roughly analogous to this in the magnetization of successive pieces of steel drawn across a lodestone? A certain co-ordination of movement in the molecules of the steel has been induced and magnetism results. So in some manner far more complex, life I believe may be epitomized as co-ordinated motion.

The subject-matter of such speculations lies so far outside our present-day chemistry as to almost require apology for their presentation, but they are well within the subject-matter of the chemistry of the future, for, to again quote the words of Pearson:

"The goal of science is clear, it is nothing short of the complete interpretation of the universe."

Or as Muir has put it:

"The great business of chemistry is to force men into close contact with some aspects of external realities and, with the help of her sister sciences, to remove everything that prevents the full vision of nature."

NOTES.

THE METHODS OF THE UNITED STATES STEEL CORPORATION FOR THE COMMERCIAL SAMPLING AND ANALYSIS OF IRON ORES.

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IRON ORE SAMPLING IN GENERAL.

Owing to the varying conditions under which iron ores must be sampled, both by the producer and the consumer, variations from any uniform procedure of sampling are necessitated. The elements of time, size of shipment, kind of ore and other considerations are determining factors in the details of procuring the sample. Hence it is the purpose to render the following methods general in their scope, promulgating those ideas applicable in the broader sense to ore sampling, without attempting to prescribe for ever varying and unforeseen contingencies in the details.

CAR SAMPLING AT PRODUCER'S END.

All samples must be taken uniformly over the surface of the cars after being loaded by taking a minimum of twelve places for wooden, and fifteen places for steel cars, by either the parallel or the zigzag system. The following diagrams illustrate these two systems and show the minimum number of points where samples are to be taken.

PARALLEL SYSTEM.

25 TON WOODEN CAR.

50 TON STEEL CAR.

ZIGZAG SYSTEM.

25 TON WOODEN CAR.

50 TON STEEL CAR.

When lumps of ore are encountered at the designated points where samples are to be taken, small portions of each lump must be chipped off, about equal in size to the first joint of the thumb. When rock occurs it must also be sampled as ore, that is, a proportionate amount of the rock

or off grade material is to be taken, not equal in amount to the regular sample for the area covered by that point, unless the entire area is such off grade material. Then the same amount of adjacent material must be taken, ore or rock, equal in amount to the portion taken at each regular point. This also applies to field boulders, unless thrown from cars.

The samples are taken with a garden trowel. Each ten cars, either steel or wooden, must be combined, as a rule, into one sample; but less than ten cars may be grouped. The weight of the sample must not run under fifteen pounds for ten wooden, and twenty pounds for ten steel cars.

Whenever very lumpy ore is to be sampled in the cars, the rope net system is used as shown below, which gives about thirty-two places on each car, the knots being eighteen inches apart. In using the net system, if a lump of ore or rock comes directly under the knot, a piece should be taken about the size of the first joint of the thumb. If fine ore occurs under a knot, an equal amount is taken with the trowel. The rope net system is used at the Marquette Range on the hard, lump ore; also at the Soudan Mine, Vermilion Range.

A moisture sample must be taken for each train load from all open pit mines; a train consists of forty to forty-five of the fifty ton steel cars, or sixty to sixty-eight of the twenty-five ton wooden cars. A moisture sample must also be taken for all the cars loaded at a shaft or stockpile during each ten hour shift. Samples must be taken from three places on top of each car as shown by the following diagram.

Care should be taken to secure the sample from well underneath the surface as soon as practicable after loading, maintaining the true proportion of lump and fine ore. The sample as taken must be immediately placed in a can with a tightly fitting lid, and brought to the crusher house. It is optional to take a moisture sample from the regular sample, provided it has been taken from well underneath the surface.

CARGO SAMPLING AT CONSUMER'S END.

Cargo ores present the most serious obstacles to a uniform method of sampling. The boats vary in size from three thousand to twelve thousand tons with one or two decks, and in the number of hatches from six to thirty-six with widths varying from twelve to twenty-four feet. The

grabs at the different unloading points vary in number, kind, size, and the rapidity of their operation. The ores vary from the very fine to the all lump, from the so-called mixed ores such as the groups to the mixed cargoes, consisting of different ores in the same boat, and with different ways of loading the boats.

Grab Sampling.—An excellent method of cargo sampling where the entire cargo will be represented, and particularly adaptable for fine ores is called grab sampling. A sampler with a small scoop attached to a handle of suitable length and holding a definite amount of ore, a quarter, a half, or a whole pound, takes a scoopful from each grab as it rises above the deck. The disadvantage of this system is its increased cost due to the extra number of samplers, one being required for each grab during the entire time of unloading.

The general plan for the sampling of all cargoes is to first sample the tops of the piles, before the grabs have started to unload; this is called sampling of the cones. After the grab has removed from the hatch all the ore within reach, the exposed faces standing on each side are sampled; this method is known as face sampling. Or when the latter practice is impracticable owing to the operation of the grabs, then the method of rounds is followed.

In sampling, a small shovel or garden trowel is used, the total length of which, including the handle, is twelve inches, and it also constitutes a measure. It is the aim to take equal sized samples from each of the points selected. When lump ore or rock is encountered at the point determined by the measure a portion is broken off equal to the amount regularly taken.

In the sampling of the cones, at a point midway between the side and the center of the boat, directly under the edge of the hatch, the first sample is taken and sampling continued one shovel length apart up the surface of the cone, over its apex and down the opposite surface to a corresponding point under the other edge of the hatch. This line is crossed from corresponding opposite points under the hatch as shown in the following sketch. Not more than one-tenth of the total sample is to be taken in the sampling of the cones.

CONE SAMPLING.

Face Sampling.—After a grab has removed from a hatch all the ore within reach and has moved to another hatch, the sampler shall measure two shovel lengths from the side of the boat and start up the exposed face of the ore taking samples one shovel length apart all the way to the top, using

a ladder if necessary. The next vertical line is measured four shovel lengths from the first and the samples taken each shovel length apart on this line as before, and so on for each succeeding line across the boat. This is repeated on

the opposite ore face, and the entire procedure continued until the ore faces of all the hatches are sampled that the character of the boat and the operation of the grabs will permit. When a bulkhead occurs, only the face opposite to it is to be sampled.

