California College of Pharmacy
A LABORATORY MANUAL
OF
QUALITATIVE
CHEMICAL ANALYSIS
FOR STUDENTS OF PHARMACY

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PREFACE.

Qualitative analysis is of use to the pharmacist in testing chemicals for identity and purity. The study of the subject is also of value because of the practical knowledge of chemicals and chemical processes acquired by the student, and because it gives training in careful observation and develops the reasoning powers as they are exercised in the interpretation of results.

This Manual was prepared as a guide for the author’s own classes, and the objects sought are to acquaint the student with the general methods of qualitative analysis and to prepare him to carry out such qualitative tests as the pharmacist may be called upon to make. The course is arranged to include one hour of lecture, one hour of recitation and about three hours of laboratory work per week for one school year. Practice on the analysis of unknown solutions is provided for throughout the course. This is important, as it not only increases the interest of the student in the work but also develops his self-reliance by constantly putting him upon his own responsibility in doing his work and in interpreting his results. The introductory section on the theory of chemistry may be omitted if it is not necessary for the class to study or review this part of the subject.

The Manual is in no sense a reference book, and in general only those things are included that are needed as a guide for the laboratory work, or which may easily be learned in connection with this work. A standard reference book on qualitative analysis will be of great service while taking the course, and such a reference book is indispensable for anyone who is to pursue the subject further.

T. J. B.

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Matter is anything which occupies space, as wood, water and air. In addition to its occupation of space, which is called its extension, matter has several other general properties, including gravitation, indestructibility, inertia, impenetrability and divisibility, which are studied in physics. Some of the things which exist but are not material, are energy in its various forms, mental processes and the emotions.

A substance is a separate kind of matter, like iron, marble, water or air. Each substance possesses all of the general properties of matter and, also, several properties which serve to distinguish it from other kinds of matter and are called specific properties. Among these are its condition of being a solid, a liquid or a gas, its density, and some of the following: color, odor, taste, melting-point, boiling-point, crystalline form, hardness, brittleness, tenacity, behavior toward other substances, and some other properties. The particular combination of these properties possessed by a substance serve to identify it. Most substances may be separated into several other substances, with more or less difficulty, and a substance is said to be composed of its constituents, which are referred to, collectively, as its composition.

Changes in Matter.—Matter is constantly undergoing change. The changes may be rapid or slow but nothing is permanent. The changes are very various, but they may all be classified into two great kinds: one kind affecting the condition of the matter and the other kind affecting its composition. The changes which affect the condition of matter are physical changes and include all forms of motion, temperature changes, magnetic changes, changes of state, like the freezing or boiling of water, and many others. In a physical change the identity of the matter undergoing the change is not affected. The changes which affect the composition of matter are called chemical changes and include all burnings or combustions, the decay of vegetable and animal matter, the rusting of iron, and
various others. A chemical change is frequently called a chemical reaction or simply a reaction. In a chemical change the identities of the matter undergoing the change are lost and new substances with new identities are formed.

Physical and chemical changes are often mutually dependent upon each other; heat, light, electricity, solution and other physical agencies, often produce chemical changes and, conversely, chemical changes often produce heat, light, electricity and other physical effects. Physical and chemical changes are all caused by energy, which is that which can produce change in matter. Energy cannot be created or destroyed, which fact is referred to as the conservation of energy. Heat, light, electricity, and the power due to the motion or the position of matter, are different forms of energy, and these agencies may produce chemical change or be produced by chemical change. Matter can neither be created nor destroyed, which fact is referred to as indestructibility or the conservation of matter.

Physical science studies the properties of matter and the effects of energy on matter. Physics is the branch of physical science which studies the general properties of matter and the changes which matter undergoes without affecting its composition. Chemistry is the branch of physical science which studies the specific properties and composition of substances and changes in their composition.

Laws of Definite and Multiple Proportions.—Early in the development of the science of chemistry two important laws were formulated from the results of experiments. The first is called the law of definite proportions and may be stated as follows: In any chemical change there is always a definite relationship between the amounts of the substances entering into and produced by the change. For example, copper and sulphur, when heated together, will react upon each other, in the proportion of 63 parts of copper to 32 parts of sulphur, any excess of either substance being left unchanged. Also, there is a definite amount of the resulting substance formed by the reaction of any given amount of copper upon the corresponding amount of sulphur.

The second law is called the law of multiple proportions and may be stated as follows: If two substances react upon each other in different proportions by weight the amounts of each substance in the different reactions bear a simple relationship to each other. For example, sulphur will react upon oxygen in two ways: in one of the reactions a given amount of sulphur, as 32 parts, reacts upon 32 parts of oxygen, and in the other 32 parts of sulphur reacts upon 48 parts of oxygen. In these two different reactions, the amounts of the sulphur being equal, the relative amounts of the oxygen are to each other as two to three, a simple relationship.


Structure of Matter.—A mass, or body, is a separate portion of matter, like an iron rod, a block of marble, the water in a reservoir, or the air in a room. Any appreciable mass may be divided into smaller masses, but it appears that, if this division were carried far enough, extremely small particles would be obtained which could not be further subdivided without changing the identity of the substance. These particles are called molecules. They are so small in size that many millions are contained in even a small mass of matter, like a grain of sand, and there is no machinery fine enough to completely separate a mass into its molecules, nor is it possible to see them even with high magnification. The specific properties of a substance are due to the properties of its molecules and their influence on each other.

Molecules may be temporarily subdivided by other than mechanical processes, but, when this is accomplished, the identity of the substance changes. The smaller particles which are contained in molecules are called atoms. Atoms are the indivisible particles of which all matter is composed. They are united with each other to form molecules, the atoms existing separately only for an instant as they pass from one molecule to another. Any collection of molecules is a mass. The attraction of molecules for each other is called cohesion or adhesion. The attraction which causes atoms to unite with each other to form molecules is called chemical affinity, chemical attraction, or chemism.

The description above of the structure of matter is a brief statement of the very important atomic theory which is the foundation of the science of chemistry and has many applications in physics. While all parts of it cannot be rigorously proved, it is the only complete explanation of the facts of chemistry that has been formulated, and it is now generally accepted as being true.

Composition of Matter.—If the molecules of a substance are all alike and the atoms in each molecule are alike, the substance is of the simplest possible composition and cannot be separated into any other substances by any known means. Such a simple substance is called an element. About eighty elements have been discovered, each of the countless number of substances known being composed of one or more of these elements.

If the molecules forming a substance are all exactly alike, but each molecule contains different kinds of atoms, the substance is called a compound. The number of possible compounds formed from the eighty elements is very large, just as the number of words formed from the twenty-six letters of the alphabet appears to be unlimited. Many compounds are found in nature and many others are manufactured. It is not possible to separate the constituents of a compound from each other by physical processes, but a compound
may be separated into simpler compounds or elements by chemical processes, such a separation being called a chemical decomposition. In a compound the constituents which compose it are united with each other in a fixed and definite proportion by weight. The compound has a separate identity, and properties which are very different from those of its constituents.

If a substance is formed of different kinds of molecules it is called a mixture. In a mixture the different constituents are merely commingled with each other, and each retains its own identity and properties, and the properties of the mixture are between the properties of the constituents. It is generally possible to separate the constituents of a mixture from each other by mechanical processes, because of their different solubilities, melting-points or other specific properties, and the relative amounts of the constituents may vary.

Many natural substances, like granite and air, are mixtures, while others, like water, salt and marble, are compounds, and a few, like gold and sulphur, are elements. An absolutely pure compound or element is very unusual, the impurities being mixed with the compound or element.

An element is said to be in the nascent state for the instant during which it is being set free from a compound and before its atoms have united with each other to form molecules. When the atoms of an element have united to form molecules, a certain amount of energy is necessary to overcome their attraction for each other, so an element is more active, chemically, in the nascent state, as its atoms then exhibit their full chemical affinities.

**Kinds of Chemical Changes.**—*Combination*, when two or more substances unite with each other chemically to produce one substance, as the burning of hydrogen in oxygen to form water.

*Decomposition*, when one substance forms two or more substances as the result of a chemical change, as the decomposition of water by an electric current to form hydrogen and oxygen.

*Double decomposition*, when two substances react upon each other to produce two other substances by an exchange of constituents. This is the commonest kind of chemical change, and many examples of it will be studied.

There are many changes which belong to more than one of these kinds of chemical changes, one, called *substitution*, being quite common. In this kind of a change an element reacts upon a compound in such a way that a part of the compound is displaced, the first element taking its place, as the reaction between zinc and sulphuric acid, in which the zinc replaces the hydrogen of the acid, forming zinc sulphate and setting hydrogen free.
Symbols.—Early in the development of the science of chemistry it was found convenient to designate elements by signs. At first these were astronomical signs and geometrical figures, but as the number of known elements increased this method was found to be faulty and the present symbols came into use. The symbol of an element is formed from the English or some other name of the element by taking the first letter, alone, or with some other letter in the name. The first letter of a symbol is always a capital, and if there is a second letter, it is always a small letter. When symbols first came into use they were mere abbreviations of the names of the elements, and they are sometimes used in this way at present. In most cases, however, they stand for one atom of an element. If several atoms are to be indicated the symbol is multiplied by methods discussed in the next paragraph.

Formulas.—Chemical formulas are used to represent molecules. Formulas are made by writing the symbols of the elements in the molecules, one after the other. If a molecule contains only one atom of a certain element the symbol of that element indicates this; more than one atom of any element in a molecule is indicated by an inferior figure written after the symbol of that element, thus H₂O is the formula for water and tells us that each molecule of water contains two atoms of hydrogen and one atom of oxygen; H₂SO₄ is the formula for sulphuric acid, whose molecules each contain two atoms of hydrogen, one atom of sulphur and four atoms of oxygen. When for any reason a part of a molecule consisting of more than one atom is considered as a group to be multiplied, it is enclosed in a parenthesis, which is followed by the inferior figure, as in ammonium sulphate (NH₄)₂SO₄, whose molecule contains two NH₄ groups, combined with the SO₄ group. A formula generally represents one molecule of a compound. More than one molecule are shown by a line figure written before the formula, thus two molecules of ammonium sulphate are written 2(NH₄)₂SO₄.

Chemical Equations.—When a chemical change occurs, it is frequently possible to express what occurs by the use of symbols and formulas written in the form of an equation. Besides being of great convenience, these equations express exact relationships between the quantities of the substances entering into the reaction and produced by it; thus the decomposition of water is expressed 2H₂O = 2H₂ + O₂, which tells us that when water is decomposed two molecules yield two molecules of hydrogen and one molecule of oxygen. Actually very many molecules are involved when any appreciable amount of water is decomposed. This equation is sometimes written H₂O = 2H + O, but the first form is preferable, as it is the simplest way of writing all atoms combined to form molecules, which they do as soon as they are liberated from other molecules.
Chemical equations, like mathematical equations, must be balanced, that is, they must have the same number of atoms, of each kind involved, on both sides of the equality sign.

Atomic Weight.—Atoms are so extremely small that their absolute weights, in terms of any ordinary unit, are very small decimals which have been calculated, approximately, but they cannot be determined directly. The relative weights of the atoms of different elements have been quite accurately determined and have been referred to the weight of an atom of hydrogen as a unit. What is known as the atomic or combining weight of an element is the number of times that the weight of its atom is heavier than the weight of an atom of hydrogen. Thus the atomic weight of oxygen is 15.88, which means that an atom of oxygen is 15.88 times as heavy as an atom of hydrogen; the atomic weight of iron is 55.5, which means that an atom of iron weighs 55.5 times as much as an atom of hydrogen, and so on. The sum of the weights of the atoms in a molecule of a compound is the molecular weight of that compound.

Valence.—The atoms of different elements differ from each other in the number of atoms of other elements that they can combine with chemically to form molecules. This quantity of combining power of the atoms of an element is called its valence and is measured by the number of atoms of hydrogen that one atom of the element can combine with, or take the place of. The unit of valence is called a bond. An element whose atom combines with, or displaces, one atom of hydrogen, as Cl in HCl and Na in NaCl, has one bond and is called a monad element or simply a monad. An element whose atom combines with, or displaces, two atoms of hydrogen, as O in H2O and Ca in CaO, has two bonds and is called a dyad. Similarly a triad has three bonds; a tetrad, four; a pentad, five; a hexad, six. There are very few compounds in which an element appears to have a valence greater than six. The valence of an element is indicated, when necessary, by the use of superior Roman numerals written after the symbol of the element, as H', O'', C'''', etc.

Some of the elements have the same valence in all of their compounds, while others vary in valence, forming two or more series of compounds. When atoms combine to form molecules all bonds must be satisfied, the number of atoms in each molecule being the smallest number that will comply with this general law. If two elements have the same valence, one atom of each will form a molecule of their compound, as two monads in HCl', Na'Cl', two dyads in Fe''O'', Ca''S'', etc. Two atoms of a monad combine with one atom of a dyad, as in H'_2O''; three atoms of a monad with one atom of a triad, as in N'''/H'_3; two atoms of a dyad with one atom of a tetrad, as in C'''/O''', two atoms of a triad with three atoms
ACIDS, BASES AND SALTS

of a dyad, as in As"2"O"5; two atoms of a pentad with five atoms of a dyad, as in P"2"O"5, and so on.

**Acids, Bases and Salts.**—Some compounds containing hydrogen have several properties in common and are called *acids*. They generally have a sour taste and change certain dyes, like litmus, from blue to red. Many of the acids contain oxygen combined with the hydrogen, and the hydrogen alone or the hydrogen and oxygen are combined with some other element, called the *characteristic element* of the acid. Another class of compounds, called bases, contain oxygen, or oxygen and hydrogen, combined with another element, called the *characteristic element* of the base. An acid will exchange hydrogen for the characteristic element of a base forming water and a compound which is called a *salt*. This reaction between an acid and a base is called a *neutralization*. Examples: CaO + 2HCl = CaCl₂ + H₂O, Ca(OH)₂ + 2HCl = CaCl₂ + 2H₂O. Salts, however, may be formed in several other ways.

An element which combines with oxygen, or oxygen and hydrogen, to form a base is called a *metal* and is electro-positive. An element which combines with hydrogen, or hydrogen and oxygen, to form an acid is called a *non-metal* and is electro-negative. The classification of elements into metals and non-metals has long been used and was formerly based on physical properties. It is not exact, as there are some elements which behave both as metals and non-metals under different conditions. The differences are in degree, some elements always forming bases, like potassium and calcium, some always forming acids, like chlorine and sulphur, and a few forming both bases and acids, like antimony. There are about fifty metals, and they vary greatly in properties. Some are familiar substances, like iron, gold, copper, etc., while others are seldom seen in the free state and can only be obtained with difficulty from their compounds.

The metals are opaque and have a lustre when they are freshly cut and clean. They are conductors of both heat and electricity. Potassium, sodium, calcium, magnesium, zinc, iron, silver, copper, mercury, lead, tin, gold and aluminum are important metals. An *alloy* is formed by fusing two or more metals together. Some alloys appear to be unstable compounds and others appear to be mixtures. There are many important and widely used alloys, such as brass, type-metal and solder.

The non-metals vary in properties even more than the metals do. The principal non-metals are hydrogen, oxygen, sulphur, chlorine, bromine, iodine, nitrogen, phosphorus, carbon, silicon and boron.

An acid containing one atom of replaceable hydrogen in the molecule is said to be *monobasic*; similarly, acids with two, three and four atoms of replaceable hydrogen in the molecules are *di-basic*, *tri-basic* and *tetra-basic*, respectively. There are few acids with
more than four replaceable hydrogen atoms in the molecules. An acid composed of hydrogen and one other element and no oxygen is called a hydracid. These are few in number: hydrochloric acid, HCl; hydrobromic acid, HBr; hydriodic acid, HI; hydrofluoric acid, HF; and hydrosulphuric acid, H₂S, comprising the entire list of hydracids. Nearly all of the many other acids contain oxygen in addition to the hydrogen and the characteristic element and are called oxyacids. An acid anhydrid is a compound of a negative element with oxygen which will combine with water to form an acid, or which is formed, along with water, when an acid is decomposed, as SO₃ combines with water to form H₂SO₄; and CO₂ is formed, along with H₂O, on the decomposition of H₂CO₃. Different acids may be obtained from the same negative element, if it combines with different amounts of oxygen to form different anhydrides, or one of its anhydrides combines with different amounts of water.

A base may be either a compound of a metal and oxygen or of a metal, oxygen and hydrogen. A base that is soluble in water is called an alkali. Alkalies turn red litmus back to blue. Dyes, like litmus, whose colors are affected by acids and alkalies, are called indicators. A substance which affects an indicator like an acid is said to have an acid reaction. A substance which has the opposite effect on an indicator, behaving like an alkali, is said to have an alkaline reaction. Substances which do not affect the color of indicators are said to be neutral in reaction.

Salts formed by the complete neutralization of an acid by a base, in which all of the hydrogen of the acid is replaced by a metal, are called normal salts, as Na₂SO₄ from H₂SO₄. Such salts are generally neutral in reaction. A salt in which the base has not entirely neutralized an acid, and the metal has replaced only a part of the hydrogen of the acid, is called an acid salt, as NaHSO₄. They are generally acid in reaction. A salt in which the acid has not entirely neutralized the base, and which retains a part of the oxygen, or oxygen and hydrogen of the base, is called a basic salt, as BiOCl. A double salt is a salt that contains more than one metal, as KNaSO₄.

**Compound Radicals.**—There are certain groups of atoms, called compound radicals, which behave like single atoms. They do not exist separately, but one of them may be found in several different compounds, and will pass from one compound to another without its parts becoming separated. A compound radical has a definite valence, which is the difference between the valences of its constituent atoms. Some are electro-positive and behave like metals, and some are electro-negative and behave like non-metals. Many of these groups have commonly used names, like hydroxyl (OH)', ammonium (NH₄)', and cyanogen (CN)' From acids there is an important class of such groups, called acid radicals, and found in the
salts of the acids, each consisting of all of an acid excepting the replaceable hydrogen. For example, \((\text{NO}_3)^-\), from \(\text{HNO}_3\), is found in salts of nitric acid; \((\text{SO}_4)^-\), from \(\text{H}_2\text{SO}_4\), in salts of sulphuric acid; \((\text{PO}_4)^{3-}\), from \(\text{H}_3\text{PO}_4\), in salts of phosphoric acid, and so on.

**Nomenclature.**—Systematic rules have been gradually adopted for naming chemical compounds, and a knowledge of them is important, as it enables one to name a compound from its formula, or to give the formula from its name. These names are confusing, as many compounds, like water, salt and marble, have long been known as separate substances and bear common names, called *synonyms*, which have no relationship to their chemical composition; also, the rules have been changed and improved from time to time, but compounds are often called by names given to them under old rules.

The only rule governing the naming of elements is that "-um" indicates a metal, though there are several important metals, like iron, copper and gold, whose commonly used names do not conform to this rule. Some of the elements, like gold, sulphur and iron, have been known for a long time and their names have long been used. Those discovered more recently have been named from some striking property, or from the localities in which they were first found.

Many of the metals vary in valence and form two distinct series of compounds. These are distinguished from each other by changing the end of the name of the metal to -ous for the compounds in which the metal has the lower valence, and to -ic for the compounds in which it has the higher valence. Examples: compounds of mercury in which the mercury has a valence of one are the mercurous compounds, and compounds in which mercury has a valence of two are the mercuric compounds; compounds of iron (ferrum) in which the iron has a valence of two are the ferrous compounds, and compounds in which iron has a valence of three are the ferric compounds.

A *binary compound* is a compound containing two elements only, like \(\text{HCl}, \text{H}_2\text{O}\), etc. A binary compound is called by the name of the positive element in the compound followed by the name of the negative element with its last part changed to -ide. Example: \(\text{NaCl}\) is sodium chloride. If two elements form two or more compounds with each other, these compounds may sometimes be distinguished by the rule for distinguishing different series of compounds of metals. Examples: \(\text{HgCl}\) is mercurous chloride and \(\text{HgCl}_2\) is mercuric chloride. Or prefixes are used to indicate the number of atoms of the negative element in a molecule of the compound, the number of atoms of the positive element remaining the same. Examples: \(\text{H}_2\text{O}\) is hydrogen monoxide; \(\text{H}_2\text{O}_2\) is hydrogen dioxide. Compound radicals are often considered as elements, and their compounds may be named like binary compounds. Examples: \(\text{NH}_4\text{Cl}\) is ammonium chloride; \(\text{Hg(CN)}_2\) is mercuric
cyanide. Bases are named by the binary rule, thus CaO is calcium oxide and Ca(OH)₂ is calcium hydroxide.

Hydracids are named by taking the name of the characteristic element, changing its last part to -ic and prefixing hydro. Examples: HCl is hydrochloric acid; H₂S is hydrosulphuric acid. By the binary rule these would be hydrogen chloride and hydrogen sulphide, respectively, and such names are sometimes used.

Each negative element generally forms several oxyacids. In naming the oxyacids formed by an element, one containing a relatively large number of oxygen atoms in its molecules is called by the name of the element with its last part changed to -ic. Example: HClO₃ is chloric acid. The acid containing the next smaller number of oxygen atoms in its molecules is named by taking the name of the characteristic element and changing its last part to -ous. Example: HClO₂ is chlorous acid. If there is an acid with still less oxygen than in the “ous” acid it is called by the name of the characteristic element, changing its last part to -ous and prefixing hypo. Example: HClO is hypochlorous acid. If there is an acid with more oxygen in the molecules than is contained in the “ic” acid, it is called by the name of the characteristic element, changing its last part to -ic and prefixing per-. Example: HClO₄ is perchloric acid.

Salts of hydro-acids are binary compounds and are named by the binary rule. Example: CaCl₂ is the calcium salt of hydrochloric acid and is named calcium chloride. Salts of oxyacids are named from the names of the acids. A salt of an acid whose name ends in -ic is called by the name of the positive element or radical followed by the name of the acid with its last part changed to -ate. Examples: KClO₃ is the potassium salt of chloric acid and its name is potassium chlorate; similarly KClO₄ is potassium perchlorate. A salt of an acid whose name ends in -ous is called by the name of the positive element, followed by the name of the acid with its last part changed to -ite. Examples: KClO₂ is the potassium salt of chlorous acid and its name is potassium chlorite; similarly KClO is potassium hypochlorite. Salts were formerly named in reverse order and the name of the metal modified in ways no longer in good usage, but these older names are often seen. Examples: sodium chloride was formerly called chloride of soda; potassium chlorate was formerly called chlorate of potash, and so on.

Acid salts are generally designated by the prefix bi-, as in sodium bi-carbonate, which is Na₂HCO₃, sometimes called sodium hydrogen carbonate or sodium acid carbonate. Basic salts are commonly designated by the prefixes sub- or oxy-, as in bismuth subchloride, which is BiOCl, sometimes called bismuth oxychloride or bismuth basic chloride. The name of a double salt contains the names of both metals in the salt, as in potassium-sodium sulphate, KNaSO₄.
There are some compounds whose names are not included in the rules above, but they may be learned separately as the compounds themselves are studied.

Solution.—All substances, when brought into contact with liquids, are affected by the liquid to a greater or less extent. A solid substance, on being mixed with a liquid, may lose its solid condition entirely and become a part of the liquid, when it is said to be dissolved, a solution being formed; or a great part of the solid may be merely suspended in the liquid in the form of small solid particles. Solids which dissolve in a liquid are said to be soluble in the liquid, which is called a solvent. Solids which will not dissolve in a liquid are said to be insoluble in that liquid, though this is a comparative matter only, as all solids will dissolve in liquids to some extent. So-called insoluble substances are only very slightly soluble. When a liquid has dissolved all of a substance that it can dissolve at the ordinary temperature the solution is said to be saturated. If the solvent is heated it will generally dissolve more of a solid than at lower temperatures, and if an excessive amount is thus dissolved the solution is said to be super-saturated, the excess being deposited from the solution on cooling. All liquids and gases dissolve in liquids to a greater or less extent, in the same way that solids do. When two liquids dissolve each other in all proportions they are said to be miscible. Solution is a phenomenon which is of great importance in natural processes and in manufacturing operations.

Insoluble matter in a liquid is called a sediment; when an insoluble substance is formed in a solution by chemical reaction or other means it is called a precipitate, and the process is called precipitation. If the constituents of a solution can react to form an insoluble substance or precipitate they will generally do so. This is called the law of precipitation, and much use is made of it in chemical processes.

To remove a sediment or precipitate from a liquid we generally employ paper filtration in analytical work. This consists of pouring the liquid containing the precipitate on a smooth folded paper filter contained in a glass funnel. The clear liquid which passes through the paper is called a filtrate. The folded paper should not extend quite to the edge of the funnel, and it is generally better to wet the paper with water before filtering if a watery liquid is to be filtered. If the filtrate does not come clear it will often do so if it is passed several times through the same filter containing the precipitate.

The nature of solution was long unknown, but it has been carefully studied during recent years. At first sight it appears, merely, that when a substance goes into solution the influences of the molecules on each other are overcome as the attraction between the molecules in a solid, or the repulsion between the molecules of a gas, disappear.
Some solutions appear to be of this character, but in others there is a much more radical change.

If two fluids, liquids or gases, which are miscible, are brought into contact with each other, they immediately begin to mix, and this spontaneous mixing will continue until a uniform mixture results. It is rapid for gases and slow for liquids. When the liquids are only superimposed, one on the other, the process is called diffusion; when they are separated by a porous membrane the process is called osmosis. Osmosis is a selective process when applied to solutions, some substances in solution passing through the porous membrane with the solvent, while others do not pass through. The substances which will osmose with their solvents are called crystalloids, and those that will not osmose are called colloids. Dialysis is the process of separating crystalloids from colloids by osmosis. Colloids and crystalloids are in quite different conditions in their solutions. Gelatin, starch, gums, soap and rubber are colloids with most solvents, also certain hydroxides and other compounds of metals, such as are found in milk of magnesia and dialyzed iron. Colloids do not form true solutions, as the molecular influences are not overcome. They appear to swell and absorb the solvent to form a pasty or liquid mixture. The boiling- and freezing-points of the solvent are not changed, as they are in true solutions.

When crystalloids dissolve there may be only a molecular dispersion, as in the solution of sugar in water, but this is exceptional. In nearly all solutions of crystalloids the molecules of the dissolved substances are not only dispersed, but they are partially broken down, or dissociated, into positive and negative parts called ions. Example: in the solution of sodium nitrate some of the molecules of NaNO₃ are separated into the electro-positive ion Na and the electro-negative ion NO₃. The relative number of molecules thus disassociated varies with the identity of the compound, the solvent, the temperature and the strength of the solution. In weak solutions the dissociation is relatively greater than in strong solutions. The electrical conductivity of solutions is due to the positive and negative characters of the ions. When a compound is decomposed by an electric current the process is called electrolysis.

Water of Crystallization.—Many chemical compounds tend to assume characteristic geometric forms, called crystals, when the compounds are deposited from a solution as the solvent evaporates, or when the compound solidifies after fusion, or is condensed from the gaseous condition. A crystalline compound is one which is in the form of crystals, and an amorphous compound is one which is not in the form of crystals. The crystals of a compound are generally alike in form, though they may vary widely in size, and they may retain some loosely combined water, which is called water of crystal-
lization, and without which the compound will not form crystals or crystallize. Example: copper sulphate in the crystalline form has a deep blue color and has the formula CuSO₄5H₂O. If this is heated moderately the water is driven off and the residue of CuSO₄ is a white amorphous powder. Not all crystals, however, contain water of crystallization. Example: common salt, sodium chloride, readily crystallizes and has the formula NaCl, without any water in the molecules.

The water of crystallization is combined in the molecules of a substance and does not appear as moisture. If the water of crystallization is expelled from a compound by heating, the process is called exsiccation and the compound is then said to be anhydrous. A compound which yields all or part of its water of crystallization to the air is said to be efflorescent. A substance which absorbs moisture from the air is hygroscopic, and if the water appears as moisture, dissolving the substance partially or completely, the substance is deliquescent. Examples: crystalline washing soda, sodium carbonate, has the formula Na₂CO₃10H₂O and effloresces in dry air, falling to an amorphous powder. Anhydrous copper sulphate will absorb water from moist air, changing from white to blue in color. Fused calcium chloride, CaCl₂, is a deliquescent substance, first absorbing water to form CaCl₂6H₂O, and then absorbing more water, becoming moist and ultimately dissolving in the water.

When a compound containing water of crystallization is dissolved in water the water of crystallization becomes a part of the solvent and is generally omitted in giving the formula of the compound, but when a crystalline compound is not dissolved the water of crystallization often must be considered. Example: alum crystals have the formula KAl(SO₄)₂ 12H₂O, being nearly one-half water, but we use the formula KAl(SO₄)₂ when referring to alum, unless there is a special reason for including the water of crystallization.

Chemical analysis is the separation of a substance into its constituents by chemical processes. Qualitative analysis is the identification of the constituents of a substance. Quantitative analysis is the determination of the relative amounts of the constituents of a substance. Qualitative analysis may be used to ascertain the composition of an unknown substance, or to find the presence or absence of impurities in a known substance.

Each element and compound radical has certain characteristic reactions which are used to separate and identify it. Any substance used to bring about a reaction is called a reagent. If it is used in the form of a solution the solution is called a test-solution. A list of the most often used test-solutions and their strengths will be found at the end of this book.
THE METALS.

It is customary to separate and identify the metals in a substance before looking for the acid radicals and non-metals. The scheme generally used for the separation and identification of the metals depends upon the fact that a large majority of the metals, in solutions of their compounds, will form precipitates with hydrogen sulphide, \( \text{H}_2\text{S} \), under various conditions, these precipitates varying widely in their properties. In carrying out this scheme it is necessary that the substances be in solution, and, at the beginning of the study of qualitative analysis, the work is done upon solutions of known and unknown composition. Afterward, some practice on the bringing of substances into solution is necessary.

In this manual the principal metals are classified into seven groups, as follows:

**Group 1.**—Metals precipitated as chlorides by hydrochloric acid, \( \text{HCl} \): silver, Ag; mercurous mercury, \( \text{Hg}^+ \); and all but a very small amount of lead, \( \text{Pb} \).

**Group 2.**—Metals precipitated as sulphides by hydrogen sulphide, \( \text{H}_2\text{S} \), in the presence of hydrochloric acid, \( \text{HCl} \), and whose sulphides are insoluble in ammonium sulphide, \( (\text{NH}_4)_2\text{S}_x \): mercuric mercury, \( \text{Hg}^{\prime} \); bismuth, \( \text{Bi} \); copper, \( \text{Cu} \); cadmium, \( \text{Cd} \); and the small amount of lead not removed with group 1.

**Group 3.**—Metals precipitated as sulphides by hydrogen sulphide, \( \text{H}_2\text{S} \), in the presence of hydrochloric acid, \( \text{HCl} \), and whose sulphides are soluble in ammonium sulphide, \( (\text{NH}_4)_2\text{S}_x \): arsenic, \( \text{As} \); antimony, \( \text{Sb} \); tin, \( \text{Sn} \); gold, \( \text{Au} \); and platinum, \( \text{Pt} \).

**Group 4.**—Metals precipitated as hydroxides by ammonium hydroxide, \( \text{NH}_4\text{OH} \), in the presence of ammonium chloride, \( \text{NH}_4\text{Cl} \): iron, \( \text{Fe} \); chromium, \( \text{Cr} \); and aluminum, \( \text{Al} \).

**Group 5.**—Metals precipitated as sulphides by ammonium sulphide, \( (\text{NH}_4)_2\text{S} \), or hydrogen sulphide, \( \text{H}_2\text{S} \), in the presence of ammonium hydroxide, \( \text{NH}_4\text{OH} \): cobalt, \( \text{Co} \); nickel, \( \text{Ni} \); manganese, \( \text{Mn} \); and zinc, \( \text{Zn} \).

**Group 6.**—Metals precipitated as carbonates by ammonium carbonate, \( (\text{NH}_4)_2\text{CO}_3 \), in the presence of ammonium chloride, \( \text{NH}_4\text{Cl} \), and ammonium hydroxide, \( \text{NH}_4\text{OH} \): barium, \( \text{Ba} \); strontium, \( \text{Sr} \); and calcium, \( \text{Ca} \).

**Group 7.**—Metals not precipitated by any group reagent: magnesium, \( \text{Mg} \); potassium, \( \text{K} \); sodium, \( \text{Na} \); lithium, \( \text{Li} \); and the compound radical ammonium, \( (\text{NH}_4)^+ \).
The general reagents used to precipitate the groups are called group reagents and they will usually precipitate the metals of the preceding groups, so, in separating the metals, each of the group reagents must be used in exactly the order given. If there is no precipitate formed when a group reagent is added, that group is absent and we pass on to the next group. If there is a precipitate formed the reagent is slowly added until precipitation appears to be complete. The precipitate is then filtered out of the liquid, and the filtrate tested first with a small amount of the last used reagent, to be sure that precipitation by it is complete; and it is then tested for the next group.

Nearly all of the tests described in this text-book are to be carried out with solutions. Unless other directions are given, from one-quarter to one-half an inch of the solution to be tested is poured into a test-tube, for each test, and a few drops of the reagent are added, which are generally sufficient, unless it is necessary to completely remove the radical being precipitated. Diluted acids and solutions of salts are used as reagents unless otherwise specified.

Success in the practice of qualitative analysis depends very largely upon the ability to recognize the reactions obtained. This ability is acquired only by experience, and much practice is necessary upon solutions whose composition is known, called known solutions. After becoming familiar with the reactions by work on known solutions, the student can undertake the analysis of unknown solutions of gradually increasing difficulty.

**GROUP 1.**

Metals precipitated as chlorides by hydrochloric acid, HCl.

Lead, Pb; mercurous mercury, Hg⁺; silver, Ag.

**LEAD (PLUMBUM), Pb^{II} = 207.10.**

Lead is largely used in the metallic state, alone and in many alloys, like solder, type-metal and pewter. The compounds of lead vary in color some being white, some yellow, red or brown. Lead acetate and lead nitrate are readily soluble in water, but all other important salts of lead are insoluble or only very slightly soluble in water. Lead compounds are dangerous poisons, in single large doses or in small doses taken over a long period of time.

**IMPORTANT COMPOUNDS OF LEAD.**

Lead carbonate, basic lead carbonate, "white lead," "cerussa," (PbCO₃)₂Pb(OH)₂.
Lead chromate, "chrome yellow," PbCrO₄.
Lead iodide, PbI₂.
Lead nitrate, Pb(NO₃)₂.
Lead dioxide, lead peroxide, PbO₂.
Lead sulphide, “galena,” PbS.

**TESTS FOR LEAD.**

Use a separate portion of a solution of lead acetate, Pb(C₂H₃O₂)₂, or of lead nitrate, Pb(NO₃)₂, for each of the following tests:

1. Add HCl, obtaining a white precipitate of lead chloride, PbCl₂.
   This precipitate is slightly soluble in water, so the lead is not all precipitated, and a weak solution of a lead salt will not give the reaction. Pour a portion of the liquid containing the precipitate of PbCl₂ on a filter, pass hot water through the filter and the precipitate will dissolve.
   Soluble chlorides, as KCl, will give the same precipitate.
2. Add KI, obtaining a yellow precipitate of lead iodide, PbI₂.
3. Pass H₂S gas through the lead solution, obtaining a black precipitate of lead sulphide, PbS.
   (NH₄)₂S will give the same precipitate.
4. Add H₂SO₄, obtaining a white precipitate of lead sulphate, PbSO₄.
   Soluble sulphates, as K₂SO₄, will give the same precipitate.
5. Add a few drops of NaOH, obtaining a white precipitate of lead hydroxide, Pb(OH)₂, which forms sodium plumbite, Na₂PbO₂, and dissolves on adding an excess of the NaOH.
6. Add K₂Cr₂O₇, obtaining a yellow precipitate of lead chromate PbCrO₄.
   Soluble chromates, as K₂CrO₄, give the same precipitate.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. Pb(C₂H₃O₂)₂ + HCl = Pb(NO₃)₂ + KCl = Pb(C₂H₃O₂)₂ + CaCl₂ =
2. Pb(C₂H₃O₂)₂ + KI = Pb(NO₃)₂ + NH₄I =
3. Pb(NO₃)₂ + H₂S = Pb(C₂H₃O₂)₂ + (NH₄)₂S =
4. Pb(NO₃)₂ + H₂SO₄ = Pb(C₂H₃O₂)₂ + (NH₄)₂SO₄ =
5. Pb(NO₃)₂ + NaOH = Pb(OH)₂ + NaNO₃ =
6. 2Pb(C₂H₃O₂)₂ + K₂Cr₂O₇ + H₂O = 2PbCrO₄ + 2K₂C₂H₃O₂
   + 2HCl + 2H₂O.
   Pb(NO₃)₂ + K₂CrO₄ =
MERCURIOUS MERCURY, $\text{Hg}^i = 200.6$.

