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EXPERIMENTS

FOR

STUDENTS IN GENERAL CHEMISTRY

SMITH AND KELLER.



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Chemistry

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EXPERIMENTS

ARRANGED FOR

STUDENTS IN GENERAL CHEMISTRY

BY

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SECOND EDITION, ENLARGED, WITH 37 ILLUSTRATIONS.



PHILADELPHIA:
P. BLAKISTON, SON & CO.

1012 WALNUT STREET.

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P R E F A C E .

This little work is designed as a guide for beginners in chemistry. The arrangement of the course is such as the authors have used with success in the instruction of their classes; its object is not to dispense with the supervision of an instructor, but rather to assist him. Although reference is made to Richter's "Inorganic Chemistry," any other text-book on the subject can be employed in its stead. The experiments have been collected from various sources, and no claim is made for originality.

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NON-METALS.

CHAPTER I.

APPARATUS, MANIPULATIONS AND OPERATIONS.

(1) *The Bunsen burner and the blowpipe.*

1. Make a borax bead. 2. Dissolve a very minute quantity of manganese dioxide in it. 3. Heat in the oxidizing flame (?). 4. In the reducing flame (?). 5. Heat oxide of lead on charcoal in the reducing flame. 6. In the oxidizing flame.

(2) *Working with glass tubing and rods.*

1. Cut various lengths of rods and tubing. 2. Round the sharp edges by softening and turning the ends in the lamp.

(3) *Construct a wash-bottle (Fig. 1).*

1. Soften a sound cork by rolling it under your foot on a clean floor. 2. Bore two parallel holes through it by means of a cork-borer. These perforations should be cylindrical and of less diameter than the glass tubes they are to receive. Use a rat-tail file in enlarging them. 3. Cut suitable lengths of glass tubing. 4. Draw the longer one to a fine point after softening in the flame. 5. Bend the tubes in an ordinary fish-tail burner, and round the sharp edges. 6. Fit the different pieces together.

(4) *Arrange some other form of apparatus for practice.*

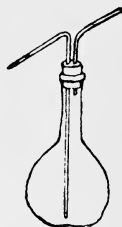
(5) *The balance.*

1. Weigh an object by placing it on the left-hand pan of the balance, and a weight judged about equal on the right-hand pan. Should the latter be found too heavy, replace it by the next smaller one; if too light, by the next heavier one. Then add systematically the smaller weights, until the needle points to the middle of the scale. The final adjustment is made with the rider. In adding or removing weights, the supports must always be raised.

(6) *Measuring vessels.*

1. Measure off 10 cc. of water (*a*) in a cylinder, (*b*) in a burette, (*c*)

FIG. 1.



in a pipette. Always read the *lower* meniscus. 2. Measure off similarly 50, 100 and 200 cc. of water, and determine their weight. 3. Measure the volume of 50 grms. of oil of vitriol, and of 65 grms. of muriatic acid. What are the specific gravities of these substances? Note the relation between weight and volume in the metric system.

(7) *Chemical operations*: Solution, evaporation, crystallization, precipitation, filtration, washing and drying.

1. Place into a test-tube pure sodium carbonate, into another cobalt chloride, and add distilled water to each. Stir. What occurs? 2. To calcium carbonate, add water. Is there any change? Now add a little hydrochloric acid. What action has it? 3. Pour 5 cc. of strong hydrochloric acid upon powdered manganese dioxide; observe appearance and odor. Note, too, in each case, whether heat has any effect. Distinguish between chemical and mechanical solution. 4. Heat the cobalt chloride and the calcium carbonate solutions, each in a separate dish, on an iron plate, until the liquids are completely driven off (?). 5. Dissolve potassium chlorate in hot water, and allow to stand and cool (?). 6. To a portion of the cobalt chloride solution, add a solution of soda; boil. 7. Allow to settle and filter. 8. Wash the precipitate until pure water runs through the filter (?). 9. Heat the filter until perfectly dry.

CHAPTER II.

GENERAL PRINCIPLES.

(1) *Changes in matter.*

1. Rub a glass rod with a piece of cloth, then touch particles of paper with it (?). 2. Through an insulated spiral of stout copper wire pass a current from two Bunsen cells. Place a piece of wrought-iron—a nail will answer—inside the spiral, and bring iron filings in contact with it. What happens? Interrupt the current and note the result; repeat. 3. Heat a platinum wire in the non-luminous flame; is there any change? What is the effect of removing it?

Are the original properties of the substances in the above experiments altered, after the action of the forces of electricity, magnetism and heat has been stopped?

4. Mix intimately four parts, by weight, of finely powdered sulphur with seven parts of very finely divided iron (filings). Pass a magnet over a portion of the mixture. Another portion treat with carbon disulphide in



a test-tube. Then heat the remaining portion in a tube over a gas flame. Note carefully what occurs in each case. Powder the mass resulting from the last operation in a dry mortar. Can you extract from it any iron with a magnet, or dissolve out any sulphur with carbon disulphide? What inference do you draw from the facts observed? 5. Decompose water in Hofmann's apparatus by an electric current. The water should be acidulated with sulphuric acid to make it a conductor of electricity. A current from four to six Bunsen cells is required. To the gas, of which a larger volume has collected, apply a flame, and to the other a glowing spark (?) 6. Heat oxide of mercury in a tube of hard glass (*Fig. 2*). Apply the spark test (?). 7. Rub some sulphur and mercury together in a mortar (?) 8. Heat sugar in a dry test-tube, at first gently, and then more strongly. Note color and odor. 9. Mix dry soda and tartaric acid in a mortar. Is there any action? What occurs when you add water? Point out in what respect the changes involved in experiments 1-3 differ essentially from those in 4-9. By what general names can you distinguish the two different kinds? With which does chemistry concern itself? Define *chemistry*.

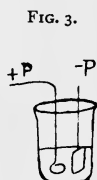


Through what agencies have the results been obtained in experiments 4-9? Has any gain or loss of matter occurred in any of them?

(2) The products resulting from 5 and 6 cannot be further simplified, *i. e.*, decomposed into dissimilar substances. They are *elements*.* What are water and red oxide of mercury?

1. Dissolve in a little nitric acid, the black powder obtained by heating an intimate mixture of powdered sulphur and copper filings.†

Evaporate the solution nearly to dryness, take up in water and filter. What remains on the filter? Place the filtrate in a beaker, dip the platinum electrodes of a battery (one or two Bunsen cells) into it (*Fig. 3*), and allow the current to act for ten minutes. What do you observe upon the platinum foil, forming the negative pole? What changes have the copper and the sulphur undergone in this experiment?



(Study pp. 18-27, in Richter's Chemistry.)

(3) *Metals and non-metals*. (See Richter, p. 20.)

* The instructor should here develop the idea of *element* more fully.

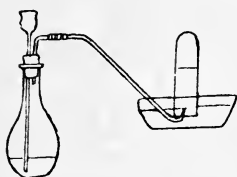
† A better substitute would be finely divided copper; such as may be obtained by the reduction of black cupric oxide in a current of hydrogen gas (see page 14).

CHAPTER III.

HYDROGEN.—H.

(1) Put several pieces of granulated zinc into a test-tube and pour dilute sulphuric acid upon them. What occurs?

FIG. 4.

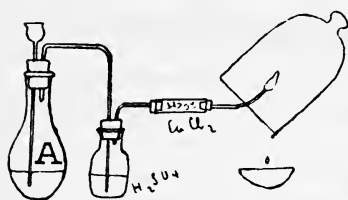


(2) Arrange the apparatus shown in Fig. 4. The flask should contain about 15 grms. of Zn, and dilute H_2SO_4 is poured through the funnel tube. When all the air in the apparatus has been displaced (*ask for precautions!*) collect six test-tubes full of the gas over water.

(3) What are its properties? Will it burn? Support combustion? Is it lighter than air?

(4) 1. To learn what becomes of hydrogen when it burns in air, arrange apparatus as in Fig. 5. The gas is led from the evolution flask *A*, into a bottle containing concentrated H_2SO_4 , and then passes through a tube filled with pieces of $CaCl_2$. The gas which escapes is free from moisture. Burn it under a cold glass jar. What do you obtain? 2. Fill a small flask with a mixture of one vol. of H and five vols. of air; cork; invert the flask several times to mix the gases; wrap a towel around it and bring its mouth to a flame. Result?

FIG. 5.

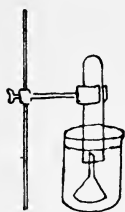


(5) *Hydrogen is not the only product of the action of H_2SO_4 upon Zn.*

Pour some of the liquid remaining in the flask, in which H was generated, into a porcelain dish. Evaporate to about one-third of the original bulk; allow to stand several hours. You will now discover that the solution is full of colorless crystals. These are zinc sulphate or white vitriol—a salt, $ZnSO_4 + 7H_2O$. Write the equation of the reaction.

(6) *Determine the weight of H generated by a given weight of Zn.*

FIG. 6.



A piece of Zn (not more than .02 gm.) is accurately weighed, and placed under a funnel in a beaker (Fig. 6). The latter is then nearly filled with water, so that the entire funnel is under the surface. A test-tube containing dilute H_2SO_4 is lowered over the stem of the funnel. Hydrogen appears and collects in the tube. When all the Zn has disappeared,* transfer the tube containing the H to a larger vessel, holding water. Measure the volume of the gas by marking

* This may be hastened by bringing a spiral of platinum wire in contact with the Zn.

the tube where the inner and outer levels of water are even, and then weighing or measuring the quantity of water that it will hold to that mark. Note the temperature of the water, and the height of the barometer.

The weight of the H is found by multiplying the vol. by the wt. of 1 cc., *i.e.*, .0000896 gr. Before this can be done, however, it is necessary to reduce the volume of the gas to 0° C. and 760 mm., as the above value has been determined under these conditions. If v = volume observed, t = temperature, and p = pressure, then

$$v_0 = \frac{v \times p}{(1 + at) \times 760}$$

and $W = v_0 \times .0000896$. $a = .003665$.*

To calculate the quantity of Zn necessary to generate a unit of H, we say—

$$\text{Wt of H : Wt of Zn :: 1 : } x.$$

x here stands for the *equivalent weight* of Zn.

The equivalent weights of some other metals, such as Fe, Cd, Mg, can be determined in the same manner. Magnesium gives the most satisfactory results.

(7) *Decompose water by electrolysis* and test the products.

(8) Take a small piece of sodium, wrap it in paper and place it, with forceps, under the mouth of a test-tube filled with water, and inverted in water (Fig. 7) contained in a dish. Repeat this until the test-tube is filled with the gas. Test it for H.

What becomes of the metal? Write the reaction.

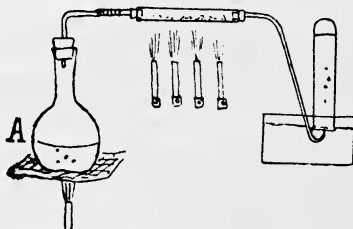
(9) Construct the apparatus shown in Fig. 8.

Water is heated to boiling in the flask *A*, and the steam led over iron filings or wire, heated to redness in a tube of hard glass. Care must be taken to prevent steam from condensing in any part of the tube. Collect the escaping gas over water. Test it for H.

FIG. 7.



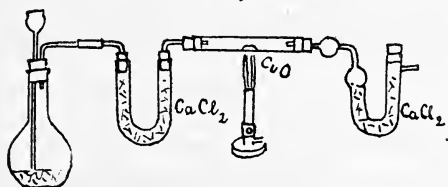
FIG. 8.



* Tension of aqueous vapor is here neglected.

Is the iron changed? Equation?

FIG. 9.



(10) Into a weighed tube of hard glass (6–8 inches in length) place a weighed quantity (1–2 grams) of cupric oxide; connect the tube with a CaCl_2 -tube of known weight (Fig. 9). Pass a current of dry H over the CuO and heat.

After the change is complete, cool and determine the loss in weight of tube + CuO , and the gain in the CaCl_2 tube. Explain the reaction.

Problems.—1. How much H can be obtained from Zn and 299 grms. of H_2SO_4 ? 2. How much Zn and H_2SO_4 are necessary to furnish 100 grms. of H? 3. Suppose you have found that .015 gm. of Mg yield 15.2 cc. of H at 20°C . and 750 mm., what is the equivalent weight of that metal? 4. How many cc. of H can be obtained from 2 grms. of Na and water? 5. How many grms. of H_2O can be decomposed by 5 grms. of Fe; by how much is the weight of the latter increased? 6. 10 grms. of CuO will yield how much Cu upon heating in H?

Give a brief summary of what you have learned about H.

(Study Richter, pp. 39–47.)

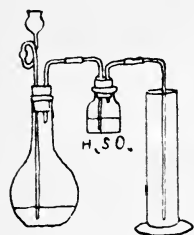
CHAPTER IV.

FIRST NATURAL GROUP OF ELEMENTS—CHLORINE, BROMINE, IODINE, FLUORINE.

CHLORINE.—Cl.

(1) Into a test-tube put MnO_2 and concentrated HCl. Note what happens both before and after heating.

FIG. 10.



(2) Use apparatus (shown in Fig. 10) for preparing larger quantities of Cl. The MnO_2 should be in the form of small lumps (not powder.) Heat the mixture gently, pass the Cl through a small quantity of water and collect it either by downward displacement or over *warm* water.

Write the reaction. How many atoms of Cl are liberated? How many molecules?

(3) 1. What is the normal condition of this element? 2. Is it lighter than air? 3. Is it inflammable? 4. Does it support combustion?



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To obtain answers to these questions, fill 5 test-tubes with dry Cl, and proceed as under H.

(4) Again fill 5 bottles with the dry gas. Cover them with glass plates. Into 1 throw a little pulverized antimony.

Into 2 carefully introduce a piece of phosphorus.

Into 3 insert tissue paper moistened with oil of turpentine.*

Into 4 introduce colored flowers.

Into 5 pour a little litmus solution.

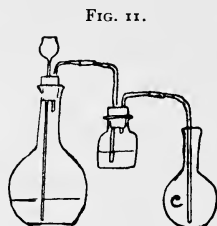
What are the results?

(5) Fill a small-sized flask one-half with chlorine, the other half with hydrogen. Wrap a towel about the flask and apply a flame to its open mouth (?). *Care.*

(6) Invert a bottle filled with Cl over water saturated with the same gas. What follows in the course of a few hours' exposure to sunlight? Can you account for results in experiments (4), (5) and (6)? Why should the gas be collected over *warm* water?

(7) *Determine the weight of a litre of chlorine.* Arrange apparatus as shown in Fig. 11.

In the evolution-flask place a mixture of equal weights of salt and manganese dioxide. Add sulphuric acid, previously diluted with its own volume of water (pour the acid into the water!). Heat gently. Chlorine is evolved, and dried by passing it through concentrated sulphuric acid, after which it is led into the perfectly dry flask *c*. When this is filled, which you ascertain by the color of the gas in the neck, slowly withdraw the tube and cork the flask at once. Weigh the flask. Read the barometer and thermometer. Determine, also, the weights of the flask filled with air and with water.



Calculation:

Capacity of flask,	a
Temperature,	t
Pressure,	p
Flask filled with air,	w
“ “ Cl,	w'
Wt. of a litre of air,	1.293 gm.
“ “ Cl,	x

* It is well, when the turpentine is old, to gently warm it, and then saturate the tissue paper.

The weight of the air filling the flask is $\frac{a \times p \times .001293}{(1 + .00367 t) 760}$. The difference between this and w is the weight of the vacuum flask. Subtract this from w' . The remainder is the weight of the Cl, (W). Reduce the vol. of the Cl to 0° C. and 760 mm. (see under H); it is $v_0 = \frac{a \times p}{(1 + .00367 t) 760}$, and the weight of 1 litre, $x = \frac{W \times 1000}{v_0}$.

