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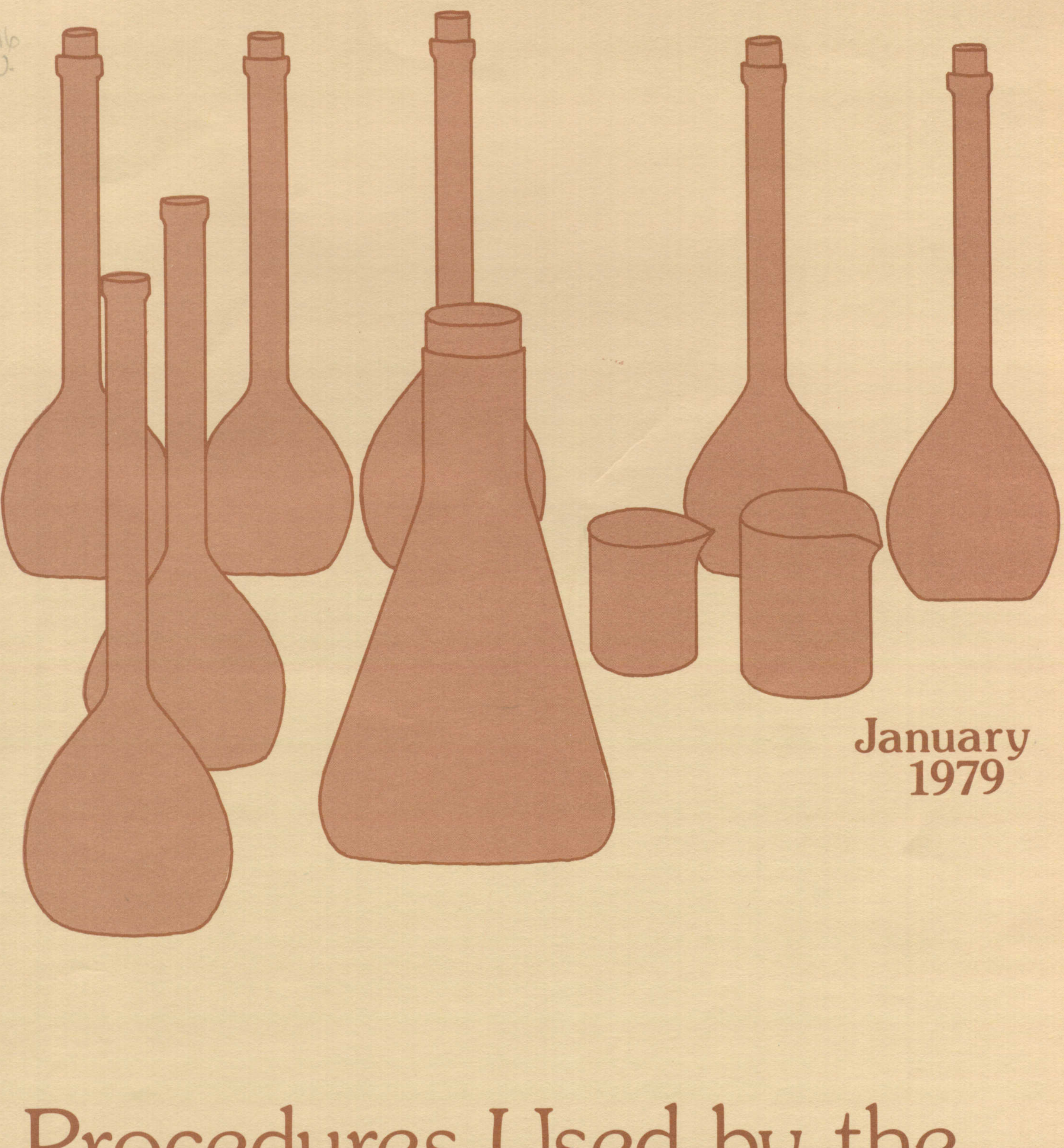

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Procedures Used by the Auburn University Soil Testing Laboratory

PROCEDURES USED BY THE AUBURN UNIVERSITY
SOIL TESTING LABORATORY

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PROCEDURES USED BY THE AUBURN UNIVERSITY SOIL TESTING LABORATORY

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I. Introduction

The soil testing program of Auburn University is a joint program of the Agricultural Experiment Station and the Cooperative Extension Service. The Agricultural Experiment Station conducts soil test calibration research, operates the soil testing laboratory, and makes recommendations. The Cooperative Extension Service has primary responsibility for education on soil testing and distribution of supplies.