FACE SAMPLING.

Round Sampling.—When the operation of the grabs makes sampling by the face method impracticable, or with boats having twenty-four foot hatch centers, and decks furnishing protection to the samplers, then sampling shall be done while the ore is being removed by the grabs. When five or six feet of the face of the ore have been exposed, the sampler shall enter the hatch and measuring two shovel lengths from the side of the boat or edge of the face, take successive samples up the face one shovel length apart. The next vertical line is measured four shovel lengths from the first,

and samples are taken all the way to the top as before, and so on across the entire face of the ore. This procedure is repeated on the opposite face, one-third of the total weight of the sample to be taken in the first round. When all the ore within reach of the grab is removed, the second round is taken, using the measurements as above, and the remaining two-thirds of the sample are secured.

Part of the regular sample is to be taken for the moisture sample, and for the fineness sample when such is desired.

CAR SAMPLING AT CONSUMER'S END.

When the ore is received in cars the greatest possible number are represented in the samples, and not less than ten equal sized samples are taken from each car. When cars are loaded with fine ore with the piles in opposite ends, at least five samples are taken from each pile, the first one at the apex of the pile, and the other four at points symmetrically arranged around the sides of the pile, two-thirds of the distance from the apex to the base of the pile or sides of the car. With cars loaded in the center, the system is the same, except that the center of the side of the pile lengthwise of the car, is the first point, the other four being symmetrically arranged around this point.

When the ten points are located in a car, each of them is supposed to represent a definite area, equal to one-tenth of the ore surface of the car. If the car contains all fine ore, then ten equal sized samples are taken, one from each of the points. If the car contains a mixture of fine and lump ore, with varying amounts of each in the areas included in the different divisions, then each area is judged separately and sampled accordingly. The fine and lump ore are taken each in its proper proportion, the former with the trowel, the latter being chipped, or selected small pieces being taken, each about the size of the first joint of the thumb. The combined sample of fine, chipped and selected pieces from

each area, equals the amount taken were it all fine ore. If the contents of the car are all lump ore, the proper sized pieces are chipped from four or five of the lumps in each of the ten areas making forty or fifty pieces from each car, the total amount of chipped pieces from each of the areas equaling the amount that would be taken were it all fine ore. All samples of fine ore are taken from well underneath the surface to obtain the ore in its natural state. A proportionate amount of the main sample is retained in a tightly closed can for the moisture determination.

PREPARATION OF SAMPLES IN GENERAL.

In the preparation of the sample for analysis, the ideal practice is to crush and quarter alternately until the desired quantity with the requisite degree of fineness is attained. A more expeditious and equally efficient method, is to crush the entire sample to the desired degree of comminution, then reduce the quantity by successive quartering as before, until the desired amount remains. It should ever be our purpose to approach as closely as possible to either of these two methods in the preparation of all samples for analysis.

PREPARATION OF SAMPLE AT PRODUCER'S END.

Samples before being quartered are brought into the crusher house where they are dried, if necessary, at 100° C. until the ore can be well mixed. Care must be taken to prevent overheating when other than low pressure steam is used for the purpose, especially with ores containing a large quantity of limonite. When sufficiently dry the larger lumps are crushed, if necessary, so that the entire sample will pass through a half-inch mesh sieve; a finer sieve may be used if desired. The sample is thoroughly mixed on an iron top table, then spread out evenly about three-fourths of an inch in depth and alternately quartered and mixed until one-fourth of the original sample remains. It is now crushed until fine enough to pass a quarter-inch mesh sieve, then mixed and quartered as before until about two pounds remain. It is again crushed until fine enough to pass a twenty mesh sieve, and spread out evenly about three-eighths of an inch in depth. About three ounces of this are taken from all over the pile, with a small spatula, dried in a small pan at 100° C. and crushed on a chrome steel plate, until it will all pass through a hundred mesh sieve. After being thoroughly mixed this is transferred to a bottle, and constitutes the sample for analysis.

The following method for the preparation of the sample is optional. All the ore is passed through a half-inch sieve, thoroughly mixed on a suitable cloth and quartered in the usual way, diagonally opposite portions being rejected until about five pounds remain. The sample is again crushed, if need be, to pass a quarter-inch mesh sieve, and mixed and quartered as before until one and one-half to two pounds remain. After being dried at 100° C. for twenty to thirty minutes, it is crushed to a fineness of twenty mesh, mixed and quartered until about three ounces remain, and the entire sample is crushed on a chrome steel plate and passed through a hundred mesh sieve. This sample is spread out in a shallow pan, dried at 100° C. for thirty minutes, again mixed and transferred to a three-ounce bottle or can for the analysis.

PREPARATION OF SAMPLE AT CONSUMER'S END.

The aggregate sample is dried at 100° C. and crushed

before any quartering whatsoever, so that the entire mass will pass through a half-inch mesh sieve. This is reduced by successive quartering and crushing until its weight is from four to eight ounces, and it will then all pass through an eighth-inch-mesh sieve. Or the entire sample is crushed so it will pass through an eighth-inch-mesh sieve, and then quartered as before until from four to eight ounces remain. And this final quantity is then further crushed with a chrome steel bucking board and muller and all passed through a hundred-mesh sieve. A sufficient amount of this powder for all the needs of the analyst is placed in a small air-tight container, dried for one hour at 100° C. and when cool it constitutes the sample for analysis. A separate, larger portion of the same sample is retained for further needs.

IRON.

By the Bichromate or Permanganate Methods.