How much heavier is one litre of Cl than an equal vol. of H?

Write the reaction involved in the above method for preparing Cl.

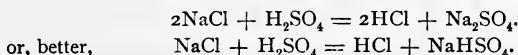
Problems.—1. How many litres of Cl can be obtained from 1 kilo of MnO_2 and HCl? 2. What weight of salt is required to prepare 100 litres of Cl? 3. How many pounds of sodium sulphate and manganese sulphate will be formed in the preparation of 100 litres of chlorine gas? 4. Calculate the number of grams of Cl that 2 litres of water will absorb, provided the latter takes up twice its volume of Cl? Write out your deductions from the above experiments on Cl.

(Read Richter, pp. 49-52.)

HYDROGEN CHLORIDE.—HCl.

(1) Repeat the explosion of equal vols. of Cl and H. Quickly cover the mouth of the flask, and immerse it under water. Does the latter rise? Put a drop of the liquid on the tongue and note the taste. Add some blue litmus solution. Is there any change?

(2) The product of the union of H and Cl is a colorless gas. It is called *hydrogen chloride*. It is usually prepared by the action of sulphuric acid upon salt, thus:—



The apparatus employed here is the same as that used for making Cl (Fig. 10).

(3) *Determine the properties of HCl as under H and Cl.*

What new property appears here? Fill a long dry glass tube with the gas, and quickly bring it into a basin containing water colored blue with litmus. What happens? What does HCl gas yield on dissolving in water?

(4) In the preparation of H by the action of Na upon water, it was observed that the liquid became soapy to the touch, and acquired the property of turning red litmus blue. Prepare such a solution. To it add a few drops of litmus, and then an HCl solution (gradually) from a

burette, until the blue color just begins to turn. Evaporate the resulting liquid to crystallization. Dissolve and recrystallize the product. It appears in cubes, and has the taste of common salt. It does not affect either red or blue litmus. We say it is *neutral* in reaction. The substance is chloride of sodium or common salt. What is a salt? An acid? A base? How can you obtain HCl and Cl from NaCl?

(5) *Burn H in an atmosphere of Cl, and Cl in hydrogen.*

Generate chlorine as already described (p. 14) and collect it in a large cylinder. Into this introduce a burning jet of hydrogen (Fig. 12). Does it continue burning? What is the appearance of the flame? To show the combustion of Cl in H arrange apparatus as in Fig. 13.

(6) To determine *the weight of a litre of HCl*, proceed exactly as under chlorine.

(7) *Determine the composition of HCl by volume.*

1. Fill a perfectly dry and graduated tube with HCl. Close the open end with the thumb, and opening the tube for a moment, quickly pour in about 10 cc. of sodium amalgam (see sodium, p. 36). Close the tube at once with the thumb, slightly moist, and shake well. Invert the tube in a large beaker of water, and remove the thumb. The amalgam will drop into the water, and the latter will rush up into the tube, filling it nearly half full. Immerse the tube so that the water in it and that in the beaker are on the same level. This is done to measure the hydrogen under atmospheric pressure. Read the residual volume of the gas and measure the volume of the mercury.

Calculation :—

Capacity of tube,	a
Vol. of mercury,	b
Vol. of H.,	c

$$c = \frac{a - b}{2}$$

(8) Add a solution of HCl to solutions of silver nitrate ; of mercurous nitrate ; and of lead acetate. What do you observe in each case? Boil the precipitate formed in the lead solution with water. Cool, and note result.

FIG. 12.

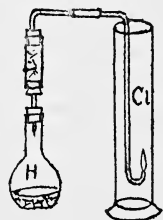


FIG. 13.

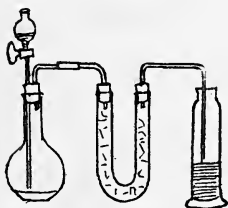


BROMINE.—Br.

(1) Allow a drop of bromine to fall upon a heated watch glass; cover it quickly with a beaker. What is the color of the vapor? Dissolve one drop of bromine in each of the following solvents contained in test-tubes: water, alcohol, ether, carbon disulphide, and chloroform. Note the relative solubilities, and the color of each solution.

(2) 1. Pass Cl through an aqueous solution of potassium bromide. What happens? 2. To one portion of the product add a few drops of CS_2 , and agitate the mixture; what is the result? 3. To another portion of the solution, containing free Br, add a few drops of a starch solution.* Result?

FIG. 14.



(4) Devise a method for preparing bromine from KBr.

(5) *Prepare hydrobromic acid.*—In a small flask cover 2 grams of amorphous phosphorus with 4 grams of H_2O , and from a funnel, provided with a stop-cock, gradually allow 20 grams of Br to run in.†

The gas is purified by conducting it through a U-tube, containing moistened pieces of phosphorus and glass (Fig. 14), and led into water to obtain the aqueous solution.

(6) Add aqueous HBr to solutions of AgNO_3 , HgNO_3 and $\text{Pb}(\text{NO}_3)_2$ —do the resulting bromide precipitates differ much from the corresponding chlorides?

IODINE.—I.

(1) 1. Place an iodine crystal upon a warm plate, and note color of vapor. 2. Test the solubility of iodine in the same solvents as were used with bromine; what are the colors of the resulting solutions?

(2) 1. Pass Cl through a solution of KI. Test the resulting liquid with ether, carbon disulphide and starch solution (as with Br). 2. Repeat this experiment, substituting Br-water for the Cl. Avoid excess of Cl as well as Br (?).

What conclusion do you draw from these experiments relative to the affinity of the halogens for potassium?

* The starch solution necessary for this purpose can be prepared as follows: One gram of starch is well ground in a mortar, with very little water, to creamy consistence. It is then poured into 200 cc. of boiling water. Allow to subside, decant the clear supernatant liquid and use it for the test.

† As it is rather difficult to weigh bromine upon a balance, calculate the volume corresponding to the weight given and measure out the same in a cylinder.

(3) Pass hydrogen sulphide gas (H_2S) into 50 cc. of water, and add powdered iodine till the brown color no longer disappears. Warm, filter (?) and distil the filtrate. The product is what?

How is gaseous HI prepared?

(4) Precipitate solutions of silver nitrate ($AgNO_3$), mercurous nitrate ($HgNO_3$), lead nitrate ($Pb(NO_3)_2$), and mercuric chloride ($HgCl_2$) with KI. Note result in each case.

FLUORINE.—Fl.

(1) In a lead dish (or platinum crucible) place 1 gram of pulverized fluor spar ($CaFl_2$). Add conc. H_2SO_4 ; cover the dish or crucible with a watch-glass coated with paraffin, through which some characters have been drawn with a fine point. Heat gently for a few minutes.

What do you observe on removing the paraffin?

(2) Can you liberate Fl from a fluoride?

Problems.—1. How much NaBr, H_2SO_4 and MnO_2 are necessary to produce 1 cu. metre of Br vapor at $20^\circ C$ and 745 mm.? 2. What per cent. of HI does a liquid contain, which represents a solution of 50 litres of the gas in 1 litre of H_2O ? 3. 10 grms. of $CaFl_2$ will give what weight of HFl? 4. How much salt and sulphuric acid will be required to prepare 6 litres of muriatic acid of sp. gr. 1.17? What volume would the HCl in these six litres occupy at 735 mm. pressure and $22^\circ C$? 5. What is the percentage of hydrochloric acid in a solution of which 17 cc. dissolve exactly 2 grams of metallic magnesium? What is the volume of hydrogen liberated at 760 mm. and 0° ?

CHAPTER V.

SECOND NATURAL GROUP OF ELEMENTS—OXYGEN, SULPHUR, SELENIUM, TELLURIUM.

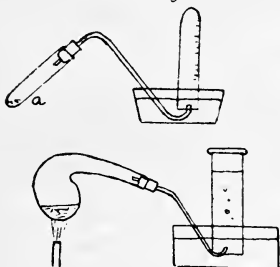
OXYGEN.—O.

(1) *Preparation.*—1. Weigh the hard glass tube *a* (Fig. 15), and introduce a weighed quantity (about .5 gm.) of red oxide of mercury. Ignite strongly; collect the liberated gas, and measure it. Weigh the tube with the residue. What are the products of the ignition?

(2) Prepare more of the gas, as follows: Mix equal parts of $KClO_3$ and pulverized MnO_2 ; heat in a tube of hard glass or small retort. Collect the gas in bottles over water (Fig. 15).

Into No. 1 lower a piece of ignited sulphur on an iron spoon. Note result. Add water after the combustion (?).

FIG. 15.



Into No. 2 introduce a small piece of burning phosphorus (care!). Proceed as in No. 1.

Into No. 3 introduce ignited charcoal. Treat as before. Add now a few drops of blue litmus to the contents of each bottle. Any change?

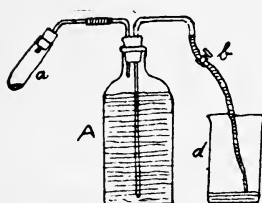
Into bottle No. 4 introduce a fine watch spring, previously heated at one end and dipped into powdered S. Result?

Is oxygen heavier or lighter than air? Has it color, taste, or odor? Will it burn? Does it support combustion?

What other methods can be used for preparing O?

(3) Determine the weight of a litre of O.

FIG. 16.



Arrange apparatus shown in Fig. 16; *a* is a tube of hard glass, whose weight is known; it contains a weighed amount of KClO_3 (about 0.3 gm.). The bottle *A* is filled with water, *b* is a clip and *d* a beaker. The exit tube should contain water as far as clip *b* at the beginning of the experiment. Open the clip, heat *a* to bright redness, and receive the water displaced by the

O in *d*. When no more gas is evolved, cool; allowing the rubber tube to dip under the water of the jar. Some of the water will be drawn back into the bottle (?). Measure the volume of the water in *d*. Note the temperature of the air, and the height of the barometer. Weigh *a*, containing residue (KCl).

Calculation:

Weight of the tube,	a
Weight of KClO_3 and tube,	b
Weight of KClO_3 ,	b - a
Volume of H_2O collected,	v
Barometric pressure,	p
Temperature,	t
Aqueous tension at t,	p'
Weight of KCl and tube,	c

$$V_0 = \frac{v \times (p - p')}{(1 + .00367 t)} \times 760 \quad \text{and} \quad x = \frac{(b - c) \times 1000}{.V_0}$$

Dissolve the residual KCl in water, and to its solution add nitrate of silver (?). How does KClO_3 behave under like conditions?



(4) Give a summary of your work upon O.

Problems.—1. How much O, by wt. and vol., can be obtained from 54 grms. of HgO ? 2. Heat will expel what vol. of O from 2.45 grms. of KClO_3 ? 3. How much HgO is necessary to yield 1 cu. d. m. of O? 4. How many times is O heavier than H?

OZONE.— O_3 .

(1) Pour water on clean pieces of phosphorus to half cover them; invert a large, clean jar over this and allow to stand for several hours. Test the air under the jar for ozone. For this purpose use paper impregnated with a mixture of starch paste and potassium iodide. What occurs? (Read Richter, p. 85–89.)

COMPOUNDS OF OXYGEN AND HYDROGEN.

WATER.— H_2O .

(1) Arrange the distillation apparatus (Fig. 17) and prepare about 100 cc. of distilled water. Note its taste and odor. Test it for chlorides with AgNO_3 . Does it leave a residue upon evaporation? What action has it on litmus?

Apply all these tests to a natural water (except rain).

(2) 1. Heat a little vegetable matter in a dry test-tube. 2. Heat fresh meat in the same manner. 3. Carefully heat crystals of zinc or copper sulphate in a test-tube. What happens in these experiments? 4. Expose clear crystals of sodium phosphate, on a watch-crystal to the air. 5. Do the same with pieces of calcium chloride. Results?

(3) *Determine the quantitative composition of water.*

1. The composition of water by weight follows from the experiment of reducing oxide of copper described under H.

2. The relative volumes with which O and H unite to form water, are determined either by analysis or synthesis. The former has been performed in electrolyzing water.

3. Fill a eudiometer (Fig. 18) with water. Through a rubber tube admit about 50 cc. of O and then a like volume of H. (If the eudiometer is not graduated, mark these with rubber bands.) Close the open end with your thumb, leaving some air to serve as a cushion beneath it, and pass the spark. Remove the thumb, and pour in enough water to

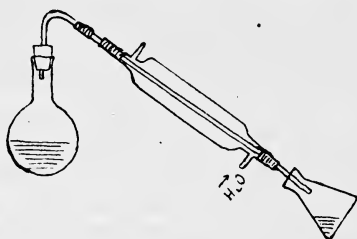
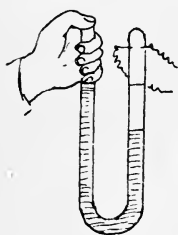


FIG. 17.

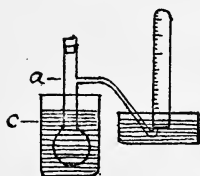
make the levels equal in both limbs. What is the amount of the contraction? What is the residual gas? Test it.

FIG. 18.



(4) *Determine the weight of a litre of steam.*—Construct apparatus shown in Fig. 19. The flask *a* is closed with a cork. *C* is a vessel containing melted paraffin. A small glass tube is weighed and filled with water (not more than .02 gm.). Heat the vessel *C* with a Bunsen burner until the temperature of *a* is constant (?). Now drop the tube containing the water through the mouth of the flask (the bottom of which should be protected with a layer of asbestos) and quickly re-cork. When the fall of water in the graduated tube ceases, read the volume of gas, and note the temperature and pressure of the air.

FIG. 19.



The calculation is analogous to that used under O.

(5) Perform experiment 2, p. 100 in Richter.

How many volumes of steam result from the combination of 2 vols. of H and 1 vol. of O?

How would you deduce the molecular formula of water from the preceding experiments?

HYDROGEN PEROXIDE.— H_2O_2 .

(1) Add moist hydrated barium peroxide to cold dilute H_2SO_4 . Filter. What does the filtrate contain?

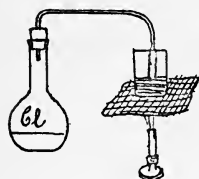
(2) 1. Add a solution of KI, containing starch, to a portion of this liquid (?). Ferrous sulphate hastens the reaction. 2. Cautiously add a dilute solution of potassium permanganate to another portion (?).

COMPOUNDS OF OXYGEN AND CHLORINE.

(1) Make a dilute solution of caustic potash, and conduct chlorine into it until the latter is no longer absorbed. Treat one portion of the product with HCl, and another with H_2SO_4 . What results?

(2) Mix 10 grms. of quicklime with 25 cc of H_2O . After the slaking is finished, conduct Cl into the mixture until it is no longer absorbed.

FIG. 20.



Add HCl to one portion and H_2SO_4 to a second portion.

What is set free? Does it bleach?

(3) Pass Cl into a hot *concentrated* solution of KOH till it ceases to be absorbed (Fig. 20). What separates upon cooling? Recrystallize the product from water. Will it give off O

upon heating? Try the action of HCl upon a crystal. Allow a drop of conc. H_2SO_4 to fall upon a *small* crystal and warm gently (?). Care!

Observe carefully the behavior of KClO_3 upon heating (?).

SULPHUR.—S.

(1) Place a few grams of powdered S in a dry test-tube, and heat gradually. Observe and describe the changes which occur.

(2) Dissolve a little S in CS_2 and allow to stand till the liquid has evaporated. What remains?

(3) Determine the sp. gr. of S (Fig. 21). Water, previously boiled is introduced into a flask. It is essential that the neck of the flask should be narrow. Weigh the flask, then place an additional 10 grm. weight upon the right-hand pan of the balance and small pieces of S upon the left-hand pan, until the pointer is again in the middle. Now introduce the S into the flask. Carefully remove water above the mark and re-weigh the flask with its contents. The loss in weight will represent the weight of a volume of water equal to that of 10 grms of S. The latter divided by the former is the specific gravity of the sulphur.