Soil samples are routinely tested for pH, lime requirement, and available P,K, Mg, and Ca. Available calcium is only reported on samples where peanuts, tomatoes, peppers, or peaches are to be grown. When requested and a need is indicated, soil organic matter, soluble salts and the micronutrients such as Zn,Mn, and Fe may be determined for additional charges. Fertilizer recommendations are made from the soil test results. In addition to routine soil tests, a pecan leaf analysis service is offered.

II. Soil Sample Preparation

1. Open samples daily. Check names, addresses, numbering of samples, information sheets, and payments ($2.00 per sample, $1.00 extra when organic matter determination is requested).

2. Place samples on shelves and number consecutively beginning with No. 1 the first of July.

3. Classify each soil sample (soil group 1 to 5) by computer based on cation exchange capacity and geographic location:

Soil Group 1	Sandy soils with CEC$<$4.3 meq. per 100 g. (Norfolk, Kalmia, Ruston, Orangeburg, Dothan, etc.)
Soil Group 2	Loams and light clays with CEC 4.3-8.4 meq. per 100 g. (Greenville, Lucedale, Savannah, Cecil, Appling, Colbert, Hartsells, etc.)

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[1] Research Associate and Associate Professor, respectively.

Soil Group 3	Blackbelt soils with CEC >8.4 meq./100g. (Houston, Sumter, Vaiden, etc.)
Soil Group 5	Limestone Valleys soils with CEC >8.4 meq./100 g. (Decatur, Dewey, etc.)

4. Place samples in lab. trays in sequence beginning on the left front row of the tray. Fill in rows of 6 deep and 6 rows to a tray and place in heated drying cabinet at 55°C (130°F) for at least 24 hours or until samples are dry.

5. After samples are dry, screen through a 10-mesh standard sieve or grind in a mechanical grinder. After thorough mixing, samples are ready for analysis.

III. Sample Extraction

1. Weigh a 5 g sample and transfer to a 50 ml Erlenmeyer flask (12, 50 ml Erlenmeyer flasks are fitted into special wooden racks which are numbered at each end).

2. Deliver a 20 ml volume of extracting solution simultaneously to each of the 12 flasks with an automatic pipette rack.

3. Place a set of racks (a maximum of 6) on a mechanical shaker set at 180 oscillations per minute with a 1½ inch stroke. Shake for 5 min.

4. Filter the samples immediately through a 9.0 cm Whatman No. 1 filter paper into 1 oz. plastic cups. The cups are fitted into racks congruent to the extraction racks.

5. Pour the filtered sample extracts into vials fitted in racks. Samples are now ready for analytical determinations.

NOTE: All soils are extracted using the double-acid extracting solution (Mehlich, 1953). However, those soils classified in soil group 3 must be re-extracted separately with the Mississippi extracting solution (Lancaster, 1970).
Soil-test P, K, Mg, and especially Ca usually read higher and the lab results will have to be corrected.

IV. Soil pH and Lime Requirement Determinations (Adams and Evans, 1962)

1. Transfer one scoop of soil (20 ml volume) to a 3 oz. waxed paper cup and add 20 ml of distilled water by means of an automatic pipette rack.

2. Place a tray of 48 samples on a stirring machine equipped to stir all simultaneously. Stirrers should be rinsed between stirring each tray. After stirring for 30 sec., let samples stand for at least 30 min.

3. Stir each sample immediately before making pH determination. The pH meter should be calibrated before use as well as regularly throughout the day's run. Buffer solutions with pH of 4.00 and 7.00 should be used.

4. After recording water pH, add 20 ml of Adams-Evans buffer solution with a similar machine as was used for adding water.

5. Stir for 4 min. on the same mechanical stirrer.

6. Set standard pH meter for expanded scale using buffer 7.0 and a blank of Adams-Evans buffer solution (1:1 buffer-water solution with pH 8.0).

7. Read buffer pH while stirring. (Changes in digital read-out during this reading indicates incomplete mixing of soil-buffer).

