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Soil Test Procedures for

Calcareous Rockland Soils of the Bahamas

The Bahamas

Recommended Soil Test Procedures for Bahamian Calcareous Rockland Soils

Introduction:

Standard chemical test procedures have neither been tested nor developed for the Bahamian calcareous Rockland soils. The chemical nature and fertility characteristics of these soils are little understood and therefore the shotgun approach to fertilization has been adopted. However, as the agriculture sector develops and grows, there will be increasing demands for soil testing to increase soil productivity and reduce fertilizer costs. Soil tests performed in other countries may employ inappropriate methods that would lead to misleading interpretation of results. Therefore, the future development and testing of already existent soil test procedures for Bahamian soils should be one of the goals of soil research in the Bahamas.

The authors have attempted to test and modify already existing soil test procedures deemed appropriate for Bahamian soils and to present these procedures as the recommended methods until further research provides ones that are even more appropriate. It should be understood, however, that sound fertilizer recommendation cannot be based on soil test values alone. The correlation between field fertility experiments and soil test values is essential before adequate recommendations can be made. Low minor elements (iron, manganese, zinc, copper, boron) availability would be forecasted for Bahamian calcareous soils. Consequently, there is a need for the testing of already existing procedures or the development of new ones for minor elements in Bahamian soils. However, procedures for extraction of available minor elements are not presented in this bulletin.

It should be noted that calcareous rockland soils which covers the entire Bahamian Archipelago, developed from oolitic limestone parent materials. This soil is of the type found in Homestead Florida which has agricultural significance in that region of Florida. It is recommended that procedures to extract and measure minor elements in Bahamian soils be conducted in the future.

Soil pH

One of the most enlightening attributes of a soil is its pH. Soil pH measurements attempt to assess the degree of acidity the plant root is subject to from the soil solution. Whether a soil is acidic, neutral, or basic has much to do with the availability of various plant nutrients and the activity of various microorganisms. Three soil pH ranges are particularly informative: a pH less than 4 indicates the presence of free acids generally from oxidation of sulfides; a pH below 5.5 suggests the likely occurrence of exchangeable aluminum, and a pH range from 7.8 -8.2 indicates the presence of calcium carbonate.

Since most of the plant essential elements in a soil reach maximal or near maximal availability in the pH range 6-7, and decrease both above and below this range, the soil pH is an indication of relative availability of plant nutrients. Thus, it seems fair to say that soil pH is both a symptom of the soil's condition and a cause of many of the reactions that occur.

Explanation:

The pH measurement in a calcium chloride solution first proposed by Schofield and Taylor (1955) provides a means of estimating the activity of hydrogen ions $[H^+]$ at a constant ionic strength for soils. Later, Peech (1965) recommended that pH measurement in soils be made by equilibrating 10g of soil with 20 mL of 0.01M calcium chloride ($CaCl_2$) solution and then inserting the calomel electrode into the supernatant solution and the glass electrode into the partially settled suspension. The results should then be reported as "soil pH measured in 0.01M calcium chloride." Junction potential apparently is not eliminated in suspensions of soils in 0.01M $CaCl_2$. Therefore, Peech's method of placing the calomel electrode in the supernatant solution serves as a safety precaution. The advantage in inserting the glass electrode in the suspension is that the suspension is more strongly buffered than the supernatant solution, and hence will be less influenced than the supernatant solution by minor disturbances, such as carryover from one sample to the next.

Peech (1965) pointed out that the pH of a soil measured in 0.01M $CaCl_2$ solution is independent of dilution over a wide range of soil: solution ratios. The proportion of the total electrolyte contributed by a non-saline soil to a suspension of one part of soil in two parts of 0.01M calcium chloride is relatively small, so that the observed pH is a better index of base saturation than is the pH of the soil measured in water, and perhaps more important. Peech (1965) noted that the electrolyte concentration of the soil solution in non-saline soils at optimum water content for plant growth is similar to that in 0.01M $CaCl_2$. The pH values obtained in 0.01M $CaCl_2$ according to this method are lower than those obtained in water, under similar conditions. Some researchers found an average difference of about 0.5 pH units.

Soil pH: Procedure for Soil pH Measurement

Apparatus:

pH meter and electrodes (*Combination electrode or a calomel and glass electrodes*); Buffer solutions (pH 4 and 7), Analytical balance, Volumetric flask (1L); Beakers (100 mL), Glass rods, Sieve (2mm)

Reagents:

0.01M Calcium chloride (CaCl$_2$) solution:
Dissolve 1.1g of CaCl$_2$ in 500 mL of distilled water and make up to volume in a volumetric flask (1L).

Method:

1. Standardize the pH meter according to the manufacturer's instructions using pH 4 and 7 buffer solutions.
2. Weigh 20g of air-dried soil that has been passed through a 2mm sieve into a beaker.
3. Add 40 mL of the 0.01M CaCl$_2$ solution, stir with a glass rod and let stand for at least 30 min.
4. Re-stir, and immediately lower the electrode(s) in to the soil suspension (note: If using a glass electrode, the glass electrode should be placed in the setting soil suspension and the calomel electrode in the supernatant solution above to reduce interference. If using a combination electrode, place the electrode in the partially settled suspension).
5. **Record the pH after the pH meter reading is stabilized.**

Sample Data:

Soil pH measurements were made in water and in 0.01M CaCl$_2$ on soil samples collected from the BARTAD—Andros and Nassau.