Hydrargyrum, U. S. P., "Quicksilver."

Mercury is a silvery white liquid metal which is largely used in the metallic state in thermometers, barometers and metallurgical processes. When thoroughly mixed with some substances, as chalk, syrup, honey, and fats, it is extinguished, that is, divided into minute globules which remain separated, and the metal in this condition has a dark gray color. Mass of mercury, mercurial ointment and mercury with chalk all contain extinguished metallic mercury. Alloys containing mercury are called amalgams.

There are two classes of mercury salts, both of which are important: the mercurous salts considered here, and the mercuric salts considered in group 2. The mercurous compounds vary in color, from white through yellow and green to black. They are nearly all insoluble in water, the nitrate being the only important salt which is readily soluble. Compounds of mercury are poisonous and are volatilized by heat, with or without decomposition.

IMPORTANT MERCURIOUS COMPOUNDS.


$\text{HgCl}$.


Mercurous nitrate, $\text{HgNO}_3$.

Mercurous oxide, $\text{Hg}_2\text{O}$.

TESTS FOR MERCURIOUS MERCURY.

Use a separate portion of a solution of mercurous nitrate, $\text{HgNO}_3$, for each of the following tests.

1. Add $\text{HCl}$, obtaining a white precipitate of mercurous chloride, $\text{HgCl}$.

Collect a small portion of the precipitate on a filter and pass hot water through the filter and the precipitate will not dissolve. Pour $\text{NH}_4\text{OH}$ through the filter and the precipitate will blacken, forming dimercurous ammonium chloride, $\text{NH}_2\text{Hg}_2\text{Cl}$.

2. Add $\text{KI}$, obtaining a greenish-yellow precipitate of mercurous iodide, $\text{HgI}$.

3. Pass $\text{H}_2\text{S}$ gas through the solution, obtaining a black precipitate, consisting of *mercuric sulphide, $\text{HgS}$, and metallic mercury. $(\text{NH}_4)_2\text{S}$ will give the same precipitate.

4. Add $\text{NaOH}$, obtaining a black precipitate of mercurous oxide, $\text{Hg}_2\text{O}$.

The same precipitate is obtained with lime water, $\text{Ca(OH)}_2$. A
mixture formed from calomel and lime water is an old-fashioned remedy, called "black wash."

5. Pour 5 mils of the mercury solution into a small beaker. Place a small piece of copper foil in the solution. The copper becomes covered with a gray deposit of metallic mercury, which brightens when rubbed. Dry the coated copper foil, put it in a dry test-tube and heat gently over the flame of a Bunsen burner. The mercury will volatilize and condense on the cooler part of the test-tube as a gray deposit, which consists of small globules of the metal.

Mercury will deposit from its compounds and amalgamate with many metals as with the copper in this test and care must be taken not to allow mercury or its compounds to come in contact with gold rings.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. \[ \text{HgNO}_3 + \text{HCl} = \]
   \[ 2\text{HgCl} + 2\text{NH}_4\text{OH} = \text{NH}_2\text{Hg}_2\text{Cl} + \text{NH}_4\text{Cl} + 2\text{H}_2\text{O} \]
2. \[ \text{HgNO}_3 + \text{KI} = \]
   \[ \text{HgNO}_3 + \text{NH}_4\text{I} = \]
3. \[ \text{HgNO}_3 + \text{H}_2\text{S} = \]
   \[ \text{HgNO}_3 + (\text{NH}_4)_2\text{S} = \]
4. \[ \text{HgNO}_3 + \text{NaOH} = \]
   \[ \text{HgCl} + \text{Ca(OH)}_2 = \]
5. \[ \text{HgNO}_3 + \text{Cu} = \]

**SILVER (ARGENTUM), \( \text{Ag}^i = 107.12 \).**

Silver is largely used in the metallic state, being generally alloyed with a small amount of copper to harden it. Its compounds are poisonous and unstable, being easily reduced to the oxide or metallic silver by reducing agents, including organic matter. This reduction is rapid in actinic light. Most of the important silver salts are colorless, but the iodide is yellow and the oxide is black. The nitrate is the only important salt which is readily soluble in water.

**IMPORTANT COMPOUNDS OF SILVER.**

Silver chloride, \( \text{AgCl} \).
Silver cyanide, \( \text{AgCN} \).
Silver iodide, \( \text{AgI} \).
Silver nitrate, \( \text{Argenti nitras, U. S. P., "lunar caustic," AgNO}_3 \).
Silver oxide, \( \text{Argenti oxidum, U. S. P., Ag}_2\text{O} \).
Silver sulphate, \( \text{Ag}_2\text{SO}_4 \).

**TESTS FOR SILVER.**

Use a separate portion of a solution of silver nitrate, \( \text{AgNO}_3 \), for each of the following tests:
1. Add HCl, obtaining a white precipitate of silver chloride, AgCl, insoluble in acids, but very soluble in ammonia water, NH₄OH, forming ammonio-silver chloride, (NH₃)₂AgCl.

   Collect a small portion of the precipitate of AgCl on a filter. Pour hot water though the filter and the precipitate will not dissolve. Pour NH₄OH through the filter and the precipitate will dissolve. Add HNO₃ to the ammonio-silver solution and the precipitate, AgCl, will reappear.

   Soluble chlorides, as NaCl, will precipitate silver as silver chloride.

2. Add KI, obtaining a light yellow precipitate of silver iodide, AgI, slowly soluble in an excess of the reagent as potassium silver iodide, KAgI₂.

3. Add KCN, obtaining a white precipitate of silver cyanide, AgCN, readily soluble in an excess of the reagent as potassium silver cyanide, KAg(CN)₂.

4. Pass H₂S gas through the silver solution, obtaining a black precipitate of silver sulphide, Ag₂S.

   (NH₄)₂S will give the same precipitate.

5. Add NaOH, obtaining a brown precipitate of silver oxide, Ag₂O, insoluble in an excess of the reagent, but readily soluble in nitric and acetic acids and ammonia water.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. \[ \text{AgNO}_3 + \text{HCl} = \]
   \[ \text{Ag}_2\text{SO}_4 + \text{NaCl} = \]
   \[ \text{AgCl} + \text{NH}_4\text{OH} = (\text{NH}_3)_2\text{AgCl} + \]
   \[ (\text{NH}_3)_2\text{AgCl} + 2\text{HNO}_3 = \text{AgCl} + 2\text{NH}_4\text{NO}_3 \]

2. \[ \text{AgNO}_3 + \text{KI} = \]
   \[ \text{AgC}_2\text{H}_3\text{O}_2 + \text{NH}_4\text{I} = \]
   \[ \text{AgI} + \text{KI} = \]

3. \[ \text{AgNO}_3 + \text{KCN} = \]
   \[ \text{Ag}_2\text{SO}_4 + \text{KCN} = \]

4. \[ \text{AgNO}_3 + \text{H}_2\text{S} = \]
   \[ \text{Ag}_2\text{SO}_4 + (\text{NH}_4)_2\text{S} = \]

5. \[ \text{AgNO}_3 + \text{NaOH} = \]
   \[ \text{Ag}_2\text{O} + \text{HNO}_3 = \]

SAMPLE NUMBER 1.

Analysis of a solution for group 1 of the metals.

The solution may contain salts of lead, mercurous mercury, or silver, or any two of these metals, or all three of them.

Dip a small piece of red litmus paper in the solution. If the color of the litmus is not affected the solution is neutral or acid in reaction,
as it should be. If the color changes to blue add HNO₃ T.S. slowly, with stirring, until the liquid will change the color of blue litmus paper to red.

A. To about 10 mils of the solution contained in a test-tube, add a few drops of HCl T.S. If no precipitate forms, silver and mercurous mercury are absent, and lead, if present, is in very small amount, which will be detected in group 2. If a white precipitate forms it consists of chlorides of metals of group 1. Continue to add the HCl T.S. until the precipitation appears to be complete. Filter the liquid and test the clear filtrate by adding a few drops of HCl T.S. If this produces a precipitate add more HCl T.S., filter through the same filter paper and test the last portion of the filtrate in the same manner as before, and repeat the filtration and testing of the filtrate until the HCl T.S. produces no precipitate in the filtrate, when precipitation is complete. A small amount of lead, however, remains in the solution, if this metal is present.

B. Heat some water to boiling in a beaker supported on wire gauze over a gas flame. Pour 20 mils of this hot water through the precipitate on the filter paper. If lead is present, PbCl₂ dissolves. Test separate portions of the filtrate for lead with KI T.S., H₂S, H₂SO₄ T.S., and K₂Cr₂O₇ T.S. as described on page 17, remembering that the precipitates, if any, are small in amount because the solution of lead is very weak.

If lead is found pass 50 mils or more of boiling water through the precipitate on the filter paper to wash out the remainder of the PbCl₂. When the PbCl₂ is all washed out a portion of the filtrate will not give a white precipitate with H₂SO₄ T.S., with which it should be tested.

If the precipitate all dissolves in hot water it consisted of PbCl₂ alone, and silver and mercurous mercury are absent.

C. If a precipitate remains on the filter paper it consists of AgCl or HgCl, or both. Pour a small amount of NH₄OH T.S. through the precipitate on the filter. If the precipitate blackens, the presence of mercurous mercury is shown, as described on page 25.

D. If silver is present the AgCl dissolves in the NH₄OH T.S. Add HNO₃ T.S. to the filtrate until it is acid in reaction to litmus. A white precipitate shows the presence of silver, as described on page 27.

GROUP 2.

Metals precipitated by hydrogen sulphide, H₂S, in presence of hydrochloric acid, HCl, and whose sulphides are insoluble in ammonium sulphide, (NH₄)₂Sₓ.

Mercuric mercury, Hg²⁺; (lead, Pb); bismuth, Bi; copper, Cu; cadmium, Cd.
MERUCRIC MERCURY (HYDRARGYRUM), $\text{Hg}^{II} = 200.6$.

Oxidizing agents readily change mercurous compounds to mercuric compounds and reducing agents readily change mercuric compounds to mercurous compounds. As mercuric compounds are much more poisonous than mercurous compounds, care must be taken not to prescribe or dispense any mercurous compound for medicinal use in such a way that it might be oxidized to the mercuric condition.

Mercuric compounds are variously colored, being white, yellow, red or black. Most of them are insoluble or only slightly soluble in water, but mercuric chloride, mercuric nitrate, and mercuric cyanide are soluble.

IMPORTANT MERCURIC COMPOUNDS.

Mercuric-ammonium chloride, $\text{Hydrargyrum ammoniatum, U. S. P.}$, ammoniated mercury, “white precipitate,” $\text{NH}_2\text{HgCl}$.

Mercuric bromide, $\text{HgBr}_2$.

Mercuric chloride, $\text{Hydrargyri chloridum corrosivum, U. S. P.}$, “corrosive sublimate,” $\text{HgCl}_2$.

Mercuric cyanide, $\text{Hg(CN)}_2$.

Mercuric iodide, $\text{Hydrargyri iodidum rubrum, U. S. P.}$, “biniodide of mercury,” $\text{HgI}_2$.

Mercuric nitrate, $\text{Hg(NO}_3)_2$.

Yellow mercuric oxide, $\text{Hydrargyri oxidum flavum, U. S. P.}$, $\text{HgO}$.

Red mercuric oxide, $\text{Hydrargyri oxidum rubrum, U. S. P.}$, “red precipitate,” $\text{HgO}$.

Mercuric sulphate, $\text{HgSO}_4$.

Mercuric subsulphate, “turpeth mineral,” $(\text{HgO})_2\text{HgSO}_4$.

Black mercuric sulphide, “ethiops mineral,” $\text{HgS}$.

Red mercuric sulphide, “cinnabar,” “vermilion,” $\text{HgS}$.

TESTS FOR MERCURIC MERCURY.

Use a separate portion of a solution of a mercuric salt, as mercuric chloride, $\text{HgCl}_2$, for each of the following tests:

1. Pass $\text{H}_2\text{S}$ through the mercuric solution. A black precipitate of mercuric sulphide, $\text{HgS}$, is formed, but several other colors, ranging from white through yellow, orange and brown, generally appear before the final black precipitate. This range of colors is due to the temporary formation of compounds of $\text{HgS}$ with the original mercuric salt.

2. Add NaOH T.S. to the mercuric solution. An excess of the alkali precipitates yellow mercuric oxide, $\text{HgO}$. When the mercuric salt is in excess, the precipitate is brown and consists of a basic salt of mercury. The same precipitate is obtained with lime water, $\text{Ca(OH)}_2$, and the mixture formed is the old fashioned “yellow wash.”
3. Add NH₄OH T.S. to the mercuric solution. A white precipitate of mercurammonium chloride, NH₂HgCl, is formed. Add HCl T.S. The precipitate will dissolve.

4. Slowly add KI T.S. to the mercuric solution. A precipitate of mercuric iodide, HgI₂, first yellow, then red, is produced, which forms potassium-mercuric iodide, (KI)₂HgI₂, and dissolves in an excess of the reagent.

5. Slowly add SnCl₂ T.S. to the mercuric solution. At first the precipitate formed is white and consists of mercurous chloride, HgCl. When the reagent is in excess, the precipitate is gray and consists of finely divided metallic mercury.

6. Acidulate a small amount of the mercuric solution in a small beaker with HCl T.S. and introduce a small piece of copper foil into the liquid. Mercury is deposited on the copper, just as was described for mercurous compounds on page 18.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. \[ \text{HgCl}_2 + \text{H}_2\text{S} = \text{Hg(NO}_3\text{)}_2 + (\text{NH}_4)_2\text{S} = \]

2. \[ \text{HgCl}_2 + \text{NaOH} = \text{HgCl}_2 + \text{Ca(OH)}_2 = \]

3. \[ \text{HgCl}_2 + \text{NH}_4\text{OH} = \text{NH}_2\text{HgCl} + \text{NH}_2\text{HgCl} + \text{HCl} = \]

4. \[ \text{HgCl}_2 + \text{KI} = \text{Hg(NO}_3\text{)}_2 + \text{NH}_4\text{I} = \]

5. \[ \text{HgCl}_2 + \text{SnCl}_2 = \text{HgCl} + \text{SnCl}_2 = \]

6. \[ \text{HgCl}_2 + \text{Cu} = \]

**BISMUTH, Bi\text{iii, v} = 208.0.**

Bismuth is a hard and brittle crystalline metal which is a constituent of some easily fusible alloys and is used in manufacturing its compounds. The salts of bismuth are generally white in color, but the oxide is yellow and the sulphide is black. Bismuth chloride and bismuth nitrate are soluble in water with some decomposition. The other important compounds are insoluble. Bismuth compounds in solution readily form insoluble basic compounds containing the monad radical bismuthyl, (BiO)'. Some organic acids or large amounts of mineral acids prevent this precipitation. Bismuthic compounds are unimportant.

**IMPORTANT COMPOUNDS OF BISMUTH.**

- Bismuth chloride, BiCl₃.
- Bismuth subcarbonate, U. S. P., mainly (BiO)₂CO₃H₂O.
- Bismuth nitrate, Bi(NO₃)₃.
- Bismuth oxide, Bi₂O₃.
- Bismuth subnitrate, U. S. P., mainly, Bi(OH)₂NO₃.
TESTS FOR BISMUTH.

Use a separate portion of a solution of either bismuth nitrate, Bi(NO$_3$)$_3$, or bismuth chloride, BiCl$_3$, for each of the following tests:

1. Pour 2 mils of the bismuth solution into 10 mils of water contained in a test-tube. A white precipitate of bismuth subnitrate, BiONO$_3$, or of bismuth subchloride, BiOCl, is produced if the liquid is not too strongly acid.

2. Pass H$_2$S through the bismuth solution. A black precipitate of bismuth sulphide, Bi$_2$S$_3$, is produced.

3. Add NaOH T.S. to the bismuth solution. A white precipitate of bismuth hydroxide, Bi(OH)$_3$, is formed. Boil the liquid containing the precipitate. The precipitate changes to yellow bismuth oxide, Bi$_2$O$_3$. If reducing agents are present the precipitate becomes black and then consists of metallic bismuth.

4. Add Na$_2$CO$_3$ T.S. to the bismuth solution. A white precipitate of bismuth subcarbonate, (BiO)$_2$CO$_3$, is produced.

5. Add K$_2$CrO$_4$ T.S. to the bismuth solution. A yellow precipitate of bismuth subchromate, (BiO)$_2$CrO$_4$ is formed, which is insoluble in NaOH T.S. Solutions of chromates, as K$_2$CrO$_4$, give the same precipitate.

6. Slowly add NaOH T.S. to 1 mil of SnCl$_2$ T. S. until the precipitate first formed dissolves, forming sodium stannite, Na$_3$SnO$_2$. Add a few drops of the bismuth solution to the Na$_2$SnO$_2$ solution. A black precipitate of metallic bismuth, Bi, is produced.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. Bi(NO$_3$)$_3$ + H$_2$O = BiCl$_3$ + H$_2$O
2. BiCl$_3$ + H$_2$S = Bi(NO$_3$)$_3$ + (NH$_4$)$_2$S
3. Bi(NO$_3$)$_3$ + NaOH = BiCl$_3$ + KOH = Bi(OH)$_3$ + heat
4. 2Bi(NO$_3$)$_3$ + 3Na$_2$CO$_3$ = (BiO)$_2$CO$_3$ + 6NaNO$_3$ + 2 CO$_2$
   BiCl$_3$ + K$_2$CO$_3$ =
5. BiCl$_3$ + K$_2$CrO$_4$ + 5H$_2$O = 2(BiO)$_2$CrO$_4$ + 2KCl + 10HCl
   Bi(NO$_3$)$_3$ + K$_2$CrO$_4$ + 2H$_2$O = (BiO)$_2$CrO$_4$ + 2KNO$_3$ + 4HNO$_3$
6. 4NaOH + SnCl$_2$ = Na$_3$SnO$_2$ + 2NaCl + 2H$_2$O
   2BiCl$_3$ + 3Na$_2$SnO$_2$ + 6NaOH = 2Bi + 6NaCl + 3Na$_2$SnO$_3$ + 3H$_2$O

COPPER (CUPRUM), Cu$^{+}$, $^{++}$ = 63.57

Copper is a red metal which is largely used in the metallic state, alone and in many alloys, of which brass, composed of copper and
zinc, is the most important. Many of the compounds of copper are green or blue in color and they impart these colors to their solutions. The cuprous compounds are unstable and unimportant. Copper sulphate, copper chloride and copper nitrate are readily soluble in water. The other important compounds of copper are insoluble or only slightly soluble in water.

**IMPORTANT COMPOUNDS OF COPPER.**

Basic copper acetate, “verdigris,” \( \text{Cu}_2\text{O} (\text{C}_2\text{H}_3\text{O}_2)_2 \).
Copper aceto-arsenite, “Paris green,” \( \text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2 + 3\text{Cu}(\text{AsO}_2)_2 \).
Copper arsenite, “Scheele’s green,” \( \text{CuHAsO}_3 \).
Copper chloride, cupric chloride, \( \text{CuCl}_2\text{H}_2\text{O} \).
Copper nitrate, cupric nitrate, \( \text{Cu(NO}_3)_2\text{H}_2\text{O} \).
Cuprous oxide, \( \text{Cu}_2\text{O} \).
Cupric oxide, \( \text{CuO} \).
Copper sulphate, cupric sulphate, *Cupri sulphas, U. S. P.*, “blue vitriol,” \( \text{CuSO}_4\text{H}_2\text{O} \).

**TESTS FOR COPPER.**

A separate portion of a solution of a copper salt, as copper sulphate, \( \text{CuSO}_4 \), should be used for each of the following tests:

1. Pass \( \text{H}_2\text{S} \) through the copper solution. A black precipitate of copper sulphide, \( \text{CuS} \), is formed. This precipitate is soluble in nitric acid and in a solution of potassium cyanide.
2. Add \( \text{NaOH} \) T.S. to the copper solution. A light blue precipitate of copper hydroxide, \( \text{Cu(OH)}_2 \), is produced, which is insoluble in an excess of the reagent. Boil the liquid containing the precipitate. The copper hydroxide is decomposed into black cupric oxide, \( \text{CuO} \), and water.

If the liquid contains citrates or tartrates or certain other organic substances, the copper hydroxide will dissolve in an excess of the alkali, forming a deep blue solution from which red cuprous oxide, \( \text{CuO} \), will precipitate on boiling if reducing agents are present. This behavior is the basis of Fehling’s test for sugar and other similar tests.

3. Slowly add \( \text{NH}_4\text{OH} \) T.S. to the solution of a copper salt. A light blue precipitate of copper hydroxide, \( \text{Cu(OH)}_2 \), is produced and immediately dissolves in an excess of the reagent, forming a deep blue solution containing ammonio-copper sulphate, \( \text{Cu(NH}_3)_2\text{SO}_4 \), or a similar compound. Slowly add KCN T.S. to the blue liquid. Potassium cuprocyanide, \( \text{K}_3\text{Cu(CN)}_4 \), is formed and the liquid becomes colorless. \( \text{H}_2\text{S} \) does not precipitate the copper from this solution, as copper sulphides are soluble in a solution of KCN.
4. Add \( \text{K}_4\text{Fe(CN)}_6 \) T.S. to the solution of a copper salt. A red-brown precipitate of copper ferrocyanide, \( \text{Cu}_2\text{Fe(CN)}_6 \), is produced, which is insoluble in diluted acids.
5. Dip a piece of platinum wire in the copper solution and heat in the blue flame of a Bunsen burner. The flame will be colored green or greenish-blue.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. \( \text{CuSO}_4 + \text{H}_2\text{S} = \text{Cu(NO}_3)_2 + \text{H}_2\text{S} = \)
2. \( \text{CuSO}_4 + \text{NaOH} = \text{Cu(C}_2\text{H}_3\text{O}_2)_2 + \text{KOH} = \text{Cu(OH)}_2 + \text{heat} = \)
3. \( \text{CuSO}_4 + 4\text{NH}_4\text{OH} = \text{Cu(NH}_3)_4\text{SO}_4 + \text{H}_2\text{O} \)
   \[ 2\text{Cu}^{2+}(\text{NH}_3)_4\text{SO}_4 + 10\text{KCN} + 8\text{H}_2\text{O} \]
   \[ = 2\text{K}_3\text{Cu}^{2+}[(\text{CN})_4] + 2\text{K}_2\text{SO}_4 + 8\text{NH}_4\text{OH} + (\text{CN})_2 \]
4. \( \text{CuSO}_4 + \text{K}_4\text{Fe(CN)}_6 = \text{Cu(NO}_3)_2 + \text{K}_4\text{Fe(CN)}_6 = \)

**CADMIUM, Cd\textsuperscript{II} = 112.4.**

Cadmium is a soft, white, crystalline metal resembling zinc and tin. It is a constituent of some easily fusible alloys. Cadmium salts are generally white in color, but the sulphide is yellow and the oxide is brown. The sulphate, the nitrate and the halogen salts are soluble in water. Most of the other cadmium salts are insoluble in water.

**IMPORTANT COMPOUNDS OF CADMIUM.**

Cadmium bromide, CdBr\textsubscript{2}\text{H}_2\text{O}.
Cadmium chloride, CdCl\textsubscript{2}\text{H}_2\text{O}.
Cadmium iodide, CdI\textsubscript{2}.
Cadmium nitrate, Cd(NO\textsubscript{3})\textsubscript{2}\text{H}_2\text{O}.
Cadmium oxide, CdO.
Cadmium sulphate, (CdSO\textsubscript{4})\textsubscript{3}8\text{H}_2\text{O}.
Cadmium sulphide, CdS.

**TESTS FOR CADMIUM.**

Use a separate portion of a solution of a cadmium salt, as CdSO\textsubscript{4}, for each of the following tests:

1. Pass H\textsubscript{2}S through the cadmium solution. A yellow precipitate of cadmium sulphide, CdS, will form. This precipitate is soluble in hot diluted H\textsubscript{2}SO\textsubscript{4}, but is not dissolved by other diluted acids, nor by solutions of alkalis, sulphides or cyanides.
2. Add NaOH T.S. to the cadmium solution. A white precipitate of cadmium hydroxide, Cd(OH)\textsubscript{2}, will form, which is insoluble.
in an excess of the reagent. The same precipitate will form with NH₄OH T.S., but is soluble in an excess of this reagent.

3. Add Na₂CO₃ T.S. to the cadmium solution. A white precipitate of cadmium carbonate, CdCO₃, is formed, which is not soluble in an excess of the reagent, but which will dissolve in diluted acids.

4. Slowly add KCN T.S. to the cadmium solution. A white precipitate of cadmium cyanide, Cd(CN)₂, will be produced and this will form potassium cadmium cyanide, K₂Cd(CN)₄, and dissolve in an excess of the reagent. Pass H₂S through the solution. Yellow cadmium sulphide, CdS, will be precipitated.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. \[ \text{CdSO}_4 + \text{H}_2\text{S} = \]
   \[ \text{Cd(NO}_3\text{)}_2 + \text{H}_2\text{S} = \]

2. \[ \text{CdSO}_4 + \text{NaOH} = \]
   \[ \text{Cd(NO}_3\text{)}_2 + \text{NH}_4\text{OH} = \]

3. \[ \text{Cd(NO}_3\text{)}_2 + \text{Na}_2\text{CO}_3 = \]
   \[ \text{CdSO}_4 + (\text{NH}_4\text{)}_2\text{CO}_3 = \]

4. \[ \text{CdSO}_4 + \text{KCN} = \]
   \[ \text{K}_2\text{Cd(CN)}_4 + \text{H}_2\text{S} = \]

**SAMPLE NUMBER 2.**

**Analysis of a solution for group 2 of the metals.**

The solution may contain salts of mercuric mercury, lead, bismuth, copper or cadmium, or of several of these metals.

Add a few drops of HCl T.S. to about 10 mils of the sample, warm and pass H₂S through the acidulated liquid. If no precipitate is produced, the metals of group 2 are absent. If a precipitate is produced, continue to pass H₂S through the liquid until precipitation is complete.

Collect the precipitate on a filter and test the filtrate by passing H₂S through it for a few minutes. If any additional precipitate is formed, continue to pass H₂S through the liquid for some time and then filter through the same filter paper, testing the new filtrate in the same manner as before. The metals of this group are all in the precipitate on the filter paper and the filtrate may be discarded, unless it is to be tested for the metals of subsequent groups.

A. Wash the precipitate on the filter with water and allow the water to drain out, discarding the washings. Pour the same portion of about 5 mils of hot HNO₃ T.S. through the filter several times. Any white or light colored residue insoluble in HNO₃ T.S. may be disregarded. A dark colored residue, insoluble in HNO₃ T.S. should
be examined for mercury by B. The filtrate is to be tested for the other members of the group.

B. If a dark colored residue is left that will not dissolve in HNO₃ T.S., mercury is indicated. Verify by puncturing the filter paper and washing the residue into a test-tube with about 3 mils of nitrohydrochloric acid. Boil the liquid until it ceases to smell of chlorine, dilute with about 5 mils of water, filter and add SnCl₂ T.S. A white or gray precipitate shows the presence of mercury.

C. Pour the filtrate obtained in A into a beaker, add about 2 mils of concentrated H₂SO₄ and boil until white fumes are given off. Cool and dilute with about 5 mils of water. A white precipitate indicates lead. If a precipitate forms, filter and save the filtrate to be tested for the remaining members of the sub-group.

D. To verify the presence of lead, wash the precipitate on the filter paper with water, allow the water to drain out and pass about 5 mils of NH₄C₂H₅O₂ T.S. through the paper several times. Add K₂Cr₂O₇ T.S. to the filtrate. A yellow precipitate shows the presence of lead.

E. To the liquid or filtrate from C, add an excess of NH₄OH T.S., shown by a strong odor of ammonia which persists after shaking the liquid. A white precipitate indicates the presence of bismuth. If a precipitate forms, filter the liquid and preserve the filtrate to be tested for Cu and Cd.

F. To verify the presence of bismuth, wash the precipitate on the filter paper with water, allow it to drain and moisten it with a few drops of NaOH T.S., followed by a few drops of SnCl₂ T.S. A brown or black coloration shows the presence of bismuth.

G. If the liquid or filtrate from E is blue, copper is present. Verify the presence or absence of copper by adding an excess of HC₂H₅O₂ T.S. followed by K₄Fe(CN)₆ T.S. to a portion of the liquid. A red coloration or precipitate shows the presence of copper.

II. If the liquid or filtrate from E is colorless, pass H₂S through it. A yellow precipitate shows the presence of cadmium. If the liquid is blue, slowly add KCN T.S. to a portion, until the blue color disappears and then pass H₂S through the liquid. A yellow precipitate shows the presence of cadmium.

**ANALYTICAL TABLES.**

Condensed directions for carrying out analytical processes are frequently given in tabular form, and such tables are very convenient. They are not often complete enough, however, to be followed successfully by inexperienced workers, so they should be used
only by those who have become familiar with the processes by following detailed directions.

In using the tables in this book the following points should be kept in mind, constantly:

Where chemical formulas are given for reagents, the test solutions should be used, unless directed otherwise.

Care should be taken to use sufficient of each reagent to accomplish its purpose, without using a large excess, and to test all filtrates after removing a metal or group by precipitation.

Only positive results are given in the tables, as if all of the metals included are present. When a negative result is obtained on testing for any metal or group, it is absent and one should pass on to the next test, often omitting filtrations or other processes which would be necessary if positive results were obtained.

**SAMPLE NUMBER 3.**

**Analysis of a solution for groups 1 and 2 of the metals.**

Examine the solution by the following tables, referring when necessary to the detailed directions for each group given on preceding pages.

The solution should be neutral or acid to litmus. If alkaline, add HNO₃ to the portion to be examined until the reaction is acid.

Add HCl as long as a precipitate is produced and filter.

<table>
<thead>
<tr>
<th>Precipitate—Pb, Hgᵢ, Ag</th>
<th>Filtrate—Hgᵢ, Pb, Bi, Cu, Cd</th>
</tr>
</thead>
<tbody>
<tr>
<td>Examine for metals of group 1 by I.</td>
<td>Warm and pass H₂S as long as a ppt. is produced. Filter. Examine for metals of group 2 by II.</td>
</tr>
</tbody>
</table>

I.

Examination of any precipitate produced by HCl in a neutral or acid solution.

Wash the precipitate on the filter with cold water, discarding the washings. Pass a portion of hot water through the washed precipitate on the filter.

<table>
<thead>
<tr>
<th>Filtrate—PbCl₂</th>
<th>Residue—HgCl, AgCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>Add K₂Cr₂O₇ and cool. Yellow ppt., PbCrO₄, shows</td>
<td>Pass the same portion of NH₄OH through the residue on the filter several times</td>
</tr>
<tr>
<td><strong>Residue—NH₄₂Hg₂Cl</strong> black, shows</td>
<td><strong>Filtrate—(NH₄)₂(AgCl)₂</strong></td>
</tr>
<tr>
<td>lead</td>
<td>silver</td>
</tr>
<tr>
<td>mercurous mercury</td>
<td></td>
</tr>
</tbody>
</table>
II.

Examination of a precipitate produced by H$_2$S in an acid liquid, after group 1 of the metals has been removed from a solution.

Wash the residue on the filter with water and drain, discarding the washings. Pour a portion of hot HNO$_3$ through the filter several times.

<table>
<thead>
<tr>
<th><strong>Residue</strong>—HgS</th>
<th><strong>Filtrate</strong>—Pb(NO$_3$)$_2$, Bi(NO$_3$)$_3$, Cu(NO$_3$)$_3$, Cd(NO$_3$)$_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Black. Dissolve in nitrohydrochloric acid, boil to expel Cl, dilute with water, filter and add SnCl$_2$. A white or gray ppt. HgCl$_2$—Hg, shows</td>
<td>Add conc. H$_2$SO$_4$ and boil until white fumes are given off, dilute with H$_2$O, cool and filter.</td>
</tr>
<tr>
<td><strong>Precipitate</strong>—PbSO$_4$</td>
<td><strong>Filtrate</strong>—Bi$_2$(SO$_4$)$_3$, CuSO$_4$, CdSO$_4$</td>
</tr>
<tr>
<td><strong>Precipitate</strong>—Bi(OH)$_3$</td>
<td><strong>Filtrate</strong>—Cu(NH$_3$)$_2$.SO$_4$, Cd(NH$_3$)$_2$.SO$_4$</td>
</tr>
<tr>
<td>White. Add NaOH and SnCl$_2$ on filter. Brown or black coloration, Bi$_2$.O$_3$, shows</td>
<td>Divide into two portions.</td>
</tr>
<tr>
<td>To one portion add an excess of HC$_2$.H$_2$.O$_2$ and then K$_4$.Fe(CN)$_6$. A red ppt. or coloration, Cu$_4$.Fe(CN)$_6$, shows</td>
<td>Pass H$_2$S through the other portion, first decolorizing with KCN if Cu is present. A yellow ppt., CdS, shows</td>
</tr>
</tbody>
</table>

**GROUP 3.**

Metals precipitated as sulphides by hydrogen sulphide, H$_2$S, in the presence of hydrochloric acid, HCl, and whose sulphides are soluble in ammonium sulphide, (NH$_4$)$_2$.S$_x$.

Gold, Au; Platinum, Pt; Arsenic, As; Antimony, Sb; Tin, Sn.

**GOLD (AURUM) Au$^+_{iii}$ = 197.2.**

Gold is a soft yellow metal that is used most largely in the metallic state in coins, jewelry, etc. It is permanent in the air and does not dissolve in any single acid, but is dissolved by nitro-hydrochloric acid and other liquids containing free chlorine, bromine or iodine. The metal is also attacked by fused alkalies and fused potassium nitrate. Gold compounds are poisonous and unstable. The only important compounds of gold are the auric salts with the halogen elements, which are soluble in water and brown or yellow in color. The aurous compounds are unimportant.
IMPORTANT COMPOUNDS OF GOLD.

Gold chloride, \( \text{AuCl}_3 \).
Gold and sodium chloride, \( \text{Auri et Sodii Chloridum, U. S. P., AuCl}_3 + \text{NaCl} \).
Bromauric acid, N. F., \( \text{HAuBr}_4 + 5\text{H}_2\text{O} \).
Chlorauric acid, \( \text{HAuCl}_4 + 4\text{H}_2\text{O} \).

TESTS FOR GOLD.

Use a separate portion of a solution of gold chloride, \( \text{AuCl}_3 \), for each of the following tests:

1. Pass \( \text{H}_2\text{S} \) gas through the gold solution, obtaining a brownish-black precipitate of gold sulphide, \( \text{Au}_2\text{S}_3 \). Collect the precipitate on a filter and pass yellow ammonium sulphide solution through the filter. The precipitate will dissolve.
2. Add \( \text{NaOH} \) T.S. A brown precipitate of gold hydroxide, \( \text{Au(OH)}_3 \), soluble in an excess of the reagent will be formed.
3. Add 2 drops of nitric acid and then \( \text{FeSO}_4 \) T.S. A brown precipitate of metallic gold will be formed.
4. Add \( \text{SnCl}_2 \) T.S. and let stand. A purple precipitate of “Purple of Cassius” is slowly formed. This is a substance of indefinite composition, containing aurous oxide, \( \text{Au}_2\text{O} \), and oxides of tin.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. \( \text{AuCl}_3 + \text{H}_2\text{S} = \)
2. \( \text{AuCl}_3 + \text{NaOH} = \)
3. \( 2\text{AuCl}_3 + 6\text{FeSO}_4 = 2\text{Au} + 2\text{FeCl}_3 + 2\text{Fe}_2(\text{SO}_4)_3 \)

PLATINUM, \( \text{Pt}^{\text{II, IV}} = 195.2 \).

Platinum is a heavy white metal with a high melting-point. It is permanent in the air and is not attacked by any single acid. Platinum vessels are used in chemical work and care should be taken not to allow them to come in contact with free chlorine or bromine, nor to ignite alkalies in them, nor mixtures that contain free iodine, sulphur, phosphorus or metals, or which will liberate these substances. The reducing gas flame forms a platinum carbide, so care should be taken to heat platinum vessels only in the oxidizing flame. Platinum ware may be cleansed by fusing borax in it and it may be polished by rubbing with moist sea-sand.

Platinum salts are unstable and readily reduced, liberating the metal as a black porous mass called “spongy platinum” or a black powder called “platinum black.”

IMPORTANT COMPOUNDS OF PLATINUM.

Platinum chloride, \( \text{PtCl}_4 + 5\text{H}_2\text{O} \).
Chloroplatinic acid, \( \text{H}_2\text{PtCl}_6 + 6\text{H}_2\text{O} \).
TESTS FOR PLATINUM.