One or more portions of one-half to one gram of the ore are placed in a beaker, and 5 to 10 cc. of the solution of stannous chloride and 5 to 25 cc. of strong hydrochloric acid are added. This variation in the amount of acid is due, aside from the variations in the weight of sample and the varying solubilities of the ores, to the fact that while in the permanganate method the minimum amount of free acid should be present, with the bichromate method an excess of acid is essential. The beaker is covered with a watch glass and heated gently on the steam bath, or hot plate, until all iron is dissolved, and if the permanganate method is to be used, the volume of the solution is reduced by evaporation so as not to exceed 10 cc. While still hot the stannous chloride solution is added from a burette, drop by drop, until the color due to the ferric chloride just disappears, and then one drop in excess. The solution is diluted to about 350 cc. with cold water, 5 cc. of mercuric chloride solution are added and the solution is thoroughly stirred. From this point the bichromate or permanganate titrations are optional. In the former case, the potassium bichromate solution is added from a burette until a drop of the solution being titrated, added to a drop of the potassium ferricyanide solution on a paraffined plate shows no ferrous iron. In the latter case, 10 cc. of the titrating solution are added for a one-half gram sample, and 20 cc. for one gram. The solution is stirred and titrated with the potassium permanganate. In either case, the number of cc. used multiplied by the respective factor gives the percentage of iron.

If the residue is suspected of containing iron it is filtered off, ignited and fused; the fusion is dissolved in water, acidified with hydrochloric acid, and the solution reduced and titrated as above; or the solution before being acidified is filtered, the ferric oxide on the filter is dissolved in hydrochloric acid and added to the main solution or titrated separately.

SOLUTIONS FOR IRON TITRATIONS.

The stannous chloride solution is made by dissolving 250 grams of the salt in 500 cc. of strong hydrochloric acid, and diluting to 1000 cc.

The mercuric chloride solution is saturated in the cold.

The potassium ferricyanide solution is made fresh each day by dissolving in the proportion of 0.100 gram of the salt in 100 cc. of water.

The potassium bichromate solution is made by dissolving the salt in the proportion of about 8.9 grams to one liter of water, and standardizing with an iron ore, iron wire or

steel, of known iron content. One cc. equals approximately 1 per cent. iron with a one gram sample taken for analysis.

The titrating solution is made up in the following proportion to the liter: 90 grams of manganous sulphate are dissolved in 650 cc. of water, 175 cc. of strong sulphuric acid are added, then 175 cc. of phosphoric acid of 1.75 sp. gr., and the solution is filtered if necessary.

The permanganate solution is made by dissolving the salt in the proportion of about 5.7 grams to 1 liter of water and standardizing in the same manner as the bichromate solution. One cc. equals approximately 1 per cent. iron with a one gram sample taken for analysis.

SILICA.

Using One Gram with Hydrofluoric Evaporation.

One gram of the sample is transferred to a porcelain dish, or a beaker, with watch glass cover, and 20 cc. of strong hydrochloric acid are added. A gentle heat is applied until the ore is dissolved and the solution is then evaporated to dryness, unless it has been conclusively shown to be unnecessary.¹ To the residue, which should not be heated above 120° C., 15 cc. of dilute hydrochloric acid (1 part acid to 1 part water) are added, heat is applied until the salts are dissolved and the solution is diluted with hot water. The precipitate is filtered off, washed, ignited at the highest temperature of the blast lamp or muffle furnace for at least five minutes, cooled and weighed. To the residue in the crucible one or two drops of strong sulphuric acid and 5 cc. of hydrofluoric acid are added, and the solution is evaporated to the expulsion of sulphuric acid. The residue is ignited as before and again weighed; the difference between the two weights is silica.

SILICA.

Using One to Five Grams when All or Part of the Filtrate Is Used for the Phosphorus or Other Determinations.

One to five grams of the sample are transferred to a dish (beaker optional) with a watch glass cover, 20 to 50 cc. of strong hydrochloric acid are added, and a gentle heat is applied, without boiling, for about one-half hour. The solution is diluted with an equal volume of water, filtered into another dish of the same size, and the visible iron stain is washed out of the filter paper. This solution containing the bulk of the iron is allowed to go to dryness on the steam bath. The residue is ignited in a platinum crucible, and then fused with about six times its weight of sodium carbonate. The fusion may be leached out of the crucible, but it is found more expeditious to let it cool around the end of a platinum rod, and on again heating the crucible the entire mass adhering to the rod is removed. It is then dissolved in the original filtrate, or placed while still hot in the covered dish, in which the original solution was made and to which about 25 cc. of hot water have previously been added. The small amount remaining in the crucible is dissolved in hot water, and finally in hot dilute hydrochloric acid (1 part acid to 1 part water), and added to the dish containing the fusion, which by this time has been disintegrated by the hot water and acidified with hydrochloric acid; this dish is then placed with the other on the steam bath. When the contents of both dishes are dry, 10 to 15 cc. of strong

¹ This clause is introduced on the insistence of the mine chemists that with certain ores evaporation to dryness is not necessary to obtain all the silica.

hydrochloric acid are added to the dish containing the residue of the original filtrate. After heating until the ferric salt is dissolved, the solution is evaporated until the excess of hydrochloric acid is expelled, as is indicated by the first appearance of insoluble ferric chloride on the bottom or sides of the dish. Seven cc. of strong nitric acid are added and heat is applied for about one minute, or until the violent reaction is over. The solution is diluted with cold water and filtered into a suitable flask. This is the procedure if all the filtrate is to be used for the phosphorus determination; but the evaporation of the excess hydrochloric acid and the addition of the nitric acid are dispensed with, if the united filtrates are to be received in a volumetric flask and aliquot portions taken for the other determinations.

To the dish containing the fusion, and which should not be heated above 120° C., just enough dilute hydrochloric acid is added to moisten the residue. Hot water is then added and heat applied until all the salts are dissolved, when the solution is filtered into the same flask with the last filtrate; the combined filtrates should not exceed 150 cc. if the solution is to be used for the phosphorus determination. In this case, the complete washing of the silica is continued in another flask until free from chlorides, and these washings are discarded. The filter and its contents are then ignited and weighed as silica. The filtrate in the flask is reserved for the phosphorus determination, or if in a volumetric flask, for the determinations which follow.

PHOSPHORUS.

By the Fusion Method.