8. Take water pH readings with one decimal, buffer pH 2 decimals.

V. Routine Elemental Analysis

A. Phosphorus (Watanabe and Olsen, 1965)

1. Add one part (.35 ml) of the soil extract to 24 parts (8.40 ml) of phosporus Reagent C (color developer) using a diluter-dispenser set for 1:24. This solution is transferred into glass vials for P determination.

2. Aliquot duplicate set of standards from 0 to 16.35 ppm P (table 3).

3. Allow 30 min. for color to develop.
   a. Allow 30 min. period of warmup after turning instrument on.

   b. Read absorbance on Bausch and Lomb Spectronic 100 at 740 nm wavelength.

B. Calcium and Magnesium

1. Using a similar diluter-dispenser, take a 1.5 ml aliquot of extract and dilute with 6.0 ml of .5% Lanthanum solution (100 ml of 5% Lanthanum solution diluted to 1 liter with acid extracting solution).

2. Dilute standards of 0 to 60 ppm Ca and 0 to 6 ppm Mg using the same procedure as extracts (table 3).

3. Read concentration on a Perkin-Elmer model 373 or 460 Atomic Absorption Spectrophotometer with air/acetylene flame adjusted for maximum sensitivity.

C. Potassium

Potassium is read on undiluted extract in concentration on the same Atomic Absorption Spectrophotometer.

VI. Special Analyses

A. Zinc

Pre-rinse glassware with 1 N HCL. Use standard extracting pro-

Lime Requirement for 8-inch Furrow Slice to pH 6.5 (Adams-Evans Buffer Procedure)

Hundreds of Pounds Ag. Lime (CaC)$_3$ X 1½)

Tons per acre	Buffer pH	Water pH																
		5.9	5.8	5.7	5.6	5.5	5.4	5.3	5.2	5.1	5.0	4.9	4.8	4.7	4.6	4.5	4.4	Tons
1.0	7.85	9	10	11	11	12	13	13	14	15	15	16	16	17	17	18	19	1.0
	7.80	12	13	14	15	16	17	18	19	19	20	21	22	22	23	24	25	1.5
	7.75	15	16	18	19	20	21	22	23	24	25	26	27	28	29	30	31	
	7.70	17	19	21	23	24	26	27	28	29	30	31	32	33	34	36	38	2.0
	7.65	20	23	25	27	28	30	31	33	34	35	37	38	39	40	42	44	
	7.60	23	26	28	30	32	34	36	37	39	40	42	43	44	46	48	50	2.5
1.5	7.55	26	29	32	34	36	38	40	42	44	46	47	49	50	52	53	56	3.0
	7.50	29	32	35	38	40	43	46	47	49	51	52	54	56	57	59	63	
	7.45	32	36	39	42	44	47	49	52	54	56	58	59	61	63	65	69	3.5
2.0	7.40	35	39	42	46	48	51	54	56	59	61	63	65	67	69	71	75	4.0
	7.35	38	42	46	49	53	56	58	61	63	66	68	70	72	75	77	81	
	7.30	41	45	49	53	57	60	63	66	68	71	73	76	78	80	83	88	4.5
	7.25	44	48	53	57	61	64	67	70	73	76	78	81	84	86	89	94	
2.5	7.20	47	52	56	61	65	68	72	75	78	81	84	86	89	92	95	100	
	7.15	49	55	60	64	69	73	76	80	83	86	89	92	95	98	101	106	
	7.10	52	58	63	68	73	77	81	84	88	91	94	97	100	103	107	113	
	7.05	55	61	67	72	77	81	85	89	93	96	99	103	106	109	113	119	
	7.00	58	65	70	76	81	85	90	94	98	101	105	108	111	115	119	125	

Auburn University Soil Testing Laboratory, October, 1974.

cedure. Read on AA in lb/acre available Zn. Use standards 0-10 ppm. Zn values must be recorded by hand on each soil test report since the computer is not programmed to report Zn.

B. Manganese Determination

Use routine extracting procedures. Read on AA and report in the same manner as Zn. Use standards of 0-20 ppm.

C. Determination of Soil Organic Matter, modified Jackson's procedure (Jackson, 1958)

1. Weigh 0.5 gram of oven-dry soil into a 250 ml flask. If soil is sandy or low in organic matter, use a 1 gram sample.

2. Add exactly 10 ml of 1 N $K_2Cr_2O_7$.

3. Add rapidly, 10 ml of conc. H_2SO_4.

4. Shake for 1 minute; let stand for 30 minutes.

5. Add 100 ml distilled water. Ten ml of 85% H_3PO_4 may be added for a clearer titration endpoint but this is optional. Solution may also be filtered if necessary.

