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**P***rocedures used  
for soil and plant  
analysis by the  
Auburn University  
Soil Testing  
Laboratory*



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PROCEDURES USED BY THE AUBURN UNIVERSITY

SOIL TESTING LABORATORY

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Information contained herein is available to all persons regardless of race, color, sex or national origin

PROCEDURES USED BY THE AUBURN UNIVERSITY  
SOIL TESTING LABORATORY

N. V. Hue and C. E. Evans<sup>1</sup>

I. Introduction

The soil testing program at Auburn University is a joint project of the Alabama Agricultural Experiment Station and the Alabama Cooperative Extension Service. The Alabama Agricultural Experiment Station conducts soil-test calibration research and operates the soil testing laboratory. The Alabama 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 extractable P, K, Mg, and Ca. Extractable Ca is only reported on samples where peanuts, tomatoes, peppers, or peaches are to be grown. When requested or a need is indicated, soil organic matter, soluble salts, and micronutrients such as Zn, Mn, Fe, Cu, and B may be determined for additional charges. Fertilizer recommendations are made from the soil test results. In addition to routine soil tests, a limited plant analysis service is offered.

II. Soil Sample Preparation

1. Checking samples. Soil samples are received daily and immediately checked for farmers' name, address, number of samples, crops to be grown, and payments (\$3 per sample for routine analysis, and an additional \$2 per sample when organic matter determination is requested). The checking operation is carried out by comparing the information listed on each soil-sample box with that written on the "information sheet." If the two sources of information do not match, an attempt is made to reconcile the differences, and if needed, a comment will be made on the report returned to the farmer.
2. Numbering samples. After checking, soil samples are placed on shelves and numbered consecutively, beginning with No. 1 on the first of July each year.

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3. Identifying Black Belt soil samples. Soils from the Black Belt area of Alabama, which are often calcareous and high in clay content, are extracted with a different solution. Therefore, an effort is made to separate them from other soils before chemical analysis is performed. The separation is carried out by visually inspecting, hand-feeling the soil texture in conjunction with the county (location) where the soil was sampled, and by the HCl effervescent test.
4. Drying samples. Soils are placed in metal trays in sequence beginning on the left front row of the tray. Each tray consists of 36 (6 x 6) samples. They are then dried in a large forced-air oven at 55°C (130°F) for at least 24 hours or until samples are dry.
5. Screening samples. Dry samples are ground in a mechanical grinder and screened through a 10-mesh sieve to obtain homogeneity of the soil. Soil samples are then ready for chemical analyses.

### III. Instrumentation Setup in the Laboratory

Since 1978, the Auburn University Soil Testing Laboratory has been equipped with a mini-computer (MODCOMP II/25). Various analytical instruments, including an electronic balance (Mettler, PE 200), an atomic absorption spectrometer (Perkin-Elmer 373), two digital pH meters with BCD output (Sargent-Welch, PAX), and a visible spectrophotometer (Bausch & Lomb, SPECTRONIC 100) are interfaced to the computer either directly or via a custom-built interface box. A block diagram of the instrumentation setup is shown in figure 1. Laboratory data, such as soil-water pH, buffer pH, and P, K, Mg, and Ca concentrations in soil extract, are acquired automatically by the computer. With this setup, a technician does not have to write down readings or punch-in numbers. The computerized process for soil-test data acquisition and its applications have been discussed elsewhere (10, 9).

#### Computer Interface

The interface serves three functions: (1) provides communication from the computer to the technician, (2) allows the technician to input test parameters and identification, and (3) provides translation and input of digital data signals from the instrument to the computer.

A pictorial diagram of an interface is presented in figure 2. The interface has 4-digit LED display by which the computer relays messages to the technician. The display shows either position number, or instrument reading, or error messages. For analysis, each batch of samples is placed in laboratory trays in which positions are numbered. These begin at "1" and are numbered in increasing order. Position numbers represent laboratory numbers

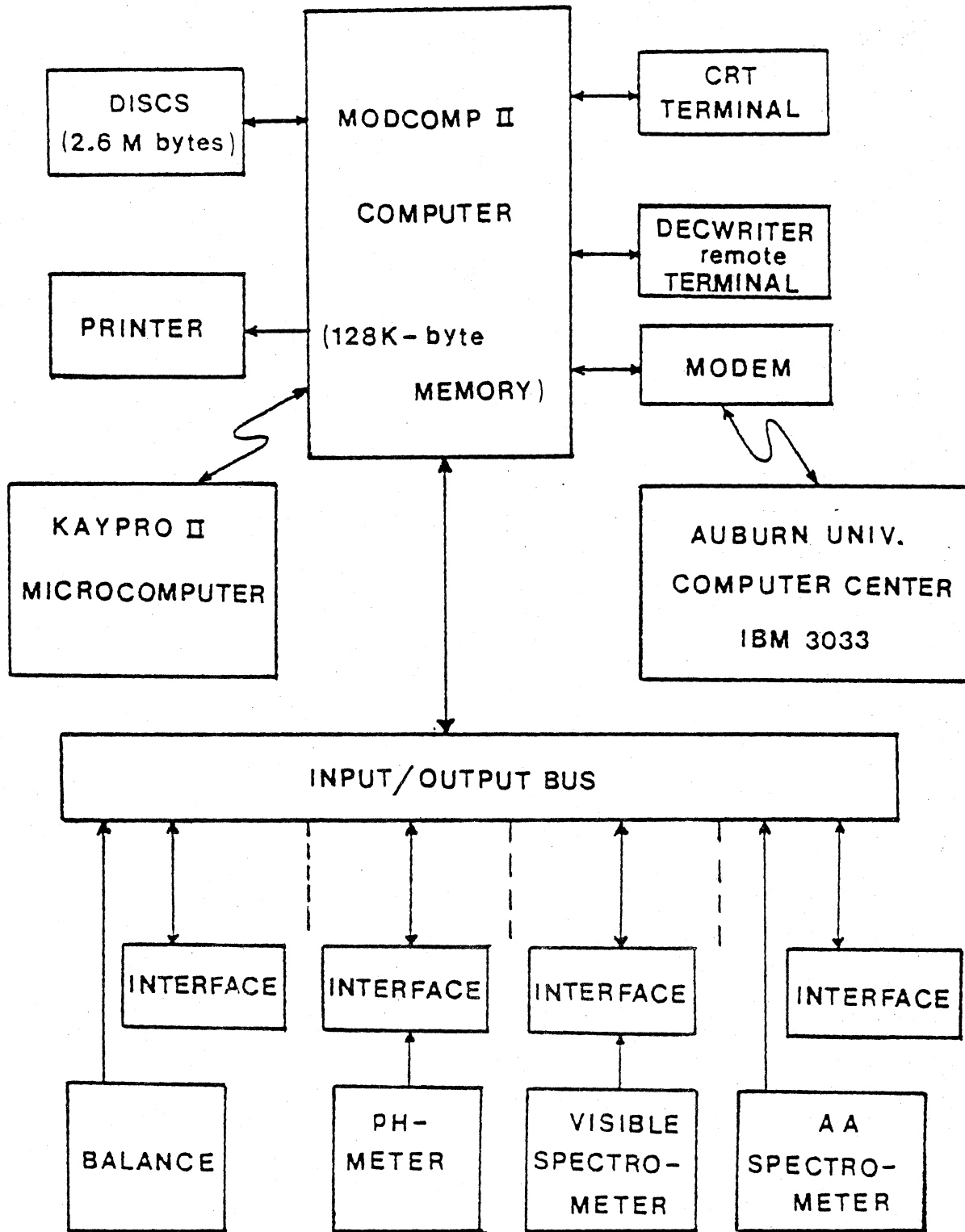


Fig. 1. Block diagram of the computer system of the Auburn University Soil Testing Laboratory

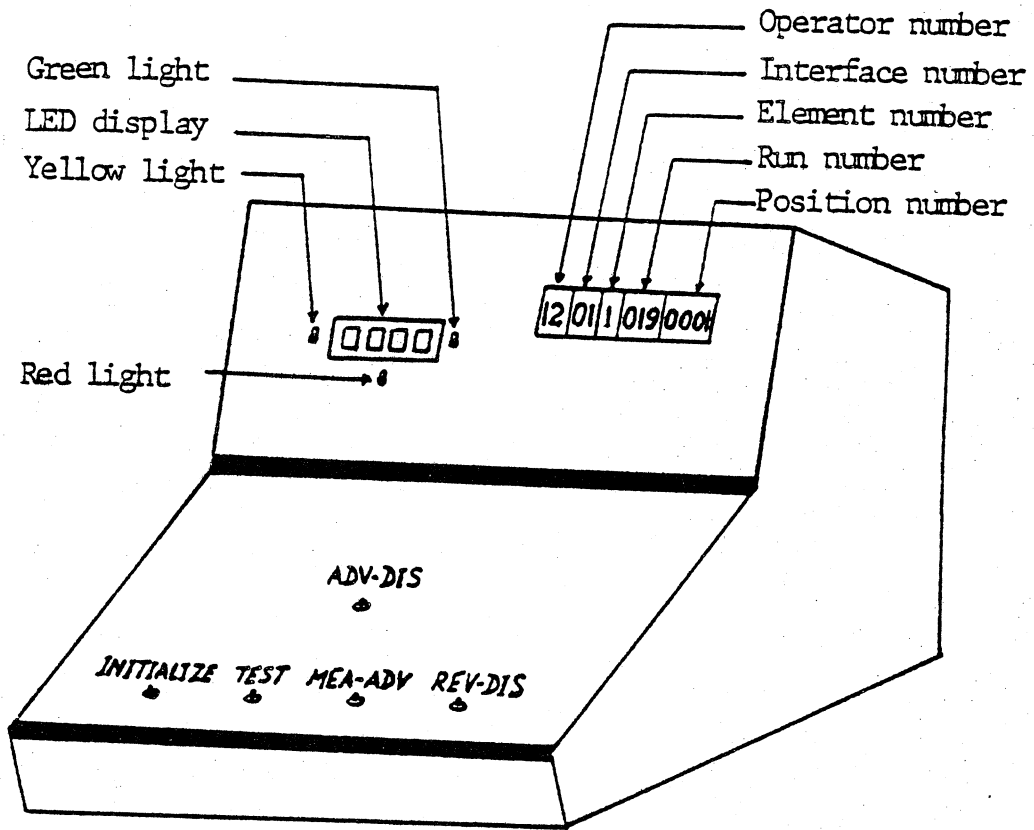


Fig. 2. Operator-and-Instrument Interface.