FIG. 21.



(4) Prepare the *monoclinic* modification of S by melting about 10 grms. of the ordinary variety in a covered Hessian crucible. Cool; and as soon as a solid crust has formed upon the surface, pierce it and allow the still liquid portion of the contents to run out. Note the shape of the crystals upon the sides of the crucible.

(5) To obtain the *plastic* variety, heat 10 grams of S in a test-tube above 230°C ., and pour the mass into cold water.

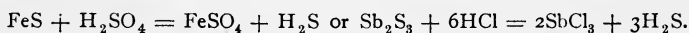
Test the solubility of the product in CS_2 . Preserve a portion of it for several days. Does it change?

(6) To a strong solution of yellow potassium sulphide, add HCl. What are the properties of the separated sulphur?

Give a brief outline of the element sulphur; compare it with the previously studied elements.

SULPHUR AND HYDROGEN.

(7) *Hydrogen sulphide* is formed with difficulty from its elements, but is readily obtained by the action of acids upon sulphides, thus:—



The apparatus to be used is the same as that employed in preparing hydrogen.

(8) What are the properties of H_2S ? Is it soluble in water? Does it

burn? What are its products of combustion? Hold a porcelain plate in the flame; what results? Pass the gas into solutions of chromic acid, permanganic acid, and ferric chloride. What changes are observed? How do these last-named reactions show its reducing power? What happens to the aqueous solution of the gas when exposed to the air? What action has the aqueous solution upon litmus? To what class of compounds does it, therefore, belong?

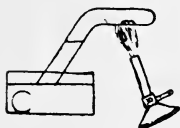
(10) Pass H_2S through solutions of the following salts, viz.:— $CuSO_4$, $SbCl_3$, $Pb(NO_3)_2$, $AsCl_3$, and $Zn(C_2H_3O_2)_2$. Note results carefully.

Can sulphides be prepared in another manner? (See Chap. II, § 1.)

(11) *Determine the composition of hydrogen sulphide.*

Into a bent tube of hard glass, filled with mercury, (Fig. 22), introduce dry hydrogen sulphide.* Place a piece of tin in the bent portion, and heat it. Is the volume of the gas changed after the experiment, and what becomes of the piece of tin? Test the gas remaining in the tube. Do your results enable you to deduce the molecular formula of H_2S ? (See Richter, 3d ed., p. 111.) Trace the similarity between H_2S and H_2O . Write a summary of your experiments on H_2S .

FIG. 22.



SULPHUR AND CHLORINE.

(12) 1. *Sulphur Monochloride*.—Prepare this compound by conducting dry chlorine over molten sulphur. The product which distils over is collected in a dry test-tube, kept cold by immersion in ice water. 2. Redistil the product. Determine its boiling point in an apparatus similar to that pictured in Fig. 23. Note the color and odor of the product. Expose some of it to the air on a watch-glass. Add water to another portion contained in a test-tube. Note carefully what happens. Write the reaction, and examine for all the products.

FIG. 23.



SULPHUR AND OXYGEN.

(13) Burn sulphur in the air. Result? Burn FeS_2 in the air. What are the properties of the resulting compound? It is *sulphur dioxide*— SO_2 .

(14) Fit a small flask, as indicated in Fig. 24. Place copper turnings in it, then add H_2SO_4 (strong) through the funnel tube. Warm. Is the product the same as that obtained in 13? Is it soluble in water? Has the aqueous solution the same properties as the gas? 2. Pass some of

* The instructor should perform this experiment.



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the gas into solutions of potassium dichromate and potassium permanganate acidulated with H_2SO_4 . Repeat these experiments with the aqueous solution instead of the gas. What happens in each case? 3. Test the aqueous solution of SO_2 with litmus. What is this solution commonly called? 4. Fill a dry jar with SO_2 gas; introduce colored flowers. Note the result.

(15) What is the formula of *sulphurous acid*? How many series of salts can it form? How would you designate the different sodium salts? Add HCl to a solution of Na_2SO_3 . What follows? Evaporate the solution to dryness and examine the residue. What is it? Write the reaction.

SULPHUR TRIOXIDE— SO_3 —and SULPHURIC ACID— H_2SO_4 . (Read Richter, p. 189).

(16) 1. Prepare sulphuric acid as described in Richter, p. 191. Study the product carefully. 2. Dilute a portion of it with water; what happens? 2. Test a portion of this diluted solution with litmus (?). 4. Another portion neutralize with $NaOH$ and evaporate. What is the residue? Does it contain any S? Prove this. 5. Add $BaCl_2$ to a third portion of the solution. What is the precipitate? Is it soluble in water or in hydrochloric acid? 6. What is the action of strong H_2SO_4 upon wood or paper? Explain the cause of this action.

(17) How many series of salts can sulphuric acid form. Prepare $(NH_4)_2SO_4$, Na_2SO_4 , $NaHSO_4$ and $CuSO_4$. (Read Richter, pp. 189–200).



CHAPTER VI.

NITROGEN GROUP—NITROGEN, PHOSPHORUS, ARSENIC, ANTIMONY AND BISMUTH.

NITROGEN.—N.

(1) *Preparation*.—1. In a dish swimming on water place a piece of phosphorus and ignite it; invert a beaker glass over it (Fig. 25). What becomes of the P? When the latter has ceased burning, restore the level of the water, and note the decrease in the volume of the air. Test the residual gas with a burning taper. 2. Heat gently in a small flask or retort a mixture of 1 part KNO_2 , 1 pt. NH_4Cl , 1 pt. $K_2Cr_2O_7$, and 3 pts. of H_2O ; collect the gas over water. Fill five bottles with this gas.

FIG. 25.



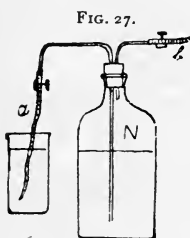
FIG. 26.



(2) Has it color, taste, odor? Does it burn or

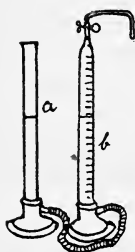
support combustion? Is the gas heavier than air? Does it unite readily with other elements?

(3) *Determine the weight of a litre of nitrogen.*—A round-bottomed flask is fitted, as shown in Fig. 26. Pour about 30 cc. of water into it, and insert the rubber cork to the mark. Boil the water, while the clip is open, until all the air has been expelled from the flask. Steam should be allowed to escape for about five minutes. Now close the tube with the clip, and remove the flame. Cool and weigh the flask. Read the temperature and barometric pressure in the balance-room.



Connect the flask with the tube, *b*, of the aspirator, containing N, and arranged as in Fig. 27. The rubber tube, *a*, is made to dip under water, and the clip is gradually opened, allowing N to enter the flask. Now raise the vessel containing the water into which the rubber tube dips, so that the water in it is at a higher level than that in the aspirator. Close the clip. Disconnect the flask and open the clip for a moment, to establish atmospheric pressure in the flask. Weigh. The calculation is identical with that given for O.

FIG. 28.



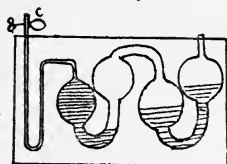
What is the ratio between the weights of equal volumes of N and H?

(4) Is *air* a chemical compound?

How would you determine the weight of a litre of air?

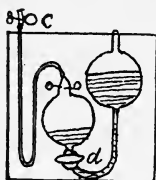
(5) 1. *Determination of the Oxygen in Air by the Pyrogallate Method.*—

FIG. 29.



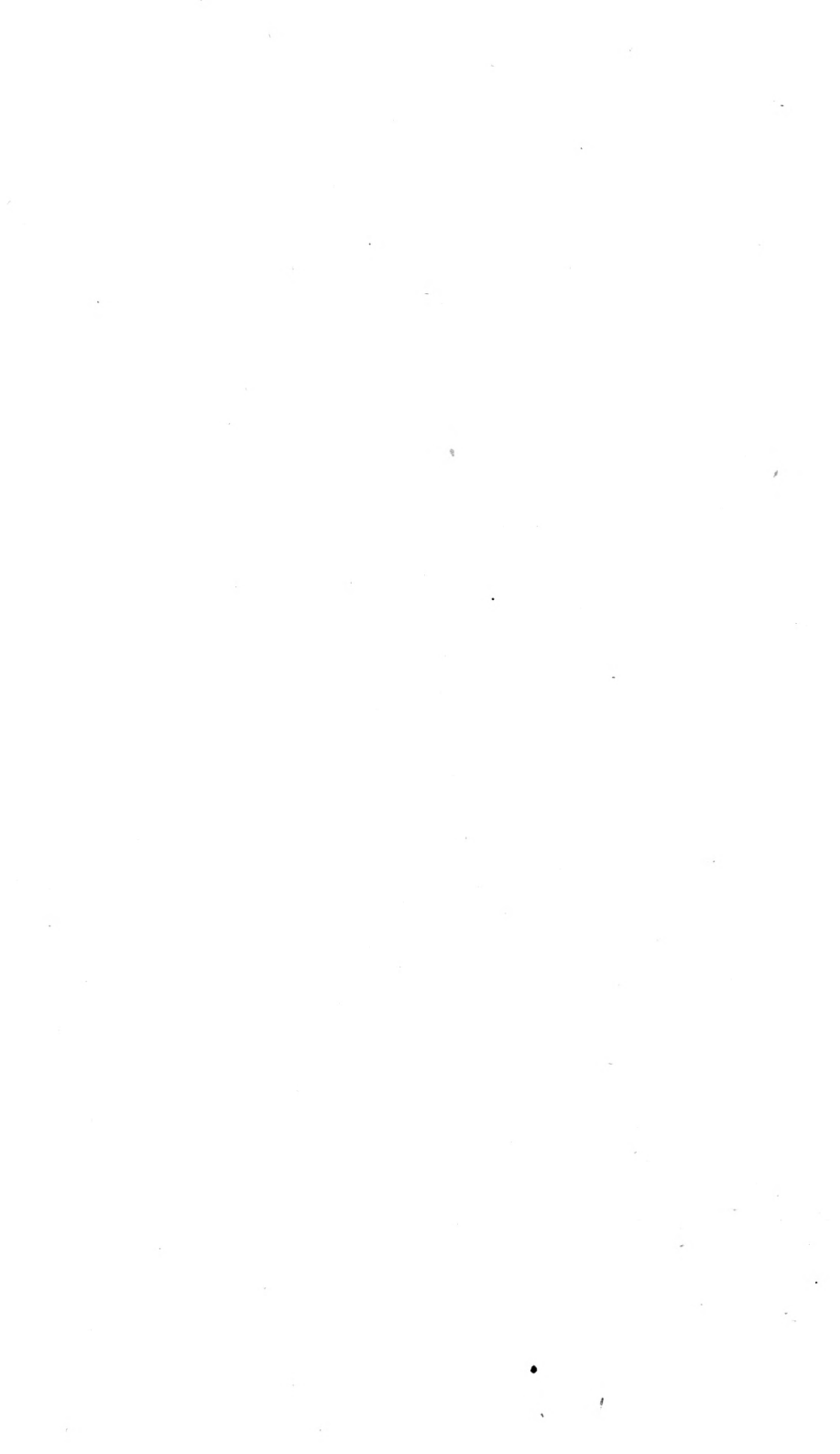
At the atmospheric temperature and pressure measure off 50 cc. of air in the Hempel burette, shown in Fig. 28. Connect this at *c* with the capillary of a Hempel's compound pipette (Fig. 29) containing an alkaline solution of pyrogallate of potash. Open the clips and transfer the air to the pipette by raising the tube, *a*. When this is accomplished, and the capillary of the pipette is filled with water from *b*, close the clips again. Disconnect the apparatus. Shake the pipette for several minutes so as to bring gas and absorbent in intimate contact. Reconnect pipette and burette, and force the residual gas into the latter. Restore atmospheric pressure and read the volume. What does the loss represent?

FIG. 30.



2. *Explosion Method.*—To 40 cc. of air contained in





the burette add 40 cc. of pure H. Pass this mixture into the Hempel explosion pipette shown in Fig. 30. Close the stop-cock, *d*, and the clip, *c*, then connect the platinum electrodes with an inductor and pass a spark. What takes place? Measure the volume of the gas remaining. How much of the contraction was due to O? What is the composition of the gas after the explosion?

(Study Richter, pp. 116-125.)

NITROGEN AND HYDROGEN.

AMMONIA.

(6) *Preparation*.—Heat an intimate mixture of finely powdered ammonium chloride and caustic lime in a flask; conduct the evolved gas through a tube filled with small pieces of lime, and collect it in jars or test-tubes over mercury.

What is the object of the lime in the tube? Why can you not dry the gas by passing it through H_2SO_4 or $CaCl_2$? Why should it be collected over mercury?

(7) Is ammonia gas combustible? Does it support combustion? 1. Conduct NH_3 through a glass tube, and insert this into a wider tube filled with oxygen (Fig. 31). Apply a flame. The ammonia gas will ignite and continue to burn. 2. Heat concentrated ammonia water in a beaker until there is an abundant disengagement of gas, then conduct a rapid current of oxygen through the liquid, and lower a glowing spiral of platinum into the beaker (as in Fig. 32).

What happens?

Note the odor of NH_3 (caution?). Is it lighter than air? Soluble in water?

(8) *Prepare an aqueous solution of ammonia.*

What are its properties?

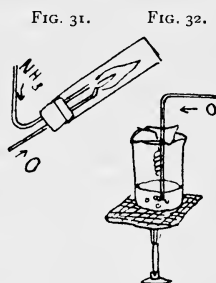
Add red litmus to some of the solution (?), and then neutralize carefully with dilute HCl. Evaporate to dryness. Compare the product with the ordinary NH_4Cl . Test it for Cl (?). Heat a little of it with sodium hydroxide (?). Heat another portion on a platinum foil (?).

(9) *Determine the weight of a litre of NH_3 .*

Fill a dry flask with the gas by *upward* displacement, and proceed exactly as under chlorine. What is the density of NH_3 ?

To determine the quantitative composition of NH_3 , perform experiments 1 and 2 on pp. 130 and 131, in Richter.

Write out summary. (Read Richter, pp. 125-131.)



NITROGEN AND THE HALOGENS.

(11) Pour a saturated alcoholic solution of iodine into strong ammonia water. Collect the precipitate on a filter and wash it with water. Open the moist filter; tear it into small pieces and spread these on a board. After they have become dry, touch them with the end of a rod (?). *Ask for instructions!* (Read Richter, pp. 132-133.)

NITROGEN AND OXYGEN.

(12) *Hyponitrous oxide*— N_2O . 1. Place about 5 grms. of ammonium nitrate in a small retort; add a little water, and apply heat. Collect the product over warm water. 2. Test it with a glimmering chip; 3. with burning phosphorus; 4. with burning sulphur. 5. Mix equal volumes of this gas and of H, and apply a flame. What other gas does it resemble in its properties? (Read Richter, pp. 212-213.)

(13) *Nitric oxide*—NO. 1. Pour dilute HNO_3 (sp. gr. 1.2) upon copper turnings contained in an evolution flask. Cool, and allow the red fumes, which form at first, to escape; then collect the colorless product over water. 2. What occurs when this gas comes in contact with the air? Is it the O or the N of the air that acts upon the gas? 3. Apply the tests given under (12) to this gas (?). How can NO be distinguished from oxygen? 4. Fill a cylinder with NO, and add a few drops of CS_2 , shake well and bring a flame to the mouth of the vessel (?). 5. Pass a current of NO into a strong solution of ferrous sulphate. What occurs? After the solution has become saturated with the gas heat it to boiling (?). 6. Pass the gas into a solution of potassium permanganate (?).