6. Add approximately 3 drops of 0.025 M Ortho-Phenanthroline ferrous sulphate solution.

7. Titrate with 0.5 N $FeSO_4$. $FeSO_4$ normality should be determined before each run since it is likely to change slightly from day to day. Use two blanks for this.

8. Make the following calculation:

$$\%C = \frac{(\text{m.e. Ox.} - \text{m.e. Red.}) \times 0.3}{\text{Wt. of dry soil}} \times 1.33$$

$$\%\text{O.M.} = \%C \times 1.72$$

$$\text{m.e. Ox.} = N \times \text{ml. } K_2Cr_2O_7$$

$$\text{m.e. Red.} = N \times \text{ml. } FeSO_4$$

Reagents:

1. 1 N $K_2Cr_2O_7$: Dissolve 49.04 grams of $K_2Cr_2O_7$, dried at 104^0, in one liter of deionized water.

2. 0.5 N $FeSO_4$: Dissolve 140 grams of $FeSO_4 \cdot 7H_2O$ in deionized water. Add 40 ml conc. H_2SO_4 and make to one liter.

3. Use commercial, reagent grade, 0.025 M Ortho-phenanthroline ferrous sulphate solution ("Ferroin").

An alternative is the use of a Carbon analyzer from LECO Inc. Co. that reads directly percent carbon from the sample based on the Infrared absorption of CO_2.

D. Soluble Salts

1. A 40 ml volume of soil is placed into a 250 ml Erlenmeyer flask.
2. Add 80 ml distilled H_2O.
3. Shake in mechanical shaker for 15 minutes.
4. Allow to stand for about 20 minutes and filter using Buchner funnel.
5. Read on Solu-bridge. Determine specific conductance of liquid and refer to table 2 for interpretations.

E. Determination of Nitrate in Soils

1. Phenoldisulfonic Acid Procedure (Sou. Coop. Series, 1965; Bremner, 1965)

Reagents:

(a) Sodium hydroxide 1 N

(b) Phenoldisulfonic acid: Dissolve 25 grams of pure, white phenol in 150 ml of concentrated H_2SO_4 in a 500-ml Erlenmeyer flask. Then add 75 ml of fuming H_2SO_4 (13% SO_3), mix the solution, and place the flask (loosely stoppered) in boiling water for 2 hours.

(c) Ammonium hydroxide solution (NH_4OH): Mix equal parts of reagent-grade NH_4OH with distilled water.

(d) Standard nitrate solution: Dissolve 3.608 grams of oven-dried KNO_3 in distilled water and dilute to 1 liter in a volumetric flask. This should contain 500 ppm nitrate. Prepare working standards of 0 to 20 with proper dilution. Standard solutions should be kept refrigerated.

Extraction of Soils:

(a) Place 2 g of soil in a 50 ml Erlenmeyer flask.

(b) Add 20 ml of distilled water.

(c) Shake for 15 min. and filter.

Procedure:

(a) Place 2 ml of the soil extract in a 50 ml pyrex beaker then add 1 drop of 1 N NaOH, and evaporate to dryness on a hot plate (110^{0}C).

Table 2. Interpretation of soluble salt measurement **

The level (reading) of soluble salts which most plants tolerate without injury is greatly influenced by the amount of available water, temperature, uniformity of soil-fertilizer mixture, amount of organic matter, soil texture, fertilizer materials used and other factors peculiar to each operation. The following is a general interpretation which will serve as a guide:

Reading (mho x 10^{-5})	Effect
0-25	Salinity effect negligible. Probably too low for good growth of most crops in greenhouse culture.
26-50	May be inadequate except where fertilizer is applied with each watering or on very light soils.
51-125	Satisfactory for well drained greenhouse soils. Germination of sensitive crops may be restricted and seedlings may be injured.
126-175	Yields of many crops may be restricted. Higher than desirable for greenhouse soils.
More than 175	Leach with enough water so that 2-4 quarts pass through each square foot of bench area or one pint of water per 6 inch pot; wait 30 to 90 minutes and repeat. A third application may be necessary if readings are excessively high.

