HAND BOOK OF QUALITATIVE CHEMICAL ANALYSIS

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Hand Book of Qualitative Chemical Analysis by Robert C. Kedzie

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ROBERT C. KEDZIE

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Trieste

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OF

QUALITATIVE CHEMICAL ANALYSIS.

SELECTED AND ARRANGED FOR THE

Students of the State Agricultural College of Michigan,

BY

ROBERT C. KEDZIE, A.M., M.D.,

PROFESSOR OF CHEMISTRY.

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INORGANIC CHEMICAL ANALYSIS.

THE object of Chemical Analysis is to ascertain the chemical composition of any given body. Inorganic chemical analysis is divided into Qualitative Analysis and Quantitative Analysis. Qualitative Analysis is the separation of the chemical components of any substance, either in a separate form or in some known state of chemical combination. It is also used to identify simple or elementary substances by bringing them into chemical combination with other substances whose chemical composition is already known. Qualitative Analysis teaches us to separate and identify the components of any substance with regard to their quality only, without regard to their quantity, and answers the question, "*What* does the substance contain?" Quantitative Analysis furnishes the methods of procedure by which we determine the relations of weight or volume which these elements bear to each other, and answers the question, "*How much* of its several given elements does the substance contain?"

The methods of Qualitative Analysis consist in bringing the substance under examination into contact with other bodies of known properties, and observing the phenomena which ensue. These phenomena consist in alterations, either in state of aggregation, form or color, depending upon chemical change. By exhibiting the constituent parts of a substance of *unknown* composition, in forms of *known* composition, the constitution of the body examined, and the presence of its several component elements may be positively inferred. The separation and identification of substances are usually accomplished by physical changes. All bodies which we employ for the purpose of producing physical changes by chemical transformations we call *reagents;* and the resulting changes are called *reactions.* Acids, bases, salts, and elementary bodies are alike used as reagents. The reader will note a difference between *analysis* and testing: we test for any particular substance by eliciting some property peculiar to itself, such as a change of color (*e.g.*, the blood-red coloration formed by adding a sulphocyanide to a solution of ferric salt), or the production of a peculiar odor (*e.g.*, the garlic odor of the vapor of metallic arsenic).

By means of reagents the chemist interrogates the substance under examination, inquiring whether it contains this or that group of chemically similar elements, or only this or that member of such group. If the question be put correctly, that is, if all the conditions under which the reaction expected can be produced by the reagent employed, I e carefully observed, the answer is decisive as to the presence or absence of the element or group of elements sought But if the properties and chemical relations of the bodies formed by the chemical changes which constitute the reaction, have been wholly or partially neglected, the answer is at least of doubtful accuracy, if not certainly erroneous.

Chemical substances act upon each other more certainly and speedily when they are in the *fluid state*, because the contact of the molecules is then more complete, and that interchange of particles which constitutes the reaction more easily effected. The fluid state may be secured either by *solution* or by *fusion*. Reagents, therefore, may be employed either in the *wet way* or in the *dry way*. In the wet way the reagent in solution is brought into contact with the substance to be analyzed, which is usually in the liquid form. In the dry way the substance to be analyzed and the reagent are brought together in the solid state and subjected to a heat sufficient to melt he reagent, or both the reagent and the assay. At a high temperature chemical reactions are secured similar to those obtained by solution: certain changes are also secured by the addition or abstraction of oxygen, according as we use the oxidizing or deoxidizing flame. The knowledge derived in the dry way by the use of the blowpipe, and by the behavior of bodies in the different flames which can be produced by it, is one of great importance in Qualitative Analysis. This method is mainly employed in the preliminary examinantion, and the details of manipulation in the dry way are described in connection with the preliminary examination.

