubbles by flushing small amounts of the sample through the lower stopcock while observing the illuminated field. Continue until no further bubbles appear in the field.

7. Place the battery switch in the No. 1 postion and observe the particles flowing across the field; as seen through the eyepiece of the microscope. Using a stop watch, determine the lenght of time in seconds that is required for each of five separate particles to flow through 20 divisions on the micrometer scale. Record the time for each particle to the nearest 0.5 second.

8. Place battery switch in the No. 2 position and time 5 particles in the reverse direction (Remark 1).

9. Check the cell for air bubbles as in Step 6 above.

10. Repeat Steps 7 and 8 above.

11. Average the twenty values in seconds obtained in Steps 7, 8, and 10 above and then calculate the "average mobility" of the particles as follows:

Calculations

$$\text{Mobility}^{*} = \frac{200}{\text{Seconds (Average)} \times 7.66}$$

* A particle has a mobility of 1 unit when it moves 1 micron per second unde a potential of 1 volt with the electrodes 1 cm. apart. (Remark 2)

Remarks

1. It is necessary to time the movement in both directions to eliminate errors due to convection currents, etc., in the cell

2. With the present set up; i.e., using 92 volts and a cell with electrodes 12 cm. apart, it has been found convenient to time the particles for 200 microns (20 divisions on the mecrometer scale). A curve may be used for determining the mobility of the particles and can be prepared by plotting time in seconds against mobility using log log paper.

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Code: WN-1a

H. W. Laboratory Manual
Water Methods

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By: W.J.M.

# NICKEL (COLORIMETRIC METHOD)
(Range 0-0.1 ppm)

## Safety Precautions

1. Observe the general laboratory Safety Rules.
2. Observe rules for handling strong acids.

## Literature References

1. Murray & Ashley, "Determination of Manganese, Nickel and Phosphorous in Iron and Steel", Ind. Eng. Chem., Anal. Ed., 10, 1 (1938).
2. Hopkins and Williams, Ltd., "Organic Reagents For Metals", 3rd Ed., 41 (1938).
3. Memo: Story, to Safranski, "Determination of Nickel in Process Water", June 6, 1945.

## Principle and Limitations

In this method, nickel in alkaline solution is determined by the addition of dimethylglyoxime, with the resultant formation of a highly colored, slightly soluble compound of nickelic dimethylglyoxime. The color is measured on a spectrophotometer at 520 mu.

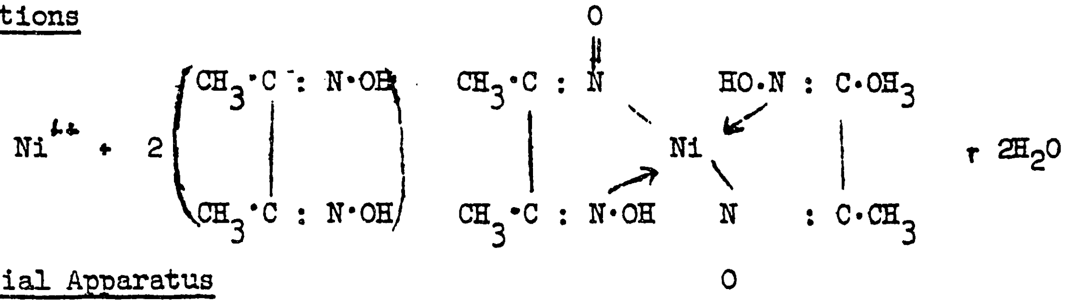
This method is nearly free of interferences. Iron interferes unless special precautions are taken. It is held in solution as the complex citrate and gives no interference providing the spectrophotometer readings are taken within one-half hour after the addition of the dimethylglyoxime, otherwise the complex citrate causes fading of the nickelic dimethylglyoxime.

This procedure gives excellent results in the range of 0.01 to 0.03 mg. of nickel. The least amount of nickel readable on the Coleman spectrophotometer is 0.5 micrograms or 0.005 ppm when a 100 ml. sample is used.

[redacted]

[redacted]

## Reactions



## Special Apparatus

1. Coleman Model 11 spectrophotometer (Apparatus Code: AS-16a)
2. Absorption cells, 5 cm. Aminco (Apparatus Code: AC-21d)
3. Filter, sintered glass (course) (apparatus Code: AF-9b)

## Chemicals and Solutions

1. Sulfuric acid, C.P.
   $H_2SO_4$, 96%
2. Sulfuric acid, 3 N (Solutions Code: SS-23a)
3. Citric acid, 10% (Solutions Code: SC-9a)
4. Bromine water, saturated (Solutions Code : SB-18a)
5. Ammonium hydroxide, C.P.
   $NH_4OH$, C. P.
6. Dimethylglyoxime, 1% (Solutions Code: SD-9a)
7. Standard nickel solution, 10 ppm (Solutions Code: SN-8a)

## Procedure

The Coleman model 11 spectrophotometer is used in this determination. See Apparatus Code: AS-16a for details on the operation and maintenance of this instrument.

### A. Preparation of the Calibration Curve

A calibration curve relating the per cent transmission to the concentration of nickel must be prepared as a reference for unknown samples. This curve must be prepared under the same conditions as the samples to be run and is good only for the specific instrument on which it is determined. If such a curve is not available (Remark 1), prepare it as follows before analyzing the sample.

[redacted]

1. Deliver into separate clean, dry evaporating dishes 0, 1, 2, 3, 4, and 5 ml. portions of the standard nickel solution. The dish containing no nickel will serve as a blank on the reagents.

2. Proceed with the color development as described in Steps 2 to 9 under B - Analysis of the Sample.

3. On semi-log co-ordinate paper, plot ppm nickel of solution against per cent transmittance corrected (the latter on the log scale.

Calculations

Calculate the ppm nickel in solution compared for each sample.

1. ppm nickel $= \frac{\text{ml. standard} \times 10}{\text{final volume of dilution}}$

2. % transmission corrected $= \frac{\text{\% transmittance of sample} \times 100}{\text{\% transmittance of blank}}$

B. Analysis of the Sample

In the analysis of a single sample, run duplicate determinations. Run at least one blank on the reagents with each sample (Remark 2).

1. To a clean 600 ml. beaker, add 500 ml. of sample.

2. Acidify the sample with 1 ml. of the 96% sulfuric acid and evaporate to dryness.

3. Take up the residue in 2 ml. of 3N sulfuric acid.

4. Add 1 ml. of the citric acid, 5 drops of saturated bromine water, and 10 drops of the ammonium hydroxide. Mix the contents of the dish by rotating the dish.

5. Filter the contents of the dish through a coarse sintered glass suction filter directly into a 10 ml. graduated cylinder (Remark 3).

6. Wash the dish three times with 1 ml. portions of distilled water, adding the washings through the filter.

7. Add 5 drops of the dimethylglyoxime solution to the cylinder.

8. Dilute the contents of the cylinder to volume and mix thoroughly.

9. Fill a 5 cm. Aminco absorption cell with the solution, insert the PC-4 filter into the spectrophotometer, and measure the per cent transmittance of the solution against the blank at 520 mu.

10. Determine the ppm of nickel present from the calibration curve.

Calculations

$$\text{ppm nickel} = \frac{\text{ppm nickel as read on curve} \times 10}{\text{original sample size (ml.)}}$$

Remarks

1. Prepare a new calibration curve whenever a new dimethylglyoxime or nickel standard solutions are introduced. A new calibration curve should be prepared at frequent intervals, the frequency to be determined by the results obtained with an old curve.

2. The blank should contain all the reagents, but the sample is omitted.

3. The final volume of solution is kept down to 10 ml. This may be conveniently done by the use of a 10 ml. graduated cylinder with the base cut down so that it will fit through the neck of a 500 ml. filter flask.

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H. W. Laboratory Manual
Water Methods

Code: WN-2a

Reissued: December, 1948
Status Standard
By: W.J.M.

## WATER

## DETERMINATION OF AMMONIA-NITROGEN

(Range: 0.02-0.12 ppm)

### Safety Precautions

Observe the general laboratory Safety Rules.

### Literature References

American Public Health Association, "Standard Methods for the Examination of Water and Sewage", 8th Ed., A.P.H.A., New York, 41-44 (1936).

### Principle and Limitations

This method depends upon a comparison of colors produced when the sample and standard ammonia solutions are treated with Nessler's reagent. Nessler's reagent reacts with the ammonia in alkaline solution to give a yellow colored compound. The ammonia is separated from interfering substances in the sample by distillation.

### Reactions

$$2(K_2H_gI_4) + ANH_3 \text{ --- } Hg_2NI + AKI + 3\ NH_4I$$

### Special Apparatus

1. Coleman spectrophotometer, Model 11 (Apparatus Code: AS-16a)
2. Absorption cells, 5 cm. sq., Coleman (Apparatus Code: AC-21b)

### Chemicals and Solutions

1. Phosphate buffer solution (pH 7.4) (Solutions Code: SB-22b)
2. Ammonium chloride solution, 10 ppm. (Solutions Code: SA-14c)
3. Nessler's reagent (Solutions Code: SN-5a)"
4. Redistilled water (Solutions Code: SW-1c)
5. Potassium sodium tartrate, 5% (Solutions Code: SP-22a)

[redacted]

## Procedure

The Coleman Model 11 Spectrophotometer is used in this determination, see Apparatus Code: AS-16a for instructions on the operation and maintenance of this instrument.

### A. Preparation of the calibration Curve

A calibration curve, relating the per cent transmission to the amount of ammonia present in the colored solution must be prepared for each instrument used. If such a curve is not available, prepare it as follows before making the determination.

1. Pipet 0.6, 1.0, 1.5, 2.0, 2.5, 3.0, and 3.6 ml. of the standard ammonium chloride solution into separate 100 ml. volumetric flasks (Remark 1).
2. Dilute each solution to the mark with ammonia-free redistilled water.
3. Add 2 ml. of Nessler's reagent to each flask, and to a flask containing only 100 ml. distilled water to serve as a blank, and shake each flask thoroughly.
4. Allow the solutions to stand for 10 minutes for complete color development.
5. Determine the per cent transmission of each soltuion against the blank on the Coleman spectrophotometer. Use the 5 cm absorption cells, a wavelength setting of 400 mu, and a PC-4 filter.
6. Plot the per cent transmission of each solution as the ordinate against the amount of ammonia nitrogen (expressed as ppm) present in each sample on semi-logarithmic graph paper.

### B. Analysis of the Sample

1. Set up a distillation apparatus, in which a 1000 ml. distillation flask is the sample container. A glass condensor may be used, and it should be so connected to the flask that it is vertical, and the concentrate may fall directly from it to the receiver.
2. Add ammonia-free distilled water to the apparatus, and boil steam through the apparatus until the condensate gives a negative test for ammonia.

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3. Empty the distilling flask and measure 500 ml. of the sample into it (Remark 2).

4. Add 10 ml. of the phosphate buffer to the contents of distillation flask.

5. Distill the contents of the flask until approximately 200 ml. of distillate have been received. The distillation rate should be not more than 10 ml. per minute and not less than 6 ml. per minute. Save the residue in the distillation flask for the albuminoid nitrogen analysis.

6. Measure the volume of the distillate in a 250 ml. graduated cylinder.

7. Pipet 100 ml. of the distillate and 1 ml. of the ammonia standard into a 100 ml. volumetric flask.

8. Proceed with the color development and measurement as described in Steps 3 to 5, Part A above (Remark 3).

9. Determine the ppm. of ammonia nitrogen present in the sample from the calibration curve.

Calculations

$$\text{ppm. ammonia nitrogen} = \frac{(A - B)\,V}{100 \times S}$$

where: A = amount of nitrogen found in the sample, ppm.

B = amount of nitrogen added to the sample = 10 ppm.

V = Volume of distillate collected, ml.

S = Volume of sample originally measured, ml.

Remarks

1. These solutions correspond, respectively to 6, 10, 15, 20, 25, 30, and 36 ppm of ammonia nitrogen.

2. If less than 500 ml. of sample is used, it should be diluted to 500 ml. with ammonia-free distilled water before proceeding.

[redacted]

3. If a turbidity develops in the solutions after the addition of the Nessler's reagent, add a few drops of 5% potassium sodium tartrate (Rochelle salts). If the solution does not clarify, discard and repeat the determination.

[redacted]

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WN-3a

Reissued: December, 1948
Status: Standard
By: W.J.M.

## WATER

## DETERMINATION OF ALBUMINOID NITROGEN
(Range: 0.02-0.12 ppm)

### Safety Precautions

Observe the general laboratory Safety Rules.

### Literature References

American Public Health Association, "Standard Methods for the Examination of Water and Sewage", 8th Ed., A.P.H.A., New York, 45, (1936).

### Principle and Limitations

This determination is a measure of the nitrogen in the form of protein or intermediate decomposition products. The ammonia nitrogen is first removed by boiling from a buffered slightly alkaline solution. After the removal of ammonia-nitrogen, alkaline potassium permanganate is added to the Kjeldahl distillation flask. This oxidizes the nitrogenous organic matter and liberates the nitrogen as ammonia. The distillation is continued and the distillate collected and analyzed for ammonia-nitrogen.

### Special Apparatus

1. Coleman spectrophotometer, Model 11 (Apparatus Code: AS-16a)
2. Absorption cells, 5 cm. sq., Coleman (Apparatus Code: AC-21b)
3. Flask, distilling, Claissen, 1000 ml.
4. Condenser, Liebig, 300 mm. (or longer)

### Chemicals and Solutions

1. Nessler's Reagent (Solutions Code: SN-5a)
2. Potassium permanganate, alkaline reagent (Solutions Code: SP-23a)
3. Potassium sodium tartarate, 5% (Solutions Code: SP-22a)

[redacted]

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[redacted]

## Procedure

The Coleman Model spectrophotometer is used in making this determination. See Apparatus Code: AS-16a for directions for the operation and maintenance of this instrument.

### A. Calibration of the Spectrophotometer

A calibration curve, relating the per cent transmission of the colored solution to the amount of ammonia nitrogen must be prepared for each instrument used in this determination. For directions for the preparation of this curve, see Water Methods Code: WN-2a, Ammonia Nitrogen. If such a calibration curve is not available, prepare it according to the directions given in the reference code.

### B. Analysis of the Sample

This determination should be made on the residue in the Kjeldahl flask remaining after the ammonia nitrogen determination. See Water Methods Code: WN-2a, Step 5.

1. To the residue remaining in the distillation flask, add 50 ml. of the alkaline potassium permanganate solution.

2. Connect the distilling flask to the condensor, and continue the distillation at a rate of not more than 10 ml. and not less than 6 ml. per minute until at least 200 ml. of the distillate has been collected.

3. Measure the volume of the distillate in a graduate cylinder, and transfer 100 ml. of it into a 100 ml. volumetric flask.

4. To another flask, add 100 ml. of distilled water. To each flask, add 2 ml. Nessler's reagent (Remark 1).

5. Allow the solutions 10 minutes for full color development, then measure the transmission of the sample against the blank in the Coleman spectrophotometer. Use a wave length of 400 mu, a PC-4 filter, and the 5 cm. absorption cells.

6. Determine the ppm of nitrogen present in the sample from the calibration curve.

[redacted]

Calculations

$$\text{ppm. albuminoid nitrogen} = \frac{A \times V}{100 \times S}$$

where: $A$ = ppm. nitrogen read from curve

$V$ = volume of distillate collected, ml.

$S$ = volume of sample, ml.

Remarks

The sample and the blank should be clear at this point. If not, add a few drops of 5% sodium potassium tartrate (Rochelle salt). If the turbidity persists, discard the samples and repeat the determination.

[redacted]

Code: WN-4a

H. W. Laboratory Manual
Water Methods

Reissued: December, 1948
Status Standard
By: W.J.M.

# WATER

## DETERMINATION OF NITRATE NITROGEN
(Range: 0.05-0.30 ppm)

### Safety Precautions

1. Observe the general laboratory Safety Rules.

2. Observe rules for handling concentrated acids.

### Literature References

American Publich Health Association. "Standard Methods for the Examination of Water and Sewage," 8th Ed., A.P.H.A., New York, 48 (1936).

### Principles and Limitations

When nitrates are treated with phenol-disulfonic acid and the resulting solution made alkaline with sodium hydroxide, a yellow compound is produced. The yellow compound formed is the sodium salt of picric acid formed by the nitration of phenol. The color produced by this method in the sample is compared with potassium nitrate standard solutions treated in a similar manner.

Chlorides greater than 130 ppm in the portion being tested, nitrites greater than 0.25, and/or carbohydrates interfere with the accuracy of this method.

### Reactions

$$(C_6H_3(OH)(SO_3H)_2 + 3HNO_3 \;{-}{-}{-}\; C_6H_2(OH)(NO_2)_3 + 2H_2SO_4 + H_2O$$

### Special Apparatus

1. Coleman spectrophotometer, Model 11 (Apparatus Code: AS-16a)

2. Absorption cells, 5 cm. sq., Coleman (Apparatus Code: AC-21b)

### Chemicals and Solutions

1. Phenol disulfonic acid (Solutions Code: SP-3a)

[redacted]

2. Nitrate standard, 10 ppm (Solutions Code: SN-10b)
3. Sodium hydroxide, 50% (Solutions Code: SS-8d)
4. Sulfuric acid, 3N (Solutions Code: SS-23a)
5. Methyl orange indicator, 0.1% (Solutions Code: SM-5a)

Procedure

The Coleman Model 11 Spectrophotometer is used in this determination. See Apparatus Code: AS-16a for details on the operation and maintenance on this instrument.

A. Preparation of the Calibration Curve

1. Pipet 0.5, 1.0, 1.5, 2.0, 2.5, and 3.0 ml. of the nitrate standard solution into separate 50 ml. volumetric flasks. To a seventh flask, add 3 ml. of distilled water to serve as a blank (Remark 1).

2. Add 3 ml. of the phenol disulfonic acid reagent to each flask, and then dilute the resulting solutions to approximately 30 ml.

3. Adjust the pH of the solutions to 3 to 6 by the addition of the strong caustic. With the aid of pH paper and a diluted caustic solution, adjust the pH of each solution to a value of approximately 8.

4. Dilute the solutions to the mark with distilled water and mix well.

5. Transfer each of the solutions to 5 cm. sq. Coleman absorption cells, and measure the transmission of each solution against the blank at 400 mu. Use a PC-4 filter in the Coleman spectrophotometer.

6. Plot the per cent transmission as ordinate against the ppm of nitrate nitrogen in the standards. Draw a smooth curve through the points obtained (Remark 2).

B. Analysis of the Sample

1. Determine the amount of 0.01 N sulfuric acid necessary to neutralize 100 ml. of the sample to the methyl orange end point.

2. Pipet 100 ml. of the sample to be analyzed into a porcelain evaporating dish, and add the amount of 0.01N sulfuric acid used in step 1 (Remark 3).

3. Add 1.0 ml. of the nitrate standard solution.

4. Evaporate the resulting solution to dryness on a steam bath.

5. Add 3 ml. of the phenol disulfonic acid solution to the residue in the evaporating dish, and rub it in well with a glass rod (Remark 3).

6. Dilute the solution with 20 ml. distilled water.

7. Add sufficient 50% sodium hydroxide to adjust the pH of the solution to 3 to 6. With the aid of pH paper and diluted caustic, adjust the pH to 8.

8. Filter the resulting solution through No. 42 Whatman paper into a 50 ml. volumetric flask.

9. Wash the dish and filter thoroughly with distilled water, adding the rinsings to the contents of the volumetric flask.

10. Dilute the solution to the mark with distilled water, and mix thoroughly.

11. Transfer the solution into the 5 cm. absorption cell, and determine the percent transmission of the sample against the blank at 400 mu, using a PC-4 filter.

12. Determine the ppm of nitrogen present in this sample from the calibration curve.

Calculations

$$\text{ppm N} = \frac{50 \times (A-B)}{100}$$

where: A = ppm. nitrate read from curve

B = ppm added in Part B, Step 3, = 0.2 ppm

Remarks

1. These solutions will contain, respectively, 0.10, 0.20, 0.30, 0.40, 0.50, and 0.60 ppm nitrogen.

2. This curve should be nearly a straight line.

3. Good contact must be obtained between the residue and the phenol disulphonic acid.

[redacted]

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[redacted]

H. W. Laboratory Manual
Water Methods

Code: WO-1a

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Status: Standard
By: W.J.M.

## DETERMINATION OF DISSOLVED OXYGEN

(Range 0.005 - 50 ppm)

### Safety Precautions

1. Observe the general laboratory Safety Rules.
2. Observe rules for handling strong acids.

### Literature References

1. A.P.H.A., "Standard Methods for the Examination of Water and Sewage", 8th Ed., Lancaster Press, 139 (1936).
2. Swartz and Gurney, A.S.T.M., 34, Part 2, 796 (1934).

### Principle and Limitations

The method is based upon the oxidation of manganous ion to the manganic state by the dissolved oxygen. The manganic ion in acid solution liberates iodine from the iodide in the solution and the iodine liberated is equivalent to the oxygen present in the sample. The iodine is determined by titration using thiosulfate with starch indicator.

Iron salts, reducing agents or strong oxidizing agents will interfere with the method.

The procedure will give accurate results to 0.01 ppm oxygen. By using extreme care in all operations, and titrating with 0.01N thiosulfate from a micro buret, it may be possible to obtain results reproducible to a 0.001 ppm. The minimum quantity detectable is about 0.005 ppm.

For routine control analyses it has been found that the Swartz-Gurney modification did not increase the accuracy enough to justify its use. Good reagents were used and the increased number of steps probably introduced as many errors as was compensated for by the modified method.

### Reactions

1. $2MnCl_2 + 4KOH + O_2 \text{ --- } 4KCl + 2MnO(OH)_2$

[redacted]

[redacted]

2. $4KI + 4H_2SO_4 + 2MnO(OH)_2 \text{ --- } 2K_2SO_4 + 2MnSO_4 + 2I_2 + 6H_2O$

3. $2I_2 + 4Na_2S_2O_3 \text{ --- } 2Na_2S_4O_6 + 4NaI$

$$1 \text{ ml. N } Na_2S_2O_3 = \frac{32}{4 \times 100} = 0.008 \text{ gm. } O_2$$

## Special Apparatus

1. Buret, semi-micro, 10 ml. (Apparatus Code: AB-21a)
2. Bottle, dissolved oxygen, 250 ml. (Apparatus Code: AB-12a)

## Chemicals and Solutions

1. Manganous chloride solution, approx. 20% (Solutions Code: SM-2a)
2. Alkaline iodide solution, approx. 10% (Solutions Code: SP-15b)
3. $H_2SO_4$, C.P., 96%
4. Sodium thiosulfate solution 0.10N or 0.01N (Solutions Code: SS-18a) or (Solutions Code: SS-18b)
5. Starch solution, 1 % with no preservative (Solutions Code: SS-21b)

## Procedure

The sample must be at a temperature of between 50 and 70°F. Collect the sample by running it into the 250 ml. glass-stoppered bottle through a glass tube which extends to the bottom of the bottle. Run enough sample through the bottle so that the total volume has been displaced several times. Regulate the flow so that no bubbles are formed and the glass tube can be removed leaving the bottle full of sample with no air bubbles trapped in the sample (Remark 1).

1. Immediately (at the point of sampling) add 2 ml. of the manganous solution followed by 2 ml. of the alkaline iodide. These solutions must be added from pipets or calibrated glass tubes which are immersed below the surface of the sample. Do not blow the solution from the pipet.
2. Stopper the bottle carefully, allowing the excess liquid to overflow, with no air bubbles left in the bottle.

[redacted]

3. Mix the solution several times by inverting the bottle and allowing the precipitate to settle at least three times.

4. Remove the stopper carefully and add 2 ml. of concentrated sulfuric acid immersing the pipet below the surface.

5. Replace the stopper without trapping any air in the bottle and mix. The precipitate will dissolve leaving a colorless or brownish solution (Remark 2).

6. Mix the solution well and transfer into a 500 ml. flask.

7. Titrate to the starch-iodide end point with thiosulfate.

   a. If the solution is dark brown, titrate with 0.1N thiosulfate to a light color, add 1 ml. of 1% starch solution and continue titrating to the end point.

   b. If the solution is light colored or colorless add 1 ml. of 1% starch solution and titrate with 0.01N thiosulfate to the end point.

   c. If no blue color develops upon the addition of starch report the oxygen content as less than 0.005 ppm.

Calculations

1. ppm dissolved oxygen = ml. of 0.1N sodium thiosulfate x 3.2

2. ppm dissolved oxygen = ml. of 0.01N sodium thiosulfate x 0.32

3. If no blue color develops upon addition of 1 ml. of starch, report as less than 0.005 ppm dissolved oxygen.

Remarks

1. If other than 250 ml. sample is used apply proper correction factor as follows:

   a. 1 ml. N sodium thiosulfate = 0.008 g. $O_2$

   b. 1 ml. N sodium thiosulfate = $\frac{0.008 \times 10^6}{\text{wt. of sample}}$ = ppm $O_2$

2. At this point the sample may be transferred into the laboratory but the titration should be completed within one hour.

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WO-2a
Reissued: December, 1948
Status: Standard
By: W.J.M.

## DETERMINATION OF OXALIC ACID

(Range 0.5 to 200 ppm)

### Safety Precautions

1. Observe general laboratory Safety Rules.

2. Potassium permanganate is an oxidizing agent and should not be allowed to come in contact with reducing material except under controlled conditions.

### Literature References

Furman, "Scott's Standard Methods of Chemical Analysis", D. Van Nostrand Co., 2253 (1939)

### Principle and Limitations

A known sample is titrated while hot with potassium permanganate to a faint pink end point. The amount of oxalic acid present is calculated and expressed as ppm of the original sample.

The reaction is slow to start by proceeds rapidly as soon as some of the permanganate is reduced since the manganous ion acts as a catalyst.

The results obtained are accurate to 0.5 ppm and should be reported as such.

### Reaction

$$5(COOH)_2 + 2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 10CO_2 + 8H_2O$$

$$1 \text{ ml. N } KMnO_4 = \frac{126.05}{2 \times 1000} = 0.06303 \text{ g. } (COOH)_2 \cdot 2H_2O$$

### Chemicals and Solutions

1. Potassium permanganate, 0.01N (Solutions Code: SP-16b)

2. Sulfuric Acid, C.P.
   $H_2SO_4$, 96%

[redacted]

[redacted]

Code: WO-2a

Procedure

1. Measure 100 ml. sample of water containing oxalic acid into a 250 ml. Erlenmeyer flask.

2. Add 5 ml. of the sulfuric acid and heat to 70 to 80°C.

3. Titrate the hot sample with the standard potassium permanganate adding the reagent in increments of 0.1 ml. and shaking between each addition.

4. Continue the titration until a faint pink end point is obtained which persists for 3 minutes.

Calculations

1. a. $\% \text{ oxalic acid} = \dfrac{\text{ml. } KMnO_4 \times N \times 0.06303}{\text{Wt. of sample}} \times 100$

   b. $\text{ppm oxalic acid} = \% \text{ oxalic acid} \times 10{,}000$

2. $\text{ppm oxalic acid} = \text{ml. } KMnO_4 \times N \times 630$

[redacted]

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WP-1a

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By: W.J.M.

## pH ON 100 AREA PROCESS WATER

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

Dole, "The Glass Electrode", John Wiley & Sons, N.Y.

### Principle and Limitations

The 100 Area process water is not well buffered and tends to loss of carbon dioxide. Temperature variations are large enough to cause serious errors unless corrected for by careful attention to proper use of the temperature compensator during analysis.

The method is designeed to standardize conditions in order to compensate as much as possible for the numberous sources of error.

On the basis of experience using this method the relative error in the determinations will be less than 0.05 pH unit.

### Special Apparatus

Flow cell (Apparatus Code: AC-5a)

### Chemicals and Solutions

1. Concentrated buffer solution (Solutions Code: SB-22a)
2. Dilute phosphate buffer solution pH 7.50 (Solutions Code: SB-22b)

### Procedure

1. Rinse the flow cell and electrodes with distilled water.
2. Flush the flow cell and electrodes once with special buffer solution which has been cooled to the temperature of the water to be tested.

[redacted]

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3. Fill the flow cell with buffer solution, regulate the temperature compensator to the temperature of the buffer solution.

4. Check the electrodes to make certain that the glass electrode is suspended in the solution just enough to completely cover the bulb, and that the calomel electrode tip is $\frac{1}{4}$ inch beneath the surface of the solution.

5. Adjust the slide wire scale to the pH of the buffer solution at that temperature.

6. Press the push button in the center of the slide wire knob down. If the needle deflects from zero, turn the "zero adjuster" with a small screw driver until the needle is zeroed. (This adjustment should be made frequently for precise work, but need be done about every 8 hours in most work).

7. Drain flow cell, then flush with distilled water for two minutes to remove all traces of buffer solution.

8. Flush cell for 2 minutes with the process water to be measured by attaching a rubber hose from the sample tap to the flow cell.

9. Disconnect rubber hose, let water stand in flow cell for $\frac{1}{2}$ minute.

10. Take the temperature of the water in the flow cell and set temperature compensator within $\frac{1}{2}$°C.

11. Take pH of sample.

12. Repeat until duplicate readings agree within 0.02 pH units.

13. After taking pH measurements, rinse the flow cell and electrodes with distilled water and discard the rinsings. Leave the cell filled with distilled water when not in use.

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WP-2a

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Status: Standard
By: W.J.M.

## PHOSPHATE IN BOILER WATER
(Range 0 - 100 ppm as $Na_3PO_4$)

### Safety Precautions

1. Observe general laboratory Safety Rules.

2. Observe rules for handling strong acids.

### Literature References

1. Snell and Snell, "Colorimetric Methods of Analysis", 2nd Edition, D. Van Nostrand Co. Chapters 49 and 50 (1943).

2. Biffen and Snell, "Commercial Methods of Analysis", Chapters on Phosphate and Water.

3. A.P.H.A. "Standard Methods of Water Analyses", A.P.H.A. 109-111 (1936).

### Principle and Limitations

This method is based upon the formation of the yellow phospho-molybdic acid. The intensity of the color is proportional to the phosphate present. By controlling the acidity and drawing up a curve with standard $PO_4$ solutions containing the approximate amount of silica found in boiler water, silica interference is practically eliminated.

The method is reproducible and if the limits of the procedure are not exceeded, spiked samples of boiler water can be checked within 2 ppm of $Na_3PO_4$. The acidity is controlled by the molybdate reagent used which is made up to 5<u>N</u> in sulfuric acid.

The color of the phospho-molybdate is read after standing 5 minutes and is stable for 20 minutes. The intensity of the color is affected by temperature, the higher the temperature the more intense the color and a correction factor must be used if the temperature of the analysis is different from the temperature at which the calibration curve was drawn. This correction is as follows:

A. Add one percent to the uncorrected transmission reading for each degree centigrade above temperature at which curve was drawn.

[redacted]

B. Substract one per cent from the uncorrected transmission reading for each degree centrigrade below temperature at which curve was drawn.

Reactions

$$24H^{+1} + PO_4^{-3} + 12\ MoO_4^{-3}\ \text{---}\ P(Mo_3O_{10})_4^{-3} + 12\ H_2O$$

Special Apparatus

| | | |
|---|---|---|
| 1. | Spectrophotometer, Coleman model II | (Apparatus Code: AS-16a) |
| 2. | Matched cuvettes, 19 mm | (Apparatus Code: AC-21a) |
| 3. | PC-4 filter for Coleman Spectrophotometer | |

Chemicals and Solutions

| | | |
|---|---|---|
| 1. | Molybdate reagent | (Solutions Code: SM-15b) |
| 2. | Standard phosphate solution - 250 ppm $Na_3PO_4$ | (Solutions Code: SP-9c) |
| 3. | Standard silica solution - approx. 60 ppm silica | (Solutions Code: SS-10d) |

Procedure

The Coleman model II Spectrophotometer is used in this determination. See Apparatus Code: AS-16a for details on the operation and maintenance of this instrument.

A. Preparation of the Calibration Curve

1. Follow the procedure for the unknown and run 10 samples each containing 25 ml. of a 60 ppm silica solution, plus 1 ml. standard sodium phosphate in the first, 2 ml. in the second, 3 ml. in the third and so on up to 10 ml. added. These correspond to 10 ppm, 20 ppm, 30 ppm, 40 ppm, 50 ppm, 60 ppm, 70 ppm, 80 ppm, 90 ppm and 100 ppm in a 25 ml. sample expressed as sodium phosphate. Run a distilled water, plus molybdate reagent, blank.

2. Draw a curve from this data basing it on a 25 ml. sample with a final dilution to 50 ml.

3. Make a notation on the curve of the temperature of the analyses at the time the readings were taken.

B. Determination of $Na_3PO_4$ in Unknown Sample

1. Measure accurately 25 ml. of clear, filtered sample.

2. To another flask, add 25 ml. distilled water for use as a blank.

3. Add 10 ml. molybdate reagent to the samples and blank, dilute to 50 ml., shake, let stand 5 minutes, read the temperature. Determine the percent transmittance at 400 mu using 19 mm cuvettes and a PC-4 filter.

Calculations

1. Correct for temperature difference by adding one percent to the transmittance for each degree above temperature shown on calibration curve and deducting one percent for each degree below.

2. % transmittance corrected =

$$\frac{\text{\% transmittance of sample (corrected for temp)} \times 100}{\text{\% transmittance of blank}}$$

3. Read directly from curve the ppm of sodium phosphate. If greater than 100 ppm, take a smaller sample.

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H. W. Laboratory Manual
Water Methods

Code: WR-1a

Reissed: December, 1948
Status: Standard
By: W.J.M.

## DETERMINATION OF $R_2O_3$
## (Range 0 - 500 ppm)

### Safety Precautions

1. Observe general laboratory Safety Rules.

2. If the modification of this method designed to give a value not including the silica is used, follow carefully all the safety rules for handling hydrofloiric acid.

### Literature References

Essential Materials, Code: ESR-2a

### Principle and Limitations of the Method

A known amount of sample is conentrated, treated with an excess of ammonium hydroxide, and the precipitate is filtered off. This precipitate is then ignited to the oxides and the residue is known as the $R_2O_3$. The weight is expressed as a percentage of the original samole.

The composition of the residue may vary widely and may contain the following elements: Al, Fe, Cr, B, Cb, Be, In, Ga, Ce, Zr, Ta, Ti, Tl, Si, V and all the rare earths. For this reason the results sometime lead to erroneous conclusions. A spectrographic analysis is the best way of determining the constituents, but, if the residue is large enough, iron and silica may be determined by standard procedures.

Phosphate, arsenate, vanadate, and silicate interfere by forming insolubles with the $R_2O_3$ elements. Organics, such as citric acid and sugar, interfere by forming soluble complexes of iron and aluminum, but the alkali or alkaline earths do not interfere providing sufficient ammonium salt is present and phosphate and carbonate are absent.

A modification has been found useful when the silica content of the water varies widely. In this procedure the ignited residue is treated with sulfuric and hydrofluoric acid and then reignited to give a value for $R_2O_3$ less silica.

[redacted]

The procedure as used is not very accurate, but better reproducibility is obtained using the above modification.

## Chemicals and Solutions

1. Hydrofluoric acid, C.P.
   HF, 48%
2. Sulfuric acid, C.P.
   $H_2SO_4$, 96%
3. Ammonium chloride, C.P.
   $NH_4Cl$. C.P.
4. Hydrochloric acid, 10% (Solution Code: SH-25a)
5. Ammonium hydroxide, 6N (Solution Code: Sa-12a)
6. Ammonium nitrate, 2% (Solution Code: SA-15a)

## Procedure

### A. Determination of Total $R_2O_3$

1. Evaporate 5 liters of the water sample (to which has been added 10 ml. of 10% hydrochloric acid) to about 100 ml. (Remark 1)
2. Wash the concentrated sample from the evaporating dish into a 400 ml. beaker using a small quantity of 10% hydrochloric acid.
3. Add 2 to 3 grams of ammonium chloride.
4. Add 6N ammonium hydroxide until the solution is alkaline.
5. Heat to boiling and let boil for a few minutes until the excess ammonia escapes.
6. Add 1 ml. of 6N ammonium hydroxide and let cool for 15 to 30 minutes.
7. Filter off the precipitate through a #40 Whatman paper.
8. Wash the precipitate with 2% ammonium nitrate until free of chlorides.
9. Transfer the paper and precipitate to a tared, ignited, platinum crucible and dry in an oven or under an infre red heater.

[redacted]

10. Ignite at 900 to 1000°C to constant weight and record the increase in weight as $R_2O_3$.

Calculation

$$\text{ppm } R_2O_3 \text{ (including silica)} = \frac{\text{weight of residue in milligrams}}{\text{liter of sample}}$$

B. Determination of $R_2O_3$ less Silica

1. To the residue obtained in Step 10 above , add 1 to 2 ml. of distilled and 2 to 3 drops of sulfuric acid.
2. Add cautiously 10 to 15 ml. of hydrofluoric acid and evaporate to a small volume under the hood.
3. Add another 5 ml. portion of hydrofluorice acid evaporate to dryness.
4. Heat carefully over a burner until all the sulfuric acid has been decomposed.
5. Ignite in a muffle at 900 to 1000°C to constant weight and record this weight as the $R_2O_3$ less silica. (Remark 2).

Calculation

$$\text{ppm } R_2O_3 \text{ less silica} = \frac{\text{Weight residue less silica in mg.}}{\text{liter of sample}}$$

Remarks

1. The sample size depends upon the amount of $R_2O_3$ present and must be varied to meet specific conditions.
2. This weight will closely represent the oxides of irom and aluminum present. The iron can be determined and the aluminum estimated by difference.

[redacted]

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WS-1a

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By: W.J.M.

# DETERMINATION OF DISSOLVED SILICA, COLORIMETRIC METHOD
(Range 0.4 - 200 ppm)

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

Schwartz, "Ind. Eng. Chem.", Anal. Ed., 14. 893 (1942)

## Principle and Limitations

This method is based upon the formation of a colored silicomolybdic acid complex. The intensity of the color is proportional to the dissolved silica present when interfering substances are absent or eliminated. Oxalic acid is added to eliminate phosphate interference.

This method is reproducible and is very rapid for control work. Errors due to silica in the reagents are most prevalent. All reagents should be kept in Pyrex bottles and a blank must be run with every determination. A freshly prepared silica solution must be used for standardizing or checking the standardization of the instrument. Under these conditions results accurate to 0.4 ppm silica can be obtained.

## Special Apparatus

| | | |
|---|---|---|
| 1. | Spectrophotometer, Coleman model II | (Apparatus Code: AS-16a)- |
| 2. | Matched curvettes, 19 mm | (Apparatus Code: AC-21a) |

## Chemicals and Solutions

| | | |
|---|---|---|
| 1. | Ammonium molybdate solution, 5% | (Solutions Code: SA-10a) |
| 2. | Hydrochloric acid, 20% | (Solutions Code: SH-25a) |
| 3. | Oxalic acid, 10% | (Solutions Code: SO-24b) |
| 4. | Standard silica solution, 100 ppm | (Solutions Code: SS-10a) |

[redacted]

Procedure

The Coleman Spectrophotometer model II is used in this determination. For details regarding its use and operation see Apparatus Code: AS-16a.

A. Standardization of Instrument

1. Transfer to a 100 ml. volumetric flask containing about 50 ml. of water 1, 3, 5, and 7.5 ml. portions of standard silica solution. Use a fifth flask containing distilled water only as a blank.

2. Add 5 ml. of the hydrochloric acid solution to the samples only and 8 ml. of the ammonium molybdate solution to both series of flasks.

3. Shake and let stand for ten minutes.

4. If phosphates are present add 3 ml. of oxalic acid to all flasks and mix again.

5. Dilute to mark with distilled water and mix well.

6. Transfer the solutions into 19 mm matched cuvettes and determine the percent transmittance against distilled water at 400 mu using the PC-4 filter.

7. On semi-log co-ordinate paper plot ppm against % transmittance corrected (the latter on the log scale).

Calculations

1. $$\text{ppm silica} = \frac{\text{ml. standard} \times 100}{\text{Volume of dilution}}$$

2. $$\text{\% transmittance corrected} = \frac{\text{\% transmittance of sample} \times 100}{\text{\% transmittance of blank}}$$

B. Determination of Silica in Unknown Sample

1. Transfer a 25 ml. sample of filtered water into a 100 ml. volumetric flask.

2. To a second flask add 75 ml. of distilled water for a blank.

3. Add 5 ml. of the hydrochloric acid and 8 ml. of the ammonium molybdate.

4. Shake and let stand for 10 minutes.

5. If phosphates are present, add 3 ml. of the oxalic acid and mix.

6. Dilute to the mark with distilled water and mix again.

7. Transfer into 19 mm matched cuvettes and determine the percent transmittance at 400 mu using the PC-4 filter.

Calculations

1. % transmittance corrected =

$$\frac{\%\ \text{transmittance of sample} \times 100}{\%\ \text{transmittance of blank}}$$

2. Using the corrected % transmittance determine the ppm silica present in the diluted sample.

3. ppm silica $= \frac{A \times C \times 10^{-6}}{B} \times 10^{6} = \frac{A \times C}{B}$

or for a 25 ml. sample diluted to 100 ml:

ppm silica $= \frac{A \times 100}{25} - 1.33\ A$

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H. W. Laboratory Manual
Water Methods

Code: WS-1b
Reissued: February, 1949
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By: W.J.M.

## DISSOLVED SILICA IN UNTREATED WATER, COLORIMETRIC METHOD
(Range 0-20 ppm)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

1. Schwartz, "Ind. Eng. Chem." Anal. Ed., 14, 893 (1942).

2. Galbraith's report on Silica.

### Principle and Limitations

This method is based upon the determination of silica in untreated water by means of gravimetric and colorimetric analysis with the subsequent comparison of results by plotting on semi-log coordinate paper. The silica is determined gravimetrically by Essential Materials Code: ES-6b, and colorimetrically by the formation of a colored silicomolybdic acid complex.

In the colorimetric method, the intensity of the color is proportional to the dissolved silica present when interfering substances are absent or eliminated and conditions are closely standardized. Oxalic acid is added when phosphate is present in order to eliminate interference of phospho-molybdic acid.

The method is reproducible providing the sample to be analyzed and the standard used in preparing the calibration curve have been treated exactly alike. Directions for preparing standard silica solutions must be followed with great care in order to avoid large errors.

It has been found that the reaction between silica and molybdate is for practical purposes of an emperical nature. For this reason this method must be fitted to each analysis by rigidly treating the standard silica solution exactly like the silica used under plant conditions.

Providing the limitations are thoroughly understood, and the proper precautions taken it is believed that results approaching an overall accuracy of 1.0 ppm can be obtained under ordinary control conditions.

[redacted]

Special Apparatus

1. Spectrophotometer, Coleman modell 11(Apparatus Code: AS-16a)
2. Matched cuvettes, 19 mm. (Apparatus Code: AC-21a)
3. PC-4 filter for Coleman spectrophotometer

Chemicals and Solutions

| | |
|---|---|
| 1. Ammonium molybdate, 5% | (Solutions Code: SA-10a) |
| 2. Hydrochloric acid, 10% | (Solutions Code: SH-25a) |
| 3. Oxalic acid, 10% | (Solutions Code: SO-24b) |

Procedure

The Coleman model 11 spectrophotometer is used in this determination. See Apparatus Code: AS-16a for details on the operation and maintenance of this instrument.

A. Preparation of the Calibration Curve

1. Gravimetric Method

   a. Add 25 ml. of the hydrochloric acid to a liter platinum dish. Add untreated water and heat the platinum dish and its contents on an infra-radiator. Continue the addition and evaporation of untreated water until a total of 15 liters have been evaporated. Run in duplicate.

   b. Analyze the contents of the dish gravimetrically by following Essential Materials Code: ES-6b, starting with Step 4.