Use a separate portion of a solution of platinum chloride, PtCl₄, for each of the following tests:

1. Pass H₂S gas through the platinum solution. A black precipitate of platinic sulphide, PtS₂, is formed slowly in a cold liquid, quickly on heating. This precipitate is insoluble in acids, but is soluble in yellow ammonium sulphide T.S.

2. Add NH₄Cl T.S. and an equal volume of alcohol. A yellow precipitate of ammonium chloroplatinate, (NH₄)₂PtCl₆ is formed.

Potassium salts give a yellow precipitate of the corresponding potassium chloroplatinate, K₂PtCl₆.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. PtCl₄ + H₂S =
2. PtCl₄ + NH₄Cl =

ARSENIC, As³⁺,v = 74.96.

Arsenic is a brittle crystalline element which has the physical properties of a metal, but generally behaves as a non-metal. The compounds of arsenic are dangerous poisons and methods for the detection of small quantities of the element in the stomach contents and in various tissues of the body are of great importance in toxicological chemistry.

The compounds of arsenic are generally white, though several of them have various colors due to the other elements present. Most of the important compounds of arsenic are more or less soluble in water. Arsenous sulphide and Paris green are insoluble. Compounds of arsenic are volatilized at high temperatures, with or without decomposition.

IMPORTANT COMPOUNDS OF ARSENIC.

Arsenic acid, ortho-arsenic acid, (H₃AsO₄)₂ + H₂O.
Arsenous iodide, U. S. P., AsI₃.
Arsenous sulphide, "orpiment," As₂S₃.
Copper aceto-arsenite, "Paris green," Cu(C₂H₃O₂)₂ + 3Cu(AsO₂)₂.
Hydrogen arsenide, arseniuretted hydrogen, "arsine," AsH₃.
Potassium arsenite, K₂HAsO₃.
Sodium arsenite, U. S. P., Na₂HAsO₄ + 7H₂O.

TESTS FOR ARSENIC.

Use a separate portion of a solution of either arsenous chloride, AsCl₃; sodium arsenite, Na₂HAsO₃; or sodium arsenate, Na₂HAsO₄; for tests 1, 2 and 3.
1. Pass H₂S gas through the warmed arsenic solution. A yellow precipitate of arsenous sulphide, As₂S₃, is formed, quickly from strong solutions of arsenous compounds and slowly from weak solutions of arsenous compounds or from solutions of arsenic compounds.

Collect the precipitate on a filter paper and pass warm (NH₄)₂S T.S. through the paper. The precipitate dissolves.

Arsenous sulphide is also dissolved by (NH₄)₂CO₃ T.S.

2. Reinsch’s Test.—Mix 20 mils of HCl T.S. with 20 mils of water and heat to boiling in a small porcelain evaporating dish, supported on a piece of wire gauze on a tripod over the flame of a Bunsen burner. When the liquid boils introduce a piece of bright copper foil about 2 cm. square and continue boiling for five minutes, adding water from time to time to keep the volume of the liquid approximately constant. If there is no dark deposit formed on the copper, the reagent and apparatus are free from arsenic.

A dark deposit on the copper shows the presence of arsenic or other easily reduced metal in the reagents. Such a preliminary trial is called a blank test, and is made when there is a possibility of misleading results being obtained from the reagents or other source.

If the copper remains bright during the preliminary test, add two or three drops of the arsenic solution and boil for ten minutes, adding water from time to time to keep the volume constant. A dark coating of copper arsenide, Cu₅As₂, will be deposited on the copper, which may scale off if much arsenic is present. Pour off the liquid, rinse the copper, first with water and then with alcohol, and then dry it by pressing between the folds of a filter paper. Cut the copper into small pieces, introduce some of them into a dry test-tube, and heat the lower end of the tube by holding it in the flame of a Bunsen burner. The arsenic will oxidize and volatilize, condensing on the cool part of the tube as a white deposit of arsenic trioxide, As₂O₃, whose characteristic octahedral crystals may be recognized under a magnifying glass.

Reinsch’s test will detect very small amounts of arsenic, and it can be used in the presence of organic matter.

3. Marsh’s Test.—Prepare a small hydrogen generator with an outlet tube drawn to a jet. Introduce a few pieces of pure zinc and some diluted hydrochloric acid into the generator and allow the generated hydrogen to escape for some time, to displace all of the air in the generator. To test this hold an inverted test-tube down over the jet until the tube is filled with hydrogen. Remove it from the jet, keeping the test-tube inverted, and bring the mouth of the tube in contact with a small flame. If the hydrogen burns quietly at the mouth of the tube it is safe to ignite it at the jet, but not otherwise. To make it doubly sure wrap a towel loosely
around the flask, when the hydrogen burns quietly in the test-tube, and then bring a flame to the jet to ignite the hydrogen. The towel can then be removed. Hold a cold piece of porcelain in the flame for a moment. If the porcelain is not discolored the reagents and apparatus are free from arsenic. If a dark spot appears on the porcelain, the reagents or apparatus contain arsenic or antimony.

If the blank test shows the reagents and apparatus to be free of arsenic, allow the hydrogen to continue burning and introduce a few drops of an arsenic solution into the generator through the funnel tube. The arsenic compound is reduced by a small part of the nascent hydrogen and the gaseous compound arsenine, As₃, is formed which mixes with the rest of the hydrogen. In a short time the blue hydrogen flame will change to white, the arsenine burning to form arsenous oxide, As₂O₃, and water. If a piece of cold porcelain is held in the flame it lowers the temperature of the burning gas, and a lustrous dark spot of arsenic will be deposited on the porcelain. This spot will dissolve in a solution of chlorinated soda, NaClO₃.

Marsh’s test is exceedingly delicate, but if a mixture containing organic matter is to be tested for arsenic, the organic matter must be destroyed before this test can be applied.

4. Add magnesia mixture (see reagents) to a solution of Na₂HAsO₄. A white precipitate of magnesium ammonium arsenate, MgNH₄AsO₄, will form.

Add magnesia mixture to a solution of Na₂HAsO₃. No precipitate will be produced.

This test will help to distinguish between arsenous and arsenic compounds.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. \[ \text{AsCl}_3 + \text{H}_2\text{S} = \]
   \[ \text{K}_2\text{HAsO}_3 + \text{H}_2\text{S} + \text{HCl} = \]
   \[ 2\text{Na}_2\text{HAsO}_4 + 5\text{H}_2\text{S} + 4\text{HCl} = \text{As}_2\text{S}_3 + 4\text{NaCl} + \text{S}_2 + 8\text{H}_2\text{O} \]

2. \[ 4\text{AsCl}_3 + 10\text{Cu} + 6\text{H}_2\text{O} = 2\text{Cu}_3\text{As}_2 + 12\text{HCl} + 3\text{O}_2 \]
   \[ 2\text{Cu}_3\text{As}_2 + 3\text{O}_2 = 10\text{Cu} + 2\text{As}_2\text{O}_3 \]

3. \[ \text{AsCl}_3 + \text{Zn} + \text{HCl} = \]
   \[ \text{K}_2\text{HAsO}_3 + \text{Zn} + \text{HCl} = \]
   \[ \text{Na}_2\text{HAsO}_4 + 4\text{Zn} + 10\text{HCl} = 2\text{NaCl} + 4\text{ZnCl}_2 + 4\text{H}_2\text{O} + \text{AsH}_3 \]
   \[ \text{As}_4 + 10\text{NaClO}_3 + 6\text{H}_2\text{O} = 4\text{H}_3\text{AsO}_3 + 10\text{NaCl} \]

4. \[ \text{Na}_2\text{HAsO}_4 + \text{NH}_4\text{OH} + \text{MgSO}_4 = \]

**ANTIMONY (STIBIUM), Sb^{III, V} = 120.2.**

Antimony is a crystalline brittle metal whose compounds are poisonous. Type-metal is an alloy of antimony and lead. Soluble salts of antimony are decomposed by water in solutions that con-
tain moderate amounts of mineral acids and basic compounds containing the monad radical antimonyl, \( \text{(SbO)}' \), or more complex oxides, are precipitated. Organic acids or large amounts of mineral acids prevent this precipitation.

Antimony salts are unstable and only a few of them are important. The oxides are feebly basic with acids and feebly acid with bases. Antimonous chloride and the other halogen salts are deliquescent and soluble in small amounts of water with some decomposition. Antimonyl-potassium tartrate is moderately soluble in water, without decomposition.

**IMPORTANT COMPOUNDS OF ANTIMONY.**

Antimonous chloride, "butter of antimony," \( \text{SbCl}_3 \).
Antimonous oxide, \( \text{Sb}_2\text{O}_3 \).
Antimonous sulphide, \( \text{Sb}_2\text{S}_3 \).
Antimonyl-potassium tartrate, *Antimony and potassium tartrate, U. S. P.*, "tartar emetic," \( \text{(KSbO}_4\text{H}_4\text{O}_6)\_2\text{H}_2\text{O} \).
Hydrogen antimonide, "stibine," \( \text{SbH}_3 \).

**TESTS FOR ANTIMONY.**

Use separate portions of a solution of antimonous chloride, \( \text{SbCl}_3 \), for each of the following tests:

1. Pour 2 mils of the antimony chloride solution into a test-tube half filled with water. A white precipitate consisting mainly of antimony oxychloride, \( \text{SbOCl} \), is formed if the solution does not contain a large excess of free acid.
2. Pass \( \text{H}_2\text{S} \) gas through the antimony solution. An orange red precipitate of antimony sulphide, \( \text{Sb}_2\text{S}_3 \), is formed. Collect a small amount of the precipitate on a filter and pass warm yellow ammonium sulphide solution through the filter. The precipitate dissolves.
3. Apply Reinsch's test, including the blank test, as described under arsenic on page 4, but using a few drops of the antimony solution instead of the arsenic solution. A dark gray deposit of metallic antimony will form on the copper. If the copper with the deposit is dried and cut into small pieces and heated in a dry test-tube the antimony will oxidize and volatilize, condensing on the cool part of the tube as a white deposit of antimonous oxide, \( \text{Sb}_2\text{O}_3 \), which is amorphous.
4. Apply Marsh's test, including the blank test, as described under arsenic on pages 40-41, but using a few drops of the antimony solution, instead of the arsenic solution. The antimony compound is reduced by a small part of the nascent hydrogen and gaseous stibine, \( \text{SbH}_3 \), is formed, which burns with the excess of hydrogen.
TIN (STANNUM), \( \text{Sn}^{\text{II,IV}} = 119.0 \).

Tin is a soft, white, crystalline metal that is largely used in the metallic state as foil and pipe, and in various alloys like bronze, solder and pewter. Tin plate, commonly called “tin,” is sheet iron coated with tin.

IMPORTANT COMPOUNDS OF TIN.

Stannous chloride, \( \text{SnCl}_2\cdot2\text{H}_2\text{O} \).
Stannic chloride, \( \text{SnCl}_3\cdot5\text{H}_2\text{O} \).
Stannous sulphide, \( \text{SnS} \).
Stannic sulphide, “Mosaic gold,” \( \text{SnS}_2 \).
Sodium stannate, \( \text{Na}_2\text{SnO}_3\cdot4\text{H}_2\text{O} \).

TESTS FOR TIN.

Use a separate portion of a solution of stannous chloride, \( \text{SnCl}_2 \), for each of the following tests:

1. Pour 2 mils of the stannous chloride solution into about 10 mils of water. A precipitate of stannous oxychloride, \( \text{Sn}_2\text{OCl}_2 \), will form, if the solution is not too strongly acid.

Oxygen from the air frequently precipitates stannous oxychloride
with formation of some stannic chloride in bottles of SnCl₂ T.S. This precipitation is retarded by acidulating the solution with HCl, and keeping some pieces of metallic tin in it.

2. Pass H₂S through the SnCl₂ solution. A brown precipitate of stannous sulphide, SnS, is formed. Collect a small amount of the precipitate on a filter and pass yellow ammonium sulphostannate, (NH₄)₂SnS₃. Add an excess of diluted HCl to the filtrate. A yellow precipitate of stannic sulphide, SnS₂, will form.

3. Add NaOH T.S. in excess. A white precipitate of stannous hydroxide, Sn(OH)₂, appears and then dissolves in the excess of the reagent, forming sodium stannite, Na₂SnO₂. Boil the solution gently. A white precipitate of stannous oxide, SnO, forms.

4. Add a few drops of diluted HCl and a few drops of HgCl₂ T.S. to the solution of SnCl₂. A white precipitate of mercurous chloride, HgCl, will form, or, if the SnCl₂ is in large excess, the precipitate will be gray and consist of metallic mercury.

5. Pour some SnCl₂ solution into a test-tube containing zinc and diluted HCl. Metallic tin is liberated as a gray, spongy precipitate which will form SnCl₂ and dissolve in HCl if the pieces of zinc are removed.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. SnCl₂ + H₂O =
2. SnCl₂ + H₂S =
   SnS + (NH₄)₂S₂ =
   (NH₄)₂SnS₃ + HCl = SnS₂ +
3. SnCl₂ + NaOH =
   Sn(OH)₂ + NaOH =
   Na₂SnO₂ + H₂O =
4. SnCl₂ + HgCl₂ = HgCl +
   SnCl₂ + HgCl₂ = Hg +
5. SnCl₂ + Zn =
   Sn + HCl =

NOTE ON STANNIC COMPOUNDS.

Stannic compounds in solution give a yellow precipitate of stannic sulphide, SnS₂, with H₂S, and a white precipitate of stannous hydroxide, Sn(OH)₄, with NaOH T.S., soluble in an excess of the reagent. Lead and hydrochloric acid reduce stannic compounds, SnCl₂ being formed, which may be recognized by applying tests for stannous compounds to the solution.

SAMPLE NUMBER 4.

Analysis of a solution for group 3 of the metals.

The solution may contain salts of arsenic, antimony, tin, gold or platinum, or of several of these metals.
A. Acidulate 10 mils of the sample with HCl T.S., warm and pass H₂S gas through it.

If no precipitate is produced on passing H₂S through the acid liquid for ten minutes or more, appreciable amounts of the metals of group 3 are absent.

If a precipitate forms, continue to pass H₂S through the liquid until precipitation appears to be complete.

Collect the precipitate on a filter and test the filtrate by passing H₂S through it for a few minutes. If no more precipitate is formed, group 3 is all precipitated, and the filtrate may be discarded, unless it is to be kept for tests for succeeding groups. If H₂S produces any additional precipitate, continue to pass the gas through the filtrate for some time and then filter through the same filter paper, testing the new filtrate in the same manner as before.

B. Allow the water to drain from the precipitate on the filter paper. Puncture the apex of the filter and wash the precipitate into a test-tube with 10 mils of (NH₄)₂CO₃ T.S., which will dissolve As₂S₃, but not the sulphides of the other metals of this group. Warm the liquid by standing the test-tube in hot water for a few minutes, filter and wash any precipitate on the filter with 10 mils of water, discarding the washings.

C. Acidulate the filtrate from B with HCl T.S. and pass H₂S through it. A yellow precipitate shows the presence of arsenic. A white precipitate consists of sulphur and may be disregarded. If arsenic is found, verify it by applying Reinsch’s test to the liquid containing the precipitate.

D. Allow any precipitate from B to drain on the filter paper. Puncture the apex of the paper and wash the residue into a test-tube with 10 mils of concentrated HCl. Heat the test-tube by standing it in hot water for ten minutes and filter. The filtrate will contain the antimony and tin, if present, and the insoluble residue will contain the gold and platinum, if present.

E. Introduce the filtrate from D into a small generating flask fitted with a funnel tube and a delivery tube. Add an equal volume of water and a small amount of metallic zinc. Conduct the gas formed through a half test-tube full of AgNO₃ T.S. for three minutes. If no precipitate forms in the AgNO₃ T.S., antimony is absent. A black precipitate indicates the presence of antimony.

F. If a black precipitate is formed in the AgNO₃ T.S. by paragraph E, collect the precipitate on a filter and wash with water, rejecting the filtrate and washings. Then pass HCl T.S. through the filter, passing the filtrate through the filter again if necessary to get a clear liquid. Pass H₂S through the filtrate. An orange-colored precipitate shows the presence of antimony.

G. Pass a small amount of the liquid in the generator, described
in paragraph E, through a filter into about 1 mil of HgCl₂ T.S. contained in a test-tube. A white or gray precipitate shows the presence of tin.

II. If the residue, insoluble in hydrochloric acid is light colored the sample does not contain gold or platinum and the residue may be discarded. If this residue is dark colored it should be tested for gold and platinum. Puncture the apex of the filter paper and wash the residue into a test-tube with 5 mils of nitrohydrochloric acid. Warm the liquid by standing the test-tube in hot water, add an equal volume of water, filter out any insoluble residue and divide the clear filtrate into two parts.

I. To one part of the filtrate add SnCl₂ and SnCl₄ test solutions and let stand. A purple coloration or precipitate indicates gold. Verify by adding SnCl₂ and SnCl₄ test solutions to the original solution.

J. Neutralize the other part of the filtrate from H with NH₄OH, add an equal volume of alcohol and then NH₄Cl T.S. A yellow or brown precipitate indicates platinum. Verify by adding alcohol and NH₄Cl T.S. to the original solution.

**SAMPLE NUMBER 5.**

**Analysis of a solution for groups 1, 2 and 3 of the metals.**

<table>
<thead>
<tr>
<th>Precipitate—Pb, Hg, Ag</th>
<th>Filtrate—</th>
</tr>
</thead>
<tbody>
<tr>
<td>Examine for metals of group 1 by I.</td>
<td>Warm and pass H₂S as long as a precipitate is produced. Collect the ppt. on a filter, wash and drain. Digest the ppt. with (NH₄)₂S₈ and filter.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Residue—Hg, Pb, Bi, Cu, Cd</th>
<th>Filtrate—</th>
</tr>
</thead>
<tbody>
<tr>
<td>Examine for metals of group 2 by II.</td>
<td>Examine for metals of group 3 by III.</td>
</tr>
</tbody>
</table>

I. Examination of any precipitate produced by HCl in a neutral or acid solution.

Wash the precipitate on the filter with cold water, discarding the washings. Pass a portion of hot water through the washed precipitate on the filter.

<table>
<thead>
<tr>
<th>Filtrate—PbCl₂</th>
<th>Residue—HgCl₂, AgCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>Add K₂CrO₄. Yellow ppt., PbCrO₄ shows</td>
<td>Pass the same portion of NH₃OH through the residue on the filter several times</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Residue—NH₃HgCl + Hg, black, shows</th>
<th>Filtrate—(NH₄)₂(AgCl)₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Add an excess of HNO₃, white ppt., AgNO₃, shows</td>
<td>lead</td>
</tr>
</tbody>
</table>
II.

Examination of any residue insoluble in \((\text{NH}_4)_2\text{S}_x\) from a precipitate produced by \(\text{H}_2\text{S}\) in an acid liquid, after group 1 of the metals has been removed from a solution.

<table>
<thead>
<tr>
<th>Residue—(\text{HgS})</th>
<th>Filtrate—(\text{Pb(NO}_3)_2, (\text{Bi(NO}_3)_3, (\text{Cu(NO}_3)_2, (\text{Cd(NO}_3)_2)</th>
<th>Add conc. (\text{H}_2\text{SO}_4) and boil until white fumes are given off, cool, dilute with (\text{H}_2\text{O}) and filter.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Black. Dissolve in nitrohydrochloric acid, boil to expel Cl, dilute with water, filter and add (\text{SnCl}_2). A white or gray ppt. (\text{HgCl}_2 + \text{Hg}) shows</td>
<td>Precipitate—(\text{PbSO}_4)</td>
<td>White Dissolve with (\text{NH}_4\text{H}_2\text{O}_2) and add (\text{K}_2\text{Cr}_2\text{O}_7). Yellow ppt., (\text{PbCrO}_4), shows</td>
</tr>
<tr>
<td>Filtrate—(\text{Bi}_2(\text{SO}_4)_3, \text{CuSO}_4, \text{CdSO}_4)</td>
<td>Add an excess of (\text{NH}_3\text{OH}) and filter.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Filtrate—(\text{Cu(NH}_3)_2\text{SO}_4, \text{Cd(NH}_3)_2\text{SO}_4)</td>
<td>Divide into two portions.</td>
</tr>
<tr>
<td></td>
<td>To one portion add an excess of (\text{HC}_2\text{H}_2\text{O}_2) and then (\text{K}_2\text{Fe(CN)}_4). A red ppt. or coloration, (\text{Cu}_2\text{Fe(CN)}_4), shows</td>
<td>Pass (\text{H}_2\text{S}) through the other portion, decolorizing with (\text{KCN}), if (\text{Cu}) is present. A yellow ppt., (\text{CdS}), shows</td>
</tr>
</tbody>
</table>

Examination of the solution in \((\text{NH}_4)_2\text{S}_x\) of any precipitate produced by \(\text{H}_2\text{S}\) in an acid liquid, after group 1 of the metals has been removed from a solution.

Acidulate the \((\text{NH}_4)_2\text{S}_x\) solution with \(\text{HCl}\) and collect any precipitate formed on a filter. Transfer the precipitate to a test tube and digest in \((\text{NH}_4)_2\text{CO}_3\) T.S. Filter.

<table>
<thead>
<tr>
<th>Filtrate—((\text{NH}_4)_2\text{As}_3)</th>
<th>Residue—(\text{Sb}_2\text{S}_3, \text{SnS}_2, \text{Au}_2\text{S}_3, \text{PtS}_2)</th>
<th>Wash with water. Digest in concentrated (\text{HCl}). Filter.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Add excess of (\text{HCl}) and pass (\text{H}_2\text{S}). A yellow ppt., (\text{AsS}_2), shows</td>
<td>Filtrate—(\text{SbCl}_3, \text{SnCl}_2)</td>
<td>Place in (\text{H}) generator. Add (\text{H}_2\text{O}) and (\text{Zn}). Conduct gas into (\text{AgNO}_3) for three minutes</td>
</tr>
<tr>
<td></td>
<td>Residue—(\text{Au}_2\text{S}_3, \text{PtS}_2)</td>
<td>If light colored (\text{Au}) and (\text{Pt}) are absent. If dark colored, digest in nitrohydrochloric acid. Add (\text{H}_2\text{O}) and filter.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>To a part of the filtrate add (\text{SnCl}_2) and (\text{SnCl}_4) and let stand. A purple ppt., (\text{AuCl}_2), etc., shows</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>arsenic</th>
<th>antimony</th>
<th>tin</th>
<th>gold</th>
<th>platinum</th>
</tr>
</thead>
<tbody>
<tr>
<td>mercuric mercury</td>
<td>lead</td>
<td>bismuth</td>
<td>copper</td>
<td>cadmium</td>
</tr>
</tbody>
</table>
METALS

GROUP 4.

Metals precipitated as hydroxides by ammonium hydroxide, \( \text{NH}_4\text{OH} \), in presence of ammonium chloride, \( \text{NH}_4\text{Cl} \):
- Iron, Fe;
- Chromium, Cr;
- Aluminum, Al.

IRON (FERRUM) \( \text{Fe}^{ii,iii} = 55.84 \)

Iron is largely used in the metallic state, nearly pure as wrought iron, less pure as steel, and still less pure as cast iron and malleable iron. Reduced iron is powdered metallic iron obtained by the action of hydrogen on heated ferric oxide.

In the ferrous compounds, iron has a valence of two, and in the ferric compounds it has a valence of three. Ferrous compounds are readily oxidized to ferric compounds and ferric compounds are nearly as readily reduced to ferrous compounds. Both series of compounds are important. Soluble ferrous compounds are generally light green in color and slightly color their solutions. Soluble ferric compounds are generally brown or yellow in color and strongly color their solutions.

IMPORTANT FERROUS COMPOUNDS.

- Ferrous bromide, \( \text{FeBr}_2\text{6H}_2\text{O} \).
- Ferrous carbonate, \( \text{FeCO}_3 \).
- Ferrous chloride, \( \text{FeCl}_2 \).
- Ferrous hydroxide, \( \text{Fe(OH)}_2 \).
- Ferrous iodide, \( \text{FeI}_2 \).
- Ferrous oxide, \( \text{FeO} \).
- Ferrous sulphate, U. S. P., "copperas," "green vitriol," \( \text{FeSO}_4\text{7H}_2\text{O} \).
- Exsiccated ferrous sulphate, U. S. P., \( (\text{FeSO}_4)_{2}\text{3H}_2\text{O} \).
- Ferrous sulphide, \( \text{FeS} \).

IMPORTANT FERRIC COMPOUNDS.

- Ferric ammonium sulphate, "ferric alum," \( \text{NH}_4\text{Fe(SO}_4)_2\text{12H}_2\text{O} \).
- Ferric chloride, U. S. P., \( \text{FeCl}_3\text{6H}_2\text{O} \).
- Ferric ferrocyanide, "Prussian blue," \( \text{Fe}_4(\text{Fe(CN)}_6)_3 \).
- Ferric hydroxide, "arsenic antidote," \( \text{Fe(OH)}_3 \).
- Ferric hypophosphite, N. F., \( \text{Fe(PH}_2\text{O}_2)_3 \).
- Ferric nitrate, \( \text{Fe(NO}_3)_3 \).
- Ferric oxide, "hematite," \( \text{Fe}_2\text{O}_3 \).
- Ferroso-ferric oxide, "magnetite," \( \text{Fe}_3\text{O}_4 \).
- Ferric pyrophosphate, N. F., composition indefinite.
- Ferric sulphate, \( \text{Fe}_2(\text{SO}_4)_3 \).
- Ferric subsulphate, "Monsel's salt," \( \text{Fe}_4\text{O(SO}_4)_5 \).
TESTS FOR FERROUS IRON.

Use a separate portion of a solution of a ferrous salt, as FeSO₄, for each of the following tests:

1. Add NH₄OH T.S. to the ferrous solution. A white precipitate of ferrous hydroxide, Fe(OH)₂, is produced if the ferrous salt is pure, but the precipitate immediately begins to oxidize and darken in color, forming first the black ferroso-ferric hydroxide, Fe₃(OH)₈, which on standing changes by further oxidation to the light brown ferric hydroxide, Fe(OH)₃. Ferrous hydroxide is slightly soluble in solutions of ammonium salts, so the precipitation of iron by this reagent is not complete. NaOH T.S. will produce the same results.

2. Add (NH₄)₂CO₃ T.S. to the ferrous solution. A white precipitate of ferrous carbonate, FeCO₃, is produced and immediately begins to decompose and oxidize and darken in color, forming first the black ferroso-ferric hydroxide, Fe₃(OH)₈, and then, by further oxidation, the light brown ferric hydroxide, Fe(OH)₃. Na₂CO₃ T.S. will produce the same results.

3. Add (NH₄)₂S T.S. to the ferrous solution. A black precipitate of ferrous sulphide, FeS, will be produced. The precipitate is readily soluble in acids.

4. Add K₄Fe(CN)₆ T.S. to the ferrous solution. A light blue precipitate of potassium-ferrous ferrocyanide, K₂FeFe(CN)₆, will be produced which will readily oxidize to form ferric ferrocyanide, Fe₄(Fe(CN)₆)₃, dark blue. The precipitate is insoluble in acids. If any ferric salt is present in the ferrous solution the precipitate will be dark blue.

5. Add K₃Fe(CN)₆ T.S. to the ferrous solution. A dark blue precipitate of ferrous ferricyanide, Fe₃(Fe(CN)₆)₂, will be formed. The precipitate is insoluble in acids.

6. Add KCNS T.S. to the ferrous solution. No coloration or precipitation will be produced, unless some ferric salt is present, which would give a bright red color with the reagent.

Complete and balance the following equations:

1. \[\text{FeSO}_4 + \text{NH}_4\text{OH} = \text{FeCl}_2 + \text{NaOH}\]
2. \[\text{FeCl}_2 + (\text{NH}_4)_2\text{CO}_3 = \text{FeSO}_4 + \text{Na}_2\text{CO}_3\]
3. \[\text{FeSO}_4 + (\text{NH}_4)_2\text{S} = \text{FeCl}_2 + \text{K}_2\text{S}\]
4. \[\text{FeSO}_4 + K_4\text{Fe(CN)}_6 = \text{FeBr}_2 + K_4\text{Fe(CN)}_6\]
5. \[\text{FeCl}_2 + K_3\text{Fe(CN)}_6 = \text{FeSO}_4 + K_3\text{Fe(CN)}_6\]
TESTS FOR FERRIC IRON.

Use a separate portion of a solution of a ferric salt, as FeCl₃, for each of the following tests:

1. Add NH₄OH T.S. to the ferric solution. A red-brown gelatinous precipitate of ferric hydroxide, Fe(OH)₃, will be formed, which is insoluble in an excess of the reagent or in solutions of ammonium salts, but which will dissolve in acids. Precipitation is not complete if glycerine, sugar, or some organic acids are present. On boiling the liquid containing the precipitate, it changes to a basic ferric hydroxide, FeO(OH), which is not gelatinous.

NaOH T.S. produces the same precipitate.

2. Add Na₂CO₃ T.S. to the ferric solution. Carbon dioxide CO₂, is given off, and ferric hydroxide, Fe(OH)₃, is precipitated. The same result is obtained with (NH₄)₂CO₃ T.S.

3. Add (NH₄)₂S T.S. to the ferric solution. The iron is reduced to the ferrous condition and a black precipitate is obtained, which consists of ferrous sulphide, FeS, and sulphur.

H₂S reduces ferric salts in solution to the ferrous condition, with precipitation of sulphur.

4. Add K₄Fe(CN)₆ T.S. to the ferric solution. A deep blue precipitate of ferric ferrocyanide, Fe₄(Fe(CN)₆)₃, is formed. The precipitate is insoluble in diluted acids, but is decomposed by alkalis.

5. Add K₃Fe(CN)₆ T.S. to the ferric solution. Ferric ferricyanide, FeFe(CN)₆, is produced and changes the color of the solution to a greenish-brown.

6. Add KCNS T.S. to the ferric solution. A blood red coloration is produced, due to the formation of ferric sulphocyanate, Fe(CNS)₃. This reaction is most dependable in the presence of moderate amounts of free HCl. Many different compounds prevent the reaction in neutral solutions.

Complete and balance the following equations:

1. \[ \text{FeCl}_3 + \text{NH}_4\text{OH} = \text{Fe}_2(\text{SO}_4)_3 + \text{NaOH} = \text{Fe(OH)}_3 + \text{heat} = \]

2. \[ \text{Fe(NO}_3)_3 + \text{Na}_2\text{CO}_3 + \text{H}_2\text{O} = \text{FeCl}_3 + (\text{NH}_4)_2\text{CO}_3 + \text{H}_2\text{O} = \]

3. \[ \text{Fe}_2(\text{SO}_4)_3 + 3(\text{NH}_4)_2\text{S} = 2\text{FeS} + \text{S} + 3(\text{NH}_4)_2\text{SO}_4 \]

4. \[ \text{FeCl}_3 + \text{K}_4\text{Fe(CN)}_6 = \text{Fe}_2(\text{SO}_4)_3 + \text{K}_4\text{Fe(CN)}_6 = \]

5. \[ \text{Fe(NO}_3)_3 + \text{K}_4\text{Fe(CN)}_6 = \]

6. \[ \text{FeCl}_3 + \text{KCNS} = \text{Fe}_2(\text{SO}_4)_3 + \text{KCNS} = \]
CHROMIUM, Cr\(^{\text{ii, iii, vi}}\) = 52.0

Chromium is a light gray crystalline metal which is seldom seen in the free state. It is contained in small amounts in some varieties of steel, to which it imparts additional hardness.

Compounds of chromium are brightly colored. When it has a valence of two, chromium forms the unimportant chromous compounds; when it has a valence of three it forms the chromic compounds; and when it has the valence of six it is an acid-forming element, in chromic acid and its anhydride and the chromates.

IMPORTANT COMPOUNDS OF CHROMIUM.

Chrome oxide, Cr\(_2\)O\(_3\).
Chromium trioxide, U. S. P., chromic anhydride, "chromic acid," CrO\(_3\).
Chromium-potassium sulphate, "chrome alum," KCr(SO\(_4\))\(_2\)12H\(_2\)O.
Chromium sulphate, Cr\(_2\)(SO\(_4\))\(_3\)H\(_2\)O.
Lead chromate, "chrome yellow," PbCrO\(_4\).
Potassium chromate, K\(_2\)CrO\(_4\).
Potassium dichromate, K\(_2\)Cr\(_2\)O\(_7\).

TESTS FOR CHROMIC SALTS.

Use a separate portion of a solution of a chromic salt, as Cr\(_2\)(SO\(_4\))\(_3\), for each of tests, 1, 2, 3 and 4.

1. Add NH\(_4\)OH T.S. to the chromium solution. A greenish-blue gelatinous precipitate of chromic hydroxide, Cr(OH)\(_3\), will form.

2. Slowly add NaOH T.S. to the chromium solution. The greenish-blue chromic hydroxide, Cr(OH)\(_3\), will form and re-dissolve in an excess of the reagent as sodium chromite, NaCrO\(_2\). Chromium hydroxide is re-precipitated upon boiling.

3. Add Na\(_2\)CO\(_3\) T.S. to the chromium solution. The greenish-blue chromic hydroxide, Cr(OH)\(_3\), will be precipitated and carbon dioxide will be given off.

4. Add (NH\(_4\))\(_2\)S T.S. to the chromium solution. The greenish-blue chromium hydroxide, Cr(OH)\(_3\), will be precipitated and H\(_2\)S will be given off.

5. Fusion Test.—Collect the precipitate from any of the preceding tests for chromium on a filter and allow to drain. Transfer a portion to the cover of a porcelain crucible, add about an equal amount of dry KNO\(_3\) and twice as much dry Na\(_2\)CO\(_3\). Support the cover on a pipe-stem triangle and heat in the Bunsen burner flame until the mass is fused. The KNO\(_3\) oxidizes the chromium to chromic acid, which forms the yellow potassium and sodium chromates, K\(_2\)CrO\(_4\) and Na\(_2\)CrO\(_4\). Allow the mass to cool and dissolve from the
cover by boiling with a small quantity of water in a beaker. Filter the solution formed, acidulate the filtrate with HC$_2$H$_5$O$_2$ and add Pb(C$_2$H$_5$O$_2$)$_2$ T.S. A yellow precipitate of lead chromate, PbCrO$_4$ will be formed.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. \[ \text{Cr}_2(\text{SO}_4)_3 + \text{NH}_4\text{OH} = \]
   \[ \text{CrCl}_3 + \text{NH}_4\text{OH} = \]
2. \[ \text{Cr}_2(\text{SO}_4)_3 + \text{NaOH} = \]
   \[ \text{Cr(OH)}_3 + \text{NaOH} = \]
   \[ \text{NaCrO}_2 + \text{H}_2\text{O} = \]
3. \[ \text{CrCl}_3 + \text{Na}_2\text{CO}_3 + \text{H}_2\text{O} = \]
   \[ \text{Cr}_2(\text{SO}_4)_3 + (\text{NH}_4)_2\text{CO}_3 + \text{H}_2\text{O} = \]
4. \[ \text{Cr}_2(\text{SO}_4)_3 + (\text{NH}_4)_2\text{S} + \text{H}_2\text{O} = \]
   \[ \text{CrCl}_3 + (\text{NH}_4)_2\text{S} + \text{H}_2\text{O} = \]
5. \[ 2\text{Cr(OH)}_3 + 2\text{KNO}_3 + \text{Na}_2\text{CO}_3 = \text{K}_2\text{CrO}_4 + \text{Na}_2\text{CrO}_4 + 3\text{H}_2\text{O} + \text{N}_2\text{O}_2 + \text{CO}_2 \]
   \[ \text{Cr(OH)}_3 + \text{KNO}_3 + \text{K}_2\text{CO}_3 = \]
   \[ \text{K}_2\text{CrO}_4 + \text{Pb(C}_2\text{H}_5\text{O}_2)_2 = \]

**ALUMINUM, Al$^{iii}$ = 27.1**

Aluminum is a white metal which is largely used in the metallic state. Its compounds are generally colorless.

Aluminum readily forms double sulphates, called alums, with monad metals or ammonium. Similar double sulphates of other triad and monad metals are also called alums.

**IMPORTANT COMPOUNDS OF ALUMINUM.**

Aluminum-ammonium sulphate, alum, U. S. P., ammonium alum, AlNH$_4$(SO$_4$)$_2$ 12H$_2$O.
Aluminum chloride, N. F., AlCl$_3$.
Aluminum oxide, native in corundum, emery, etc., Al$_2$O$_3$.
Aluminum-potassium sulphate, alum, U. S. P., potassium alum, AlK(SO$_4$)$_2$12H$_2$O.
Aluminum silicates, native in clays, etc., composition variable.
Aluminum-sodium sulphate, sodium alum, AlNa(SO$_4$)$_2$12H$_2$O.
Aluminum sulphate, N. F., Al$_2$(SO$_4$)$_3$16H$_2$O.
Exsiccate alum, U. S. P., “burnt alum,” “dried alum,” AlNH$_4$(SO$_4$)$_2$ or AlK(SO$_4$)$_2$.