for the samples in a "run". The upcoming position number is displayed for the technician before each analysis. After each analysis, the computer momentarily shows the reading back to the technician on the LED display, indicating that it has been recorded, and then it advances to the next position number. Error codes are shown when there is a hardware or software problem or if the technician is attempting to perform an unacceptable task. The LED display is surrounded by three lights, which are cues to the technician. A yellow light indicates that the computer is ready to accept data, a green light indicates that the computer has acquired valid data and is doing the bookkeeping to accept the next data, and a red light indicates an error or an invalid operation.

The interface has five sets of thumbwheel switches located on the right side of the upper-front panel. These digits are set by the technician at the beginning of each run so that data can be correctly collected by the computer. Beginning on the left, the first two digits identify the operator, the third and fourth identify the interface, and the fifth identifies the element being analyzed. Digits 6-8 identify the run number. A run contains from 1 to 499 samples, and the run number is advanced consecutively with groups of samples analyzed each year. Digits 9-12 set the beginning position number for startup of analyses on the instrument. By using run numbers to identify groups of samples, the position numbers can be repeated for each group of samples to represent different laboratory numbers.

The interface has five pushbuttons (Figure 2): Initialize, Test, Mea/Adv, Rev/Dis, and Adv/Dis located on the lower-front panel. Upon pressing one of these buttons, the associated software program will be activated, and certain operations will be carried out:

| <u>Function</u> | <u>Operation description</u>  |
|-----------------|---|
| TEST            | Allows the operator to check for proper computer communication and for proper hardware operation of the interface.  |
| INITIALIZE      | Commands the computer to obtain information from the thumbwheel switches, check for the validity of the information, and set up the necessary data files on disc for the data acquisition process.    |
| MEA/ADV         | Initiates the transfer of an analytical measurement to the computer. The computer then performs the necessary bookkeeping chores, stores the data on disc, and advances its internal position by one. |

REV/DIS

Allows the operator to decrement the position by one with respect to the current position and shows on the LED display the measurement that has been stored. If the operator does not like the reading, the MEA/ADV switch can be pressed to take a new reading.

ADV/DIS

Allows the operator to advance the position by one with respect to the current position and displays the reading that had been taken, if any.

#### IV. Soil Sample Extraction

Two extracting solutions are being used by the laboratory to handle the diversity of soils throughout the State. The Mehlich 1 (12) solution, also known as the Double-Acid (0.05 N HCl + 0.025 N H<sub>2</sub>SO<sub>4</sub>) solution, is used for soils other than Black Belt. The latter soil group is extracted with the Mississippi solution (11).

##### 1. Reagents (extracting solutions)

- a. 0.05 N HCl + 0.025 N H<sub>2</sub>SO<sub>4</sub> solution (12). Pour approximately 30 L deionized H<sub>2</sub>O into 40 L bottle. Add 28 mL concentrated H<sub>2</sub>SO<sub>4</sub> and 166 mL concentrated HCl. Dilute to 40 L with deionized H<sub>2</sub>O. Mix thoroughly.
- b. Mississippi solution (11). Add 900 mL glacial acetic acid, 65 g of malonic acid (Eastman practical or equivalent), CH<sub>2</sub>(CO<sub>2</sub>H)<sub>2</sub>, 120 g of malic acid (Eastman practical), CH<sub>2</sub>CHOH(CO<sub>2</sub>H)<sub>2</sub>, and 13.8 g of NH<sub>4</sub>F, to 7500 mL of deionized H<sub>2</sub>O and mix well to dissolve reagents. Add 30 g of AlCl<sub>3</sub>·6H<sub>2</sub>O, mix well, and adjust pH to 4.0 with NH<sub>4</sub>OH. Dilute to 10 L with deionized H<sub>2</sub>O. Mix thoroughly.

##### 2. Procedure

- a. Scoop 4 cubic cm of soil into a weighing pan so that soil weight can be recorded automatically by the computer (the weight is approximately 5 g, depending on the soil texture, i.e., sandy soils are heavier and clayey soils are lighter). Pour it into a 50-mL Erlenmeyer flask (12 flasks are fitted into a special wooden rack which is numbered at both ends).
- b. Deliver a 20-mL aliquot of extracting solution simultaneously to each of the 12 flasks with an automatic pipette assembly.

- c. Place a set of racks (a maximum of 6) on a mechanical shaker (180 oscillations/minute, 3.8-cm displacement/stroke). Cover them with a thin polyethylene sheet. Shake for 5 minutes for the Double-Acid, and 10 minutes for the Mississippi.
- d. Filter the samples immediately through a 9-cm Whatman No. 1 filter paper into 1-oz plastic cups. The cups are fitted into racks congruent to the extractant racks.
- e. Pour the filtrate into a 10-mL plastic vial. The samples are now ready for P, K, Mg, and Ca determinations.

## V. Routine Elemental Analyses

### 1. Phosphorus Determination (14, 13).

Principle. Phosphate reacts with sulfomolybdic acid in the presence of a reducing reagent (ascorbic acid) to yield a blue compound (phosphomolybdic). The intensity of the color is linearly proportional to the concentration of solution phosphate.

#### Phosphorus Reagents (14)

- a. Reagent A. Dissolve 100 g of ammonium molybdate,  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$ , in approximately 500 mL deionized  $\text{H}_2\text{O}$  in 2 L volumetric flask. Dissolve 2.425 g of antimony potassium tartrate,  $\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_4\cdot \frac{1}{2}\text{H}_2\text{O}$ , in molybdate solution. Add 1,400 mL concentrated  $\text{H}_2\text{SO}_4$ , cool, and make to volume with distilled  $\text{H}_2\text{O}$ . Store in polyethylene or pyrex glass bottle in a dark, cool compartment.
- b. Reagent B. Dissolve 88 g of ascorbic acid in deionized  $\text{H}_2\text{O}$ . Dilute to 1 L. Store in dark glass in cool compartment.
- c. Reagent C (color developer). Dilute needed quantity of Reagents A and B, using the following ratio: 2 mL A + 1 mL B + 97 mL extracting solution for a total volume of 100 mL. Prepare fresh solution daily, and allow the solution to stand a minimum of 1 hour before use (the solution is good for @ 48 hours).

#### Procedure

- a. Add one part (0.85 mL) of the soil extract to 9 parts (7.65 mL) of phosphorus reagent C (color developer), using a diluter/dispenser (MANOSTAT, DILUTOR II) set for 1:9. This solution is transferred into a glass vial for P determination. Allow 30 minutes for color to develop.

- b. Aliquot duplicate sets of P standards: 0, 0.5, 1.0, 2.5, 5.0 and 10.0 ppm P (as  $\text{KH}_2\text{PO}_4$  solutions), and mix them with color developer as described in [a]. These standards correspond to soil-test P of 0, 4, 8, 20, 40, and 80 pounds per acre, respectively.
- c. Turn on the B & L SPECTRONIC 100, and allow it to warm-up for 15 minutes. Set the wavelength at 740 nm. Read absorbance of the standards and samples.

## 2. Potassium Determination

Potassium is determined by atomic absorption spectroscopy on the undiluted soil extract.

Principle. At elevated temperatures (@ 2,000°C with  $\text{C}_2\text{H}_2$ /air flame), K salt in soil extract is converted into atomic  $\text{K}$  and simple K compounds. Potassium atoms in the ground state will absorb certain energy (provided by a K hollow cathode lamp). The energy difference between the unabsorbed reference light beam and the absorbed sample beam is proportional to K concentration in the liquid extract.

### Procedure

- a. AA (Perkin-Elmer 373) operating parameters. Wavelength = 766.5 nm, slit width = 2, burner angle = 30° from the light path.
- b. Standards. 20 and 50 ppm K in the Double-Acid solution. The standards, however, are entered as 160 and 400 (pounds per acre of K in soil). The instrument absorbance is first zeroed with the Double-Acid solution.

## 3. Calcium and Mg Determinations

Calcium is determined by using a  $\text{C}_2\text{H}_2$ /air flame in La solution. The La addition is to help reduce interferences from other elements, especially P, on Ca determination.

Magnesium is also determined on the La diluted solution for convenience. This matrix, however, is not required for Mg determination.

### Reagent (lanthanum solution)

5% Lanthanum Solution. Wet 58.65 g of  $\text{La}_2\text{O}_3$  with  $\text{H}_2\text{O}$ . Add 250 mL of concentration HCl very slowly until the material is dissolved. Dilute to 1 L with deionized  $\text{H}_2\text{O}$ .

