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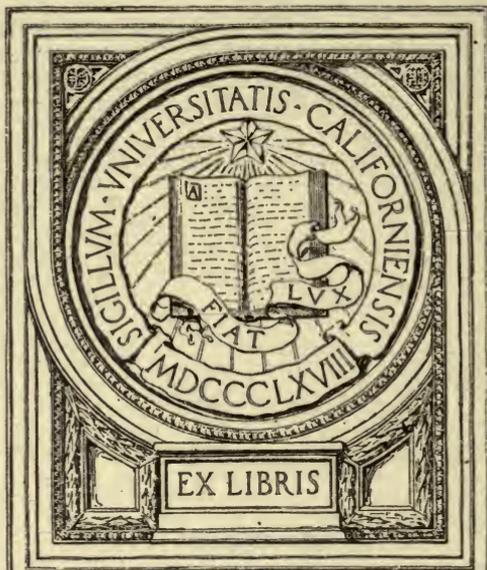


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LABORATORY MANUAL
FOR
SOIL FERTILITY

By
CYRIL G. HOPKINS
AND
J. H. PETTIT

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LABORATORY MANUAL FOR SOIL FERTILITY

By
CYRIL G. HOPKINS
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J. H. PETTIT

UNIVERSITY OF
CALIFORNIA

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NOTE.—The student practices described in this laboratory manual are the result of five years' experience by the authors in conducting classes in a course of study in soil fertility. With some modifications the usual chemical methods are employed, specific chemical directions being commonly based upon those adopted by the Association of Official Agricultural Chemists. Otherwise these practices were originated in this University.

The increasing number of students in this institution, and the fact that some other institutions also desire to use our manual, induced us to put it in printed form.

Suggestions from other teachers of soil fertility regarding possible improvements in the manual will be gladly received.

THE AUTHORS.

College of Agriculture, University of Illinois,
Urbana, Ill., July, 1905.

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LIST OF STUDENT APPARATUS.

- | | |
|------------------------------------|---|
| 2 Bunsen burners with rubber hose. | 2 Evaporating dishes, 10 cm. |
| 2 Ring stands. | 1 Graduated cylinder, 100 cc. |
| 6 Rings (3 sizes). | 1 Graduated cylinder, 25 cc. |
| 1 Burette clamp. | 1 Burette, 50 cc. |
| 2 Triangles, pipe-stem. | 1 Pipette, 25 cc. |
| 2 Erlenmeyer flasks, 200 cc. | 1 Graduated pipette, 10 cc. |
| 2 Erlenmeyer flasks, 300 cc. | 1 Desiccator. |
| 2 Beakers, 250 cc. | 2 Test tubes. |
| 2 Beakers, 400 cc. | 1 Double condenser with connect-
ing tubing. |
| 2 Beakers, 600 cc. | 2 Watch glasses. |
| 2 Kjeldahl flasks, 500 cc. | 2 Pinch cocks. |
| 2 Copper flasks, 500 cc. | 1 Percolator, 500 cc. |
| 1 Bottle, 2500 cc. | 1 Measuring flask, 250 cc. |
| 2 Bottles, 1000 cc. | 1 Bone spoon. |
| 2 Bottles, 500 cc. | 2 Safety distillation bulbs with rub-
ber stoppers. |
| 2 Bottles, 250 cc. | 1 Pair forceps. |
| 2 Crucibles, 25 cc. | 2 Bottles, 400 cc., with corks. |
| 2 Crucibles, 14 cc. | 1 Thermometer, 100° C. |
| 1 Wash bottle, 1000 cc. | 10 Glass battery jars, 5 liters, with
1 cm. drainage hole in the side,
1 cm. from the bottom. |
| 1 Funnel, 15 cm. | 2 pieces of iron gauze with asbestos
center. |
| 2 Funnels, 10 cm. | |
| 4 Funnels, 6 cm. | |
| 1 Pair crucible tongs. | |
| 2 Evaporating dishes, 8 cm. | |

PRACTICE I.

PREPARATION OF A STANDARD HYDROCHLORIC ACID SOLUTION.*

By the use of a hydrometer and specific gravity tables prepare five liters or more of approximately one-half normal hydrochloric acid, using chemically pure concentrated acid and ammonia-free water.

Standardize by the silver nitrate method:—Place exactly 25 cc. (note temperature of stock solution when measured out) of the acid solution, measured with a pipette, in a 300 cc. Erlenmeyer flask, dilute to 75 cc., add at once from a burette sufficient 5% silver nitrate solution to nearly, but not quite, precipitate all the chlorin. Close the flask with a clean rubber stopper and shake till the precipitate will settle nearly completely in a short time. Then add the silver nitrate in 1 cc. portions, shaking after each addition, until the precipitation is complete, avoiding more than 1 cc. excess of silver nitrate solution.

Shake until the silver chlorid settles well, wash three times by decantation (after shaking each time) using about 100 cc. of water containing 1 cc. concentrated nitric acid per liter and decanting the liquid through a 9 cm. filter. Transfer the precipitate to the filter, dry, transfer the bulk of the precipitate to a watch-glass or crucible, and burn the paper in a weighed crucible. Add 2 to 5 drops of concentrated nitric acid to dissolve reduced silver and then 2 to 5 drops of concentrated hydrochloric acid. Evaporate to dryness without spattering, add the main precipitate, dry to constant weight at 120° to 130°, cool in a desiccator, and weigh.