**TESTS FOR ALUMINUM.**

Use a separate portion of a solution of an aluminum salt, Al$_2$(SO$_4$)$_3$, for each of the following tests:
1. Add NH₄OH T.S. to the aluminum solution and boil. A white gelatinous precipitate of aluminum hydroxide, Al(OH)₃ will form, which becomes flocculent upon boiling.

2. Slowly add NaOH T.S. to the aluminum solution. A white gelatinous precipitate of aluminum hydroxide, Al(OH)₃, will be produced which will dissolve in an excess of the reagent forming sodium aluminate, NaAlO₂.

3. Add Na₂CO₃ T.S. to the aluminum solution. A white gelatinous precipitate of aluminum hydroxide, Al(OH)₃, will be formed which will dissolve in an excess of the reagent forming sodium aluminate, NaAlO₂.

4. Add (NH₄)₂S T.S. to the aluminum solution. The white gelatinous aluminum hydroxide, Al(OH)₃, will be precipitated and H₂S will be generated.

5. Add Na₂HPO₄ T.S. to the aluminum solution. A white precipitate of aluminum phosphate, AlPO₄, will form. This precipitate is soluble in mineral acids and in solutions of fixed alkalies, but is insoluble in acetic acid.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. \[
\text{Al}_2\text{(SO}_4\text{)}_3 + \text{NH}_4\text{OH} = \\
\text{AlCl}_3 + \text{NH}_4\text{OH}
\]

2. \[
\text{AlCl}_3 + \text{NaOH} = \\
\text{KAl(SO}_4\text{)}_2 + \text{KOH} = \\
\text{Al(OH)}_3 + \text{NaOH}
\]

3. \[
\text{Al}_2\text{(SO}_4\text{)}_3 + \text{Na}_2\text{CO}_3 + \text{H}_2\text{O} = \\
\text{AlCl}_3 + (\text{NH}_4\text{)}_2\text{CO}_3 + \text{H}_2\text{O}
\]

4. \[
\text{AlCl}_3 + (\text{NH}_4\text{)}_2\text{S} + \text{H}_2\text{O} = \\
\text{NH}_4\text{Al(SO}_4\text{)}_2 + (\text{NH}_4\text{)}_2\text{S} + \text{H}_2\text{O}
\]

5. \[
\text{Al}_2\text{(SO}_4\text{)}_3 + \text{Na}_2\text{HPO}_4 = \\
\text{AlCl}_3 + \text{Na}_2\text{HPO}_4
\]

**SAMPLE NUMBER 6.**

**Analysis of a solution for group 4 of the metals.**

The solution may contain salts of aluminum, chromium, ferrous iron or ferric iron.

A. To about 10 mils of the solution add a few drops of HNO₃ T.S. and boil for a moment to oxidize ferrous iron, if present. Then add an equal volume of NH₄Cl T.S. and an excess of NH₄OH T.S. to the hot liquid, boil again, filter and wash the precipitate on the filter paper. The filtrate may be discarded after observing that it smells of ammonia, which shows that the necessary excess of this reagent was used.
**B.** Puncture the filter and transfer the precipitate to a small porcelain evaporating dish with about 5 mils of water. Add about 2 mils of NaOH T.S., boil for several minutes and filter, if necessary.

**C.** Acidulate the filtrate from B with HCl T.S., then add an excess of \((\text{NH}_4)_2\text{CO}_3\) T.S. and boil. A white precipitate shows the presence of aluminum.

**D.** Wash the precipitate from B on the filter paper and allow it to drain. Transfer a part of the moist residue to the cover of a porcelain crucible, cover with about the same amount of dry KNO\(_3\) and about twice as much dry Na\(_2\)CO\(_3\), place the cover on a pipette stem triangle supported on a tripod and heat in a gas flame until the mass is fused. Cool, then heat the cover and adhering mass with about 10 mils of water contained in a small beaker. Remove the cover from the liquid and filter.

**E.** Acidulate the filtrate from D with HC\(_2\)H\(_3\)O\(_2\) T.S. and add Pb(Cl\(_2\)H\(_3\)O\(_2\))\(_2\) T.S. A yellow precipitate shows the presence of chromium.

**F.** Wash any residue from D on the filter and allow it to drain. Pass about 5 mils of HCl T.S. through the filter and add KCNS T.S. to the filtrate. A red coloration shows the presence of iron.

**G.** If iron is found, test separate portions of the original solution for ferrous and ferric salts as follows:

Acidulate one portion with HCl T.S. and add K\(_3\)Fe(CN)\(_6\) T.S. A dark blue precipitate shows the presence of ferrous iron.

Acidulate another portion with HCl T.S. and add KCNS T.S. A red coloration shows the presence of ferric iron.

**NOTE ON GROUP 4.**

Some metals of the fourth and fifth groups are precipitated with the fourth group if either phosphoric acid or oxalic acid is present, as their phosphates and oxalates are insoluble in alkaline liquids; so, in the separation of all the groups, it is necessary to test the original solution for phosphoric acid and oxalic acid and remove these acids, if present, by methods to be described later, before precipitating Group 4.
SAMPLE NUMBER 7.

Analysis of a solution for groups 1, 2, 3 and 4 of the metals.

The solution should be neutral or acid in reaction to litmus. If alkaline add HNO₃ to the portion to be examined until the reaction is acid. Add HCl as long as a precipitate is produced and filter.

<table>
<thead>
<tr>
<th>Precipitate</th>
<th>Filtrate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pb, Hg', Ag.</td>
<td>Warm and pass H₂S as long as a precipitate is produced. Filter.</td>
</tr>
<tr>
<td><strong>Examine for metals of group 1 by I</strong></td>
<td><strong>Precipitate</strong></td>
</tr>
<tr>
<td></td>
<td>Digest with (NH₄)₂Sₓ and filter.</td>
</tr>
<tr>
<td><strong>Residue</strong></td>
<td><strong>Filtrate</strong></td>
</tr>
<tr>
<td>Hg”, Pb, Bi, Cu, Cd.</td>
<td>Sb, Sn, As, Au, Pt.</td>
</tr>
<tr>
<td><strong>Examine for metals of group 2 by II</strong></td>
<td><strong>Filtrate</strong></td>
</tr>
<tr>
<td><strong>Residue</strong></td>
<td><strong>Filtrate</strong></td>
</tr>
<tr>
<td></td>
<td>Boil to expel H₂S. Add a few drops of HNO₃ and boil again, then add NH₄Cl and an excess of NH₄OH and boil again. Filter.</td>
</tr>
<tr>
<td><strong>Precipitate</strong>, Al, Cr, Fe.</td>
<td><strong>Examine for metals of group 4 by IV</strong></td>
</tr>
</tbody>
</table>

I.

Examination of any precipitate produced by HCl in a neutral or acid solution.

Wash the precipitate on the filter with cold water, discarding the washings. Pass a portion of hot water through the washed precipitate on the filter.

<table>
<thead>
<tr>
<th>Filtrate—PbCl₂</th>
<th>Residue—HgCl, AgCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>Add K₂CrO₄. Yellow ppt., PbCrO₄ shows</td>
<td>Pass the same portion of NH₄OH through the residue on the filter several times</td>
</tr>
<tr>
<td><strong>Residue—NH₄HgCl+Hg</strong> black, shows</td>
<td><strong>Filtrate, (NH₄)₂(AgCl)₂</strong></td>
</tr>
</tbody>
</table>

lead mercurous mercury silver
II.

Examination of any residue insoluble in \((\text{NH}_4)_2\text{S_x}\) from a precipitate produced by \(\text{H}_2\text{S}\) in an acid liquid, after group 1 of the metals has been removed from a solution.

Wash the residue on the filter, first with an additional portion of \((\text{NH}_4)_2\text{S_x}\) and then with water, and drain, discarding the washings.

Pour a portion of hot \(\text{HNO}_3\) through the filter several times.

<table>
<thead>
<tr>
<th>Residue—(\text{HgS})</th>
<th>Filtrate—(\text{Pb(NO}_3)_2, \text{Bi(NO}_3)_3, \text{Cu(NO}_3)_2, \text{Cd(NO}_3)_2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Black. Dissolve in nitrohydrochloric acid, boil to expel Cl, dilute with water, filter and add (\text{SnCl}_2). A white or gray ppt. (\text{HgCl} + \text{Hg}) shows</td>
<td>Add conc. (\text{H}_2\text{SO}_4) and boil until white fumes are given off, cool, dilute with (\text{H}_2\text{O}) and filter.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Precipitate—(\text{PbSO}_4)</th>
<th>Filtrate—(\text{Bi(SO}_4)_3, \text{CuSO}_4, \text{CdSO}_4)</th>
</tr>
</thead>
<tbody>
<tr>
<td>White Dissolve with (\text{NH}_2\text{C}_2\text{H}_2\text{O}_2) and add (\text{K}_2\text{Cr}_2\text{O}_7). Yellow ppt., (\text{PbCr}_2\text{O}_4), shows</td>
<td>Add an excess of (\text{NH}_2\text{OH}) and filter.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Precipitate, (\text{Bi(OH)}_3)</th>
<th>Filtrate—(\text{Cu(NH}_3)_2\text{SO}_4, \text{Cd(NH}_3)_2\text{SO}_4)</th>
</tr>
</thead>
<tbody>
<tr>
<td>White. Add (\text{NaOH}) and (\text{SnCl}_2) on filter. Brown or black coloration, (\text{Bi}_2\text{O}_3), shows</td>
<td>Divide into two portions. To one portion add an excess of (\text{HC}_2\text{H}_2\text{O}_2) and then (\text{K}_2\text{Fe(CN)}_4). A red ppt. or coloration, (\text{Cu}_2\text{Fe(CN)}_6), shows</td>
</tr>
</tbody>
</table>

**mercuric mercury** | **lead** | **bismuth** | **copper** | **cadmium** |

III.

Examination of the solution in \((\text{NH}_4)_2\text{S_x}\) of any precipitate produced by \(\text{H}_2\text{S}\) in an acid liquid, after group 1 of the metals has been removed from a solution.

Acidulate the \((\text{NH}_4)_2\text{S_x}\) solution with \(\text{HCl}\) and collect any precipitate formed on a filter. Transfer the precipitate to a test tube and digest in \((\text{NH}_4)_2\text{CO}_3\) T.S. Filter.

<table>
<thead>
<tr>
<th>Filtrate—((\text{NH}_4)_2\text{AsS}_4)</th>
<th>Residue—(\text{Sb}_2\text{S}_3, \text{SnS}_2, \text{AuS}_3, \text{PtS}_2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Add excess of (\text{HCl}) and pass (\text{H}_2\text{S}). A yellow ppt., (\text{AsS}_3), shows</td>
<td>Wash with water. Digest in concentrated (\text{HCl}). Filter.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Filtrate—(\text{SbCl}_3, \text{SnCl}_2)</th>
<th>Residue—(\text{AuS}_3, \text{PtS}_2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Place in (\text{H}) generator. Add (\text{H}_2\text{O}) and (\text{Zn}). Conduct gas into (\text{AgNO}_3) for three minutes If black ppt. forms in (\text{AgNO}_3) dissolve in (\text{HCl}) and pass (\text{H}_2\text{S}). An orange ppt., (\text{SbS}_3), shows</td>
<td>Filter liquid from generator and add few drops to (\text{HgCl}_2). A white or gray ppt., (\text{HgCl} + \text{Hg}), shows</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>arsenic</th>
<th>antimony</th>
<th>tin</th>
<th>gold</th>
<th>platinum</th>
</tr>
</thead>
<tbody>
<tr>
<td>Neutralize another part of the filtrate with (\text{NH}_2\text{OH}), add alcohol and (\text{NH}_2\text{Cl}). A yellow ppt., ((\text{NH}_4)_2\text{PtCl}_6), shows</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
IV.

Examination of any precipitate produced by NH₄OH, after groups 1, 2 and 3 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings.
Boil the precipitate with NaOH for several minutes and filter.

<table>
<thead>
<tr>
<th>Filtrate, NaAlO₂</th>
<th>Residue—Cr(OH)₃, Fe(OH)₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acidulate with HCl, then add an excess of (NH₄)₂CO₃ and boil. A white ppt., Al(OH)₃ shows</td>
<td>Wash with H₂O and drain, discarding the washings. Transfer a part of the residue to a porcelain crucible cover, add about the same amount of dry KNO₃ and about twice as much dry Na₂CO₃ and ignite until fused. Cool, heat the mass with H₂O and filter.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Filtrate, K₂CrO₄</th>
<th>Residue—Fe₂O₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acidulate with HC₂H₃O₂ and add Pb(C₃H₅O₂)₂. A yellow ppt., PbCrO₄ shows</td>
<td>Wash on the filter with water, discarding the washings. Dissolve in HCl and add KCNS. A red coloration, Fe(CNS)₂ shows</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>aluminum</th>
<th>chromium</th>
<th>ferrous iron</th>
<th>ferric iron</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filtrate, Fe₂(CN)₆</td>
<td>Residue—Fe₂O₃</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acidulate with HCl and K₂Fe(CN)₆. A dark blue ppt., Fe₃(Fe(CN)₆)₂ shows</td>
<td>Wash on the filter with water, discarding the washings. Dissolve in HCl and add KCNS. A red coloration, Fe(CNS)₂ shows</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

GROUP 5.

Metals precipitated as sulphides by ammonium sulphide, (NH₄)₂S, in the presence of ammonium hydroxide, NH₄OH:
Cobalt, Co; nickel, Ni; manganese, Mn; zinc, Zn.

COBALT, Co²⁺,³⁺ = 58.97

Cobalt is a light colored metal, resembling nickel, which is seldom used in the metallic state. Its oxide is a constituent of the pigment “smalt,” which is used for coloring glass blue. The cobaltic compounds, in which the metal has a valence of three, are unimportant. In the more important cobaltous salts the metal has a valence of two. Soluble cobalt compounds are generally red in color when hydrated or in solution and deep blue when anhydrous.

IMPORTANT COMPOUNDS OF COBALT.

Cobalt chloride, CoCl₂6H₂O.
Cobalt nitrate, Co(NO₃)₂6H₂O.
Cobalt oxide, CoO.
Cobalt sulphate, CoSO₄7H₂O.
TESTS FOR COBALT.

Use a separate portion of a solution of a cobalt salt, as cobalt nitrate, Co(NO₃)₂, for each of the following tests:

1. Add (NH₄)₂S T.S. to the cobalt solution. A black precipitate of cobalt sulphide, CoS, will form, which is insoluble in an excess of the reagent and in diluted HCl, but is soluble in concentrated HCl and in diluted HNO₃.

2. Add NaOH T.S. to the cobalt solution and boil the liquid. A blue precipitate of a basic salt is obtained which forms cobalt hydroxide, Co(OH)₂, and becomes red in color upon boiling.

3. Slowly add NH₄OH T.S. to the cobalt solution. A precipitate of cobalt hydroxide, Co(OH)₂, is produced, but it immediately dissolves in an excess of the reagent to form a brown solution containing complex double compounds of variable composition.

4. Acidulate a portion of the cobalt solution with acetic acid, and add twice its volume of KNO₂ T.S. Warm the mixture and allow it to stand for some time. A yellow crystalline precipitate of potassium cobaltic nitrite, K₃Co(NO₂)₆, will slowly form. This compound is soluble in water but is insoluble in an excess of KNO₂.

5. Bend the end of a piece of platinum wire around the point of a lead pencil to form a loop about 3 millimeters in diameter. Heat this loop in the oxidizing blowpipe flame for a moment and plunge it into some powdered borax. Heat the adhering borax in the blowpipe flame until it fuses to form a clear and colorless glass-like bead. Dip this bead in the cobalt solution and again heat it in the blowpipe flame. The bead will be colored dark blue. If too much cobalt is present the color will appear to be black.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. Co(NO₃)₂ + (NH₄)₂S =
   CoSO₄ + (NH₄)₂S =
2. Co(NO₃)₂ + NaOH =
   CoCl₂ + KOH =
3. CoCl₂ + NH₄OH =
   CoSO₄ + NH₄OH =
4. CoCl₂ + 7KNO₂ + 2HC₂H₃O₂ = K₃Co(NO₂)₆ + 2KCl + 2KC₂H₃O₂ + H₂O + NO

NICKEL, Ni^{ii,iii} = 58.68

Nickel is a silvery white metal which is largely used in the metallic state for electroplating and as a constituent of various alloys. German silver is an alloy of copper, zinc and nickel. United States "nickel" coins are 75 per cent. copper and 25 per cent. nickel.
While nickel forms two series of compounds, only those in which it has a valence of two are important. Compounds of nickel generally have a bright green color which they impart to their solutions.

**IMPORTANT COMPOUNDS OF NICKEL.**

Nickel-ammonium sulphate, \( \text{Ni}(\text{NH}_4)_2(\text{SO}_4)_2 \).

Nickel chloride, \( \text{NiCl}_2 \cdot 6\text{H}_2\text{O} \).

Nickel oxide, \( \text{NiO} \).

Nickel sulphate, \( \text{NiSO}_4 \cdot 7\text{H}_2\text{O} \).

**TESTS FOR NICKEL.**

Use a separate portion of a solution of a nickel salt, as nickel chloride, \( \text{NiCl}_2 \), for each of the following tests:

1. Add \((\text{NH}_4)_2\text{S}\) T.S. to the nickel solution. A black precipitate of nickel sulphide, \( \text{NiS} \), will be obtained. This precipitate is insoluble in acetic acid and in diluted hydrochloric acid, but is readily soluble in nitric acid or nitrohydrochloric acid.

2. Add \( \text{NaOH} \) T.S. to the nickel solution. A pale green precipitate of nickel hydroxide, \( \text{Ni(OH)}_2 \), will be formed, which is insoluble in an excess of the reagent.

3. Slowly add \( \text{NH}_4\text{OH} \) T.S. to the nickel solution. The pale green precipitate of nickel hydroxide, \( \text{Ni(OH)}_2 \), will be formed which will form a double salt and dissolve to a blue solution in an excess of the reagent.

4. Make a borax bead, dip it in the nickel solution and heat it in the oxidizing flame of the blowpipe. The bead will be colored violet while hot, changing to brown upon cooling.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. \[ \text{NiCl}_2 + (\text{NH}_4)_2\text{S} = \text{NiSO}_4 + (\text{NH}_4)_2\text{S} \]
2. \[ \text{Ni(NO}_3)_2 + \text{NaOH} = \text{NiSO}_4 + \text{KOH} \]
3. \[ \text{NiCl}_2 + \text{NH}_4\text{OH} = \text{NiSO}_4 + 2\text{NH}_4\text{Cl} + 2\text{NH}_4\text{OH} = (\text{NH}_3)_4\text{NiCl}_2 + 4\text{H}_2\text{O} \]

**MANGANESE, Mn**

Manganese is a brittle gray metal, resembling cast iron, which is seldom seen in the metallic state. It is a constituent of some varieties of steel. The element varies widely in valence, forming several series of compounds, of which the most important are the manganous
compounds in which it has a valence of two, and the permanganates in which it has a valence of seven.

The manganous compounds generally have a light pink color. Other compounds of manganese have various bright colors.

**IMPORTANT COMPOUNDS OF MANGANESE.**

Manganese chloride, manganous chloride, \( \text{MnCl}_2 \cdot 4\text{H}_2\text{O} \).
Manganese dioxide, precipitated manganese dioxide, U. S. P., pyrolusite, \( \text{MnO}_2 \).
Manganese hypophosphite, N.F., manganous hypophosphite, \( \text{Mn}[(\text{PH}_2\text{O}_2)_2\text{H}_2\text{O}] \).
Manganese sulphate, N.F., manganous sulphate, \( \text{MnSO}_4 \cdot 4\text{H}_2\text{O} \).

**TESTS FOR MANGANOUS SALTS.**

Use a separate portion of a solution of a manganous salt, as \( \text{MnCl}_2 \), for each of tests 1, 2, 3 and 4:

1. Add \( (\text{NH}_4)_2\text{S} \) T.S. to the manganese solution. A flesh-colored precipitate of manganous sulphide, \( \text{MnS} \), will be formed. Manganous sulphide is readily soluble in diluted mineral acids and in acetic acid.

2. Add \( \text{NaOH} \) T.S. to the manganese solution. A white precipitate of manganous hydroxide, \( \text{Mn(OH)}_2 \), will be formed, which oxidizes to form basic manganic hydroxide, \( \text{MnO(OH)} \), and turns brown upon exposure to air. Both hydroxides are insoluble in an excess of the reagent.

Collect some of the precipitated \( \text{Mn(OH)}_2 \) on a filter paper for use in tests 5 and 6.

3. Add \( \text{NH}_4\text{OH} \) T.S. to the manganese solution. A part of the manganese is precipitated as the white manganous hydroxide, \( \text{Mn(OH)}_2 \), which oxidizes and turns brown upon exposure to air. The other part forms a double salt and remains in solution.

To a portion of the manganese solution add double its volume of \( \text{NH}_4\text{Cl} \) T.S. and then add \( \text{NH}_4\text{OH} \) T.S. The manganese forms a soluble double salt, manganous ammonium chloride, \( \text{Mn(NH}_4)_2\text{Cl} \), and precipitation does not occur until oxidation takes place when the brown basic manganic hydroxide, \( \text{MnO(OH)} \), is slowly precipitated.

4. Add \( \text{Na}_2\text{CO}_3 \) T.S. to the manganese solution. A white precipitate of manganous carbonate, \( \text{MnCO}_3 \), will form, which oxidizes to form the brown basic manganic hydroxide, \( \text{MnO(OH)} \), upon exposure to air.

5. Mix about one-quarter of a teaspoonful of lead peroxide, \( \text{PbO}_2 \), with about 5 mils of \( \text{HNO}_3 \) T.S. and a small quantity of the pre-
ZINC precipitated Mn(OH)$_2$ obtained in test 2, and boil. Allow the powder to settle and observe that the supernatant liquid has the pink or purplish color of permanganic acid, H${\text{MnO}_4}$. The Mn(OH)$_2$ dissolves to form Mn(NO$_3$)$_2$, which is then oxidized to H${\text{MnO}_4}$.

6. Fusion Test.—Place some of the precipitated Mn(OH)$_2$ from test 2 on the cover of a porcelain crucible and cover it with about the same amount of dry KNO$_3$ and twice as much dry Na$_2$CO$_3$. Support the cover on a pipe-stem triangle and heat in the flame of a Bunsen burner until fused. Allow to cool. The manganese is oxidized to manganic acid by the potassium nitrate and a green mass containing potassium and sodium manganates will be obtained.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. MnCl$_2$ + (NH$_4$)$_2$S =
   MnSO$_4$ + (NH$_4$)$_2$S =
2. MnSO$_4$ + NaOH =
   MnCl$_2$ + KOH =
   4Mn(OH)$_2$ + O$_2$ = 4MnO(OH) + 2H$_2$O
3. 2MnSO$_4$ + 2NH$_4$OH = Mn(NH$_4$)$_2$(SO$_4$)$_2$ + Mn(OH)$_2$
   MnCl$_2$ + 2NH$_4$Cl = Mn(NH$_4$)$_2$Cl$_4$
4. Mn(NO$_3$)$_2$ + Na$_2$CO$_3$ =
   MnSO$_4$ + (NH$_4$)$_2$CO$_3$ =
5. 2Mn(NO$_3$)$_2$ + 5PbO$_2$ + 6HNO$_3$ = 2HMnO$_4$ + 5Pb(NO$_3$)$_2$ + 2H$_2$O
6. 3Mn(OH)$_2$ + 4KNO$_3$ + Na$_2$CO$_3$ = 2K$_2$MnO$_4$ + Na$_2$MnO$_4$ + 4NO + CO$_2$ + 3H$_2$O

ZINC, Zn$^{ii}$ = 65.37

Zinc is a bluish-white crystalline metal which is largely used in the metallic state. Brass is an alloy of copper and zinc. Galvanized iron is sheet iron coated with zinc. Zinc salts are poisonous and are generally colorless.

IMPORTANT COMPOUNDS OF ZINC.

Zinc acetate, U. S. P., Zn(C$_2$H$_5$O$_2$)$_2$.
Zinc bromide, ZnBr$_2$.
Zinc sub-carbonate, precipitated zinc carbonate, U. S. P., basic zinc carbonate, (ZnCO$_3$)$_2$(Zn(OH)$_2$)$_3$.
Zinc chloride, U. S. P., ZnCl$_2$.
Zinc iodide, ZnI$_2$.
Zinc oxide, U. S. P., “zinc white,” ZnO.
Zinc sulphate, U. S. P., “white vitriol,” ZnSO$_4$7H$_2$O.
THE METALS

TESTS FOR ZINC.

Use a separate portion of a solution of a zinc salt, as ZnSO₄, for each of tests 1, 2, 3 and 4.

1. Add \((\text{NH}_4)_2\text{S}\) T.S. to the zinc solution. A white precipitate of zinc sulphide, ZnS, will be obtained. The precipitate is soluble in mineral acids, but is not soluble in acetic acid.

2. Slowly add NaOH T.S. to the zinc solution. A white precipitate of zinc hydroxide, Zn(OH)₂, is obtained, which forms sodium zincate, Na₂ZnO₂, and dissolves in an excess of the reagent. The zinc is partially re-precipitated as zinc oxide, ZnO, upon boiling the solution.

3. Slowly add NH₄OH T.S. to the zinc solution until the precipitate formed is re-dissolved, then boil the solution obtained. The first precipitate formed is zinc hydroxide, Zn(OH)₂. This dissolves in an excess of NH₄OH to form ammonium zincate, \((\text{NH}_4)_2\text{ZnO}_2\). Finally a precipitate of zinc oxide, ZnO, is formed upon boiling the solution.

4. Add Na₂CO₃ T.S. to the zinc solution. A white precipitate of zinc sub-carbonate, \((\text{ZnCO}_3)_2\text{Zn(OH)}_2\), is obtained. This precipitate is soluble in acids and in alkali hydroxides.

Collect some of the precipitated zinc sub-carbonate on a small filter paper, wash with water and allow to drain for use in test 5.

5. Fold the filter paper containing the precipitate collected from test 4, place it on the cover of a porcelain crucible with a small amount of dry Na₂CO₃ and add a few drops of Co(NO₃)₂ T.S. Place the cover on a pipe-stem triangle supported on a tripod and ignite in the flame of a gas burner. A green mass consisting of zinc and cobalt oxides will be obtained.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. \(\text{ZnSO}_4 + (\text{NH}_4)_2\text{S} =\)
   \(\text{Zn(C}_2\text{H}_3\text{O}_2)_2 + \text{K}_2\text{S} =\)

2. \(\text{ZnCl}_2 + \text{NaOH} =\)
   \(\text{Zn(C}_2\text{H}_3\text{O}_2)_2 + \text{KOH} =\)
   \(\text{Zn(OH)}_2 + \text{NaOH} =\)

3. \(\text{ZnSO}_4 + \text{NH}_4\text{OH} =\)
   \(\text{Zn(OH)}_2 + \text{NH}_4\text{OH} =\)
   \((\text{NH}_4)_2\text{ZnO}_2 + \text{heat} =\)

4. \(5\text{ZnSO}_4 + 5\text{Na}_2\text{CO}_3 + 3\text{H}_2\text{O} = (\text{ZnCO}_3)_2(\text{Zn(OH)}_2)_3 + 5\text{Na}_2\text{SO}_4 + 3\text{CO}_2\)
   \(\text{ZnCl}_2 + \text{K}_2\text{CO}_3 =\)
The sample may contain salts of any of the following metals: cobalt, nickel, manganese, zinc.

A. To about 10 mils of the solution add (NH₄)₂S T.S. If no precipitate is produced, the metals of group 5 are absent. If a precipitate is produced, continue to add the reagent until precipitation appears to be complete. Filter and test the filtrate. If precipitation is complete, the filtrate may be discarded.

B. Wash the precipitate on the filter with cold water, discarding the washings, then pass the same portion of about 5 mils of HCl T.S. through the precipitate on the filter several times. Cobalt and nickel sulphides, if present, remain undissolved, and zinc and manganese, if present, form chlorides and dissolve.

C. Boil the filtrate from B until it ceases to smell of H₂S. Add an excess of NaOH T.S. and filter, if necessary. Manganese, if present, is precipitated as Mn(OH)₂, and zinc, if present, forms Na₂ZnO₂ and remains in solution.

D. Pass H₂S through the filtrate from C. If no precipitate is produced, zinc is absent. A white or gray precipitate, ZnS, shows the presence of zinc.

E. If NaOH produced no precipitate in C, manganese is absent. If a precipitate was produced by NaOH in C, transfer some of it from the filter to a test-tube, add a small amount of powdered lead peroxide, PbO₂, and about 5 mils of HNO₃ T.S., and boil. Allow the powder to settle. If the supernatant liquid has a pink color, NaMnO₄, the presence of manganese is shown.

F. If there is no dark-colored residue on the filter paper from B, cobalt and nickel are absent. If there is a dark-colored residue insoluble in HCl T.S., in B, puncture the filter and wash the residue into a test-tube with about 5 mils of nitro-hydrochloric acid. Add an excess of NaOH T.S. and collect the precipitate produced on a filter and wash with water, discarding the filtrate and washings. Pass the same portion of about 5 mils of HC₂H₃O₂ T.S. through the precipitate on the filter several times.

G. To the solution in HC₂H₃O₂, from F, add about 10 mils each of KCl T.S. and KNO₃ T.S., let stand for half an hour and filter if necessary. If no precipitate is produced cobalt is absent. If KCl and KNO₃ produce a yellow precipitate, K₂Co(NO₃)₆, the presence of cobalt is shown.

H. To the filtrate from G, add an excess of NaOH. If no precipitate is produced, nickel is absent. A light green precipitate Ni(OH)₂ shows the presence of nickel.
SAMPLE NUMBER 9.

Analysis of a solution for groups 1, 2, 3, 4 and 5 of the metals.

The solution should be neutral or acid in reaction to litmus. If alkaline add HNO₃ to the portion to be examined for metals until the reaction is acid. Add HCl as long as a precipitate is produced and filter.

<table>
<thead>
<tr>
<th>Precipitate</th>
<th>Filtrate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pb, Hg', Ag.</td>
<td>Warm and pass H₂S as long as a precipitate is produced. Filter</td>
</tr>
</tbody>
</table>

Examine for metals of group 1 by I.

<table>
<thead>
<tr>
<th>Precipitate</th>
<th>Filtrate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Digest with (NH₄)₂S₧ and filter.</td>
<td>Boil to expel H₂S. Add a few drops of HNO₃ and boil again then add NH₄Cl and an excess of NH₄OH and boil again. Filter</td>
</tr>
</tbody>
</table>

Residue Hg", Pb, Bi, Cu, Cd. | Filtrate Sb, Sn, As, Au, Pt. |
Examine for metals of group 2 by II. | Examine for metals of group 3 by III |

Precipitate | Filtrate |
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Al, Cr, Fe.</td>
<td>Pass H₂S as long as a ppt is produced. Filter</td>
</tr>
</tbody>
</table>

Prencipitate Zn, Mn, Co, Ni. | Filtrate |
Examine for metals of group 5 by V. |

I.

Examination of any precipitate produced by HCl in a neutral or acid solution.

Wash the precipitate on the filter with cold water, discarding the washings. Pass a portion of hot water through the washed precipitate on the filter.

Filtrate—PbCl₂ | Residue—HgCl, AgCl |
Add H₂SO₄ and cool. White ppt., PbSO₄, shows lead | Pass the same portion of NH₄OH through the residue on the filter several times |

Residue—NH₄HgCl + Hg, black, shows mercurous mercury | Filtrate, (NH₄)₂(AgCl)₂, Add an excess of HNO₃ white ppt., AgCl, shows silver |
II.

Examination of any residue insoluble in $\text{(NH}_4\text{)}_2\text{S}_x$ from a precipitate produced by $\text{H}_2\text{S}$ in an acid liquid, after group 1 of the metals has been removed from a solution.

Wash the residue on the filter, first with an additional portion of $\text{(NH}_4\text{)}_2\text{S}_x$ and then with water, and drain, discarding the washings.

Pour a portion of hot $\text{HNO}_3$ through the filter several times.

**Residue—HgS**

Black. Dissolve in nitrohydrochloric acid, boil to expel Cl, dilute with water, filter and add SnCl. A white or gray ppt. $\text{HgCl}_2 + \text{Hg}$ shows

<table>
<thead>
<tr>
<th>Precipitate—PbSO$_4$</th>
<th>Filtrate—$\text{Pb(NO}_3\text{)}_2$, $\text{Bi(NO}_3\text{)}_3$, $\text{Cu(NO}_3\text{)}_2$, $\text{Cd(NO}_3\text{)}_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>White</td>
<td>Add conc. $\text{H}_2\text{SO}_4$ and boil until white fumes are given off, cool, dilute with $\text{H}_2\text{O}$ and filter.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Precipitate, Bi(OH)$_3$</th>
<th>Filtrate—$\text{Bi}_2(\text{SO}_4)_3$, $\text{CuSO}_4$, $\text{CdSO}_4$</th>
</tr>
</thead>
<tbody>
<tr>
<td>White</td>
<td>Add NaOH and SnCl$_3$ on filter. Brown or black coloration, Bi$_2$O$_3$, shows</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Precipitate, $\text{Cu(NO}_3\text{)SO}_4$, $\text{Cd(NO}_3\text{)SO}_4$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Divide into two portions. Pass $\text{H}_2\text{S}$ through the other portion, decolorizing with KCN, if Cu is present. A yellow ppt., CdS, shows</td>
</tr>
</tbody>
</table>

Examination of the solution in $\text{(NH}_4\text{)}_2\text{S}_x$ of any precipitate produced by $\text{H}_2\text{S}$ in an acid liquid, after group 1 of the metals has been removed from a solution.

Acidulate the $\text{(NH}_4\text{)}_2\text{S}_x$ solution with HCl and collect any precipitate formed on a filter. Transfer the precipitate to a test tube and digest in $\text{(NH}_4\text{)}_2\text{CO}_3$ T.S. Filter.

<table>
<thead>
<tr>
<th>Filtrate—$\text{(NH}_4\text{)}_2\text{AsS}_4$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wash with water. Digest in concentrated HCl. Filter.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Residue—$\text{Sb}_2\text{S}_3$, $\text{SnS}_2$, $\text{Au}_2\text{S}_3$, $\text{PtS}_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wash with water. Digest in concentrated HCl. Filter.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Filtrate—$\text{SbCl}_3$, $\text{SnCl}_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Place in H generator. Add $\text{H}_2\text{O}$ and Zn. Conduct gas into $\text{AgNO}_3$ for three minutes</td>
</tr>
</tbody>
</table>

If black ppt. forms in $\text{AgNO}_3$ dissolve in HCl and pass $\text{H}_2\text{S}$. An orange ppt., $\text{Sb}_2\text{S}_3$, shows

| Filter liquid from generator and add few drops to $\text{HgCl}_2$. A white or gray ppt., $\text{HgCl}_2 + \text{Hg}$, shows |

<table>
<thead>
<tr>
<th>Residue—$\text{Au}_2\text{S}_3$, $\text{PtS}_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>If light colored Au and Pt are absent. If dark colored, digest in nitrohydrochloric acid. Add $\text{H}_2\text{O}$ and filter.</td>
</tr>
</tbody>
</table>

To a part of the filtrate add SnCl and SnCl$_3$ and let stand. A purple ppt., Au$_2$O$_3$, etc., shows

Neutralize another part of the filtrate with NH$_2$OH, add alcohol and NH$_2$Cl. A yellow ppt., $\text{(NH}_4\text{)}_2\text{PtCl}_6$, shows

<table>
<thead>
<tr>
<th>arsenic</th>
<th>antimony</th>
<th>tin</th>
<th>gold</th>
<th>platinum</th>
</tr>
</thead>
</table>


IV.

Examination of any precipitate produced by NH₄OH, after groups 1, 2 and 3 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings.

Boil the precipitate with NaOH for several minutes and filter.

<table>
<thead>
<tr>
<th>Filtrate, NaAlO₂</th>
<th>Residue—Cr(OH)₃, Fe(OH)₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acidulate with HCl, then add an excess of (NH₄)₂CO₃ and boil. A white ppt., Al(OH)₃ shows</td>
<td>Wash with H₂O and drain, discarding the washings. Transfer a part of the residue to a porcelain crucible cover, add about the same amount of dry KNO₃ and about twice as much dry Na₂CO₃ and ignite until fused. Cool, heat the mass with H₂O and filter.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Filtrate, K₂CrO₄</th>
<th>Residue—Fe₂O₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acidulate with HCl₂H₂O₂ and add Pb(C₂H₃O₂)₃. A yellow ppt., PbCrO₄ shows</td>
<td>Wash on the filter with water, discarding the washings. Dissolve in HCl and add KCNS. A red coloration, Fe(CNS)₃, shows iron</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>aluminum</th>
<th>chromium</th>
</tr>
</thead>
<tbody>
<tr>
<td>filtration</td>
<td>filtration</td>
</tr>
</tbody>
</table>

| To one portion add HCl and K₃Fe(CN)₆. A dark blue ppt., Fe₃(Fe(CN)₆)₁₂ shows | To another portion add HCl and KCNS. A red coloration, Fe(CNS)₃, shows |

V.