Working solution. Dilute La solution to 0.5% La as needed (100 mL of 5% La solution to 1 L with the Double-Acid solution).

### Procedure

- a. Using a diluter/dispenser, take a 1.5-mL aliquot of soil extract and mix with 6 mL of 0.5% La solution.
- b. AA operating parameters.

| Parameter             | Ca    | Mg    |
|-----------------------|-------|-------|
| Wavelength, nm        | 422.7 | 285.2 |
| Slit width            | 0.7   | 0.7   |
| Burner angle (degree) | 30    | 30    |
| Low standard, ppm     | 30    | 3     |
| Hi standard, ppm      | 60    | 6     |

Note: Ca and Mg standards should be prepared in La matrix.

### VI. Soil pH and Lime Requirement Determinations

Apace of soil extraction, soil pH and lime requirement (for acid soils) are determined.

#### Reagent [Adams-Evans (1) buffer for lime requirement]

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

#### Procedure

1. Transfer one scoop of soil (20 mL) to a 3-oz waxed paper cup, and add 20 mL of deionized H<sub>2</sub>O by means of an automatic pipette assembly.
2. Place a tray of 48 samples on a mechanical stirrer which is equipped to stir all samples simultaneously. Stirring rods should be rinsed between trays. After 30-seconds stirring, samples are allowed to stand for at least 30 minutes.

3. Stir each sample immediately before pH determination. The pH meter should be calibrated before use and regularly throughout the day's run. Buffer solutions of pH 4.00 and 7.00 should be used for calibration.
4. After soil-water pH has been taken, add 20 mL of Adams-Evans (1) buffer solution with a similar automatic pipette assembly.
5. Stir samples for 4 minutes on the same mechanical stirrer.
6. Standardize pH meter. Set the Adams-Evans buffer at pH 8.00 from a "blank" of the buffer solution (1:1 by volume of the buffer-water mixture).
7. Read buffer pH while stirring (changes in reading during this measurement indicate incomplete mixing of soil and buffer).
8. Take soil-water pH measurements with one decimal, buffer pH with 2 decimals.
9. Lime requirement is calculated from soil pH and buffer pH for an acid soil (tables 1 to 4).

Table 1. The Amount of Agricultural Lime Needed to Raise the pH of Acid Soils to 6.5 Based on Soil pH and the "Adams-Evans" Buffer pH

|        |    | Hundreds of pounds ag. lime at different pH <sup>1</sup> |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |               |
|--------|----|--|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|---------------|
|        |    | 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/<br>acre |
| 7.90 - | 6  | 6  | 7   | 8   | 8   | 9   | 9   | 9   | 10  | 10  | 10  | 11  | 11  | 11  | 12  | 13  |     |               |
| 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  | 20  | 20  | 21  | 22  | 22  | 23  | 24  | 25  |     |               |
| 7.75 - | 15 | 16   | 18  | 19  | 20  | 21  | 22  | 23  | 24  | 25  | 26  | 27  | 28  | 29  | 30  | 31  |     | 1.5           |
| 7.70 - | 17 | 19   | 21  | 23  | 24  | 26  | 27  | 28  | 29  | 30  | 31  | 32  | 33  | 34  | 36  | 38  |     |               |
| 7.65 - | 20 | 23   | 25  | 27  | 28  | 30  | 31  | 33  | 34  | 35  | 37  | 38  | 39  | 40  | 42  | 44  |     | 2.0           |
| 7.60 - | 23 | 26   | 28  | 30  | 32  | 34  | 36  | 37  | 39  | 40  | 42  | 43  | 45  | 46  | 48  | 50  |     | 2.5           |
| 7.55 - | 26 | 29   | 32  | 34  | 36  | 38  | 40  | 42  | 44  | 46  | 47  | 49  | 50  | 52  | 53  | 56  |     |               |
| 7.50 - | 29 | 32   | 35  | 38  | 40  | 43  | 45  | 47  | 49  | 51  | 52  | 54  | 56  | 57  | 59  | 63  |     | 3.0           |
| 7.45 - | 32 | 36   | 39  | 42  | 44  | 47  | 49  | 52  | 54  | 56  | 58  | 59  | 61  | 63  | 65  | 69  |     | 3.5           |
| 7.40 - | 35 | 39   | 42  | 46  | 48  | 51  | 54  | 56  | 59  | 61  | 63  | 65  | 67  | 69  | 71  | 75  |     |               |
| 7.35 - | 38 | 42   | 46  | 49  | 53  | 56  | 58  | 61  | 63  | 66  | 68  | 70  | 72  | 75  | 77  | 81  |     | 4.0           |
| 7.30 - | 41 | 45   | 49  | 53  | 57  | 60  | 63  | 66  | 68  | 71  | 73  | 76  | 78  | 80  | 83  | 88  |     |               |
| 7.25 - | 44 | 48   | 53  | 57  | 61  | 64  | 67  | 70  | 73  | 76  | 78  | 81  | 84  | 86  | 89  | 94  |     |               |
| 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   | 71  | 76  | 81  | 85  | 90  | 94  | 98  | 101 | 105 | 108 | 111 | 115 | 119 | 125 |     |               |

<sup>1</sup> Pure CaCO<sub>3</sub> x 1.5; for tillage depth of 8 inches.

Table 2. The Amount of Agricultural Lime Needed to Raise the pH of Acid Soils to 6.0 Based on the Soil pH and the "Adams-Evans" Buffer pH

Hundreds of pounds ag. lime at different pH<sup>1</sup>

|        | 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/<br>acre |
|--------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|---------------|
| 7.90 - | 1   | 2   | 3   | 4   | 4   | 5   | 6   | 6   | 7   | 7   | 8   | 8   | 9   | 9   | 10  | 11  | 1.0           |
| 7.85 - | 2   | 3   | 4   | 5   | 7   | 8   | 9   | 9   | 10  | 11  | 12  | 13  | 13  | 14  | 15  | 16  |               |
| 7.80 - | 2   | 4   | 6   | 7   | 9   | 10  | 11  | 13  | 14  | 15  | 16  | 17  | 18  | 19  | 20  | 22  |               |
| 7.75 - | 3   | 5   | 7   | 9   | 11  | 13  | 14  | 16  | 17  | 18  | 20  | 21  | 22  | 23  | 25  | 27  | 1.5           |
| 7.70 - | 3   | 6   | 9   | 11  | 13  | 15  | 17  | 19  | 20  | 22  | 24  | 25  | 27  | 28  | 30  | 33  |               |
| 7.65 - | 4   | 7   | 10  | 13  | 15  | 18  | 20  | 22  | 24  | 26  | 28  | 29  | 31  | 33  | 35  | 38  | 2.0           |
| 7.60 - | 4   | 8   | 11  | 15  | 17  | 20  | 23  | 25  | 27  | 29  | 31  | 33  | 35  | 38  | 40  | 43  |               |
| 7.55 - | 5   | 9   | 13  | 16  | 20  | 23  | 26  | 28  | 31  | 33  | 35  | 38  | 40  | 42  | 45  | 49  | 2.5           |
| 7.50 - | 5   | 10  | 14  | 18  | 22  | 25  | 28  | 31  | 34  | 37  | 39  | 42  | 44  | 47  | 50  | 54  |               |
| 7.45 - | 6   | 11  | 16  | 20  | 24  | 28  | 31  | 34  | 38  | 40  | 43  | 46  | 49  | 52  | 55  | 60  | 3.0           |
| 7.40 - | 6   | 12  | 17  | 22  | 26  | 30  | 34  | 38  | 41  | 44  | 47  | 50  | 53  | 56  | 60  | 65  | 3.5           |
| 7.35 - | 7   | 13  | 19  | 24  | 28  | 33  | 37  | 41  | 44  | 48  | 51  | 54  | 58  | 61  | 65  | 71  |               |
| 7.30 - | 7   | 14  | 20  | 25  | 35  | 31  | 40  | 44  | 48  | 51  | 55  | 59  | 62  | 66  | 70  | 76  | 4.0           |
| 7.25 - | 8   | 15  | 21  | 27  | 33  | 38  | 43  | 47  | 51  | 55  | 59  | 63  | 67  | 70  | 75  | 81  |               |
| 7.20 - | 8   | 16  | 23  | 29  | 35  | 40  | 45  | 50  | 55  | 59  | 63  | 67  | 71  | 75  | 80  | 87  |               |
| 7.15 - | 9   | 17  | 24  | 31  | 37  | 43  | 48  | 53  | 58  | 62  | 67  | 71  | 75  | 80  | 85  | 92  |               |
| 7.10 - | 9   | 18  | 26  | 33  | 39  | 45  | 51  | 56  | 61  | 66  | 71  | 75  | 80  | 84  | 90  | 98  |               |
| 7.05 - | 10  | 19  | 27  | 35  | 41  | 48  | 54  | 59  | 65  | 70  | 75  | 79  | 84  | 89  | 94  | 103 |               |
| 7.00 - | 10  | 20  | 28  | 36  | 44  | 50  | 57  | 63  | 68  | 74  | 79  | 84  | 89  | 94  | 99  | 109 |               |

<sup>1</sup> Pure CaCO<sub>3</sub> x 1.5; for tillage depth of 8 inches.