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(b) Cool. Add 1 ml of phenoldisulfonic acid and rotate to dissolve the residue.

(c) After 10 minutes, add 5 ml of distilled water. Let it cool down, and add 6 ml of the ammonium hydroxide solution.

(d) Read absorbance on B & L Spectronic 100 at 420 nm wavelength after 15 min. standing.

(e) Standards of 0 to 20 ppm of $N\text{-}NO_3$ should be run with the samples. The nitrate content of the samples is determined directly from the calibration graph. Extractions reading greater than 20 ppm should be diluted and analyzed again with new standards.

VII. Preparation of Reagents

A. Adams-Evans Buffer for Lime Requirement (Adams and Evans, 1962)

1. Dissolve 360 g p-Nitrophenol (practical grade) in approximately 4 l. of hot, deionized H_2O. Use low heat to dissolve.
2. Dissolve 270 g of boric acid in 3 l. hot, deionized H_2O. Use low heat if necessary.
3. Dissolve 189 g of potassium hydroxide (KOH) in approximately 200 ml deionized H_2O.
4. Place 1,332 g of potassium chloride (KCl) in 18-l. bottle. To this add about 6 liters distilled H_2O. Mix thoroughly.
5. To the KCl solution, add the previously mixed solutions in same order as listed. Make to 18 liters with deionized H_2O. Adjust the pH to 8.00 with KOH or HCl if necessary.

B. Extracting Solutions (Mehlich, 1953; Lancaster, 1970)

1. 0.05 N HCl and 0.025 N H_2SO_4 mixture. Pour approximately 30 l. distilled H_2O into 40-l. bottle. Add 28 ml conc. H_2SO_4 and 166 ml conc. HCl. Make to 40 l. with distilled H_2O. Mix thoroughly.
2. Mississippi Method (for group 3 soils). Add 900 ml glacial acetic acid, 65 g malonic acid (Eastman practical of equivalent), $CH_2(CO_2H)_2$, 120 g malic acid (Eastman practical), $CH_2CHOH(CO_2H_2)_2$, and 13.8 g ammonium fluoride, NH_4F, to 7500 ml deionized H_2O and mix well to dissolve reagents. Add 30 g aluminum chloride hexahydrate, $AlCl_3 \cdot 6H_2O$, mix well, and adjust pH to 4.0 with NH_4OH. Dilute to 10 l. with deionized H_2O. Mix thoroughly.

C. Phosphorus Reagents (Watanabe and Olsen, 1965)

1. Reagent A. Dissolve 100 g ammonium molybdate, $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$, in approximately 500 ml deionized H_2O in 2-liter volumetric flask. Dissolve 2.425 g antimony potassium tartrate, $K(SbO)C_4H_4O_4 \cdot \frac{1}{2}H_2O$, in

molybdate solution. Add 1,400 ml conc. H_2SO_4, cool, and make to volume with distilled H_2O. Store in polyethylene or pyrex glass bottle in a dark, cool compartment.

2. Reagent B. Dissolve 88.0 g reagent grade ascorbic acid in distilled H_2O dilute to 1 l. Store in dark glass in cool compartment.

3. Reagent C (Working Solution). Dilute needed quantity of Reagents A and B using the following ratio: 1 ml B + 2 ml A + 97 ml extracting solution for a total volume of 100 ml. Prepare fresh daily allowing to stand a minimum of 2 hr and a maximum of 24 hrs.

D. Calcium Reagents

5% Lanthanum Solution. Wet 58.65 grams La_2O_3 with distilled H_2O. Add 250 ml conc. HCl very slowly until material is dissolved. Dilute to 1 liter with distilled H_2O. Working solution - Dilute Lanthanum solution to 0.5% as needed (100 ml of 5% La solution to 1 l. with acid extracting solution).

VIII. Preparation of Stock Solutions and Standards

A. Phosphorus Standard (1,000 ppm P). Place 4.392 g of oven dry potassium biphosphate (KH_2PO_4) in 1 liter volumetric flask. Dissolve in acid extracting solution and make to volume with the same extracting solution. Dilute according to data in table 3.

B. Potassium Standard (1,000 ppm K). Place 3.8132 g of oven-dry potassium chloride (KCl) in 2-l. volumetric flask. Dissolve and make to volume with acid extracting solution. Dilute according to data in table 3.