2. Colorimetric Method

   a. Transfer to 100 ml. volumetric flasks, 5, 10, 15, 25, 40, and 50 ml. of the untreated water. Dilute the flasks to a total volume of 50 ml. with distilled water. Add 50 ml. of distilled water to a seventh flask for use as a blank.

   b. Add 8 ml. of the ammonium molybdate solution and 10 ml. of the hydrochloric acid to each flask, stopper and shake vigorously.

   c. Let stand for ten minutes.

[redacted]

d. Dilute to the mark with distilled water and mix thoroughly.

e. Transfer the solutions to 19 mm. matched cuvettes and determine the percent transmittance against the blank at 400 mu using the PC-4 filter.

f. On semi-log co-ordinate paper plot ppm silica against % transmittance corrected.

Calculations

XY (0.02) = ppm silica in each aliquot of untreated water

where: X = cc. of untreated water used
Y = ppm silica in untreated water as found by gravimetric analysis

$$\% \text{ transmittance corrected} = \frac{\% \text{ transmittance of sample} \times 100}{\% \text{ transmittance of blank}}$$

B. Determination of Silica in Unknown Sample

1. Transfer 50 ml. sample of untreated water to a 100 ml. volumetric flask.
2. To a second flask add 50 ml. of distilled water for use as a blank.
3. Add 8 ml. of the ammonium molybdate and 10 ml. of the hydrochloric acid.
4. Shake vigorously and let stand for ten minutes.
5. If phosphates are present, add 3 ml. of the oxalic acid and mix.
6. Dilute to the mark with distilled water and mix again.
7. Transfer to 19 mm. matched cuvettes and determine the percent transmittance at 400 mu using the PC-4 filter.

Calculations

1. $\% \text{ transmittance corrected} = \dfrac{\% \text{ transmittance of sample} \times 100}{\% \text{ transmittance of blank}}$
2. Read directly the ppm silica from the curve.
3. If other than 50 ml. sample is taken:
   $\text{ppm } SiO_2 \text{ in sample} = \dfrac{\text{ppm } SiO_2 \text{ from curve} \times 50}{\text{ml. sample}}$

[redacted]

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WS-2a

Reissued: December, 1948
Status: Standard
By: W.J.M.

# DETERMINATION OF SUSPENDED SILICA
(Range 0 - 1000 ppm)

## Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe all rules for handling hydrofluoric acid.

## Principle and Limitations of Method

The suspended solids are filtered from the sample with filter paper. Silica is determined by the weight loss of the ignited suspended solids residue upon treatment with hydrofluoric acid to volatilize the silica.

The method is a standard silica procedure. In the range of 0.10 ppm $SiO_2$, results can be duplicated to within 5%.

## Reactions

$$SiO_2 + 4HF \text{ - - - } SiF_4 + 2H_2O$$

## Chemicals and Solutions

1. Hydrofluoric acid, C.P.
   HF, 48%
2. Sulfuric acid, C.P.
   $H_2SO_4$, 96%

## Procedure

1. Measure a sample which will contain at least 2 mg. of suspended silica and filter through a No. 42 filter paper.
2. Place the filter paper in a platinum crucible and ignite in a muffle furnace at 1000 to 1100° C.
3. Remove the crucible from the muffle furnace, cool, add 3 to 5 drops of the sulfuric acid and sufficient distilled water to cover the solid residue.

[redacted]

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4. Place the crucible in a fused silica triangle on a hot plate, and evaporate all of the water and acid.

5. Ignite in the muffle furnace and then place in the hottest flame of a gas burner for 15 minutes.

6. Cool in a desiccator and weigh accurately.

7. Add 3 drops of the sulfuric acid and then cautiously add enough hydrofluoric acid to cover all of the solid residue.

8. Evaporate to dryness on the hot plate.

9. Ignite in the muffle furnace and then place in the hottest flame of a gas burner for 15 minutes.

10. Cool in a desiccator and weigh accurately.

Calculations

$$\text{ppm Suspended } SiO_2 = \frac{\text{Loss in wt. of crucible during HF treatment} \times 10^6}{\text{ml. of sample}}$$

[redacted]

| | |
|---|---|
| Code: | WS-3a |

H. W. Laboratory Manual
Water Methods

Reissued: December, 1948
Status: Standard
By: W.J.M.

## DETERMINATION OF SILICA IN RESIDUES, GRAVIMETRIC METHOD

Range (0 - 1000 ppm)

### Safety Precautions

1. Observe general laboratory Safety Rules.

2. Observe all rules for handling hydrofluoric acid.

### Literature References

1. Kolthoff J.M., and Sandell E.B., "Textbook of Quanitative Inorganic Analysis" 381 (1937)

2. Grasselli Chemicals Experimental Laboratory Methods No. 66.05, June 7, 1940.

3. Willard H.H., and Diehl H., "Advanced Quantitative Analyses", D. Van Nostrand Co. 180-184 (1943).

### Principle and Limitations of Method

The silica is dehydrated in the presence of hydrochloric acid ignited and weighed. It is then treated with hydroflouric acid and ignited again causing the volatization of silicon tetrafluoride. The ignited residue is weighed and the loss in weight is calculated as silica and expressed as a percentage of the original sample. The procedure is applicable to soluble (basic) silicates and to insoluble silicates after fusion with sodium carbonate.

### Reactions

$$SiO_2 + 4HF \overset{\text{Heat}}{---} SiF_4 + 2H_2O$$

### Chemicals and Solutions Required

1. Sulfuric acid, C.P.
   $H_2SO_4$, 96%

2. Hydrofluoric acid, C.P.
   HF, 48%

[redacted]

3. Hydrochloric acid, 1:1 (Solutions Code: SH-25a)

4. Hydrochloric acid, 1:100 (Solutions Code: SH-25a)

5. Methyl orange indicator, 0.1% (Solutions Code: SM-5a)

6. Sodium carbonate, C.P.
$Na_2CO_3$, C.P.

Procedure

| Soluble Silicates | Insoluble Silicates | |
|---|---|---|
| 1. | 1. | Weigh by difference a sample containing approximately 0.5 g. of silica. |
| | 2. | Transfer the sample into a platinum crucible and add about 5 times its weight of sodium carbonate. |
| | 3. | Fuse the mixture until the reaction is complete. |
| | 4. | Dissolve the melt in 1:1 hydrochloric acid. |
| 2. | | Transfer the sample to aplatinum evaporating dish and dilute with water until the dish is about half full. |
| 3. | | Warm until the sample dissolves. |
| 4. | 5. | Proceed as in step 6. |

6. Add 20 ml. of concentrated hydrochloric acid with constant stirring.

7. Evaporate to dryness on the steam bath. (Do not heat over 120°C)

8. Heat the residue at 110 to 120°C for 30 minutes.

9. Add 25 to 50 ml. of 1:1 hydrochloric acid.

10. Filter through a #40 ashless paper as soon as the soluble salts have dissolved.

[redacted]

11. Policeman the dish and rinse the dish and filter paper several times with 1:100 hydrochloric acid. (Remark 1)

12. Wash with hot distilled water until the filter paper is alkaline to methyl orange. (Remark 2) Test by adding a drop of indicator to the top edge of the paper.

13. Collect the filtrate and washings in the original platinum dish and evaporate to dryness.

14. Repeat steps 6 through 12 on this residue using a separate paper for the filtration. (Remark 3)

15. Transfer the filter papers to an ignited platinum crucible, and dry in an oven or under an infra-red heater.

16. Ignite in a muffle furnace at 1000 to 1100°C until free of carbon.

17. Remove the crucible from the furnace and ignite at the highest temperature of a blast lamp for 15 minutes.

18. Cool in a desiccator and weigh. The weight recorded is the weight of the silica plus the impurities. (Remark 4)

19. Moisten the residue with about 5 ml. of distilled water and add 3 to 5 drops of the sulfuric acid.

20. Cautiously add 10 to 15 ml. of the hydrofluoric acid.

21. Evaporate to a small volume, cool, and add 5 ml. more of hydrofluoric acid.

22. Evaporate to dryness, fuming off all the sulfuric acid.

23. Ignite in a blast lamp gradually increasing the temperature to full heat, Continue the ignition at full heat for 15 minutes.

24. Cool in the desiccator and weigh. The loss in weight represents the silica present.

Calculations

1. $\%SiO_2 = \frac{\text{loss in weight} \times 100}{\text{weight of sample}}$

2. $\text{ppm } SiO_2 = \%SiO_2 \times 10^4$

[redacted]

Remarks

1. The filter paper is washed with dilute hydrochloric to prevent hydrolysis of iron and aluminum salts and subsequent retention on the paper. If such salts are present in amounts considerably less than the amount of silica present, this operation may be omitted.

2. Filter paper containing hydrochloric is not easily ashed. Consequently all acid must be washed from the paper.

3. If the amount of silica present is small or if an accuracy less than 95% is acceptable this second evaporation and filtration may be neglected.

4. If the amount of occluded material is small the first weight less the weight of the crucible may be taken as the weight of the silica present. This procedure gives reasonably accurate results, but a check should be made with both procedures before omitting the volatization.

[redacted]

Code: WS-4a

H. W. Laboratory Manual
Water Methods

Reissued: December, 1948
Status: Standard
By: W.J.M.

DETERMINATION OF: (1) TOTAL DISSOLVED SOLIDS
(2) LOSS ON IGNITION OF DISSOLVED SOLIDS
(3) NON VOLATILE DISSOLVED SOLIDS
(Range 0 - 100 ppm)

## Safety Precautions

Observe general laboratory Safety Rules.

## Principle and Limitations of Method

The residue from an evaporation of a filtered water sample is the dissolved solids that were present. From an ignition of the dissolved solids, the Non-Volatile Dissolved Solids and Loss on Ignition of Dissolved Solids can be calculated.

In order to speed up the test, the evaporation is made beneath an infra-red heater instead of over a steam or water bath. The drying operation is made in a 105°C oven.

## Procedure

### A. Total Dissolved Solids

1. Ignite a clean 100 ml. platinum dish to constant weight.
2. Evaporate a 300 ml. sample in the dish by making 50 ml. additions with a 50 ml. pipet. Use an infra-red heater for the evaporation (Remark 1).
3. Dry to constant weight in the 105°C oven.

## Calculations

$$\text{ppm total dissolved solids} = \frac{\text{g. gain in wt. by dish} \times 10^6}{\text{ml. of sample}}$$

### B. Loss on Ignition of Dissolved Solids

1. Ignite at 850 to 900°C the platinum dish that contains the weighed residue from the dissolved solids determination.

[redacted]

2. Cool the dish and moisten with a few drops of distilled water.

3. Dry to constant weight in the 105°C oven.

Calculations

$$\text{ppm loss on ignition of dissolved solids} = \frac{\text{loss in wt. of dish after ignition, g.} \times 10^6}{\text{ml. of sample}}$$

C. Non-Volatile Dissolved Solids

Calculations

$$\text{ppm non-volatile dissolved solids} = \frac{\text{wt. of residue in dish after ignition g.} \times 10^6}{\text{ml. of sample}}$$

D. Total Solids in Process Water

a. Preparation of Calibration Curve

1. Determine the ppm total solids on a sample of raw water as in WS-4a, part A.

2. Titrate at least five samples each of raw and distilled water using 0.01N hydrochloric acid with 2 drops methyl red indicator for 50 ml. samples.

3. Make at least five mixtures of raw and distilled water (i.e. one part raw, four parts distilled; two parts raw, three parts distilled, etc.) and titrate as before with 0.01N hydrochloric acid. The total solids in each sample should be known.

4. Plot a curve of ppm total solids against ml. 0.01N hydrochloric acid.

b. Analyses of the Sample

1. Place 50 ml. of process water in a clean, dry 100 ml. beaker.

2. Add two drops of methyl red indicator and titrate with 0.01N hydrochloric acid.

3. Refer to calibration chart to determine ppm total solids.

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Remarks

1. Never allow the dish to go dry while under the heater since the temperature would be high enough to drive off volatile matter.

2. The endpoint using 0.01N hydrochloric acid is not very distinct and a little practice is necessary before consistent results can be obtained.

3. In practice it is usually found that the 50 ml. sample will stay pink on the addition of two drops methyl red indicator. This normally indicates less than 10 ppm.

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WS-4b

Reissued: December, 1948
Status: Standard
By: W.J.M.

# TOTAL SOLIDS IN 200 AREA PROCESS WATER

## Safety Precautions

Observe general laboratory Safety Rules.

## Principle and Limitations

The total solids in the process water are titrated with hydrochloric acid and the amount of the solids read from a predetermined calibration chart.

## Chemicals and Solutions

### A. Preparation of Calibration Curve

1. Determine the ppm total solids on a sample of raw water as in Water Methods Code: WS-4a, Part A.
2. Titrate at least five samples each of raw and distilled water with hydrochloric acid with 2 drops of the methyl red indicator for 50 ml. samples.
3. Make at least five mixtures of raw and distilled water (i.e., one part raw, four parts distilled; two parts raw, three parts distilled, etc.) and titrate as before with the hydrochloric acid. The total solids in each sample should be known.
4. Plot a curve of ppm total solids against ml. of 0.01 N hydrochloric acid.

### B. Analysis of the Sample

1. Place 50 ml. of process water in a clean, dry 100 ml. beaker.
2. Add 2 drops of methyl red indicator and titrate with the hydrochloric acid.
3. Refer to the calibration chart to determine ppm total solids.

[redacted]

Remarks

1. The end point using 0.01 N hydrochloric acid is not very distinct and a little practice is necessary before consistent results can be obtained.

2. In practice it is usually found that the 50 ml. sample will stay pink on the addition of 2 drops of methyl red indicator. This normally indicates less than 10 ppm.

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WS-5a

Reissued: December, 1948
Status: Standard
By: W.J.M.

## DETERMINATION OF:

(1) SUSPENDED SOLIDS
(2) LOSS ON IGNITION OF SUSPENDED SOLIDS
(3) NON-VOLATILE SUSPENDED SOLIDS
(Range 0 - 100 ppm)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

Theroux, Eldridge, and Mallmann, "Analysis of Water and Sewage", 2nd Ed., McGraw - Hill Book Co., Inc. 5-6 (1936).

### Principle and Limitations

The suspended solids are filtered from solution on the ignited asbestos mat of a Gooch crucible and are weighed directly. Ignition of the solids drives off the matter which can be volatilized and leaves the non-volatile suspended solids. These quantities are calculated to parts per million based on the original sample.

The procedure has been in use as standard practice in water laboratories for a number of years. In the range of 0-10 ppm suspended solids, results can be duplicated to within 10%.

### Special Apparatus

Crucible, Gooch, with asbestos mat (Apparatus Code: AC-18a)

### Procedure

A. Suspended Solids

1. Dry a Gooch crucible in the 105°C oven and then ignite it in the 850 to 900°C muffle furnace (Remark 1).

2. Cool the crucible, moisten the mat with a few drops of distilled water, and dry in the 105°C oven to constant weight.

[redacted]

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[redacted]

3. Measure a sample which will contain at least 2 mg. of suspended solids and filter the sample through the prepared Gooch.

4. Follow the sample filtration with several distilled water rinsings of the sample flask to insure that all of the suspended matter has been transferred to the crucible.

5. Dry to constant weight in the 105°C oven.

Calculations

$$\text{ppm total suspended solids} = \frac{\text{gain in wt. by crucible, g.} \times 10^6}{\text{ml. of sample}}$$

B. Loss on Ignition of Suspended Solids

1. Ignite for one hour at 850 to 900°C the crucilbe that contains the weighed solids from the suspended solids determination.

2. Cool the crucible and moisten with a few drops of distilled water.

3. Dry to constant weight in the 105°C oven.

Calculations

$$\text{ppm loss on ignition of suspended solids} = \frac{\text{loss in wt. of crucible during ignition g.} \times 10^6}{\text{ml. of sample}}$$

C. Non-Volatile Suspended Solids

Calculations

$$\text{ppm non-volatile suspended solids} = \frac{\text{wt. of residue in crucible after ignition g.} \times 10^6}{\text{ml. of sample}}$$

Remarks

1. The ignition may be omitted if the loss on ignition of suspended solids is not to be determined.

[redacted]

[redacted]

Code: WS-6a

H. W. Laboraoty Manual
Water Methods

Reissued; December, 1948
Status: Standard
By: W.J.M.

# DETERMINATION OF SULFATES, TURBIDIMETRIC METHOD

(Range 0.5 - 100 ppm)

## Safety Precautions

Observe general laboratory Safety Rules.

## Principle and Limitations

This method depends on the formation of highly insoluble barium sulfate which is precipitated in the presence of excess salt and acid and held in suspension by glycerol. The percent of light transmitted by the suspension is proportional to the sulfate concentration.

This is a very rapid method for control analysis. It gives reproducidble results and will accurately detect as low as 0.025 mg of sulfate in a 50 ml. sample.

## Reactions

$SO_4^{-2} + Ba^{+2} --- BaSO_4$

## Special Apparatus

1. Spectrophotometer, Coleman model II (Apparatus Code: AS-16a)
2. Matched cuvettes, 19 mm (Apparatus Code: AC-21a)

## Reagents and Chemicals

1. Barium chloride crystals, C.P., 60 mesh
   $BaCl_2 \cdot 2H_2O$, C.P., 60 mesh
2. Glycerol solution, 33.3% by vol. (Solutions Code: SG-12a)
3. Sodium chloride-hydrochloric acid solution, 1.6% NaCl-2.4% HCl (Solutions Code: SS-4c)
4. Standard sulfate solution, 100 ppm (Solutions Code: SS-24a)

[redacted]

## Procedure

A Coleman Spectrophotometer model 11 is used in this determination. For details as to its use and operation see Apparatus Code: AS-16a.

### A. Standardization of Instrument

A standard transmittance curve is prepared from a sulfate solution of known strength and is used as a reference for unknown samples. This curve must be prepared under the same conditions as the samples to be run and is good only for the instrument for which it is determined.

1. Deliver into 100 ml. volumetric flasks, 5, 10, 15 and 20 ml. portions of standard sulfate solution and deliver 10 ml. of distilled water into a fifth flask which serves as a blank.
2. Add 10 ml. of sodium chloride-hydrochloric acid solution and 10 ml. of glycerol solution to each flask and mix.
3. Add about 0.25 g. of barium chloride to the sample and the blank.
4. Shake until the crystals dissolve.
5. Dilute all samples to the mark with distilled water and mix.
6. Transfer a portion of standards and blank successively to one of a matched pair of 19 mm round cuvettes and determine the percent transmittance against distilled water at 450 millimicrons using the PC-4 filter.
7. On semi-log paper plot ppm sulfate against the corrected % transmittance with the latter on the log scale.

## Calculations

1. $$\text{ppm sulfate} = \frac{\text{ml. standard} \times 100}{\text{volume of dilution}}$$

2. $$\text{\%transmittance corrected} = \frac{\text{\%transmittance of sample} \times 100}{\text{\%transmittance of blank}}$$

B. Determination of Sulfates in Unknown Sample

1. Transfer duplicate 50 ml. portions of filtered sample to 100 ml. volumetric flasks, one portion for a blank and the other for the development of turbidity.

2. Add 10 ml. of sodium chloride-hydrochloric acid solution and 10 ml. of glycerol solution to both samples and mix well.

3. Add about 0.25 g. of barium chloride to the turbidity sample only.

4. Shake until the crystals dissolve.

5. Dilute both samples to the mark with distilled water and mix well.

6. Transfer to 19 mm round cuvettes and determine the percent transmittance against distilled water at 450 mu using the PC-4 filter.

Calculations

1. $\%\text{transmittance corrected} = \dfrac{\%\text{transmittance of sample} \times 100}{\%\text{transmittance of blank}}$

2. From the standard curve read the ppm of sulfate for the corrected percent transmittance.

3. a. $\text{ppm of sulfate} = \dfrac{A \times C \times 10^{-6} \times 10^{6}}{B} = \dfrac{A \times C}{B}$

or for the conditions above

b. $\text{ppm of sulfate} = \dfrac{100\,A}{50} = 2\,A$

where A = ppm sulfate of solutions compared(from curve)

B = Volume of original sample, ml.

C = Volume of sample as prepared for comparison, ml.

Remarks

1. The barium sulfate suspension prepared in this procedure is stable for about 2 hours.

2. If the sample does not give a percent transmittance between 55 and 97 use a smaller or larger sample as follows:

   a. If the percent transmittance is less than 55 use a 10ml. sample and proceed as above but multiplying the result obtained by 5.

   b. If the percent transmittance is more than 97 use a 100 ml. sample and evaporate to about 50 ml. Proceed as above but multiply the result obtained by 0.5.

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WS-7a
Reissued: January, 1949
Status: Standard
By: W.J.M.

## SODIUM DICHROMATE (DIPHENYLCARBAZIDE METHOD)

(Range 0.1 - 4 ppm)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

Furman, "Scott's Standard Methods of Chemical Analysis", I, 5th Ed., D. Van Nostrand Co., Inc., New York, 290 (1939).

### Principle and Limitations

An acid solution of dichromate ion reacts with diphenylcarbazide to form a violet color which is stable for more than two hours. In the presence of sulfuric acid the color develops immediately and the intensity is proportional to the amount of chromium (VI) present. The color is measured on a spectrophotometer at 540 mu.

### Reactions

$$\mathrm{Cr_2O_7^{-2}} + \text{-}\underset{\mathrm{N}}{\mathrm{N}}\text{-}\underset{\mathrm{H}}{\mathrm{N}}\text{-}\underset{\mathrm{O}}{\mathrm{C}}\text{-}\underset{\mathrm{H}}{\mathrm{N}}\text{-}\underset{\mathrm{H}}{\mathrm{N}}\text{-} \quad \text{---} \quad \text{Violet colored complex}$$

### Special Apparatus

1. Coleman Model 11 Spectrophotometer (Apparatus Code: AS-16a)
2. Filter PC-4 for Coleman spectrophotometer
3. Matched cuvettes; 19 mm. (Apparatus Code: AC-21a)

### Chemicals and Solutions

1. Chromium standard, 50 ppm (Solutions Code: SC-10b)
2. Sulfuric acid, 6N (Solutions Code: SS-23a)
3. Diphenylcarbazide, 0.2% (Solutions Code: SD-10b)

[redacted]

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The Coleman Model 11 Spectrophotometer is used in this determination. See Apparatus Code: AS-16a for details on the operation and maintenance of this instrument.

A. Preparation of the Calibration Curve

A calibration curve relating the percent transmission to the concentration of the chromium must be prepared for each instrument used. If such a curve is not available, prepare it as follows before analyzing the sample (Remark 1).

1. Pipet 50 ml. of the chromium standard solution into a 500 ml. volumetric flask and dilute to the mark with distilled water. This solution is not stable for very long and must be made fresh each time.

2. Pipet 2.5 ml, 5 ml, 7.5 ml, 10 ml, 12.5 ml, 15 ml, 17.5 ml, and 20 ml samples of the diluted standard into 50 ml. glass stoppered graduated mixing cylinders (Remark 2). These correspond to 0.5 ppm, 1 ppm, 1.5 ppm, 2.0 ppm, 2.5 ppm, 3.0 ppm, 3.5 ppm and 4.0 ppm dichromate solutions.

3. Proceed with the color development and measurement as described in Steps 2 to 6 under Part B below.

4. Plot the percent transmittance of each solution as ordinate against the amount of chromium (expressed as ppm of sodium dichromate on semi-log graph paper. Draw a smooth curve (Remark 3) through the various points.

B. Analysis of the Sample

Prepare a blank with each set of samples analyzed.

1. Pipet a 25 ml. sample into a 50 ml. graudated glass stoppered mixing cylinder (Remark 2).

2. Add 1 ml. of the sulfuric acid to the contents of the cylinder.

3. Add 0.5 ml. of diphenylcarbazide solution to the cylinder, and swirl the contents to insure thorough mixing.

4. Dilute to the mark with distilled water. Mix well.

5. Allow to stand 5 minutes but not more than 10 minutes.

6. Transfer the solution into a 19 mm Coleman cuvette.

7. Determine the percent transmission of the sample solution agains distilled water. Use the PC-4 filter in the spectrophotometer, and make the measurement at 540 mu.

Calculations

$$\%\ \text{transmittance corrected} = \frac{\%\ \text{transmittance read} \times 100}{\%\ \text{transmittance of blank}}$$

Remarks

1. Check the calibration curve whenever a new chromium standard solution is introduced, or at least once each month.

2. Do not clean the glassware used in this determination with chromic acid cleaning solution. Glassware may be satisfactorily cleaned by rinsing in concentrated sulfuric acid.

3. This curve should be a straight line. Consult supervision if it does not follow a straight line between 100% and 30% transmission.

Code: WS-7b

H. W. Laboratory Manual
Water Methods

Reissued: December, 1948
Status: Standard
By: W.J.M.

## SODIUM DICHROMATE DIRECT COLOR METHOD

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

Memoranda, Rasmussen and Stehney to Kitson, "The Effect of pH on the Absorption of Dichromate", March 26 and April 10, 1945.

### Principles and Limitations

Sodium dichromate solutions possess an orange or yellow color, depending upon the pH or acidity of the system. In any case, the radiation absorption in the near ultra violet wave lengths (350-400 mu) is relatively intense, and permits "colorimetric" measurement of the amount of dichromate in the system. Since the intensity of the absorption depends on the wave length and the pH of the system, these variables must be carefully controlled.

In the present method, a Beckman spectrophotometer is used for the color measurements. The pH of the system is adjusted to a value of 7.0 with the aid of buffers. Since the amount of dichromate in the sample is extremely small, a 10 cm. absorption cell is used and dilution of the original sample avoided as much as possible.

Since no color development is necessary, the accuracy of the method is limited only by the accuracy of the spectrophotometric measurements, and is of the order of about 1%.

### Special Apparatus

| | |
|---|---|
| 1. Beckman spectrophotometer | (Apparatus Code: AS-16b) |
| 2. Cells, absorption, 10 cm., Beckman | (Apparatus Code: AC-21j) |

### Chemicals and Solutions

| | |
|---|---|
| 1. Phosphate buffer, pH 7 | (Solutions Code: SB-22c) |

## Procedure

The Beckman spectrophotometer is used in this determination. See Apparatus Code: AS-16b for details on the maintenance and operation of this instrument.

### A. Preparation of the Calibration Curve

A calibration curve relating the per cent transmission to the concentration of sodium dichromate must be prepared for each instrument used. If such a curve is not available (Remark 1), prepare it as follows:

1. Pipet 50 ml. of the standard dichromate solution into a 250 ml. volumetric flask.

2. Dilute the solution in the flask to the mark with distilled water, and mix thoroughly.

3. Pipet 1, 2, 3, 4, 5, 10, 15, and 20 ml. portions of this diluted standard into individual 50 ml. volumetri flasks.

4. Dilute the contents of each flask to the mark with distilled water. Fill an extra flask with distilled water to serve as the blank.

5. To each of the solutions prepared in Step 4 add 2 ml. of the phosphate buffer. Mix each solution well, and transfer each into a clean 10 cm. absorption cell.

6. Measure the transmission of each solution against the blank at 370 mu.

7. Plot the per cent transmission of each solution as the ordinate against the amount of chromium (expressed as ppm sodium dichromate) (Remark 2) on semi-log graph paper (Remark 3). Draw a smooth curve through the various points.

### B. Analysis of the Sample

Run duplicate determinations on each sample. Run a blank with each sample analyzed (Remark 4).

1. Fill a 50 ml. volumetric flask to the mark with the sample.

2. Add to the volumetric flask 2 ml. of the phosphate buffer.

3. Mix the solutions thoroughly. Transfer each solution into a clean 10 cm. absorption cell.

4. Measure the transmission of the sample against that of the blank at 370 mu.

5. Determine the amount of chromium in the sample (expressed as ppm sodium dichromate) from the calibration curve.

Remarks

1. Prepare a new calibration curve whenever a new phosphate buffer is used.

2. The solutions correspond, in order, to 0.2, 0.4, 0.6, 0.8, 1.0, 2.0, 3.0, and 4.0 ppm sodium dichromate.

3. This curve should be a straight line. If it is not, consult supervision.

4. Prepare this blank following the given procedure, but substituting distilled water for the sample in Step 1.

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WS-8a
Reissued: December, 1948
Status Standard
By: W.J.M.

# SODIUM CHLORIDE
(Range: 0-300 ppm)

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

Theroux, Eldridge, and Mallmann, "Analysis of Water and Sewage", 3rd. Ed., McGraw-Hill Book Co., New York, 94 and 163 (1943).

## Principle and Limitations

Sodium chloride in boiler water is determined by titration of the neutralized sample used in Water Methods Code: WA-2a with standard silver nitrate solution in the presence of potassium chromate indicator. The chloride is precipitated as silver chloride. The end point of the titration is reached when a red precipitate of silver chromate ($Ag_2CrO_4$) first appears owing to the reaction of a slight excess of silver nitrate with the potassium chromate.

## Reactions

$$NaCl + AgNO_3 \text{ --- } AgCl + NaNO_3$$

$$K_2CrO_4 + 2AgNO_3 \text{ --- } Ag_2CrO_4 + 2KNO_3$$

## Special Apparatus

Buret, semi-micro 10 ml. (Apparatus Code: AB-21a)

## Chemicals and Solutions

1. Potassium chromate, 10% (Solutions Code: SP-12a)
2. Silver nitrate, 0.5 mg. $Cl^-$/ml. (Solutions Code: SS-9d)

## Procedure

1. To the sample neutralized to the methyl orange end point add 0.5 ml. of the potassium chromate indicator.

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[redacted]

2. Mix the contents of the container thoroughly and titrate with standard silver nitrate from a buret, a few drops at a time, with constant stirring until the first permanent reddish color is obtained (Remark 1). Record the ml. of silver nitrate used.

Calculations

ppm sodium chloride = 10 x (ml. of silver nitrate - 0.2)

Remarks

1. This color change may be more readily determined by comparison with a distilled water sample to which 0.5 ml. of potassium chromate indicator has been added.

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[redacted]

Code: WS-9a

H. W. Laboratory Manual
Water Methods

Reissued: December, 1948
Status: Standard
By: W.J.M.

## SODIUM SULFATE
(Range 0-1500 ppm)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

Theroux, F.R., Eldridge, E.F., and Mallmann, W.L., "Analysis of Water and Sewage", 3rd. Ed., pp. 93, 94, and 159, McGraw - Hill Book Co., Inc., New York, (1943).

### Principle and Limitations

Benzidine hydrochloride reacts with sulfates in a hydrochloric acid solution to form a slightly soluble compound of benzidine and sulfuric acid. This compound is filtered and washed entirely free of excess hydrochloric acid. The amount of sulfuric acid in the compound is then determined by titration with standard sodium hydroxide.

### Reactions

$$Na_2SO_4 + C_{12}H_8(NH_2)_2 \cdot 2HCl \text{ - - - } C_{12}H_8(NH_2)_2 \cdot H_2SO_4 + 2NaCl$$

$$C_{12}H_8(NH_2)_2 \cdot H_2SO_4 + 2NaOH \text{ - - - } Na_2SO_4 + C_{12}H_8(NH_2)_2 \cdot 2H_2O$$

### Special Apparatus

| | |
|---|---|
| Buret, semi-micro 10 ml. | (Apparatus Code: AB-21a) |

### Chemicals and Solutions

| | |
|---|---|
| 1. Benzidine dihydrochloride, 3% | (Solutions Code: SB-4b) |
| 2. Hydrochloric acid, 6N | (Solutions Code: SH-25a) |
| 3. Phenolphthalein indicator, 0.5% | (Solutions Code: SP-8b) |
| 4. Sodium hydroxide, 0.02N | (Solutions Code: SS-8a) |

[redacted]

Procedure

1. Filter at least 100 ml. of the boiler water sample through a filter paper.

2. Measure 100 ml. of the filtered sample into a 250 ml. Erlenmeyer flask.

3. Add sufficient hydrochloric acid to the sample to make neutral or slightly acid to litmus paper.

4. Add 10 ml. of 3% benzidine dihydrochloride solution to the contents of the flask and mix by swirling.

5. Allow the mixture to stand for about 10 minutes or sufficiently long so that the precipitate settles.

6. Decant the supernatant liquid through a Whatman No. 1 filter paper without unnecessary mixing of the liquid and precipitate. Collect the filtrate in a second 250 ml. Erlenmeyer flask (Remark 1). Discard the filtrate, but retain the flask for Step (8).

7. Transfer the precipitate (benzidine sulfate) to the filter paper by mixing with small quantities of distilled water and pouring the precipitate onto the filter paper. Rinse the flask several times with distilled water, adding the rinsings to the precipitate contained on the filter paper. Allow each rinse to drain through the paper before the next is added (Remark 2).

8. Transfer the filter paper containing the benzidine sulfate to the original flask, add about 50 ml. of distilled water, stopper the flask and shake thoroughly to disintegrate the filter paper.

9. Add several drops of 0.5% phenolphthalein indicator to the contents of the flask and titrate with the sodium hydroxide solution, using a 10 ml. semi-micro buret, to a pink end point.

10. Heat the sample to boiling and continue the titration until a permanent pink end point results.

11. Record the ml. of standard sodium hydroxide used.

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[redacted]

Code: WS-9a

Calculations

ppm sodium sulfate = 14.2 X ml. 0.02N sodium hydroxide.

Remarks

1. Prior to discarding the filtrate contained in the 250 ml. Erlenmeyer flask, test for the presence of sodium sulfate by adding 1 ml. of benzidine dihydrochloride. If a precipitate is found, repeat steps (4) through (9) adding the precipitate to the original filter paper.

2. The washing removes undesirable hydrochloric acid.

[redacted]

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WS-10a
Reissued: December, 1948
Status: Standard
By: W.J.M.

## SODIUM SULFITE
(Range 0-30 ppm)

### Safety Precautions

1. Observe general laboratory Safety Rules.

2. Observe rules for handling acetic acid.

### Literature References

Treadwell and Hall, "Analytical Chemistry", II 9th Ed. John Wiley and Sons, New York, 623 (1942).

### Principle and Limitations

Iodate oxidizes sulfite to sulfate in the presence of sufficient acid. Iodide must be present to indicate the complete oxidization of the sulfite, which is evident by the production of free iodine to form the intensely blue-colored complex in the presence of starch solution.

### Reactions

$$3SO_3^{-2} + IO_3 \; --- \; 3SO_4^{-2} + I^-$$

$$IO_3^- + I^- + 6H^+ \; --- \; I_2 + 3H_2O$$

### Special Apparatus

| | |
|---|---|
| Buret, semi-micro 10 ml. | (Apparatus Code: AB-21a) |

### Chemicals and Solutions

| | |
|---|---|
| 1. Potassium iodate, 0.01586N | (Solutions Code: SP-20b) |
| 2. Hydrochloric acid, 20% | (Solutions Code: SH-25a) |
| 3. Potassium iodide, 3% | |
| 4. Starch indicator solution, 1.0% | (Solutions Code: SS-21a) |

[redacted]

[redacted]

Code: WS-10a

Procedure

1. Measure 100 ml. of the freshly collected and cooled, but unfiltered, sample and transfer into a 250 ml. Erlenmeyer flask.

2. Add 6 ml. of 20% hydrochloric acid and 5 ml. of 3% potassium iodide to the flask containing the sample.

3. Add 2 ml. of strach indicator and mix the contents of the flask by swirling.

4. Titrate the solution with standard potassium iodate solution until a faint blue color results from the liberation of free iodine.

Calculation

ppm sodium sulfite = 10 X ml. 0.01586$\underline{N}$ potassium iodate.

[redacted]

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WS-11a
Reissued: December, 1948
Status: Standard
By: W.J.M.

# SODIUM, COLORIMETRIC METHOD
(Range 10 -100 ppm)

## Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules for handling alcohol.

## Literature References

1. Furman, N. H., "Scott's Standard Methods of Chemical Analysis", I 5th Ed., D. Van Nostrand Co., Inc., New York, 879-880 (1939)
2. McCormick, D. R., and Carlson, W. E., Chemist Analyst, 31, 15, (1942).

## Principle and Limitations of Method

Sodium may be precipitated from slightly acid solutions as the complex sodium magnesium uranyl acetate. This precipitation must be done in the cold in the presence of ethyl alcohol to reduce the solubility of the precipitate. The filtered precipitate may be dissolved in warm water and estimated directly be means of the yellow color of the solution. The color obeys the Beers-Lambert law over the range 10-100 ppm of sodium. The accuracy of this method, if care is exercised to insure complete precipitation, is 5%.

## Reactions

$$Na^+ + Mg(C_2H_3O_2)_2 + 3UO_2(C_2H_3O_2)_2 + C_2H_3O_2^- \text{ ---}$$

$$NaC_2H_3O_2 \cdot Mg(C_2H_3O_2)_2 \cdot 3UO_2(C_2H_3O_2)_2$$

## Special Apparatus

1. Coleman Model 11 Spectrophotometer (Apparatus Code: AS-16a)
2. Absorption cells, 5 cm. Aminco (Apparatus Code: AC-21d)
3. PC-4 filter for spectrophotometer
4. Filtration tube (Apparatus Code: AT-20c)

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[redacted]

Chemicals and Solutions

1. Standard sodium solution, 1000 ppm (Solutions Code: SS-22a)
2. Ethanol, C.P.
   $C_2H_5OH$, 95%
3. Magnesium uranyl acetate (Solutions Code: SM-1b)
4. Sodium magnesium uranyl acetate, saturated (Solutions Code: SS-29a)

Procedure

The Coleman Model 11 Spectrophotometer is used in this determination. See Apparatus Code: AS-16a for details on the operation and maintenance of this instrument.

A. Preparation of the Calibration Curve

A calibration curve relating the per cent transmission to the concentration of sodium must be prepared for each instrument. If such a curve is not available (Remark 1), prepare it as follows before analyzing the sample.

1. Deliver into a 6 inch test tube 1 ml. of the standard sodium solution.
2. Proceed with the color development as described in Steps 2 to 10 under B-Analysis of the sample.
3. Transfer into separate 10 ml. volumetric flasks, 0.1, 0.5, 1, 2.5, and 5 ml. of the solution and dilute to the mark with distilled water.
4. Mix the flasks thoroughly and transfer the solution to clean, dry 5 cm. Aminco absorption cells, Insert the PC-4 filter into the spectrophotometer, set the wavelength at 425 mu, and measure the transmission of the solutions.
5. Plot, on single cycle semi-log papter, the per cent transmission of each solution as the ordinate against the sodium in each solution.

B. Analysis of the Sample

In the analysis of a single sample, run duplicate determinations. Run at least one blank on the reagents (Remark 2).

1. Pipet 1 ml. of the sample into a 6 inch test tube.

2. Add 1 ml. of 95% ethanol to the test tube.

3. Cool the contents of the test tube to 0°C by placing the test tube into a beaker containing ice-salt. Assemble the cooling medium on a ring stand, clamp the test tube firmly into position in the beaker, and insert the agitator of a mechanical stirrer into the test tube. Clamp the stirrer into position.

4. Start the mechanical agitator and add 10 ml. of the magnesium uranyl acetate reagents. Continue the stirring, allowing the precipitation to proceed for 45 minutes.

5. Discontinue the stirring and remove the agitator from the test tube, first being sure to wash off all particles of precipitate with the alcohol wash solution.

6. Transfer the test tube to a centrifuge and centrifuge for 5 minutes at 2000 to 2500 r.p.m. Decant off as much of the supernatatant liquid as possible.

7. Transfer the precipitate contained in the test tube to a 3 ml. "F" fritted-glass funnel by means of the saturated sodium magnesium uranyl acetate wash solution and filter with suction. Do not allow the solution to warm up during the filtration.

8. Wash the precipitate with several portions of the saturated sodium magnesium uranyl acetate wash solution and then allow the precipitate to suck dry. Discard the filtrate.

9. Insert the 3 ml. fritted-glass filtering funnel into the filtration tube (AT-20c) and insert the assembly into a clean, dry 10 ml. volumetric flask. Add several small portions of hot distilled water to the precipitate contained on the filter and collect the dissolved preipitate in the 10 ml. volumetric flask.

10. When the precipitate has been completely dissolved off the filter, remove the filtering funnel from the flask, dilute the contents of the flask to the mark and mix thoroughly.

11. Transfer the solution to a clean, dry 5 cm. Aminco absorption cell. Insert the PC-4 filter into the spectrophotometer, set the wavelength at 425 mu, and measure the transmission of the solution.

[redacted]

12. Determine the ppm of sodium in the sample by comparison with the standard transmittance curve.

Remarks

1. Prepare a new calibration curve whenever new magnesium uranyl acetate or sodium standard solutions are introduced. A new calibration curve should be prepared at frequent intervals, frequency to be determined by the results obtained with an old curve.

2. Prepare this blank by following the given procedure, omitting the sample addition and filtration.

[redacted]

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WT-1a

Reissued: December, 1948
Status: Standard
By: W.J.M.

## DETERMINATION OF TURBIDITY
(Range 2 - 500 ppm)

### Safety Precautions

Observe general laboratory Safety Rules.

### Principle and Limitations of the Method

The particles which make up the turbidity of water absorb and scatter light. The amount of light transmitted by such a turbid solution is proportional to the turbidity.

This method is entirely empirical. It is believed to give results which are correct to about 1 ppm and is reproducible to 1 ppm. Although it is known that particle size has a very pronounced effect on the absorption and scattering of light, it is believed that the photoelectric method is as good as any other method of observation.

### Special Apparatus

1. Spectrophotometer, Coleman Model 11 (Apparatus Code: AS-16a)
2. Absorption cells, 50 mm., Coleman (Apparatus Code: AC-21b)

### Chemicals and Solutions

Standard turbidity solution, 100 ppm (Solutions Code: ST-23a)

### Procedure

The Coleman Spectrophotometer Model 11 is used in this determination. For details about its use and operation see (Apparatus Code: AS-16a).

#### A. Standardization of Instrument

1. Deliver into 25 ml. volumetric flasks 5, 10, 15, 20, and 25 ml. samples of standard silica solution.
2. Dilute to the mark with distilled water and mix well.

[redacted]

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3. Transfer to marched 50 mm absorption cells and determine the transmittance of each sample at 520 mu using the PC-4 filter.

4. On semi-log co-ordinate paper plot ppm against percent transmittance as read on the spectrophotometer.

Calculations

$$\text{ppm} = \frac{\text{ml. std.} \times 100}{25} = 4 \times \text{ml. standard}$$

B. Determination of Turbidity in Unknown Sample

1. Shake and mix the sample well.

2. Determine its percent transmittance at 520 mu using 50 mm absorption cells and the PC-4 filter.

Calculations

Read the turbidity in ppm directly from the standard curve.

[redacted]

H. W. Laboratory Manual
Water Methods

Code: WT-1b

Reissued: December, 1948
Status: Stantard
By: W.J.M.

# DETERMINATION OF ARTIFICIAL TURBIDITY
(Range 0-100 ppm)

## Safety Precaution

Observe general laboratory Safety Rules.

## Principles and Limitations of Method

The particles which are added to water to give aritifical turbidity absorb and scatter light. The amount of light transmitted by such a solution is proportional to its turbidity.

The method gives results reproducible to about 1 ppm providing the solution is well mixed in the absorption cell. A settling time of one minute without agitation causes an error of 5 to 10 ppm.

Dichromate up to 15 ppm does not interfere.

## Special Apparatus

1. Spectrophotometer, Coleman Model 11 (Apparatus Code: AS-16a)
2. Matched cuvettes, 50 mm (Apparatus Code: AC-21a)

## Chemicals and Solutions

Artificial turbidity material simiar to that in sample.

## Procedure

A Coleman Universal Spectrophotometer is used in this determination. For details regarding its use and operation see Apparatus Code AS-16a.