Record the weights of silver chlorid from duplicate 25 cc. portions of the standard hydrochloric acid.

*To be done by the instructor.

PRACTICE 2.

PREPARATION OF A STANDARD AMMONIA SOLUTION.

Determine, by hydrometer, the specific gravity of concentrated ammonia and calculate, by the use of a specific gravity table, the number of cubic centimeters necessary to make two liters of approximately one-fifth normal ammonia solution.

Sp. Gr.
 Grams NH_3 per cc
 Grams NH_3 per liter in normal solution
 Grams NH_3 in two liters of 1-5 normal solution
 Cc. of conc. NH_3 equivalent to g. NH_3

Measure out the required amount of concentrated ammonia, add distilled water to make the total volume up to two liters, and mix thoroughly. Standardize by titrating 10 cc. of the standard hydrochloric acid with the ammonia solution, using lacmoid as an indicator. Make three titrations.

(1) 10 cc. HCl is equivalent to cc. NH_3
 (2) 10 cc. HCl is equivalent to cc. NH_3
 (3) 10 cc. HCl is equivalent to cc. NH_3
 Average c.c. NH_3
 1 cc. NH_3 is equivalent to Mg N.

Give reactions in first and second practices, and explain the computations involved in ascertaining the weight of nitrogen in 1 cc. of the standard ammonia solution.

PRACTICE 3.

BLANK DETERMINATION OF NITROGEN IN REAGENTS USED IN DISTILLING.

Place 250 cc. of ammonia-free water in a copper flask, add 10 cc. of concentrated alkali solution (made by dissolving 1000 gm. sodium hydroxid and 25 gm. potassium sulfid in 1000 cc. water), pouring carefully down the side of the flask, connect with the condenser, shake the flask thoroughly, heat up slowly and distill into a 300 cc. Erlenmeyer flask containing 10 cc. of the standard hydrochloric acid and about 15 cc. ammonia-free water. The end of the delivery tube should dip into the acid solution. Distill to a volume of 200 cc. Add lacmoid and titrate with standard NH_3 .

Titration (1)cc. NH_3

Titration (2)cc. NH_3

Averagecc. NH_3

State correction in cc. of standard NH_3 .

Explain all reactions involved, including the use of the indicator.

PRACTICE 4.

PREPARATION OF AN AMMONIUM SULFATE SOLUTION.

Weigh out exactly in a weighed crucible the number of grams of chemically pure ammonium sulfate (assuming the salt to be dry) equivalent to 500 cc. of the standard ammonia solution. Dry in the air bath at 115° to 120° for thirty minutes, cool in a desiccator, and weigh. Dissolve in ammonia-free water in a 250 cc. measuring flask. Dilute to exactly 250 cc. Mix well, and transfer to a dry 250 cc. bottle. Label and keep stoppered when not in use.

500 cc. standard NH_3 contains.....	gm. N
Percent N in $(\text{NH}_4)_2\text{SO}_4$ by theory is.....	
500 cc. NH_3 is equivalent to	gm. $(\text{NH}_4)_2\text{SO}_4$
	Before After
	Heating Heating
Weight of crucible+ $(\text{NH}_4)_2\text{SO}_4$ =.....
Weight of crucible	=.....
Weight of $(\text{NH}_4)_2\text{SO}_4$	=.....
Percent dry matter in salt is.....	

How much of the ammonium sulfate will it be necessary to weigh out in order to have exactly 5 gm. of the dry salt?

PRACTICE 5.

DETERMINATION OF NITROGEN IN AMMONIUM SULFATE.

Place 10 cc. of the ammonium sulfate solution in a 500 cc. copper flask, add 240 cc. ammonia-free water and then add carefully, by pouring down the side of the flask, 10 cc. of the concentrated alkali. Connect immediately with the condenser, shake the flask thoroughly, heat slowly, and distill into a 300 cc. Erlenmeyer flask containing 10 cc. of the standard hydrochloric acid and about 15 cc. of ammonia-free water, to a volume of 200 cc. Add lacmoid and titrate the excess acid with standard ammonia.

Titration (1)	cc. NH_3
(2)	cc. NH_3
Average	cc. NH_3
Mgs. N in sample	
Percent N in dry salt ..	
The percentage purity of the dry salt is.....	

Explain all reactions.

Does the percentage of nitrogen vary directly or inversely with the titration readings?

PRACTICE 6.

FIXATION OF BASES IN SOILS.

(a) Place a small bunch of glass wool in a percolator, cover with 1 cm. of clean sand, and add 100 gm. of clayey soil. Upon this carefully pour 250 cc. of dilute ammonium sulfate solution (50 cc. of the solution prepared in Practice 4, plus 200 cc. of ammonia-free water). When percolation ceases, mix the percolate thoroughly and determine nitrogen in two 50 cc. portions.

Titrations (1)cc. NH_3
 (2) cc. NH_3
 Average.....cc. NH_3
 Mg. N per cc. in solution used
 Mg. N per cc. in percolate
 Percent N fixed by soil

(b) Repeat the experiment, using 200 gm. of the same soil.

Titrations (1)cc. NH_3
 (2)cc. NH_3
 Average.....cc. NH_3
 Mg. N per cc. in solution used
 Mg. N per cc. in percolate
 Percent N fixed by soil.....

(c) Repeat the experiment, using 200 gm. of sandy soil.