Many reagents exhibit the same or similar behavior with a group of chemically similar elements, and with most of the compounds of these elements; and can therefore be used to divide the elements into groups. Such reagents are called *group reagents*. Others serve for the further distinction of the several members of such groups, and are termed *special reagents*. Their selection depends upon the knowledge of the behavior of such reagents to each single element of the group, or of each of its several compounds. The number of special reagents is much greater than that of the group reagents, their nature being as various as that of the substances which come under examination. Their selection depends on the solubility or insolubility, color, or other physical or chemical properties of the new compounds to which they give rise. Certain special reagents produce reactions which are entirely characteristic of a given substance or compound. Such reagents and reactions are distinguished by being printed in SMALL CAPS.

It is the task of the analyst not only to establish that this or that body is present in a compound, but he is to prove that no other body is present beside those which he has actually found. Hence it is evident that he must not treat the substance under examination with reagents indiscriminately. He must follow a certain fixed order, a methodical system in the application of reagents. This systematic method consists in the employment of group reagents for the successive separation of groups of elements possessing certain common chemical properties, and finally, in the recognition of each member in such group by the use of special reagents which are characteristic.

The first thing to be done in the Qualitative Analysis of a solid body is to subject it to a preliminary examination in the dry way, whereby important information as to its composition may often be obtained. The substance is then dissolved and its constituents determined by analysis in the wet way. The course of Qualitative Analysis therefore embraces three parts:

I. Preliminary examination in the dry way.

II. Solution, or conversion into the liquid form.

III. Analysis of the solution in the wet way.

TABLE I.

The substance under examination is non-metallic, being neither a metal nor an alloy.

Reduce the substance to a fine powder in a porcelain mortar.

FIRST STEP.

Place a few grains of the substance in a clean, dry test tube, and heat, at first gently, but if no change ensues, to a full red heat, and watch the changes.

Substance unaltered.	Absence of organic matter; volatile bodies; fusible bodies; salts containing water of crystallization; salts containing interstitial water; substances whose color is changed by heat.
Changes color.	Many metallic oxides; White to yellow, h., to white, c., ZnO. White to yellowish brown, h., to dirty white, c., SnO_2. Yellow to brownish red, h., to yellow, c., PbO. White to orange red, h., to yellow, c., Bi_2O_3. Red to black, h., to red, c., Fe_2O_3. Brown to black, h., to red, c., $Fe_2O_3.3H_2O$. Black after intense heating, salts of Co. and Cu.
Aqueous vapors are expelled.	Substances containing water of crystallization; holding water mechanically enclosed (decrepitate); decomposable hydrates (alum and borax intumesce.)
Sublimate forms.	Gray tarnish or distinct globules easily united, Hg. Substance melts, sublimes, giving crystalline sublimate white, c.,—probably $HgCl_2$. Sublimes without melting, sublimate yellow, h., white, c., Hg_2Cl_2. White sublimate without melting or changing color, evolving ammoniacal odor and the vapor changing red litmus to blue when heated with Na_2CO_3—some salts of ammonia; White sublimate without melting or changing color, octrohedral crystals (use lens) As_2O_3; Substance melts, emits dense white vapors, forming crystalline sublimate which gives a strong acid reaction with blue litmus paper, $H_2C_2O_4 2H_2O$.; Reddish-brown drops, S.; Black sublimate, becoming red by rubbing with glass rod, HgS.
Carbonization.	Organic bodies; Cyanogen compounds. Blackening is not necessarily carbonization—usually a burnt odor and escape of combustible gases. The organic substances burn vividly when heated in test tube with KNO_3. with formation of K_2CO_3.

TABLE I.—First Step, Continued.

Gases escape when strongly heated.	O.—from Nitrates, Chlorates, Bromates, Iodates, and peroxides (ignites spark on splinter); SO_2—from Sulphates, Sulphites, Hyposulphites, or oxidation of Sulphur or Sulphides (odor of burning sulphur); NO_2.—from decomposition of the Nitrates of all metals except the alkalies (reddish-brown fumes); CO_2.—from decomposition of carbonates (lime water); CN,—from decomposition of metallic Cyanides (odor and peach blossom flame); H_2S.—from Sulphides containing water (odor and lead paper); NH_3,—from salts of ammonia, and some Cyanogen compounds (odor and alkaline reaction with red litmus paper).