For this determination, five grams of the sample are taken for all ores when the phosphorus content is under 0.100 per cent., two and one-half grams when the phosphorus content is between 0.100 and 0.200 per cent., and one gram when the phosphorus exceeds 0.200 per cent.

An aliquot part of the filtrate, the volume of which should not exceed 150 cc., representing one gram, or two and a half grams of the sample, from the five-gram portion for silica in the volumetric flask, is boiled in a suitable flask for a few minutes with the addition of 10 cc. of strong nitric acid. To this solution, or to the entire filtrate in the flask from the five-gram portion, a slight excess of strong ammonia is added over the amount necessary to precipitate the iron and alumina, and after mixing enough strong nitric acid to dissolve the precipitate with from 5 to 10 cc. in excess.

The solution is heated to a temperature of 80° C., 50 cc. of the solution of ammonia molybdate are added, and the flask is shaken continuously for five minutes. The solution is allowed to stand until the precipitate has subsided. If the yellow precipitate is to be weighed, it is caught on a 9 cm. filter paper that has been dried for at least half an hour at 110° to 120° C. and weighed between watch glasses, washed thoroughly with a 2 per cent. solution of nitric acid, dried for one hour at the above temperature and weighed between glasses; 1.63 per cent of its weight is phosphorus.

If the yellow precipitate is to be titrated by the acid alkali method, the weighing of the filter is dispensed with, the washing with the 2 per cent. nitric acid is continued as before to the complete removal of the iron and molybdenum salts, and then with a 1 per cent. solution of potassium nitrate until free from acid. The filter and its contents

are then transferred to a small flask or beaker and an excess of the standard alkali added. When the precipitate is all dissolved by shaking or stirring, about 150 cc. of water and a couple of drops of a saturated solution of phenolphthalein in alcohol are added, and the solution is back titrated with the standard nitric acid to the disappearance of the color. It is optional to again add the standard alkali, the end point being a faint, permanent, pink color.

The yellow precipitate may also be dissolved in dilute ammonia, and the phosphorus determined by the permanganate titration method as subsequently described. Or it may be dissolved in a hot ammoniacal citrate solution and the phosphorus precipitated as ammonium magnesium phosphate, as hereafter described.

Ammonium Molybdate Solution.—This solution is made in the following proportions: to 225 grams of pure molybdic acid in a suitable flask there is added 500 cc. of water, and then 500 cc. of strong ammonia. When in solution, this is poured into a flask containing 2500 cc. of nitric acid of 1.20 sp. gr., the solution being kept agitated, and three or four drops of ammonium or sodium phosphate solution are added to coagulate the suspended impurities. The solution is kept in a warm place over night or until the supernatant liquid is clear, when it is filtered into a bottle for use.

Stock Solution of Caustic Soda.—145 grams are dissolved in water and the solution is diluted to 2200 cc.; 200 cc. of this solution diluted to 2000 cc. will be of approximate strength.

Nitric Acid.—20 cc. of strong nitric acid are diluted to 2000 cc. These solutions are made exactly agreeing and standardized on steels or ores of known phosphorus content so that each cc. used corresponds to 0.01 per cent. phosphorus, when two grams of the sample are used. This weight has been found the most convenient for steel analysis, and a simple calculation enables it to be used for varying weights of either steel or ore.

PHOSPHORUS.

Direct Determination of the Soluble Phosphorus Using the Permanganate or the Acid Alkali Titration.

This method is particularly applicable to ores wherein all or a fairly constant proportion of the phosphorus is soluble in strong acid. The insoluble phosphorus is determined at less frequent intervals, and the amount so obtained added to the soluble gives the total phosphorus.

From one to five grams of the sample, according to the phosphorus content, are placed in a beaker with a watch glass cover, 20 to 50 cc. of strong hydrochloric acid are added and a gentle heat is applied until the ore is dissolved. Two or three drops of nitric acid are added and the solution is boiled a minute or two, and then the excess acid evaporated. The solution is diluted and filtered, the filtrate being received in an 8 to 16 ounce Erlenmeyer flask; the filter and residue are washed with dilute hydrochloric acid (1 part acid to 4 parts water) and water until free from iron discoloration.

Strong ammonia is now added until a slight precipitate persists, or until the iron is completely precipitated, and in either case this is dissolved with strong nitric acid, too great an excess being avoided. The solution is heated to 80° C., 30 to 50 cc. of ammonium molybdate solution are added, the flask is shaken and placed in a warm place to allow the precipitate to subside. The greater part of the supernatant

liquid is siphoned off, the remainder being filtered, or the entire solution is filtered. The precipitate is washed with ammonium sulphate solution or a 2 per cent. sulphuric acid solution, until free from iron and molybdenum salts.

The funnel containing the precipitate is transferred to the flask in which the precipitation was made, and the precipitate dissolved in dilute ammonia (1 part ammonia to 3 parts water); the resulting solution should not exceed 25 cc. Eight grams of granulated zinc (20 mesh) are added, then 75 cc. of sulphuric acid (1 part acid to 4 parts water), and the solution is boiled gently until reduction is complete. It is then filtered through glass wool or cotton, the filtrate being received in a beaker, and the flask and filter are washed thoroughly with cold water. The solution is titrated with potassium permanganate until a slight, permanent, pink tint is obtained. From the number of cc. used, the percentage of phosphorus is calculated.

If the acid alkali titration is to be used, the yellow precipitate is washed as before with a 2 per cent. nitric acid solution until free from iron and molybdenum salts, then with a 1 per cent. solution of potassium nitrate until free from acid, and the determination is completed in the regular manner.

SOLUTIONS.

Standard Permanganate.

204.5 grams potassium permanganate,
1 carboy (48 liters) distilled water.

Ammonium Molybdate.

300 grams molybdic acid dissolved in
1200 cc. ammonium hydrate, sp. gr. 0.96, this to be
added slowly to
4500 cc. nitric acid, sp. gr. 1.20, keeping the solution
agitated.

Ammonium Sulphate.

40000 cc. distilled water,
1100 cc. ammonium hydrate, sp. gr. 0.96,
960 cc. sulphuric acid, sp. gr. 1.84.