Table 1: Soil pH measured in water and 0.01M CaCl$_2$

Sample Site	pH in water	pH in 0.01M CaCl$_2$
Field 3-12A, Andros	7.90	7.50
Farm (No. 4), Andros	7.92	7.51
Camperdown, Nassau	7.95	7.4
Lyford Cay, Nassau	7.99	7.3

We would recommend that for a more realistic assessment of pH in the root zone, the soil pH measurements for Bahamian soils be made in 0.01M CaCl$_2$.

Soil Organic Matter

Soil organic matter includes plant and animal residues, microorganism tissue (both living and dead) and a very complex group of strictly soil compounds known as "humus." By far the largest portion of soil organic matter is made up of these humic substances; recently added plant residues and microorganism tissue make up only a small part of the total. Soil organic matter has a major influence on soil aggregation, moisture retention, cation exchange capacity, nutrient reserve and its availability, and biological activity.

The amount of organic matter in the soil varies widely. Some soils may be practically devoid of organic carbon, while others may be 80% organic matter. Most agricultural soils are in a group somewhere between these two extremes. The average organic matter content of the soils of the U.S. is 2.06% in the surface and 0.83% in the subsoil.

Soil Organic Matter: Procedure for Measurement of Soil Organic Matter

Soil Organic Matter: Determination using Loss-On-Ignition Method

Apparatus:
Muffle Furnace, Oven, Analytical Balance, Crucibles

Method:
1. Weigh the empty crucible and record the weight.
2. Add about 2 g of air-dried and sieved soil to the crucible.
3. Place the crucible in an oven at 105°C for 4 h.
4. Remove the crucible from the oven and place them in a dessicator to cool to room temperature. Weigh accurately and record the weight.
5. Place the crucible in a muffle furnace at 360°C and heat for 2 h.
6. Turn off the furnace after 2 h and leave the crucible in the furnace until temperature of the furnace comes down to 200°C.
7. Remove the crucible from the furnace using tong and place them in a dessicator. Allow sample to cool to room temperature in a dessicator.

7. Remove the crucible from the furnace using tong and place them in a dessicator. Allow sample to cool to room temperature in a dessicator.
8. Weigh crucible and record the weight.

$$\% \, OM = \frac{Wt. \, of \, Soil \, at \, 105 \, °C - Wt. \, of \, Soil \, at \, 360 \, °C}{Wt \, of \, Soil \, at \, 105 \, °C} \times 100 \qquad Eq. \, 1$$

Sample Data:

Table 2. Soil organic matter content (%)

Soil (Source)	Organic Matter (%)
Camperdown, Nassau	19
Lyford Cay, Nassau	11

The Average organic matter content of soils in the U.S. is 2.06% in the surface, and 0.83% in the subsoil. However, the two soils in the above table have relatively high percentage of organic matter. It has been noted that CEC measurements of Bahamian surface soils usually yield values which are high. The generally high CEC values of the Bahamian calcareous rock land soil may be due to high organic matter content. Soils with high organic matter content generally have high CEC and the capacity to hold large amounts of nutrients cations (positively charged nutrients).

More studies need to be done on the organic matter content of Bahamian soils.

Cation Exchange Capacity

Cation exchange capacity (CEC) is a measure of the quantity of readily exchangeable cations neutralizing negative charge in the soil and is usually expressed in milliequivalents (meq) per 100 g of soil or centimol of charge per kilogram soil ($cmol_c$ kg^{-1}). Both units are numerically equal (1 meq/100g = $cmol_c$ kg^{-1}). It is an important chemical property of soils, which provides information on the storage capacity of soils to hold cations, essential for plant growth.   Clay and organic matter have a large number of negative charges on their surfaces, thus they attract cations and contribute to higher CEC.

Cation Exchange Capacity: Procedure for Measurement of Cation Exchange Capacity by Direct Method

Apparatus:

Atomic Absorption Spectrometer, Inductively Coupled Plasma-Optical Emissions Spectrometer, Rotary shaker, Centrifuge machine, Volumetric flask

Method:

1. Weigh 2 gram of air-dried and sieved soil and transfer into a 50 mL plastic tube.
2. Add 20 mL of 1N sodium acetate solution (pH 8.9) and shake for 5 min on a rotary shaker. Centrifuge the sample at 3200 rpm for 10 minutes and discard the supernatant. Repeat the process three more times.
3. Add 20 mL of 70% ethanol to the sample and shake the sample on a rotary shaker at a slow speed for 2 min. Centrifuge the sample at 3200 rpm for 10 minutes and discard the supernatant. Repeat the process two more times.
4. Add 20 mL of 1N ammonium acetate (pH 8.9) to the sample and shake the sample on a rotary shaker for 5 min. Centrifuge the sample at 3200 rpm for 10 minutes and collect the supernatant in a 200 mL volumetric flask.
5. Repeat the process three more times.