Examination of any precipitate produced by H₂S in an alkaline liquid, after groups 1, 2, 3 and 4 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings. Pass the same portion of HCl through the filter several times.

<table>
<thead>
<tr>
<th>Filtrate—ZnCl₂, MnCl₂</th>
<th>Residue—CoS, NiS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boil to expel H₂S, cool, add excess of NaOH and filter. Filtrate, Na₂ZnO₂ Pass H₂S. A white ppt. ZnS, shows</td>
<td>If light colored, cobalt and nickel are absent. If dark colored, dissolve in nitro-hydrochloric acid, add an excess of NaOH, collect the ppt. produced on a filter and wash, discarding the washings. Dissolve the ppt. in HCl₂H₂O₂, add an excess of KCl and of KNO₃, let stand for half an hour and filter.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Precipitate, K₃Co(NO₃)₆, yellow, shows</th>
<th>Filtrate, Ni(C₂H₃O₂)₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>zinc</td>
<td>manganese</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>cobalt</th>
<th>nickel</th>
</tr>
</thead>
<tbody>
<tr>
<td>filtration</td>
<td>filtration</td>
</tr>
</tbody>
</table>
Metals precipitated as carbonates from a neutral or alkaline solution by ammonium carbonate, \((\text{NH}_4)_2\text{CO}_3\) in the presence of ammonium chloride, \(\text{NH}_4\text{Cl}\):

Calcium, Ca; strontium, Sr; barium, Ba.

**CALCIUM, Ca\(^{ii}\) = 40.07**

Calcium is a light colored metal which quickly oxidizes in the air; it is seldom seen in the metallic state, but many of its compounds are important. Many of the compounds of calcium were formerly named as salts of lime, for example; calcium phosphate was formerly called "phosphate of lime."

**IMPORTANT COMPOUNDS OF CALCIUM.**

- Calcium acetate, \(\text{Ca(C}_2\text{H}_3\text{O}_2)_2\).
- Calcium bromide, U. S. P., \(\text{CaBr}_2\).
- Calcium carbonate, precipitated calcium carbonate, U. S. P., prepared chalk, U. S. P., marble, chalk, limestone, whiting, \(\text{CaCO}_3\).
- Calcium carbide, \(\text{CaC}_2\).
- Calcium chloride, U. S. P., \(\text{CaCl}_2\).
- Calcium hydroxide, slaked lime, \(\text{Ca(OH)}_2\).
- Calcium hypophosphite, U. S. P., \(\text{Ca(PH}_2\text{O}_2)_2\).
- Calcium iodide, \(\text{CaI}_2\).
- Calcium oxide, U. S. P., lime, quicklime, \(\text{CaO}\).
- Calcium phosphate, precipitated calcium phosphate, N.F., \(\text{Ca}_3(\text{PO}_4)_2\).
- Calcium sulphate, gypsum, \(\text{CaSO}_4\cdot2\text{H}_2\text{O}\).
- Exsiccated calcium sulphate, plaster of Paris, \(\text{CaSO}_4\).
- Calcium sulphide, \(\text{CaS}\).
- Calcium sulphite, \(\text{CaSO}_3\cdot2\text{H}_2\text{O}\).
- Chlorinated lime, U. S. P., bleaching powder, "chloride of lime," \(\text{CaOCl}_2\).

**TESTS FOR CALCIUM.**

Use separate portions of a solution of a calcium salt, as \(\text{CaCl}_2\), for each of the following tests:

1. Add \((\text{NH}_4)_2\text{CO}_3\) T.S. to the calcium solution. A white precipitate of calcium carbonate, \(\text{CaCO}_3\), will be obtained, which is not soluble in an excess of the reagent or in alkalis, but is readily soluble in acids. Any soluble carbonate will give the same precipitate.

2. Add \(\text{NaOH}\) T.S. to the calcium solution. A white precipitate of calcium hydroxide, \(\text{Ca(OH)}_2\), will be obtained if the solutions are sufficiently concentrated. No precipitate is obtained from dilute solutions, as \(\text{Ca(OH)}_2\) is slightly soluble in water. It is less soluble
in hot water than it is in cold water. Ammonium hydroxide does not precipitate calcium from solutions of calcium salts.

3. Add \( \text{H}_2\text{SO}_4 \) T.S. to the calcium solution. A white precipitate of calcium sulphate, \( \text{CaSO}_4 \), will be obtained, unless the solution is very dilute. \( \text{CaSO}_4 \) is slightly soluble in water. Soluble sulphates, as \( \text{Na}_2\text{SO}_4 \), will give the same precipitate.

4. Add \( \text{Na}_2\text{HPO}_4 \) T.S. to the calcium solution. A white precipitate of calcium hydrogen phosphate, \( \text{CaHPO}_4 \), will be obtained. The precipitate is readily soluble in acids.

5. Add \( \text{(NH}_4\text{)}_2\text{C}_2\text{O}_4 \) T.S. to the calcium solution. A white precipitate of calcium oxalate, \( \text{CaC}_2\text{O}_4 \), will be obtained. The precipitate is readily soluble in mineral acids or in oxalic acid, but is not soluble in acetic acid.

6. Clean a piece of platinum wire by holding it in a blue gas flame until it ceases to give any color to the flame. Dip the wire in the calcium solution and heat again. The flame will be colored yellowish-red.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. \[
\text{CaCl}_2 + (\text{NH}_4)_2\text{CO}_3 = \text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2 + \text{Na}_2\text{CO}_3
\]

2. \[
\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2 + \text{NaOH} = \text{CaBr}_2 + \text{KOH}
\]

3. \[
\text{CaBr}_2 + \text{K}_2\text{SO}_4 = \text{CaCl}_2 + (\text{NH}_4)_2\text{SO}_4
\]

4. \[
\text{CaCl}_2 + \text{Na}_2\text{HPO}_4 = \text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2 + \text{Na}_2\text{HPO}_4
\]

5. \[
\text{CaCl}_2 + (\text{NH}_4)_2\text{C}_2\text{O}_4 = \text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2 + \text{K}_2\text{C}_2\text{O}_4
\]

**STRONTIUM, \( \text{Sr}^{\text{ii}} = 87.63 \)**

Strontium is a yellow metal which readily oxidizes in moist air and actively decomposes water. It is seldom seen in the metallic state.

**IMPORTANT COMPOUNDS OF STRONTIUM.**

Strontium bromide, U. S. P., \( \text{SrBr}_2\text{H}_2\text{O} \).

Strontium carbonate, \( \text{SrCO}_3 \).

Strontium chloride, \( \text{SrCl}_2\text{H}_2\text{O} \).

Strontium iodide, U. S. P., \( \text{SrI}_2\text{H}_2\text{O} \).

Strontium nitrate, \( \text{Sr(NO}_3)_2 \).

**TESTS FOR STRONTIUM.**

Use separate portions of a solution of a strontium salt, as \( \text{SrCl}_2 \), for each of the following tests:
1. Add \((\text{NH}_4)_2\text{CO}_3\) T.S. to the strontium solution. A white precipitate of strontium carbonate, \(\text{SrCO}_3\), will form, which is insoluble in an excess of the reagent or in alkalies, but is readily soluble in acids.

2. Add \(\text{H}_2\text{SO}_4\) T.S. to the strontium solution. A white precipitate of strontium sulphate, \(\text{SrSO}_4\), will form if the solution is not too dilute. The precipitate is insoluble in acids or alkalies.

3. Add \(\text{CaSO}_4\) T.S. to the strontium solution and warm the mixture. A white precipitate of strontium sulphate, \(\text{SrSO}_4\), will form slowly. As \(\text{CaSO}_4\) is only slightly soluble in water, the reagent is very dilute, so the amount of the precipitate is small. As this reagent will not precipitate calcium it can be used to detect strontium in the presence of calcium.

4. Add \(\text{Na}_2\text{HPO}_4\) T.S. to the strontium solution. A white precipitate of strontium hydrogen phosphate, \(\text{SrHPO}_4\), will form, which is soluble in acids.

5. Add \((\text{NH}_4)_2\text{C}_2\text{O}_4\) T.S. to the strontium solution. A white precipitate of strontium oxalate, \(\text{SrC}_2\text{O}_4\), will form. The precipitate is readily soluble in mineral acids, but only very slightly soluble in acetic or oxalic acid solutions.

6. Hold a piece of platinum in a non-luminous gas flame until it ceases to give any color to the flame. Dip the cleaned wire in the strontium solution and again hold it in the flame. The flame will be colored deep red.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. \[\text{SrCl}_2 + (\text{NH}_4)_2\text{CO}_3 = \]
   \[\text{Sr(NO}_3)_2 + \text{Na}_2\text{CO}_3 = \]

2. \[\text{Sr(NO}_3)_2 + \text{H}_2\text{SO}_4 = \]
   \[\text{SrCl}_2 + \text{Na}_2\text{SO}_4 = \]

3. \[\text{SrCl}_2 + \text{CaSO}_4 = \]
   \[\text{Sr(NO}_3)_2 + \text{CaSO}_4 = \]

4. \[\text{Sr(NO}_3)_2 + \text{Na}_2\text{HPO}_4 = \]
   \[\text{SrCl}_2 + (\text{NH}_4)_2\text{HPO}_4 = \]

5. \[\text{SrCl}_2 + (\text{NH}_4)_2\text{C}_2\text{O}_4 = \]
   \[\text{Sr(NO}_3)_2 + \text{K}_2\text{C}_2\text{O}_4 = \]

**BARIUM, \(\text{Ba}^{ii} = 137.37\)**

Barium is a yellowish-white metal which is not used in the metallic state. Its compounds are poisonous.

**IMPORTANT COMPOUNDS OF BARIUM.**

Barium carbonate, \(\text{BaCO}_3\).

Barium chloride, \(\text{BaCl}_2\text{H}_2\text{O}\).

Barium dioxide, barium peroxide, \(\text{BaO}_2\).
Barium hydroxide, \( \text{Ba(OH)}_2 \).
Barium nitrate, \( \text{Ba(NO}_3\text{)}_2 \).
Barium sulphate, permanent white, \( \text{BaSO}_4 \).
Barium sulphide, \( \text{BaS} \).

**Tests for Barium.**

Use a separate portion of a solution of a barium compound, as \( \text{BaCl}_2 \), for each of the following tests:

1. Add \((\text{NH}_4)_2\text{CO}_3\) T.S. to the barium solution. A white precipitate of barium carbonate, \( \text{BaCO}_3 \), will be obtained. Other soluble carbonates, as \( \text{Na}_2\text{CO}_3 \), will give the same precipitate.

2. Add \(\text{H}_2\text{SO}_4\) T.S. to the barium solution. A white precipitate of barium sulphate, \( \text{BaSO}_4 \), will be obtained, which is insoluble in acids or alkalis. Soluble sulphates, as \( \text{K}_2\text{SO}_4 \), give the same precipitate.

3. Add \((\text{NH}_4)_2\text{C}_2\text{O}_4\) T.S. to the barium solution. A white precipitate of barium oxalate, \( \text{BaC}_2\text{O}_4 \), will be obtained. The precipitate is soluble in acetic acid or in a solution of oxalic acid.

4. Add \(\text{Na}_2\text{HPO}_4\) T.S. to the barium solution. A white precipitate of barium hydrogen phosphate, \( \text{BaHPO}_4 \), will be obtained.

5. Add \(\text{K}_2\text{Cr}_2\text{O}_7\) T.S. to the barium solution. A yellow precipitate of barium chromate, \( \text{BaCrO}_4 \), will be obtained. This precipitate is soluble in nitric and hydrochloric acids, but is insoluble in acetic acid. \( \text{K}_2\text{CrO}_4 \) T.S. will give the same precipitate.

6. Make a flame test of the barium solution with platinum wire. The flame will be colored a yellowish-green.

**Complete and Balance the Following Equations:**

1. \( \text{BaCl}_2 + (\text{NH}_4)_2\text{CO}_3 = \)
\( \text{Ba(NO}_3\text{)}_2 + \text{Na}_2\text{CO}_3 = \)

2. \( \text{Ba(NO}_3\text{)}_2 + \text{H}_2\text{SO}_4 = \)
\( \text{BaCl}_2 + \text{K}_2\text{SO}_4 = \)

3. \( \text{Ba(C}_2\text{H}_5\text{O}_2\text{)}_2 + (\text{NH}_4)_2\text{C}_2\text{O}_4 = \)
\( \text{BaCl}_2 + \text{K}_2\text{C}_2\text{O}_4 = \)

4. \( \text{BaCl}_2 + \text{Na}_2\text{HPO}_4 = \)
\( \text{Ba(NO}_3\text{)}_2 + (\text{NH}_4)_2\text{HPO}_4 = \)

5. \( \text{BaCl}_2 + \text{K}_2\text{Cr}_2\text{O}_7 + \text{H}_2\text{O} = \)
\( \text{Ba(NO}_3\text{)}_2 + \text{K}_2\text{CrO}_4 = \)

**Sample Number 10.**

**Analysis of a solution for group 6 of the metals.**

The solution may contain salts of any of the following metals: calcium, strontium, barium.

A. To about 10 mils of the sample add about 5 mils of \( \text{NH}_4\text{Cl} \) T.S. and about 5 mils of \( \text{NH}_4\text{OH} \) T.S. and then \( (\text{NH}_4)_2\text{CO}_3 \) T.S. If no
precipitate is produced, calcium, strontium and barium are absent. If a precipitate is produced, continue to add \((\text{NH}_4)_2\text{CO}_3\) T.S. until precipitation appears to be complete. Filter and test the filtrate.

**B.** Wash any precipitate obtained in A on the filter with water, discarding the washings. Pour the same portion of about 5 mils of \(\text{HC}_2\text{H}_3\text{O}_2\) T.S. through the filter several times to dissolve the precipitate. Add a few drops of \(\text{K}_2\text{Cr}_2\text{O}_7\) T.S. to the solution. If no precipitate is produced, barium is absent. A yellow precipitate shows the presence of barium. If barium is present continue to add \(\text{K}_2\text{Cr}_2\text{O}_7\) T.S. until precipitation appears to be complete. Filter and test the filtrate. Divide the liquid or filtrate into two portions.

**C.** To one portion of the liquid or filtrate from B add \(\text{CaSO}_4\) T.S., boil and let stand. If no precipitate is produced, strontium is absent. A small amount of white precipitate, slowly formed, shows the presence of strontium.

**D.** To the other portion of the filtrate from B, add \(\text{K}_2\text{SO}_4\) T.S., boil, let stand for ten minutes and filter, to remove strontium if present. To the filtrate add \((\text{NH}_4)_2\text{C}_2\text{O}_4\) T.S. If no precipitate is produced, calcium is absent. A small amount of white precipitate slowly formed shows the presence of calcium.

**SAMPLE NUMBER 11.**

**Analysis of a solution for groups 1, 2, 3, 4, 5 and 6 of the metals.**

The solution should be neutral or acid in reaction to litmus. If alkaline add \(\text{HNO}_3\) to the portion to be examined for metals until the reaction is acid. Add \(\text{HCl}\) as long as a precipitate is produced and filter.

<table>
<thead>
<tr>
<th>Precipitate</th>
<th>Filtrate</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\text{Pb}, \text{Hg}, \text{Ag})</td>
<td>Warm and pass (\text{H}_2\text{S}) as long as a precipitate is produced. Filter.</td>
</tr>
<tr>
<td>Examine for metals of group 1 by I.</td>
<td></td>
</tr>
<tr>
<td><strong>Residue</strong></td>
<td></td>
</tr>
<tr>
<td>(\text{Hg}^+, \text{Pb}, \text{Bi}, \text{Cu}, \text{Cd})</td>
<td></td>
</tr>
<tr>
<td>Examine for metals of group 2 by II.</td>
<td></td>
</tr>
<tr>
<td><strong>Precipitate</strong></td>
<td></td>
</tr>
<tr>
<td>Digest with ((\text{NH}_4)_2\text{S}_x) and filter.</td>
<td></td>
</tr>
<tr>
<td><strong>Filtrate</strong></td>
<td></td>
</tr>
<tr>
<td>Examine for metals of group 3 by III.</td>
<td></td>
</tr>
<tr>
<td><strong>Precipitate</strong></td>
<td></td>
</tr>
<tr>
<td>Boil to expel (\text{H}_2\text{S}). Add a few drops of (\text{HNO}_3) and boil again, then add (\text{NH}_4\text{Cl}) and an excess of (\text{NH}_4\text{OH}) and boil again. Filter.</td>
<td></td>
</tr>
<tr>
<td><strong>Filtrate</strong></td>
<td></td>
</tr>
<tr>
<td>Pass (\text{H}_2\text{S}) as long as a ppt. is produced. Filter.</td>
<td></td>
</tr>
<tr>
<td><strong>Precipitate</strong></td>
<td></td>
</tr>
<tr>
<td>(\text{Zn}, \text{Mn}, \text{Co}, \text{Ni})</td>
<td></td>
</tr>
<tr>
<td>Examine for metals of group 5 by V.</td>
<td></td>
</tr>
<tr>
<td><strong>Filtrate</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Precipitate</strong></td>
<td></td>
</tr>
<tr>
<td>(\text{Ba}, \text{Sr}, \text{Ca})</td>
<td></td>
</tr>
<tr>
<td>Examine for metals of group 6 by VI.</td>
<td></td>
</tr>
</tbody>
</table>
I.

Examination of any precipitate produced by HCl in a neutral or acid solution.

Wash the precipitate on the filter with cold water, discarding the washings. Pass a portion of hot water through the washed precipitate on the filter.

<table>
<thead>
<tr>
<th>Filtrate—PbCl₂</th>
<th>Residue—HgCl, AgCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>Add H₂SO₄ and cool. White ppt. PbSO₄ shows</td>
<td>Pass the same portion of NH₄OH through the residue on the filter several times</td>
</tr>
<tr>
<td></td>
<td>Residue—NH₄HgCl+Hg, lead</td>
</tr>
<tr>
<td></td>
<td>Filtrate, (NH₄)₂(AgCl)₂ silver</td>
</tr>
</tbody>
</table>

II.

Examination of any residue insoluble in (NH₄)₂Sₓ from a precipitate produced by H₂S in an acid liquid, after group 1 of the metals has been removed from a solution.

Wash the residue on the filter, first with an additional portion of (NH₄)₂Sₓ and then with water, and drain, discarding the washings. Pour a portion of hot HNO₃ through the filter several times.

<table>
<thead>
<tr>
<th>Residue, HgS</th>
<th>Filtrate—Pb(NO₃)₂, Bi(NO₃)₃, Cu(NO₃)₂, Cd(NO₃)₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Black. Dissolve in nitrohydrochloric acid, boil to expel Cl, dilute with water, filter and add SnCl₂. A white or gray ppt. HgCl+Hg shows</td>
<td>Add conc. H₂SO₄ and boil until white fumes are given off, dilute with H₂O, cool and filter.</td>
</tr>
<tr>
<td>Precipitate, PbSO₄</td>
<td>Precipitate, Bi(OH)₃</td>
</tr>
<tr>
<td>White. Dissolve with NH₄C₂H₅O₂ and add K₂Cr₂O₇. Yellow ppt., PbCrO₄, shows</td>
<td>White. Add NaOH and SnCl₂ on filter. Brown or black coloration, Bi₂O₃, shows</td>
</tr>
<tr>
<td>mercuric mercury</td>
<td>lead</td>
</tr>
<tr>
<td>bismuth</td>
<td>copper</td>
</tr>
<tr>
<td>cadmium</td>
<td></td>
</tr>
</tbody>
</table>

Divide into two portions.

To one portion add an excess of HC₂H₅O₂ and then K₄Fe(CN)₆. A red ppt. or coloration Cu₂Fe(CN)₅ shows |

Pass H₂S through the other portion, first decolorizing with KCN if Cu is present. A yellow ppt., CdS, shows
III.

Examination of the solution in \((\text{NH}_4)_2\text{S}_x\) of any precipitate produced by \(\text{H}_2\text{S}\) in an acid liquid, after group 1 of the metals has been removed from a solution.

Acidulate the \((\text{NH}_4)_2\text{S}_x\) solution with \(\text{HCl}\) and collect any precipitate formed on a filter. Transfer the precipitate to a test tube and digest in \((\text{NH}_4)_2\text{CO}_3\) T.S. Filter.

<table>
<thead>
<tr>
<th>Residue—(\text{Sb}_2\text{S}_3, \text{SnS}_2, \text{Au}_2\text{S}_3, \text{PtS}_2)</th>
<th>Solution in ((\text{NH}_4)_2\text{S}_x) of any precipitate produced by (\text{H}_2\text{S}) in an acid liquid, after group 1 of the metals has been removed from a solution.</th>
</tr>
</thead>
</table>

Filtrate—

\((\text{NH}_4)_2\text{AsS}_4\)

Add excess of \(\text{HCl}\) and pass \(\text{H}_2\text{S}\). A yellow ppt., \(\text{AsS}_3\), shows

Residue—\(\text{Sb}_2\text{S}_3, \text{SnS}_2, \text{Au}_2\text{S}_3, \text{PtS}_2\)

Wash with water. Digest in concentrated \(\text{HCl}\). Filter.

Filtrate—\(\text{SbCl}_5, \text{SnCl}_2\)

Place in \(\text{H}\) generator. Add \(\text{H}_2\text{O}\) and \(\text{Zn}\). Conduct gas into \(\text{AgNO}_3\) for three minutes

If black ppt. forms in \(\text{AgNO}_3\) dissolve in \(\text{HCl}\) and pass \(\text{H}_2\text{S}\). An orange ppt., \(\text{SbS}_2\), shows

Filter liquid from generator and add few drops to \(\text{HgCl}_2\). A white or gray ppt., \(\text{HgCl}_2\). \(\text{Hg}\), shows

arsenic

antimony

tin

gold

platinum

IV.

Examination of any precipitate produced by \(\text{NH}_4\text{OH}\), after groups 1, 2 and 3 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings. Boil the precipitate with \(\text{NaOH}\) for several minutes and filter.

Filtrate, \(\text{NaAlO}_2\).

Acidulate with \(\text{HCl}\), then add an excess of \((\text{NH}_4)_2\text{CO}_3\) and boil. A white ppt., \(\text{Al(OH)}_3\), shows

Residue—\(\text{Cr(OH)}_3, \text{Fe(OH)}_3\)

Wash with \(\text{H}_2\text{O}\) and drain, discarding the washings. Transfer a part of the residue to a porcelain crucible, add about the same amount of dry \(\text{KNO}_3\) and about twice as much dry \(\text{Na}_2\text{CO}_3\) and ignite until fused. Cool, heat the mass with \(\text{H}_2\text{O}\) and filter.

Filtrate, \(\text{K}_2\text{CrO}_4\).

Acidulate with \(\text{HCl}_2\text{H}_2\text{O}_2\) and add \(\text{Pb(C}_2\text{H}_5\text{O}_2)_2\). A yellow ppt., \(\text{PbCrO}_4\), shows

Residue—\(\text{Fe}_2\text{O}_3\)

Wash on the filter with water, discarding the washings. Dissolve in \(\text{HCl}\) and add \(\text{KCNS}\). A red coloration, \(\text{Fe(CNS)}_3\), shows

iron

If iron is present test separate portions of the original solution as follows:

To one portion add \(\text{HCl}\) and \(\text{K}_2\text{Fe(CN)}_4\). A dark blue ppt., \(\text{Fe}_3\text{(Fe(CN)}_5\text{)}_2\), shows

To another portion add \(\text{HCl}\) and \(\text{KCNS}\). A red coloration, \(\text{Fe(CNS)}_3\), shows

ferrous iron

ferric iron

aluminum

chromium
Examination of any precipitate produced by $H_2S$ in an alkaline liquid, after groups 1, 2, 3 and 4 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings. Pass the same portion of $HCl$ through the filter several times.

<table>
<thead>
<tr>
<th>Filtrate—ZnCl$_2$, MnCl$_2$.</th>
<th>Residue—CoS, NiS.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boil to expel $H_2S$, cool, add excess of NaOH and filter.</td>
<td>If light colored, cobalt and nickel are absent. If dark colored, dissolve in nitro-hydrochloric acid, add an excess of NaOH, collect the ppt. produced on a filter and wash, discarding the washings. Dissolve the ppt. in $HC_2H_3O_2$, add an excess of KCl and of KNO$_2$, let stand for half an hour and filter.</td>
</tr>
<tr>
<td>Filtrate, Na$_2$ZnO$_2$.</td>
<td>Precipitate, K$_2$Co(NO$_3$)$_6$</td>
</tr>
<tr>
<td>Pass $H_2S$. A white ppt. ZnS, shows</td>
<td>yellow, shows</td>
</tr>
<tr>
<td>Boil with conc. $HNO_3$ and PbO$_2$ and let stand. A pink supernatant liquid shows</td>
<td>Filtrate, Ni(C$_2$H$_3$O$_2$)$_2$</td>
</tr>
<tr>
<td>zinc</td>
<td>manganese</td>
</tr>
</tbody>
</table>

VI.

Examination of any precipitate produced by (NH$_4$)$_2$CO$_3$, after groups 1, 2, 3, 4 and 5 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings. Dissolve the washed precipitate in $HC_2H_3O_2$, add an excess of K$_2$Cr$_2$O$_7$ and filter.

<table>
<thead>
<tr>
<th>Precipitate, BaCrO$_4$.</th>
<th>Filtrate—Sr(C$_2$H$_3$O$_2$)$_2$, Ca(C$_2$H$_3$O$_2$)$_2$.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yellow shows</td>
<td>Divide into two portions.</td>
</tr>
<tr>
<td>barium</td>
<td>To one portion add CaSO$_4$, boil and let stand. White ppt. SrSO$_4$, shows</td>
</tr>
<tr>
<td>strontium</td>
<td>To the other portion, add K$_2$SO$_4$, boil, cool and filter, if necessary. To the filtrate add (NH$_4$)$_2$C$_2$O$_4$. White ppt. CaC$_2$O$_4$, shows</td>
</tr>
</tbody>
</table>

calcium

GROUP 7.

Metals not precipitated by any group reagent:

Magnesium, Mg; potassium, K; sodium, Na; lithium, Li; ammonium, (NH$_4$)$_1$.

MAGNESIUM, Mg$^{ii} = 24.32$

Magnesium is a silvery-white metal of low specific gravity which is permanent in dry air but oxidizes in moist air. It burns in the air,
giving an intense white light. It is a constituent of flashlight powders, but is most important in its compounds.

**IMPORTANT COMPOUNDS OF MAGNESIUM.**

Magnesium carbonate, native as magnesite, MgCO₃.
Magnesium sub-carbonate, magnesium carbonate, U. S. P., magnesia alba, (MgCO₃)₄Mg(OH)₂5H₂O.
Magnesium chloride, N.F., MgCl₂6H₂O.
Magnesium hydroxide, Mg(OH)₂.
Magnesium oxide, U. S. P., magnesia, calcined magnesia, MgO.
Heavy magnesium oxide, U. S. P., heavy magnesia, MgO.
Magnesium sulphate, U. S. P., epsom salt, MgSO₄7H₂O.
Magnesium silicates, talcum, asbestos, meerschaum, soapstone; variable in composition.

**TESTS FOR MAGNESIUM.**

Use a separate portion of a solution of a salt of magnesium, as MgCl₂, for each of the following tests:

1. Add (NH₄)₂CO₃ T.S. to the magnesium solution. A white precipitate of magnesium-ammonium carbonate, MgCO₃(NH₄)₂CO₃, is formed when the reagent is in excess. No precipitate will form if excessive amounts of ammonium salts are present.

To a portion of the magnesium solution add an equal volume of NH₄Cl T.S. and then (NH₄)₂CO₃ T.S. Soluble magnesium-ammonium chloride is formed and this is not precipitated by the carbonate.

2. Add Na₂CO₃ T.S. to the magnesium solution. A white precipitate of magnesium sub-carbonate, principally (MgCO₃)₄Mg(OH)₂ is formed. The precipitate is soluble in solutions of ammonium salts and will not form if they are present.

3. Add NH₄OH T.S. to the magnesium solution. Half of the magnesia is precipitated as the white gelatinous magnesium hydroxide, Mg(OH)₂. The other half forms a double salt, as (NH₄)₂MgCl₄, and remains in solution. The precipitate forms soluble double compounds with ammonium salts and no precipitation occurs if an excess of ammonium salts is present in the solution.

4. Add NaOH T.S. to the magnesium solution. A gelatinous white precipitate of magnesium hydroxide, Mg(OH)₂, will form. The precipitate is soluble in ammonium salts and will not form if they are present.

5. To a portion of the magnesium solution add NH₄Cl T.S., NH₄OH T.S., and Na₂HPO₄ T.S. in the order given. A white crystalline precipitate of magnesium-ammonium phosphate, MgNH₄PO₄, will form slowly. NH₄Cl does not take part in the reaction, but is used to prevent precipitation of magnesium hydroxide by NH₄OH.
THE METALS

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. \( \text{MgCl}_2 + (\text{NH}_4)_2\text{CO}_3 = \)
   \( \text{MgSO}_4 + (\text{NH}_4)_2\text{CO}_3 = \)
   \( \text{MgCl}_2 + \text{NH}_4\text{Cl} = \)

2. \( \text{MgCl}_2 + \text{K}_2\text{CO}_3 = \)
   \( \text{MgSO}_4 + \text{Na}_2\text{CO}_3 = \)

3. \( \text{MgBr}_2 + \text{NH}_4\text{OH} = \)
   \( \text{MgSO}_4 + \text{NH}_4\text{OH} = \)

4. \( \text{MgSO}_4 + \text{NaOH} = \)
   \( \text{MgCl}_2 + \text{KOH} = \)

5. \( \text{MgCl}_2 + \text{NH}_4\text{OH} + \text{Na}_2\text{HPO}_4 = \)
   \( \text{MgSO}_4 + \text{NH}_4\text{OH} + (\text{NH}_4)_2\text{HPO}_4 = \)

POTASSIUM (KALIUM), \( K^+ = 39.10 \)

Potassium is the most electropositive of the metals which are found in any considerable quantity. It is a soft white metal which must be kept under kerosene, as it rapidly oxidizes and takes fire in contact with water or when exposed to the air. The element is but little used in the metallic state. The salts of potassium are very important and all of them are more or less soluble in water.

IMPORTANT COMPOUNDS OF POTASSIUM.

Potassium acetate, U. S. P., KC\(_2\)H\(_3\)O\(_2\).
Potassium bicarbonate, U. S. P., saleratus, KHCO\(_3\).
Potassium bitartrate, U. S. P., cream of tartar, KHC\(_4\)H\(_4\)O\(_6\).
Potassium bromate, KBrO\(_3\).
Potassium bromide, U. S. P., KBr.
Potassium carbonate, U.S. P., potash, pearlash, salt of tartar, K\(_2\)CO\(_3\).
Potassium chlorate, U. S. P., KClO\(_3\).
Potassium chloride, N.F., KCl.
Potassium chromate, K\(_2\)CrO\(_4\).
Potassium cyanide, prussiate of potash, KCN.
Potassium dichromate, K\(_2\)Cr\(_2\)O\(_7\).
Potassium ferricyanide, red prussiate of potash, K\(_3\)Fe(CN)\(_6\).
Potassium ferrocyanide, yellow prussiate of potash, K\(_4\)Fe(CN)\(_6\).
Potassium hydroxide, U. S. P., potassium hydrate, potassa, caustic potash, KOH.
Potassium hypophosphite, U. S. P., KPH\(_2\)O\(_2\).
Potassium iodide, U. S. P., KI.
Potassium nitrate, U. S. P., saltpetre, nitre, KNO\(_3\).
Potassium perchlorate, KClO\(_4\).
Potassium permanganate, U. S. P., KMnO\(_4\).
Potassium sulphate, N.F., K\(_2\)SO\(_4\).
Potassium sulphocyanate, potassium thiocyanate, KCNS.
Potassium and sodium tartrate, U. S. P., rochelle salt, KNaC₄H₆O₆·4H₂O.
Sulphuretted potash, U.S.P., liver of sulphur, composition indefinite.

TESTS FOR POTASSIUM.

Use a separate portion of a solution of a potassium salt, as KCl for each of the following tests:

1. Add H₂C₄H₄O₆ T.S. to the potassium solution. A white precipitate of potassium bi-tartrate, KHC₄H₄O₆, will form if the solution is not too dilute. The addition of an equal volume of alcohol increases the delicacy of the test, as the precipitate is less soluble in diluted alcohol than it is in water. The precipitate is soluble in alkalis, and will not form if they are present in the solution until an excess of the reagent has been added. Sodium hydrogen tartrate, NaHC₄H₄O₆, will produce the same precipitate in neutral or acid solutions.

2. Acidulate a portion of the potassium solution with HC₂H₅O₂ T.S., add Na₃Co(NO₂)₆ T.S. and let stand. A yellow precipitate of potassium-cobaltic nitrite, K₃Co(NO₂)₆, will form slowly, if the solution is not too dilute.

3. Heat some of the potassium solution on platinum wire in a blue gas flame and observe the color of the flame, directly and through blue glass. A characteristic violet flame, visible through blue glass, is obtained.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. KCl + H₂C₄H₄O₆ =
K₂SO₄ + NaHC₄H₄O₆ =

2. KC₂H₅O₂ + Na₃Co(NO₂)₆ =
KBr + Na₃Co(NO₂)₆ =

SODIUM (NATRIUM), Na⁺ = 23.0

Sodium is a soft white metal, closely resembling potassium. It decomposes water and rapidly oxidizes in the air, so it must be kept under kerosene or some other liquid which does not contain oxygen. The compounds of sodium are very important and all of them are soluble in water.

IMPORTANT COMPOUNDS OF SODIUM.

Sodium acetate, U. S. P., NaC₂H₃O₂·3H₂O.
Sodium arsenate, U. S. P., Na₂HAsO₄·7H₂O.
Exsiccated sodium arsenate, U. S. P., Na₂HAsO₄.
Sodium bicarbonate, U. S. P., baking soda, NaHCO₃.
Sodium bisulphite, NaHSO₃.
Sodium borate, U. S. P., sodium tetraborate, sodium pyroborate, borax, Na₈B₄O₁₀H₂O.
Sodium bromide, U. S. P., NaBr.
Sodium carbonate, sal soda, washing soda, Na₂CO₃10H₂O.
Monohydrated sodium carbonate, U. S. P., Na₂CO₃H₂O.
Sodium chlorate, NaClO₃.
Sodium chloride, U. S. P., salt, NaCl.
Sodium cyanide, U. S. P., NaCN.
Sodium hydroxide, U. S. P., sodium hydrate, caustic soda, soda, NaOH.
Sodium hypochlorite, NaOCl.
Sodium hypophosphite, U. S. P., NaPH₂O₂H₂O.
Sodium iodide, U. S. P., NaI.
Sodium nitrate, U. S. P., Chili saltpetre, NaNO₃.
Sodium nitrite, U. S. P., NaNO₂.
Sodium oxalate, Na₂C₂O₄.
Sodium perborate, U. S. P., NaBO₃4H₂O.
Sodium peroxide, Na₂O₂.
Tri-sodium phosphate, Na₃P0₄.
Sodium phosphate, U. S. P., di-sodium hydrogen phosphate, Na₂HPO₄12H₂O.
Exsiccated sodium phosphate, U. S. P., Na₂HPO₄.
Sodium di-hydrogen phosphate, NaH₂PO₄.
Sodium pyrophosphate, Na₄P₂O₇10H₂O.
Sodium silicates, etc., water glass, soluble glass, Na₂SiO₃, Na₂SiO₄.
Sodium sulphate, U. S. P., Glauber's salt, Na₂SO₄10H₂O.
Sodium sulphide, Na₂S9H₂O.
Sodium sulphite, Na₂SO₃7H₂O.
Exsiccated sodium sulphite, U. S. P., Na₂SO₃.
Sodium tartrate, Na₂C₄H₄O₆2H₂O.
Sodium thiosulphate, U. S. P., "hyposulphite of soda," Na₂S₂O₅5H₂O.

TEST FOR SODIUM.

As all compounds of sodium are soluble in water to an appreciable extent, there are no dependable precipitation tests for the metal. The flame test is very sensitive, however, if properly applied.