Titrations (1)cc. NH_3
 (2)cc. NH_3
 Average.....cc. NH_3
 Mg. N per cc. in solution used
 Mg. N per cc. in precolate.....
 Percent N fixed by soil.....

Give a general reaction for the fixation of bases by soils and explain fully what chemical elements that are important in soil fertility may be retained in soils by means of this reaction.

PRACTICE 7.

NITRIFICATION.

Dilute 20 cc. of the standard ammonium sulfate solution to 500 cc., add 3 gm. of dipotassium phosphate, 5 gm. of calcium carbonate, and about 2 gm. of fresh rich garden soil. Mix well, let settle, and draw off two 100 cc. portions.

Place 500 gm. of clean washed and dried white sand in a percolator. Upon this pour 100 cc. of the above solution and immediately wash the sand with about 500 cc. of ammonia-free water, collect the washings and make up to exactly 500 cc. Place 250 cc. portions in copper flasks, add 10 cc. of alkali and determine the nitrogen in the usual way. Compare the amount of nitrogen distilled as ammonia with that originally applied in the ammonium sulfate.

Titration (1)	cc. NH_3
(2)	cc. NH_3
Average	cc. NH_3
Mg. N found	

Add the other 100 cc. portion of ammonium sulfate solution to 500 grams of clean, washed and dried sand in a percolator and allow to stand in a dark place at warm room temperature for four weeks. Then wash out and determine the ammonia nitrogen as directed above.

Titration (1)	cc. NH_3
(2)	cc. NH_3
Average	cc. NH_3
Mg. N found	
Percent N nitrified	

What change has been brought about and how?

Explain fully.

PRACTICE 8.

DETERMINATION OF NITROGEN IN REAGENTS.

Measure out exactly 10 cc. of the concentrated alkali in a beaker and dilute to 200 cc. with distilled water. Stir and add slowly (finally drop by drop) concentrated sulfuric acid until the alkali is neutralized, as shown by the change of color.

10 cc. alkali are equivalent to cc. H_2SO_4

How many cc. of alkali are necessary to neutralize 20 cc. H_2SO_4 ?

Place approximately 2 grams of pure sugar in a Kjeldahl flask, add by measure approximately .650 gm. metallic mercury and 20 cc. sulfuric acid. Digest in a ventilated hood over a low flame till colorless, add carefully, while still boiling hot, powdered potassium permanganate until the solution is green. Allow to cool. Transfer with 200 cc. of ammonia-free water to a copper flask by means of a large funnel supported on an iron ring. Add carefully sufficient concentrated alkali to neutralize 20 cc. of concentrated sulfuric acid, connect with the condenser, shake until thoroughly mixed, and distill as usual.

Titration (1) cc. NH_3

(2) cc. NH_3

Average cc. NH_3

Check up the standard ammonia solution by titrating against the standard hydrochloric acid solution.

Correction for nitrogen in reagents in terms of standard ammonia solution cc.

Explain the use of the sugar. Give the reaction between the sugar and the sulfuric acid. Why is the mercury used? the potassium permanganate? the potassium sulfid?

1877

Received of _____

the sum of _____ Dollars

for _____

PRACTICE 10.

DETERMINATION OF NITROGEN IN ANIMAL EXCREMENTS: SOLID AND LIQUID.

Each group of students will work upon one of the following:

1. Horse excrements.
2. Steer excrements.
3. Cow excrements.
4. Sheep excrements.
5. Swine excrements.
6. Poultry excrement and fresh cow's milk.
7. Human excrements.
8. Wheat straw and manger refuse (for bedding).

Record age and condition of animals and food rations as nearly as possible in all cases.

For solid excrements: Weigh out 10 grams of fresh substance on filter paper, placed on a watch-glass, and transfer both paper and excrement to a Kjeldahl flask.

For liquid excrement: Measure out 10 cc. and place in a Kjeldahl flask. Compute weight from specific gravity.....gm.

Solid	Liquid.
Titration (1).....cc. NH_3	Titration (1).....cc. NH_3
(2).....cc. NH_3	(2).....cc. NH_3
Average.....cc. NH_3	Average.....cc. NH_3
%N.....	%N.....

Calculate the results obtained and with these record the results obtained by three members of each group as indicated upon the following page. Value N at 15 cents per pound.

10 tons alfalfa hay contain.....lb. N

1 ton fresh cow dung contains.....lb. N

How many tons of fresh cow dung would be required to furnish nitrogen for 10 tons of alfalfa hay?

PRACTICE II.

DETERMINATION OF NITROGEN IN FERTILIZERS.

Weigh out $\frac{1}{2}$ gram of each of the following materials and use 20 cc. of standard hydrochloric acid in the receiver.

(a) Ammonium sulfate.

(b) Dried blood.

(c) Sodium nitrate. (Use the Kjeldahl method modified for nitrates. Mix 2 gm. salicylic acid with the sodium nitrate in flask and add 30 cc. sulfuric acid. Use $1\frac{1}{2}$ times the usual amount of alkali in the distillation to neutralize the larger amount of sulfuric acid.)

Titration cc. NH_3	Average corrected	cc. NH_3 from sample	Percent N	Value per ton (N 15 c. per lb.)
(a) (1)				
(2)				
(b) (1)				
(2)				
(c) (1)				
(2)				

How would the reaction of the soil be affected by the residues left by each of these materials when used to supply nitrogen for plant growth?

PRACTICE 12.