6. Make up the final volume to 200 mL with 1N ammonium acetate
7. Analyze the sample for sodium using AAS or ICP-OES.

$$CEC\ (meq/100g) = \frac{concentration\ of\ Na\ (mgL^{-1}) \times 200}{2 \times 230} \qquad Eq.\ 2$$

Sample Data

Table 3. Comparison of CEC determined by summation and direct methods

Soil Source	CEC by Summation Method (meq/100g)	CEC by Direct Method (meq/100g)
Camperdown, Nassau	51.47	52.33
Lyford Cay, Nassau	43.27	42.75

The CEC values shown in the table above are considered high for agricultural soils

Calcium Carbonate in Soils

Carbonates occur naturally in many soils either as calcium (calcite) or magnesium (dolomite) carbonate or mixture of both. Soils that contain free calcium carbonate and have pH values in the range of 7.3 to 8.5 are known as calcareous soils. Most soils of arid and semi-arid regions are calcareous because of relatively little leaching. The presence of $CaCO_3$ in soils directly or indirectly affects the chemistry and availability of several plant nutrients including phosphorus, magnesium, potassium, manganese, zinc, copper, iron, and boron. Calcareous soils can be used to grow any crop, if managed properly. The knowledge of carbonate amount in the soil is necessary to develop management practices.

Calcium Carbonate: Procedure for Measurement of Calcium Carbonate in Soils

Apparatus:

Analytical balance, Erlenmeyer flask, burette

Method:

1. Weigh 1 g of air-dried and sieved soil in a 150 mL of Erlenmeyer flask. Add 10 mL of 1 N HCl to the flask.
2. Swirl the flask and leave it overnight.
3. Add 100 mL of deionized water and shake it thoroughly.
4. Add 4 drops of phenolphthalein indicator to the flask.
5. Titrate with 1 N NaOH solution while swirling the flask. Continue the titration until a faint pink color persists and note the volume of NaOH consumed.

$$\% \, CaCO_3 = \frac{[(10 \times N \, of \, HCL - (R \times N \, of \, NaOH)] \times 0.05}{Wt \, of \, Soil} \times 100 \qquad Eq. \, 3$$

Sample Data:

Table 4: Calcium carbonate concentration in selected soil samples

Soil Source	CaCO₃ (%)
Camperdown, Nassau	22.88
Lyford Cay, Nassau	0.67

Soluble Salt Content of Soil and Water

The growing plant derives its nutrients directly from the air and indirectly from the soil, through the medium of the soil solution. Soluble salts will increase the salt content of the soil solution until it may become harmful to plant growth. In nature, soils in arid regions and soils which are subjected to a rising water table may be saline (have excess salts). Under artificial conditions, many green house soils may become saline, due to use of large quantities of fertilizers. Pure water is a very poor conductor of electric current, whereas, water containing the dissolved salts ordinarily found in soils conducts current approximately in proportion to the amount of salt present. Based on this fact, the measurement of the electrical conductivity of an extract gives a satisfactorily accurate indication of the total concentration of soluble salts. Since many of the saline soils occur in arid regions, irrigation water quality is usually considered with soluble salt analysis. Although many analyses may be made on irrigation water, one of the most useful measurements is the electrical conductivity. The salinity hazard of the irrigation water may be estimated from this measurement.

> **EC = 1/r**
> EC= Electrical conductivity, r= Electrical resistivity (ohm cm)
> Basic unit of conductivity is Siemen (S) and Siemen = 1/ohm = mho
> ***EC =1/ohm cm or S per cm***
> ***1 μS per cm = 0.001 mS per cm = 0.000001 S per cm***
> The electrical conductivity is measured with a conductivity bridge (solu-bridge)

Most soil extracts have conductivity much less than one unit. For instance, measurement of one sample of soil extract gave EC that equals 0.000694 S per cm. For similar cases, with physical and chemical measurements, it is customary to choose a small subunit that gives a more convenient location of the decimal point when recording or expressing the data. For example, the unit EC x 10^3 is called the _millisiemen per centimeter (mS per cm)_. This is a convenient and practical conductivity unit for most soil salinity work. Until recently, EC x 10^5 (or K x 10^5) was in common use. EC x 10^6 denotes conductivity expressed in _μS per centimeter._ This is the unit most generally used for expressing the conductivity of irrigation waters.

The conductivity of the soil extract (0.000694 S per cm) mentioned above, when expressed in these several units is:

$$EC=0.000694 \text{ Siemen(S) per cm}$$
$$EC \text{ x } 10^3 = 0.694 \text{ mS per cm}$$
$$EC \text{ x } 10^5 = 69.4 \text{ μS per cm}$$
$$EC \text{ x } 10^6 = 694 \text{ μS per cm}$$

The electrical conductivity of aqueous salt solutions increases with increase in temperature, the increase per centigrade degree being very close to 2%. The standard temperature for reporting electrical conductivity is 25^0C.

Soluble Salts: Procedure for Measurement of Soluble Salts in Soil and Water

Apparatus & Reagents:

Conductivity Meter; Standard conductivity solutions for Calibration; Beaker (500 mL); Balance; Buchner funnel and Filter flask; Water aspirator; Sieve (2mm); Balance

Method:

a. *Measurement of Salt content in soil*:

Prepare a saturated paste of soil extract as follows:

1. Weigh 250g of air-dried and sieved soil to a 500 mL beaker.
2. Add distilled deionized water to the soil, while stirring with a spatula. Consolidate the mixture from time-to-time during the mixing process, by tapping the container on the workbench. At saturation, the soil paste glistens as it reflects light, flows slightly when the container is tipped, and slides freely and clearly off a spatula for all soils except those of high clay content.
3. After the mixing has been completed, cover the container, and allow the sample to stand for an hour or more; then recheck the criteria for saturation. If the paste has stiffened markedly or lost its glisten, add more water, and mix it again. Alternatively, if free water has collected on the surface of the paste, add an additional weighed quantity of dry soil, and mix again (The moisture contained at saturation can be determined by oven-drying a weighed subsample of the saturated soil paste).
4. Filter with a Buchner funnel under suction until approximately 30mL have been obtained.
5. Calibrate the conductivity meter according to the manufacturer's instruction.
6. Determine the electrical conductivity of the 30mL or more extract from the saturated paste.

b. *Measurement of Salt content of irrigation water*:

1. If the irrigation water has a high quantity of particulate matter, it can be filtered through a filter paper.
2. The electrical conductivity can then be determined directly using a calibrated conductivity meter.