Table 3. The Amount of Agricultural Lime Needed to Raise the pH of Acid Soils to 5.5 Based on the Soil pH and the "Adams-Evans" Buffer pH

|        | Hundreds of pounds ag. lime at different pH <sup>1</sup> |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     | Tons/<br>acre |
|--------|--|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|---------------|
|        | 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 |               |
| 7.90 - | 0  | 0   | 0   | 0   | 0   | 1   | 2   | 3   | 3   | 4   | 5   | 6   | 6   | 7   | 8   | 9   |               |
| 7.85 - | 0  | 0   | 0   | 0   | 0   | 1   | 3   | 4   | 5   | 6   | 7   | 8   | 9   | 10  | 12  | 13  | 1.0           |
| 7.80 - | 0  | 0   | 0   | 0   | 0   | 2   | 4   | 5   | 7   | 8   | 10  | 11  | 12  | 14  | 15  | 18  |               |
| 7.75 - | 0  | 0   | 0   | 0   | 0   | 2   | 4   | 7   | 8   | 10  | 12  | 14  | 15  | 17  | 19  | 22  |               |
| 7.70 - | 0  | 0   | 0   | 0   | 0   | 3   | 5   | 8   | 10  | 12  | 14  | 17  | 19  | 21  | 23  | 27  | 1.5           |
| 7.65 - | 0  | 0   | 0   | 0   | 0   | 3   | 6   | 9   | 12  | 14  | 17  | 19  | 22  | 24  | 27  | 31  |               |
| 7.60 - | 0  | 0   | 0   | 0   | 0   | 4   | 7   | 10  | 14  | 16  | 19  | 22  | 25  | 28  | 31  | 36  | 2.0           |
| 7.55 - | 0  | 0   | 0   | 0   | 0   | 4   | 8   | 12  | 15  | 18  | 22  | 25  | 28  | 31  | 35  | 40  |               |
| 7.50 - | 0  | 0   | 0   | 0   | 0   | 5   | 9   | 13  | 17  | 21  | 24  | 28  | 31  | 34  | 38  | 45  |               |
| 7.45 - | 0  | 0   | 0   | 0   | 0   | 5   | 10  | 14  | 19  | 23  | 26  | 30  | 34  | 38  | 42  | 49  | 2.5           |
| 7.40 - | 0  | 0   | 0   | 0   | 0   | 6   | 11  | 16  | 20  | 25  | 29  | 33  | 37  | 41  | 46  | 54  |               |
| 7.35 - | 0  | 0   | 0   | 0   | 0   | 6   | 12  | 17  | 22  | 27  | 31  | 36  | 40  | 45  | 50  | 58  | 3.0           |
| 7.30 - | 0  | 0   | 0   | 0   | 0   | 6   | 13  | 18  | 24  | 29  | 34  | 39  | 43  | 48  | 54  | 63  |               |
| 7.25 - | 0  | 0   | 0   | 0   | 0   | 7   | 13  | 20  | 25  | 31  | 36  | 41  | 46  | 52  | 58  | 67  | 3.5           |
| 7.20 - | 0  | 0   | 0   | 0   | 0   | 7   | 14  | 21  | 27  | 33  | 39  | 44  | 50  | 55  | 61  | 71  |               |
| 7.15 - | 0  | 0   | 0   | 0   | 0   | 8   | 15  | 22  | 29  | 35  | 41  | 47  | 53  | 59  | 65  | 76  |               |
| 7.10 - | 0  | 0   | 0   | 0   | 0   | 8   | 16  | 23  | 30  | 37  | 43  | 50  | 56  | 62  | 69  | 80  | 4.0           |
| 7.05 - | 0  | 0   | 0   | 0   | 0   | 9   | 17  | 25  | 32  | 39  | 46  | 52  | 59  | 66  | 73  | 85  |               |
| 7.00 - | 0  | 0   | 0   | 0   | 0   | 9   | 18  | 26  | 34  | 41  | 48  | 55  | 62  | 69  | 77  | 89  |               |

<sup>1</sup> Pure CaCO<sub>3</sub> x 1.5; for tillage depth of 8 inches.

Table 4. The Amount of Agricultural Lime Needed to Raise the pH of Acid Soils to 7.0 Based on the Soil pH and the "Adams-Evans" Buffer pH

|        |    | Hundreds of pounds ag. lime at different pH <sup>1</sup> |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |           |
|--------|----|--|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----------|
|        |    | 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/acre |
| 7.95 - | 3  | 4  | 4   | 4   | 5   | 5   | 5   | 5   | 5   | 6   | 6   | 6   | 6   | 6   | 6   | 6   | 6   | 1.0       |
| 7.90 - | 7  | 7  | 8   | 9   | 9   | 10  | 10  | 10  | 11  | 11  | 11  | 12  | 12  | 12  | 12  | 13  |     |           |
| 7.85 - | 10 | 11   | 12  | 13  | 14  | 14  | 15  | 16  | 16  | 17  | 17  | 17  | 18  | 18  | 19  | 19  |     |           |
| 7.80 - | 13 | 15   | 16  | 17  | 18  | 19  | 20  | 21  | 22  | 22  | 23  | 23  | 24  | 24  | 25  | 25  |     | 1.5       |
| 7.75 - | 17 | 19   | 20  | 22  | 23  | 24  | 25  | 26  | 27  | 28  | 29  | 29  | 30  | 30  | 31  | 31  |     |           |
| 7.70 - | 20 | 22   | 24  | 26  | 28  | 29  | 30  | 31  | 32  | 33  | 34  | 35  | 36  | 36  | 37  | 38  |     | 2.0       |
| 7.65 - | 23 | 26   | 28  | 30  | 32  | 34  | 35  | 37  | 38  | 39  | 40  | 41  | 42  | 42  | 43  | 44  |     |           |
| 7.60 - | 27 | 30   | 32  | 35  | 37  | 39  | 40  | 42  | 43  | 44  | 46  | 47  | 48  | 49  | 49  | 50  |     | 2.5       |
| 7.55 - | 30 | 33   | 36  | 39  | 41  | 43  | 45  | 47  | 49  | 50  | 51  | 52  | 54  | 55  | 56  | 57  |     |           |
| 7.50 - | 33 | 37   | 40  | 43  | 46  | 48  | 50  | 52  | 54  | 55  | 57  | 58  | 59  | 61  | 62  | 63  |     | 3.0       |
| 7.45 - | 37 | 41   | 45  | 48  | 51  | 53  | 55  | 57  | 59  | 61  | 63  | 64  | 65  | 67  | 68  | 69  |     | 3.5       |
| 7.40 - | 40 | 45   | 49  | 52  | 55  | 58  | 60  | 63  | 65  | 67  | 68  | 70  | 71  | 73  | 74  | 75  |     | 4.0       |
| 7.35 - | 43 | 48   | 53  | 56  | 60  | 63  | 65  | 68  | 70  | 72  | 74  | 76  | 77  | 79  | 80  | 82  |     |           |
| 7.30 - | 47 | 52   | 57  | 61  | 64  | 68  | 70  | 73  | 75  | 78  | 80  | 82  | 83  | 85  | 86  | 88  |     | 4.5       |
| 7.25 - | 50 | 56   | 61  | 65  | 69  | 72  | 75  | 78  | 81  | 83  | 85  | 87  | 89  | 91  | 93  | 94  |     |           |
| 7.20 - | 53 | 59   | 65  | 69  | 74  | 77  | 80  | 83  | 86  | 89  | 91  | 93  | 95  | 97  | 99  | 101 |     |           |
| 7.15 - | 57 | 63   | 69  | 74  | 78  | 82  | 86  | 89  | 92  | 94  | 97  | 99  | 101 | 103 | 105 | 107 |     |           |
| 7.10 - | 60 | 67   | 73  | 78  | 83  | 87  | 91  | 94  | 97  | 100 | 102 | 105 | 107 | 109 | 111 | 113 |     |           |

<sup>1</sup> Pure CaCO<sub>3</sub> x 1.5; for tillage depth of 8 inches.

## VII. Data Summary and Quality Control

Since laboratory results are collected automatically as an integral part of a computer-assisted data acquisition system, a unique opportunity is presented for using the computer to control the quality of analytical work.

In addition to the use of blank and check samples, which are randomly but regularly (every 20 to 40 samples) inserted into a run, table 5, to detect such problems as contamination, error in instrument calibration, and/or misplacement of soil samples, the CEC of each soil sample is calculated by two independent methods. One is based on the sum of extractable cations and buffer pH CEC+, Table 5; the other on soil-water pH and buffer pH CEC, Table 5 (9). For most Alabama soils, where Mehlich 1 is used as the extracting solution and the buffer pH is determined by the Adams-Evans method, the two estimated CECs should be practically identical. On the other hand, a discrepancy between them would mean one of these possibilities:

- (1) The inappropriateness of the testing procedures, e.g., the use of Mehlich 1 and buffer solutions in calcareous soils, and/or
- (2) The dissolution of unreacted lime giving high Ca readings, and/or
- (3) Errors in analytical measurements, especially soil pH, buffer pH, and extractable Ca.

The computer is programmed to pinpoint the discrepancy, when it exceeds a set tolerance limit (50%), and to print a warning such as an asterisk. Actions can be promptly taken to recheck, identify, and correct errors or to identify the reason for the discrepancy.

Upon completion of a "run" and all quality-control measures are satisfied, soil-test data and associated information (e.g., farmer number, run number) are sent through a MODEM (2400 baud) to an IBM mainframe computer for execution of the fertilizer recommendations program and print-out of recommendations to be returned to the farmer (7).