Phenolphthalein.

Saturated solution in alcohol.

PHOSPHORUS.

By Ignition of the Insoluble Residue.

One to five grams of the sample in a porcelain dish, with watch glass cover, are digested in a 20 to 50 cc. of strong hydrochloric acid with the aid of a gentle heat. After diluting slightly with cold water, the solution is filtered into another dish of the same size, and the precipitate washed free of the visible iron discoloration. The filtrate is rapidly evaporated and while this is in progress, the residue is transferred to a platinum crucible and ignited in a muffle furnace or over a blast lamp. The residue is broken up, reignited for a few minutes, then transferred into the original filtrate and the solution is evaporated to dryness.

Fifteen cc. of strong hydrochloric acid are now added and the solution is evaporated until ferric chloride begins to separate in the solid state. To the covered dish, 7 cc. of strong nitric acid are added and heating is continued for about one minute. The solution is diluted with cold water and filtered into a suitable flask. The filter and precipitate are washed with a 2 per cent. nitric acid solution until the iron discoloration is removed.

With unknown ores, and at regular intervals with

all ores, as a check, the insoluble residue is fused with sodium carbonate. The fusion is dissolved in dilute hydrochloric acid (1 part acid to 1 part water), and the solution is evaporated to dryness. The residue is redissolved in dilute hydrochloric acid and the solution is filtered into the main filtrate, which has also been evaporated to dryness, and the residue treated as described in the previous paragraph after the evaporation to dryness. The filtrate, or the combined filtrates in the flask are made ammoniacal, and the precipitate is dissolved in strong nitric acid with 5 to 10 cc. in excess. The solution is brought to a temperature of 80° C., 50 cc. of ammonium molybdate solution are added and the flask is shaken for five minutes.

When the precipitate has subsided, it is filtered off and washed with a 2 per cent. solution of nitric acid. From this point the yellow precipitate may be weighed, or titrated by the acid alkali or permanganate methods as previously described, or dissolved, reprecipitated, and weighed as magnesium pyrophosphate in the following manner.

The yellow precipitate is dissolved on the filter with hot ammonium citrate solution (175 cc. strong ammonia to 325 cc. water containing 25 grams citric acid), the filtrate being received in a No. 0 beaker. The filter is washed several times with hot water, then 5 to 10 cc. of magnesia mixture (100 grams magnesium sulphate plus 300 grams ammonium chloride in 800 cc. hot water plus 400 cc. strong ammonia) are poured through the filter. The filtrate in the small beaker is thoroughly mixed and allowed to stand for at least three hours, and if possible over night. The precipitate is filtered off, washed with dilute ammonia (1 part ammonia to 4 parts water), ignited at a low temperature until the carbon of the filter is destroyed, then at the highest temperature of the muffle furnace or burner, cooled and weighed as magnesium pyrophosphate containing 27.84 per cent. phosphorus.

PHOSPHORUS.

When Titanium is Present.

Two grams of the sample are intimately mixed with eight to ten times its weight of sodium carbonate in a large platinum crucible and fused over a blast lamp, the fusion being maintained at the highest temperature for fifteen to twenty minutes. The fusion is then transferred to a beaker containing hot water and thoroughly disintegrated; the solution is then filtered and the residue washed with hot water, or preferably with a 1 per cent. solution of sodium carbonate. As an extra precaution, not deemed necessary in every-day work, the residue may be ignited until the filter is destroyed, crushed in the crucible, fused, disintegrated and filtered off as before, the filtrates being combined. The filtrate, or the combined filtrates are acidified with hydrochloric acid and evaporated to dryness. The residue is moistened with dilute hydrochloric acid, dissolved in hot water and the solution filtered into a suitable flask.

The filtrate is made ammoniacal, then strong nitric acid is added with from 5 to 10 cc. in excess. The solution is brought to a temperature of 80° C., 50 cc. of ammonium molybdate solution are added, the flask is shaken for five minutes, and the phosphorus determined by any of the methods previously described.

When it is desired to separate the phosphorus from the large amount of sodium salts in the filtrate after the evapo-

ration to dryness, a weighed amount of iron, about 0.05 gram, of known phosphorus content, and in the ferric condition, is added and precipitated in the hot solution with ammonia. The precipitate is filtered off, dissolved in warm, dilute nitric acid, and if the resulting filtrate is somewhat cloudy from particles of filter paper in suspension, and it is desired to remove them, it is refiltered after the addition of a little paper pulp. This filtrate is now made ammoniacal, 5 to 10 cc. excess nitric acid are added, and the determination is completed as above described.

ALUMINA.

Phosphate Method.

For this determination the hydrochloric acid filtrate from the silica of one gram of the sample, or the hydrochloric acid solution of the precipitated iron and alumina from the gravimetric determination of manganese is used. To the cool solution diluted to about 400 cc. in a No. 5 beaker, 30 cc. of a 10 per cent. solution of ammonium phosphate are added, and then ammonia until a faint, permanent precipitate is formed. One and one-half cc. of strong hydrochloric acid are now added, the solution is stirred until the precipitate is dissolved, and 50 cc. of a 20 per cent. solution of sodium hyposulphite are added. The solution is heated just to the boiling point, at which 8 cc. of strong acetic acid and 15 cc. of a 20 per cent. solution of ammonium acetate in the same measure are added, and the boiling is continued ten minutes. The precipitate is allowed a few minutes to subside, is filtered off as quickly as possible and washed ten times with hot water. The filter and its contents are ignited at a low temperature until the carbon is destroyed, then at the highest temperature of the muffle furnace, cooled and weighed as aluminum phosphate, 41.85 per cent. of which is alumina.

ALUMINA.

Ether Method.