Example: Here are conductivity measurements made on a soil sample taken from one of the fields on Satellite Farm No. 4 where a potato experiment is located as well as two irrigation wells on the research station.

Table 5: Conductivity measurements on some soil and irrigation water samples from BARTAD, Andros

Sample (Source)	EC (mS per cm) at 25 ^{0}C	Salt concentration (mg/L)
Soil (Farm No 4)	1.10	704
Water (Field 3-12A Eastern Well)	0.92	589
Water (Field 3-11A Eastern Well)	0.50	320

This soil should not have any salinity effect on plants. The water from both wells would be good for irrigation.

General Information on plant responses to soil electrical conductivity and quality rating of irrigation water, based on electrical conductivity measurements

Table 6: Response of plants associated with different ranges of electrical conductivity of saturation extracts of soils

Electrical Conductivity of soil saturation extract (mS per cm) at 25 ^{0}C	Plant response
0-2	Salinity effects usually negligible
2-4	Yield of very salt-sensitive crops may be restricted
4-8	Yield of salt-sensitive crops restricted
8-16	Only salt-tolerant crops yield satisfactorily
> 16	Only a very few salt-tolerant crops yield satisfactorily

Note: Most fruits are salt intolerant. Most vegetables have medium salinity tolerance. Field beans are very intolerant to salts.

Table 7: Quality rating of irrigation water from electrical conductivity measurements

Conductivity (EC x10^6)	Conductivity (EC x10^3)	Salinity Hazard
100-250	0.1-0.25	**Low**
250-750	0.25-0.75	**Moderate**
750-2250	0.75-2.25	**Medium-High**
2250-4000	2.25-4.00	**High**
4000-6000	4-6	**Very High**

Note: *The following relations are frequently employed:*

Salt concentration (mg/L) = 640 x EC (mS per cm)
Total cation concentration (meq/L) = 10 x EC (mS per cm)
Osmotic Pressure (atm) = 0.36 x EC (mS per cm)

Soil Phosphorus

Phosphorus (P) is classed as one of the nutrients required in the largest quantity, but its content in plants is considerably less than that of nitrogen, potassium and calcium. As a limiting factor; however, phosphorus is more important than calcium and probably more important than potassium. Phosphorus forms sparingly soluble compounds with cations in the soil such as Ca^{2+}, Al^{3+}, and Fe^{3+}. Because of this, the amount of P in soil solution at any one time is very small. Plants growing in the soil apparently absorb P only from the soil solution, and simple calculations show that in order for normal plant growth and P uptake to occur, the soil solution must be renewed several times each day, during the growing season. The limiting soil factor in P uptake by plants is likely to be renewal of the soil solution near the plant roots by processes of dissolution and diffusion. The factors involved in renewal of the soil solution are the amount of P that can be solubilized, its degree of solubility, and the rate of its diffusion from the solid surface to the plant root surface.

An extractant for soil P can be designed to estimate the first two of these factors, P supply and solubility. Although even these are measured grossly and empirically, diffusion rate is not measured at all. Therefore, even with the better extractants of soil P, correlation of the numbers obtained with other soil measurements and plant uptake and yield is necessary in order to make the soil test procedure usable over a wide range of soils.

Labile P is the portion of the total soil P that is relatively loosely bound onto or associated with soil minerals or amorphous materials. Usually, it is a small percent of the total P and is related to the quantity of P that can be utilized by crops growing on the soil. Ideally, for an extractant to provide a best index for the quantity of fertilizer P that is required for some range of soil-crop-climate combinations, it should do the following:

1. Dissolve and/or desorb some reproducible and consistent proportions of the labile P.
2. Reflect the extent and nature of reaction between the soils and any fertilizer P that may be added.

The $NaHCO_3$ extraction is generally recommended for calcareous soils, since it appears to be more effective than H_2SO_4-HCl and NH_4F-HCl for soils having high degree of base saturation and moderate-to-high amounts of calcium-P and free $CaCO_3$.

Phosphorus is extracted from the soil with 0.5M $NaHCO_3$ at a nearly constant pH of 8.5 (Olsen et al., 1954). In calcareous, alkaline or neutral soils containing calcium phosphates, this extractant decreases the concentration of calcium in solution by causing precipitation of calcium as $CaCO_3$; as a result, the concentration of P in solution increases. In acid soils, containing aluminum or iron phosphates, such as variscite and strengite, P concentration in solution increases as the pH rises. Secondary precipitation reactions in acid and calcareous soils are reduced to a minimum, because the concentration of aluminum, calcium, and iron remains at a low level in the extractant.