Table 5. Soil-test Data Summary

A.U. SOIL TEST LABORATORY DATA SUMMARY FOR RUN 104 PAGE 1 \*\* 12:19:27 \*\* 2/19/85 \*\*

| POSNO | MPOSNO | FARMER | LABNO | NR | PH   | LIME | P              | K   | Mg   | Ca   | NOTE     | CEC   | CEC+  | K-RATIO |
|-------|--------|--------|-------|----|------|------|----------------|-----|------|------|----------|-------|-------|---------|
|       |        |        |       |    |      |      | -----Lb/A----- |     |      |      | MEQ/100g |       | %     |         |
| 1     |        | 6983   | 34162 | 1  | 5.59 | 7.59 | 1              | 123 | 146  | 780  |          | 6.59  | 5.93  | 6.30    |
| 2     |        | 6983   | 34163 | 1  | 5.68 | 7.65 | 5              | 138 | 186  | 920  |          | 5.95  | 6.04  | 7.23    |
| 3     |        | 6983   | 34164 | 1  | 5.19 | 7.61 | 1              | 62  | 123  | 400  |          | 4.94  | 4.70  | 3.62    |
| 4     |        | 6983   | 34165 | 1  | 5.63 | 7.67 | 3              | 104 | 124  | 730  |          | 5.44  | 5.08  | 5.81    |
| 5     |        | 6984   | 34166 | 1  | 4.24 | 7.61 | 30             | 31  | 31   | 180  |          | 5.44  | 3.67  | 1.86    |
| 6     |        | 6984   | 34167 | 1  | 4.96 | 7.70 | 62             | 115 | 64   | 400  |          | 3.31  | 3.80  | 7.82    |
| 7     |        | 6984   | 34168 | 1  | 5.18 | 7.80 | 43             | 60  | 69   | 430  |          | 2.52  | 2.95  | 4.65    |
| 8     |        | 6984   | 34169 | 1  | 5.30 | 7.71 | 171            | 158 | 47   | 430  |          | 3.92  | 3.71  | 10.37   |
| 9     |        | 6985   | 34170 | 2  | 5.40 | 7.68 | 126            | 165 | 112  | 570  |          | 4.59  | 4.63  | 9.85    |
| 10    |        | 6985   | 34171 | 1  | 6.39 | 7.76 | 88             | 414 | 1101 | 4450 | *        | 6.72  | 18.13 | 15.06   |
| 11    |        | 6986   | 34172 | 1  | 6.10 | 7.75 | 270            | 116 | 266  | 1530 |          | 5.61  | 7.04  | 5.91    |
| 12    |        | 6987   | 34173 | 2  | 5.28 | 7.74 | 18             | 100 | 69   | 400  |          | 3.47  | 3.48  | 6.88    |
| 13    |        | 6987   | 34174 | 2  | 5.72 | 7.78 | 26             | 65  | 47   | 360  |          | 3.83  | 2.93  | 4.53    |
| 14    |        | 6988   | 34175 | 1  | 5.41 | 7.64 | 5              | 74  | 149  | 470  |          | 5.19  | 4.69  | 4.27    |
| 15    |        | 6989   | 34176 | 3  | 5.70 | 7.78 | 19             | 114 | 77   | 380  |          | 3.78  | 3.12  | 7.87    |
| 16    |        | 6990   | 34177 | 1  | 6.70 | 7.81 | 102            | 156 | 467  | 1430 |          | 7.06  | 7.16  | 7.50    |
| 17    |        | 6990   | 34178 | 1  | 6.95 | 7.83 | 104            | 196 | 443  | 1360 |          | 8.39  | 6.85  | 9.10    |
| 18    |        | 6990   | 34179 | 1  | 5.13 | 7.56 | 45             | 226 | 72   | 440  |          | 5.38  | 5.20  | 12.60   |
| 19    |        | 6991   | 34180 | 1  | 6.98 | 7.83 | 178            | 341 | 222  | 5290 |          | 8.72  | 15.91 | 12.46   |
| 20    |        | 6992   | 34181 | 1  | 5.25 | 7.63 | 121            | 116 | 51   | 460  |          | 4.85  | 4.41  | 6.91    |
| 21    |        | 6993   | 34182 | 1  | 5.77 | 7.77 | 61             | 164 | 84   | 870  |          | 4.14  | 4.50  | 10.12   |
| 22    |        | 6994   | 34183 | 1  | 5.40 | 7.59 | 54             | 112 | 46   | 880  |          | 5.88  | 5.81  | 5.94    |
| 23    |        | 6995   | 34184 | 1  | 5.52 | 7.57 | 71             | 239 | 262  | 1310 |          | 6.62  | 8.03  | 11.32   |
| 24    |        | 6995   | 34185 | 1  | 7.40 | 7.78 | 280            | 276 | 3460 | 6660 | *        | 24.30 | 5.82  | 9.12    |
| 25    |        | 6996   | 34186 | 1  | 6.44 | 7.85 | 91             | 80  | 31   | 980  |          | 4.38  | 3.82  | 5.07    |
| 26    |        | 6996   | 34187 | 1  | 6.42 | 7.74 | 61             | 50  | 17   | 1010 |          | 7.47  | 4.71  | 2.60    |
| 27    |        | 6996   | 34188 | 1  | 6.67 | 7.85 | 120            | 97  | 35   | 1090 |          | 5.41  | 4.16  | 5.69    |
| 28    |        | 6997   | 34189 | 1  | 5.97 | 7.86 | 92             | 76  | 64   | 340  |          | 2.87  | 2.27  | 6.08    |
| 29    |        | 6997   | 34190 | 1  | 5.82 | 7.80 | 50             | 47  | 141  | 660  |          | 3.71  | 3.84  | 3.10    |
| 30    |        | 6997   | 34191 | 1  | 5.70 | 7.81 | 31             | 91  | 54   | 310  |          | 3.27  | 2.55  | 6.84    |
| 31    |        | 6997   | 34192 | 1  | 5.36 | 7.76 | 96             | 35  | 32   | 280  |          | 3.36  | 2.79  | 2.56    |
| 32    |        | 6998   | 34193 | 1  | 5.48 | 7.64 | 297            | 108 | 65   | 950  |          | 5.41  | 5.58  | 5.91    |
| 33    |        | 6998   | 34194 | 1  | 6.14 | 7.99 | 0              | 1   | 6    | 30   | BL* 1    | 0.23  | 0.10  | 0.32    |
| 34    |        | 6998   | 34195 | 1  | 5.65 | 7.75 | 69             | 109 | 94   | 440  | CK 51    | 4.17  | 3.62  | 7.08    |
| 35    |        | 6999   | 34196 | 1  | 5.76 | 7.79 | 26             | 66  | 54   | 290  |          | 3.75  | 2.68  | 4.72    |
| 36    |        | 6999   | 34197 | 1  | 5.78 | 7.77 | 28             | 76  | 73   | 320  |          | 4.16  | 3.03  | 5.14    |
| 37    |        | 6999   | 34198 | 2  | 6.14 | 7.78 | 49             | 49  | 70   | 320  |          | 5.08  | 2.91  | 3.14    |
| 38    |        | 6999   | 34199 | 2  | 6.57 | 7.74 | 17             | 81  | 169  | 520  | *        | 8.52  | 4.18  | 4.12    |
| 39    |        | 7000   | 34200 | 1  | 4.59 | 7.44 | 152            | 165 | 83   | 560  |          | 4.81  | 6.43  | 8.92    |
| 40    |        | 7000   | 34201 | 1  | 6.46 | 7.63 | 77             | 66  | 290  | 5990 |          | 10.99 | 19.14 | 2.18    |

\*Indicates discrepancy in CEC calculations.

## VIII. Special Analyses

Upon request, several non-routine analyses can be performed. These determinations are classified as "special" because additional cost will be charged for their services and results are not kept by the computer.

1. Micro-nutrients (Cu, Fe, Mn, Zn, B, Mo) and toxic elements (Al, Ni, Cr, Pb).

These elements are determined with an Inductively-coupled argon Plasma Spectrometer (Jarrell-Ash, ICAP 9000).

### Principles of Operation

Inductively-coupled argon plasma spectroscopy is a "finely tuned" version of emission spectroscopy. In this technique, radio frequency (RF) radiation is used to heat a flow of argon gas into a plasma by means of an induction coil, figure 3. When a sample is introduced into this extremely hot (5,000 to 10,000°K) argon plasma, the sample is broken down into individual atoms, which are then further excited by the plasma. These excited atoms subsequently re-emit the excitation energy as electromagnetic radiation (light) which is characteristic of their respective chemical elements.

The emitted light is then passed into the spectrometer, which disperses the light across its focal curve, separating the various emission lines present in the radiation. At appropriate locations on the focal curve are positioned photomultiplier tubes, one for each analytical line desired on the system (total 16 in our ICAP). Light energy is converted into electrical current, which is integrated over a period of time (a few seconds), and then digitized so that data may be fed into a computer (Apple IIe) for calculations and storage. Analytical results can then be printed out on the computer terminal or displayed on a video screen.

The computer, besides collecting data and calculating results, also controls the overall functioning of the spectrometer, making the system reasonably "automatic" and fail-safe.