### A. Standardization of Instrument

A standard transmittance curve is prepared from a solution containing a known amount of the material used to produce the turbidity. This curve is good only on the instrument on which it is determined and under the conditions of standardization. It should also be checked for each separate material.

[redacted]

1. Dry a sample of the material in am oven at 150°C.
2. Weigh out a portions of 0.1000 g. of the material. Transfer into a 1 liter volumetric flask, and dilute to the mark with distilled water, mix well.
3. Deliver into 100 ml. volumetric flasks 10, 20, 30, 50, 75, and 100 ml. portions of the suspension being sure that it is kept well mixed.
4. Dilute to the mark with distilled water and mix well.
5. Transfer each sample into a 50 ml. absorption cell and compare immediately against distilled water in the spectrophotometer at 600 mu. (Remark 1)
6. On semi-log paper plot turbidity as micrograms per ml. of solutions against percent transmittance with the latter on the log scale.

Calculations

Calculate the turbidity in ppm of solution compared for sample.

$$\text{ppm} = \frac{\text{ml. standard} \times 100}{\text{volume of dilution}}$$

B Determination of Turbidity in Unknown Sample

1. Shake the sample thoroughly and transfer into a 50 mm. absorption cell and compare immediately against distilled water at 600 mu. (Remark 1)
2. Read the percent transmittance on the spectrophotometer.
3. From the curve read the turbidity in ppm corresponding to the observed percent transmittance.

Remarks

1. The sample must be kept thoroughly agitated at all times to prevent errors due to settling.

[redacted]

## H. W. LABORATORY MANUAL

## 100 AREA LABORATORY SOLUTIONS SECTION

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| CODE | TITLE | PAGES |
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| ST-5b | Tellurium Solution, 10 mg. Te/ml. | 2 |
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| ST-9a | Titan Yellow Indicator, 0.05% | 1 |
| ST-15a | Orthotolidine, 0.1% | 1 |
| ST-23a | Turbidity Standard, 100 ppm | 1 |
| SW-1a | Redistilled Water | 1 |
| SW-1b | Redistilled Water, Silica Free | 1 |
| SW-1c | Redistilled Water, Ammonia Free | 1 |
| SZ-7c | Zirconium-Alizarin Indicator | 2 |
| SZ-9c | Zirconium Nitrate, 50 mg. Zr/ml. in 3N Nitric Acid | 1 |

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H. W. Laboratory Manual
Laboratory Solutions

Code: SA-6a

Reissued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

# AQUA REGIA

(1 Vol. Conc. $HNO_3$)
(3 Vol. Conc. $HCl$)

## Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe the rules for handling corrosive chemicals, particularly those pertaining to concentrated nitric and hydrochloric acids.
3. Aqua regia is a corrosive, volatile liquid with a suffocating odor. It attacks all metals including gold and platinum.

## Literature References

Welcher, Frank, "Chemical Solutions", D. Van Nostrand Co., Inc., New York, 24 (1943).

## Quantity and Container

100 ml. in a 125 ml. glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Hydrochloric acid, C.P. $HCl$, 36% | 750 ml. | 75 ml. |
| 2. Nitric acid, C.P. $HNO_3$, 70% | 250 ml. | 25 ml. |

## Preparation

1. Measure 25 ml. concentrated nitric acid and 75 ml. of concentrated hydrochloric acid and add together into a 250 ml. beaker.
2. Transfer the acid into a 125 ml. glass-stoppered Pyrex bottle and store in a dark, cool place (Remark 1).

[redacted]

Remarks

1. When the aqua regia is to be stored for sometime, add 25 ml. of distilled water to the acid mixture. The addition of water reduces the quantity of chlorine and other objectionable gases which are evolved.

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

| | |
|---|---|
| Code: | SA-10a |
| Reissued: | August, 1948 |
| Status: | Standard |
| By: | D.W.T.-L.F.W. |

# AMMONIUM MOLYBDATE
(5%)

## Safety Precautions

Observe general laboratory Safety Rules.

## Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Ammonium Molybdate, C.P.<br>$(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ | 50 g. | 25 g. |

## Preparation

1. Transfer the 25 g. of ammonium molybdate into a 500 ml. volumetric flask.
2. Add approximately 400 ml. of distilled water and dissolve the salt.
3. Dilute to volume with distilled water.
4. Transfer the solution into a 500 ml. glass-stoppered Pyrex bottle for storage.

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[redacted]

H. W. Laboratory Manual
Laboratory Solutions

| | |
|---|---|
| Code: | SA-10c |
| Reissued: | August, 1948 |
| Status: | Standard |
| By: | D.W.T.-L.F.W. |

# AMMONIUM MOLYBDATE
(1%)

## Safety Precautions

Observe general laboratory Safety Rules.

## Quantity and Container

50 ml. in a 60 ml. medicine dropping bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Ammonium molybdate, C.P.<br>$(NH_4)_6Mo_7O_{24}.4H_2O$, C.P. | 10 g. | 0.5 g. |

## Preparation

1. Transfer 0.5 g. of ammonium molybdate into a 50 ml. volumetric flask.
2. Add approximately 40 ml. of distilled water and dissolve the salt.
3. Dilute to volume with distilled water.
4. Transfer the solution into a 60 ml. medicine dropping bottle.

[redacted]

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H. W. Laboratory Manual
Laboratory Solutions

Code: SA-12a
Reissued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

# AMMONIUM HYDROXIDE SOLUTIONS

## Safety Precautions

1. Observe general laboratory Safety Rules.

2. Observe the rules for handling corrosive chemicals, particularly those pertaining to concentrated ammonium hydroxide.

## Reagents

### For Preparation

Ammonium hydroxide, C.P.
$NH_4OH$, 28% $NH_3$

## Preparation

1. Calculate the volume of concentrated ammonium hydroxide required to make the desired concentration as follows:

   a. For normality or molarity.

   ml. of concentrated $NH_4OH = 67.7\ (A \times B)$

   where A = desired normality or molarity.
   B = desired total volume in liters.

   Example: $A = 6\underline{N}$ or $6M$ and $B = 0.5$

   thus $67.7(6 \times 0.5) = 203$ ml. of concentrated $NH_4OH$

   b. For percentage of $NH_3$.

   ml. of concentrated $NH_4OH = (\frac{A}{28})B$

   where A = desired percentage of $NH_3$
   B = desired total volume in ml.

   Example: $A = 10\%$ and $B = 250$.

   thus $(\frac{10}{28})250 = 90$ ml. of concentrated $NH_4OH$

[redacted]

c. For Parts.

ml. of concentrated $NH_4OH = A(\frac{a}{b})$

where A = desired total volume in ml.
a = desired parts of hydroxide.
b = desired parts of hydroxide <u>plus</u> desired parts of water.

Example: For 1:2 where A = 1000, then a = 1 and b = 3.

thus $1000(\frac{1}{3}) = 333$ ml. of concentrated $NH_4OH$

2. Using a graduated cylinder, slowly add the required amount of concentrated ammonium hydroxide into a volumetric flask of the proper size which contains at least 50% of the total volume of distilled water. This work must be done in a well-ventilated hood.

3. Mix well and dilute to volume with distilled water.

4. After the solution is thoroughly mixed, transfer it into a dry, glass-stoppered Pyrex bottle of the proper size.

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SA-14b

Reissued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

## AMMONIUM CHLORIDE
(0.38%)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

"Standard Methods of the Examination of Water and Sewage", Eighth Edition, American Public Health Association, New York, 42 (1936).

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Ammonium chloride, C.P.<br>$NH_4Cl$, C.P. | 3.818 g. | 3.818 g. |
| 2. Distilled water, ammonia-free<br>(Solutions Code: SW-1c) | | |

### Preparation

1. Obtain the tare weight of a 100 ml. beaker.

2. With the tared beaker on the balance pan, add ammonium chloride until a total of 3.818 g. have been transferred into the beaker.

3. Add approximately 60 ml. of ammonia-free distilled water and dissolve the salt.

4. Transfer the solution quantitatively to a 1 liter volumetric flask. Rinse the beaker at least three times with ammonia-free distilled water, adding the rinsings to the flask without loss.

[redacted]

5. Dilute to volume with ammonia-free distilled water, and mix well.

6. Transfer the solution into a 1 liter glass-stoppered Pyrex bottle for storage.

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

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|---|---|
| Code: | SA-14c |
| Reissued: | August, 1948 |
| Status: | Standard |
| By: | D.W.T-L.F.W. |

## AMMONIUM CHLORIDE

(1 ml. = .01 mg. of nitrogen)
(1 ml. = .01288 mg. of $NH_4$ )

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

"Standard Methods for the Examination of Water and Sewage", Eighth Edition, American Public Health Association, New York, 42 (1936).

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Ammonium chloride, 0.38% (Solutions Code: SA-14b) | 10 ml. | 10 ml. |
| 2. Distilled water, ammonia-free (Solutions Code: SW-1c) | | |

### Preparation

1. Using a pipet, deliver 10 ml. of 0.38% ammonium chloride into a 1 liter volumetric flask.
2. Dilute to volume with ammonia-free distilled water, and mix well.
3. Transfer the solution into a 1 liter glass-stoppered Pyrex bottle for storage.

### Remarks

1. This solution should not be used after 2 weeks and should be kept away from light.

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Code: SA-15b

H. W. Laboratory Manual
Laboratory Solutions

Reissued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

## AMMONIUM NITRATE
## (4M in 5N $HNO_3$)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules for handling corrosive chemicals, particularly those pertaining to strong acids.

### Quantity and Container

100 ml. in a 125 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Ammonium nitrate, C.P.<br>$NH_4NO_3$, C.P. | 320 g. | 32 g. |
| 2. Nitric acid, C.P.<br>$HNO_3$, 70% | 375 ml. | 37.5 ml. |

### Preparation

1. Place about 50 ml. distilled water in a 250 ml. beaker.
2. Dissolve 37.5 ml. concentrated nitric acid in the water, mixing thoroughly.
3. Dissolve 32 g. ammonium nitrate in the nitric acid solution, heating if necessary.
4. Transfer the solution into a 100 ml. volumetric flask, and dilute to volume with distilled water.
5. Transfer the solution into a 125 ml. glass-stoppered Pyrex bottle for storage (Remark 1).

### Remark

1. If the solution crystallizes on cooling, store in an oven at 50 to 60°C.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SA-15c

Issued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

## AMMONIUM NITRATE
## (5M in 6.25N $HNO_3$)

### Safety Precautions

1. Observe general laboratory Safety Rules.

2. Observe the rules pertaining to the handling of concentrated nitric acid.

### Quantity and Container

100 ml. in a 125 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Ammonium nitrate, C.P.<br>$NH_4NO_3$, C.P. | 400 g. | 40 g. |
| 2. Nitric acid, C.P. conc.<br>$HNO_3$, C.P. conc. | 390 ml. | 39 ml. |

### Preparation

1. Dissolve 39 ml. of concentrated nitric acid in approximately 40 ml. of distilled water contained in a 250 ml. beaker. Mix well.

2. Weigh out 40 g. of ammonium nitrate, and dissolve in the nitric acid solution, heating if necessary.

3. Transfer the solution into a 100 ml. volumetric flask, and dilute to volume with distilled water.

4. Transfer the solution into a 100 ml. glass-stoppered Pyrex bottle for storage (Remark 1).

### Remarks

1. In case the solution crystallizes out on cooling, store in an oven at 50 to 60°C.

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|---|---|
| Code: | SA-16a |
| Reissued: | August, 1948 |
| Status: | Standard |
| By: | D.W.T.-L.F.W. |

H. W. Laboratory Manual
Laboratory Solutions

## AMMONIUM OXALATE
(4%)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

1. Griffin, R. C., "Technical Methods of Analysis", McGraw-Hill, 2 (1927).
2. Hodgman, C. D., "Handbook of Chemistry and Physics" 27th. Edition, Chemical Rubber Publishing Co., 1268 (1943).

### Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Ammonium oxalate, C.P.<br>$(NH_4)_2C_2O_4 \cdot H_2O$, C.P. | 40 g. | 20 g. |

### Preparation

1. Transfer the 20 g. of ammonium oxalate into a 500 ml. volumetric flask.
2. Add about 400 ml. of distilled water and dissolve the salt.
3. Dilute to volume with distilled water, and mix well.
4. Transfer the solution into a 500 ml. glass-stoppered Pyrex bottle for storage.

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| H. W. Laboratory Manual<br>Laboratory Solutions | Code: SA-16b |
|---|---|
| | Reissued: August, 1948 |
| | Status: Standard |
| | By: D.W.T.-L.F.W. |

# AMMONIUM OXALATE
## (Saturated)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Ammonium oxalate, C.P.<br>$(NH_4)_2C_2O_4.H_2O$, C.P. | 50 g. | 25 g. |

### Preparation

1. Transfer the 25 g. of ammonium oxalate into a 500 ml. volumetric flask.
2. Add about 400 ml. of distilled water and dissolve the salt.
3. Dilute to volume with distilled water, and mix well.
4. Transfer the solution into a 500 ml. glass-stoppered Pyrex bottle for storage.

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|---|---|
| Code: | SB-1b |
| Reissued: | August, 1948 |
| Status: | Standard |
| By: | D.W.T.-L.F.W. |

H. W. Laboratory Manual
Laboratory Solutions

## BARIUM CHLORIDE
## (0.025N)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

A. P. H. A. "Standard Methods for the Examination of Water and Sewage", 108 (1936).

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Barium chloride, C.P.<br>$BaCl_2$, C.P. | 3.1 g. | 3.1 g. |

For Standardization

Sulfuric acid, 5% (Solutions Code: SS-23a)

### Preparation

1. Dissolve 3.1 g. of barium chloride in a small amount of water contained in a 1 liter volumetric flask.

2. Dilute to mark with distilled water, and mix well.

3. Transfer the solution into a 1 liter glass-stoppered Pyrex bottle for storage.

### Standardization

1. Pipet a 50 ml. sample of barium chloride solution into a 250 ml. beaker.

2. Heat to boiling and add an excess of 5% sulfuric acid.

[redacted]

3. Cover with a watch glass and digest on a steam bath until the precipitate has completely settled.

4. Filter by decantation, through an ignited Gooch crucible, (Apparatus Code: AC-18a).

5. Wash the precipitate, by decantation, with three 10 ml. portions of hot acidified water and three portions of hot water.

6. Dry, ignite at about 900°C to constant weight.

7. Calculate the normality of the barium chloride as follows:

$$\underline{N} \text{ of } BaCl_2 = \frac{\text{wt. } BaSO_4 \times 1000}{50 \times 116.71}$$

8. Adjust the normality of the solution as follows:

$$\text{Total volume required} = \frac{\text{Actual volume} \times \text{actual normality}}{\text{desired normality}}$$

9. Dilute the solution to the required volume as calculated above. This dilution must be made accurately as the solution should be $0.025\underline{N} \pm 1.0\%$.

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Laboratory Solutions

Code: SB-4b
Reissued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

# BENZIDINE DIHYDROCHLORIDE
(3%)

## Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe the safety rules pertaining to the handling of strong acids.

## Quantity and Container

1000 ml. in a 1 liter brown Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Benzidine dihydrochloride, $C_{12}H_{12}N_2.2HCl$, E.K. #33 | 30 g. | 30 g. |
| 2. Hydrochloric acid HCl, 36% | 50 ml. | 50 ml. |

## Preparation

1. Weigh 30 g. of benzidine dihydrochloride into a 100 ml. beaker.
2. Add 50 ml. of concentrated hydrochloric acid to the contents of the beaker and mix to a paste.
3. Rinse the paste into a 1 liter volumetric flask with distilled water. Rinse the beaker thoroughly with distilled water, adding the rinsings to the volumetric flask.
4. Dilute the flask to volume with distilled water and mix thoroughly.
5. Filter the solution through a Whatman No. 5 filter paper into a brown Pyrex bottle for storage.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SB-5a
Reissued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

# BENZIDINE
(About 2%)

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

A. P. H. A. "Standard Methods for the Examination of Water and Sewage", 106, (1936).

## Quantity and Container

500 ml. in 500 ml. glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Benzidine hydrochloride, C.P. $C_{12}H_{12}N_2 . 2HCl$ | 22.4 g. | 11.2 g. |
| 2. Hydrochloric acid, 10% HCl (Solutions Code: SH-25a) | 100 ml. | 50 ml. |

## Preparation

1. Dissolve the weighed portion of reagent in 450 ml. of distilled water.
2. Add 50 ml. of 10% hydrochloric acid.
3. Shake and mix well and store in a 500 ml. glass-stoppered Pyrex bottle.

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Code: SB-7c

H. W. Laboratory Manual
Laboratory Solutions

Reissued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

## BISMUTH NITRATE
(10 mg. $Bi^{+3}$/ml.)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to the handling of concentrated acids.

### Literature References

Coryell, C.D., Hume, D.N., Ballou, N.E. and Glendenin, L., Clinton Secret Report, CL-CDC-4 or 5211 (March 15, 1944).

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Bismuth nitrate, C.P.<br>$Bi(NO_3)_3 \cdot 5H_2O$, C.P. | 23.2 g. | 23.2 g. |
| 2. Nitric acid<br>$HNO_3$, 70% | 63.0 ml. | 63.0 ml. |

### Preparation

1. Transfer the bismuth nitrate into a 1 liter volumetric flask.
2. Add approximately 750 ml. of distilled water containing 63.0 ml. of concentrated nitric acid, and dissolve the salt.
3. Dilute to volume with distilled water.
4. Transfer the solution into a 1 liter glass-stoppered Pyrex bottle for storage.

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Laboratory Solutions

Code: SB-18a
Reissued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

# BROMINE WATER
## (Saturated)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Bromine is a very corrosive liquid and great care should be excercised in handling it.
3. As many operations as possible should be carried out in the hood with the window lowered.
4. If bromine is received in the laboratory in an ampoule it is opened as follows:
    a. Cool the ampoule of bromine to about 10°C.
    b. Score the neck of the ampoule, about one inch from the top, with a triangular file.
    c. Cover the ampoule with a cloth and break it at the file mark by placing the thumbs together on the opposite side from the file mark and exerting a slight downward pressure.
    d. Drain the ampoule into a pint glass-stoppered bottle.

### Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Bromine, C.P.<br>$Br_2$, C.P. | 10 ml. | 5 ml. |

### Preparation

1. Pipet 5 ml. of bromine into 500 ml. of cold distilled water contained in a 500 ml. glass-stoppered Pyrex bottle. Hold

[redacted]

the tip of the pipet beneath the liquid surface of the water during delivery.

Remarks

1. A slight excess of liquid bromine must be visible in the bottom of the bottle in order that complete saturation may be assured.

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Laboratory Solutions

Code: SB-22b
Reissued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

## PHOSPHATE BUFFER
## (pH = 7.4)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

American Public Health Association "Standard Methods for the Examination of Water and Sewage", A.P.H.A., New York, 8th. Ed. 42 (1936).

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Potassium monobasic phosphate $KH_2PO_4$, C.P. | 14.300 g. | 14.300 g. |
| 2. Potassium diobasic phosphate $K_2HPO_4$, C.P. | 90.150 g. | 90.150 g. |
| 3. Redistilled water (Solutions Code: SW-1a). | | |

### Preparation

1. Transfer 14.300 g. of potassium dibasic phosphate and 90.150 g. of potassium monobasic phosphate into a 1 liter volumetric flask.
2. Add approximately 900 ml. of redistilled water and dissolve the salt.
3. Dilute to volume with redistilled water, and mix well.
4. Transfer the solution into a 1 liter glass-stoppered Pyrex bottle for storage.
5. Check the pH of the solution with the Beckman pH meter.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SB-22c
Reissued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

## PHOSPHATE BUFFER
## (pH = 7)

### Safety Precautions

Observe the general laboratory Safety Rules.

### Quantity and Container

560 ml. in a glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Sodium monobasic phosphate, C.P.<br>$NaH_2PO_4$, C.P. | 300 g. | 167 g. |
| 2. Sodium dibasic phosphate, C.P.<br>$Na_2HPO_4$, C.P. | 38 g. | 25 g. |

### Preparation

1. Prepare solution A by dissolving 167 g. of sodium monobasic phosphate in 500 ml. of distilled water.

2. Prepare solution B by dissolving 25 g. of sodium dibasic phosphate in 75 ml. of distilled water.

3. Pour 500 ml. of solution A into a dry, glass-stoppered Pyrex bottle.

4. Add 60 ml. of solution B to the contents of the bottle. Mix the resulting solution well.

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Laboratory Solutions

Code: SB-22d

Reissued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

## DILUTED PHOSPHATE BUFFER
(pH = 7.00)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Phosphate buffer, concentrated (Solutions Code: SB-22g) | 100 ml. | 100 ml. |

### Preparation

1. Pipet 100 ml. concentrated phosphate buffer into a 1 liter volumetric flask.
2. Dilute to volume with distilled water before transferring into the Pyrex bottle for storage.
3. Check the pH of the solution on a Beckman pH meter.

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|---|---|
| Code: | SB-22f |

H. W. Laboratory Manual
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Reissued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

## BUFFER, PHOSPHATE (DILUTED)
## (7.50 pH)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

20 liters in a 20 liter rubber-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Buffer, phosphate, concentrated (Solutions Code: SB-22g) | 100 ml. | 2000 ml. |
| 2. National Technical Laboratory Concentrated Buffer (7.00 pH) | | |

### Preparation

1. Using a 1 liter graduated cylinder, deliver 2 liters of the concentrated phosphate buffer into the 20 liter Pyrex bottle (with a graduation mark at 20 liters).

2. Add approximately 10 liters of distilled water, and mix well with an electric stirrer.

3. Dilute to 20 liters with distilled water; mix well.

4. Standardize a Beckman pH meter with the National Technical Laboratory concentrated buffer at room temperature.

5. Determine the value of the pH of the diluted buffer at the same temperature.

6. If the deviation from 7.50 is greater than 0.02 pH, follow the procedure given in step 5 of the Standardization in Solutions Code: SB-22g.

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| H. W. Laboratory Manual | Code: | SB-22g |
| Laboratory Solutions | Reissued: | August, 1948 |
| | Status: | Standard |
| | By: | D.W.T.-L.F.W. |

## BUFFER, PHOSPHATE (CONCENTRATED)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

Lang, N. A., "Handbook of Chemistry", Handbook Publishers, Inc., 1121 (1944).

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Sodium hydroxide pellets<br>NaOH, Reagent Grade | 16.56 g. | 16.56 g. |
| 2. Sodium monobasic phosphate | | |
| $NaH_2PO_4.H_2O$ | 69.0 g. | 69.0 g. |
| $NaH_2PO_4.2H_2O$ | 78.0 g. | 78.0 g. |

For Standardization

National Technical Laboratory
Concentrated Buffer (7.00 pH)

### Preparation

1. Obtain the weight of a 200 ml. beaker. With this beaker on the pan of a torsion balance, add sodium hydroxide pellets until a total of 16.56 g. have been transferred into the beaker (Remark 1).

2. Slowly add, with stirring and with cooling, about 100 ml. of distilled water. After the pellets have dissolved, transfer the solution into a 1 liter volumetric flask. Rinse the beaker three times with distilled water, delivering these washings into the volumetric flask without loss.

[redacted]

3. Similarly; weigh out 69.0 g. (or 78.9 g.) of sodium monobasic phosphate, dissolve it in distilled water, and transfer into the volumetric flask.

4. Add about 650 ml. of distilled water, and mix well.

5. Allow the solution to cool. Then dilute to 1 liter with distilled water; mix well and transfer to a dry, 1 liter glass-stoppered Pyrex bottle for storage.

Standardization

1. Pipet 10 ml. of the concentrated phosphate buffer solution into a 100 ml. volumetric flask.

2. Dilute to 100 ml. with distilled water; mix well by shaking.

3. Standardize a Beckman pH meter with the National Technical Laboratory concentrated buffer at 25°C.

4. Determine the pH of the phosphate buffer at 25°C.

5. If the deviation from 7.50 is greater than 0.02 pH, proceed as follows:

   a. For a pH less than 7.50, the product of the difference multiplied by 10 is numerically equal to the number of grams of sodium hydroxide pellets that must be added.

   b. For a pH more than 7.50, the product of the difference multiplied by 75 is numerically equal to the number of grams of sodium monobasic phosphate that must be added.

   c. Mix the solution thoroughly after dissolving the additional reagents. Then repeat the standardization procedure.

6. Repeat steps 3, 4 and 5 having the temperature of the solutions at 20, 15, 10 and 5°C.

Remarks

1. Weigh the sodium hydroxide pellets as rapidly as possible, for the hydroxide is very hydroscopic.

2. Each new lot of reagents may cause variations in the final pH of the buffer.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SC-1a

Reissued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

## CALCIUM CHLORIDE, STANDARD
(1 mg. $CaCO_3$/ml.)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

A.P.H.S., "Standard Methods for the Examination of Water and Sewage", 59 (1936).

### Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| | Per Liter | Total |
|---|---|---|
| 1. Calcium carbonate, C.P.<br>$CaCO_3$, C.P. | 1.0 g. | 0.50 g. |
| 2. Hydrochloric acid, 3N<br>(Solutions Code: SH-25a) | | |
| 3. Ammonium hydroxide, 6N<br>(Solutions Code: SA-12a) | | |

### Preparation

1. Dissolve the accurately weighed portion of carbonate in about 5 ml. 3N hydrochloric acid, being careful to avoid splattering.
2. Wash down the dish with carbon dioxide-free, distilled water.
3. Neutralize with 6N ammonium hydroxide to a slight alkalinity using litmus as an indicator.
4. Transfer the solution into a 500 ml. volumetric flask and dilute to mark with carbon dioxide-free, distilled water.
5. After the solution is thoroughly mixed, transfer it into a dry 500 ml. glass-stoppered Pyrex bottle for storage.

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H. W. Laboratory Manual
Laboratory Solutions

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|---|---|
| Code: | SC-2a |
| Reissued: | August, 1948 |
| Status: | Standard |
| By: | D.W.T.-L.F.W. |

# CADMIUM CHLORIDE (AMMONICAL)
(2%)

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

Furman, N. H., "Scott's Standard Methods of Chemical Analysis", D. Van Nostrand Co., Inc., 1, 619 (1939).

## Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Cadmium chloride, C.P.<br>$CdCl_2 \cdot 2H_2O$, C.P. | 22 g. | 22 g. |
| 2. Ammonium hydroxide<br>$NH_4OH$, 28% $NH_3$ | 550 ml. | 550 ml. |

## Preparation

1. Weigh into a 500 ml. tared beaker about 22 g. of cadmium chloride.
2. Add 250 ml. of distilled water to dissolve the cadmium chloride.
3. Transfer into a 1 liter volumetric flask.
4. Measure in a graduate, 550 ml. of concentrated ammonium hydroxide and add it to the flask.
5. Dilute the solution to 1000 ml. with distilled water.
6. Transfer the solution into a 1 liter glass-stoppered Pyrex bottle for storage.

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Remarks

1. This solution is of such strength that 50 ml. will precipitate approximately 0.175 g. of sulfur evolved as $H_2S$, which is equivalent to about 3.5% sulfur on a 5 g. sample.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SC-3a
Reissued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

## CALCIUM SULFATE
(Saturated)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Calcium sulfate, anhydrite $CaSO_4$, C.P. | 5.0 g. | 2.5 g. |

### Preparations

1. Place the weighed portion of the salt into a 1 liter bottle.
2. Add 500 ml. of distilled water.
3. Allow to stand, with frequent shaking for 24 hours.
4. Filter off the clear supernatant and store in a 500 ml. bottle.

### Remarks

1. Calcium sulfate is more soluble in cold water than in hot. The solution should be prepared at normal room temperature to insure a saturated solution.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SC-5b

Reissued: August, 1948
Status: Standard
By: D.W.T.-L.F.W.

# CESIUM CHLORIDE
(10 mg. Cs/ml.)

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

Coryell, C. D., Hume, D. N., Ballou, N. E. and Glendenin, L., Clinton Secret Report, CL-CDC-4 or 5211 (March 15, 1944).

## Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Cesium chloride, C.P.<br>CsCl, C.P. | 12.7 g. | 12.7 g. |

## Preparation

1. Transfer 12.7 g. of cesium chloride into a 1 liter volumetric flask.

2. Add approximately 900 ml. of distilled water, and dissolve the salt.

3. Dilute to volume with distilled water, and mix well.

4. Transfer the solution into a 1 liter glass-stoppered Pyrex bottle for storage.

[redacted]

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H. W. Laboratory Manual
Laboratory Solutions

Code: SC-7c
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Status: Standard
By: L.F.W.

## CERIUM AMMONIUM NITRATE, STANDARDIZED
(10 mg. $Ce^{++4}$/ml.)

### Safety Precautions

1. Observe general laboratory Safety Rules.

2. Observe the rules for handling ether.

### Literature References

Coryell, C.D., Hume, D.N., Ballou, N.E. and Glendenin, L., Clinton Secret Report, CL-CDC-4 or 5211 (March 15, 1944).

### Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Cerium ammonium nitrate<br>$Ce(NH_4)_2(NO_3)_6$ | 39.2 g. | 19.6 g. |

For Standardization

1. Ethanol, C.P.
   $C_2H_5OH$, 95%

2. Ether, anhydrous
   $(C_2H_5)_2O$, C.P.

3. Oxalic acid, 10% (Solutions Code: SO-24b)

### Special Apparatus

Crucible, sintered glass (fine) (Apparatus Code: AC-18c)

### Preparation

1. Transfer 19.6 g. of ceric ammonium nitrate into a 500 ml. volumetric flask.

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2. Add approximately 400 ml. of distilled water, and dissolve the salt.

3. Dilute to volume with distilled water, and mix well.

4. Transfer the solution into a 500 ml. glass-stoppered Pyrex bottle for storage.

Standardization

1. Prepare two sintered glass crucibles in the following manner:

    a. Wash with 5 ml. of water.

    b. Wash three times with 5 ml. portions of 95% ethanol.

    c. Wash three times with 5 ml. portions of anhydrous ether.

    d. Place the crucibles in a vacuum desiccator (containing no desiccant).

    e. Evacuate for two minutes and open the desiccator to the air.

    f. Repeat step (e) twice.

    g. Weigh the crucibles accurately.

    h. Replace the crucibles in the desiccator until ready for use.

2. Pipet duplicate 5 ml. portions of ceric ammonium nitrate into 150 ml. beakers.

3. Add 20 ml. of water and heat to boiling.

4. Add 15 ml. of 10% oxalic acid with stirring.

5. Cool in an ice bath for exactly 10 minutes.

6. Filter through the sintered glass crucibles.

7. Wash the precipitate by following the procedure outlined for the crucibles alone in step (1), (a) through (c). The portions of water and alcohol washes should be poured first into the beakers and from there on to the filter.

8. Dry and weigh the crucibles and precipitates by exactly the same procedure used for the crucible alone in step (1), (d) through (g).

## 9. CALCULATIONS

where x = Weight of the crucible and precipitate in g.

y = Weight of the crucible in g.

$$\frac{1000\,(x-y)}{5} = \text{mg. Ce}^{+4}/\text{ml.}$$

NOTE: Duplicate results should agree within 1% and the average value should be recorded as the final result.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SC-9a
Reissued: August, 1948
Status: Standard
By: L.F.W.

## CITRIC ACID
(10%)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

250 ml. in a 250 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Citric acid, C.P.<br>$C_6H_8O_7$, C.P. | 100 g. | 25 g. |

### Preparation

1. Transfer 25 g. of citric acid into a 250 ml. volumetric flask.
2. Add approximately 200 ml. of distilled water, and dissolve the acid.
3. Dilute to volume with distilled water, and mix well.
4. Transfer the solution into a 250 ml. glass-stoppered Pyrex bottle for storage.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

| | |
|---|---|
| Code: | SC-10a |
| Reissued: | August, 1948 |
| Status: | Standard |
| By: | L.F.W. |

# CHROMIUM STANDARD
(0.100 g. Cr/l.)

## Safety Precautions

Observe general laboratory Safety Rules.

## Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Potassium dichromate $K_2Cr_2O_7$, Reagent, recrystallized from water. | 0.2828 g. | 0.2828 g. |

## Preparation

1. Powder 1-2 g. of the recrystallized potassium dichromate in a mortar.
2. Dry the ground material at 105-110°C. for several hours. Allow to cool in a desiccator.
3. Obtain the tare weight of a 100 ml. beaker.
4. With the tared beaker on the balance pan, add potassium dichromate until a total of 0.2828 g. have been transferred into the beaker.
5. Add approximately 60 ml. of distilled water, and dissolve the salt.
6. Transfer the solution into a 1 liter volumetric flask. Rinse the beaker at least 3 times with distilled water adding the rinsings into the beaker.
7. Dilute to volume with distilled water, and mix well.
8. Transfer the solution into a 1 liter glass-stoppered Pyrex bottle for storage.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SC-10b
Reissued: August, 1948
Status: Standard
By: L.F.W.

## CHROMIUM STANDARD
(50 mg. $Na_2Cr_2O_7.2H_2O$/1.)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Potassium dichromate<br>$K_2Cr_2O_7$, primary standard | 0.9871 g. | 0.9871 g. |

### Preparation

1. Dry 1-2 g. of powdered primary standard potassium dichromate at 110°C. for at least 6 hours. Allow the dried salt to cool in a suitable desiccator.

2. Obtain the tare weight of a 100 ml. beaker.

3. With the tared beaker of the balance pan, add potassium dichromate until a total of 0.9871 g. have been transferred into the beaker.

4. Add approximately 60 ml. of distilled water, and dissolved the salt.

5. Transfer the solution into a 1 liter volumetric flask. Rinse the beaker at least three times with distilled water, adding the rinsings into the beaker.

6. Dilute to volume with distilled water, and mix well.

7. Transfer the solution into a 1 liter glass-stoppered Pyrex bottle for storage. This is the stock solution.

8. Pipet 50 ml. of the stock solution into a 1 liter volumetric flask.

[redacted]

9. Dilute to volume with distilled water, and mix well.

10. Transfer the diluted solution into a 1 liter glass-stoppered Pyrex bottle for storage.

Remarks

1. The stock solution contains dichromate ion equivalent to 1000 p.p.m. of $Na_2Cr_2O_7.2H_2O$.

2. The diluted solution contains dichromate ion equivalent to 50 p.p.m. of $Na_2Cr_2O_7.2H_2O$.

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Laboratory Solutions

Code: SC-15a

Reissued: August, 1948
Status: Standard
By: L.F.W.

## PLATINUM-COBALT COLOR STANDARD
(Color = 500)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe the rules for handling corrosive chemicals, particularly those pertaining to concentrated hydrochloric acid.

### Literature References

Am. Public Health Assoc., "Standard Methods for the Examination of Water and Sewage", Am. Public Health Assoc., 13 (1936).

### Quantity and Container

100 ml. in a 100 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Potassium chloro platinate<br>$K_2PtCl_6$, C.P. | 1.245 g.<br>(0.5 g. Pt) | 0.1245 g.<br>(0.05 g. Pt) |
| 2. Cobalt chloride, C.P.<br>$CoCl_2.6H_2O$, C.P. | 1.0 g.<br>(0.248 g. Co) | 0.10 g.<br>(0.0248 g. Co) |
| 3. Hydrochloric acid<br>$HCl$, 36% | 100 ml. | 10 ml. |

### Preparation

1. Obtain the tare weight of a 100 ml. beaker.
2. With the tared beaker on the balance pan, add potassium chloro platinate until a total of 0.1245 g. has been transferred into the beaker.
3. Continuing with the tared beaker on the balance pan, add cobalt chloride until a total of 0.10 g. has been transferred into the beaker.

[redacted]

4. Add approximately 60 ml. of distilled water, and dissolve the salts.

5. Quantitatively transfer the solution into a 100 ml. volumetric flask. Rinse the beaker at least 3 times with distilled water, adding the rinsings into the flask.

6. Dilute to volume with distilled water, and mix well.

7. Transfer the solution into a dry 100 ml. glass-stoppered Pyrex bottle for storage.

Remarks

1. Solutions of other color values can be prepared by diluting a specific volume of this 500 standard.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SC-16b

Reissued: September, 1948
Status: Standard
By: L.F.W.

## COPPER STANDARD
## (100 p.p.m. Cu)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Copper sulfate, C.P.<br>$CuSO_4 \cdot 5H_2O$, C.P. | 0.3930 g. | 0.3930 g. |

### Preparation

1. Obtain the tare weight of a 100 ml. beaker.
2. With the tared beaker on a balance pan, add copper sulfate crystals until a total of 0.3930 g. has been transferred into the beaker.
3. Add approximately 60 ml. of distilled water, and dissolve the salt.
4. Transfer the solution into a 1 liter volumetric flask. Rinse the beaker at least 3 times with distilled water, adding the rinsings into the flask.
5. Dilute to volume with distilled water, and mix well.
6. Transfer the solution into a dry 1 liter glass-stoppered Pyrex bottle for storage.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SC-19a
Reissued: September, 1948
Status: Standard
By: L.F.W.

## CLEANING SOLUTION
(Chromic Acid)

### Safety Precautions

1. Observe general laboratory Safety Rules.

2. Observe the rules for handling concentrated acids, i.e., wear a rubber apron, rubber gloves, safety glasses, and a face shield.

### Quantity and Container

7-1/2 liters in a 2 gallon porcelain crock and cover.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Sodium dichromato, Commercial<br>$Na_2Cr_2O_7.2H_2O$, | 40 g. | 305 g. |
| 2. Sulfuric acid, C.P.<br>$H_2SO_4$, C.P. | 1 l. | 7-1/2 l. |
| 3. Distilled water<br>$H_2O$ | 18 ml. | 135 ml. |

### Preparation

1. Add approximately 135 ml. of distilled water and the 305 g. of sodium dichromate into a 600 ml. beaker.

2. Place the beaker on a hot plate and boil the solution until all the sodium dichromate has dissolved.

3. Pour the hot dichromate solution into a 2 gallon porcelain crock, and add cautiously, with constant stirring, three (2-1/2 liter) bottles of concentrated sulfuric acid.

4. Continue stirring until the solution is well mixed.

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Laboratory Solutions

Code: SC-23a
Reissued: September, 1948
Status: Standard
By: L.F.W.

# CUPFERRON
(6%)

## Safety Precautions

Observe general laboratory Safety Rules.

## Quantity and Container

50 ml. in a 60 ml. medicine dropping bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Ammonium salt of N-nitroso N-phenylhydroxylamine $C_6H_5N(ONH_4)NO$, C.P. | 60 g. | 3.0 g. |

## Preparation

1. Transfer 3.0 g. of cupferron (the ammonium salt of N-nitroso N-phenylhydroxylamine) into a 50 ml. volumetric flask.

2. Add approximately 25 ml. of distilled water, and dissolve the salt.

3. Dilute to volume with distilled water, and mix well.

4. Transfer the solution into a 60 ml. medicine dropping bottle for storage.

[redacted]

[redacted]

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Laboratory Solutions

Code: SD-9a

Reissued: September, 1948
Status: Standard
By: L.F.W.

# DIMETHYLGLYOXIME
## (1%)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. The alcohol solution is inflammable and must be kept away from open flames.

### Quantity and Container

250 ml. in a 250 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Dimethylglyoxime, C.P.<br>$(CH_3)_2C_2(NOH)_2$, C.P. | 10 g. | 2.5 g. |
| 2. Ethanol<br>$C_2H_5OH$, 95% | | |

### Preparation

1. Transfer the 2.5 g. of dimethylglyoxime into a 250 ml. volumetric flask.
2. Add approximately 200 ml. of ethanol, and dissolve the dimethylglyoxime.
3. Dilute to volume with ethanol, and mix well.
4. Transfer the solution into a 250 ml. glass-stoppered Pyrex bottle for storage.

[redacted]

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|---|---|
| Code: | SF-8a |
| Reissued: | November, 1948 |
| Status: | Standard |
| By: | W.J.M. |

H. W. Laboratory Manual
Laboratory Solutions

## FERROUS AMMONIUM SULFATE
## (0.01N)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to the handling of strong acids.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Ferrous ammonium sulfate, C.P. $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ | 4.0 g. | 4.0 g. |
| 2. Sulfuric acid, 0.1N (Solutions Code: SS-23a) | 1000 ml. | 1000 ml. |

For Standardization

1. Sulfuric acid, 6N (Solutions Code: SS-23a)
2. Sulfuric acid, silica free, 1N (Solutions Code: SS-23k)
3. Sodium oxalate, 0.0100N (Solutions Code: SS-11a)
4. Ceric sulfate, 0.01N (Solutions Code: SC-6b)
5. Ferroin indicator, 0.0025 M (Solutions Code: SF-7b)

### Preparation

1. Weigh out exactly 4.000 g. of ferrous ammonium sulfate, and transfer to a 250 ml. beaker.
2. Dissolve in 0.1N sulfuric acid, and transfer the solution into a 1 liter volumetric flask.

[redacted]

3. Rinse the beaker with several portions of 0.1N sulfuric acid, and add the rinsings to the volumetric flask.

4. Dilute the solution to the mark with 0.1N sulfuric acid, and after thorough mixing, transfer into a 1 liter glass-stoppered Pyrex bottle for storage.

Standardization

The following directions for the standardization of the ferrous ammonium sulfate solution are given for the use of ultra-micro equipment. For certain uses, it is desirable to standardize the solution with ordinary micro equipment, and certain adjustments of the amount of reagents used will have to be made.

A. Determination of the Ceric-Ferrous Equivalence Value

Run duplicate determinations until consecutive results within 0.4 λ are obtained.

1. Add 100 λ of 6 N sulfuric acid to a micro titration dish.

2. Add 50 λ of the 0.01 N ceric sulfate solution to the titration dish. Rinse the pipet used with 1 N sulfuric acid, and add the rinsings to the titration dish.

3. Dilute the solution to about 3/4 the capacity of the dish.

4. Add 5 λ of the 0.0025 M Ferroin indicator to the contents of the titration dish.

5. Insert the stirrer thread into the solution and start the magnetic stirrer.

6. Fill the capillary buret with the ferrous ammonium sulfate solution and, after noting the zero reading, insert the buret tip into the solution in the titration dish.

7. Titrate the solution to the first appearance of a pink color.

8. Note the final buret reading and convert the number of cm. of ferrous ammonium sulfate solution used to microliters.

B. Standardization of the Ferrous Ammonium Sulfate Solution

Run duplicate determinations until consecutive checks within 0.0001 N are obtained.

1. Pipet 100 λ of the 6 N sulfuric acid into a micro titration dish.

2. Add 25 λ of the 0.0100 N sodium oxalate solution to the titration dish.

3. Add 50 λ of the ceric sulfate solution. Rinse the pipet used to make the addition with 1 N sulfuric acid, and add the rinsings to the titration dish.

4. Dilute the contents of the dish with distilled water until their total volume is about 3/4 that of the dish.

5. Insert the stirrer thread into the solution and start the magnetic stirrer.

6. Heat the solution to 60° C., and allow to cool, while stirring, for 3 minutes.

7. Add 5 λ of the 0.0025 M Ferroin indicator to the solution in the titration dish.

8. Fill the capillary buret with the ferrous ammonium sulfate solution to be standardized, and after noting the zero reading of the buret, insert the buret tip into the solution in the titration dish.

9. Titrate the solution to the first appearance of a pink color.

10. Note the final buret reading and convert the number of cm. of ferrous ammonium sulfate solution used into microliters.

Calculations

$$N_f = \frac{25 \times 0.01000}{A - B}$$

where $A$ = amount of ferrous ammonium sulfate solution (expressed as microliters) used in section A above.

$B$ = amount of ferrous ammonium sulfate solution (expressed as microliters) used in section B above.

$N_f$ = normality of ferrous ammonium sulfate solution

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SF-12a

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Status: Standard
By: W.J.M.