Phosphorus Extraction and Analysis Procedure

Apparatus:

UV/VIS Spectrophotometer, spectrophotometer cuvettes Orbital shaker, Analytical Balance, pipettes (micro and macro, as required)

Chemicals and Reagents:

1. Extracting solution -0.5 M Sodium bicarbonate ($NaHCO_3$) solution: Dissolve 42.0g of $NaHCO_3$ in distilled deionized water (500mL) in a 1L volumetric flask, and bring to volume with distilled deionized water. Adjust the pH of the solution to 8.5 with 1M NaOH or 0.5N HCl. Store the solution in a polyethylene container for periods longer than 1 month; check the pH of the solution each month. Prepare a fresh solution, if the solution has been standing over 1 month in a glass container.
2. Reagents for phosphorus analysis:
a. ***Reagent A***- Dissolve 12.0g of ammonium molybdate [$(NH_4)_6Mo_7O_{24}.4H_2O$] in 250 mL of distilled water. Separately, dissolve 0.2908g of antimony potassium tartrate in 100 mL of distilled deionized water. Add both dissolved reagents to 1000 mL of 5N H_2SO_4 (148 mL of concentrated H_2SO_4/L), mix thoroughly and make up to 2L in a volumetric flask using distilled deionized water. Store in a Pyrex glass bottle in a dark and cool compartment.
b. ***Reagent B***- Dissolve 1.056g of ascorbic acid in 200 mL of reagent A prepared above, and mix. This reagent should be prepared as required, as it does not keep for more than 24 hours.
3. Carbon black (free of phosphorus)
4. 1% p-nitrophenol solution
5. Phosphorus standard (1000 mg/L P)

Extraction Method:

Add 5g of soil and 40 mL of 0.5M $NaHCO_3$ extracting solution to 125 mL Erlenmeyer flasks. Shake the flasks in an orbital shaker for 30 min. Filter the suspension through a Whatman # 42 filter paper. Add 1g of carbon black (phosphate free), shake immediately before pouring the suspension into the funnel, if the filtrate is dark colored.

Phosphorus analysis:

The amount of phosphorus in soil extracts is measured using the Murphy and Riley (1962) colorimetric method. This method is based on reaction of orthophosphate ions in the extracts with ammonium molybdate and antimony potassium tartrate reagents to form a complex at acidic pH. Subsequently, the complex formed is reduced with ascorbic acid to form a blue complex. Absorption of light by the blue complex is measured at 880 nm wavelength. The absorbance is proportional to the concentration of orthophosphate in the sample.

Phosphorus Extraction and Analysis Procedure

Preparation of P standards:

1. Prepare a 100 mg/L P solution by diluting a 1000 mg/L P standard. Pipette 100, 200, 300, 400, and 500 □L of 100 mg/L P standard into 50 mL volumetric flasks to prepare a series of P standards (0.2-1.0 mg/L).
2. Add 8 mL of Reagent B to each flask, and make up to volume with distilled water. Cover the flask and invert to mix, wait 5 min to develop the blue color.
3. Pipette sufficient amount of standard solution into a spectrophotometer cuvette, place the cuvette in the spectrophotometer cuvette holder and read the absorbance at 880 nm.
4. Read the absorbance for each standard using spectrophotometer wavelength set at 880 nm. The spectrophotometer should be zeroed against a blank containing the extraction reagent prior to taking absorbance measurements of standards.
5. Calibration curve: Plot Absorbance *vs.* concentration of standard.

Measurement of P in standards and soil extracts:

Prior to P determination in $NaHCO_3$ extracts, the extracts should be acidified to pH 5 using 5N H_2SO_4. Use p-nitrophenol as an indicator for pH adjustment. Add a drop of indicator to the extract and add 5N H_2SO_4 drop wise until the solution turns from yellow to clear. Now the pH of extract should be approximately 5. Use this pH adjusted extract for P measurement.

1. Pipette 1-10 mL of aliquots from the acidified (pH 5) extracts into 50 mL volumetric flasks (*it may be necessary to use all of the filtrate if the concentration of P is low in the extracting solution or to dilute the filtrate if the concentration is too high*).
2. Add distilled de-ionized water to make the volume to 40 mL (if necessary), and then add 8 mL of reagent B.
3. Make the volume up to 50 mL using distilled de-ionized water and mix. Prepare a blank with extractant solution and 8 mL of reagent B.
4. Allow 10 minutes for development of blue color.
5. Zero the spectrophotometer using the blank. Pipette sufficient amount of extract into a spectrophotometer cuvette, place the cuvette in spectrophotometer cuvette holder and read the absorbance at 880 nm.
6. Use the P calibration curve to calculate amount of P in each soil extract. To calculate final concentration of P in soil extracts include the dilution factor in the calculations.
7. Express soil P as mg of P per kg of soil.

Phosphorus Extraction and Analysis Procedure

Example:

Here are extractable P measurements in a soil taken from one of the research plots at BARTAD research station. Two methods were used to extract P from the soil: the first is the Bray P1 method (NH_4F-HCl method) and the second is the 0.5M $NaHCO_3$ extraction method.

Table 8: Phosphorus in soils from BARTAD Research Station

Soil Sample Source	Extraction Method	P Concentration in soil[a] (mg/kg)
Field 3-12A, Andros	Bray P1	4.72
Field 3-12A, Andros	0.5M $NaHCO_3$ (pH 8.5)	68.40
Camperdown, Nassau	0.5M $NaHCO_3$ (pH.8.5)	9.7
Lyford Cay, Nassau	0.5 M $NaHCO_3$ (pH 8.5)	9.5

[a] Concentration of P in the extracts was measured by the Murphy and Riley Ascorbic Acid method (Murphy and Riley, 1962).