### ICAP Operating Parameters

|                        |   |
|------------------------|---|
| Incident RF power      | 1.1 KW  |
| Reflected RF power     | minimum ( 20W)                                |
| Nebulizer type         | cross flow                                    |
| Nebulizer pressure     | 30 p.s.i.                                     |
| Argon flow rates       |   |
| coolant                | 18 L/minute                                   |
| sample                 | 0.4 L/minute                                  |
| plasma (not required)  | 0.5 L/minute                                  |
| Sample aspiration rate | 1.5 mL/minute                                 |
| Observation height     | 18 mm above load coil                         |
| Profiling element      | Cu (channel #13 at center of the focal curve) |

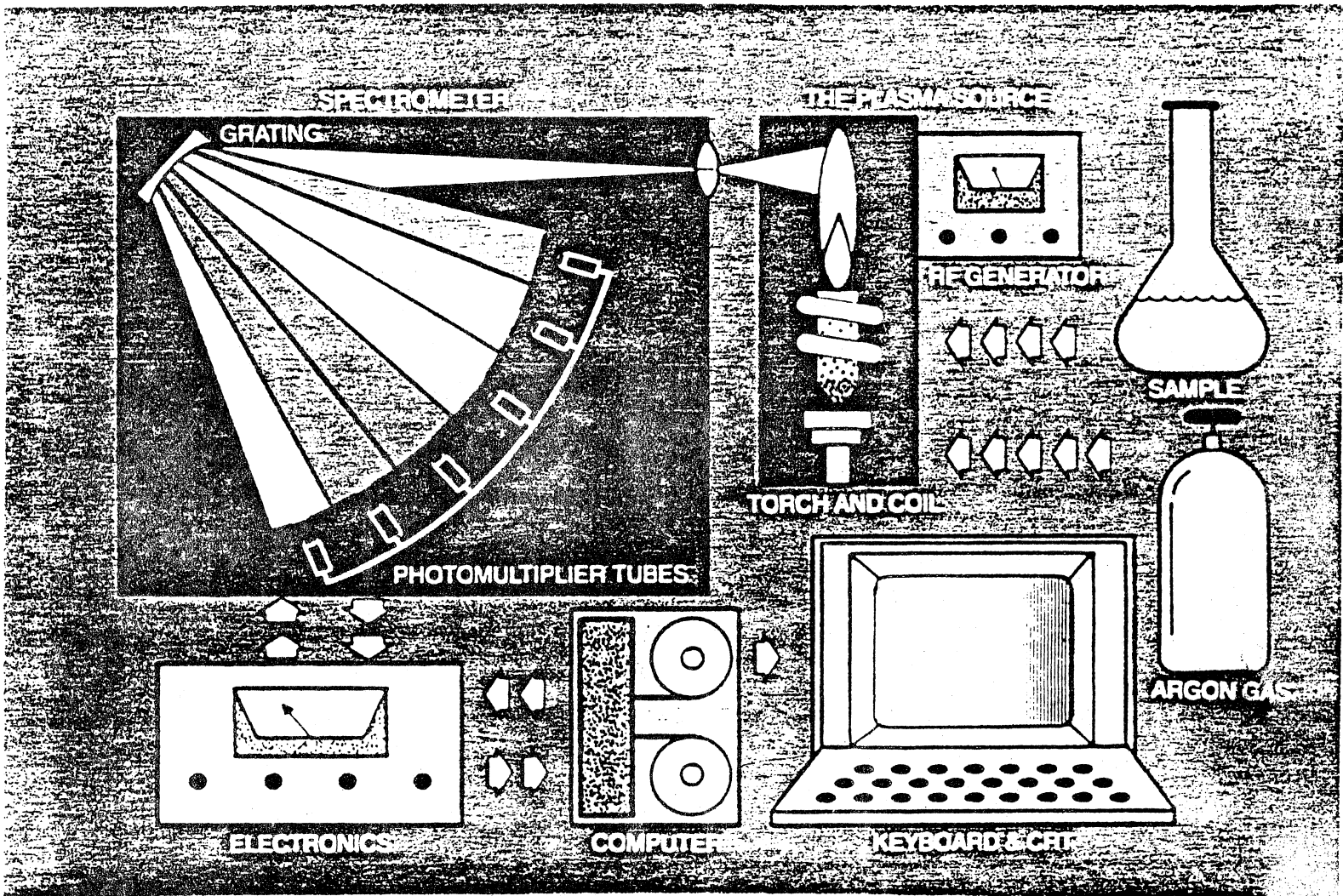


Fig. 3. Block diagram of an ICP.

## 2. Organic Matter Determination

Organic carbon is determined by a dry combustion method with a LECO WR-12 carbon analyzer. Then, organic matter is calculated by multiplying organic carbon by 1.90 (6).

### Principle of Operation

Soil sample is mixed with an electrically conductive material and burned under pure  $O_2$  atmosphere in an inductive furnace. Carbon dioxide produced is measured with a thermal conductivity cell, which consists of a Wheatstone bridge that responds to the heat difference carried by  $O_2$  and  $CO_2$  gas.

### Operating parameters

|                      |                     |
|----------------------|---------------------|
| $O_2$ pressure       | 35 p.s.i.           |
| $O_2$ flow rate      | 1.2 to 1.5 L/minute |
| burn time            | 70 seconds          |
| detector temperature | 50°C                |

### Procedure

- Weight 0.25 g of carbonate-free soil into a ceramic crucible.
- Add 1 scoop (@ 2 g) of iron chip (to make sample electrically conductive).
- Add 1 scoop of granular copper.
- Load the crucible into the inductive furnace.
- Press ANALYZE button on the C ANALYZER.

### NOTE:

- Digital readout on the display is based on 1 g sample. Therefore, if soil weight is 0.25 g, multiply the reading by 4 to get percent C in soil.
- If sample contains carbonates, a pre-treatment is required, as follows: slowly add 1 mL of 4 N  $H_2SO_4$  to 0.25 g of soil. The mixture is allowed to stand for 1 hour, then is placed in a forced-air oven at 80°C until completely dried. Organic carbon can then be determined on the treated sample.

### 3. Nitrate Determination

The phenoldisulfonic acid method is used for  $\text{NO}_3\text{-N}$  determination (5,2).

#### Soil Extraction

- a. Place 5 g of soil in a 50-mL Erlenmeyer flask
- b. Add 20 mL of deionized  $\text{H}_2\text{O}$
- c. Shake for 15 minutes, then filter.

#### Preparation of reagents

- a. Sodium hydroxide 1N. Dissolve 4 g of NaOH granules in 100 ml of deionized  $\text{H}_2\text{O}$ .
- b. Phenoldisulfonic acid. Dissolve 25 g of pure, white phenol in 150 mL of concentrated  $\text{H}_2\text{SO}_4$  in a 500-mL Erlenmeyer flask. Then, add 75 mL of fuming  $\text{H}_2\text{SO}_4$  (13 percent  $\text{SO}_3$ ). Mix the solution. Place the flask (loosely stoppered) in hot water ( $90^\circ\text{C}$ ) for 2 to 4 hours.
- c. 1:1  $\text{NH}_4\text{OH}$ . Mix equal parts of concentrated  $\text{NH}_4\text{OH}$  and  $\text{H}_2\text{O}$ .
- d.  $\text{NO}_3\text{-N}$  standards. Dissolve 7.216 g of oven-dried  $\text{KNO}_3$  in deionized  $\text{H}_2\text{O}$  and dilute to 1 L in a volumetric flask. This solution contains 1000 ppm  $\text{NO}_3\text{-N}$ . Prepare working standards of 0 to 10 ppm  $\text{NO}_3\text{-N}$  with proper dilution. Standard solutions can be kept for about 2 weeks. Low concentration  $\text{NO}_3\text{-N}$  standards (5 p.p.m.) go bad easily.

#### Procedure

- a. Aliquot 1 mL of soil extract into a 50-ml Pyrex beaker, add 1 drop of 1 N NaOH, evaporate to dryness on a hot plate. Cool beaker to room temperature.
- b. Add 1 mL of phenoldisulfonic acid, rotate the beaker to dissolve the residue. Allow the mixture to stand for 10 minutes for the reaction to complete.
- c. Add 5 mL of deionized  $\text{H}_2\text{O}$ . Let it cool (@ 5 minutes), add 6 mL of 1:1  $\text{NH}_4\text{OH}$  solution. Cool to room temperature.
- d. Read absorbance on a B & L spectrometer at 420 nm.
- e. Standards of 0, 1, 2, 5, and 10 p.p.m.  $\text{NO}_3\text{-N}$  should always be run along with samples.



#### 4. Sulfate Determination

An indirect method is used for  $\text{SO}_4^{2-}$  determination (8); in this method  $\text{SO}_4^{2-}$  is precipitated completely with an excess but known amount of  $\text{BaCl}_2$ . The unreacted Ba will then be measured with an AA spectrometer, using a  $\text{C}_2\text{H}_2/\text{N}_2\text{O}$  flame.

##### Soil Extraction

Calcium monophosphate is used to extract soil  $\text{SO}_4^{2-}$  (3).