## FLUORIDE STANDARD
(1.000 mg. F/l.)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Sodium fluoride is a toxic chemical. Care should be used to avoid transmission into the body.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Sodium fluoride, C.P.<br>NaF, C.P. | 2.2103 g. | 2.2103 g. |
| 2. Water, redistilled<br>(Solution Code: SW-1c) | | |

### Preparation

1. Dry 4 g. of sodium fluoride in an oven at 110°C.
2. Weigh out 2.2103 g. of the dried and cooled salt, and transfer into a 1 liter volumetric flask.
3. Dissolve the salt with redistilled water, and dilute the solution to the mark with redistilled water.
4. Transfer the solution after thorough mixing, to a 1 liter glass-stoppered Pyrex bottle for storage.

### Standardization

This solution is prepared to contain exactly 1 mg.F/ml.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SG-12a

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Status: Standard
By: W.J.M.

## GLYCEROL SOLUTION
(33.3% by Volume)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Glycerol, White<br>$C_3H_8O_3$ | 333 ml. | 167 ml. |

### Preparation

1. Using a graduated cylinder deliver 167 ml. of glycerol into a 500 ml. volumetric flask and dilute to the mark.
2. Transfer the solution to a 500 ml. glass-stoppered Pyrex bottle for storage.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

| | |
|---|---|
| Code: | SG-23a |
| Reissued: | October, 1948 |
| Status: | Standard |
| By: | W.J.M. |

# GUM ARABIC
## (5%)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Prepartion | Per Liter | Total |
|---|---|---|
| 1. Gum arabic | 50 g. | 25 g. |
| 2. Salicylic acid, U.S.P. $C_7H_6O_3$ | 0.10 g. | 0.05 g. |

### Preparation

1. Dissolve 25 g. of gum arabic and 0.05 g. of Salicylic acid in distilled water in a 500 ml. volumetric flask.
2. Dilute to the mark.
3. Shake and mix well.
4. Transfer to a 500 ml. glass-stoppered Pyrex bottle for storage.

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Code: SH-25a

H. W. Laboratory Manual
Laboratory Solutions

Reissued: October, 1948
Status: Standard
By: W.J.M.

## HYDROCHLORIC ACID SOLUTIONS

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe the rules for handling corrosive chemicals, particularly those pertaining to concentrated hydrochloric acid.

### Reagents

#### For Preparation

Hydrochloric acid
$HCl$, 36%

#### For Standardization

1. Sodium hydroxide (Solutions Code: SS-8a)
2. Sodium carbonate, C.P.
   $Na_2CO_3$, C.P.
3. Methyl red indicator, 0.1% Alcoholic (Solutions Code: SM-6a)
4. Modified methyl red, 0.1% (Solutions Code: SM-6c)

### Special Apparatus

Semi-microburet, 10 ml. (Apparatus Code: AB-21a)

### Preparation

1. Calculate the volume of concentrated hydrochloric acid required to make the desired concentration as follows:

   a. For normality or molarity.

   ml. of 36% $HCl = 86.2(A \times B)$

   where $A$ = desired normality or molarity.
   $B$ = desired total volume in liters.

[redacted]

[redacted]

Example: $A = 0.33\underline{N}$ or $0.33M$ and $B = 1$.

thus $86.2(0.33 \times 1) = 28$ ml. of 36% HCl

b. For percentage of HCl.

ml. of 36% HCl $= (\frac{A}{36})B$

where A = desired percentage.
B = desired total volume in ml.

Example: A = 15% and B = 500.

thus $(\frac{15}{36})500 = 208$ ml. of 36% HCl

c. For parts.

ml. of 36% HCl $= A(\frac{a}{b})$

where A = desired total volume in ml.
a = desired parts of acid.
b = desired parts of acid <u>plus</u> desired parts of water.

Example: For 1:1 where A = 500, then a = 1 and b = 2.

thus $500(\frac{1}{2}) = 250$ ml. of 36% HCl.

2. By means of a graduated cylinder, slowly add the required amount of concentrated hydochloric acid into a volumetric flask of the proper size, which contains at least 50% of the total volume of distilled water. This work must be done in a well ventilated hood.

3. Mix well and dilute to the mark with distilled water.

4. After the solution is well mixed, store it in a dry, glass-stoppered Pyrex bottle of the proper size.

## Standardization

One of the methods given below will be suitable for standardizing hydrochloric acid solutions used on this project. It is important in accurate work to select a standardization using the same indicator as will be involved in the subsequent uses of the solution.

A. Against primary standard sodium carbonate.

1. Place the solution in a 10 ml. semi-microburet and thoroughly flush the system.

[redacted]

2. Dry about 1g. of the standard primary sodium carbonate to constant weight in an oven held between 135 and 150°C

3. Cool in a desiccator containing sticks of potassium hydroxide.

4. Obtain the tare weights of three 100 ml. beakers. Weigh accurately and transfer into these beakers 0.13 to 0.15 g. of the dried carbonate.

5. Add approximately 15 ml. of distilled water and dissolve the sodium carbonate.

6. Add 1 drop of 0.1% alcoholic methyl red indicator.

7. Titrate with the hydrochloric acid to the characteristic red end point. Add about 1 ml. excess of acid. Record this volume.

8. Cover the beakers and boil the solution for at least 5 minutes in order to completely expel the carbon dioxide. Do not evaporate to dryness.

9. Cool to room temperature and add 1 drop of 0.1% alcoholic methyl red indicator.

10. Back titrate with standard sodium hydroxide (Remark 1) to the first yellow tint, using split drops as the end point is approached. Record this volume.

11. Titrate a known amount of the standard sodium hydroxide with the hydrochloric acid, using 0.1% alcoholic methyl red indicator (Calculation 1.).

Calculations

1. ml. of HCl/ml. of NaOH = $\frac{\text{ml. of HCl}}{\text{ml. of NaOH}}$

2. ml. of HCl = A - (B x ml. of HCl/ml. of NaOH)

   where A = Volume of HCl from Step 7.
   B = Volume of NaOh from Step 10.

3. $\underline{N}$ of HCl = $\frac{\text{wt. of } Na_2CO_3}{0.053 \times \text{ml. of HCl}}$

   the $\underline{N}$ values obtained from the triplicate samples should not vary more than 0.0002.

B. Against standard sodium hydroxide with modified methyl red as indicator.

1. Pipet 10 ml. of the hydrochloric acid into a 250 ml. Erlenmeyer flask. Add 50 ml. of distilled water.

2. Add 1 drop of modified methyl red indicator. Addition of excess indicator tends to give a purple or black color at the end point. If this occurs repeat the determination, using less indicator.

3. Titrate the solution with standard sodium hydroxide (Remark 1) from a 10 ml. semi-microburet. The end point is reached when the solution goes from red to colorless or a slight grayish tinge. Addition of excess base turns the indicator green.

4. The normality of the sodium hydroxide should correspond to the normality of the hydrochloric acid being prepared.

Calculations

$$\underline{N} \text{ of HCl} = \frac{N_b \times B}{10}$$

where $N_b$ = Normality of the sodium hydroxide.

B = ml. of sodium hydroxide used.

Standardization should be repeated until 3 consecutive titrations give $\underline{N}$ values within 0.0002.

C. Gravimetric standardization using silver nitrate.

1. Pipet 10 ml. of the hydrochloric acid into a 250 ml. beaker.

2. Add 100 ml. of distilled water.

3. Calculate the volume of silver nitrate of any normality required to completely precipitate a given volume of hydrochloric acid of any normality as follows:

$$Vs = \frac{Va \times Na}{Ns}$$

where Vs = Volume of silver nitrate in ml.
Va = Volume of hydrochloric acid.
Na = Normality of hydrochloric acid.
Ns = Normality of silver nitrate.

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[redacted]

4. Using a graduated cylinder add the calculated amount of silver nitrate to the hydrochloric acid solution.

5. Add a 20% excess of the silver nitrate to insure complete precipitation.

6. Heat carefully to gentle boiling. Stir occasionally and continue heating with stirring until the silver chloride precipitate coagulates, leaving a nearly clear supernatant solution.

7. Allow the precipitate to settle for 5 minutes in a dark place.

8. Obtain the tare weight of a fine sintered-glass crucible.

9. Decant the clear liquid through the sintered-glass using gentle suction and catch the filtrate in a 250 ml. Erlenmeyer flask.

10. Quantitatively transfer the precipitate left in the beaker into the sintered-glass.

11. Rinse out the beaker with several portions of prepared solutions and add the washings to the sintered-glass. (Remark 1)

12. Test the filtrate with a few drops of silver nitrate.

13. If a white cloudiness appears repeat Steps 6,7,9,10, and 11 until the filtrate remains clear upon addition of silver nitrate.

Calculations

$$Na = \frac{Ws \times 1000}{143.34 \times Va}$$

where Na = Normality of the hydrochloric acid
Ws = Weight of the silver chloride precipitate
Va = Volume of the hydrochloric acid

Remarks

Owing to the slight solubility of silver chloride it has been recommended to wash first with water containing silver nitrate (0.05 g./l approximately) then with 1% solution of nitric acid and finally twice with redistilled water.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SH-26a

Reissued: October, 1948
Status: Standard
By: W.J.M.

## HYDROGEN PEROXIDE
## ($5 \times 10^{-4}$N)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to the handling of strong acids.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Hydrogen Peroxide, C.P.<br>$H_2O_2$, 30% | 0.028 g. | 0.028 g. |

For Standardization

1. Potassium Permanganate, 0.1N (Solution Code: SP-16a)
2. Sulfuric acid, 6N (Solution Code: SS-23a)

### Preparation

1. Pipet 2 ml. of the 30% peroxide into a 500 ml. volumetric flask and dilute to the mark with distilled water.
2. Shake and mix well.
3. This procedure gives a solution about 0.07N in peroxide.

### Standardization

1. Pipet 10 ml. of the approximately 0.07N peroxide into a 50 ml. beaker.
2. Add 10 ml. of water and 10 ml. of 6N $H_2SO_4$.

[redacted]

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3. Titrate with the standard $.1\underline{N}$ potassium permanganate to an end point that will hold for 30 seconds.

4. Calculate the normality of the peroxide by the following formula:

$$Np = \frac{Vm \times Nm}{Vp}$$

where Np = Normality of the hydrogen peroxide
Vm = Volume of the potassium permanganate
Nm = Normality of the potassium permanganate
Vp = Volume of the hydrogen peroxide

5. From this normality calculate the volume of hydrogen peroxide necessary to dilute to 1000 ml. to produce a solution of $5 \times 10^{-4}\underline{N}$ as follows:

$$\text{Volume hydrogen peroxide needed} = \frac{1000 \times 5 \times 10^{-4}}{Np} = \frac{5 \times 10^{-1}}{Np}$$

6. Using a Mohr pipet accurately measure the amount of hydrogen peroxide calculated and transfer it into a 1000 ml. volumetric flask.

7. Dilute to mark with water and mix well. This solution should be $5 \times 10^{-4}\underline{N}$ in hydrogen peroxide.

**Remarks**

This solution must be prepared and standardized each time it is to be used since it can not be stored without deteriorating.

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

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|---|---|
| Code: | SH-26c |
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| By: | W.J.M. |

# HYDROGEN PEROXIDE SOLUTION
(7.5% Superoxol)

## Safety Precautions

1. Observe general laboratory Safety Rules.

2. Strong hydrogen peroxide (30%) is a corrosive chemical and should not be brought in contact with the skin.

## Quantity and Container

1000 ml. in a liter glass-stoppered amber bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Hydrogen peroxide, C.P. $H_2O_2$, 30% | 250 ml. | 250 ml. |

## Preparation

1. Using a graduated cylinder transfer the 250 ml. of the 30% hydrogen peroxide solution into a 1 liter volumetric flask.

2. Dilute the solution to the mark with distilled water, and mix thoroughly.

3. Transfer the solution to a 1 liter glass-stoppered amber bottle for storage.

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[redacted]

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|---|---|
| Code: | SH-26a |
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| By: | W.J.M. |

H. W. Laboratory Manual
Laboratory Solutions

## HYDROGEN PEROXIDE
## (3%)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules for handling hydrogen peroxide.

### Quantity and Container

2000 ml. in a 2 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Hydrogen peroxide, C.P.<br>$H_2O_2$, 32% | 107 ml. | 214 ml. |

### Preparation

1. Using a graduated cylinder, measure 214 ml. of the hydrogen peroxide into a 3000 ml. beaker.
2. Add slowly and with constant stirring 1786 ml. of distilled water.
3. Transfer into a 2 liter glass-stoppered Pyrex bottle for storage.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SH-28a

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By: W.J.M.

## 8-HYDROXYQUINOLINE
(2% Alcoholic Solution)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

250 ml. in a 250 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. 8-Hydroxyquinoline, C.P.<br>$C_9H_7ON$, C.P. | 20 g. | 5 g. |
| 2. Ethyl Alcohol, C.P.<br>$C_2H_5OH$, 95% | 1000 ml. | 250 ml. |

### Preparation

1. Weigh 5.0 g. of the 8-Hydroxyquinoline on a torsion balance and transfer into a 250 ml. volumetric flask.
2. Dissolve in the ethyl alcohol and dilute to the mark with the ethyl alcohol.
3. Mix well and transfer into a 250 ml. glass-stoppered bottle for storage.

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| H. W. Laboratory Manual | Code: SI-14e |
| Laboratory Solutions | Reissued: November, 1948 |
| | Status: Standard |
| | By: W.J.M. |

# IODINE
## (0.2N Solution)

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

1. Furman, N. H., "Scott's Standard Methods of Analysis", I, 5th Edition, D. Van Nostrand Co., Inc., 1207 (1939).
2. Griffin, R. C., "Technical Methods of Analysis", McGraw-Hill Publishing Co., 10-12 (1927).

## Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Iodine, resublimed<br>$I_2$, resublimed | 25.4 g. | 25.4 g. |
| 2. Potassium iodide, C.P.<br>KI, C.P. | 40 g. | 40 g. |

### For Standardization

| | |
|---|---|
| 1. Arsenous acid, .1N | (Solutions Code: SA-18a) |
| 2. Starch indicator, stabilized 1.0% | (Solutions Code: SS-21a) |

## Special Apparatus

| | |
|---|---|
| Buret, semi-micro, 10 ml. | (Apparatus Code: AB-21a) |

## Preparation

1. Dissolve 40 g. of the potassium iodide in as little distilled water as possible in a 1 liter volumetric flask.

[redacted]

2. Add 25.4 g. of the iodine and dissolve in the potassium iodide solution.

3. Dilute to the mark with distilled water.

4. Mix well and transfer into a 1 liter glass-stoppered Pyrex bottle for storage.

Standardization

1. Deliver from a 10 ml. semi-micro buret, about 8 ml. of .1N arsenous acid into a 50 ml. beaker.

2. Add 1 ml. of the 1% starch indicator.

3. Using a 10 ml. semi-micro buret titrate with the iodine solution to a light blue end point.

4. Repeat Steps 1 through 3, using 9 ml. of arsenous acid.

Calculations

$$N_1 = \frac{N_2 \times V_2}{V_1}$$

where $N_1$ = Normality of the iodine

$N_2$ = Normality of the arsenous acid

$V_2$ = Volume of the arsenous acid

$V_1$ = Volume of the iodine

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| H. W. Laboratory Manual<br>Laboratory Solutions | Code: SI-15a<br>Reissued: November 1948<br>Status: Standard<br>By: W.J.M. |
|---|---|

## IODINE MONOCHLORIDE
(0.005 M)

### Safety Precautions

1. Observe general laboratory Safety Rules.

2. Observe rules pertaining to the handling of strong acids.

### Literature References

1. Smith, G. F., "Cerate Oxidimetry", G. F. Smith Chemical Co., 38 (1942).

2. Oesper, R. E., "Newer Methods of Volumetric Analysis", D. Van Nostrand Co., 30 (1938).

### Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Potassium iodide, C.P.<br>KI, C.P. | 0.558 g. | 0.279 g. |
| 2. Potassium iodate, C.P.<br>$KIO_3$, C.P. | 0.356 g. | 0.178 g. |
| 3. Hydrochloric acid, C.P.<br>HCl, 36% | 500 ml. | 250 ml. |
| 4. Chloroform, C.P.<br>$CH Cl_3$, C.P. | 10 ml. | 10 ml. |

### Preparation

1. Dissolve 0.279 g. of potassium iodide and 0.178 g. of potassium iodate in 250 ml. of distilled water contained in a 500 ml. volumetric flask.

2. Add immediately 250 ml. of the hydrochloric acid.

[redacted]

3. Adjust to equivalence of potassium iodide and potassium iodate by adding 10 ml. of chloroform.

4. Mix well and if the chloroform layer is discolored add dropwise a very dilute solution of the iodate until the color disappears sharply. Then add an equally dilute solution of the iodide until one drop restores the color. If the chloroform layer is colorless add dropwise a dilute solution of the iodide until the color appears.

5. Dilute to the mark with distilled water.

6. Mix well and transfer to a 500 ml. glass-stoppered Pyrex bottle for storage.

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SI-18c

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# FERRIC NITRATE
## (10 mg. $Fe^{+3}$/ml.)

## Safety Precautions

1. Observe general laboratory Safety Rules.

2. Observe rules pertaining to the handling of strong acids.

## Literature References

Coryell, C. D., Hume, D. N., Ballou, N. E., and Glendenin, L., Clinton Secret Report, CL-CDC-4 or 5211 (March 15, 1944).

## Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Ferric nitrate, C.P.<br>$Fe(NO_3)_3 \cdot 9H_2O$, C.P. | 72 g. | 72 g. |
| 2. Nitric acid, C.P.<br>$HNO_3$, 70% | 1 ml. | 1 ml. |

## Preparation

1. Dissolve the 72 g. of the ferric nitrate, weighed on a torsion balance, in distilled water in a 1 liter volumetric flask containing 1 ml. of concentrated nitric acid.

2. Dilute to the mark with distilled water.

3. Mix well and transfer into a 1 liter glass-stoppered Pyrex bottle for storage.

[redacted]

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Laboratory Solutions

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|---|---|
| Code: | SM-1b |
| Reissued: | November, 1948 |
| Status: | Standard |
| By: | W.J.M. |

## MAGNESIUM URANYL ACETATE

### Safety Precautions

1. Observe general laboratory Safety Rules.

2. Acetic acid produces burns on the skin; avoid contact.

### Literature References

Furman, N. H., "Scott's Standard Methods of Chemical Analysis", 5th Ed., I, D. Van Nostrand Co., Inc., New York, 880 (1939).

### Quantity and Container

2000 ml. in a 2 liter glass-stoppered amber-colored bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Glacial acetate acid, C.P. | 60 ml. | 120 ml. |
| 2. Magnesium acetate, C.P.<br>$Mg(C_2H_3O_2)_2 \cdot 4H_2O$, C.P. | 300 g. | 600 g. |
| 3. Uranium acetate, C.P.<br>$UO_2(C_2H_3O_2)_2 \cdot 2H_2O$, C.P. | 45 g. | 90 g. |

### Preparation

#### Solution A

1. Weigh 90 g. of uranium acetate and transfer into a 2 liter Erlenmeyer flask.

2. Using a graduated cylinder measure 60 ml. of glacial acetic acid and add to the contents of the flask.

3. Add sufficient distilled water to the contents of the flask to make 1 liter (Remark 1).

4. Warm the flask and its contents to 70° C and stir until solution is complete.

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Solution B

1. Weigh 600 g. of magnesium acetate and transfer into a 2 liter Erlenmeyer flask.

2. Using a graduated cylinder measure 60 ml. of glacial acetic acid and add to the contents of the flask.

3. Add sufficient distilled water to the contents of the flask to make 1 liter (Remark 1).

4. Warm the flask and its contents to 70° C and stir until solution is complete.

5. Mix together solutions A and B at 70° C, and then cool to 20° C.

6. Allow the solution to stand at this temperature for 2 hours or longer.

7. Filter the solution through filter paper into a glass-stoppered 2 liter amber-colored bottle (Remarks 2 and 3).

Remarks

1. Prior to preparing the solution, mark a 2 liter Erlenmeyer flask at the liter level by measuring a volume of water and adding to the flask.

2. Preserve this reagent away from direct sunlight.

3. If further precipitation occurs, the solution should be filtered again before it is used.

[redacted]

[redacted]

| | |
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| H. W. Laboratory Manual<br>Laboratory Solutions | Code: SM-3a<br>Reissued: November 1948<br>Status: Standard<br>By: W.J.M. |

## MANGANOUS SULFATE
(1 ml. = .050 mg. Mn)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

A.P.H.A., "Standard Methods for the Examination of Water and Sewage," A.P.H.A. and A.W.A.A., 83 (1936).

### Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Potassium permanganate, C.P.<br>$KMnO_4$, C.P. | 0.1438 g. | 0.0719 g. |
| 2. Sodium bisulfite, C.P.<br>$NaHSO_3$, C.P. | 0.4 g. | 0.2 g. |
| 3. Sulfuric acid, 1N<br>(Solutions Code: SS-23a) | 4-6 ml. | 2-3 ml. |

### Preparation

1. Add 2 - 3 ml. of 1N sulfuric acid to about 100 ml. of distilled water.
2. Dissolve the accurately weighed 0.0719 g. of the potassium permanganate in the acid solution.
3. Add the .2 g. of the sodium bisulfite to reduce the permanganate.
4. Boil to remove the excess sulfur dioxide and transfer into a 500 ml. volumetric flask.
5. Dilute to the mark with distilled water and mix well.

[redacted]

6. Transfer into a 500 ml. glass-stoppered Pyrex bottle for storage.

## Standardization

The procedure is designed to give a solution containing 50 micrograms of manganese per ml.

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SM-4a

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Status: Standard
By: W.J.M.

# MAGNESIUM STANDARD

(1 ml. = 100 micrograms Mg/ml.)

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

Snell, F. D., and Snell, C. T., "Colorimetric Methods of Analysis", I, 2nd Edition, D. Van Nostrand Co., Inc., 477 (1943).

## Quantity and Container

1000 ml. in four 250 ml. glass-stoppered Pyrex bottles.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Magnesium sulfate, hydrated, C.P.<br>$MgSO_4 \cdot H_2O$, C.P. | 1.014 g. | 1.014 g. |

## Preparation

1. Dissolve the 1.014 g. of the magnesium sulfate in distilled water in a 1 liter volumetric flask.

2. Dilute to the mark and mix well.

3. Transfer into four 250 ml. glass-stoppered Pyrex bottles.

## Standardization

This preparation is designed to give a solution containing 100 micrograms magnesium per ml.

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Laboratory Solutions

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|---|---|
| Code: | SM-5a |
| Reissued: | November, 1948 |
| Status: | Standard |
| By: | W.J.M. |

## METHYL ORANGE
(0.1%)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

Furman, N. H., "Scott's Standard Methods of Chemical Analysis," II, 5th. Edition, D. Van Nostrand Co., Inc., 2192 (1939)

### Quantity and Container

250 ml. in a 250 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Methyl Orange, commercial dye | 1.0 g. | 0.25 g. |

### Preparation

1. Dissolve the .25 g. portion of methyl orange in 250 ml. of water contained in a 250 ml. volumetric flask.

2. Shake and mix well.

3. Transfer into a 250 ml. glass-stoppered Pyrex bottle for storage.

[redacted]

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H. W. Laboratory Manual
Laboratory Solutions

Code: SM-6a
Reissued: November, 1948
Status: Standard
By: W.J.M.

# METHYL RED, ALCOHOLIC
(0.1%)

## Safety Precautions

1. Observe general laboratory Safety Rules.

2. Keep the alcohol away from open flames.

## Literature References

Furman, N. H., "Scott's Standard Methods of Analysis", II, 5th. Edition, D. Van Nostrand Co., Inc., 2192 (1939)

## Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Methyl red, commercial dye | 1 g. | 0.5 g. |
| 2. Ethyl alcohol, C.P.<br>$C_2H_5OH$, C.P. | 600 ml. | 300 ml. |

## Preparation

1. Dissolve the 0.5 g. of the methyl red in 300 ml. of the 95% ethyl alcohol in a 500 ml. volumetric flask.

2. Dilute to the mark with distilled water.

3. Shake and mix well.

4. Transfer into a 500 ml. glass-stoppered Pyrex bottle for storage.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SM-6b

Reissued: November, 1948
Status: Standard
By: W.J.M.

## METHYL RED
(0.1%, ½ neutralized)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

Kolthoff, I. M., and Laitinen, H. A., "pH and Electro Titrations," John Wiley and Sons, 28 (1941).

### Quantity and Container

100 ml. in a 100 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Methyl red, Commercial Dye | 1.0 g. | 0.10 g. |
| 2. Sodium hydroxide solution 0.05 N (Solutions Code: SS-8a) | 74 ml. | 7.4 ml. |

### Preparation

1. Grind to powder 0.10 g. methyl red in an agate or "Mullite" mortar with 7.4 ml. 0.05 N sodium hydroxide solution.
2. After the indicator is dissolved, transfer the solution into a 100 ml. volumetric flask.
3. Wash out the mortar and pestle well with distilled water adding the washings to the flask.
4. Dilute to the mark with distilled water.
5. Mix well and transfer into a 100 ml. glass-stoppered Pyrex bottle for storage.

[redacted]

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H. W. Laboratory Manual
Laboratory Solutions

Code: SM-10b

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By: W.J.M.

# MERCURIC CHLORIDE
(0.35%)

## Safety Precautions

1. Observe general laboratory Safety Rules.

2. Mercuric chloride is extremely poisonous, and care must be exercised that it is not taken internally.

## Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Mercuric chloride, C.P.<br>$HgCl_2$, C.P. | 3.5 g. | 3.5 g. |

## Preparation

1. Weigh the 3.500 g. of the mercuric chloride on a watch glass and transfer it into a 1 liter volumetric flask, rinsing the watch glass with distilled water and adding the rinsings to the flask.

2. Add about 500 ml. of distilled water to the flask.

3. Shake the flask until the mercuric chloride is in solution.

4. Dilute to the mark with distilled water and mix well.

5. Transfer into a 1 liter glass-stoppered Pyrex bottle for storage.

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| Code: | SM-10c |
| Reissued: | November, 1948 |
| Status: | Standard |
| By: | W.J.M. |

## MERCURIC CHLORIDE
(Saturated Aqueous)

### Safety Precautions

1. Observe general laboratory Safety Rules.

2. Mercuric chloride is extremely poisonous. Avoid oral contact.

### Literature References

Merck and Co., "The Merck Index", 5th Ed., Merck and Co., Inc., Rahway, N.J., 1027 (1940).

### Quantity and Container

100 ml. in a 125 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Mercuric chloride, C.P.<br>$HgCl_2$, C.P. | 75 g. | 7.5 g. |

### Preparation

1. Weigh 7.5 g. of the mercuric chloride and transfer into a 100 ml. volumetric flask.

2. Dissolve in distilled water and dilute to the mark.

3. Mix well and transfer into a 125 ml. glass-stoppered Pyrex bottle.

[redacted]

[redacted]

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Laboratory Solutions

Code: SM-15b

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# MOLYBDATE REAGENT

## Safety Precautions

1. Observe general laboratory Safety Rules.

2. Observe rules pertaining to the handling of strong acids.

## Literature References

Snell, F. D., and Snell, C. T., "Colorimetric Methods of Analysis", I, Second Edition, D. Van Nostrand Co., 509 (1943).

## Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Ammonium molybdate, C.P.<br>$(NH_4)_2$MoO4, C.P. | 128 | 64 |
| 2. Sulfuric Acid, 10N<br>Solutions Code: SS-23a | 1000 ml. | 500 ml. |

## Preparation

1. Weigh on a torsion balance 64 g. of the ammonium molybdate.

2. Transfer into a 1000 ml. beaker and dissolve in 500 ml. of distilled water.

3. Transfer the solution into a 1 liter volumetric flask.

4. Using a graduated cylinder, add slowly the 500 ml. of the 10N sulfuric acid.

5. Mix well and transfer into a 1 liter glass-stoppered Pyrex bottle for storage.

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Code: SN-5a

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Laboratory Solutions

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## NESSLER'S SOLUTION

### Safety Precautions

1. Observe the general laboratory Safety Rules.
2. Mercuric chloride is a poisonous and corrosive chemical; avoid any oral contact.
3. Observe the rules pertaining to handling strong acids.

### Literature References

Snell, F. D., and Snell, C. T., "Colorimetric Methods of Analysis", I, D. Van Nostrand Co., Inc., 653 (1943)

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Potassium iodide, C.P.<br>KI, C.P. | 50 g. | 50 g. |
| 2. Mercuric chloride, saturated aqueous solution (Solutions Code: SM-10c) | | |
| 3. Potassium hydroxide, clear 50% solution (Standards Code: SG-P-5b; omit the standardization) | 400 ml. | 400 ml. |
| 4. Redistilled water, ammonia free (Solutions Code: SW-1c) | | |

[redacted]

[redacted]

Code: SN-5a

Preparation

1. Weigh on a torsion balance 50 g. of potassium iodide.
2. Transfer the 50 g. of potassium iodide into a 1 liter volumetric flask.
3. Add approximately 35 ml. of cold ammonia-free distilled water and dissolve the potassium iodide.
4. Alternately, add the saturated mercuric chloride solution, and swirl the flask until a very slight, red precipitate is formed and remains.
5. Using a graduated cylinder, add 400 ml. of the 50% potassium hydroxide solution, and swirl the flask.
6. Dilute to the mark with ammonia-free distilled water, and mix well by shaking (Remark 1).
7. Allow the solution to stand for 24 hours and then decant the clear solution into a 1 liter glass-stoppered Pyrex bottle for storage.

Remarks

1. If a heavy precipitate remains, a new Nessler's solution should be made up.

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Laboratory Solutions

Code: SN-8a
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# NICKEL SOLUTION STANDARD
(1 ml. = 10 micrograms Mi/ml.)

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

1. Snell, F. D. and Snell, C. T., "Colorimetric Methods of Analysis", I, D. Van Nostrand Co., Inc., 316 (1943)

## Quantity and Container

1. 1000 ml. in four 250 ml. glass-stoppered Pyrex bottles.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Nickel ammonium sulfate, C.P.<br>$NiSO_4 (NH_4)_2 \cdot 6H_2O$, C.P. | 0.0673 g. | 0.0673 g. |

## Preparation

1. Transfer the 0.0673 g. of nickel ammonium into a sulfate 1000 ml. volumetric flask.
2. Add about 500 ml. distilled water to the volumetric flask and mix until the salt is dissolved.
3. Dilute to the mark with distilled water and mix well.
4. Store in four 250 ml. glass-stoppered Pyrex bottles.

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Code: SN-9a

H. W. Laboratory Manual
Laboratory Solutions

Reissued: November, 1948
Status: Standard
By: W.J.M.

# NITRIC ACID SOLUTIONS

## Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe the rules pertaining to handling strong acids.

## Reagents and Specifications

### For Preparation

Nitric acid, C.P.
$HNO_3$, 70%

## Preparation

1. Calculate as follows the volume of concentrated nitric acid required to make the desired volume and concentration of dilute acid:

   a. For normality or molarity:

   $$V_1 = 63.2\ (V_2 \times N_2)$$

   where $V_1$ = ml. of 70% nitric acid to be used.
   $V_2$ = desired total volume in liters of dilute solution.
   $N_2$ = desired normality or molarity of dilute solution.

   Example: To make up 1 liter of 0.1 N $HNO_3$

   $$V_1 = 63.2\ (1 \times 0.1) = 6.3 \text{ ml. of } 70\%\ HNO_3$$

   b. For percentage of $HNO_3$:

   $$V_1 = \left(\frac{N}{70}\right) V_2$$

   where $V_1$ = ml. of 70% nitric acid to be used
   N = desired percentage
   $V_2$ = desired total volume in ml.

[redacted]

Example: To make up 500 ml. of 15% $HNO_3$

$$V_1 = \left(\frac{15}{70}\right) 500 = 107 \text{ ml. of } 70\% \; HNO_3$$

c. For parts:

$$V_1 = \left(\frac{a}{b}\right) V_2$$

where $V_1$ = ml. of 70% $HNO_3$ to be used
$V_2$ = desired total volume in ml.
a = desired parts of acid.
b = desired parts of acid <u>plus</u> desired parts of water

Example: To make up 500 ml. of 1:3 $HNO_3$

$$V_1 = \left(\frac{1}{4}\right) 500 = 125 \text{ ml. } 70\% \; HNO_3$$

2. By means of a graduated cylinder slowly add the required amount of concentrated nitric acid into a volumetric flask of the proper size containing at least half of the total volume of distilled water to be used. This work must be done in a well ventilated hood.

3. Mix well and dilute to the mark with distilled water.

4. Mix well and transfer into a glass-stoppered Pyrex bottle of the proper size for storage.

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## NITRATE SOLUTION STANDARD
(0.01 mg. N/ml.)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

American Public Health Association, "Standard Methods for the Examination of Water and Sewage", 8th Edition, A.P.H.A., New York, 45 (1936).

### Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Potassium nitrate, C.P.<br>$KNO_3$, C.P. | 0.07216 g. | 0.7216 g. |
| 2. Phenoldisulfphonic acid, C.P. | 4 ml. | 2 ml. |

### Preparation

1. Weigh out exactly 0.7216 g. of the potassium nitrate, and transfer into a 1 liter volumetric flask.

2. Dissolve the salt in a little distilled water, then dilute the solution to the mark and mix thoroughly.

3. Pipet 50 ml. of this stock solution into a small evaporating dish, and evaporate it to dryness on a steam bath. Do not allow the material to spatter.

4. Add 2 ml. of phenoldisulfonic acid to the contents of the dish, and rub the mixture thoroughly with a glass rod to insure intimate contact.
5. Dilute the solution with distilled water, and transfer it into a 500 ml. volumetric flask.
6. Dilute the solution to the mark with distilled water, and mix thoroughly.
7. Transfer the solution into a 500 ml. glass-stoppered [redacted] bottle for storage.

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Code: SO-24a
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By: W.J.M.

# OXALIC ACID
## (.1N)

### Safety Precautions

1. Observe general laboratory Safety Rules
2. Oxalic acid is poisonous and care should be exercised to prevent taking it internally.

### Literature Reference

Griffin, R. C., "Technical Methods of Analysis", McGrew and Hill Co , 9 (1927).

### Quantity and Container

1 liter in 1 liter dark bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Oxalic axid, anhydrous, C.P. $H_2C_2O_4$, C. P. | 4,501 g. | 4,501 g. |
| or | | |
| 2. Oxalic acid, crystalline, C.P, $H_2C_2O_4 \cdot 2H_2O$, C.P | 6,302 g. | 6,302 g. |

### Preparation

1. Weigh accuretly the required amount of oxalic acid and transfer into a 100 ml. beaker.

2. Add about 50 ml. of distilled water to dissolve the acid and transfer into a 1 liter volumetric flask. Rinse the beaker and add the washings to the flask.

3. Dilute to the mark with distilled water, mix well, and transfer into a dark bottle for storage.

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Code: SO-24b

H. W. Laboratory Manual
Laboratory Solutions

Reissued: November, 1948
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By: W.J.M.

# OXALIC ACID
(10%)

## Safety Precautions

1. Observe general laboratory Safety Rules.
2. Oxalic acid is poisonous and care should be exercised to avoid taking it internally.

## Quantity and Container

500 ml. in 500 ml. soft glass bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Oxalic acid, C.P.<br>$H_2C_2O_4 \cdot 2N_2O$, C.P. | 100 g. | 50 g. |

## Preparation

1. Weigh on a torsion balance the required amount of the oxalic acid.
2. Transfer into a 500 ml. beaker, adding about 300 ml. of distilled water to dissolve the oxalic acid.
3. Transfer the solution into a 500 ml. volumetric flask, dilute to the mark with distilled water.
4. Mix well and transfer into a 500 ml. soft glass bottle for storage.

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Code: SP-3a

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# PHENOL DISULFONIC ACID

## Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to handling strong acids and bases.
3. The fumes from fuming sulfuric acid are very irritating; mix acids and phenol under the hood.

## Literature References

American Public Health Association, "Standard Methods for the Examination of Water and Sewage", 8th Edition, A.P.H.A., New York 45 (1936)

## Quantity and Container

225 ml. in 250 ml. glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Phenol, C.P., colorless<br>$C_6H_5OH$, C.P. colorless | 111 g. | 25 g. |
| 2. Sulfuric acid, C.P.<br>$H_2SO_4$, 96% | 667 ml. | 150 ml. |
| 3. Fuming sulfuric acid, Technical grade<br>$H_2SO_4$, 15% free $SO_3$ | 333 ml. | 75 ml. |

## Preparation

1. Dissolve 25 grams of colorless phenol in 150 ml. of pure concentrated sulfuric acid in a 400 ml. beaker.
2. Add 75 ml. of fuming sulfuric acid containing at least 15% free sulfur trioxide.
3. Stir well and heat in a flask on a water bath for two hours.

[redacted]

The reagents are then free of mono- and tri-sulfonic acids.

4. Transfer the reagent into a 250 ml. glass-stoppered Pyrex bottle for storage.

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Code: SP-4a

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By: W.J.M.

# POTASSIUM HYDROXIDE, ALCOHOLIC
## (0.5N)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to handling strong bases.

### Literature References

Furman, N.H., "Scott's Standard Methods of Chemical Analysis", II, 5th Edition, D. Van Nostrand, New York, 1771 (1939).

### Quantity and Container

1000 ml. in a 1 liter cork-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Potassium hydroxide, C.P.<br>KOH, C.P. | 29 g. | 29 g. |
| 2. Ethanol, 95%, Purified<br>(Solutions Code: SE-21b) | 1 liter | 1 liter |

For Standardization

| | |
|---|---|
| 1. Hydrochloric acid<br>0.33N | (Solutions Code: SH-25a) |
| 2. Phenolphthalein indicator<br>1.0% | (Solutions Code: SP-8a) |

### Special Apparatus

Pipet, special calibrations, 25 ml. (Apparatus Code: AP-9a)

### Preparation

1. Weigh out 29 ± 1.0 g. of C.P. potassium hydroxide rapidly into a 750 ml. Pyrex beaker on a rough scale or balance.

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2. Dissolve in 500 ml. of 95% purified ethyl alcohol.

3. Allow the solution to settle in a dark place.

4. Filter through an asbestos filter.

5. Transfer into a 1 liter volumetric flask.

6. Dilute to the mark with the 95% purified alcohol. Shake and mix well.

7. Transfer into a paraffin-lined cork-stoppered bottle and store in a dark place.

## Standardization

1. Using the calibrated pipet, transfer 25 ml. of the solution to a 100 ml. Erlenmeyer flask.

2. Add 1 drop of 1.0% phenolphthalein indicator solution.

3. Titrate, using a 50 ml. buret, with standard 0.33<u>N</u> hydrochloric acid to a faint pink color.

4. Duplicate determination should check within 0.005.

## Calculations

$$N_1 = \frac{V_2 \times N_2}{V_1}$$

where $N_1$ = Normality of potassium hydroxide.

$V_1$ = Volume of potassium hydroxide.

$N_2$ = Normality of 0.33N hydrochloric acid.

$V_2$ = Volume of hydrochloric acid.

## Remarks

1. This solution should stand at least 24 hours before standardizing.

2. Restandardize before each determination.

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Laboratory Solutions

Code: Sp-5a

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Status: Standard
By: W.J.M

## PHENOL RED, ALCOHOLIC
(0.1%)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Do not use alcohol near a flame.

### Literature References

Rieman, W., Neuss, J. D., and Naimann, B., "Quantitative Analysis" McGraw - Hill, 454 (1942).

### Quantity and Container

500 ml. in 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Phenol red, commercial dye | 1 g. | 0.5 g. |
| 2. Ethyl alcohol, C. P. $C_2H_5OH$, 95% | 1000 ml. | 500 ml. |

### Preparation

1. Dissolve 0.5 g. of phenol red crystals in about 200 ml. of the alcohol in a 600 ml. beaker.
2. Transfer into a 500 ml. volumetric flask and dilute to the mark with the alcohol.
3. Mix thoroughly and transfer into a 500 ml. glass-stoppered Pyrex bottle for storage.

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Code: SP-8a
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## PHENOLPHTHALEIN INDICATOR
(1.0%)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules for handling alcohol.

### Literature References

Furman, N. H., "Scott's Standard Methods of Chemical Analysis", II, 5th Edition, D. Van Nostrand Co. 2192 (1939)

### Quantity and Container

500 ml. in 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Phenolphthalein, commercial dye<br>$C_{20}H_{14}O_4$, commercial dye | 10 g. | 5 g. |
| 2. Ethyl alcohol, C.P.<br>$C_2H_5OH$, 95% | 1000 ml. | 500 ml. |

### Preparation

1. Dissolve the 5 g. phenolphthalein in 250 ml. of the ethyl alcohol in a 500 ml. volumetric flask.
2. Dilute to the mark with alcohol.
3. Mix well and transfer into a 500 ml. glass-stoppered Pyrex bottle for storage.

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Code: SP-8b

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## PHENOLPHTHALEIN INDICATOR

(0.50%)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to handling alcohol.

### Quantity and Container

100 ml. in 125 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Phenolphthalein, Commercial Dye, $C_{20}H_{14}O_4$ | 5.0 g. | 0.5 g. |
| 2. Ethanol, C.P. $C_2H_5OH$, 95% | 1 liter | 100 ml. |

### Preparation

1. Weigh out 0.50 g. phenolphthalein, and dissolve in about 50 ml. of the ethanol in a 100 ml. volumetric flask.
2. Dilute to the mark with ethanol and mix well.
3. Transfer solution into a 125 ml. glass-stoppered Pyrex bottle for storage.

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## PHOSPHATE STANDARD
## (250 ppm $Na_3PO_4$)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sodium dihydrogen phosphate, C.P.<br>$NaH_2PO_4 \cdot H_2O$, C.P. | 2.10 g. | 2.10 g. |

### Preparation

1. Weigh 2.10 g. of the sodium dihydrogen phosphate and transfer to a 1 liter volumetric flask.

2. Dissolve in distilled water, dilute to the mark with distilled water, and mix well.

3. Pipet 100 ml. of this solution into another 1 liter volumetric flask and dilute to the mark with distilled water.

4. Mix well and transfer into a dry 1 liter glass-stoppered Pyrex flask for storage.

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Code: SP-12a

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By: W.J.M.

## POTASSIUM CHROMATE
(10%)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

50 ml. in a 60 ml. dropping bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Potassium chromate, C.P.<br>$K_2CrO_4$, C.P. | 100 g. | 5 g. |

### Preparation

1. Weigh 5 g. of the salt into a 60 ml. beaker.
2. Add 50 ml. of distilled water to the beaker and mix until the salt is in solution.
3. Transfer the contents of the beaker into a 60 ml. dropping bottle for storage.

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Code: SP-13c

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By: W.J.M.

## POTASSIUM CHLORIDE
(4% in 0.5% HCl)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to handling strong acids.

### Quantity and Container

5000 ml. in a 5 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Potassium chloride, C.P.<br>KCl, C.P. | 40 g. | 200 g. |
| 2. Hydrochloric acid, C.P.<br>HCl, 36% | 5 ml. | 25 ml. |

### Preparation

1. Weigh on a torsion balance 200 g. of the potassium chloride and transfer into the 5 liter storage bottle containing about 4975 ml. of distilled water.
2. Mix the contents of the bottle, using a stirring assembly, until the salt is in solution.
3. Acidify the solution with concentrated hydrochloric acid using litmus paper, then add 25 ml. of concentrated hydrochloric acid.
4. Mix the solution thoroughly and store for use.

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## POTASSIUM BI-IODATE
### (0.025 N)

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

American Public Health Association, "Standard Methods for the Examination of Water and Sewage", 8th Edition, A.P.H.A., 142 (1936).

## Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Potassium bi-iodate, C.P.<br>$KIO_3 \cdot HIO_3$, C.P. | 0.8124 g. | 0.4062 g. |

## Preparation

1. Transfer the accurately weighed portion of the potassium bi-iodate into a 400 ml. beaker, and dissolve in about 200 ml. distilled water.

2. Transfer into a 500 ml. volumetric flask, rinsing the beaker and adding washings to the flask.

3. Dilute to the mark with distilled water.

4. Mix well and store in a 500 ml. glass-stoppered Pyrex bottle.

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Code: SP-15a
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## POTASSIUM IODIDE
(10%, 20%)

### Safety Precautions

Observe general laboratory Safety Rules.

### 10% Solution

#### Quantity and Container

250 ml. in a 250 ml. glass-stoppered amber bottle.

#### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Potassium iodide, C.P.<br>KI, C.P. | 100 g. | 25 g. |

#### Preparation

1. Transfer the 25 g. of potassium iodide into a 250 ml volumetric flask and add about 150 ml. of distilled water.
2. Dissolve the crystals, dilute to 250 ml. with distilled water, and mix well by shaking.
3. Transfer the solution into a dry, 250 ml. glass-stoppered amber bottle for storage.

### 20% Solution

#### Quantity and Container

250 ml. in a 250 ml. glass-stoppered amber bottle.

#### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Potassium iodide, C.P.<br>KI, C.P. | 200 g. | 50 g. |

[redacted]

Preparation

1. Transfer the 50 g. of potassium iodide into a 250 ml. volumetric flask and add about 150 ml. of distilled water.

2. Dissolve the crystals, dilute to 250 ml. with distilled water, and mix well by shaking.

3. Transfer the solution into a dry, glass-stoppered amber bottle for storage.

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Code SP-15b

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## POTASSIUM IODIDE, ALKALINE
(about 10%)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to handling strong bases.

### Literature References

American Public Health Association, "Standard Methods for the Examination of Water and Sewage", 8th Edition, A.P.H.A., 143(1936).

### Quantity and Container

100 ml. in 4 oz. rubber-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Potassium iodide, C.P.<br>KI, C.P. | 150 g. | 15 g. |
| 2. Potassium hydroxide, C.P.<br>KOH, C.P. | 700 g. | 70 g. |

### Preparation

1. Dissolve the 70 g. of potassium hydroxide in 50 ml. of freshly boiled distilled water in a 250 ml. beaker.
2. Add the 15 g. of potassium iodide and mix until solution is complete.
3. Transfer into a 100 ml. volumetric flash and dilute to the mark with distilled water.
4. Mix well and transfer into a 4 oz. rubber-stoppered Pyrex bottle.

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## POTASSIUM IODIDE -- BUFFERED
(About 6.5%)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

1000 ml. in 1 liter glass-stoppered amber bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Potassium iodide, C.P.<br>KI, C.P. | 65 g. | 65 g. |
| 2. Ammonium chloride, C.P.<br>$NH_4Cl$, C.P. | 2 g. | 2 g. |

### Preparation

1. Dissolve the weighed potassium iodide and ammonium chloride in 100 ml. of distilled water in a 125 ml. beaker.

2. Transfer to a 1 liter volumetric flask and dilute to mark with distilled water.

3. Mix well and store in a glass-stoppered amber bottle.

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Code: SP-16a
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## POTASSIUM PERMANGANATE
## (0.1N)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Potassium permanganate is a strong oxidizing agent and should not be allowed to come in contact with reducing agents except under controlled conditions.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle painted black.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Potassium permanganate, C.P.<br>$KMnO_4$, C.P. | 3.2 g. | 3.2 g. |

For Standardization

1. Sulfuric acid, C.P.
   $H_2SO_4$, 96%
2. Sodium oxalate, C.P.
   $Na_2C_2O_4$, C.P.

### Special Apparatus

Buret, 10 ml. (Apparatus Code: AB-21a)

### Preparation

1. Weigh the 3.2 g. of the potassium permanganate and transfer into a 1 liter volumetric flask.
2. Dissolve the weighed salt in distilled water and dilute to the mark with distilled water.
3. Transfer to a 2000 ml. beaker and boil the solution vigorously for 10 minutes.

[redacted]

4. Filter through ignited asbestos and catch the solution in a 1 liter glass-stoppered clean dark bottle for storage.

5. Allow to age in the dark bottle for several days before standardizing.

## Standardization

1. Dry in a weighing bottle about 1 g. of primary standard sodium oxalate at 105°C in the oven.

2. Cool bottle in disiccator for about 30 minutes.

3. Weigh accuretly 0.50 to 0.625 g. portion of the dried salt and transfer into a 250 ml. Erlenmeyer flask.

4. Dissolve in about 200 ml. of distilled water and add 10 ml. of 96% sulfuric acid.

5. Cool to room temperature, transfer into a 250 ml. volumetric flask and dilute to the mark with distilled water.

6. Pipet 25 ml. of this solution into a 100 ml. becker and heat to 80 to 90°C. (Remark 1)

7. Using a 10 ml. buret add potassium permanganate solution dropwise until each drop is decolorized immediately. (Remark 2.)

8. Then add the permanganate rapidly until about seven ml. has been run in.

9. Allow all the color to become discharged and then titrate dropwise to a 30 second, light pink endpoint.

## Calculations

$$\text{Normality } KMNO_4 = \frac{\text{wt. of } Na_2C_2O_4 \text{ g.}}{0.0670 \times \text{ml. } KMNO_4}$$

Duplicate determinations should agree to within 0.0001

## Remarks

1. A temperature of 80 to 90°C must be maintained throughout the standardization in order to obtain the sharpest possible end point.
2. The titration is started by dropwise addition to prevent side reactions taking place. While this is not experienced in this reaction, it is possible in certain other permanganate determinations.

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## POTASSIUM IODATE
## (0.01586N)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Potassium iodate, C.P.<br>$KIO_3$, C.P. | 0.5655 g. | 0.5655 g. |
| 2. Sodium bicarbonate, C.P.<br>$NaHCO_3$, C.P. | 0.25 g. | 0.25 g. |

### Preparation

1. Weigh 0.5655 g. of the potassium iodate and 0.25 g. of the sodium bicarbonate and transfer into a 1 liter volumetric flask.
2. Dilute the contents of the volumetric flask to the mark with distilled water and mix thoroughly.
3. Transfer the solution into a 1 liter glass-stoppered Pyrex bottle for storage.

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# POTASSIUM IODATE AS PRIMARY STANDARD IN ACIDIMETRY

## Safety Precautions

Observe general laboratory Safety Rules.

## Literature References

Koltoff, I. M., and Sandell, E. B., "Quantitative Inorganic Analysis", The Macmillan Co., New York, 550 (1943).

## Quantity and Container

100 ml. in a 125 ml. glass-stoppered Pyrex bottle.

## Reagents

### For Preparation

Potassium iodate, Primary Standard (Remark 1)
$KIO_3$, Primary Standard

### For Standardization

1. Sodium thiosulfate, 0.1N (Solutions Code: SS-18a)
2. Potassium iodide, C.P.
   KI, C.P.
3. Methyl red, alcoholic, 0.1% (Solutions Code: SM-6a)

## Special Apparatus

1. Pipet, 10 ml. calibrated (Apparatus Code: Ap-9a)
2. Buret, 10 ml. semi-micro (Apparatus Code: AB-21a)

## Preparation

1. Weigh the calculated amount of primary standard potassium iodate to the fourth decimal place, into a 100 ml. beaker (Remark 2).

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2. Add about 50 ml. of distilled water to the beaker and mix until the salt is in solution.

3. Transfer the solution into a 100 ml. volumetric flask. Rinse the beaker at least three times with distilled water, adding the rinsings to the flask.

4. Dilute the contents of the volumetric flask to the mark with distilled water, mix thoroughly and transfer the solution into a 125 ml. glass-stoppered Pyrex bottle for storage.

## Standardization

1. By means of a calibrated 10 ml. pipet, transfer an aliquot of the standard potassium iodate solution into a 100 ml. Erlenmeyer flask.

2. Add 2 ml. of the 1N sodium thiosulfate, 0.17 g. of the potassium iodide crystals, and 1 drop of the 0.1% methyl red indicator to the flask and mix thoroughly (Remark 4).

3. Titrate the contents of the Erlenmeyer flask with the hydrochloric acid to be standardized, using a 10 ml. semi-micro buret.

4. Repeat steps (1) through (3) until three successive titrations agree within 0.1%.

## Calculations

$$N_1 = \frac{N_2 \times V_2}{V_1}$$

where $N_1$ = Normality of the hydrochloric acid.

$N_2$ = Normality of the potassium iodate.

$V_1$ = Volume of the hydrochloric acid.

$V_2$ = Volume of the potassium iodate.

## Remarks

1. Calculate the g. of the potassium iodate required to make the desired normality and volume as follows:

$$\text{g. } KIO_3 = \frac{A \times B \times C}{D \times E}$$

where A = 214.02 (M.W. $KIO_3$)

B = Desired normality

C = Desired volume (100 ml.)

D = 6 ($H^+$ equivalent)

E = 1000

2. The primary standard potassium iodate should be dried to constant weight in a sulfuric acid desiccator. Heating the salt above 100°C causes decomposition with resultant liberation of iodine. Therefore, the former means of drying should be resorted too.

3. Potassium iodate may be used for the direct standardization of acids by making use of the reaction:

$$IO_3^- + 5I^- + 6H^+ \text{ - - - } 3I_2 + 3H_2O$$

By adding an excess of neutral alkali iodide and sodium thiosulfate to a solution of potassium iodate, hydrogen ions are removed upon addition of hydrochloric acid, until all of the iodate has been converted into iodine. The iodine is removed by the excess sodium thiosulfate:

$$2S_2O_3^= + I_2 \text{ - - - } S_4O_6^= + 2I^-$$

Therefore, the liquid remains neutral and colorless during the titration with hydrochloric acid until the end-point is reached. As soon as all of the iodate has been used up, the solution becomes acid, as indicated by the color change of methyl red.

4. The values given are based on the use of 0.1N potassium iodate solution. For other than a 0.1N solution, vary these values accordingly.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SP-22a
Reissued: November, 1948
Status: Standard
By: W.J.M.

## POTASSIUM SODIUM TARTRATE
(5%)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

100 ml. in 125 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Potassium sodium tartrate, C.P. $KNaC_4H_4O_6\cdot 4H_2O$ (Rochelle Salts) | 50 g. | 5 g. |

### Preparation

1. Weigh out 5 g. of the potassium sodium tartrate, and transfer the weighed crystals to a 100 ml. volumetric flask.

2. Dissolve the crystals in a small amount of distilled water, and dilute to the mark with distilled water.

3. Transfer the solution into a 125 ml. glass-stoppered Pyrex bottle for storage.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SP-23a

Reissued: November, 1948
Status: Standard
By: W.J.M.

## POTASSIUM PERMANGANATE
(Alkaline Reagent)

### Safety Precautions

Observe the general laboratory Safety Rules.

### Literature References

American Public Health Association, "Standard Methods for the Examination of Water and Sewage", 8th Edition, A.P.H.A., New York, 45 (1936).

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered amber bottle.

### Reagents and Specification

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Potassium permanganate, C.P.<br>$KMnO_4$, C.P. | 8 g. | 8 g. |
| 2. Sodium hydroxide, C.P.<br>NaOH, C.P. | 180 g. | 180 g. |

### Preparation

1. Boil 600 ml. of distilled water in a 1000 ml. beaker for 10 minutes.
2. Allow the water to cool, and add 8 g. of the potassium permanganate.
3. In a separate 600 ml. beaker, dissolve 180 g. of the sodium hydroxide in 500 ml. of redistilled water. Allow the solution to stand until the insoluble material settles.
4. Pour the permanganate solution into a 2000 ml. beaker, and add 400 ml. of the clear supernatant from the sodium hydroxide solution prepared in step 3.
5. Add 250 ml. distilled water.
6. Concentrate the solution to 1 liter by evaporation. Transfer the cooled solution to a 1 liter glass-stoppered amber bottle for storage.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SP-25a

Reissued: November, 1948
Status: Standard
By: W.J.M.

# POTASSIUM PYROGALLATE
(Alkaline Pyrogallol)

## Safety Precautions

1. Observe general laboratory Safety Rules.
2. Pyrogallic acid is extremely poisonous. Avoid internal and external contact.
3. Observe rules pertaining to handling strong bases.

## Literature References

Welcher, F., "Chemical Solutions", D. Van Nostrand Co., Inc., New York, 280 (1942).

## Quantity and Container

200 ml. in a 250 ml. glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Pyrogallol, C.P.<br>$C_6H_6O_3$, C.P. | 50 g. | 10 g. |
| 2. Potassium hydroxide, C.P.<br>KOH, C.P. | 500 g. | 100 g. |

## Preparation

1. Weigh 100 g. of the potassium hydroxide into a 400 ml. Pyrex beaker.
2. Add 200 ml. of distilled water to the contents of the beaker and mix thoroughly.
3. Weigh out 10 g. of the solid pyrogallic acid on paper and pour into a funnel inserted in the 250 ml. glass-stoppered Pyrex bottle.
4. Pour the potassium hydroxide upon the pyrogallic acid. The latter will dissolve at once, and the solution is ready for use.

[redacted]

Remarks

1. This amount of potassium pyrogallate solution is sufficient for one determination when used with the Tin Apparatus Code: AT-9a. A fresh batch should be made up for each determination.

2. The concentration of the potassium pyrogallate is suitable for mixture of gases containing less than 28% oxygen.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SR-23b

Reissued: November, 1948
Status: Standard
By: W.J.M.

## RUTHENIUM CHLORIDE
(10 mg. Ru/ml.)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

Hume, D.N., Glendenin, I.E., Ballow, N.E., Coryell, C.D., "Elements of Methods for the Fission Product in Plant Process Solutions", Clinton Secret Report CN-1312 or A-3044.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Ruthenium chloride, C.P.<br>$RuCl_3$, C.P. | 28 g. | 28 g. |

### Preparation

1. Weigh 28 g. of the ruthenium chloride into a 400 ml. beaker and dissolve in about 200 ml. distilled water.
2. Transfer into a 1 liter volumetric flask and dilute to the mark with distilled water.
3. Transfer into a 1 liter glass-stoppered Pyrex bottle for storage.

[redacted]

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H. W. Laboratory Manual
Laboratory Solutions

Code: SS-1a
Reissued: November, 1948
Status: Standard
By: W.J.M.

## SOAP SOLUTION
### (1 ml. = 1 mg. $CaCO_3$)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to handling alcohol.

### Literature References

American Public Health Association, "Standard Methods for the Examination of Water and Sewage," A.P.H.A., 60 (1936).

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Stock soap solution, 10% (Solutions Code: SS-1b) | 110-125 ml. | 110-125 ml. |
| 2. Ethanol, C.P. $C_2H_5OH$, 80% | about 900 ml. | about 900 ml |

For Standardization

Calcium carbonate solution, 1 ml. = 1 mg. $CaCO_3$.
(Solutions Code: SC-1a)

### Preparation

1. Using a graduated cylinder measure out 110 - 125 ml. of stock soap solution into a 1000 ml. Erlenmeyer flask.
2. Dilute to about a liter with 80% ethanol.
3. Adjust the solution according to the procedure given under the standardization.

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## Standardization

### A. Determination of Lather Factor

1. Using a graduated cylinder, measure 50 ml. of distilled water into an 8 oz. glass-stoppered bottle.

2. Add from a buret 0.1 to 0.2 ml. of soap solution and shake the bottle vigorously.

3. Let bottle stand on its side until lather fades.

4. Continue adding soap solution in 0.1 to 0.2 ml. increments until a lather lasting 5 minutes is obtained.

5. Record the volume of soap solution used as the lather factor.

6. Repeat steps 1 - 5 until five determinations agreeing withing 0.2 ml. are obtained.

### B. Determination of $CaCO_3$ Titer

1. Place 45 ml. of distilled water in an 8 oz. glass-stoppered bottle and add 5 ml. of the standard $CaCO_3$ solution.

2. Titrate with standard soap solution as above until a 5 minute lather is obtained.

3. Deduct the lather factor from the titration and compare the volumes of soap and $CaCO_3$.

4. Adjust the volume of the soap solution until 1 ml. of soap solution is equivalent to 1 ml. of $CaCO_3$. (Remark 1).

## Remarks

1. The lather factor must be determined at each adjustment of the soap solution.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

| | |
|---|---|
| Code: | SS-1b |
| Reissued: | November, 1948 |
| Status: | Standard |
| By: | W.J.M. |

## SOAP SOLUTION
(10%)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature Reference

A.P.H.A., "Standard Methods for the Examination of Water and Sewage", A.P.H.A. and A.W.A.A., 60 (1936).

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Castile soap, pure powdered. | 100 g. | 100 g. |
| 2. Ethanol, C.P.<br>$C_2H_5OH$, 80% | 1000 ml. | 1000 ml. |

### Preparation

1. Dissolve about 100 g. of the soap in about 500 ml. of the 80% ethanol in a 1 liter volumetric flask.
2. Dilute to the mark with 80% ethanol mix, and let stand at least overnight.
3. Decant the clear solution into a 1 liter glass-stoppered Pyrex bottle for storage.

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Code: SS-3a

H. W. Laboratory Manual
Laboratory Solutions

Reissued: November, 1948
Status: Standard
By: W.J.M.

# SODIUM CARBONATE
## (10%)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

Griffin, R. C., "Technical Methods of Analysis", 2nd Edition, McGraw-Hill, 5(1927).

### Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sodium carbonate, C.P.<br>$Na_2CO_3$, C.P. | 100 g. | 50 g. |

### Preparation

1. Dissolve 50 g. of the sodium carbonate in about 200 ml. of distilled water in a 500 ml. volumetric flask, and dilute to the mark with distilled water.
2. Mix well and transfer into a 500 ml. glass-stoppered Pyrex bottle.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SS-4b

Reissued: November, 1948
Status: Standard
By: W.J.M.

# STANDARD CHLORIDE (STOCK SOLUTION)
## (0.005 g. Cl/ml.)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

500 ml. in 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sodium chloride, C.P.<br>NaCl, C.P. | 8.240 g. | 4.120 g. |

### Preparation

1. Weigh out exactly 4.120 g. of dry sodium chloride and dissolve in about 200 ml. distilled water in a 400 ml. beaker.

2. Transfer quantitatively into a 500 ml. volumetric flask and dilute to volume.

3. Transfer into a 500 ml. glass-stoppered Pyrex bottle for storage.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SS-4c

Reissued: November, 1948
Status: Standard
By: W.J.M.

## SODIUM CHLORIDE - HYDROCHLORIC ACID
(1.6% NaCl)
(2.4% HCl)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to the handling of strong acids.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Sodium chloride, C.P.<br>NaCl, C.P. | 16 g. | 16 g. |
| 2. Hydrochloric acid, C.P.<br>HCl, 36% | 20 ml. | 20 ml. |

### Preparation

1. Dissolve the weighed sodium chloride in 500 ml. of distilled water in a 600 ml. beaker.
2. Add the hydrochloric acid slowly with stirring.
3. Transfer into a 1 liter volumetric flask and dilute to the mark with distilled water.
4. Mix well and transfer into a 1 liter glass-stoppered Pyrex bottle for storage.

[redacted]

[redacted]

H. E. Laboratory Manual
Laboratory Solutions

Code: SS-4e

Reissued: November, 1948
Status: Standard
By: W.J.M.

## SODIUM CHLORIDE-STANDARD
(1 ml. = 5 micrograms)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sodium chloride<br>1 ml. = 0.005 g. Cl/ml.<br>(Solutions Code: SS-4b) | 1 ml. | 1 ml. |

### Preparation

1. Pipet the 1 ml. of the sodium chloride into a 1 liter volumetric flask.
2. Dilute to mark with chloride-free distilled water.
3. Transfer into a 1 liter glass-stoppered Pyrex bottle for storage.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SS-6a

Reissued: November, 1948
Status: Standard
By: W.J.M.

## SODIUM DIETHYLDITHIOCARBAMATE
(0.1%)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

AmericanPublic Health Association, "Standard Methods for the Examination of Water and Sewage", A.P.H.A., 25 (1936).

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered amber bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sodium diethyldithiocarbamate, C.P.<br>$N(C_2H_5)_2CS_2Na$, Eastman, C.P. | 1.0 g. | 1.0 g. |

### Preparation

1. Dissolve 1.0 g. of the sodium dethyldithiocarbamate in about 50 ml. distilled water in a 125 ml. beaker.

2. Transfer into a 1 liter volumetric flask, dilute to the mark with distilled water and mix well.

3. Filter into a 1 liter glass-stoppered amber bottle protected from light for storage.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SS-7a

Reissued: November, 1948
Status: Standard
By: W.J.M.

## SODIUM SULFATE
## (0.2M)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

250 ml. in a 250 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sodium sulfate, C.P.<br>$Na_2SO_4$, C.P. | 28.40 g. | 7.1 g. |

### Preparation

1. Weigh out 7.1 g. of the sodium sulfate and transfer into a 250 ml. volumetric flask.

2. Dilute to the mark with distilled water and mix well.

3. Transfer into a 250 ml. glass-stoppered Pyrex bottle for storage.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SS-8a
Issued: November, 1948
Status: Standard
By: W.J.M.

## SODIUM HYDROXIDE SOLUTIONS

### Safety Precautions

1. Observe general laboratoy Safety Rules.
2. Observe rules pertaining to handling strong bases.

### Reagents and Specifications

#### For Preparation

Sodium hydroxide, 50% solution
(Solutions Code: SS-8d)

#### For Standardization

1. Potassium acid phthalate, primary standard
   $KHC_8H_4O_4$, primary standard
2. Hydrochloric acid, 0.33N primary standard or secondary standard. (Remark 7)
3. Phenolpthalein indicator, 0.5% (Solutions Code: SP-8b)
4. Methyl red indicator, 0.1% (Solutions Code: SM-6a)

### Special Apparatus

Burets, semi-micro, 10 ml. (Apparatus Code: AB-21a).

### Preparation

1. Calculate the volume of the sodium hydroxide solution required to make the desired concentration as follows:

   a. For normality or molarity
   ml. of 50% sodium hydroxide = 52.6(A x B)

   where A = Desired normality or molarity.

   B = Desired total volume in liters.

   Example: to make 1 liter of 0.25N sodium hydroxide.

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ml. of 50% sodium hydroxide = $52.6(0.25 \times 1)$

= 13.2 ml. 50% sodium hydroxide.

b. For percentage of sodium hydroxide ml. of 50% sodium hydroxide = $\frac{(A)}{(50)}B$

where A = Desired percentage

B = Desired total volume in liters.

Example: to make 500 ml. of 20% sodium hydroxide

ml. of 50% sodium hydroxide = $\frac{(20)}{(50)}500$ = 200 ml. of 50% sodium hydroxide.

c. For parts

ml. of 50% sodium hydroxide = $A\frac{(a)}{(b)}$

where A = Desired total volume in ml.

a = Desired parts of base.

b = Desired parts of base plus desired parts of water.

Example: to make 500 ml. of 1:3 sodium hydroxide.

ml. of 50% sodium hydroxide = $500\ \left(\frac{1}{4}\right)$ = 125 ml. sodium hydroxide.

2. Slowly add rhe required amount of 50% sodium hydroxide to a volumetric flask of the proper size, containing at least 50% of the total required volume of distilled water.
3. Mix well and dilute to the mark with distilled water.
4. Transfer into a dry rubber-stoppered Pyrex bottle of the proper size. (Remarks 1,2 and 3)

[redacted]

## Standardization

A great many methods are available for standardizing alkali solutions. However, one of the methods given below will be found suitable for standardizing sodium hydroxide which is intended for any common use on this project. It is important in accurate work to select a standardization using the same indicator as will be involved in the subsequent uses of the solution (Remark 5).

## Against Primary Standard Potassium Acid Phthalate

1. Determine the approximate weight (in g.) of potassium acid phthalate samples to be weighed as follows:

   g. Potassium acid phthalate = A x B x C

   where A = 0.2

   B = Estimated normality of sodium hydroxide.

   C = Desired volume of alkali for titration (approximately 3/4 buret capacity)

2. Weigh accurately to four decimal places into suitable titrating containers several samples of about the above determined size.

3. Add about 50 ml. of distilled water (neutral to phenolphthalein) to each of the weighed samples, then add one drop of phenolphthalein indicator.

4. Titrate each of the phthalate samples with the alkali solution to be standardized to a light pink end-point which is permanent for 30 seconds.

5. Read the buret and apply temperature corrections (see below and Remark 4) to the observed volumes used.

## Calculations (Remark 6)

$$N_1 = \frac{W_1}{0.20406 \times V_1}$$

where $N_1$ = Normality of sodium hydroxide

$W_1$ = Weight of potassium acid phthalate

$V_1$ = Volume of sodium hydroxide

## Against Standard Hydrochloric Acid

This method is applicable to standardizations against either primary or secondary standard hydrochloric acid (Remark 7).

1. Measure accurately from a buret into a 100 ml. beaker or flask the required amount of hydrochloric acid standard.

2. Dilute with about 30 ml. of distilled water which is neutral to the indicator to be used.

3. Add one drop of phenolphthalein or methyl red indicator solution (Remark 5).

4. Titrate with the sodium hydroxide solution to be standardized.

5. Record the buret reading and apply temperature corrections to both buret readings if required (Remark 4).

## Calculations (Remark 6)

$$N_1 = \frac{V_2 \times N_2}{V_1}$$

where $N_1$ = Normality of sodium hydroxide

$V_1$ = ml. of sodium hydroxide

$N_2$ = Normality of hydrochloric acid

$V_2$ = ml. of hydrochloric acid

## Temperature Corrections

Temperature corrections for standard solutions are calculated as follows:

$$\text{Correction to be added (ml.)} = V \times (e - a) \times (20 - t)$$

where V = observed volume (ml.)

t = temperature of the solution at time of reading.

a = Cubical co-efficient of expansion of glass. For our purpose an average value of 0.00002 is applicable to either soft glass or Pyrex.

e = Cubical co-efficient of expansion of the solution which may be obtained for various strengths of sodium hydroxide solutions from the following table:

| Normality of sodium hydroxide solution | Cubical co-effecient of expansion ml. per ml. per °C. |
|---|---|
| 0 - 0.20 | 0.00025 |
| 0.20 - 0.40 | 0.00027 |
| 0.40 - 0.60 | 0.00029 |
| 1.0 | 0.00034 |
| 1.5 | 0.00037 |
| 2.0 | 0.00040 |

Tables of corrections to be applied to solutions having normalities between 0.20 and 0.40 and 0 and 0.20 have been worked out and will be found in this manual code: SA - 18a.

## Remarks

1. Solutions of strength greater than 0.33 N should be stored in paraffin-lined Pyrex bottles stoppered with corks which have been soaked in melted paraffin or with rubber stopperss which have been boiled in 10% sodium hydroxide solution and thoroughly washed with distilled water..

2. It is best to allow sodium hydroxide solutions to age for at least a week with daily shakings before attempting standardization.

3. Sodium hydroxide solutions of strength greater than 0.33 N should not be allowed to stand in a buret as etching of the buret and freezing of the stopcock will occur.

4. If the solutions are at the same temperature and of approximately the same strength no temperature corrections need be applied.

5. The chief important difference between the use of methyl red and phenolphthalein indicators is caused by the presence of small amounts of carbonate in nearly all alkali solutions. When using methyl red indicator this carbonate is titrated and included in the normality of the solution. This yields a value which is not greatly affected by carbon dioxide. In using phenolphthalein indicator the value of the carbonate is not included in the normality of the solution. Variations in normality obtained using the two indicators will be found to be in the order of 0.1%.

6. Duplicate determinations of normality should agree within 0.0002.

7. For best results the acid solution should have a strength of

within a factor of 2 of that of the solution to be standardized. Primary standard 0.33 N hydrochloric acid refers to material prepared directly from constant boiling hydrochloric acid.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SS-8d
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Status: Standard
By: W.J.M.

# SODIUM HYDROXIDE
## (50% = 19N)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to handling strong bases

### Quantity and Container

1000 ml. in 1 liter rubber-stoppered wax-lined Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sodium hydroxide pellets, C. P.<br>NaOH, C. P. | 760 g. | 760 g. |

### Preparation

1. Dissolve the 760 g. of the sodium hydroxide in about 700 ml. of distilled water in a 1000 ml. beaker and allow to cool.
2. Transfer into a 1 liter volumetric flask, dilute to the mark with distilled water, mix well and let stand until supernatant is clear.
3. Decant of supernatant liquid and transfer into a 1 liter Pyrex bottle wax-lined for storage.
4. Stopper with a cork previously soaked in paraffin or a rubber stopper which was boiled in 10% sodium hydroxide and carefully washed in distilled water.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SS-4b

Reissued: November, 1948
Status: Standard
By: W.J.M.

## STANDARD CHLORIDE (STOCK SOLUTION)
(0.005 g. Cl/ml.)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

500 ml. in 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sodium chloride, C.P.<br>NaCl, C.P. | 8.240 g. | 4.120 g. |

### Preparation

1. Weigh out exactly 4.120 g. of dry sodium chloride and dissolve in about 200 ml. distilled water in a 400 ml. beaker.

2. Transfer quantitatively into a 500 ml. volumetric flask and dilute to volume.

3. Transfer into a 500 ml. glass-stoppered Pyrex bottle for storage.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SS-4c

Reissued: November, 1948
Status: Standard
By: W.J.M.

# SODIUM CHLORIDE - HYDROCHLORIC ACID

(1.6% NaCl)
(2.4% HCl)

## Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to the handling of strong acids.

## Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Sodium chloride, C.P.<br>NaCl, C.P. | 16 g. | 16 g. |
| 2. Hydrochloric acid, C.P.<br>HCl, 36% | 20 ml. | 20 ml. |

## Preparation

1. Dissolve the weighed sodium chloride in 500 ml. of distilled water in a 600 ml. beaker.
2. Add the hydrochloric acid slowly with stirring.
3. Transfer into a 1 liter volumetric flask and dilute to the mark with distilled water.
4. Mix well and transfer into a 1 liter glass-stoppered Pyrex bottle for storage.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SZ-9c
Reissued: December, 1948
Status: Standard
By: W.J.M.

## ZIRCONIUM NITRATE
(50 mg. $Zr^{+4}$ ml. in $3\underline{N}$ $HNO_3$)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to handling strong acids.

### Quantity and Container

2000 ml. in a 2 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Zirconium nitrate, C.P.<br>$Zr(NO_3)_4$ | 186 g. | 372 g. |
| 2. Nitric acid, C.P.<br>$HNO_3$, 70% | 190 ml. | 380 ml. |

### Preparation

1. Weigh 372 g. of zirconium nitrate and add to a 600 ml. beaker containing 380 ml. of the nitric acid and 220 ml. distilled water.
2. Mix well and transfer into a 2 liter volumetric flask, dilute to the mark with distilled water.
3. After thorough mixing transfer into a 2 liter glass-stoppered Pyrex bottle for storage.

[redacted]

[redacted]

| | |
|---|---|
| Code: | SS-4e |

H. E. Laboratory Manual
Laboratory Solutions

Reissued: November, 1948
Status: Standard
By: W.J.M.

## SODIUM CHLORIDE STANDARD
(1 ml. = 5 micrograms)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sodium chloride<br>1 ml. = 0.005 g. Cl/ml.<br>(Solutions Code: SS-4b) | 1 ml. | 1 ml. |

### Preparation

1. Pipet the 1 ml. of the sodium chloride into a 1 liter volumetric flask.
2. Dilute to mark with chloride-free distilled water.
3. Transfer into a 1 liter glass-stoppered Pyrex bottle for storage.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SS-6a
Reissued: November, 1948
Status: Standard
By: W.J.M.

# SODIUM DIETHYLDITHIOCARBAMATE
## (0.1%)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

American Public Health Association, "Standard Methods for the Examination of Water and Sewage", A.P.H.A., 25 (1936).

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered amber bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sodium diethyldithiocarbamate, C.P.<br>$N(C_2H_5)_2CS_2Na$, Eastman, C.P. | 1.0 g. | 1.0 g. |

### Preparation

1. Dissolve 1.0 g. of the sodium dethyldithiocarbamate in about 50 ml. distilled water in a 125 ml. beaker.

2. Transfer into a 1 liter volumetric flask, dilute to the mark with distilled water and mix well.

3. Filter into a 1 liter glass-stoppered amber bottle protected from light for storage.

[redacted]

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Code: SS-7a

H. W. Laboratory Manual
Laboratory Solutions

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By: W.J.M.

## SODIUM SULFATE
(0.2M)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

250 ml. in a 250 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sodium sulfate, C.P.<br>$Na_2SO_4$, C.P. | 28.40 g. | 7.1 g. |

### Preparation

1. Weigh out 7.1 g. of the sodium sulfate and transfer into a 250 ml. volumetric flask.

2. Dilute to the mark with distilled water and mix well.

3. Transfer into a 250 ml. glass-stoppered Pyrex bottle for storage.

[redacted]

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H. W. Laboratory Manual
Laboratory Solutions

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# SODIUM HYDROXIDE SOLUTIONS

## Safety Precautions

1. Observe general laboratoy Safety Rules.
2. Observe rules pertaining to handling strong bases.

## Reagents and Specifications

### For Preparation

Sodium hydroxide, 50% solution
(Solutions Code: SS-8d)

### For Standardization

1. Potassium acid phthalate, primary standard $KHC_8H_4O_4$, primary standard
2. Hydrochloric acid, 0.33N primary standard or secondary standard. (Remark 7)
3. Phenolpthalein indicator, 0.5% (Solutions Code: SP-8b)
4. Methyl red indicator, 0.1% (Solutions Code: SM-6a)

## Special Apparatus

Burets, semi-micro, 10 ml. (Apparatus Code: AB-21a).

## Preparation

1. Calculate the volume of the sodium hydroxide solution required to make the desired concentration as follows:

    a. For normality or molarity
    ml. of 50% sodium hydroxide = 52.6(A x B)

    where A = Desired normality or molarity.

    B = Desired total volume in liters.

    Example: to make 1 liter of 0.25N sodium hydroxide.

[redacted]

[redacted]

ml. of 50% sodium hydroxide = 52.6(0.25 x 1)

= 13.2 ml. 50% sodium hydroxide.

b. For percentage of sodium hydroxide ml. of 50% sodium hydroxide = $\left(\frac{A}{50}\right)B$

where A = Desired percentage

B = Desired total volume in liters.

Example: to make 500 ml. of 20% sodium hydroxide

ml. of 50% sodium hydroxide = $\left(\frac{20}{50}\right)500$ = 200 ml. of 50% sodium hydroxide.

c. For parts

ml. of 50% sodium hydroxide = $A\left(\frac{a}{b}\right)$

where A = Desired total volume in ml.

a = Desired parts of base.

b = Desired parts of base <u>plus</u> desired parts of water.

Example: to make 500 ml. of 1:3 sodium hydroxide.

ml. of 50% sodium hydroxide = $500\left(\frac{1}{4}\right)$ = 125 ml. sodium hydroxide.

2. Slowly add rhe required amount of 50% sodium hydroxide to a volumetric flask of the proper size, containing at least 50% of the total required volume of distilled water.
3. Mix well and dilute to the mark with distilled water.
4. Transfer into a dry rubber-stoppered Pyrex bottle of the proper size. (Remarks 1,2 and 3)

[redacted]

[redacted]

## Standardization

A great many methods are available for standardizing alkali solutions. However, one of the methods given below will be found suitable for standardizing sodium hydroxide which is intended for any common use on this project. It is important in accurate work to select a standardization using the same indicator as will be involved in the subsequent uses of the solution (Remark 5).

## Against Primary Standard Potassium Acid Phthalate

1. Determine the approximate weight (in g.) of potassium acid phthalate samples to be weighed as follows:

   g. Potassium acid phthalate = A x B x C

   where A = 0.2

   B = Estimated normality of sodium hydroxide.

   C = Desired volume of alkali for titration (approximately 3/4 buret capacity)

2. Weigh accurately to four decimal places into suitable titrating containers several samples of about the above determined size.

3. Add about 50 ml. of distilled water (neutral to phenolphthalein) to each of the weighed samples, then add one drop of phenolphthalein indicator.

4. Titrate each of the phthalate samples with the alkali solution to be standardized to a light pink end-point which is permanent for 30 seconds.

5. Read the buret and apply temperature corrections (see below and Remark 4) to the observed volumes used.

## Calculations (Remark 6)

$$N_1 = \frac{W_1}{0.20406 \times V_1}$$

where $N_1$ = Normality of sodium hydroxide

$W_1$ = Weight of potassium acid phthalate

$V_1$ = Volume of sodium hydroxide

[redacted]

## Against Standard Hydrochloric Acid

This method is applicable to standardizations against either primary or secondary standard hydrochloric acid (Remark 7).

1. Measure accurately from a buret into a 100 ml. beaker or flask the required amount of hydrochloric acid standard.

2. Dilute with about 30 ml. of distilled water which is neutral to the indicator to be used.

3. Add one drop of phenolphthalein or methyl red indicator solution (Remark 5).

4. Titrate with the sodium hydroxide solution to be standardized.

5. Record the buret reading and apply temperature corrections to both buret readings if required (Remark 4).

## Calculations (Remark 6)

$$N_1 = \frac{V_2 \times N_2}{V_1}$$

where $N_1$ = Normality of sodium hydroxide

$V_1$ = ml. of sodium hydroxide

$N_2$ = Normality of hydrochloric acid

$V_2$ = ml. of hydrochloric acid

## Temperature Corrections

Temperature corrections for standard solutions are calculated as follows:

$$\text{Correction to be added (ml.)} = V \times (e - a) \times (20 - t)$$

where V = observed volume (ml.)

t = temperature of the solution at time of reading.

a = Cubical co-efficient of expansion of glass. For our purpose an average value of 0.00002 is applicable to either soft glass or Pyrex.

e = Cubical co-efficient of expansion of the solution which may be obtained for various strengths of sodium hydroxide solutions from the following table:

| Normality of sodium hydroxide solution | Cubical co-effecient of expansion ml. per ml. per °C. |
|---|---|
| 0 - 0.20 | 0.00025 |
| 0.20 - 0.40 | 0.00027 |
| 0.40 - 0.60 | 0.00029 |
| 1.0 | 0.00034 |
| 1.5 | 0.00037 |
| 2.0 | 0.00040 |

Tables of corrections to be applied to solutions having normalities between 0.20 and 0.40 and 0 and 0.20 have been worked out and will be found in this manual code: SA - 18a.

Remarks

1. Solutions of strength greater than 0.33 N should be stored in paraffin-lined Pyrex bottles stoppered with corks which have been soaked in melted paraffin or with rubber stopperss which have been boiled in 10% sodium hydroxide solution and thoroughly washed with distilled water..

2. It is best to allow sodium hydroxide solutions to age for at least a week with daily shakings before attempting standardization.

3. Sodium hydroxide solutions of strength greater than 0.33 N should not be allowed to stand in a buret as etching of the buret and freezing of the stopcock will occur.

4. If the solutions are at the same temperature and of approximately the same strength no temperature corrections need be applied.

5. The chief important difference between the use of methyl red and phenolphthalein indicators is caused by the presence of small amounts of carbonate in nearly all alkali solutions. When using methyl red indicator this carbonate is titrated and included in the normality of the solution. This yields a value which is not greatly affected by carbon dioxide. In using phenolphthalein indicator the value of the carbonate is not included in the normality of the solution. Variations in normality obtained using the two indicators will be found to be in the order of 0.1%.

6. Duplicate determinations of normality should agree within 0.0002.

7. For best results the acid solution should have a strength of

[redacted]

within a factor of 2 of that of the solution to be standardized. Primary standard 0.33 N hydrochloric acid refers to material prepared directly from constant boiling hydrochloric acid.

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H. W. Laboratory Manual
Laboratory Solutions

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# SODIUM HYDROXIDE
## (50% = 19N)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to handling strong bases

### Quantity and Container

1000 ml. in 1 liter rubber-stoppered wax-lined Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sodium hydroxide pellets, C. P.<br>$NaOH$, C. P. | 760 g. | 760 g. |

### Preparation

1. Dissolve the 760 g. of the sodium hydroxide in about 700 ml. of distilled water in a 1000 ml. beaker and allow to cool.
2. Transfer into a 1 liter volumetric flask, dilute to the mark with distilled water, mix well and let stand until supernatant is clear.
3. Decant of supernatant liquid and transfer into a 1 liter Pyrex bottle wax-lined for storage.
4. Stopper with a cork previously soaked in paraffin or a rubber stopper which was boiled in 10% sodium hydroxide and carefully washed in distilled water.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

| | |
|---|---|
| Code | SS-9a |
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| By: | W.J.M. |

# SILVER NITRATE

## Safety Precautions

1. OObserve general laboratory Safety Rules.

2. Silver nitrate is caustic and poisonous. Care should be exercised while handling the material.

## Quantity and Container

1000 ml. in a 1 liter glass-stoppered dark bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Silver nitrate<br>$AgNO_3$, C. P. | 17 g. | 17 g. |

## Preparation

1. Dissolve 17 g. of the silver nitrate in distilled water in a 1 liter volumetric flask and dilute to the mark with distilled water.

2. Transfer into a 1 liter glass-stoppered dark bottle for storage.

[redacted]

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H. W. Laboratory Manual
Laboratory Solutions

Code: SS-9d

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By: W.J.M.

## SILVER NITRATE

(1 ml. = 0.5 mg. Cl)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Silver nitrate is caustic and poisonous. Care should be exercised when handling this salt.

### Literature Reference

A.P.H.A. "Standard Methods for the Examination of Water and Sewage", A.P.H.A., 34 (1936).

### Quantity and Container

1000 ml. in 1 liter glass-stoppered Pyrex bottle painted black.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Silver nitrate, C.P.<br>$AgNO_3$, C.P. | 2.4 g. | 2.4 g. |

For Standardization

| | | |
|---|---|---|
| 1. | Sodium chloride, 0.001 g. Cl/ml | (Solutions Code: SS-4d) |
| 2. | Phenolphthalein indicator, 1.0% | (Solutions Code: SP-8a) |
| 3. | Methyl orange indicator 0.1% | (Solutions Code: SM-5a) |
| 4. | Sodium carbonate 0.02N | (Solutions Code: SS-3d) |
| 5. | Sodium chromate indicator, 5% | (Solutions Code: SS-5a) |
| 6. | Sulfuric acid, 0.02N | (Solutions Code: SS-23a) |

[redacted]

## Preparation

1. Dissolve 2.4 g. of the silver nitrate in about 50 ml. distilled water in a 125 ml. beaker.

2. Transfer into a 1 liter volumetric flask and dilute to the mark with distilled water.

3. Shake and mix well and transfer into a 1 liter glass-stoppered Pyrex bottle, painted black, for storage.

## Standardization

1. Pipet 10 ml. of the standard chloride solution into a 6-inch white porcelain evaporating dish containing 40 ml. of distilled water.

2. Add 1 drop of phenolphthalein indicator, and if a red color appears neutralize with .02N $H_2SO_4$.

3. Add 1 drop of methyl orange indicator and if a red color appears neutralize with .02N $Na_2CO_3$.

4. Add 1 ml. of sodium chromate indicator.

5. Titrate with the silver nitrate solution until a faint red coloration is perceptable.

6. Calculate the correction due to variations in volume of the liquid as follows:

$$X = 0.003V + 0.02$$

where X = correction in ml. to be deducted from silver nitrate volume.

V = volume of solution after titration.