Interpretation:

The results of this extraction and of others (not presented here) indicate that the $NaHCO_3$ method extracts more P than the Bray P1 method (Bray and Kurtz, 1945) in Bahamian Rockland calcareous soil. Previous studies show that the isotope method of measuring labile P correlates best with plant available P in calcareous soils (Dalal and Hallsworth, 1977). The $NaHCO_3$ method is also excellent in assessing the P fertility as related to plant growth potential on these types of soils. Therefore, 0.5 M $NaHCO_3$ (pH 8.5) should be used instead of Bray P1 in testing for P in Bahamian calcareous soils.

Calcium, Magnesium, Potassium and Sodium in Calcareous Soils

Calcareous soils present serious difficulties because calcium carbonate is partially soluble even in the more neutral extractants. Bower et al. (1952) discuss both calcareous and saline soils in this respect and advocated a high pH sodium acetate extractant. Tobia and Milad (1956) discussed exchangeable calcium in particular and reviewed several methods. They recommended dilute hydrochloric acid brought into equilibrium with solid calcium carbonate. The simplest approach seems to be to raise the pH of the 1M ammonium acetate extractant from pH 7-9 by addition of ammonia. The amount of extractable Ca obtained is quite arbitrary though it includes only a minimum of carbonate-Ca. There is probably little point in separating "true" exchangeable calcium and carbonate-calcium dissolved by mild extraction since the latter is part of the soil nutrient potential. Magnesium is also affected in a similar way, although to a lesser extent, in the extraction of soils containing magnesium carbonate.

This extractant is suitable for the extraction of sodium, potassium, calcium, and magnesium in calcareous soils.

Extraction method using Ammonium Acetate (pH 9)

Apparatus:

Atomic Absorption or Inductively Coupled Plasma Spectrometer; pH meter; Orbital shaker; Erlenmeyer flasks (125 mL); Analytical Balance; Filter funnel and # 42 Whatman filter paper; Sieve (2mm)

Reagents:

Extracting reagent- 1M Ammonium acetate (pH 9):

Dilute glacial acetic acid (99.5%, 114 mL) with water to a volume of approximately 1L. Then add concentrated ammonium hydroxide (139 mL), and add de-ionized water to obtain a volume of about 1980 mL. Mix well and check the pH of the resulting solution, and add more ammonium hydroxide as needed to obtain a solution pH of 9. Dilute the solution with deionized water to a final volume of 2L using deionized water in a volumetric flask.

Method:

Mineral soils:

1. Weigh 10g of air-dried and sieved soils into a 500 mL Erlenmeyer flask. Add 250 mL of ammonium acetate and shake for 1h on a rotary shaker. Filter through a Whatman #42 filter paper in to borosilicate or polyethylene bottles and reject the first 20-30 mL. Run two blanks with extractant only. (Note: The moisture content should be determined at the time of weighing in order to express the results in a dry-weight basis.)
2. Measure Ca, Mg, K, and Na by atomic absorption or inductively coupled plasma (ICP) spectrometer.

Calcium, Magnesium, Potassium, and Sodium in Calcareous Soils

The low, medium, and high ranges for exchangeable bases in Bahamian soils have not been established to date. However, the ranges used for the Baker's soil test for Pennsylvania soils, which is supposed to work for even calcium saturated soils, may be used as a guide. The ranges are given in **Table 1.**

Table 9. Guide for Ca, Mg, K, and Na ranges (based on Baker's soil test for Pennsylvania soils)

Range	---------------meq/100g[a]--------------				
	Ca	Mg	K	Na	CEC[b]
Low	3.0-5.0	0.2-0.5	0.1-0.2	0-0.22	4.0-7.0
Normal	5.0-10.0	0.5-2.0	0.2-0.7	0.22-0.65	7.0-15.0
High	10.0-15.0	2.0-4.0	0.7-1.0	0.65-1.08	15.0-20.0

[a]meq- Milliequivalent

[b]Cation exchange capacity (CEC) is a measure of the soil total capacity to hold positively charged nutrients. In Bahamian soils due to the slightly alkaline condition, it is assumed that exchangeable hydrogen in negligible. Therefore, CEC = $\sum$ (Ca, Mg, Na, K) in meq/100g

Example:

Table 10. Ca, Mg, K, and Na in soils from one of the research plots at the BARC Research Station

Field 3-12A	CEC (meq/100g)	---------------meq/100g[a]--------------			
		Ca	Mg	K	Na
	16.8	14.8	1.25	0.3	0.52
Rating	High	High	Normal	Normal	Normal

[a] It should be noted that 1 meq/100g = 1 $cmol_{(+)}$/kg

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APPENDIX 1 - Soil Analysis Data

Some soil analysis data obtained for soils in Nassau, Bahamas

Mineralogical properties of these soils were determined by X-ray Diffraction (XRD) Spectroscopy.