- Place 5 g of soil in a 50-mL Erlenmeyer flask
- Add 20 mL of 0.01 M  $\text{Ca}(\text{H}_2\text{PO}_4)_2$
- Shake for 30 minutes. Then, allow to stand overnight.
- Filter

##### Preparation of Reagents

- $\text{Ca}(\text{H}_2\text{PO}_4)_2$  - 0.01 M. Pour slowly 1 g of  $\text{CaCO}_3$  into 40 mL of 0.5 M  $\text{H}_3\text{PO}_4$  (concentrated  $\text{H}_3\text{PO}_4 = 14.7$  M). Dilute to 1 L with  $\text{H}_2\text{O}$ .
- Stock 1,000 p.p.m. Ba solution. Dissolve 1.7786 g  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  in deionized water and dilute to 1 L.
- Working Ba solutions. Dilute 10 and 25 mL of the stock Ba solution to 100 mL with deionized water to give 100 and 250 p.p.m. Ba solutions.
- Stock 1,000 p.p.m.  $\text{SO}_4$  solution. Dissolve 1.814 g of  $\text{K}_2\text{SO}_4$  in deionized water and dilute to 1 L. Prepare working solutions of 1.0 to 40.0 p.p.m.  $\text{SO}_4$  by dilution of the stock solution.
- $\text{ClCH}_2\text{COOH}$ -KOH solution. Dissolve 47.25 g  $\text{ClCH}_2\text{COOH}$  (monochloroacetic acid) in deionized water; add 3.40 g KOH and dilute to 250 mL.
- $\text{BaSO}_4$  seeding suspension. Mix 0.1 g  $\text{BaSO}_4$  powder with 100 mL of deionized water. Shake well before use.

##### Procedure

- Aliquot 5 mL of soil extract into a 50-mL Erlenmeyer flask.
- Add 1.5 mL of 100 p.p.m. Ba (as  $\text{BaCl}_2$ )
- Add 1 mL of monochloroacetic acid-KOH mixture

- d. Add 0.1 mL of BaSO<sub>4</sub> seeding solution
- e. Add 7.5 mL of 95% Ethanol
- f. Stopper and shake for 15 minutes
- g. Transfer about half of the solution into 10-mL centrifuge tube and centrifuge samples (maximum 24 samples per batch) at 10,000 g for 30 minutes.
- h. Decant the supernatant and save it.
- i. Measure Ba in the supernatant with an AA
- j. Sulfate standards: 0 to 15 p.p.m. SO<sub>4</sub>-S should always accompany samples

## 5. Soluble Salts

Specific conductance (indicator of soluble salts) is determined in the supernatant of 1:2 soil:water slurry (13).

### Procedure

Measure 40 cm<sup>3</sup> of 2-mm sieved soil into a beaker, add 80 mL of water, stir thoroughly and allow suspension to settle for at least 30 minutes or long enough for the solids to settle.

Draw supernatant into the conductivity pipette to slightly above the constricted part of pipette. Avoid drawing liquid into rubber bulb. If this occurs, rinse bulb before continuing with the next sample.

### Calculations

Specific conductance (SC) of the soil extract is calculated as follows:

$$SC, \text{ mmhos per cm at } 25^{\circ}\text{C} = \frac{1.4118 \times C_{\text{ext}}}{C_{\text{std}}}$$

where the value of 1.4118 is the specific conductance of the standard 0.01 N KCl solution in mmho per cm at 25°C and C<sub>std</sub> and C<sub>ext</sub> refer to conductance in mhos of the standard (0.01 N KCl) solution and extract, respectively.

See tables 6 and 7 for interpretation of the results.

Table 6. Soluble Salt Reading in mmho/cm and Corresponding Approximate ppm of Salt in 1:2 Air-dried Soil (Volume to Volume) Water Extract and Salinity Effects

| Reading<br>mmho/cm* | Salt<br>p.p.m. | Salinity<br>effects  |
|---------------------|----------------|--|
| 0.40                | 512            | Salinity effects mostly negligible   |
| 0.40-0.80           | 512-1024       | <u>Very slightly saline.</u> Yield of crops of low salt tolerance may be reduced by 50% all classes of crops (see table 2).  |
| 0.81-1.60           | 1025-2048      | <u>Slightly saline.</u> Yields of fruit and vegetable crops of medium salt tolerance may be reduced by 50%. Similar yield reductions may occur in the more sensitive forage and field crops of medium salt tolerance. Lower half (0.81-1.20) of range satisfactory for well-drained mineral greenhouse soils. Upper half (1.21-1.60) of range higher than desirable for greenhouse soils except for peat and lightweight mixes.  |
| 1.61-2.40           | 2049-3072      | <u>Moderately saline.</u> Yields of virtually all fruit crops significantly reduced. Yield reductions of 50% may occur in the more sensitive forage and field crops of high salt tolerance. Similar yield reductions occur in the more highly salt-tolerant vegetable crops. For greenhouse crops ( 2.0) leach soil with enough water so that 2-4 quarts pass through each square foot of bench area of 1 pint of water per 6-inch pot; repeat after about 1 hour. Repeat again if readings are still in the high range. |
| 2.41-3.20           | 3073-4096      | <u>Strongly saline.</u> Only highly salt-tolerant forage and field crops will yield satisfactorily.  |
| 3.20                | 4096           | <u>Very strongly saline.</u> Only a few highly salt-tolerant grasses, herbaceous plants and certain shrubs and trees will grow.  |

\*mmho/cm x 1000 = mg salt/dm<sup>3</sup>

Table 7. Range of Salt Tolerance of Crops

| SC,<br>mmho/cm         | Rating | Salt tolerance   |
|------------------------|--------|--|
| <u>Forage Crops</u>    |        |  |
| 0.5-0.8                | Low*   | Ladino clover, burnet, red clover, alsike clover, meadow foxtail, white dutch clover   |
| 0.81-2.6               | Medium | Milkvetch, sourclover, tall meadow oatgrass, smooth brome, big trefoil, reed canary, meadow fescue, blue grama, orchardgrass, oats (hay), wheat (hay), rye (hay), alfalfa, hubam clover, sudan grass, dallis grass, strawberry clover, mountain brome, beardless wildrye, birdsfoot trefoil, perennial ryegrass, yellow sweetclover, white sweetclover |
| 2.61-3.6               | High   | Hardinggrass, barley (hay), tall fescue, crested wheatgrass, canada wildrye, tall wheatgrass, rescue grass, rhodes grass, bermuda grass, nuttall alkaligrass, saltgrass, alkali sacaton  |
| <u>Field Crops</u>     |        |  |
| 0.5-0.8                | Low    | Field peas, soybeans, field beans  |
| 0.81-2.6               | Medium | Castorbeans, sunflower, flax, broadbean lima, pinto, etc., corn (field), rice, sesbania, soybean, sorghum (grain), oats (grain), wheat (grain), rye (grain), safflower   |
| 2.61-3.6               | High   | Cotton, rape, sugarbeet, barley  |
| <u>Vegetable Crops</u> |        |  |
| 0.5-0.8                | Low    | Beans, celery, radish  |
| 0.81-1.6               | Medium | Cucumber, squash, pea, onion, carrot, lettuce, cauliflower, bell pepper, potato, sweet potato, sweet corn, cabbage, broccoli, tomato   |
| 1.61-2.4               | High   | Spinach, asparagus, kale, beets  |

\*No crop injury at low end of range to as much as 50% yield reduction at high end of range.

## 6. Particle Size Analysis

Purpose. A particle size analysis is made for determining the texture of a soil. It consists of obtaining the percentages of sand, silt, and clay in the soil.

Principle. When the mineral fragments of soil are suspended in water, they tend to sink. The rate at which they settle depends on the relative size of the particles. The largest particles settle first and the smallest particles settle last.

Two steps are necessary to obtaining satisfactory particle size analysis:

1. Thorough shaking of the sample in water with a chemical dispersing agent so that each particle is functioning as nearly separately as possible.
2. Placing the suspension of the sample in a suitable amount of water in preparation for unequal settling.

There are several methods for making particle size analysis of the soil. One of the most rapid is the Bouyoucos Hydrometer method (4). This method consists of placing a hydrometer, which is calibrated to read grams per liter, in the soil suspension in a special sedimentation cylinder at the desired time. Theoretically, the hydrometer measures the density of a suspension at a given depth.

### Procedure

- a. Place 40 g of soil in a special baffled metal cup. Fill the cup to within 4 cm of the rim with water. Add 50 mL of Calgon dispersing agent (a solution of 100 g Calgon per liter) to break up the soil aggregates.
- b. Stir with electric motor for 10 minutes. Then pour the soil and water suspension into a special settling cylinder, wash the coarse fraction from the cup with a stream of water.
- c. Bring the final volume to 1000 mL. Stopper the cylinder, and shake while in a horizontal position. Place upright on the bench and note the time immediately.
- d. Place the hydrometer in the suspension, and take a reading at 40 seconds from the time placed on bench.

This reading is: \_\_\_\_\_

- e. A second reading is taken at the settling time of 6.0 hours.

This reading is: \_\_\_\_\_

Explanation:

The first hydrometer reading represents the concentration of silt and clay (all sand having settled in 40 seconds), and the second reading the concentration of clay (silt having settled in 6 hours).

Calculations:

$$\% \text{ (silt + clay)} = (100/40) * (40\text{-sec reading})$$

$$\% \text{ (clay)} = (100/40) * (6\text{-h reading})$$

$$\% \text{ (silt)} = \% \text{ (silt + clay)} - \% \text{ (clay)}$$

$$\% \text{ (sand)} = 100 - \% \text{ (silt + clay)}$$

Textural Class Name: \_\_\_\_\_

See figure 4 for textural class name.