(14) *Nitrogen trioxide*— N_2O_3 . (Read Richter, pp. 205-206.)

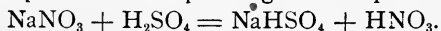
Nitrous Acid— HNO_2 . (Richter, p. 206.)

(15) *Nitrogen tetroxide*, N_2O_4 , and *dioxide*, NO_2 . 1. Heat 10 grams of dry lead nitrate in a test tube; condense the escaping vapors in a well-cooled receiver. What are the vapors, and what is the condensed liquid? Note the color. 2. What is the action of cold water, and of aqueous solutions of the alkalies upon N_2O_4 ? What do these reactions indicate in respect to the composition of this compound? (Richter, pp. 207-208.) 3. What is its action upon potassium iodide?

(16) *Nitrogen pentoxide*, N_2O_5 . (Richter, p. 205.)

NITRIC ACID.— HNO_3 .

1. *Preparation*.—In a retort heat a mixture of sodium nitrate and sulphuric acid in proportions corresponding to the equation (?):



Collect the product in a cold receiver.

2. What are the physical properties of HNO_3 ? Color? Odor? Action on litmus (dilute with H_2O)? 3. What action has it on indigo? Upon the skin? 4. Notice the effect of the acid upon the following metals: Cu, Fe, Pb, Zn, Sn. Write the reaction for each one. 5. Cover powdered sulphur with the acid, and warm (?). Dilute with water, filter, and test the liquid with BaCl_2 (?). 6. Add a few drops of HNO_3 to a solution of ferrous sulphate (?); warm the solution (?).

Problems.—1. Required 1 cu. m. of N. How much air is to be deprived of O; and how much P must be burned, if 62 pts. of P unite with 80 pts. of O?

2. How much HNO_3 , containing 46 per cent. of water, may be obtained from 1,700 grms. of NaNO_3 , and how much water must be taken?

3. How many grams of NH_3 will be absorbed by 5 litres of H_2O , if the latter absorbs 500 times its volume of the gas? 4. Ten litres of water having absorbed 700 times their volume of ammonia, what are the least amounts of NH_4Cl and CaO necessary for producing this solution?

PHOSPHORUS.—P.

(1) 1. Determine the physical properties of the *active* and the *red* varieties. 2. Allow a small piece of the active variety to ignite in the air. Will the red variety do this? 3. Throw a small piece of the yellow variety into a jar of dry Cl (?). Repeat with the red variety (?). 4. Bring a small dry piece of active P in contact with iodine (?). 5. Heat a flask containing a small piece of P and water until the former is melted, then pass a current of oxygen through a delivery tube into the melted phosphorus (?). *Care!* (Study Richter, pp. 133-136.)

PHOSPHORUS AND HYDROGEN.

(2) *Phosphine*— PH_3 . 1. Fill a flask almost full with a moderately concentrated NaOH solution. Add a few pieces of P, and heat carefully. When the air in the neck of the flask has been expelled by the escaping gas, insert a cork with a delivery tube the other end of which dips under *warm* water. What becomes of the gas as it escapes into the air? Write the reaction involved.

(Richter, pp. 136-139.)

Is there any similarity between PH_3 and NH_3 ?

PHOSPHORUS AND THE HALOGENS.

(3) 1. Pass a current of dry CO_2 gas into a retort, the bottom of which is covered with dry sand. When all the air has been expelled, introduce some well-dried pieces of P, and replace the CO_2 by a stream of dry Cl. Connect the neck of the retort with a Liebig's condenser, and collect the product in a receiver. It is phosphorus trichloride. What are its properties? Pour some of it into water(?).

2. Place a little PCl_3 in a dry test-tube, and pass a stream of dry Cl upon its surface. What is the result?

PHOSPHORUS AND OXYGEN.

(Richter, pp. 214-219.)

(4) 1. Prepare *phosphorus pentoxide*, P_2O_5 , by burning a carefully dried piece of P under a dry bell-jar. 2. Drop a portion of the product into water (?).

(5) *Orthophosphoric acid*, H_3PO_4 ; *metaphosphoric acid*, HPO_3 ; and *pyrophosphoric acid*, $\text{H}_4\text{P}_2\text{O}_7$. How are these acids obtained? How many series of salts are derived from them? By what names would you distinguish the different salts?

1. Dissolve some Na_2HPO_4 in water and test the solution with AgNO_3 , and FeCl_3 . What do you observe in each case? 2. Dissolve *fused* Na_2HPO_4 in water, and perform the same tests with its solution. 3. Heat salt of phosphorus ($\text{NaNH}_4\text{HPO}_4$) until it no longer effervesces; cool, crush the residue in a mortar, and dissolve it in water. How does this solution behave upon treating with the above reagents? 4. Acidify a portion of the last-named solution with acetic acid, and add a solution of albumen to it. Result?

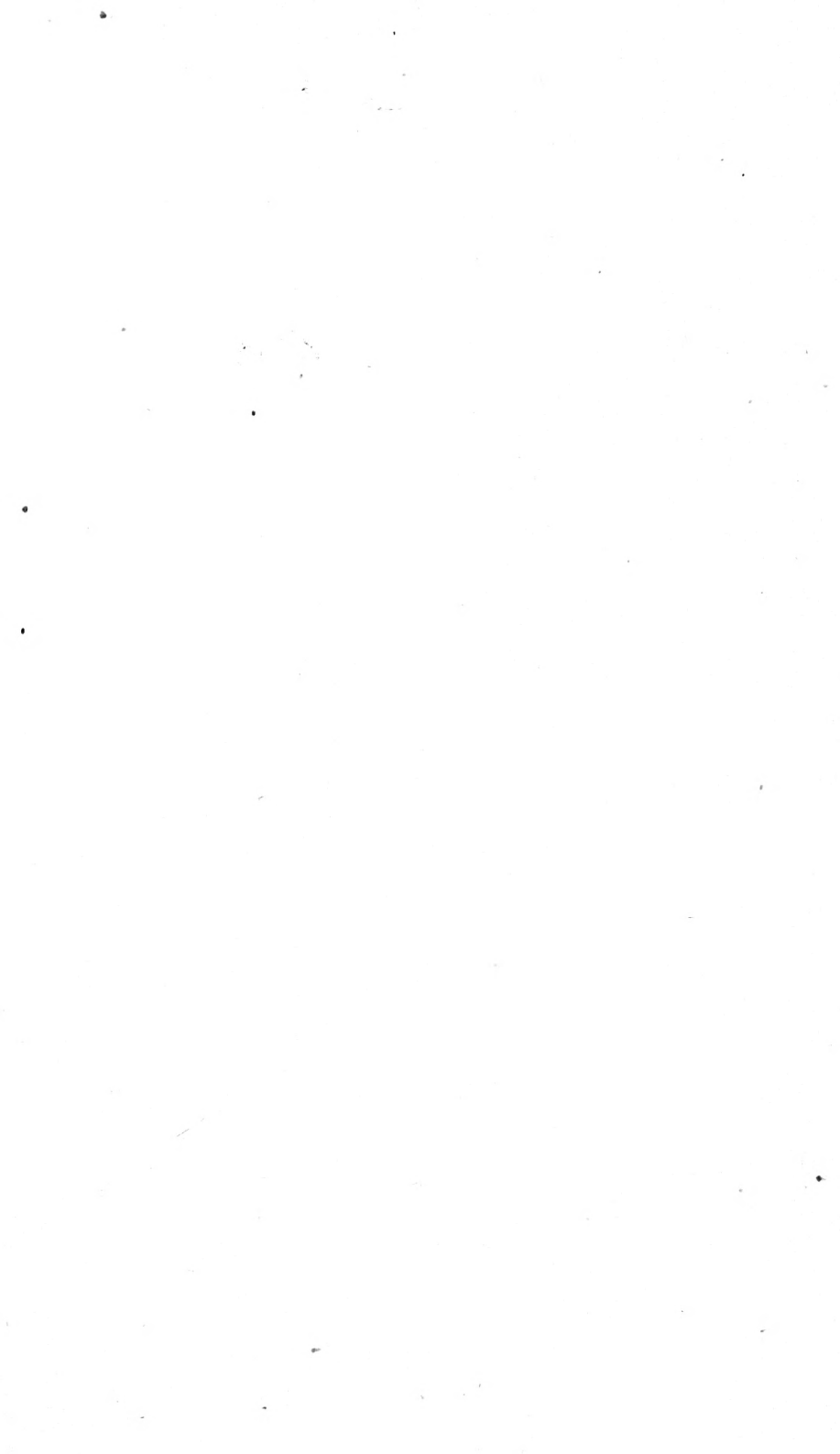
(6) *Phosphorus trioxide*— P_2O_3 , and *phosphorous acid*— H_3PO_3 .

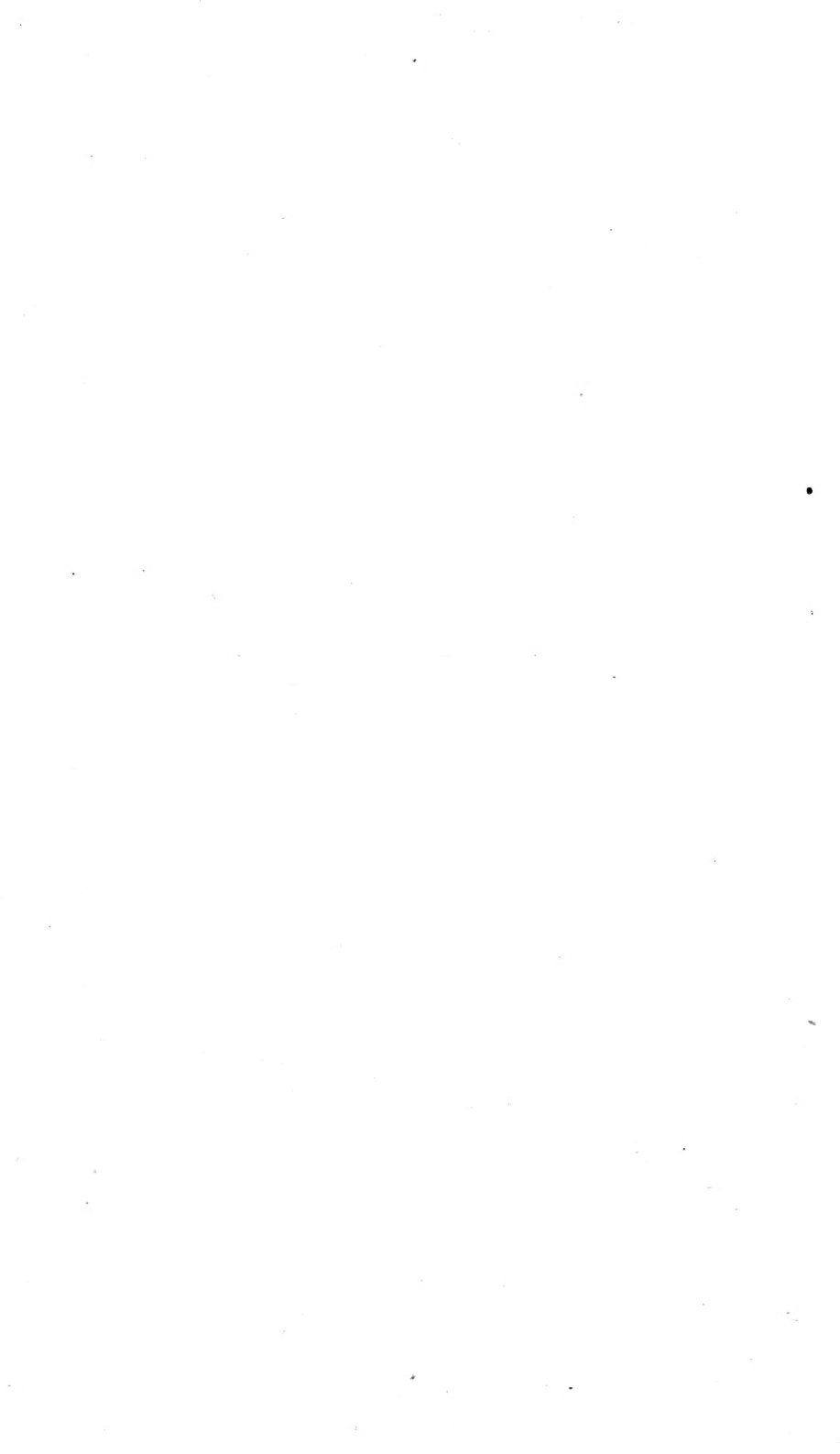
Pour PCl_3 into water. Evaporate the solution to syrupy consistency (?). (Study Richter, p. 216.)

(7) *Hypophosphorous acid*— H_3PO_2 .

Heat pieces of phosphorus in a porcelain dish with a moderately strong baryta solution (see p. 29). When no more PH_3 is formed, cool, filter, and pass CO_2 into the solution until it shows a neutral reaction to litmus. Toward the end, the solution should be warmed. Filter and evaporate to suitable concentration. Hypophosphite of barium will crystallize.

How may the free acid be obtained from this salt?





ARSENIC.—As.

(1) Study the physical and chemical properties of this element. (Richter, pp. 142 and 143.) Are they analogous to those of phosphorus?

1. In a tube of hard glass heat a small piece of As to redness. Result?
2. Heat As with the oxidizing flame upon charcoal (?).
3. Dissolve powdered As in strong HNO_3 (?).

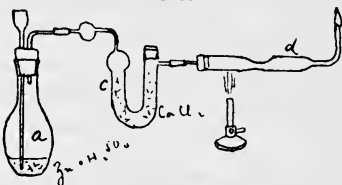
ARSENIC AND HYDROGEN.

(2) *Perform Marsh's test for As.**

Arrange the apparatus shown in Fig.

33. To the mixture of Zn and dilute H_2SO_4 contained in *a*, add a small portion of the solution to be tested for As. The liberated gas contains H and AsH_3 (arsine). It is passed through *c*, filled with CaCl_2 (?), and then through *d*, a tube of hard glass, contracted at several places. After all the air has been expelled from the apparatus, ignite the hydrogen. If As is present it will burn with a bluish white flame, and white vapors will be given off. Hold a cold porcelain plate in the flame (?). Heat the tube *d*, as shown in the figure (?).

Fig. 33.



Great care must be exercised in performing this test, as the arsine gas is extremely poisonous!

ANTIMONY.—Sb.

(1) Study this element in the same manner as As. Distinguish between SbH_3 and AsH_3 .

1. Treat the metallic mirrors obtained in Marsh's apparatus, with a freshly prepared solution of hypochlorite of sodium: As dissolves readily, while Sb is scarcely acted upon.
2. Heat a piece of the tube in which a mirror has formed, in the flame of the Bunsen burner. Dissolve the product in dilute, warm HCl , and add H_2S water (?).
3. Treat the spot formed upon a cold porcelain plate with yellow ammonium sulphide, and evaporate the solution at a gentle heat (?).

Problems.—(1) How much P can be obtained from 250 grms. of bones? (See Richter, p. 134.) (2) 10 grms. of P give what vol. phosphine? (3) What is the weight of the product remaining, after evaporating a solution of 10 grms. of As in HNO_3 ?

* Ask for instructions.

CHAPTER VI.

CARBON GROUP—CARBON AND SILICON.

CARBON.—C.

(1) How many allotropic modifications of this element are known? What are their principal properties? 1. Boil a dilute litmus solution with powdered animal charcoal; filter. Result? 2. Substitute indigo for the litmus in the preceding experiment (?).