One to five grams of the sample are placed in an 8 ounce beaker and dissolved in 20 to 50 cc. of strong hydrochloric acid with the aid of a gentle heat. The solution is diluted and filtered, the residue washed, and the filtrate evaporated to dryness. The residue on the filter is ignited in a platinum crucible, 2 to 6 drops of sulphuric acid (1 part acid to 1 part water) and 5 to 10 cc. of hydrofluoric acid are added and the solution is evaporated to dryness. The residue in the crucible is fused with one-half gram of potassium or sodium bisulphate and dissolved in 10 cc. of dilute hydrochloric acid (1 part acid to 1 part water). The residue from the original filtrate, after its evaporation to dryness, is dissolved in 20 to 30 cc. of hydrochloric acid, 1.13 sp. gr., cooled and transferred to a separatory funnel, 40 to 100 cc. of ether are added and the funnel is shaken for one minute, the solution being kept cool. The acid layer is drawn off into an 8 ounce beaker, and to this is added the solution obtained from the treatment of the insoluble residue. The combined solutions are heated until the ether is driven off, a few crystals of sodium or potassium chlorate are added, and the heating is continued until free chlorine is expelled. The solution is diluted to about 75 cc., ammonia is added until a slight precipitate persists, then 10 cc. of a 20 per cent. solution of ammonium acetate. The solution is boiled about one minute and filtered; the precipitate is washed

with hot water, then dissolved in warm dilute hydrochloric acid. After being diluted to about 75 cc. and a slight excess of ammonia added, the solution is boiled, filtered and the precipitate washed as before. The combined filtrates are reserved for the determination of manganese, lime and magnesia. From this point, the alumina may be determined by either of the following methods:

The precipitate is ignited, and weighed as oxides of iron, alumina and phosphorus. The ignited oxides are dissolved in 3 cc. of stannous chloride and 10 cc. of strong hydrochloric acid, and the iron is titrated with a weak bichromate solution, 1 cc. of which is equal to 0.2 per cent. iron. Or the precipitate is fused with sodium carbonate, the fusion is dissolved in hydrochloric acid and the iron titrated as above. The iron calculated as the sesquioxide, plus the phosphorus as pentoxide is deducted from the total weight of the precipitate, leaving the alumina.

Or the filter and precipitate are ignited at a low temperature until the carbon is destroyed. The precipitate is crushed in the crucible and thoroughly fused with about 5 grams of sodium carbonate. The fusion is dissolved in hot water, a little paper pulp is added to reduce any manganese that may be present, the residue is filtered off and washed. The filtrate received in a small beaker is acidified with hydrochloric acid, heated to boiling, and ammonia is added in slight excess. After boiling for a minute or two, the precipitate is filtered off, washed with hot water, ignited and weighed. This weight minus the weight of the phosphorus as pentoxide is alumina.

MANGANESE.

Gravimetric Method.

The filtrate from the one-gram portion for silica, or an aliquot part of the filtrate, equal to one gram of the ore, from the five-gram portion for silica in the volumetric flask, is boiled for a few minutes in a No. 5 beaker with the addition of 10 cc. of strong nitric acid. A basic acetate separation is then made as follows. The solution is diluted with cold water to about 150 cc. and the greater part of the free acid is neutralized with strong ammonia, then cautiously with dilute ammonia (1 part ammonia to 4 parts water) with vigorous stirring until a faint precipitate persists. The solution is then diluted with hot water to about 400 cc. 25 cc. of a 20 per cent. solution of ammonium acetate are added, the solution is heated to boiling and boiled about one minute. After the precipitate has subsided, the solution is filtered through a 185 mm. filter on a 4-inch ribbed funnel into a No. 6 beaker.

When the manganese is under one per cent., only one basic acetate separation is made and the precipitate is washed several times with hot water. If over one per cent., the precipitate is washed but once, then dissolved on the filter in hot, dilute hydrochloric acid and the basic acetate separation made as before, except that the dilution before precipitating need not be so great. The filtrate and washings, or the combined filtrates in this determination, or from the iron and alumina in the ether alumina method are heated to boiling and 25 cc. of strong ammonia added, and then cautiously about 1 gram of ammonium persulphate. One gram is sufficient to precipitate the manganese in any iron ore. The solution is boiled about ten minutes and the precipitate filtered off, washed with hot water, ignited at

a gentle heat and then at the highest temperature of the muffle furnace or blast lamp, and weighed as manganomanganic oxide, containing 72.05 per cent. manganese. This precipitate may be dissolved before or after ignition, reprecipitated, and weighed as manganese pyrophosphate.

The ignited precipitate of manganomanganic oxide is usually contaminated with a small amount of silica sufficient to cause an appreciable error in a small percentage of manganese. In this case it is dissolved in the crucible in warm, dilute hydrochloric acid; the residue, if any, is filtered off, washed, burned and the weight obtained is deducted from the previous weight.

The precipitate of iron and alumina on the filter obtained in this determination is to be retained for the determination of alumina by the phosphate method.

MANGANESE.

Volhard's Method.

One to two grams of the sample are placed in a No. 2 beaker with cover, 15 to 25 cc. of strong hydrochloric acid are added, and heat is applied until the ore is dissolved. Five cc. of nitric acid or a few crystals of potassium chlorate are now added, and the solution is boiled until nitrous fumes or free chlorine are expelled. Evaporation is continued to the expulsion of free acid, the solution is diluted to about 100 cc. and washed into a volumetric flask. Zinc oxide emulsion is added in excess, the solution is diluted to the mark and mixed thoroughly; the precipitate is allowed to subside and an aliquot portion is decanted off, equal to one-half or one gram of the ore. For ores low in manganese, the solution is made slightly turbid with zinc oxide emulsion to coagulate the manganese dioxide set free in the titration. The solution is heated to boiling and titrated with permanganate until a faint, pink color persists. Or, after the dilution to about 100 cc., the solution is washed into a half liter flask, an excess of zinc oxide emulsion is added as before, the solution is heated to boiling and titrated directly in the presence of the iron precipitate.

Strength of permanganate solution for iron ores: 1 cc. equals 0.1 per cent. manganese, standardized on iron ore of about one-half per cent. manganese content.