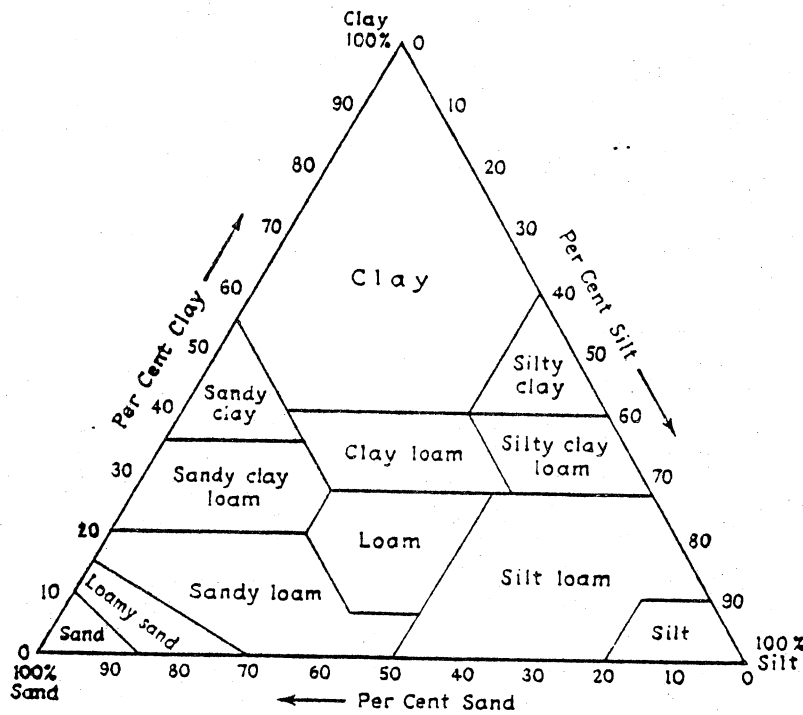


Fig. 4. Guide for textural classification.

## IX. Plant Analysis

The Soil Testing Laboratory offers a pecan leaf analysis service, which includes a routine soil test, and a leaf analysis for N, S, P, K, Ca, Mg, and Zn. The package costs \$11. Samples received for a pecan leaf analysis are numbered consecutively beginning with number 1 for the first sample of the calendar year (e.g., 85-1, 85-2, 85-3, etc.). Soil and leaf samples from the same orchard receive the same number.

"Trouble shooting" samples including several plant species and plant parts, which are often brought in by Extension specialists, are also analyzed by the laboratory similarly to pecan leaves plus Fe, Mn, Cu, and B.

### 1. Dry Ashing of Plant Samples

- a. Weigh 0.5 g of dry plant material into 50 mL pyrex beaker. Plant material should be ground to pass a 40-mesh (0.60 mm) stainless steel sieve.
- b. Cover with watch glass and place in muffle furnace. Heat to 450°C and hold at that temperature until all carbon is burned off (@ 4 hours). The ash should be greyish white.
- c. Add 10 mL of 1 N HNO<sub>3</sub>, and evaporate to dryness slowly on a hot plate. Take just to dryness and do not bake.
- d. Add 10 mL 1 N HCl to dissolve the residue.
- e. Warm nearly to boiling then transfer to a 100-mL volumetric flask. Wash beaker 3 times with small amounts of water.
- f. Bring to 100 mL, and filter. Do not wash filter paper or add more water in this step.

NOTE: 1 N HCl = 83 mL concentrated HCl per liter.  
1 N HNO<sub>3</sub> = 64 mL concentrated HNO<sub>3</sub> per liter.

- g. Use the ICAP to determine P, K, Ca, Mg, Mn, Cu, Fe, and Zn.
- h. If B is determined, Pyrex beaker must be replaced with a special ceramic crucible and step [c] is omitted.

### 2. Wet Ashing of Plant Samples

#### Reagents

Acid mix: 70 percent concentrated HNO<sub>3</sub> + 30 percent HClO<sub>4</sub>  
(60-72 percent)  
1 N HCl  
Whatman #40 filter paper  
Deionized H<sub>2</sub>O

## Apparatus

250-mL digestion tubes and rack  
100-mL volumetric flasks  
Steam bath in hood  
Block digester in hood  
Balance (to 0.01 g)

## Procedure

- a. Weigh 0.5 g of plant material into a 250-mL digestion tube.
- b. Add 10 mL of the Acid Mix and heat for 1 hour on the steam bath or let stand overnight.
- c. Transfer the tubes to the block digester and heat at 190°C until the material is digested (@ 2 hours). Add more Acid Mix as needed to keep the tubes from going dry.
- d. Remove the tubes from the block, cool down.
- e. Add 10 mL of 1 N HCl and bring to 100 mL with deionized H<sub>2</sub>O.
- f. Filter a portion of the solution through a Whatman No. 40 filter paper.
- g. Carry a blank through the procedure.
- h. Store the solutions in glass bottles.
- i. Use the ICAP to determine P, K, Ca, Mg, Mn, Cu, Fe, and Zn.

Note: Wet ashing procedure is not suitable for B determination because a portion of B would be lost as H<sub>3</sub>BO<sub>3</sub>.

## 3. Total N Determination.

A Leco CHN-600 is being used for total N determination. This instrument has an advantage over the "conventional" Kjeldahl method because the digestion of the sample is not required. The time it takes to obtain total N content of a solid sample is @ 5 minutes.

### Principle of Operation

The determination of N is made by burning a weighed quantity of sample (solid or liquid) in pure O<sub>2</sub> at @ 950°C; CO<sub>2</sub>, H<sub>2</sub>O vapor, oxides of nitrogen, N<sub>2</sub>, and oxides of sulfur are possible products of combustion. Oxides of sulfur are removed with CaO in the secondary combustion zone so that water vapor cannot combine to form H<sub>2</sub>SO<sub>4</sub>. The remaining gases of combustion are collected in a ballast and are allowed to mix



thoroughly. Then, 10-mL aliquot is taken for N determination. The aliquot is carried by He into a reagent train consisting of hot Cu for the removal of O<sub>2</sub> and the reduction of NO<sub>x</sub> to N<sub>2</sub>, NaOH for the removal of CO<sub>2</sub>, and Mg(ClO<sub>4</sub>)<sub>2</sub> for the removal of H<sub>2</sub>O. The remaining elemental N<sub>2</sub> is measured by the thermal conductivity cell. This detector cell has the ability to detect the difference in the thermal conductivity of gases. The cell consists of two pairs of matched filaments used in four legs of a Wheatstone bridge. The "reference" filaments are maintained in a constant gas (pure He), pressure, flow, and temperature environment, while the "measure" filament is maintained in a constant pressure, flow, and temperature environment, but the gas type is allowed to vary (a mixture of He and N<sub>2</sub>).

#### Operating Parameters

|                                  |  |
|----------------------------------|--|
| O <sub>2</sub> profile           | 3, 4, 1  |
| O <sub>2</sub> gas back pressure | 6 - 10 psi   |
| Combustion pressure              | 3 - 8 psi  |
| He pressure                      | 12 psi   |
| N <sub>2</sub> Blank             | <0.2 percent (ultra pure O <sub>2</sub> gas - 99.99) |

#### Procedure

- Press the GAS ON key on the computer keyboard to turn on the He gas.
- Weigh out a known quantity of plant tissue (0.07 to 0.15 g) into a tin capsule.
- Crimp end of the capsule to keep sample from falling out.
- Enter sample weight either automatically or manually by the keyboard.
- Place sample on the automatic sampling disc.
- Press ANALYZE to start.

#### Note:

- Halt the operation immediately if WARNING light comes on.
- Try to determine the possible cause; if the problem is found and solved, press RESET key to resume the operation.
- Press GAS ON key again to shut He off.

#### 4. Total S Determination.

A LECO IR-32 sulfur analyzer is used to determine total S in plants and soils.

##### Principle of Operation

Solid sample is mixed with an electrically conductive material (Fe powder) and with catalysts (W and  $V_2O_5$ ). Then, the mixture is burned under pure  $O_2$  atmosphere in an inductive furnace to produce  $SO_2$ ,  $CO_2$ , and  $H_2O$ . Water vapor is then eliminated with  $Mg(ClO_4)_2$ ,  $SO_2$  in the remaining gaseous mixture is detected with an IR detector. Because  $CO_2$  gas absorbs at some of the same infrared wavelengths as  $SO_2$ , a  $CO_2$  filter is needed. This  $CO_2$  filter is a sealed chamber containing  $CO_2$  gas, which completely absorbs energy at the  $CO_2$  wavelengths. Between the  $CO_2$  filter and the shutter mechanism are two chambers, one is called the reference cell, the other is called the measure cell. During calibration, both cells are filled with pure  $O_2$ . However, when  $SO_2$  from the sample enters the measure cell, the IR radiation in that portion of the system is partially absorbed by the sample  $SO_2$  gas. The difference in IR energy between two cells is then converted electronically into percent S in the sample.

##### Operating Parameters

|                  |                                       |
|------------------|---------------------------------------|
| $O_2$ pressure   | 20 p.s.i.                             |
| Integration time | 60 seconds                            |
| Standard         | cotton leaf containing 0.60 percent S |

##### Procedure

- Weigh 0.100 g of plant material or 0.250 g of soil into a ceramic crucible.
- Add 1 scoop (2 g) of iron powder, 1 scoop of tungsten fine granules, and 1 disk of vanadium pentoxide ( $V_2O_5$ ), in this order.
- Place a lid on the crucible, and then place the crucible into the inductive furnace.
- Press ANALYZE button on the S analyzer.

##### Note:

- Always standardize the machine with comparable standards before running samples.
- The reading is displayed as percent of sample on a 1-g basis. Therefore, if only 0.1-g sample is used, the result (percent) is 10 times the reading.

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