Heat a piece of platinum wire in the blue gas flame until it ceases to impart any color to the flame. Dip the wire in a solution of sodium chloride and heat again, observing the color directly and through blue glass. An intense yellow color is imparted to the flame, but this color is cut off by blue glass.

As sodium is widely distributed in small amounts, it is necessary to
be especially careful in cleaning the platinum wire before applying the flame test for it. Also it is necessary to test the original solution for sodium, in separations, because some of the reagents used contain its salts. The flame test for sodium is so delicate that the metal is often found and reported, when its compounds are present only as impurities in a substance.

**LITHIUM, Li**\(^{+}\) = 6.94

Lithium is a rare metal which is only of importance in a few of its salts, used in medicine. Its compounds are generally colorless and soluble in water.

**IMPORTANT COMPOUNDS OF LITHIUM.**
Lithium bromide, U. S. P., LiBr.
Lithium carbonate, U. S. P., Li\(_2\)CO\(_3\).
Lithium chloride, LiCl.

**TESTS FOR LITHIUM.**
Use a separate portion of a solution of a lithium salt, as LiCl, for each of the following tests:

1. Add Na\(_2\)CO\(_3\) T.S. to the lithium solution. A white precipitate of lithium carbonate, Li\(_2\)CO\(_3\), is formed if the solution is sufficiently concentrated.

2. Add Na\(_3\)HPO\(_4\) T.S. and NaOH T.S. to the lithium solution and boil the mixture. A white precipitate of lithium phosphate, Li\(_3\)PO\(_4\), will form, if the solution is not too dilute.

3. Heat some of the lithium solution on platinum wire in a blue gas flame and observe the color of the flame, directly and through blue glass. The flame is colored a deep red, which is visible through a single piece of blue glass of ordinary thickness, but is not visible through several thicknesses of blue glass.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. LiCl + Na\(_2\)CO\(_3\) =
   LiBr + K\(_2\)CO\(_3\) =

2. LiBr + Na\(_3\)HPO\(_4\) =
   LiCl + (NH\(_4\))\(_2\)HPO\(_4\) =

**AMMONIUM, (NH\(_4\))**\(^{+}\) = 18.042

Ammonium is a positive compound radical which forms a series of salts that are very similar to the salts of potassium and sodium. All ammonium salts are volatilized or decomposed by heat and most of them are soluble in water.
THE METALS

IMPORTANT AMMONIUM COMPOUNDS.

Ammonia, NH₃.
Ammonium acetate, NH₄C₂H₃O₂.
Ammonium bromide, U. S. P., NH₄Br.
Ammonium carbonate, U.S.P., sal volatile, NH₄HCO₃ + NH₄NH₂CO₂.
Ammonium chloride, U. S. P., sal ammoniac, NH₄Cl.
Ammonium hydroxide, NH₄OH (in solution only).
Ammonium hypophosphite, N. F., NH₄PH₂O₂.
Ammonium iodide, U. S. P., NH₄I.
Ammonium nitrate, NH₄NO₃.
Ammonium oxalate, (NH₄)₂C₂O₄H₂O.
Ammonium phosphate, N. F., (NH₄)₂HPO₄ + NH₄H₂PO₄.
Ammonium sulphate, (NH₄)₂SO₄.
Ammonium sulphide, (NH₄)₂S.
Ammonium polysulphide, yellow ammonium sulphide, (NH₄)₂Sₓ.

TESTS FOR AMMONIUM.

Use a separate portion of a solution of an ammonium salt, as NH₄Cl, for each of the following tests:

1. Add NaOH T.S. to the ammonium solution until the reaction is alkaline to litmus, and boil the mixture. Ammonia gas, NH₃, is evolved. Observe the odor of ammonia in the vapor. Moisten a piece of red litmus paper and hold it in the vapor: the color will be changed to blue. Dip a glass rod in concentrated HCl and hold it in the vapor: white fumes of ammonium chloride, NH₄Cl, are formed.

2. Add H₂C₄H₆O₆ T.S. to the ammonium solution. A white precipitate of ammonium bi-tartrate, NH₄HC₄H₆O₆, will be formed, if the solution is not too dilute. The precipitate is soluble in alkalies.

3. Acidulate a portion of the ammonium solution with HC₂H₃O₂ T.S., add Na₃Co(NO₃)₆ T.S. and let stand. A yellow precipitate of ammonium-cobaltic nitrite, (NH₄)₃Co(NO₃)₆, will form slowly.

4. Add alkaline HgK₂I₄ T.S. (Nessler’s reagent) to the ammonium solution. A brown precipitate of nitrogen dimercuric iodide, NHg₂I, is formed if the solution is concentrated. With dilute solutions of ammonium salts this reagent gives a deep brown color, but no precipitate. Several of the metals interfere with this reaction.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. \( \text{NH}_4\text{Cl} + \text{NaOH} = \)
   \( (\text{NH}_4)\text{SO}_4 + \text{KOH} = \)
   \( \text{NH}_3 + \text{HCl} = \)
2. \( \text{NH}_4\text{Br} + \text{H}_2\text{C}_4\text{H}_6\text{O}_6 = \)
   \( \text{NH}_4\text{NO}_3 + \text{NaHC}_2\text{H}_4\text{O}_6 = \)
3. \( \text{NH}_4\text{Cl} + \text{Na}_3\text{Co(NO}_2)_6 = \text{NH}_4\text{NO}_3 + \text{Na}_3\text{Co(NO}_2)_6 \)

4. \( \text{NH}_4\text{Cl} + 2\text{HgK}_2\text{I}_4 + 4\text{KOH} = \text{NH}_2\text{I} + 7\text{KI} + \text{KCl} + 4\text{H}_2\text{O} \)

**SAMPLE NUMBER 12.**

**Analysis of a solution for group 7 of the metals.**

The sample may contain compounds of any of the following metals: magnesium, lithium, sodium, potassium, ammonium.

A. Acidulate about 5 mils of the sample with HCl T.S., evaporate the liquid to a small volume on a water-bath, transfer to a porcelain crucible and ignite gently, to volatilize any ammonium compounds. Dissolve the residue in about 10 mils of water in a beaker, adding a few drops of HCl T.S., to obtain a clear solution, if necessary.

B. Add an excess of NaOH T.S. to the solution obtained in A and boil. If no precipitate is produced, magnesium is absent. A white precipitate, Mg(OH)\(_2\), shows the presence of magnesium. Filter and test the filtrate.

C. To the clear filtrate obtained in B, add Na\(_2\)HPO\(_4\) T.S. and boil. If no precipitate is produced, lithium is absent. A white precipitate, Li\(_3\)PO\(_4\), which may appear as no more than a turbidity in the liquid, shows the presence of lithium.

D. Hold the end of a piece of platinum wire in the blue flame of a gas burner until the flame no longer shows a color from the wire. Dip the wire in the original solution and hold it in the flame again. If the flame is bright yellow when looked at directly but shows no color through blue glass, sodium is present and potassium is absent.

If the flame is violet when looked at directly and through double thick blue glass, potassium is present and sodium is absent.

If the flame is bright yellow when looked at directly and shows a violet color through double thick blue glass, both sodium and potassium are present.

If lithium is present these flame reactions are affected by its deep red flame, but this can be allowed for after experience with the tests described above.

E. To a portion of the original solution add an excess of NaOH T.S. and boil. If no odor of ammonia can be detected and the steam will not turn red litmus paper blue, ammonium is absent. If the steam smells of ammonia, or turns red litmus paper blue, ammonium is present.
SAMPLE NUMBER 13.

Analysis of a solution for all groups of the metals.

Separation of the metals into groups.

The solution should be neutral or acid in reaction to litmus. If alkaline add 
HNO₃ to the portion to be examined for metals until the reaction is acid. Add 
HCl as long as a precipitate is produced and filter.

<table>
<thead>
<tr>
<th>Precipitate</th>
<th>Filtrate</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>Pb, Hg', Ag.</td>
<td>Warm and pass H₂S as long as a precipitate is produced. Filter.</td>
</tr>
<tr>
<td>Examine for metals of group 1 by I.</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>Residue</td>
<td>Precipitate</td>
</tr>
<tr>
<td>-----------</td>
<td>-------------</td>
</tr>
<tr>
<td>Hg', Pb, Bi, Cu, Cd.</td>
<td>Digest with (NH₄)₂S, and filter.</td>
</tr>
<tr>
<td>Examine for metals of group 2 by II.</td>
<td>Examine for metals of group 3 by III.</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>Filtrate</td>
<td>Precipitate</td>
</tr>
<tr>
<td>-----------</td>
<td>-------------</td>
</tr>
<tr>
<td>Zn, Mn, Co, Ni.</td>
<td>Digest with (NH₄)₂CO₃, and filter.</td>
</tr>
<tr>
<td>Examine for metals of group 5 by V.</td>
<td>Examine for metals of group 6 by VI.</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Examination of any precipitate produced by HCl in a neutral or acid solution.

Wash the precipitate on the filter with cold water, discarding the washings. Pass a portion of hot water through the washed precipitate on the filter.

| Filtrate—PbCl₂ | 
|---------------| Add H₂SO₄ and cool. White ppt. PbSO₄, shows |

| Residue—HgCl, AgCl | 
|----------------------| Pass the same portion of NH₄OH through the residue on the filter several times |

<table>
<thead>
<tr>
<th>Residue—NH₄HgCl⁺Hg⁺</th>
<th>Filtrate, (NH₄)⁺(AgCl)₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>black, shows</td>
<td>Add an excess of HNO₃, white ppt., AgCl, shows</td>
</tr>
</tbody>
</table>

lead | mercurosus mercury | silver
II.

Examination of any residue insoluble in \((\text{NH}_4)_2\text{S}_x\) from a precipitate produced by \(\text{H}_2\text{S}\) in an acid liquid, after group 1 of the metals has been removed from a solution.

Wash the residue on the filter, first with an additional portion of \((\text{NH}_4)_2\text{S}_x\) and then with water, and drain, discarding the washings.

Pour a portion of hot \(\text{HNO}_3\) through the filter several times.

**Residue, \(\text{HgS}\)**
- Black. Dissolve in nitrohydrochloric acid, boil to expel \(\text{Cl}\), dilute with water, filter and add \(\text{SnCl}_2\). A white or gray ppt. \(\text{HgCl} \cdot \text{Hg}\) shows

**Filtrate—\(\text{Pb(NO}_3)_2, \text{Bi(NO}_3)_3, \text{Cu(NO}_3)_2, \text{Cd(NO}_3)_2\)**
- Add conc. \(\text{H}_2\text{SO}_4\) and boil until white fumes are given off, dilute with \(\text{H}_2\text{O}\), cool and filter.

**Filtrate—\(\text{Bi}_2(\text{SO}_4)_3, \text{CuSO}_4, \text{CdSO}_4\)**
- Add an excess of \(\text{NH}_4\text{OH}\) and filter.

<table>
<thead>
<tr>
<th>Precipitate, (\text{PbSO}_4) White. Dissolve with (\text{NH}_4\text{Cl}, \text{H}_2\text{O}) and add (\text{K}_2\text{Cr}_2\text{O}_7). Yellow ppt., (\text{PbCrO}_4), shows</th>
<th>Precipitate, (\text{Bi(OH)}_3) White. Add (\text{NaOH}) and (\text{SnCl}_2) on filter. Brown or black coloration, (\text{Bi}_2\text{O}_3), shows</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Filtrate—(\text{Cu(NH}_3)_4\text{SO}_4, \text{Cd(NH}_3)_4\text{SO}_4)</strong></td>
<td><strong>Filtrate—(\text{Cu(NH}_3)_4\text{SO}_4, \text{Cd(NH}_3)_4\text{SO}_4)</strong></td>
</tr>
<tr>
<td>Divide into two portions. To one portion add an excess of (\text{HC}_2\text{H}_3\text{O}_2) and then (\text{K}_4\text{Fe(CN)}_6). A red ppt. or coloration, (\text{Cu}_2\text{Fe(CN)}_6), shows</td>
<td></td>
</tr>
<tr>
<td>Pass (\text{H}_2\text{S}) through the other portion, first decolorizing with (\text{KCN}) if (\text{Cu}) is present. A yellow ppt., (\text{CdS}), shows</td>
<td></td>
</tr>
</tbody>
</table>

| mercuric mercury | lead | bismuth | copper | cadmium |

III.

Examination of the solution in \((\text{NH}_4)_2\text{S}_x\) of any precipitate produced by \(\text{H}_2\text{S}\) in an acid liquid, after group 1 of the metals has been removed from a solution.

Acidulate the \((\text{NH}_4)_2\text{S}_x\) solution with \(\text{HCl}\) and collect any precipitate formed on a filter. Transfer the precipitate to a test tube and digest in \((\text{NH}_4)_2\text{CO}_3\) T.S. Filter.

**Filtrate—\((\text{NH}_4)_2\text{As}_2\)**
- Add excess of \(\text{HCl}\) and pass \(\text{H}_2\text{S}\). A yellow ppt., \(\text{As}_2\text{S}_3\), shows

**Residue—\(\text{Sb}_2\text{S}_3, \text{SnS}_3, \text{AuS}_3, \text{PtS}_2\)**
- Wash with water. Digest in concentrated \(\text{HCl}\). Filter.

**Filtrate—\(\text{SbCl}_3, \text{SnCl}_2\)**
- Place in \(\text{H}\) generator. Add \(\text{H}_2\text{O}\) and \(\text{Zn}\). Conduct gas into \(\text{AgNO}_3\) for three minutes
- If black ppt. forms in \(\text{AgNO}_3\) dissolve in \(\text{HCl}\) and pass \(\text{H}_2\text{S}\). An orange ppt., \(\text{Sb}_2\text{S}_3\), shows
- Filter liquid from generator and add few drops to \(\text{HgCl}_2\). A white or gray ppt., \(\text{HgCl} \cdot \text{Hg}\) shows

**Residue—\(\text{AuS}_3, \text{PtS}_2\)**
- If light colored \(\text{Au}\) and \(\text{Pt}\) are absent. If dark colored, digest in nitrohydrochloric acid. Add \(\text{H}_2\text{O}\) and filter.
- To a part of the filtrate add \(\text{SnCl}_3\) and \(\text{SnCl}_4\) and let stand. A purple ppt., \(\text{AuS}_3\), etc., shows
- Neutralize another part of the filtrate with \(\text{NH}_4\text{OH}\), add alcohol and \(\text{NH}_4\text{Cl}\). A yellow ppt., \(\text{(NH}_4)_2\text{PtCl}_6\), shows

<table>
<thead>
<tr>
<th>arsenic</th>
<th>antimony</th>
<th>tin</th>
<th>gold</th>
<th>platinum</th>
</tr>
</thead>
</table>
IV.

Examination of any precipitate produced by NH₄OH, after groups 1, 2 and 3 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings. Boil the precipitate with NaOH for several minutes and filter.

<table>
<thead>
<tr>
<th>Filtrate, NaAlO₂</th>
<th>Residue—Cr(OH)₂, Fe(OH)₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acidulate with HCl, then add an excess of (NH₄)₂CO₃ and boil. A white ppt., Al(OH)₃ shows</td>
<td>Wash with H₂O and drain, discarding the washings. Transfer a part of the residue to a porcelain crucible cover, add about the same amount of dry KNO₃ and about twice as much dry Na₂CO₃ and ignite until fused. Cool, heat the mass with H₂O and filter.</td>
</tr>
<tr>
<td>aluminum</td>
<td>chromium</td>
</tr>
</tbody>
</table>

Examination of any precipitate produced by H₂S in an alkaline liquid, after groups 1, 2, 3 and 4 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings. Pass the same portion of HCl through the filter several times.

<table>
<thead>
<tr>
<th>Filtrate—ZnCl₂, MnCl₂</th>
<th>Residue—CoS, NiS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boil to expel H₂S, cool, add excess of NaOH and filter.</td>
<td>If light colored, cobalt and nickel are absent. If dark colored, dissolve in nitrohydrochloric acid, add an excess of NaOH, collect the ppt. produced on a filter and wash, discarding the washings. Dissolve the ppt. in HCl₂H₂O₂, add an excess of KCl and of KNO₃, let stand for half an hour and filter.</td>
</tr>
<tr>
<td>Filtrate, Na₂ZnO₂</td>
<td>Precipitate, K₃Co(NO₃)₆</td>
</tr>
<tr>
<td>-------------------</td>
<td>--------------------------</td>
</tr>
<tr>
<td>Pass H₂S. A white ppt. ZnS, shows</td>
<td>yellow, shows</td>
</tr>
<tr>
<td>Zinc</td>
<td>Cobalt</td>
</tr>
</tbody>
</table>

V.

Examination of any precipitate produced by H₂S in an alkaline liquid, after groups 1, 2, 3 and 4 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings. Pass the same portion of HCl through the filter several times.
VI.

Examination of any precipitate produced by \((\text{NH}_4)_2\text{CO}_3\), after groups 1, 2, 3, 4 and 5 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings. Dissolve the washed precipitate in \(\text{HC}_2\text{H}_3\text{O}_2\), add an excess of \(\text{K}_2\text{Cr}_2\text{O}_7\) and filter.

<table>
<thead>
<tr>
<th>Precipitate, (\text{BaCrO}_4)</th>
<th>Filtrate—(\text{Sr(C}_2\text{H}_3\text{O}_2)_2), (\text{Ca(C}_2\text{H}_3\text{O}_2)_2).</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yellow shows barium</td>
<td>To one portion add (\text{CaSO}_4), boil and let stand. White ppt. (\text{SrSO}_4), shows strontium</td>
</tr>
</tbody>
</table>

VII.

Examination of a solution for metals not precipitated with groups 1, 2, 3, 4, 5 or 6.

Add an excess of \(\text{HCl}\) to the filtrate from group 6 and evaporate the liquid to dryness and ignite. Dissolve the residue in water with \(\text{HCl}\). Add an excess of \(\text{NaOH}\) and boil. A white ppt., \(\text{Mg(OH)}_2\), shows magnesium.

Filter out any \(\text{Mg(OH)}_2\), add \(\text{NaH}_2\text{PO}_4\) and boil. A white ppt., \(\text{Li}_2\text{PO}_4\), shows lithium.

Make a flame test of the original solution on platinum wire.

A yellow flame, not visible through blue glass shows sodium.

A violet flame, visible through double thick blue glass shows potassium.

Add an excess of \(\text{NaOH}\) to a portion of the original solution and boil. An alkaline vapor, \(\text{NH}_3\), having the odor of ammonia, shows ammonium.
The acid radicals cannot be separated from each other by grouping as is done with the metals, and they must often be tested for separately in the original sample. The reactions of the soluble salts of the most important acids are given here. Some others will be noticed later when the preliminary treatment of solids is given.

Three groups of the acids are recognized, as follows:

Group A.—Acids whose radicals are precipitated by silver nitrate, in the presence of nitric acid; hydrochloric acid, HCl; hydrobromic acid, HBr; hydriodic acid, HI; hydrocyanic acid, HCN; hydrosulphuric acid, H₂S; and others.

Group B.—Acids whose radicals are precipitated by barium chloride, from a neutral solution: sulphuric acid, H₂SO₄; boric acid, H₃BO₃; sulphurous acid, H₂SO₃; carbonic acid, H₂CO₃; oxalic acid, H₂C₂O₄; phosphoric acid, H₃PO₄; and others.

Group C.—Acids whose radicals are not precipitated in groups A and B: acetic acid, H₄C₂H₅O₂; nitric acid, HNO₃; and others.

**GROUP A.**

Acids whose radicals are precipitated as silver salts by silver nitrate in the presence of nitric acid.

The principal members of this group are hydrochloric acid, HCl; hydrobromic acid, HBr; hydriodic acid, HI; hydrocyanic acid, HCN; and hydrosulphuric acid, H₂S.

**HYDROCHLORIC ACID, HCl.**

Muriatic acid. Spirit of salt.

Hydrochloric acid, U. S. P., 31–33%.

Diluted Hydrochloric acid, U. S. P., 9.5–10.5%.

Hydrochloric acid is a colorless gas with a sharp suffocating odor, which is exceedingly soluble in water, and the name is generally applied to the solution. The acid and its solution are irritant or corrosive poisons when inhaled or swallowed. It is monobasic and its salts are called chlorides, formerly muriates. The whole acid combines with vegetable alkaloids and such salts are called hydrochlorides or hydrochlorates.
The chlorides are generally colorless and most of them are soluble in water. Silver chloride and mercurous chloride are insoluble, and lead chloride is only very slightly soluble; also the basic chlorides of bismuth, antimony and tin are insoluble in water.

**IMPORTANT SALTS OF HYDROCHLORIC ACID.**

Ammonium chloride, U. S. P., sal ammoniac, NH₄Cl.
Barium chloride, BaCl₂H₂O.
Calcium chloride, U. S. P., CaCl₂2H₂O.
Ferric chloride, U. S. P., FeCl₃6H₂O.
Gold chloride, AuCl₃.
Magnesium chloride, MgCl₂6H₂O.
Mercuric chloride, corrosive mercuric chloride, U. S. P., corrosive sublimate, HgCl₂.
Mercurous chloride, mild mercurous chloride, U. S. P., calomel, HgCl₁.
Platinum chloride, PtCl₄.
Potassium chloride, KCl.
Sodium chloride, U. S. P., salt, NaCl.
Stannic chloride, SnCl₄.
Stannous chloride, SnCl₂2H₂O.
Zinc chloride, U. S. P., ZnCl₂.

**TESTS FOR SALTS OF HYDROCHLORIC ACID.**

Use separate portions of a solution of a soluble chloride, as NaCl, for each of the following tests:

1. Add AgNO₃ T.S. to the chloride solution. A curdy white precipitate of silver chloride, AgCl, is formed. The precipitate soon darkens upon exposure to light and is insoluble in acids, but is readily soluble in ammonia water.

2. Add Pb(C₂H₂O₂)₂ T.S. to the chloride solution. A white precipitate of lead chloride, PbCl₂, is formed if the solution is not too dilute. The precipitate is slightly soluble in cold water, and more soluble in hot water.

3. Evaporate a portion of the chloride solution to dryness, or nearly so, add concentrated H₂SO₄ and heat. HCl gas will be given off, which can be recognized by its odor and by the white fumes of NH₄Cl if it forms if a glass rod or stopper wet with NH₄OH T.S. is held in it.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. NaCl⁻ + AgNO₃ = 
   HCl + AgNO₃ =
   CaCl₂ + AgNO₃ =
2. \[ \text{KCl} + \text{Pb}((\text{C}_2\text{H}_3\text{O}_2)_2) = \]
\[ \text{HCl} + \text{Pb(NO}_3)_2 = \]
\[ \text{BaCl}_2 + \text{Pb}((\text{C}_2\text{H}_3\text{O}_2)_2) = \]

3. \[ \text{NaCl} + \text{H}_2\text{SO}_4 = \]
\[ \text{MgCl}_2 + \text{H}_2\text{SO}_4 = \]
\[ \text{HCl} + \text{NH}_3 = \]

**HYDROBROMIC ACID, HBr.**

Diluted hydrobromic acid, U. S. P., 9.5–10.5%.

Hydrobromic acid is a gas which resembles hydrochloric acid, but is less stable than that acid. Its salts are called bromides.

The bromides are generally colorless and soluble in water. Silver bromide and mercurous bromide are insoluble and lead bromide is only slightly soluble. All bromides are decomposed by chlorine and bromine is set free.

**IMPORTANT SALTS OF HYDROBROMIC ACID.**

Ammonium bromide, U. S. P., \(\text{NH}_4\text{Br}\).
Calcium bromide, U. S. P., \(\text{CaBr}_2\text{H}_2\text{O}\).
Lithium bromide, U. S. P., \(\text{LiBr}\).
Potassium bromide, U. S. P., \(\text{KBr}\).
Silver bromide, \(\text{AgBr}\).
Sodium bromide, U. S. P., \(\text{NaBr}\).
Strontium bromide, U. S. P., \(\text{SrBr}_2\).

**TESTS FOR SALTS OF HYDROBROMIC ACID.**

Use a separate portion of a solution of a bromide, as \(\text{KBr}\), for each of the following tests:

1. Add \(\text{AgNO}_3\) T.S. to the bromide solution. A yellow-white precipitate of silver bromide, \(\text{AgBr}\), will be formed, which slowly darkens upon exposure to light. The precipitate is insoluble in nitric acid, but is slowly soluble in ammonia water.

2. Add \(\text{Pb}((\text{C}_2\text{H}_3\text{O}_2)_2)\) T.S. to the bromide solution. A white precipitate of lead bromide, \(\text{PbBr}_2\), will be formed, which is slightly soluble in water.

3. To a few drops of the bromide solution add a few drops of carbon disulphide, \(\text{CS}_2\), and slowly add chlorine T.S. Bromine will be set free and will dissolve in the \(\text{CS}_2\) to make a brown solution. The color will not disappear when the chlorine is in excess.
COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. \( \text{KBr} + \text{AgNO}_3 = \text{CaBr}_2 + \text{AgNO}_3 \)  
2. \( \text{NaBr} + \text{Pb(C}_2\text{H}_5\text{O}_2)_2 = \text{NH}_4\text{Br} + \text{Pb(NO}_3)_2 \)  
3. \( \text{KBr} + \text{Cl}_2 = \text{CaBr}_2 + \text{Cl}_2 \)

HYDRIODIC ACID, HI.

Diluted hydriodic acid, U. S. P., 9.5–10.5 %.

Hydriodic acid is a heavy colorless gas, which is readily soluble in water. It is unstable and readily reacts with oxygen, forming water and setting iodine free when oxidized. The salts of hydriodic acid are called iodides.

Silver iodide, mercurous iodide, mercuric iodide and lead iodide are insoluble in water. The iodides of other important metals are soluble. All iodides are decomposed by chlorine or bromine and iodine is set free.

IMPORTANT SALTS OF HYDRIODIC ACID.

Ammonium iodide, U. S. P., NH\(_4\)I.
Arsenous iodide, U. S. P., AsI\(_3\).
Ferrous iodide, FeI\(_2\).
Lead iodide, N.F., PbI\(_2\).
Mercuric iodide, red mercuric iodide, U. S. P., biniodide of mercury, HgI\(_2\).
Mercurous iodide, yellow mercurous iodide, U. S. P., protoiodide of mercury, HgI.
Potassium iodide, U. S. P., KI.
Silver iodide, AgI.
Sodium iodide, U. S. P., NaI.
Strontium iodide, U. S. P., SrI.
Zinc iodide, ZnI\(_2\).

TESTS FOR SALTS OF HYDRIODIC ACID.

Use a separate portion of a solution of an iodide, as KI, for each of the following tests:

1. Add AgNO\(_3\) T.S. to the iodide solution. A yellow precipitate of silver iodide, AgI, will be obtained. The precipitate slowly blackens in the light and is insoluble in HNO\(_3\) or NH\(_4\)OH.
2. Add Pb(C\(_2\)H\(_5\)O\(_2\))\(_2\) T.S. to the iodide solution. A yellow precipitate of lead iodide, PbI\(_2\), will be obtained.
3. To a few drops of the iodide solution add about the same
amount of CS₂, and slowly add chlorine T.S. Iodine will be set free and will dissolve in the CS₂ to make a pink solution. When the chlorine is in excess the iodine will be oxidized to iodic acid, HIO₃, and the solution will be decolorized.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. \( \text{KI} + \text{AgNO}_3 = \text{NH}_4\text{I} + \text{AgC}_2\text{H}_5\text{O}_2 = \)
2. \( \text{NaI} + \text{Pb(C}_2\text{H}_3\text{O}_2\text{)}_2 = \text{ZnI}_2 + \text{Pb(NO}_3\text{)}_2 = \)
3. \( \text{KI} + \text{Cl}_2 = \text{CaI}_2 + \text{Cl}_2 = \text{I}_2 + \text{Cl}_2 + \text{H}_2\text{O} = \text{HIO}_3 + \)

**HYDROCYANIC ACID, HCN.**

Prussic acid.

Diluted hydrocyanic acid, U. S. P., 1.9–2.1%.

Hydrocyanic acid is a colorless liquid with an odor resembling that of bitter almonds. It is soluble in water, but the solution slowly decomposes through the influence of light and air. The salts of hydrocyanic acid are called cyanides, formerly prussiates.

The cyanides of the alkali metals, the alkaline earth metals, and mercuric cyanide are soluble in water. The cyanides of other important metals are insoluble. The acid itself and all of its salts are deadly poisons, especially dangerous because their action is so rapid that there is little time for the administration of antidotes. Care should be taken not to inhale the gas evolved when an acid is added to a cyanide.

**IMPORTANT SALTS OF HYDROCYANIC ACID.**

Mercuric cyanide, Hg(CN)₂.
Potassium cyanide, KCN.
Silver cyanide, AgCN.
Sodium cyanide, U. S. P., NaCN.

**TESTS FOR SALTS OF HYDROCYANIC ACID.**

Use a separate portion of a solution of a cyanide, as KCN, for each of the following tests:

1. Slowly add AgNO₃ T.S. to the cyanide solution. A white precipitate of silver cyanide, AgCN, will be formed when the AgNO₃ is in excess. Add an excess of NH₄OH T.S. The precipitate will
form a double compound and dissolve. Silver cyanide is insoluble in diluted HNO₃.

2. To about 2 mils of the cyanide solution in a test-tube, add about the same amount of each of FeSO₄ T.S. and KOH T.S., and warm, when potassium ferrocyanide K₄Fe(CN)₆ will be formed. Then add two drops of FeCl₃ T.S. and acidulate with HCl T.S. A blue precipitate of ferric ferrocyanide, Fe₄(Fe(CN)₆)₃, will be formed.

3. To about 2 mils of the cyanide solution in a porcelain evaporating dish, add 2 drops of yellow ammonium sulphide and heat on a water-bath until the liquid is colorless, potassium sulphocyanate, KCNS, being formed. Then acidulate with HCl T.S. and add a drop of FeCl₃ T.S. A red coloration, due to ferric sulphocyanate, Fe(CNS)₃, will be produced.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. KCN + AgNO₃ =
   HCN + AgNO₃ =

2. 6KCN + FeSO₄ = K₄Fe(CN)₆ + K₂SO₄
   FeCl₃ + K₄Fe(CN)₆ =

3. KCN + (NH₄)₂S₂ = KCNS + 2NH₃ + H₂S
   FeCl₃ + KCNS =

**HYDROSULPHURIC ACID, H₂S**

Hydrogen sulphide. Sulphuretted hydrogen.

Hydrosulphuric acid is a colorless gas having a characteristic disagreeable odor; it is moderately soluble in water. The gas in solution readily oxidizes and deposits sulphur. The salts of hydrosulphuric acid are called sulphides, formerly sulphurets. Many metals readily combine with additional atoms of sulphur to form polysulphides, which yield hydrogen sulphide and sulphur on decomposition.

The sulphides of the alkali metals—potassium, sodium, lithium and ammonium; and of the alkaline earth metals—magnesium, calcium, strontium and barium; are soluble in water. The sulphides of the other important metals are insoluble.

**IMPORTANT SALTS OF HYDROSULPHURIC ACID.**

Ammonium sulphide, colorless ammonium sulphide, (NH₄)₂S.

Ammonium polysulphide, yellow ammonium sulphide, (NH₄)₂Sₓ.

Antimonous sulphide, SbₓS₃.

Barium sulphide, BaS.

Calcium sulphide, CaS.
Ferrous sulphide, FeS.
Lead sulphide, galena, PbS.
Mercuric sulphide, cinnabar, vermilion, HgS.
Sodium sulphide, Na₂S.
Zinc sulphide, ZnS.

**TESTS FOR SALTS OF HYDROSPHURIC ACID.**

Use a separate portion of a solution of a sulphide, as Na₂S, for each of the following tests:

1. Add AgNO₃ T.S. to the sulphide solution. A black precipitate of silver sulphide, Ag₂S, will be obtained, which is insoluble in diluted acids.
2. Add Pb(C₂H₃O₂)₂ T.S. to the sulphide solution. A black precipitate of lead sulphide, PbS, will be obtained, which is insoluble in diluted acids.
3. To a few drops of the sulphide solution add H₂SO₄ T.S., and warm. Hold a piece of filter paper moistened with Pb(C₂H₃O₂)₂ T.S. in the gas evolved, and observe the odor. Lead sulphide, PbS will be formed and will blacken the paper, and the odor of H₂S will be observed.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. Na₂S + AgNO₃ = CaS + AgNO₃ =
2. Na₂S + Pb(C₂H₃O₂)₂ = (NH₄)₂S + Pb(NO₃)₂ =
3. K₂S + H₂SO₄ = BaS + HCl =
   H₂S + Pb(C₂H₃O₂)₂ =

**SAMPLE NUMBER 14.**

Analysis of a solution for the important metals and for group A of the acids.

The sample may contain chlorides, bromides, iodides, cyanides or sulphides of any of the important metals.

A. Examine a portion of the sample for metals by the tables on pages 82–85.

B. To about 5 mils of the sample add H₂SO₄ T.S. and heat, observing the odor of the vapor given off. Hold a piece of lead acetate test-paper in the vapor. If the test-paper is not blackened, sulphides are absent. If the odor of H₂S is perceived and the test-paper is blackened, sulphides are present.
C. To about 3 mils of the sample add a small crystal of FeSO₄ and an excess of NaOH T.S. and heat. Then add two drops of FeCl₃ T.S. and acidulate with HCl. If no blue precipitate is obtained, cyanides are absent. A blue precipitate, Fe₄(Fe(CN)₆)₃, shows the presence of cyanides.

D. To a few drops of the sample, add about the same amount of CS₂ and then slowly add chlorine T.S. until the chlorine is in excess. If the CS₂ is not colored, iodides and bromides are absent. If the CS₂ is colored pink at first and becomes colorless or brown when the chlorine is in excess, iodides are present. If the CS₂ is colored brown when the chlorine is in excess, bromides are present.

E. If sulphides, cyanides, iodides and bromides are absent, acidulate a small portion of the sample with HNO₃ T.S. and add AgNO₃ T.S. If no precipitate is obtained, chlorides are absent. A white precipitate which readily dissolves in NH₄OH T.S. shows the presence of chlorides.

If sulphides or cyanides are present, add an excess of HNO₃ T.S. to a small portion of the sample and boil, under a hood, to expel H₂S or HCN. Then add AgNO₃ T.S. A white precipitate shows the presence of chlorides.

If bromides or iodides are present, add HNO₃ T.S. to about 5 mils of the sample and boil if necessary to expel H₂S or HCN. Add an excess of AgNO₃ T.S. and filter. Pass about 2 mils of NH₄OH T.S. once through the precipitate on the filter. Any silver chloride quickly dissolves as ammonio-silver chloride. Acidulate the filtrate with HNO₃. A white precipitate shows the presence of chlorides. This test effectually separates chlorides from iodides, but the separation from bromides is not complete, as silver bromide slowly dissolves in ammonia water. To completely separate chlorides from bromides is difficult of accomplishment, and the methods used are beyond the scope of this manual.

GROUP B.

Acids whose radicals are precipitated from a neutral solution as barium salts by barium chloride or barium nitrate.

The principal members of this group are sulphuric acid, H₂SO₄; sulphurous acid, H₂SO₃; carbonic acid, H₂CO₃; oxalic acid, H₂C₂O₄; and phosphoric acid, H₃PO₄.

SULPHURIC ACID, H₂SO₄.

Oil of vitrol.

Sulphuric acid, U.S.P., 93–95%. Diluted sulphuric acid, U.S.P. 9.5–10.5%.

Sulphuric acid is a heavy, colorless and odorless liquid which is hygroscopic and non-volatile at ordinary temperatures, but may be
distilled unchanged. The concentrated acid attacks and destroys organic matter and inflicts serious burns if it comes in contact with the skin. It has a strong affinity for water and much heat is generated when the strong acid and water are mixed. On this account, the acid should always be poured into the water, when they are being mixed, so that the rise in temperature will be gradual.

Sulphuric acid, being dibasic, forms normal salts called sulphates, and acid salts called bisulphates. The sulphates and bisulphates are generally colorless and soluble in water. Lead sulphate, barium sulphate and strontium sulphate are insoluble, and mercurous sulphate and calcium sulphate are but slightly soluble.

**IMPORTANT SALTS OF SULPHURIC ACID.**

Aluminum and ammonium sulphate, ammonia alum, $\text{AlNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

Aluminum and potassium sulphate, potash alum, $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

Aluminium sulphate, $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$.

Ammonium sulphate, $(\text{NH}_4)_2\text{SO}_4$.

Barium sulphate, heavy spar, permanent white, $\text{BaSO}_4$.

Cadmium sulphate, $\text{CdSO}_4$.

Calcium sulphate, gypsum, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$.

Calcium sulphate, anhydrous, plaster of Paris, $\text{CaSO}_4$.

Chromium potassium sulphate, chrome alum, $\text{CrK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

Cobalt sulphate, $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$.