3. *Determination of the composition of coal.*

1. Volatile matter and coke. Weigh out 2 grms. of powdered coal in a platinum crucible provided with a well-fitting cover. Heat with a large flame, until the escaping gases cease to burn between the lid and the crucible. A blast lamp flame is applied for a minute longer. Cool and weigh. Loss in weight represents the *volatile* matter. The residue is called *coke*.

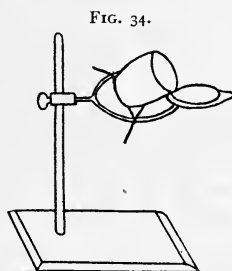


FIG. 34.

2. Ash.—A second portion of coal (1 gm.) is gently heated over the Bunsen flame, until the volatile constituents are expelled. The heat is then raised and the lid of the crucible placed in the position indicated in Fig. 34. The residue is the *ash*.

(Read Richter, pp. 150-152.)

CARBON AND HYDROGEN.

(2) *Methane* (Marsh gas)— CH_4 .

1. *Preparation*.—Heat a dried mixture of sodium acetate and sodium hydroxide in an iron tube.* Collect the gas over water. Note its color, odor and taste. Does it burn? 2. Mix 1 vol. of it with 7 to 8 times its vol. of air and explode by applying a flame. (Ask for instructions!)

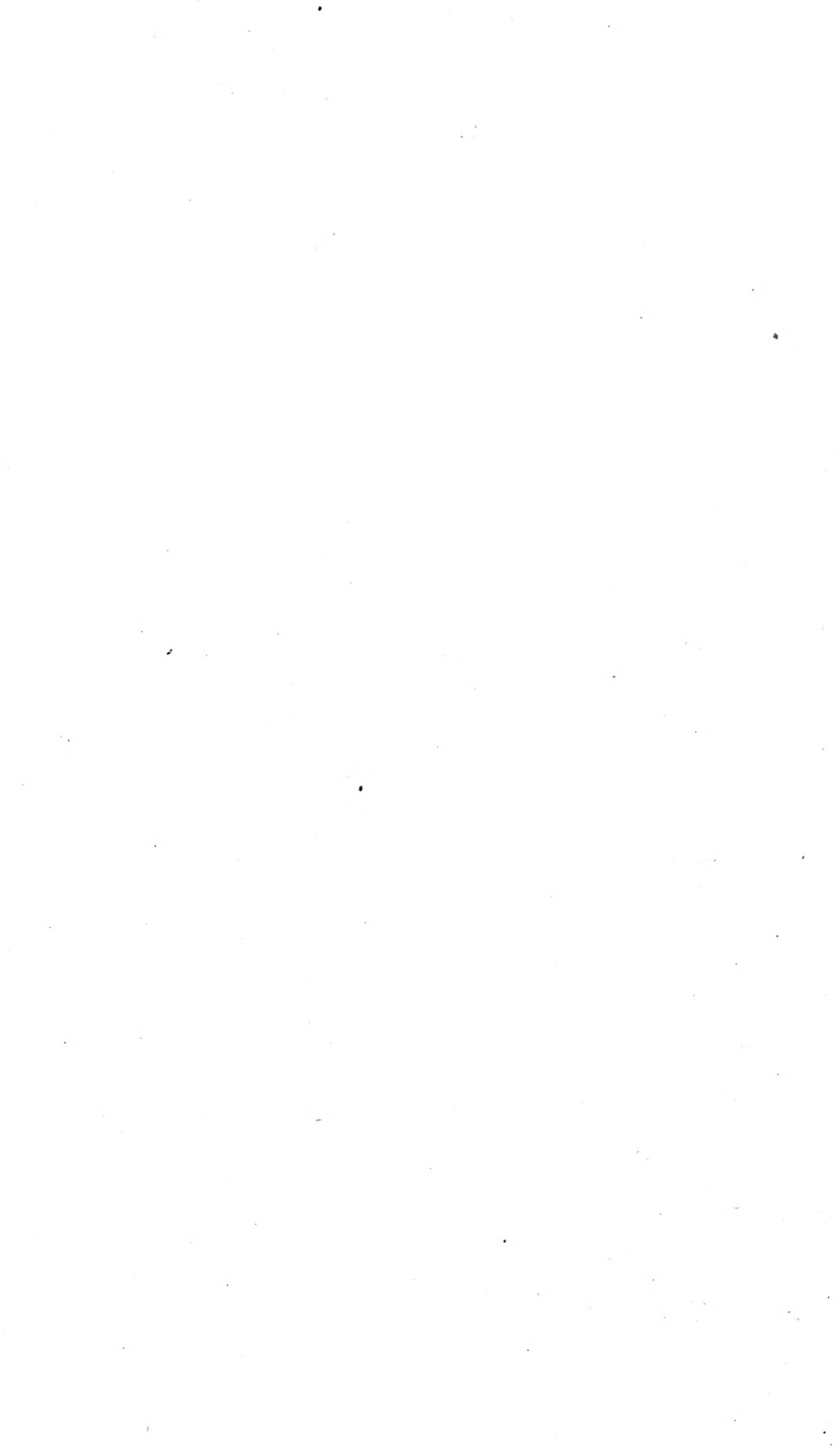
How would you determine the molecular weight of this compound?

(3) Make a eudiometric combustion of 1 vol. of CH_4 with 2 vols. of O as described in Richter, p. 121.

(4) *Ethane*— C_2H_6 . (Richter, p. 153.)

(5) *Acetylene*— C_2H_2 . Light a Bunsen burner at the base and turn it

* A hard glass tube will answer.



down, so that the flame is small. Acetylene can be recognized, among the products of combustion, by its characteristic odor.

(6) CARBON AND THE HALOGENS. (Richter, p. 160.)

CARBON AND OXYGEN.

(7) *Carbon dioxide*— CO_2 .

1. *Preparation*.—Upon pieces of marble, contained in an evolution flask, pour dilute HCl (1 HCl : 1-2 H_2O). Conduct the resulting gas through water and through conc. H_2SO_4 . It may be collected either by downward displacement of the air, or over mercury. 2. Note color, taste and odor of this gas. Is it soluble in water? How does its weight compare with that of air? Does it burn or support combustion? 3. Conduct a current of CO_2 into a solution of NaOH, evaporate the liquid, and test the residue for Na_2CO_3 (?). 4. To different portions of Na_2CO_3 solution, add solutions of MgSO_4 , BaCl_2 , $\text{Pb}(\text{NO}_3)_2$, ZnSO_4 (?).

(Study Richter, pp. 228-232.)

(8) *Carbon monoxide*— CO .

Preparation.—1. In a tube of hard glass heat zinc dust to faint redness, while conducting a slow current of CO_2 over it. In what respect does the product differ from CO_2 . 2. Heat crystals of oxalic acid with conc. H_2SO_4 in a flask, and wash the product with a NaOH solution. Write the reaction: Study the properties of this gas. (Richter, p. 233.)

(9) *Carbon disulphide*— CS_2 .

Perform some of the experiments indicated in Richter, p. 234.

(10) CARBON AND NITROGEN.

1. In a dry test-tube heat a nitrogenous carbon compound with a small piece of K. Cool and add water. KCN is formed and can be tested with AgNO_3 . 2. Convert a portion of the KCN into KCNS by evaporating with $(\text{NH}_4)_2\text{S}$. Test with FeCl_3 . 3. To a solution of FeSO_4 add potassium ferrocyanide. What results? 4. What is the action of the ferrocyanide upon solutions of ferric salts?

(11) Study the nature of flame. Make the experiments described in Richter, pp. 155-160.

SILICON.—Si.

(1) *Preparation*.—Make an intimate mixture of 1 grm. magnesium powder and 4 grms. of finely powdered quartz-sand. Heat this to bright redness in a wide tube of hard glass. It is best to use the blast lamp for this purpose. The part of the tube containing the mixture should be rotated in the flame. The residue, after a few minutes' heating, is allowed to cool, and treated with water containing HCl. The product

consists of amorphous silicon and undecomposed quartz. 2. Test the action of the following reagents upon Si: sulphuric, nitric and hydrofluoric acids, potash solution and chlorine. (Read Richter, p. 161.)

SILICON AND OXYGEN.

(2) *Silicon dioxide* (Silica, Quartz)— SiO_2 .

1. Test its solubility in the various acids and alkalies. 2. Fuse a mixture of 1 grm. of finely powdered quartz with 4 grms. of Na_2CO_3 , in a platinum crucible. Dissolve the product in water. 3. To a portion of this solution add HCl, and evaporate to complete dryness. Take up the residue with water and filter off the insoluble portion. 4. To another portion of the aqueous solution of the fusion add NH_4Cl . (?). Make a bead of *salt of phosphorus*; bring a fragment of a silicate or of quartz into it, and heat in the blow-pipe flame for a few minutes (?).

BORON.—B.

(1) Preparation similar to that of Si. What are its properties? Does it unite directly with other elements? Is it known in several allotropic modifications? What is the valency of this element?

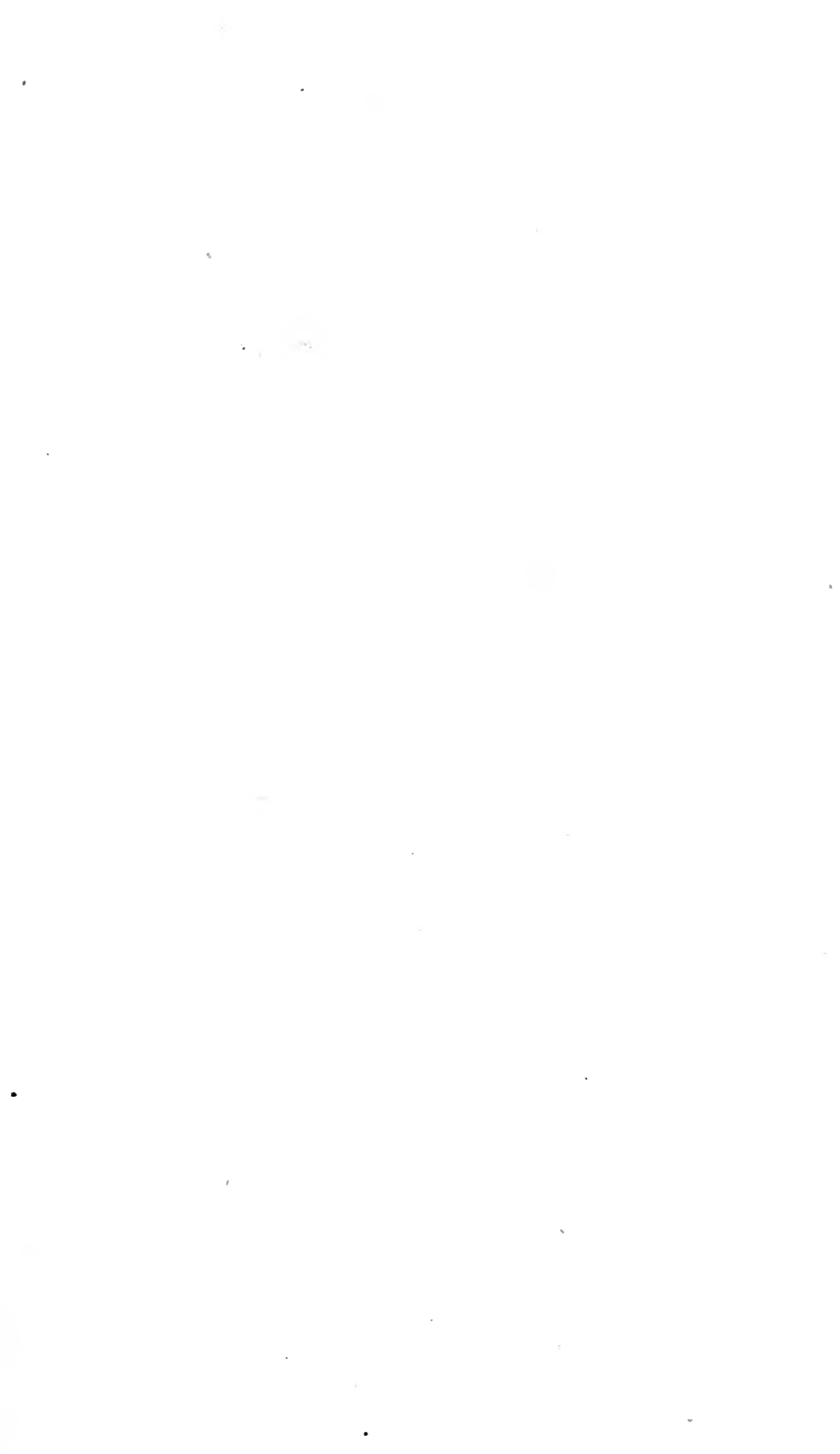
(Read Richter, pp. 240 and 241.)

BORON AND OXYGEN.

(2) *Boric Acid*— BO_3 .

1. Dissolve borax in 5 parts of boiling water, add HCl to acid reaction, and allow to cool. What crystallizes out of the solution? Dry some of the product by pressing it between filter paper. Test its solubility in water and in alcohol. What do you observe on igniting the alcoholic solution? Moisten a piece of turmeric paper with an aqueous solution of boric acid, and dry at a gentle heat. What happens?

Problems.—(1) How much CO_2 results from the combustion of 12 grms. of carbon? (2) How much CO_2 will an indefinite quantity of CaCO_3 give, when acted upon by 4.666 grms. of muriatic acid, containing 30 per cent. of pure HCl? (3) How many cubic decimeters of CO can be obtained from 90 grms. of oxalic acid? (4) What amount of SiO_2 can be obtained from 2 grms. of Wollastonite (CaSiO_3)? (5) What is the theoretical quantity of boric acid obtainable from 15 grms. of borax ($\text{Na}_2\text{B}_4\text{O}_7 + 10\text{H}_2\text{O}$)?





METALS.

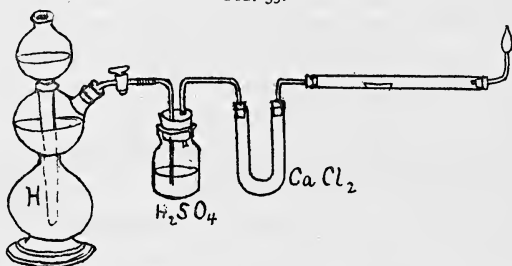
CHAPTER VII.

METALS OF THE ALKALIES—POTASSIUM, SODIUM, [AMMONIUM].

POTASSIUM.—K.

(1) *Preparation.*—Arrange apparatus as shown in Fig. 35. Into a tube of hard glass, *c*, introduce a porcelain boat containing about 1 gm. of a mixture of 138 pts. (1 mol.) of dry (?) K_2CO_3 and 72 pts. (3 at.) of Mg powder. Pass a current of dry H over it, and after all the air has been displaced in the apparatus (?), light the escaping gas; heat the part of the tube surrounding the boat to incipient redness. Observe the brilliant metallic mirror which is formed, and drive it away from the boat by increasing the temperature: it is *potassium*. Note also the green color of the vapor and the violet coloration it imparts to the burning hydrogen. What is the residue left in the boat? Test its reaction with litmus (?).

FIG. 35.



Formulate the reaction involved in this method of preparation.

(2) 1. Cut a piece of K with a knife, and observe the color and lustre of the fresh surface. *Care!* 2. To ascertain whether the metal is fusible, heat a small piece of it in a stream of H. 3. Is it heavier or lighter than water?

(3) 1. Expose a thin slice of K to the air. What takes place? 2. Throw a *small* piece of it upon H_2O (?). In this experiment it is advisable to use a tall beaker and to cover the same with a glass plate. 3. What is the action of the halogens upon K? *Ask for instructions.*

POTASSIUM AND OXYGEN.