DETERMINATION OF NITROGEN IN SOILS.

Each group of students will work upon one of the following:

1. Surface of gray silt loam.
2. Subsoil of gray silt loam.
3. Surface of brown silt loam.
4. Subsoil of brown silt loam.
5. Surface of black clay loam.
6. Subsoil of black clay loam.
7. Sandy soil.
8. Peaty soil (use 5 gm. soil and 20 cc. standard HC in receiver.)

For all soils except peat weigh out 10 gm. air-dry soil and use 10 cc. of standard hydrochloric acid in the receiver.

Titrations (1).....	cc. NH_3
(2).....	cc. NH_3
Average.....	cc. NH_3
Percent N

Calculate the results obtained and with these record the results obtained by three members of each group as indicated upon the following page.

Assuming there are 2,000,000 lb. in an acre to the depth of seven inches, how many pounds of nitrogen are there in this plowed soil? How many 100-bushel crops of corn will this produce if the total crop is removed?

PRACTICE 12.—(Cont.)

NAME OF STUDENT	Kind of Soil	Percent N	Pounds of N Per Stratum	No. of 100-bu. Crops of Corn Equivalent
AVERAGE				
AVERAGE				
AVERAGE				
AVERAGE				
AVERAGE				
AVERAGE				
AVERAGE				

PRACTICE 14.

PREPARATION OF STANDARD SODIUM HYDROXID AND NORMAL POTASSIUM NITRATE SOLUTIONS.*

These solutions are to be used in the determination of soil acidity.

(a) Weigh out enough sodium hydroxid (sticks, purified over alcohol, about 75 % NaOH) to make 3 liters of solution of such strength that 1 cc. shall be equivalent to 4 mg. of calcium carbonate. Dissolve in ammonia-free water, dilute to exactly 1000 cc., add 100 cc. of a saturated barium hydroxid solution, shake well, and allow to stand over night, or until clear. Draw off 100 cc. of the clear solution, place in a 250 cc. bottle containing 100 cc. of the saturated solution of barium hydroxid. If a precipitate occurs, add the 200 cc. to the main solution, shake and repeat the above operations until no further precipitate occurs. (If no precipitate is formed throw away the 200 cc.) Always keep an exact record of the amount of liquid remaining in the stock bottle. When the solution is free from carbon dioxid, draw off 100 cc. and place in a 100 cc. bottle. Fill the burette with this and titrate against 10 cc. portions of standard hydrochloric acid. Add ammonia-free water so that 1 cc. of the standard sodium hydroxid solution shall be exactly equivalent to 4 mg. of calcium carbonate.

(b) Prepare 5 liters of a normal potassium nitrate solution, assuming the salt to be pure.

If 100 grams of an acid soil are placed in 250 cc. of normal potassium nitrate solution and shaken for three hours a reaction takes place between the potassium nitrate and the acid constituents of the soil, giving, as one of the products, soluble acid salts and so making the acidity determinable. An equilibrium is reached, however, before this reaction runs to an end and if, after having drawn off 125 cc. to titrate, 125 cc. of fresh potassium nitrate are added to the bottle and the bottle again shaken for three hours, 125 cc. drawn off will give a titration, which is more than one-half of the first. By continuing this process until the last 125 cc. shows practically no acidity, we have a series of titrations the sum of which represents the total acidity of the 100 gm. of soil. It has been found by working with a number of different soils that as an average the sum of such a series is $2\frac{1}{2}$ times the first titration.

Consequently when the sodium hydroxid is made up so that 1 cc. is equivalent to 4 mg. of calcium carbonate and 125 cc. (which represents 50 gm. of soil) are titrated, each 0.1 cc. required to neutralize corresponds to 1 mg. of calcium carbonate required by the 100 gm. of soil, or to 0.001 % of calcium carbonate required by the soil tested.

*To be done by the instructor.

PRACTICE 15.

DETERMINATION OF ACIDITY (OR LIME REQUIREMENT) OF SOILS.

Each group of students will work upon one of a group of soils selected by the instructor using surface, sub-surface, and subsoil samples.

Place 100 gm. of soil in a 400 cc. (or 12 oz.) wide-mouthed bottle, add 250 cc. normal potassium nitrate solution, stopper, and shake continuously for three hours in a shaking-machine, or every five minutes by hand. Let stand over night. Draw off 125 cc. of the clear supernatant liquid, boil 10 minutes to expel carbon dioxid, cool, and titrate with the standard sodium hydroxid, using phenolphthalein as indicator.

Surface. Subsurface. Subsoil.

Titration (1)	cc. NaOH
(2)	cc. NaOH
Average	cc. NaOH

Are carbonates present in the soil? Explain the test for carbonates.

Calculate the results obtained and with these record the results obtained by three members of each group as indicated on the following page. Consider 7 acre-inches to weigh 2,000,000 lb.

What kind of crops, and in what way, does ground limestone mainly benefit?

PRACTICE 16.

PREPARATION OF PLANT FOOD SOLUTIONS.*

Solution No. 1.—Nitrogen: Dissolve 80 gm. of ammonium nitrate in 2500 cc. of distilled water. Use 10 cc. per pot.

Solution No. 2.—Phosphorus: Dissolve 25 gm. of monocalcium phosphate in 2500 cc. of ammonia-free water. Use 10 cc. per pot.

Solution No. 3.—Potassium: Dissolve 50 gm. of potassium sulfate in 2500 cc. of ammonia-free water. Use 10 cc. per pot.