Copper sulphate, blue vitriol, blue stone, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.

Ferric ammonium sulphate, ferric alum, $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

Ferric subsulphate, Monsel's salt, $\text{Fe}_2\text{O}(\text{SO}_4)_3$.

Ferric sulphate, iron tersulphate, $\text{Fe}_2(\text{SO}_4)_3$.

Ferrous sulphate, green vitriol, copperas, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$.

Magnesium sulphate, Epsom salt, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$.

Manganous sulphate, $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$.

Mercuric sulphate, $\text{HgSO}_4$.

Mercurous sulphate, $\text{Hg}_2\text{SO}_4$.

Nickel sulphate, $\text{NiSO}_4$.

Potassium sulphate, $\text{K}_2\text{SO}_4$.

Silver sulphate, $\text{Ag}_2\text{SO}_4$.

Sodium sulphate, Glauber's salt, $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$.

Strontium sulphate, $\text{SrSO}_4$.

Zinc sulphate, white vitriol, $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$.

**TESTS FOR SALTS OF SULPHURIC ACID.**

Use a separate portion of a solution of a sulphate, as $\text{Na}_2\text{SO}_4$, for each of the following tests:

1. Add $\text{BaCl}_2$ T.S. to the sulphate solution. A finely divided
white precipitate of barium sulphate, BaSO₄, will be obtained, which is insoluble in HCl or HNO₃.

2. Add Pb(C₂H₃O₂)₂ T.S. to the sulphate solution. A white precipitate of lead sulphate, PbSO₄, will be obtained, which is insoluble in acids.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. \( \text{Na}_2\text{SO}_₄ + \text{BaCl}_₂ = \)
\( \text{MgSO}_₄ + \text{Ba(NO)}₃₂ = \)

2. \( \text{K}_₂\text{SO}_₄ + \text{Pb(C}_₂\text{H}_₃\text{O}_₂)₂ = \)
\( \text{ZnSO}_₄ + \text{Pb(NO)}₃₂ = \)

**SULPHUROUS ACID, \( \text{H}_₂\text{SO}_₃ \).**

Sulphurous acid is formed when its anhydride, SO₂, is dissolved in water, but the acid is so unstable that it cannot be isolated.

The acid, being dibasic, forms normal salts, called sulphites, and acid salts, called bisulphites. The sulphites and bisulphites of the alkali metals and the bisulphites of the alkaline earth metals are soluble in water. The other metals do not generally form bisulphites and their sulphites are insoluble. Sulphites and bisulphites are decomposed by most acids, the acid anhydride, sulphur dioxide, SO₂, being generated.

**IMPORTANT SALTS OF SULPHUROUS ACID.**

Calcium sulphite, CaSO₄·2H₂O.
Calcinium bisulphite, bisulphite of lime, Ca(HSO₃)₂.
Sodium sulphite, Na₂SO₃·7H₂O.
Exsiccated sodium sulphite, U. S. P., Na₂SO₃.
Sodium bisulphite, NaHSO₃.

**TESTS FOR SALTS OF SULPHUROUS ACID.**

Use a separate portion of a solution of a sulphite, as Na₂SO₃, for each of the following tests:

1. Add BaCl₂ T.S. to the sulphite solution, followed by an excess of HCl and then by a few drops of nitrohydrochloric acid. A white precipitate of barium sulphite, BaSO₄, will be obtained, which will dissolve in the HCl; and a precipitate of barium sulphate, BaSO₄, will be formed upon adding the nitrohydrochloric acid.

2. Add H₂SO₄ T.S. to a small quantity of the sulphite solution and heat the liquid, observing the odor of the gas evolved. The characteristic choking odor is that of sulphur dioxide, SO₂. The bleaching effect of the gas on organic coloring matter may also be obtained, most easily by holding a colored flower, as a violet, in the gas.
COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. \( \text{Na}_2\text{SO}_3 + \text{BaCl}_2 = \text{Ca(HSO}_3)_2 + \text{Ba(NO}_3)_2 \)
2. \( \text{Na}_2\text{SO}_3 + \text{H}_2\text{SO}_4 = \text{NaHSO}_3 + \text{HCl} = \)

CARBONIC ACID \( \text{H}_2\text{CO}_3 \).

Carbonic acid appears to be formed when carbon dioxide is dissolved in water, as in "soda water." The compound, however, is so unstable that it cannot be isolated. The acid forms normal salts called carbonates, and acid salts called bicarbonates. Carbonates and bicarbonates are decomposed by nearly all other acids, yielding water and carbon dioxide. The carbonates of the alkali metals are soluble in water. All other carbonates are insoluble in water, though several of them will form unstable bicarbonates and dissolve in water containing carbon dioxide in solution.

IMPORTANT SALTS OF CARBONIC ACID.

Ammonium carbonate, U. S. P., ammonium bicarbonate + ammonium carbamate, \( \text{NH}_4\text{HCO}_3 + \text{NH}_4\text{NH}_2\text{CO}_2 \).
Calcium carbonate, limestone, marble, chalk, prepared chalk, U. S. P., precipitated calcium carbonate, U. S. P., \( \text{CaCO}_3 \).
Ferrous carbonate, \( \text{FeCO}_3 \).
Lead sub-carbonate, white lead, \( (\text{PbCO}_3)_2\text{Pb(OH)}_2 \).
Lithium carbonate, U. S. P., \( \text{Li}_2\text{CO}_3 \).
Magnesium carbonate, magnesite, \( \text{MgCO}_3 \).
Magnesium carbonate, U. S. P., magnesia alba, \( (\text{MgCO}_3)_4\text{Mg(OH)}_2 \cdot 5\text{H}_2\text{O} \).
Potassium carbonate, U. S. P., potash, salt of tartar, \( \text{K}_2\text{CO}_3 \).
Potassium bicarbonate, U. S. P., saleratus, \( \text{KHCO}_3 \).
Sodium carbonate, soda, washing soda, \( \text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O} \).
Monohydrated sodium carbonate, U. S. P., \( \text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O} \).
Sodium bicarbonate, U. S. P., baking soda, \( \text{NaHCO}_3 \).
Zinc carbonate, calamine, \( \text{ZnCO}_3 \).

TESTS FOR SALTS OF CARBONIC ACID.

Use a separate portion of a solution of a carbonate, as \( \text{Na}_2\text{CO}_3 \), for each of the following tests:
1. Add \( \text{BaCl}_2 \) T.S. to the carbonate solution. A white precipitate of barium carbonate, \( \text{BaCO}_3 \), will be formed, which is soluble in acids.
2. Add \( \text{CaCl}_2 \) T.S. to the carbonate solution. A white precipitate of calcium carbonate, \( \text{CaCO}_3 \), will be formed, which is soluble in acids.
3. To a portion of the carbonate solution contained in a test-tube provided with a delivery tube, add H₂SO₄ T.S. and conduct the gas generated into lime water, Ca(OH)₂. The carbonate will be decomposed, yielding carbon dioxide, CO₂, and this will precipitate calcium carbonate, CaCO₃, from the lime water.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. Na₂CO₃ + BaCl₂ =
   KHCO₃ + Ba(NO₃)₂ =

2. K₂CO₃ + CaCl₂ =
   (NH₄)₂CO₃ + CaSO₄ =

3. Na₂CO₃ + H₂SO₄ =
   NaHCO₃ + HCL =
   Ca(OH)₂ + CO₂ =

**SAMPLE NUMBER 15.**

**Analysis of a solution for the important metals and for the radicals of sulphuric, sulphurous and carbonic acids.**

The sample may contain any of the important metals, as sulphates, sulphites or carbonates.

A. Examine a portion of the sample for metals by the tables on pages 82–85.

B. To a portion of the sample, add HCl until the reaction is acid and then add BaCl₂ T.S. If no precipitate is produced sulphates are absent. A white precipitate shows the presence of sulphates.

C. To a portion of the sample, contained in a test-tube provided with a delivery tube, add H₂SO₄ T.S., and heat. If a gas is evolved, observe its odor and pass it through lime water. If there is no effervescence of an odororous gas and the lime water is not rendered turbid, sulphites and carbonates are absent. If there is an effervescence of a gas having the odor of burning sulphur, sulphites are present. If there is an effervescence of an odorless gas which renders lime water turbid, carbonates are present. If the odor of burning sulphur is observed and the lime water is rendered turbid, both sulphites and carbonates are present.

**OXALIC ACID, H₂C₂O₄·2H₂O.**

Oxalic acid is an organic acid, whose salts are frequently used as reagents. It is a soluble crystalline solid. The acid is dibasic and forms normal and acid salts, called oxalates, which are generally colorless. The acid and its salts are poisonous. The oxalates of the alkali metals and of chromium are soluble in water. The
oxalates of all other important metals are insoluble in water, but will dissolve in mineral acids, if the metals present form soluble salts with the acids.

**IMPORTANT SALTS OF OXALIC ACID.**

Ammonium oxalate, \((\text{NH}_4)_2\text{C}_2\text{O}_4\cdot\text{H}_2\text{O}\).
Calcium oxalate, \(\text{CaC}_2\text{O}_4\cdot\text{H}_2\text{O}\).
Cerium oxalate, \(\text{Ce}_2(\text{C}_2\text{O}_4)_3\cdot10\text{H}_2\text{O}\).
Potassium acid oxalate, salt of lemon, \(\text{KHC}_2\text{O}_4\cdot2\text{H}_2\text{O}\).
Sodium oxalate, \(\text{Na}_2\text{C}_2\text{O}_4\).

**TESTS FOR SALTS OF OXALIC ACID.**

Use a separate portion of a solution of an oxalate, as \((\text{NH}_4)_2\text{C}_2\text{O}_4\), for each of the following tests:

1. Add \(\text{BaCl}_2\) T.S. to the oxalate solution. A white precipitate of barium oxalate, \(\text{BaC}_2\text{O}_4\), will be formed; the precipitate is soluble in acids which form soluble salts with barium, including acetic acid.
2. Add \(\text{CaCl}_2\) T.S. to the oxalate solution. A white precipitate of calcium oxalate, \(\text{CaC}_2\text{O}_4\), will be formed, which is soluble in mineral acids but not in acetic acid.

**COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**

1. \((\text{NH}_4)_2\text{C}_2\text{O}_4 + \text{BaCl}_2 = \text{Na}_2\text{C}_2\text{O}_4 + \text{Ba(NO}_3)_2\)
2. \(\text{K}_2\text{C}_2\text{O}_4 + \text{CaCl}_2 = \text{KHC}_2\text{O}_4 + \text{CaSO}_4\)

**PHOSPHORIC ACID, \(\text{H}_2\text{PO}_4\).**

Phosphoric acid, U.S.P., 85-88%. Dilute phosphoric acid, U.S.P., 9.5-10.5%.

Orthophosphoric acid.

Orthophosphoric acid is a colorless deliquescent crystalline solid commonly seen as a syrupy solution in water and whose salts are the phosphates. It is not volatile but when heated loses water and changes, first to pyrophosphoric acid, \(\text{H}_4\text{P}_2\text{O}_7\), and then to metaphosphoric acid, \(\text{HPO}_3\).

Orthophosphoric acid is tri-basic and forms normal salts and two series of acid salts. Salts in which only two of the three hydrogen atoms are replaced by metals, as \(\text{Na}_2\text{HPO}_4\), are common, and are frequently called phosphates. The phosphates of the alkali metals and the acid phosphates of a few other metals are soluble in water. All other phosphates are insoluble.
IMPORTANT SALTS OF PHOSPHORIC ACID.

Ammonium phosphate, \((\text{NH}_4)_2\text{HPO}_4\).
Calcium phosphate, \(\text{Ca}_3(\text{PO}_4)_2\).
Ferric phosphate, \(\text{FePO}_4\).
Sodium phosphate, U. S. P., \(\text{Na}_2\text{HPO}_4\cdot 12\text{H}_2\text{O}\).

TESTS FOR SALTS OF PHOSPHORIC ACID.

Use a separate portion of a solution of a phosphate, as \(\text{Na}_2\text{HPO}_4\), for each of the following tests:

1. Add \(\text{BaCl}_2\) T.S. to the phosphate solution. A white precipitate of barium phosphate, \(\text{BaHPO}_4\), will form, which is soluble in acids.

2. To the phosphate solution add \(\text{CaCl}_2\) T.S., followed by an excess of \(\text{HC}_2\text{H}_3\text{O}_2\) T.S. A white precipitate of calcium phosphate, \(\text{CaHPO}_4\), will be obtained, which will dissolve in the acetic acid.

3. To the phosphate solution add \(\text{NH}_4\text{Cl}\) T.S., \(\text{NH}_4\text{OH}\) T.S. and \(\text{MgSO}_4\) T.S. in the order given. A white precipitate of magnesium ammonium phosphate, \(\text{MgNH}_4\text{PO}_4\), will form, which is soluble in acids.

4. To about 2 mils of \((\text{NH}_4)_2\text{MoO}_4\) T.S. add a few drops of the phosphate solution and let stand. A yellow precipitate of ammonium phosphomolybdate, \((\text{NH}_4)_3\text{PO}_4(\text{MoO}_3)_{12}\), will form slowly. The precipitate is soluble in alkalis.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. \(\text{Na}_2\text{HPO}_4 + \text{BaCl}_2 = (\text{NH}_4)_2\text{HPO}_4 + \text{Ba(NO}_3)_2\)

2. \(\text{K}_2\text{HPO}_4 + \text{CaCl}_2 = \text{CaHPO}_4 + \text{HC}_2\text{H}_3\text{O}_2 = \text{Ca(H}_2\text{PO}_4)_2 + \)

3. \(\text{NaHPO}_4 + \text{NH}_4\text{OH} + \text{MgSO}_4 = (\text{NH}_4)_2\text{HPO}_4 + \text{MgCl}_2\)

4. \(\text{Na}_2\text{HPO}_4 + 12(\text{NH}_4)_2\text{MoO}_4 + 23\text{HNO}_3 = (\text{NH}_4)_3\text{PO}_4(\text{MoO}_3)_{12} + 21\text{NH}_4\text{NO}_3 + 2\text{NaNO}_3 + 12\text{H}_2\text{O}\)

SAMPLE NUMBER 16.

Analysis of a solution for the principal metals and for the radicals of oxalic and phosphoric acids.

The sample may contain any of the important metals as oxalates or phosphates.

A. Add \(\text{NaOH}\) T.S. to a portion of the sample until the reaction is alkaline, then \(\text{HC}_2\text{H}_3\text{O}_2\) T.S. until the reaction is acid, then \(\text{CaCl}_2\) T.S. and let stand. If no precipitate is produced oxalic acid is
absent. A white precipitate, which may be formed slowly, shows the presence of oxalates.

B. To about 3 mils of (NH₄)₂MoO₄ T.S. in a test-tube add a few drops of the sample and let stand. If no precipitate is formed phosphates are absent. A yellow precipitate, slowly formed, shows the presence of phosphates.

About 10 mils of the sample should be examined for the metals by the tables on pages 82–85, with the following changes if oxalates or phosphates, or both, are present.

C. If oxalates are present evaporate the filtrate from the second group to a small volume, transfer to a porcelain crucible, add concentrated HNO₃ and gently ignite, which treatment will destroy oxalic acid. Then dissolve the residue in HCl T.S. and proceed with the examination for group 3 of the metals.

D. If phosphates are present concentrate the filtrate from the second group of metals to about 5 mils, or use the solution obtained in C after destroying oxalic acid, if present. Mix with three times its volume of (NH₄)₂MoO₄ T.S., warm the mixture and let it stand for some time. Filter and test the filtrate. Evaporate the filtrate nearly to dryness to expel nitric acid. Dilute with about 10 mils of water, add a few drops of HCl and pass H₂S through the solution to precipitate the excess of molybdenum, then filter and proceed with the examination for group 3 of the metals, etc.

GROUP C.

Acids whose radicals are not precipitated by any group reagent.

The principal members of this group are boric acid, H₃BO₃; acetic acid, HC₂H₃O₂; and nitric acid, HNO₃.

BORIC ACID, H₃BO₃.

Boric acid, U. S. P.

Boracic acid. Orthoboric acid.

Boric acid is a crystalline solid which is moderately soluble in water. The acid decomposes when heated, forming successively metaboric acid, HBO₂; pyroboric acid or tetraboric acid, H₂B₄O₇; and finally the acid anhydride, boron trioxide, B₂O₃. The salts of ortho-boric acid are unstable and seldom seen. The borates, generally, are the salts of tetraboric acid or metaboric acid, but when decomposed in the presence of water they yield orthoboric acid. The borates are decomposed by nearly all other acids. The borates of the alkali metals are soluble in water. The borates of other metals are insoluble in water but many of them become soluble in the presence of an excess of boric acid.
IMPORTANT SALTS OF BORIC ACID.

Glyceryl borate, boroglycerin, C₃H₅BO₃.
Sodium borate, U. S. P., sodium tetraborate, sodium pyroborate, borax, Na₂B₄O₇·10H₂O.

TESTS FOR SALTS OF BORIC ACID.

Use a separate portion of a solution of a borate, as Na₂B₄O₇, for each of the following tests:
1. Add BaCl₂ T.S. to the borate solution. A white precipitate of barium metaborate, Ba(BO₂)₂, will form if the solution is not too dilute. The precipitate is soluble in an excess of the reagent and in HNO₃ or HCl.
2. Add H₂SO₄ T.S. to a portion of borate solution in a porcelain evaporating dish; then add about twice the volume of alcohol, stir and ignite. The outer mantle of the flame will show a green color. Before applying this test it is necessary to remove copper and barium from the solution, if they are present, by H₂S and H₂SO₄, respectively.
3. Add HCl T.S. to the borate solution, dip a piece of turmeric paper in the liquid and dry the paper. The paper will be colored red-brown, changing to red upon drying.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. Na₂B₄O₇ + BaCl₂ + H₂O = Ba(BO₂)₂ + Na₂B₄O₇ + Ba(NO₃)₂ + H₂O =
2. Na₂B₄O₇ + H₂SO₄ + H₂O = H₃BO₃ +
3. Na₂B₄O₇ + HCl + H₂O = H₃BO₃ +

ACETIC ACID, HC₂H₃O₂.

Acetic acid, U. S. P., 36-37%.
Diluted acetic acid, U.S.P., 5.7-6.3%. Glacial acetic acid, U.S.P., 99+%.

Acetic acid is a colorless liquid with a characteristic pungent odor. The pure acid corrodes the skin and freezes to a glass-like solid at temperatures below 15°C. The diluted acid has about the strength of vinegar, from which it was first prepared.

The acid is monobasic and its salts are called acetates. All the normal acetates are soluble in water, but insoluble basic acetates are formed by several of the metals.

IMPORTANT SALTS OF ACETIC ACID.

Ammonium acetate, NH₄C₂H₃O₂.
Copper acetate, Cu(C₂H₃O₂)₂·H₂O.
Copper aceto-arsenite, paris green, Cu(C₂H₃O₂)₂ + 3Cu(AsO₂)₂.
Copper subacetate, verdigris, Cu₂O(C₂H₃O₂)₂.
Ferric acetate, Fe(C₂H₃O₂)₃.
Lead acetate, U. S. P., sugar of lead, Pb(C₂H₃O₂)₂3H₂O.
Lead sub-acetate, Pb₂O(C₂H₃O₂)₂.
Potassium acetate, U. S. P., KC₂H₃O₂.
Sodium acetate, U. S. P., NaC₂H₃O₂3H₂O.
Zinc acetate, U. S. P., Zn(C₂H₃O₂)₂2H₂O.

TESTS FOR SALTS OF ACETIC ACID.

Use a separate portion of a solution of an acetate, as NaC₂H₃O₂, for each of the following tests:

1. To a portion of the acetate solution add H₂SO₄ T.S., boil the mixture and observe the odor of the vapor, which will be that of the acetic acid, HC₂H₃O₂, set free.

2. To about 2 mils of the acetate solution, add a few drops of alcohol, C₂H₅OH, and about 1 mil of H₂SO₄ T.S. and heat, observing the odor of the vapor. The characteristic fruity odor of ethyl acetate, C₂H₅C₂H₃O₂ will be observed.

3. Add FeCl₃ T.S. to the acetate solution which should be neutral or only slightly acid in reaction, and boil the mixture. Ferric acetate, Fe(C₂H₃O₂)₃, is formed and colors the liquid red. Upon boiling a brown-red precipitate of a basic ferric acetate, FeO(C₂H₃O₂)₂, will be obtained.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. NaC₂H₃O₂ + H₂SO₄ =
   Pb(C₂H₃O₂)₂ + HCl =
2. NaC₂H₃O₂ + H₂SO₄ + C₂H₅OH = NaHSO₄ +
3. KC₂H₃O₂ + FeCl₃ =
   Zn(C₂H₃O₂)₂ + FeCl₃ =
   Fe(C₂H₃O₂)₃ + H₂O =

NITRIC ACID, HNO₃.

Aqua fortis.
Nitric acid, U.S.P., 67-69%.

Nitric acid is a colorless fuming liquid which is unstable and difficult to prepare in a pure state. The official acid is a concentrated solution and is a dangerously corrosive and poisonous liquid. It is a strong acid and also a powerful oxidizing agent. "Fuming nitric acid" is brown in color and is a solution of nitrogen peroxide, NO₂, in concentrated nitric acid.

The acid is monobasic and its salts are called nitrates. All normal nitrates are soluble in water.
NITRIC ACID

IMPORTANT SALTS OF NITRIC ACID.

Ammonium nitrate, $\text{NH}_4\text{NO}_3$.
Barium nitrate, $\text{Ba(NO}_3)_2$.
Bismuth nitrate, $\text{Bi(NO}_3)_3$.
Bismuth sub-nitrate, U.S.P., approximately $\text{Bi(OH)}_2\text{NO}_3$.
Ferric nitrate, $\text{Fe(NO}_3)_3$.
Lead nitrate, $\text{Pb(NO}_3)_2$.
Mercuric nitrate, $\text{Hg(NO}_3)_2\cdot 4\text{H}_2\text{O}$.
Potassium nitrate, U.S.P., saltpetre, nitre, $\text{KN}_0_3$.
Silver nitrate, U.S.P., lunar caustic, $\text{AgNO}_3$.
Sodium nitrate, Chili saltpetre, $\text{NaNO}_3$.
Strontium nitrate, $\text{Sr(NO}_3)_2$.

TESTS FOR SALTS OF NITRIC ACID.

Use a separate portion of a solution of a nitrate, as $\text{KNO}_3$, for each of the following tests:

1. To a portion of the nitrate solution add an equal volume of $\text{H}_2\text{SO}_4$ T.S. and a small amount of metallic copper and heat the mixture. The nitrate is decomposed and gaseous nitric oxide, NO, is formed. This oxidizes in the air and the brown gas, nitrogen peroxide, NO$_2$, is obtained.

2 BROWN RING TEST. Mix about 1 mil of the nitrate solution with about the same volume of concentrated $\text{H}_2\text{SO}_4$, and cool the mixture. Then carefully pour some freshly made FeSO$_4$ T.S. down the side of the test-tube so that the liquids do not mix and let stand. The liberated nitric acid will oxidize some of the iron to form ferric sulphate and this will give a brown ring between the two layers.

The test may also be applied by mixing the solution to be tested with FeSO$_4$ T.S. and superimposing the mixture on concentrated $\text{H}_2\text{SO}_4$ in a test-tube.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $2\text{KNO}_3 + 4\text{H}_2\text{SO}_4 + 3\text{Cu} = \text{K}_2\text{SO}_4 + 3\text{CuSO}_4 + 4\text{H}_2\text{O} + 2\text{NO}$
   
   $2\text{NO} + \text{O}_2 = 2\text{NO}_2$

2. $\text{KNO}_3 + \text{H}_2\text{SO}_4 =$
   
   $6\text{FeSO}_4 + 3\text{H}_2\text{SO}_4 + 2\text{HNO}_3 = 3\text{Fe}_2(\text{SO}_4)_3 + 4\text{H}_2\text{O} + 2\text{NO}$

SAMPLE NUMBER 17.

Analysis of a solution for the principal metals and for the radicals of boric, acetic and nitric acids.

The sample may contain any of the important metals, as borates, acetates or nitrates.
A. Examine a portion of the sample for metals by the tables on pages 82–85.

B. To a portion of the sample add HCl T.S., dip a piece of turmeric paper in the liquid and dry the paper. If the color of the paper is unchanged borates are absent. If the paper is turned red-brown, changing to dark red upon drying, borates are present.

C. To a portion of the sample add a few drops of alcohol followed by an excess of H₂SO₄ T.S., heat the liquid and observe the odor of its vapor. If no odor of ethyl acetate can be observed, acetates are absent. If the odor of ethyl acetate is observed, acetates are present.

D. Mix some of the sample with about the same volume of concentrated H₂SO₄ in a test-tube, cool the liquid, superimpose FeSO₄ T.S. on it and let stand. If no brown ring forms at the junction of the two liquids, nitrates are absent. If a brown ring forms at the junction of the liquids, nitrates are present.

**SAMPLE NUMBER 18.**

**Analysis of a solution for the important metals and acids.**

The sample may contain any of the important metals as salts of any of the acids studied.

A. Add NaOH T.S. to a portion of the sample until the reaction is alkaline, then HC₂H₃O₂ T.S. until the reaction is acid, then CaCl₂ T.S. and let stand. If no precipitate is produced, oxalates are absent. A white precipitate, which may be formed slowly, shows the presence of oxalates.

B. To about 3 mils of (NH₄)₂MoO₄ T.S. in a test-tube add a few drops of the sample, warm and let stand. If no precipitate forms, phosphates are absent. A yellow precipitate, slowly formed, shows the presence of phosphates.

C. Examine a portion of the sample for metals by the tables on pages 82–85, removing oxalates and phosphates if present as directed on pages 99–100.

D. To about 5 mils of the sample add an excess of H₂SO₄ T.S. and heat. If a gas is given off observe its odor, hold a piece of paper wet with Pb(C₂H₃O₂)₂ T.S. in the gas and pass some of it through lime water.

A gas having the odor of burning sulphur shows the presence of sulphites.  
A gas having the odor of H₂S and which blackens Pb(C₂H₃O₂)₂ shows the presence of sulphides.  
An odorless gas which renders lime water turbid shows the presence of carbonates.

E. Add a slight excess of HNO₃ T.S. to a portion of the sample, followed by AgNO₃ T.S. If no precipitate is produced, the acids of
NITRIC ACID

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group A are absent: pass to paragraph I. If a precipitate is produced, proceed by paragraph F, remembering that sulphides, if present, will have been detected in D.

F. To about 3 mils of the sample add a small crystal of FeSO₄ and an excess of NaOH T.S. and heat. Then add two drops of FeCl₃ T.S. and acidulate with HCl. A blue precipitate, Fe₄(Fe(CH)₆)₃, shows the presence of cyanides.

G. To a few drops of the sample, add about the same amount of CS₂ and then slowly add chlorine T.S. until the chlorine is in excess. If the CS₂ is not colored, iodides and bromides are absent. If the CS₂ is colored pink at first and becomes colorless or brown when the chlorine is in excess, iodides are present. If the CS₂ is colored brown when the chlorine is in excess, bromides are present.

H. If sulphides, cyanides, iodides and bromides are absent, acidulate a small portion of the sample with HNO₃ T.S. and add AgNO₃ T.S. A white precipitate which readily dissolves in NH₄OH T.S. shows the presence of chlorides.

If sulphides or cyanides are present, add an excess of HNO₃ T.S. to a small portion of the sample and boil, under a hood, to expel H₂S or HCN. Then add AgNO₃ T.S. A white precipitate shows the presence of chlorides.

If bromides or iodides are present, add HNO₃ T.S. to about 5 mils of the sample and boil if necessary to expel H₂S or HCN. Add an excess of AgNO₃ T.S. and filter. Pass about 2 mils of NH₄OH T.S. once through the precipitate on the filter. Any silver chloride quickly dissolves as ammonio-silver chloride. Acidulate the filtrate with HNO₃. A white precipitate shows the presence of chlorides.

I. Acidulate a portion of the sample, add HCl T.S., and add BaCl₂ T.S. A white precipitate shows the presence of sulphates. If a precipitate is formed upon adding HCl T.S., it should be filtered out and the BaCl₂ T.S. should be added to the clear filtrate.

J. Add H₂SO₄ T.S. and an equal volume of alcohol to a portion of the sample contained in a porcelain evaporating dish and set fire to the alcohol. If the flame is colored green, borates are present.

Barium and copper, if present, must be removed before applying this test for boric acid.

K. Add an excess of H₂SO₄ T.S. and a few drops of alcohol to a portion of the sample, boil and observe the odor of the vapor. An odor of ethyl acetate, C₂H₅C₂H₅O₂, shows the presence of acetates.

L. Mix a portion of the sample with about the same volume of concentrated H₂SO₄, superimpose FeSO₄ T.S. on the liquid and let stand. A brown zone between the two liquids shows the presence of nitrates.
SOLIDS.

Solid substances must generally be put into solution before they can be examined for bases and acids, and the tests must be applied to the solutions obtained; but the behavior of the solid, alone or with reagents, will often give information leading to the identification of its constituents; and various methods for the examination of solids have been devised. The method given here is aimed at bringing the solid into solution by the use of various solvents. Incidentally, some information of analytical value may be obtained by observing the directions closely.

SAMPLES NUMBERS 19 AND 20.

Qualitative analysis of a solid.

The sample may contain any of the bases or acids considered in the course. Interfering organic substances are absent.

A. The sample should be finely powdered with a mortar and pestle, or otherwise, if it is not already in a powdered condition.

B. Place a small portion of the powdered sample in a small dry test-tube and heat in a gas flame, carefully observing its behavior and the color and odor of any gas or vapor given off, also any condensation of vapors on the cool portion of the tube.

If no apparent change occurs in the solid and no odor is observed, and there is no condensation of vapors, then organic matter, volatile bodies and water of crystallization are absent.

If the solid blackens and there is an odor of something burning, organic matter is present. If organic matter were present it would be necessary to destroy it, by methods which will be described later, as it would interfere with the analytical scheme.

Compounds of some of the metals change color or fuse when heated, but the changes are not significant enough to be of much analytical value. Such metals, if present, will be found when the groups are separated.

If the substance gives off a vapor which condenses on the cool part of the tube, volatile substances are contained in the solid, or are formed upon heating it. These may include water; any compounds of ammonium, arsenic, antimony and mercury; or some compounds of sulphur, iron and iodine. The water may generally be recognized as minute drops and the other substances will be found in their proper places when the scheme of analysis is applied.
If a gas is evolved, observe its odor and test it by holding a splinter of wood or a cord with a spark at the end in it, and by passing some of it through lime water. H₂S, SO₂, NH₃, NO₂ and some other gases may be recognized by their odors. If the gas makes a spark glow more vividly it contains more oxygen than the air and this indicates the presence of nitrates, chlorates, bromates, iodates or peroxides. If organic matter is absent and the gas renders lime water turbid, it contains carbon dioxide and the presence of carbonates is indicated.

C. Boil about 1 gram of the finely divided sample with about 15 mils of water in a beaker. If the solid does not completely dissolve allow the residue to settle and decant the water through a filter. Evaporate a few drops of the filtrate to dryness on a watch-glass. If no appreciable deposit is left on the watch-glass the sample is insoluble in water. In such a case proceed with paragraph D, discarding the rest of the filtrate. If any appreciable deposit is left on the watch-glass, the rest of the filtrate, or the solution if the sample all dissolved in water, should be examined for the bases by the tables on pages 82–85 and for acids as directed on pages 92, 97, 99 and 103.

D. If there is any residue insoluble in water from C, wash it with water by decantation and filtration and allow to drain, discarding the washings. Transfer two small portions of the moist powder to test-tubes and boil one with HCl T.S. and the other with HNO₃ T.S., observing which of the acids appears to be the better solvent for the powder.

If there is an effervescence upon adding the HCl T.S. to the powder, observe the color and odor of the gas and pass some of it through lime water. A greenish-yellow gas indicates a chlorate, a brown gas indicates a nitrate, and a purple gas indicates an iodide. Gases with characteristic odors are yielded by salts of H₂S, H₂SO₃, HBr, HI, HCN and HC₂H₃O₂. If the gas renders lime water turbid, carbonates are indicated. It should be remembered that chlorine might be generated from the HCl used, if certain oxidizing agents are present.

Boil the rest of the moist powder with about 10 mils of the acid which appears to dissolve it the better. If neither acid appears to dissolve any of the powder, or both dissolve it entirely, use HCl T.S. in preference to HNO₃ T.S. If there is any residue insoluble in the acids, allow it to settle and decant the supernatant liquid through the filter used for the water solution and test a few drops for dissolved matter by evaporating on a watch-glass as before. If any appreciable deposit is left on the watch-glass, the rest of the filtrate, or the solution if all the residue dissolved in the acid, should be examined for bases by the tables on pages 82–85, and for acids as directed on paged 92, 97, 99 and 103.
E. If there is any residue insoluble in HCl or HNO₃, from D, it should be washed by decantation and filtration as before and digested with about 5 mils of nitro-hydrochloric acid. Then add about 10 mils of water and filter, testing the filtrate for dissolved matter by evaporation on a watch-glass and examine for bases and acids, if any appreciable amount of the solid goes into solution.

F. If sufficient quantities of the solvents have been used and there is still some residue left from E, it consists of insoluble sulphates, chlorides or silicates. To decompose these transfer the washed and drained residue to a porcelain crucible, mix it with about twice its bulk of dry Na₂CO₃ and heat to fusion. Then allow the mass to cool and boil it with about 10 mils of water. Filter the liquid and examine the filtrate for acids by paragraph G. Dissolve any residue in HCl or HNO₃, as in paragraph D and examine the solution for bases by the tables on pages 82–85.

G. To the filtrate from F add an excess of HNO₃ T.S. and boil. If no precipitate is formed, silicates are absent. A white gelatinous precipitate shows the presence of silicates. If a precipitate is formed filter the liquid. Divide the liquid or filtrate into two portions.

To one portion add BaCl₂ T.S. If no precipitate is formed, sulphates are absent. A white precipitate shows the presence of sulphates.

To the other portion, add AgNO₃ T.S. If no precipitate is formed, chlorides are absent. A white precipitate, readily soluble in ammonia water, shows the presence of chlorides.
QUALITATIVE EXAMINATION OF OFFICIAL INORGANIC CHEMICALS.

In speaking of the purity of a chemical substance we refer to the kinds and amounts of other substances that it contains. These other substances are called impurities.

To manufacture an absolutely pure chemical compound, it would be necessary to use nothing but absolutely pure substances and not allow the compound to come into contact with anything that would contaminate it at any step in the process of manufacture. Such ideal conditions are seldom or never found, as natural substances are not often chemically pure and most chemicals will mix with more or less of contaminating substances during their processes of manufacture. Compounds approximating chemical purity, however, are obtained by working as nearly as possible under the ideal conditions, or by purifying compounds of a lower degree of purity. In general, water is considered as an impurity only to the extent that it affects the strength or concentration of a chemical.

Many grades of the same chemical are manufactured. The lowest commercial grade is commonly said to be technically pure and contains enough of the chemical to give the substance identity, but may contain considerable amounts of impurities. Chemicals of this grade are made in large quantities and are used in many kinds of manufacturing work. The highest grade of a chemical is generally labelled chemically pure and is as nearly absolutely pure as can be obtained under manufacturing conditions. Such chemicals are relatively high in price and are only used when such a high grade of purity is essential, as in the preparation of reagents for analytical work.

For medicinal use, chemicals are required to be free from appreciable amounts of objectionable impurities and of proper strength, but the United States Pharmacopoeia and the National Formulary do not require that they be of chemically pure grade in many cases, as the excessive cost of such chemicals is not warranted, provided that the impurities are not harmful or excessive in amount. A similar rule can properly be applied to unofficial chemicals. The allowable amount of impurities is stated in the purity rubric, which is a part of the definition of official chemicals and states the minimum strength required for concentrated chemicals and the minimum and maximum strengths allowed for solutions of chemicals.
Qualitative tests on official chemicals are of two kinds, namely, *identity tests* and *purity tests*, these being used, respectively, to establish the identity of the substance under examination, and to ascertain the presence or absence of excessive amounts of objectionable impurities. The strengths of different chemicals are determined by assays, which belong to the domain of quantitative analysis.

The directions for work on samples 21–25 are taken from the United States *Pharmacopoeia*, with some omissions and modifications, made to keep the work within the scope of an elementary course in qualitative analysis.

SAMPLE NUMBER 21.

**Examination of granulated ferrous sulphate for identity and purity.**  
(U. S. P., pp. 172 and 170.)

The tests should be carried out as directed, the following report form being copied and filled out as the results of each test are obtained.

Name............................................ Date................

Record of work and report on sample No. 21.

Qualitative examination of granulated ferrous sulphate,  
(U. S. P., pages 172 and 170).