(4) *Preparation of Potassium Hydroxide.*—In an iron vessel dissolve 50 grms. of crystallized $Ba(OH)_2$ in 160 cc. of water. Cautiously add a hot concentrated solution of 20 grms. of K_2SO_4 until a sample of the supernatant liquid is no longer precipitated by either K_2SO_4 or $Ba(OH)_2$. Filter rapidly through a plaited filter, and evaporate the solution in an iron or silver dish over a large flame. Continue heating the residue till it appears in a state of quiet fusion. *During this operation protect the eyes with a glass plate.* Now pour the product upon a clean iron surface, and

while still warm put it into a bottle provided with a well-fitting stopper. Examine its fracture and color. Try its solubility in water and in alcohol. What is the reaction of the aqueous solution with litmus? What is an alkali?

Salts.

(5) *Potassium Chlorate*.— KClO_3 . (See p. 22).

(6) *Potassium Nitrate*.— KNO_3 . To a hot concentrated solution of 20 grms. of NaNO_3 add a solution of 18 grms. of KCl . Boil. What separates from the *warm* mixture? What crystallizes from the mother liquor on cooling? Recrystallize the latter product. Examine its crystalline form. Is it more soluble in hot than in cold water? Explain the method of preparation.

(7) Into a red-hot platinum crucible throw small portions of an intimate mixture of 10 grms. of KNO_3 and $1\frac{1}{2}$ grms. of charcoal powder. What takes place? Write the reaction. What is gunpowder?

Reactions.

(8) Use KNO_3 for the following tests.

1. Place a little of the salt upon the end of a clean platinum wire and introduce it into a non-luminous flame. What color do you observe? View the flame through a cobalt glass (?). 2. To the aqueous solution of the potassium salt add HCl and boil. Concentrate by evaporation and add PtCl_4 . What is the composition of the resulting precipitate? Try its solubility in hot and in cold water, also in alcohol. 3. To the concentrated solution of the salt add a saturated solution of tartaric acid; either at once, or on shaking, a white crystalline precipitate appears (?).

SODIUM.—Na.

(1) How is this metal usually prepared?

(2) Study its physical and chemical properties (Richter, p. 285). Wherein does it differ from K?

(3) *Prepare Sodium Amalgam.*

To 500 grms. of dry mercury, contained in a Wedgewood mortar add gradually 5–10 grms. of Na in thin slices. Perform this operation in a good draught chamber, as the union of the two metals is attended with the evolution of light and heat, and poisonous vapors are given off. Stir well with the pestle, allow to cool, and transfer the product to a well-stoppered bottle. What is its action on H_2O or dilute H_2SO_4 ?

SODIUM AND OXYGEN.

(4) *Preparation of Sodium Hydroxide solution.*

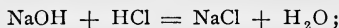
Add a little water to 10 grms of fresh quicklime contained in an iron (or porcelain) vessel. Cover the latter, and in a second iron pot dissolve 25 grms. of soda ash (Na_2CO_3), using about 100 cc. of water.



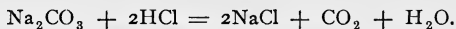
Heat the solution to boiling; stir the quicklime—which should have broken up to a white powder—with enough water to form a thin paste (milk of lime), and add this gradually to the boiling liquid. Stir well with an iron wire; transfer the mixture to a bottle; cork, and allow it to stand. After the supernatant liquid has become perfectly clear, decant it by means of a glass siphon filled with water. It should be preserved in a tightly corked bottle (?). Test a few drops of the solution with BaCl_2 (?). What should the solution contain, and of what does the precipitate, from which it was separated, consist? Write the equation representing the reaction.

(5) *Determine the amount of NaOH contained in the solution.*

Measure off accurately 20 cc. into a porcelain dish; add a drop or two of phenolphthalein solution, and dilute with water. From a burette carefully add dilute hydrochloric acid until the red color has *just* disappeared. Read off the volume of the acid used; it is the exact quantity needed to neutralize the alkali:—



that is, 40 pts. (1 mol.) of NaOH require 36.5 pts. (1 mol.) of HCl, and if we know the weight of the HCl contained in the volume of the dilute acid consumed, a simple proportion will give the weight of the alkali in 20 cc. of the solution. The *strength* of the acid is determined as follows: In a porcelain dish, dissolve 1.06 grms. of pure Na_2CO_3 , previously ignited and accurately weighed; add a little phenolphthalein, heat to boiling and introduce acid from the burette until the liquid *remains* colorless after continued boiling. The carbonate is then exactly neutralized:—



It takes, therefore, 73 pts. of HCl for 106 pts. of Na_2CO_3 . Suppose, now, 20 cc. of the acid had been used to decolorize the indicator, then 1 cc. would equal $\frac{106}{20} = .53$ grms. of Na_2CO_3 , or .365 grms. of HCl. The latter number is the *standard* or *strength* of the dilute acid.

The phenolphthalein takes no part in these reactions; it merely *indicates* by its change of color the complete neutralization of the alkali. Why is it necessary to boil the solution when the acid is standardized with a carbonate?

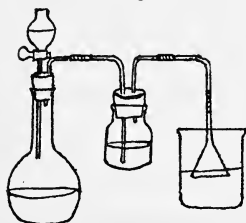
Salts.

(6) *Sodium chloride.*— NaCl .

Purify common salt.—Grind 50 grms. of salt in a mortar with 150 cc. of water. Filter into a beaker, and conduct HCl gas into the solution,

as shown in Fig. 36.

FIG. 36.



Pure NaCl separates out. Collect it on a platinum cone, remove the liquid with the aid of a filter pump, and dry the salt by warming it in a porcelain dish, while stirring it with a glass rod.

(7) *Sodium carbonate*.— Na_2CO_3 .

Recrystallize some of the commercial carbonate. Heat a portion of the product in a porcelain dish. What do you observe?

Reactions.

(8) Use the purified chloride for the tests. 1. What color do sodium salts give to the flame? 2. Mix a drop of the aqueous solution with 10 drops of a PtCl_4 solution on a watch-glass. Evaporate *very carefully* to a small volume. On cooling, a red colored salt crystallizes out in long monoclinic needles (?). Is it soluble in water? in alcohol? 3. Are there any salts of sodium which are not soluble in water? Can compounds of sodium be precipitated by any reagent?

AMMONIUM.

(1) What is the composition of ammonium? Can it be obtained in a free state? (See Richter, p. 295.)

(2) Dissolve commercial sal ammoniac in a little water, add ammonia in slight excess, warm, filter if a precipitate is formed, and evaporate to crystallization; stir constantly. Ammonium chloride is thus obtained in the form of a fine powder.

Reactions.

(3) 1. On a piece of platinum foil heat successively small portions of the chloride, the sulphate, and the nitrate. What occurs in each case? 2. Mix a little NH_4Cl with burnt lime in a small mortar. Note the odor of the escaping gas and its reaction with litmus. 3. Heat a small portion of NH_4Cl with a caustic soda solution. What is given off? Explain the action of strong bases upon ammonium salts. 4. Add PtCl_4 to a solution of NH_4Cl . Result? 5. To a concentrated solution of the ammonium salt add tartaric acid and shake the mixture (?). 6. Do compounds of ammonium impart a color to the flame?

Compare the metals of the alkalis with each other. How can the compounds of potassium, sodium, and ammonium be distinguished?

Problems.—1. How much KNO_3 is theoretically obtainable from 2 kilos

of Chili saltpetre of 97%, and what amount of Sylvite containing 98% of KCl is required? 2. Suppose that 75 cc. of dilute HNO_3 were required to saturate 50 cc. of a potash lye; further, that 10 cc. of the acid neutralized 1.06 grms. of Na_2CO_3 , what amount of KOH would the lye contain? 3. In the valuation of a pearl ash (impure K_2CO_3), 29.1 cc. of a sulphuric acid were used to neutralize 5 grms. of the sample; the acid contained 98 grms. of H_2SO_4 per litre; calculate the percentage of impurities in the product. 4. Required the minimum amount of marble that should be burnt to liberate the NH_3 from 50 grms. of NH_4NO_3 .

CHAPTER VIII.

METALS OF THE ALKALINE EARTHS—CALCIUM, STRONTIUM, BARIUM.

CALCIUM.—Ca.

CALCIUM AND OXYGEN.

(1) 1. Ignite 2 grms. of powdered marble in a platinum crucible to the highest temperature obtainable with the aid of the blast lamp. Continue this for 15 minutes, occasionally stirring the mass with a platinum wire; what is the residue? Explain the reaction. 2. Add about 5 cc. of water to the product. What do you observe? Test the reaction of the product with litmus paper.

(2) 1. *Prepare lime water.*—To the slaked lime obtained from 20 grms. of quicklime (see p. 37) add 1 litre of water; transfer the mixture to a bottle. Cork tightly, shake and allow to stand. When the solution has become clear, draw it off by means of a siphon? What does it contain? Of what does the undissolved portion consist? 2. Place a portion of the lime water on a watch glass and expose to the air (?). 3. Through a second portion blow air from your lungs (?). 4. Conduct a stream of CO_2 through a third portion and observe carefully the successive changes. Explain them. 5. What takes place upon boiling the clear solution which is obtained as the final product in the preceding experiment?

Salts.

(3) *Calcium Chloride.*— CaCl_2 .

1. Evaporate some of the spent acid of a CO_2 generator to dryness. What is the residue? 2. Expose a little of the salt to the air (?). 3. What use have you made of CaCl_2 previously? 4. *Prepare porous CaCl_2* ($\text{CaCl}_2 + 2\text{H}_2\text{O}$). Dissolve the residue obtained in 1 in lime water, filter, and neutralize exactly with HCl. Evaporate the filtrate to dryness

in a porcelain dish, and heat the residue for some time on the sand-bath. The solution of the product must show a neutral reaction.

(4) *Calcium Hypochlorite*.— $\text{Ca}(\text{ClO})_2$. (See p. 22.)

(5) *Calcium Sulphate*.— CaSO_4 .

1. *Carefully* heat a few grms. of gypsum in a porcelain dish until the water of crystallization is completely expelled. Pulverize the residue. What happens when it is made into a paste with water and allowed to stand?

Reactions.

Use the pure CaCl_2 for the following tests:—

1. Introduce a small portion of the salt into the Bunsen flame by means of a platinum wire (?). 2. To the aqueous solution add $(\text{NH}_4)_2\text{CO}_3$. Result? 3. To another portion add dilute H_2SO_4 . What is the composition of the precipitate? Why does it not form in very dilute solutions? 3. Add $(\text{NH}_4)_2\text{C}_2\text{O}_4$ to the filtrate from the CaSO_4 (?).

STRONTIUM.—Sr.

Reactions.

1. What color is imparted to the Bunsen flame by compounds of this element? 2. Add a CaSO_4 solution to the solution of a strontium salt (?).

BARIUM.—Ba.

Reactions.

1. Observe what color Ba compounds give to the flame. Moisten the sample with HCl before heating it (?). 2. To a portion of the aqueous solution of the chloride add $(\text{NH}_4)_2\text{CO}_3$. What results? 2. Add dilute H_2SO_4 to a second portion (?).

Point out how the elements of this group may be distinguished (*a*) from those of the preceding group; (*b*) from each other.

Problems.—1. How much nitric acid of 20 per cent. will effect the solution of 1 gram. of Iceland spar (CaCO_3)? How much CO_2 is given off, and what volume would it occupy at 20°C . under a pressure of 750 mm.? 2. Suppose .5 gram. of sulphur were dissolved in HNO_3 , what quantity of BaCl_2 must be added until it ceases to produce a precipitate? 3. One gram. of a mineral consisting of the carbonates of Ca, Sr, and Ba, in the proportion of their molecular weights, will leave what weight of the mixed sulphates on treating and evaporating with an excess of H_2SO_4 ?



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CHAPTER IX.

MAGNESIUM GROUP—MAGNESIUM, ZINC, CADMIUM.

MAGNESIUM.—Mg.

(1) Examine the metal in the forms of ingot, ribbon and powder. Note its color, lustre and specific gravity. 2. Introduce a piece of the ribbon into the flame with the forceps (?). What is the product? 3. Treat a piece of the ribbon with dilute H_2SO_4 . Reaction?

Salts.(2) *Magnesium Chloride.*— $MgCl_2$.

Prepare the ANHYDROUS salt.—Dissolve about 50 grms. of the crystallized (?) chloride and 50 grms. of NH_4Cl in as little water as possible. Evaporate to dryness in a porcelain dish. Reduce the mass while hot to small pieces in a mortar, dry it carefully, so as to remove every trace of moisture. It is best to do this by heating small portions of the material in a porcelain crucible until it no longer sinters. A small sample should not give off moisture when heated in a dry test-tube. Be careful also to prevent re-absorption of moisture. Quickly transfer the warm powder to a platinum crucible provided with a well-fitting cover. Heat, at first gently, to expel the NH_4Cl , then increase the temperature until the mass is in a state of quiet fusion. It is the anhydrous salt which, being extremely hygroscopic, should be preserved in a tightly stoppered bottle. It should dissolve in water to a *clear* liquid.

Why cannot the anhydrous chloride be obtained by evaporation of the aqueous solution?

(3) *Magnesium Sulphate.*— $Mg SO_4 + 7H_2O$.

Recrystallize some of the commercial salt. What is the form of the crystals? Taste?

(4) *Reactions.*

1. Heat a portion of the sulphate or chloride on a platinum wire in the Bunsen flame; moisten with $Co(NO_3)_2$ solution and heat again. A *pink-colored* mass results. 2. Add some caustic soda to a little of the solution of the chloride (?). The resulting precipitate dissolves on addition of an ammonium salt (?). 3. Mix a second portion of the chloride solution with NH_3 and NH_4Cl , add Na_2HPO_4 and agitate the liquid. What is the composition of the precipitate? Examine it with the aid of a lens.

ZINC.—Zn.

(1) How is this metal obtained from its ores?

(2) Study the physical and chemical properties of Zn (see Richter, p. 316). 1. Treat a small piece of *pure* Zn with dilute H_2SO_4 (?). 2. Repeat this experiment, substituting the impure commercial metal. What difference do you observe? What causes it?

(3) *Granulate commercial zinc*.—Melt 100 grms. of the metal in a well-covered Hessian crucible. The blast lamp may be used for this purpose, but it is better to perform the operation in a wind furnace. The crucible is then removed from the source of heat, and allowed to cool until the melted metal no longer takes fire when the cover is lifted. Pour the metal, in a thin stream, into a pail filled with cold water. Drain the product and dry at a moderate heat.

Salts.

(4) *Zinc sulphate*.— $Zn SO_4 + 7H_2O$. (See p. 12). 1. Prepare some of this salt and recrystallize it carefully from water. 2. Examine the crystals. What other salt have you prepared that exhibits similar forms? Is there any analogy in the composition of the two salts?

Reactions.

(5) 1. Heat a small piece of Zn on charcoal in the oxidizing flame.(?) 2. Moisten the incrustation obtained with a drop of $Co(NO_3)_2$, and heat again. Result? 3. To a solution of $ZnSO_4$ add $(NH_4)_2 S$. What is the color of the precipitate? Try its solubility in dilute HCl and in $HC_2H_3O_2$ (acetic acid). 4. Study the action of caustic alkalies, *e. g.*, NaOH upon the Zn solution.

How could you distinguish between Zn and Mg? What differences are there between this and the preceding groups?

Problems.—1. What is the strength of a sulphuric acid of which 20 cc. dissolve exactly .048 gm. of Mg? 2. Suppose it was found that 1 gm. of Zn gave with H_2SO_4 , 325 cc. of H at $16^\circ C.$ and 755 mm., and, further, that .369 gm. of Mg produced the same amount of the gas. Knowing the atomic weight of Mg to be 24, and remembering that the two sulphates are isomorphous, how is it possible to deduce the at. wt. of Zn from the data given?