Table A1: Mineralogy of some soils from Nassau, Bahamas

Soil Source	Mineralogy
Boyd's Sub Division	Calcite, Aragonite, Quartz Chlorite[a], Kaolinite, Bohemite[b]
Chippingham area (Red Soil)[c]	Quartz, Calcite, Aragonite Chlorite[a], Kaolinite, Bohemite[b]
Buttonwood area	Aragonite, Calcite, Quartz Kaolinite
Chippingham area	Calcite, Aragonite, Quartz Chlorite[a], Kaolinite, Bohemite[b]

[a]Chlorite may contain vermiculite layers (XRD data)

[b] Further characterization is required to confirm presence of bohemite

[c]May contain iron oxides or hydroxides

Table A2: Soil pH

Soil (Source)	In 0.01 M CaCl$_2$
Boyd's Sub Division	7.3
Chippingham area (Red Soil)	7.3
Buttonwood area	7.3
Chippingham area	7.2

Table A3: Sodium bicarbonate (pH 8.5) extractable P

| Soil (Source)	P (mg/kg)
Boyd's Sub Division	74.6
Chippingham area (Red Soil)	56.8
Buttonwood area	63.4
Chippingham area	48.1

APPENDIX 1 - Soil Analysis Data

Table A4: Amount of Na, Mg, Ca, and K in selected soils (Ammonium acetate extraction, pH 9)

Soil Source	Na (meq/100g)	Mg (meq/100g)	Ca (meq/100g)	K (meq/100g)
Boyd's Sub Division	0.59	1.59	14.5	0.85
Chippingham area (Red Soil)	0.59	0.54	25.7	2.21
Buttonwood area	0.55	1.64	18.1	1.23
Chippingham area	0.50	6.41	59.7	1.59
Camperdown	0.24	4.40	46.52	0.31
Lyford Cay	0.46	4.17	37.39	1.25

XRD Spectrum of Clay Fraction from Boyd's Sub Division Soils
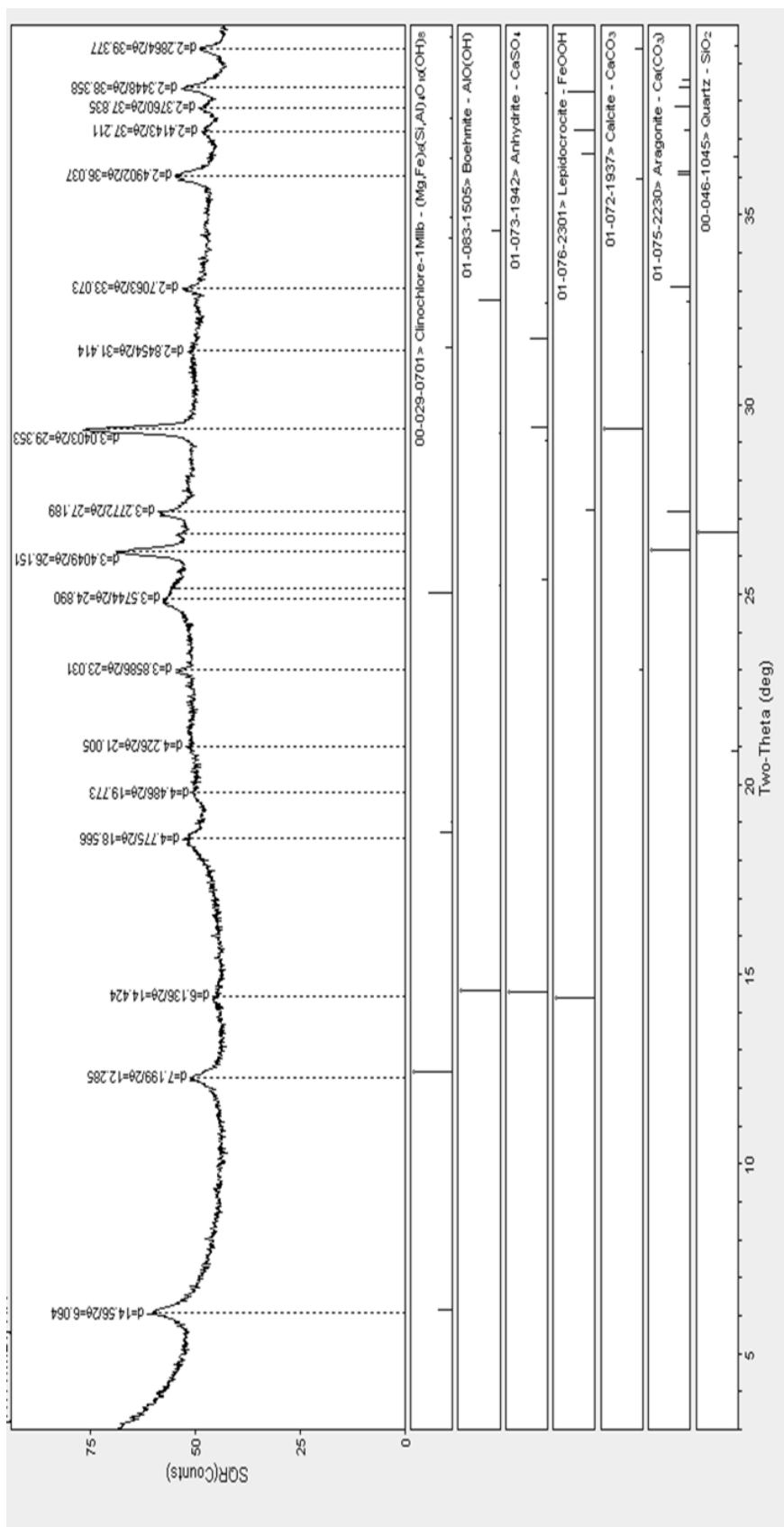
XRD Spectrum of Clay Fraction from Chippingham Soils