Tests for identity:

- Color—
- Odor—
- Taste—
- Reaction to litmus—
  - \( + K_3\text{Fe(CN)}_6 \text{T.S.} \)
  - \( + \text{BaCl}_2 \text{T.S. and HCl T.S.} \)

Tests for impurities:

- Heavy metals—
- Free acid—

**TESTS FOR IDENTITY.**

Granulated ferrous sulphate is a very pale bluish-green crystalline powder, without odor, and having a saline, styptic taste.

An aqueous solution of the salt (1 in 20) is acid to litmus.

An aqueous solution of the salt, even when highly diluted, gives with potassium ferricyanide T.S. a blue color or precipitate, and with barium chloride T.S. a white precipitate insoluble in hydrochloric acid.
TESTS FOR IMPURITIES.

Dissolve 1 Gm. of the salt in about 50 mils of distilled water containing 1 mil of diluted sulphuric acid, heat the solution to boiling, oxidize it with nitric acid and then mix it with a slight excess of ammonia water and filter. The filtrate is colorless, and, after acidulating with hydrochloric acid, it does not respond to the test for heavy metals (see general test No. 3, p. 118).

Agitate 1 Gm. of ferrous sulphate in small fragments during four or five minutes, with 10 mils of alcohol, and filter the mixture; the filtrate does not immediately redden moistened blue litmus paper (free acid).

SAMPLE NUMBER 22.

Examination of ammonium bromide for identity and purity. (U. S. P., p. 43.)

Tests should be carried out as described, and a written record and report made as the work progresses, similar to that directed for sample No. 21.

TESTS FOR IDENTITY.

Ammonium bromide occurs in colorless, transparent, prismatic crystals, or as a white, crystalline or granular powder; odorless, of a pungent, saline taste.

When heated, ammonium bromide volatilizes without fusing.

An aqueous solution of the salt (1 in 20) is neutral or not more than slightly acid to litmus.

An aqueous solution of ammonium bromide when gently heated with potassium hydroxide T.S. evolves ammonia.

Silver nitrate T.S. added to an aqueous solution of the salt (1 in 10) produces a yellowish-white precipitate, insoluble in nitric acid or in a moderate excess of ammonia water.

TESTS FOR IMPURITIES.

Add 1 mil of chloroform to 10 mils of an aqueous solution of the salt (1 in 20) and cautiously introduce chlorine water, which has been diluted with an equal volume of distilled water, drop by drop and with constant agitation. The liberated bromine dissolves in the chloroform and imparts to it a yellow or orange color, which is free from any violet tint (iodide).

Drop 1 mil of diluted sulphuric acid upon about 1 Gm. of the powdered salt; no yellow color appears at once (bromate).

A blue color is not produced at once on adding potassium ferrocyanide T.S. to 20 mils of an aqueous solution of the salt (1 in 250) (iron).
Add 1 mil of potassium sulphate T.S. to 10 mils of an aqueous solution of ammonium bromide (1 in 20), acidulated with acetic acid; no turbidity is produced immediately (barium); under similar conditions the addition of barium chloride T.S. produces no turbidity (sulphate).

An aqueous solution of the salt does not respond to the test for heavy metals (see general test No. 3, p. 118).

SAMPLE NUMBER 23.

Examination of diluted hydrochloric acid for identity and purity. (U. S. P., p. 14.)

The tests should be carried out as described, and a written record and report made as the work progresses, similar to that directed for sample No. 21.

TESTS FOR IDENTITY.

Diluted hydrochloric acid is a colorless, odorless liquid; it has a strongly acid taste and is strongly acid to litmus.

With silver nitrate T.S. it yields a white, curdy precipitate, insoluble in nitric acid, but readily soluble in ammonia water.

TESTS FOR IMPURITIES.

Add 1 mil of chloroform, to 10 mils of diluted hydrochloric acid and then cautiously introduce chlorine water, which has been diluted with an equal volume of distilled water, a drop at a time, with constant agitation; the chloroform remains free from any yellow, orange or violet color (bromide or iodide).

Add 1 mil of potassium iodide T.S. and 1 mil of chloroform to 10 mils of diluted hydrochloric acid and agitate the mixture; the chloroform remains free from any violet coloration (free chlorine or bromine).

Two mils of diluted hydrochloric acid, diluted with seven volumes of distilled water, does not respond to the test for heavy metals (see general test No. 3, p. 118).

Five mils of diluted hydrochloric acid diluted with 12 mils of distilled water (without the treatment with sulphuric and sulphurous acids) meets the requirements of the test for arsenic (see general test No. 1, p. 115).

Add 5 drops of barium chloride T.S. to a mixture of 3 mils of diluted hydrochloric acid and 5 mils of distilled water; neither turbidity nor precipitation appears within one hour (sulphuric acid or sulphates). At the end of this period, the further addition to the liquid of 2 drops of tenth-normal iodine V.S. produces neither turbidity nor decoloration of the iodine (sulphurous acid).
SAMPLE NUMBER 24.

Examination of bismuth subnitrate for identity and purity.
(U. S. P., pp. 82-83.)

The tests should be carried out as described and a written record
and report made as the work progresses, similar to that directed for
sample No. 21.

TESTS FOR IMPURITIES.

Boil about 1 Gm. of bismuth subnitrate with 20 mils of a mixture
of equal parts of acetic acid and distilled water, cool the solution
and filter. Free the filtrate from bismuth by the addition of hydro-
gen sulphide, boil the mixture and again filter. The latter filtrate
leaves no appreciable residue on evaporation and gentle ignition
(alkalies or alkali earths).

Boil about 0.1 Gm. of the salt with 5 mils of potassium hydroxide
T.S.; no odor of ammonia is perceptible nor does the vapor turn
moistened red litmus paper blue.

The residue resulting from the ignition of 2 Gm. of the salt does
not respond to Bettendorf's test for arsenic (see general test No. 2,
p. 117).

Add 3 Gm. of the salt to 3 mils of warm nitric acid; no efferves-
cence occurs (carbonate), and no residue remains (insoluble foreign
salts). Pour this solution into 100 mils of distilled water, a white
precipitate is produced. Filter, evaporate the filtrate on a water-
bath to 30 mils, again filter the liquid and divide the new filtrate
into portions of 5 mils each.

Mix one portion with an equal volume of diluted sulphuric acid;
it does not become cloudy (lead).

Precipitate another portion with a slight excess of ammonia water;
the supernatant liquid does not exhibit a bluish tint (copper).

Another portion is not immediately affected by barium nitrate
T.S. (sulphate). With hydrochloric acid no precipitate is formed
which is insoluble in a slight excess of the latter, but soluble in ammonia water (silver).

SAMPLE NUMBER 25.

Examination of alum for identity and purity.
(U. S. P., pp. 39-40).

The tests should be carried out as described and a written record and report made as the work progresses, similar to that directed for sample No. 21.

TESTS FOR IDENTITY.

Ammonium alum and potassium alum both occur in large, colorless crystals, crystalline fragments, or as white powders; alum is odorless and has a sweetish and strongly astringent taste.

Ammonium alum.—Potassium hydroxide T.S. added to an aqueous solution of ammonium alum (1 in 20) at first causes a precipitate, which completely dissolves in an excess of the reagent, ammonia being evolved.

Potassium alum.—Potassium alum imparts a violet color to a non-luminous flame.

The addition of sodium bitartrate T.S. to a saturated solution of potassium alum produces, sometimes slowly, a white crystalline precipitate.

Potassium hydroxide T.S. added to an aqueous solution of potassium alum (1 in 20) at first causes a precipitate, which completely dissolves in an excess of the reagent, but no ammonia is evolved.

Both ammonium alum and potassium alum conform to the following tests:

An aqueous solution of alum (1 in 20) is acid to litmus.

An aqueous solution of alum (1 in 20) yields with ammonia water a white gelatinous precipitate almost insoluble in an excess of ammonia water.

With barium chloride T.S., an aqueous solution of alum (1 in 20) yields a white precipitate insoluble in hydrochloric acid.

TESTS FOR IMPURITIES.

An aqueous solution of alum does not respond to the test for heavy metals (see general test No. 3, p. 118). An aqueous solution of alum meets the requirements of the test for arsenic (see general test No. 1, p. 116).

Add 5 drops of potassium ferrocyanide T.S. to 20 mils of an aqueous solution of alum (1 in 150); no blue color is produced at once (iron).
GENERAL QUALITATIVE TESTS OF THE UNITED STATES PHARMACOPÆIA.

1. Arsenic Test.—Test Apparatus.—Prepare several generators, equipped with tubes, etc., as described below. Select as a generator a bottle of about 50 mls capacity, having a mouth about 2.5 cm. in diameter and provide a well-fitting rubber stopper, suitably perforated. In the perforation in this stopper insert a vertical exit tube about 13 cm. in total length and 1 cm. in diameter throughout the upper portion (for about 10 cm.) and constricted at its lower extremity to a tube of about 3 cm. in length and about 5 mm. in diameter. This latter tube should extend but slightly below the stopper. In the lower part of the wider exit tube insert a small pledget of dry glass wool and then a strip of the freshly prepared but dry lead acetate test-paper rolled into a coil, and above this a plug of the moist (not wet) lead acetate glass wool. In the upper extremity of this tube insert through a perforated cork stopper, a glass tube 12 cm. in length, having an internal diameter of about 3 mm. Place the mercuric bromide test-paper in this tube, bending or creasing the upper portion of the strip so that it will retain its position. The strip should extend within about 2 cm. of the perforated cork stopper and must not be introduced into the tube until ready to start the test. This tube should be thoroughly cleaned and dried each time it is used.

Standard Arsenic Test-solution.—Dissolve 0.1 Gm. of arsenic trioxide, which has been finely pulverized, dried in a desiccator, and accurately weighed, in about 5 mls of a 20 per cent. solution of sodium hydroxide (free from arsenic). Neutralize the solution with diluted sulphuric acid (free from arsenic), add 10 mls of the same acid and sufficient recently boiled distilled water to bring the volume of the solution to exactly 1000 mls at 25° C. Accurately measure 10 mls of this solution, transfer it to a 1000 mil flask, add 10 mls of diluted sulphuric acid (free from arsenic) and make up the volume with recently boiled distilled water to exactly 1000 mls at 25° C. Employ this solution, containing 0.001 mg. of arsenic trioxide in each 1 mil (at 25° C.), in preparing the standard stain. Keep this solution in a glass-stoppered bottle. It is advisable to make fresh solutions whenever new standard stains are to be prepared.
Preparation of the Chemical to be Tested.—Add 1 mil of a mixture of equal volumes of concentrated sulphuric acid and distilled water, to 5 mils of an aqueous solution of the chemical (1 in 25) or to a solution in 5 mils of distilled water of the residue remaining after any special treatment that may be directed. This acidulation is not necessary in the case of inorganic acids. Now, unless specially directed, add 10 mils of a saturated aqueous solution of sulphurous acid. Evaporate this liquid in a small beaker, on a water-bath, until it is free from sulphurous acid and has been reduced to about 2 mils in volume. Dilute this evaporated liquid to about 5 mils with distilled water.

The Test for Arsenic.—Preparation of the Standard Stain.—Introduce into the generator from 8 to 10 Gm. of zinc followed by 25 mils of dilute sulphuric acid, prepared by mixing 1 part of concentrated sulphuric acid, free from arsenic, with 4 parts of distilled water, and 5 drops of acid stannous chloride T.S. Add at once 2 mils (accurately measured at 25° C.) of the standard arsenic T.S. and immediately insert the stopper containing the exit tube, into which have been placed the glass-wool pledget, the dry lead acetate test-paper, the moist lead acetate glass wool, and the mercuric bromide test-paper, as described under the Test Apparatus. Should the evolution of the gas be violent at first, check the reaction by immersing the bottle in cold water. Should the reaction subside, increase it by placing the bottle in warm water. If the reaction be too violent, the stain will spread and not form a distinctive band, thus making the comparisons of color intensity difficult. After the test has continued for forty-five minutes, remove the mercuric bromide test-paper and place it in a clean, dry tube for comparison. This stain represents 0.002 mg. of arsenic trioxide in addition to any stain produced by the reagents. The stain from the reagents should scarcely be perceptible when determined by a blank experiment. Since light, heat and moisture cause the stain to fade rapidly, comparison should be made as soon as possible. The stained test-papers may be preserved by either dipping in hot, melted paraffin or keeping them over phosphoric anhydride protected from light.

Testing the Chemical.—Introduce into another generator from 8 to 10 Gm. of the zinc, followed by 25 mils of the dilute sulphuric acid (1 to 4) and 5 drops of acid stannous chloride T.S. Insert the stopper containing the exit tube charged with the test-papers and glass wool, as just described. Then add 5 mils of the solution to be tested, previously treated as directed, under Preparation of the Chemical, and immediately insert the stopper charged with the test-papers and glass wool. When the evolution of hydrogen has pro-
ceeded actively for forty-five minutes, remove the mercuric bromide test-paper and carefully compare it with the standard stain prepared as described above. The stain produced by the chemicals tested does not exceed in length or intensity of color that prepared as the standard, indicating not more than 1 part of arsenic in 100,000 parts of the substance tested.

Interfering Chemicals.—Antimony, if present in the substance tested, will produce a gray stain. Sulphites, sulphides, thiosulphates, and other compounds which liberate hydrogen sulphide or sulphurous acid when treated with sulphuric acid, must be oxidized by means of nitric acid and then reduced by means of sulphurous acid as directed under Preparation of the chemical to be tested, before introducing them into the apparatus. Sulphur compounds as well as hydrogen phosphide give a bright yellow band on the test paper. If sulphur compounds are present, a simultaneous darkening of the lead acetate test paper and glass wool will occur. If such is the case, the operation as directed under Preparation of the chemical to be tested must be repeated upon a fresh portion of the solution being tested, using greater care in effecting the complete removal of the sulphurous acid. In testing hypophosphites special care should be observed to completely oxidize the solution being tested as directed, otherwise a yellow stain may be produced through the evolution of hydrogen phosphide, which might be confused with the orange-yellow color produced by arsenic. Compounds containing antimony should be tested for arsenic by Bettendorf's Test (see Test No. 2).

The test apparatus should be thoroughly cleaned and dried immediately before and after use.

2 Arsenic test, Bettendorf's.—This test is employed only in testing salts of bismuth and compounds containing antimony for the presence of arsenic.

To a solution of the prescribed quantity of the substance to be tested in 5 mils of concentrated hydrochloric acid contained in a clean test tube, add 10 mils of saturated stannous chloride T.S. which has been freshly prepared, and set it aside for thirty minutes. If arsenic is present in non-permissible amounts, a brownish tint or precipitate will be seen when the tube is placed over a white surface and the solution viewed from above, comparison being made with a mixture of 5 mils of concentrated hydrochloric acid and 10 mils of concentrated stannous chloride T.S., prepared as directed above.

Note: It is absolutely necessary that for this test the solution of stannous chloride be freshly prepared, and that nitrates, sulphates, sulphites, sulphides and compounds of mercury, gold, and selenium be absent from the reagents, and from the chemicals being tested.
3 Test for heavy metals. — Chemicals. — This test is to be used to
detect the presence of undesirable metallic impurities in official
chemical substances or their solutions; these must not respond
affirmatively within the stated time. Acidulate 10 mils of a solution
of the substance in distilled water (1 in 50), contained in a test tube
of about 40 mils capacity and of about 2.5 cm. diameter, with 1 mil
of diluted hydrochloric acid (unless otherwise directed), warm it
to about 50° C., add an equal volume of freshly prepared hydrogen
sulphide T.S., stopper, and allow the mixture to stand at 35° C.
for half an hour. At the end of this time the mixture should still
possess the odor of hydrogen sulphide; if not, it should be thoroughly
saturated with the gas and again set aside for half an hour. The
color produced, if any, is not greater than that observed by a blank
test made in the same manner and with the same quantities of the
reagents (omitting the solution to be tested); the solutions being
viewed crosswise by reflected light while held against a white sur-
face. A slight turbidity due to separation of sulphur from the
hydrogen sulphide may occur.
DESTROYING OF ORGANIC MATTER.

If organic matter is present in a sample, it is necessary to destroy it before proceeding with a qualitative analysis for inorganic bases and acids. There are several methods for accomplishing this, of which the most generally useful are the following:

A. Mix the sample with an excess of concentrated HCl, add some crystals of KClO₃ and boil under a hood until the mixture is decolorized or a portion does not carbonize upon drying and igniting. Add additional HCl and KClO₃ as necessary, avoiding a large excess. Then evaporate the liquid to dryness on a water bath and examine the residue by the method for the qualitative analysis of a solid.

This method destroys the organic matter, or changes it so that it does not interfere with the analysis. The metals present are oxidized to their highest valences, and ammonium salts are volatilized. Allowance must also be made for the potassium and chlorine compounds added.

B. Mix the sample with about 4 times its weight of concentrated H₂SO₄ and twice its weight of concentrated HNO₃, digest at the ordinary temperature for several hours, and then heat in a hood until the white fumes of H₂SO₄ are given off. If the mixture still contains black carbonized organic matter, add more HNO₃ and repeat the heating until white fumes are given off, repeating this treatment with additional HNO₃ and heating several times if necessary. When the mixture ceases to show the presence of carbon, cautiously add about an equal volume of water and concentrate until the white fumes of H₂SO₄ are given off, then cautiously add four or five times the volume of water and filter, if necessary. Examine the filtrate by the method for the qualitative analysis of a liquid. Any residue, on the filter paper, may contain silica or the sulphates of lead, barium, strontium and calcium. Examine it by the method for the qualitative analysis of a solid, beginning with paragraph F on page 108.

This is the most dependable method for the destruction of organic matter. Allowance must be made, however, for the H₂SO₄ and HNO₃ used, and it is often necessary to use both of these methods, or others, in making a careful qualitative analysis of a substance containing organic matter.
REAGENTS AND TEST SOLUTIONS.

On the following pages will be found directions for preparing the test solutions called for in this book. Most of these are of the same strength as is specified in the United States Pharmacopoeia, but for performing the Pharmacopoeial tests on official chemicals, the reagents should be made according to the directions of the Pharmacopoeia IX, pages 521–583.

In preparing reagents and test solutions, only pure chemicals and distilled water should be used.

**Acetic acid, HC\textsubscript{2}H\textsubscript{3}O\textsubscript{2}.—** Mix official acetic acid, containing about 36 per cent. of absolute HC\textsubscript{2}H\textsubscript{3}O\textsubscript{2} with twice its volume of distilled water.

**Ammonium hydroxide test solution, NH\textsubscript{4}OH or NH\textsubscript{3} + H\textsubscript{2}O.—** Use the official ammonia water, containing 10 per cent. of NH\textsubscript{3}.

**Ammonium carbonate test solution, (NH\textsubscript{4})\textsubscript{2}CO\textsubscript{3}.—** Dissolve 20 Gm. of official ammonium carbonate in a mixture of 20 mils of ammonia water and 65 mils of distilled water, and add sufficient distilled water to measure 100 mils.

**Ammonium chloride test solution, NH\textsubscript{4}Cl.—** Dissolve 10 Gm. of ammonium chloride in sufficient distilled water to make 100 mils.

**Ammonium molybdate test solution, (NH\textsubscript{4})\textsubscript{6}Mo\textsubscript{7}O\textsubscript{24} + 4H\textsubscript{2}O.—** Mix 6.5 Gm. of finely powdered molybdic acid with 14 mils of distilled water and 14.5 mils of stronger ammonia water to effect solution. Cool and slowly add the solution, in small portions with agitation, to a well cooled mixture of 32 mils of nitric acid and 40 mils of distilled water. Allow the solution to stand for twenty-four hours and then filter through asbestos.

Preserve the test solution in the dark, and, if a sediment should form in it after some days, carefully decant the clear solution. This solution should be tested at frequent intervals. Add 2 mils of sodium phosphate T.S. to 5 mils of the reagent; an abundant yellow precipitate forms either at once or upon slight warming. If only a slight precipitation or yellow opalescence results, the reagent must be rejected. When employed as a reagent, ammonium molybdate T.S. is always added in large excess to the solution being tested, the latter having previously been strongly acidified with nitric acid.

**Ammonium oxalate test solution (NH\textsubscript{4})\textsubscript{2}C\textsubscript{2}O\textsubscript{4} + H\textsubscript{2}O.—** Dissolve 4 Gm. of ammonium oxalate in sufficient distilled water to measure 100 mils.
Ammonium polysulphide test solution, Yellow Ammonium Sulphide, \((NH_4)_2S_x\).—Saturate three parts of ammonia water with hydrogen sulphide, forming a solution of ammonium hydrogen sulphide, \(NH_4HS\). Add to the solution two additional parts of ammonia water, forming a solution of normal or colorless ammonium sulphide, \((NH_4)_2S\). Finally add a small quantity of precipitated sulphur and preserve the solution in small, amber, glass-stoppered bottles.

Barium chloride test solution, \(BaCl_2 + 2H_2O\).—Dissolve 10 Gm. of barium chloride in sufficient distilled water to make 100 mils.

Barium hydroxide test solution, \(Ba(OH)_2 + 8H_2O\).—A freshly made saturated solution of barium hydroxide in distilled water.

Barium nitrate test solution, \(Ba(NO_3)_2\).—Dissolve 5 Gm. of barium nitrate in sufficient distilled water to make 100 mils.

Bromine test solution (bromine water), Br.—A saturated solution of bromine, prepared by adding 3 mils of bromine to 100 mils of cold water. The solution should be stored in a glass-stoppered bottle in a cool place, protected from light and should be shaken well, allowing the excess of bromine to settle before using.

Calcium chloride test solution, \(CaCl_2 + 2H_2O\).—Dissolve 10 Gm. of calcium chloride in sufficient distilled water to measure 100 mils.

Calcium hydroxide test solution (lime water), \(Ca(OH)_2\).—A nearly saturated aqueous solution of calcium hydroxide.

Calcium sulphate test solution, \(CaSO_4 + 2H_2O\).—Mix 5 mils of diluted sulphuric acid with 200 mils of distilled water, add 1 Gm. of calcium carbonate and shake: when the acid is completely neutralized filter the mixture.

Chlorine test solution (chlorine water), Cl.—A saturated aqueous solution of chlorine, prepared by generating the gas with manganese and hydrochloric acid, washing and conducting it into cold water until saturated. The solution should be preserved in small amber glass-stoppered bottles in a cool, dark place and it should have a strong odor of chlorine when used.

Cobaltous chloride test solution, \(CoCl + 6H_2O\).—Dissolve 2 Gm. of cobaltous chloride with the aid of 1 mil of hydrochloric acid in sufficient distilled water to measure 100 mils.

Copper sulphate test solution, \(CuSO_4 + 5H_2O\).—Dissolve 10 Gm. of copper sulphate in sufficient distilled water to make 100 mils.

Ferric chloride test solution, \(FeCl_3 + 6H_2O\).—Dissolve 10 Gm. of ferric chloride in sufficient water to make 100 mils.

Ferrous sulphate test solution, \(FeSO_4 + 7H_2O\).—Dissolve 10 Gm. of clear crystals of ferrous sulphate in sufficient distilled water to make 100 mils. This solution is to be freshly prepared immediately before use.
Hydrochloric acid test solution, HCl.—Use the official diluted hydrochloric acid of 10 per cent. strength.

Hydrogen sulphide test solution, H₂S.—A saturated, aqueous solution of hydrogen sulphide. To prepare about 1000 mls of the solution, treat 20 Gm. of ferrous sulphide in a suitable apparatus with a mixture of 20 mls of sulphuric acid and 250 mls of distilled water, pass the gas through a drying tube filled with granulated calcium chloride, then from this through a tube of about 8 millimeters diameter and 40 centimeters in length, which contains about 5 Gm. of coarsely pulverized iodine mixed with glass wool, and finally through a wash bottle which contains a small quantity of potassium iodide T.S. The gas thus purified is conducted nearly to the bottom of a bottle of the capacity of about 1500 mls containing 1000 mls of cold distilled water. Shake the bottle occasionally to facilitate the solution of the gas. When the gas is no longer absorbed, transfer the solution to small, dark, amber-colored bottles, fill nearly to the top; pass a stream of purified hydrogen sulphide for a few minutes through each, and then at once stopper them tightly, and preserve them afterwards in a cool and dark place. Do not use this solution unless it retains a strong odor of hydrogen sulphide, and, when added to an equal volume of ferric chloride T.S., produces at once a copious precipitate of sulphur.

Iodine test solution, I.—Dissolve 1 Gm. of iodine and 3 Gm. of potassium iodide in 50 mls of distilled water.

Lead acetate glass wool, Pb(C₂H₃O₂)₂ + 3H₂O.—Immerse glass wool in a mixture of equal parts of lead acetate T.S. and water and remove the excess of liquid by pressing it between filter paper. It should be prepared immediately before it is to be used.

Lead acetate test paper, Pb(C₂H₃O₂)₂ + 3H₂O.—Immerse strips of heavy white filter paper, 6 cm. in width and 8 cm. in length, in a mixture of equal parts of lead acetate T.S. and distilled water, drain off the excess of liquid and dry the paper in an oven at 100° C., avoiding contact with metal.

Lead acetate test solution, Pb(C₂H₃O₂)₂ + 3H₂O.—Dissolve 10 Gm. of clear transparent crystals of lead acetate, free from adhering lead carbonate, in sufficient distilled water to make 100 mls. Preserve the solution in well-stoppered bottles.

Litmus paper and test solution.—Exhaust powdered litmus with three separate and successive portions (each equal to about 4 times its weight) of boiling alcohol (which removes the undesirable color erythrolitmin), each extraction lasting for about one hour. After draining off the alcohol, digest the residue with about an equal weight of cold water and filter. (This blue solution, which contains some alkali, after being acidulated, may be used to make red litmus paper.) Finally, extract the residue with about 5 times its weight of boiling distilled water, and, after thoroughly cooling, filter. The
addition of 1 drop of hundredth-normal acid or alkali V.S. to 50 mils of distilled water containing 5 drops of the indicator produces a distinct change in color. Preserve the filtrate, as a test solution, in wide-mouthed bottles stoppered with loose plugs of purified cotton so as to exclude dust but admit air. The latter must be free from acid or ammoniacal vapors. The blue color of litmus test solution is changed by acids to red, and this red color by the addition of alkalies is restored to blue.

Litmus Paper, Blue.—Impregnate with the test solution just described strips of white filter paper, and dry them by suspending them on lines of clean twine, in an atmosphere free from acid or ammoniacal vapors. This paper must quickly respond to a two hundred and fiftieth-normal acid V.S.

Litmus Paper, Red.—Prepare this with the same kind of paper and in the manner described under Litmus Paper, Blue, having added to the test solution used to impregnate the paper just sufficient of a highly diluted solution of hydrochloric acid to impart to it a faint red tint. Neither blue nor red litmus paper should have an intense color. Preserve the test paper in bottles, so as to exclude dust and acid or ammoniacal vapors. This paper must quickly respond to a two hundred and fiftieth-normal alkali V.S.

Magnesia mixture, MgCl₂ + 6H₂O, NH₄Cl, NH₄OH.—Dissolve 5.5 Gm. of magnesium chloride and 7 Gm. of ammonium chloride in 65 mils of distilled water, add 35 mils of ammonia water, set the mixture aside for a few days in a well-stoppered vessel, and filter. If not perfectly clear, filter the solution before using.

Magnesium sulphate test solution, MgSO₄ + 7H₂O.—Dissolve 10 Gm. of magnesium sulphate in sufficient distilled water to measure 100 mils.

Manganese sulphate test solution, MnO₄ + 4H₂O.—Dissolve 10 Gm. of manganese sulphate in 50 mils of distilled water and add sufficient diluted sulphuric acid to make 100 mils.

Mercuric bromide test paper, HgBr₂.—Cut stiff, heavy quantitative filter paper into strips 3 mm. in width and about 12 cm. in length. Immerse these strips for five minutes in alcoholic mercuric bromide T.S. Remove the excess of solution by pressing the strips between filter paper and then dry them quickly on glass in an oven heated to 100° C. Place the strips at once in a wide mouthed bottle and stopper it securely.

Mercuric bromide test solution, alcoholic, HgBr₂.—Dissolve 5 Gm. of mercuric bromide in 100 mils of alcohol, employing a gentle heat to facilitate solution. Keep it in glass stoppered bottles protected from the light.

Mercuric chloride test solution, HgCl₂.—Dissolve 5 Gm. of mercuric chloride in sufficient distilled water to measure 100 mils.
Mercuric potassium iodide test solution (Mayer's reagent), $\text{HgI}_2 + 2\text{KI}$.—Dissolve 1.358 Gm. of mercuric chloride in 60 mils of distilled water, and 5 Gm. of potassium iodide in 10 mils of distilled water. Mix the two solutions, and then add sufficient distilled water to measure 100 mils.

Mercuric potassium iodide test solution, alkaline (Nessler's Reagent).—Dissolve 10 Gm. of potassium iodide in 10 mils of distilled water, and add gradually in portions a saturated aqueous solution of corrosive mercuric chloride with constant agitation, until a slight red precipitate remains undissolved; to this mixture add 30 Gm. of potassium hydroxide and, when solution has taken place, 1 mil more of the saturated aqueous solution of mercuric chloride. Dilute this solution with distilled water until it measures 200 mils. Allow the precipitate to subside, and draw off the clear fluid. Two mils of this reagent, when added to 50 mils of distilled water containing 0.05 mg. of ammonia, produces at once a yellowish brown coloration.

Mercurous nitrate test solution, $\text{HgNO}_3$.—Mix 10 Gm. of pure mercury with 5 mils of nitric acid and 5 mils of distilled water, in a porcelain evaporating dish and set it aside for twenty-four hours in a cool, dark room. Separate and drain the crystals of mercurous nitrate, and dissolve them in 100 mils of distilled water. Preserve the solution in a dark amber-colored bottle in which a small quantity of mercury has been placed.

Nitric acid test solution, $\text{HNO}_3$.—Add 36 mils of concentrated nitric acid to 200 mils of distilled water and mix. This solution contains approximately 10 per cent. of absolute $\text{HNO}_3$.

Oxalic acid test solution, $\text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$.—Dissolve 5 Gm. of oxalic acid in sufficient distilled water to measure 100 mils.

Phenolphthalein test solution.—Dissolve 1 Gm. of phenolphthalein in 100 mils of alcohol. It gives a red color with alkali hydroxides or carbonates, and acids render the solution colorless. It is not suitable as an indicator for ammonia nor in the presence of large quantities of ammonium salts. Phenolphthalein paper is prepared by impregnating white, unsized paper with the test solution and drying it.

Potassium carbonate test solution, $\text{K}_2\text{CO}_3$.—Dissolve 10 Gm. of potassium carbonate in sufficient distilled water to measure 100 mils.

Potassium chloride test solution, $\text{KCl}$.—Dissolve 10 Gm. of potassium chloride in sufficient distilled water to measure 100 mils.

Potassium chromate test solution, $\text{K}_2\text{CrO}_4$.—Dissolve 10 Gm. of potassium chromate in sufficient distilled water to measure 100 mils.

Potassium cyanide test solution, $\text{KCN}$.—Dissolve 5 Gm. of potassium cyanide in sufficient distilled water to measure 100 mils.

Potassium dichromate test solution, $\text{K}_2\text{Cr}_2\text{O}_7$.—Dissolve 10 Gm. of potassium dichromate in sufficient distilled water to measure 100 mils.
Potassium ferricyanide test solution, $K_3Fe(CN)_6$.—Dissolve 1 part of potassium ferricyanide in about 10 parts of distilled water. This solution should be freshly made when required, as it undergoes decomposition with formation of ferrocyanide on standing. A freshly prepared aqueous solution mixed with ferric chloride T.S. which has been well diluted with distilled water shows a brown tint, free from turbidity or a shade of green.

Potassium ferrocyanide test solution, $K_4Fe(CN)_6 + 3H_2O$.—Dissolve 10 Gm. of potassium ferrocyanide in sufficient distilled water to measure 100 mls.

Potassium hydroxide test solution, KOH.—Use the official solution of potassium hydroxide containing approximately 5 per cent. of KOH.

Potassium iodide test solution, KI.—Dissolve 20 Gm. of potassium iodide in sufficient distilled water to measure 100 mls, and preserve the solution in dark, amber-colored, well-stoppered bottles. The solution should be frequently renewed.

Potassium sulphate test solution, $K_2SO_4$.—Dissolve 1 Gm. of potassium sulphate in sufficient distilled water to measure 100 mls.

Potassium sulphocyanate test solution, KCNS.—Dissolve 1 Gm. of potassium sulphocyanate in sufficient distilled water to make 100 mls.

Silver nitrate test solution, AgNO$_3$.—Dissolve 2 Gm. of silver nitrate in sufficient distilled water to measure 100 mls.

Sodium acetate test solution, NaC$_2$H$_3$O$_2 + 3H_2O$.—Dissolve 10 Gm. of sodium acetate in sufficient distilled water to make 100 mls.

Sodium carbonate test solution, Na$_2$CO$_3 + H_2O$.—Dissolve 10 Gm. of monohydrated sodium carbonate in sufficient distilled water to measure 100 mls.

Sodium cobaltic nitrite test solution, $(NaNO_2)_6 Co_2(NO_2)_6 + H_2O$.—Dissolve 4 Gm. of cobaltous chloride and 10 Gm. of sodium nitrite in about 50 mls of distilled water, add 2 mls of acetic acid and dilute with sufficient distilled water to measure 100 mls. A few drops of acetic acid should be added to the solution from time to time. The reagent must not be kept longer than three months. Should any precipitate form on standing, filter.

Sodium cyanide test solution, NaCN.—Dissolve 1 Gm. of sodium cyanide in sufficient distilled water to measure 10 mls. The solution must be freshly prepared when required.

Sodium hydroxide test solution, NaOH.—Dissolve 10 Gm. of sodium hydroxide in sufficient distilled water to measure 100 mls.

Sodium hypobromite test solution, NaBrO.—To a solution of 40 Gm. of sodium hydroxide in about 150 mls of distilled water add 10 mls of bromine and, after solution has taken place, add sufficient distilled water to measure 200 mls. The solution must be freshly prepared when required for use.
Sodium nitroprusside test solution, Na$_2$FeNO(CN)$_5$ + 2H$_2$O.—Dissolve 1 part of sodium nitroprusside in 19 parts of distilled water immediately before using.

Sodium phosphate test solution, NaHPO$_4$ + 12H$_2$O.—Dissolve 10 Gm. of sodium phosphate, in clear crystals, in sufficient distilled water to measure 100 mils.

Sodium tartrate test solution, Na$_2$C$_4$H$_4$O$_6$ + 2H$_2$O.—Dissolve 16 Gm. of sodium tartrate in sufficient distilled water to measure 100 mils.

Sodium thiosulphate test solution, Na$_2$S$_2$O$_3$ + 5H$_2$O.—Dissolve 2.5 Gm. of sodium thiosulphate in sufficient distilled water to measure 100 mils.

Stannous chloride test solution, SnCl$_2$ + 2H$_2$O.—Dissolve 10 Gm. of stannous chloride crystals in 100 mils of distilled water to which a small amount of hydrochloric acid has been added, and preserve the solution in glass-stoppered bottles in which a fragment of tin has been placed. The solution must be renewed at frequent intervals.

Stannous chloride test solution, saturated, SnCl$_2$ + 2H$_2$O.—(For Bettendorf's test for arsenic.) A saturated solution of stannous chloride crystals in concentrated hydrochloric acid. After filtering the solution through asbestos it has not more than a pale yellow color.

Starch test solution.—Triturate 1 Gm. of cornstarch with 10 mils of cold distilled water, add boiling distilled water with constant stirring to make about 200 mils, then boil the mixture for a few minutes until a thin, translucent fluid is obtained. This solution must be freshly prepared when required.

Sulphuric acid test solution, H$_2$SO$_4$.—Use the official diluted sulphuric acid, containing about 10 per cent. of absolute H$_2$SO$_4$.

Tartaric acid test solution, H$_2$C$_4$H$_4$O$_6$.—Dissolve 1 part of tartaric acid in 3 parts of distilled water. This solution must be frequently renewed.

Turmeric tincture—Digest any convenient quantity of ground turmeric root repeatedly with small quantities of distilled water and discard the liquids. Then digest the dried residue for several days with six times its weight of alcohol, and filter.

Turmeric paper.—Impregnate white, unsized paper with the tincture, and dry it. The tincture, as well as the paper, turns brown with alkalies, and the original yellow color is restored by acids, with the exception of boric acid, which, especially in the presence of hydrochloric acid, turns the color to reddish-brown, which is changed to bluish-black by ammonia.

Zinc sulphate test solution, ZnSO$_4$.—Dissolve 10 Gm. of zinc sulphate in sufficient distilled water to measure 100 mils.
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