CHAPTER X.

MERCURY, COPPER, SILVER, GOLD.

MERCURY.—Hg.

(1) Study the physical and chemical properties of the metal. Wherein does it differ from the other metals?

MERCURY AND OXYGEN.

(2) *Mercuric oxide*.— HgO .

How is this substance prepared? What is its behavior on heating?

Mix a little powdered S with dry Na_2CO_3 and HgO . Ignite the mixture in a dry test-tube. Extract the residue with water, filter, acidify with HCl and add BaCl_2 . What has become of the oxide of mercury in this experiment?

Salts.

(3) *Mercurous Nitrate*.— HgNO_3 .

An *excess* of metallic mercury (use 10–15 grms.) is treated in the cold with moderately strong HNO_3 until the formation of crystals is no longer noticeable. Redissolve the crystals by warming, filter, and allow to crystallize.

To prepare a solution of the salt take it up with water acidulated with HNO_3 (?).

(4) *Mercuric chloride*.— HgCl_2 .

Dissolve about 5 grms. of Hg in aqua regia. Evaporate to dryness on a water bath. Place the residue into a small dry flask, cover the latter with a watch-glass, and heat cautiously on a sand-bath. What is the sublimate formed in the upper part of the flask? Dissolve it in four parts of boiling water and allow to crystallize.

Reactions.

(5) *Mercurous compounds*. Use the solution of the nitrate. 1. Add a few drops of HCl to 2 or 3 cc. of the solution. What takes place? Filter, and add NH_3 to the precipitate (?). 2. Add stannous chloride to another portion of the nitrate solution (?). 3. In a third portion immerse a slip of Cu foil. Examine the stain on the metal; is it changed when you hold it in the flame?

(6) *Mercuric compounds*. The chloride will answer for the tests.

1. Pass H_2S through a dilute solution and observe the gradation of colors through which the precipitate passes. What is the final product?

2. Add SnCl_2 , drop by drop, to the mercury solution. Explain the changes which occur.

COPPER.—Cu.

1. *Preparation.*—Ignite the pure oxide in a current of dry H (see p. 14). Examine the color and the lustre of the product; test its solubility in HCl , H_2SO_4 (both strong and dilute), and HNO_3 . Write equations representing the reactions.

Salts.

(2) *Copper Sulphate.*— $\text{CuSO}_4 + 5\text{H}_2\text{O}$.

To 10 grms. of Cu in a flask add 45 grms. of conc. H_2SO_4 , and heat. When the metal has completely disappeared and the gas (?) ceases to be given off, allow to cool, place the white crystalline residue (?) into a porcelain dish, rinse the flask with hot water. Now add a few drops of HNO_3 to the hot water solution, and filter. From the filtrate the sulphate crystallizes on standing. Recrystallize the product.

Does this salt suffer decomposition on exposure to the atmosphere? Heat a small quantity in a porcelain crucible, first moderately, then more strongly (?).

(3) *Sulphate of Copper and Potassium.*— $\text{CuK}_2(\text{SO}_4)_2 + 6\text{H}_2\text{O}$.

Prepare solutions of 10 grms. of blue vitriol and 7 grms. of K_2SO_4 , both saturated at 70° . The latter should also contain a few drops of H_2SO_4 . Mix the solutions; on cooling the double salt separates in whitish-blue crystals. Examine their form.

Reactions.

(4) Use either of the salts you have prepared.

1. Mix a little of the salt with Na_2CO_3 , and heat on charcoal in the reducing flame (?). 2. Make a borax bead and dissolve a minute quantity of a Cu-compound in it. What color does it give (a) in the oxidizing flame? (b) in the reducing flame? (c) when the bead is reduced with a small piece of tin? 3. Through a dilute Cu-solution pass H_2S . Is the resulting precipitate soluble in HCl or in HNO_3 ? 4. Add ammonia, drop by drop, to the solution. What changes do you observe? 5. To a portion of the very dilute solution add potassium ferrocyanide (?).

(5) To a solution of copper sulphate in a porcelain dish add a small piece of Zn. Allow to stand over night. Note the result. Has the Zn disappeared? Does the solution contain any of this metal? In what form? Where is the Cu?

(6) Repeat the experiment, weighing the copper sulphate (.5 gm.) and the Zn (.2 gm.). Add HCl in quantity sufficient to insure the

entire solution of the Zn, collect the Cu on a filter, wash with alcohol, dry, heat gently and weigh it in a porcelain crucible. The filtrate should be colorless.

Compare the weight of the metallic Cu obtained with that of the Zn employed (?). How does the found Cu accord with the calculated amount of that metal in .5 gm. $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$?

Repeat the experiment using Cd in place of Zn. Compare the weights of the metals as before. What deduction can you make?

SILVER.—Ag.

(1) *Prepare pure Silver from a coin.*

Dissolve a 25-cent piece in nitric acid of sp. gr. 1.2, filter (?), and evaporate the blue (?) solution to dryness. Fuse the residue till it blackens, extract with 250 cc. of water; filter. Now add ammonia in large excess, and then, cautiously, a sodium bisulphite solution (of about 40 %) until on boiling a small portion of the liquid, it is completely decolorized.

The greater part of the Ag separates from the solution on standing in the cold; it is well crystallized. The remainder may be precipitated by warming to 70° . Digest the product with strong ammonia (?), wash, dry and ignite it.

Examine the metal carefully. What are its physical and chemical characteristics?

SILVER AND SULPHUR.

(2) *Silver Sulphide.*— Ag_2S .

Into a dilute solution of AgNO_3 (see next experiment), containing about 2 grms. of the metal, pass H_2S . When the liquid smells of the gas, filter off the black precipitate, wash it with water and dry at 100° .

Salts.

(3) *Silver Nitrate.*— AgNO_3 .

Dissolve the Ag obtained in (1) in dilute HNO_3 and evaporate to dryness on the water bath. Dissolve the residue in 80 cc. of distilled water, and preserve the solution in a dark bottle (?). What is its reaction with litmus?

Reactions.

(4) 1. Compounds of Ag on charcoal before the blow-pipe give a white metallic globule (?). 2. To a silver solution—use the nitrate—add HCl. Collect the precipitate on a small filter, wash, dissolve it in ammonia, and add an excess of HNO_3 to the solution (?). Explain these

reactions. 3. Expose a small portion of the chloride to direct sunlight. Any change? What practical application is made of this reaction? (Read Richter, p. 340.)*

(5) Place strips of the metals Zn, Fe, Sn, Pb, and Cd in a solution of silver nitrate. What is the result in each case? Explain.

GOLD.—Au.

(1) How could you distinguish the metal Au from Hg, Ag, and Cu?

Reactions.

(2) 1. Dissolve a small piece of gold (or of a substance containing gold) in aqua regia, concentrate the solution at a gentle heat and pour it into a porcelain dish. Add a solution of FeCl_3 to an SnCl_2 solution until the latter is permanently yellow. After diluting, dip a glass rod into this and then into the gold solution. A purple streak (purple of Cassius) is formed. 2. Add ferrous sulphate to some of the AuCl_3 solution (?).

In what respects do the members of this group differ from each other, and how can they be distinguished from the metals of the preceding groups?

Problems.—1.5 grms. of HgO gave on ignition with carbon 4.63 grms. of metallic mercury; the specific gravity of the vapor of HgCl_2 referred to H, was found to be 135.5. What is the atomic weight of Hg? 2. The molecule of Hg contains how many atoms, if the vapor density equals 100? 3. On analysis a chalcocite was found to contain 20.15 per cent. of S and 79.85 per cent. of Cu. Deduce the molecular formula of the mineral. 4. What quantities of Ag, Au, and Hg can be precipitated from their respective solutions by 1 gram. of Cu?

CHAPTER XI.

ALUMINIUM, TIN, LEAD, BISMUTH.

ALUMINIUM.—Al.

(1) By what methods is this metal obtained on a large scale? What are its properties? Try the action of the following reagents upon Al: HCl , HNO_3 , and NaOH solution. Write the reactions.

* If practicable, the instructor should here show and explain the preparation of a photographic negative.

Salts.

(2) *Sulphate of Aluminium and Potassium.*— $KAl(SO_4)_2 + 12H_2O$.

Prepare saturated solutions of $Al_2(SO_4)_3$ and K_2SO_4 ; mix these so that the resulting liquid contains the two sulphates approximately in the proportion of their molecular weights. The double sulphate crystallizes on standing. Why? Recrystallize it from water. What is the form of the crystals?

What is an *alum*? (See Richter, p. 351.)

Reactions.

(3) Use alum. 1. Heat a little of the salt on a platinum wire in the oxidizing flame, moisten with $Co(NO_3)_2$, and heat again. A blue mass (?) is the product. 2. To an aqueous solution add ammonia (?). Add $(NH_4)_2S$ to another portion of the solution. What do you observe? 4. To the diluted solution add $NaOH$, drop by drop. Note the successive changes (?).

TIN.—Sn.

(1) Examine a bar of this metal. 1. Note the sound it emits on bending (?). 2. Etch a smooth surface with HCl (?). 3. Try the solubility of Sn in hot HCl . 4. What action have moderately dilute, and concentrated, HNO_3 upon it? Write the reactions.

(2) *Determine the specific heat of Tin.*

A thin glass beaker of about 200 cc. capacity is carefully covered on the outside with a moderately thin layer of cotton wool. This may be called the *calorimeter*. Pour 100 cc. of distilled water into the beaker. Suspend a thermometer in the water. Place 25 grms. of granulated tin into a test-tube, close the mouth of the latter with a plug of cotton. Introduce the test-tube with its contents into a beaker glass containing boiling water. A stout copper wire will serve as a handle. After ten or fifteen minutes the tin will have acquired the temperature of the boiling water— 100° . The tube is then rapidly removed from the latter and its outer surface freed from moisture by quickly passing a towel over it. Remove the cotton from the mouth, and transfer the tin to the calorimeter. While the metal is being introduced raise the thermometer from the water, and replace it as soon as all the metal has been added; stir the liquid well and observe, as accurately as possible, the highest point

reached by the mercury column. Approximate results can be obtained from these data. Calculate as follows:—

Let y = temperature of water before introducing the tin.

“ z = “ “ “ after “

“ w = weight of the water.

“ v = “ of the metal.

“ x = sp. heat—then

$$x = \frac{100(z-y)}{25(100-z)}$$

(Study Richter pp. 256–259.) Would the specific heat found for tin, when multiplied by the constant 6.4 give the same value as that found in experiment (3) for the equivalent of tin? Explain. How many series of tin compounds are there?

(3) *Determine the equivalent weight of Tin.*

Place about 3 grms. of tin in a porcelain crucible that has been previously weighed. Cover the metal with 5–10 cc. of concentrated HNO_3 . Then carefully apply heat by means of an iron plate. The tin is dissolved, while fumes of NO_2 are set free. When the acid has been entirely expelled, heat the crucible with the white stannic oxide over a Bunsen burner; allow to cool and weigh.

Let w = weight of crucible and SnO_2

“ v = “ “ “ “ metallic tin.

“ y = “ “ “

Then $w - v$ = weight of O ,

and $v - y$ = “ “ Sn.

$$\text{Equiv. of Sn} = \frac{(v-y) \times 8}{w-v}$$

Salts.

(4) *Stannous Chloride.*— SnCl_2 .

Dissolve 10 grms. of granulated Sn in warm conc. HCl with the addition of a few drops of PtCl_4 (?). Put the solution into a well-stoppered bottle.

Reactions.

(5) *Stannous Compounds.* Use the chloride solution.

1. Conduct H_2S through a portion of the diluted liquid. A brown precipitate (?) is thrown down. Is it soluble in yellow ammonium sulphide? What does HCl precipitate from the sulphide solution? 2. What is the action of HgCl_2 upon SnCl_2 (see p. 44).

(6) *Stannic Compounds.* Add a few drops of Br to a portion of the SnCl_2 solution, and boil (?). Use the diluted liquid for the tests.

1. Pass H_2S into a portion of the solution. What is the color of the precipitate? Is it soluble in HCl? in $(\text{NH}_4)_2\text{S}$?

2. Add Cu-turnings, boil, decant the liquid, and add HgCl_2 . What happens? Explain.

LEAD.—Pb.

(1) How can this metal be obtained from the oxide? By what physical properties can it be distinguished from other metals? Is it soluble in the mineral acids? (2) In a solution of 5 grms. of lead nitrate in about 50 cc. of water, suspend a strip of metallic Zn and let stand for a few days (?).

Salts.

(3) Dissolve 5 grms. of granulated lead (test-lead) by warming with dilute HNO_3 . Concentrate by evaporation and allow to crystallize.

Reactions.

(4) 1. Before the blowpipe, on charcoal, lead compounds are reduced to metallic beads, which are sectile with the knife. 2. Add HCl to a solution of the nitrate. Boil the precipitate with water. (?) What takes place on cooling? 3. To another portion add dilute H_2SO_4 (?). 4. Pass H_2S into a third portion (?).

BISMUTH.—Bi.

Reactions.

(1) 1. Mix a little of the oxide or nitrate of Bi with Na_2CO_3 and heat in the reducing flame on charcoal. Does the resulting metallic globule resemble lead? Is it sectile? 2. Pass H_2S into a solution of the chloride or nitrate in HCl (?). 3. Add a large volume of water to a bismuth solution. What occurs? What reactions distinguish Al, Sn, Pb and Bi from each other, and from the metals previously studied?

Problems.—1. What is the molecular formula of a mineral containing

SiO_2	=	43.08
Al_2O_3	=	36.82
CaO	=	20.10
		100.00

2. A compound of tin and chlorine yielded on analysis 29.42 parts of Sn and 35.40 parts of Cl; its vapor density was ascertained to be 132.85. What is the atomic weight of tin? 3. Deduce the formula of *Cosalite* from the following analysis:—

S	=	15.27
Bi	=	41.76
Pb	=	40.32
Ag	=	2.65
		100.00

CHAPTER XII.

CHROMIUM, MANGANESE, IRON, NICKEL, COBALT.

CHROMIUM.—Cr.

CHROMIUM AND OXYGEN.

(1) *Chromic oxide*.— Cr_2O_3 .

1. *Preparation*.—Mix intimately 20 grms. of potassium dichromate and 4 grms. of sulphur. Heat the mixture in a porcelain crucible over the blast lamp for about 20 minutes. Cool, extract the residue with boiling water and dry it at a gentle heat. What is its color; is it soluble in dilute HCl? 2. Fuse a portion of it with six times its weight of NaHSO_4 in a platinum crucible. What takes place? 3. Repeat this experiment with some finely powdered *chromite*. (?)

Salts.

(2) *Chromic Chloride*.— CrCl_3 .

Prepare the anhydrous salt. Intimately mix 10 grams of Cr_2O_3 , prepared as described, and 3 grms. of powdered charcoal, and convert this into a dough with a little starch paste. Form the product into balls of the size of a pea; dry, and then ignite these (covered with charcoal powder) in a Hessian crucible, provided with well-fitting lid. Place the residue into a tube of hard glass, and heat it in a current of CO_2 to expel every trace of moisture. With the aid of a blast lamp increase the temperature and replace the CO_2 by a current of Cl. The excess of Cl should be absorbed by conducting it into a bottle filled with caustic soda. (?) The resulting CrCl_3 sublimes to the cooler portions of the tube. Describe its appearance. Is it soluble in water?

What other chlorides are prepared in a similar way? Write the equation, expressing the reaction.

(3) *Chrome Alum*.— $\text{Cr}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4 + 24 \text{H}_2\text{O}$.

Dissolve 10 grms. of $\text{K}_2\text{Cr}_2\text{O}_7$ in a little water; acidify with H_2SO_4 , pass SO_2 into the liquid until the latter is saturated with the gas. Allow to stand; the double salt crystallizes. What is its crystalline form? Dissolve some of it in cold water and note the color of the solution; now warm it. What takes place (see Richter, p. 374)?