7. Deduct this correction from the total volume of silver nitrate to determine volume of silver nitrate equivalent to the chloride added.
8. Make final adjustment of the silver nitrate solution such that, after application of the correction, 1 ml. silver nitrate solution is equivalent to 0.5 mg. of chloride radical (0.5 ml. of standard chloride solution).

## Remark

The volume at the start of the titration must be 50 ml. to duplicate conditions of use.

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## SILICA SOLUTION STANDARD
(1 ml. = 0.10 mg. $SiO_2$)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents

#### For Preparation

Sodium silicate commercial grade,
$SiO_2$, 28%

### Preparation

1. Solution A

Dissolve 35 g. of the sodium silicate in a 100 ml. volumetric flask with distilled water and dilute to the mark with distilled water. Solution A is stable for at least 24 hours.

2. Solution B

Pipet 10 ml. of solution A into a 1 liter volumetric flask and dilute to the mark with distilled water. Work rapidly; this solution is very unstable.

3. Solution C

Pipet 100 ml. of solution B into a 1 liter volumetric flask and dilute to the mark with distilled water. Solution C must be made within 5 minutes after the preparation of solution B. Solution C is used for the preparation of the calibration curve for use in analysing treated water and is stable for at least 4 hours.

[redacted]

Standardization

1. Pipet triplicate 25 ml. samples of solution B into platinum dishes and determine silica by Water Methods Code: WS-3a. Calculate ppm silica in solution B.

2. Divide ppm silica in solution B by 10 to obtain ppm silica in solution C.

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|---|---|
| H. W. Laboratory Manual | Code: SS-10d |
| Laboratory Solutions | Reissued: November, 1948 |
| | Status: Standard |
| | By: W.J.M. |

# SILICA SOLUTION
(Approximately 60 ppm)

## Safety Precautions

Observe general laboratory Safety Rules.

## Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sodium silicate, commerical grade $SiO_2$, 28% | 1.3 ml. | 1.3 ml. |

## Preparation

1. Dilute 1.3 ml. of 28% commercial sodium silicate to the mark with distilled water in a 1 liter volumetric flask.

2. Pipet 100 ml. of the solution into another 1 liter volumetric flask and dilute to the mark with distilled water.

3. Transfer into a 1 liter glass-stoppered Pyrex bottle for storage.

## Standardization

Approximately 60 ppm sodium silicate. Check by a colorimetric silica analysis, (Water Methods Code: WS-1b).

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

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## SODIUM PARA PERIODATE
(0.12% Periodate in 11% $H_2SO_4$)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to handling strong acids.

### Quantity and Container

About 2000 ml. in a 2 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Sodium para periodate, C.P.<br>$Na_2H_3IO_6$, C.P. | 1.2 g. | 2.4 g. |
| 2. Sulfuric acid, C.P.<br>$H_2SO_4$, 96% | 60 ml. | 120 ml. |

### Preparation

1. Measure out the 120 ml. of the sulfuric acid and add it slowly with stirring to 1500 ml. of distilled water in a 3000 ml. beaker.
2. Add 380 ml. distilled water.
3. Add the 2.4 g. of the sodium para periodate to the solution.
4. Heat to boiling and keep hot for 30 minutes.
5. Cool and transfer into a 2 liter glass-stoppered Pyrex bottle for storage.

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Code SS-18a

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# SODIUM THIOSULFATE
## (0.1N)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules for handling strong acids.

### Literature References

1. Treadwell, F. P., and Hall, W. T., "Analytical Chemistry", II, John Wiley & Sons, 585-589 (1942).
2. Hillebrand, W. F., and Lundell, G. E. F., "Applied Inorganic Analysis", John Wiley & Sons, 152-154 (1929)

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Sodium thiosulfate, C.P.<br>$Na_2S_2O_3.5H_2O$, C.P. | 25 g. | 25 g. |
| 2. Borax, C.P.<br>$Na_2B_4O_7.10H_2O$, C.P. | 3.8 g. | 3.8 g. |

For Standardization

1. Potassium iodide solution, 10% (Solutions Code: SP-15a)
2. Hydrochloric acid, C.P. HCl, 36%
3. Potassium permanganate, 0.1N (Solutions Code: SP-16a)
4. Starch solution, 1.0% (Solutions Code: SS-21a)

[redacted]

[redacted]

Special Apparatus

Burets, semi-micro, 10 ml., two (Apparatus Code: AB-21a)

Preparation

1. Dissolve the 25 g. sodium thiosulfate in about 300 ml. distilled water in a 1 liter volumetric flask.
2. Add the 3.8 g. of borax. (Remark 1)
3. Dilute to mark with distilled water and mix well.
4. If the solution is turbid, filter, and finally transfer into a 1 liter glass-stoppered Pyrex bottle for storage.

Standardization

1. Place in a 100 ml. Erlenmeyer flask 20 ml. of the 10% KI solution, 20 ml. of distilled water, and 1 ml. of 36% HCl. (Remark 2)
2. Measure accurately from a 10 ml. buret about 8 ml. of standard 0.1N $KMnO_4$ solution and add it to the flask.
3. Let stand in the dark for 5 minutes.
4. Titrate slowly with the sodium thiosulfate to a light straw color.
5. Add 1 ml. of the 1% starch indicator.
6. Continue the titration, using split drops, until the blue color of the starch is just destroyed.
7. Run a blank using the same amounts of reagents but substituting 8 ml. of water for the $KMnO_4$.
8. Repeat steps 1 through 7 using 9 ml. of $KMnO_4$.

Calculation

$$N_1 = \frac{V_2 \times N_2}{V_1}$$

where $N_1$ = normality of sodium thiosulfate

[redacted]

$V_1$ = ml. sodium thiosulfate corrected = ml. in titration - ml. of blank

$N_2$ = normality of 0.1N potassium permanganate

$V_2$ = ml. potassium permanganate used.

Remarks

1. The borax is used to keep the solution sterile. Decomposition of thiosulfate solution is due to bacterial action which converts thiosulfate into sulfate and sulfur. If the solution is made and kept sterile the titer will not change under normal conditions.

2. This standardization should be carried out in a diffused light as iodine is released by the action of direct sunlight.

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SS-18b
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By: W.J.M.

# SODIUM THIOSULFATE
## (0.01N)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sodium thiosulfate, 0.1N<br>(Solutions Code: SS-18a) | about 100 ml. | about 100 ml. |

### Preparation

1. From the exact normality of the 0.1N sodium thiosulfate calculate the volume needed to make 1000 ml. of 0.01N solution as follows:

$$V = \frac{10}{N}$$

where V = volume of 0.1N sodium thiosulfate required.

N = exact normality of 0.1N sodium thiosulfate.

2. Measure out exactly this amount of 0.1N standard solution and place in a 1 liter volumetric flask.
3. Dilute to mark with distilled water and mix well.
4. Transfer into a 1 liter glass-stoppered bottle for storage.

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H. W. Laboratory Manual
Laboratory Solutions

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# SODIUM SOLUTION STANDARD
(1 mg. Na/ml.)

## Safety Precautions

Observe general laboratory Safety Rules.

## Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

## Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sodium chloride, C.P.<br>NaCl, C.P. | 2.5413 g. | 2.5413 g. |

## Preparation

1. Dry the sodium chloride in the 110° C oven for about 30 minutes, then cool in a desiccator.
2. Weigh out exactly 2.5413 g. of dry sodium chloride and transfer into a 1 liter volumetric flask.
3. Dilute the contents of the volumetric flask to the mark with distilled water and mix thoroughly.
4. Transfer into a 1 liter glass-stoppered Pyrex bottle for storage.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SS-21b

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## STARCH SOLUTION (NOT STABILIZED)
## (1%)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature Reference

A.P.H.A., "Standard Methods for the Examination of Water and Sewage", A.P.H.A., 141 (1936)

### Quantity and Container

100 ml. in 100 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Starch, potato | 10 g. | 1 g. |

### Preparation

1. Grind 1 gram of the starch in a mortar with a little water.
2. Pour the paste into 100 ml. of boiling water.
3. Stir while boiling, and then allow to settle overnight.
4. Decant off the clear supernatant into a 100 ml. glass-stoppered Pyrex bottle for storage.

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Laboratory Solutions

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# SULFURIC ACID SOLUTIONS

## Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to handling strong acids.
3. Observe rules pertaining to handling strong bases.
4. The addition of sulfuric acid to water generates large quantities of heat, therefore such additons should be made very slowly and with constant stirring.

## Reagents

### For Preparation

Sulfuric acid, C.P.
$H_2SO_4$, 96%

### For Standardization

1. Sodium hydroxide (Solutions Code: SS-8a)
2. Phenolphthalein indicator, 1% (Solutions Code: SP-8a)
3. Hydrochloric acid, C.P.
   HCl, 36%
4. Barium chloride, 10% (Solutions Code: SB-1a)

## Special Apparatus

1. Buret, semi-micro, 10 ml. (Apparatus Code: AB;21a)
2. Crucible, Gooch, with asbestos mat (Apparatus Code: AC-18a)

## Preparation

1. Calculate the volume of 96% sulferic acid required to make the desired concentration as follows:

[redacted]

a. For normality

$$\text{ml. of } 96\% \; H_2SO_4 = 27.8(A \times B)$$

where A = Desired normality

B = Desired total volume in liters

Example:

To make 1 liter of 5N sulfuric acid
ml. of 96% $H_2SO_4$ = 27.8(5 X 1)

= 139 ml. of 96% $H_2SO_4$

b. For molarity

$$\text{ml. of } 96\% \; H_2SO_4 = 55.5(A \times B)$$

where A = Desired normality

B = Desired total volume in liters

Example:

To make 500 ml. of 3M sulfuric acid
ml. of 96% $H_2SO_4$ = 55.5(0.5 X 3)

= 83.5 ml. 96% $H_2SO_4$

c. For percentage of $H_2SO_4$

$$\text{ml. of } 96\% \; H_2SO_4 = \left(\frac{A}{96}\right)B$$

where A = Desired percentage

B = Desired total volume in ml.

Example:

To make 1 liter of 20% sulfuric acid
ml. of 96% $H_2SO_4$ = $\left(\frac{20}{96}\right)1000$

= 20.8 ml. of 96% $H_2SO_4$

d. For parts

$$\text{ml. of } 96\%\ H_2SO_4 = A(\tfrac{a}{b})$$

where A = Desired total volume in ml.

a = Desired parts of acid

b = Desired parts of acid <u>plus</u> desired parts of water

Example:

To make 500 ml. of 1:3 sulfuric acid

$$\text{ml. of } 96\%\ H_2SO_4 = 500(\tfrac{1}{4})$$

$$= 125 \text{ ml. } 96\%\ H_2SO_4$$

2. Slowly add the required amount of the 96% sulfuric acid to a volumetric flask of the proper size containing at least half of the required volume of distilled water. This work should be done in a well ventilated hood.

3. Mix well and dilute to the mark with distilled water.

4. After the solution is well mixed transfer into a dry, glass-stoppered Pyrex bottle of the proper size for storage.

Standardization

One of the methods given below will be suitable for standardizing sulfuric acid solutions used on this project. It is important in accurate work to select a standardization using the same indicator as will be involved in the subsequent uses of the solution.

A. Against standardized sodium hydroxide, using phenolpthalien indicator.

1. The normality of the sodium hydroxide used should be within a factor of 2 of the sulfuric acid being standardized.

2. Measure accuretyl from a specially calibrated pipet 5 ml. of acid and place in a 50 ml. beaker.

3. Add 2 drops of the 1% phenolphthalein solution.

4. Titrate with the standard sodium hydroxide to a faint pink color.

Calculation:

$$N_1 = \frac{N_2 \times V_2}{V_1}$$

where $N_1$ = Normality of sulfuric acid

$V_1$ = Volume of sulfuric acid, ml.

$N_2$ = Normality of sodium hydroxide

$V_2$ = Volume of sodium hydroxide, ml.

Remarks

If a solution of a certain exact normality is desired, the volume may be adjusted after standardization as follows:

Calculation:

$$V_1 = \frac{V_2 \times N_2}{N_1}$$

where $V_1$ = Total volume to which standardized solution must be diluted to give exact normality desired

$N_1$ = Desired normality

$V_2$ = Original volume minus sum of aliquots taken for standardization

$N_2$ = Actual normality as found by standardization

B. Gravimetric precipitation as barium sulfate.

1. Ignite the Gooch crucible to constant weight and place in a desiccator until ready for use.

2. Pipet 25 ml. of the sulfuric acid into a 400 ml. beaker and add about 300 ml. distilled water.

3. Add 4 ml. of the hydrochloric acid and heat to boiling.

4. Add dropwise with stirring 10 ml. of the barium chloride.

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[redacted]

5. Digest on a steam bath for 1 hour.

6. Filter through the ignited and weighed Gooch crucible, wash thoroughly and heat to constant weight.

7. After calculation, the volume may be adjusted, if desired, as described under Remarks.

Calculation:

$$N_1 = \frac{W_1 \times 1000}{25 \times 116.7}$$

where $N_1$ = Normality of the sulfuric acid

$W_1$ = Weight of the barium sulfate

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SS-23k

Reissued: December, 1948
Status: Standard
By: W.J.M.

## SULFURIC ACID, SILICA FREE
## (1N)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to handling strong acids.

### Quantity and Container

1000 ml. in a 1 liter cork-stoppered paraffin-lined Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Sulfuric acid, 10N<br>(Solutions Code: SS-23a) | 100 ml. | 100 ml. |
| 2. Redistilled water.<br>(Solutions Code: SW-1a) | | |

### Preparation

1. Add slowly 100 ml. sulfuric acid to 900 ml. redistilled water contained in a 1 liter volumetric flask.
2. After cooling, dilute to the mark with redistilled water.
3. Mix thoroughly and transfer into a 1 liter cork-stoppered paraffin-lined Pyrex bottle for storage.

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H. W. Laboratory Manual
Laboratory Solutions

Code: SS-24a

Reissued: November, 1948
Status: Standard
By: W.J.M.

## SULFATE SOLUTION
(1 ml. = 100 micrograms $SO_4$)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

1000 ml. in four 250 ml. glass-stoppered Pyrex bottles.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| Sulfuric acid, 0.04N<br>(Solutions Codes: SS-23a) | 52 ml. | 52 ml. |

### Preparation

1. Transfer into a 1 liter volumetric flask the 52 ml. of the sulfuric acid.
2. Dilute to the mark with distilled water and mix well.
3. Transfer into four 250 ml. glass-stoppered Pyrex bottles for storage.

### Standardization

This method is designed to give a solution containing 100 micrograms of $SO_4$ = /ml.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Reagents

Code: SS-27a

Reissued: December, 1948
Status: Standard
By: W.J.M.

## SULFANILIC ACID
## (0.33N)

### Safety Precautions

1. Observe the general laboratory Safety Rules.
2. Observe rules pertaining to handling strong bases.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Sulfanilic acid, C.P.<br>$C_6H_7SNO_3$, 99.8% | 57.72 g. | 57.72 g. |
| 2. Sodium hydroxide, C.P.<br>NaOH, C.P., pellets | About 40 pellets | |

For Purification and Assay

| | |
|---|---|
| 1. Sodium hydroxide, Standard 0.33N | (Solutions Code: SS-8a) |
| 2. Phenol red indicator, 0.1% | (Solutions Code: SP-5a) |
| 3. Posdered charcoal ("Nuchar" is satisfactory) | |

### Preparation

1. Dry about 60 g. of the sulfanilic acid to constant weight at 135°C.
2. Weigh accurately to the third decimal place into a 250 ml. beaker 57.5-58.0 g. of the dried material.
3. Place in the beaker about 20 pellets of sodium hydroxide.

[redacted]

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4. Fill the beaker about 3/4 full with distilled water.

5. Heat the beaker gently until the sodium hydroxide is dissolved, then bring the temperature to about 80°C.

6. Pour the solution with as much of the undissolved sulfanilic acid as possible into a 1 liter volumetric flask containing about 400 ml. of cool distilled water. Take care not to lose any of the solution or crystals.

7. Add another 20 pellets of sodium hydroxide to the beaker and again fill 3/4 full of distilled water.

8. Heat to about 80°C and dissolve all traces of sulfanilic acid in the beaker. Wash down the sides of the beaker well.

9. Transfer the solution into the flask and rinse the beaker well, adding the rinsings to the flask.

10. Mix until all of the sulfanilic acid has dissolved.

11. Cool the solution to room temperature.

12. Dilute to the mark with distilled water and mix well.

13. Insert a thermometer into the solution and record the temperature at which the dilution was made.

14. Apply temperature correction for 0.33N solutions to the volume of the flask.

Calculations

$$N_1 = \frac{W_1 \times P}{17.318 \times V_1}$$

where $N_1$ = Normality of sulfanilic acid.

$W_1$ = Weight of sulfanilic acid used.

$V_1$ = Corrected dilution volume in ml.

$P$ = Percentage purity of the sulfanilic acid.

Remarks

1. Unless the assay of the sulfanilic acid is accurately known it must be determined as outlined below. Material of less than 99.8% purity should be recrystallized before using.

[redacted]

## Purity of Sulfanic Acid

1. Dry a small portion of sulfanilic acid for several hours at 135°C.

2. Weigh accurately into a 250 ml. beaker 0.50-0.55 g. of the dried sulfanilic acid.

3. Add about 100 ml. of distilled water.

4. Measure into the beaker from a buret 95% of the calculated required amount of $0.33\underline{N}$ standard sodium hydroxide.

5. Boil the sample solution for several minutes to expel carbon dioxide (Remark 1).

6. Remove the beaker from the flame and add 2-3 drops of phenol red indicator.

7. Complete the titration with standard sodium hydroxide.

## Calculations

$$P = \frac{V_1 \times N_1 \times 17.318}{W_1}$$

where $P$ = Percentage purity of sulfanilic acid.

$V_1$ = ml. of sodium hydroxide used

$\underline{N}_1$ = Exact normality of sodium hydroxide used.

$W_1$ = Weight of sulfanilic acid.

## Remarks

1. Since the end point of the titration occurs at about pH 8, any carbon dioxide in the water or standard sodium hydroxide will be neutralized. This carbon dioxide must therefore be removed by boiling just before the solution goes alkaline.

2. Should the purity of the sulfanilic acid be below 99.8% the material should be recrystallized as outlined below.

## Purification of Sulfanilic Acid

1. Measure into a large beaker about 25% more than the amount of sulfanilic acid required.

2. Add about 20 ml. of distilled water for each g. of sulfanilic acid (Remark 1).

3. Add 1-2 g. of powdered charcoal.

4. Boil the mixture for 15-30 minutes adding distilled water to offset evaporation if necessary.

5. Filter quickly while boiling hot through a Buchner funnel containing two layers of Whatman No. 1 filter paper covered with about 1 inch of powdered charcoal. (Remarks 2 and 3)

6. Cool the filtrate to 5°C.

7. Filter off the sulfanilic acid and wash with a little cold water.

8. Allow to dry at room temperature for several hours and finally complete the drying at 135°C (Remark 4).

9. Determine purity as above.

10. The recrystallization may be repeated until a purity of more than 99.8% is attained.

Remarks

1. This is the approximate amount of water needed to form a saturated solution at 90°C which is desirable.

2. Boiling water should be poured through the filter to heat it just before filtering the sulfanilic acid solution. This will minimize loss from crystallization in the filter.

3. A watch glass may be placed on top of the charcoal to prevent it from splattering.

4. If the wet material is placed directly in the oven it will cake badly.

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: SS-27b

Reissued: December, 1948
Status: Standard
By: W.J.M.

# SULFANILIC ACID
## (1%)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to handling acetic acid.

### Literature References

Feigl, F., "Spot Tests", Nordeman, 215 (1939).

### Quantity and Container

100 ml. in 125 ml. glass-stoppered Pyrex dropping bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Sulfanilic acid, C.P.<br>$C_6H_7SNO_3$, C.P. | 10 g. | 1 g. |
| 2. Acetic acid, 30%<br>(Solutions Code: SA-4c) | 1000 ml. | 100 ml. |

### Preparation

1. Weigh out 1.0 g. sulfanilic acid, and transfer into a 250 ml. beaker containing 100 ml. of 30% acetic acid.
2. Gently warm the solution and stir until all the sulfanilic acid has dissolved.
3. Cool solution and transfer into a 125 ml. glass-stoppered Pyrex dropping bottle for storage.

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| H. W. Laboratory Manual | Code: SS-29a |
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| | Status: Standard |
| | By: W.J.M. |

## SODIUM MAGNESIUM URANYL ACETATE
(Saturated in 95% Ethanol)

### Safety Precautions

Observe general laboratory Safety Rules.

### Literature References

Furman, N.H., "Scott's Standard Methods of Chemical Analysis", 5th Ed., I, D. Van Nostrand Co., Inc., New York, 830 (1939).

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Magnesium uranyl acetate solution (Solutions Code: SM-1b) | | |
| 2. Sodium chloride, C.P.<br>NaCl, C.P. | 10 g. | 10 g. |
| 3. Ethanol, C.P.<br>$C_2H_5OH$, 95% | 1 liter | 1 liter |

### Preparation

1. Prepare a 10% solution of sodium chloride by dissolving 10 g. of C.P. sodium chloride in 100 ml. of distilled water.

2. Add the 10% sodium chloride solution, with stirring in a 1500 ml. beaker containing approximately 1000 ml. of the magnesium uranyl acetate solution until no further precipitation of sodium magnesium uranyl acetate occurs.

3. Mix the contents of the beaker thoroughly and allow the precipitate to settle.

[redacted]

4. Decant the supernatant through a Buchner funnel containing Whatman No. 42 filter paper, and finally wash the precipitate onto the filter. Rinse the beaker several times with the supernatant contained in the filtering flask, adding the rinsings to the funnel.

5. Dry the precipitate in a low temperature oven.

6. Dissolve the dried sodium magnesium uranyl acetate in approximately 900 ml. of 95% ethanol until a saturated solution is obtained.

7. Filter the saturated alcoholic solution into a 1 liter glass-stoppered Pyrex bottle for storage.

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Code: ST-5b

H. W. Laboratory Manual
Laboratory Solutions

Reissued: December, 1948
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By: W.J.M.

## TELLURIUM SOLUTION
(10 mg. Te/ml)

### Safety Precautions

1. Observe general laboratory Safety Rules.

2. Tellurium compounds are extremely toxic if taken internally and some are volatile. Use a hood when heating the solutions.

3. Observe rules pertaining to handling strong acids.

### Literature References

Coryell, C.D., Hume, D.N., Ballow, N.E., and Glendenin, I.E., "Clinton Secret Report", CL-CDC-4 (March 15, 1944).

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Tellurium metal, C.P.<br>Te, C.P. | 10 g. | 10 g. |
| 2. Nitric acid, C.P.<br>$HNO_3$, 70% | 10 ml. | 10 ml. |
| 3. Hydrochloric acid, C.P.<br>HCl, 36% | 30 ml. | 30 ml. |
| 4. Hydrochloric acid solution, 3M<br>(Solutions Code: SH-25a) | 1 liter | 1 liter |

### Preparation

1. Place the weighed tellurium metal in a 250 ml. Erlenmeyer flask.

2. Add 10 ml. of nitric acid and heat gently until no more metal appears to dissolve (An insoluble white residue forms).

[redacted]

3. Evaporate to a volume of 2 to 3 ml. by swirling over a burner.
4. Add 10 ml. of concentrated hydrochloric acid and evaporate to a volume of 2 to 3 ml. (The white residue should dissolve).
5. Repeat Step (4) twice.
6. Transfer into a 1 liter volumetric flask and dilute to the mark with 3M hydrochloric acid.
7. Filter the solution if it is not clear.
8. Transfer into a 1 liter glass-stoppered Pyrex bottle for storage.

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| H. W. Laboratory Manual | Code: ST-8a |
| Laboratory Solutions | Reissued: November, 1948 |
| | Status: Standard |
| | By: W.J.M. |

## THIOGLYCOLIC ACID (NEUTRALIZED)
(1%)

### Safety Precautions

Observe the general laboratory Safety Rules.

### Quantity and Container

1000 ml. in a 1 liter glass-stoppered amber bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Thioglycolic acid, C.P.<br>$C_2H_4O_2S$, C.P. | 10 ml. | 10 ml. |
| 2. Ammonium hydroxide, C.P.<br>$NH_4OH$, C.P. | | |

### Preparation

1. Measure out 10 ml. of the thioglycolic acid into a 1 liter volumetric flask.
2. Add the ammonium hydroxide until the solution is neutralized, then add 1 ml. in excess. It should just turn red litmus blue.
3. Dilute the solution to the mark with distilled water, and mix thoroughly.
4. Transfer the solution into a 1 liter glass-stoppered amber bottle for storage.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Solutions

Code: ST-9a

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By: W.J.M.

## TITAN YELLOW INDICATOR
(0.05%)

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

500 ml. in a 500 ml. glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. Titan yellow, commercial dye | 0.50 g. | 0.25 g. |

### Preparation

1. Dissolve the 0.25 g. of the titan yellow in 300 ml. of distilled water in a 500 ml. volumetric flask.

2. Dilute to the mark with distilled water and transfer into a 500 ml. glass-stoppered Pyrex bottle for storage.

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H. W. Laboratory Manual
Laboratory Solutions

Code: ST-15a

Reissued: November, 1948
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By: W.J.M.

## ORTHOTOLIDINE
(0.1%)

### Safety Precautions

1. Observe general laboratory Safety Rules.
2. Observe rules pertaining to handling strong acids.

### Quantity and Container

1000 ml. in 1 liter glass-stoppered Pyrex bottle.

### Reagents and Specifications

| For Preparation | Per Liter | Total |
|---|---|---|
| 1. o-Tolidine, C.P.<br>$C_{14}H_{16}N_2$, C.P. | 1 g. | 1 g. |
| 2. Hydrochloric acid, 1:4 HCl<br>(Solutions Code: SH-25a) | 500 ml. | 500 ml. |

### Preparation

1. Place 1 g. of o-tolidine in a large mortar and add 5 ml. of 1:4 hydrochloric acid.
2. Grind to a thin paste and add 150 ml. of distilled water.
3. Stir until the o-tolidine is dissolved.
4. Transfer into a 1 liter graduated cylinder and dilute with distilled water to 505 ml.
5. Add 495 ml. of 1:4 HCl to make volume 1000 ml.
6. Transfer into a 1 liter glass-stoppered Pyrex bottle for storage.

[redacted]

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H. W. Laboratory Manual
Laboratory Solutions

Code: ST-23a

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By: W.J.M.

# TURBIDITY SOLUTION STANDARD

(100 ppm)

## Reagents and Specifications

### For Preparation

Standard solution purchased from Eimer and Amend.

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Code: SW-1a

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Laboratory Solutions

Reissued: December, 1948
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By: W.J.M.

## REDISTILLED WATER

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

5000 ml. in a 5 liter glass-stoppered Pyrex bottle.

### Reagents

#### For Preparation

Distilled water, ordinary laboratory reagent originally distilled and condensed in block tin.

### Special Apparatus

Glass still (Apparatus Code: AS-19b)

### Preparation

1. Fill the boiler of the still about 3/4 full with distilled water.

2. Bring the water to a boil with an electric heater or a gas flame, and distil off about 300 ml. water into a 500 ml. beaker.

3. Continue the distillation, collecting the condensate in a 5 liter glass-stoppered Pyrex bottle, reserved exclusively for this purpose.

4. When the boiler is nearly dry, refill with distilled water. Continue the distillation until approximately 5 liters of water have been collected.

[redacted]

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H. W. Laboratory Manual
Laboratory Solutions

Code: SW-
Reissued: December, 1
Status: Stand
By: W.J.

## REDISTILLED WATER, SILICA FREE

### Safety Precautions

Observe general laboratory Safety Rules.

### Quantity and Container

5000 ml. in a 5 liter cork-stoppered paraffin-lined Pyrex bott

### Reagents

#### For Preparation

Distilled water, ordinary laboratory reagent, originally distilled and condensed in block tin.

### Special Apparatus

Still, with glass boiler and block tin condensor.

### Preparation

1. Fill the boiler of the still about 3/4 full with distilled water.
2. Bring the water to a boil with an electric heater or a gas flame, and distil off about 300 ml. water into a 500 ml. be
3. Continue the distillation, collecting the condensate in a 5 liter, paraffin-lined cork-stoppered Pyrex bottle, reserved exclusively for this purpose.
4. When the boiler is nearly dry, refill with distilled water. Continue the distillation until approximately 5 liters of w er have been collected.
5. Cork the bottle with a cork previously soaked in paraffin.

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## H. W. LABORATORY MANUAL

## 100 AREA APPARATUS

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| AT-21b | Thermometer, ASTM E1, -5°C to 300°C | 1 |

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H. W. Laboratory Manual
Laboratory Apparatus

Code: AB-5a
Reissued: January, 1949
Status: Standard
By: W.J.M.

# BECKMAN pH METER MODEL G

## Uses

1. The meter is used for measurement of pH of many types of solutions.
2. It may be used for measurement of various E.M.F. of various types of systems where fitted with suitable electrodes.

## Manufacturer

National Technical Laboratories, Beckman Instruments Division, South Pasadena, California.

## Advantages

1. Simply constructed and designed for rugged use.
2. Small and compact construction.
3. Semimicro electrodes permit measurements on small volumes of solution.
4. Simple and rapid in operation.

## Disadvantages

1. Lacks high accuracy when compared to standard potentiometric methods of measuring pH.
2. Does not give straight line calibration over entire range.
3. Electrode system is relatively fragile.

## Discussion

The following paragraphs are intended only as a guide to the use of the instrument in the laboratory. A complete discussion of the theory of electrometric measurements will be found elsewhere in this manual and in the literature references cited.

[redacted]

## A. Measurement of pH

### 1. Choice of Electrodes

pH measurements on the Beckman are generally made with a glass and calomel electrode. Although various size electrodes are available the No 290 and 270 electrodes are used since they are able to measure the pH of a relatively small volume of liquid.

2. The glass electrode should be protected from any shock or direct physical contact with another object. The glass bulb, or membrane, is fragile, and a small crack in it is enough to obviate measurements.

Whenever a new electrode is placed in use, the bulb should be allowed to soak in distilled water at least 12 hours. Once placed in use, it should be kept in distilled water when not in use. If an electrode is once used and then put away for a long storage period, it should be thoroughly rinsed with distilled water and allowed to dry before storing in a suitable container.

The calomel electrode is much sturdier than the glass electrode, and is less subject to breakage. It need not be soakod before use although it should be inserted in water for 5 to 10 minutes before use. It containes a saturated potassium chloride solution which continually, though very slowly, seeps out of the electrode. It must be refilled from time to time. Potassium chloride crystals should always be present in the electrode.

### 3. Measurement

a. Put the operating lever in the (1) position.

b. Set the range switch to "pH".

c. Set the temperature compensator to the temperature of the test solution.

d. Adjust the milliammeter needle to zero by rotating knob 1.

e. Hold the operating lever in position 2, and adjust the needle to zero again with the knob 2. (This adjustment must be made each time a change is made in the temperature compensator.)

f. Allow the operating lever to return to the vertical position.

g. Fill the test dish with a solution of known pH. A buffer of pH 7.0 is usually supplied with the Beckman instruments, but any solution with known pH between 3.0 and 9.0 will be satisfactory.

h. Adjust the slide wire scale to the pH of the solution.

i. Press the push button in the center of the slide wire knob down. If the needle deflects from zero, turn the "zero adjuster" with a small screw driver until the needle is zeroed. (This adjustment should be made frequently for precise work, but need be done only about every 8 hours in most work.)

j. Remove the electrodes from the solution, rinse thoroughly with distilled water and dry lightly with absorbant tissue.

k. Immerse the electrodes in the solution whose pH is to be determined. Thissolution may be contained in the test dish or in another container.

l. Hold down the push button and rotate the slide wire to zero the needle. Release the button.

m. If the needle does not remain at zero, zero it with knob 1 and repeat step l. Read the pH of the solution directly from the scale.

n. If the pH determined is above 9.5 use a type E electrode and repeat the measurement.

## A. Use of the Instruement as a Potentiometer.

Although the Beckman pH meter cannot be compared to a good potentiometer for measurement of absolute E.M.F. values, it is occasionally of value in locating the end point of a potentiometric titration. The instrument is compact, rugged and easier to operate than most potentiometers.

### 1. Choice and Care of Electrodes

The choice of electrode for a potentiometric titration is entirely dependent uppn the type of titration. In many acid base titrations the regular glass-calomel setup can be used.

In a large number of other cases a platinum electrode may be substituted for the glass electrode. A platinum electrode for use with the instrument is sold by the National Technical Laboratories. Other types of electrodes may be used also.

Care of the glass and calomel electrodes has been described above. A platinum electrode should be cleaned with hot chromic acid and rinsed with distilled water before use. A tungsten electrode should be heated to a dull red heat and then drawn quickly over a lump of fused sodium nitrite. Silver electrodes should be dipped in dilute nitric acid until they gas freely, then rinsed with distilled water. For more details on the care of individual electrodes, see the individual apparatus writeups covering them.

2. E.M.F. Measurements.

   a. Connect the two electrodes to the jacks in the electrode compartment. Connect the positive half cell to the upper jack.

   b. Set the range switch to "+MV".

   c. Carry out the balancing exactly as described under the measurements of pH but omit steps 7, 8, and 9. The scale reads 0 to 1.300 volts, the smallest subdivision being 0.01 V.

   d. If the instrument cannot be balanced, set the range switch at "-MV", and repeat step 3, If it cannot still be balanced it indicates the voltage of the cell is beyond the range of the instrument. In this case, the meter is not applicable to the system, and a regular potentiometer must be substituted for it.

## Care of the Instrument.

Like any fine instrument, the pH meter deserves the best possible care. It should be protected at all times from fumes and from electrical and mechanical damage. It contains a standard cell, and should not be inverted. The cover, which may be removed for convenience in making measurements, should always be replaced when the instrument is not in use. If the instrument is not to be used over a long period of time, the electrodes should be removed, cleaned and stored.

1. Replacement of the Batteries

   The Beckman is equipped with several dry batteries. Failure of these batteries is indicated by inability to balance the instrument with the 1 and 2 knobs. They should be replaced as follows:

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a. The A batteries are Burgess Batteries No. 4FH. This or an equivalent battery should be used for replacement.

b. The B battery is a 22 1/2 Burgess No. 5156.

c. The C batteries are Burgess No. 5540, 7 1/2 V.

2. Replacement of the Amplifying Tube

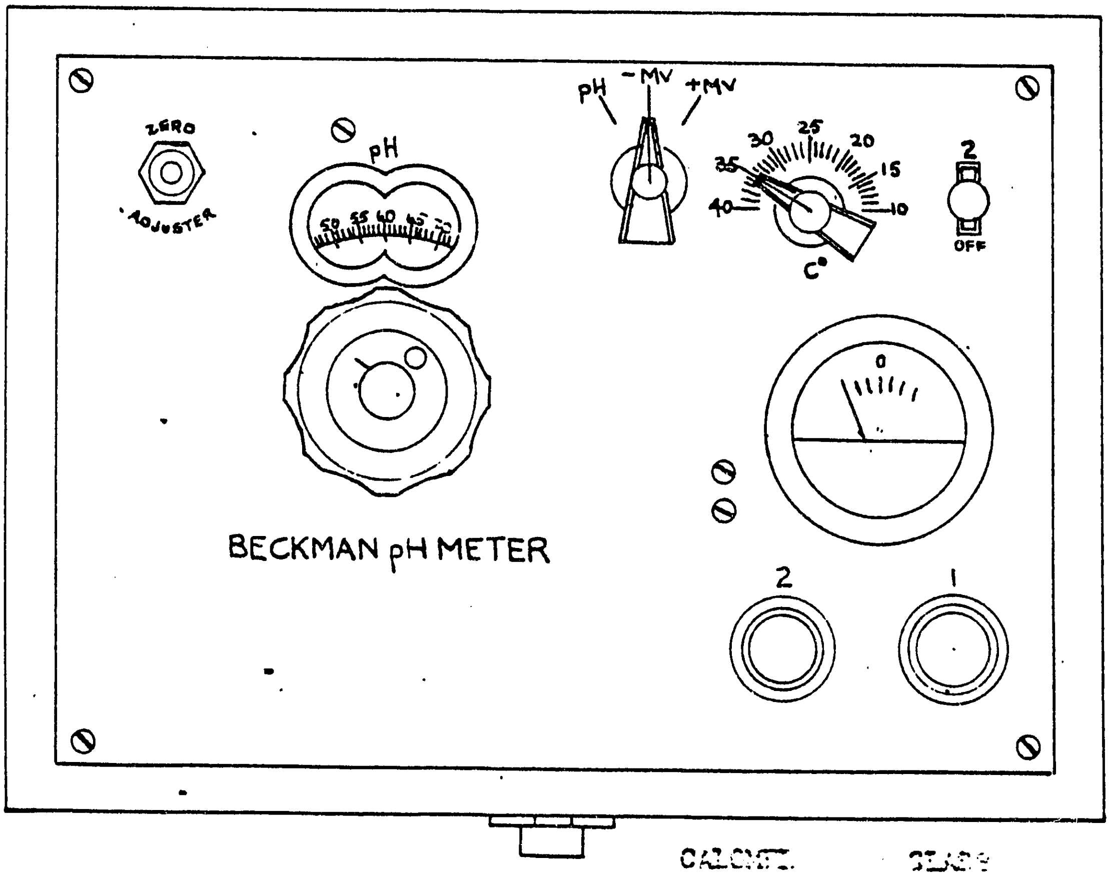
a. The amplifying tubes are operated far under their rated capacity and should not require replacements over long periods of time. If replacement is necessary, consult supervision who will contact the proper repair man.

3. Replacement of Electrodes

a. The replacement of the electrodes is a frequently performed, simple operation. New electrodes need only be inserted when the old electrodes have been physically damaged. Most frequent source of trouble is a small crack on the glass electrode. This is indicated by erratic and drifting pH values. It is most easily confirmed by measuring the pH of a known solution.

[redacted]

Code: AZ-5a



CALOMEL
ELECTRODE

GLASS
ELECTRODE

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H. W. Laboratory Manual
Laboratory Apparatus

Code: AB-5b

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## INSTRUCTIONS FOR BECKMAN MODEL H, AC OPERATED pH METER

### Installing the Electrodes

1. Glass Electrode Soak a new glass electrode several hours in water. If time does not permit this, the new electrode may be used with satisfactory accuracry if buffer standardization is repeated frequently. Insert the glass electrode terminal in the upper terminal jack at the rear of the instrument and tighten clamping screw.

2. Calomel Electrode Remove the rubber protector from lower end, wipe the electrode and shake out any air bubbles trapped in the lower end. Electrical contact between the electrode and the test solution is established by a small porous fiber sealed into the lower tip. Replenish the saturated KCl solution through the side tube when necessary--LEAVE THIS TUBE OPEN DURING MEASUREMENTS. Insert the pin terminal in the pin jack below the glass electrode terminal jack and tighten clamping screw.

3. Electrode Assembly· Mount the electrodes in their holder so that the calomel electrode projects slightly lower than the glass electrode and be sure that they cannot touch the bottom of the beaker or sample container when the holder is lowered against the rod support.

### Using the pH Meter

4. Warm-up Set the range switch on NEUTRAL and be sure that the push-botton switch is in the IN position. (Press and rotate), Connect the instrument to the power line and allow a five minute warm-up period before proceeding with the measurements.

   While range switch is on NEUT. the meter needle should always read exactly 7 pH; if necessary adjust the needle position with the screw just over the meter pivot.

5. Standardization Against Buffer After turning on the instrument as described in section 4, place some fresh buffer in a clean beaker and immerse the electrodes. Set TEMPERATURE knob to the temperature of the buffer; switch to proper range and release push-button. By use of the STANDARDIZATION control set the meter needle to the pH of the buffer.

[redacted]

Important: The exact pH of the buffer will depend upon temperature as indicated on the buffer label. Standardization should always be performed with buffer at a temperature within $10^{\circ}$C of the solutions to be tested.

Lock IN the push-button and set the pointer to mark the position of the meter needle. If meter reads off scale, switch to the other range before setting the pointer, Use this range position when subsequently resetting needle to pointer position with standardization control. This procedure is used to correct for any changes in the amplifier which may occur subsequent to standardization. (cf. Section 6).

It is essential to standardize against a buffer solution daily. For most accurate work, checks should be made before and after each series of tests.

6. Measuring pH of Samples With the push-button locked IN, spray the electrodes with distilled water and wipe them with absorbent tissue. Clean the beaker and half fill with test sample. Immerse the electrodes and if necessary use the standardization control to bring meter needle into coincidence with the pointer. Release the push-button and the meter will indicate the pH automatically. If the meter reads off scale switch to the other range.

   The push-button may be left OUT to read pH as long as the electrodes are immersed but should preferably be locked IN when the electrodes are being handled or are not immersed. This procedure will prevent the possibility of polarization of the glass electrode which will be apparent if the readings drift for several minutes after re-immersion.

Operation Suggestions

7. a. When making measurements at temperatures much different from room temperature it is desirable to work with larger quantities of solution--at least 250 ml.--so the temperature will not change during the measurement. To obtain the most accurate results, time must be allowed for the electrodes to reach the temperature of the solution, and they should be immersed almost their whole length in the solution. Care must be exercised however to keep the top portion of the electrodes dry to prevent electrical contact of the solution with any metal parts of the electrodes or the electrode holder.

b. It is very desirable to standardize the meter at a temperature close to that of the solutions to be tested (at least within 10°C) because of small potentials associated with the electrode which fail to follow exactly the temperature correction provided. At a given temperature the effect of these small potentials is eliminated by the use of STANDARDIZATION control in the standardization procedure.

c. When measuring pH of poorly buffered solutions it is usually worthwhile to rinse both the electrodes and beaker with the solution to be tested before finally filling the beaker and making the measurement.

d. Polarization of the glass electrode may occur if either or both of the electrodes are removed from the solution without first depressing the push-button. To avoid this possibility the push-button should normally be left in the IN position and released only while readings are actually being made.

Such polarization has no permanent harmful effect on the electrode but temporarily causes the pH readings to be high by a few tenths of a pH unit. The effect will disappear within a few minutes (one to five minutes) depending upon the extent of the polarization and the temperature of the electrode. Cold electrodes (below 20°C) are most liable to polarization effects while at temperatures above 50°C the effect is virtually absent.

## Measuring Oxidation Reduction Potentials and E.M.F.'s

8. Set the TEMPERATURE control at 25°C. and the range switch at 7 to 0 pH. With the push-button IN, use the STANDARDIZATION control to bring the meter needle to zero on the milivolt scale. Insert connections from the source to be measured and release the push-button. The meter will then read directly in milivolts. If the meter reads off scale below zero, the lead connected to the glass electrode terminal is negative instead of positive. If this happens the range switch can be changed to the 7 to 14 pH position and the reading made without reconnecting the terminals. Between measurements the push-button should be locked IN, and, if necessary, the STANDARDIZATION control should be used to bring the needle to zero on the milivolt scale before releasing the push-button for a reading.

Since the TEMPERATURE control affects the sensitivity of the instrument it is possible to extend the range of the milivolt scale to approximately 520 milivolts by setting the TEMPERATURE control to 100°C and multiplying the milivolt reading by 5/4 (e.g. 5/4 x 414 = 517.5 mv.).

Maintaining The pH Meter and Electrodes

9. a. Electrodes The KCl solution in the calomel electrode should never be allowed to dry out or become unsaturated. Separation of the inner calomel-mercury column should not effect the accuracy of the measurement. When not in use the electrodes should be immersed in water. The electrodes should be kept clean. If the calomel electrode is to be stored for an extended period, the rubber protector on the end should be replaced.

b. Fuses The power plug accomodates two, 1 ampere, 3 AG fuses which are available from radio supply dealers, automobile supply houses, etc. To replace, force the fuse out from the end with a pencil point or similar object.

c. Tubes With the exception of the #932 Electrometer Tube, all tubes are standard and can be obtained from any radio supply dealer. The #932 tube has been specially aged, processed and selected and is available only from Beckman Dealers or National Technical Laboratories, South Pasadena, California.