Solution No. 4.—Magnesium: Dissolve 20 gm. of magnesium sulfate in 2500 cc. of ammonia-free water. Use 10 cc. per pot.

Solution No. 5.—Iron: Dissolve 0.1 gm. ferric chlorid in 250 cc. of ammonia-free water. Use 1 cc. per pot.

Prepare these solutions carefully, using chemically pure salts, and label each bottle.

*To be done by the instructor.

PRACTICE 17.

PREPARATION OF POT CULTURES.

Use clean, white sifted sand in 5-liter heavy glass battery jars, having a 1 cm. hole within 1 cm. of the bottom. Into the hole fit a drain tube made of glass-tubing with a glass-wool filter at the inner end, so that it will take liquid from the lowest place in the jar. Put up a series of ten of these pots, eight to be used as indicated in the table below and two in an experiment to be devised by the student. The previous treatment of the sand in the latter two will depend upon the experiment to be made.

To extract the sand, fill the jar within 1 cm. of the top with dry sifted sand and add to this dilute sulfuric acid (made by adding 100 cc. of concentrated chemically pure sulfuric acid to 900 cc. of ammonia-free water) until the sand is saturated. Let stand two hours and then add ammonia-free water, allowing the drainage to flow into a second jar until it is saturated. Allow this jar to stand two hours and then wash both with ammonia-free water until free of acid. In this way any soluble plant food is removed from the sand and one portion of acid extracts two jars. The sand for two of the jars, in which experiments are to be made to show the effect of nitrogen-gathering bacteria, is first heated to 120° to 130° for one-half hour and then extracted and washed as above.

Before planting, mix with the sand of each pot 10 gm. of pure calcium carbonate.

In making applications of plant food as indicated in the following table and in such amounts as are shown in Practice 16 the solutions to be applied to each pot are to be mixed together, and diluted to 1000 cc. Mix thoroughly and apply the whole amount to the pot, allowing any water present to be forced out through the drain.

The first application of the plant food solutions is to be made at the time of planting, the second three weeks later, the third two weeks later, and subsequent applications at intervals of one week, each time making the application as directed above.

Each student in each group will prepare and care for a series of pots as indicated in the following table.

PRACTICE 17.—(Cont.)

Pot No.	Preparation of sand	Plant food added	SEEDS PLANTED							
			Group 1	2	3	4	5	6	7	8
1.	Extract and wash	None	Corn	Oats	Wheat	Cow Peas	Red Clover	Soy Beans	Rape	Beets
2.	"	All but N	"	"	"	"	"	"	"	"
3.	"	All but P	"	"	"	"	"	"	"	"
4.	"	All but K	"	"	"	"	"	"	"	"
5.	None	All	"	"	"	"	"	"	"	"
6.	None	All	Red Clover	Cow Peas	Soy Beans	Vetch	Alfalfa	Sweet Clover	Crimson Clover	Alsike Clover
7.	Heat, extract and wash	All but N	"	"	"	"	"	"	"	"
8.	"	All but N Bacteria *	"	"	"	"	"	"	"	"

*Obtain about ½ kilo of soil which has recently grown the infected legume and shake it up with about one liter of water. Let settle and to each seed as it is planted add 10 cc. of the supernatant liquid, before the seed is covered.

Why is the CaCO₃ added? Why were two of the pots heated? Make observations and at least weekly notes of any differences in growth and explain.

PRACTICE 18.

PREPARATION OF AMMONIUM MOLYBDATE SOLUTION.*

Dissolve 100 gm. of molybic acid in 400 cc. of ammonium hydroxid of .96 specific gravity and pour this solution slowly and with constant stirring into 1250 cc. of nitric acid of 1.20 specific gravity. It is best to cool the acid after the addition of each small amount of ammonium hydroxid. Keep the mixture in a warm place several days, or until a portion heated to 40° deposits no yellow precipitate of ammonium phosphomolybdate.

PRACTICE 19.

PREPARATION OF A STANDARD POTASSIUM HYDROXID SOLUTION.*

Dissolve the number of grams of chemically pure potassium hydroxid (usually about 85%) sufficient to make 4 liters of a solution, one cc. of which will be equivalent to .5 mg. of phosphorus, in 400 cc. distilled water. Remove carbonates with barium hydroxid, as in Practice 14. Then make up to 500 cc. and titrate 10 cc. portions with standard hydrochloric acid, using phenolphthalein as indicator. Compute the exact weight of potassium hydroxid in the remaining solution and dilute with a sufficient quantity of water to reduce the strength to exactly 2.0809 gm. potassium hydroxid per 100 cc., so that 1 cc. is equivalent to .5 mg. phosphorus.

Mix well, check up by again titrating, and label: Standard Potassium Hydroxid (1cc.—5 mg. P.)

PRACTICE 20.

PREPARATION OF A STANDARD NITRIC ACID SOLUTION.*

Determine the specific gravity of concentrated nitric acid. Measure out sufficient to make 4 liters of solution of a strength equivalent per cc. to the standard potassium hydroxid solution. Dilute with ammonia-free water to 3½ liters and titrate 25 cc. portions of the standard potassium hydroxid with the dilute nitric acid, using phenolphthalein as indicator. Then add sufficient ammonia-free water to make the nitric acid of the same titrating strength as the standard alkali. Mix thoroughly and check up by another titration.

*To be done by the instructor.