(4) 1. Examine crystals of *potassium dichromate*, $\text{K}_2\text{Cr}_2\text{O}_7$. How is it obtained? 2. Dissolve 10 grms. of this salt in water, and from a burette carefully add a caustic soda solution until the color is changed to yellow (?).

What crystallizes from the solution on evaporation? How can you reconvert the product into the dichromate?

Reactions.

(5) 1. Dissolve a minute quantity of a chromium compound in a borax bead. Heat in the oxidizing and in the reducing flame. Results? 2. Heat a little of the compound with KNO_3 on a platinum foil (?)

(6) *Chromic compounds.* Use chrome alum for the tests. 1. Add caustic soda, drop by drop, to a little of the solution. (?) Continue the addition of the reagent till the precipitate is redissolved. What takes place on boiling the solution? 2. What is the action of ammonia on the solution of the chromium salt?

(7) *Chromates.* Use a solution of potassium chromate. 1. Add lead acetate solution. Note the color of the precipitate. Is it soluble in acetic acid? 2. Substitute BaCl_2 for the lead salt in the preceding experiment. (?) 3. Acidify the chromate solution with H_2SO_4 and add H_2O_2 to the liquid. What happens? 4. To some of the chromate solution add a few drops of HCl and about 1 cc. of alcohol. What occurs when the mixture is heated to boiling?

MANGANESE.—Mn.

MANGANESE AND OXYGEN.

(1) In what proportions do these two elements unite with each other? Enumerate the oxides which occur in nature. What is formed when the oxides of manganese are heated in H ? When they are ignited in the air?

Salts.

(2) *Manganous Chloride.*— $\text{MnCl}_2 + 4\text{H}_2\text{O}$.

Evaporate in a porcelain dish the solution obtained in the preparation of Cl from MnO_2 and HCl . Heat the dry residue over a small flame for some time. Add much water and boil. Filter, and to $\frac{1}{10}$ of the filtrate add a solution of Na_2CO_3 in excess. Allow the precipitate (?) to settle, draw off the supernatant liquid with a siphon, and wash the remaining precipitate several times with water by decantation. Add the precipitate then to the principal solution and digest at a gentle heat until a small filtered sample mixed with $(\text{NH}_4)_2\text{S}$ gives a flesh-colored precipitate which is completely dissolved by dilute acetic acid. Now filter and evaporate to crystallization.

(3) *Potassium Manganate*— K_2MnO_4 and

Potassium Permanganate— $\text{K}_2\text{Mn}_2\text{O}_8$.

In a porcelain crucible fuse a mixture of 5 grms. KOH and 2.5 grms. KClO_3 ; gradually add 5 grms. finely powdered MnO_2 . Maintain a moderate red heat for 15 minutes. Dissolve the dark-green residue in a *little* water. Observe the color of the solution. What does it contain? Then dilute with much water and conduct CO_2 into the liquid. Is there any change? If so, write the equation expressing it.

$\text{K}_2\text{Mn}_2\text{O}_8$ as well as K_2MnO_4 are powerful oxidizing agents. Warm a little of the alkaline K_2MnO_4 solution with a few drops of alcohol (?). To a little of the permanganate solution, acidified with H_2SO_4 , add sulphurous acid (?). Treat the acidified solution also with solutions of ferrous sulphate and oxalic acid (?).

Reactions.

(4) 1. What color do Mn-compounds impart to a borax bead in the oxidizing flame? What is the effect of the reducing flame? 2. Heat a little of an Mn-compound with Na_2CO_3 and KNO_3 on a platinum foil. What does the resulting mass contain? 3. To a little of the solution of the chloride in water add $(\text{NH}_4)_2\text{S}$. What is the color of the precipitate. Test its solubility in acids (including acetic acid). 4. Add caustic soda to another portion of the chloride solution. Is the precipitate soluble in an excess of the reagent? Is its color affected by exposure to the air? Explain.

IRON.—Fe.

(1) *Preparation.*—Into a tube of Bohemian glass place a porcelain boat filled with the finely powdered oxide. Pass a current of dry H through the tube, and when all the air is expelled (how could you test it?), apply heat to that part of the tube which contains the boat. What is formed in the anterior portion of the tube? After a red heat has been maintained for 10 minutes allow the boat to cool in H, and examine its contents. Are they attracted by the magnet? Expose the product to air (?).

How is iron obtained from its ores on a large scale? What are its properties? (see Richter, pp. 393 and 394). Distinguish between cast-iron, steel, and wrought-iron.

Salts.

(2) *Ferrous Sulphate.*— $\text{Fe SO}_4 + 7\text{H}_2\text{O}$.

To 25 grms. of Fe in the form of nails or wire, free from rust, contained in a flask, add 200 cc. of dilute (1 : 4) sulphuric acid. When the evolution of the gas (? Note its odor!) is no longer violent, warm, and finally boil until the liberation of gas ceases. A sample of the solution poured



into a test-tube should, on cooling, give a copious separation of crystals. Filter into a casserole containing 2-3 cc. of conc. H_2SO_4 , and let stand for 8 hours. Collect the crystallized product in a funnel the stem of which is closed with a loose plug of glass wool,* allow the mother liquor to drain off, wash with very little cold water (?), and dry between sheets of filter paper. Examine the product carefully. Note its color, taste, solubility in water and crystal form. What other salts of analogous composition are isomorphous with it?

What is observed when some of the salt is heated, first moderately, then strongly, in a tube of hard glass?

Expose the aqueous solution of the salt to the air for several hours (?).

(3) *Ferrous Ammonium Sulphate*.— $Fe(NH_4)_2(SO_4)_2 + 6H_2O$.

In 100 cc. of dilute sulphuric acid dissolve clean iron wire till no more hydrogen is given off; neutralize a like quantity of the acid exactly with ammonia water, and add to it a few drops of dilute sulphuric acid. Filter the iron solution into that of the ammonium salt. Let the salt crystallize, drain it on a funnel provided with a perforated platinum cone, wash and dry as described under (2). Preserve in a well-stoppered bottle. What metals can replace the iron in this salt without altering its crystalline form?

(4) *Ferric Ammonium Sulphate*.— $Fe_2(SO_4)_3 \cdot (NH_4)_2SO_4 + 24H_2O$.

Place 20 grms. of crystallized ferrous sulphate into a porcelain dish together with a few cc. of water and 3.5 grms. of oil of vitriol. Warm on an asbestos plate, adding nitric acid, drop by drop, until no further change of color (?) is observed. Evaporate the excess of HNO_3 , dissolve the residue in hot water and add 3.5 grms. of $(NH_4)_2SO_4$; filter, and set the solution aside for crystallization. Separate the crystals from the mother liquor, and wash and dry them as under (2). To what class of substances does this salt belong? Why?

Reactions.

(5) In a borax bead dissolve a small quantity of an iron compound, and treat it successively in the oxidizing and reducing flames. What changes do you observe?

(6) *Ferrous Compounds*.—Use a freshly prepared solution of ferrous sulphate for the following tests: 1. To a few drops of it, diluted with water, add ammonia. Note the color of the precipitate, and the changes which occur on exposure to the air (?). 2. Add $(NH_4)_2S$ to another portion (?). Is the resulting precipitate soluble in HCl ? 3. In a porcelain capsule bring together a little of the ferrous solution and a drop of

* It is better to use a perforated platinum cone, and to remove the adhering solution with the aid of a filter pump.

Next, dissolve 1 gm. of ferrous ammonium sulphate in 100 cc. distilled water, add 5 cc. H_2SO_4 , and then introduce the permanganate until the final reaction is observed. Calculate the percentage of iron in this salt and compare the experimental result with the theoretical value.

How much oxygen will each molecule of $K_2Mn_2O_8$ give up in oxidizing? How many molecules of FeO can be changed to Fe_2O_3 by a molecule of $K_2Mn_2O_8$?

COBALT.—Co AND NICKEL.—Ni.

Reactions.

1. Dissolve a minute quantity of a cobalt compound in a borax bead. Heat first in the oxidizing, then in the reducing flame (?). 2. What is the behavior of nickel compounds under like conditions? 3. Add caustic alkali to a solution of $Co(NO_3)_2$, warm the mixture (?). What action have caustic alkalies on solutions of nickel salts? 5. To the cobalt solution cautiously add ammonia. After a precipitate (?) has formed, add more of the reagent. What takes place? Expose the resulting solution to the air in a shallow dish (?). 6. Treat a nickel solution in an analogous manner (?). 7. To the solutions of Co and Ni, each in a separate test-tube, add $(NH_4)_2S$. Filter and wash the precipitated sulphides, and test their solubility in acids (?).

Note the colors of cobalt and nickel salts, in the hydrated as well as in the anhydrous state.

Is there any marked difference between Co and Ni in respect to their chemical department?

Point out the differences in the reactions of Cr, Mn, Fe, Co, and Ni.

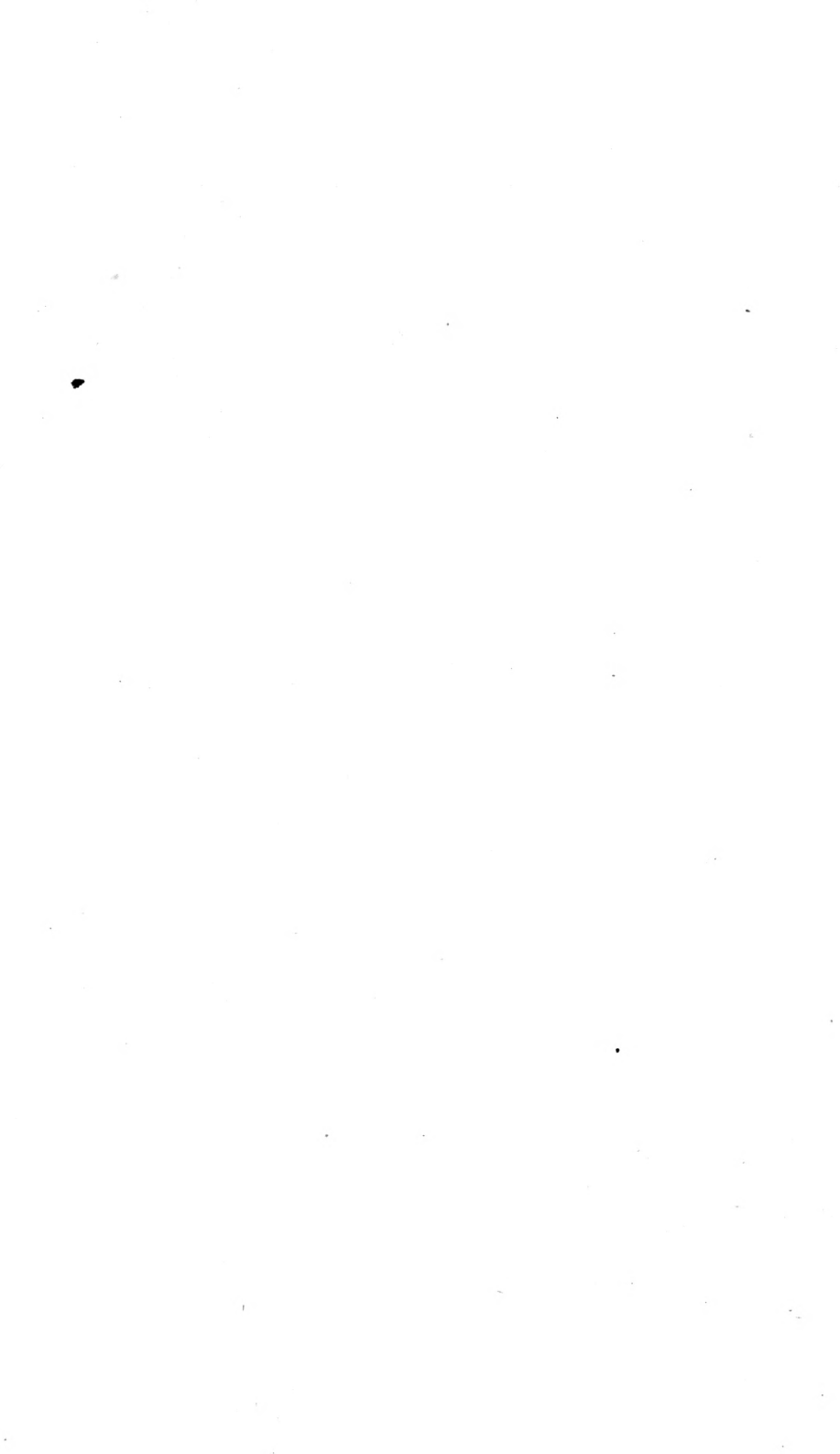
How may ferrous compounds be distinguished from ferric? What conditions are favorable to the conversion of the former into the latter? The latter into the former?

By what means may chromic salts be changed into compounds of chromic acid? How may the reverse change be effected?

Devise a method for separating the elements treated in this chapter.

Problems.—1. How much $K_2Cr_2O_7$ can be obtained theoretically from 100 kilos of a chromite containing 58.6 per cent. of Cr_2O_3 ? 2. 100 grms. of a pyrolusite which was found to contain 4 per cent. of impuri-

ties, will give what volume of O_2 , measured at $20^\circ C$ and 745 mm., when strongly ignited? What is the weight of the residue, assuming that one-half of the impurities was moisture, the other half quartz? 3. How many grms. of HNO_3 are required to oxidize 12 grms. of crystallized ferrous sulphate? 4. What percentage of metallic iron is contained in a salt, of which .7 gm. are exactly oxidized by 17.8 cc. of permanganate solution (standard : 1 cc. = .0056 gm. Fe)?



APPENDIX.

TABLE OF METRIC WEIGHTS AND MEASURES.

MEASURES OF LENGTH.

1 metre = 10 decimetres = 100 centimetres = 1000 millimetres.

1 metre = 1.09363 yards = 3.2809 feet = 39.3709 inches.

MEASURES OF CAPACITY.

1 cubic metre = 1000 litres = 1,000,000 cubic centimetres = 1,000,000,000 cubic millimetres.

1 litre = 61.02705 cubic inches = .035317 cubic foot = 1.76077 pints = .22097 gallon.

MEASURES OF WEIGHT.

1 gram = weight of 1 cc. of water at 4° C.

1 Kilogram = 1000 grams = 100.000 centigrams = 1,000,000 milligrams.

1 Kilogram = 2.20462 lbs. = 35.2739 ounces = 15432.35 grains.

TABLE OF ATOMIC WEIGHTS OF ELEMENTS.

Aluminium . . . Al	27.0	Lead Pb	207.0
Antimony . . . Sb	120.0	Magnesium . . . Mg	24.0
Arsenic As	75.0	Manganese . . . Mn	55.0
Barium Ba	137.0	Mercury Hg	200.0
Bismuth Bi	208.0	Molybdenum . . Mo	96.0
Boron B	11.0	Nickel Ni	59.0
Bromine Br	80.0	Nitrogen N	14.0
Cadmium Cd	112.0	Oxygen O	16.0
Calcium Ca	40.0	Phosphorus . . . P	31.0
Carbon C	12.0	Platinum Pt	195.0
Chlorine Cl	35.5	Potassium K	39.0
Chromium . . . Cr	52.5	Silicon Si	28.0
Cobalt Co	59.0	Silver Ag	108.0
Copper Cu	63.3	Sodium Na	23.0
Fluorine Fl	19.0	Strontium Sr	87.5
Gold Au	197.0	Sulphur S	32.0
Hydrogen H	1.0	Tin Sn	118.0
Iodine I	127.0	Zinc Zn	65.0
Iron Fe	56.0		



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