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CODE: AB-5b

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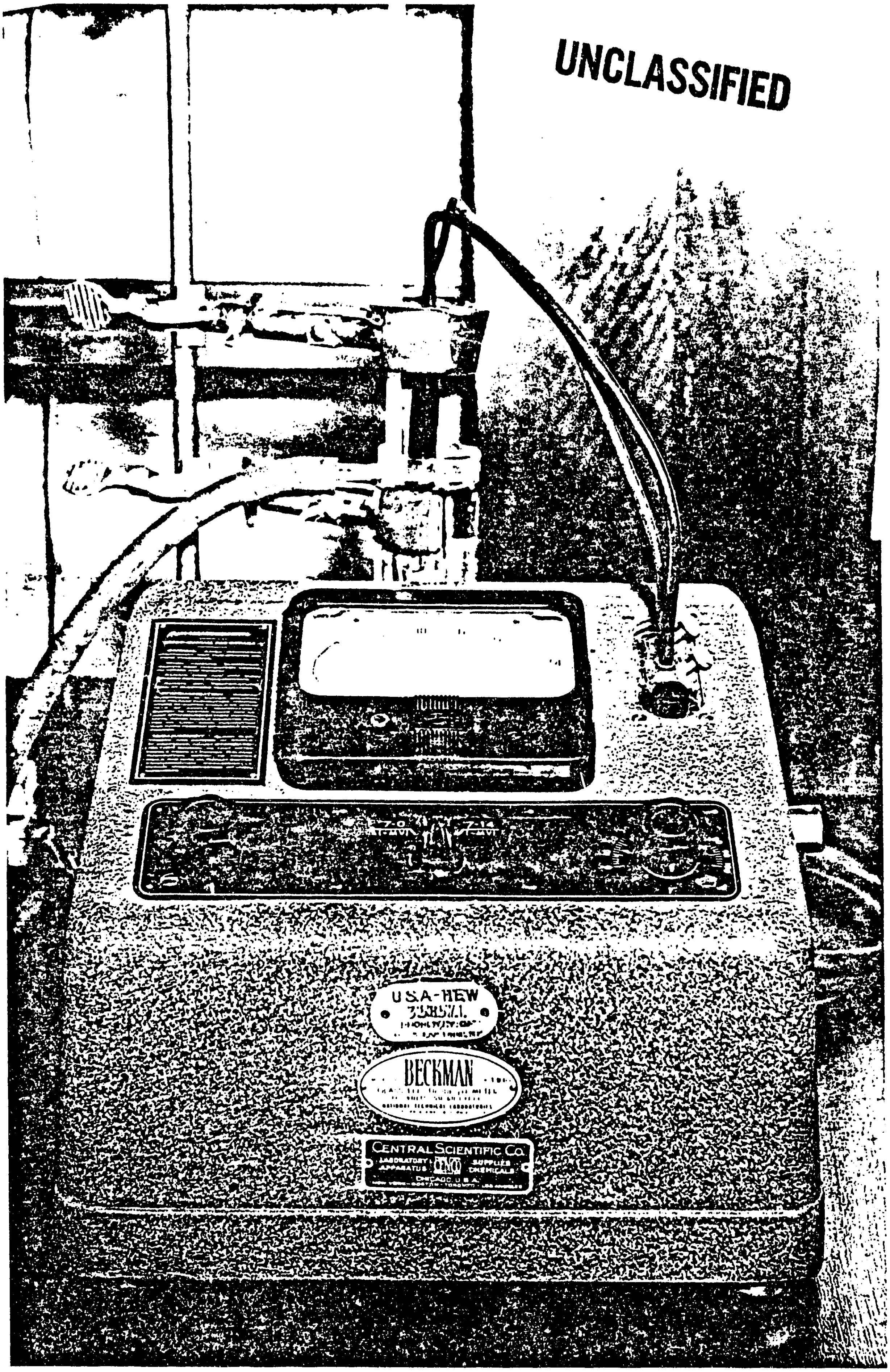

BECKMAN pH METER, MODEL H

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H. W. Laboratory Manual
Laboratory Apparatus

Code: AB-12a
Reissued: January, 1949
Status: Standard
By: W.J.M.

# BOTTLE DISSOLVED OXYGEN

## Uses

This bottle is used in the determination of dissolved oxygen in water.

## Manufacturers

| | | |
|---|---|---|
| 1. | Fisher Scientific Company | Catalogue No. 2-926 |
| 2. | Burrell Technical Supply Company | Catalogue No. A83-900 |

## Advantages

1. Cone shaped stopper prevents the entrapment of air below it.
2. Flared lip gives water seal after filling to prevent air being drawn into bottle.

## Literature References

1. A.P.H.A. "Standard Methods for the Examination of Water and Sewage".
2. Public Health Reports, Supplement No. 90.

## Description

The bottle as shown in the sketch is of glass and has a capacity of 250 to 300 ml. The stopper is accurately ground and is cone shaped to prevent air from being trapped beneath it when the bottle is closed. The neck of the bottle is flared and extends past the stopper to provide a water seal thus preventing air from being drawn into the bottle after sampling.

## Preparation for Use

A bottle of this type may be purchased or can be prepared from an 8 oz. tincture bottles by grinding the bottom of the stopper to a conical point.

## Use

1. The bottle is filled with sample to the very top as specified

[redacted]

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by the A.P.H.A. "Standard Methods of Examination of Water and Sewage".

2. The stopper is carefully inserted, displacing the liquid in the neck, thus leaving no air space above the sample.

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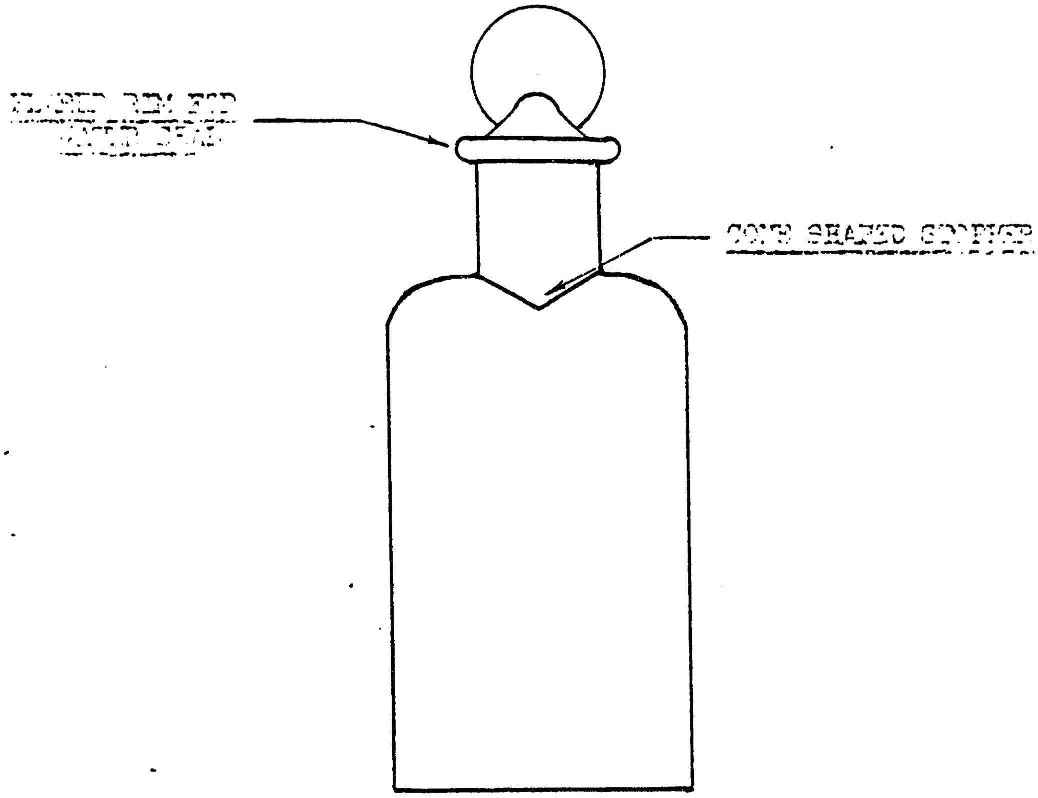
Code: AE-12A

# DISSOLVED OXYGEN BOTTLE



CAPACITY 250 ML

ALL GLASS CONSTRUCTION

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[redacted]

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[redacted]

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Laboratory Apparatus

Code: AB-14a
Reissued: January, 1949
Status: Standard
By: W.J.M.

## BOTTLE, RESERVOIR
(1000 ml. capacity)

### Uses

This bottle is used as reservoir for a 10 ml. semi-micro buret, Apparatus Code: AB-21a.

### Manufacturers

Eck and Krebs.

### Advantages

Interchangeable

### Description

This Pyrex bottle, of 1000 ml. capacity, is fitted with a ST 29/42 glass joint and may be fitted with glass stopper. It is interchangeable with the reservoir of 10 ml. semi-micro burets Apparatus Code: AB-21a.

### Use

This bottle is used for the storage of standard solutions as well as a buret reservoir. Consequently, solutions can be changed by changing the reservoir and flushing the buret thus eliminating the transfer of the liquid.

[redacted]

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Laboratory Apparatus

Code: AB-21a
Reissued: January, 1949
Status: Standard
By: W.J.M.

## BURET, SEMI-MICRO, 10 ML.

### Uses

This buret is used for the accurate delivery of standard solutions in volumes of 10 ml. or less.

### Manufacturers

Eck and Krebs.

### Advantages

1. Volume readings to 0.002 ml. are possible.
2. Standard reagents are conserved by returning excess solution to the reservoir.
3. Space and reagents are saved by using semi-micro scale.

### Disadvantages

1. Fine delivery tube is apt to clog with dust or dirt.
2. Small samples or aliquot samples must be used.
3. Excess reagent returned to reservoir.

### Description

This buret, as shown in the attached sketch, is of all Pyrex construction 96 cm. in overall length. The graduated portion is 52 cm. long and is calibrated and accurate at all points to 0.01 ml. or better. The buret is surrounded by an evacuated jacket free of water vapor. A one liter bottle, Apparatus Code: AB-14a is supplied as a reservoir.

### Preparation for Use

1. Fill the reservoir with standard solution.
2. Turn the stopcock to connect the reservoir and buret.
3. Apply air pressure until the buret is filled above the mark.

[redacted]

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4. By controlling the stopcock, return the excess solution to the reservoir until the meniscus is on the mark.

5. Place receiving vessel underneath the buret tip.

6. Turn stopcock to deliver solution to tip.

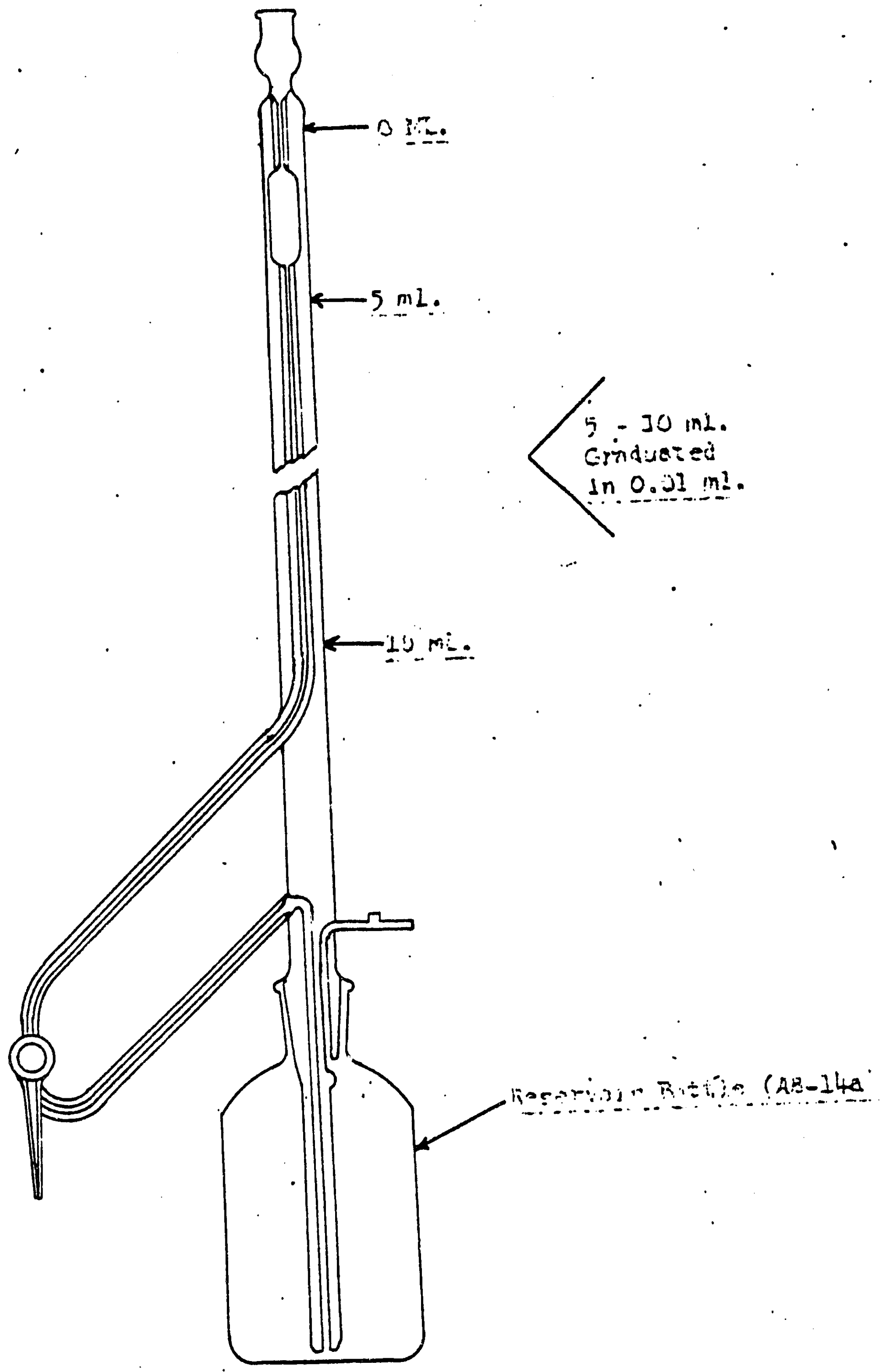
Remarks

Never leave the buret empty or partially empty. Always fill the buret to above the zero mark. Almost all chemical solutions corrode glass to some extent and this corrosion is greatest at the interface of the liquid. By filling the buret to above the zero mark this corrosion is kept above the calibrated portion.

[redacted]

Code: AB-21a

Buret 10 ml



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Laboratory Apparatus

Code: AC-5a

Reissued: January, 1949
Status: Standard
By: W.J.M.

## FLOW CELL FOR pH MEASUREMENTS

### Uses

The flow cell is used for taking pH of water samples that are unstable with regard to dissolved gases and therefore to pH.

### Manufacturers

Fabricated on the plant site.

### Advantages

1. Convenient to use.
2. Prevents deviation of the water sample.

### Disadvantages

Relatively fragile.

### Discussion

Consistent pH readings can be obtained only when a flow cell of some type is used. Figure 1 and 2 represents types which have proven satisfactory over a period of time.

### Description

The flow cells are fabricated from ordinary glassware of the dimensions given in the attached drawings. In the cell shown in Figure 1, the water sample flows into the cell through the bottom right angle tube and overflows at the top of the cell walls. In Figure 2, the water sample enters the cell as in Figure 1, but exits through the side-arm attachment.

### Operation

Use a Beckman Model G or L & N portable pH meter equipped with standard electrodes and an auxillary flow cell attachment. Standardize the pH meter by the established method using 0.05M potassium acid phthalate solution. This solution should be prepared from the stable 0.2M stock solution by diluting daily. Fill the flow cell with the phthalate buffer solution and

[redacted]

determine the temperature. Any temperature between 1°C and 30°C may be used. Set the temperature compensator on the instrument at the temperature of the buffer solution. Adjust the instrument at the No's 1 and 2 position and use it for determining the proper setting according to the buffer. Adjust the instrument and use it for determining the pH of the process water in the usual manner. Flow process water through the cell until it is thoroughly washed and set the temperature compensator at the temperature of the water to be tested and take the pH. Repeat until duplicate readings are obtained. Check the electrodes to make certain that the glass electrode is suspended in the solution just enough to completely cover the bulb, and that the calomel electrode tip is 1/4 inch beneath the surface of the solution.

After using the pH meter for determining the pH, wash the flow cell with distilled water and leave filled with distilled water with the electrodes dipping in the water.

Use

Use as directed in Water Methods Code: WP-1a.

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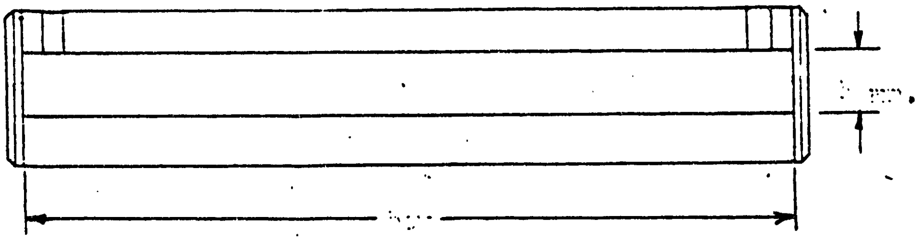
Code: [illegible]

Contents (Class)



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H. W. Laboratory Manual
Laboratory Apparatus

Code: AC-21f

Reissued: January, 1949
Status: Standard
By: W.J.M.

## ABSORPTION CELLS
(5 cm. Plastic, 4 mm. I.D.)

### Uses

Used in the Beckman spectrophotometer to hold the solution whose transmission is being measured. Use only when the volume of the solution available is less than 10 ml.

### Advantages

1. Relatively long length (5 cm.) combined with a very small volume (0.6 ml.).
2. Interchangable end windows permit use with most aqueous and non-aqueous solutions, including those containing hydrofluoric acid.
3. Cells are easier to fill and handle than corresponding all-glass cells.
4. Recessed end windows easy to keep clean, and difficult to damage except by deliberate action.
5. Uses same adapter as other types of cells, notably the 5 cm. Aminco (AC-21d).

### Disadvantages

Windows, held in place mechanically, may be source of leaks.

### Literature References

Rosenfels, Kirk, Kitson, Patton and Curtis, CN Report.

### Notes on the Care and Use of Capillary Absorption Cells

Cells of this type are a recent development, and have not been tested and evaluated thoroughly. In general, the notes on handling other absorption cells apply (Apparatus Code: AC-21e). The following points will be found useful.

1. The only convenient way to clean the optical faces is to unscrew the ends and remove the small glass end windows.

[redacted]

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# FLOW CELLS FOR BECKMAN pH METER

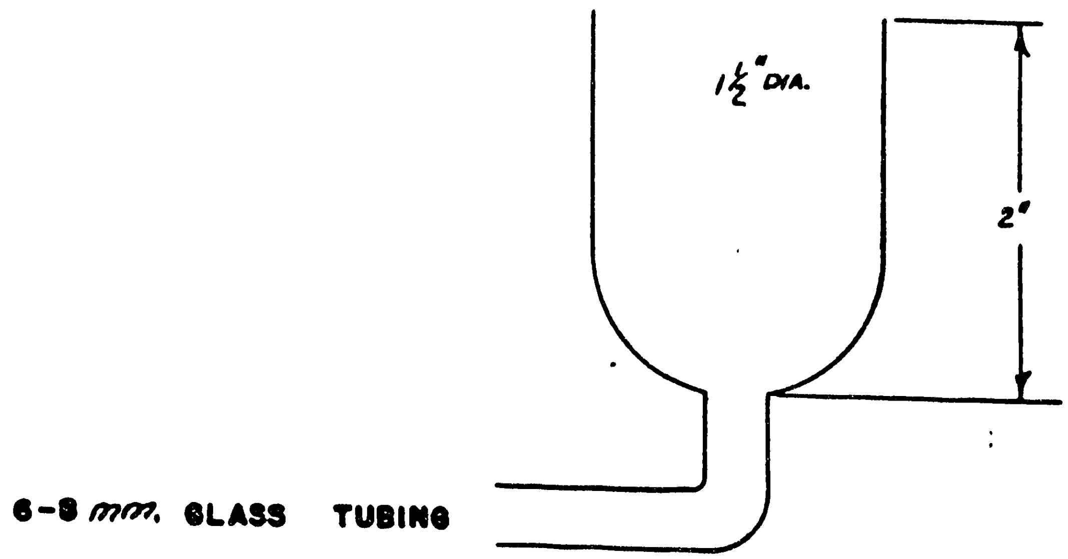

FIG. 1

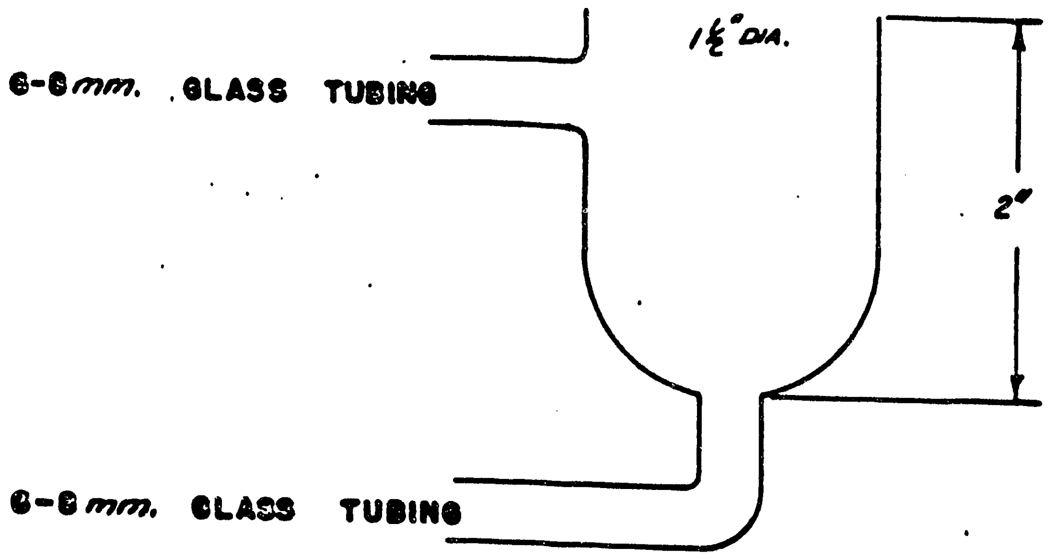

FIG. 2

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H. W. Laboratory Manual
Laboratory Apparatus

Code: AC-7a
Reissued: January, 1949
Status: Standard
By: W.J.M.

# TAYLOR CHLORIMETER

## Uses

Used in the rapid determination of free chlorine in such samples as water, sewage, industrial wastes, etc.

## Manufacturer

Distributed by Burrell Technical Supply Co., Pittsburgh, Pa., Cat. No. A 83-600.

## Advantages

1. Compact, simple, and rugged design.
2. Standards nearly permanent.
3. Simple to use.

## Disadvantages

Standards occasionally not accurate or permanent.

## Discussion

The comparator is shown in the attached cut.

### 1. Description

The comparator consists of two principle parts, a base and a slide, both molded of black plastic. The base contains a run for the slide, and holes, near the center, for two reagents bottles and three small test tubes or cells. The slide contains a series of solutions in sealed ampules. Alternate ampules contain water, while the other tubes contain a colored solution, whose color corresponds to that developed by definite amounts of free chlorine in water and/or the tolidine reagent. The slide is built to cover the range 0.1 to 1.0 ppm free chlorine.

[redacted]

## II. Theory

The theory of the comparator is simple. The ortho tolidine reagent is so prepared that when the water sample is added to the tube provided, only 1/2 ml. of the reagent must be prepared to secure color development. Three tubes are provided, and each of these is filled with the sample to be analyzed. The color is developed in the center tube only, however. Then the slide is moved into position, and the color of the various solutions viewed through it. When a blank position on the slide is over the center tube, two of the color standards, one on either side, are over the tubes filled with sample but without reagent. Thus the light coming through the center tube is absorbed to some extent by (a) the color of the sample, (b) the color due to the free chlorine in the water, and (c) the color of the clear water in the two tubes. The light coming through either side comes through the same absorbers, (a) the color of the sample itself, (b) the color of the standard, corresponding to a certain amount of chlorine, and (c) the color of clear water in the two tubes. Only difference is that in the case of the unknown, absorbers (a) and (b) are in one tube; and in the case of the standards, absorber (a) is in the first tube, and (b) is in the second. Thus correction is automatically made for the color of the sample. The position of the slide should be varied until the color of the unknown is the same as one of the standards, or between two consecutive standards. The chlorine concentration is then read from the slide. In the case where the color lays between, average the two values on either side.

## III. Operation

1. Fill the three cells provided with the instrument to the mark with the water sample and place in the slots provided in the rack.

2. To the center cell, add 0.5 ml. of ortho tolidine.

3. Mix the contents of the center cell well, replace the cell in its slot and let stand for exactly two minutes.

4. Move the slide of the comparator, until the color of the center tube is matched by one or is between two, of the standards.

5. Report in ppm of chlorine the value of the standard which most nearly matches the sample.

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Code AC-7a

## TAYLOR CHLORIMETER

[redacted]

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H. W. Laboratory Manual
Laboratory Apparatus

Code: AC-18a
Reissued: January, 1949
Status: Standard
By: W.J.M.

# CRUCIBLE, GOOCH, WITH ASBESTOS MAT

## Uses

This type of filtering medium is used where no further treatment of the precipitate is necessary, or where the presence of organic material (paper) is undesirable.

## Manufacturers

Coors Porcelain Company

## Advantages

1. Eliminates necessity of burning off paper and correcting for ash.

2. Provides a control over the filter medium.

## Disadvantages

The crucible must be prepared for each determination.

## Literature References

1. Hillebrand and Lundell "Applied Inorganic Analysis" John Wiley and Son, 91 and 92 (1929).

2. Treadwell and Hall "analytical Chemistry", John Wiley and Son, 30 and 31 (1942).

## Description

A Gooch crucible is a porcelain container having a perforated bottom which for use is covered by an asbestos mat. A perforated porcelain plate is sometimes used to cover the mat to prevent dislodgement of the fibers.

## Preparation for Use

1. Prepare a thin slurry of acid washed asbestos fiber in water and shake well.

2. Place the crucible in a Walter crucible holder and insert in a suction flask connected to a vacuum source.

[redacted]

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3. Apply a gentle vacuum to the crucible and add the slurry in small amounts until an even mat is spread over the bottom of the crucible. If desired, suspensions of varying size of fibers can be prepared and placed in the crucible with the coarse asbestos on the bottom and the fine asbestos on the top.

4. Continue adding small amounts of asbestos slurry until the pad is about 2 to 3 mm. thick. A small perforated porcelain disk may be used to cover the pad.

5. Wash the mat three times with water or until no further asbestos fibers are carried through in the filtrate.

6. Suck as dry as possible.

7. Before being weighed the crucible must be dried in exactly the same manner as the final precipitate is to be treated. A precipitate which is to be ignited should never be weighed in a crucible which has only been dried in an oven.

<u>Use</u>

1. The crucible is used in the same manner as described above. Attention should be paid to the following points:

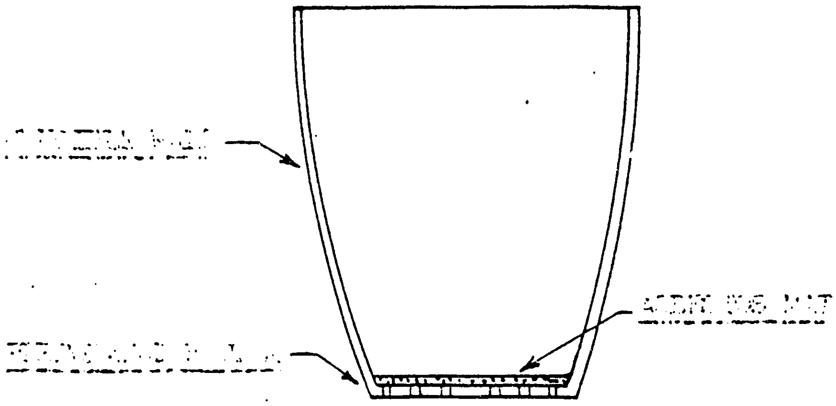
    a. Do not pour a liquid directly on the mat as some fibers may be carried through.

    b. Do not fill the crucible with liquid unless the suction is applied in order to prevent floating the mat with consequent loss of the precipitate.

[redacted]

Code; [illegible]-15a

[illegible] CRUCIBLE WITH ASBESTOS MAT



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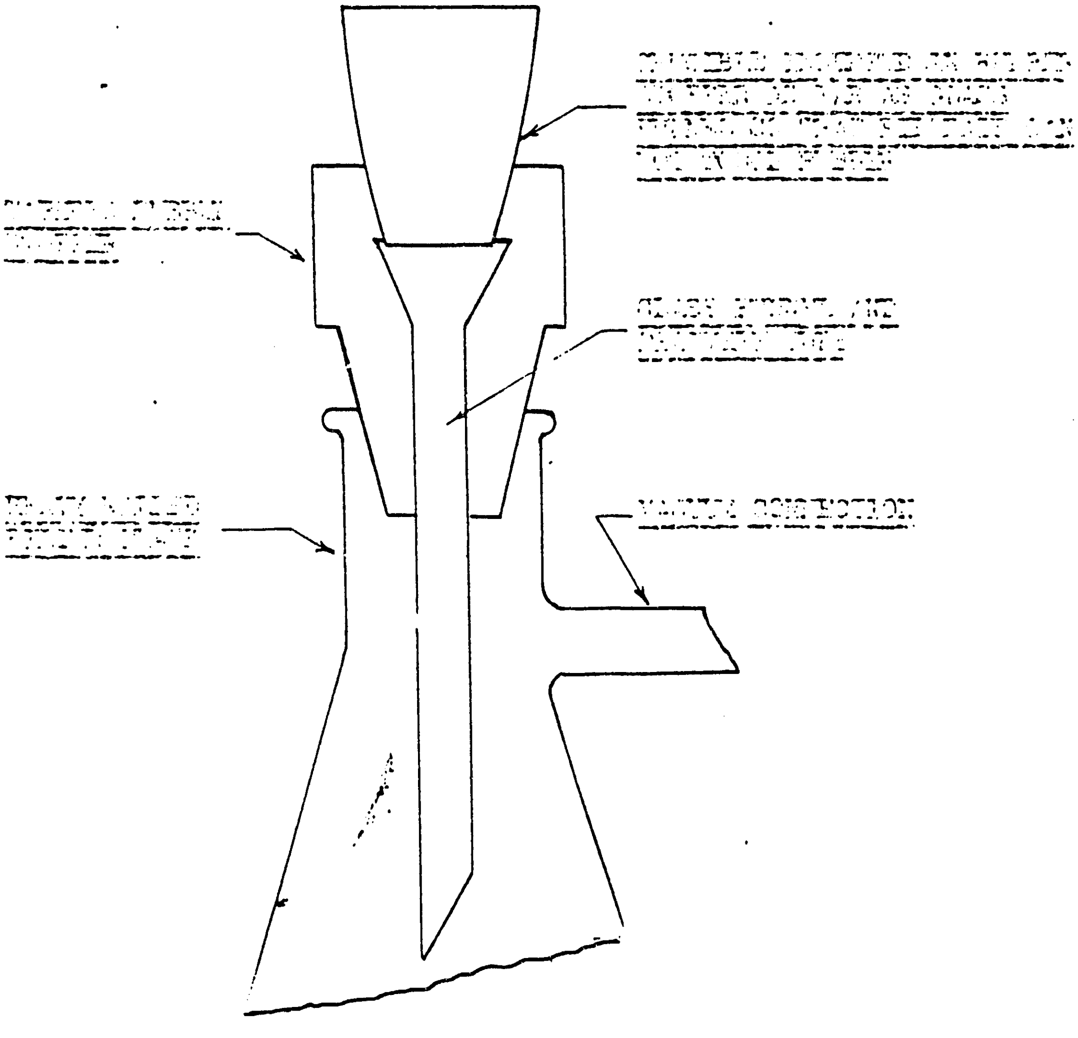

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H. W. Laboratory Manual
Laboratory Apparatus

Code: AC-18c
Reissued: January, 1949
Status: Standard
By: W.J.M.

## CRUCIBLE, SINTERED GLASS

### Uses

These crucibles are used to replace Gooch crucibles where high temperature ignitions are not necessary.

### Manufacturers

Corning Glass Company

### Advantages

Eliminates asbestos mat.

### Disadvantages

1. Can not be ignited at high temperatures.
2. Sudden temperature changes must be avoided.
3. Filter disk is difficult to clean.

### Description

This crucible is composed of a Pyrex glass vessel equipped with a sintered glass filtering disc. The filter can be obtained in three degrees of fineness and the disks are fused directly into the glass walls.

### Preparation for Use

1. Wash and dry the crucible in the same manner to be used in the analysis.
2. This crucible is used in the same manner as a Gooch crucible, except in regard to excessive heating.

[redacted]

## Cleaning

The cleaning of sintered glass crucibles is of especial importance. The best method of cleaning is to leach out the precipitate or residue by means of a liquid in which it is soluble. The solvent is then removed by inverting the crucible over the filter flask assembly and washing with water in the reverse direction of that normally used.

Any solvents which might corrode or etch glass must be avoided to prevent increasing the porosity of the disk.

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H. W. Laboratory Manual
Laboratory Apparatus

Code: AC-21a

Reissued: January, 1949
Status: Standard
By: W.J.M.

## MATCHED CUVETTES, 19 MM.

### Uses

Used in the Coleman spectrophotometer to hold the solution whose transmission is being measured.

### Manufacturers

Wilkens-Anderson Co., Chicago, Ill.

### Advantages

1. Low cost
2. Easy to use

### Disadvantages

1. Difficult to match accurately.
2. Easily scratched and marked. Such marks cause poor, inaccurate readings.

### Literature References

Manufacturer's Technical Literature

### Discussion

#### I. Procedure for Matching Cuvettes

The cuvettes can be obtained from the manufacturer in matched pairs, but the high cost of these cuvettes makes a cheaper substitute desirable. Unmatched cuvettes may be obtained from the manufacturer at a fraction of the cost of the matched ones They are readily grouped into matched sets by the following procedure.

1. Select about 80 cuvettes from several boxes of the unmatched cuvettes.
2. Clean the cuvettes thoroughly. Wash them with soap and water first, using a brush if necessary. Rinse the cuvettes thoroughly, then immerse them in warm cleaning

[redacted]

solution, rinse thoroughly, first under the tap and then with distilled water.

3. Dry the outside of each cuvette with absorbent tissue.

4. Fill one of the cuvettes about 2/3 full with distilled water. Fill the remaining tubes about 2/3 full of 0.00034 M potassium dichromate in 1 M sulfuric acid.

5. Insert the water-filled cuvette into the spectrophotometer, which has previously been prepared for operation (use a wave length setting of 430 mu). Adjust the spectrophotometer to read about 98 percent with the cuvette in the light path. Slowly rotate the cuvette until a maximum transmission reading is obtained. Mark the cuvette with a wax pencil to indicate the center of the range of maximum transmission. It is most convenient to make a vertical mark on the side of the cuvette facing the operator.

6. Readjust the spectrophotometer, so that the blank, aligned to give its maximum reading, reads 100 percent.

7. Insert each of the potassium dichromate-filled cuvettes into the spectrophotometer in turn. Slowly rotate the cuvettes in the holder until their position of maximum transmission is located. Record the maximum transmission value, and mark the cuvettes to indicate the center of the range of maximum transmission as was done in Step 5.

8. Repeat Steps 5, 6, and 7 twice, using different operators each time, so that three transmission values, each by a different operator, are obtained for each cuvette.

9. Average the transmission values for each cuvette.

10. Select a set of eight tubes having maximum transmission values within + 0.05%. This set of cuvettes may be considered matched.

11. Replace the wax pencil mark with a permanent mark made with a diamond tipped glass marker. Mark the set so that it can be distinguished readily.

12. Repeat Steps 10 and 11, choosing as many sets of cuvettes as desired or possible.

13. Store the remaining cuvettes retaining the original numbers and data for future reference.

14. Clean and dry the selected matched cuvettes. They are now ready for use.

II. Notes on the Care and Use of Absorption Cells

In the use of these cuvettes, observe the general precautions for handling all absorption cells.

1. Never touch the optical faces with the bare fingers or with any harsh tools such as tongs. Finger marks are difficult to remove from the faces, and scratches cannot be removed.

2. Always clean and dry the cell thoroughly immediately after use. Never allow any liquid except distilled water to remain in contact with the cells for longer time than is necessary to make the desired measurements.

3. Never use absorption cells with corrosive solutions, particularly those containing hydrofluoric acid.

If the cells become dirty, they may be cleaned as follows:

1. Immerse the cell briefly in warm dichromate cleaning solution. (Warm sulfuric acid alone if the cells are to be used in the chromium determinations).

2. After removing the cell from the cleaning solution, rinse it several times with distilled and redistilled water.

3. The cells may be dried by rinsing in alcohol and ether (provided traces of these materials do not interfere with the color next to be measured), but they should never be dried by heating.

   Cells should be stored in a small cabinet or box especially reserved for the purpose. If the storage is to be for long periods of time, the cells should be wrapped in a soft tissue and placed in a small, covered box. This in turn should be stored in some out of the way place.

[redacted]

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Code: AC-21b

H. W. Laboratory Manual
Laboratory Apparatus

Reissued: January, 1949
Status: Standard
By: W.J.M.

## ABSORPTION CELLS
(5 cm. Square Coleman)

### Uses

Used in the Coleman Model 11 spectrophotometer to hold the solution whose transmission is being measured.

### Manufacturer

Wilkens-Anderson Co., Chicago, Ill.

### Disadvantages

1. Large volume
2. Difficult to handle

### Literature References

Manufacturer's Technical Literature

### Discussion

Refer to notes given in Apparatus Code: AC-21a.

[redacted]

[redacted]

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Laboratory Apparatus

Code: AC-21d

Reissued: January, 1949
Status: Standard
By: W.J.M.

# ABSORPTION CELLS
## (5 cm. Aminco)

### Uses

Used in a spectrophotometer to hold the solution whose transmission is being measured.

### Manufacturer

American Instrument Co., Silver Springs, Maryland

### Advantages

Relatively long length (5 cm) combined with small volume (7.5 ml.).

### Disadvantages

1. High cost
2. It is not designed to fit any spectrophotometer, and adapters must be used when the cell is used with either the Coleman or Beckman spectrophotometer.

### Literature References

Manufacturer's Technical Literature

### Discussion

Refer to notes given in Apparatus Code: AC-21a.

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Code: AC-21e

H. W. Laboratory Manual
Laboratory Apparatus

Reissued: January, 1949
Status: Standard
By: W.J.M.

# ABSORPTION CELLS
(5 cm. Glass, 4 mm. I.D.)

## Uses

Used in the Beckman spectrophotometer to hold the solution whose transmission is being measured. Use only when the volume of solution available is less than 10 ml.

## Manufacturers

American Instrument Co., Silver Springs, Maryland.

## Advantages

Relatively long length (5 cm.) combined with a very small volume (0.6 ml.).

## Disadvantages

1. Need special adapter for use in the Beckman Spectrophotometer.

2. Small size requires careful handling and filling techniques.

## Literature References

Rosenfels, Kirk, Kitson, Patton, and Curtis, CN Report.

## Discussion

Refer to notes given in Apparatus Code: AC-21a.

Cells of this size require special handling and filling techniques as compared to conventional sized absorption cells.

1. In filling the cells, it is necessary to transfer the liquid to the cell with a small pipet. After filling, the cell must be kept level.

2. The cell is most conveniently emptied by using the same pipet used to fill it.

Cells of this type are most conveniently stored by immersing them in a small bottle of distilled water. Before use, they must be

[redacted]

dried to remove excess water, both inside and out, and then rinsed with the colored solution to be measured.

The cells may be stored in the same manner as other absorption cells, however, if this is desired or preferred.

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Obviously this will have to be done before the cells are filled. During the filling and handling operations, the faces must be kept clean.

2. The cells are best stored in a clean and dry state. No experiments have been carried out to determine whether they can be allowed to soak in water between uses as is recommended with the 4 mm. I.D. glass cells.

3. In using these cells, it is important that the same cells be used for the analysis as were used for preparing the calibration curve, that the same cells are used for blank and sample, and that the same end of the cell always is in the same position in the optical path.

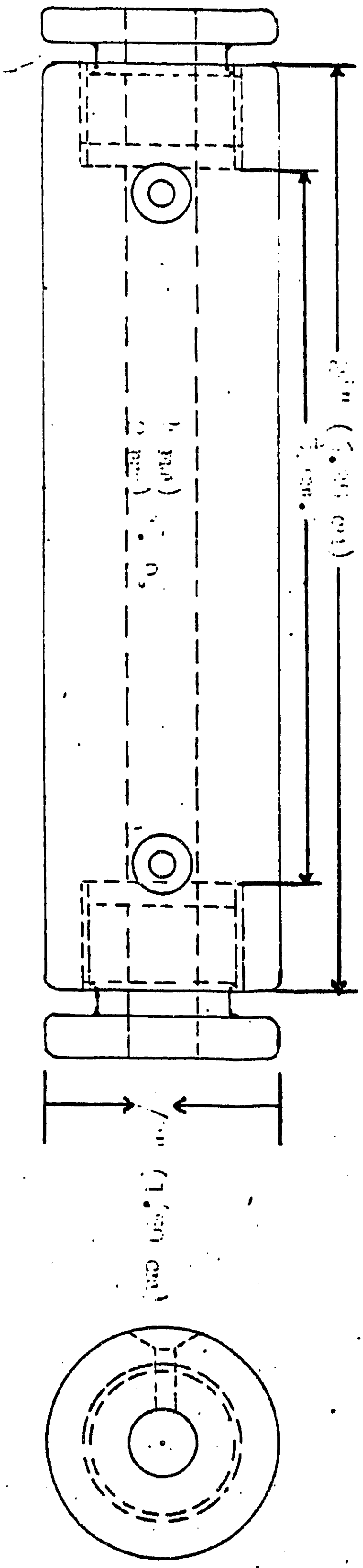
4. Always keep the rotational position of the cells in the holder the same. It is most convenient to keep the ports vertical.

5. A special spectrophotometer adapter is necessary for these cells. Use the same adapter as for the Aminco glass cells, Apparatus Code: AC-21d.

[redacted]

Code: AC-21[illegible] and [illegible]

[illegible] ([illegible])



AC-21[illegible] = [illegible] cm, 1 cm I.D.
AC-21[illegible] = [illegible] cm, 2 cm I. D.

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[redacted]

Code: AF-9b'

H. W. Laboratory Manual
Laboratory Apparatus

Reissued: January, 1949
Status: Standard
By: W.J.M.

## SINTERED GLASS FILTER

### Uses

Used in the filtration of chemical precipitates, where glass filters and a micro scale are desired.

### Manufacturers

Fabricated on the plant site from materials from Corning Glass Co., Corning, New York.

### Advantages

1. Permits rapid suction filtering.

2. Permits ready removal of precipitate, either chemically or mechanically.

### Disadvantages

Readily contaminated and difficult to decontaminate.

### Discussion

The filter is made as follows: Draw off one end of a pyrex sealing tube (Corning 10 F) and blow a test tube bottom on it. This should be done as close to the filter as possible. In the bottom of the test tube, blow a small hole and attach a piece of 7 or 8 mm. tubing to it. Anneal thoroughly in a flame.