PRACTICE 21.

DETERMINATION OF TOTAL PHOSPHORUS IN FERTILIZERS.

Each group of students will work upon one of the following materials:

- | | |
|--------------------------|-------------------------------|
| 1. Bone Ash. | 5. Raw Rock Phosphate. |
| 2. Raw Bone Meal. | 6. Acidulated Rock Phosphate. |
| 3. Steamed Bone Meal. | 7. Double Superphosphate. |
| 4. Acidulated Bone Meal. | 8. Basic Slag Phosphate. |

For materials containing more than 9 percent of phosphorus use 1 gm., for lower percentages use 2 gm. Ignite in a crucible to destroy organic matter. Transfer to a beaker and dissolve in 15 cc. of hydrochloric acid (concentrated hydrochloric acid plus an equal volume of ammonia-free water) by the aid of gentle heat. Transfer to a 250 cc. measuring flask, cool, and dilute to exactly 250 cc. Mix well, transfer to a dry bottle, and let settle.

Place 25 cc. in a 250 cc. beaker, add 5 cc. of nitric acid (concentrated nitric acid plus an equal volume of ammonia-free water), just neutralize with ammonia, and clear up with a few drops of nitric acid, using heat if necessary but avoiding more than a few drops in excess. Heat to 50°-60° on a water-bath, add 35 cc. of the clear molybdate solution, stir, keep at 50°-60° for two hours, let stand in the desk over night. Filter, wash twice by decantation, using 25 cc. portions of distilled water, stirring thoroughly, and then allowing the precipitate to settle before decanting upon a 9 cm. filter. Transfer the precipitate to the filter, and wash the beaker and filter seven or eight times with small amounts of ammonia-free water until free of acid. Place the filter containing the precipitate in the beaker and add standard potassium hydroxid in 10 cc. portions until the precipitate is dissolved. Titrate the excess alkali with standard nitric acid, using phenolphthalein as an indicator.

cc. HNO ₃ used	cc. KOH to dissolve ppt.	mg. P	%
.....
.....
Ave.

PRACTICE 22.

PREPARATION OF A NEUTRAL AMMONIUM CITRATE SOLUTION.*

To 370 grams of commercial citric acid add commercial ammonia, specific gravity .96, until nearly neutral; reduce the specific gravity to nearly 1.09 and make exactly neutral, testing as follows: Prepare a solution of fused calcium chlorid, 200 grams to the liter, and add one-fourth volume of strong alcohol. Make the mixture exactly neutral, using a small amount of freshly prepared corallin solution as a preliminary indicator, and test finally by withdrawing a portion, diluting with an equal volume of water, and testing with cochineal solution; 50 cc. of this solution will precipitate the citric acid from 10 cc. of the citrate solution. To 10 cc. of the nearly neutral citrate solution add 50 cc. of the alcoholic calcium chlorid solution, stir well, filter at once through a folded filter, dilute with an equal volume of water, and test the reaction with a neutral solution of cochineal. If acid or alkaline, add ammonia or citric acid, as the case may be, mix, and test again, as before. Repeat this process until a neutral reaction is obtained. Add sufficient water to make the specific gravity 1.09 at 20°.

*To be done by the instructor.



PRACTICE 23.

DETERMINATION OF CITRATE-INSOLUBLE PHOSPHORUS.

Use the same amounts and materials as in Practice 21. If the material is acid, wash the weighed sample on a 9 cm. filter with water until free of acid.

Heat in a water-bath 100 cc. of the neutral ammonium citrate solution to 65° in a 200 cc. Erlenmeyer flask, loosely stoppered with a stopper holding a 100° thermometer. When 65° is reached put in the sample and shake thoroughly. Place in the bath at 65° and let stand for 30 minutes, shaking every five minutes. At the end of 30 minutes filter and wash thoroughly with water at 65° until all soluble phosphorus is removed (test for soluble phosphorus with 1 cc. of ammonium molybdate solution). Transfer the filter and its contents to a crucible, dry, and then ignite until all organic matter is destroyed, transfer to a beaker, add about 15 cc. of hydrochloric acid, and heat until all phosphorus is dissolved. Make up to 250 cc., mix well, transfer to a dry bottle, and let settle. Determine phosphorus in 25 cc. portions according to the directions given in Practice 21.

cc. HNO ₃ used	cc. KOH to dissolve ppt.	mg. P	%P
.....
.....
Ave.

Calculate the results obtained in Practices 21 and 23 and with these record the results obtained by three members of each group as indicated in the table on the following page. Value citrate-soluble phosphorus at 12 cents and insoluble phosphorus at 5 cents a pound.

PRACTICE 23.--(Con't.)

NAME OF STUDENT	Material	Citrate Soluble Percent	Insoluble Percent	Total Percent	Value per Ton
AVERAGE					
AVERAGE					
AVERAGE					
AVERAGE					
AVERAGE					
AVERAGE					
AVERAGE					
AVERAGE					

PRACTICE 24.

DETERMINATION OF PHOSPHORUS IN FARM PRODUCE.