SECOND STEP.

A moistened Platinum wire is dipped into the powdered substance and strongly heated in the reducing flame. (Should the previous examination indicate the presence of any fusible metal, *e.g.*, Pb, Sb, Bi., the experiment under this step should be omitted entirely, or a thread of asbestos used in place of the Platinum wire.)

Colors the flame	Yellow	Sodium. Molybdic troxide—greenish yellow. Substances rich in carbon.
	Red	Strontium (carmine.) Calcium (orange red.)
	Green	Copper (excepting $CuCl_2$) bright green. Barium, yellowish green. Molybdic acid, yellowish green Boric acid, " " Phosphoric acid, bluish "
	Blue	Lead. Arsenic. Chloride, Bromide and Iodide of Copper. Antimony (greenish blue.)
	Violet	Potassium (easily obscured.) Cyanides (peach-blossom.)

THIRD STEP.

A few grains of the substance are placed on charcoal and heated in the reducing flame.

Volatile (without incrustation or the formation of metalic globule.)	Salts of NH_4; As_2O_3 (odor of garlic); Some compounds of Hg.

TABLE I.—THIRD STEP, CONTINUED.

Volatile (or partially volatile) with incrustation.	Incrustation yellow, c.—PbO Incrustation dark yellow, c.—Bi_2O_3. Incrustation white, c.—Sb_2O_3. Incrustation blue and yellow—MoO_3. Incrustation reddish-brown, or a variegated tarnish—CdO. Incrustation yellow, h. white, c.—ZnO. Incrustation difficult to volatilize, yellowish, h. dirty white, c.—SnO_2.

NOTE.—Many substances which sublime in the test-tube under *First Step*, will give an incrustation when treated on charcoal, but these incrustations should be disregarded, and the indications from the sublimation followed.

Fusible, non-volatile, absorbed by the coal or forms a bead. (Residue pressed on moistened turmeric paper stains it brown.)	Compounds of { K. Na. Si. Ba. Sr. Some Salts of Ca.
Infusible and color unchanged.	SiO_2. Al_2O_3. Some compounds of { Ba. Sr. Ca. Mg. Luminous when heated intensely.

FOURTH STEP.

Place some of the substance on charcoal and heat in Reducing flame, moisten with a drop of $Co2(NO_3)$, then heat again intensely.

Colored Mass—(color best observed when assay is cold.)	Blue glass { alkaline Phosphates, " Borates, " Silicates Blue mass. (infusible). { Al_2O_3, SiO_2—(pale blue) Green { ZnO. yellowish green c. SnO_2. bluish green c. Sb_2O_3 dirty green c. Pink MgO.

FIFTH STEP.

Mix some of the finely powdered substance with twice that amount of dry Na_2CO_3. place on charcoal and heat strongly in the Reducing flame, (a mixture of KCy and Na_2CO_3 is a powerful reducing agent and should be used in treating compounds of Sn.)

Metallic globule with incrustation.	White incrustation. Globule easily formed and volatilized—brittle—Sb. Slight white incrustation. Globule formed with difficulty, malleable, Sn. Yellow incrustation. Globule easily formed—soft, very malleable. Pb. Yellow incrustation. Globule easily formed, very fusible, brittle. Bi.
Metallic globule without incrustation.	Red globule, Cu. Yellow globule, Au. Silvery-white globule, Ag. } Malleable.
Metallic powder or grains without incrustation.	Infusible and Magnetic, Fe.Ni.Co. Infusible and non-Magnetic, Pt.Ir.W.
Incrustation without metallic grains or globule.	White, very volatile, odor of garlic--As. Yellow h. white c.—Zn. Dark red, or variegated--Cd.