The filter is used as is any fritted glass filter. The same precautions observed in the use of these filters should be observed with this filter.

The filter may be made from other porosities or other O.D. sealing tubes, if desired.

[redacted]

[redacted]

H. W. Laboratory Manual
Laboratory Apparatus

Code: AF-12a

Issued: January, 1949
Status: Tentative
By: W.J.M.

# KLETT FLUORIMETER

## Uses

To measure fluorescence.

## Manufacturer

Klett Manufacturing Co., New York, N.Y.

## Advantages

1. High sensitivity.
2. Two photo-cell construction minimizes line-voltage fluctuations.
3. Rapid analysis.

## Disadvantages

1. Requires a transformer and a galvanometer with transformer.
2. Bulky.

## Literature References

Photo-stat copy: "Klett Fluorimeter, Model 2070".

## Description

A more detailed description is given in the photostatted copy. See also, the diagram.

1. Lamphouse holds a type H-4 high pressure mercury lamp.
2. Housing at right contains the reference photo cell, the green (Corning 4080) light filter, and an adjustable diaphragm.
3. Housing at left contains the measuring photocell, the condenser lenses, the lamp filter (#5970), the photo cell filter (#3389), the cuvette holder, and the shutter.

[redacted]

4. Galvanometer switch has three positions: low sensitivity when the handle is in the "L" position; high, in the "H" position; and off when the handle points up.

5. Potentiometer is a precision, linear-wound one with a 360 division linear scale.

6. Photo cells are of the barrier-layer type and respond to light of wave length from 4000 A° to 6600 A°.

Operation

1. Plug in the fluorimeter lamp at least 5 minutes before readings are made.
2. Fill the cuvette with a standard of fluorescence and place it in the instrument.
3. Set the potentiometer at some value between 100 and 300.
4. Open the shutter.
5. Move the galvanometer switch to "L" and adjust the variable slit until the galvanometer is "zeroed" in the middle of the scale.
6. Repeat with the switch at "H".
7. Close the shutter.
8. Replace the cuvette with one containing the unknown, the fluorescence of which is to be measured.
9. Open the shutter.
10. Move the galvanometer switch to "L" and turn the potentiometer until the galvanometer beam is brought back to "zero".
11. Repeat with the switch at "H".
12. Turn the switch off.
13. Close the shutter.
14. The ratio of the potentiometer scale reading to that of the standard (which was set between 100 and 300) gives the ratio of the fluorescences of the unknown to that of the standard.

15. The concentration of the standard and the potentiometer setting for it should be so chosen that a deflection of 1 mm. of the galvanometer is obtained per 2 or 3 division move of the potentiometer.

[redacted]

[illegible]

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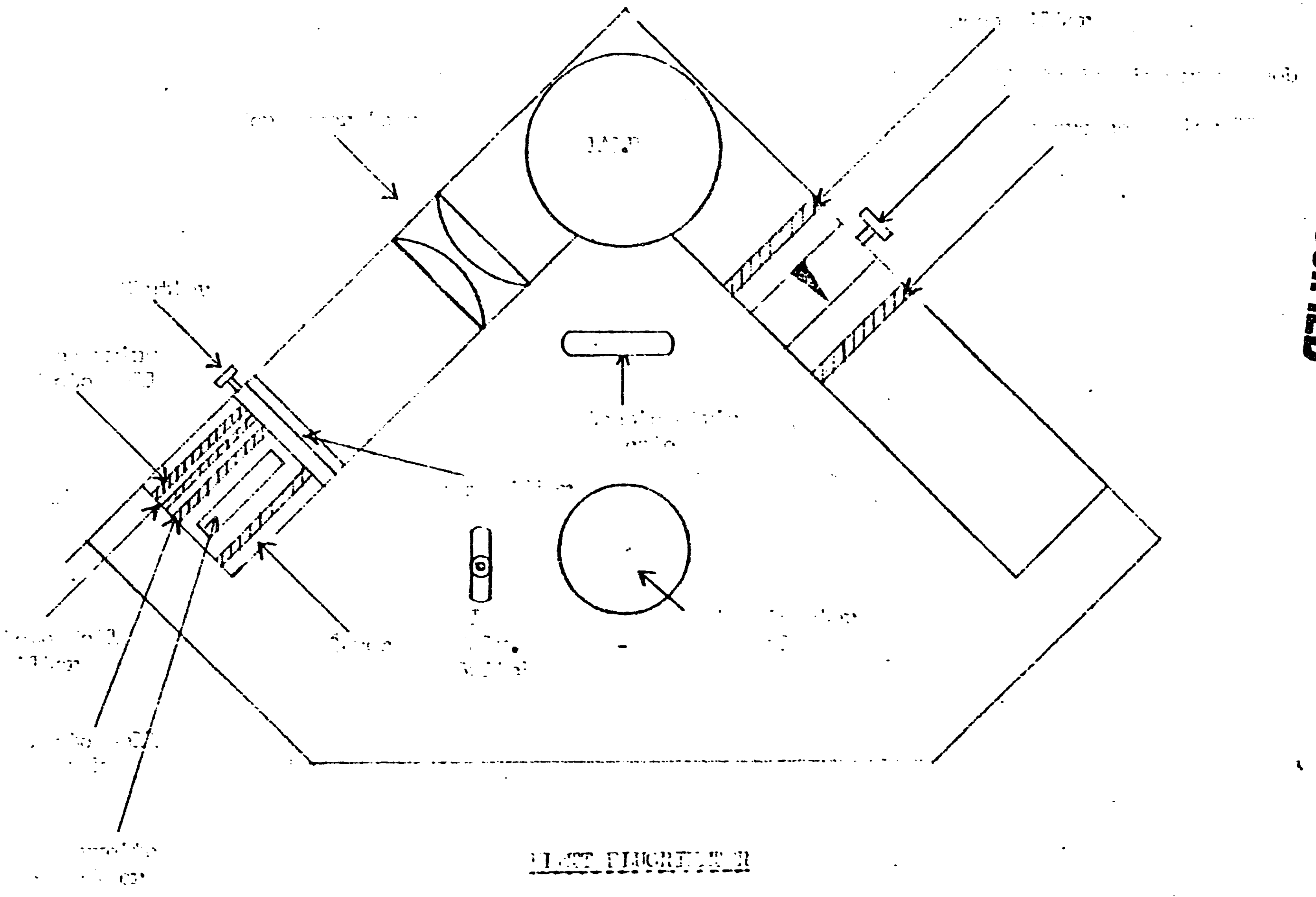

[illegible] FLUORIMETER

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Code: AF-12a

Drawn by: W. J. Major 2-1-50

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CODE: AF-12b

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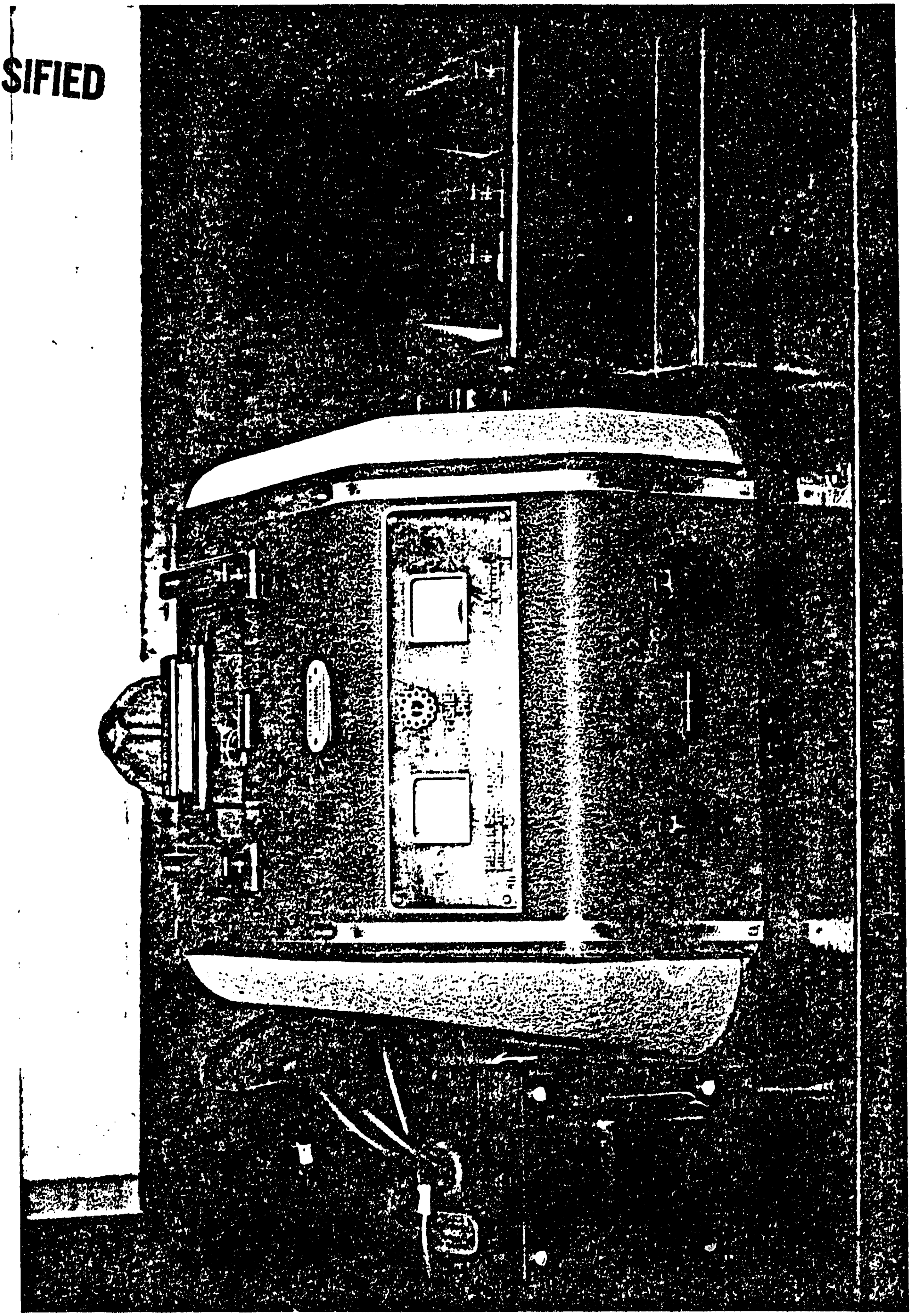

FISHER NEFLUORO - PHOTOMETER

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H. W. Laboratory Manual
Laboratory Apparatus

Code: AH-1a
Reissued: January, 1949
Status: Standard
By: W.J.M.

# RADIANT HEATER

## Uses

Used for evaporating solutions, drying precipitates and for general laboratory use.

## Manufacturer

General Electric Supply Co.

## Advantages

1. Construction assures long life and efficiency because fumes cannot reach the enclosed reflector.
2. Evaporation is more rapid because the light rays travel directly to the solution and penetrate it.
3. Bumping and spattering minimized.
4. Chars filter paper rapidly without danger of paper firing.

## Description

The radiant heater consists of a single 250 watt infra-red reflector drying lamp. The lamp is attached to an ordinary ring stand for adjustable heights as required in the laboratory.

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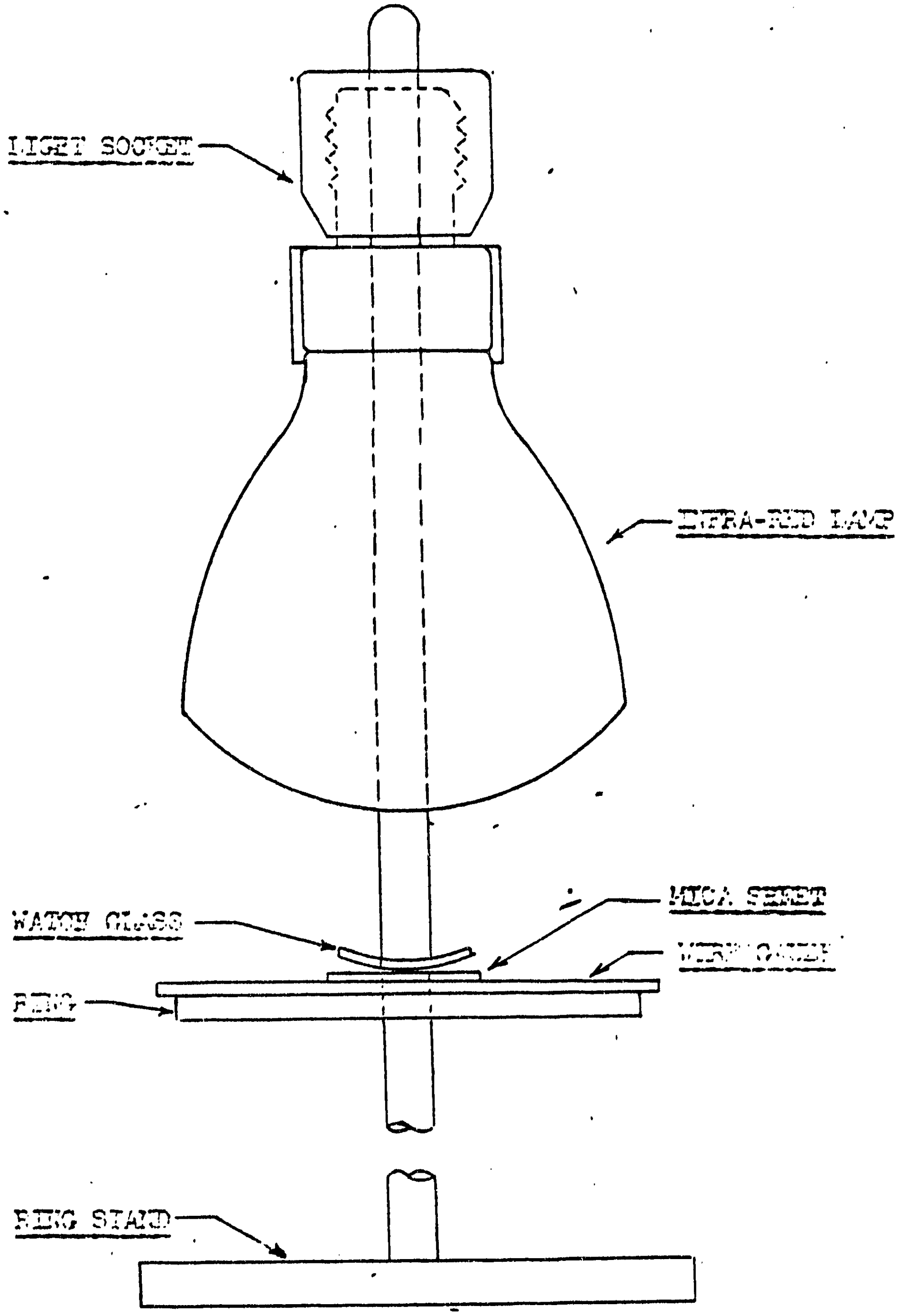
Code; AF-1a

# RADIANT HEATER



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H. W. Laboratory Manual
Laboratory Apparatus

Code: AS-16a

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Status: Standard
By: W.J.M.

## COLEMAN MODEL 11 SPECTROPHOTOMETER

### Uses

Used in the colorimetric determination of numerous ions throughtout the entire process.

### Manufacturer

Coleman Electrical Company, Maywood, Illinois

### Advantages

1. Low Cost
2. Ruggedness
3. Low cost of absorption
4. Ease of manipulation

### Disadvantages

1. Poor resolution by monochromator
2. Insensitive measuring system
3 Limited applicability

### Literature Refernces

Technical Literature, Coleman Electrical Company Maywood, Illinois.

### Discussion

The optical and electrical circuits of this spectrophotometer, and its application in analysis, have been described elsewhere in this manual. The following instructions are intended to aid in the care and use of the spectrophotometer. They inclued instructions for the preliminary set-up of the instrument its operation and calibration, replacement of expended parts and other miscellaneous information.

### I. Preliminary Set-Up

[redacted]

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1. Remove the new instrument from its box, and set upon a firm level table. Place the sponge rubber pads beneath each corner. These will serve to minimize external vibrations which would otherwise obviate the results.

2. Connect the two rubber-covered leads from the instrument to the "Output poles of the transformer. To the Input poles, connect the ends of the light cord which accompanies the transformer. In either case, the polarity is emmaterial.

3. Check the wavelength calibration, and the setting of the absorption cell carrier as described under sections III and VII of these directions.

4. Cover the snstrument with a standard cover or with some material which is relatively impervious to dust. Dust and dirt in the optic and electrical parts of any instrument are a major source of difficulty.

I. Operation of the Instrument

1. Direct Reading Method

This method of measuring transmission with the Coleman spectrophotometer is more rapid and as accurate and precise as the null-point method It will be used in all determinations unless otherwise specified. Measurements made by the two methods cannot be compared, since they may not agree even when taken from the same instrument.

a.; Set the drum dial, seen through the window in the right hand side of the top panel, to about 3 on the back scale. Turn the main switch to "Galv.", and adjust the galvanometer reading on the scale in the upper left hand corner of the panel to approximately zero by adjusting the "Adjust Zero" knob. Complete the zero adjustment by moving the drum dial slightly. When the adjustment in complete, the reading (black scale) on the drum scale shoudl not be more than 5 If it is greater, it should be returned to 3, and the galvanometer reset with the "Adjust Zero knob The setting of the galvanometer is reasonably constant, but although it seldom changes, it should be checked occasionally

b. Turn the main switch to the "On position and allow the instrument to warm up for approximately five minutes.

c. While the instrument is warming up, check to see that the correct absorption cell carried is in place. If the test tube type cella are used the vertical carried should be inserted through the top of the panel.

[redacted]

[redacted]

A small light guard, which slides with the carrier and prevents stray light from entering the sample chamber, must also be in place. If the horizontal cells are to be used, the horizontal cell holder, together with the necessary adapted, sgould be inserted through the small door on the right hand side of the instrument. In the latter case, the opening in the panel must be entirely closed by a suitable light guard.

d. Before making measurements, insert the proper filter for the region to be measured into the slot behind the sample chamber. The slot opens near the hinged end of the side door to the sample chamber. The proper filters for the various wavelengths are; 350-400 mu, PC-6; 400-650, PC-4mu; 650-800, PC-5.

e. Adjust the wavelength to the desired value by turning the wavelength knob. Turn the "Fluor" knove to its most counterclockwise position.

f. After the necessary warm-up period, insert the absorption cells, filled with the liquid whose transmission is desired, and a blank, into the cell holder. Position the latter so that the blank-containing cell is in the light path.

g. By varying the position of the "Galv. Coarse" knob, adjust the galvanometer reading to approximately 100 per cent. Adjust it exactly to this value by varying the position of the "Galv. Fine" knob.

h. Insert into the light path the absorption cell containing the unknown, and when the galvanometer comes to rest, read the percentage transmission directly off the scale.

i. Repeat steps g and h until 4 reading on one absorption cell have been made. Average the four values.

j. When through with the instrument, turn the main switch "Off", remove the absorption cells and clean if necessary.

2. Null-Point Method

a. Set the drum dial, seen through the window in the right hand side of the top panel to zero. Turn the main switch to "Galv", and adjust the galvanometer reading on the scale in the upper left hand corner of the panel to zero by moving the "Adjust Zero" knob.

[redacted]

[redacted]

b. Turn the main switch to the "On" position, and allow the instrument to warm up for approximately 5 minutes.

c. While the instrument is warming up, check to see that the correct absorption cell carrier is in place. If the test tube type cells are used, the vertical carrier should be inserted through the top of the panel. A small light guard, which slides with the carrier and prevents stray light from entering the sample chamber, must also be in place. If the horizontal cells are to be used. the horizontal cell holder, together with the necessary adapter, should be inserted through the small door on the right hand side of the instrument. In the latter case, the opening in the top panel must be entirely closed by a suitable light guard.

d. Before making measurements. insert the proper filter for the region to be measured in to the slot behing the sample chamber. The slot opens near the hinged end of the side door to the sample chamber. The proper filters for the various wavelengths are: 350-400 mu, PC-6; 400-650 mu, PC-4; 650-800 mu, PC-mu.

e. Adjust the wavelength to the desired value by turning the wavelength knob. Trun the "Flur" knob to its most counterclockwise position.

f, After the necessary warm-up porion, insert the absorption cells, filled with the liquid whose transmission is desired, and a blank, into the cell holder. Position the latter so that the blank-containing cell is in the light path.

g. Set the drum dial, by means of the "Bal" knob on the right hand side of the instrument, to 100 percent transmission.

h. Vary the position of the "Galv Course" knob until the galvanometer reads approximately zero. Complete the adjustment to zero with the "Galv Fine" knov. If the galvanometer cannot be brought on the scale with thsos adjustments, vary the position of the "Flour" knob until an approximate zero is abrained, and again complete the adjustment with the "Galv Fine" knob.

i. Insert into the light path the absorption cell containing the unknown, and readjust the galvanometer to zero by varying the position of the drum dial. Read the percent transmission from the dial.

j. Repeat steps g, h, and i until 4 readings on one absorption cell have been made. Average the four values.

k. Any number of readings may now be made. The 100 percent setting of the blank should be checked before each reading, however.

l. When through with the instrument, turn the main switch "Off", remove the absorption cells, and clean if necessary.

## III. Calibration of Wavelength with Didymium Filter

It is occasionally desirable to check the wavelength setting. This should always be done when a new instrument is received and when a new exciter lamp is installed.

1. Turn the main switch "On" and allow the instrument to warm up for five minutes.
2. Insert the filter PC-7 in the filter holder.
3. Set the wavelength scale at the value stamped on filter (usually 587 mu, but occasionally different).
4. Set the "Fluor" knob to its extreme counterclosckwise position.
5. After the necessary warm-up period, turn the "Galv. Coarse" knob until the galvanometer reads 80 to 100 percent.
6. Slowly rotate the wavelength scale until a minimum transmission value is obtained. If this value is less than 80 percent, readjust "Galv. Coarse" knob so the reading is between 80 and 100 percent and relocate the point of minimum transmission.
7. The wavelength of minimum transmission should be that wavelength marked on the filter ± 2 mu. If the value falls within this range, the setting may be assumed to be correct. If the value does not fall within this range, proceed as follows:
8. Set the wavelength scale at the correct value marked on the filter.
9. Remove the screen from the back of the instrument.

10. Loosen slightly, through the hole in the bottom of the instrument case, the clamp screw on the botton of the lamp arm.

11. Adjust the headless screw in the side of the lamp arm, by means of a long screw driver inserted through the screen opening, until the galvanometer reads a minimum. This should not be more than a fraction of a turn. It may be necessary, if the screw turns stiffly, to loosen the rosin lock with a drop of alcohol.

12. Tighten the clamp screw (see step 10).

13. Check wavelength setting again, and, if correct, replace the screen.

## IV. Replacement of the Exciter Lamp

The exciter lamp is an 8.5 volt, 36 watt prefocused coiled line filament lamp. These lamps have a life expectancy of 300 hours, and should be replaced as follows upon failure.

1. Remove the screen from the back of the instrument.

2. Loosen the knurled bakelite binding post which holds the spaghetti covered lamp lead. Remove the lead.

3. Grasp the exciter bulb and turn counter clockwise (looking down), then pull straight up and remove from the instrument.

4. Insert new bulb, after wiping clean and after threading the lead through the hole in the lamp arm.

5. Reverse directions 2 and 3. Avoid touching the side of the bulb toward which the filament faces.

6. Before replacing screen, check the wavelength calibration as described in above. More often than not, some adjustment will have to be made.

## V. Replacement of the Galvanometer Lamp

The galvanometer lamp is a 6 to 8 V, 6 cp, doublecontact bayonet base, automobile type bulb. Replace this bulb as follows:

1. Remove the screen from the back of the instrument.

2. Remove one screw and loosen the other in the clamp which holds the galvanometer lamp in place.

3. Remove the bulb and socket from the holder, and remove the bulb from its socket.

4. Insert a new bulb into the socket. Wipe bulb free of finger marks.

5. Replace socket in clamp. Rescrew clamp so that socket and bulb can be moved vertically, but will not do so of their own weight.

6. Set the main switch of the instrument at "Galv." and adjust the height of the bulb and socket until a sharp, clear image is obtained on the galvanometer scale.

7. Tighten the clamp, and replace the wire screen.

## VI. Replacement of Slide Wire Battery

A small Burgess 4 FJ $1\frac{1}{2}$ V battery is mounted inside the instrument. Replacement is seldom necessary unless the instrument is extensively used for fluorimetric work. Replace as follows:

1. Remove the six screws around the extreme edge of the top panel. Loosen the set screw holding the "Bal" knob and remove the knob and its spring washer. Set the instrument on its front edge. Push the transformer cable back several inches into the hole by which it emerges from the bottom of the box. Pull forward on the edge of the panel farthest from the drum dial until the rheostats, etc. on the under side of the panel clear the side of the box. Then slide sideways to disengage the drum dial shaft from the box, and finally pull the panel forward and lay it on its face on the table.

2. Disconnect the battery leads.

3. Remove the battery hold-down strap.

4. Remove the battery, and replace with a new one.

5. Replace battery clamp, and draw down screws snugly.

6. Connect the battery leads, red to the positive or center terminal, black to the negative terminal.

7. Replace panel by reversing the directions in 1 above.

[redacted]

## VII. Centering of the Vertical (ST-10-C) Absorption Cell Carrier

The Model 11 Universal Spectrophotometer is provided with stop screws located at either end of the rectangular panel slot through which the cuvette carrier is inserted. These screws serve to position the carrier accurately in either extreme of its travel and thereby insure that the respective cuvettes occupy exactly the same position in the monochromatic light beam, a condition that is particularly important when round cuvettes are used.

When an ST-10-C cuvette carrier is ordered together with the Spectrophotometer, these locating screws have been adjusted at the factory and the corresponding cuvette carrier then engraved with the serial number of the particular instrument adjusted for its use. However, when the ST-10-C carrier is purchased separately, these stop screw adjustments must be completed by the operator before exact spectrophotometric measurements are undertaken. Failure to observe this fact will nullify the inherent precision of the instrument and of the most carefully matched cuvettes. When necessary proceed with the adjustment as follows:

1. Turn the instrument switch to "On", adjust wavelength dial to about 550 mu., remove the photocell filter and observe that the projected light beam is centered on the sensitive surface of the photocell. Now it will be apparent that if a cylindrical cuvette is inserted squarely in the axis of this beam, the beam will remain straight, but if the cuvette is off center the beam will be bent and will no longer properly center on the photocell. The object of the following procedure is to so adjust the cuvette carrier locating screws that the interposed cuvettes cause the minimum beam deviation and that the effect be exactly the same in either extreme cuvette position.

2. Check for gross misadjustment of the locating screws by moving the carrier first to one and then to the other extreme position with filled cuvettes in place. Notice if there occurs any shift in the position of the illumiated spot on the photocell surface. (This is best observed by inserting the cuvette carrier without its light guard slider, the photocell surface then being visible past the edge of the carrier).

3. Adjust the locating screws until there is no perceptible shift in the position of the illuminated spot at the extreme positions of the carrier and the beam is properly centered on the photocell. (Use the filled round cuvettes for this test also).

[redacted]

4. Turn the drum dial to 100 by means of the "Bal" knob and then turn the "Fluor" knob to the extreme counterclockwise position. Insert the PC-4 filter.

5. Adjust the "Galv. Coarse" knob until the galvanometer reads between 80 and 90 with a filled cuvette in the side of the carrier intercepting the beam. NOTE THIS READING.

6. Now slide the cuvette carrier to the opposite extreme position and transfer the same cuvette to the opposite cavity so that it again intercepts the light beam, being particularly careful to insert in the same angular position as before and to avoid finger prints on the surfaces exposed to the light. The galvanometer should now read the same value as at (5) if the stop screws are fully adjusted. However, the two readings may possibly fail to agree, in which instance proceed as follows:

7. First note if moving the carrier slightly away from the adjacent stop screw brings the second galvanometer reading to better or worse accord with (5). Adjust the adjacent stop screw accordingly, until the readings exactly agree. If more than one-half turn of the stop screw is necessary for agreement, turn the screw one-half turn, transfer the cuvette to the other side of the carrier and complete adjustment in the same manner with the other stop screw. Return the carrier to the original position, transfer the cuvette and check again for agreement, which should be practically within the limit of reading of the instrument.

8. Finally, again check visually, to be sure that the cuvette is properly centered in the beam.

## VIII. Remote Control Equipment

In some of the applications of this instrument, it is necessary for the operator to remain at some distance from the solutions being analyzed, because of the unusual hazards in these solutions. For this purpose, certain equipment has been developed, and is shown in the photograph attached to this discussion and to that of AS-16b.

1. Cuvette Shield In the back row, center and right, of the photograph at the end of this method, is shown a cuvette shield, to be used when the cuvettes are being placed in or taken from the holder. The right hand view shows the apparatus cut away, and containing a standard cuvette carrier. Two small metal posts push the cuvettes up out

of the carrier enough to permit grasping with tongs. In operation, the empty carrier is put in the shield, and then each cuvette, filled in another place, is placed in the carrier with tongs. The whole carrier is then lifted up, the cuvettes dropping into position. For removal of the cuvettes, the procedure is merely reversed.

See University of Chicago, Metallurgical Laboratory Drawing No. CAN-114 for constructional details.

2. Absorption Cell Adapter and Carrier In order to use the 5 cm. Aminco (AC-21d) absorption cells in the Coleman spectrophotometer, an adapter is needed. The photograph attached to Apparatus Code: AS-16b shows the combined adapter and carrier developed for this purpose. It is second from the right. The cells may either be set in the carrier before filling, or lowered in with tongs after filling. The whole carrier can then be set in the side door of the instrument, the door closed, measurements made in the usual fashion, the holder and cells removed, etc.

See University of Chicago, Metallurgical Laboratory Drawing No. CAN-155 for constructional details.

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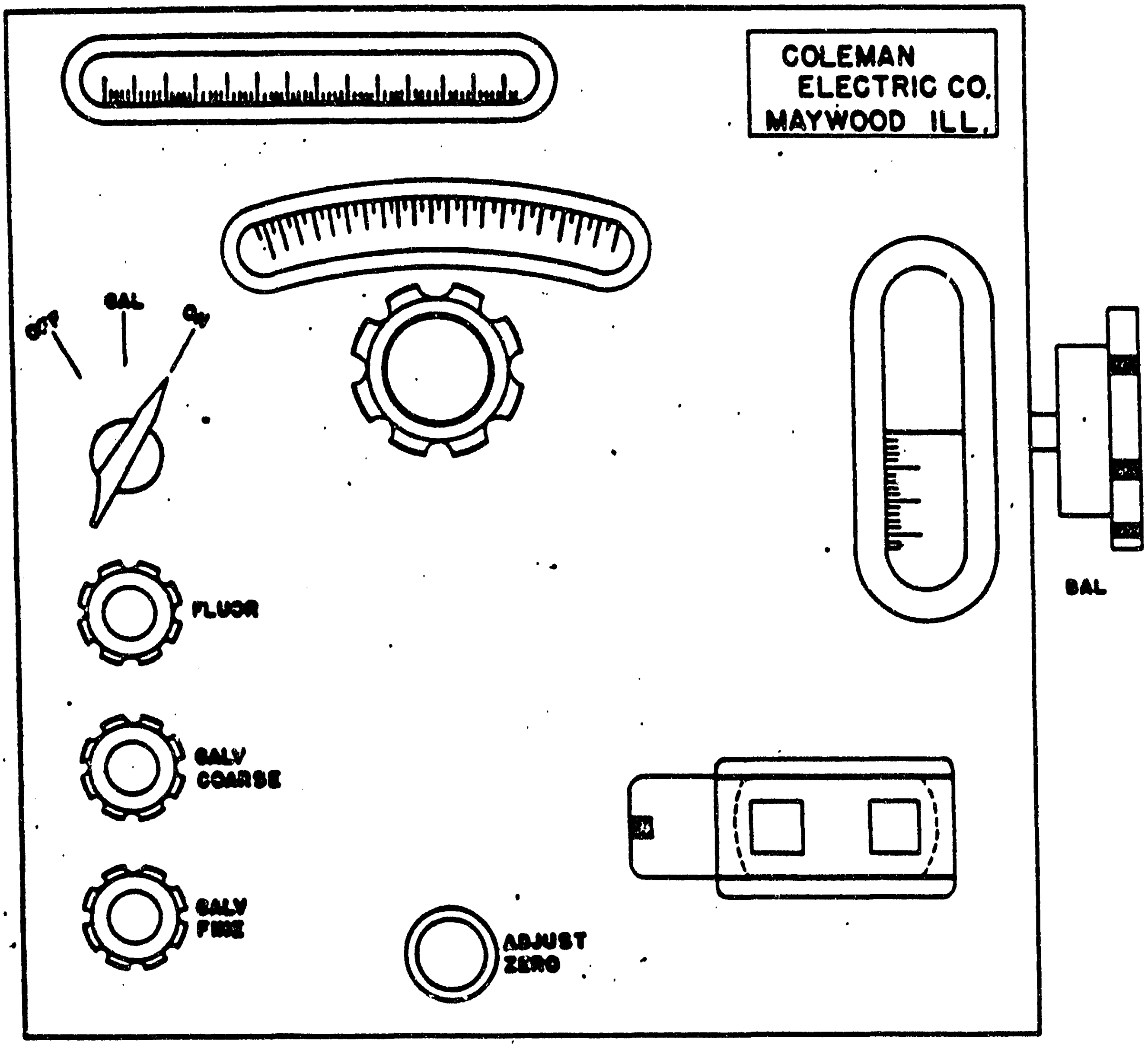
Code AS-16a



MODEL II
UNIVERSAL SPECTROPHOTOMETER
COLEMAN

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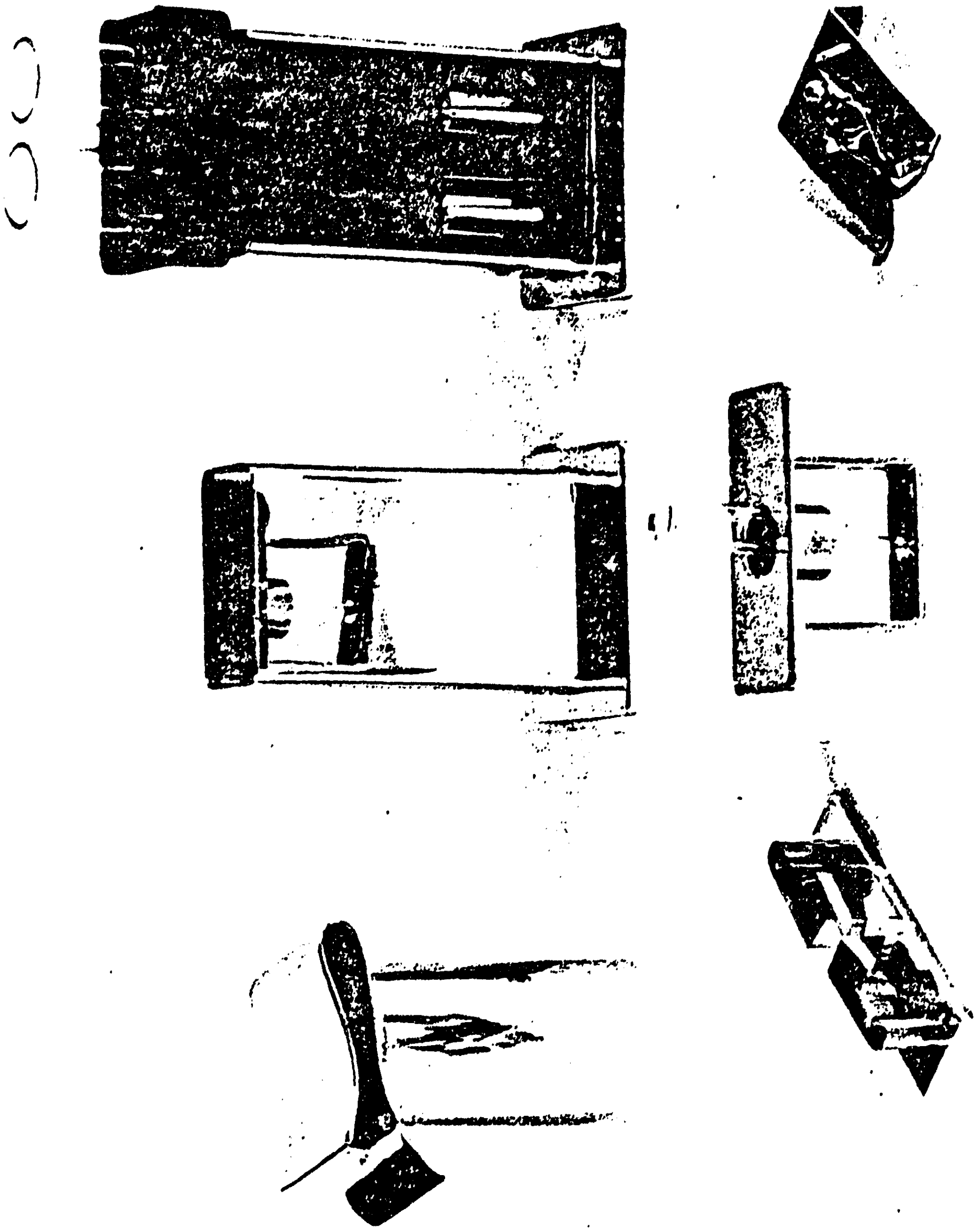

FIGURE II - REMOTE CONTROL EQUIPMENT

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H. W. Laboratory Manual
Laboratory Apparatus

Code: AS-19b
Reissued: January, 1949
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By: W.J.M.

## DISTILLATION APPARATUS, ALL-GLASS WITH FRIEDRICHS CONDENSER

### Uses

Used in the distillation of many liquids where freedom of rubber contacts in the distillation apparatus is essential to the purity of the final product.

### Manufacturer

Corning Glass Co., Corning, N.Y.

### Advantages

1. Simple to assemble.
2. Requires less desk space than most models.
3. Gives maximum protection against carry over of the liquid being distilled.

### Disadvantages

Must be disassembled to fill the boiler.

### Discussion

1. Mount the apparatus over a large electric hot plate, and connect it firmly to a ring stand.
2. Two or more stills may be set up and arranged to deliver their output to the same receiver, thus saving additional desk space.
3. A few boiling chips, glass beads, or a debumping rod should be inserted in the boiler to prevent bumping.

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Code: AT-2a

H. W. Laboratory Manual
Laboratory Apparatus

Issued: December, 1948
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By: W.J.M.

## DIRECTIONS FOR REUNITING THE MERCURY IN THERMOMETERS

Thermometers should not be rejected because the mercury column has become separated. In shipment it is almost inevitable that the mercury thread in a thermometer should become separated. This is especially true with gas filled thermometers and with those having a reservoir in the top. Such thermometers should not be condemned as it is not difficult in most cases to reunite the thread. Surface tension makes it practically impossible to jar the parts together or to make one part touch the other by heating the bulb. The following remedies will ususally solve the difficulty and should be tried in the order in which they are given:

(1) Immerse the bulb of the thermometer in a freezing mixture until all of the mercury has been drawn into the bulb by its contraction. Tap the thermometer to dislodge gas bubbles in the bulb. This treatment is usually effective and is not likely to result in breaking the thermometer.

(2) If the thermometer has a reservoir in the top, either of the following methods may be used:

a. If the capillary is fairly large, invert the thermometer and by jarring, start the mercury thread traveling down. Once started, it will usually flow until the reservoir is full. This causes the two parts to flow together in the reservoir. Turn the thermometer over and by jarring again, start the mercury traveling toward the bubl. Usually it will continue until the column stands at the normal height.

b. Hold the thermometer at a considerable height over a low flame and by moving it back and forth, apply heat gently until the broken place in the thread has moved upward into the reservoir. Usually on cooling, the mercury will remain united. Caution! This method is more likely to result in breakage and extreme care should be used.

(3) By holding the thermometer at arm's length in a horizontal position and rapidly (but not suddenly) swinging the arm down in a circular motion, centrifugal force may cause the upper part of the broken thread to unite with the lower. Be careful not to "snap" the arm when starting the motion as the thermometer may snap off.

[redacted]

(4) If a proper centrifugal arrangement is available, the thermometer can be placed in it, bulb outward, and whirled. A simple arrangement for producing the same result is to tie a string in the ring at the top, start whirling slowly and gradually increase the speed. Be careful not to jerk the thermometer as the small ring may break off.

(5) The above methods should be given several trials before admitting failure. The method to be described now is so likely to ruin the thermometer that it should be employed only as a last resort in preference to discarding the thermometer. Holding the thermometer in a slanting position with the bulb upward, very gently and cautiously heat the upper end of the detached part of the mercury thread, making sure that the glass is gently heated over a sufficient portion on each side to avoid breakage. Be careful not to heat sufficiently to soften the glass. If this is done carefully, the mercury can be distilled from the detached thread and may be driven upward to unite with the main portion of the mercury column.

[redacted]

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Code: AT-20c

H. W. Laboratory Manual
Laboratory Apparatus

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By: W.J.M.

# FILTRATION TUBE

## Uses

Used as an adapter for filtering directly into a 10 ml. volumetric flask.

## Manufacturer

1. Fabricated on the plant site.
2. Stopcock A.H. Thomas Co., Cat. No. 9289.

## Advantages

1. Affords rapid, controlled means of collecting filtrates.
2. Compact
3. Easily manipulated

## Description

The unit consists of a regular 5 inch Pyrex side-arm test tube modified to fit a 10 ml. volumetric flask by cutting off the bottom of the tube and sealing to it a short length of glass tubing (about 2-3 cm.). A No. 1 Pyrex stopcock (1 mm. bore, 6 mm. tubing) is fused to the test tube extension.

## Operation

A 3 ml. fritted-glass filtering funnel is equipped with a No. 0 rubber stopper, and inserted into the filtration tube. The assembled unit is secured to a ring stand with the exit end of the filtration tube inserted into the neck of a 10 ml. volumetric flask. With the stopcock closed, the solution is poured into the filter, and vacuum is applied to the side-arm. When a sufficient amount of filtrate has collected in the filtration tube, vacuum is discontinued and the filtrate collected in the vessel by manipulation of the stopcock.

Several filtration tubes may be conveniently connected through a manifold to the vacuum system. A bleeder line should be inserted in each vacuum connection so that the filter tube may be drained without disconnecting any connections.

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Use

Use as directed in Water Methods Code: WS-11a.

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H. W. Laboratory Manual
Laboratory Apparatus

Code: AT-21a
Reissued: January, 1949
Status: Standard
By: W.J.M.

## ERMOMETER
## ASTM E1 (-20 C to 150 C)

### Uses

For general use in A.S.T.M. tests.

### Manufacturers

1. Central Scientific Co., Chicago, Ill. (Cat. No. 19270)
2. Fisher Scientific Co., Pittsburgh, Pa. (Cat. No. 15-166-G1)

### Description

Ordinary mercury thermometer, calibrated and made according to A.S.T.M. specifications. Use according to specific instructions accompanying each method.

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H. W. Laboratory Manual
Laboratory Apparatus

Code: AT-21b

Reissued: January, 1949
Status: Standard
By: W.J.M.

## THERMOMETER
ASTM El (-5 C to 300°C)

### Uses

For general use in A.S.T.M. tests.

### Manufacturers

1. Central Scientific Co., Chicago, Ill. (Cat. No. 19270)
2. Fischer Scientific Co., Pittsburgh, Pa. (Cat. No. 15-166-G3)

### Description

Ordinary mercury thermometer, calibrated and made according to A.S.T.M. specifications. Use according to specific instructions accompanying each method.

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# END

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