Each group of students will work upon one of the following products:

- | | |
|-----------------|--------------------|
| 1. Wheat. | 5. Oats. |
| 2. Corn. | 6. Oat Straw. |
| 3. Corn Stover. | 7. Red Clover Hay. |
| 4. Corn Cobs. | 8. Alfalfa Hay. |

Weigh out 2 gm. of the material in a 25 cc. crucible, moisten with a 20% solution of ammonium nitrate, and ignite in a muffle furnace at a low red heat for two hours. Transfer to a 250 cc. beaker, dissolve in about 15 cc. of hydrochloric acid, dilute to about 40 cc., filter and wash. Evaporate the filtrate and washings to about 25 cc. and determine phosphorus according to directions given in Practice 21, using only 10 cc. of the ammonium molybdate solution.

cc. HNO_3 used	cc. KOH to dissolve ppt.	mg. P	%P
.....
.....
Ave.

Calculate the results obtained and record with these the results obtained by three members of each group as indicated on the following page. Value phosphorus at 12 cents per pound.

How many pounds of steamed bone meal will it require to replace the phosphorus removed from the soil in a 100-bushel crop of corn, a 75-bushel crop of oats, and a 3-ton crop of clover hay (see Practice 9)?

PRACTICE 24 (Cont.)

NAME OF STUDENT	Material	Percent P	Pounds P Per Ton	Value Per Ton
AVERAGE				
AVERAGE				
AVERAGE				
AVERAGE				
AVERAGE				
AVERAGE				
AVERAGE				
AVERAGE				

PRACTICE 25.

FIXATION OF PHOSPHORUS BY SOILS.

Each group of students will use one of the following soils:

- | | |
|--------------------------------|--------------------------------|
| 1. Surface of gray silt loam. | 5. Surface of black clay loam. |
| 2. Subsoil of gray silt loam. | 6. Subsoil of black clay loam. |
| 3. Surface of brown silt loam. | 7. Sandy soil. |
| 4. Subsoil of brown silt loam. | 8. Peaty soil. |

Dissolve 1 gm. of double superphosphate in 500 cc. of water. Filter and keep in a stoppered bottle.

(a) Determine the phosphorus in 50 cc. portions of this solution by concentrating to 25 cc. and following the directions given in Practice 21.

cc. HNO ₃ used	cc. KOH to dissolve ppt.	mg. P	%P
.....
.....
Ave.

(b) Dilute 50 cc. of the double superphosphate solution to 250 cc. and percolate through 100 gm. of soil held in a percolator as in Practice 6. Determine the phosphorus in 100 cc. portions by concentrating to 25 cc. and following the directions given in Practice 21, using only 15 cc. of the ammonium molybdate solution.

cc. HNO ₃ used	cc. KOH to dissolve ppt.	mg. P	%P
.....
.....
Ave.

Percent phosphorus fixed by soil.....

(c) Thoroughly mix 10 gm. of CaCO₃ with 100 gm. of the same soil, dilute 50 cc. of the double superphosphate solution to 250 cc., percolate through soil and determine the phosphorus in 100 cc. portions of the percolate in the same manner as above.

cc. HNO ₃ used	cc. KOH to dissolve ppt.	mg. P	%P
.....
.....
Ave.

Percent phosphorus fixed by soil.....

Calculate the results obtained and with these record the results obtained by three members of each group as indicated in the table on the following page.

Give the general reaction. Explain the effect of the CaCO₃. How was the fixation brought about in the non-calcareous soils?

PRACTICE 26.

DETERMINATION OF PLANT FOOD IN SOILS.

Each student will select a soil in which he is especially interested.

(a) *Nitrogen.*

Determine according to directions given in Practice 12.

(b) *Dry Matter.*

Weigh out 5 gm. of the air-dried soil in a small porcelain dish, dry at 100° for 5 hours, cool in a desiccator, and weigh.

Weight of vessel plus dry matter
Weight of vessel
Weight of dry matter
Percent dry matter
Average

(c) *Insoluble matter.*

Place 10 gm. of the air-dried soil in a 200 cc. Erlenmeyer flask, add 100 cc. of hydrochloric acid (sp. gr. 1.115), close with a rubber stopper in which is a glass tube 18 inches long, and digest for ten hours on the water-bath, at the temperature of boiling water, shaking once every hour. Dilute, filter through a 15 cm. filter, wash free of chlorids with distilled water, and evaporate the filtrate and washings to dryness with 5 cc. of nitric acid to destroy organic matter. Take up with about 10 cc. of hydrochloric acid and about 25 cc. of water, again run to dryness and heat three hours in an air-oven at 110°. Add about 15 cc. of hydrochloric acid, heat 30 minutes on the water-bath, dilute, filter through a 9 cm. filter and wash with distilled water. Ignite the filter and contents with the main portion of the insoluble matter.

Weight of crucible plus insoluble matter
Weight of crucible
Weight of insoluble matter
Percent of insoluble matter
Average

Make the filtrate and washings up to exactly 500 cc., mix thoroughly and put in dry stoppered bottle. Label it Solution A.

(d) *Phosphorus.*

Evaporate 200 cc. of Solution A to about 20 cc. and determine phosphorus according to the directions given in Practice 21, using 15 cc. of the clear ammonium molybdate solution.

cc. HNO ₃ used	cc. KOH to dissolve ppt.	mg. P	%P
.....
.....
Ave.

(e) *Potassium, Sodium, and Iron.*

Evaporate 100 cc. of Solution A to complete dryness. Add about 25 cc. of hot water and 50 cc. of a saturated barium hydroxid solution, let stand 30 minutes on the water-bath, filter through an 11 cm. filter, and

PRACTICE 26.—(Con't.)

wash with hot water until free of chlorids. Dissolve the precipitate from the filter with as small an amount as possible of warm dilute sulfuric acid (1 to 6), catch the solution and washings in a 200 cc. Erlenmeyer flask and treat this solution as directed below for the determination of iron.