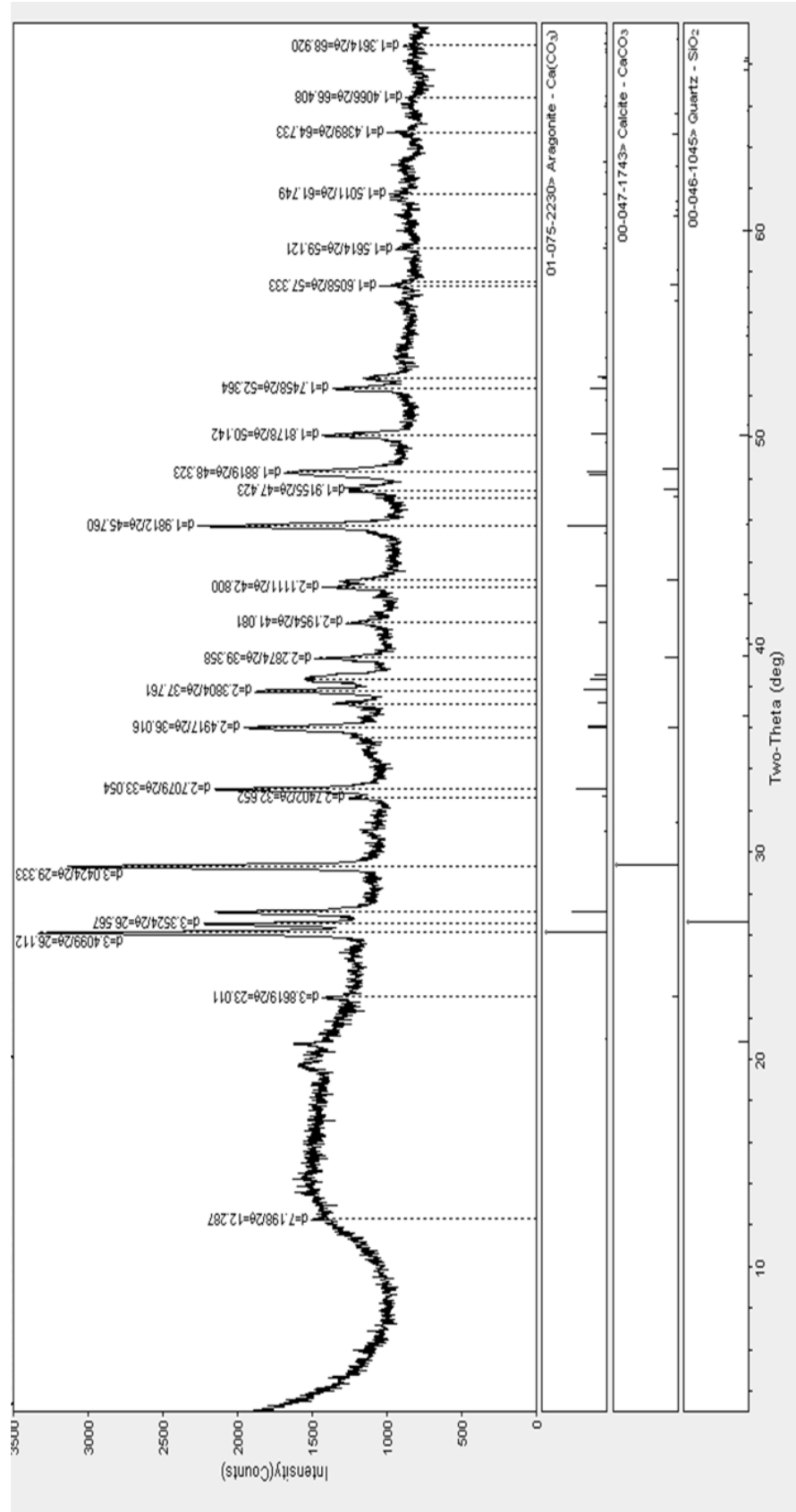# XRD Spectrum of Clay Fraction from Chippingham Area (Red Soil)

XRD Spectrum of Clay Fraction from Chippingham Area (Red Soil)

XRD Spectrum of Soils from Buttonwood Area

XRD Spectrum of Soils from Buttonwood Area

Intensity(Counts) vs Two-Theta (deg)

01-075-2230> Aragonite - Ca(CO₃)
00-047-1743> Calcite - CaCO₃
00-046-1045> Quartz - SiO₂

Peak labels:
d=7.198/2θ=12.287
d=3.8619/2θ=23.011
d=3.4099/2θ=26.112
d=3.3524/2θ=26.567
d=3.0424/2θ=29.333
d=2.7079/2θ=33.054
d=2.7402/2θ=32.652
d=2.4917/2θ=36.016
d=2.3804/2θ=37.761
d=2.2874/2θ=39.358
d=2.1954/2θ=41.081
d=2.1111/2θ=42.800
d=1.9812/2θ=45.760
d=1.9155/2θ=47.423
d=1.8819/2θ=48.323
d=1.8178/2θ=50.142
d=1.7458/2θ=52.364
d=1.6058/2θ=57.333
d=1.5614/2θ=59.121
d=1.5011/2θ=61.749
d=1.4389/2θ=64.733
d=1.4066/2θ=66.408
d=1.3614/2θ=68.920

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