For manganese ores, the procedure is the same up to the removal of the free acid and the addition of zinc oxide emulsion, when the solution is diluted to the mark in a 500 cc. volumetric flask and mixed. Then 50 cc. are transferred to a flask, diluted to about 250 cc., heated to boiling, and titrated as before. No zinc oxide emulsion need be added here, the voluminous precipitate of manganese dioxide acting as coagulant.

Strength of permanganate solution for manganese ores: 1 cc. equals 0.3 per cent. manganese, standardized on manganese ore of known manganese content.

MANGANESE.

Color Comparison or Titration Method.

One gram of the sample in a porcelain dish, or beaker, is warmed after the addition of 25 cc. of strong hydrochloric acid and 5 cc. of strong sulphuric acid, and when in solution, the heating is continued until fumes of sulphuric acid appear. The solution is allowed to cool, 25 cc. of water are added, heat is applied until the salts are dissolved and the solution is transferred to a 50 cc. volumetric flask, diluted to the mark

and mixed. If the color comparison method is to be used, the solution should be poured through a dry filter after mixing; if the titration method is to be used, the filtration may be omitted. With a pipette 10 cc., equal to 0.2 gram of the ore, are transferred into a 1-in. by 8-in. test tube, if for comparison, or into a 150 cc. Erlenmeyer flask, if to be titrated. Fifteen cc. of the nitrate of silver solution are added, the solution is heated over a flame or in a water bath, and about one gram of ammonium persulphate is added. The reaction takes place equally well in a nitric or sulphuric acid solution or a mixture of the two. The essential point is the presence of a sufficient amount of silver nitrate. As soon as the formation of the permanganic acid is well under way, the test tube or flask is removed from the source of heat and placed in a cold water bath. This solution may be compared against the color obtained from an ore or a steel of known manganese content, in the latter case by dissolving 0.2 gram of the steel in 10 cc. of nitric acid of 1.20 sp. gr., adding 15 cc. of the silver nitrate solution and the persulphate as above.

If the solution in the 150 cc. Erlenmeyer flask is to be titrated, it is diluted to about 100 cc., 7 cc. of a 0.2 per cent. solution of C. P. sodium chloride are added and the sodium arsenite solution is run in until the color is just discharged.

If the insoluble residue is suspected of containing manganese, it is filtered off, burned, 2 or 3 drops of sulphuric acid (1 part acid to 1 part water) and about 5 cc. of hydrofluoric acid are added and the solution is evaporated until sulphuric acid fumes appear; 5 cc. of the same dilute sulphuric acid are added, and the crucible is warmed till the residue is in solution, when it is added to the main solution.

The stock solution of silver nitrate is made in the proportion of 66.66 grams of the salt to the liter. Twenty cc. of this solution are diluted to one liter for use; each 15 cc. contains 0.02 gram of silver nitrate.

The sodium arsenite solution is made by boiling 10 grams of arsenious acid until dissolved in an aqueous solution of 30 grams of sodium carbonate and diluting to 1 liter. About 125 cc. of the stock solution are diluted to 2000 cc., and this solution is standardized on steels or ores of known manganese content so that 1 cc. equals 0.1 per cent. manganese on a 0.2-gram sample.

MANGANESE.

Peroxide Reduction Method.

One-half to three grams of the sample in a beaker are digested in 20 to 50 cc. of strong hydrochloric acid. If the insoluble residue is suspected of containing manganese, a few drops of hydrofluoric acid are added, and the solution is evaporated almost to dryness. Manganese-free glass must be used if hydrofluoric acid is employed. Seventy-five cc. of strong nitric acid are added, the solution is boiled until clear, the manganese is precipitated as dioxide by the addition of a slight excess of potassium chlorate, and the boiling is continued for about five minutes. The solution is cooled, diluted to about 300 cc. with cold water, and a definite amount of hydrogen peroxide solution is added from a pipette. After the reduction of the dioxide, the excess of hydrogen peroxide is determined by titration with standard potassium permanganate solution. The value of the peroxide solution in terms of permanganate is determined, and from the total volume of peroxide used, less the

excess determined by the permanganate titration, the percentage of manganese is calculated.

The relative value of these two solutions is determined as follows: About 60 cc. of strong nitric acid are boiled for about five minutes in a beaker, then cooled and diluted to about 300 cc. To the cold solution, a definite amount of the peroxide solution is added and titrated with the standard permanganate.

The hydrogen peroxide solution is made by diluting one pound of hydrogen peroxide to 9000 cc., containing 200 cc. of strong sulphuric acid.

The permanganate solution contains 1.7375 grams per liter and is standardized against an ore of known manganese content.

LIME.

The filtrate from the persulphate or bromine precipitation of the manganese, after the basic acetate or ammonia separation of the iron and alumina, is heated to boiling in a No. 6 beaker, 10 cc. of strong ammonia and 5 cc. of a saturated solution of ammonium oxalate are added, and the heating is continued until the volume of the solution is reduced to about 150 cc. When the lime is low it is essential that the evaporation be continued to this point to insure complete precipitation. Five cc. of strong ammonia are now added and the solution is boiled for about ten minutes. The precipitate is then filtered off, washed with hot water, burned and weighed as calcium oxide. In normal ores when the lime and magnesia are low, only the first few washings are allowed to flow into the filtrate, the object being to keep the filtrate as concentrated as possible for the magnesia determination which follows. The calcium oxide is also liable to be contaminated with a small amount of silica and when extreme accuracy is desired, the ignited and weighed precipitate is dissolved in warm, dilute hydrochloric acid and the solution is evaporated to dryness. The residue is dissolved in dilute hydrochloric acid, the silica is determined and deducted from the previous weight.

If the lime is to be titrated, it is dissolved on the filter in 25 cc. of hot, dilute sulphuric acid (1 part acid to 4 parts water), the solution is diluted with 150 cc. of hot water and titrated with permanganate of such strength that 1 cc. equals about 0.100 per cent. lime.

MAGNESIA.