Concentrate the filtrate from the barium hydroxid precipitation to about 50 cc., add a few drops of ammonium hydroxid and 10 cc. of ammonium carbonate solution, let stand 30 minutes on the water-bath, filter through an 11 cm. filter into a 10 cm. evaporating dish, and wash with hot water until free of chlorids. Evaporate the filtrate to dryness and carefully ignite the dish over a Bunsen burner to expel ammonium salts. Take up with about 10 cc. of hot water, add a few drops of ammonium hydroxid and ammonium carbonate, filter through a 9 cm. filter into a 250 cc. beaker, and wash with hot water. Concentrate to about 25 cc., add 5 cc. of ammonium sulfate solution (75 gm. per liter), digest on a water-bath 2 hours, and filter into a 250 cc. beaker through a 9 cm. filter. Transfer to a small weighed evaporating dish or large crucible, and evaporate to dryness. Ignite over a Bunsen burner to remove ammonium salts. Add 1 gm. of powdered ammonium carbonate, heat to full redness, cool in a desiccator, and weigh.

Weight of dish plus K_2SO_4 and Na_2SO_4
Weight of dish
Weight of K_2SO_4 and Na_2SO_4
Weight of K_2SO_4 (computed)
Weight of Na_2SO_4
Weight of Na
Percent Na
Average

Take up with a little hot water, add a few drops of hydrochloric acid and platonic chlorid solution (10 cc. contain 1 gm. platinum), using .1 cc. of the platinum solution for every 5 mg. of sulfates found, evaporate to a thick syrup, wash seven times with 4 cc. of 80% alcohol, decanting each time through a 9 cm. filter, let the filter dry thoroughly at room temperature, and dissolve any of the potassium platonic chlorid on the filter with hot water, catching the solution in the dish containing the greater part of the salt, evaporate to dryness, heat 30 minutes at 110° in an air-bath, cool in the desiccator, and weigh.

Weight of dish plus K_2PtCl_6
Weight of dish
Weight of K_2PtCl_6
Weight of K
Percent of K
Average

To the 200 cc. Erlenmeyer flask, containing the iron in sulfuric acid solution, add 1 gm. of zinc dust, stopper with a cork carrying a delivery-tube bent so as to dip into a beaker of distilled water. Let stand overnight and titrate the reduced iron with potassium permanganate. (Solu-

PRACTICE 26.—(Con't.)

tion to be made by the instructor so that 1 cc. shall be equivalent to 2 mg. of iron).

cc. KMnO_4 used
Percent Fe
Average

(f) *Aluminum.*

Heat to boiling 100 cc. of Solution A in a 250 cc. beaker, add ammonium hydroxid carefully, until an odor of ammonia is distinguishable after blowing into the beaker, let settle, and decant supernatant liquid through an 11 cm. filter. Wash with about 50 cc. of hot water by decantation, dissolve in hydrochloric acid, dilute to 100 cc., heat to boiling, and again precipitate by adding ammonium hydroxid as above. Decant through the same filter, transfer to the filter, and wash with hot water until free of chlorids. Dry and ignite in a weighed crucible over a Bunsen burner.

Weight of crucible plus Al_2O_3 , Fe_2O_3 , P_2O_5
Weight of crucible
Weight of Al_2O_3 , Fe_2O_3 , P_2O_5
Weight Fe_2O_3 and P_2O_5 (computed)
Weight of Al_2O_3
Weight Al
Percent Al
Average

(g) *Calcium.*

Concentrate the filtrate from (f) to 50 cc., add a few drops of ammonium hydroxid and then ammonium oxalate until all the calcium is precipitated.

Digest on the water-bath 2 hours, filter through a 9 cm. filter, wash with hot water until free of chlorids, dry, and ignite in a weighed crucible over a Bunsen burner, finishing to constant weight over the blast lamp.

Weight of crucible plus CaO
Weight of crucible
Weight of CaO
Weight of Ca
Percent Ca
Average

(h) *Magnesium.*

Concentrate the filtrate from (g) to 50 cc., cool, add a few drops of ammonium hydroxid, then add slowly while stirring disodium phosphate solution until the magnesium is all precipitated, add 10 cc. of ammonium hydroxid, cover, and let stand over night. Filter through a 9 cm. filter, wash with dilute ammonium hydroxid (1 liter of ammonium hydroxid of .96 sp. gr. diluted to 5 liters), dry, and ignite in a weighed crucible over a Bunsen burner, finishing to constant weight over the blast lamp.

PRACTICE 26.—(Con't.)

Weight of crucible plus $Mg_2P_2O_7$
Weight of crucible
Weight of $Mg_2P_2O_7$
Weight of Mg
Percent Mg
Average

PERCENTAGE COMPOSITION OF SOIL.

	Air-dry Basis.	Dry Basis.
Nitrogen
Phosphorus
Potassium
Calcium
Magnesium
Iron
Aluminum
Sodium
Insoluble Matter
Undetermined } C,H,O, etc. }

Are carbonates present in the soil?.....

If the soil is acid, what percent of calcium carbonate is required to correct the acidity?.....

Suggest a practical method of treatment, of unlimited application, by which the productive capacity of this soil could be profitably increased and permanently maintained.

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