C. Magnesium Standard (1,000 ppm Mg). Dissolve 1.000 g. pure magnesium (Mg) in a minimum volume of conc. HCl dilute to 1 l. Dilute according to data in table 3.

D. Calcium Standard (1,000 ppm Ca). Weigh 5.0 g calcium carbonate ($CaCO_3$) into a 2-liter volumetric flask. Dissolve in 50 ml of 3 N HCl. Make to volume with acid extracting solution.

IX. Computer Data

For each farmer that sends in a soil sample, at least 3 computer files are punched with information which will be used by the computer program in reporting test results and in making fertilizer recommendations. The 3 files contain the following information:

Name file: This file contains the farmer's name, address, county code

Table 3. Preparation of P, K, Mg, and Ca standards from stock solutions [1]

Std. sol. number	Volume of 1,000 ppm stock std. diluted to 1 liter with acid extracting solution (ml per liter)			
	P	K	Mg	Ca[2]
1	0	0	0	0
2	1	5	1	10
3	5	10	2	20
4	10	20	4	40
5	15	30	6	80
6	20	40	8	120
7	25	50	10	160

[1] Above volumes of P, K, Ca, and Mg stock solutions taken per liter are also equivalent to ppm.

[2] To convert ppm in sample extracts to lb. per acre, multiply by 8. Calcium standards contain 100 ml 5% La per liter, making them 0.5% La.

number, number of samples, number of copies, and farmer number.

Crop file: This file contains the sample name or number, crop to be grown, crop code number (for fertilizer recommendation), laboratory number, number of crops for that sample, and farmer number.

Sample file: This file contains the farmer number, the laboratory number, number of crops for which recommendations are given, soil pH, buffer pH, Ca, P, K, and Mg, and special comment number which can be added if desired.

A computer program is written to convert the absorbance of P readings and Ca reading to lb./A.

Soil test reports are computer printed for each farmer on special forms (Soil Test Form B). The report contains his sample designation, pH value, P, K, Ca, and Mg values reported in percent sufficiency (fertility index). Each value is rated excessively high, very high, high, medium, low, or very low for each element. Fertilizer and lime recommendations are made for each crop to be grown for that sample. On samples where special analyses such as Zn, Mn, organic matter, soluble salts, etc. are made, the results are written on the report by hand.

X. Plant Analysis

The Soil Testing Laboratory offers a pecan leaf analysis service where a routine soil test plus Ca and Zn and a pecan leaf analysis for N, P, K, Mg, Ca, Zn and An are made for $10.00. Samples received for a pecan leaf analysis are numbered consecutively beginning with number 1 for the first sample of the calendar year (i.e., 73-1, 73-2, 73-3, etc.). Soil and leaf samples from the same orchard receive the same number.

Soil samples are also numbered and included in a routine "run". Procedures for analysis are the same as previously described. Ca and Zn are determined and reported in addition to the routine analysis.

Pecan leaf samples are tested for N, P, K, Ca, Mg, and Zn. Results are reported on the enclosed sheet. Recommendations based on the soil test results and leaf analysis are made by the Auburn University Department of Horticulture.

Dry ash is prepared as follows:

1. Weigh 1 gram (or 0.5 gram if instructed) of dry plant material into 50 ml pyrex beaker. (Plant material should be ground to pass a 40-mesh, stainless steel sieve).
2. Cover with watch glass and place in muffle furnace. Heat to 450°C and hold at that temperature until all carbon is burned off. The ash should be greyish white.

3. Cool. Add 10 ml of 1 N nitric acid (HNO_3) and evaporate to dryness slowly on hot plate. Take just to dryness and do not bake.

4. Cool. Add 10 ml 1 N HCl to dissolve the residue.

5. Warm nearly to boiling then transfer to 100 ml volumetric flask. Wash beaker 3 times with small amounts of water.

6. Bring to volume of 100 ml and filter. Do not wash filter paper or add more water in this step.

7. Always run a blank sample.
   Solutions: 1 N HCl = 166 ml conc. HCl per 2 liters
   1 N HNO_3 = 128 ml conc. HNO_3 per 2 liters

C. Determinations of P, K, Mg, Ca, and Zn on the ashed samples are made according to routine procedures for each.

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*Information contained
herein is available to
all regardless of race,
color, or national origin.*