The filtrate from the calcium oxalate, which should not exceed 200 cc. in volume, is made slightly acid with strong hydrochloric acid, and 5 cc. of a 10 per cent. solution of ammonium phosphate are added, then to the cool solution, 25 cc. of strong ammonia, drop by drop, with continuous stirring during the addition. The solution is stirred at intervals for two or three hours, or continuously on a machine for one hour, and if permissible it is allowed to stand in a cool place over night. The precipitate is filtered off, washed thoroughly with dilute ammonia (1 part ammonia to 4 parts water), ignited at a low temperature until the filter is destroyed, and finally at the highest temperature of the muffle furnace or blast lamp, and weighed as magnesium pyrophosphate containing 36.24 per cent. magnesia.

SULPHUR.

Fusion Method.

One gram of the sample is intimately mixed in a capacious platinum crucible with about ten grams of sulphur-free

sodium carbonate, and not more than a half gram of potassium nitrate. The crucible is heated cautiously over a blast lamp until the fusion is quiet, the last few minutes of the fusion being at the highest temperature of the blast. If sulphur bearing gas is used, the contents of the crucible must be protected from the flame gases with a suitable shield. The fusion is completely disintegrated in hot water, to which a few drops of alcohol may be added to reduce and precipitate any manganese present. The solution is filtered and the residue washed with hot water, or preferably with a 1 per cent. solution of sodium carbonate.

To the filtrate, the volume of which is about 150 cc., there is cautiously added a slight excess of hydrochloric acid. The solution is heated to the boiling point, 5 cc. of a 10 per cent. solution of barium chloride are added, and the beaker is placed on the steam bath or hot plate until the precipitate has subsided. The precipitate is then filtered off, washed with hot water till free from chlorides, burned in an open crucible and weighed; 13.73 per cent. of the weight is sulphur.

If the precipitate is suspected of containing silica, which is rarely the case, it can be purified after ignition by adding a drop of sulphuric acid and 2 or 3 cc. of hydrofluoric acid, evaporating and igniting as usual.

If sulphur-free sodium peroxide is used for the decomposition of the ore in place of the sodium carbonate and niter, the fusion is made in a nickel crucible with about 5 grams of the peroxide; the remainder of the treatment is the same.

SULPHUR.

Wet Method.

Five to ten grams of the sample in a porcelain dish or beaker, with a watch glass cover, are dissolved in 50 to 100 cc. of aqua regia (1 part nitric acid to 9 parts hydrochloric acid) and the solution is evaporated to dryness. The residue is dissolved in 20 to 40 cc. of strong hydrochloric acid and the solution evaporated until the first appearance of insoluble ferric chloride. This is dissolved by the addition of a slight excess of strong hydrochloric acid, the solution is diluted to about 75 cc., and filtered into a suitable beaker, the residue being washed with hot water and the least possible amount of dilute hydrochloric acid.

If the insoluble residue is suspected of containing sulphur compounds insoluble in aqua regia, it is fused with the least possible amount of sodium carbonate and niter, as previously described in the Fusion Method for Sulphur. The fusion is disintegrated in hot water, the solution is filtered, acidified with hydrochloric acid and added to the main filtrate, or the sulphur may be estimated separately.

The filtrate is heated nearly to boiling, and after the addition of 5 cc. of a 10 per cent. solution of barium chloride it is again evaporated until a film of ferric chloride appears. This is dissolved by the addition of a few drops of strong hydrochloric acid and, with a wash bottle, a stream of cold water is forced into the solution until its volume is from 175 to 250 cc. It is then set aside in a cool place over night. The precipitate is filtered off, washed with hot water and dilute hydrochloric acid until free from iron, burned and weighed as usual.

IGNITION LOSS.

One gram of the sample is placed in a platinum crucible with a tightly fitting cover and ignited at a bright red heat

for fifteen minutes. It is then allowed to cool in a desiccator and weighed. This is repeated with five minutes' ignition until constant weight is obtained.

MOISTURE.

A portion of the entire sample reserved for the moisture determination, in either case not less than two kilograms, is transferred to a suitable pan and weighed, dried at 100° C. until constant weight is obtained and the percentage of moisture calculated.

REPORT OF THE COMMITTEE OF THE AMERICAN CHEMICAL SOCIETY APPOINTED TO CO-OPERATE WITH THE NATIONAL CONSERVATION COMMISSION.

In May, 1908, a meeting of the governors of the different states was held at the White House in Washington to consider the conservation of our rapidly wasting natural resources. Following this meeting, a commission was appointed by the President of the United States to investigate the subject, and the principal scientific societies of the United States were invited to coöperate with it. The committee of the American Chemical Society, appointed in response to this invitation, now has the honor to submit the following preliminary report.

On December 8, 9, and 10 the National Conservation Commission met in Washington in joint conference with the delegates of other organizations and the governors of more than twenty states. The commission, in its elaborate investigations, had, so to speak, taken stock of our natural resources, and its report, therefore, was essentially statistical in character. It had estimated the magnitude of each particular resource, and had studied the rate of consumption of such substances as lumber, coal, iron, etc. It discussed the wastage of the land by preventable erosion, and its effects not only upon agriculture but also in reducing the navigability of streams. Questions like these were treated at considerable length, and their general character is all that need be mentioned just here. The data of the commission were mainly classified under four headings, namely, minerals, forests, lands, and waters, and under each one the evils to be remedied were pointed out with all the emphasis and clearness which the statistical method of investigation made possible. The commission cleared the ground for study into the prevention or limitation of future waste; and the problem of conservation can now be taken up in a more intelligent manner than has been possible hitherto. We now know better than ever before what the evils and dangers really are; the next step is to discover remedies, and then, finally, to apply them. The public attention has been aroused; the people of the country are awakening to the necessity of greater prudence and economy in the use of our resources, and definite lines of action can now be laid down with a reasonable probability that they will be followed. Fortunately, the reports of the commission are neither sensational nor unduly pessimistic; the results of their conferences are presented seriously, and in such a manner as to compel consideration; they are therefore all the more likely to produce permanent effects of great benefit to the American people. The utterances of the mere alarmists rarely carry conviction, but disclosures like these made by the Conservation Commission can not be disregarded.